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SUMTER COUNTY LANDFILL
QUALITY ASSURANCE PROJECT PLAN
LANDFILL COMPOST ANALYSES

QUALITY ASSURANCE PROJECT PLAN FOR A PRELIMINARY CHEMICAL ASSESSMENT OF LANDFILL COMPOST FROM THE SUMTER COUNTY LANDFILL

Prepared for

SPRINGSTEAD ENGINEERING, INC.
Leesburg, Florida

Prepared by

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Ormond Beach, Florida

October 1988

D. E. R.

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SOUTHWEST DISTRICT
TAMPA

	Approval	Date
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Project Manager (Springstead Engineering)	Paul Bradley	
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3.0 PROJECT DESCRIPTION

On March 28, 1988 Envirolab, Inc. received a request for proposal from Springstead Engineering, Inc. for the performance of a variety of chemical tests related to compost materials generated at the Sumter County Volume Reduction and Compost Facility (Figure 1). Sumter County was in the process of revising its Solid Waste Facility Operating Permit SC60-132701 to include composting. Based on comments from Florida Department of Environmental Regulation personnel, Springstead Engineering (SE) developed a list of proposed parameters to be analyzed in the compost. The results of this testing was to be reviewed by FDER in order to assess the potential environmental risk associated with the composting process.

On April 11, 1988 Envirolab replied to Springstead Engineering's RFP with a proposal of analytical services. These services included chemical and microbiological testing in Envirolab's Ormond Beach Laboratory.

On September 4, 1988 Mr. Francis Corden spoke with Mr. Ron Peoples of Waste Recovery Systems (WRS), and Mr. Paul Bradley of SE. The purpose of the discussions was to inform Envirolab that SE had selected Envirolab as the contract laboratory for the project. Envirolab was instructed to develop a site specific quality control plan to cover the sampling and analyses of materials at the Compost Facility.

At that time Envirolab with the consent of WRS arranged a meeting with the local FDER representative to thoroughly define the scope of work for the project. Mr. Kim Ford of FDER indicated that certain revisions had been suggested by FDER staff in Tallahassee pertaining to the testing needed. During the meeting a scope of work was outlined by FDER and is outlined in the following subsections.

3.1 SAMPLING AND ANALYSIS

The composting will be performed on the composting pad (figure 1) in a series of segregated rows. The end of the composing time will be determined by the drop in temperature of the compost to 20 degrees Celsius above ambient. Francine Joyal of DER - Tallahassee indicated that the sampling will follow 17-709.530 (1)(e) (compost rule) for the compost as it is ready to leave the Landfill. Please refer to Section 6 for sampling methodology.

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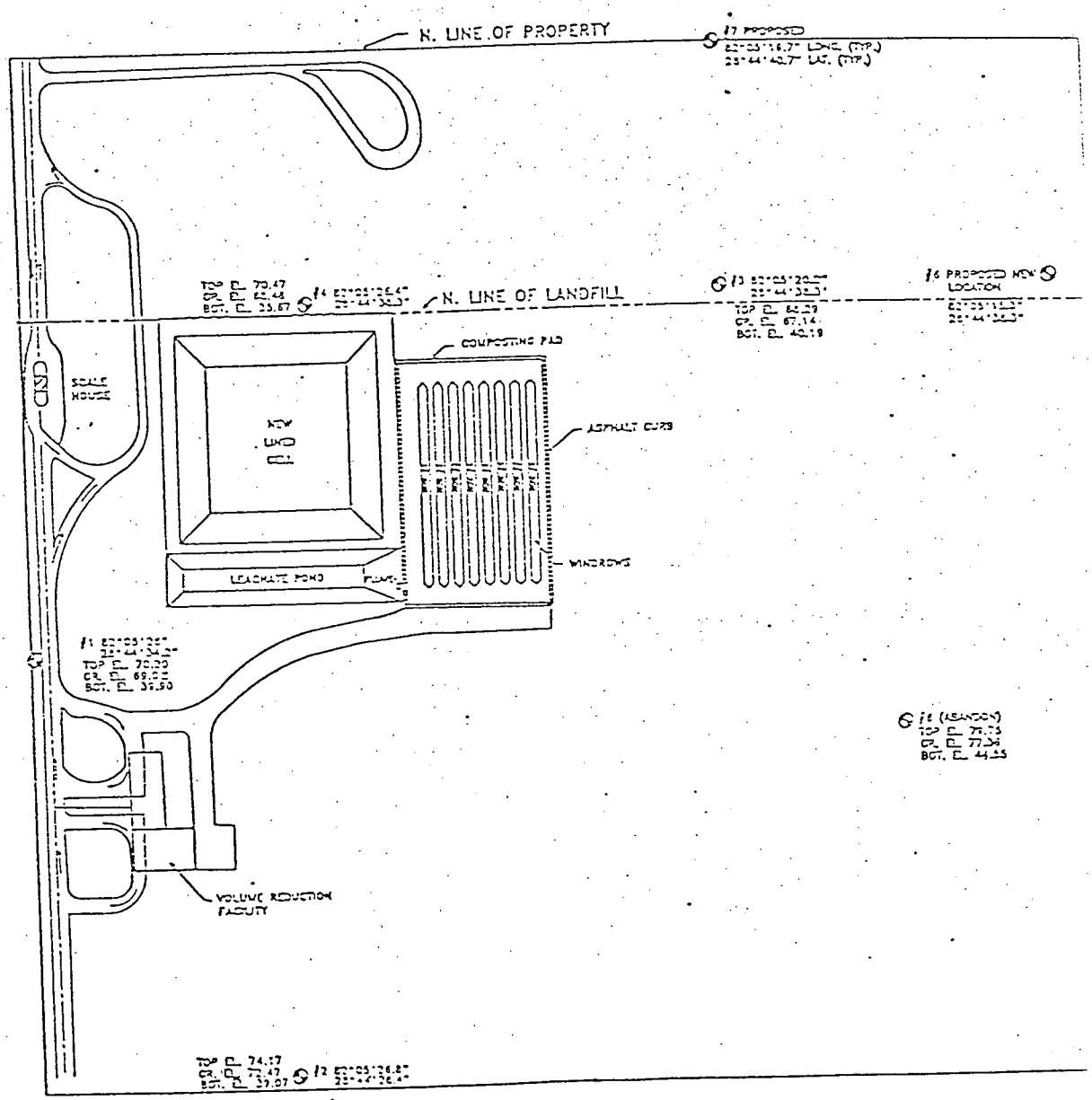
Figure 1



3.2 Data Analysis and Report

Within 45 days after collection of the samples, a report will be prepared and delivered to Springstead Engineering. Reports of spike samples will be reported as well as any excursions of parameters above the applicable regulatory maximum contaminate levels.

