CITRUS COUNTY
CENTRAL SANITARY LANDFILL EXPANSION
Citrus County, Florida

Batch PACT® Leachate Treatment System

OPERATION MANUAL

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Operations Manual

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LANDFILL
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SECTION NO. 2
PROCESS DESCRIPTION

1.0 Narrative

The Citrus County Central Sanitary Landfill Leachate Treatment System consists of three (3) Model B140 Batch PACT® structures, one (1) Sludge Storage/Decant Equalization Structure, and three (3) decant pumps. Each of the two first stage Model B140 structures includes aeration tank, diffusers, aeration blower, electric decant winch, waste sludge pump, polymer feed system, level sensors, and control panel. The second stage Model B140 structure includes these items plus three turbine agitators. The sludge storage/decant equalization structure includes sludge storage tank, diffusers, aerated sludge blower, manual decant winch, sludge pump, chemical feed system, decant equalization tank, filter feed pumps, level sensors, and control panel.

The Engineering Flow Diagrams, found in Section 1, depict the process flow scheme for the batch PACT® Leachate Treatment System.

The PACT® Leachate Treatment System is a biological treatment and physical adsorption process combined into a single unit process. Powdered activated carbon is added to the conventional activated sludge process to adsorb non-biodegradable compounds and provide improved removal of biodegradable materials.

The Citrus County Central Sanitary Landfill Batch PACT® system is designed to treat leachate generated at the site. The leachate is sequentially pumped to one of two first stage aeration tanks where the required nutrient (phosphorous) and carbon are added to maintain optimum treatment. When the aeration tank is filled with leachate, treatment proceeds in the following sequence:

Aerobic Treatment - One positive displacement blower provides air to the coarse bubble diffusers for dispersion throughout the aeration tank. In this step, carbonaceous BOD is removed and nitrification takes place via biological assimilation. Also, nondegradable, adsorbable material is adsorbed on the powdered carbon.

Clarification - The aeration tank also acts as a clarifier. After aerobic treatment, the aeration blower is restarted and the polymer pump provides polymer from a 100 gallon tank. The polymer is added to the aeration tank and allowed to mix for 10-15 minutes. The blower is then shut off and the entire tank is allowed to settle.

Decantation - After a settling time of approximately 30-60 minutes, the clear liquid is decanted off the top of the tank and pumped to the anoxic tank.

Sludge Removal - Solids concentrations are controlled in the aeration tank by periodic wasting of the sludge to the aerated sludge storage tank via the sludge wasting pump. Sludge is normally wasted from the aeration tank after decanting. Carbon must be added to the aeration tanks to replace carbon removed from the tank during sludge wasting. Carbon from the anoxic tank is normally wasted to the two aeration tanks to provide make-up carbon to the tanks or virgin carbon can be added directly to the aeration tank daily via 50 pound water soluble bags.

A second stage tank accepts decant from the two first stage tanks sequentially and virgin carbon is added to maintain optimum treatment.

The anoxic tank is filled with decant from the two first stage aeration tanks.

Anoxic Treatment - Decant from either of the two first stage aeration tanks is pumped to the second stage anoxic tank. During this fill step, methanol is also added to provide a carbon source for denitrification reaction. Agitation is provided by three vertical turbine mixers in the anoxic tank. Methanol feed and mixing continues until the predetermined amount of methanol is fed and denitrification is complete.

Re-aeration - The methanol pump and mixers stop and the aeration blower starts to re-aerate the contents of the anoxic tank.

Clarification - The aeration tank also acts as a clarifier. After anoxic treatment and before re-aeration is complete, the polymer pump provides polymer from a 100 gallon tank. The

polymer is added to the anoxic tank and allowed to mix for 10-15 minutes. The blower is then shut off and the entire tank is allowed to settle.

Decantation - After a settling time of 30-60 minutes, the clear liquid is decanted off the top of the tank and pumped to the decant equalization tank.

Sludge Removal - Solids concentrations are controlled in the anoxic tank by periodic wasting of the sludge to the two first stage aeration tanks via the sludge wasting pump. Sludge is normally wasted following decanting. Carbon must be added to the anoxic tank to replace carbon removed during sludge wasting. Virgin carbon is added to the anoxic tank directly via 50 pound water soluble bags.

2.0 Flow To The Aeration Tank

Leachate:

Three pumping stations supply leachate through force mains to the two batch PACT® aeration tanks for treatment.

Plant Drains:

One plant drain pump station supplies wastewater through force mains to the batch PACT® aeration tanks for treatment.

Carbon Addition:

Make-up powdered activated carbon is added to the aeration tank using 50 pound water soluble bags as needed. The 50 pound bags are placed directly in the aeration tank after first removing the outer paper bag. Aeration blower or mixer should be operating to provide mixing.

Nutrient Addition:

Nutrient (phosphorous) requirements are provided by adding phosphoric acid as needed via the chemical feed system.

3.0 Aerobic/Anoxic Treatment

Aerobic Reaction (Carbonaceous Oxidation and Nitrification):

In the aeration tank, the wastewater will aerate in the presence of powdered activated carbon, bio-mass and non-volatile material (ash) to remove BOD and COD and convert ammonia to nitrate (nitrification).

The bacteria in the aeration tank remove the organic compounds that are measured by the BOD analysis by consuming it as food. This is called metabolism. Metabolism consists of two separate on-going processes; respiration and synthesis. The desired bacteria are aerobic. That is, they require free oxygen dissolved in the mixed liquor for respiration. In the respiration process the bacteria consume organic compounds and oxygen dissolved in the wastewater and convert (oxidize) it to carbon dioxide and water. The bacteria also reproduce, using a portion of the food for synthesis or new cell growth. The required oxygen is made available to the bacteria by bubbling air into the water (mixed liquor) in the aeration tank. Some of the oxygen is dissolved in the water and in this dissolved form becomes available to the bacteria. The available oxygen is measured as D.O. (dissolved oxygen) and should be at least 2 PPM to maintain adequate biological activity and promote the predomination of the proper species of aerobic bacteria. Low D.O. levels in the two aeration tanks (less than 1.0) as well as other factors, promote the reproduction of filamentous, poorly settling bacteria.

The oxygen requirement for biological uptake is provided by one positive displacement aeration blower. The blower will provide compressed air through coarse bubble diffusers located throughout the aeration tank.

Anoxic Reaction (Denitrification):

In the anoxic step, the wastewater in the aeration tank is subjected to anoxic (no oxygen) conditions. The aeration blower is shut off and a 10 HP submerged mixer provides the required mixing of the wastewater. Under these conditions, nitrate in the wastewater is removed by converting nitrate to nitrogen gas (denitrification).

Sludge Wasting:

It is in the aeration tanks and the anoxic tank that the substrate in the wastewater is consumed as food by the biomass or absorbed on the carbon. The substrate represents food, and air is added to supply oxygen needed for respiration. In the case of the anoxic tank, oxygen is supplied to the organisms by the nitrate ion rather than by aeration. The micro-organisms will multiply (increase in population) as long as there is flow (substrate) to the aeration tank, oxygen and the appropriate environmental conditions such as temperature, pH, etc., exist. The solids resident time (SRT) is determined by the mass of solids removed from the aeration tank each day. The SRT must be controlled at some desirable value. In this case 15 days is the target SRT so the biological solids retain good settling characteristics. This means that every day one-fifteenth of the solids in the aeration tank must be removed from the system.

The required solids removal is accomplished by pumping sludge to the aerated sludge storage tank. The biomass fraction will be replaced by new cell growth and the carbon fraction will be replaced by adding carbon from the anoxic tank or virgin carbon.

Clarification:

In the Batch PACT® system the aeration and anoxic tanks also act as clarifiers. This is accomplished by shutting off the aeration blower and/or mixers after polymer addition and allowing the solids to settle. The treated effluent from the system is decanted from the top of the aeration tank and pumped to the decant equalization tank via the decant pump.

4.0 Polymer Addition

Polymer is fed to the aeration tank using a constant speed gear pump for injection of a dilute polymer solution from a mixed tank.

The pump flow is constant at 7 gpm, therefore polymer addition is controlled by regulating the amount of time that the pump is operating. The polymer injection pump can be operated either manually or automatically. In the automatic mode the polymer pump run time is set in register C03 of the controller located in the batch PACT® control panel.

A 100 gallon tank is supplied to hold a minimum of one day's usage of polymer solution at a polymer concentration of ½%. The polymer dose required is estimated to be 2 mg/l which represents approximately 3 minutes of polymer injection pump operation per cycle.

5.0 Carbon Addition

The addition of make-up powdered activated carbon (PAC) is done intermittently (once per day for example) to replace PAC losses and PAC wasted from the system. Make-up carbon is added directly to the anoxic tank from 50 pound water soluble bags. Makeup carbon to the aeration tanks should be first from solids wasted from the anoxic tank, and if additional carbon is needed to meet treatment goals, by virgin carbon.

Carbon addition is manually carried out each day with the aeration blower and/or mixers operating to insure good mixing. A dust mask should be used when working with the powdered activated carbon.

6.0 Nutrient Addition

A nutrient solution of phosphoric acid supplies the required phosphorous to the aeration tank. The chemical feed system is required to supply the nutrient concentration required to maintain optimum biomass growth. Note that nutrient nitrogen is available in the leachate influent as ammonia (NH_3).

Calculation of the amount of nutrient required is detailed in Section 3.

7.0 Aerated Sludge Storage Tank

The aerated sludge storage tank is used to hold wasted mixed liquor solids under aerobic conditions until they can be pumped to sludge drying beds. Diffusers are used to aerate and mix the contents of the aerated sludge holding tank and may decrease the volatile solids content by up to one third. To maximize the solids storage capacity of the holding tank a decant pipe is used to decant off clear supernatant. Decanting is accomplished manually. In the decanting cycle, the aerated sludge blower is shut off and the tank contents are then allowed to settle. If the tank level is sufficient to warrant decanting, the swing-pipe suction line is positioned so the open end is just above the sludge blanket level, the decant discharge

valve (No. 34) is opened, and the tank is decanted to the plant drain pump station. When the supernatant level falls to near the sludge blanket or when solids are carried out of the tank, the decant discharge valve (No. 34) is closed and the aerated sludge blower is restarted.

8.0 Decant Equalization Tank

The decant equalization tank is used to store the treated leachate from the anoxic tank. The leachate is then pumped at a steady flow rate to a tertiary filter (supplied by others) by one of two filter feed pumps. After filtration, the treated leachate is discharged to the landfill percolation pond site.

9.0 Equipment List

Aeration Tank	Capacity Dimensions HDT (Nominal) Quantity	45,000 gallons 12'W x 12'H x 48'L 108 hours (4.5 days) 2
Decant Equalization Tank	Capacity Dimensions HDT (Nominal) Quantity	29,000 gallons 12'W x 12'H x 31'L 23 hours (0.97 days) 1
Aerated Sludge Storage Tank	Capacity Dimensions Quantity	11,200 gallons 12'W x 12'H x 12'L 1
Aeration Blowers	Capacity Discharge Pressure Motor Quantity	1000 SCFM 6.0 PSI 40 HP / 460 V 3
Aerated Sludge Blower	Capacity Discharge Pressure Motor Quantity	60 SCFM 6.0 PSI 5 HP / 460 B 1
Electric Decant Winches	Capacity Motor Quantity	700 lbs 1/2 HP / 115 V 3
Mixers	Capacity Motor Quantity	12,500 GPM 3 HP / 460 V 3

Waste Sludge Pumps	Capacity Motor Quantity	70 GPM @ 30' TDH 1 1/2 HP / 460 V 3
Decant Pumps	Capacity Motor Quantity	200 GPM @ 12' TDH 2.1 HP / 460 V 3
Filter Feed Pumps	Capacity Motor Quantity	30 GPM @ 23' TDH 1/2 HP / 460 V 2
Sludge Pumps	Capacity Motor Quantity	180 GPM @ 12' TDH 2.1 HP / 460 V 1
Polymer Systems	Quantity Pump Tank Mixer	3 7 GPM / 1 HP / 460 V 100 gallons 1/3 HP/ 115 V
Chemical Feed System	Quantity Pump Tank Mixer	1 11 GPH/ 1/3 HP / 460 V 100 gallons 1/3 HP / 115 V
Control Panels	Quantity Size Features	4 (3) @ 48"H x 37"W x 12"D (1) @ 36"H x 31"W x 8"D (3) w/Modicon PC-0085 Programmable Controllers
Methanol Tank	Capacity Quatity	11,900 gallons 1
Methanol Pump	Capacity Motor Quantity	5.5 GPH 1/4 HP 1

SECTION NO. 3
PROCESS CONTROL

1.0 Narrative

In this section the control of solids in the aeration tanks is discussed. The reader should recognize that solids must also be controlled in the anoxic tank. Solids are wasted from the anoxic tank to the two aeration tanks. Solids are wasted from the aeration tank to the aerated sludge storage tank. Virgin carbon is added to the anoxic tank. The aeration tanks receive carbon from the anoxic tank when solids are wasted, and may be supplemented by virgin carbon.

A. A control scheme - whether for activated sludge or PACT® - should maintain a balance between sorption and stabilization. Such schemes will also be based on sludge wasting (and addition of virgin carbon in the required amounts).

B. While PAC affects both sorption and stabilization, its major contribution is to the sorptive capacity of the system. PAC concentration or dosage levels will thus be primarily determined by the pollutant removals required, which may be measured by effluent color or COD, for example.

C. Control of the PACT® system thus involves two different but related elements: Addition of virgin PAC; and wasting of excess Mixed Liquor Suspended Solids (MLSS) to solids disposal.

In PACT® systems where the PAC is not regenerated and returned to the system, sufficient virgin powdered activated carbon (PAC) must be added either continuously or incrementally to replace whatever PAC has been intentionally wasted or lost in the effluent. This amount is termed the Carbon Dose and is defined as:

$$\text{Carbon Dose (mg/l)} = \frac{\text{(MLVCS mg/l) HDT}}{\text{SRT}}$$

Where MLVCS = Mixed liquor volatile carbon solids, mg/l

HDT = Hydraulic detention time, days

SRT = Solids residence time, days

HDT may be further defined as the time required for a unit volume influent to the aeration tank to pass through the tank, expressed in days:

$$\text{HDT, days} = \frac{\text{Va (volume of aeration tank, gallons}}{\text{Q (rate of flow into tank, gallons/day)}}$$

For this example: Va = 45,000 gallons

Q = 15,000 gallons/day

HDT = 4.5 days

SRT is defined as:

$$\text{SRT, days} = \frac{\text{Total solids in PACT® system, lbs.}}{\text{Waste rate, lb./day}} = \frac{\text{MLSS}}{\text{WCS}}$$

The weight of total solids is calculated as follows:

MLSS lbs. = Va (volume of aeration tank, MG) x 8.34 x MLSS, mg/l

(8.34 is the well known factor which converts concentrations in mg/l and flows in MGD into lbs/day.)

Solids wasting is directly from the aeration tank. The waste rate is calculated as follows (neglecting solids lost to the effluent):

WCS, Waste Rate; lb. solids to be wasted per day =

$$\frac{\text{MLSS, mg/l x 8.34 x Va, MG}}{\text{SRT (desired)}}$$

Since the aeration tank is well mixed, there is a direct relationship between solids and volume in the mixed liquor.

If wasting is to be accomplished directly from the aeration tank:

$$\text{W, waste flow rate; GPD} = \frac{\text{Va, Gal.}}{\text{SRT}}$$

NOTE: If decant effluent suspended solids are significant, they must be considered or over wasting will result and the actual SRT will be lower than desired. In this case:

$$WCS = \frac{MLSS \times 8.34 \times Va}{SRT} - Q, MGD \times 8.34 \times Effl.\ SS, mg/l$$

Wasting is accomplished by pumping solids to the aerated sludge storage tank. This requires that the thickened carbon solids concentration (TCS) be known. So:

$$W, GPD = \frac{WCS, lb/day \times 10^6}{8.34 \times TCS, mg/l}$$

Wasting can also be done directly from the aeration tank during aeration. In that case, the MLSS, mg/l concentration is substituted for TCS in the above equation.

Once the sludge waste pump flow rate is known, the time required for wasting can be calculated.

$$\frac{W\ GPD}{70\ GPM\ (for\ example)} = \frac{Min.}{Day}\ Total\ time\ solids\ must\ be\ pumped$$

Volatile carbon must be added each day to replace carbon wasted and that lost in the effluent.

Carbon in the mixed liquor is measured as carbon by the PAC/BIO test procedure.

$$Carbon\ Wasted, = W, GPD \times MLVCS \times 8.34 \times 10^{-6}$$

Carbon lost in the effluent is estimated as follows:

$$Lb/day = Q, MGD \times 8.34 \times Eff.\ SS, mg/l \times \frac{MLVCS, mg/l}{MLSS, mg/l}$$

Make-up volatile carbon must replace carbon lost to these two areas.

Powdered activated carbon can vary in inert (ash) content from 5% to 35%. Therefore;

$$PAC\ added = \frac{Lbs.\ volatile\ carbon\ added/day}{1 - \frac{\%\ ash}{100}}$$

Once the appropriate "initial charge" of PAC has been added to the system, only makeup carbon need be added. Makeup is necessary to compensate for effluent PAC losses and the amount of spent PAC wasted from the system.

Wasting of sludge from the system is done to maintain a biological Sludge Age which will permit growth of desired flora, such as nitrifying bacteria, including Nitrobacter but not so great as to provide conditions suitable for the excess growth of scum-foam producing organisms such as Nocardia. In this respect - control by solids wasting - PACT® is exactly the same as activated sludge, as one would expect based on the inherently biological nature of PACT®. Sludge Age is defined as the age, in days, which results when the system total solids mass is divided by the daily rate at which solids are removed from the system. This parameter is thus seen to be the same as Solids Residence Time (SRT) or Mean Cell Residence Time (MCRT) often seen in wastewater treatment reference literature. The initial goal for this application will be a Sludge Age of 15 days. This figure may change as more information and operating data become available, and as loadings to the plant change. Seasonal changes in the Sludge Age or SRT may also be required to achieve the effluent quality desired.

D. In addition to the control of the system as just discussed, the following operational parameters should be monitored closely:

1. **Dissolved Oxygen (D.O.)** - It is recommended that D.O. levels be kept at a minimum of 2 mg/l at all sample locations in the aeration tank, and should not be allowed to drop to less than 1.0 mg/l for any reason. Levels below 1.0 mg/l may result in poor effluent quality which may persist well beyond restoration of proper D.O. levels. In the anoxic tank, the D.O. level should normally be near zero, except during the reaeration step, just prior to decanting when the D.O. is elevated to 4-6 mg/l.

2. **Polymer Dosage** - It will be necessary to determine an approximate range of polymer dosage based on bench-scale testing (which can and should be done on-site by prospective polymer suppliers). The polymer is added to increase

capture of fine solids which would otherwise be lost in the effluent, and to improve sludge settleability overall. When scaling up to plant requirements, calculations should be based on the aeration tank volume.

Polymer required (lbs/day) = (Volume MG) (8.34) (mg/l)*

Normally 2 mg/l of Cationic polymer is required to produce an acceptable effluent suspended solids concentration.

*mg/l = dosage determined from bench jar tests.

3. **Nutrient Addition** - The supplemental addition of nutrient phosphorous may be required to supply this key element to the biomass. The addition of nutrient nitrogen is not required since it is supplied in the leachate influent as ammonia (NH_3).

Bacteria require a source of soluble nitrogen and phosphorous for cell growth and reproduction. If there are insufficient nutrients in the incoming wastewater, two problems can result:

a. Filamentous bacteria (fungi) will predominate resulting in poor settleability of the mixed liquor (bulking sludge).

b. Biological activity will be capable of assimilating only a fraction of the organic loading (food) to the PACT® system. BOD/COD reductions will be hindered.

If excess nutrients are fed to the PACT® system to overcompensate for a nutrient deficiency, a large fraction of these nutrients may not be incorporated into the mixed liquor solids and will pass from the PACT® system in the effluent. It is important to monitor the effluent for excess nutrients, and then make adjustments to the amount of nutrients added. A change of no more

than 5 percent (5%) per week is advised to avoid adverse effects on the biomass.

Operational experience with BOD removal at the lowest possible nutrient dosage rates will allow the operator to properly control the addition rate. Soluble ammonia-nitrogen (NH_3-N) and soluble phosphorous as measured in the PACT® system effluent need not exceed 1-2 mg/l. Many systems have demonstrated adequate treatment at 25-50 percent of these values.

Nutrient requirements are based on BOD_5 loading to the PACT® system. For every 100 parts of BOD_5 in the incoming wastewater, 5 parts of soluble nitrogen and 1 part of phosphorous need to be available to the biomass. Nitrogen in the incoming wastewater should be determined by a TKN (Total Kjeldahl Nitrogen) analysis in the laboratory. Phosphorous in the incoming wastewater should be determined as total phosphorous.

To determine the amount of commercial chemical nutrients to be added to correct a nutrient deficiency, the following information is required:

a. PACT® influent BOD_5, mg/l
b. PACT® influent TKN, mg/l
c. PACT® influent P, mg/l (as total phosphorous)
d. Average daily wastewater flow, mg/d
e. Suggested weight ratio, $BOD_5/N = 100/5 = 20$
f. Suggested weight ratio, $BOD_5/P = 100/1 = 100$
g. Atomic weight ratio for commercial nutrient chemicals used (given in Table 3-1)
h. Density ratios for liquid chemicals (given in Table 3-1)

Examples of nutrient dosage calculations are given in Section 3-2, F.

E. Operation and control of the PACT® system have been discussed in terms of the following parameters:

1. Wasting (proper SRT)

2. Carbon addition (Carbon Dose)
3. D.O. control (minimum of 2 mg/l D.O.)
4. Polymer or coagulant dosage (proper feed rate for actual plant flows).
5. Maintaining proper nutrient (Nitrogen and Phosphorous) levels.

Any problems which arise during operation of a PACT® Wastewater Treatment System will be found to have its cause in one or more of these five parameters.

2.0 Sample Calculations

A. Biomass Level

The influent stream contains pollutants (suspended, colloidal, and dissolved solids) which the PACT® system must remove to meet the required plant effluent limitations. The portion of the waste measured as COD will be adsorbed by the PAC in the aeration system. The portion of the waste measured as BOD will have to be stabilized (converted to CO_2, water and cell structure material) biologically.

The question arises: How much biomass is needed to stabilize a given amount of BOD loading? A commonly used parameter which relates influent loadings and the biomass which results from those loadings is the F/M ratio. F = food or BOD loading, M = microorganism mass.

F/M may be applied to PACT® much in the same way it is used in activated sludge systems. The normal activated sludge F/M is in the range of 0.2 - 0.5.

To calculate a target MLVSS the F/M ratio chosen should be within this range.

Example:

1. Let's assume that the BOD loading to the plant is projected at 2,000 mg/l.

2. The average plant flow is projected at 30,000 gal/day (GPD) or 0.030 million gallons/day (MGD). For each of the two aeration tanks, the average flow is then 15,000 GPD or 0.015 MGD.

3. The volume of each of the three aeration tanks is 0.045 million gallons (MG).

4. Convert BOD loading in mg/l to lbs/day.

   F = 2,000 mg/l x 8.34 lbs/gal x 0.15 MGD = 2500 lbs/day

5. For an F/M of 0.35 (midway between 0.2 and 0.5).

   $M = \frac{2500 \text{ lbs/day BOD}}{0.35 \text{ (per tank)}} = 7149$ lbs MLVSS will be required

6. To avoid problems associated with relatively higher values of F/M (analogous to low SRT) such as turbid effluent containing significant solids and BOD due to the dispersed bacterial growth which characterizes operation at high F/M (short SRT), it is recommended that M (pounds of MLVSS in the system) be increased. This may be accomplished by decreasing wasting rates (raising SRT). In the next section we will consider the constraints on values of F/M which can actually be attained in any given plant. To illustrate the point here, however, assume we choose a biomass which will make F/M=0.24 (M=10,400 lb). This allows BOD excursions up to 5200 lbs/day without exceeding F/M=0.50, but we assume that the average BOD remains at about 2500 lbs/day. This is saying that with an F/M of 0.24 we can double the short term loading on the plant without causing bleed through of BOD or change the biology to an extent that would hamper treatment assuming ample oxygen, nutrients, hydraulic capacity and a readily degradable wastewater are provided.

B. **Carbon (PAC) Level**

The solids carried in the aeration tank of course are not just MLVSS. Associated with the living portion of the biological floc are many inert, inorganic materials like grit. A portion of each living organism is also inorganic. We commonly differentiate these fractions by burning or ashing a MLSS sample in a muffle furnace at 550° or 660° in the laboratory. In a PACT® system, about 5-35% of the virgin PAC added as initial charge or make-up PAC is ash. When a sample of MLSS is analyzed for its various components - PAC, biomass and ash - there is no distinction

made regarding where the ash in the sample came from. Therefore, the following applies equally whether there is PAC in the system or not.

We choose, for example, to operate the system with a mixed liquor volatile carbon concentration at say 2,100 mg/l because experience has shown acceptable treatment results with this level.

1. Given an aeration tank volume of 45,000 gallons then:
   Vol. Carbon = 0.045 MG x 8.34 x 2,100 mg/l = 788 lbs./tank

2. Virgin carbon, as delivered, may be approximately 15% ash, 85% PAC. To get 788 lbs. of volatile carbon will require:
   lbs. virgin carbon = $\frac{788}{0.85}$ = 927 lbs./tank

C. Wasting

Again, this is the means by which the process is controlled. PAC levels in the PACT® system are determined by operating experience. Based on this information, PAC addition to the system is essentially an independent variable, which can be maintained at as high or low a level as required (or desired). Biomass level, on the other hand, is not an independent variable because the amount of biomass which can be developed in any system depends upon the amount of food available to that biomass.

Therefore, the concept of control of PACT® by wasting reflects the fact that what is really being controlled is the biomass. We can add as much PAC as we want, but we will only get as much biomass as the influent loading will support.

Empirically, it has been determined that the biomass which results from a wasting level based on a 5-15 day SRT performs well, and development of nuisance biogrowths are minimized. As dictated by actual operation the SRT may change to provide optimum treatment.

The calculation of the daily waste quantity (W) is quite simple, no matter what SRT is chosen:

W lbs/day = M (MLSS) lbs/SRT days

1. The MLSS concentration is measured at 15,000 mg/l, for example. Then:
   M (MLSS) lbs = 15,000 mg/l MLSS x 8.34 x 0.045 MG = 5,630 lbs.

2. Choose SRT = 15 days (design value), then:

3. $W = \frac{M - \text{Effluent S.S.}}{SRT}$, if effluent S.S. concentration is low, we can neglect this term.

   $$\frac{M}{SRT} = \frac{5{,}630 \text{ lbs}}{15 \text{ days}} = 375 \text{ lbs/day}$$

   This amount can be wasted as mixed liquor, whose concentration has been measured at 15,000 mg/l.

4. $$W \text{ (MGD)} = \frac{375 \text{ lbs/ day}}{\text{MLSS mg/l} \times 8.34}$$

5. $$W \text{ (MGD)} = \frac{375 \text{ lbs/day}}{8.34 \times 15{,}000 \text{ mg/l}} = 0.003 \text{ MGD/tank}$$

   (Also calculated as one fifteenth of the aeration tank volume.)

6. W = 3,000 gallons/day/tank of mixed liquor at 15,000 mg/l SS

7. Let the waste flow rate = 70 gpm (for this example).

   Then the total time in minutes to waste (during aeration sequence) for the day is:

   $$\frac{3{,}000 \text{ gal.}}{70 \text{ gpm}} = 43 \text{ minutes/tank}$$

8. Wasting can be done at the end of the settling sequence or during the aeration sequence. Note that wasting time will be less if done at the end of settling sequence (higher solids concentration). Volume to be wasted if done after decanting 15,000 gallons from the aeration tank is

$$0.003 \text{ max} * \frac{45{,}000 - 15{,}000}{45{,}000} = 0.002 \text{ MGD}$$

D. **Carbon Addition Rate**

1. Carbon addition = Carbon wasted (given negligible effluent losses).

2. The amount of volatile PAC wasted is based on the amount of volatile PAC in the MLSS.

From B.1.: The mixed liquor contains 788 lbs. of volatile carbon (per tank). Since we are wasting one fifteenth (1/SRT) of the total mass of MLSS every day, we will remove:

$$1/\text{SRT} \times \text{MLVC} = 1/15 \times 788 \text{ lbs.} = 53 \text{ lbs of volatile carbon/day}$$

3. To get an equivalent amount of virgin PAC (15% ash):

$$\frac{53 \text{ lbs. vol.}}{0.85 \frac{\text{lb. vol. PAC}}{\text{lb. PAC}}} = 61 \text{ lbs. virgin PAC}$$

4. The carbon makeup required can be added at the start of the cycle. The carbon concentration in the mixed liquor should be routinely analyzed and the carbon feed adjusted accordingly.

E. **Polymer Addition**

1. A cationic polyelectrolyte (polymer) of high molecular weight, applied at the correct dosage, can significantly reduce the amount of suspended solids in the effluent and enhance settling.

Dosage requirements can be (roughly) determined by a bench jar test. Your polymer supplier will usually be glad to demonstrate his/her product and suggest dosage rates.

A wide range of brands and types of polymer should be tested initially keeping in mind the economics involved. One supplier's "cheap" polymer may work just as well as another supplier's "expensive" brand or even exceed the high priced polymer's performance. Polymer dosage is usually expressed in terms of parts per million.

2. Example

Let's say that the polymer chosen demonstrates good particulate capture at 2 ppm, as demonstrated by the jar test.

The polymer will be applied to the entire tank contents of 45,000 gallons. The pounds of polymer can be calculated as follows:

$$0.045 \text{ MG} \times 8.34 \text{ lb/gal} \times 2 \text{ ppm} = 0.75 \text{ lbs/day}$$

Convert 0.75 lbs. of polymer to gallons of polymer:

$$\text{Total days dosage} = \frac{0.75 \text{ lbs/day}}{9.0 \text{ lbs/gal}} = 0.083 \text{ gallons of polymer per day as supplied}$$

3. Polymer Dilution:

The concentrated polymer must be diluted prior to blending into the wastewater stream. This occurs in the mix tank supplied as part of the polymer addition system. The neat polymer solution is pumped into the mix tank containing the proper quantity of dilution water to yield a solution of about 0.5% wt. polymer.

At an injection rate of 7 gpm, approximately 2 to 3 minutes are required to inject 2 mg/l of polymer respectively. Experience will dictate what dilution and dosage are most effective.

F. Nutrient Addition

The following example shows how to determine whether there is a nutrient deficiency in the PACT® system influent, and how to calculate the quantity of additional nutrients required to correct the deficiency. Once the nutrient chemical feed rate (lb/day) is determined, the chemical feed equipment must be set to feed the calculated amount of nutrient based on a 24-hour period. (In continuous flow PACT® systems the nutrients should be fed, preferably, in proportion to flow. In batch PACT® systems the nutrients should be added during the wastewater feed step of the process cycle.)

Example: Calculate the required amount of additional nutrients to correct a nutrient deficiency.

Given: (for this example only)

a. PACT® influent BOD_5 = 2000 mg/l
b. PACT® influent TKN = 250 mg/l
c. PACT® influent P = 2 mg/l
d. PACT® average daily influent flow = 0.028 mgd (27,600 gallons/day)
e. Suggested weight ratio, BOD_5/N = 100/5 = 20
f. Suggested weight ratio, BOD_5/P = 100/1 = 100
g. Nutrient Nitrogen = Commercial liquid ammonium hydroxide (NH_4OH) 29% by weight. Atomic weight ratio from (Table 3-1) = 2.5
   Nutrient Phosphorous = Commercial liquid phosphoric acid (H_3PO_4) 75% by weight. Atomic weight ratio (from Table 3-1) = 3.2
h. Density of 29% aqueous NH_4OH = 0.95 (from Table 3-1)
   Density of 75% H_3PO_4 solution = 1.58 (from Table 3-1)

1.0 Calculate nutrient needed to achieve the suggested BOD/N/P ratios.

$$\text{Nutrient Needed} = \frac{\text{PACT® influent BOD, mg/l}}{\text{suggested ratio, } BOD_5/\text{nutrient}}$$

$$\text{N needed, mg/l} = \frac{2000}{20} = 100 \text{ mg/l}$$

$$\text{P needed, mg/l} = \frac{2000}{100} = 20 \text{ mg/l}$$

2.0 Calculate the nutrient addition. If the answer is zero or a negative number, no nutrient need be added.

Nutrient Addition = (Nutrient needed, mg/l) - (Nutrient in PACT® influent, mg/l)

N addition, mg/l = 100-250 (TKN) = -150 mg/l

P addition, mg/l = 20-2 (Total P) = 18 mg/l

3.0 Calculate the weight of nutrients that need to be added.

Nutrient to add, lb/day = (Added Nutrient, mg/l)(Q, mgd)(8.34 lb/gal)

P to add, lb/day = (18)(.020)(8.34) = 1.5 lb/day/tank

4.0 Calculate weight of commercial chemical to be added per day

$$\text{Nutrient chemical, lb/day} = \frac{\text{(nutrient to add, lb/day)(Atomic weight ratio)(100\%)}}{\text{concentration of chemical \%}}$$

$$75\%\ H_3PO_4\ \text{gal/day} = \frac{(1.5)(3.2)(100)}{75} = 6.4\ \text{lbs/day, solution/tank}$$

5.0 Convert lbs/day solution to gallons per day

$$\text{Nutrient chemical, gal/day} = \frac{\text{Solution lbs/day}}{\text{(Density)(8.34 lbs/gal)}}$$

$$75\%\ H_3PO_4\ \text{gal/day} = \frac{6.4}{(1.58)(8.34)} = 0.5\ \text{gal/day/tank}$$

Note: If dry chemicals are to be used, the weight of the chemical is determined directly; Step 5.0 is omitted.

6.0 Nutrient addition using estimated BOD and nitrogen values.

As stated previously, a desirable BOD:N:P weight ratio based on influent BOD is 100:5:1. For practical purposes an estimate of organic loading to the PACT® system can be used especially if nutrient addition rates have to be adjusted on a daily basis. Therefore, analysis such as chemical oxygen demand (COD) is necessary to provide quick analytical results. COD analyses can be run within several hours and an estimated BOD value can be calculated once a BOD:COD ratio has been established. To establish a BOD/COD ratio, parallel COD and BOD analyses of the PACT® influent should be conducted over a period of 90 days or longer. Once the

BOD:COD ratio is known, a BOD can be estimated based on a measured COD.

Estimated BOD, mg/l = (COD, mg/l)(Ratio BOD/COD)

As an estimate for nitrogen content in the PACT® influent, ammonia-nitrogen (NH_3-N) can be used since it is easier to run in the lab than the Total Kjeldahl Nitrogen (TKN) analysis. However, a correlation between NH_3-N and TKN must be developed also. Again, this ratio of TKN/NH_3-N should be established over a period of 90 days or longer using parallel TKN and NH_3-N analyses of the PACT® influent.

Estimated N, mg/l = (NH_3-N, mg/l)(Ration TKN/NH_3-N)

a. Example: Estimate the BOD loading based on a COD analysis.

For this example, let's say that over a 3-month period of time, the influent BOD/COD ratio is consistently 0.33. In other words, the organic loading to the PACT® system can be expressed as: BOD = (COD)(.33)

Given:

COD = 6,000 mg/l

Estimated BOD, mg/l = (COD, mg/l) ratio $\frac{\text{BOD}}{\text{COD}}$

Estimated BOD, mg/l = (6,000)(.33) = 1980 mg/l

Use the estimated BOD in the nutrient calculation in place of actual BOD_5.

b. Example: Estimate N available in the PACT® influent based on the TKN/NH_3-N ratio.

For this example, let's say that over a 3-month period of time the TKN/NH_3-N ratio is consistently 10.0.

Given:

NH_3-N = 25 mg/l

Estimated N, mg/l = (NH_3-N, mg/l) ratio $\frac{\text{TKN}}{\text{NH}_3\text{-N}}$

Estimated N, mg/l = (25)(10) = 250 mg/l

Use the estimated N in the nutrient calculation in place of the actual TKN.

Note: TKN measures organic nitrogen plus ammonia-nitrogen in the sample. This analysis better quantifies total nitrogen available to the biomass.

7.0 De-nitrification

De-nitrification occurs in the absence of free oxygen if the bacteria present in the tank (anoxic tank) are provided with an adequate food source. Methanol has been chosen as the food source. Although there are other food sources available, methanol has been chosen because it does not contain any nitrogen, it is non-toxic at low concentrations, it reacts easily and it leaves no significant by-product (COD).

The quantity of methanol required to provide an adequate food supply is approximated using the following equation.

Methanol required in mg/l	=	[Initial nitrate X 2.47] [in mg/l]	+	[Initial nitrate X 1.53] [in mg/l]	+	[Initial D.O. X 0.87] [in mg/l]

For the following example, we will assume a nitrate concentration of 200 mg/l, a nitrite concentration of zero (which is typical), and a dissolved oxygen concentration of 2 mg/l.

Methanol required in mg/l = (200 mg/l X 2.47) + (0 mg/l X 1.53) + (2 mg/l X 0.87)

The results of the sample equation indicates that 496 mg/l of methanol will be required to satisfy food requirements in the anoxic tank. The following equation is used to convert mg/l to lbs. per day.

lbs of methanol required per day	=	Methanol required in mg/l	X	Weight of one gallon in water	X	Daily flow in MGD

Lbs. of methanol required per day = 496 mg/l X 8.34 X 0.03 MGD

The result of the sample equation indicates that 124 lbs. of methanol will be required per day. To determine the quantity of methanol required in gallons, we will have to divide 124 lbs. by the weight of one gallon of methanol. A gallon of methanol weighs 6.6 lbs per gallon. The result of division indicates that 18.8 gallons of methanol will be required per day in four batches in the second stage tank. To provide the appropriate quantity of methanol (for this example), the discharge flow of our metering pump must be adjusted to provide a flow of 4.7 gph for one hour during the fill/mix portion of each cycle.

TABLE 3-1

PACT® Nutrient Atomic Weight Ratios
for Commonly Used Commercial Chemicals

Source	Formula	Atomic Wt	Calc.	Atomic Wt. Ratio	Density
Nitrogen					
Elemental Nitrogen	N	14	14/14 =	1.0	-
Ammonia	NH_3	17	17/14 =	1.2	-
Ammonium Hydroxide	NH_4OH	35	35/14 =	2.5	-
10% solution		-	-	-	.98
18% solution		-	-	-	.96
29% solution		-	-	-	.95
Ammonium Chloride	NH_4Cl	53.5	43.5/14 =	3.8	-
Ammonium Sulfate	$(NH_4)_2SO_4$	132	132/(2)(14) =	4.7	-
Phosphorus					
Elemental Phosphorus	P	31	31/31 =	1.0	-
Phosphoric Acid	H_3PO_4	98	98/31 =	3.2	-
50% solution		-	-	-	1.34
75% solution		-	-	-	1.58
Trisodium Phosphate	Na_3PO_4	164	164/31 =	5.3	-
Combination N-P					
Mono-basic Ammonium Phosphate	$(NH_4)H_2PO_4$	115	115/14 = 115/31 =	8.2 (nitrogen) 3.7 (phosphorus)	
Di-basic Ammonium Phosphate	$(NH_4)_2H_2PO_4$	132	132/(2)(14) = 132/31 =	4.7 (nitrogen) 4.3 (phosphorus)	

SECTION NO. 4

PLANT COMPONENT DESCRIPTION AND OPERATION

Important - This section explains in detail the description, function and operation instructions for every major piece of equipment in the plant. It is not a sequential listing of start-up and shut-down instructions for the plant. Sequential start-up and shut-down instructions are found in Sections 5 and 8 respectively of this manual. This section should be referenced in conjunction with sequential plant start-up and shut-down instructions. Further information for each piece of equipment is found in the Equipment section of the Equipment/Control/Instrument/Valve Manual.

1.0 Operating Sequence

Batch PACT® Units No. 1, 2, and 3 can be operated in either manual or automatic mode. In the manual mode, all pieces of equipment will operate independently. In the automatic mode most of the equipment will operate automatically once the operator starts the cycle. In the automatic mode the equipment operation is controlled by a series of adjustable timers in the PC0085 programmable controller. The registers are preset and times are adjusted by using a TC0085 keypad located on the front face of the Batch PACT® control panel.

The A.S.S.T./Decant equalization tank is operated in the manual mode only. That is, each piece of equipment operates independently.

Automatic Mode

The operating sequence for the automatic operating mode is controlled using a PC0085 programmable controller located in each control panel. Additional information, including the Gould PC0085 programmable control system users manual, the Quartech TCR085 Data Access Panel Products Manual, and a Zimpro/Passavant software ladder diagram of the automatic operating mode as outlined below, is given in the Control Section of the Equipment/Control/Instrument/Valve Manual. Operators should become familiar with the Control/Instrumentation sections of this manual before attempting to operate the Batch PACT® unit. Operation of the Batch PACT® unit in the manual mode would be similar to the operator having the responsibility to turn-on or shut-off equipment at the appropriate time.

Sequence of Operation
First Stage Batch PACT® Unit 1 or 2

1. When power is applied to panel, unit is off-line and influent valve is closed. Place all selection switches in the "auto" position.
2. Operator presses START, CYCLE RUN lamps lights, unit enters IDLE mode.
3. Unit waits for both other Batch PACT® influent valves to close. This cycle will continue until both other influent valves close. Influent valve opens.
   a. In Aerated Fill mode (normal operation), blower starts (if not already running).
4. Blower continues to run until tank fills (level control mode)
5. Aerobic React Timer startes when tank completes filling.
6. Aerobic React Timer expires. Polymer addition Timer starts. Polymer pump starts. Blower continues operation.
7. Polymer Addition Timer expires. Polymer pump stops. Flush Timer starts. Flush Valve opens.
8. Flush Timer expires. Flush Valve closes. Polymer Mix Timer starts.
9. Polymer Mix Timer expires. Blower stops. Settling Timer starts.
10. Settling Timer expires. Decant pipe lowers.
11. Decant pump starts. Tank is pumped to LOW level. Decant pump stops. Decant pipe rises.
12. Go to Step 3.

NOTE: Wasting is manual. Phosphoric acid addition is manual.

Sequence of Operation
Second Stage Batch Tank Unit 3

1. When power is applied to panel, unit is off-line and influent valve is closed. Place all selection switches in the "auto" position.
2. Operator presses START, CYCLE RUN lamps lights, unit enters IDLE mode.
3. Unit fills from either of the first stage tank decant lines.
4. When tank low level is exceeded, the mixer starts for a timed period. The methanol Feed pump also starts for its own timeed period, and the Fill Timer also starts.
5. When Fill Timer times out, the first stage decant pump stops transferring decant to the second stage tank.
6. When the methanol Feed Timer expires, the methanol pump stops.
7. When the Mixer Timer expires, (Anoxic React Timer) the mixers stop and the aeration blower starts for a timed period.
8. When the React Timer expires, the Polymer Addition Timer starts. Blower continuees to operated.
9. Flush Timer expires. Flush Valve closes. Polymer Mix Timer starts.
10. Polymer Mix Timer expires. Blower stops. Settling Timer starts.
11. Settling Timer expires. Decant pipe lowers.
12. Decant pump starts. Tank is pumped to LOW level. Decant pump stops. Decatn pipe rises.
13. Go to Step 3.

Modicon 0085 Programmable Controller PACT® Registers				
	Name	Register	(1st Stage) Preset (One Cycle/Day)	(2nd Stage) Preset (One Cycle/Day)
1.	Fill Timer	C00	N/A	
2.	Aerobic React Timer/ Anaerobic Reset Timer	C01		
3.	Anoxic React Timer (MeOH Feed)	C02	N/A	
4.	Polymer Addition Timer	C03		
5.	Polymer Line Flush Timer	C04		
6.	Polymer Mix Timer	C05		
7.	Settling Timer	C06		
8.	Horn Blower Timer	C07		
9.	Anoxic Idle Timer (Mixer Run)	C10		
10.	Aerobic Idle Timer	C11		N/A

All register values are in minutes.

Enable Fill Timer by setting register].40 to 1. If the Fill timer is disabled, the Influent Valve will close when the tank fills to the High Level Sensor. If the Fill Timer is enabled, the Influent Valve will close when the Fill Timer expires or the tank fills to the High Level Sensor, whichever comes first.

If register].41 is set to 0 (default setting), the unit will run the Aeration Blower during filling (Aerobic Fill). If register].41 is set to 1 (by using the register access module), the unit will run the Mixer during filling (Anoxic Fill).

The Aeration Blower will always run during steps 2, 4, 5 and 6. The Mixer will always run during Step 3.

In Idle mode, if this unit is waiting for influent for longer than the Anoxic Idle Timer, due to low flows, the Aeration Blower will run for the time set in the Aerobic Idle Timer. This cycle will continue until the unit is needed to process influent or the unit is taken off line.

Citrus County Cycle Description

1. Start mixers (starting mixers enables methanol feed pump).
2. Start feed from tank 1 and 2.
3. Start methanol feed pump. Operate for timed period. Pump may time out during fill or after fill has been completed.
4. Mixer times out, with methanol pump stopped, start aeration blower for timed period.
5. Near end of aeration period, add polymer. Let aeration blower mix the polymer into the tank contents.
6. Stop aeration blower. Let settle for timed period.
7. Decant
8. Add carbon if required (manual operation).
9. Ready for a new batch.

Operation of Batch PACT®
Unit 1, 2 and 3

Start-up

Apply power to all three units (120 VAC 1ø and 480 VAC 3ø). Press Cycle Stop button twice to ensure each unit is off line.

Press Cycle Start on unit to be filled first. Wait for influent valve to open. Press Cycle Start on other tanks to be used.

Operation

Press Cycle Stop once to pause a unit. While the unit is paused, the Cycle Run lamp will flash, all motors and pumps will stop, and the influent valve will close.

Press Cycle Stop again to take unit off line and reset PACT® cycle position to beginning.

NOTE: No motors or pumps will be automatically started if the unit is off line. If aeration of the tank is required when the unit is off line, place the Aeration Blower control selector in the Hand position.

To continue a paused cycle, press the Cycle Start button. The Cycle Run lamp will go to a steady-on state, and the PACT® cycle will continue from the paused point.

NOTE: If the unit is paused during a timed event (such as the aeration step), the total time spend in that step may vary by as much as one minute per each time the cycle is paused. Pressing Cycle Stop while the polymer line contains polymer is strongly discouraged.

Shutdown

To take an individual unit off line, press Cycle Stop twice. No pumps or motors will be automatically started, although the Hand Off Auto and Open Close Auto switches can still be used to manually operate the pumps, motors and valves.

NOTE:

1. Chemical feed (nutrient phosphorous) to the aeration tank is done manually by running the chemical pump for a predetermined length of time.
2. Sludge wasting from the aeration tank is done manually by running the sludge waste pump for a predetermined length of time. Sludge is pumped to the sludge storage tank.
3. Carbon addition is done manually by placing 50 lb. bags of PAC, as required, in aeration tank.

2.0 Non-Potable Water Supply System

Description: Non-potable (clean) water is supplied to the following users via a network of piping and valves:

Equipment	Use
Polymer System	Polymer dilution, line flushing
Waste Sludge Pump	Seal Water, line flushing
Chemical Tank	Chemical mixing

Start-Up: Before charging the non-potable water system, make sure the following valves are closed:

No. 1, 7 (Polymer System)

No. 6, 11 (Waste Sludge Pump)

No. 37 (Chemical Tank)

No. 32 (Sludge Pump)

No. 19 (Non-Potable Water Strainer)

3.0 Controls/Instrumentation

Description: The controls/instrumentation consists of four field control panels and miscellaneous instruments for local control of Zimpro/Passavant furnished equipment.

A. Batch PACT® Unit 1, 2, & 3 Control Panels - One panel is located in the equipment area of each unit. Each panel houses the programmable controller, electrical equipment, and manual operating controls for:

-- Aeration Blower

-- Tank Mixer

- -- Influent Valves
- -- Polymer System
- -- Waste Sludge Pump
- -- Decant Pump & Winch
- -- Alarms

B. A.S.S.T./Decant Equalization Control Panel - Located in the equipment area of the unit. This panel houses the electrical equipment and manual operating controls for:

- -- Aerated sludge blower
- -- Sludge pump
- -- Chemical feed system
- -- Filter feed pumps
- -- Alarms

C. Miscellaneous field instruments and electrical controls including:

- -- All hand switches (HS) for 120 volt, 1ø, Zimpro/Passavant supplied motors
- -- Pressure Indicators (PI)
- -- Temperature Indicators (TI)
- -- Level Switches (LS)
- -- Limit Switches (ZS)
- -- Electrically actuated valves (EV)
- -- And other miscellaneous instruments as shown on the engineering flow diagrams.

Start-Up

It should be noted here that once the plant instrumentation is energized, it should remain energized unless the plant is shut down for an extended period of time.

Shut-Down

De-energize all power supplies.

4.0 Polymer System

Description: Polymer is mixed in a tank and fed to the system by a constant speed gear pump rated at 7 gpm. Add neat polymer to the polymer tank containing dilution water to form a ½% (wt) polymer solution. The mixer should be on as the polymer is added and allowed to mix for 30 minutes after polymer addition. Mixing is not required during operation. Polymer quantities should be limited to what can be used in a 24-hour period.

Polymer addition to the aeration tank per cycle:

Polymer Dose, mg/l	Volume at ½%, gal	Pump Run Time, min.
2	14	2
3	21	3
5	35	5

Start-Up:

Prerequisites:

-- Non-potable water available.

-- Power supply energized.

To prepare the polymer system for use: Close valve No. 15, open valve No. 1, and add the desired quantity of water to the polymer tank.

Open valve No. 19 and flush strainer (No. 18) until clean water is observed. Operate polymer pump momentarily (HS-130 to HAND position) to insure water flow through the pump. Turn on the polymer tank mixer (HS-120) and add neat polymer to the mix tank. Allow to mix for 30 minutes and then turn off mixer (HS-120).

Open valve No. 2 & 7. Turn on the polymer pump (HS-130) and run the pump for the time required to provide the polymer dosage. In the automatic mode the run time is set on register C03 of the PC0085 programmable controller.

Shut-Down: Turn off the polymer pump (HS-130), and flush the lines with dilution water by opening valve EV-135 with hand switch HS-135. In the automatic mode the pump will stop and lines will be flushed in accordance with the Operating Sequence.

Note: If the system is shut down for a long period of time, it should be thoroughly flushed with water and drained.

5.0 Aeration Blower

Description: Air to the aeration tank diffusers is supplied from one aeration blower. The blower is a Roots Type RAI-U, Size 718, rotary positive displacement blower rated at 1000 cfm. The blower is equipped with inlet filter, inlet and discharge silencers, inlet and discharge temperature gauges, inlet and discharge expansion joints, discharge relief valve, discharge check valve, and discharge pressure gauge.

Start-Up:

Prerequisites:

-- Blower pre-startup maintenance, including lubrication, must be completed (see Equipment Manual for details).

-- Electric power supply energized.

Before starting the blower, open Valve No. 10. Turn on the blower (HS-180 to HAND position). In the automatic mode, the blower will start in accordance with the Operating Sequence.

Shut-Down: Turn off the blower (HS-180 to OFF position). In the automatic mode, the blower will stop in accordance with the Operating Sequence.

6.0 Waste Sludge Pump

Description: PACT® sludge is drawn from the bottom of the aeration tank and pumped to the aerated sludge storage tank by the waste sludge pump. The pump is Dean Bros. Model DL201, in-line vertical, centrifugal pump rated at 70 gpm.

Start-Up:

Prerequisites:

-- Waste sludge available.

-- Power supply energized.

Before starting the waste sludge pump, open the following valves:

No. 9, pump suction.

No. 11, seal water. Establish seal water flow at 5-10 GPH. Readjust once the pump is running.

No. 24, pump discharge.

Turn on the waste sludge pump (HS-190) and run until the required volume of sludge has been pumped.

Shut-Down: Turn off the waste sludge pump (HS-190). If the shutdown is to be an extended one, close valve No. 9 and flush the pump suction and discharge lines with non-potable water by opening valve No. 6.

7.0 Mixers (Unit #3)

Description: Contents of the aeration tank are kept mixed during the anoxic cycle by the submersible mixer. The three dirven Lightnin mixers are rated at 12500 gpm.

Start-Up:

Prerequisites:

-- Wastewater available.

-- Power supply energized.

-- Mixer pre-startup maintenance, including lubrication, is completed (see Equipment Manual for details).

Turn on the mixer (HS-175 to HAND position). In the automatic mode, the mixer will start in accordance with the Operating Sequence.

Shut-Down: Turn off the mixer (HS-175). In the automatic mode, the mixer will stop in accordance with the Operating Sequence.

8.0 Decant Pump

Description: Decant supernatant is drawn from the top of the aeration tank and pumped to the decant equalization tank by the decant pump. The pump is a Flygt Model CT3085/82, dry pit vertical mount, centrifugal pump rated for 200 gpm.

Start-Up:

Prerequisites:

- Wastewater available for decanting.
- Decant pipe lowered below wastewater to decant level.
- Power supply energized.

Before starting the decant pump, open the following valves:

No. 8, pump suction

No. 21, pump discharge

Turn on the decant pump (HS-170) to HAND position and run until aeration tank is decanted to the desired level. In the automatic mode, the decant pump will start in accordance with the Operating Sequence.

Shut-Down: Turn off the decant pump (HS-170). In the automatic mode, the decant pump will stop in accordance with the Operating Sequence.

9.0 Decant Winch

Description: The decant pipe is lowered into the aeration tank for draw-off of the decant supernatant and raised out of the liquid after decanting is completed by the decant winch. The winch is a Thern Model 473A 1/2 B electric power winch (worm gear type).

Start-Up:

Prerequisites:

-- Power supply energized.

Turn local operating station hand switch (HS1-150) to manual position and raise or lower decant pipe by turning hand switch (HS2-150). In the automatic mode, the decant winch will be raised and lowered in accordance with Operating Sequence.

NOTE: For automatic operation the limit switches furnished with decant winch must be adjusted to the required lowered position and raised position.

Initial Start-Up Raised Position -

Set end of pipe @ 0'-6" from top of aeration tank.

Initial Start-Up Lowered Position -

Set end of pipe @ 4'-0" from top of aeration tank.

10.0 Carbon Addition

Description: Virgin powdered activated carbon (PAC) is added directly to the anoxic tank on a daily basis as required to meet leachate treatment performance levels. PAC is furnished in 50 lb. water soluble bags with an over-pack made of paper. These bags are to be stored indoors in a dry area. NOTE: Virgin PAC may also be added directly to the aeration tanks as needed.

To Add Carbon: PAC bag(s) are transported from storage to the Batch PACT® tank platform. At this time the outer paper bag can be cut at one end, allowing inside water soluble bag to be carefully removed and slipped into the aeration tank.

Safety: Note that some dusting of carbon will occur and that a dust mask is recommended while handling PAC. See Activated Carbon Material Safety Data Sheet found in Section 9 of this manual for additional information regarding activated carbon.

11.0 Chemical Feed System

Description: Phosphoric acid (nutrient) is fed from a tank to one of two (2) aeration tanks by a Neptune Chemical proportioning pump, Model No. 532-A-N3, diaphragm type, rated at 0-11 GPH. Add 50% or 75% commercially available solution of phosphoric acid to the chemical tank. (A mixer is supplied for the mixing of dry chemicals, if required.)

NOTE: Be sure that Valve No. 39 (Chemical Tank Drain) is closed before filling tank.

Safety: A phosphoric acid Material Safety Data Sheet must be obtained from the supplier of phosphoric acid. This safety data sheet will provide detailed information on the

phosphoric acid used at your facility. For reference, a "sample" phosphoric acid Material Safety Data Sheet can be found in Section 9 of this manual.

CAUTION: Eye protection and special clothing are required when handling phosphoric acid. See Section 9 on working safely with chemicals.

Start-Up:

Prerequisites:

- Non-potable water available.
- Power supply energized.
- Solution in chemical tank.
- Wastewater in aeration tank.

Open pump suction valve No. 38 and discharge valve No. 22 to the selected aeration tank. Turn on the chemical pump (HS-440) and run the pump at the required stroke rate and length of time to provide the needed phosphoric acid dosage.

Shut-Down: Turn off the chemical pump (HS-440) and close valve No. 38 and 22.

12.0 Aerated Sludge Blower

Description: Air to the aerated sludge storage tank diffusers is supplied from one aerated sludge blower. The blower is a Roots Type RAI-U, Size 36, rotary positive displacement blower rated at 60 cfm. The blower is equipped with inlet filter, inlet and discharge silencers, inlet and discharge temperature gauges, inlet and discharge expansion joints, discharge relief valve, discharge check valve, and discharge pressure gauge.

Start-Up:

Prerequisites:

- Blower pre-startup maintenance, including lubrication, must be completed (see Equipment Manual for details).
- Electric power supply energized.

Before starting blower, open valve No. 28. Turn on blower (HS-460).

Shut-Down: Turn off blower (HS-460) and close Valve No. 28.

13.0 Sludge Pump

Description: Solids from the bottom of aerated sludge storage tank are pumped to the sludge drying beds adjacent to the leachate treatment system. The pump is a Flygt Model CT3085/82, dry pit vertical mount, centrifugal pump rated for 180 gpm at 12' head or 220 gpm at 7.5' head.

Before pumping sludge to drying beds, the aerated sludge blower should be turned off (HS-460) and the sludge allowed to settle. At this time the liquid near the top of the tank can be decanted to the pump drain station. This is done by using the manual decant winch to position the decant pipe just above the sludge level and opening valve No. 34.

Before the sludge pump, open the pump suction and discharge valve, No. 33 and 30 respectively.

Turn on the sludge pump (HS-430) and run until the required amount of sludge has been pumped or pump automatically shuts off at low level.

Shut-Down: Turn off the sludge pump (HS-430) and close pump suction valve No. 33. If shutdown is to be an extended one, flush the pump suction and discharge lines with non-potable water by opening Valve No. 32.

Restart aerated sludge blower (HS-460).

14.0 Filter Feed Pumps

Description: Treated leachate from the decant equalization tank is pumped to a tertiary sand filter by the two filter feed pumps. These pumps are Goulds Model GL887 submersible sewage pumps rated for 30 GPM at 23' TDH. In the automatic mode these pumps are cycled on and off by level controls as follows:

- lead pump ON @ High Level
- lag pump ON @ High High Level
- lead and lag pump OFF @ Low Level

Note: Lead and Lag pumps are alternated.

In the manual mode the filter feed pumps can be operated by turning HS-420 and/or HS-425 to HAND position.

Start-Up:

Prerequisites:

-- Wastewater available.

-- Power supply energized.

-- Tertiary filter on-line.

Turn pump(s) ON (HS-420 and/or HS-425).

Shut-Down:

Turn pump(s) OFF (HS-420 and/or HS-425).

15.0 Prepare the Methanol System

15.1 Open the following valves:

No. 75, methanol tank outlet to the methanol pumps.

No. 77, (2) methanol pump outlet.

SECTION NO. 5
PLANT START-UP

1.0 Narrative

This section contains a sequential, step-by-step procedure to start-up the PACT[R] Wastewater Treatment Plant. This procedure should be followed anytime the plant is put on line, such as after an extended shut-down.

A. Due to initial slow biomass growth, the plant should be started, if possible, at a decreased influent loading rate.

B. An initial charge of 800 lbs. (16 bags) of virgin PAC is to be added to each aeration tank. This amount is placed directly in aeration tank.

C. It will be very important to control wastewater flows into the plant during start-up. In no case should the full flow/load be applied before PAC and biomass levels have reached at least the lower design limit concentrations and the polymer used has been proven to be effective. Premature introduction of the full hydraulic load may result in biomass die off and the possible loss of weeks of effort in developing an acclimated biomass.

D. Biomass can be introduced into the system in two ways - either by developing naturally in the system from inoculating organisms carried in with the waste stream or by seeding the system with sludge from an operating biological treatment plant. Seeding with a large inoculum of either activated sludge or trickling filter humus will allow development of the biomass much more quickly than will starting from "scratch".

If sludge can be trucked in from a neighboring plant, add enough sludge to bring the biomass level to about 500 mg/l, after which it can develop on its own to whatever level the influent feed can support.

The recommended start-up schedule is outlined below. Percentages listed represent percent of the total daily flow (30,000 GPD).

% Waste	Duration, Days
25	2
50	4
75	4

The schedule may be shortened or waste flow percentages increased as dictated by the performance results.

E. Polyelectrolyte or "polymer" should also be added from the beginning of plant operation in the proper dosage for the flows/loadings existing during the start-up period. It may be necessary to perform jar tests and change feed rates accordingly, even day-to-day, as flows and loads change and the system approaches design or "steady state" operation. This will be especially important when the plant is started from "scratch" or with very low levels of biomass.

F. It will not be necessary to waste sludge immediately, especially when the biomass is difficult to establish, but wasting and PAC addition to maintain SRT and the desired carbon dose should begin as soon as biomass is detectable microscopically, and not later than one SRT after beginning operation of waste (i.e., if the design SRT is 15 days, the wasting of 1/15 of the solids in the system daily should begin within 15 days after the introduction of waste - in whatever quantity or flow - to the PACT^{R} system).

G. As biomass levels increase, the system oxygen demand will also increase. D.O. checks at various points and depths in the aeration tank should be made 2-3 times per day, and additional aeration provided if D.O. concentrations are seen to fall consistently between 1-2 mg/l D.O.

H. It will be necessary to sample and test the system daily during start-up. Table 5-1 shows the recommended sample points and analyses. While many plants may be run without all recommended analyses, baseline data and periodic samples are invaluable in solving problems which may arise even long after start-up. Analytical procedures for the recommended analyses will be found in Section 10 of this manual.

2.0 Initial Start-Up Instructions

A. Pre-Start-Up Checks & Procedures

These checks should be made at initial start-up and also by all new operating personnel (or whenever the system has been idle or mothballed for an extended period).

1. Locate and trace all flow streams in the system. Use the equipment drawings supplied with the system equipment manual. These streams include:
   a. Plant influent stream
   b. Air flow
   c. Waste sludge flow
   d. Polymer feed lines
   e. Non-potable water
   f. Nutrient system
   g. Waste sludge transport and storage

   Inspect all connections and fittings to be sure they are correct and tight.

2. Inspect the equipment, piping and valves associated with each stream to ensure free operation, no obstructions or broken lines, and in the case of rotating equipment, jog to confirm the proper direction of rotation. Repair or adjust as necessary.

3. Check all lubricant levels and grease fittings, determine whether fresh lubricant is needed or has recently been added.

4. Check packings, seals and seal water flows to ensure proper installation and operation. Packings and seal water flows will have to be readjusted after operation on process flows.

5. Check all timers for proper initial setting (see equipment manual for initial setpoints). Visually confirm that contacts and electrical connections are secure and clean (not burned or corroded).

6. Visually check the entire system for loose or broken gratings, braces, belt guards, etc. Insure that all protective coatings and paint are intact, cover the area completely as required, and are free from wrinkles, bubbles, and flaking or peeling especially on submerged surfaces.

7. Read and become thoroughly familiar with all manufacturers' manuals and instructions in the system equipment section.

B. **Check-Out on Water**

Fill all the tanks with water, then run all equipment and instrumentation for a minimum of 8 hours following manufacturers' instructions and instructions found in Section 4 of this manual. Monitor all equipment closely and note the following:

1. Current draw on each leg of 3 phase equipment.
2. Any unusual noise or vibration.
3. Overheating.
4. Make operational adjustments to equipment and instrumentation per instructions given in manufacturers' manual.

C. **Detailed Plant Start-Up**

Once the equipment and instrumentation has performed satisfactorily on water, prepare the plant to go on-line as follows:

NOTE: Reference sections in parenthesis ( ).

1. Drain the test water from all tanks except the aeration tank. The aeration tank should be drained to about two thirds full to accommodate the initial charge of carbon slurry and seed biomass.
2. Energize the instrumentation system (Section 4-3).
3. Charge the non-potable water system (Section 4-2).

Important

It should be noted here that the operator should take all prescribed safety precautions for handling powdered activated carbon, acid, nutrients, and polymer before proceeding. See Section 9 on Personnel Safety.

4. Prepare the polymer addition system (Section 4-4).
5. Start the aeration blower at minimum flow (Section 4-5).
6. Charge the aeration tank with PAC (Section 4-10).
7. Add seed sludge to aeration tank. The aeration tank should now be nearly full. (If not, add water.)
8. Add wastewater to the aeration tank at 25% of normal flow.
9. Add nutrients (Section 4-11).
10. Begin wasting sludge to the aerated sludge storage tank after 10-15 days of sustained biological growth.

Monitor all plant functions. Maintain the following operating parameters during the start-up phase:

MLSS D.O.	2.0 mg/l (ppm) minimum
MLSS pH	6.5 - 7.5

Effluent ammonia nitrogen and phosphorous at 1-3 ppm.

Influent flow - increase gradually as biological activity increases (no more than 25% increase per day).

TABLE 5-1

SUGGESTED ANALYTICAL SCHEDULE

	BOD	COD	SS	PAC/BIO	pH	NH_2-N	TOTAL P	SETTABILITY	NO_3-N
1. PACT Feed	D	D	D	N	D	D	D	N	N
2. Mixed Liquor (1A,B,C)	N	N	D	TH	D	N	N	D	N
3. Effluent (1A,B)	D	D	D	N	D	D	D	N	D
4. Effluent	D	D	D	N	D	D	D	N	D
D = Daily (week days)									
TH = 2-3 times per week									
W = Once per week									
N = Not required									
NOTE: Analytical work required by the Discharge Permit is not considered in this manual.									
Use TOC of COD to monitor treatment level. Use microscopic exam to monitor biology. Use NH_3 and total P to regulate nutrient addition.									

SECTION NO. 6
ROUTINE OPERATION

1.0 Routine Operation

Maintain the following process control parameters during normal operation of the plant.

Influent flow range - minimal to 10,000 GPD per aeration tank at 6000 mg/l COD. (flow to each aeration tank can be increased proportionally as influent COD concentration falls below 6000 mg/l, maximum hydraulic capacity is 27,000 gallons per batch)

Mixed liquor D.O. - 2 ppm minimum (aeration tanks)

Mixed liquor pH - 6.5 to 7.5 is optimal

Nutrient addition - maintain effluent phosphorous (P) in the range of 1-3 ppm.

Polymer feed rate - maintain minimal "suspended solids" in effluent.

SRT - 15-20 days (10 day SRT recommended to start with. SRT may be increased slowly if nuisance biogrowths do not cause effluent quality to deteriorate.)

The operator should perform the following recommended duties in developing an operations routine for the plant:

A. Collect samples and perform analyses as recommended (see Section 5) to monitor plant operation and meet any applicable Federal, State or local regulatory agency requirements.

B. Perform maintenance as recommended by equipment manufacturers. (See Equipment Manual.)

C. Check air flow and distribution to ensure proper D.O. and mixing.

D. Fill out log sheets as required.

E. Check that required chemical additives are available in storage tanks. Check and record all feed rates. Ensure delivery at feed point. Recalibrate system as required.

F. Check the pump flow of waste sludge to ensure that proper waste rates are achieved.

G. Add carbon daily to the anoxic tank to replace carbon wasted. (Add to aeration tanks as or if required, to achieve treatment goals).

H. Methanol Feed Rate: Monitor level in the methanol storage tank to confirm daily use.

NOTE: It is strongly recommended that a preventative maintenance program, for all plant equipment, be developed and implemented. Such a preventative maintenance program should contain a schedule for cleaning, lubrication, inspections, adjustments, calibrations and maintaining protective coatings for plant equipment. With proper maintenance, the equipment can be expected to last its full anticipated service life.

OPERATING RECORDS

1. Waste Processed
   a. Influent or effluent flow data
   b. pH to the PACT^{R} unit
   c. Temperature to the PACT^{R} unit

2. Carbon Added
   a. Carbon added to system

3. Polymer Dose Rate
   a. Polymer pump operating time
   b. Change in polymer tank level
   c. Polymer usage per day (neat polymer)

4. Wasting Rate
   a. Pump flow rate and operating duration
   b. Sludge storage tank levels

5. Nutrient Feed Rate
   a. Nutrient pump operating time
   b. Change in nutrient tank level
   c. Nutrient usage per day

6. Records of equipment operation should be kept also.

7. Methanol Feed Rate
   a. Methanol pump operation time
   b. Change in methanol tank level
   c. Methanol use per day

2.0 Lubrication Schedule

Points of Lubrication	Mfr's Recommended Lubr.	Amount	Freq.
Aeration Blower, Aerated Sludge Blower (Roots)			
a) Bearing Lubrication	a) Chevron SRI No. 2 Grease	As Req'd	5 weeks
b) Gear Housing	b) Oil - SAE40 (Winter)		
	Oil - SAE50 (Summer)	Full	See Manual
c) Motor, 40HP, 5HP	c) Baldor Motor Chevron SRI No. 2	As Req'd	3 months
Polymer Pump (Liquiflo)			
a) Motor, 1HP	a) U.S. Motor Chevron SRI No. 2	As Req'd	Yearly
Polymer Mixer (Neptune)			
a) Gear Housing	a) Lithium #2 Grease		
b) Motor, 1/3HP	b) Prelubricated for life of motor	16 ounces	Yearly
Waste Sludge Pump (Dean Brothers)			
a) Motor, 1½HP	a) Lithium #2 Grease	3/4 Full	Yearly
Turbine Mixer (Lightnin)			
a) Gear Drive	a) Mobil SHC626 or Exxon Spartin EP 100	9 Quart	After 1st 200 hrs of operation
Decant Pump, Sludge Pump (Flygt)			
a) Oil Casing	a) SAE10W30 Motor Oil Mobil Whiterex 309 or equivalent paraffin oil	1.1 qt.	Inspect monthly
Decant Winch (Thern)			
a) Gear Case	a) AGMA #8 Oil	Fill to middle oil plug	Inspect Monthly
b) Open Gears	b) NLGI #2 EP Grease	2.0 oz.	Monthly
c) All other points of friction	c) medium wt. oil	As Req'd	Monthly
Chemical Pump (Neptune)			
a) Gear Box	a) SAE90 or AGMA NO. 5EP	As Req'd	As Required
b) Motor, 1/3HP	b) Prelubricated for life of motor	-----	-----
Chemical Mixer (Neptune)			
a) Motor, 1/4 HP	b) Prelubricated for life of motor	-----	-----
Filter Feed Pumps (Goulds, G&L)			
a) Submersible pump/motor	a) Oil - filled w/no further lubrication required		

Section 1

CITRUS COUNTY
CENTRAL SANITARY LANDFILL EXPANSION
Citrus County, Florida

Batch PACT® Leachate Treatment System

OPERATION MANUAL

March 1990

ZIMPRO PASSAVANT
INNOVATIVE WASTE TREATMENT TECHNOLOGY
301 W. Military Rd.
Rothschild, WI 54474
Telephone (715) 359-7211
FAX: (715) 355-3219

Shop Dwg. No. CC-47
Our Job No. 22-081.91

Date 4/4/90

[X] Approved.
[ ] Approved Subject to Notations and Corrections as Indicated.
[ ] Disapproved. Revise as Indicated by Notations and Corrections and Resubmit.

Checking of shop drawing is limited to general design and general arrangement only, and is not intended to be a verification of the items or total material required. Approval shall not relieve the Contractor from the responsibility of details of design, correct dimensions for proper fitting, capacity, performance, construction or any other requirement of the Contract.

POST, BUCKLEY, SCHUH & JERNIGAN, INC.
Consulting Engineers and Planners

By Gary M. Granata

REVIEWED AND APPROVED FOR SUBMITTAL

TO THE ENGINEER

CONE CONSTRUCTORS, INC.

By _____ DATE 3/26

BEFORE ATTEMPTING TO START OR OPERATE THE ZIMPRO UNIT OR ANY OF THE EQUIPMENT OR SYSTEMS COMPRISING THE UNIT, A THOROUGH UNDERSTANDING OF ITS USE AND OPERATION IS MANDATORY. ALL OPERATORS ARE REQUIRED TO THOROUGHLY READ AND UNDERSTAND THIS MANUAL AND ALL MANUALS FURNISHED WITH THE UNIT RELATIVE TO THE EQUIPMENT TO BE OPERATED.

TABLE OF CONTENTS

Section No. 1

INTRODUCTION

1.0 Introduction

Understanding the operation of a Zimpro/Passavant PACT[R] Wastewater Treatment System requires an appreciation for the dual nature of the system. It is both biological and physicochemical. The equipment used to operate and control the process has been designed to allow controlled variation in the influence of either of these dual facets to accommodate a wide range of load conditions while maintaining a given level of superior performance. The process is able to provide this level of superior performance because of a synergistic* interaction between Powdered Activated Carbon (PAC) and the microorganisms or biomass which constitute the physicochemical and biological components of the system, respectively.

All activated sludge systems function by converting suspended, colloidal and dissolved solids into settleable biological floc. The mechanism for this conversion is generally described as occurring in two steps:

1. "Sorption" - Whether adsorption, absorption, chemisorption, electrostatic interaction, or some similarly-named phenomenon, the term refers to the mechanism by which molecules and larger sorbate particles are initially removed from the wastewater stream in which they are suspended or dissolved.

* The combined action of the two together is greater than the sum of the action of the two occurring separately.

2. Stabilization - Incorporation of sorbed materials into the floc. Those materials, largely organic contaminants, which can be decomposed or directly assimilated are said to be biodegradable and are used by the microorganisms for new cell material and to obtain energy. These materials are converted to suspended biomass and harmless carbon dioxide and water when wastewater is properly seeded and aerated in the PACT^R process. Nondegradable materials - largely grit, plastics, etc. - also accumulate in the floc and form a part of the matrix upon which biological growth occurs.

Similar to activated sludge, the biophysical PACT^R process also converts these same suspended, colloidal and dissolved solids into a settleable PAC/biological floc. The conversion mechanism may be considered to be the same: i.e., as occurring in two steps-sorption and stabilization. The differences between PACT^R and activated sludge processes can be viewed as being differences in degree rather than as being entirely different phenomena. Accordingly, whatever one would do to properly operate/control the activated sludge process applies to PACT^R in the same way, and for the same reasons. However, PACT^R has additional characteristics which favor its use when compared with the activated sludge.

1. The synergism referred to earlier allows PACT^R to "sorb" a greater variety of types and sizes of molecules and particles than can be achieved by either carbon (whether powdered or granular) or activated sludge acting alone. This synergistic sorption also occurs much more quickly. This "more and faster" removal allows greater volumetric loadings - whether hydraulic, organic, or both - than are possible with activated sludge. Equivalent loadings produce an effluent of higher quality from PACT^R than would result from activated sludge.

2. Present of the PAC buffers the effect of toxic and shock loads on the biomass. The environment seen by the microorganisms is thus more constant, and "steady state" becomes a more realistic concept than when applied to the typical activated sludge ecosystem.

3. Presence of PAC improves the oxygen environment seen by the biomass organisms. Activated carbon exhibits a rather strong affinity for gaseous oxygen, which may help to account for a greater transfer efficiency of aeration devices when PAC is present in the PACT^{R} system.

From the operating point of view, this means that higher D.O. levels are possible, that demand can be more readily satisfied, and most important, that the micro-environment in and around the individual floc particles can be more easily maintained as truly aerobic, rather than fluctuating between aerobic, micro-aerophilic, and anaerobic - as occurs in activated sludge systems.

2.0 Basis of Design

Table 1-1
Basis of Design
Citrus County, Florida
Leachate Treatment System

	Influent	PACT® Effluent
Flow Rate, GPD	30,000	30,000
pH	6.5-8.5	6.5-8.5
COD, mg/l	6,000	
BOD_5, mg/l	2,000	20
TSS, mg/l	400	20
NH_3, mg/l	400	-
NO_3, mg/l	-	12
SRT, days	15	
HDT, days	4.5	
Carbon Dose, mg/l	650	

Refer to page 1-6 for definition of abbreviated terms.

3.0 Abbreviations

COD	Chemical Oxygen Demand
BOD	Biochemical Oxygen Demand
DO	Dissolved Oxygen
F/M	Food to Microorganism Ratio (Biomass)
HDT	Hydraulic Detention Time Days
MG	Million Gallons
MGD	Million Gallons Per Day
MLSS	Mixed Liquor Suspended Solids
MLVSS	Mixed Liquor Volatile Suspended Solids
MLVCS	Mixed Liquor Volatile Carbon Solids
PAC	Powdered Activated Carbon
Q	Rate of Flow
TCS	Thickened Carbon Sludge Concentration
SRT	Solids Residence Time, Days
Va	Volume of Aeration Basin
W	Waste Flow, MGD
WCS	Waste Rate, lbs/day
P	Phosphorous
TKN	Total Kjeldahl Nitrogen
DOC	Dissolved Organic Carbon
NH_3-N	Ammonia Nitrogen
TSS	Total Suspended Solids
PO_4	Phosphate
NH_3	Ammonia
NO_3	Nitrate

4.0 Drawings

1. Piping Symbols & Identification, 30.1-2343-D-1001
2. P&ID Symbols & Identification, 30.1-2343-D-1002
3. Engineering Flow Diagram, 30.1-2343-D-1003, Sheet 1-4
4. General Arrangement Dwgs, 30.1-2343-D-7302, -7303, & -7304

GRAPHIC SYMBOLS FOR VALVES

- GATE VALVE
- GLOBE VALVE
- BALL VALVE
- BUTTERFLY VALVE
- PLUG VALVE
- LUBRICATED PLUG VALVE
- CHECK VALVE
- DIAPHRAGM VALVE
- HOSE BIB
- NEEDLE VALVE
- ANGLE VALVE
- RELIEF VALVE
- THREE WAY VALVE
- FOUR WAY VALVE
- DIAPHRAGM OPERATED VALVE
- PRESSURE REGULATOR
- SOLENOID OPERATED VALVE
- MOTOR OPERATED VALVE
- PISTON OPERATED VALVE
- LOUVERED DAMPER
- DAMPER
- VALVE SUPPLIED BY ZIMPRO (ZP)
- METERING ORIFICE VALVE

LC – INDICATES THAT A VALVE IS NORMALLY LOCKED IN THE CLOSED POSITION
LO – INDICATES THAT A VALVE IS NORMALLY LOCKED IN THE OPEN POSITION

PIPING SYMBOLS

- PROCESS LINE
- SECONDARY LINE
- CAPILLARY TUBING
- PNEUMATIC TUBING
- ELECTRICAL SIGNAL
- HYDRAULIC SIGNAL
- INSULATED PIPE
- ELECTRICALLY TRACED LINE
- STEAM JACKETED LINE
- ① THREADED CONNECTIONS
- ① FLANGED CONNECTIONS
- ① BUTT WELD CONNECTIONS
- ① SOCKET WELD CONNECTIONS
- ① CONCENTRIC REDUCER
- ① ECCENTRIC REDUCER
- CAPPED PIPE
- HOSE CONNECTION
- BLIND FLANGE
- ① REDUCING FLANGE
- ORIFICE
- SPECTACLE BLIND (CLOSED / OPEN)
- ① UNION
- RUPTURE DISK
- DIAPHRAGM SEAL
- Y STRAINER
- OPEN FLOOR DRAIN
- CLOSED FLOOR DRAIN
- FLOOR TRENCH
- SENSING BULB
- SIGHT FLOW INDICATOR
- SIGHT GLASS
- CONDENSATE TRAP
- SPRAY NOZZLE
- EXPANSION JOINT
- FLEXIBLE CONNECTOR
- PIGTAIL SYPHON
- EXPANSION CHAMBER
- EDUCTOR
- PILOT GAS MIXER
- IN LINE AIR FILTER
- AIR INTAKE FILTER
- SURGE CHAMBER
- VENT WITH WEATHER HOOD
- EXPANSION JOINT (BELLOWS TYPE)
- FILTER (FIL)
- SILENCER (SIL)
- SLIDE GATE
- ROTARY FEEDER
- ① PIPE SUPPORT LOCATION
- ① PIPE SUPPORT DESIGNATION (22)

NOTES:
① NORMALLY APPLIES TO PIPING LAYOUTS ONLY

MISC IDENTIFICATION

ABBREVIATIONS – SERVICE DESIGNATIONS

AIR	A	OXIDIZED LIQUOR	OL
AIR SUPPLY	AS	OXIDIZED LIQUOR & VAPORS	OLV
AMBIENT AIR INLET	AA	OXIDIZED SLUDGE	OS
		OXIDIZED SLUDGE & VAPORS	OSV
BACKWASH	BKH	OXIDIZED WASTE	OW
BOILER BLOWDOWN	BBD	OXIDIZED WASTE & VAPORS	OWV
BOILER FEED WATER	BFW		
BRINE	B	PLANT WATER	PLW
BURNER COMBUSTION AIR	BCA	POTABLE WATER	PW
		PILOT GAS	PG
CARBON SLURRY	CS	PROCESS WATER	PRW
CHEMICAL FEED	CHF	PHOSPHORIC ACID	PA
CHEMICAL RECYCLE	CR	REACTOR SPRAY WATER	RSW
CHEMICAL RECYCLE BLEED	CRB	REGENERATED CARBON	RC
CLARIFIER EFFLUENT	CE	RAW LIQUOR	RL
CONDENSATE	C	RAW LIQUOR & AIR	RLA
		RAW WASTE	RW
DIGESTOR GAS	DG	RAW SLUDGE & AIR	RSA
DIGESTOR OVERFLOW	DO	RECYCLE SLUDGE	RS
DRAIN	D	RAW WASTE & AIR	RWA
EFFLUENT	EFF	REACTOR BLOWDOWN	RBD
EFFLUENT WATER	EW	RECOVERED LIQUOR	RCL
		RELIEF VENT	RV
FEED WATER	FW		
FILTER PRESS EFFLUENT	FPE	SEAL WATER	SW
FILTRATE	F	SLUDGE	S
FLASH STEAM	FS	SOFTENED FEED WATER	SFW
FLUSHING WATER	FW	SOFTENED FEED WATER RETURN	SFR
FUEL OIL	FO	SOLVENT	SOL
FUEL OIL SUPPLY	FOS	STEAM	ST
FUEL OIL RETURN	FOR	SPENT CARBON	SC
FLUIDIZING AIR	FA	SHAFT COOLING AIR	SCA
		SLUDGE COMBUSTION AIR	CA
HOT OIL	HO	SCUM	SCM
HOT WATER	HW	SCRUBBER EFFLUENT	SE
		SODA ASH	SA
INSTRUMENT AIR	IA	VACUUM	VAC
LEACHATE	LTE	VAPORS	V
LIQUID PROPANE GAS	LPG	VENT	VT
LIME SLUDGE	LS	VIRGIN CARBON	VC
LIME	L		
NITRIC ACID	NA	WASTE SLUDGE	WS

ABBREVIATIONS – MATERIALS

BRASS	BS	HASTELLOY	HST
BRONZE	BR	INCONEL	INC
CARBON STEEL	CS	INCOLOY	INL
CAST IRON	CI	KYNAR	KY
CHLORINATED		MALLEABLE IRON	MI
POLYVINYL CHLORIDE	CPVC	POLYPROPYLENE	PP
COPPER	CU	POLYVINYL CHLORIDE	PVC
DUCTILE IRON	DI	REINFORCED	
FIBERGLASS		THERMOSETTING RESIN	RTR
REINFORCED PLASTIC	FRP	SEMI STEEL	SIS
FORGED STEEL	FS	STAINLESS STEEL	SS
GALVANIZED IRON	GI	TANTALUM	TA
GALVANIZED STEEL	GS	TITANIUM	TI

LINE CONTINUATIONS

- INDICATES A LINE GOING TO OR COMING FROM ANOTHER SYSTEM.
- 5 | A | 2 — INDICATES CONTINUATION OF LINE IS ON SHEET NUMBER 5, SAME DRAWING NUMBER, IN ZONE A – 2.
- 501 — INDICATES A FLOW STREAM NUMBER AND/OR DIRECTION.

REV	DATE	BY	DESCRIPTION	CH'KED	APPR'D

PREFABRICATED MODEL B140 BATCH PACT® LEACHATE TREATMENT SYSTEM FOR CITRUS COUNTY, FLORIDA

PIPING SYMBOLS & IDENTIFICATION

DRAWN: Larry J. Doepke
CHECKED: [illegible]
APPROVED: [illegible]
DATE: 2-13-90
SCALE: NONE
NEXT ASSEMBLY: CC-PSYM
SHEET 1 OF 1

ZIMPRO PASSAVANT
ROTHSCHILD, WISCONSIN 54474

DRAWING NUMBER: 30.1-2343-D-1001
REV. 0

INSTRUMENT SYMBOLS

- FIELD MOUNTED INSTRUMENT
- PANEL MOUNTED INSTRUMENT (PANEL FACE)
- PANEL MOUNTED INSTRUMENT (PANEL REAR)
- FIELD PANEL MOUNTED INSTRUMENT (PANEL FACE)
- FIELD PANEL MOUNTED INSTRUMENT (PANEL REAR)
- CONTROL INTERLOCK NOT CONNECTED TO COMPUTER
- CONTROL INPUT INTERLOCK ALSO CONNECTED TO COMPUTER
- CONTROL OUTPUT INTERLOCK ALSO CONNECTED TO COMPUTER
- INTERLOCK BRANCH
- TURBINE METER

INSTRUMENT LETTER DESIGNATIONS

	FIRST LETTER		SUCCEEDING LETTER		
	MEASURED OR INITIATING VARIABLE	MODIFIER	READOUT OR PASSIVE FUNCTION	OUTPUT FUNCTION	MODIFIER
A	ANALYSIS		ALARM		
B	BURNER FLAME		USER'S CHOICE	USER'S CHOICE	USER'S CHOICE
C	CONDUCTIVITY (ELECTRICAL)			CONTROL	
D	DENSITY (MASS) OR SPECIFIC GRAVITY	DIFFERENTIAL			
E	VOLTAGE (EMF)		PRIMARY ELEMENT		
F	FLOW RATE	RATIO			
G	GAGING (DIMENSIONAL)		GLASS		
H	HAND (MANUALLY INITIATED)				HIGH (OPEN)
I	CURRENT (ELECTRICAL)		INDICATE		
J	POWER	SCAN			
K	TIME OR TIME-SCHEDULE			CONTROL STATION	
L	LEVEL		LIGHT (PILOT)		LOW (CLOSED)
M	MOISTURE OR HUMIDITY				MIDDLE OR INTERMEDIATE
N	USER'S CHOICE		USER'S CHOICE	USER'S CHOICE	USER'S CHOICE
O	USER'S CHOICE		ORIFICE (RESTRICTION)		
P	PRESSURE OR VACUUM		POINT (TEST CONNECTION)		
Q	QUANTITY OR EVENT	INTEGRATE OR TOTALIZE			
R	RADIOACTIVITY		RECORD OR PRINT		
S	SPEED OR FREQUENCY	SAFETY		SWITCH	
T	TEMPERATURE			TRANSMIT	
U	MULTIVARIABLE		MULTIFUNCTION	MULTIFUNCTION	MULTIFUNCTION
V	VISCOSITY			VALVE, DAMPER, OR LOUVER	
W	WEIGHT OR FORCE		WELL		
X	UNCLASSIFIED		UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED
Y	USER'S CHOICE			RELAY OR COMPUTE	
Z	POSITION			DRIVE, ACTUATE, UNCLASSIFIED FINAL CONTROL ELEMENT	

RELAYS INCLUDE ALL COMPUTATION, SELECTION, AND TRANSMISSION DEVICES.
FUNCTIONAL SYMBOLS GIVEN BELOW DESIGNATE THE EXACT FUNCTIONS OF THE RELAY.

- [Σ] – ADD
- [Δ] – SUBTRACT
- [×] – MULTIPLY
- [÷] – DIVIDE
- [√] – SQUARE ROOT
- [AVG] – AVERAGE
- [REV] – REVERSE
- [1:3] – RATIO
- [>] – HIGH SELECT
- [<] – LOW SELECT
- [~] – INTEGRATE
- [D] – DERIVATIVE

[I/P] – INPUT/OUTPUT CONVERSION

- E – VOLTAGE
- I – CURRENT
- P – PNEUMATIC
- H – HYDRAULIC
- R – RESISTANCE
- O – ELECTROMAGNETIC OR SONIC
- A – ANALOG
- D – DIGITAL

ABBREVIATIONS

CO	CONTROLLER OUTPUT
PV	PROCESS VARIABLE
SP	SET POINT
L.O.R.	LOCAL/OUT OF SERVICE/REMOTE
S/S	START/STOP
F.C.	FAIL CLOSED
F.O.	FAIL OPEN
E	ENERGIZED STATE
DE	DE-ENERGIZED STATE

REV.	DATE	BY	DESCRIPTION	CH'KED	APPR'D

PREFABRICATED MODEL B140 BATCH PACT® LEACHATE TREATMENT SYSTEM FOR CITRUS COUNTY, FLORIDA

P&ID SYMBOLS AND IDENTIFICATION

DRAWN Larry J. Doepke
CHECKED [illegible]
APPROVED [illegible]
DATE 2-12-90
SCALE NONE
NEXT ASSEMBLY CC-PIDS
SHEET 1 OF 1

ZIMPRO PASSAVANT
ROTHSCHILD, WISCONSIN 54474

DRAWING NUMBER 30.1-2343-D-1002
REV 0

NON POTABLE WATER
1 1/2" GI
CHEMICAL
1/2"
INFLUENT
4" SS
DPI 175
TI 175
HS 180 HOA
SIL
6" CS
8"
AERATION BLOWER
17
TI 185
PI 185
16
10
VACUUM BREAKER
14
8" CS
HS 160 OPEN/CLOSE/AUTO
EV 160
XL 160 CLOSED
22
18
19
1"
1
HS 120 OFF/ON
M
POLYMER TANK
2
15
3
1" CPVC
3/4" SS
25
1/2" SS
HS 130 HOA
PI 125
POLYMER PUMP
4
5
7
HS 135 OPEN/CLOSE/AUTO
XL 135 OPEN
EV 135
1" GI
1/2" GI
MAN/AUTO
HS1 150
RAISE/LOWER
HS2 150
M
ZSLH 150
DECANT WINCH
1" CPVC
1 1/2" GI
HIGH LEVEL
LAHH 140
LSHH 140
8" SS
LSH 140
LSL 140
RUN
XL 175
HS 175 HOA
M
MIXER
BATCH PACT® TANK NO. 1
8
4"
12
DRAIN
HS 190 S/S
6
2" CS
1 1/2"
23
24
TO AERATED SLUDGE STORAGE TANK
3" CS
9
SLUDGE WASTING PUMP
6"
FI 195
13
EV 195
11
4"
OVERFLOW
CYCLE RUN
XL 100
CYCLE START
HS 100
CYCLE STOP
HS 100
HORN SILENCE
HS 110
HORN
XX 110
HS 170 HOA
3"
20
21
4"
TO DECANT EQUALIZATION TANK
43
44
DECANT PUMP

REV	DATE	BY	DESCRIPTION	CH'KED	APPR'D
1	10-12-89	LJD	MISC REVISIONS	DAH	

PREFABRICATED MODEL B140 BATCH PACT® LEACHATE TREATMENT SYSTEM FOR CITRUS COUNTY, FLORIDA

ENGINEERING FLOW DIAGRAM

DRAWN Larry J. Doepke
CHECKED DAH
APPROVED DAH
DATE 10-12-89
SCALE NONE
NEXT ASSEMBLY CC-EFD1
SHEET 1 OF 4

ZIMPRO PASSAVANT
ROTHSCHILD, WISCONSIN 54474
DRAWING NUMBER 30.1-2343-D-1.003
REV. 1

NON POTABLE WATER
1 1/2" GI
CHEMICAL
1/2"
INFLUENT
4" SS
DPI 275
TI 275
SIL
6" CS
HS 280
HOA
8"
AERATION BLOWER
17
TI 285
PI 285
6" CS
16
10
VACUUM BREAKER
14
8" CS
HS 260
OPEN/CLOSE/AUTO
EV 260
XL 260
CLOSED
22
MAN/AUTO
HS1 250
RAISE/LOWER
HS2 250
ZSLH 250
DECANT WINCH
18
19
1"
1
HS 220
OFF/ON
M
POLYMER TANK
2
15
3
1" CPVC
25
1/2" SS
HS 230
HOA
PI 225
4
5
3/4" SS
3/4" SS
POLYMER PUMP
HS 235
OPEN/CLOSE/AUTO
XL 235
OPEN
7
EV 235
1" GI
1/2" GI
1" CPVC
HIGH LEVEL
LAHH 240
LSHH 240
6" SS
LSH 240
LSL 240
RUN
XL 275
HS 275
HOA
M
MIXER
BATCH PACT® TANK NO. 2
8
4"
12
DRAIN
1 1/2" GI
HS 290
S/S
6
1 1/2"
2" CS
3" CS
9
23
24
TO AERATED SLUDGE STORAGE TANK
SLUDGE WASTING PUMP
EV 295
13
11
FI 295
6"
4"
OVERFLOW
HS 270
HOA
3"
20
21
4"
43
44
TO DECANT EQUALIZATION TANK
DECANT PUMP
CYCLE RUN
XL 200
CYCLE START
HS 200
CYCLE STOP
HS 200
HORN SILENCE
HS 210
HORN
XX 210

REV.	DATE	BY	DESCRIPTION	CH'KED	APPR'D
1	12-20-89	LJD	MISC REVISIONS	DAH	RMH

PREFABRICATED MODEL B140 BATCH PACT® LEACHATE TREATMENT SYSTEM FOR CITRUS COUNTY, FLORIDA

ENGINEERING FLOW DIAGRAM

DRAWN Larry J. Doepke
CHECKED RJD DAH
APPROVED RMH
DATE 10-12-89
SCALE NONE
NEXT ASSEMBLY CC-EFD2
SHEET 2 OF 4

ZIMPRO PASSAVANT
ROTHSCHILD, WISCONSIN 54474

DRAWING NUMBER 30.1-2343-D-1003
REV. 1

NON POTABLE WATER
1 1/2" GI
CHEMICAL
1/2"
INFLUENT
4" SS
DPI 375
TI 375
HS 380 HOA
SIL
6" CS
8"
AERATION BLOWER
17
TI 385
PI 385
16
10
VACUUM BREAKER
14
8" CS
HS 360 OPEN/CLOSE/AUTO
EV 360
XL 360 CLOSED
22
18
19
1"
1
HS 320 OFF/ON
M
POLYMER TANK
2
15
3
1" CPVC
HS 330 HOA
25
1/2" SS
PI 325
3/4" SS
POLYMER PUMP
4
5
7
HS 335 OPEN/CLOSE/AUTO
XL 335 OPEN
EV 335
1" GI
1/2" GI
MAN/AUTO
HS1 350
RAISE/LOWER
HS2 350
M ZSLH 350
DECANT WINCH
1" CPVC
1 1/2" GI
HIGH LEVEL
LAHH 340
LSHH 340
8" SS
LSH 340
LSL 340
RUN
XL 375
HS 375 HOA
M
MIXER
HS 390 S/S
6
1 1/2"
2" CS
3" CS
9
23
24
TO AERATED SLUDGE STORAGE TANK
SLUDGE WASTING PUMP
EV 395
FI 395
13
11
6"
4"
8
4"
12
DRAIN
OVERFLOW
HS 370 HOA
3"
20
21
4"
43
44
TO DECANT EQUALIZATION TANK
DECANT PUMP
BATCH PACT® TANK NO. 3

XL 300 CYCLE RUN
CYCLE START
HS 300
CYCLE STOP
HS 300
HORN SILENCE
HS 310
XX 310 HORN

REV.	DATE	BY	DESCRIPTION	CH'KED	APP'D
1	8-2-90	LJD	MISC REVISIONS	DHH	KMD

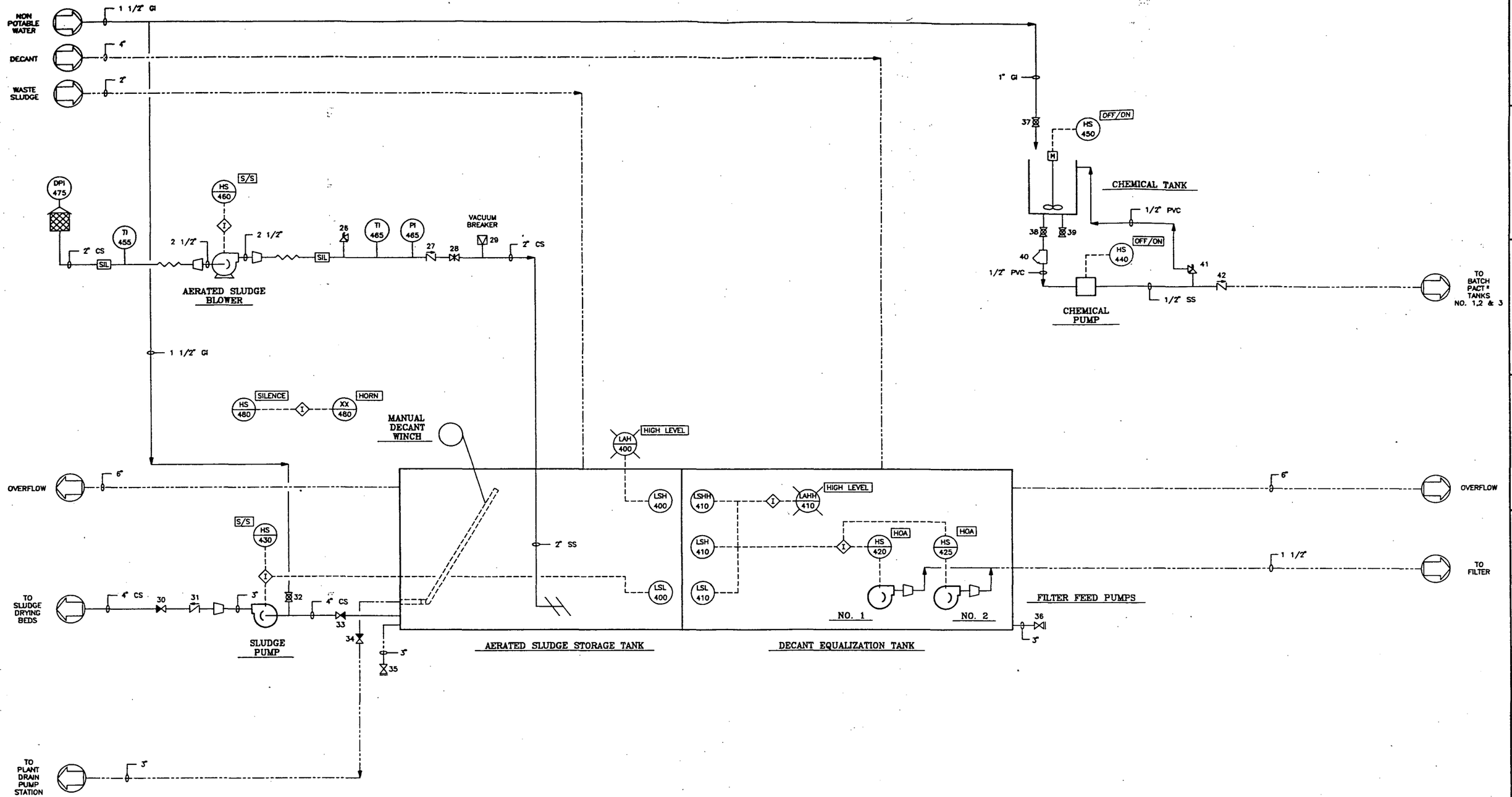
PREFABRICATED MODEL B140 BATCH PACT® LEACHATE TREATMENT SYSTEM FOR CITRUS COUNTY, FLORIDA

ENGINEERING FLOW DIAGRAM

DRAWN Larry J. Doepke
CHECKED DHH
APPROVED
DATE 10-12-89
SCALE NONE
NEXT ASSEMBLY CC-EFD3
SHEET 3 OF 4

ZIMPRO PASSAVANT
ROTHSCHILD, WISCONSIN 54474

DRAWING NUMBER 30.1-2343-D-1003
REV 1



REV.	DATE	BY	DESCRIPTION	CH'KED	APPR'D
1	12-12-89	LJD	MISC REVISIONS	DAH	RMH

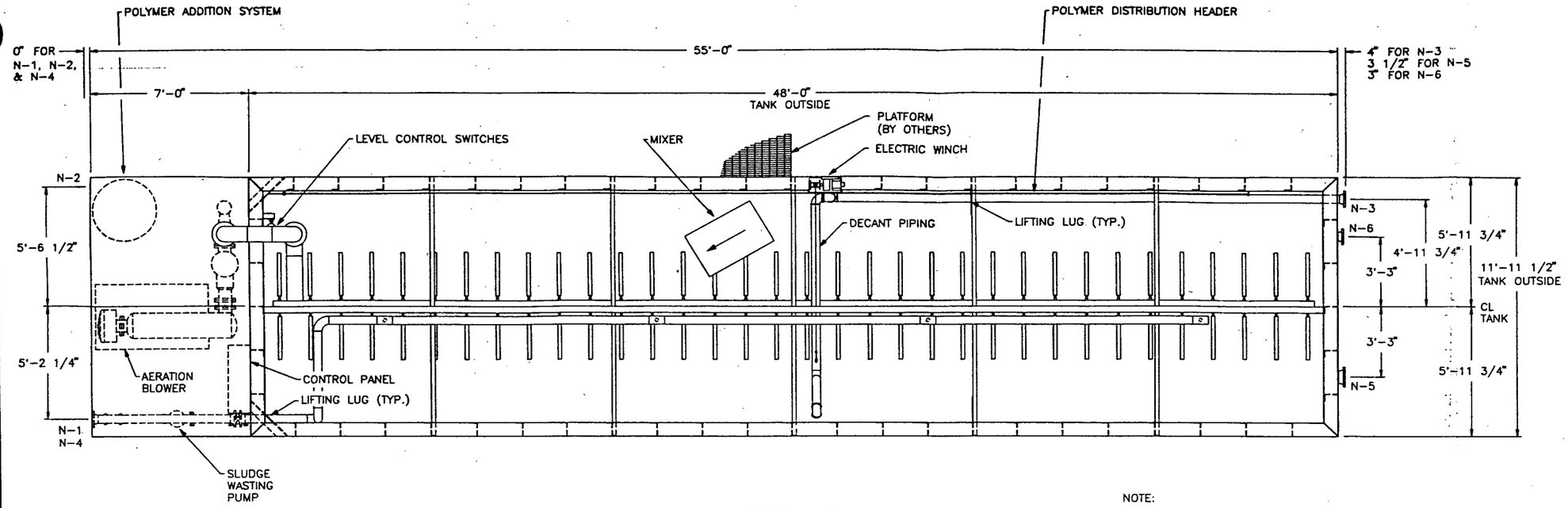
PREFABRICATED MODEL B140 BATCH PACT R
LEACHATE TREATMENT SYSTEM FOR
CITRUS COUNTY, FLORIDA

ENGINEERING FLOW DIAGRAM

DRAWN	Larry J. Doepke
CHECKED	LJD DAH
APPROVED	RMH
DATE	10-12-89
SCALE	NONE
NEXT ASSEMBLY	CC-EFD4
SHEET	4 OF 4

ZIMPRO PASSAVANT
ROTHSCHILD, WISCONSIN 54474

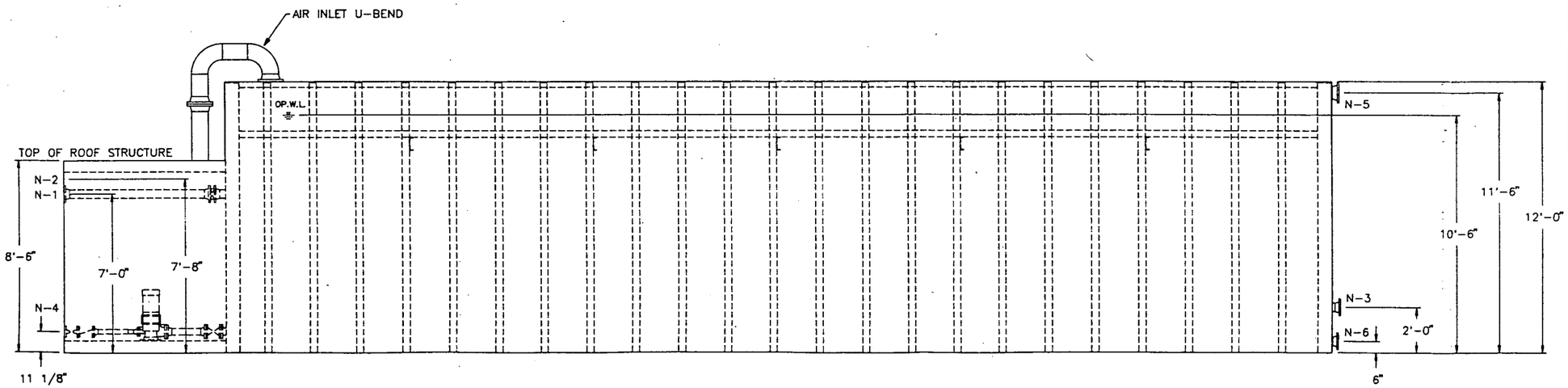
DRAWING NUMBER 30.1-2343-D-1.003 REV. 1



PLAN VIEW

NOTE:

CHEMICAL INLET PIPING (N-7) SHALL BE ROUTED BY CUSTOMER OVER THE TOP OF THE TANK AT ANY LOCATION IN ORDER TO OPTIMIZE THE FIELD PIPING ARRANGEMENT.



ELEVATION VIEW

GENERAL NOTES

1. GENERAL MATERIALS OF FABRICATION:

C.S. –	PLATE	– ASTM A36
	STRUCTURAL SHAPES	– ASTM A36
	PIPING	– ASTM A120
	BOLTS	– ASTM A307
S.S. –	PLATE	– ASTM A240–304L
	PIPING	
	WELDED	– ASTM A312–TP304L
	THREADED	– ASTM A312–TP304
	BOLTS	– 18–8

2. FINISHING:

C.S. – EXTERIOR – SURFACE PREP. – SSPC–SP6
– PRIMER – TNEMEC 66–BLACK HI–BUILD EPOXOLINE ONE COAT @ 4.0 MIL D.F.T.
– PAINT – TNEMEC 71–BLACK ENDURA–SHIELD ONE COAT @ 1.5 MIL D.F.T.

INTERIOR – SURFACE PREP. – SSPC–SP10
– PRIMER – TNEMEC 66–BLACK HI–BUILD EPOXOLINE ONE COAT @ 4.0 MIL D.F.T.
– PAINT – TNEMEC 66–BLACK HI–BUILD EPOXOLINE ONE COAT @ 4.0 MIL D.F.T.

UNDERSIDE – SURFACE PREP. – SSPC–SP10
– PAINT – TNEMEC 46H–413 HI–BUILD TNEME–TAR ONE COAT @ 16.0 MIL D.F.T.

3. APPROXIMATE WEIGHTS:

EMPTY	–	40,000 LBS.
OPERATING	–	415,000 LBS.
FLOODED	–	470,000 LBS.

NOZZLE SCHEDULE

MARK	SIZE	TYPE	DESCRIPTION
N–1	4"	150 LB. FLANGE	TANK INLET
N–2	1 1/2"	NPT UNION	NON–POTABLE WATER INLET
N–3	4"	150 LB. FLANGE	DECANT OUTLET
N–4	2"	150 LB. FLANGE	SLUDGE WASTING OUTLET
N–5	6"	150 LB. FLANGE	TANK OVERFLOW
N–6	4"	150 LB. FLANGE	TANK DRAIN
N–7	BY CUSTOMER	BY CUSTOMER	CHEMICAL INLET

ALL FLANGE BOLT HOLES STRADDLE CENTER LINES, U.O.N.