TABLE 3.1 PARAMETER LIST FOR COMPOST ANALYSES

Parameter	Method	Precision	Accuracy	Completeness
Moisture * (100% T.Solids%)	160.3	5.3,3.6	99.2,6.3	90
Total Nitrogen*	351+353	N/A	N/A	
Total Kjehdahl Nitr.	351.3	11.6,8.1	92.8,6.2	90
Nitrate + Nitrite	353.2	2.11,0.77	100,6.23	90
Total Phosphorous	365.2	22.1,0.2	105.3,4.04	90
Potassium	3050/7610	10.2,3.2	102,7.07	90
Cadmium	3050/7130	3.2,1.8	99.2,7.3	90
Copper	3050/7210	25.8,10.0	102,3.9	90
Lead	3050/7420	4.56,2.03	96.8,4.5	90
Nickel	3050/7520	4.8,3.2	98.1,6.8	90
Zinc	3050/7950	38.5,5.67	99.4,6.5	90
Fecal Coliform	SM 908	N/A	N/A	
pH	9045	2.4,1.7	99.8,0.46	90
Organic Matter (volatile solids)	160.4	Insufficient data for this type of matrix.		
Foreign Matter** DER 17-709	530(1)(F)	N/A	N/A	

Compost for Total Kjehdahl Nitrogen, Nitrate + Nitrite, Total Phosphorous, are prepared by blending 1 gram compost into 100 mls, de-ionized H2O using stainless steel blender. Multiple aliquots (0.1 ml to 5 mls) of mixture are used for analyses. An aliquot of the sample will be digested by using H2SO4 and Nitric Acid digestion to Sulfuric Acid fuming to bring the compost into solution for the total phosphorus analyses. This digestion method is referenced from USEPA/US Army Corp of Engineers Technical Committee on Criteria for Dredge and Fill Material " Procedures for Handling and Chemical Analysis of Sediment and Water Samples" May 1981, page 3-225, digestion method B (EPA contract EPA 4805572010). The TKN digestion is included in the TKN method. An aliquot of the 1 gm to 100 mls sample will be filtered through 0.45 micron filter and analyzed for Nitrate-Nitrite.

Compost Total Solids are prepared by ten grams of compost sample and weighed on an analytical balance to the nearest milligram. The sample is analyzed as in EPA 160.2 without the filtering.

* Total Nitrogen is the sum of TKN and NO3 + NO2.
** Foreign Matter is described in FL DER Rule 17-709 530(1)(F).

Fecal Coliform has dilution water (phosphate buffer as described in method) run as a negative control and a fecal strep negative culture control and a sample of purified culture of fecal coliform run as positive control. The positive and negative controls are recorded as just 'positive' and 'negative', due to nonavailability of Quantifiable standards. The cultures are obtained from EPA as Quality Assurance check samples and/or from the Micro Section at Halifax Hospital in Daytona Beach, FL.

The compost samples are prepared for analyses by blending 25g of sample and 250 mls of dilution water (phosphate buffer water as described in SM 908) using a stomacher device. The homogenized sample is further diluted to appropriate dilutions so that the analyses will not yield TNTC results. The sample size could be as high as one milliliter, to as low as 10 (-5 power) milliliter. All equipment used for sample preparation will be sterilized prior to use, using an autoclave.

The MPN method will be a 5 tube method using three dilutions, Envirolab will actually use five dilutions to keep the fecal coliform concentration in the range of the test. The dilutions are of the blended sample and are 10mls (1gm), 1ml (0.1g), 0.1ml (0.01gm). 0.01ml (0.001gm) and 0.001ml (0.0001gm). If the first three concentrations are in the method range they will be used, if not then the next three concentrations will be used and so on. The MPN Value from the S.M. MPN Table will be factored by 10g divided by the highest concentration (in grams) dilution used, divided by 100. This result is MPN per gram. Divide this value by the % volatile solid to yield MPN fecal coliform per gram of volatile solid.

4.0 PROJECT ORGANIZATION AND RESPONSIBILITY

A proposed organizational chart for the project is shown in Figure 4.1.

The Project Coordinator for Sumter County will be Mr. Gary Breeden. He is ultimately responsible for the activities of the subcontractor groups of Waste Recovery Systems (WRS), and Springstead Engineering (SE). Mr. Paul Bradley of SE will act as the contact between Sumter County, Waste Recovery Systems and Envirolab. Though Envirolab will be working closely with the WRS representatives including Mr. Ron Peoples he will have no direct responsibilities related to the project.

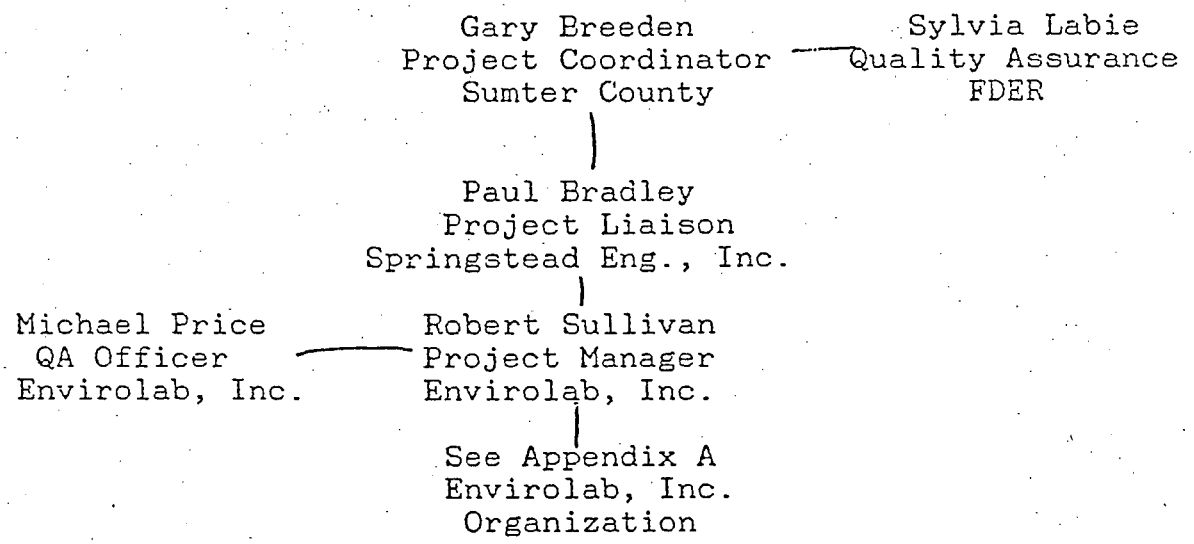
Mr. Robert Sullivan as Laboratory Director will act as Project Manager for the project. Mr. Sullivan has the authority to commit all required resources to this project as needed. Mr. Sullivan will be ultimately responsible for coordinating the administrative, field sampling, and laboratory tasks.