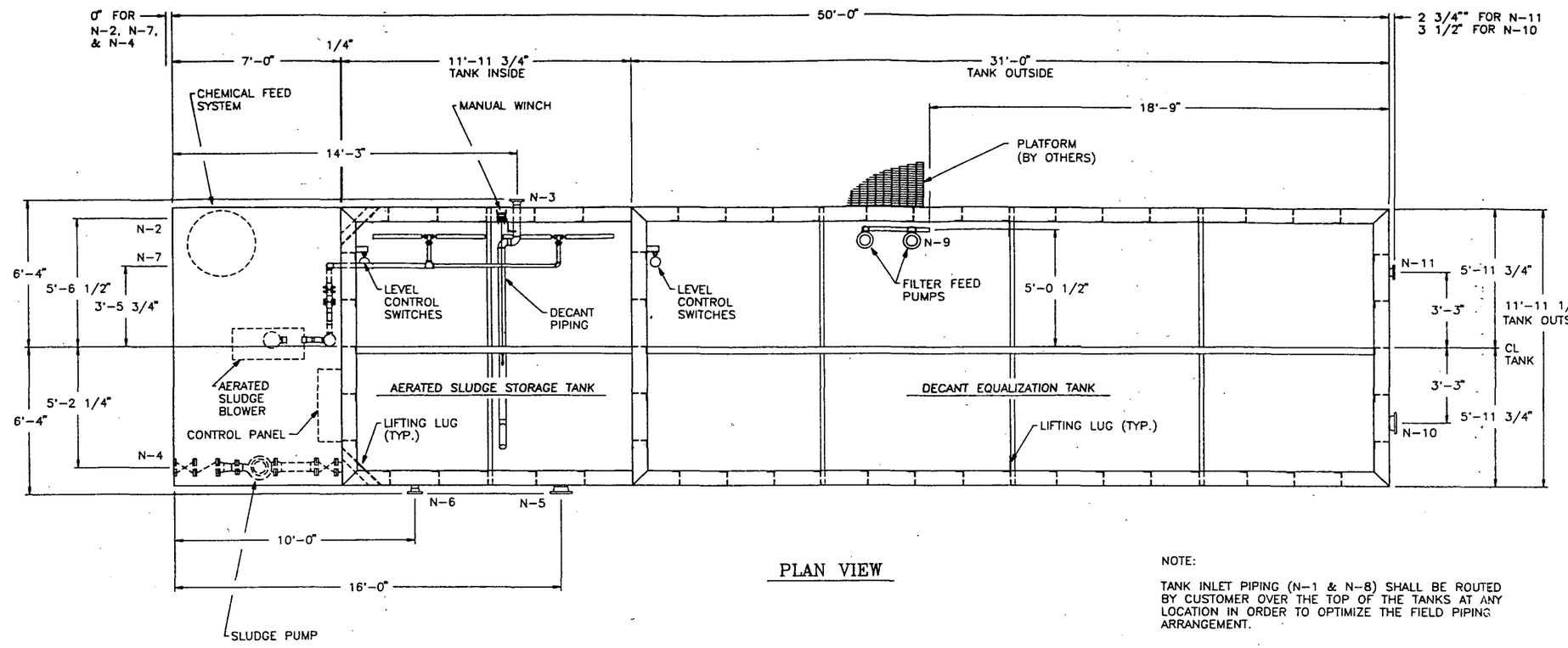
REV.	DATE	BY	DESCRIPTION	CH'KED	APPR'D
3	5-16-90	LJD	MISC REVISIONS	DMK	fmH

PREFABRICATED MODEL B140 BATCH PACT® LEACHATE TREATMENT SYSTEM FOR CITRUS COUNTY, FLORIDA

BATCH PACT® TANK NO. 1
GENERAL ARRANGEMENT

DRAWN	Larry J. Doepke
CHECKED	DMK
APPROVED	fmH
DATE	10-24-89
SCALE	3/8"=1'-0"
NEXT ASSEMBLY	CC-7302
SHEET	1 OF 1
DRAWING NUMBER	30.1-2343-D-7302
REV.	3

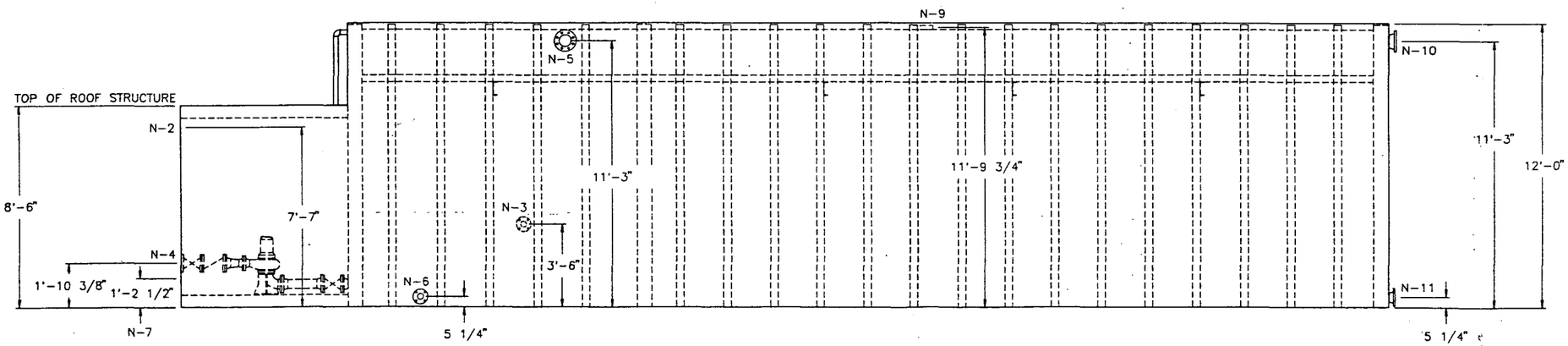
ZIMPRO PASSAVANT
ROTHSCHILD, WISCONSIN 54474



PLAN VIEW

NOTE:

TANK INLET PIPING (N-1 & N-8) SHALL BE ROUTED BY CUSTOMER OVER THE TOP OF THE TANKS AT ANY LOCATION IN ORDER TO OPTIMIZE THE FIELD PIPING ARRANGEMENT.



ELEVATION VIEW

GENERAL NOTES

1. GENERAL MATERIALS OF FABRICATION:

C.S. –	PLATE	– ASTM A36
	STRUCTURAL SHAPES	– ASTM A36
	PIPING	– ASTM A120
	BOLTS	– ASTM A307
S.S. –	PLATE	– ASTM A240-304L
	PIPING	
	WELDED	– ASTM A312-TP304L
	THREADED	– ASTM A312-TP304
	BOLTS	– 18-8

2. FINISHING:

C.S. – EXTERIOR
- SURFACE PREP. – SSPC-SP6
- PRIMER – TNEMEC 66-BLACK HI-BUILD EPOXOLINE ONE COAT @ 4.0 MIL D.F.T.
- PAINT – TNEMEC 71-BLACK ENDURA-SHIELD ONE COAT @ 1.5 MIL D.F.T.

INTERIOR
- SURFACE PREP. – SSPC-SP10
- PRIMER – TNEMEC 66-BLACK HI-BUILD EPOXOLINE ONE COAT @ 4.0 MIL D.F.T.
- PAINT – TNEMEC 66-BLACK HI-BUILD EPOXOLINE ONE COAT @ 4.0 MIL D.F.T.

UNDERSIDE
- SURFACE PREP. – SSPC-SP10
- PAINT – TNEMEC 46H-413 HI-BUILD TNEME-TAR ONE COAT @ 16.0 MIL D.F.T.

3. APPROXIMATE WEIGHTS:

EMPTY	–	36,000 LBS.
OPERATING	–	372,000 LBS.
FLOODED	–	421,000 LBS.

NOZZLE SCHEDULE

MARK	SIZE	TYPE	DESCRIPTION
N-1	BY CUSTOMER	BY CUSTOMER	AERATED SLUDGE STORAGE TANK INLET
N-2	1 1/2"	NPT UNION	NON-POTABLE WATER INLET
N-3	3"	150 LB. FLANGE	DECANT OUTLET
N-4	4"	150 LB. FLANGE	SLUDGE OUTLET
N-5	6"	150 LB. FLANGE	AERATED SLUDGE STORAGE TANK OVERFLOW
N-6	3"	150 LB. FLANGE	AERATED SLUDGE STORAGE TANK DRAIN
N-7	1/2"	NPT UNION	CHEMICAL OUTLET
N-8	BY CUSTOMER	BY CUSTOMER	DECANT EQUALIZATION TANK INLET
N-9	1 1/2"	MALE NPT	DECANT EQUALIZATION TANK EFFLUENT OUTLET
N-10	6"	150 LB. FLANGE	DECANT EQUALIZATION TANK OVERFLOW
N-11	3"	150 LB. FLANGE	DECANT EQUALIZATION TANK DRAIN

ALL FLANGE BOLT HOLES STRADDLE CENTER LINES, U.O.N.

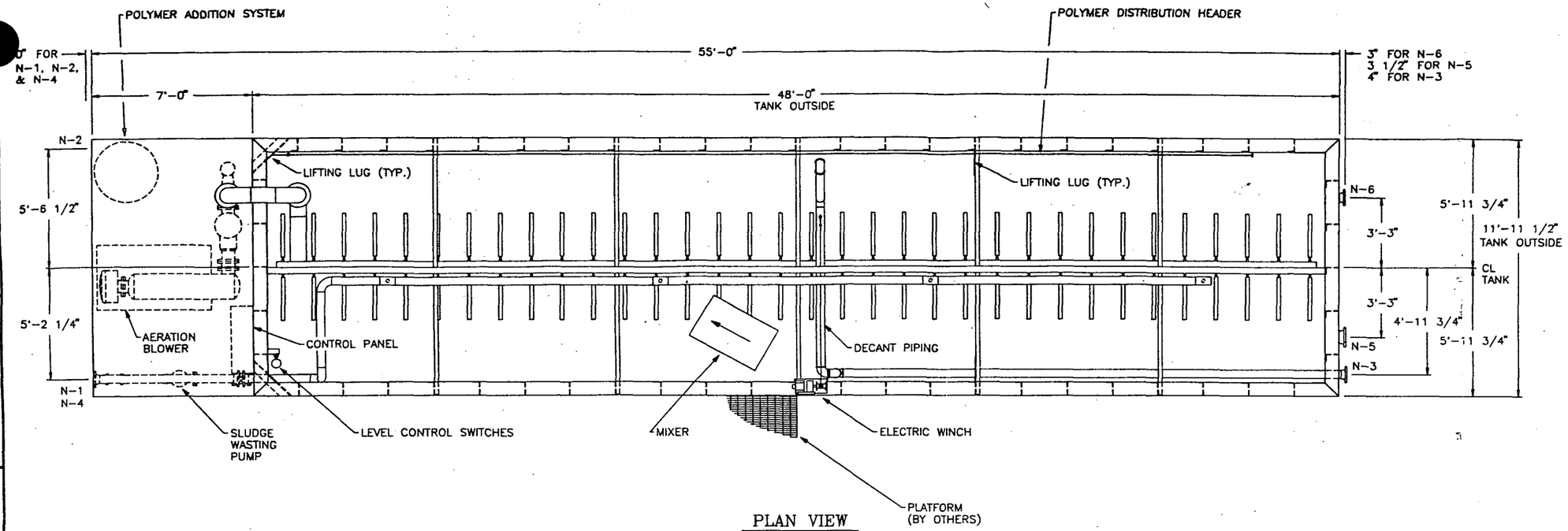
REV	DATE	BY	DESCRIPTION	CHKED	APPRD
E	10-27-89	LJD	MISC REVISIONS	MH	RWH

PREFABRICATED MODEL B140 BATCH PACT® LEACHATE TREATMENT SYSTEM FOR CITRUS COUNTY, FLORIDA

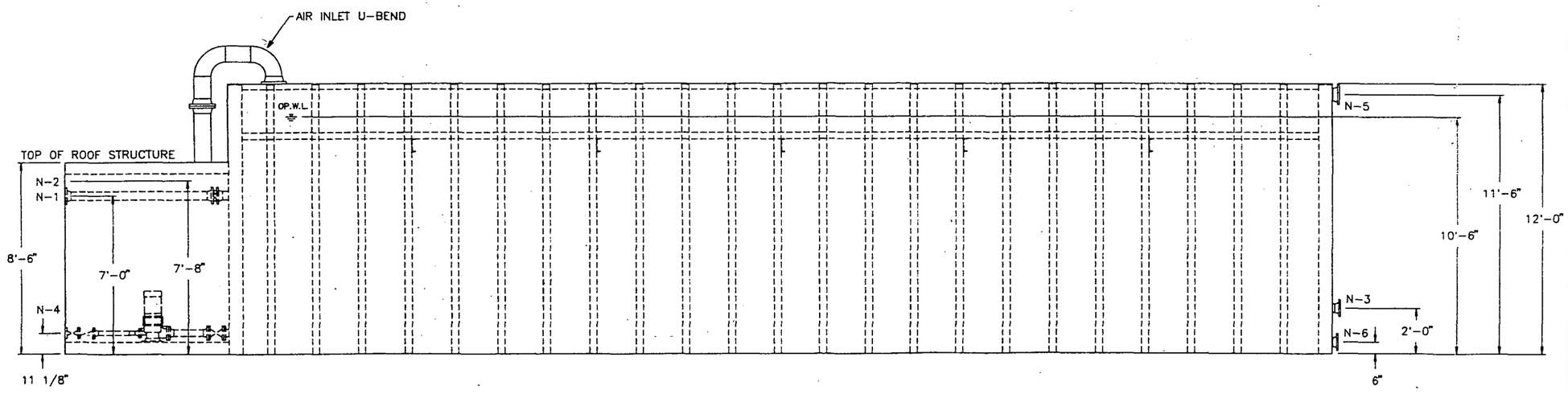
A.S.S.T. / DECANT EQUALIZATION TANK GENERAL ARRANGEMENT

DRAWN	Larry J. Doepke
CHECKED	DJT
APPROVED	RWH
DATE	10-24-89
SCALE	3/8"=1'-0"
NEXT ASSEMBLY	CC-7304
SHEET	1 OF 1
DRAWING NUMBER	30.1-2343-D-7304
REV	2

ZIMPRO PASSAVANT
ROTHSCHILD, WISCONSIN 54474



PLAN VIEW



ELEVATION VIEW

GENERAL NOTES

1. GENERAL MATERIALS OF FABRICATION:

C.S. –	PLATE	– ASTM A36
	STRUCTURAL SHAPES	– ASTM A36
	PIPING	– ASTM A120
	BOLTS	– ASTM A307
S.S. –	PLATE	– ASTM A240–304L
	PIPING	
	WELDED	– ASTM A312–TP304L
	THREADED	– ASTM A312–TP304
	BOLTS	– 18–8

2. FINISHING:

C.S. – EXTERIOR – SURFACE PREP. – SSPC–SP6
– PRIMER – TNEMEC 66–BLACK HI–BUILD EPOXOLINE ONE COAT @ 4.0 MIL D.F.T.
– PAINT – TNEMEC 71–BLACK ENDURA–SHIELD ONE COAT @ 1.5 MIL D.F.T.

INTERIOR – SURFACE PREP. – SSPC–SP10
– PRIMER – TNEMEC 66–BLACK HI–BUILD EPOXOLINE ONE COAT @ 4.0 MIL D.F.T.
– PAINT – TNEMEC 66–BLACK HI–BUILD EPOXOLINE ONE COAT @ 4.0 MIL D.F.T.

UNDERSIDE – SURFACE PREP. – SSPC–SP10
– PAINT – TNEMEC 46H–413 HI–BUILD TNEME–TAR ONE COAT @ 16.0 MIL D.F.T.

3. APPROXIMATE WEIGHTS:

EMPTY	–	40,000 LBS.
OPERATING	–	415,000 LBS.
FLOODED	–	470,000 LBS.

NOZZLE SCHEDULE

MARK	SIZE	TYPE	DESCRIPTION
N–1	4"	150 LB. FLANGE	TANK INLET
N–2	1 1/2"	NPT UNION	NON–POTABLE WATER INLET
N–3	4"	150 LB. FLANGE	DECANT OUTLET
N–4	2"	150 LB. FLANGE	SLUDGE WASTING OUTLET
N–5	6"	150 LB. FLANGE	TANK OVERFLOW
N–6	4"	150 LB. FLANGE	TANK DRAIN

ALL FLANGE BOLT HOLES STRADDLE CENTER LINES, U.O.N.

REV.	DATE	BY	DESCRIPTION	CH'KED	APPR'D
3	10-30		MISC REVISIONS	DAK	RMH

PREFABRICATED MODEL B140 BATCH PACT® LEACHATE TREATMENT SYSTEM FOR CITRUS COUNTY, FLORIDA

BATCH PACT® TANKS NO. 2 & 3
GENERAL ARRANGEMENT

DRAWN	Larry J. Doepke
CHECKED	DAK
APPROVED	RMH
DATE	10-24-89
SCALE	3/8"=1'-0"
NEXT ASSEMBLY	CC-7303
SHEET	1 OF 1

ZIMPRO PASSAVANT
ROTHSCHILD, WISCONSIN 54474

DRAWING NUMBER 30.1-2343-D-7303 REV. 3

Section No. 2

PROCESS DESCRIPTION

1.0 Narrative

The Citrus County Central Sanitary Landfill Leachate Treatment System consists of three (3) Model B140 Batch PACT^{R} structures, one (1) Sludge Storage/Decant Equalization Structure, and three (3) decant pumps. Each Model B140 structure includes aeration tank, diffusers, aeration blower, submersible mixer, electric decant winch, waste sludge pump, polymer feed system, level sensors, and control panel. The sludge storage/decant equalization structure includes sludge storage tank, diffusers, aerated sludge blower, manual decant winch, sludge pump, chemical feed system, decant equalization tank, filter feed pumps, level sensors, and control panel.

The Engineering Flow Diagrams, found in Section 1, depict the process flow scheme for the batch PACT^{R} Leachate Treatment System.

The PACT^{R} Leachate Treatment System is a biological treatment and physical adsorption process combined into a single unit process. Powdered activated carbon is added to the conventional activated sludge process to adsorb non-biodegradable compounds and provide improved removal of biodegradable materials.

The Citrus County Central Sanitary Landfill Batch PACT^{R} system is designed to treat leachate generated at the site. The leachate is pumped to one of three aeration tanks where the required nutrient (phosphorous) and carbon are added to maintain optimum treatment. When the aeration tank is filled with leachate, treatment proceeds in the following sequence:

Aerobic treatment – One positive displacement blower provides air to the coarse bubble diffusers for dispersion throughout the aeration tank. In this step, carbonaceous BOD is removed and nitrification takes place via biological assimilation. Also, nondegradable, adsorbable material is adsorbed on the powdered carbon.

Anoxic treatment – With the aeration blower off, a submerged mixer provides mechanical agitation. In this step, denitrification takes place in an anoxic (without air) environment.

Clarification – The aeration tank also acts as a clarifier. After aerobic and anoxic treatment steps, the aeration blower is restarted and the polymer pump provides polymer from a 100 gallon tank. The polymer is added to the aeration tank and allowed to mix for 10–15 minutes. The blower is then shut off and the entire tank is allowed to settle.

Decantation – After a settling time of approximately 30–60 minutes, the clear liquid is decanted off the top of the tank and pumped to the decant equalization tank.

Sludge Removal – Solids concentrations are controlled in the aeration tank by periodic wasting of the sludge to the aerated sludge storage tank via the sludge wasting pump. Sludge is normally wasted from the aeration tank after decanting. Virgin carbon must be added to the aeration tank to replace carbon removed from the tank during sludge wasting. Virgin carbon is added directly to the aeration tank daily via 50 pound water soluble bags.

2.0 Flow To The Aeration Tank

A. Leachate:

Three pumping stations supply leachate through force mains to the three batch PACT[R] aeration tanks for treatment.

B. Plant Drains:

One plant drain pump station supplies wastewater through force mains to the batch PACT[R] aeration tanks for treatment.

C. Carbon Addition:

Make-up powdered activated carbon is added to the aeration tank using 50 pound water soluble bags as needed. The 50 pound bags are placed directly in the aeration tank after first removing the outer paper bag. Aeration blower or submerged mixer should be operating to provide mixing.

D. Nutrient Addition:

Nutrient (phosphorous) requirements are provided by adding phosphoric acid as needed via the chemical feed system.

3.0 Aerobic/Anoxic Treatment

Aerobic Reaction (Carbonaceous Oxidation and Nitrification):

In the aeration tank, the wastewater will aerate in the presence of powdered activated carbon, bio-mass and non-volatile material (ash) to remove BOD and COD and convert ammonia to nitrate (nitrification).

The bacteria in the aeration tank remove the organic compounds that are measured by the BOD analysis by consuming it as food. This is called metabolism. Metabolism consists of two separate on-going processes; respiration and synthesis. The desired bacteria are aerobic. That is, they require free oxygen dissolved in the mixed liquor for respiration. In the respiration process the bacteria consume organic compounds and oxygen dissolved in the wastewater and convert (oxidize) it to carbon dioxide and water. The bacteria also reproduce, using a portion of the food for synthesis or new cell growth. The required oxygen is made available to the bacteria by bubbling air into the water (mixed liquor) in the aeration tank. Some of the oxygen is dissolved in the water and in this dissolved form becomes available to the bacteria. The available oxygen is measured as D.O. (dissolved oxygen) and should be at least 2 PPM to maintain adequate biological activity and promote the predomination of the proper species of aerobic bacteria. Low D.O. levels (less than 1.0) as well as other factors, promote the reproduction of filamentous, poorly settling bacteria.

The oxygen requirement for biological uptake is provided by one positive displacement aeration blower. The blower will provide compressed air through coarse bubble diffusers located throughout the aeration tank.

Anoxic Reaction (Denitrification):

In the anoxic step, the wastewater in the aeration tank is subjected to anoxic (no oxygen) conditions. The aeration blower is shut off and a 10 HP submerged mixer provides the required mixing of the wastewater. Under these conditions, nitrate in the wastewater is removed by converting nitrate to nitrogen gas (denitrification).

Sludge Wasting:

It is in the aeration tank that the substrate in the wastewater is consumed as food by the biomass or absorbed on the carbon. The substrate represents food, and air is added to supply oxygen needed for respiration. The micro-organisms will multiply (increase in population) as long as there is flow (substrate) to the aeration tank, oxygen and the appropriate environmental conditions such as temperature, pH, etc., exist. The solids resident time (SRT) is determined by the mass of solids removed from the aeration tank each day. The SRT must be controlled at some desirable value. In this case 15 days is the target SRT so the biological solids retain good settling characteristics. This means that every day one-fifteenth of the solids in the aeration tank must be removed from the system.

The required solids removal is accomplished by pumping sludge to the aerated sludge storage tank. The biomass fraction will be replaced by new cell growth and the carbon fraction will be replaced by adding virgin carbon.

Clarification:

In the Batch PACT^{R} system the aeration tank also acts as the clarifier. This is accomplished by shutting off the aeration blower after polymer addition and allowing the solids to settle. The treated effluent from the system is decanted from the top of the aeration tank and pumped to the decant equalization tank via the decant pump.

4.0 Polymer Addition

Polymer is fed to the aeration tank using a constant speed gear pump for injection of a dilute polymer solution from a mixed tank.

The pump flow is constant at 7 gpm, therefore polymer addition is controlled by regulating the amount of time that the pump is operating. The polymer injection pump can be operated either manually or automatically. In the automatic mode the polymer pump run time is set in register C03 of the controller located in the batch PACT^{R} control panel.

A 100 gallon tank is supplied to hold a minimum of one day's usage of polymer solution at a polymer concentration of ½%. The polymer dose required is estimated to be 2 mg/l which represents approximately 3 minutes of polymer injection pump operation per cycle.

5.0 Carbon Addition

The addition of make-up powdered activated carbon (PAC) is done intermittently (one per day for example) to replace PAC losses and PAC wasted from the system. Make-up carbon is added directly to the aeration tank from 50 pound water soluble bags.

Carbon addition is manually carried out each day with the aeration blower operating to insure good mixing. A dust mask should be used when working with the powdered activated carbon.

6.0 Nutrient Addition

A nutrient solution of phosphoric acid supplies the required phosphorous to the aeration tank. The chemical feed system is required to supply the nutrient concentration required to maintain optimum biomass growth. Note that nutrient nitrogen is available in the leachate influent as ammonia (NH_3).

Calculation of the amount of nutrient required is detailed in Section 3.

7.0 Aerated Sludge Storage Tank

The aerated sludge storage tank is used to hold wasted mixed liquor solids under aerobic conditions until they can be pumped to sludge drying beds. Diffusers are used to aerate and mix the contents of the aerated sludge holding tank and may decrease the volatile solids content by up to one third. To maximize the solids storage capacity of the holding tank a decant pipe is used to decant off clear supernatant. Decanting is accomplished manually. In the decanting cycle, the aerated sludge blower is shut off and the tank contents are then allowed to settle. If the tank level is sufficient to warrant decanting, the swing-pipe suction line is positioned so the open end is just above the sludge blanket level, the decant discharge valve (No. 34) is opened, and the tank is decanted to the plant drain pump station. When the supernatant level falls to near the sludge blanket or when solids are carried out of the tank, the decant discharge valve (No. 34) is closed and the aerated sludge blower is restarted.

8.0 Decant Equalization Tank

The decant equalization tank is used to store the treated leachate from each of the three aeration tanks. The leachate is then pumped at a steady flow rate to a tertiary filter (supplied by others) by one of two filter feed pumps. After filtration, the treated leachate is discharged to the landfill percolation pond site.

9.0 Equipment List

Aeration Tank	Capacity	45,000 gallons
	Dimensions	12'Wx12'Hx48'L
	HDT (Nominal)	108 hrs (4.5 days)
	Quantity	3
Decant Equalization Tank	Capacity	29,000 gallons
	Dimensions	12'Wx12'Hx31'L
	HDT (Nominal)	23 hrs (0.97 days)
	Quantity	1
Aerated Sludge Storage Tank	Capacity	11,200 gallons
	Dimensions	12'Wx12'Hx12'L
	Quantity	1
Aeration Blowers	Capacity	1000 SCFM
	Disch. Press.	6.0 PSI
	Motor	40 HP / 460 V
	Quantity	3
Aerated Sludge Blower	Capacity	60 SCFM
	Disch. Press.	6.0 PSI
	Motor	5 HP / 460 V
	Quantity	1
Electric Decant Winches	Capacity	700 lbs
	Motor	½ HP / 115 V
	Quantity	3
Submersible Mixers	Capacity	9400 GPM
	Motor	10 HP / 460 V
	Quantity	3

Waste Sludge Pumps	Capacity	70 GPM @ 30' TDH
	Motor	1½ HP / 460 V
	Quantity	3
Decant Pumps	Capacity	200 GPM @ 12' TDH
	Motor	2.1 HP / 460 V
	Quantity	3
Filter Feed Pumps	Capacity	30 GPM @ 23' TDH
	Motor	½ HP / 460 V
	Quantity	2
Sludge Pump	Capacity	180 GPM @ 12' TDH
	Motor	2.1 HP / 460 V
	Quantity	1
Polymer Systems	Quantity	3
	Pump	7 GPM/1 HP/460 V
	Tank	100 Gallons
	Mixer	1/3 HP/115 V
Chemical Feed System	Quantity	1
	Pump	11 GPH/1/3 HP/115 V
	Tank	100 Gallons
	Mixer	¼ HP/115 V
Control Panels	Quantity	4
	Size	(3) @ 48"Hx37"Wx12"D
		(1) @ 36"Hx31"Wx8"D
	Features	(3) w/Modicon PC-0085 Programmable Controllers

Section No. 3

PROCESS CONTROL

1.0 Narrative

A. A control scheme - whether for activated sludge or PACT^R - should maintain a balance between sorption and stabilization. Such schemes will also be based on sludge wasting (and addition of virgin carbon in the required amounts).

B. While PAC affects both sorption and stabilization, its major contribution is to the sorptive capacity of the system. PAC concentration or dosage levels will thus be primarily determined by the pollutant removals required, which may be measured by effluent color or COD, for example.

C. Control of the PACT^R system thus involves two different but related elements: Addition of virgin PAC; and wasting of excess Mixed Liquor Suspended Solids (MLSS) to solids disposal.

In PACT^R systems where the PAC is not regenerated and returned to the system, sufficient virgin powdered activated carbon (PAC) must be added either continuously or incrementally to replace whatever PAC has been intentionally wasted or lost in the effluent. This amount is termed the Carbon Dose and is defined as:

$$\text{Carbon Dose (mg/l)} = \frac{\text{(MLVCS mg/l) HDT}}{\text{SRT}}$$

Where MLVCS = Mixed liquor volatile carbon solids, mg/l

HDT = Hydraulic detention time, days

SRT = Solids residence time, days

HDT may be further defined as the time required for a unit volume influent to the aeration tank to pass through the tank, expressed in days:

$$\text{HDT, days} = \frac{\text{Va (volume of aeration tank, gallons}}{\text{Q (rate of flow into tank, gallons/day)}}$$

For this example: Va = 45,000 gallons

Q = 10,000 gallons/day

HDT = 4.5 days

SRT is defined as:

$$\text{SRT, days} = \frac{\text{Total solids in PACT}^{R}\text{ system, lbs.}}{\text{Waste rate, lb./day}} = \frac{\text{MLSS}}{\text{WCS}}$$

The weight of total solids is calculated as follows:

MLSS lbs. = Va (volume of aeration tank, MG) x 8.34 x MLSS, mg/l

(8.34 is the well known factor which converts concentrations in mg/l and flows in MGD into lbs/day.)

Solids wasting is directly from the aeration tank. The waste rate is calculated as follows (neglecting solids lost to the effluent):

$$\text{WCS, Waste Rate; lb. solids to be wasted per day} = \frac{\text{MLSS, mg/l x 8.34 x Va, MG}}{\text{SRT (desired)}}$$

Since the aeration tank is well mixed, there is a direct relationship between solids and volume in the mixed liquor.

If wasting is to be accomplished directly from the aeration tank:

$$\text{W, waste flow rate; GPD} = \frac{\text{Va, Gal.}}{\text{SRT}}$$

NOTE: If decant effluent suspended solids are significant, they must be considered or over wasting will result and the actual SRT will be lower than desired. In this case:

$$WCS = \frac{MLSS \times 8.34 \times Va}{SRT} - Q, MGD \times 8.34 \times Effl.\ SS, mg/l$$

Wasting is accomplished by pumping solids to the aerated sludge storage tank. This requires that the thickened carbon solids concentration (TCS) be known. So:

$$W, GPD = \frac{WCS, lb/day \times 10^6}{8.34 \times TCS, mg/l}$$

Wasting can also be done directly from the aeration tank during aeration. In that case, the MLSS, mg/l concentration is substituted for TCS in the above equation.

Once the sludge waste pump flow rate is known, the time required for wasting can be calculated.

$$\frac{W\ GPD}{70\ GPM\ (for\ example)} = \frac{Min.}{Day}\ Total\ time\ solids\ must\ be\ pumped$$

Volatile carbon must be added each day to replace carbon wasted and that lost in the effluent.

Carbon in the mixed liquor is measured as carbon by the PAC/BIO test procedure.

$$Carbon\ Wasted, = W, GPD \times MLVCS \times 8.34 \times 10^{-6}$$

Carbon lost in the effluent is estimated as follows:

$$Lb/day = Q, MGD \times 8.34 \times Eff.\ SS, mg/l \times \frac{MLVCS, mg/l}{MLSS, mg/l}$$

Make-up volatile carbon must replace carbon lost to these two areas.

Powdered activated carbon can vary in inert (ash) content from 5% to 35%. Therefore;

$$PAC\ added = \frac{Lbs.\ volatile\ carbon\ added/day}{1 - \frac{\%\ ash}{100}}$$

Once the appropriate "initial charge" of PAC has been added to the system, only makeup carbon need be added. Makeup is necessary to compensate for effluent PAC losses and the amount of spent PAC wasted from the system.

Wasting of sludge from the system is done to maintain a biological Sludge Age which will permit growth of desired flora, such as nitrifying bacteria, including Nitrobacter but not so great as to provide conditions suitable for the excess growth of scum-foam producing organisms such as Nocardia. In this respect - control by solids wasting - PACT^{R} is exactly the same as activated sludge, as one would expect based on the inherently biological nature of PACT^{R}. Sludge Age is defined as the age, in days, which results when the system total solids mass is divided by the daily rate at which solids are removed from the system. This parameter is thus seen to be the same as Solids Residence Time (SRT) or Mean Cell Residence Time (MCRT) often seen in wastewater treatment reference literature. The initial goal for this application will be a Sludge Age of 15 days. This figure may change as more information and operating data become available, and as loadings to the plant change. Seasonal changes in the Sludge Age or SRT may also be required to achieve the effluent quality desired.

D. In addition to the control of the system as just discussed, the following operational parameters should be monitored closely:

1. **Dissolved Oxygen (D.O.)** - It is recommended that D.O. levels be kept at a minimum of 2 mg/l at all sample locations in the aeration tank, and should not be allowed to drop to less than 1.0 mg/l for any reason. Levels below 1.0 mg/l may result in poor effluent quality which may persist well beyond restoration of proper D.O. levels.

2. **Polymer Dosage** – It will be necessary to determine an approximate range of polymer dosage based on bench-scale testing (which can and should be done on-site by prospective polymer suppliers). The polymer is added to increase capture of fine solids which would otherwise be lost in the effluent, and to improve sludge settleability overall. When scaling up to plant requirements, calculations should be based on the aeration tank volume.

Polymer required (lbs/day) = (Volume MG) (8.34) (mg/l)*

Normally 2 mg/l of Cationic polymer is required to produce an acceptable effluent suspended solids concentration.

*mg/l = dosage determined from bench jar tests.

3. **Nutrient Addition** – The supplemental addition of nutrient phosphorous may be required to supply this key element to the biomass. The addition of nutrient nitrogen is not required since it is supplied in the leachate influent as ammonia (NH_3).

Bacteria require a source of soluble nitrogen and phosphorous for cell growth and reproduction. If there are insufficient nutrients in the incoming wastewater, two problems can result:

a. Filamentous bacteria (fungi) will predominate resulting in poor settleability of the mixed liquor (bulking sludge).
b. Biological activity will be capable of assimilating only a fraction of the organic loading (food) to the PACT^R system. BOD/COD reductions will be hindered.

If excess nutrients are fed to the PACT^R system to overcompensate for a nutrient deficiency, a large fraction of these nutrients may not be incorporated into the mixed liquor solids and will pass from the PACT^R system in the effluent. It is important to monitor the effluent for excess nutrients, and then make adjustments to the amount of nutrients added. A change of no more than 5 percent (5%) per week is advised to avoid adverse effects on the biomass.

Operational experience with BOD removal at the lowest possible nutrient dosage rates will allow the operator to properly control the addition rate. Soluble ammonia-nitrogen (NH_3-N) and soluble phosphorous as measured in the PACT^R system effluent need not exceed 1-2 mg/l. Many systems have demonstrated adequate treatment at 25-50 percent of these values.

Nutrient requirements are based on BOD_5 loading to the PACT^R system. For every 100 parts of BOD_5 in the incoming wastewater, 5 parts of soluble nitrogen and 1 part of phosphorous need to be available to the biomass. Nitrogen in the incoming wastewater should be determined by a TKN (Total Kjeldahl Nitrogen) analysis in the laboratory. Phosphorous in the incoming wastewater should be determined as total phosphorous.

To determine the amount of commercial chemical nutrients to be added to correct a nutrient deficiency, the following information is required:

a. PACT^R influent BOD_5, mg/l
b. PACT^R influent TKN, mg/l
c. PACT^R influent P, mg/l (as total phosphorous)
d. Average daily wastewater flow, mg/d
e. Suggested weight ratio, $BOD_5/N = 100/5 = 20$
f. Suggested weight ratio, $BOD_5/P = 100/1 = 100$

g. Atomic weight ratio for commercial nutrient chemicals used (given in Table 3-1)

h. Density ratios for liquid chemicals (given in Table 3-1)

Examples of nutrient dosage calculations are given in Section 3-2, F.

E. Operation and control of the PACT^R system have been discussed in terms of the following parameters:

1. Wasting (proper SRT)
2. Carbon addition (Carbon Dose)
3. D.O. control (minimum of 2 mg/l D.O.)
4. Polymer or coagulant dosage (proper feed rate for actual plant flows).
5. Maintaining proper nutrient (Nitrogen and Phosphorous) levels.

Any problems which arise during operation of a PACT^R Wastewater Treatment System will be found to have its cause in one or more of these five parameters.

2.0 Sample Calculations

A. Biomass Level

The influent stream contains pollutants (suspended, colloidal, and dissolved solids) which the PACT^R system must remove to meet the required plant effluent limitations. The portion of the waste measured as COD will be adsorbed by the PAC in the aeration system. The portion of the waste measured as BOD will have to be stabilized (converted to CO_2, water and cell structure material) biologically.

The question arises: How much biomass is needed to stabilize a given amount of BOD loading? A commonly used parameter which relates influent loadings and the biomass which results from those loadings is the F/M ratio. F = food or BOD loading, M = microorganism mass.

F/M may be applied to PACT^R much in the same way it is used in activated sludge systems. The normal activated sludge F/M is in the range of 0.2 - 0.5.

To calculate a target MLVSS the F/M ratio chosen should be within this range. Example:

1. Let's assume that the BOD loading to the plant is projected at 2,000 mg/l.

2. The average plant flow is projected at 30,000 gal/day (GPD) or 0.030 million gallons/day (MGD). For each of the three aeration tanks, the average flow is then 10,000 GPD or 0.010 MGD.

3. The volume of each of the three aeration tanks is 0.045 million gallons (MG).

4. Convert BOD loading in mg/l to lbs/day.
   F = 2,000 mg/l x 8.34 lbs/gal x 0.10 MGD = 1668 lbs/day

5. For an F/M of 0.35 (midway between 0.2 and 0.5).
   $M = \frac{1668 \text{ lbs/day BOD}}{0.35}$ = 4766 lbs MLVSS will be required (per tank)

6. To avoid problems associated with relatively higher values of F/M (analogous to low SRT) such as turbid effluent containing significant solids and BOD due to the dispersed bacterial growth which characterizes operation at high F/M (short SRT), it is recommended that M (pounds of MLVSS in the system) be increased. This may be accomplished by decreasing wasting rates (raising SRT). In the next section we will consider the constraints on values of F/M which can actually be attained in any given plant. To illustrate the point here, however, assume we choose a biomass which will make F/M = 0.24 (M=6950 lb). This allows BOD excursions up to 3,475 lbs/day without exceeding F/M = 0.50, but we assume that the average BOD remains at about 1668 lbs/day. This is saying that with an F/M of 0.24 we can double the short term loading on the plant without causing bleed through of BOD or change the biology to an extent that would hamper treatment assuming ample oxygen, nutrients, hydraulic capacity and a readily degradable wastewater are provided.

B. Carbon (PAC) Level

The solids carried in the aeration tank of course are not just MLVSS. Associated with the living portion of the biological floc are many inert, inorganic materials like grit. A portion of each living organism is also inorganic. We commonly differentiate these fractions by burning or ashing a MLSS sample in a muffle furnace at 550° or 660° in the laboratory. In a PACT^{R} system, about 5-35% of the virgin PAC added as initial charge or make-up PAC is ash. When a sample of MLSS is analyzed for its various components - PAC, biomass and ash - there is no distinction made regarding where the ash in the sample came from. Therefore, the following applies equally whether there is PAC in the system or not.

We choose, for example, to operate the system with a mixed liquor volatile carbon concentration at say 2,100 mg/l because experience has shown acceptable treatment results with this level.

1. Given an aeration tank volume of 45,000 gallons then:
   Vol. Carbon = 0.045 MG x 8.34 x 2,100 mg/l = 788 lbs./tank

2. Virgin carbon, as delivered, may be approximately 15% ash, 85% PAC. To get 788 lbs. of volatile carbon will require:
   lbs. virgin carbon = $\frac{788}{0.85}$ = 927 lbs./tank

C. Wasting

Again, this is the means by which the process is controlled. PAC levels in the PACT^{R} system are determined by operating

experience. Based on this information, PAC addition to the system is essentially an independent variable, which can be maintained at as high or low a level as required (or desired). Biomass level, on the other hand, is not an independent variable because the amount of biomass which can be developed in any system depends upon the amount of food available to that biomass.

Therefore, the concept of control of PACT^{R} by wasting reflects the fact that what is really being controlled is the biomass. We can add as much PAC as we want, but we will only get as much biomass as the influent loading will support.

Empirically, it has been determined that the biomass which results from a wasting level based on a 5-15 day SRT performs well, and development of nuisance biogrowths are minimized. As dictated by actual operation the SRT may change to provide optimum treatment.

The calculation of the daily waste quantity (W) is quite simple, no matter what SRT is chosen:

W lbs/day = M (MLSS) lbs/SRT days

1. The MLSS concentration is measured at 15,000 mg/l, for example. Then:
   M (MLSS) lbs = 15,000 mg/l MLSS x 8.34 x 0.045 MG = 5,630 lbs.

2. Choose SRT = 15 days (design value), then:

3. $W = \frac{M - \text{Effluent S.S.}}{SRT}$, if effluent S.S. concentration is low, we can neglect this term.

   $\frac{M}{SRT} = \frac{5{,}630 \text{ lbs}}{15 \text{ days}} = 375 \text{ lbs/day}$

   This amount can be wasted as mixed liquor, whose concentration has been measured at 15,000 mg/l.

4. $W\ (MGD) = \frac{375\ lbs/\ day}{MLSS\ mg/l \times 8.34}$

5. $W\ (MGD) = \frac{375\ lbs/day}{8.34 \times 15{,}000\ mg/l} = 0.003\ MGD/tank$

   (Also calculated as one fifteenth of the aeration tank volume.)

6. W = 3,000 gallons/day/tank of mixed liquor at 15,000 mg/l SS

7. Let the waste flow rate = 70 gpm (for this example).

   Then the total time in minutes to waste (during aeration sequence) for the day is:

   $\frac{3{,}000\ gal.}{70\ gpm} = 43$ minutes/tank

8. Wasting can be done at the end of the settling sequence or during the aeration sequence. Note that wasting time will be less if done at the end of settling sequence (higher solids concentration).

D. Carbon Addition Rate

1. Carbon addition = Carbon wasted (given negligible effluent losses).

2. The amount of volatile PAC wasted is based on the amount of volatile PAC in the MLSS.

   From B.1.: The mixed liquor contains 788 lbs. of volatile carbon (per tank). Since we are wasting one fifteenth (1/SRT) of the total mass of MLSS every day, we will remove:

1/SRT x MLVC = 1/15 X 788 lbs. = 53 lbs of volatile carbon/day

3. To get an equivalent amount of virgin PAC (15% ash):

$$\frac{53 \text{ lbs. vol.}}{0.85 \frac{\text{lb. vol.}}{\text{lb. PAC}}} = 61 \text{ lbs. virgin PAC}$$

4. The carbon makeup required can be added at the start of the cycle. The carbon concentration in the mixed liquor should be routinely analyzed and the carbon feed adjusted accordingly.

E. Polymer Addition

1. A cationic polyelectrolyte (polymer) of high molecular weight, applied at the correct dosage, can significantly reduce the amount of suspended solids in the effluent and enhance settling.

   Dosage requirements can be (roughly) determined by a bench jar test. Your polymer supplier will usually be glad to demonstrate his/her product and suggest dosage rates.

   A wide range of brands and types of polymer should be tested initially keeping in mind the economics involved. One supplier's "cheap" polymer may work just as well as another supplier's "expensive" brand or even exceed the high priced polymer's performance. Polymer dosage is usually expressed in terms of parts per million.

2. Example

   Let's say that the polymer chosen demonstrates good particulate capture at 2 ppm, as demonstrated by the jar test.

The polymer will be applied to the entire tank contents of 45,000 gallons. The pounds of polymer can be calculated as follows:

$$0.045 \text{ MG} \times 8.34 \text{ lb/gal} \times 2 \text{ ppm} = 0.75 \text{ lbs/day}$$

Convert 0.75 lbs. of polymer to gallons of polymer:

$$\text{Total days dosage} = \frac{0.75 \text{ lbs/day}}{9.0 \text{ lbs/gal}} = 0.083 \text{ gallons of polymer per day as supplied}$$

3. Polymer Dilution:

The concentrated polymer must be diluted prior to blending into the wastewater stream. This occurs in the mix tank supplied as part of the polymer addition system. The neat polymer solution is pumped into the mix tank containing the proper quantity of dilution water to yield a solution of about 0.5% wt. polymer.

At an injection rate of 7 gpm, approximately 2 to 3 minutes are required to inject 2 mg/l of polymer respectively. Experience will dictate what dilution and dosage are most effective.

F. Nutrient Addition

The following example shows how to determine whether there is a nutrient deficiency in the PACT^R system influent, and how to calculate the quantity of additional nutrients required to correct the deficiency. Once the nutrient chemical feed rate (lb/day) is determined, the chemical feed equipment must be set to feed the calculated amount of nutrient based on a 24-hour period. (In continuous flow PACT^R systems the nutrients should be fed, preferably, in proportion to flow. In batch

PACT^R systems the nutrients should be added during the wastewater feed step of the process cycle.)

Example: Calculate the required amount of additional nutrients to correct a nutrient deficiency.

Given: (for this example only)

a. PACT^R influent BOD_5 = 2000 mg/l
b. PACT^R influent TKN = 250 mg/l
c. PACT^R influent P = 2 mg/l
d. PACT^R average daily influent flow = 0.028 mgd (27,600 gallons/day)
e. Suggested weight ratio, $BOD_5/N = 100/5 = 20$
f. Suggested weight ratio, $BOD_5/P = 100/1 = 100$
g. Nutrient Nitrogen = Commercial liquid ammonium hydroxide (NH_4OH) 29% by weight. Atomic weight ratio from (Table 3-1) = 2.5

   Nutrient Phosphorous = Commercial liquid phosphoric acid (H_3PO_4) 75% by weight. Atomic weight ratio (from Table 3-1) = 3.2
h. Density of 29% aqueous NH_4OH = 0.95 (from Table 3-1)

   Density of 75% H_3PO_4 solution = 1.58 (from Table 3-1)

1.0 Calculate nutrient needed to achieve the suggested BOD/N/P ratios.

$$\text{Nutrient Needed} = \frac{\text{PACT}^R \text{ influent BOD, mg/l}}{\text{suggested ratio, } BOD_5/\text{nutrient}}$$

$$\text{N needed, mg/l} = \frac{2000}{20} = 100 \text{ mg/l}$$

$$\text{P needed, mg/l} = \frac{2000}{100} = 20 \text{ mg/l}$$

2.0 Calculate the nutrient addition. If the answer is zero or a negative number, no nutrient need be added.

Nutrient Addition =
(Nutrient needed, mg/l) - (Nutrient in PACT^R influent, mg/l)

N addition, mg/l = 100-250 (TKN) = -150 mg/l

P addition, mg/l = 20-2 (Total P) = 18 mg/l

3.0 Calculate the weight of nutrients that need to be added.

Nutrient to add, lb/day =
(Added Nutrient, mg/l)(Q, mgd)(8.34 lb/gal)

P to add, lb/day = (18)(.020)(8.34) = 1.5 lb/day/tank

4.0 Calculate weight of commercial chemical to be added per day

Nutrient chemical, lb/day =

$$\frac{\text{(nutrient to add, lb/day)(Atomic weight ratio)(100\%)}}{\text{concentration of chemical \%}}$$

75% H_3PO_4 gal/day = $\frac{(1.5)(3.2)(100)}{75}$ = 6.4 lbs/day, solution/tank

5.0 Convert lbs/day solution to gallons per day

Nutrient chemical, gal/day = $\frac{\text{Solution lbs/day}}{\text{(Density)(8.34 lbs/gal)}}$

75% H_3PO_4 gal/day = $\frac{6.4}{(1.58)(8.34)}$ = 0.5 gal/day/tank

Note: If dry chemicals are to be used, the weight of the chemical is determined directly; Step 5.0 is omitted.

TABLE 3-1

PACT[R] Nutrient Atomic Weight Ratios
for Commonly Used Commercial Chemicals

Source	Formula	Atomic Wt	Calc.	Atomic Wt. Ratio	Density
Nitrogen					
Elemental Nitrogen	N	14	14/14 =	1.0	-
Ammonia	NH_3	17	17/14 =	1.2	-
Ammonium Hydroxide	NH_4OH	35	35/14 =	2.5	-
10% solution		-	-	-	.98
18% solution		-	-	-	.96
29% solution		-	-	-	.95
Ammonium Chloride	NH_4Cl	53.5	43.5/14 =	3.8	-
Ammonium Sulfate	$(NH_4)_2SO_4$	132	132/(2)(14) =	4.7	-
Phosphorus					
Elemental Phosphorus	P	31	31/31 =	1.0	-
Phosphoric Acid	H_3PO_4	98	98/31 =	3.2	-
50% solution		-	-	-	1.34
75% solution		-	-	-	1.58
Trisodium Phosphate	Na_3PO_4	164	164/31 =	5.3	-
Combination N-P					
Mono-basic Ammonium Phosphate	$(NH_4)H_2PO_4$	115	115/14 =	8.2 (nitrogen)	
			115/31 =	3.7 (phosphorus)	
Di-basic Ammonium Phosphate	$(NH_4)_2H_2PO_4$	132	132/(2)(14) =	4.7 (nitrogen)	
			132/31 =	4.3 (phosphorus)	

6.0 Nutrient addition using estimated BOD and nitrogen values.

As stated previously, a desirable BOD:N:P weight ratio based on influent BOD is 100:5:1. For practical purposes an estimate of organic loading to the PACT^R system can be used especially if nutrient addition rates have to be adjusted on a daily basis. Therefore, analysis such as chemical oxygen demand (COD) is necessary to provide quick analytical results. COD analyses can be run within several hours and an estimated BOD value can be calculated once a BOD:COD ratio has been established. To establish a BOD/COD ratio, parallel COD and BOD analyses of the PACT^R influent should be conducted over a period of 90 days or longer. Once the BOD:COD ratio is known, a BOD can be estimated based on a measured COD.

Estimated BOD, mg/l = (COD, mg/l)(Ratio BOD/COD)

As an estimate for nitrogen content in the PACT^R influent, ammonia-nitrogen (NH_3-N) can be used since it is easier to run in the lab than the Total Kjeldahl Nitrogen (TKN) analysis. However, a correlation between NH_3-N and TKN must be developed also. Again, this ratio of TKN/NH_3-N should be established over a period of 90 days or longer using parallel TKN and NH_3-N analyses of the PACT^R influent.

Estimated N, mg/l = (NH_3-N, mg/l)(Ration TKN/NH_3-N)

a. Example: Estimate the BOD loading based on a COD analysis.

For this example, let's say that over a 3-month period of time, the influent BOD/COD ratio is consistently 0.33. In other words, the organic loading to the PACT^R system can be expressed as: BOD = (COD)(.33)

Given:

COD = 6,000 mg/l

Estimated BOD, mg/l = (COD, mg/l) ratio $\frac{BOD}{COD}$

Estimated BOD, mg/l = (6,000)(.33) = 1980 mg/l

Use the estimated BOD in the nutrient calculation in place of actual BOD_5.

b. Example: Estimate N available in the PACT^R influent based on the TKN/NH_3-N ratio.

For this example, let's say that over a 3-month period of time the TKN/NH_3-N ratio is consistently 10.0.

Given:

NH_3-N = 25 mg/l

Estimated N, mg/l = (NH_3-N, mg/l) ratio $\frac{TKN}{NH_3\text{-}N}$

Estimated N, mg/l = (25)(10) = 250 mg/l

Use the estimated N in the nutrient calculation in place of the actual TKN.

Note: TKN measures organic nitrogen plus ammonia-nitrogen in the sample. This analysis better quantifies total nitrogen available to the biomass.

Section No. 4

PLANT COMPONENT DESCRIPTION AND OPERATION

Important - This section explains in detail the description, function and operation instructions for every major piece of equipment in the plant. It is not a sequential listing of start-up and shut-down instructions for the plant. Sequential start-up and shut-down instructions are found in Sections 5 and 8 respectively of this manual. This section should be referenced in conjunction with sequential plant start-up and shut-down instructions. Further information for each piece of equipment is found in the Equipment section of the Equipment/Control/Instrument/Valve Manual.

1.0 Operating Sequence

Batch PACT^R Units No. 1, 2, and 3 can be operated in either manual or automatic mode. In the manual mode, all pieces of equipment will operate independently. In the automatic mode most of the equipment will operate automatically once the operator starts the cycle. In the automatic mode the equipment operation is controlled by a series of adjustable timers in the PC0085 programmable controller. The registers are preset and times are adjusted by using a TC0085 keypad located on the front face of the Batch PACT^R control panel.

The A.S.S.T./Decant equalization tank is operated in the manual mode only. That is, each piece of equipment operates independently.

Automatic Mode

The operating sequence for the automatic operating mode is controlled using a PC0085 programmable controller located in each control panel. Additional information, including the Gould PC0085 programmable control system users manual, the Quartech TCR085 Data

Access Panel Products Manual, and a Zimpro/Passavant software ladder diagram of the automatic operating mode as outlined below, is given in the Control Section of the Equipment/Control/Instrument/Valve Manual. Operators should become familiar with the Control/Instrumentation sections of this manual before attempting to operate the Batch PACT^{R} unit. Operation of the Batch PACT^{R} unit in the manual mode would be similar to the operator having the responsibility to turn-on or shut-off equipment at the appropriate time.

Sequence of Operation
Batch PACT^R Unit 1, 2, or 3

1. When power is applied to panel, unit is off-line and influent valve is closed. Place all selection switches in the "auto" position.

2. Operator presses START, CYCLE RUN lamps lights, unit enters IDLE mode.

3. Unit waits for both other Batch PACT^R influent valves to close. If this unit must wait longer than the Anoxic Idle Timer, the blower will start and run for the duration of the Aerobic Idle Timer. This cycle will continue until both other influent valves close. Influent valve opens. Tank begins to fill.
   a. In Aerobic Fill mode (normal operation), blower starts (if not already running).
   b. In Anoxic Fill mode (operator selectable), blower stops (if running) and mixer starts.

4. Blower or mixer continues to run until tank fills (level control mode) or Fill Timer expires, if enabled (timed control mode). Operator can disable Fill Timer.

5. Mixer stops (if running) and blower starts (if not already running). Aerobic React Timer starts.

6. Aerobic React Timer expires. Blower stops, Anoxic React Timer starts. Mixer starts.

7. Anoxic React Timer expires. Mixer stops. Polymer addition Timer starts. Polymer pump starts. Blower starts.

8. Polymer Addition Timer expires. Polymer pump stops. Flush Timer starts. Flush Valve opens.

9. Flush Timer expires. Flush Valve closes. Polymer Mix Timer starts.

10. Polymer Mix Timer expires. Blower stops. Settling Timer starts.

11. Settling Timer expires. Decant pipe lowers.

12. Decant pump starts. Tank is pumped to LOW level. Decant pump stops. Decant pipe rises.

13. Go to Step 3.

Modicon 0085 Programmable Controller
PACT[R] Registers

	Name	Register	Preset (One cycle/day)
1.	Fill Timer	C00	480
2.	Aerobic React Timer	C01	600
3.	Anoxic React Timer	C02	240
4.	Polymer Addition Timer	C03	3
5.	Polymer Line Flush Timer	C04	3
6.	Polymer Mix Timer	C05	5
7.	Settling Timer	C06	60
8.	Horn Blow Timer	C07	5
9.	Anoxic Idle Timer	C10	120
10.	Aerobic Idle Timer	C11	30

All register values are in minutes.

Enable Fill Timer by setting register].40 to 1. If the Fill timer is disabled, the Influent Valve will close when the tank fills to the High Level Sensor. If the Fill Timer is enabled, the Influent Valve will close when the Fill Timer expires or the tank fills to the High Level Sensor, whichever comes first.

If register].41 is set to 0 (default setting), the unit will run the Aeration Blower during filling (Aerobic Fill). If register].41 is set to 1 (by using the register access module), the unit will run the Mixer during filling (Anoxic Fill).

The Aeration Blower will always run during steps 2, 4, 5 and 6. The Mixer will always run during Step 3.

In Idle mode, if this unit is waiting for influent for longer than the Anoxic Idle Timer, due to low flows, the Aeration Blower will run for the time set in the Aerobic Idle Timer. This cycle will continue until the unit is needed to process influent or the unit is taken off line.

Operation of Batch PACT^{R} Unit 1, 2 and 3

Start-up

Apply power to all three units (120 VAC 1ϕ and 480 VAC 3ϕ). Press Cycle Stop button twice to ensure each unit is off line.

Press Cycle Start on unit to be filled first. Wait for influent valve to open. Press Cycle Start on other tanks to be used.

Operation

Press Cycle Stop once to pause a unit. While the unit is paused, the Cycle Run lamp will flash, all motors and pumps will stop, and the influent valve will close.

Press Cycle Stop again to take unit off line and reset PACT^{R} cycle position to beginning.

NOTE: No motors or pumps will be automatically started if the unit is off line. If aeration of the tank is required when the unit is off line, place the Aeration Blower control selector in the Hand position.

To continue a paused cycle, press the Cycle Start button. The Cycle Run lamp will go to a steady-on state, and the PACT^{R} cycle will continue from the paused point.

NOTE: If the unit is paused during a timed event (such as the aeration step), the total time spend in that step may vary by as much as one minute per each time the cycle is paused. Pressing Cycle Stop while the polymer line contains polymer is strongly discouraged.

Shutdown

To take an individual unit off line, press Cycle Stop twice. No pumps or motors will be automatically started, although the Hand Off Auto and Open Close Auto switches can still be used to manually operate the pumps, motors and valves.

NOTE:

1. Chemical feed (nutrient phosphorous) to the aeration tank is done manually by running the chemical pump for a predetermined length of time.

2. Sludge wasting from the aeration tank is done manually by running the sludge waste pump for a predetermined length of time. Sludge is pumped to the sludge storage tank.

3. Carbon addition is done manually by placing 50 lb. bags of PAC, as required, in aeration tank.

2.0 Non-Potable Water Supply System

Description: Non-potable (clean) water is supplied to the following users via a network of piping and valves:

Equipment	Use
Polymer System	Polymer dilution, line flushing
Waste Sludge Pump	Seal Water, line flushing
Chemical Tank	Chemical mixing

Start-Up: Before charging the non-potable water system, make sure the following valves are closed:

No. 1, 7 (Polymer System)
No. 6, 11 (Waste Sludge Pump)
No. 37 (Chemical Tank)
No. 32 (Sludge Pump)
No. 19 (Non-Potable Water Strainer)

3.0 Controls/Instrumentation

Description: The controls/instrumentation consists of four field control panels and miscellaneous instruments for local control of Zimpro/Passavant furnished equipment.

A. Batch PACT^{R} Unit 1, 2, & 3 Control Panels - One panel is located in the equipment area of each unit. Each panel houses the programmable controller, electrical equipment, and manual operating controls for:
   - — Aeration Blower
   - — Tank Mixer
   - — Influent Valves
   - — Polymer System
   - — Waste Sludge Pump
   - — Decant Pump & Winch
   - — Alarms

B. A.S.S.T./Decant Equalization Control Panel - Located in the equipment area of the unit. This panel houses the electrical equipment and manual operating controls for:
   - — Aerated sludge blower
   - — Sludge pump
   - — Chemical feed system
   - — Filter feed pumps
   - — Alarms

C. Miscellaneous field instruments and electrical controls including:
   - — All hand switches (HS) for 120 volt, 1ϕ, Zimpro/Passavant supplied motors
   - — Pressure Indicators (PI)
   - — Temperature Indicators (TI)
   - — Level Switches (LS)
   - — Limit Switches (ZS)
   - — Electrically actuated valves (EV)
   - — And other miscellaneous instruments as shown on the engineering flow diagrams.

Start-Up

It should be noted here that once the plant instrumentation is energized, it should remain energized unless the plant is shut down for an extended period of time.

Shut-Down

De-energize all power supplies.

4.0 Polymer System (Unit #1)

Description: Polymer is mixed in a tank and fed to the system by a constant speed gear pump rated at 7 gpm. Add neat polymer to the polymer tank containing dilution water to form a ½% (wt) polymer solution. The mixer should be on as the polymer is added and allowed to mix for 30 minutes after polymer addition. Mixing is not required during operation. Polymer quantities should be limited to what can be used in a 24-hour period.

Polymer addition to the aeration tank per cycle:

Polymer Dose, mg/l	Volume at ½%, gal	Pump Run Time, min.
2	14	2
3	21	3
5	35	5

Start-Up:

Prerequisites:

— Non-potable water available.

— Power supply energized.

To prepare the polymer system for use: Close valve No. 15, open valve No. 1, and add the desired quantity of water to the polymer tank.

Open valve No. 19 and flush strainer (No. 18) until clean water is observed. Operate polymer pump momentarily (HS-130 to HAND position) to insure water flow through the pump. Turn on the polymer tank mixer (HS-120) and add neat polymer to the mix tank. Allow to mix for 30 minutes and then turn off mixer (HS-120).

Open valve No. 2 & 7. Turn on the polymer pump (HS-130) and run the pump for the time required to provide the polymer dosage. In the automatic mode the run time is set on register C03 of the PC0085 programmable controller.

Shut-Down: Turn off the polymer pump (HS-130), and flush the lines with dilution water by opening valve EV-135 with hand switch HS-135. In the automatic mode the pump will stop and lines will be flushed in accordance with the Operating Sequence.

Note: If the system is shut down for a long period of time, it should be thoroughly flushed with water and drained.

5.0 Aeration Blower (Unit #1)

Description: Air to the aeration tank diffusers is supplied from one aeration blower. The blower is a Roots Type RAI-U, Size 718, rotary positive displacement blower rated at 1000 cfm. The blower is equipped with inlet filter, inlet and discharge silencers, inlet and discharge temperature gauges, inlet and discharge expansion joints, discharge relief valve, discharge check valve, and discharge pressure gauge.

Start-Up:

Prerequisites:

-- Blower pre-startup maintenance, including lubrication, must be completed (see Equipment Manual for details).

-- Electric power supply energized.

Before starting the blower, open Valve No. 10. Turn on the blower (HS-180 to HAND position). In the automatic mode, the blower will start in accordance with the Operating Sequence.

Shut-Down: Turn off the blower (HS-180 to OFF position). In the automatic mode, the blower will stop in accordance with the Operating Sequence.

6.0 Waste Sludge Pump (Unit #1)

Description: PACT^{R} sludge is drawn from the bottom of the aeration tank and pumped to the aerated sludge storage tank by the waste sludge pump. The pump is Dean Bros. Model DL201, in-line vertical, centrifugal pump rated at 70 gpm.

Start-Up:

Prerequisites:

— Waste sludge available.

— Power supply energized.

Before starting the waste sludge pump, open the following valves:

No. 9, pump suction.

No. 11, seal water. Establish seal water flow at 5-10 GPH. Readjust once the pump is running.

No. 24, pump discharge.

Turn on the waste sludge pump (HS-190) and run until the required volume of sludge has been pumped.

Shut-Down: Turn off the waste sludge pump (HS-190). If the shutdown is to be an extended one, close valve No. 9 and flush the pump suction and discharge lines with non-potable water by opening valve No. 6.

7.0 Submersible Mixer (Unit #1)

Description: Contents of the aeration tank are kept mixed during the anoxic cycle by the submersible mixer. The mixer is a Flygt Model 4451 submersible, motor driven mixer rated at 9400 gpm.

Start-Up:

Prerequisites:

- — Wastewater available.
- — Power supply energized.
- — Mixer pre-startup maintenance, including lubrication, is completed (see Equipment Manual for details).

Turn on the mixer (HS-175 to HAND position). In the automatic mode, the mixer will start in accordance with the Operating Sequence.

Shut-Down: Turn off the mixer (HS-175). In the automatic mode, the mixer will stop in accordance with the Operating Sequence.

8.0 Decant Pump (Unit #1)

Description: Decant supernatant is drawn from the top of the aeration tank and pumped to the decant equalization tank by the decant pump. The pump is a Flygt Model CT3085/82, dry pit vertical mount, centrifugal pump rated for 200 gpm.

Start-Up:

Prerequisites:

- Wastewater available for decanting.
- Decant pipe lowered below wastewater to decant level.
- Power supply energized.

Before starting the decant pump, open the following valves:

No. 8, pump suction

No. 21, pump discharge

Turn on the decant pump (HS-170) to HAND position and run until aeration tank is decanted to the desired level. In the automatic mode, the decant pump will start in accordance with the Operating Sequence.

Shut-Down: Turn off the decant pump (HS-170). In the automatic mode, the decant pump will stop in accordance with the Operating Sequence.

9.0 Decant Winch (Unit #1)

Description: The decant pipe is lowered into the aeration tank for draw-off of the decant supernatant and raised out of the liquid after decanting is completed by the decant winch. The winch is a Thern Model 473A 1/2 B electric power winch (worm gear type).

Start-Up:

Prerequisites:

— Power supply energized.

Turn local operating station hand switch (HS1-150) to manual position and raise or lower decant pipe by turning hand switch (HS2-150). In the automatic mode, the decant winch will be raised and lowered in accordance with Operating Sequence.

NOTE: For automatic operation the limit switches furnished with decant winch must be adjusted to the required lowered position and raised position.

Initial Start-Up Raised Position -

Set end of pipe @ 0'-6" from top of aeration tank.

Initial Start-Up Lowered Position -

Set end of pipe @ 4'-0" from top of aeration tank.

10.0 Carbon Addition

Description: Virgin powdered activated carbon (PAC) is added directly to the aeration tank on a daily basis as required to meet leachate treatment performance levels. PAC is furnished in 50 lb. water soluble bags with an over-pack made of paper. These bags are to be stored indoors in a dry area.

To Add Carbon: PAC bag(s) are transported from storage to the Batch PACT^{R} tank platform. At this time the outer paper bag can be cut at one end, allowing inside water soluble bag to be carefully removed and slipped into the aeration tank.

Safety: Note that some dusting of carbon will occur and that a dust mask is recommended while handling PAC. See Activated Carbon Material Safety Data Sheet found in Section 9 of this manual for additional information regarding activated carbon.

11.0 Chemical Feed System

Description: Phosphoric acid (nutrient) is fed from a tank to one of three (3) aeration tanks by a Neptune Chemical proportioning pump, Model No. 532-A-N3, diaphragm type, rated at 0-11 GPH. Add 50% or 75% commercially available solution of phosphoric acid to the chemical tank. (A mixer is supplied for the mixing of dry chemicals, if required.)

NOTE: Be sure that Valve No. 39 (Chemical Tank Drain) is closed before filling tank.

Safety: A phosphoric acid Material Safety Data Sheet must be obtained from the supplier of phosphoric acid. This safety data sheet will provide detailed information on the phosphoric acid used at your facility. For reference, a "sample" phosphoric acid Material Safety Data Sheet can be found in Section 9 of this manual.

CAUTION: Eye protection and special clothing are required when handling phosphoric acid. See Section 9 on working safely with chemicals.

Start-Up:

Prerequisites:

— Non-potable water available.
— Power supply energized.
— Solution in chemical tank.
— Wastewater in aeration tank.

Open pump suction valve No. 38 and discharge valve No. 22 to the selected aeration tank. Turn on the chemical pump (HS-440) and run the pump at the required stroke rate and length of time to provide the needed phosphoric acid dosage.

Shut-Down: Turn off the chemical pump (HS-440) and close valve No. 38 and 22.

12.0 Aerated Sludge Blower

Description: Air to the aerated sludge storage tank diffusers is supplied from one aerated sludge blower. The blower is a Roots Type RAI-U, Size 36, rotary positive displacement blower rated at 60 cfm. The blower is equipped with inlet filter, inlet and discharge silencers, inlet and discharge temperature gauges, inlet and discharge expansion joints, discharge relief valve, discharge check valve, and discharge pressure gauge.

Start-Up:

Prerequisites:

— Blower pre-startup maintenance, including lubrication, must be completed (see Equipment Manual for details).
— Electric power supply energized.

Before starting blower, open valve No. 28. Turn on blower (HS-460).

Shut-Down: Turn off blower (HS-460) and close Valve No. 28.

13.0 Sludge Pump

Description: Solids from the bottom of aerated sludge storage tank are pumped to the sludge drying beds adjacent to the leachate treatment system. The pump is a Flygt Model CT3085/82, dry pit vertical mount, centrifugal pump rated for 180 gpm at 12' head or 220 gpm at 7.5' head.

Before pumping sludge to drying beds, the aerated sludge blower should be turned off (HS-460) and the sludge allowed to settle. At this time the liquid near the top of the tank can be decanted to the pump drain station. This is done by using the manual decant winch to position the decant pipe just above the sludge level and opening valve No. 34.

Before the sludge pump, open the pump suction and discharge valve, No. 33 and 30 respectively.

Turn on the sludge pump (HS-430) and run until the required amount of sludge has been pumped or pump automatically shuts off at low level.

Shut-Down: Turn off the sludge pump (HS-430) and close pump suction valve No. 33. If shutdown is to be an extended one, flush the pump suction and discharge lines with non-potable water by opening Valve No. 32.

Restart aerated sludge blower (HS-460).

14.0 Filter Feed Pumps

Description: Treated leachate from the decant equalization tank is pumped to a tertiary sand filter by the two filter feed pumps. These pumps are Goulds Model GL887 submersible sewage pumps rated for 30 GPM at 23' TDH. In the automatic mode these pumps are cycled on and off by level controls as follows:

- lead pump ON @ High Level
- lag pump ON @ High High Level
- lead and lag pump OFF @ Low Level

Note: Lead and Lag pumps are alternated.

In the manual mode the filter feed pumps can be operated by turning HS-420 and/or HS-425 to HAND position.

Start-Up:

Prerequisites:

— Wastewater available.
— Power supply energized.
— Tertiary filter on-line.

Turn pump(s) ON (HS-420 and/or HS-425).

Shut-Down:

Turn pump(s) OFF (HS-420 and/or HS-425).

Section No. 5

PLANT START-UP

1.0 Narrative

This section contains a sequential, step-by-step procedure to start-up the PACT[R] Wastewater Treatment Plant. This procedure should be followed anytime the plant is put on line, such as after an extended shut-down.

A. Due to initial slow biomass growth, the plant should be started, if possible, at a decreased influent loading rate.

B. An initial charge of 800 lbs. (16 bags) of virgin PAC is to be added to each aeration tank. This amount is placed directly in aeration tank.

C. It will be very important to control wastewater flows into the plant during start-up. In no case should the full flow/load be applied before PAC and biomass levels have reached at least the lower design limit concentrations and the polymer used has been proven to be effective. Premature introduction of the full hydraulic load may result in biomass die off and the possible loss of weeks of effort in developing an acclimated biomass.

D. Biomass can be introduced into the system in two ways- either by developing naturally in the system from inoculating organisms carried in with the waste stream or by seeding the system with sludge from an operating biological treatment plant. Seeding with a large inoculum of either activated sludge or trickling filter humus will allow development of the biomass much more quickly than will starting from "scratch".

If sludge can be trucked in from a neighboring plant, add enough sludge to bring the biomass level to about 500 mg/l, after which it can develop on its own to whatever level the influent feed can support.

The recommended start-up schedule is outlined below. Percentages listed represent percent of the total daily flow (30,000 GPD).

% Waste	Duration, Days
25	2
50	4
75	4

The schedule may be shortened or waste flow percentages increased as dictated by the performance results.

E. Polyelectrolyte or "polymer" should also be added from the beginning of plant operation in the proper dosage for the flows/loadings existing during the start-up period. It may be necessary to perform jar tests and change feed rates accordingly, even day-to-day, as flows and loads change and the system approaches design or "steady state" operation. This will be especially important when the plant is started from "scratch" or with very low levels of biomass.

F. It will not be necessary to waste sludge immediately, especially when the biomass is difficult to establish, but wasting and PAC addition to maintain SRT and the desired carbon dose should begin as soon as biomass is detectable microscopically, and not later than one SRT after beginning operation of waste (i.e., if the design SRT is 15 days, the wasting of 1/15 of the solids in the system daily should begin within 15 days after the introduction of waste - in whatever quantity or flow - to the PACT^{R} system).

G. As biomass levels increase, the system oxygen demand will also increase. D.O. checks at various points and depths in the aeration tank should be made 2-3 times per day, and additional aeration provided if D.O. concentrations are seen to fall consistently between 1-2 mg/l D.O.

H. It will be necessary to sample and test the system daily during start-up. Table 5-1 shows the recommended sample points and analyses. While many plants may be run without all recommended analyses, baseline data and periodic samples are invaluable in solving problems which may arise even long after start-up. Analytical procedures for the recommended analyses will be found in Section 10 of this manual.

2.0 Initial Start-Up Instructions

A. Pre-Start-Up Checks & Procedures

These checks should be made at initial start-up and also by all new operating personnel (or whenever the system has been idle or mothballed for an extended period).

1. Locate and trace all flow streams in the system. Use the equipment drawings supplied with the system equipment manual. These streams include:
   a. Plant influent stream
   b. Air flow
   c. Waste sludge flow
   d. Polymer feed lines
   e. Non-potable water
   f. Nutrient system
   g. Waste sludge transport and storage

   Inspect all connections and fittings to be sure they are correct and tight.

2. Inspect the equipment, piping and valves associated with each stream to ensure free operation, no obstructions or broken lines, and in the case of rotating equipment, jog to confirm the proper direction of rotation. Repair or adjust as necessary.

3. Check all lubricant levels and grease fittings, determine whether fresh lubricant is needed or has recently been added.

4. Check packings, seals and seal water flows to ensure proper installation and operation. Packings and seal water flows will have to be readjusted after operation on process flows.

5. Check all timers for proper initial setting (see equipment manual for initial setpoints). Visually confirm that contacts and electrical connections are secure and clean (not burned or corroded).

6. Visually check the entire system for loose or broken gratings, braces, belt guards, etc. Insure that all protective coatings and paint are intact, cover the area completely as required, and are free from wrinkles, bubbles, and flaking or peeling especially on submerged surfaces.

7. Read and become thoroughly familiar with all manufacturers' manuals and instructions in the system equipment section.

B. **Check-Out on Water**

Fill all the tanks with water, then run all equipment and instrumentation for a minimum of 8 hours following manufacturers' instructions and instructions found in Section 4 of this manual. Monitor all equipment closely and note the following:

1. Current draw on each leg of 3 phase equipment.
2. Any unusual noise or vibration.
3. Overheating.
4. Make operational adjustments to equipment and instrumentation per instructions given in manufacturers' manual.

C. **Detailed Plant Start-Up**

Once the equipment and instrumentation has performed satisfactorily on water, prepare the plant to go on-line as follows:

NOTE: Reference sections in parenthesis ().

1. Drain the test water from all tanks except the aeration tank. The aeration tank should be drained to about two thirds full to accommodate the initial charge of carbon slurry and seed biomass.

2. Energize the instrumentation system (Section 4-3).

3. Charge the non-potable water system (Section 4-2).

Important

It should be noted here that the operator should take all prescribed safety precautions for handling powdered activated carbon, acid, nutrients, and polymer before proceeding. See Section 9 on Personnel Safety.

4. Prepare the polymer addition system (Section 4-4).

5. Start the aeration blower at minimum flow (Section 4-5).

6. Charge the aeration tank with PAC (Section 4-10).

7. Add seed sludge to aeration tank. The aeration tank should now be nearly full. (If not, add water.)

8. Add wastewater to the aeration tank at 25% of normal flow.

9. Add nutrients (Section 4-11).

10. Begin wasting sludge to the aerated sludge storage tank after 10-15 days of sustained biological growth.

Monitor all plant functions. Maintain the following operating parameters during the start-up phase:

MLSS D.O.	2.0 mg/l (ppm) minimum
MLSS pH	6.5 - 7.5

Effluent ammonia nitrogen and phosphorous at 1-3 ppm.

Influent flow - increase gradually as biological activity increases (no more than 25% increase per day).

Tabe 5-1

Suggested Analytical Schedule

	BOD	COD	SS	PAC/ BIO	pH	NH_3-N	Total P	Settleability
1. PACT Feed	D	D	D	N	D	D	D	N
2. Mixed Liquor	N	N	D	Th	D	N	N	D
3. Effluent	D	D	D	N	D	D	D	N

D = Daily (week days)

Th = 2-3 times per week

W = Once per week

N = Not required

NOTE: Analytical work required by the Discharge Permit is not considered in this manual.

Use TOC or COD to monitor treatment level.

Use microscopic exam to monitor biology.

Use NH_3 and total P to regulate nutrient addition.

Section No. 6

ROUTINE OPERATION

1.0 Routine Operation

Maintain the following process control parameters during normal operation of the plant.

Influent flow range - minimal to 10,000 GPD per aeration tank at 6000 mg/l COD.

Mixed liquor D.O. - 2 ppm minimum

Mixed liquor pH - 6.5 to 7.5 is optimal

Nutrient addition - maintain effluent phosphorous (P) in the range of 1-3 ppm.

Polymer feed rate - maintain minimal "suspended solids" in effluent.

SRT - 15 days (10 day SRT recommended to start with. SRT may be increased slowly if nuisance biogrowths do not cause effluent quality to deteriorate.)

The operator should perform the following recommended duties in developing an operations routine for the plant:

A. Collect samples and perform analyses as recommended (see Section 5) to monitor plant operation and meet any applicable Federal, State or local regulatory agency requirements.

B. Perform maintenance as recommended by equipment manufacturers. (See Equipment Manual.)

C. Check air flow and distribution to ensure proper D.O. and mixing.

D. Fill out log sheets as required.

E. Check that required chemical additives are available in storage tanks. Check and record all feed rates. Ensure delivery at feed point. Recalibrate system as required.

F. Check the pump flow of waste sludge to ensure that proper waste rates are achieved.

G. Add carbon daily to the aeration tank to replace carbon wasted.

NOTE: It is strongly recommended that a preventative maintenance program, for all plant equipment, be developed and implemented. Such a preventative maintenance program should contain a schedule for cleaning, lubrication, inspections, adjustments, calibrations and maintaining protective coatings for plant equipment. With proper maintenance, the equipment can be expected to last its full anticipated service life.

OPERATING RECORDS

1. Waste Processed

    a. Influent or effluent flow data
    b. pH to the PACT^{R} unit
    c. Temperature to the PACT^{R} unit

2. Carbon Added

    a. Carbon added to system

3. Polymer Dose Rate

    a. Polymer pump operating time
    b. Change in polymer tank level
    c. Polymer usage per day (neat polymer)

4. Wasting Rate

    a. Pump flow rate and operating duration
    b. Sludge storage tank levels

5. Nutrient Feed Rate

    a. Nutrient pump operating time
    b. Change in nutrient tank level
    c. Nutrient usage per day

6. Records of equipment operation should be kept also.

2.0 Lubrication Schedules

Points of Lubrication	Mfr's Recommended Lubr.	Amount	Freq.
Aeration Blower, Aerated Sludge Blower (Roots)			
a) Bearing Lubrication	a) Chevron SRI No. 2 Grease	As Req'd	5 weeks
b) Gear Housing	b) Oil - SAE40 (Winter) SAE50 (Summer)	Full	See Manual
c) Motor, 40HP, 5HP	c) Baldor Motor Chevron SRI No. 2	As Req'd	3 months
Polymer Pump (Liquiflo)			
a) Motor, 1HP	a) U.S. Motor Chevron SRI No. 2	As Req'd	Yearly
Polymer Mixer (Neptune)			
a) Gear Housing	a) Lithium #2 Grease	16 oz.	Yearly
b) Motor, 1/3HP	b) Prelubricated for life of motor	-----	-----
Waste Sludge Pump (Dean Brothers)			
a) Motor, 1½HP	a) Lithium #2 Grease	3/4 Full	Yearly
Submersible Mixer (Flygt)			
a) Oil Casing	a) Mobil SHC630	2.65 qt.	After 1st 200 hrs of operation
b) Bearing Casing	b) Mobil SHC630	0.5 qt	After 1st 200 hrs of operation

Points of Lubrication	Mfr's Recommended Lubr.	Amount	Freq.
Decant Pump, Sludge Pump (Flygt)			
a) Oil Casing	a) SAE10W30 Motor Oil Mobil Whiterex 309 or equivalent paraffin oil	1.1 qt.	Inspect Monthly
Decant Winch (Thern)			
a) Gear Case	a) AGMA #8 Oil	Fill to middle oil plug	Inspect Monthly
b) Open Gears	b) NLGI #2 EP Grease	2.0 oz.	Monthly
c) All other points of friction	c) medium wt. oil	As Req'd	Monthly
Chemical Pump (Neptune)			
a) Gear Box	a) SAE90 or AGMA No. 5EP	As Req'd	Yearly
b) Motor, 1/3HP	b) Prelubricated for life of motor	-----	-----
Chemical Mixer (Neptune)			
a) Motor, ¼ HP	b) Prelubricated for life of motor	-----	-----
Filter Feed Pumps (Goulds, G&L)			
a) Submersible pump/motor	a) Oil - filled w/no further lubrication required	-----	-----

PACT® TROUBLESHOOTING GUIDE

OBSERVATIONS	PROBABLE CAUSE	REQUIRED CHECK	REMEDY
1. Sludge floating to surface.	1. Denitrification occuring; nitrogen gas bubbles attaching to sludge particles; sludge rises in clumps.	1a. Nitrate concentration; if no measurable NO_3, then 1 (1a) is not the cause.	1a. Increase DO in aeration tank.
		1b. Check SRT, do a microscopic examination.	1b. Reduce SRT.
	2. Filamentous growth causing floating solids.		1c. See 3a below.
2. Straggler floc in effluent settling is good, but effluent is turbid.	2a. Excessive turbulence in aeration tanks.	2a. DO in aeration tank.	2a. Reduce aeration agitation.
	2b. Carbon fines not flocculating.	2b. Polymer addition.	2b. Assure proper delivery and dose.
	2c. Toxic shock load.	2c. Microspocically examine sludge for inactive protozoa.	2c. Re-seed sludge with sludge from another plant if possible.
	2d. Large increase in loading, causing predominance of flagellates.	2d. Plant influent loading.	2d. Increase PACT addition.

OBSERVATIONS	PROBABLE CAUSE	REQUIRED CHECK	REMEDY
3. Very stable foam on aeration tank which sprays cannot break up.	3a. SRT is too long, filamentous organisms predominating.	3a. Check SRT, also microscopic examination can be used to determine presence of filamentous organisms.	3a. (1) Increase sludge wasting so as to reduce SRT. (2) Increase DO in aeration tank if less than 1 mg/l. (3) Increase pH to 7. (4) Supplement deficiency of nutrients so that BOD to nutrient ratio is no more than 100 mg/l BOD to 5 mg/l total nitrogen; to 1 mg/l phosphorus.
4. Thick billows of sudsy foam on aeration tank.	4a. MLSS too low.	4a. MLSS	4a. (1) Decrease sludge wasting so as to increase MLSS. (2) Increase daily carbon addition.

	OBSERVATIONS	PROBABLE CAUSE	REQUIRED CHECK	REMEDY
5.	pH of mixed liquor decreases to 6.5 or lower. Sludge becomes less dense.	5a. Nitrification occurring and wastewater alkalinity is low. 5b. Improper pH adjustment.	5a. Effluent NH_3 & NO_3. 5b. Influent pH records.	5a. (1) Add source of alkalinity such as lime or sodium bicarbonate. 5b. Add caustic.
6.	Pin floc in effluent - SVI is good, effluent is clear, but some floating material. Floc is granular in appearance.	6a. Long SRT. 6b. Anaerobic side streams recycled.	6a. MLSS	6a. Increase sludge wasting to decrease SRT. 6b. Identify and correct sources of anaerobic conditions.

Section No. 8

PLANT SHUTDOWN

1.0 Plant Shutdown

A. Short intermittent disruptions (several hours) of influent should not affect plant performance.

B. If a flow disruption is to be a day or two in length, continue to add carbon and waste sludge to the sludge storage tank, and continue aeration.

C. For a feed flow interruption of more than two days up to about a week, the instructions in Item B above should be followed for the first two days. Thereafter carbon addition and sludge wasting should be discontinued.

The bacteria in the aeration tankage will be deprived of soluble food and will then consume any available food stored inside their cell walls. The bacteria will begin to die off and the more hearty bacteria will live off of the dead cell matter. During this period the bacteria will decrease in number. The longer the unit is held in this mode, the slower it will have to be restarted to minimize bleed-through of soluble BOD. For restarting, seed sludge will speed up the restart.

D. For a shutdown of a month or two, the plant can be left filled with water if there is no danger of freezing. Water should be added to flush all of the MLSS from the tank. The other plant feeds should also be discontinued, i.e., polymer, nutrients, and carbon. The feed pumps should be shut down and discharge lines flushed with water.

E. If the shutdown is to be long term or if freezing will be a problem, remove solids from the system as described in Item D, above. Then drain all tankage.

Section No. 9
SAFETY

1.0 Plantwide Safety Guidelines

A. Introduction

A safe workplace is important - important to all of us. The National Safety Council reports that over 13,000 deaths and 2,300,000 injuries occur on the job every year. The estimated annual cost of these accidents is $20.7 billion, but the financial impact is really secondary. Each accident involves a worker - a person like you and me. Someone is hurt, and suffers or is killed. A family suffers too, as do friends and co-workers.

This section on safety is intended to be a means of helping you become familiar with specific safety rules, helping you recognize the potential hazards in your work area and helping you develop safe working habits. Everyone should read the Plantwide Safety portion of this manual, then you should study the section for your zone. If it creates questions, see your supervisor.

B. Responsibility

Safety is a shared responsibility. The roles of all concerned are critical. Safety programs can be created, training conducted, safety rules written, and a safe environment provided; but accidents can only be prevented if you take responsibility for your work habits.

1. Responsibilities of Management:

It is the responsibility of each manager and supervisor to insure that all of his or her employees are familiar with the safety precautions that are outlined in Standard Operating Procedures (SOP's) covering their work.

To provide proper safety equipment whenever necessary.

To correct unsafe working conditions promptly.

To encourage practice of safe working habits and compliance with safety regulations through discipline if necessary.

2. Responsibilities of Employees:

   It is your responsibility to understand and work within the safe practices indicated in this manual and department SOP's.

   If you are called upon to perform work that you consider hazardous or which, in your judgement, is not protected with sufficient safeguards, the matter must be brought to the attention of your supervisor.

   Be aware of what your neighbors do as you may be a victim of their carelessness. Do not hesitate to tactfully point out your concern to a neighbor whom you observe engaging in an unsafe practice. The efforts of our federal government to legislate safe industrial practice have been relatively ineffective in reducing the numbers of injuries. Many accidents continue to occur due to carelessness or failure to use common sense.

C. Some "Safe" Assumptions:

1. The prevention of accidents can be accomplished only through full support of every employee.

2. Employees who disregard safe work procedures are a danger to themselves, their fellow employees and the equipment with which they work.

3. A capable, mentally-alert employee avoids accidents by learning all he or she can about his or her work, using proper safeguards and protective equipment, and by avoiding shortcuts and makeshift work methods and equipment.

4. Safe operations are good operations. This is true of both employees and equipment.

5. Accidents do not just happen. Accidents are the natural result of unsafe conditions or unsafe acts, often a combination of both. To eliminate all unsafe conditions and unsafe acts is to eliminate accidents.

6. Safety is infectious: A safe operation and positive safety attitudes lead to even better conditions. An unsafe careless operation gets worse.

D. Common Sense

1. Enter restricted areas only with permission of the area supervisor.

2. Practical jokes and horseplay lead to accidents and are forbidden.

3. Walk carefully. Do not run in hallways or catwalks.

4. Obey warnings on tags and signs.

5. Keep work areas well lighted and clean.

6. Watch for wet floors.

7. Observe "NO SMOKING" signs.

8. Report any potential hazardous condition to your supervisor.

9. Know the location of eyewash stations, safety showers, first aid cabinets and fire extinguishers in your work area. See your supervisor if you do not know where this safety equipment is located.

10. Only trained operators may operate forklifts.

E. Housekeeping:

1. Keep all aisles and work places clear.

2. Keep all tools and material neatly and securely stored to avoid accidents.

3. Store waste, oily rags and other flammable materials only in receptacles provided for the purpose.

4. Keep tops of lockers clean and avoid protrusions from lockers.

5. Use ash trays and waste receptacles provided, not the floor or equipment.

6. Use a pan and brush to clean up broken glass.

7. Clean up immediately any spills of oil, grease, water, etc., to prevent slips and falls.

8. Do your part to keep washrooms, toilets, drinking fountains and locker rooms clean and neat.

F. Protective Clothing and Equipment:

Eye Protection:

These eye protection rules should be enforced for the safety of your vision:

1. If you are working with potentially hazardous liquid chemicals, you must wear waterproof goggles or faceshields when there is potential for splashing or explosive reaction.

2. Visitors to the various areas of the plant should wear shatterproof eyeglasses, safety glasses or plastic "visitors specs".

3. Each operating zone department may identify hazards unique to their operation and may define additional protection requirements.

4. Contact Lenses:

   Contact lenses are generally considered inappropriate for industrial occupations. They provide no protection to your eyes.

5. Miscellaneous Protective Garb:

   a. When working with hazardous material, check with your supervisor regarding safety precautions to be taken and equipment necessary to perform the job.

   b. Wear designated safety equipment, i.e., goggles, gloves, shoes, safety glasses, shield, respirator, etc. when needed.

   c. Wear only approved shoes or safety shoes. Tennis shoes and other casual footwear should not be allowed.

   d. Plant uniforms and protective garments must be worn as designated.

G. Fire Prevention:

1. Employees should shut down equipment and evacuate the building immediately.

2. All doors should be closed when leaving rooms.

3. Smoking is allowed only in areas designated. Use ash trays, never a waste paper basket.

4. Report any signs of defects in electrical appliances, such as sparking or heat. All equipment should be grounded.

5. Prevent combustible dust accumulations by periodic use of a vacuum cleaner.

6. Know the location of fire extinguishers and read directions frequently.

7. Keep all fire stations and extinguishers free and clear at all times.

8. Report any seal found broken on a fire extinguisher to your supervisor.

9. Report use of a fire extinguisher to the supervisor so it can be promptly refilled.

H. Electrical Hazards:

1. Unless it is a part of your regular work, do not attempt to repair or adjust any electrical equipment. Report all electrical problems to your supervisor immediately.

2. Treat electricity with respect, report bare or dangling wires to your supervisor.

3. Ground wires leading from electric apparatus must not be disconnected or broken.

4. Do not operate electrical equipment when it is wet, or when you are standing in a wet location.

5. Before using an extension cord, be certain it is grounded and there are no breaks in the insulation.

I. Lifting Tips

Back injury is the single most common industrial disabler in the United States. Whether you are involved in heavy lifting, or just occasional lifting of a few pounds, take it seriously. It only takes a small load to injure yourself if you lift it incorrectly.

1. Size up the job. Get help if you need it.

2. Know where you are placing articles, before lifting.

3. Keep your feet apart.

4. Hold your back straight.

5. Tuck in the chest.

6. Get a firm grip with your entire hand.

7. Distribute the weight evenly.

8. Keep your elbows and arms close to your sides.

9. Pull the load in close to your body.

10. Press up with your legs.

J. Compressed Gas Cylinder Precaution

1. Cylinder contents must be properly identified by name on the container.

2. Before using, secure the cylinder with chains, check the label identification tag.

3. Never tamper with the safety device on a cylinder.

4. Never use an oxygen regulator on any other gas cylinder.

5. Make sure the connections match the valve outlet; never try to force fittings that do not match. Forcing a valve may cause a leaky connection. A check for leaks with a soap solution or detector can be performed if necessary-never use a flame. Use soap if a lubricant is required.

6. Never use flammable gases where an open flame or sparks could ignite the fumes.

7. Cylinders full or empty should be transported on cylinder transport hand truck by trained personnel. All cylinders must be securely strapped to the truck. Do not roll cylinders.

8. Keep the cap securely fastened to the cylinder during all handling. Remove the cap only after the cylinder has been securely chained and supported for use. Never hoist a cylinder by its protective cap.

9. All compressed gas cylinders must be handled as if full. Keep the cylinders upright and chain them securely at all times.

10. Cylinders are stored only in designated areas and must be secured with chains. Keep empty cylinders separate from the full ones.

11. If a cylinder valve appears to be stuck, the cylinder should be tagged and returned to the supplier for proper disposition.

12. Cylinders should be protected at all times from excessive heat or physical shock. Even though empty, never drop cylinders or let one cylinder jar against another.

13. Toxic or irritating gases should be used only in a hood or in an area with forced ventilation. Face shields should be worn while changing cylinders.

14. Replace the protective cap on empty cylinders. The empty cylinders should be tagged "Empty".

15. Store pure O_2 cylinders away from flammable gas cylinders.

16. If cylinders are partially used, but not empty, they should be labeled with remaining pressure, date and person's initials.

K. First Aid and Accident Reporting

1. All job related injuries and illnesses must be reported to your supervisor immediately. Although initially some injuries may appear to be minor in nature, it is very possible they could develop into more serious conditions.

2. Your supervisor can help determine whether the extent of the injury necessitates immediate treatment.

2.0 Operators' Safety Guidelines

A. Introduction

Safe practice is an attitude and a knowledgeable awareness of potential hazards. The sewage plant is filled with potential hazards. Thoroughly acquaint yourself with the location and use of safety facilities and become familiar with safety precautions and emergency procedures before undertaking any work. Familiarize yourself with the method of operation and all hazards involved before commencing work and see that necessary safety equipment is readily available and in usable condition.

Unauthorized experiments and horseplay will not be tolerated. Never run through the area. Anticipate sudden backing or change in direction from others.

Avoid hazards by keeping drawings and cabinets closed while working. Keep aisles free of obstructions, e.g., chairs, unauthorized stools, boxes and waste receptacles. Avoid slipping hazards by picking up ice, stoppers, glass beads, glass rods, and other small items, from the floor.

Keep the work space uncluttered, with only authorized materials, instructions, notebook and pen or pencil present. Keep work space clear of chemicals, and scraps of paper. Keep measuring equipment, such as glass cylinders, far back where they will not be easily knocked over, lying on the side when not in actual use.

1. Resistant gloves should be worn when using strong chemical solutions.

2. Smoking is prohibited except in designated areas unless approved by your supervisor.

3. Corrosive solutions must be pipetted mechanically rather than by mouth. Any spillage must be neutralized as necessary and cleaned up immediately.

4. Fragments of broken glass must be removed from bench tops or floors as quickly as possible. Any liquids spilled on the floors must be wiped up immediately.

5. Heat-resistant gloves must be worn when emptying ovens.

6. When using carts, allow clearance for hands when moving through doorways or past obstacles. Do not open doors by pushing with a cart.

7. Hands must be dry when operating electrical equipment.

8. Eating and drinking beverages should be prohibited except in designated areas.

A. Handling Chemicals

1. Explosions.

   Immediately turn off open flames and other heaters. Stop addition of reagents, assist in treating victims, and vacate the area. Since toxic fumes may be present, everyone except those with designated responsibilities should vacate the area until it is decontaminated. Injured persons should be sent to the hospital.

2. Electrical Shock and Asphyxiation (Suspension of Breathing).

   The supervisor should call for medical assistance and an ambulance and use an approved resuscitation technique.

3. Ingestion of a Chemical.

   Injured should be encouraged to drink large amounts of water. Be certain to find out exactly what substance was ingested and have someone inform a hospital of exactly what chemicals are involved. Do not induce vomiting.

4. Broken Glassware.

   Alert nearby persons that broken glass may be present on the bench or on the floor. Carefully and promptly pick up large pieces with heavy leather gloves or a towel and use a dust pan and brush to remove small pieces. Discard unusable parts in a solids disposal.

C. Personal Protective Equipment

1. You should wear safety glasses whenever appropriate to do so.

2. You should not wear rings, bracelets or other jewelry which may become entangled in moving machinery.

3. Hair nets or caps should be worn to keep long hair out of moving parts.

4. Gloves should be worn when handling rough or sharp objects. Rubber gloves for handling strong chemicals should be used as needed, but never worn around moving machinery.

5. Safety shoes and hard hats should be utilized for your protection. Safety shoes, if assigned, must be worn at all times. Tennis shoes or leisure shoes should not be permitted.

6. Hearing protection must be worn in areas where designated.

7. When working in high places or entering a manhole, bin, or tank, wear a life belt, with the lifeline attached to some permanent support and held by another person. In any case, on jobs involving such risks, an extra person should be stationed close by within visual contact of the confined worker.

8. When hazardous materials are encountered, you must determine the need for special protective equipment, such as chemical-type goggles, rubber suits, rubber gloves and possibly a breathing apparatus.

9. Personal protective equipment must be worn when instructed to do so by the supervisor or in the run instructions.

D. Hand Tools - And Use

1. Select the right tool for the job.

   Examples of unsafe practices are: Pounding hardened faces of hand tools together (such as using a carpenter's hammer to hit another hammer, hatchet, or metal chisel), using a file or a screw driver for a pry, a wrench for a hammer, and pliers instead of the proper wrench.

2. Keep tools in good condition.

   Unsafe tools include wrenches with cracks or worn jaws; screw drivers with broken points, or split or broken handles; hammers with loose heads, broken or split handles; mushroomed heads on chisels, dull saws; and extension cords or electric tools with broken plugs, improper or removed grounding system, or split insulation. Return defective tools immediately to the maintenance coordinator for repairs or replacement.

3. Use tools correctly.

   Screw drivers applied to objects held in the hand; knives pulled toward the body, and failure to ground electrical equipment are common causes of accidents.

4. Keep tools in a safe place.

   Many accidents have been caused by tools falling from overhead and by knives, chisels and other sharp tools being carried in pockets or left in tool boxes with cutting edges exposed.

E. Hand Trucks

1. Do not use hand trucks with broken wheels or other defects that could cause injury.

2. Lift the pallets on trucks high enough to clear the floor.

3. Do not ride hand trucks scooter style.

4. Load the truck in such a way that you can see what is in your path. Do not load higher or wider than openings.

5. Watch where you are going when pushing or pulling a hand truck. Do not walk backwards when pulling a truck. Always be able to stop without spilling the load or loosing your balance.

6. Allow clearance for your hands when moving through doorways or past other objects. Use truck handles. Do not open doors by pushing with the load.

7. Park trucks where people will not stumble over them; leave handles in a vertical position.

8. Do not leave empty pallets in hall passageways or doorways. Do not set pallets on end against wall or other objects; always lay them flat on the floor.

9. Do not use damaged or broken pallets.

F. Lifting

1. Always use good lifting techniques as explained in the Plantwide Safety section.

2. Utilize all available powered equipment whenever possible, including the job crane, slings, or Johnson Bars.

3. Use the proper slings for the job. Do not use nylon slings on loads with sharp edges. Do not use damaged slings. Make sure your load is balanced and secure before lifting the load high. Do not walk or work under a hanging load. Keep hands and feet from under the load.

G. Spillage

If any caustic or acidic chemicals are spilled, call the supervisor immediately for assistance. All drums in the solvents area should be grounded.

H. Safety Equipment

Safety equipment must be worn at all times in the areas designated by plant policy.

I. Ladders

1. Never use a defective ladder. Tag or mark it so that it will be repaired or destroyed.

2. Be sure that a step ladder is fully open and locked before you start to climb it.

3. Tie top of extension ladder to prevent tipping or slipping or use a second person to secure the ladder.

4. Be sure that your shoes are not greasy, muddy, or slippery before you climb.

5. Hold on with both hands when going up or down. If material must be handled, raise or lower it with a rope either before going down or after climbing to the desired level.

6. Always face the ladder when ascending or descending.

7. Do not climb higher than the third rung from the top on straight or extension ladders on the second tread from the top on stepladders.

8. Never slide down a ladder.

9. Make sure the floor is clean and dry under the ladder.

10. When you are finished with a ladder, lay it down.

11. Do not set up a ladder on a wet or oily floor or if the floor has sand or oil dry on it.

J. Material Handling

1. General

a. Inspect materials for slivers, jagged edges, burrs, rough or slippery surfaces.

b. Always use proper lifting techniques as explained in the Plantwide Safety section of this manual.

c. Keep fingers away from pinch points, especially when setting down materials.

2. Electrical Hazards

a. In confined places, cover or arrange cables to prevent contact with falling sparks.

b. Never change electrodes with bare hands or wet gloves, or when standing on wet floors or grounded surfaces.

c. Ground the frames of welding units, portable or stationary, in accordance with the National Electrical Code.

d. Arrange receptacles of power cables for portable welding units so that it is impossible to remove the plug without opening the power supply switch or use plugs and receptacles which have been approved to brake full load circuits of the unit.

e. When possible, keep extension cords out of aisle ways.

K. Compressed Gas Cylinders

Consult the instructions on gas cylinder handling and use in the Plantwide Safety section of the manual.

L. Oxidizers and Flammables

Specific characteristics of oxidizers and flammables vary. Their handling and storage requirements are different and you must learn the proper methods for handling each.

Oxidizers should be stored to avoid contact with incompatible materials such as ordinary combustibles, flammable liquids, greases and other materials which might cause a reaction.

Personnel should be instructed in proper storage, handling and disposition of spilled material.

Read labels before using any chemical.

M. Electricity

1. General

a. Unless it is part of your regular work, do not attempt to repair or adjust any electrical equipment.

b. Treat all electric wires as live wires. Do not touch dangling or bare wires; report them to your supervisor.

c. Do not operate electrical equipment when it is wet or when you are standing in a wet area.

d. When using an extension cord, make sure there are no breaks in the insulation, and that plug, socket and lamp guard are in good condition.

e. Report all suspected faulty equipment to your supervisor.

f. Do not use extension cords unless absolutely necessary.

g. Keep the cords out of water.

h. Keep the cords off the floor and out of the aisles.

i. Keep all electrical boxes closed.

j. De-energize the circuit before working on equipment. "Lock Out" and remove fuses with an appropriate fuse puller.

k. Do not use heat tapes with damaged insulation or lead wires.

N. Sanitation

Accident prevention is the responsibility of all employees. Good sanitation practices can reduce the number of accidents in a plant. Poor sanitation accounts for two thirds or more of all floor falls. It is the responsibility of each employee to maintain good housekeeping in their work area.

1. Post warning signs near wet floors.

2. Don't place articles on top of stepladders, cabinets, or lockers.

3. Report any unsafe condition immediately to your supervisor.

4. Don't pick up broken glass. Sweep it up, and at once. Pick up fine splinters and chips with damp cloth or paper towel.

5. Don't empty waste baskets by reaching into them. Hold them by the sides and turn them upside down.

6. Take personal responsibility for seeing that slipping or tripping conditions anywhere on the floors or stairways are taken care of at once.

7. Unusual odors should be reported immediately to your supervisor.

8. Smoke only in designated areas.

9. Report burned-out light bulbs or florescent lamps to your supervisor.

10. Use proper lifting techniques as explained in the Plantwide Safety section.

11. Immediately mop or vacuum any water, or other liquids that are found on the floors.

12. Avoid tripping accidents by making sure that mats and runners lay flat.

13. Use caution with hot water or strong chemicals.

14. Use only pails with secure bails.

15. Be careful of spills when pushing or carrying a bucket of hot water or strong chemicals.

16. Containers which have held flammable materials should be rinsed before disposal.

17. Don't put chemicals or any solutions in unlabeled containers (all containers must be labeled unless they are empty).

18. Don't mix chemicals unless you are specifically instructed to do so.

19. Store material in carts in an orderly manner with labels visible.

20. Oily rags and dust mops must be stored in a metal container, and never be allowed to accumulate.

21. Check for and report all defective equipment, especially electric cords and plugs, etc. to your supervisor.

22. Keep equipment close to one side of a hallway, but not near a corner.

23. Do not block doorways with equipment.

24. When pushing carts or other equipment, stay to the right of the corridor.

25. Pull carts or other equipment through swinging doors. Don't ram them forward.

26. Push carts or other equipment with your hands away from the edge.

27. Approach corridor intersections slowly.

28. Unplug electrical equipment before wiping with damp cloth.

29. Handle electrical equipment only with dry hands. Never yank out extension cords, but pull by the plug, after turning the equipment off.

30. Make sure electrical equipment is grounded.

31. Make certain the switch on equipment is off, before plugging in any equipment.

32. Use proper tools and equipment for a job. Do not make substitutions for the sake of expediency.

33. When using stepladders, don't stand on the top step. Rest ladders on secure base, and be sure locking devices on stepladders are in order and are used. Never stand on chairs.

O. Pressure Fittings

1. Respect pressure, especially when air, steam or gas is contained.

2. Open blowdown or bleed valves slowly and carefully. Stand to one side. Wear a face shield whenever you are operating a blowdown or bleed valve.

3. Never open or work on tube or pipe fittings if you suspect there is pressure in the line.

4. Make sure the component parts of the tube fittings are compatible as to manufacture and material of construction.

5. Never use a ferrule or fitting of unknown identity.

6. When making new connections, check to make sure the ferrule has been crimped on the tubing properly before pressurizing.

7. Never force a fitting. If a fitting or tube nut does not engage easily, check the thread type and size to be sure the components are compatible.

8. Never use damaged or corroded tubing or fittings.

9. Use anti-seize compound whenever titanium is threaded into titanium or stainless steel into stainless steel.

10. Always pressure test new or re-worked connections with water before starting up.

11. Make sure safety valve, blowdown and bleed line tail pipes are fastened securely.

12. If severe leaks develop during a run, report them to your supervisor immediately. This could be a sign of corrosion problems.

13. Never rebend tubing. This can cause stress cracking.

14. Never use tubing of unknown identity.

15. Double check all connections for tightness before starting up.

16. When using pipe fittings, be sure of pressure rating before installation.

17. Be careful when replacing pressure gauges and pressure controllers to be sure they are rated for the pressure setting of the relief valve.

18. Do not use carbon steel or brass fittings on acid wash systems.

19. Do not use aluminum fittings (especially hose fittings) when handling caustics.

20. Temperature is as important as pressure rating when selecting fittings.

21. Valve packing must be compatible with waste, temperature and pressure.

22. High pressure can be dangerous, that is by definition anything over 10 psi.

P. General Safety Rules

1. Report to work rested and physically fit to perform your job.

2. Report all accidents and injuries to your supervisor immediately.

3. Protect your eyes with safety glasses, face shields, or goggles, as hazards exist.

4. Wear hard hats in all areas indicated.

5. Use hearing protection in areas where noise levels are excessive, over 90 dBa.

6. Wear proper respiratory equipment when toxic hazards are present.

7. Take the following precautions:

   a. Wear protective clothing which should completely cover any exposed skin and prevent liquid from coming in direct contact with the skin.

   b. Wear safety glasses and a plastic face shield.

c. Protect your hands by wearing rubber gloves or rubber coated canvas gloves while handling acids or caustic soda.

d. The sleeves of jackets or shirts should be buttoned over the glove wrists. Wear rubber boots for footwear.

e. Always pour the acid or caustic into water.

f. When disconnecting equipment for repairs, make certain that there is no internal pressure on the equipment and that the equipment has been drained, washed, and locked out.

g. Keep the equipment clean, washing off all accumulations of acid or caustic.

h. If any of these liquids should come in contact with the skin or eyes, wash off immediately with water and flood the injured area with water. In case of eye contamination, consult a physician as soon as possible.

8. Make good plant housekeeping a part of your job.

9. Always keep and maintain equipment in first class condition.

10. Use lockouts when repairing, cleaning, or inspecting machinery. Supervisors must see to it that hasps and locks are provided.