Mr. Michael Price, QA Officer will act as QA Officer for this project. He will be responsible for acting as the liaison with ABC as well as conducting field audits, laboratory audits, generating deficiency and corrective action reports. He will also be responsible for validation of the data and ensuring that the final reports are complete and accurate.

Mr. Michael Price, Laboratory Manager, will oversee the day to day operations of the laboratory and field staff. His responsibility will be to adequately schedule field and analytical tasks as well as supervise data collection, and reduction.

Personnel qualifications of Envirolab personnel are included in Section 17.0 and in Envirolab's Generic QA Plan.

Figure 4.1


Gary Breeden
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Michael Price
QA Officer
Envirolab, Inc.
Robert Sullivan
Project Manager
Envirolab, Inc.
See Appendix A
Envirolab, Inc.
Organization

5.0 QUALITY ASSURANCE OBJECTIVES

The quality assurance objectives for this project are based on the referenced values given in Section 3.0 of Envirolab's Generic QA Plan.

Where standards do not exist the EPA specified detection limit referenced in the method will apply (see Table 1, Section 3.0).

Instances may occur when the sample matrix will not permit the laboratory to attain the desired detection limit either due to matrix interferences or high interfering analyte concentrations requiring dilution. In these cases the best achievable detection limit will be reported along with a brief summary of the reason for the higher detection limit.

6.0 SAMPLING PROCEDURES

Envirolab, Inc. will perform all sampling procedures. Analytical data generated in the laboratory is only as good as the quality of the field sample delivered to the lab. Field collection procedures therefore, become critical in ensuring a successful and accurate environmental investigation. Specific protocols for sample collection, preservation, handling, shipping and storage must be specified, documented, and followed.

The general sampling procedures that will be followed are outlined in Section 4.0 of Envirolab's Generic QA Plan Table 1 summarizes the sample containers, and preservatives to be used. All information collected in the field will be entered in the field log. All entries will be in ink. Erasures are not allowed, and all changes must be initialed by the individual making the change. Samples will be returned to the cooler and placed on ice as soon as possible after collection.

Equipment decontamination procedures are also specified in Section 4.0, Appendix B(B3, B4) of Envirolab's QA Plan. Envirolab will provide enough sampling equipment so that no field decontamination is needed. Refer to Section 13.1 for field Contingency Plans.

Due to specific requirements of this site certain procedures in addition to those referenced will be followed for this project.

6.1 A. Compost Sampling

The sampling follows the compost rule 17.709.530 (1)(e).The composite will be generated using the following protocol:

At three points on the compost pile a core sample two feet into the pile will be taken using a stainless steel scoop.

A sample from each point will be placed in a presterilized specimen container for the fecal coliform. The fecal coliform sample will not be composited.

All core samples will be combined in a large glass mixing bowl and stirred gently to homogenize.

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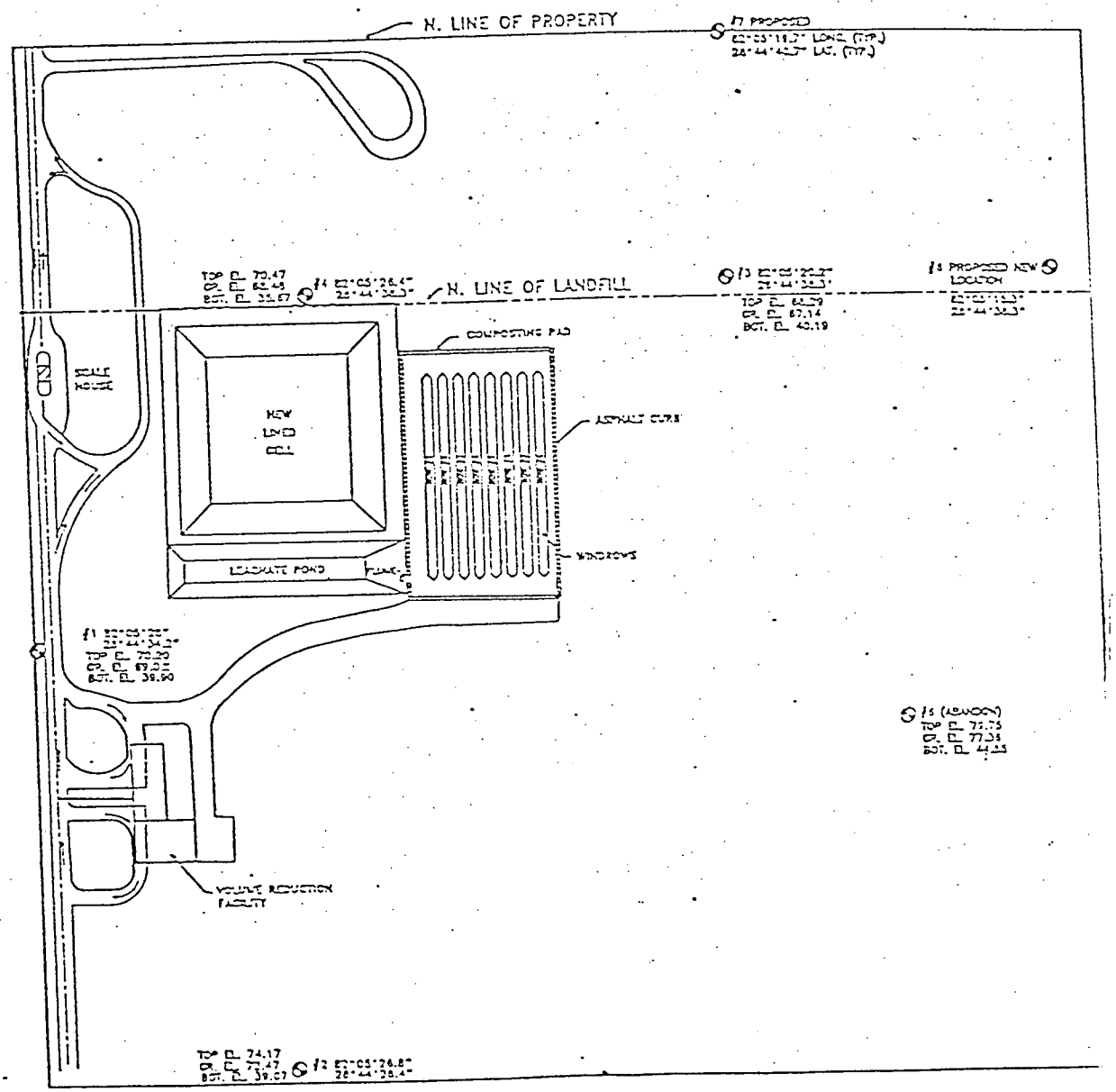
Figure 6.1



Therefore at the end of the sampling, the team should have one wide mouth amber jar, and 12 discrete fecal coliform samples in presterilized containers. All fecal coliform samples will be returned to the Laboratory as discrete samples.