11. Exercise care in the handling and storage of compressed gas cylinders; all cylinders must be secured.

12. Properly ground all electrical equipment.

13. Keep loose materials off stairs, walkways, ramps, and platforms.

14. Do not block aisles, traffic lanes, fire exits.

15. When entering different work areas, familiarize yourself with any required safety precautions.

16. Read and thoroughly understand the maintenance and operating manuals provided by the manufacturer of any equipment.

17. Be aware of work going on around you. Keep clear of suspended loads and traffic areas.

18. Face ladders when climbing. Use only sturdy ladders on a firm base.

19. Before starting equipment, have all guards in place.

20. Operate machinery within rated capacity and at safe speeds.

21. Know locations or fire extinguishing equipment and evacuation routes.

22. Report any unsafe conditions or equipment to your supervisor.

23. Keep "horseplay" and rough housing out of your work.

24. Remember that intoxicants and non-prescribed drugs are not permitted on the job.

25. Work with care and good judgement at all times to avoid accidents - whether or not a specific rule is contained in this manual.

26. Give your wholehearted support to safety activities. Preventing your accident depends mostly on you!

MATERIAL SAFETY DATA SHEET

ICI Americas Inc.
Wilmington, Delaware 19897
Phone (302) 575-3000 (24 hours)

Form No.: M3720
Date: 12/16/81

SECTION 1 NAME & PRODUCT

Material name:
HYDRODARCO® (all grades, granular or powder)

Formula of primary component(s):
Activated carbon

SECTION 2 INGREDIENTS

	%	TLV (ACGIH)
Activated carbon	100	5 mg/m^3*
*Respirable dust. Total respirable and non-respirable dust normally contains less than 1% quartz.		
(not specification values)		

SECTION 3 PHYSICAL DATA

Boiling point: Not applicable
Vapor pressure (mm Hg at 20°C): Not applicable
Vapor density (air = 1): Not applicable
Solubility in water: Not applicable
Specific gravity: No data
% Volatile by volume: Negligible
Appearance and odor: Black granules or powder

SECTION 4 FIRE AND EXPLOSION HAZARD DATA

Flash point (and method):
Not applicable

Flammable limits (STP):
Not applicable

Extinguishing media:
Water fog, foam, carbon dioxide, dry chemical, Halon 1211.

Special fire fighting protective equipment:
Self-contained breathing apparatus in confined areas.

Unusual fire and explosion hazards:
Airborne dust is a weak explosion hazard.

SECTION 5 REACTIVITY DATA

Stability:
Stable under normal and fire conditions.

Incompatibility (materials to avoid):
Oxidizing material.

Hazardous decomposition products:
Thermal decomposition: carbon dioxide, carbon monoxide.

Hazardous polymerization:
Will not occur.

SECTION 6 HEALTH HAZARD DATA

Oral ingestion:
Relatively harmless.

Eye contact:
Nonirritating.

Skin contact:
Nonirritating.

Skin absorption:
None expected.

Inhalation (TLV or suggested control figure):
TLV 5 mg/m^3. Repeated or prolonged exposure to excessive dust may cause pulmonary disorders.

Effects of overexposure:
None.

First aid procedures:
Not applicable.

SECTION 7 SPILL OR LEAK PROCEDURES

Steps to be taken in case material is released or spilled:
Sweep or vacuum up material.

Disposal method:
Dispose of waste material in a permitted landfill for nonhazardous waste (RCRA 40 CFR 260ff). Warning: Spent product may have adsorbed hazardous materials.

SECTION 8 SPECIAL PROTECTION INFORMATION

Ventilation:
Provide local exhaust to maintain dust level below the TLV.

Respiratory protection (specify type):
MSHA-NIOSH approved respirator for dusts of substances with a TLV greater than 0.05 mg/m^3.

Protective clothing:
None.

Eye protection:
Safety glasses with side shields.

Other protective equipment:
None.

SECTION 9 SPECIAL PRECAUTIONS OR OTHER COMMENTS

Precautions to be taken in handling or storing:
An oxygen deficiency may be created when activated carbon is exposed to moisture in an enclosed space. Ventilate or wear self-contained breathing apparatus.

SAMPLE

MATERIAL SAFETY DATA SHEET

CORPORATE RESEARCH & DEVELOPMENT

SCHENECTADY, N. Y.

MATERIALS INFORMATION SERVICES

No. 8

PHOSPHORIC ACID
(75 to 85%)
Revision A

Date September 1977

SECTION I. MATERIAL IDENTIFICATION

MATERIAL NAME: PHOSPHORIC ACID (75 to 85%)

OTHER DESIGNATIONS: Orthophosphoric Acid (75 to 85%), 75 to 85% Aqueous Hydrogen Phosphate, GE Material D4A1, CAS# 007 664 382

MANUFACTURER: Available from many sources

SECTION II. INGREDIENTS AND HAZARDS	%	HAZARD DATA
Hydrogen Phosphate (H_3PO_4) Water (A 75-85% solution can be prepared by mixing 54.1 to 61.6% of P_4O_{10} in water. Impurities vary for each manufacturing plant.)	75-85 Balance	TLV 1 mg/m^3 for phosphoric acid Rat, oral LD_{50} 1530 mg/kg

SECTION III. PHYSICAL DATA

H_3PO_4 content	75%	85%	100%
Boiling pt, 1 atm, deg F	275	316	ca 500
Specific gravity (20/4C)	1.579	1.689	1.83
Melting pt, deg F	0.5	70	108
Vapor pressure, mm Hg 70 F	6.4*	2.2*	0.03
Water solubility	Completely soluble		

pH	Less than 1
Volatiles above 650 F, %	ca 100
Molecular Wt (H_3PO_4)	98.0

Apperance: 75-85% acid is water-white liquid, syrup-like in viscosity at 20C.

* High vapor pressure is due to solvent water.

SECTION IV. FIRE AND EXPLOSION DATA

Flash Point and Method	Autoignition Temp.	Flammability Limits In Air	LOWER	UPPER
None - non-flammable	N/A	N/A	N/A	N/A

Phosphoric acid, though non-flammable, can cause hazardous conditions when involved in a fire. Avoid exposure of the skin and eyes to splashes and mists; use respiratory protection as a precaution when phosphoric acid is in a fire area. Cool sealed tanks of phosphoric acid with water spray to avoid rupture from heat generated pressure.

Phosphoric acid can react with metals to liberate hydrogen, which is a flammable gas that can readily form explosive mixtures with air.

SECTION V. REACTIVITY DATA

Phosphoric acid is a stable chemical under normal conditions of storage and use. It reacts with alkalis (bases) to form phosphate salts and is corrosive (especially when hot) to many metals and alloys. (It liberates hydrogen gas when reacting with metals.)

It is classed as a strong mineral acid.

SECTION VI. HEALTH HAZARD INFORMATION

TLV 1 mg/m^3

As a strong mineral acid, phosphoric acid is corrosive and can cause irritation or burns on contact with any body tissue. (It is less hazardous in this respect than nitric or sulfuric acids.)

The low vapor pressure of H_3PO_4 at room temperature gives very little inhalation hazard, except when the acid has been formed into a mist.

Overexposure results in irritation or burns of contacted body tissue. There are no reported cases of systemic effects; it does not cause "phosphorus poisoning".

First Aid:

Eye contact - Immediately flush (including under eye lids) with much running water for 15 minutes or more. Contact physician as soon as possible.

Skin contact - Flush affected areas with much water. Remove contaminated clothing under the safety shower. Get medical assistance, except when burn is minor.

Inhalation - Remove to clean air. Restore breathing. Call physician immediately.

Ingestion - Immediately dilute the acid by ingesting large amounts of water or milk; then give milk of magnesia. Vomiting can be induced; if it occurs, continue to administer fluid, especially milk. Get medical assistance as soon as possible.

SECTION VII. SPILL, LEAK, AND DISPOSAL PROCEDURES

Neutralize spilled acid with an alkaline material, such as soda ash or lime, and/or dilute with water. Flush small amounts of the neutralized or highly diluted material to the sewer, if this is in accordance with local law. (Note that this procedure adds phosphates to surface water and may not be desirable even if it is legal.)

Phosphoric acid can be absorbed on excess alkaline solid, such as soda ash; and the solid disposed of in a suitable landfill.

Set up predetermined plans for handling large spills. Contact supplier for assistance in this planning to meet local requirements and to dispose of large amounts.

SECTION VIII. SPECIAL PROTECTION INFORMATION

Use general ventilation to keep below the TLV concentration of 1 mg/m^3. Respiratory protection is not required at normal room temperatures, except when phosphoric acid mists are formed and ventilation is inadequate to meet the TLV limits. Under these conditions, respiratory protection, such as an approved mask and canister or (for high levels of mist) self-contained breathing apparatus or masks served with an air line, should be used.

Eye protection - Use chemical safety goggles or a face shield to avoid splashes of acid into the eyes.

Other protection - Use rubber gloves and, if necessary, rubber apron, rubber clothing, etc. to avoid contact of acid with the body under the use conditions. Safety showers and eye wash fountains should be immediately available where phosphoric acid is used.

SECTION IX. SPECIAL PRECAUTIONS AND COMMENTS

Store 85% acid above 70 F, 80% acid above 40 F, and 75% acid above 0 F to prevent crystallization from occurring. (Phosphoric acid solutions will normally supercool without crystallization, but this effect is unpredictable.)

Phosphoric acids should be stored in clean ventilated storage areas with good drainage. The acid should be stored away from reactive materials, metal powders, etc. Soda ash or lime should be kept in same general areas for emergency use.

Corrosion of equipment and surfaces should be considered in areas where hot or "misted" phosphoric acid is present.

DOT classification - CORROSIVE

APPROVALS: MIS, CRD J. M. Nielsen

Industrial Hygiene and Safety

MEDICAL REVIEW:

Section 10

ANALYTICAL METHODS

ANALYSIS	METHOD #
SS	160.2
SA	160.4
PAC/BIO	ZP-115
NH_3-N	350.1, 350.2, 350.3
TKN	351.1, 351.2, 351.3, 351.4
Dissolved Oxygen	360.1, 360.2
Total Phosphorus	365.1, 365.2, 365.3, 365.4
BOD	405.1, ZP-193
COD	410.1, 410.2, 410.3, 410.4

RESIDUE, NON-FILTERABLE

Method 160.2 (Gravimetric, Dried at 103–105°C)

STORET NO. 00530

1. Scope and Application
   - 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   - 1.2 The practical range of the determination is 4 mg/l to 20,000 mg/l.
2. Summary of Method
   - 2.1 A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103–105°C.
   - 2.2 The filtrate from this method may be used for Residue, Filterable.
3. Definitions
   - 3.1 Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103–105°C.
4. Sample Handling and Preservation
   - 4.1 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
   - 4.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.
5. Interferences
   - 5.1 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.
   - 5.2 Samples high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter (7.5) minimizes this potential interference.
6. Apparatus
   - 6.1 Glass fiber filter discs, without organic binder, such as Millipore AP-40, Réeves Angel 934-AH, Gelman type A/E, or equivalent.
     NOTE: Because of the physical nature of glass fiber filters, the absolute pore size cannot be controlled or measured. Terms such as "pore size", collection efficiencies and effective retention are used to define this property in glass fiber filters. Values for these parameters vary for the filters listed above.
   - 6.2 Filter support: filtering apparatus with reservoir and a coarse (40–60 microns) fritted disc as a filter support.

Approved for NPDES
Issued 1971

NOTE: Many funnel designs are available in glass or porcelain. Some of the most common are Hirsch or Buchner funnels, membrane filter holders and Gooch crucibles. All are available with coarse fritted disc.

6.3 Suction flask.

6.4 Drying oven, 103–105°C.

6.5 Desiccator.

6.6 Analytical balance, capable of weighing to 0.1 mg.

7. Procedure

7.1 Preparation of glass fiber filter disc: Place the glass fiber filter on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible with wrinkled surface up. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus or both crucible and filter if Gooch crucible is used, and dry in an oven at 103–105°C for one hour. Remove to desiccator and store until needed. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg). Weigh immediately before use. After weighing, handle the filter or crucible/filter with forceps or tongs only.

7.2 Selection of Sample Volume

For a 4.7 cm diameter filter, filter 100 ml of sample. If weight of captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 1.0 mg of residue. If other filter diameters are used, start with a sample volume equal to 7 ml/cm^2 of filter area and collect at least a weight of residue proportional to the 1.0 mg stated above.

NOTE: If during filtration of this initial volume the filtration rate drops rapidly, or if filtration time exceeds 5 to 10 minutes, the following scheme is recommended: Use an unweighed glass fiber filter of choice affixed in the filter assembly. Add a known volume of sample to the filter funnel and record the time elapsed after selected volumes have passed through the filter. Twenty-five ml increments for timing are suggested. Continue to record the time and volume increments until fitration rate drops rapidly. Add additional sample if the filter funnel volume is inadequate to reach a reduced rate. Plot the observed time versus volume filtered. Select the proper filtration volume as that just short of the time a significant change in filtration rate occurred.

7.3 Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support.

7.4 Shake the sample vigorously and quantitatively transfer the predetermined sample volume selected in 7.2 to the filter using a graduated cylinder. Remove all traces of water by continuing to apply vacuum after sample has passed through.

7.5 With suction on, wash the graduated cylinder, filter, non-filterable residue and filter funnel wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through.

NOTE: Total volume of wash water used should equal approximately 2 ml per cm^2. For a 4.7 cm filter the total volume is 30 ml.

7.6 Carefully remove the filter from the filter support. Alternatively, remove crucible and filter from crucible adapter. Dry at least one hour at 103–105°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg).

8. Calculations

8.1 Calculate non-filterable residue as follows:

$$\text{Non-filterable residue, mg/l} = \frac{(A - B)\times 1{,}000}{C}$$

where:

A = weight of filter (or filter and crucible) + residue in mg
B = weight of filter (or filter and crucible) in mg
C = ml of sample filtered

9. Precision and Accuracy

9.1 Precision data are not available at this time.

9.2 Accuracy data on actual samples cannot be obtained.

Bibliography

1. NCASI Technical Bulletin No. 291, March 1977. National Council of the Paper Industry for Air and Stream Improvement, Inc., 260 Madison Ave., NY.

RESIDUE, VOLATILE

Method 160.4 (Gravimetric, Ignition at 550°C)

STORET NO. Total 00505
Non-Filterable 00535
Filterable 00520

1. Scope and Application
   1.1 This method determines the weight of solid material combustible at 550°C.
   1.2 The test is useful in obtaining a rough approximation of the amount of organic matter present in the solid fraction of sewage, activated sludge, industrial wastes, or bottom sediments.
2. Summary of Method
   2.1 The residue obtained from the determination of total, filterable or non-filterable residue is ignited at 550°C in a muffle furnace. The loss of weight on ignition is reported as mg/l volatile residue.
3. Comments
   3.1 The test is subject to many errors due to loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion.
   3.2 The results should not be considered an accurate measure of organic carbon in the sample, but may be useful in the control of plant operations.
   3.3 The principal source of error in the determination is failure to obtain a representative sample.
4. Sample Handling and Preservation
   4.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decompostion of solids is recommended.
5. Precision and Accuracy
   5.1 A collaborative study involving three laboratories examining four samples by means of ten replicates showed a standard deviation of ±11 mg/l at 170 mg/l volatile residue concentration.
6. Reference
   6.1 The procedure to be used for this determination is found in:
   Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 95, Method 208E, (1975).

Approved for NPDES
Issued 1971

ZP-115

Determination of Volatile Carbon/Biomass

Method: Nitric Digestion

Reagents/Special Equipment: Nitric Acid
Gooch Crucibles

References:

Issued: 1975

Determination of Biomass

The determination of biomass and carbon is done by comparing the volatile suspended solids of a nitric acid digested sample with the normal volatile suspended solids.

Procedure

1. Run normal suspended solids and ash using a 10 ml sample.
2. To another 10 ml aliquot of the sample add 20 ml concentrated nitric acid in a beaker.
3. Heat acidified sample to just under boiling (DO NOT BOIL).
4. Continue heating until sample volume has been reduced to approximately 10 ml.
5. Cool sample to room temperature (See Note).
6. Determine suspended solids and ash on the digested sample. Being careful to transfer all carbon from the beaker to the gooch (a rubber policeman will aid in the transfer).
7. Calculate results.

Calculation

$$\text{Carbon, g/l} = \frac{C-D}{0.95} \qquad \text{Biomass, g/l} = A-(B + \text{Carbon})$$

A = Normal Suspended Solids, g/l

B = Normal Suspended Ash, g/l

C = Nitric Treated Suspended Solids, g/l

D = Nitric Treated Suspended Ash, g/l

Notes

1. Nitric acid fumes are harmful if inhaled. It is required that the digestion be done in a hood, and it is recommended that filtration also be done in a hood if possible.

NITROGEN, AMMONIA

Method 350.1 (Colorimetric, Automated Phenate)

STORET NO. Total 00610
Dissolved 00608

1. Scope and Application
   1.1 This method covers the determination of ammonia in drinking, surface, and saline waters, domestic and industrial wastes in the range of 0.01 to 2.0 mg/1 NH_3 as N. This range is for photometric measurements made at 630–660 nm in a 15 mm or 50 mm tubular flow cell. Higher concentrations can be determined by sample dilution. Approximately 20 to 60 samples per hour can be analyzed.
2. Summary of Method
   2.1 Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside.
3. Sample Handling and Preservation
   3.1 Preservation by addition of 2 ml conc. H_2SO_4 per liter and refrigeration at 4°C.
4. Interferences
   4.1 Calcium and magnesium ions may be present in concentration sufficient to cause precipitation problems during analysis. A 5% EDTA solution is used to prevent the precipitation of calcium and magnesium ions from river water and industrial waste. For sea water a sodium potassium tartrate solution is used.
   4.2 Sample turbidity and color may interfere with this method. Turbidity must be removed by filtration prior to analysis. Sample color that absorbs in the photometric range used will also interfere.
5. Apparatus
   5.1 Technicon AutoAnalyzer Unit (AAI or AAII) consisting of:
      5.1.1 Sampler.
      5.1.2 Manifold (AAI) or Analytical Cartridge (AAII).
      5.1.3 Proportioning pump.
      5.1.4 Heating bath with double delay coil (AAI).
      5.1.5 Colorimeter equipped with 15 mm tubular flow cell and 630–660 nm filters.
      5.1.6 Recorder.
      5.1.7 Digital printer for AAII (optional).

Approved for NPDES following preliminary distillation, Method 350.2.
Issued 1974
Editorial revision 1978

6. Reagents

6.1 Distilled water: Special precaution must be taken to insure that distilled water is free of ammonia. Such water is prepared by passage of distilled water through an ion exchange column comprised of a mixture of both strongly acidic cation and strongly basic anion exchange resins. The regeneration of the ion exchange column should be carried out according to the instruction of the manufacturer.

NOTE 1: All solutions must be made using ammonia-free water.

6.2 Sulfuric acid 5N: Air scrubber solution. Carefully add 139 ml of conc. sulfuric acid to approximately 500 ml of ammonia-free distilled water. Cool to room temperature and dilute to 1 liter with ammonia-free distilled water.

6.3 Sodium phenolate: Using a 1 liter Erlenmeyer flask, dissolve 83 g phenol in 500 ml of distilled water. In small increments, cautiously add with agitation, 32 g of NaOH. Periodically cool flask under water faucet. When cool, dilute to 1 liter with distilled water.

6.4 Sodium hypochlorite solution: Dilute 250 ml of a bleach solution containing 5.25% NaOCl (such as "Clorox") to 500 ml with distilled water. Available chlorine level should approximate 2 to 3%. Since "Clorox" is a proprietary product, its formulation is subject to change. The analyst must remain alert to detecting any variation in this product significant to its use in this procedure. Due to the instability of this product, storage over an extended period should be avoided.

6.5 Disodium ethylenediamine-tetraacetate (EDTA) (5%): Dissolve 50 g of EDTA (disodium salt) and approximately six pellets of NaOH in 1 liter of distilled water.

NOTE 2: On salt water samples where EDTA solution does not prevent precipitation of cations, sodium potassium tartrate solution may be used to advantage. It is prepared as follows:

6.5.1 Sodium potassium tartrate solution: 10% $NaKC_4H_4O_6 \cdot 4H_2O$. To 900 ml of distilled water add 100 g sodium potassium tartrate. Add 2 pellets of NaOH and a few boiling chips, boil gently for 45 minutes. Cover, cool, and dilute to 1 liter with ammonia-free distilled water. Adjust pH to 5.2 ±.05 with H_2SO_4. After allowing to settle overnight in a cool place, filter to remove precipitate. Then add 1/2 ml Brij-35[4] (available from Technicon Corporation) solution and store in stoppered bottle.

6.6 Sodium nitroprusside (0.05%): Dissolve 0.5 g of sodium nitroprusside in 1 liter of distilled water.

6.7 Stock solution: Dissolve 3.819 g of anhydrous ammonium chloride, NH_4Cl, dried at 105°C, in distilled water, and dilute to 1000 ml. 1.0 ml = 1.0 mg NH_3–N.

6.8 Standard Solution A: Dilute 10.0 ml of stock solution (6.7) to 1000 ml with distilled water. 1.0 ml = 0.01 mg NH_3–N.

6.9 Standard solution B: Dilute 10.0 ml of standard solution A (6.8) to 100.0 ml with distilled water. 1.0 ml = 0.001 mg NH_3–N.

6.10 Using standard solutions A and B, prepare the following standards in 100 ml volumetric flasks (prepare fresh daily):

NH_3–N, mg/l	ml Standard Solution/100 ml
	Solution B
0.01	1.0
0.02	2.0
0.05	5.0
0.10	10.0
	Solution A
0.20	2.0
0.50	5.0
0.80	8.0
1.00	10.0
1.50	15.0
2.00	20.0

NOTE 3: When saline water samples are analyzed, Substitute Ocean Water (SOW) should be used for preparing the above standards used for the calibration curve; otherwise, distilled water is used. If SOW is used, subtract its blank background response from the standards before preparing the standard curve.

Substitute Ocean Water (SOW)

NaCl	24.53 g/l	$NaHCO_3$	0.20 g/l
$MgCl_2$	5.20 g/l	KBr	0.10 g/l
Na_2SO_4	4.09 g/l	H_3BO_3	0.03 g/l
$CaCl_2$	1.16 g/l	$SrCl_2$	0.03 g/l
KCl	0.70 g/l	NaF	0.003 g/l

7. Procedure

7.1 Since the intensity of the color used to quantify the concentration is pH dependent, the acid concentration of the wash water and the standard ammonia solutions should approximate that of the samples. For example, if the samples have been preserved with 2 ml conc. H_2SO_4/liter, the wash water and standards should also contain 2 ml conc. H_2SO_4/liter.

7.2 For a working range of 0.01 to 2.00 mg NH_3–N/l (AAI), set up the manifold as shown in Figure 1. For a working range of .01 to 1.0 mg NH_3–N/l (AAII), set up the manifold as shown in Figure 2. Higher concentrations may be accommodated by sample dilution.

7.3 Allow both colorimeter and recorder to warm up for 30 minutes. Obtain a stable baseline with all reagents, feeding distilled water through sample line.

7.4 For the AAI system, sample at a rate of 20/hr, 1:1. For the AAII use a 60/hr 6:1 cam with a common wash.

7.5 Arrange ammonia standards in sampler in order of decreasing concentration of nitrogen. Complete loading of sampler tray with unknown samples.

7.6 Switch sample line from distilled water to sampler and begin analysis.

8. Calculations

8.1 Prepare appropriate standard curve derived from processing ammonia standards through manifold. Compute concentration of samples by comparing sample peak heights with standard curve.

9. Precision and Accuracy

9.1 In a single laboratory (EMSL), using surface water samples at concentrations of 1.41, 0.77, 0.59 and 0.43 mg NH_3–N/l, the standard deviation was ±0.005.

9.2 In a single laboratory (EMSL), using surface water samples at concentrations of 0.16 and 1.44 mg NH_3–N/l, recoveries were 107% and 99%, respectively.

Bibliography

1. Hiller, A., and Van Slyke, D., "Determination of Ammonia in Blood", J. Biol. Chem. 102, p 499 (1933).
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3. Fiore, J., and O'Brien, J. E., "Ammonia Determination by Automatic Analysis", Wastes Engineering 33, p 352 (1962).
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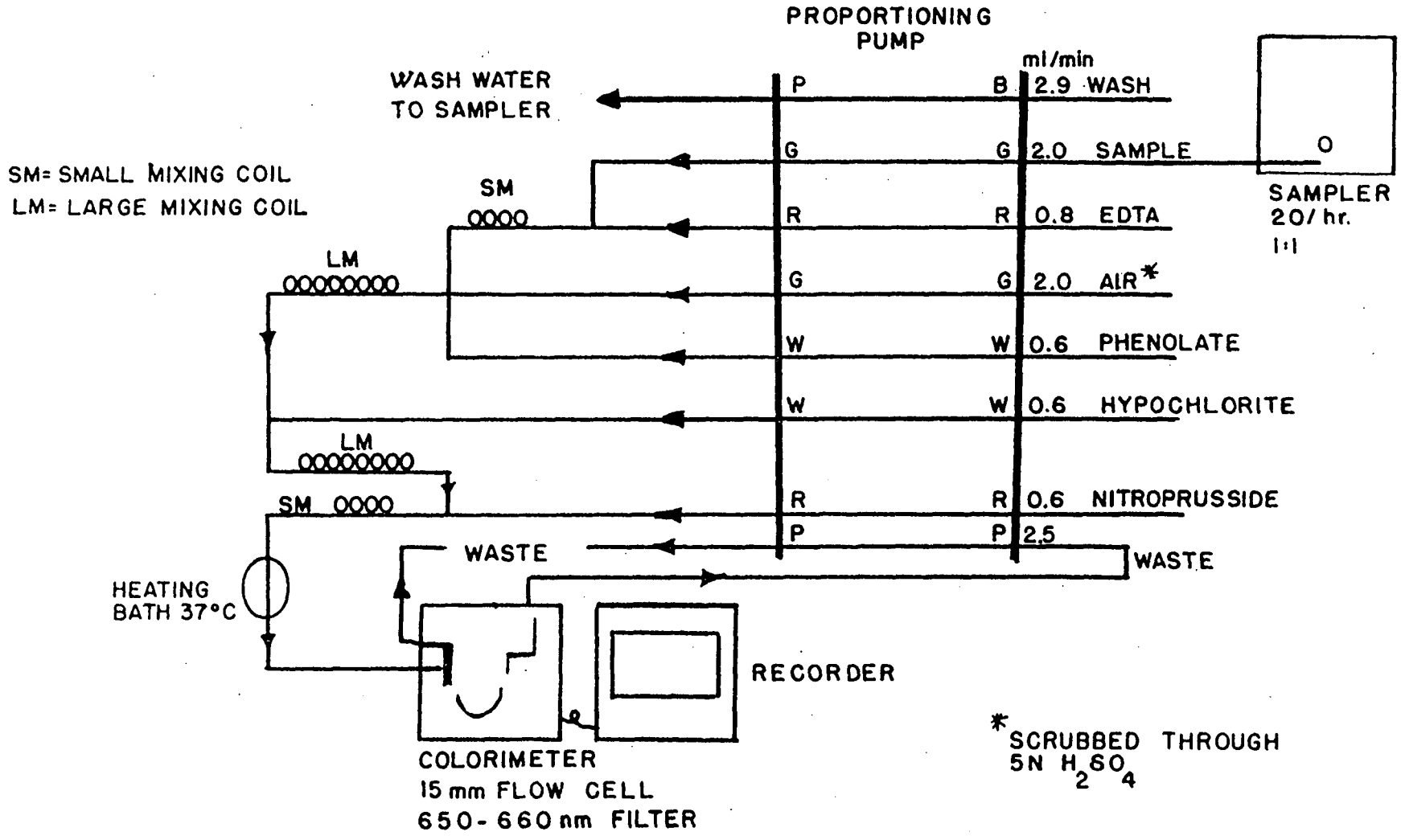
5. ASTM "Manual on Industrial Water and Industrial Waste Water", 2nd Ed., 1966 printing, p 418.
6. Booth, R. L., and Lobring, L. B., "Evaluation of the AutoAnalyzer II: A Progress Report" in Advances in Automated Analysis: 1972 Technicon International Congress, Vol. 8, p 7–10, Mediad Incorporated, Tarrytown, N.Y., (1973).
7. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 616, Method 604 (1975).



350.1-5

FIGURE 1 AMMONIA MANIFOLD AA I

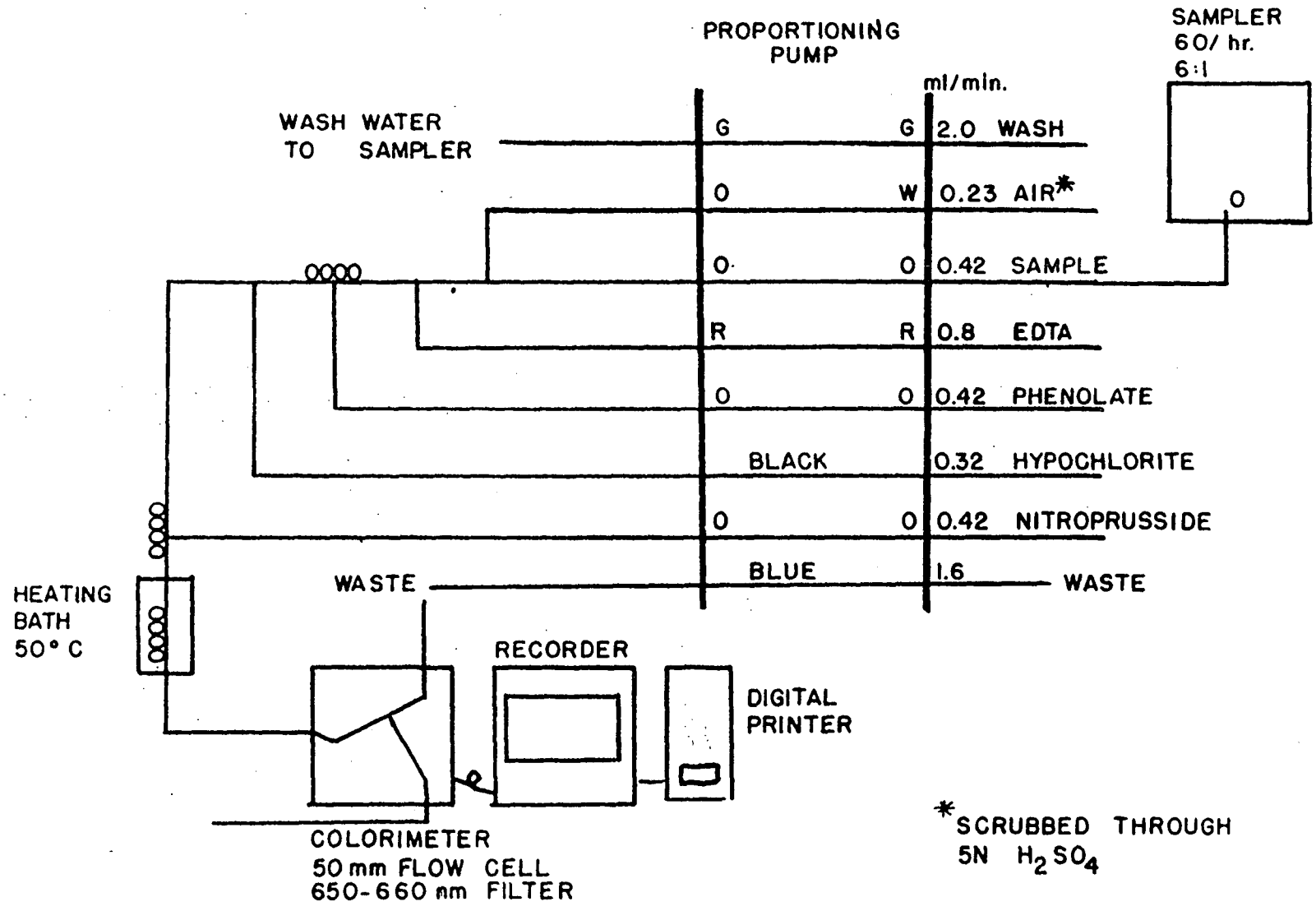


FIGURE 2. AMMONIA MANIFOLD AA II

NITROGEN, AMMONIA

Method 350.2 (Colorimetric; Titrimetric; Potentiometric – Distillation Procedure)

STORET NO. Total 00610
Dissolved 00608

1. Scope and Application
   1.1 This distillation method covers the determination of ammonia-nitrogen exclusive of total Kjeldahl nitrogen, in drinking, surface and saline waters, domestic and industrial wastes. It is the method of choice where economics and sample load do not warrant the use of automated equipment.
   1.2 The method covers the range from about 0.05 to 1.0 mg NH_3–N/l for the colorimetric procedure, from 1.0 to 25 mg/l for the titrimetric procedure, and from 0.05 to 1400 mg/l for the electrode method.
   1.3 This method is described for macro glassware; however, micro distillation equipment may also be used.
2. Summary of Method
   2.1 The sample is buffered at a pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds, and is then distilled into a solution of boric acid. The ammonia in the distillate can be determined colorimetrically by nesslerization, titrimetrically with standard sulfuric acid with the use of a mixed indicator, or potentiometrically by the ammonia electrode. The choice between the first two procedures depends on the concentration of the ammonia.
3. Sample Handling and Preservation
   3.1 Samples may be preserved with 2 ml of conc. H_2SO_4 per liter and stored at 4°C.
4. Interferences
   4.1 A number of aromatic and aliphatic amines, as well as other compounds, both organic and inorganic, will cause turbidity upon the addition of Nessler reagent, so direct nesslerization (i.e., without distillation), has been discarded as an official method.
   4.2 Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out. Volatile alkaline compounds, such as certain ketones, aldehydes, and alcohols, may cause an off-color upon nesslerization in the distillation method. Some of these, such as formaldehyde, may be eliminated by boiling off at a low pH (approximately 2 to 3) prior to distillation and nesslerization.
   4.3 Residual chlorine must also be removed by pretreatment of the sample with sodium thiosulfate before distillation.

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5. Apparatus

5.1 An all-glass distilling apparatus with an 800–1000 ml flask.

5.2 Spectrophotometer or filter photometer for use at 425 nm and providing a light path of 1 cm or more.

5.3 Nessler tubes: Matched Nessler tubes (APHA Standard) about 300 mm long, 17 mm inside diameter, and marked at 225 mm ±1.5 mm inside measurement from bottom.

5.4 Erlenmeyer flasks: The distillate is collected in 500 ml glass-stoppered flasks. These flasks should be marked at the 350 and the 500 ml volumes. With such marking, it is not necessary to transfer the distillate to volumetric flasks.

6. Reagents

6.1 Distilled water should be free of ammonia. Such water is best prepared by passage through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin. Regeneration of the column should be carried out according to the manufacturer's instructions.

NOTE 1: All solutions must be made with ammonia-free water.

6.2 Ammonium chloride, stock solution: 1.0 ml = 1.0 mg NH_3–N. Dissolve 3.819 g NH_4Cl in distilled water and bring to volume in a 1 liter volumetric flask.

6.3 Ammonium chloride, standard solution: 1.0 ml = 0.01 mg. Dilute 10.0 ml of stock solution (6.2) to 1 liter in a volumetric flask.

6.4 Boric acid solution (20 g/1): Dissolve 20 g H_3BO_3 in distilled water and dilute to 1 liter.

6.5 Mixed indicator: Mix 2 volumes of 0.2% methyl red in 95% ethyl alcohol with 1 volume of 0.2% methylene blue in 95% ethyl alcohol. This solution should be prepared fresh every 30 days.

NOTE 2: Specially denatured ethyl alcohol conforming to Formula 3A or 30 of the U.S. Bureau of Internal Revenue may be substituted for 95% ethanol.

6.6 Nessler reagent: Dissolve 100 g of mercuric iodide and 70 g of potassium iodide in a small amount of water. Add this mixture slowly, with stirring, to a cooled solution of 160 g of NaOH in 500 ml of water. Dilute the mixture to 1 liter. If this reagent is stored in a Pyrex bottle out of direct sunlight, it will remain stable for a period of up to 1 year.

NOTE 3: This reagent should give the characteristic color with ammonia within 10 minutes after addition, and should not produce a precipitate with small amounts of ammonia (0.04 mg in a 50 ml volume).

6.7 Borate buffer: Add 88 ml of 0.1 N NaOH solution to 500 ml of 0.025 M sodium tetraborate solution (5.0 g anhydrous $Na_2B_4O_7$ or 9.5 g $Na_2B_4O_7 \cdot 10H_2O$ per liter) and dilute to 1 liter.

6.8 Sulfuric acid, standard solution: (0.02 N, 1 ml = 0.28 mg NH_3–N). Prepare a stock solution of approximately 0.1 N acid by diluting 3 ml of conc. H_2SO_4 (sp. gr. 1.84) to 1 liter with CO_2-free distilled water. Dilute 200 ml of this solution to 1 liter with CO_2-free distilled water.

NOTE 4: An alternate and perhaps preferable method is to standardize the approximately 0.1 N H_2SO_4 solution against a 0.100 N Na_2CO_3 solution. By proper dilution the 0.02 N acid can then be prepared.

6.8.1 Standardize the approximately 0.02 N acid against 0.0200 N Na_2CO_3 solution. This last solution is prepared by dissolving 1.060 g anhydrous Na_2CO_3, oven-dried at 140°C, and diluting to 1000 ml with CO_2-free distilled water.

6.9 Sodium hydroxide, 1 N: Dissolve 40 g NaOH in ammonia-free water and dilute to 1 liter.

6.10 Dechlorinating reagents: A number of dechlorinating reagents may be used to remove residual chlorine prior to distillation. These include:

a. Sodium thiosulfate (1/70 N): Dissolve 3.5 g $Na_2S_2O_3 \cdot 5H_2O$ in distilled water and dilute to 1 liter. One ml of this solution will remove 1 mg/l of residual chlorine in 500 ml of sample.

b. Sodium arsenite (1/70 N): Dissolve 1.0 g $NaAsO_2$ in distilled water and dilute to 1 liter.

7. Procedure

7.1 Preparation of equipment: Add 500 ml of distilled water to an 800 ml Kjeldahl flask. The addition of boiling chips which have been previously treated with dilute NaOH will prevent bumping. Steam out the distillation apparatus until the distillate shows no trace of ammonia with Nessler reagent.

7.2 Sample preparation: Remove the residual chlorine in the sample by adding dechlorinating agent equivalent to the chlorine residual. To 400 ml of sample add 1 N NaOH (6.9), until the pH is 9.5, checking the pH during addition with a pH meter or by use of a short range pH paper.

7.3 Distillation: Transfer the sample, the pH of which has been adjusted to 9.5, to an 800 ml Kjeldahl flask and add 25 ml of the borate buffer (6.7). Distill 300 ml at the rate of 6–10 ml/min. into 50 ml of 2% boric acid (6.4) contained in a 500 ml Erlenmeyer flask.

NOTE 5: The condenser tip or an extension of the condenser tip must extend below the level of the boric acid solution.

Dilute the distillate to 500 ml with distilled water and nesslerize an aliquot to obtain an approximate value of the ammonia-nitrogen concentration. For concentrations above 1 mg/l the ammonia should be determined titrimetrically. For concentrations below this value it is determined colorimetrically. The electrode method may also be used.

7.4 Determination of ammonia in distillate: Determine the ammonia content of the distillate titrimetrically, colorimetrically or potentiometrically as described below.

7.4.1 Titrimetric determination: Add 3 drops of the mixed indicator to the distillate and titrate the ammonia with the 0.02 N H_2SO_4, matching the end point against a blank containing the same volume of distilled water and H_3BO_3 solution.

7.4.2 Colorimetric determination: Prepare a series of Nessler tube standards as follows:

ml of Standard 1.0 ml = 0.01 mg NH_3–N	mg NH_3–N/50.0 ml
0.0	0.0
0.5	0.005
1.0	0.01
2.0	0.02
3.0	0.03
4.0	0.04
5.0	0.05
8.0	0.08
10.0	0.10

Dilute each tube to 50 ml with distilled water, add 2.0 ml of Nessler reagent (6.6) and mix. After 20 minutes read the absorbance at 425 nm against the blank. From the values obtained plot absorbance vs. mg NH_3–N for the standard curve. Determine the ammonia in the distillate by nesslerizing 50 ml or an aliquot diluted to 50 ml and reading the absorbance at 425 nm as described above for the standards. Ammonia-nitrogen content is read from the standard curve.

7.4.3 Potentiometric determination: Consult the method entitled Nitrogen, Ammonia: Selective Ion Electrode Method (Method 350.3) in this manual.

7.5 It is not imperative that all standards be distilled in the same manner as the samples. It is recommended that at least two standards (a high and low) be distilled and compared to similar values on the curve to insure that the distillation technique is reliable. If distilled standards do not agree with undistilled standards the operator should find the cause of the apparent error before proceeding.

8. Calculations

8.1 Titrimetric

$$\text{mg/l } NH_3 - N = \frac{A \times 0.28 \times 1{,}000}{S}$$

where:

A = ml 0.02 N H_2SO_4 used.

S = ml sample.

8.2 Spectrophotometric

$$\text{mg/l } NH_3 - N = \frac{A \times 1{,}000}{D} \times \frac{B}{C}$$

where:

A = mg NH_3–N read from standard curve.

B = ml total distillate collected, including boric acid and dilution.

C = ml distillate taken for nesslerization.

D = ml of original sample taken.

8.3 Potentiometric

$$mg/l\ NH_3 - N = \frac{500}{D} \times A$$

where:

A = mg NH_3–N/l from electrode method standard curve.

D = ml of original sample taken.

9. Precision and Accuracy

9.1 Twenty-four analysts in sixteen laboratories analyzed natural water samples containing exact increments of an ammonium salt, with the following results:

Increment as Nitrogen, Ammonia mg N/liter	Precision as Standard Deviation mgN/liter	Accuracy as Bias, %	Accuracy as Bias, mg N/liter
0.21	0.122	-5.54	-0.01
0.26	0.070	-18.12	-0.05
1.71	0.244	+0.46	+0.01
1.92	0.279	-2.01	-0.04

(FWPCA Method Study 2, Nutrient Analyses)

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NITROGEN, AMMONIA

Method 350.3 (Potentiometric, Ion Selective Electrode)

STORET NO. Total 00610
Dissolved 00608

1. Scope and Application
   1.1 This method is applicable to the measurement of ammonia-nitrogen in drinking, surface and saline waters, domestic and industrial wastes.
   1.2 This method covers the range from 0.03 to 1400 mg NH_3–N/1. Color and turbidity have no effect on the measurements, thus, distillation may not be necessary.
2. Summary of Method
   2.1 The ammonia is determined potentiometrically using an ion selective ammonia electrode and a pH meter having an expanded millivolt scale or a specific ion meter.
   2.2 The ammonia electrode uses a hydrophobic gas-permeable membrane to separate the sample solution from an ammonium chloride internal solution. Ammonia in the sample diffuses through the membrane and alters the pH of the internal solution, which is sensed by a pH electrode. The constant level of chloride in the internal solution is sensed by a chloride selective ion electrode which acts as the reference electrode.
3. Sample Handling and Preservation
   3.1 Samples may be preserved with 2 ml of conc. H_2SO_4 per liter and stored at 4°C.
4. Interferences
   4.1 Volatile amines act as a positive interference.
   4.2 Mercury interferes by forming a strong complex with ammonia. Thus the samples cannot be preserved with mercuric chloride.
5. Apparatus
   5.1 Electrometer (pH meter) with expanded mV scale or a specific ion meter.
   5.2 Ammonia selective electrode, such as Orion Model 95–10 or EIL Model 8002–2.
   5.3 Magnetic stirrer, thermally insulated, and Teflon-coated stirring bar.
6. Reagents
   6.1 Distilled water: Special precautions must be taken to insure that the distilled water is free of ammonia. This is accomplished by passing distilled water through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin.
   6.2 Sodium hydroxide, 10N: Dissolve 400 g of sodium hydroxide in 800 ml of distilled water. Cool and dilute to 1 liter with distilled water (6.1).
   6.3 Ammonium chloride, stock solution: 1.0 ml = 1.0 mg NH_3–N. Dissolve 3.819 g NH_4Cl in water and bring to volume in a 1 liter volumetric flask using distilled water (6.1).

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Approved for NPDES following preliminary distillation (Method 350.2)

6.4 Ammonium chloride, standard solution: 1.0 ml = 0.01 mg NH_3–N. Dilute 10.0 ml of the stock solution (6.3) to 1 liter with distilled water (6.1) in a volumetric flask.

NOTE 1: When analyzing saline waters, standards must be made up in synthetic ocean water (SOW); found in Nitrogen, Ammonia: Colorimetric, Automated Phenate Method (350.1).

7. Procedure

7.1 Preparation of standards: Prepare a series of standard solutions covering the concentration range of the samples by diluting either the stock or standard solutions of ammonium chloride.

7.2 Calibration of electrometer: Place 100 ml of each standard solution in clean 150 ml beakers. Immerse electrode into standard of lowest concentration and add 1 ml of 10N sodium hydroxide solution while mixing. Keep electrode in the solution until a stable reading is obtained.

NOTE 2: The pH of the solution after the addition of NaOH must be above 11.

Caution: Sodium hydroxide must not be added prior to electrode immersion, for ammonia may be lost from a basic solution.

7.3 Repeat this procedure with the remaining standards, going from lowest to highest concentration. Using semilogarithmic graph paper, plot the concentration of ammonia in mg NH_3–N/1 on the log axis vs. the electrode potential developed in the standard on the linear axis, starting with the lowest concentration at the bottom of the scale.

7.4 Calibration of a specific ion meter: Follow the directions of the manufacturer for the operation of the instrument.

7.5 Sample measurement: Follow the procedure in (7.2) for 100 ml of sample in 150 ml beakers. Record the stabilized potential of each unknown sample and convert the potential reading to the ammonia concentration using the standard curve. If a specific ion meter is used, read the ammonia level directly in mg NH_3–N/1.

8. Precision and Accuracy

8.1 In a single laboratory (EMSL), using surface water samples at concentrations of 1.00, 0.77, 0.19, and 0.13 mg NH_3–N/1, standard deviations were ±0.038, ±0.017, ±0.007, and ±0.003, respectively.

8.2 In a single laboratory (EMSL), using surface water samples at concentrations of 0.19 and 0.13 mg NH_3–N/1, recoveries were 96% and 91%, respectively.

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NITROGEN, KJELDAHL, TOTAL

Method 351.1 (Colorimetric, Automated Phenate)

STORET NO. 00625

1. Scope and Application
   1.1 This automated method may be used to determine Kjeldahl nitrogen in surface and saline waters. The applicable range is 0.05 to 2.0 mg N/1. Approximately 20 samples per hour can be analyzed.
2. Summary of Method
   2.1 The sample is automatically digested with a sulfuric acid solution containing potassium sulfate and mercuric sulfate as a catalyst to convert organic nitrogen to ammonium sulfate. The solution is then automatically neutralized with sodium hydroxide solution and treated with alkaline phenol reagent and sodium hypochlorite reagent. This treatment forms a blue color designated as indophenol. Sodium nitroprusside, which increases the intensity of the color, is added to obtain necessary sensitivity for measurement of low level nitrogen.
3. Definitions
   3.1 Total Kjeldahl nitrogen is defined as the sum of free-ammonia and of organic nitrogen compounds which are converted to $(NH_4)_2SO_4$ under the conditions of digestion which are specified below.
   3.2 Organic Kjeldahl nitrogen is defined as the difference obtained by subtracting the free-ammonia value from the total Kjeldahl nitrogen value. Also, organic Kjeldahl nitrogen may be determined directly by removal of ammonia before digestion.
4. Sample Handling and Preservation
   4.1 Samples may be preserved by addition of 2 ml of conc. H_2SO_4 per liter and refrigeration at 4°C. Even when preserved in this manner, conversion of organic nitrogen to ammonia may occur. Therefore, samples should be analyzed as soon as possible.
5. Interferences
   5.1 Iron and chromium ions tend to catalyze while copper ions tend to inhibit the indophenol color reaction.
6. Apparatus
   6.1 Technicon AutoAnalyzer consisting of:
      6.1.1 Sampler II, equipped with continuous mixer.
      6.1.2 Two proportioning pumps.
      6.1.3 Manifold I.
      6.1.4 Manifold II.
      6.1.5 Continuous digester.
      6.1.6 Planetary pump.

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6.1.7 Five-gallon Carboy fume-trap.

6.1.8 80°C Heating bath.

6.1.9 Colorimeter equipped with 50 mm tubular flow cell and 630 nm filters.

6.1.10 Recorder equipped with range expander.

6.1.11 Vacuum pump.

7. Reagents

7.1 Distilled water: Special precaution must be taken to insure that distilled water is free of ammonia. Such water is prepared by passage of distilled water through an ion exchange column comprised of a mixture of both strongly acidic cation and strongly basic anion exchange resins. Furthermore, since organic contamination may interfere with this analysis, use of the resin Dowex XE-75 or equivalent which also tends to remove organic impurities is advised. The regeneration of the ion exchange column should be carried out according to the instruction of the manufacturer.

NOTE 1: All solutions must be made using ammonia-free water.

7.2 Sulfuric acid: As it readily absorbs ammonia, special precaution must also be taken with respect to its use. Do not store bottles reserved for this determination in areas of potential ammonia contamination.

7.3 EDTA (2% solution): Dissolve 20 g disodium ethylenediamine tetraacetate in 1 liter of distilled water. Adjust pH to 10.5–11 with NaOH (7.4).

7.4 Sodium hydroxide (30% solution): Dissolve 300 g NaOH in 1 liter of distilled water.

NOTE 2: The 30% sodium hydroxide should be sufficient to neutralize the digestate. In rare cases it may be necessary to increase the concentration of sodium hydroxide in this solution to insure neutralization of the digested sample in the manifold at the water jacketed mixing coil.

7.5 Sodium nitroprusside, (0.05% solution): Dissolve 0.5 g $Na_2Fe(CN)_5NO{\cdot}2H_2O$ in 1 liter distilled water.

7.6 Alkaline phenol reagent: Pour 550 ml liquid phenol (88–90%) slowly with mixing into 1 liter of 40% (400 g per liter) NaOH. Cool and dilute to 2 liters with distilled water.

7.7 Sodium hypochlorite (1% solution): Dilute commercial "Clorox"–200 ml to 1 liter with distilled water. Available chlorine level should be approximately 1%. Due to the instability of this product, storage over an extended period should be avoided.

7.8 Digestant mixture: Place 2 g red HgO in a 2 liter container. Slowly add, with stirring, 300 ml of acid water (100 ml H_2SO_4 + 200 ml H_2O) and stir until cool. Add 100 ml 10% (10 g per 100 ml) K_2SO_4. Dilute to 2 liters with conc. sulfuric acid (approximately 500 ml at a time, allowing time for cooling). Allow 4 hours for the precipitate to settle or filter through glass fiber filter.

7.9 Stock solution: Dissolve 4.7193 g of pre-dried (1 hour at 105°C) ammonium sulfate in distilled water and dilute to 1.0 liter in a volumetric flask. 1.0 ml = 1.0 mg N.

7.10 Standard solution: Dilute 10.0 ml of stock solution (7.9) to 1000 ml. 1.0 ml = 0.01 mg N.

7.11 Using the standard solution (7.10), prepare the following standards in 100 ml volumetric flasks:

Conc., mg N/l	ml Standard Solution/100 ml
0.00	0.0
0.05	0.5
0.10	1.0
0.20	2.0
0.40	4.0
0.60	6.0
0.80	8.0
1.00	10.0
1.50	15.0
2.00	20.0

8. Procedure

8.1 Set up manifolds as shown in Figures 1, 2, and 3.

8.1.1 In the operation of manifold No. 1, the control of four key factors is required to enable manifold No. 2 to receive the mandatory representative feed. First, the digestant flowing into the pulse chamber (PC-1) must be bubble free; otherwise, air will accumulate in A-7, thus altering the ratio of sample to digestant in digestor. Second, in maintaining even flow from the digestor helix, the peristaltic pump must be adjusted to cope with differences in density of the digestate and the wash water. Third, the sample pick-up rate from the helix must be precisely adjusted to insure that the entire sample is aspirated into the mixing chamber. And finally, the contents of the "Mixing Chamber" must be kept homogeneous by the proper adjustment of the air bubbling rate.

8.1.2 In the operation of manifold No. 2, it is important in the neutralization of the digested sample to adjust the concentration of the NaOH so that the waste from the C-3 debubbler is slightly acid to Hydrion B paper.

8.1.3 The digestor temperature is 390°C for the first stage and 360°C for the second and third stages.

8.2 Allow both colorimeter and recorder to warm up for 30 minutes. Run a baseline with all reagents, feeding distilled water through the sample line. Adjust dark current and operative opening on colorimeter to obtain stable baseline.

8.3 Set sampling rate of Sampler II at 20 samples per hour, using a sample to wash ratio of 1 to 2 (1 minute sample, 2 minute wash).

8.4 Arrange various standards in sampler cups in order of increasing concentration. Complete loading of sampler tray with unknown samples.

8.5 Switch sample line from distilled water to sampler and begin analysis.

9. Calculation

9.1 Prepare standard curve by plotting peak heights of processed standards against concentration values. Compute concentration of samples by comparing sample peak heights with standard curve.

9.2 Any sample that has a computed concentration that is less than 10% of the sample run immediately prior to it must be rerun.

10. Precision and Accuracy

10.1 Six laboratories analyzed four natural water samples containing exact increments of organic nitrogen compounds, with the following results:

Increment as Kjeldahl-Nitrogen mg N/liter	Precision as Standard Deviation Kjeldahl–N mg N/liter	Accuracy as Bias, %	Accuracy as Bias, mg N/liter
1.89	0.54	–24.6	–0.46
2.18	0.61	–28.3	–0.62
5.09	1.25	–23.8	–1.21
5.81	1.85	–21.9	–1.27

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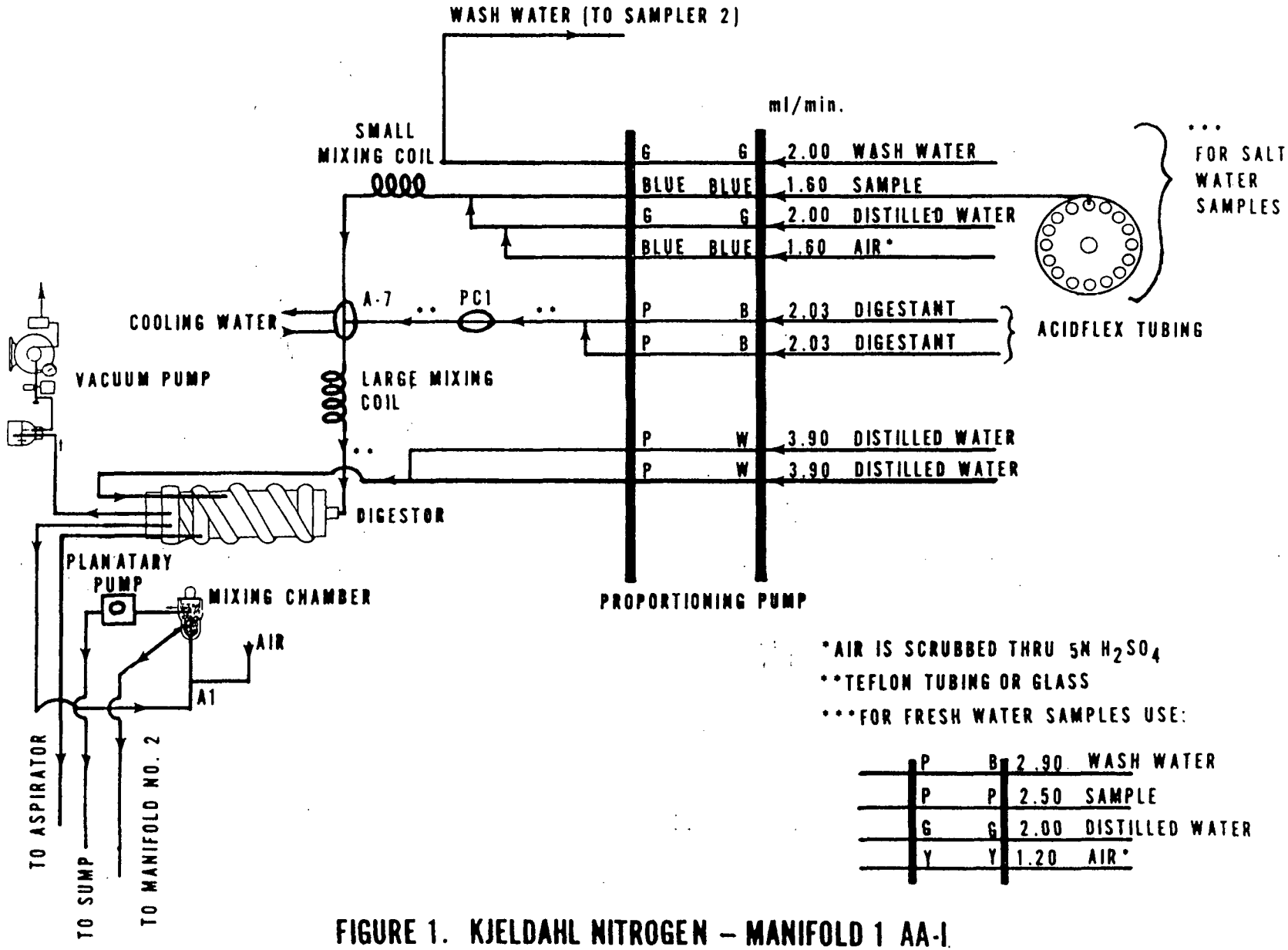
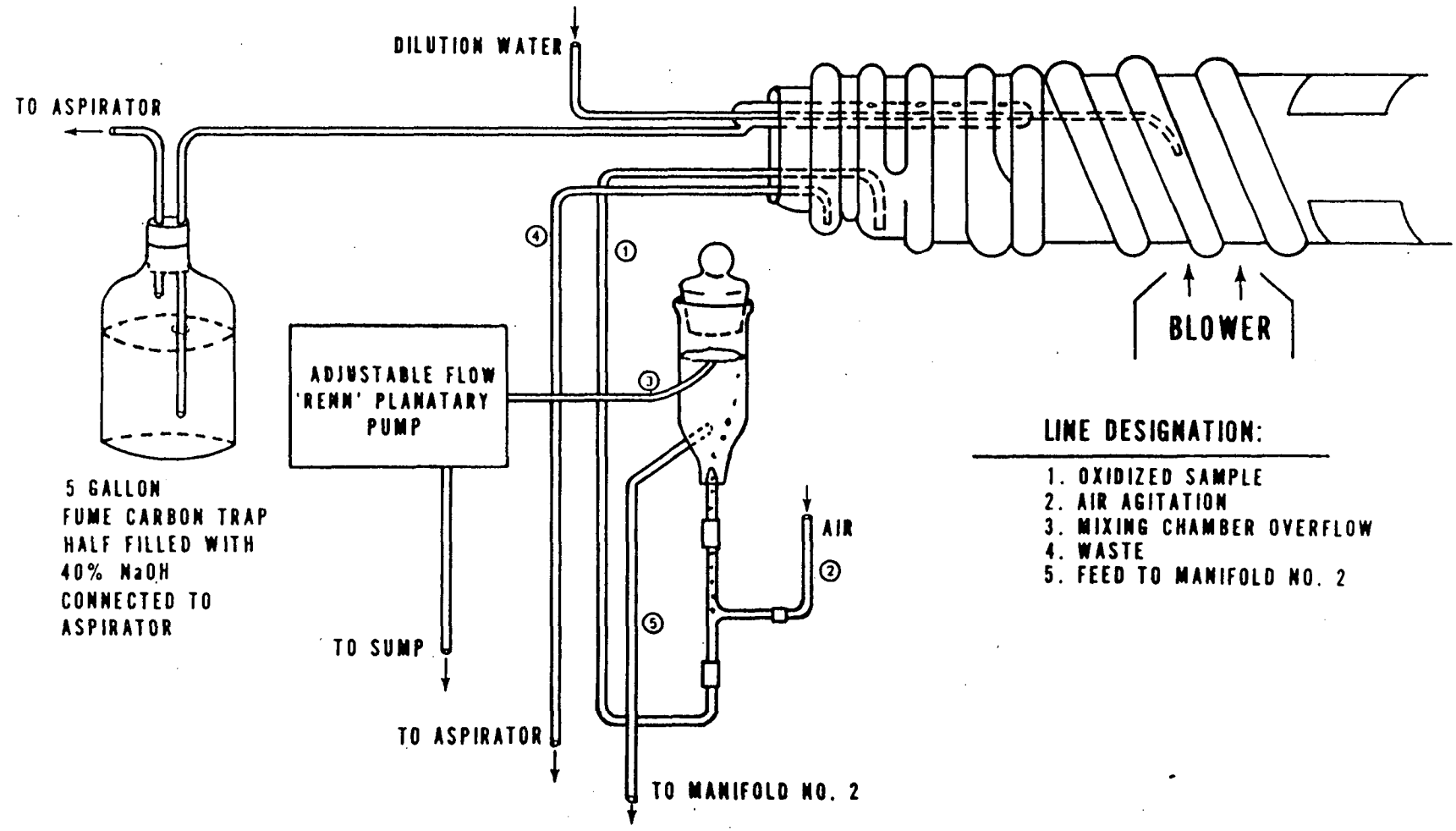


351.1-5

FIGURE 1. KJELDAHL NITROGEN – MANIFOLD 1 AA-I

CONTINUOUS DIGESTER & MIXING CHAMBER ASSEMBLY



351.1-6

FIGURE 2. KJELDAHL NITROGEN AA-I

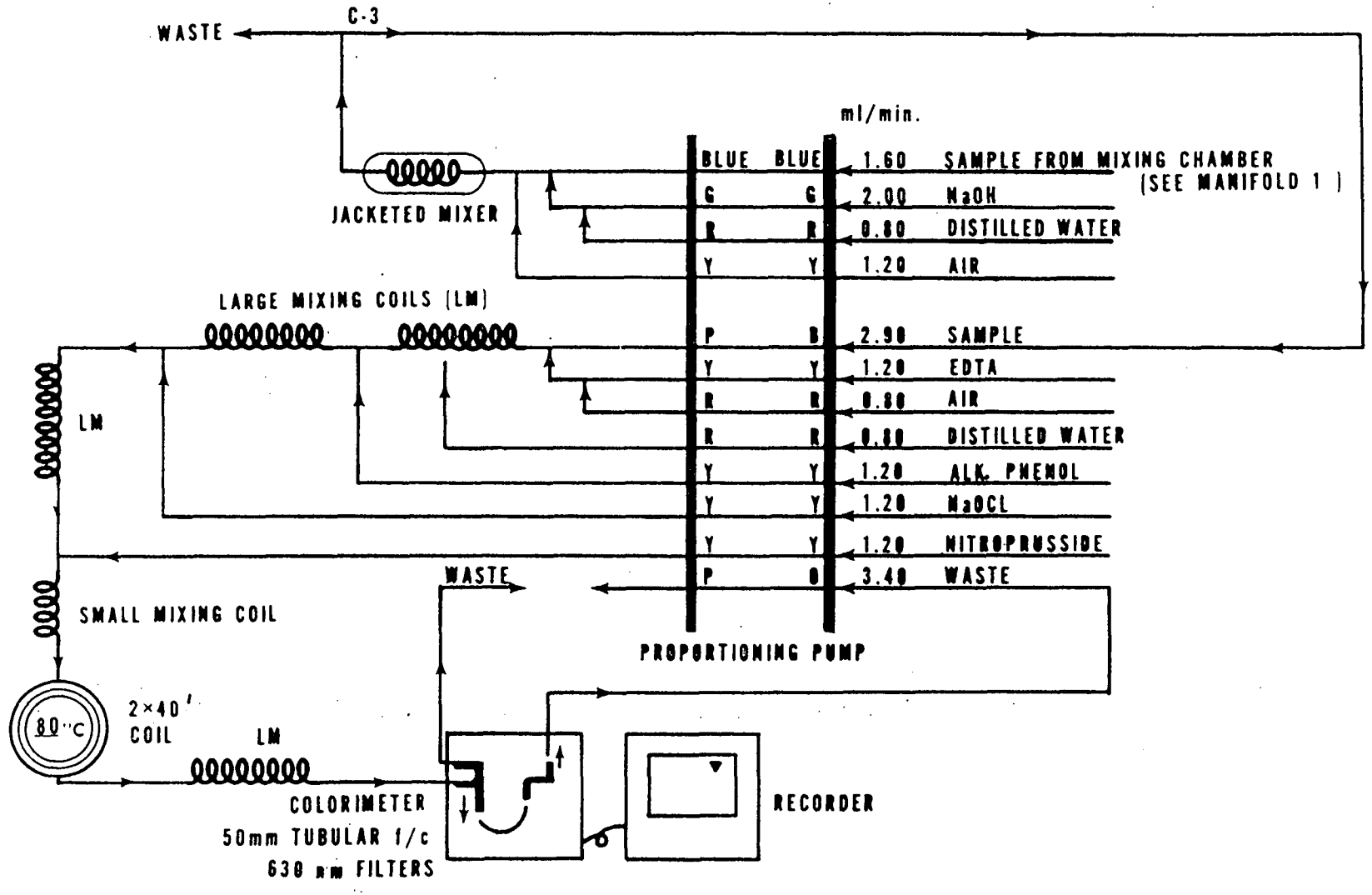


FIGURE 3. KJELDAHL NITROGEN MANIFOLD 2. AA-I

NITROGEN, KJELDAHL, TOTAL

Method 351.2 (Colorimetric, Semi-Automated Block Digester, AAII)

STORET NO. 00625

1. Scope and Application
   1.1 This method covers the determination of total Kjeldahl nitrogen in drinking and surface waters, domestic and industrial wastes. The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert the nitrogeneous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines. The applicable range of this method is 0.1 to 20 mg/1 TKN. The range may be extended with sample dilution.
2. Summary of Method
   2.1 The sample is heated in the presence of sulfuric acid, K_2SO_4 and $HgSO_4$ for two and one half hours. The residue is cooled, diluted to 25 ml and placed on the AutoAnalyzer for ammonia determination. This digested sample may also be used for phosphorus determination.
3. Definitions
   3.1 Total Kjeldahl nitrogen is defined as the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate $(NH_4)_2SO_4$, under the conditions of digestion described below.
   3.2 Organic Kjeldahl nitrogen is defined as the difference obtained by subtracting the free-ammonia value (Method 350.2, Nitrogen, Ammonia, this manual) from the total Kjeldahl nitrogen value.
4. Sample Handling and Preservation
   4.1 Samples may be preserved by addition of 2 ml of conc H_2SO_4 per liter and stored at 4°C. Even when preserved in this manner, conversion of organic nitrogen to ammonia may occur. Therefore, samples should be analyzed as soon as possible.
5. Apparatus
   5.1 Block Digestor–40
   5.2 Technicon Manifold for Ammonia (Figure 1)
   5.3 Chemware TFE (Teflon boiling stones), Markson Science, Inc., Box 767, Delmar, CA 92014)
6. Reagents
   6.1 Mercuric Sulfate: Dissolve 8 g red mercuric oxide (HgO) in 50 ml of 1:4 sulfuric acid (10 ml conc H_2SO_4: 40 ml distilled water) and dilute to 100 ml with distilled water.
   6.2 Digestion Solution: (Sulfuric acid-mercuric sulfate-potassium sulfate solution): Dissolve 133 g of K_2SO_4 in 700 ml of distilled water and 200 ml of conc H_2SO_4. Add 25 ml of mercuric sulfate solution and dilute to 1 liter.

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6.3 Sulfuric Acid Solution (4%): Add 40 ml of conc. sulfuric acid to 800 ml of ammonia free distilled water, cool and dilute to 1 liter.

6.4 Stock Sodium Hydroxide (20%): Dissolve 200 g of sodium hydroxide in 900 ml of ammonia-free distilled water and dilute to 1 liter.

6.5 Stock Sodium Potassium Tartrate Solution (20%): Dissolve 200 g sodium potassium tartrate in about 800 ml of ammonia-free distilled water and dilute to 1 liter.

6.6 Stock Buffer Solution: Dissolve 134.0 g of sodium phosphate, dibasic (Na_2HPO_4) in about 800 ml of ammonia free water. Add 20 g of sodium hydroxide and dilute to 1 liter.

6.7 Working Buffer Solution: Combine the reagents in the stated order; add 250 ml of stock sodium potassium tartrate solution (6.5) to 200 ml of stock buffer solution (6.6) and mix. Add xx ml sodium hydroxide solution (6.4) and dilute to 1 liter. See concentration ranges, Table I, for composition of working buffer.

6.8 Sodium Salicylate/Sodium Nitroprusside Solution: Dissolve 150 g of sodium salicylate and 0.3 g of sodium nitroprusside in about 600 ml of ammonia free water and dilute to 1 liter.

6.9 Sodium Hypochlorite Solution: Dilute 6.0 ml sodium hypochlorite solution (clorox) to 100 ml with ammonia free distilled water.

6.10 Ammonium chloride, stock solution: Dissolve 3.819 g NH_4Cl in distilled water and bring to volume in a 1 liter volumetric flask. 1 ml = 1.0 mg NH_3-N.

7. Procedure

Digestion

7.1 To 20 or 25 ml of sample, add 5 ml of digestion solution (6.2) and mix (use a vortex mixer).

7.2 Add (4–8) Teflon boiling stones (5.3). Too many boiling chips will cause the sample to boil over.

7.3 With Block Digestor in manual mode set low and high temperature at 160°C and preheat unit to 160°C. Place tubes in digestor and switch to automatic mode. Set low temperature timer for 1 hour. Reset high temperature to 380°C and set timer for 2 1/2 hours.

7.4 Cool sample and dilute to 25 ml with ammonia free water.

Colorimetric Analysis

7.5 Check the level of all reagent containers to ensure an adequate supply.

7.6 Excluding the salicylate line, place all reagent lines in their respective containers, connect the sample probe to the Sampler IV and start the proportioning pump.

7.7 Flush the Sampler IV wash receptacle with about 25 ml of 4.0% sulfuric acid (6.3).

7.8 When reagents have been pumping for at least five minutes, place the salicylate line in its respective container and allow the system to equilibrate. If a precipitate forms after the addition of salicylate, the pH is too low. Immediately stop the proportioning pump and flush the coils with water using a syringe. Before restarting the system, check the concentration of the sulfuric acid solutions and/or the working buffer solution.

TABLE I

CONCENTRATION RANGES
(NITROGEN)

No.	Dilution loops: Initial sample — Sample line	Initial sample — Diluent line	Resample — Resample line	Resample — Diluent line	Approx. std. cal. setting	Range PPM N (±10%)	ml stock NaOH per liter working buffer solution
1	.80 (RED/RED)	.80 (RED/RED)	.32 (BLK/BLK)	.80 (RED/RED)	700	0–0.5	250
2	.80 (RED/RED)	.80 (RED/RED)	.32 (BLK/BLK)	.80 (RED/RED)	100	0–1.5	250
3	.16 (ORN/YEL)	.80 (RED/RED)	.32 (BLK/BLK)	.80 (RED/RED)	700	0–1	120
4	.16 (ORN/YEL)	.80 (RED/RED)	.32 (BLK/BLK)	.80 (RED/RED)	100	0–5	120
5	.16 (ORN/YEL)	.80 (RED/RED)	.16 (ORN/YEL)	.80 (RED/RED)	700	0–2	80
6	.16 (ORN/YEL)	.80 (RED/RED)	.16 (ORN/YEL)	.80 (RED/RED)	100	0–10	80

7.9 To prevent precipitation of sodium salicylate in the waste tray, which can clog the tray outlet, keep the nitrogen flowcell pump tube and the nitrogen Colorimeter "To Waste" tube separate from all other lines or keep tap water flowing in the waste tray.

7.10 After a stable baseline has been obtained start the Sampler.

8. Calculations

8.1 Prepare standard curve by plotting peak heights of processed standards against concentration values. Compute concentrations by comparing sample peak heights with standard curve.

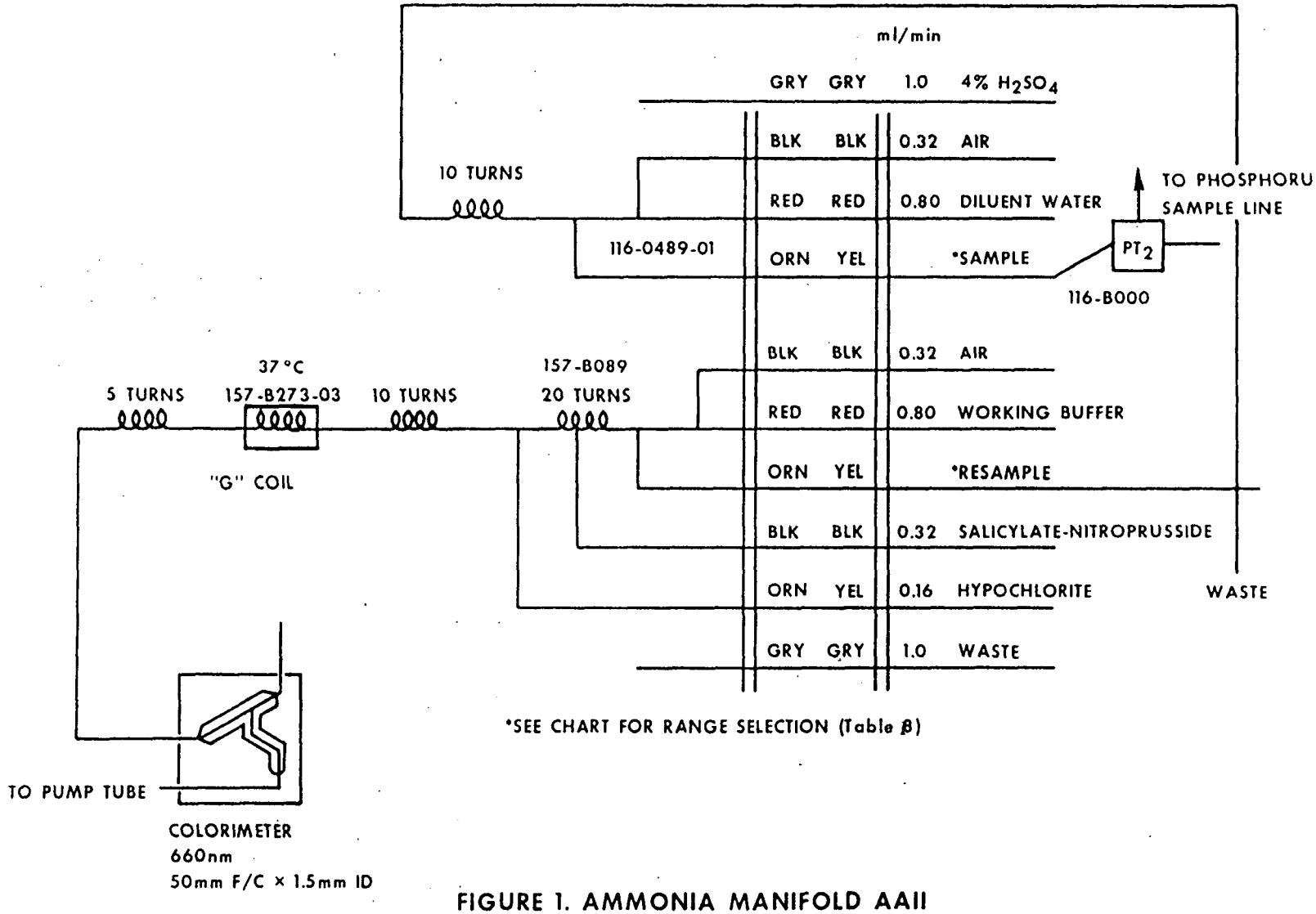
9. Precision and Accuracy

9.1 In a single laboratory (EMSL), using sewage samples of concentrations of 1.2, 2.6, and 1.7 mg N/1, the precision was ±0.07, ±0.03 and ±0.15, respectively.

9.2 In a single laboratory (EMSL), using sewage samples of concentrations of 4.7 and 8.74 mg N/1, the recoveries were 99 and 99%, respectively.

Bibliography

1. McDaniel, W.H., Hemphill, R.N. and Donaldson, W.T., "Automatic Determination of Total Kjeldahl Nitrogen in Estuarine Water", Technicon Symposia, pp. 362–367, Vol. 1, 1967.
2. Gales, M.E., and Booth, R.L., "Evaluation of Organic Nitrogen Methods", EPA Office of Research and Monitoring, June, 1972.
3. Gales, M.E. and Booth, R.L., "Simultaneous and Automated Determination of Total Phosphorus and Total Kjeldahl Nitrogen", Methods Development and Quality Assurance Research Laboratory, May, 1974.
4. Technicon "Total Kjeldahl Nitrogen and Total Phosphorus BD-40 Digestion Procedure for Water", August, 1974.
5. Gales, M.E., and Booth, R.L., "Evaluation of the Block Digestion System for the Measurement of Total Kjeldahl Nitrogen and Total Phosphorus", EPA-600/4–78–015, Environmental Monitoring and Support Laboratory, Cinncinnati, Ohio.



351.2-5

FIGURE 1. AMMONIA MANIFOLD AAII

Broad variations in the kinds and concentrations of salts in samples can make the use of a membrane probe difficult.

4.3 Reactive compounds can interfere with the output or the performance of dissolved oxygen probes.

4.3.1 Reactive gases which pass through the membrane probes may interfere. For example, chlorine will depolarize the cathode and cause a high probe-output. Long-term exposures to chlorine will coat the anode with the chloride of the anode metal and eventually desensitize the probe. Alkaline samples in which free chlorine does not exist will not interfere. Hydrogen sulfide will interfere with membrane probes if the applied potential is greater than the half-wave potential of the sulfide ion. If the applied potential is less than the half-wave potential, an interfering reaction will not occur, but coating of the anode with the sulfide of the anode metal can take place.

4.4 Dissolved oxygen probes are temperature sensitive, and temperature compensation is normally provided by the manufacturer. Membrane probes have a temperature coefficient of 4 to 6 percent/°C dependent upon the membrane employed.

5. Apparatus

5.1 No specific probe or accessory is especially recommended as superior. However, probes which have been evaluated or are in use and found to be reliable are the Weston & Stack DO Analyzer Model 30, the Yellow Springs Instrument (YSI) Model 54, and the Beckman Fieldlab Oxygen Analyzer.

6. Calibration

Follow manufacturer instructions.

7. Procedure

Follow manufacturer instructions.

8. Calculation

Follow manufacturer instructions.

9. Precision and Accuracy

Manufacturer's specification claim 0.1 mg/l repeatability with ±1% accuracy.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 450, Method 422F (1975).

OXYGEN, DISSOLVED

Method 360.1 (Membrane Electrode)

STORET NO. 00299

1. Scope and Application
   - 1.1 The probe method for dissolved oxygen is recommended for those samples containing materials which interfere with the modified Winkler procedure such as sulfite, thiosulfate, polythionate, mercaptans, free chlorine or hypochlorite, organic substances readily hydrolyzed in alkaline solutions, free iodine, intense color or turbidity and biological flocs.
   - 1.2 The probe method is recommended as a substitute for the modified Winkler procedure in monitoring of streams, lakes, outfalls, etc., where it is desired to obtain a continuous record of the dissolved oxygen content of the water under observation.
   - 1.3 The probe method may be used as a substitute for the modified Winkler procedure in BOD determinations where it is desired to perform nondestructive DO measurements on a sample.
   - 1.4 The probe method may be used under any circumstances as a substitute for the modified Winkler procedure provided that the probe itself is standardized against the Winkler method on samples free of interfering materials.
   - 1.5 The electronic readout meter for the output from dissolved oxygen probes is normally calibrated in convenient scale (0 to 10, 0 to 15, 0 to 20 mg/1 for example) with a sensitivity of approximately 0.05 mg/liter.
2. Summary of Method
   - 2.1 The most common instrumental probes for determination of dissolved oxygen in water are dependent upon electrochemical reactions. Under steady-state conditions, the current or potential can be correlated with DO concentrations. Interfacial dynamics at the probe-sample interface are a factor in probe response and a significant degree of interfacial turbulence is necessary. For precision performance, turbulence should be constant.
3. Sample Handling and Preservation
   - 3.1 See 4.1, 4.2, 4.3, 4.4 under Modified Winkler Method (360.2).
4. Interferences
   - 4.1 Dissolved organic materials are not known to interfere in the output from dissolved oxygen probes.
   - 4.2 Dissolved inorganic salts are a factor in the performance of dissolved oxygen probe.
     - 4.2.1 Probes with membranes respond to partial pressure of oxygen which in turn is a function of dissolved inorganic salts. Conversion factors for seawater and brackish waters may be calculated from dissolved oxygen saturation versus salinity data. Conversion factors for specific inorganic salts may be developed experimentally.

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7.3.4 After the temperature of the block has reached 380°C, the time should be set for 30 minutes. Longer time and higher temperature may result in complete loss of the acid.

7.3.5 Cool, add 25 ml of ammonia-free water and mix.

7.4 Electrode analysis

7.4.1 All standards should be treated as the samples and should contain the same concentration of sulfuric acid-mercuric sulfate-potassium sulfate solution (6.3).

7.4.2 Macro Kjeldahl system

To a 100 ml aliquot, add 15 ml of 10 N NaOH (6.4), mix and cool to room temperature. Immerse the electrode in the sample solution and add 4 ml of NaOH-NaI-EDTA reagent (6.5) while mixing. Allow the electrode to remain immersed in the solution until a stable reading is obtained.

7.4.3 Micro Kjeldahl system

Add 6 ml of 10 N NaOH solution (6.4), cool to room temperature and transfer the sample to a 100 ml beaker. Immerse the electrode in the sample solution and add 4 ml of NaOH-NaI-EDTA reagent (6.5) while mixing. Allow the electrode to remain immersed in the solution until a stable reading is obtained.

7.4.4 Block Digestor

Add 3 ml of 10 N NaOH (6.4), cool to room temperature, dilute to 50 ml and transfer to a 100 ml beaker. Immerse the electrode in the sample and add 2 ml of NaOH-NaI-EDTA reagent (6.5) while mixing. Allow the electrode to remain immersed in the solution until a stable reading is obtained.

8. Calculation

Using semilogarithmic graph paper, plot the concentration of ammonia in mg NH_3-N on the log axis vs. the electrode potential developed in the standard on the linear axis, starting with the lowest concentration at the bottom of the scale.

$$\text{mg TKN/l} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = mg NH_3-N read from standard curve

B = mg NH_3-N in blank

C = ml of original sample taken

9. Precision and Accuracy

9.1 Precision and accuracy data are not available at this time.

Bibliography

1. Schlueter, A., "Nitrate Interference in Total Kjeldahl Nitrogen Determinations and its Removal by Anion Exchange Resin", EPA-600/7-77-017.

6.3 Sulfuric acid-mercuric sulfate-potassium sulfate solution: Dissolve 267 g K_2SO_4 in 1300 ml distilled water and 400 ml conc H_2SO_4. Add 50 ml mercuric sulfate solution (6.2) and dilute to 2 liters with distilled water.

6.4 Sodium hydroxide 10N: Dissolve 400 g NaOH in 600 ml of ammonia-free water, cool and dilute to 1 liter.

6.5 Sodium Hydroxide, Sodium Iodide and EDTA Solution: Dissolve 400 g of NaOH, 300 g NaI and 2 g of EDTA in 700 ml of ammonia-free water, cool and dilute to 1 liter.

6.6 Ammonium chloride, stock solution: 1.0 ml = 1.0 mg NH_3-N. Dissolve 3.819 g NH_4Cl in water and make up to 1 liter in a volumetric flask with distilled water.

6.7 Ammonium chloride, standard solution: 1.0 ml = 0.01 mg NH_3-N. Dilute 10.0 ml of the stock solution (6.6) to 1 liter with distilled water in a volumetric flask.

7. Procedure

7.1 Macro Kjeldahl system

7.7.1 Place a measured sample or the residue from the distillation in the ammonia determination (for Organic Kjeldahl only) into an 800 ml Kjeldahl flask. The sample size can be determined from the following table:

Kjedahl Nitrogen in Sample, mg/l	Sample Size ml
0–5	500
5–10	250
10–20	100
20–50	50.0
50–500	25.0

Dilute the sample, if required, to 500 ml with distilled water, and add 100 ml sulfuric acid-mercuric sulfate-potassium sulfate solution (6.3) and evaporate the mixture in the Kjeldahl apparatus until SO_3 fumes are given off and the solution turns colorless or pale yellow. Continue heating for 30 additional minutes. Cool the residue and add 500 ml distilled water and mix.

7.2 Micro Kjeldahl system

7.2.1 Place 50.0 ml of sample, or an aliquot diluted to 50 ml, in a 100 ml Kjeldahl flask and add 10 ml sulfuric acid-mercuric sulfate-potassium sulfate solution (6.3). Evaporate the mixture in the Kjeldahl apparatus until SO_3 fumes are given off and the solution turns colorless or pale yellow. Then digest for an additional 30 minutes. Cool the residue, add 44 ml distilled water and mix.

7.3 Block Digestor

7.3.1 Place 20 ml of sample, or an aliquot diluted to 20 ml, in the digestion tube. Add 5 ml of sulfuric acid-mercuric sulfate-potassium sulfate solution (6.3) and mix. Add 4–8 Teflon boiling stones.

7.3.2 Place tubes in digestor that has been preheated to 200°C.

7.3.3 Set low temperature at 200°C for 1 hour, the high temperature at 380°C and total time for two and one half hours.

NITROGEN, KJELDAHL, TOTAL

Method 351.4 (Potentiometric, Ion Selective Electrode)

STORET NO. 00625

1. Scope and Application
   1.1 This method is applicable to the measurement of TKN in drinking and surface water, domestic and industrial wastes.
   1.2 This method covers the range from 0.03 to 25 mg TKN/1.
2. Summary of Method
   2.1 Following digestion and cooling, distilled water is added to the digestion flask and the pH adjusted to between 3 and 4.5 by the addition of 10 N NaOH. The sample is cooled and transferred to a 100 ml beaker. After inserting the electrode into the sample, NaOH-NaI-EDTA is added and the ammonia measured. (Ethylene diamine tetraacetic acid (EDTA) is added to the alkaline reagent (NaOH-NaI) to prevent precipitation of hydroxides, thereby avoiding deposition on the electrode membrane).
3. Sample Handling and Preservation
   3.1 Samples may be preserved by addition of 2 ml of conc. H_2SO_4 per liter and stored at 4°C. Even when preserved in this manner, conversion of organic nitrogen to ammonia may occur. Preserved samples should be analyzed as soon as possible.
4. Interferences
   4.1 Interference from metals is eliminated with the addition of NaI.
   4.2 High nitrate concentrations (10X or more than the TKN level) result in low TKN values. The reaction between nitrate and ammonia can be prevented by the use of an anion exchange resin (chloride form) to remove the nitrate prior to the TKN analysis.
5. Apparatus
   5.1 Electrometer (pH meter) with expanded mV scale.
   5.2 Ammonia selective electrode, such as Orion Model 95–10.
   5.3 Magnetic stirrer, thermally insulated and Teflon-coated stirring bar.
   5.4 Digestion apparatus: A Kjeldahl digestion apparatus with 800 or 100 ml flasks and suction take off to remove SO_3 fumes and water.
   5.5 Technicon Block Digestor BD-40.
6. Reagents
   6.1 Distilled water should be free of ammonia. Such water is best prepared by passing distilled water through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin. Regeneration of the column should be carried out according to the manufacturer's instructions.
   NOTE 1: All solutions must be made with ammonia-free water.
   6.2 Mercuric sulfate solution: Dissolve 8 g red mercuric oxide (HgO) in 50 ml of 1:4 sulfuric acid (10.0 ml conc H_2SO_4: 40 ml distilled water) and dilute to 100 ml with distilled water.

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9.4 Potentiometric determination: Calculate Total Kjeldahl Nitrogen, in mg/l, in the original sample as follows:

$$\text{TKN, mg/l} = \frac{B}{D} \times A$$

where:
A = mg NH_3–N/l from electrode method standard curve.
B = volume of diluted distillate in ml.
D = ml of original sample taken.

10. Precision

10.1 Thirty-one analysts in twenty laboratories analyzed natural water samples containing exact increments of organic nitrogen, with the following results:

Increment as Nitrogen, Kjeldahl mg N/liter	Precision as Standard Deviation mg N/liter	Accuracy as Bias, %	Accuracy as Bias, mg N/liter
0.20	0.197	+15.54	+0.03
0.31	0.247	+ 5.45	+0.02
4.10	1.056	+ 1.03	+0.04
4.61	1.191	- 1.67	-0.08

(FWPCA Method Study 2, Nutrient Analyses)

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 437, Method 421 (1975).
2. Schlueter, Albert, "Nitrate Interference In Total Kjeldahl Nitrogen Determinations and Its Removal by Anion Exchange Resins", EPA Report 600/7–77–017.

and compared to similar values on the curve to insure that the digestion-distillation technique is reliable. If treated standards do not agree with untreated standards the operator should find the cause of the apparent error before proceeding.

9. Calculation

9.1 If the titrimetric procedure is used, calculate Total Kjeldahl Nitrogen, in mg/l, in the original sample as follows:

$$\text{TKN, mg/l} = \frac{(A - B)N \times F \times 1{,}000}{S}$$

where:

A = milliliters of standard 0.020 N H_2SO_4 solution used in titrating sample.
B = milliliters of standard 0.020 N H_2SO_4 solution used in titrating blank.
N = normality of sulfuric acid solution.
F = milliequivalent weight of nitrogen (14 mg).
S = milliliters of sample digested.

If the sulfuric acid is exactly 0.02 N the formula is shortened to:

$$\text{TKN, mg/l} = \frac{(A - B) \times 280}{S}$$

9.2 If the Nessler procedure is used, calculate the Total Kjeldahl Nitrogen, in mg/l, in the original sample as follows:

$$\text{TKN, mg/l} = \frac{A \times 1{,}000}{D} \times \frac{B}{C}$$

where:

A = mg NH_3–N read from curve.
B = ml total distillate collected including the H_3BO_3.
C = ml distillate taken for Nesslerization.
D = ml of original sample taken.

9.3 Calculate Organic Kjeldahl Nitrogen in mg/l, as follows:
Organic Kjeldahl Nitrogen = TKN –(NH_3–N.)

8.3 Micro Kjeldahl system

8.3.1 Place 50.0 ml of sample or an aliquot diluted to 50 ml in a 100 ml Kjeldahl flask and add 10 ml sulfuric acid-mercuric sulfate-potassium sulfate solution (7.3). Evaporate the mixture in the Kjeldahl apparatus until SO_3 fumes are given off and the solution turns colorless or pale yellow. Then digest for an additional 30 minutes. Cool the residue and add 30 ml distilled water.

8.3.2 Make the digestate alkaline by careful addition of 10 ml of sodium hydroxide-thiosulfate solution (7.4) without mixing. Do not mix until the digestion flask has been connected to the distillation apparatus.

8.3.3 Connect the Kjeldahl flask to the condenser with the tip of condenser or an extension of the condenser tip below the level of the boric acid solution (7.6) in the receiving flask or 50 ml short-form Nessler tube.

8.3.4 Steam distill 30 ml at the rate of 6–10 ml/min., into 5 ml of 2% boric acid (7.6).

8.3.5 Dilute the distillate to 50 ml. For concentrations above 1 mg/1 the ammonia can be determined titrimetrically. For concentrations below this value, it is determined colorimetrically. The potentiometric method is applicable to the range 0.05 to 1400 mg/1.

8.4 Determination of ammonia in distillate: Determine the ammonia content of the distillate titrimetrically, colorimetrically, or potentiometrically, as described below.

8.4.1 Titrimetric determination: Add 3 drops of the mixed indicator (7.5) to the distillate and titrate the ammonia with the 0.02 N H_2SO_4 (7.7), matching the endpoint against a blank containing the same volume of distilled water and H_3BO_3 (7.6) solution.

8.4.2 Colorimetric determination: Prepare a series of Nessler tube standards as follows:

ml of Standard 1.0 ml = 0.01 mg NH_3–N	mg NH_3–N/50.0 ml
0.0	0.0
0.5	0.005
1.0	0.010
2.0	0.020
4.0	0.040
5.0	0.050
8.0	0.080
10.0	0.10

Dilute each tube to 50 ml with ammonia free water, add 1 ml of Nessler Reagent (7.10) and mix. After 20 minutes read the absorbance at 425 nm against the blank. From the values obtained for the standards plot absorbance vs. mg NH_3-N for the standard curve. Develop color in the 50 ml diluted distillate in exactly the same manner and read mg NH_3-N from the standard curve.

8.4.3 Potentiometric determination: Consult the method entitled Nitrogen, Ammonia: Potentiometric, Ion Selective Electrode Method, (Method 350.3) in this manual.

8.4.4 It is not imperative that all standards be treated in the same manner as the samples. It is recommended that at least 2 standards (a high and low) be digested, distilled,

NOTE 3: Reagents 7.7, 7.8, 7.9, and 7.10 are identical to reagents 6.8, 6.2, 6.3, and 6.6 described under Nitrogen, Ammonia (Colorimetric; Titrimetric; Potentiometric-Distillation Procedure, Method 350.2).

8. Procedure

8.1 The distillation apparatus should be pre-steamed before use by distilling a 1:1 mixture of distilled water and sodium hydroxide-sodium thiosulfate solution (7.4) until the distillate is ammonia-free. This operation should be repeated each time the apparatus is out of service long enough to accumulate ammonia (usually 4 hours or more).

8.2 Macro Kjeldahl system

8.2.1 Place a measured sample or the residue from the distillation in the ammonia determination (for Organic Kjeldahl only) into an 800 ml Kjeldahl flask. The sample size can be determined from the following table:

Kjeldahl Nitrogen in Sample, mg/l	Sample Size ml
0–5	500
5–10	250
10–20	100
20–50	50.0
50–500	25.0

Dilute the sample, if required, to 500 ml with distilled water, and add 100 ml sulfuric acid-mercuric sulfate-potassium sulfate solution (7.3). Evaporate the mixture in the Kjeldahl apparatus until SO_3 fumes are given off and the solution turns colorless or pale yellow. Continue heating for 30 additional minutes. Cool the residue and add 300 ml distilled water.

8.2.2 Make the digestate alkaline by careful addition of 100 ml of sodium hydroxide – thiosulfate solution (7.4) without mixing.

NOTE 5: Slow addition of the heavy caustic solution down the tilted neck of the digestion flask will cause heavier solution to underlay the aqueous sulfuric acid solution without loss of free-ammonia. Do not mix until the digestion flask has been connected to the distillation apparatus.

8.2.3 Connect the Kjeldahl flask to the condenser with the tip of condenser or an extension of the condenser tip below the level of the boric acid solution (7.6) in the receiving flask.

8.2.4 Distill 300 ml at the rate of 6–10 ml/min., into 50 ml of 2% boric acid (7.6) contained in a 500 ml Erlenmeyer flask.

8.2.5 Dilute the distillate to 500 ml in the flask. These flasks should be marked at the 350 and the 500 ml volumes. With such marking, it is not necessary to transfer the distillate to volumetric flasks. For concentrations above 1 mg/1, the ammonia can be determined titrimetrically. For concentrations below this value, it is determined colorimetrically. The potentiometric method is applicable to the range 0.05 to 1400 mg/1.

6. Apparatus

6.1 Digestion apparatus: A Kjeldahl digestion apparatus with 800 or 100 ml flasks and suction takeoff to remove SO_3 fumes and water.

6.2 Distillation apparatus: The macro Kjeldahl flask is connected to a condenser and an adaptor so that the distillate can be collected. Micro Kjeldahl steam distillation apparatus is commercially available.

6.3 Spectrophotometer for use at 400 to 425 nm with a light path of 1 cm or longer.

7. Reagents

7.1 Distilled water should be free of ammonia. Such water is best prepared by the passage of distilled water through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin. Regeneration of the column should be carried out according to the manufacturer's instructions.

NOTE 1: All solutions must be made with ammonia-free water.

7.2 Mercuric sulfate solution: Dissolve 8 g red mercuric oxide (HgO) in 50 ml of 1:4 sulfuric acid (10.0 ml conc. H_2SO_4 : 40 ml distilled water) and dilute to 100 ml with distilled water.

7.3 Sulfuric acid-mercuric sulfate-potassium sulfate solution: Dissolve 267 g K_2SO_4 in 1300 ml distilled water and 400 ml conc. H_2SO_4. Add 50 ml mercuric sulfate solution (7.2) and dilute to 2 liters with distilled water.

7.4 Sodium hydroxide-sodium thiosulfate solution: Dissolve 500 g NaOH and 25 g $Na_2S_2O_3 \cdot 5H_2O$ in distilled water and dilute to 1 liter.

7.5 Mixed indicator: Mix 2 volumes of 0.2% methyl red in 95% ethanol with 1 volume of 0.2% methylene blue in ethanol. Prepare fresh every 30 days.

7.6 Boric acid solution: Dissolve 20 g boric acid, H_3BO_3, in water and dilute to 1 liter with distilled water.

7.7 Sulfuric acid, standard solution: (0.02 N) 1 ml = 0.28 mg NH_3–N. Prepare a stock solution of approximately 0.1 N acid by diluting 3 ml of conc. H_2SO_4 (sp. gr. 1.84) to 1 liter with CO_2-free distilled water. Dilute 200 ml of this solution to 1 liter with CO_2-free distilled water. Standardize the approximately 0.02 N acid so prepared against 0.0200 N Na_2CO_3 solution. This last solution is prepared by dissolving 1.060 g anhydrous Na_2CO_3, oven-dried at 140°C, and diluting to 1 liter with CO_2-free distilled water.

NOTE 2: An alternate and perhaps preferable method is to standardize the approximately 0.1 N H_2SO_4 solution against a 0.100 N Na_2CO_3 solution. By proper dilution the 0.02 N acid can the be prepared.

7.8 Ammonium chloride, stock solution: 1.0 ml = 1.0 mg NH_3–N. Dissolve 3.819 g NH_4Cl in water and make up to 1 liter in a volumetric flask with distilled water.

7.9 Ammonium chloride, standard solution: 1.0 ml = 0.01 mg NH_3–N. Dilute 10.0 ml of the stock solution (7.8) with distilled water to 1 liter in a volumetric flask.

7.10 Nessler reagent: Dissolve 100 g of mercuric iodide and 70 g potassium iodide in a small volume of distilled water. Add this mixture slowly, with stirring, to a cooled solution of 160 g of NaOH in 500 ml of distilled water. Dilute the mixture to 1 liter. The solution is stable for at least one year if stored in a pyrex bottle out of direct sunlight.

NITROGEN, KJELDAHL, TOTAL

Method 351.3 (Colorimetric; Titrimetric; Potentiometric)

STORET NO. 00625

1. Scope and Application
   1.1 This method covers the determination of total Kjeldahl nitrogen in drinking, surface and saline waters, domestic and industrial wastes. The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert the nitrogenous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines.
   1.2 Three alternatives are listed for the determination of ammonia after distillation: the titrimetric method which is applicable to concentrations above 1 mg N/liter; the Nesslerization method which is applicable to concentrations below 1 mg N/liter; and the potentiometric method applicable to the range 0.05 to 1400 mg/1.
   1.3 This method is described for macro and micro glassware systems.
2. Definitions
   2.1 Total Kjeldahl nitrogen is defined as the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate $(NH_4)_2SO_4$, under the conditions of digestion described below.
   2.2 Organic Kjeldahl nitrogen is defined as the difference obtained by subtracting the free-ammonia value (Method 350.2, Nitrogen, Ammonia, this manual) from the total Kjeldahl nitrogen value. This may be determined directly by removal of ammonia before digestion.
3. Summary of Method
   3.1 The sample is heated in the presence of conc. sulfuric acid, K_2SO_4 and $HgSO_4$ and evaporated until SO_3 fumes are obtained and the solution becomes colorless or pale yellow. The residue is cooled, diluted, and is treated and made alkaline with a hydroxide-thiosulfate solution. The ammonia is distilled and determined after distillation by Nesslerization, titration or potentiometry.
4. Sample Handling and Preservation
   4.1 Samples may be preserved by addition of 2 ml of conc. H_2SO_4 per liter and stored at 4°C. Even when preserved in this manner, conversion of organic nitrogen to ammonia may occur. Preserved samples should be analyzed as soon as possible.
5. Interference
   5.1 High nitrate concentrations (10X or more than the TKN level) result in low TKN values. The reaction between nitrate and ammonia can be prevented by the use of an anion exchange resin (chloride form) to remove the nitrate prior to the TKN analysis.

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OXYGEN, DISSOLVED

Method 360.2 (Modified Winkler, Full-Bottle Technique)

STORET NO. 00300

1. Scope and Application
   1.1 This method is applicable for use with most wastewaters and streams that contain nitrate nitrogen and not more than 1 mg/l of ferrous iron. Other reducing or oxidizing materials should be absent. If 1 ml of fluoride solution is added before acidifying the sample and there is no delay in titration, the method is also applicable in the presence of 100–200 mg/l ferric iron.
   1.2 The Dissolved Oxygen (DO) Probe technique gives comparable results on all samples types.
   1.3 The azide modification is not applicable under the following conditions: (a) samples containing sulfite, thiosulfate, polythionate, appreciable quantities of free chlorine or hypochlorite; (b) samples high in suspended solids; (c) samples containing organic substances which are readily oxidized in a highly alkaline solution, or which are oxidized by free iodine in an acid solution; (d) untreated domestic sewage; (e) biological flocs; and (f) where sample color interferes with endpoint detection. In instances where the azide modification is not applicable, the DO probe should be used.
2. Summary of Method
   2.1 The sample is treated with manganous sulfate, potassium hydroxide, and potassium iodide (the latter two reagents combined in one solution) and finally sulfuric acid. The initial precipitate of manganous hydroxide, $Mn(OH)_2$, combines with the dissolved oxygen in the sample to form a brown precipitate, manganic hydroxide, $MnO(OH)_2$. Upon acidification, the manganic hydroxide forms manganic sulfate which acts as an oxidizing agent to release free iodine from the potassium iodide. The iodine, which is stoichiometrically equivalent to the dissolved oxygen in the sample is then titrated with sodium thiosulfate or phenylarsine oxide (PAO).
3. Interferences
   3.1 There are a number of interferences to the dissolved oxygen test, including oxidizing and reducing agents, nitrate ion, ferrous iron, and organic matter.
   3.2 Various modifications of the original Winkler procedure for dissolved oxygen have been developed to compensate for or eliminate interferences. The Alsterberg modification is commonly used to successfully eliminate the nitrite interference, the Rideal-Stewart modification is designed to eliminate ferrous iron interference, and the Theriault procedure is used to compensate for high concentration of organic materials.
   3.3 Most of the common interferences in the Winkler procedure may be overcome by use of the dissolved oxygen probe.

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4. Sample Handling and Preservation

4.1 Where possible, collect the sample in a **300 ml BOD** incubation bottle. Special precautions are required to avoid entrainment or solution of atmospheric oxygen or loss of dissolved oxygen.

4.2 Where samples are collected from shallow depths (less than 5 feet), use of an APHA-type sampler is recommended. Use of a Kemmerer type sampler is recommended for samples collected from depths of greater than 5 feet.

4.3 When a Kemmerer sampler is used, the **BOD** sample bottle should be filled to overflowing. (overflow for approximately 10 seconds). Outlet tube of Kemmerer should be inserted to bottom of **BOD** bottle. Care must be taken to prevent turbulence and the formation of bubbles when filling bottle.

4.4 At time of sampling, the sample temperature should be recorded as precisely as required.

4.5 Do not delay the determination of dissolved oxygen in samples having an appreciable iodine demand or containing ferrous iron. If samples must be preserved either method (4.5.1) or (4.5.2) below, may be employed.

4.5.1 Add 2 ml of manganous sulfate solution (6.1) and then 2 ml of alkaline iodide-azide solution (6.2) to the sample contained in the **BOD** bottle. Both reagents must be added well below the surface of the liquid. Stopper the bottle immediately and mix the contents thoroughly. The sample should be stored at the temperature of the collection water, or water sealed and kept at a temperature of 10 to 20°C, in the dark. Complete the procedure by adding 2 ml H_2SO_4 (see 7.1) at time of analysis.

4.5.2 Add 0.7 ml of conc. H_2SO_4 (6.3) and 1 ml sodium azide solution (2 g NaN_3 in 100 ml distilled water) to sample in the **BOD** bottle. Store sample as in (4.5.1). Complete the procedure using 2 ml of manganous sulfate solution (6.1), 3 ml alkaline iodide-azide solution (6.2), and 2 ml of conc. H_2SO_4 (6.3) at time of analysis.

4.6 If either preservation technique is employed, complete the analysis within 4–8 hours after sampling.

5. Apparatus

5.1 Sample bottles-300 ml ±3 ml capacity **BOD** incubation bottles with tapered ground glass pointed stoppers and flared mouths.

5.2 Pipets-with elongated tips capable of delivering 2.0 ml ±0.10 ml of reagent.

6. Reagents

6.1 Manganous sulfate solution: Dissolve 480 g manganous sulfate ($MnSO_4 \cdot 4H_2O$ in distilled water and dilute to 1 liter.

6.1.1 Alternatively, use 400 g of $MnSO_4 \cdot 2H_2O$ or 364 g of $MnSO_4 \cdot H_2O$ per liter. When uncertainty exists regarding the water of crystallization, a solution of equivalent strength may be obtained by adjusting the specific gravity of the solution to 1.270 at 20°C.

6.2 Alkaline iodide-azide solution: Dissolve 500 g of sodium hydroxide (NaOH) or 700 g of potassium hydroxide (KOH) and 135 g of sodium iodide (NaI) or 150 g of potassium iodide (KI) in distilled water and dilute to 1 liter. To this solution add 10 g of solution azide (NaN_3) dissolved in 40 ml of distilled water.

6.3 Sulfuric acid: concentrated.

6.4 Starch solution: Prepare an emulsion of 10 g soluble starch in a mortar or beaker with a small quantity of distilled water. Pour this emulsion into 1 liter of boiling water, allow to boil a few minutes, and let settle overnight. Use the clear supernate. This solution may be preserved by the addition of 5 ml per liter of chloroform and storage in a 10°C refrigerator.

6.4.1 Dry, powdered starch indicators such as "thyodene" may be used in place of starch solution.

6.5 Potassium fluoride solution: Dissolve 40 g $KF \cdot 2H_2O$ in distilled water and dilute to 100 ml.

6.6 Sodium thiosulfate, stock solution, 0.75 N: Dissolve 186.15 g $Na_2S_2O_3 \cdot 5H_2O$ in boiled and cooled distilled water and dilute to 1 liter. Preserve by adding 5 ml chloroform.

6.7 Sodium thiosulfate standard titrant, 0.0375 N: Prepare by diluting 50.0 ml of stock solution to 1 liter. Preserve by adding 5 ml of chloroform. Standard sodium thiosulfate, exactly 0.0375 N is equivalent to 0.300 mg of DO per 1.00 ml. Standardize with 0.0375 N potassium biiodate.

6.8 Potassium biiodate standard, 0.0375 N: For stock solution, dissolve 4.873 g of potassium biiodate, previously dried 2 hours at 103°C, in 1000 ml of distilled water. To prepare working standard, dilute 250 ml to 1000 ml for 0.0375 N biiodate solution.

6.9 Standardization of 0.0375 N sodium thiosulfate: Dissolve approximately 2 g (±1.0 g) KI in 100 to 150 ml distilled water; add 10 ml of 10% H_2SO_4 followed by 20.0 ml standard potassium biiodate (6.8). Place in dark for 5 minutes, dilute to 300 ml, and titrate with the standard sodium thiosulfate (6.7) to a pale straw color. Add 1–2 ml starch solution and continue the titration drop by drop until the blue color disappears. Run in duplicate. Duplicate determinations should agree within ±0.05 ml.

6.10 As an alternative to the sodium thiosulfate, phenylarsine oxide (PAO) may be used. This is available, already standardized, from commercial sources.