B. Raw Material Sampling

The sampling of the incoming garbage to composted will be as follows:

Four grab samples of about 200 gms each will be collected from the shredder per day for 5 days. The samples will be cooled to 4°C. The samples will be placed in quart Amber Jars with Teflon Lined Caps. Envirolab will blend the raw material to obtain an homogeneous sample. The raw material will be blended using a waring blender. The attempt is to make the raw material sample as uniform as possible and as such all twenty grab samples will be blended, combined and reblended, so that a 50 gm sample will used for the Volatile Solid Analyses that is representative of the whole composite sample. This sample is needed to calculate the reduction in organic matter.

Table 4 Sample Containers, Preservatives, and Holding Times

Matrix	Parameter Group	Container Type	Preservative	Holding Time
Compost	All Metals	1 liter Amber Glass,Teflon Lid	Ice	6 months
	Fecal Coliform	pre-sterile specimen container 125 ml size	Ice	6 hours**
	Moisture,Organic Matter	1 liter amber glass Teflon lined lid	Ice	7 days
	Nitrogen(TKN,NO3, NO2) Total Phosphorus	1 liter amber glass Teflon lined lid	Ice	48 hours

** There are no published holding times for Fecal Coliform in Compost. Sample analyses will be run within 6 hours.

7.0 SAMPLE CUSTODY

Documentation of sample custody is a critical aspect of any project assessing the potential for environmental contamination. It is critical that possession of the samples be traceable from the time of collection, until the time the analysis of the sample is complete. For this reason, Envirolab will collect the samples. Envirolab will maintain accurate and complete chain of custody records.

7.1 Field Logs

During field activities the sampling crew leader will assure that the sampling log book is completed in its entirety. Entries into the field log include, but will not be limited to:

A sketch of the sampling site
A description of each site to be sampled
All equipment used to collect the sample
All sample bottles collected at the site
The date and time of sample collection
Any preparation procedures required
The weather conditions at the time of sampling
All field measurements taken at the site
The results of calibration checks of field instruments
Whether any QC samples were taken at the site
Any problems, difficulties or unusual situations encountered
Name and signature of the sampling team members

The field log becomes a part of the permanent record of the sampling event.

7.2 Sample Labels

Sample containers will be labeled at the time of collection with a minimum of the following information:

Project Number
Sampling Date/Time
Sample Site Identification
Parameters of Interest
Preservatives
Initials of the Collector

7.3 Chain of Custody Form

The final task to be completed in the field is the completion of the sample chain of custody form. An example of the form is included in Appendix A.

All samples will remain in the custody of the sampling team from the time of collection until they are delivered to the laboratory and logged in by the sample custodian. At that time the custodian will sign off on the chain of custody form. The original and all copies will remain with the master log sheet and become part of the permanent record.

7.4 Internal Laboratory Chain of Custody

Internal chain of custody procedures are described in Envirolab's Generic QA Plan, Section 5.0 pages one through four. Briefly, they include the use of a master log. This form identifies the sample group and the parameters as well a serves as a hand written record of the analysts final results. While in the possession of the laboratory the samples are kept in a locked walk in refrigerator until needed by the analyst. At the end of each day all samples are returned.

The metal samples are locked in a storage cabinet within the metals section. The sample containers are removed only long enough to measure the required amount for the digestions. The samples are then returned to the cabinet. The sample storage cabinet and digestates are locked in the metals section room. Unauthorized access in not allowed into the room. The lower two shelves of the locked walk-in refrigerator are dedicated to the organic section. The organic section includes an extraction room and the instrument room. Both sets of doors are keyed alike and only that sections members and management have the keys. The organic samples are removed from the walkin refrigerator long enough to measure the required amount for sample prep. After sample prep, the samples are hand delivered to the organic analyst. The samples can be tracked by looking at the metals prep log book, the organics prep log book and the analyses log books. This is the manner that Envirolab tracks who has been involved with the samples.

8.0 CALIBRATION FREQUENCY

Field equipment is calibrated according to the manufacturers directions. Calibrations are performed for each sample event.

Laboratory equipment is calibrated according to the manufacturers directions and is consistent with the approved analytical method utilized. In general, laboratory instruments are calibrated once each day with the calibration curve, and checked periodically throughout the analytical run.

Please refer to Envirolab's Generic QAP, Section 6, Tables 6.1 for field equipment and Table 6.2 for laboratory equipment.

9.0 ANALYTICAL PROCEDURES

All procedures are referenced in Section 3.1.

10.0 DATA REDUCTION, VALIDATION AND REPORTING

Please refer to Envirolab's Generic QAP Section 8, pages 1 through 3.

10.1 Data Reduction

The process of converting raw data to sample concentration in the appropriate units is accomplished by the analyst responsible for the work. The specific calculation involved are described in Envirolab's Generic QA Plan.

GC and GC/MASS SPEC procedures incorporate automatic quantitation routines using known internal standards and surrogate standards. All parameters within the individual EPA methods (ie 625) are compared to known internal standards. This comparison yields an analyte relative response factor. This analyte relative response factor is compared to a standard relative repsonse factor of a standard run during that day. This comparison multiplied by the standard concentration yield the sample analyte concentration. Surrogate standards are analytes of known concentrations that are added to the samples before the extractions begin.The surrogate standards are used to calculate the extraction efficiency, (recovery) of the procedure. Internal standards are not extracted but are added to the extracts before the instrumental analyses begins. G.C. Automatic Quantitation is by Nelson Analytical while GC/MS is Finnigans Auto Quant routine. Envirolab's data reduction involves counting colonies and correcting for dilutions used, or counting MPN tubes with gas formation and using the MPH Tables and correcting for the dilutions used.

10.2 Data Validation

The data generated by the analyst is reviewed at two different stages of the data flow. The analyst is responsible for ascertaining the correctness of all calculations performed in his/her area. Prior to submitting the data to report generation, the data and associated QC information is checked by the QA Manager to verify consistency and compliance with control ranges.Results are not acceptable if QC (spiked and duplicated samples) are outside ± 3 S.D. from the mean. All analyses include controls and/or spikes and/or surrogate and internal standards that must fall within ± 3 S.D. for the analyses to be deemed valid.

10.3 Data Reporting

Data reporting is accomplished by using a key punch operator to enter data from test sheets completed by the analysts into a

commercial lab data management software package. The report generated is checked against the master log-in sheet for accuracy and any discrepancies are resolved by the QA Manager or Lab Manager. All Final reports will be generated by Envirolab and sent to the project manager. The QA Reports will also be generated by Envirolab.

11.0 INTERNAL QUALITY CONTROLS CHECKS

Please refer to Envirolab's generic Quality Assurance Plan- Section 9, Revision 1, Feb 2, 1988, pages 1 through 4.