7. Procedure

7.1 To the sample collected in the BOD incubation bottle, add 2 ml of the manganous sulfate solution (6.1) followed by 2 ml of the alkaline iodide-azide solution (6.2), well below the surface of the liquid; stopper with care to exclude air bubbles, and mix well by inverting the bottle several times. When the precipitate settles, leaving a clear supernatant above the manganese hydroxide floc, shake again. When settling has produced at least 200 ml of clear supernatant, carefully remove the stopper and immediately add 2 ml of conc. H_2SO_4 (6.3) (sulfamic acid packets, 3 g may be substituted for H_2SO_4)[1] by allowing the acid to run down the neck of the bottle, re-stopper, and mix by gentle inversion until the iodine is uniformly distributed throughout the bottle. Complete the analysis within 45 minutes.

7.2 Transfer the entire bottle contents by inversion into a 500 ml wide mouth flask and titrate with 0.0375 N thiosulfate solution (6.7) (0.0375 N phenyarsine oxide (PAO) may be substituted as titrant) to pale straw color. Add 1–2 ml of starch solution (6.4) or 0.1 g of powdered indicator and continue to titrate to the first disappearance of the blue color.

7.3 If ferric iron is present (100 to 200 mg/l), add 1.0 ml of KF (6.5) solution before acidification.

7.4 Occasionally, a dark brown or black precipitate persists in the bottle after acidication. This precipitate will dissolve if the solution is kept for a few minutes longer than usual or, if particularly persistent, a few more drops of H_2SO_4 will effect dissolution.

8. Calculation

8.1 Each ml of 0.0375N sodium thiosulfate (or PAO) titrant is equivalent to 1 mg DO when the entire bottle contents are titrated.

8.2 If the results are desired in milliliters of oxygen gas per liter at 0°C and 760 mm pressure multiply mg/l DO by 0.698.

8.3 To express the results as percent saturation at 760 mm atmospheric pressure, the solubility data in Table 422:1 (Whipple & Whipple, p 446–447, Standard Methods, 14th Edition) may be used. Equations for correcting the solubilities to barometric pressures other than mean sea level are given below the table.

8.4 The solubility of DO in distilled water at any barometric pressure, p (mm Hg), temperature, T°C, and saturated vapor pressure, u (mm Hg), for the given T, may be calculated between the temperature of 0° and 30°C by:

$$\text{ml/l DO} = \frac{(P - u) \times 0.678}{35 + T}$$

and between 30° and 50°C by:

$$\text{ml/l DO} = \frac{(P - u) \times 0.827}{49 + T}$$

9. Precision and Accuracy

9.1 Exact data are unavailable on the precision and accuracy of this technique; however, reproducibility is approximately 0.2 mg/l of DO at the 7.5 mg/l level due to equipment tolerances and uncompensated displacement errors.

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PHOSPHORUS, ALL FORMS

Method 365.1 (Colorimetric, Automated, Ascorbic Acid)

STORET NO. See Section 4

1. Scope and Application
   - 1.1 These methods cover the determination of specified forms of phosphorus in drinking, surface and saline waters, domestic and industrial wastes.
   - 1.2 The methods are based on reactions that are specific for the orthophosphate ion. Thus, depending on the prescribed pre-treatment of the sample, the various forms of phosphorus given in Figure 1 may be determined. These forms are defined in Section 4.
     - 1.2.1 Except for in-depth and detailed studies, the most commonly measured forms are phosphorus and dissolved phosphorus, and orthophosphate and dissolved orthophosphate. Hydrolyzable phosphorus is normally found only in sewage-type samples. Insoluble forms of phosphorus are determined by calculation.
   - 1.3 The methods are usable in the 0.01 to 1.0 mg P/l range. Approximately 20-30 samples per hour can be analyzed.
2. Summary of Method
   - 2.1 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.
   - 2.2 Only orthophosphate forms a blue color in this test. Polyphosphates (and some organic phosphorus compounds) may be converted to the orthophosphate form by manual sulfuric acid hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by manual persulfate digestion[2]. The developed color is measured automatically on the AutoAnalyzer.
3. Sample Handling and Preservation
   - 3.1 If benthic deposits are present in the area being sampled, great care should be taken not to include these deposits.
   - 3.2 Sample containers may be of plastic material, such as cubitainers, or of Pyrex glass.
   - 3.3 If the analysis cannot be performed the same day of collection, the sample should be preserved by the addition of 2 ml conc. H_2SO_4 per liter and refrigeration at 4°C.
4. Definitions and Storet Numbers
   - 4.1 Total Phosphorus (P) – all of the phosphorus present in the sample regardless of form, as measured by the persulfate digestion procedure. (00665)
     - 4.1.1 Total Orthophosphate (P-ortho)–inorganic phosphorus $[(PO_4)^{-3}]$ in the sample as measured by the direct colorimetric analysis procedure. (70507)

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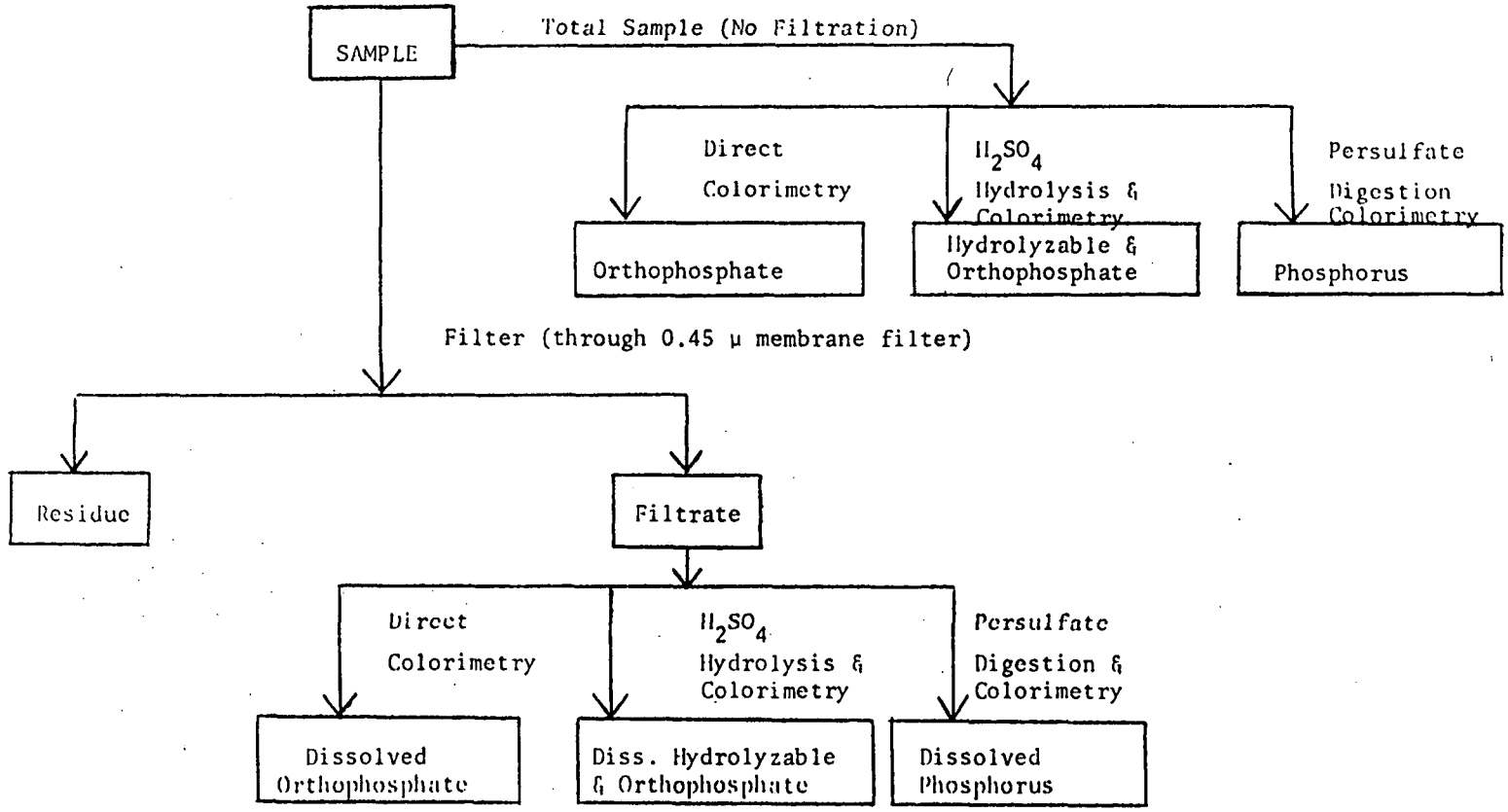

FIGURE 1. ANALYTICAL SCHEME FOR DIFFERENTIATION OF PHOSPHORUS FORMS

4.1.2 Total Hydrolyzable Phosphorus (P-hydro)–phosphorus in the sample as measured by the sulfuric acid hydrolysis procedure, and minus predetermined orthophosphates. This hydrolyzable phosphorus includes polyphosphates [$(P_2O_7)^{-4}$, $(P_3O_{10})^{-5}$, etc.] plus some organic phosphorus. (00669)

4.1.3 Total Organic Phosphorus (P-org)–phosphorus (inorganic plus oxidizable organic) in the sample as measured by the persulfate digestion procedure, and minus hydrolyzable phosphorus and orthophosphate. (00670)

4.2 Dissolved Phosphorus (P–D) – all of the phosphorus present in the filtrate of a sample filtered through a phosphorus-free filter of 0.45 micron pore size and measured by the persulfate digestion procedure. (00666)

4.2.1 Dissolved Orthophosphate (P–D, ortho) – as measured by the direct colorimetric analysis procedure. (00671)

4.2.2 Dissolved Hydrolyzable Phosphorus (P–D, hydro) – as measured by the sulfuric acid hydrolysis procedure and minus predetermined dissolved orthophosphates. (00672)

4.2.3 Dissolved Organic Phosphorus (P–D, org) – as measured by the persulfate digestion procedure, and minus dissolved hydrolyzable phosphorus and orthophosphate. (00673)

4.3 The following forms, when sufficient amounts of phosphorus are present in the sample to warrant such consideration, may be calculated:

4.3.1 Insoluble Phosphorus (P–I) = (P) – (P–D). (00667)

4.3.1.1 Insoluble orthophosphate (P–I, ortho) = (P, ortho) – (P–D, ortho). (00674)

4.3.1.2 Insoluble Hydrolyzable Phosphorus (P–I, hydro) = (P, hydro) – (P–D, hydro). (00675)

4.3.1.3 Insoluble Organic Phosphorus (P–I, org) = (P, org) – (P–D, org). (00676)

4.4 All phosphorus forms shall be reported as P, mg/l, to the third place.

5. Interferences

5.1 No interference is caused by copper, iron, or silicate at concentrations many times greater than their reported concentration in sea water. However, high iron concentrations can cause precipitation of and subsequent loss of phosphorus.

5.2 The salt error for samples ranging from 5 to 20% salt content was found to be less than 1%.

5.3 Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than phosphorus. However, at concentrations found in sea water, it does not interfere.

5.4 Sample turbidity must be removed by filtration prior to analysis for orthophosphate. Samples for total or total hydrolyzable phosphorus should be filtered only after digestion. Sample color that absorbs in the photometric range used for analysis will also interfere.

6. Apparatus

6.1 Technicon AutoAnalyzer consisting of:

6.1.1 Sampler.

6.1.2 Manifold (AAI) or Analytical Cartridge (AAII).

6.1.3 Proportioning pump.

6.1.4 Heating bath, 50°C.

6.1.5 Colorimeter equipped with 15 or 50 mm tubular flow cell.

6.1.6 650–660 or 880 nm filter.

6.1.7 Recorder.

6.1.8 Digital printer for AAII (optional).

6.2 Hot plate or autoclave.

6.3 Acid-washed glassware: All glassware used in the determination should be washed with hot 1:1 HCl and rinsed with distilled water. The acid-washed glassware should be filled with distilled water and treated with all the reagents to remove the last traces of phosphorus that might be adsorbed on the glassware. Preferably, this glassware should be used only for the determination of phosphorus and after use it should be rinsed with distilled water and kept covered until needed again. If this is done, the treatment with 1:1 HCl and reagents is only required occasionally. Commercial detergent should never be used.

7. Reagents

7.1 Sulfuric acid solution, 5N: Slowly add 70 ml of conc. H_2SO_4 to approximately 400 ml of distilled water. Cool to room temperature and dilute to 500 ml with distilled water.

7.2 Antimony potassium tartrate solution: Weigh 0.3 g $K(SbO)C_4H_4O_6 \cdot 1/2H_2O$, dissolve in 50 ml distilled water in 100 ml volumetric flask, dilute to volume. Store at 4°C in a dark, glass-stoppered bottle.

7.3 Ammonium molybdate solution: Dissolve 4 g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 100 ml distilled water. Store in a plastic bottle at 4°C.

7.4 Ascorbic acid, 0.1M: Dissolve 1.8 g of ascorbic acid in 100 ml of distilled water. The solution is stable for about a week if prepared with water containing no more than trace amounts of heavy metals and stored at 4°C.

7.5 Combined reagent (AAI): Mix the above reagents in the following proportions for 100 ml of the mixed reagent: 50 ml of 5N H_2SO_4 (7.1), 5 ml of antimony potassium tartrate solution (7.2), 15 ml of ammonium molybdate solution (7.3), and 30 ml of ascorbic acid solution (7.4). Mix after addition of each reagent. All reagents must reach room temperature before they are mixed and must be mixed in the order given. If turbidity forms in the combined reagent, shake and let stand for a few minutes until the turbidity disappears before processing. This volume is sufficient for 4 hours operation. Since the stability of this solution is limited, it must be freshly prepared for each run.

NOTE 1: A stable solution can be prepared by not including the ascorbic acid in the combined reagent. If this is done, the mixed reagent (molybdate, tartrate, and acid) is pumped through the distilled water line and the ascorbic acid solution (30 ml of 7.4 diluted to 100 ml with distilled water) through the original mixed reagent line.

7.6 Sulfuric acid solution, 11 N: Slowly add 310 ml conc. H_2SO_4 to 600 ml distilled water. When cool, dilute to 1 liter.

7.7 Ammonium persulfate.

7.8 Acid wash water: Add 40 ml of sulfuric acid solution (7.6) to 1 liter of distilled water and dilute to 2 liters. (Not to be used when only orthophosphate is being determined).

7.9 Phenolphthalein indicator solution (5 g/l): Dissolve 0.5 g of phenolphthalein in a solution of 50 ml of ethyl or isopropyl alcohol and 50 ml of distilled water.

7.10 Stock phosphorus solution: Dissolve 0.4393 g of pre-dried (105°C for 1 hour) KH_2PO_4 in distilled water and dilute to 1000 ml. 1.0 ml = 0.1 mg P.

7.11 Standard phosphorus solution: Dilute 100.0 ml of stock solution (7.10) to 1000 ml with distilled water. 1.0 ml = 0.01 mg P.

7.12 Standard phosphorus solution: Dilute 100.0 ml of standard solution (7.11) to 1000 ml with distilled water. 1.0 ml = 0.001 mg P.

7.13 Prepare a series of standards by diluting suitable volumes of standard solutions (7.11) and (7.12) to 100.0 ml with distilled water. The following dilutions are suggested:

ml of Standard Phosphorus Solution (7.12)	Conc., mg P/l
0.0	0.00
2.0	0.02
5.0	0.05
10.0	0.10

ml of Standard Phosphorus Solution (7.11)	mg P/l
2.0	0.20
5.0	0.50
8.0	0.80
10.0	1.00

8. Procedure

8.1 Phosphorus

8.1.1 Add 1 ml of sulfuric acid solution (7.6) to a 50 ml sample and/or standard in a 125 ml Erlenmeyer flask.

8.1.2 Add 0.4 g of ammonium persulfate.

8.1.3 Boil gently on a pre-heated hot plate for approximately 30–40 minutes or until a final volume of about 10 ml is reached. Do not allow sample to go to dryness. Alternately, heat for 30 minutes in an autoclave at 121°C (15–20 psi).

8.1.4 Cool and dilute the sample to 50 ml. If sample is not clear at this point, filter.

8.1.5 Determine phosphorus as outlined in (8.3.2) with acid wash water (7.8) in wash tubes.

8.2 Hydrolyzable Phosphorus

8.2.1 Add 1 ml of sulfuric acid solution (7.6) to a 50 ml sample and/or standard in a 125 ml Erlenmeyer flask.

8.2.2 Boil gently on a pre-heated hot plate for 30–40 minutes or until a final volume of about 10 ml is reached. Do not allow sample to go to dryness. Alternatively, heat for 30 minutes in an autoclave at 121°C (15–20 psi).

8.2.3 Cool and dilute the sample to 50 ml. If sample is not clear at this point, filter.

8.2.4 Determine phosphorus as outlined in (8.3.2) with acid wash water (7.8) in wash tubes.

8.3 Orthophosphate

8.3.1 Add 1 drop of phenolphthalein indicator solution (7.9) to approximately 50 ml of sample. If a red color develops, add sulfuric acid solution (7.6) drop-wise to just discharge the color. Acid samples must be neutralized with 1 N sodium hydroxide (40 g NaOH/1).

8.3.2 Set up manifold as shown in Figure 2, AAI or Figure 3, AAII.

8.3.3 Allow both colorimeter and recorder to warm up for 30 minutes. Obtain a stable baseline with all reagents, feeding distilled water through the sample line.

8.3.4 For the AAI system, sample at a rate of 20/hr, 1 minute sample, 2 minute wash. For the AAII system, use a 30/hr, 2:1 cam, and a common wash.

8.3.5 Place standards in Sampler in order of decreasing concentration. Complete filling of sampler tray with unknown samples.

8.3.6 Switch sample line from distilled water to Sampler and begin analysis.

9. Calculation

9.1 Prepare a standard curve by plotting peak heights of processed standards against known concentrations. Compute concentrations of samples by comparing sample peak heights with standard curve. Any sample whose computed value is less than 5% of its immediate predecessor must be rerun.

10. Precision and Accuracy (AAI system)

10.1 Six laboratories participating in an EPA Method Study, analyzed four natural water samples containing exact increments of orthophosphate, with the following results:

Increment as Orthophosphate mg P/liter	Precision as Standard Deviation mg P/liter	Accuracy as Bias, %	Accuracy as Bias, mg P/liter
0.04	0.019	+16.7	+0.007
0.04	0.014	− 8.3	−0.003
0.29	0.087	−15.5	−0.05
0.30	0.066	−12.8	−0.04

10.2 In a single laboratory (EMSL), using surface water samples at concentrations of 0.04, 0.19, 0.35, and 0.84 mg P/1, standard deviations were ±0.005, ±0.000, ±0.003, and ±0.000, respectively.

10.3 In a single laboratory (EMSL), using surface water samples at concentrations of 0.07 and 0.76 mg p/1, recoveries were 99% and 100%, respectively.

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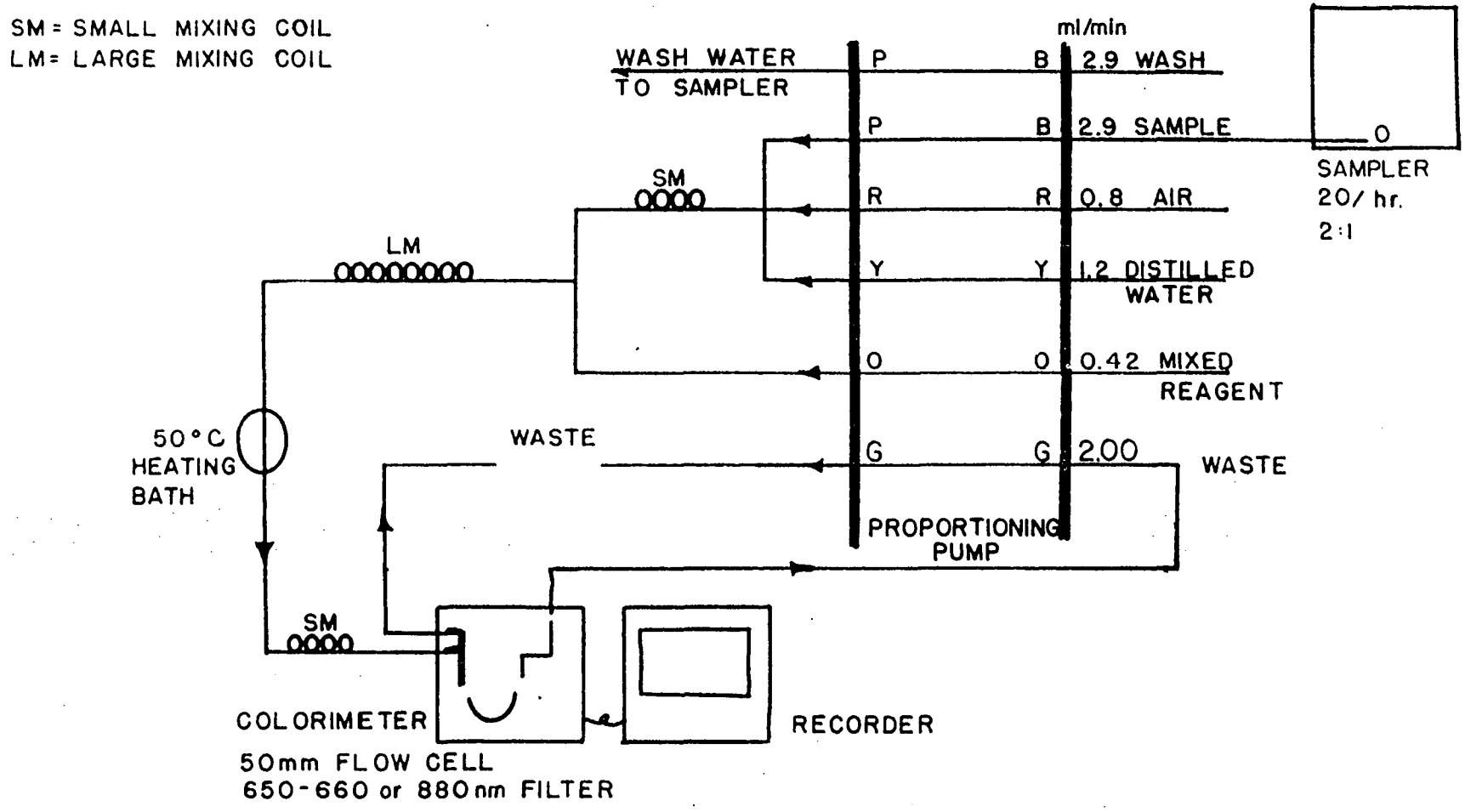
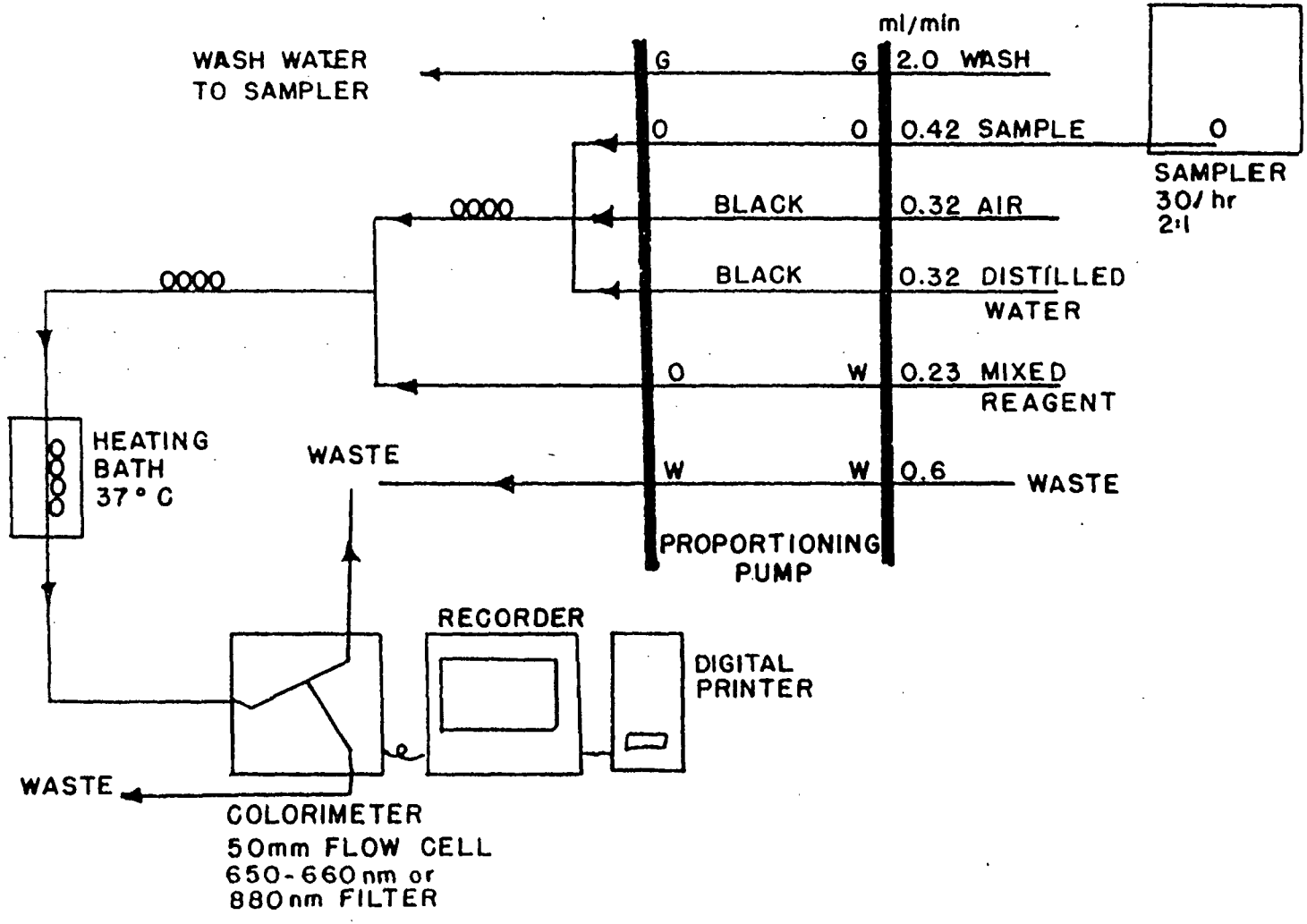


365.1-8

FIGURE 2 PHOSPHORUS MANIFOLD AA I



365.1-9

FIGURE 3 PHOSPHORUS MANIFOLD AA II

8.3.3 After 5 minutes, measure the absorbance at 650 nm with a spectrophotometer and determine the phosphorus concentration from the standard curve. The color is stable for at least one hour. For concentrations in the range of 0.01 to 0.3 mg P/l, a 5 cm cell should be used. A one cm cell should be used for concentrations in the range of 0.3 to 1.2 mg P/l.

9. Calculation

9.1 Prepare a standard curve by plotting the absorbance values of standards versus the corresponding phosphorus concentrations on linear graph paper.

9.2 Obtain concentration value of sample directly from prepared standard curve. Report results as P, mg/l.

10. Precision and Accuracy

10.1 Precision data is not available at this time.

10.2 In a single laboratory (EMSL) using industrial waste and sewage samples at concentrations of 7.6 and 0.55 mg P/l, recoveries were 99 and 100%, respectively.

7. Reagents

7.1 Ammonium molybdate-antimony potassium tartrate solution: Dissolve 8 g of ammonium molybdate and 0.2 g antimony potassium tartrate in 800 ml of distilled water and dilute to 1 liter.

7.2 Ascorbic acid solution: Dissolve 60 g of ascorbic acid in 800 ml of distilled water and dilute to 1 liter. Add 2 ml of acetone. This solution is stable for two weeks.

7.3 Sulfuric acid, 11 N: Slowly add 310 ml of conc. H_2SO_4 to approximately 600 ml distilled water. Cool and dilute to 1000 ml.

7.4 Sodium bisulfite ($NaHSO_3$) solution: Dissolve 5.2 g of $NaHSO_3$ in 100 ml of 1.0 N H_2SO_4.

7.5 Ammonium persulfate.

7.6 Stock phosphorus solution: Dissolve 0.4393 g of predried (105°C for one hour) KH_2PO_4 in distilled water and dilute to 1000 ml. 1.0 ml = 0.1 mg P.

7.7 Standard phosphorus solution: Dilute 100 ml of stock phosphorus solution to 1000 ml with distilled water. 1.0 ml = 0.01 mg P. Prepare an appropriate series of standards by diluting suitable volumes of standard or stock solutions to 100 ml with distilled water.

8. Procedure

8.1 Total Phosphorus

8.1.1 Transfer 50 ml of sample or an aliquot diluted to 50 ml into a 125 ml Erlenmeyer flask and add 1 ml of 11 N sulfuric acid (7.3).

8.1.2 Add 0.4 g ammonium persulfate (7.5), mix and boil gently for approximately 30–40 minutes or until a final volume of about 10 ml is reached. Alternatively heat for 30 minutes in an autoclave at 121°C (15–20 psi). Cool, dilute to approximately 40 ml and filter.

8.1.3 For samples containing arsenic or high levels of iron, add 5 ml of sodium bisulfite (7.4), mix and place in a 95°C water bath for 30 minutes (20 minutes after the temperature of the sample reaches 95°C). Cool and dilute to 50 ml.

8.1.4 Determine phosphorus as outlined in (8.3) orthophosphate.

8.2 Hydrolyzable Phosphorus

8.2.1 Add 1 ml of H_2SO_4 solution (8.3) to a 50 ml sample in a 125 ml Erlenmeyer flask.

8.2.2 Boil gently on a pre-heated hot plate for 30–40 minutes or until a final volume of about 10 ml is reached. Do not allow sample to go to dryness. Alternatively, heat for 30 minutes in an autoclave at 121°C (15–20 psi). Cool, dilute to approximately 40 ml and filter.

8.2.3 Treat the samples as in 8.1.3.

8.2.4 Determine phosphorus as outlined in (8.3) orthophosphate.

8.3 Orthophosphate

8.3.1 To 50 ml of sample and/or standards, add 1 ml of 11 N sulfuric acid (7.3) and 4 ml of ammonium molybdate-antimony potassium tartrate (7.1) and mix.

NOTE: If sample has been digested for total or hydrolyzable phosphorus do not add acid.

8.3.2 Add 2 ml of ascorbic acid solution (7.2) and mix.

4.1.3 Total Organic Phosphorus (P, org) — phosphorus (inorganic plus oxidizable organic) in the sample measured by the persulfate digestion procedure, and minus hydrolyzable phosphorus and orthophosphate. (00670)

4.2 Dissolved Phosphorus (P–D) — all of the phosphorus present in the filtrate of a sample filtered through a phosphorus-free filter of 0.45 micron pore size and measured by the persulfate digestion procedure. (00666)

4.2.1 Dissolved Orthophosphate (P–D, ortho) — as measured by the direct colorimetric analysis procedure. (00671)

4.2.2 Dissolved Hydrolyzable Phosphorus (P–D, hydro) — as measured by the sulfuric acid hydrolysis procedure and minus pre-determined dissolved orthophosphates. (00672)

4.2.3 Dissolved Organic Phosphorus (P–D, org) — as measured by the persulfate digestion procedure, and minus dissolved hydrolyzable phosphorus and orthophosphate. (00673)

4.3 The following forms, when sufficient amounts of phosphorus are present in the sample to warrant such consideration, may be calculated:

4.3.1 Insoluble Phosphorus (P-I)=(P)-(P-D). (00667)

4.3.1.1 Insoluble orthophosphate (P-I, ortho)=(P, ortho) – (P-D, ortho). (00674)

4.3.1.2 Insoluble Hydrolyzable Phosphorus (P–I, hydro)=(P, hydro) – (P–D, hydro). (00675)

4.3.1.3 Insoluble Organic Phosphorus (P–I, org)=(P, org) – (P-D, org). (00676)

4.4 All phosphorus forms shall be reported as P, mg/1, to the third place.

5. Interferences

5.1 Arsenate is determined similarily to phosphorus and should be considered when present. This interference may be eliminated by reducing the arsenic acid to arsenious acid with sodium bisulfite (7.4).

5.2 When high concentrations of iron are present low recovery of phosphorus will be obtained because it will use some of the reducing agent. The bisulfite treatment will also eliminate this interference.

6. Apparatus

6.1 Photometer–A spectrophotometer or filter photometer suitable for measurements at 660 or 880 nm with a light path of 1 cm or longer.

6.2 Acid-washed glassware: All glassware used should be washed with hot 1:1 HCl and rinsed with distilled water. The acid-washed glassware should be filled with distilled water and treated with all the reagents to remove the last traces of phosphorus that might be absorbed on the glassware. Preferably, this glassware should be used only for the determination of phosphorus and after use it should be rinsed with distilled water and kept covered until needed again. If this is done, the treatment with 1:1 HCl and reagents is only required occasionally. Commercial detergents should never be used.

6.3 Water bath, 95°C.

PHOSPHORUS, ALL FORMS

Method 365.3 (Colorimetric, Ascorbic Acid, Two Reagent)

STORET NO. See Section 4

1. Scope and Application
   - 1.1 These methods cover the determination of specified forms of phosphorus in drinking, surface and saline waters, domestic and industrial wastes.
   - 1.2 The methods are based on reactions that are specific for the orthophosphate ion. Thus, depending on the prescribed pretreatment of the sample, the various forms may be determined.
     - 1.2.1 Except for in-depth and detailed studies, the most commonly measured forms are phosphorus and dissolved phosphorus, and orthophosphate and dissolved orthophosphate. Hydrolyzable phosphorus is normally found only in sewage-type samples and insoluble forms of phosphorus are determined by calculation.
   - 1.3 The methods are usable in the 0.01 to 1.2 mg P/1 range.
2. Summary of Method
   - 2.1 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.
   - 2.2 Only orthophosphate forms a blue color in this test. Polyphosphates (and some organic phosphorus compounds) may be converted to the orthophosphate form by sulfuric-acid-hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by persulfate digestion.
3. Sample Handling and Preservation
   - 3.1 If benthic deposits are present in the area being sampled, great care should be taken not to include these deposits.
   - 3.2 Sample containers may be of plastic material, such as cubitainers, or of Pyrex glass.
   - 3.3 If the analysis cannot be performed the day of collection, the sample should be preserved by the addition of 2 ml conc. H_2SO_4 per liter and refrigeration at 4°C.
4. Definitions and Storet Numbers
   - 4.1 Total Phosphorus (P) — all of the phosphorus present in the sample, regardless of form, as measured by the persulfate digestion procedure. (00665)
     - 4.1.1 Total Orthophosphate (P, ortho) — inorganic phosphorus $[(PO_4)^{-3}]$ in the sample as measured by the direct colorimetric analysis procedure. (70507)
     - 4.1.2 Total Hydrolyzable Phosphorus (P, hydro) - phosphorus in the sample as measured by the sulfuric acid hydrolysis procedure, and minus pre-determined orthophosphates. This hydrolyzable phosphorus includes polyphosphorus. $[(P_2O_7)^{-4}, (P_3O_{10})^{-5}$, etc.] plus some organic phosphorus. (00669)

Approved for NPDES
Issued 1978

10. Precision and Accuracy

10.1 Thirty-three analysts in nineteen laboratories analyzed natural water samples containing exact increments of organic phosphate, with the following results:

Increment as Total Phosphorus mg P/liter	Precision as Standard Deviation mg P/liter	Accuracy as Bias, %	Accuracy as Bias mg P/liter
0.110	0.033	+3.09	+0.003
0.132	0.051	+11.99	+0.016
0.772	0.130	+2.96	+0.023
0.882	0.128	-0.92	-0.008

(FWPCA Method Study 2, Nutrient Analyses)

10.2 Twenty-six analysts in sixteen laboratories analyzed natural water samples containing exact increments of orthophosphate, with the following results:

Increment as Orthophosphate mg P/liter	Precision as Standard Deviation mg P/liter	Accuracy as Bias, %	Accuracy as Bias, mg P/liter
0.029	0.010	-4.95	-0.001
0.038	0.008	-6.00	-0.002
0.335	0.018	-2.75	-0.009
0.383	0.023	-1.76	-0.007

(FWPCA Method Study 2, Nutrient Analyses)

Bibliography

1. Murphy, J., and Riley, J., "A modified Single Solution for the Determination of Phosphate in Natural Waters", Anal. Chim. Acta., 27, 31 (1962).
2. Gales, M., Jr., Julian, E., and Kroner, R., "Method for Quantitative Determination of Total Phosphorus in Water", Jour. AWWA, 58, No. 10, 1363 (1966).
3. Annual Book of ASTM Standards, Part 31, "Water", Standard D515-72, Method A, p 389 (1976).
4. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 476 and 481, (1975).

8. Procedure

8.1 Phosphorus

8.1.1 Add 1 ml of H_2SO_4 solution (7.6) to a 50 ml sample in a 125 ml Erlenmeyer flask.

8.1.2 Add 0.4 g of ammonium persulfate.

8.1.3 Boil gently on a pre-heated hot plate for approximately 30–40 minutes or until a final volume of about 10 ml is reached. Do not allow sample to go to dryness. Alternatively, heat for 30 minutes in an autoclave at 121°C (15–20 psi).

8.1.4 Cool and dilute the sample to about 30 ml and adjust the pH of the sample to 7.0 ±0.2 with 1 N NaOH (7.10) using a pH meter. If sample is not clear at this point, add 2–3 drops of acid (7.6) and filter. Dilute to 50 ml.
Alternatively, if autoclaved see **NOTE 1.**

8.1.5 Determine phosphorus as outlined in 8.3.2 Orthophosphate.

8.2 Hydrolyzable Phosphorus

8.2.1 Add 1 ml of H_2SO_4 solution (7.6) to a 50 ml sample in a 125 ml Erlenmeyer flask.

8.2.2 Boil gently on a pre-heated hot plate for 30–40 minutes or until a final volume of about 10 ml is reached. Do not allow sample to go to dryness. Alternatively, heat for 30 minutes in an autoclave at 121°C (15–20 psi).

8.2.3 Cool and dilute the sample to about 30 ml and adjust the pH of the sample to 7.0 ±0.2 with NaOH (7.10) using a pH meter. If sample is not clear at this point, add 2–3 drops of acid (7.6) and filter. Dilute to 50 ml.
Alternatively, if autoclaved see **NOTE 1.**

8.2.4 The sample is now ready for determination of phosphorus as outlined in 8.3.2 Orthophosphate.

8.3 Orthophosphate

8.3.1 The pH of the sample must be adjusted to 7±0.2 using a pH meter.

8.3.2 Add 8.0 ml of combined reagent (7.5) to sample and mix thoroughly. After a minimum of ten minutes, but no longer than thirty minutes, measure the color absorbance of each sample at 650 or 880 nm with a spectrophotometer, using the reagent blank as the reference solution.

NOTE 1: If the same volume of sodium hydroxide solution is not used to adjust the pH of the standards and samples, a volume correction has to be employed.

9. Calculation

9.1 Prepare a standard curve by plotting the absorbance values of standards versus the corresponding phosphorus concentrations.

9.1.1 Process standards and blank exactly as the samples. Run at least a blank and two standards with each series of samples. If the standards do not agree within ±2% of the true value, prepare a new calibration curve.

9.2 Obtain concentration value of sample directly from prepared standard curve. Report results as P, mg/l. SEE **NOTE 1.**

kept covered until needed again. If this is done, the treatment with 1:1 HCl and reagents is only required occasionally. Commercial detergents should never be used.

7. Reagents

7.1 Sulfuric acid solution, 5N: Dilute 70 ml of conc. H_2SO_4 with distilled water to 500 ml.

7.2 Antimony potassium tartrate solution: Weigh 1.3715 g $K(SbO)C_4H_4O_6 \cdot 1/2H_2O$, dissolve in 400 ml distilled water in 500 ml volumetric flask, dilute to volume. Store at 4°C in a dark, glass-stoppered bottle.

7.3 Ammonium molybdate solution: Dissolve 20 g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 500 ml of distilled water. Store in a plastic bottle at 4°C.

7.4 Ascorbic acid, 0.1M: Dissolve 1.76 g of ascorbic acid in 100 ml of distilled water. The solution is stable for about a week if stored at 4°C.

7.5 Combined reagent: Mix the above reagents in the following proportions for 100 ml of the mixed reagent: 50 ml of 5N H_2SO_4, (7.1), 5 ml of antimony potassium tartrate solution (7.2), 15 ml of ammonium molybdate solution (7.3), and 30 ml of ascorbic acid solution (7.4). Mix after addition of each reagent. All reagents must reach room temperature before they are mixed and must be mixed in the order given. If turbidity forms in the combined reagent, shake and let stand for a few minutes until the turbidity disappears before proceeding. Since the stability of this solution is limited, it must be freshly prepared for each run.

7.6 Sulfuric acid solution, 11 N: Slowly add 310 ml conc. H_2SO_4 to 600 ml distilled water. When cool, dilute to 1 liter.

7.7 Ammonium persulfate.

7.8 Stock phosphorus solution: Dissolve in distilled water 0.2197 g of potassium dihydrogen phosphate, KH_2PO_4, which has been dried in an oven at 105°C. Dilute the solution to 1000 ml; 1.0 ml = 0.05 mg P.

7.9 Standard phosphorus solution: Dilute 10.0 ml of stock phosphorus solution (7.8) to 1000 ml with distilled water; 1.0 ml = 0.5 ug P.

7.9.1 Using standard solution, prepare the following standards in 50.0 ml volumetric flasks:

ml of Standard Phosphorus Solution (7.9)	Conc., mg/l
0	0.00
1.0	0.01
3.0	0.03
5.0	0.05
10.0	0.10
20.0	0.20
30.0	0.30
40.0	0.40
50.0	0.50

7.10 Sodium hydroxide, 1 N: Dissolve 40 g NaOH in 600 ml distilled water. Cool and dilute to 1 liter.

4.1.3 Total Organic Phosphorus (P, org) — phosphorus (inorganic plus oxidizable organic) in the sample measured by the persulfate digestion procedure, and minus hydrolyzable phosphorus and orthophosphate. (00670)

4.2 Dissolved Phosphorus (P–D) — all of the phosphorus present in the filtrate of a sample filtered through a phosphorus-free filter of 0.45 micron pore size and measured by the persulfate digestion procedure. (00666)

4.2.1 Dissolved Orthophosphate (P–D, ortho) — as measured by the direct colorimetric analysis procedure. (00671)

4.2.2 Dissolved Hydrolyzable Phosphorus (P–D, hydro) — as measured by the sulfuric acid hydrolysis procedure and minus pre-determined dissolved orthophosphates. (00672)

4.2.3 Dissolved Organic Phosphorus (P–D, org) — as measured by the persulfate digestion procedure, and minus dissolved hydrolyzable phosphorus and orthophosphate. (00673)

4.3 The following forms, when sufficient amounts of phosphorus are present in the sample to warrant such consideration, may be calculated:

4.3.1 Insoluble Phosphorus (P-I)=(P)-(P-D). (00667)

4.3.1.1 Insoluble orthophosphate (P-I, ortho)=(P, ortho)-(P-D, ortho). (00674)

4.3.1.2 Insoluble Hydrolyzable Phosphorus (P-I, hydro)=(P, hydro)-(P-D, hydro). (00675)

4.3.1.3 Insoluble Organic Phosphorus (P-I, org)=(P, org) – (P-D, org). (00676)

4.4 All phosphorus forms shall be reported as P, mg/1, to the third place.

5. Interferences

5.1 No interference is caused by copper, iron, or silicate at concentrations many times greater than their reported concentration in sea water. However, high iron concentrations can cause precipitation of and subsequent loss of phosphorus.

5.2 The salt error for samples ranging from 5 to 20% salt content was found to be less than 1%.

5.3 Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than phosphorus. However, at concentrations found in sea water, it does not interfere.

6. Apparatus

6.1 Photometer – A spectrophotometer or filter photometer suitable for measurements at 650 or 880 nm with a light path of 1 cm or longer.

6.2 Acid-washed glassware: All glassware used should be washed with hot 1:1 HCl and rinsed with distilled water. The acid-washed glassware should be filled with distilled water and treated with all the reagents to remove the last traces of phosphorus that might be adsorbed on the glassware. Preferably, this glassware should be used only for the determination of phosphorus and after use it should be rinsed with distilled water and

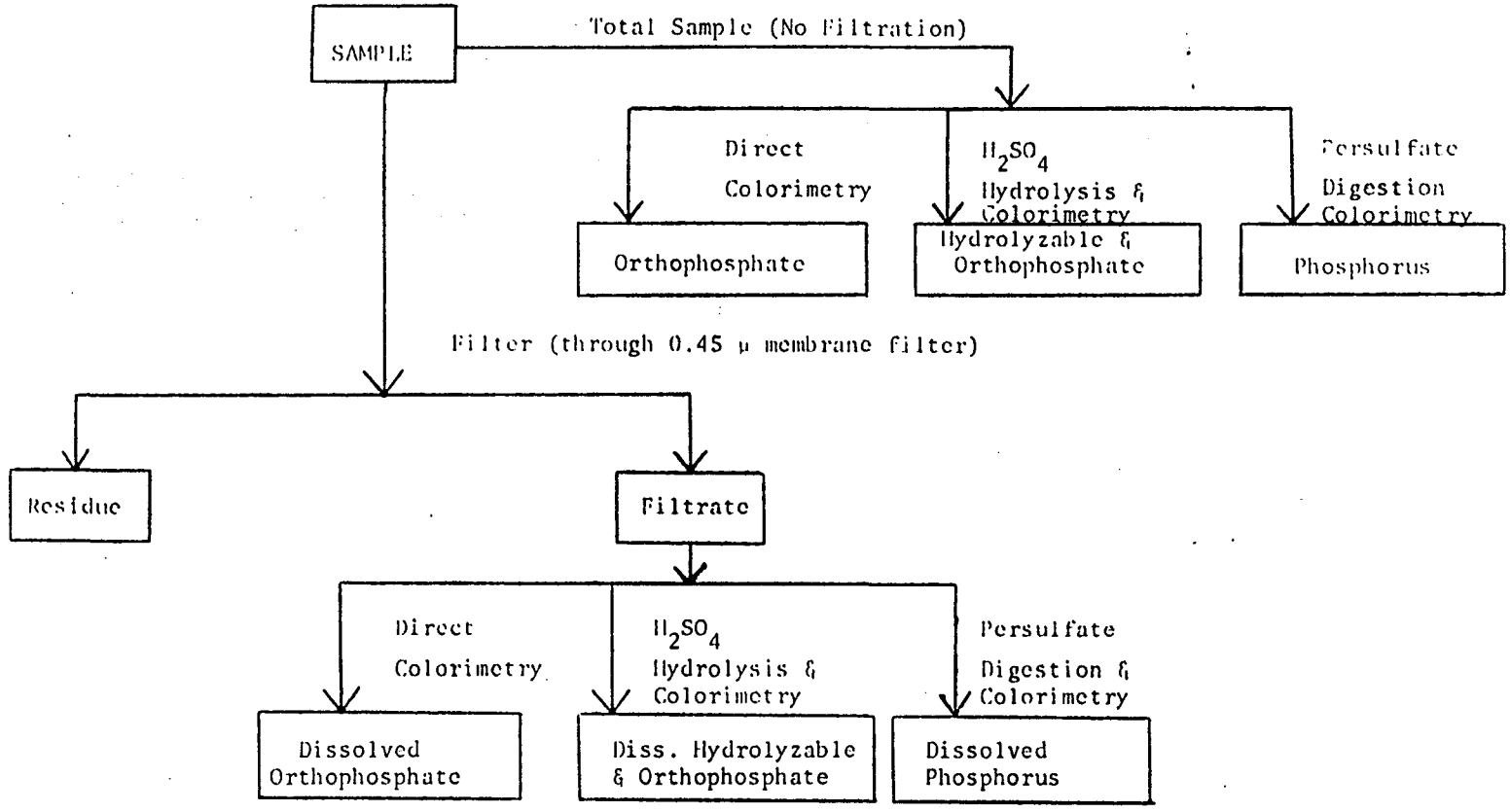


FIGURE 1. ANALYTICAL SCHEME FOR DIFFERENTIATION OF PHOSPHORUS FORMS

PHOSPHORUS, ALL FORMS

Method 365.2 (Colorimetric, Ascorbic Acid, Single Reagent)

STORET NO. See Section 4

1. Scope and Application
   - 1.1 These methods cover the determination of specified forms of phosphorus in drinking, surface and saline waters, domestic and industrial wastes.
   - 1.2 The methods are based on reactions that are specific for the orthophosphate ion. Thus, depending on the prescribed pre-treatment of the sample, the various forms of phosphorus given in Figure 1 may be determined. These forms are defined in Section 4.
     - 1.2.1 Except for in-depth and detailed studies, the most commonly measured forms are phosphorus and dissolved phosphorus, and orthophosphate and dissolved orthophosphate. Hydrolyzable phosphorus is normally found only in sewage-type samples and insoluble forms of phosphorus are determined by calculation.
   - 1.3 The methods are usable in the 0.01 to 0.5 mg P/1 range.
2. Summary of Method
   - 2.1 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.
   - 2.2 Only orthophosphate forms a blue color in this test. Polyphosphates (and some organic phosphorus compounds) may be converted to the orthophosphate form by sulfuric acid hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by persulfate digestion[2].
3. Sample Handling and Preservation
   - 3.1 If benthic deposits are present in the area being sampled, great care should be taken not to include these deposits.
   - 3.2 Sample containers may be of plastic material, such as cubitainers, or of Pyrex glass.
   - 3.3 If the analysis cannot be performed the day of collection, the sample should be preserved by the addition of 2 ml conc. H_2SO_4 per liter and refrigeration at 4°C.
4. Definitions and Storet Numbers
   - 4.1 Total Phosphorus (P) — all of the phosphorus present in the sample, regardless of form, as measured by the persulfate digestion procedure. (00665)
     - 4.1.1 Total Orthophosphate (P, ortho) — inorganic phosphorus [$(PO_4)^{-3}$] in the sample as measured by the direct colorimetric analysis procedure. (70507)
     - 4.1.2 Total Hydrolyzable Phosphorus (P, hydro) - phosphorus in the sample as measured by the sulfuric acid hydrolysis procedure, and minus pre-determined orthophosphates. This hydrolyzable phosphorus includes polyphosphorus. [$(P_2O_7)^{-4}$, $(P_3O_{10})^{-5}$, etc.] plus some organic phosphorus. (00669)

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2) It is critical that the following reagents be added as follows and in this order: to each of the remaining two bottles add 2 ml manganous sulfate and 2 ml alkali iodide azide. Place the tip of the pipet below the water surface and drain the contents along the side of the bottle to avoid adding any air bubbles.

3) Cap the bottles and invert to mix the contents thoroughly. Let the solids settle.

4) Dissolve approximately 2 g KI, free from iodate, in a 500 ml Erlenmeyer flask with 100 ml distilled water. Add 10 ml 10% H_2SO_4 and 20 ml standard potassium biodate solution (.025 N) by pipette. Dilute to 200 ml.

5) Invert the two BOD bottles again and mix thoroughly. Allow the solids to settle.

6) Rinse a 50 ml buret with two 5 ml portions of 0.025 N sodium thiosulfate. Fill the buret and record the initial volume.

7) To the two BOD bottles, add 2 ml concentrated H_2SO_4 to the side of the bottle. Cap and invert several times to mix.

8) Titrate the potassium bi-iodate solution with sodium thiosulfate to a light yellow. Add a dropper-full of starch solution, and the potassium biodate will turn blue. Titrate to a clear, colorless solution - about 20 ml. Record the volume used. Refill the buret.

9) Measure into 203 ml cut-off volumetric flasks the solutions in the BOD bottles. Then transfer to 500 ml Erlenmeyer flasks. Titrate to the same endpoint as above. Record the volume used - about 8 ml. Refill the buret as needed.

Preparation

Dry glucose and glutamic acid at 103°C for two hours. Cool in a dessicator. Weigh 0.150 g of each on an analytical balance, dissolve in about 500 ml distilled water and dilute to 1000 ml. Keep refrigerated and prepare fresh every two weeks.

Procedure

Sample Preparation Dilutions

1) The pH of the sample should be checked. If not in the range of 6.5 to 7.5 the pH should be adjusted with dilute acid or base.

2) The sample should be diluted so the depletion due to the sample will be in the range of 1.0-6.5 mg/l after a 5 day incubation. See the attached sheet for BOD estimates and dilutions. The BOD bottles have a 300 ml capacity so the final dilution will always be to 300 ml.

   In cases where an estimate of the BOD is not given, a good estimate for the BOD can be obtained from the BOD result.

   1/2 COD mg/l = BOD estimate mg/l

   This method works best for sewage runs and will not work as well on other samples. Especially for industrial wastes, the BOD may be less than half of the COD. Based on the estimate, most samples should be set up with two different dilutions in order to ensure correct depletion. In some cases and particularly for new industrial samples, more than two dilutions should be used.

Calibration of the Dissolved Oxygen Meter (do daily)

1) Fill 3 BOD bottles with distilled water that has been kept at 20°C. Cap one bottle and set aside in incubator.

3) The COD of the seed should be determined weekly, on Tuesdays. Based on the COD results, determine the concentration of seed to be added to the dilution water.

COD of seed, g/l	ml of seed per liter of dilution water
<0.20	12
0.20-0.25	10
0.25-0.30	7
>0.30	5

Standards

Sodium Thiosulfate, Stock Standard, 0.1 N

Sodium Thiosulfate - purchased concentrate. Prepare according to the directions on the label.

Sodium Thiosulfate, Working Standard, 0.025 N

Sodium Thiosulfate, 0.1 N	250 ml
Distilled Water	750 ml

Preparation

Dilute 250 ml sodium thiosulfate to 1000 ml in a volumetric flask.

Potassium Bi-iodate, Stock Standard, 0.025 N

Potassium Bi-iodate, purchased as 0.025 N.

BOD Standard

Glucose (dextrose)	0.150 g
Glutamic Acid	0.150 g
Distilled Water	1000 ml

Dilution Water

Phosphate Buffer Solution	20 ml
Magnesium Sulfate Solution	20 ml
Calcium Chloride Solution	20 ml
Ferric Chloride Solution	20 ml
Distilled Water	19 l

Preparation

Fill a 20 l bottle with 19 l distilled water and aerate gently for 20 minutes. Cover the water and store at 20°C overnight. Remove from incubator immediately before use, and add the four nutrients listed above. Stir thoroughly. The correct amount of seed should be added after the water check and seed correction bottles have been filled.