11.1 FIELD ACTIVITIES

Measures to ensure the quality of the field samples collected have been described throughout the document. In summary these measures include:

- Pre and Post Sampling Organizational Meetings
- Field Equipment Checks
- Preparation of Trip Blanks
- Sampling Instructions and the use of Approved Methods
- Collection of Field Blanks at a rate of at least 5%, or at least once per sampling trip.
- Collection of Replicate samples
- Collection of equipment blanks at a rate of 5% of the equipment that is decontaminated in the field.

11.2 LABORATORY ACTIVITIES

To ensure reliable, accurate, and reproducible data, the laboratory will utilize strict internal quality control checks as follows for the chemical analyses:

- Batch Duplicates
- Batch Spikes
- Performance Samples
- Calibration and Method Check Samples, (From EPA or NBS)
- Blanks
- Reagent Checks

Atomic Absorption, and wet chemistry Quality Control Checks are in Envirolab's generic QAP Section 9, 6.2.1. through 6.2.7. All standards are purchased from Supelco, CMS, Fisher, ChemService and are analytical grade or better. Quality control samples are from EPA Quality Assurance. Documentation of Q.C. Checks including standards and controls (spikes, duplicates, EPA control samples) are incorporated into QC charts and tables, and also in each method workbook. The QA officer reviews all documentation and if acceptable, initials the workbook page, or the analyses must be redone.

The Quality Assurance for the fecal MPN analyses are as follows:

Chemical analyses annually for Laboratory pure water for metals.
Bactericidal quality annually for Laboratory pure water.
Daily checks of Laboratory pure water for pH and conductivity.
Inclusion of negative controls with each day of analysis.
Inclusion of positive controls.
Inclusion of negative culture controls.
Annual analyses of washing procedures for inhibitory residue.

12.0 PERFORMANCE AND SYSTEM AUDITS

12.1 FIELD SYSTEM AUDITS

The QA office and the project manager may make routine visits to project sites to evaluate the performance of field personnel and general field operations and progress. They will observe the performance of the field operations personnel during each kind of activity. A formal systems audit of field operations personnel by the project QA officer will be performed,once every six months and a field audit report of the sampling team will be maintained on file by Envirolab.The audit will follow the Field Audit form obtained from DER Quality Assurance Section. The audit closest to this project date will be submitted to the Project Manager.

12.2 LABORATORY SYSTEMS AUDIT

A laboratory systems audit is routinely conducted monthly by Envirolab. These audits assure that systems and operational capability is maintained and test methodology and quality control measures are being followed as specified in the laboratory written standard operating procedures (SOP) and generic Quality Assurance Plans (QAP). The audit is initiated by the QA officer by sending blind samples through the Laboratory that only the QA officer knows the true value. Compliance to extraction and analyses times, plus qualitative and quantitative criteria are as certained with these samples.

12.3 PERFORMANCE EVALUATION AUDITS

A performance evaluation (PE) audit is an audit performed to evaluate a laboratory's ability to obtain an accurate and precise result in the analysis of known check samples by a specific analytical method. PE audits may include a review of all raw data developed by the laboratory and not reported and the submission by the Project Manager of blank spiked check samples for the analysis of the parameters in question. These check samples for the analysis of the parameters in question. These check samples may be submitted disguised as field samples.In which case, the laboratory will not know the purpose of the samples or the samples may be obvious (known) check samples (EPA or NBS traceable).

PE Audits also will be conducted by reviewing the laboratory's results from "round-robin" certification testing, and/or EPA performance evaluation samples, which occurs four times a year. An additional component of PE audits includes the review and evaluation of raw data generated from the analysis of PE samples

and actual field samples. that may be in question.

More detail concerning laboratory initiated audits can be found in Section 10.0 of Envirolab's Generic QA Plan,pages 1 through 4.

13.0 PREVENTIVE MAINTENANCE

Please refer to Envirolab's Generic QAP Section 2, pages 1 - 3.

13.1 FIELD EQUIPMENT

All field instruments are checked in the laboratory prior to each use. In the case of instrument failure, routine maintenance can be accomplished in the lab. In the event routine procedures do not restore instrument performance, the device will be repaired by qualified service personnel. Extra equipment will be bought by Envirolab as spares. In the event that the field meters are nonoperable, the sampler will call Envirolab's Laboratory Manager to inform the manager of the problem. The manager will decide if the sampling is to be rescheduled, or to continue. If the sampling continues, all deviations will be documented and submitted with the sample results.

Periodic maintenance of instrumentation is carried out by the individual analysts. This may include replacement of seals, frits, septa, O rings and other disposable parts. Major repairs are carried out by authorized service personnel. A service contract is maintained on all GC, GC/MS and AA equipment currently in use.

14.0 ASSESSMENT OF DATA PRECISION, ACCURACY, AND COMPLETENESS

Data completeness will be expressed both as a percentage of total tests conducted that are deemed valid and as the percentage of the total tests required in the scope of work that are deemed valid.

Methods for assessing data precision, accuracy, and completeness by Envirolab are described in Section 12.0, pages 1 through 6 of Envirolab's Generic QA Plan.

Quantifiable Accuracy is unable to be determined for the fecal coliform due to the nonavailability of Quantifiable Standards.

Accuracy is qualitative for the fecal coliform by analyzing with two different types of broth or media. Precision for the fecal coliform is accomplished by reading duplicate samples and comparing the numeric results.

15.0 CORRECTIVE ACTION

During the course of any investigation, the field personnel are responsible for seeing that field instruments are functioning properly and that work progresses satisfactorily. The field personnel also are responsible for ensuring performance of routine preventive maintenance and quality control procedures, thereby ensuring collection of valid field data.

If a problem is detected by the field personnel, the project manager shall be notified immediately, at which time the problem will be investigated further and corrective action will begin. Similarly, if a problem is identified during a routine audit by the project Quality Assurance Officer or the FDER QA Officer, an immediate investigation will be undertaken and corrective action deemed necessary will be taken as early as possible.

Laboratory quality control is the responsibility of both the laboratory analysts and the QA Manager. The analysts are responsible for monitoring the quality of reagents, solvents, and gases while assuring that the appropriate sample handling procedures, preservatives and containers are used based on the data required. Analysts are also responsible for the status of instruments under their care.

The QA Manager is responsible for monitoring compliance with proper chain of custody and log-in procedures as well as reviewing the QA data for compliance with accepted QC ranges. The QA Manager is also responsible for reviewing the final data package for completeness and consistency of results.

Any deviations or problems encountered with instrumentation are to be reported immediately to the Laboratory Manager. He shall recommend corrective action, initiate instrument diagnosis and the necessity of repair. Any other problems with sample analysis and/or the accuracy and completeness of a project report will also be directed to the Laboratory Managers attention. The necessity of split samples or a second analysis shall be the Laboratory Managers decision.

16.0 QUALITY ASSURANCE REPORT TO MANAGEMENT

16.1 INTERNAL REPORTS

The Quality Assurance Officer will provide status reports to the Project Manager. The reports may address the following:

- quality assurance activities and quality of collected data
- equipment calibration and preventative maintenance activities
- results of data precision and accuracy calculations
- evaluation of data completeness
- QA problems and recommended and/or implemented corrective actions
- QA audit findings

16.2 REPORT TO FDER

In accordance with DER-QA-001/85, Envirolab will provide quality assurance reports to the appropriate FDER district office on a quarterly basis if required for the length of the project. These reports will consist of applicable portions of Envirolab's internal QA reports. A final quality assurance Summary Report will be submitted at the conclusion of the project if necessary.

17.0 PERSONNEL QUALIFICATIONS

Resumes of Envirolab personnel are found in Envirolab's generic QAP Section 15.

5.1.6 Digital printer for AAII (Optional).

6. Reagents

MCB Reagents

6.1 Granulated cadmium: 40–60 mesh (E M Laboratories, Inc., 500 Exec. Blvd., Elmsford, NY 10523, Cat. 2001 Cadmium, Coarse Powder).

6.2 Copper-cadmium: The cadmium granules (new or used) are cleaned with dilute HCl (6.7) and copperized with 2% solution of copper sulfate (6.8) in the following manner:

6.2.1 Wash the cadmium with HCl (6.7) and rinse with distilled water. The color of the cadmium so treated should be silver.

6.2.2 Swirl 10 g cadmium in 100 ml portions of 2% solution of copper sulfate (6.8) for five minutes or until blue color partially fades, decant and repeat with fresh copper sulfate until a brown colloidal precipitate forms.

6.2.3 Wash the cadmium-copper with distilled water (at least 10 times) to remove all the precipitated copper. The color of the cadmium so treated should be black.

6.3 Preparation of reduction column AAI: The reduction column is an 8 by 50 mm glass tube with the ends reduced in diameter to permit insertion into the system. Copper-cadmium granules (6.2) are placed in the column between glass wool plugs. The packed reduction column is placed in an up-flow 20° incline to minimize channeling. See Figure 1.

6.4 Preparation of reduction column AAII: The reduction column is a U-shaped, 35 cm length, 2 mm I.D. glass tube (Note 1). Fill the reduction column with distilled water to prevent entrapment of air bubbles during the filling operations. Transfer the copper-cadmium granules (6.2) to the reduction column and place a glass wool plug in each end. To prevent entrapment of air bubbles in the reduction column be sure that all pump tubes are filled with reagents before putting the column into the analytical system.

NOTE 1: A 0.081 I.D. pump tube (purple) can be used in place of the 2 mm glass tube.

6.5 Distilled water: Because of possible contamination, this should be prepared by passage 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 manufacturer's instructions.

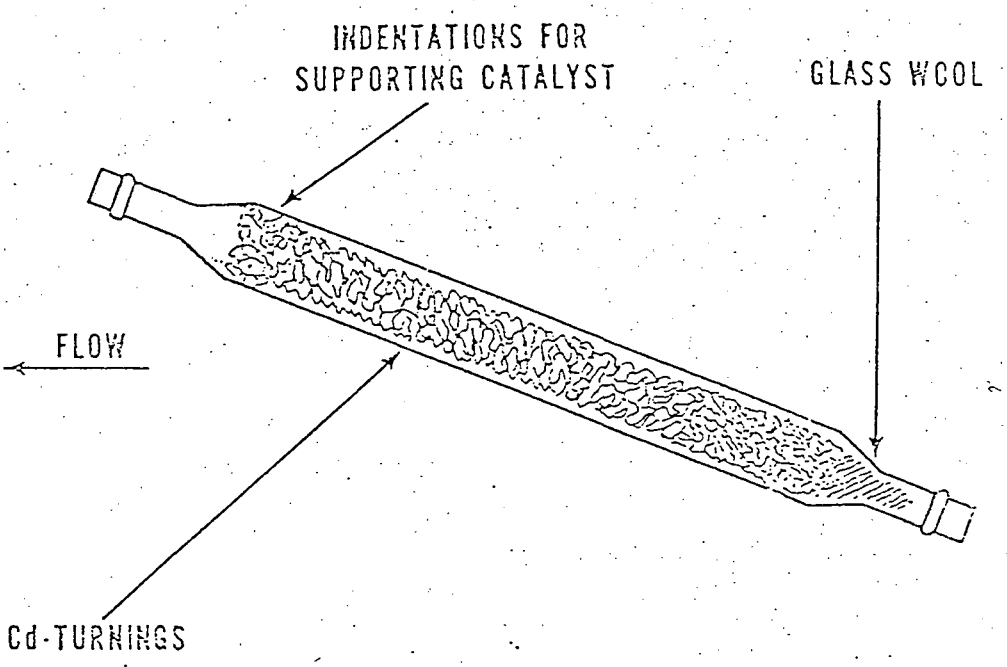
6.6 Color reagent: To approximately 800 ml of distilled water, add, while stirring, 100 ml conc. phosphoric acid, 40 g sulfanilamide, and 2 g N-1-naphthylethylenediamine dihydrochloride. Stir until dissolved and dilute to 1 liter. Store in brown bottle and keep in the dark when not in use. This solution is stable for several months.

6.7 Dilute hydrochloric acid, 6N: Dilute 50 ml of conc. HCl to 100 ml with distilled water.

6.8 Copper sulfate solution, 2%: Dissolve 20 g of $CuSO_4 \cdot 5H_2O$ in 500 ml of distilled water and dilute to 1 liter.

6.9 Wash solution: Use distilled water for unpreserved samples. For samples preserved with H_2SO_4, use 2 ml H_2SO_4 per liter of wash water.

6.10 Ammonium chloride–EDTA solution: Dissolve 85 g of reagent grade ammonium chloride and 0.1 g of disodium ethylenediamine tetracetate in 900 ml of distilled water. Adjust the pH to 8.5 with conc. ammonium hydroxide and dilute to 1 liter. Add 1/2 ml Brij-35 (available from Technicon Corporation).



TILT COLUMN TO 20° POSTION.

FIGURE 1. COPPER CADMIUM REDUCTION COLUMN
(1 1/2 ACTUAL SIZE)

6.11. Stock nitrate solution: Dissolve 7.218 g KNO_3 and dilute to 1 liter in a volumetric flask with distilled water. Preserve with 2 ml of chloroform per liter. Solution is stable for 6 months. 1 ml = 1.0 mg NO_3–N.

6.12 Stock nitrite solution: Dissolve 6.072 g KNO_2 in 500 ml of distilled water and dilute to 1 liter in a volumetric flask. Preserve with 2 ml of chloroform and keep under refrigeration. 1.0 ml = 1.0 mg NO_2–N.