Nitrification Inhibition

2-Chloro-6(trichloromethyl) pyridine (Hach Formula 2533), purchased solid.

Add 0.16 g to each BOD bottle (including standards) before filling bottles with dilution water.

Seed

Stale, filtered sewage from the local Sewage Treatment Plant is used as seed material.

1) Three acid bottles of sewage should be picked up every three weeks, or when needed. The seed should be allowed to settle one week before use, and should be kept refrigerated.

2) This seed should be filtered through glass wool, one bottle at a time, when needed.

Alkali Iodide Azide

Sodium Hydroxide (NaOH)	500 g
Sodium Iodide (NaI)	135 g
Sodium Azide (NaN_3)	10 g
Distilled Water	1040 ml

Preparation

Dissolve the sodium hydroxide in 500 ml distilled water. To this add the sodium iodide, dissolve and dilute to 1000 ml. To this solution add 10 g sodium azide dissolved in 40 ml distilled water. Total: 1040 ml. ****CAUTION****

Sulfuric Acid, Concentrated (H_2SO_4)

Sulfuric Acid, 10%

Sulfuric Acid (H_2SO_4)	100 ml
Distilled Water	900 ml

Preparation

Measure distilled water into a 1 liter pyrex glass stoppered bottle. Add the sulfuric acid VERY SLOWLY as it will generate considerable heat, and mix well. ****CAUTION****

Starch Solution

Starch	2 g
Distilled Water	100 ml
Salicylic Acid	0.2 g

Preparation

Dissolve the starch and salicylic acid in 100 ml hot distilled water. Refrigerate, and prepare fresh weekly. The starch solution should be discarded if there is any sign of biological growth in the reagent bottle.

Magnesium Sulfate

Magnesium Sulfate ($MgSO_4.7H_2O$)	22.5 g
Distilled Water	

Preparation

Dissolve the magnesium sulfate in about 500 ml distilled water and dilute to 1000 ml.

Calcium Chloride

Calcium Chloride ($CaCl_2$)	27.5 g
Distilled Water	

Preparation

Dissolve the calcium chloride in about 500 ml distilled water and dilute to 1000 ml.

Ferric Chloride

Ferric Chloride $FeCl_2.6H_2O$)	0.25 g
Distilled Water	

Preparation

Dissolve the ferric chloride in about 500 ml distilled water and dilute to 1000 ml.

Manganous Sulfate

Manganous Sulfate ($MnSO_4.H_2O$)	364 g
Distilled Water	

Preparation

Dissolve the manganous sulfate in about 500 ml distilled water, filter through white ribbon filter paper and dilute to 1000 ml.

f) Adjust the pH of the solution to 6.5 to 7.5.

g) Transfer to the appropriate size volumetric flask and dilute to the mark.

h) As a check, pour a small amount of the first dilution into a beaker. Add several drops of sodium sulfide. If the solution turns brown or there is more precipitate the solution was not completely decoppered and the procedure must be repeated from step (a).

i) Test the first dilution for excess sulfide with lead acetate paper. In the presence of excess sulfide, the lead acetate paper will turn black.

j) If too much sodium sulfide was added the sample should be aerated for 10 minutes then checked as in step (h) and step (i).

Reagents

Phosphate Buffer

Preparation

Potassium Dihydrogen Phosphate (KH_2PO_4)	8.5 g
Dipotassium Hydrogen Phosphate (K_2HPO_4)	21.75 g
Disodium Hydrogen Phosphate Heptahydrate ($Na_2HPO_4.7H_2O$)	33.4 g
Ammonium Chloride (NH_4Cl)	1.7 g
Distilled Water	

Preparation

Dissolve all reagents in about 500 ml distilled water and dilute to 1000 ml. The pH of this buffer should be 7.2 without further adjusting. Discard the solution if there is any sign of biological growth in the stock bottle.

2. NH_3, Chromium, Cyanide

The presence of these chemicals in a waste may lower the BOD or produce a toxic effect and kill the seed. If this is suspected, the waste should be set up in 4 to 5 different dilutions, covering a wide concentration range. An unusual dependence of the concentration of the waste on the BOD will often suggest such an effect is being observed, and it should be noted carefully.

3. Copper

Copper also is toxic to seeding organisms, but may be removed by the following procedure:

a) Place the volume of sample to be used for the first dilution in a 150 ml beaker and add 40-50 ml distilled water. Stir on a magnetic stirrer.

b) Attach sulfide electrodes to a pH meter. Use the millivolt (mV) scale.

c) Add sodium sulfide (1 M) dropwise to the solution. Stir the solution slowly and monitor with the sulfide electrodes. The sodium sulfide solution should smell strongly of sulfur. If not, make up fresh solution by dissolving 1.5 g of sodium sulfide in distilled water and dilute to 200 ml.

d) Add sodium sulfide slowly until no more brown precipitate forms upon addition or until there is a large jump in the mV readings.

e) Filter the solution through MILLIPORE rinsing everything thoroughly.

Discussion

The biochemical oxygen demand procedure is used to determine the relative oxygen requirements of municipal and industrial wastewaters. The test measure the biological oxygen demand of carbonaceous compounds and inorganic materials such as sulfides and ferrous iron. It may also measure the oxygen demand of nitrogenous compounds, but this can be prevented by the use of an inhibitor.

The sample of waste, or an appropriate dilution, is incubated for 5 days at 20°C in the dark. The reduction in dissolved oxygen concentration during the incubation period yields a measure of the biochemical oxygen demand.

Performance

Unseeded Dilution Water	<0.2 mg/l
Seed Correction	0.6 to 1.0 mg/l
BOD Standard	199 ± 37.0 mg/l
Minimum Depletion of Sample	1.0 mg/l
Maximum Depletion of Sample	1.0 mg/l remaining (approximately 6.5 mg/l)

Sample Handling and Preparation

1. All BOD samples should be stored at or below 4°C.

2. Standard Methods specifies that samples should be set up within 24 hours of time of collection. When this is not possible, the sample may be set up within one week of receipt.

Interferences

1. pH

   The pH of the sample or dilution may affect the BOD determination, and it must be between 6.5 and 7.5. If the pH does not fall in this range, it should be adjusted using dilute HCl or NaOH.

ZP-193

Determination of: Biochemical Oxygen Demand (BOD)

Method: Five day dissolved oxygen reduction - membrane electrode method

Reagents: Phosphate Buffer
Magnesium Sulfate
Calcium Chloride
Ferric Chloride
Manganous Sulfate
Alkali Iodide Azide
Sulfuric Acid
Starch Solution
Sodium Thiosulfate
Potassium Bi-iodate
Nitrification Inhibitor

Interferences: Ammonia, copper, chromium and cyanide will inhibit the BOD.

References: Standard Methods for the Examination of Water and Wastewater, 15th Edition, p. 483, Method 507, (1980)

EPA Method 405.1

Issued: November, 1980

Technical Revision: April, 1982

5. References

5.1 The procedure to be used for this determination is found in: Standard Methods for the Examination of Water and Wastewater, 15th Edition, p. 483, Method 507 (1980).

5.2 Young, J. C., "Chemical Methods for Nitrification Control," J. Water Poll. Control Fed., 45, p. 637 (1973).

BIOCHEMICAL OXYGEN DEMAND

Method 405.1 (5 Days, 20°C)

STORET NO. 00310
Carbonaceous 80082

1. Scope and Application
   1.1 The biochemical oxygen demand (BOD) test is used for determining the relative oxygen requirements of municipal and industrial wastewaters. Application of the test to organic waste discharges allows calculation of the effect of the discharges on the oxygen resources of the receiving water. Data from BOD tests are used for the development of engineering criteria for the design of wastewater treatment plants.
   1.2 The BOD test is an empirical bioassay-type procedure which measures the dissolved oxygen consumed by microbial life while assimilating and oxidizing the organic matter present. The standard test conditions include dark incubation at 20°C for a specified time period (often 5 days). The actual environmental conditions of temperature, biological population, water movement, sunlight, and oxygen concentration cannot be accurately reproduced in the laboratory. Results obtained must take into account the above factors when relating BOD results to stream oxygen demands.
2. Summary of Method
   2.1 The sample of waste, or an appropriate dilution, is incubated for 5 days at 20°C in the dark. The reduction in dissolved oxygen concentration during the incubation period yields a measure of the biochemical oxygen demand.
3. Comments
   3.1 Determination of dissolved oxygen in the BOD test may be made by use of either the Modified Winkler with Full-Bottle Technique or the Probe Method in this manual.
   3.2 Additional information relating to oxygen demanding characteristics of wastewaters can be gained by applying the Total Organic Carbon and Chemical Oxygen Demand tests (also found in this manual).
   3.3 The use of 60 ml incubation bottles in place of the usual 300 ml incubation bottles, in conjunction with the probe, is often convenient.
4. Precision and Accuracy
   4.1 Eighty-six analysts in fifty-eight laboratories analyzed natural water samples plus an exact increment of biodegradable organic compounds. At a mean value of 2.1 and 175 mg/1 BOD, the standard deviation was ±0.7 and ±26 mg/1, respectively (EPA Method Research Study 3).
   4.2 There is no acceptable procedure for determining the accuracy of the BOD test.

Approved for NPDES CBOD: pending approval for Section 304(h), CWA
Issued 1971
Editorial revision 1974

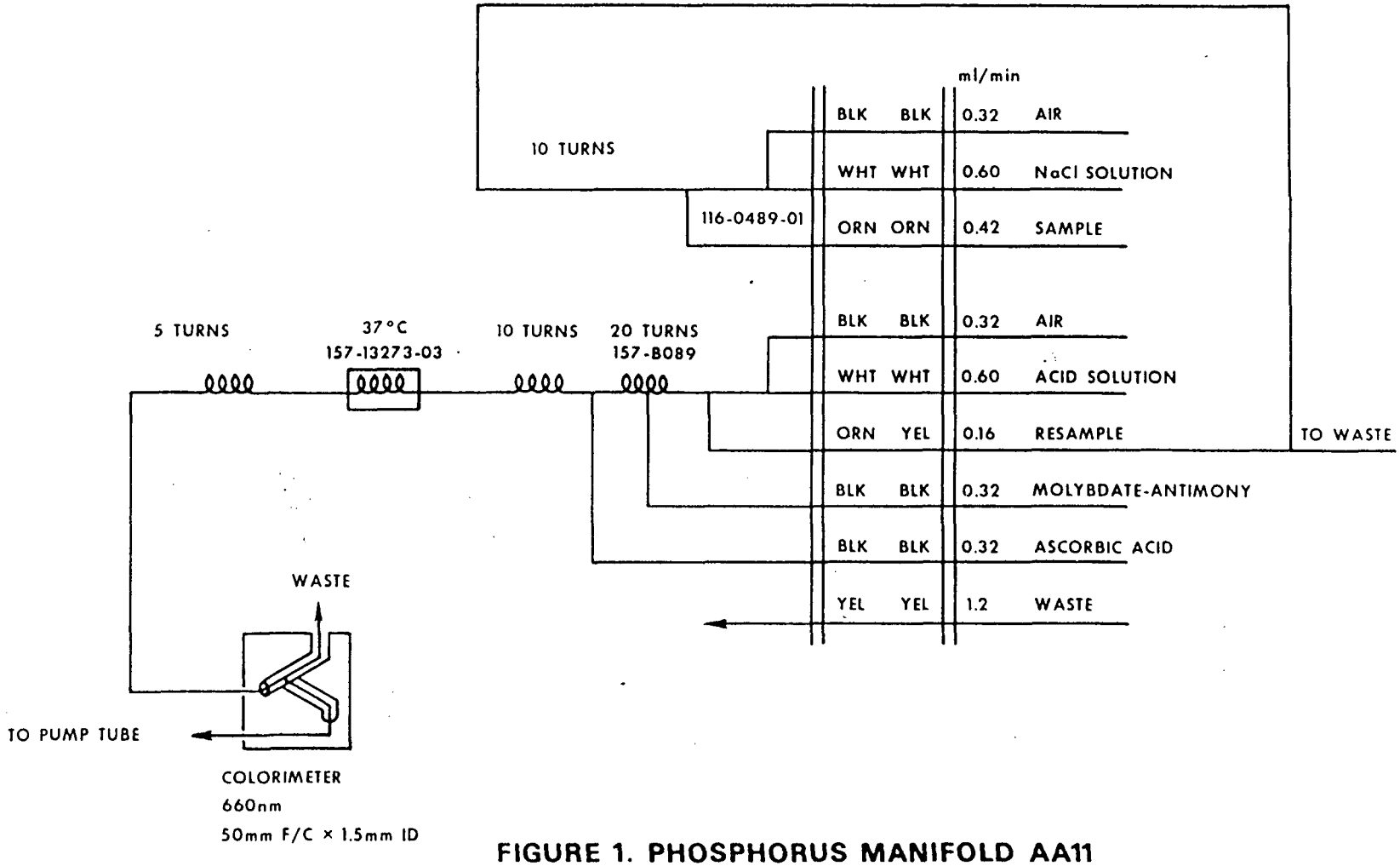

FIGURE 1. PHOSPHORUS MANIFOLD AA11

6.3 With Block Digestor in manual mode set low and high temperature at 160°C and preheat unit to 160°C. Place tubes in digestor and switch to automatic mode. Set low temperature timer for 1 hour. Reset high temperature to 380°C and set timer for 2 1/2 hours.

6.4 Cool sample and dilute to 25 ml with distilled water. If TKN is determined the sample should be diluted with ammonia-free water.

Colorimetric Analysis

6.4.1 Check the level of all reagent containers to ensure an adequate supply.

6.4.2 Excluding the molybdate/antimony line, place all reagent lines in their respective containers, connect the sample probe to the Sampler IV and start the proportioning pump.

6.4.3 Flush the Sampler IV wash receptacle with about 25 ml of 4% sulfuric acid (5.7).

6.4.4 When reagents have been pumping for at least five minutes, place the molybdate/antimony line in its container and allow the system to equilibrate.

6.4.5 After a stable baseline has been obtained, start the sampler.

7. Calculations

7.1 Prepare a standard curve by plotting peak heights of processed standards against concentration values. Compute concentrations by comparing sample peak heights with the standard curve.

8. Precision and Accuracy

8.1 In a single laboratory (EMSL) using sewage sample containing total P at levels of 0.23, 1.33, and 2.0, the precision was ±0.01, ±0.04, and ±0.06, respectively.

8.2 In a single laboratory (EMSL) using sewage samples of concentration 1.84 and 1.89, the recoveries were 95 and 98%, respectively.

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2. Gales, M.E. and Booth, R.L., "Evaluation of Organic Nitrogen Methods", EPA Office of Research and Monitoring, June, 1972.
3. Gales, M.E. and Booth, R.L., "Simultaneous and Automated Determination of Total Phosphorus and Total Kjeldahl Nitrogen", Methods Development and Quality Assurance Research Laboratory, May, 1974.
4. Technicon "Total Kjeldahl Nitrogen and Total Phosphorus BD-40 Digestion Procedure for Water", August, 1974.
5. Gales, M.E., and Booth, R.L., "Evaluation of the Technicon Block Digestor System for the Measurement of Total Kjeldahl Nitrogen and Total Phosphorus", EPA-600 /4–78–015, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

PHOSPHORUS, TOTAL

Method 365.4 (Colorimetric, Automated, Block Digestor AA II)

STORET NO. 00665

1. Scope and Application
   1.1 This method covers the determination of total phosphorus in drinking water, surface water and domestic and industrial wastes. The applicable range of this method is 0.01 to 20 mg P/1.
2. Summary of Method
   2.1 The sample is heated in the presence of sulfuric acid, K_2SO_4 and $HgSO_4$ for two and one half hours. The residue is cooled, diluted to 25 ml and placed on the AutoAnalyzer for phosphorus determination.
3. Sample Handling and Preservation
   3.1 Sample containers may be of plastic material, such as a cubitainer, or of Pyrex glass.
   3.2 If the analysis cannot be performed the day of collection, the sample should be preserved by the addition of 2 ml of conc. H_2SO_4 per liter and refrigeration at 4°C.
4. Apparatus
   4.1 Block Digestor BD–40
   4.2 Technicon Method No. 327–74W for Phosphorus
5. Reagents
   5.1 Mercuric sulfate: Dissolve 8 g red mercuric oxide (HgO) in 50 ml of 1:4 sulfuric acid (10 conc. H_2SO_4: 40 ml distilled water) and dilute to 100 ml with distilled water.
   5.2 Digestion solution: (Sulfuric acid-mercuric sulfate-potassium sulfate solution): Dissolve 133 g of K_2SO_4 in 600 ml of distilled water and 200 ml of conc. H_2SO_4. Add 25 ml of mercuric sulfate solution (5.1) and dilute to 1 liter.
   5.3 Sulfuric acid solution (0.72 N): Add 20 ml of conc. sulfuric acid to 800 of distilled water, mix and dilute to 1 liter.
   5.4 Molybdate/antimony solution: Dissolve 8 g of ammonium molybdate and 0.2 g of antimony potassium tartrate in about 800 ml of distilled water and dilute to 1 liter.
   5.5 Ascorbic acid solution: Dissolve 60 g of ascorbic acid in about 600 ml of distilled water. Add 2 ml of acetone and dilute to 1 liter.
   5.6 Diluent water: Dissolve 40 g of NaCl in about 600 ml of distilled water and dilute to 1 liter.
   5.7 Sulfuric acid solution, 4%: Add 40 ml of conc. sulfuric acid to 800 ml of ammonia-free distilled water, cool and dilute to 1 liter.
6. Procedure

   Digestion

   6.1 To 20 or 25 ml of sample, add 5 ml of digestion solution and mix. (Use a vortex mixer).
   6.2 Add 4–8 Teflon boiling chips. Too many boiling chips will cause the sample to boil over.

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10) From the attached BOD Sodium Thiosulfate Standardization Factor chart and the volume used to titrate the potassium bi-iodate solution, the correction factor for the second titration is obtained. By multiplying the correction factor and the average value obtained for the second titration, the value for standardizing the DO meter is determined. Record all values in the journal.

Example:

19.90 ml = volume of sodium thiosulfate to titrate potassium bi-iodate solution

8.15 ml = volume of sodium thiosulfate to titrate manganous sulfate solution

1.004 = correction factor (from chart)

Meter setting = 1.004 x 8.15

= 8.18

Standardization of BOD Meter (Orion)

Standardization of Meter:

1) With the electrode off, turn the meter to the pH mode and set the slope control to 100% and the temperature control to 25°C.

2) Set the reading to 7.00 using the meters calibration control. Meter is now calibrated. The meter should be turned to standby mode when samples are not being read or the probe is transferred to another bottle.

Standarization of BOD Probe:

1) Turn the electrode mode switch to BT CK (battery check). Good battery operation is indicated by a meter reading of 13.00 or greater.

2) Turn the mode switch to zero. Use the probe's zero calibration control to set the meter display at 0.00.

3) Turn the mode switch to H_2O. Place the DO probe in the BOD bottle of distilled water from step 1, page 6. Use the air calibration control to set the display to the meter setting determined from the titrations.

4) Recheck calibration at least once per hour, or more often if necessary.

5) The electrode will automatically compensate for sample temperature.

NOTE: When a fresh membrane electrolyte module has been installed, wait 15 to 30 minutes before calibrating.

Setting Up

1) Samples are generally set up on Wednesday, Thursday, and Friday for a 5-day BOD. Incubated samples are read back 5 days later on Monday, Tuesday, and Wednesday.

2) Each sample is set up with at least two dilutions. Every fifteenth sample should be set up in duplicate, both dilutions being duplicated. If the depletion for both dilutions falls in the appropriate range, both results should be recorded.

3) Using the BOD worksheets (sample attached) record sample numbers, dilutions and dilution factors. Indicate whether seeding, neutralization, or inhibiting has been done. Unless otherwise requested, all samples are seeded and inhibited.

4) The first worksheet is reserved for seed and dilution water checks and all are set up in duplicate.

a) Unseeded dilution water, containing nutrients and inhibitor.
b) Seed, 10 ml, dilution factor is 30X.
c) Seed, 15 ml, dilution factor is 20X.
d) Seed, 20 ml, dilution factor is 15X.
e) BOD Standard, 5 ml.

The seed and BOD standard should be allowed to warm to room temperature before sampling.

5) Starting with the standards sheet, record the bottle numbers used for each sample.

6) Transfer the appropriate volume of standard, seed, and samples to the appropriate bottles. Inhibitor should then be added to all bottles.

7) Using dilution water previously prepared fill the first 8 standard bottles. The bottles are filled by inserting a tubing to the bottom of the bottle and allowing the dilution water to fill the bottle from the bottom up without creating any turbulence. Stopper the bottles and set aside. The initial dissolved oxygen measurement should be made on all bottles about 15 minutes after filling. After determining and recording the initial dissolved oxygen, any displaced dilution water should be carefully replenished and the bottles re-stoppered and water-sealed. Plastic caps should then be placed on the bottles, and all bottles should be placed in the incubator.

8) Determine the volume of seed to be added to the remaining dilution water.

ml seed = 1 dilution water x ml seed used per liter of dilution water.

For example, if 7 ml of seed were used per liter of dilution water, 19 l of water would require 7 x 19 or 133 ml of seed.

9) Add the seed to the dilution water and mix thoroughly. Fill the remaining 2 standard bottles (BOD standard), and all samples with seeded dilution water. Stopper the bottles, and read the initial dissolved oxygen 15 minutes after filling, as directed in Step 7. After reading and recording, replenish dilution water if necessary, stopper, water-seal, cap and place bottles in incubator.

10) After five days, remove the bottles from the incubator and determine the dissolved oxygen remaining. Record the values on the BOD worksheets.

Calculations

A. Calculation of Seed Correction:

1) The seed is normally set up with 3 different dilutions and is recorded on the standards page. Subtract the final D.O. from the initial D.O. reading. This is the depletion due to seed and must be between 1.00 and 6.50 mg/l to be used in the following calculations.

2) Determine the BOD of the seed: Multiply the depletion due to seed by the appropriate dilution factor. For example, a 10 ml sample of seed produced a depletion of 3.60 mg/l. Multiplying by 30 (dilution factor), the BOD of the seed would be 108 mg/l.

3) Divide the BOD of the seed by the dilution factor for the seed in the dilution water. This is the seed correction.

ml of Feed in 1 l of Dilution Water	Dilution Factor
5	200X
7	143X
10	100X
12	83X

For example, if 7 ml of seed were used per liter of dilution water, 108 mg/l (BOD from step 2) would be divided by 143. The seed correction would be 0.75.

4) Determine the average seed correction for each of the six seed dilutions in the BOD bottles, and use this number in determining sample BOD's.

B. Calculation of Sample BOD:

1) The final D.O. meter reading of a sample must be 1.00 mg/l or greater. If not, repeat the 5-day BOD using a smaller sample size.

2) Subtract the final D.O. from the initial D.O. reading. This result is the total depletion and it should be 2.00 mg/l or greater.

3) Subtract the seed correction for the day from the total depletion to give depletion due to sample. This number must be at least 1.00 mg/l. If less than this, repeat the 5-day BOD using a larger sample size.

4) Multiply the depletion due to sample by the dilution factor, and record the answer.

Cleaning BOD Bottles and Glassware - Use No Soap

1) Wash BOD bottles with Micro cleaning solution each time they are used.

2) Rinse each bottle at least twice with hot water and twice with distilled water.

3) 20 liter plastic bottles for dilution water should be washed with Micro cleaning solution once a month.

NOTES

1) Keep the bottles in order.
   - a bucket will hold 12 bottles
   - a shelf in the incubator will hold bottles 6 deep

2) Cleanliness of the glassware is very important.

3) Set up samples the same week they are received.

4) Change membrane module every 6 months. Check batteries in probe daily and change every 4 months.

5) Water seal and cover the BOD bottles to be incubated.

6) Check and record the temperature of the incubators at least daily.

7) Keep close check on the values of the seed correction, BOD standard and unseeded dilution water. If these values exceed the limits described under the section on Performance, the BOD results for the day may have to be discarded and the source of error should be determined and corrected.

BOD Dilutions

Assumptions:

1. Initial Dissolved Oxygen $\geq$ 7.5 mg/l
2. Dissolved Oxygen after depletion is between 1.0 and 6.5 mg/l

BOD Est. mg/l	Dilution Factor	1st Dilution	2nd Dilution (into BOD Bottle)
3-19.5	3	-	100-300
6-39.5	6	-	50-300
10-65	10	-	30-300
30-195	30	-	10-300
60-390	60	-	5-300
150-975	150	-	2-300
300-1950	300	10-50	5-300
600-3900	600	5-100	10-300
1000-6500	1000	5-100	6-300
2000-13,000	2000	5-200	6-300
5000-32,500	5000	5-500	6-300
10,000-65,000	10000	5-500	3-300

FIVE-DAY 20°C BIOCHEMICAL OXYGEN DEMAND

INITIAL STANDARDIZATION: _____ FINAL STANDARDIZATION: _____

DATA IN: _____ DATA OUT: _____

_____ MLS OF SEED PER _____ LITERS OF DILUTION WATER

SAMPLE NUMBER										
PROJECT NUMBER										
CHEMIST										
ESTIMATED BOD										
SPECIAL INSTRUCTION										
DILUTION FACTOR										
FIRST DILUTION										
BOTTLE DILUTION										
BOTTLE NUMBER										
INITIAL READING										
FINAL READING										
TOTAL DEPLETION										
SEED CORRECTION										
SAMPLE DEPLETION										
DILUTION FACTOR										
BOD mg/l										
CORRECTION FACTOR	x1.02	x1.02	x1.02	x1.02	x1.02	x1.02	x1.02	x1.02	x1.02	x1.02
RECORDED BOD										

B.O.D. SODIUM THIOSULFATE STANDARDIZATION FACTORS

ml	N	X Factor
19.50	.0256	1.024
19.55	.0256	1.024
19.60	.0255	1.020
19.65	.0254	1.016
19.70	.0254	1.016
19.75	.0253	1.012
19.80	.0253	1.012
19.85	.0252	1.008
19.90	.0251	1.004
19.95	.0251	1.004
20.00	.0250	1.000
20.05	.0249	.996
20.10	.0249	.996
20.15	.0248	.992
20.20	.0248	.992
20.25	.0247	.988
20.30	.0246	.984
20.35	.0246	.984
20.40	.0245	.980
20.45	.0244	.976
20.50	.0244	.976
20.55	.0243	.972
20.60	.0243	.972
20.65	.0242	.968
20.70	.0242	.968
20.75	.0241	.964
20.80	.0241	.964
20.85	.0240	.960
20.90	.0239	.956
20.95	.0239	.956
21.00	.0238	.952

CHEMICAL OXYGEN DEMAND

Method 410.1 (Titrimetric, Mid-Level)

STORET NO. 00340

1. Scope and Application
   - 1.1 The Chemical Oxygen Demand (COD) method determines the quantity of oxygen required to oxidize the organic matter in a waste sample, under specific conditions of oxidizing agent, temperature, and time.
   - 1.2 Since the test utilizes a specific chemical oxidation the result has no definite relationship to the Biochemical Oxygen Demand (BOD) of the waste or to the Total Organic Carbon (TOC) level. The test result should be considered as an independent measurement of organic matter in the sample, rather than as a substitute for the BOD or TOC test.
   - 1.3 The method can be applied to domestic and industrial waste samples having an organic carbon concentration greater than 50 mg/1. For lower concentrations of carbon such as in surface water samples, the Low Level Modification should be used. When the chloride concentration of the sample exceeds 2000 mg/1, the modification for saline waters is required.
2. Summary of Method
   - 2.1 Organic and oxidizable inorganic substances in the sample are oxidized by potassium dichromate in 50% sulfuric acid solution at reflux temperature. Silver sulfate is used as a catalyst and mercuric sulfate is added to remove chloride interference. The excess dichromate is titrated with standard ferrous ammonium sulfate, using orthophenanthroline ferrous complex as an indicator.
3. Sampling and Preservation
   - 3.1 Collect the samples in glass bottles, if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.
   - 3.2 Biologically active samples should be tested as soon as possible. Samples containing settleable material should be well mixed, preferably homogenized, to permit removal of representative aliquots.
   - 3.3 Samples should be preserved with sulfuric acid to a pH < 2 and maintained at 4°C until analysis.
4. Interferences
   - 4.1 Traces of organic material either from the glassware or atmosphere may cause a gross, positive error.
     - 4.1.1 Extreme care should be exercised to avoid inclusion of organic materials in the distilled water used for reagent preparation or sample dilution.
     - 4.1.2 Glassware used in the test should be conditioned by running blank procedures to eliminate traces of organic material.

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4.2 Volatile materials may be lost when the sample temperature rises during the sulfuric acid addition step. To minimize this loss the flask should be cooled during addition of the sulfuric acid solution.

4.3 Chlorides are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate is added to the digestion flask to complex the chlorides, thereby effectively eliminating the interference on all but brine and estuarine samples.

5. Apparatus

5.1 Reflux apparatus: Glassware should consist of a 500 ml Erlenmeyer flask or a 300 ml round bottom flask made of heat-resistant glass connected to a 12 inch Allihn condenser by means of a ground glass joint. Any equivalent reflex apparatus may be substituted provided that a ground-glass connection is used between the flask and the condenser.

6. Reagents

6.1 Distilled water: Special precautions should be taken to insure that distilled water used in this test be low in organic matter.

6.2 Standard potassium dichromate solution (0.250 N): Dissolve 12.259 g $K_2Cr_2O_7$, primary standard grade, previously dried at 103°C for two hours, in distilled water and dilute to 1000 ml.

6.3 Sulfuric acid reagent: Conc. H_2SO_4 containing 23.5g silver sulfate, Ag_2SO_4, per 4.09kg bottle. With continuous stirring, the silver sulfate may be dissolved in about 30 minutes.

6.4 Standard ferrous ammonium sulfate (0.25 N): Dissolve 98.0 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in distilled water. Add 20 ml of conc. H_2SO_4 (6.8), cool and dilute to 1 liter. This solution must be standardized daily against standard $K_2Cr_2O_7$ solution (6.2).

6.4.1 Standardization: To approximately 200 ml of distilled water add 25.0 ml of 0.25 N $K_2Cr_2O_7$ (6.2) solution. Add 20 ml of H_2SO_4 (6.8) and cool. Titrate with ferrous ammonium sulfate (6.4) using 3 drops of ferroin indicator (6.6). The color change is sharp, going from blue-green to reddish-brown.

$$\text{Normality} = \frac{(\text{ml } K_2Cr_2O_7)(0.25)}{\text{ml } Fe(NH_4)_2(SO_4)_2}$$

6.5 Mercuric sulfate: Powdered $HgSO_4$.

6.6 Phenanthroline ferrous sulfate (ferroin) indicator solution: Dissolve 1.48 g of 1–10 (ortho) phenanthroline monohydrate, together with 0.70 g of $FeSO_4 \cdot 7H_2O$ in 100 ml of water. This indicator may be purchased already prepared.

6.7 Silver sulfate: Powdered Ag_2SO_4.

6.8 Sulfuric acid (sp. gr. 1.84): Concentrated H_2SO_4.

7. Procedure

7.1 Place several boiling stones in the reflux flask, followed by 50.0 ml of sample or an aliquot diluted to 50.0 ml and 1 g of $HgSO_4$ (6.5). Add 5.0 ml conc. H_2SO_4 (6.8); swirl until the mercuric sulfate has dissolved. Place reflux flask in an ice bath and slowly add, with swirling, 25.0 ml of 0.25 N $K_2Cr_2O_7$ (6.2). Now add 70 ml of sulfuric acid-silver

sulfate solution (6.3) to the cooled reflux flask, again using slow addition with swirling motion.

Caution: Care must be taken to assure that the contents of the flask are well mixed. If not, superheating may result, and the mixture may be blown out of the open end of the condenser.

7.1.1 If volatile organics are present in the sample, use an allihn condenser and add the sulfuric acid-silver sulfate solution through the condenser, while cooling the flask, to reduce loss by volatilization.

7.2 Apply heat to the flask and reflux for 2 hours. For some waste waters, the 2-hour reflux period is not necessary. The time required to give the maximum oxidation for a wastewater of constant or known composition may be determined and a shorter period of refluxing may be permissible.

7.3 Allow the flask to cool and wash down the condenser with about 25 ml of distilled water. If a round bottom flask has been used, transfer the mixture to a 500 ml Erlenmeyer flask, washing out the reflux flask 3 or 4 times with distilled water. Dilute the acid solution to about 300 ml with distilled water and allow the solution to cool to about room temperature. Add 8 to 10 drops of ferroin indicator (6.6) to the solution and titrate the excess dichromate with 0.25 N ferrous ammonium sulfate (6.4) solution to the end point. The color change will be sharp, changing from a blue-green to a reddish hue.

7.4 Blank–Simultaneously run a blank determination following the details given in (7.1) and (7.2), but using low COD water in place of sample.

8. Calculation

8.1 Calculate the COD in the sample in mg/1 as follows:

$$\text{COD, mg/liter} = \frac{(A - B)N \times 8{,}000}{S}$$

where:

A = milliliters of $Fe(NH_4)_2(SO_4)_2$ solution required for titration of the blank,

B = milliliters of $Fe(NH_4)_2(SO_4)_2$ solution required for titration of the sample,

N = normality of the $Fe(NH_4)_2(SO_4)_2$ solution, and

S = milliliters of sample used for the test.

9. Precision and Accuracy

9.1 Eighty-six analysts in fifty-eight laboratories analyzed a distilled water solution containing oxidizable organic material equivalent to 270 mg/1 COD. The standard deviation was ±17.76 mg/1 COD with an accuracy as percent relative error (bias) of -4.7%. (EPA Method Research Study 3).

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 550, Method 508 (1975).
2. Annual Book of ASTM Standards, Part 31, "Water", Standard D1252–67, p 473 (1976).

CHEMICAL OXYGEN DEMAND

Method 410.2 (Titrimetric, Low Level)

STORET NO. 00335

1. Scope and Application
   - 1.1 The scope of this modification of the Chemical Oxygen Demand (COD) test is the same as for the high level test. It is applicable to the analysis of surface waters, domestic and industrial wastes with low demand characteristics.
   - 1.2 This method (low level) is applicable for samples having a COD in the range of 5–50 mg/l COD.
2. Summary of Method
   - 2.1 Organic and oxidizable inorganic substances in an aqueous sample are oxidized by potassium dichromate solution in 50 percent (by volume) sulfuric acid in solution. The excess dichromate is titrated with standard ferrous ammonium sulfate using orthophenanthroline ferrous complex (ferroin) as an indicator.
3. Sampling and Preservation
   - 3.1 Collect the samples in glass bottles, if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.
   - 3.2 Biologically active samples should be tested as soon as possible. Samples containing settleable material should be well mixed, preferably homogenized, to permit removal of representative aliquots.
   - 3.3 Samples should be preserved with sulfuric acid to a pH < 2 and maintained at 4°C until analysis.
4. Interferences
   - 4.1 Traces of organic material either from the glassware or atmosphere may cause a gross, positive error.
     - 4.1.1 Extreme care should be exercised to avoid inclusion of organic materials in the distilled water used for reagent preparation or sample dilution.
     - 4.1.2 Glassware used in the test should be conditioned by running blank procedures to eliminate traces of organic material.
   - 4.2 Volatile materials may be lost when the sample temperature rises during the sulfuric acid addition step.
   - 4.3 Chlorides are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate is added to the digestion flask to complex the chlorides, thereby effectively eliminating the interference on all but brine and estuarine samples.
5. Apparatus
   - 5.1 Reflux apparatus: Glassware should consist of a 500 ml Erlenmeyer flask or a 300 ml round bottom flask made of heat-resistant glass connected to a 12 inch Allihn condenser

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by means of a ground glass joint. Any equivalent reflux apparatus may be substituted provided that a ground-glass connection is used between the flask and the condenser.

6. Reagents

6.1 Distilled water: Special precautions should be taken to insure that distilled water used in this test be low in organic matter.

6.2 Standard potassium dichromate solution (0.025 N): Dissolve 12.259 g $K_2Cr_2O_7$, primary standard grade, previously dried at 103°C for two hours, in distilled water and dilute to 1000 ml. Mix this solution thoroughly then dilute 100.0 ml to 1000 ml with distilled water.

6.3 Sulfuric acid reagent: Conc. H_2SO_4 containing 23.5g silver sulfate, Ag_2SO_4, per 4.09kg bottle. (With continuous stirring, the silver sulfate may be dissolved in about 30 minutes.)

6.4 Standard ferrous ammonium sulfate (0.025 N): Dissolve 98 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in distilled water. Add 20 ml of conc. H_2SO_4 (6.8), cool and dilute to 1 liter. Dilute 100 ml of this solution to 1 liter with distilled water. This solution must be standardized daily against $K_2Cr_2O_7$ solution.

6.4.1 Standardization: To approximately 200 ml of distilled water add 25.0 ml of 0.025 N $K_2Cr_2O_7$ (6.2) solution. Add 20 ml of H_2SO_4 (6.8) and cool. Titrate with ferrous ammonium sulfate (6.4) using 3 drops of ferroin indicator (6.6). The color change is sharp, going from blue-green to reddish-brown.

$$\text{Normality} = \frac{(\text{ml } K_2Cr_2O_7)(0.025)}{\text{ml Fe } (NH_4)_2\ (SO_4)_2}$$

6.5 Mercuric sulfate : Powdered $HgSO_4$.

6.6 Phenanthroline ferrous sulfate (ferroin) indicator solution: Dissolve 1.48 g of 1–10 (ortho)phenanthroline monohydrate, together with 0.70 g of $FeSO_4 \cdot 7H_2O$ in 100 ml of water. This indicator may be purchased already prepared.

6.7 Silver sulfate : Powdered Ag_2SO_4.

6.8 Sulfuric acid (sp. gr. 1.84) : Concentrated H_2SO_4.

7. Procedure

7.1 Place several boiling stones in the reflux flask, followed by 50.0 ml of sample or an aliquot diluted to 50.0 ml and 1 g of $HgSO_4$ (6.5). Add 5.0 ml conc. H_2SO_4 (6.8); swirl until the mercuric sulfate has dissolved. Place reflux flask in an ice bath and slowly add, with swirling, 25.0 ml of 0.025 N $K_2Cr_2O_7$ (6.2). Now add 70 ml of sulfuric acid-silver sulfate solution (6.3) to the cooled reflux flask, again using slow addition with swirling motion.

Caution: Care must be taken to assure that the contents of the flask are well mixed. If not, superheating may result, and the mixture may be blown out of the open end of the condenser.

7.1.1 If volatile organics are present in the sample, use an Allihn condenser and add the sulfuric acid-silver sulfate solution through the condenser, while cooling the flask, to reduce loss by volatilization.

7.2 Apply heat to the flask and reflux for 2 hours. For some waste waters, the 2-hour reflux period is not necessary. The time required to give the maximum oxidation for a wastewater of constant or known composition may be determined and a shorter period of refluxing may be permissible.

7.3 Allow the flask to cool and wash down the condenser with about 25 ml of distilled water. If a round bottom flask has been used, transfer the mixture to a 500 ml Erlenmeyer flask, washing out the reflux flask 3 or 4 times with distilled water. Dilute the acid solution to about 300 ml with distilled water and allow the solution to cool to about room temperature. Add 8 to 10 drops of ferroin indicator (6.6) to the solution and titrate the excess dichromate with 0.025 N ferrous ammonium sulfate (6.4) solution to the end point. The color change will be sharp, changing from a blue-green to a reddish hue.

7.4 Blank—Simultaneously run a blank determination following the details given in (7.1) and (7.2), but using low COD water in place of sample.

8. Calculation

8.1 Calculate the COD in the sample in mg/l as follows:

$$\text{COD, mg/l} = \frac{(A - B)N \times 8{,}000}{S}$$

where:

A = milliliters of $Fe(NH_4)_2(SO_4)_2$ solution required for titration of the blank,
B = milliliters of $Fe(NH_4)_2(SO_4)_2$ solution required for titration of the sample,
N = normality of the $Fe(NH_4)_2(SO_4)_2$ solution, and
S = milliliters of sample used for the test.

9. Precision and Accuracy

9.1 Eighty-six analysts in fifty-eight laboratories analyzed a distilled water solution containing oxidizable organic material equivalent to 12.3 mg/l COD. The standard deviation was ±4.15 mg/l COD with an accuracy as percent relative error (bias) of 0.3%. (EPA Method Research Study 3.)

CHEMICAL OXYGEN DEMAND

Method 410.3 (Titrimetric, High Level for Saline Waters)

STORET NO. 00340

1. Scope and Application
   1.1 When the chloride level exceeds 1000 mg/l the minimum accepted value for the COD will be 250 mg/l. COD levels which fall below this value are highly questionable because of the high chloride correction which must be made.
2. Summary of Method
   2.1 Organic and oxidizable inorganic substances in an aqueous sample are oxidized by potassium dichromate solution in 50 percent (by volume) sulfuric acid solution. The excess dichromate is titrated with standard ferrous ammonium sulfate using orthophenanthroline ferrous complex (ferroin) as an indicator.
3. Sample Handling and Preservation
   3.1 Collect the samples in glass bottles, if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.
   3.2 Biologically active samples should be tested as soon as possible. Samples containing settleable material should be well mixed, preferably homogenized, to permit removal of representative aliquots.
   3.3 Samples should be preserved with sulfuric acid to a pH < 2 and maintained at 4°C until analysis.
4. Interferences
   4.1 Traces of organic material either from the glassware or atmosphere may cause a gross, positive error.
      4.1.1 Extreme care should be exercised to avoid inclusion of organic materials in the distilled water used for reagent preparation or sample dilution.
      4.1.2 Glassware used in the test should be conditioned by running blank procedures to eliminate traces of organic material.
   4.2 Volatile materials may be lost when the sample temperature rises during the sulfuric acid addition step.
   4.3 Chlorides are quantitatively oxidized by dichromate and represent a positive interference. A chloride correction is made using the procedure outlined in 7.7 of this method.
5. Apparatus
   5.1 Reflux apparatus: Glassware should consist of a 500 ml Erlenmeyer flask or a 300 ml round bottom flask made of heat-resistant glass connected to a 12 inch Allihn condenser by means of a ground glass joint. Any equivalent reflux apparatus may be substituted provided that a ground-glass connection is used between the flask and the condenser.

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6. Reagents

6.1 Standard potassium dichromate solution, (0.25 N): Dissolve 12.2588 g of $K_2Cr_2O_7$, primary standard grade, previously dried for 2 hours at 103°C in water and dilute to 1000 ml.

6.2 Sulfuric acid reagent: Conc. H_2SO_4 containing 23.5 g silver sulfate, Ag_2SO_4, per 4.09kg. bottle. (With continuous stirring, the silver sulfate may be dissolved in about 30 minutes.)

6.3 Standard ferrous ammonium sulfate, 0.250 N: Dissolve 98 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in distilled water. Add 20 ml of conc. H_2SO_4, (6.7), cool and dilute to 1 liter. This solution must be standardized against the standard potassium dichromate solution (6.1) daily.

6.3.1 Standardization: Dilute 25.0 ml of standard dichromate solution (6.1) to about 250 ml with distilled water. Add 20 ml conc. sulfuric acid (6.7). Cool, then titrate with ferrous ammonium sulfate titrant (6.3), using 10 drops of ferroin indicator (6.5).

$$\text{Normality} = \frac{(\text{ml } K_2Cr_2O_7)(0.25)}{\text{ml } Fe(NH_4)_2(SO_4)_2}$$

6.4 Mercuric sulfate : Powdered $HgSO_4$.

6.5 Phenanthroline ferrous sulfate (ferroin) indicator solution: Dissolve 1.48 g of 1–10-(ortho) phenanthroline monohydrate, together with 0.70 g of $FeSO_4 \cdot 7H_2O$ in 100 ml of water. This indicator may be purchased already prepared.

6.6 Silver sulfate : Powdered Ag_2SO_4.

6.7 Sulfuric acid (sp. gr. 1.84) : Concentrated H_2SO_4.

7. Procedure

7.1 Pipet a 50.0 ml aliquot of sample not to exceed 800 mg/l of COD into a 500 ml, flat bottom, Erlenmeyer flask. Add $HgSO_4$ (6.4) in the ratio of 10 mg to 1 mg chloride, based upon the mg of chloride in the sample aliquot and 5 ml of sulfuric acid (6.7). Swirl until all the mercuric sulfate has dissolved. Add 25.0 ml of 0.25N $K_2Cr_2O_7$ (6.1). Carefully add 70 ml of sulfuric acid-silver sulfate solution (6.2) and gently swirl until the solution is thoroughly mixed. Glass beads should be added to the reflux mixture to prevent bumping, which can be severe and dangerous.

Caution: The reflux mixture must be thoroughly mixed before heat is applied. If this is not done, local heating occurs in the bottom of the flask, and the mixture may be blown out of the condenser.

7.1.1 If volatile organics are present in the sample, use an Allihn condenser and add the sulfuric acid-silver sulfate solution through the condenser, while cooling the flask, to reduce loss by volatilization.

7.2 Attach the flask to the condenser and reflux the mixture for two hours.

7.3 Cool, and wash down the interior of the condenser with 25 ml of distilled water. Disconnect the condenser and wash the flask and condenser joint with 25 ml of distilled water so that the total volume is 350 ml. Cool to room temperature.

7.4 Titrate with standard ferrous ammonium sulfate (6.3) using 10 drops of ferroin (6.5) indicator. (This amount must not vary from blank, sample and standardization.) The color change is sharp, going from blue-green to reddish-brown and should be taken as the end point although the blue-green color may reappear within minutes.

7.5 Run a blank, using 50 ml of distilled water in place of the sample together with all reagents and subsequent treatment.

7.6 For COD values greater than 800 mg/l, a smaller aliquot of sample should be taken; however, the volume should be readjusted to 50 ml with distilled water having a chloride concentration equal to the sample.

7.7 Chloride correction[1]: Prepare a standard curve of COD versus mg/l of chloride, using sodium chloride solutions of varying concentrations following exactly the procedure outlined. The chloride interval, as a minimum should be 4000 mg/l up to 20,000 mg/l chloride. Lesser intervals of greater concentrations must be run as per the requirements of the data, but in no case must extrapolation be used.

8. Calculation

$$\text{mg/l COD} = \frac{[(A - B)C \times 8{,}000] - 50D}{\text{ml of sample}} \times 1.2$$

where:

A = ml $Fe(NH_4)_2(SO_4)_2$ for blank;
B = ml $Fe(NH_4)_2(SO_4)_2$ for sample;
C = normality of $Fe(NH_4)_2(SO_4)_2$;
D = chloride correction from curve (step 7.7)
1.2 = compensation factor to account for the extent of chloride oxidation which is dissimilar in systems containing organic and non-organic material.

9. Precision and Accuracy

9.1 Precision and accuracy data are not available at this time.

Bibliography

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CHEMICAL OXYGEN DEMAND

Method 410.4 (Colorimetric, Automated; Manual)

STORET NO. 00340

1. Scope and Application
   - 1.1 This method covers the determination of COD in surface waters, domestic and industrial wastes.
   - 1.2 The applicable range of the automated method is 3–900 mg/1 and the range of the manual method is 20 to 900 mg/1.
2. Summary of Method
   - 2.1 Sample, blanks and standards in sealed tubes are heated in an oven or block digestor in the presence of dichromate at 150°C. After two hours, the tubes are removed from the oven or digestor, cooled and measured spectrophotometrically at 600 nm.
3. Sample Handling and Preservation
   - 3.1 Collect the samples in glass bottles if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.
   - 3.2 Samples should be preserved with sulfuric acid to a pH < 2 and maintained at 4°C until analysis.
4. Interferences
   - 4.1 Chlorides are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate is added to the digestion tubes to complex the chlorides.
5. Apparatus
   - 5.1 Drying oven or block digestor, 150°C
   - 5.2 Corning culture tubes, 16 x 100 mm or 25 x 150 mm with Teflon lined screw cap
   - 5.3 Spectrophotometer or Technicon AutoAnalyzer
   - 5.4 Muffle furnace, 500°C.
6. Reagents
   - 6.1 Digestion solution: Add 10.2 g $K_2Cr_2O_7$, 167 ml conc. H_2SO_4 and 33.3 g $HgSO_4$ to 500 ml of distilled water, cool and dilute to 1 liter.
   - 6.2 Catalyst solution: Add 22 g Ag_2SO_4 to a 4.09kg bottle of conc. H_2SO_4. Stir until dissolved.
   - 6.3 Sampler wash solution: Add 500 ml of conc H_2SO_4 to 500 ml of distilled water.
   - 6.4 Stock potassium acid phthalate: Dissolve 0.850 g in 800 ml of distilled water and dilute to 1 liter. 1 ml = 1 mg COD
     - 6.4.1 Prepare a series of standard solutions that cover the expected sample concentrations by diluting appropriate volumes of the stock standard.
7. Procedure
   - 7.1 Wash all culture tubes and screw caps with 20% H_2SO_4 before their first use to prevent contamination. Trace contamination may be removed from the tubes by igniting them in a muffle oven at 500°C for 1 hour.

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7.2 Automated

7.2.1 Add 2.5 ml of sample to the 16 x 100 mm tubes.

7.2.2 Add 1.5 ml of digestion solution (6.1) and mix.

7.2.3 Add 3.5 ml of catalyst solution (6.2) carefully down the side of the culture tube.

7.2.4 Cap tightly and shake to mix layers.

7.2.5 Process standards and blanks exactly as the samples.

7.2.6 Place in oven or block digestor at 150°C for two hours.

7.2.7 Cool, and place standards in sampler in order of decreasing concentration. Complete filling sampler tray with unknown samples.

7.2.8 Measure color intensity on AutoAnalyzer at 600 nm.

7.3 Manual

7.3.1 The following procedure may be used if a larger sample is desired or a spectrophotometer is used in place of an AutoAnalyzer.

7.3.2 Add 10 ml of sample to 25 x 150 mm culture tube.

7.3.3 Add 6 ml of digestion solution (6.1) and mix.

7.3.4 Add 14 ml of catalyst solution (6.2) down the side of culture tube.

7.3.5 Cap tightly and shake to mix layers.

7.3.6 Place in oven or block digestor at 150°C for 2 hours.

7.3.7 Cool, allow any precipitate to settle and measure intensity in spectrophotometer at 600 nm. Use only optically matched culture tubes or a single cell for spectrophotometric measurement.

8. Calculation

8.1 Prepare a standard curve by plotting peak height or percent transmittance against known concentrations of standards.

8.2 Compute concentration of samples by comparing sample response to standard curve.

9. Precision and Accuracy

9.1 Precision and accuracy data are not available at this time.

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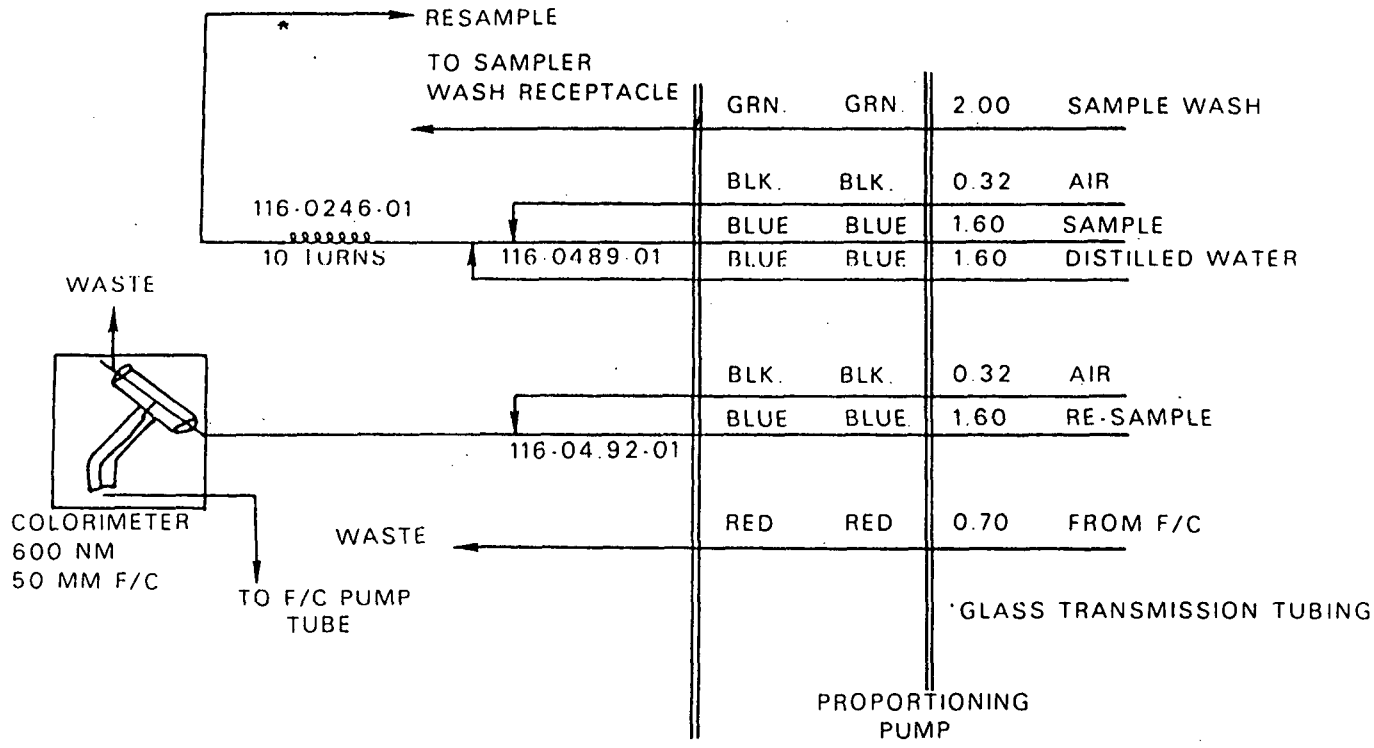

FIGURE 1 C O D MANIFOLD AA1 OR AA 11