6.13 Standard nitrate solution: Dilute 10.0 ml of stock nitrate solution (6.11) to 1000 ml. 1.0 ml = 0.01 mg NO_3-N. Preserve with 2 ml of chloroform per liter. Solution is stable for 6 months.

6.14 Standard nitrite solution: Dilute 10.0 ml of stock nitrite (6.12) solution to 1000 ml. 1.0 ml = 0.01 mg NO_2-N. Solution is unstable; prepare as required.

6.15 Using standard nitrate solution (6.13), prepare the following standards in 100.0 ml volumetric flasks. At least one nitrite standard should be compared to a nitrate standard at the same concentration to verify the efficiency of the reduction column.

Conc., mgNO_2–N or NO_3-N/l	ml Standard Solution/100 ml
0.0	0
0.05	0.5
0.10	1.0
0.20	2.0
0.50	5.0
1.00	10.0
2.00	20.0
4.00	40.0
6.00	60.0

NOTE 2: When the samples to be analyzed are saline waters, Substitute Ocean Water (SOW) should be used for preparing the standards; otherwise, distilled water is used. A tabulation of SOW composition follows:

NaCl – 24.53 g/l	$MgCl_2$ – 5.20 g/l	Na_2SO_4 – 4.09 g/l
$CaCl_2$ – 1.16 g/l	KCl – 0.70 g/l	$NaHCO_3$ – 0.20 g/l
KBr – 0.10 g/l	H_3BO_3 – 0.03 g/l	$SrCl_2$ – 0.03 g/l
NaF – 0.003 g/l		

7. Procedure

7.1 If the pH of the sample is below 5 or above 9, adjust to between 5 and 9 with either conc. HCl or conc. NH_4OH.

7.2 Set up the manifold as shown in Figure 2 (AAI) or Figure 3 (AAII). Note that reductant column should be in 20° incline position (AAI). Care should be taken not to introduce air into reduction column on the AAII.

7.3 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.

NOTE 3: Condition column by running 1 mg/l standard for 10 minutes if a new reduction column is being used. Subsequently wash the column with reagents for 20 minutes.

7.4 Place appropriate nitrate and/or nitrite standards in sampler in order of decreasing concentration of nitrogen. Complete loading of sampler tray with unknown samples.

7.5 For the AAI system, sample at a rate of 30/hr, 1:1. For the AAII, use a 40/hr, 4:1 cam and a common wash.

7.6 Switch sample line to sampler and start analysis.

8. Calculations

8.1 Prepare appropriate standard curve or curves derived from processing NO_2 and/or NO_3 standards through manifold. Compute concentration of samples by comparing sample peak heights with standard curve.

9. Precision and Accuracy

9.1 Three laboratories participating in an EPA Method Study, analyzed four natural water samples containing exact increments of inorganic nitrate, with the following results:

Increment as Nitrate Nitrogen mg N/liter	Precision as Standard Deviation mg N/liter	c/o RSD	Accuracy as Bias, %	Accuracy as Bias, mg N/liter
0.29	0.012	4.13	+ 5.75	+0.017
0.35	0.092	26.2	+18.10	+0.063
2.31	0.318	13.8	+ 4.47	+0.103
2.48	0.176	7.10	− 2.69	−0.067

Avg 6.41

$\overline{RSD}$ = 12.8 %

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3. Annual Book of ASTM Standards, Part 31, "Water", Standard D1254, p 366 (1976).
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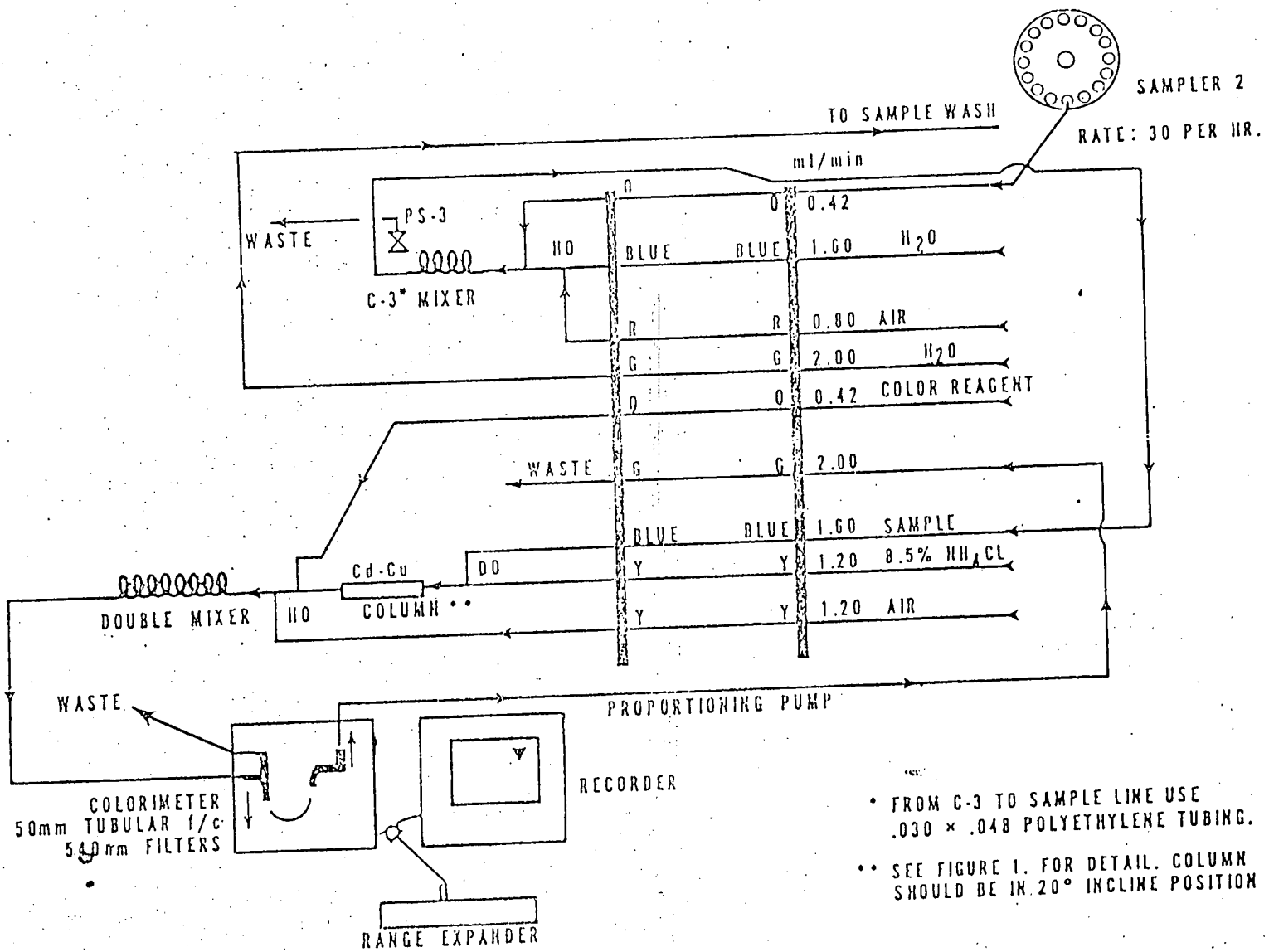

FIGURE 2. NITRATE - NITRITE MANIFOLD AA-I

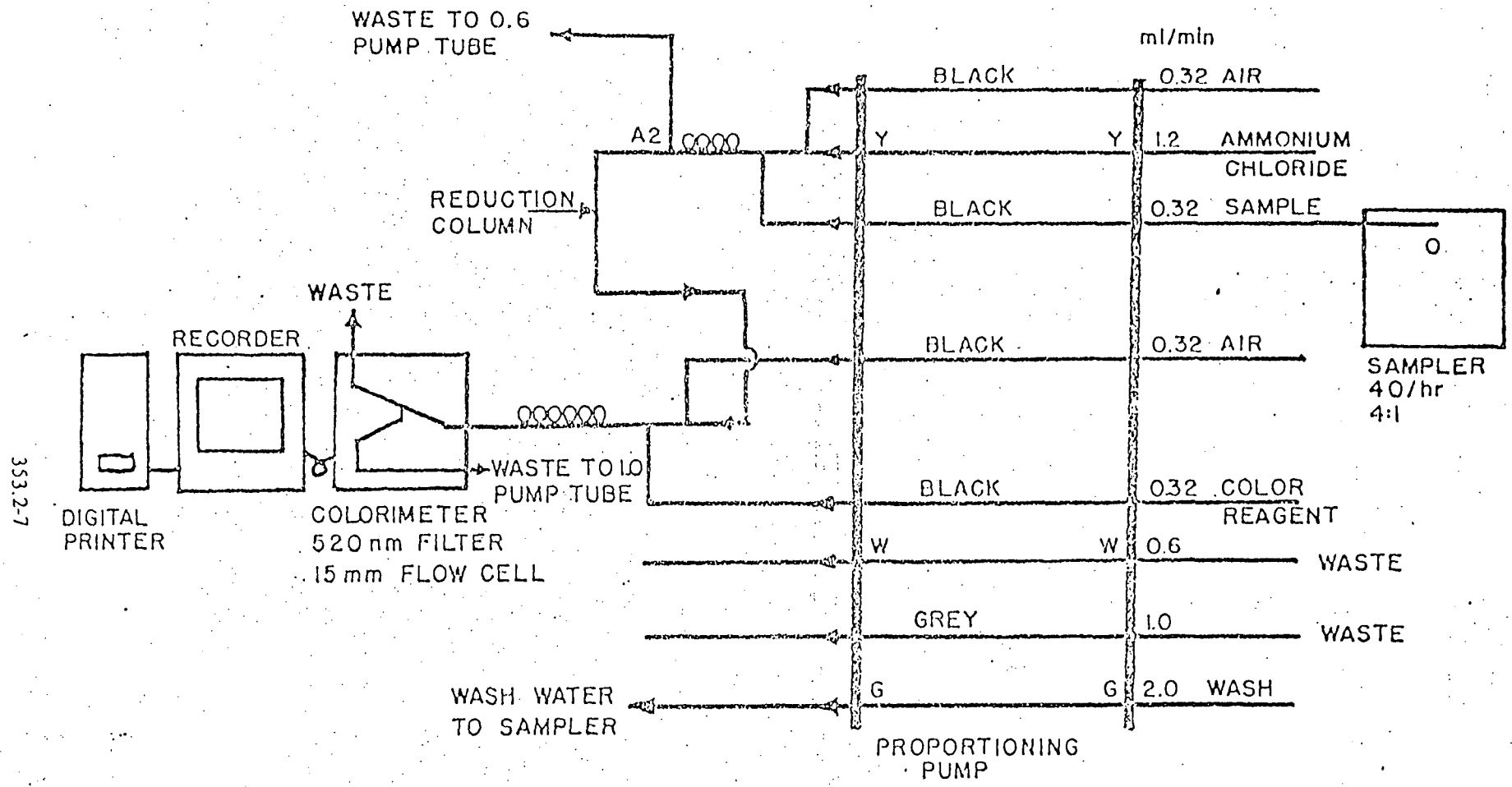

FIGURE 3 NITRATE-NITRITE MANIFOLD AA II

METHOD 8240

GAS CHROMATOGRAPHY/MASS SPECTROMETRY FOR VOLATILE ORGANICS

1.0 SCOPE AND APPLICATION

1.1 Method 8240 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds may be determined by this method:

Analyte	CAS No.[b]	Appropriate Technique	
		Purge-and-Trap	Direct Injection
Acetone	67-64-1	pp	a
Acetonitrile	75-05-8	pp	a
Acrolein	107-02-8	pp	a
Acrylonitrile	107-13-1	pp	a
Allyl alcohol	107-18-6	pp	a
Allyl chloride	107-05-1	a	a
Benzene	71-43-2	a	a
Benzyl chloride	100-44-7	pp	a
Bromoacetone	598-31-2	pp	a
Bromochloromethane (I.S.)	74-97-5	a	a
Bromodichloromethane	75-27-4	a	a
4-Bromofluorobenzene (surr.)	460-00-4	a	a
Bromoform	75-25-2	a	a
Bromomethane	74-83-9	a	a
2-Butanone	78-93-3	pp	a
Carbon disulfide	75-15-0	pp	a
Carbon tetrachloride	56-23-5	a	a
Chlorobenzene	108-90-7	a	a
Chlorobenzene-d_5 (I.S.)	108-90-7	a	a
Chlorodibromomethane	124-48-1	a	a
Chloroethane	75-00-3	a	a
2-Chloroethanol	107-07-3	pp	a
2-Chloroethyl vinyl ether	110-75-8	a	a
Chloroform	67-66-3	a	a
Chloromethane	74-87-3	a	a
Chloroprene	126-99-8	a	pc
3-Chloropropionitrile	542-76-7	ND	pc
1,2-Dibromo-3-chloropropane	96-12-8	pp	a
1,2-Dibromoethane	106-93-4	a	a
Dibromomethane	74-95-3	a	a
1,4-Dichloro-2-butene	764-41-0	pp	a
Dichlorodifluoromethane	75-71-8	a	a
1,1-Dichloroethane	75-34-3	a	a

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CHAIN OF CUSTODY RECORD

PROJ. NO.	PROJECT NAME					NO. OF CON-TAINERS							REMARKS
SAMPLERS: (Signature)													
STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION								

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)

Relinquished by: (Signature)	Date/Time	Received for Laboratory by: (Signature)	Date/Time	Remarks