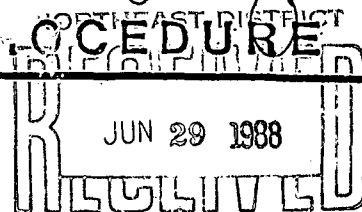


## STANDARD LABORATORY PROCEDURE



DER-JACKSONVILLE

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS  
AND CHARACTERIZATION OF HAZARDOUS WASTE SAMPLES

The following description outlines the typical treatment of hazardous waste samples as generated by the Institutional Waste program. This procedure describes steps taken during the initial generator approval through sample generation and final sample analysis and characterization. This procedure makes no attempt to describe the actual steps taken to process the waste solvent.

As written in Section D-2, Part 1 of the Temporary Operation Permit #HT01-136, Quadrex HPS Inc. is allowed to store the following waste types: D001, F001, F002, F003, and F005. The State of Florida DER has also approved a specific list contained on Attachment A in Permit/Certification No. HT01-136540. This list contains the "F" series waste and a number of characteristic D001 waste solvents and "listed" hazardous waste solvents.

QHPS may accept RCRA "listed" hazardous waste (40 CFR 261.11, 261.20) if, a) the waste is listed for its ignitability, and b) if the chemical waste has been approved by the Florida DER prior to receipt by QHPS. Upon DER approval, the operating permit will be amended and the waste will be added to Attachment A.

Due to the specificity of the permit, QHPS will not be able to store or treat hazardous waste not specifically listed on Attachment A unless the waste is a D001 characteristic waste.

If a generator wishes to dispose of trade name products, he/she must submit MSDSs listing the constituents with physical and chemical properties of the product. If the product's constituents are listed on Attachment A or the waste is D001 in nature, the material may be stored by QHPS. This criterion also applies for non-labeled waste and generic names like "oil" and "dyes". If the contents of the waste is not known, it is the generators responsibility to have laboratory analysis performed and provide QHPS with documentation.

DOCKET # 31

# STANDARD LABORATORY PROCEDURE

## Waste Approval

Waste approval is accomplished as follows:

Prior to receipt of any waste, the generator (or his waste broker) must submit a complete list for approval. If the waste type is listed on QHPS's Attachment A, the waste is given a pre-approval control number. Upon receipt of a completed waste profile sheet, the waste stream is assigned an "approval to ship" number.

To obtain an Approval To Ship Number, a waste profile sheet must be submitted. This sheet identifies the generator (EPA ID #) and his waste stream. If the waste was previously approved, that pre-approval number will convert to a "Approval To Ship Number". Additional waste added to the profile after pre-approval will be handled on a case by case basis.

The generator may by-pass the pre-approval stage by submitting the waste profile sheet directly. In this case, the waste will be reviewed (as above). If the complete profile can be accepted, an Approval To Ship control number will be assigned. If a particular waste "fails" approval, the item will be marked and the profile sheet will be returned to the generator.

## Waste Sampling

A waste stream sample may be obtained prior to bulking the fluid and/or after the solvents have been transferred to the bulk tank. Periodically samples will be removed and characterized as received from the generators waste. This will serve as a quality assurance measure. The generators waste will be verified in this manner at least quarterly.

A bulk tank sample will be characterized prior to shipping the fluid off site for disposal.

The "Sample Collection/Request For Analysis/Lab Data-Form" must be completed each time a sample is collected. This form will serve to identify the samples and maintain the traceability of the waste. Pre-numbered identification labels will be used to identify each sample.

# STANDARD LABORATORY PROCEDURE

## Analysis and Characterization

Prior to analysis, all sample identification is verified and logged into the system. If the sample is from a specific waste stream, that identification is verified against the waste profile and approval forms generated prior to receiving the waste. Once the sample is sufficiently logged into the system, the waste is assigned for analysis. The following test are performed to characterize the waste:

- Specific Gravity - 4000-005
- Flash Point - 4000-002
- Moisture Content - 4000-003
- pH - 4000-004
- Chloride Content - 4000-006
- Gas Chromatography - 4000-001

See specific SOPs for details regarding each method.

If the waste sample was generated from a specific waste stream as a QA check, the results of analysis will be compared to the waste profile sheet. If there are discrepancies between the results of analysis and the generators profile sheet, the generator will be required to submit an accurate profile sheet. If after analysis the waste is discovered not to match that on the profile sheet, QHPS reserves the right to return the waste to the generator.

A sample of the bulk fluid is taken each time the fluid is shipped off site for disposal. The results of analysis are compared to previous waste samples and used to maintain QHPS's own waste profile sheet. An estimated mass balance is maintained for each compound managed by QHPS.

Following is a flow diagram depicting that described above. Following the flow diagram is a copy of each Standard Laboratory Procedure currently being used to characterize the waste.

DRAFT

PERMITTEE:  
Quadrex HPS Inc.  
Post Office Box 4100  
Gainesville, Florida 32606

I.D. Number: FLD 980 711 071  
Permit/Certification No. HT 01-136540  
Date of Issue: 8  
Expiration Date: 8

ATTACHMENT A

<u>Name</u>	<u>Hazardous Waste Code Number</u>
1,1,1-Trichloroethane	F001/F002/U226
1,1,2-Trichloroethane	F002/U227
1,1,2-Trichlorotrifluoroethane	F002/U227
1,2-Dichlorobenzene	F002/U070
1,4-Dioxane	U108/D001
2-Ethoxyethanol (ethylene glycol monoethyl ether)	F005/U359
2-Nitropropane	F005/U171
Acetone	F003/U002
Benzene	F003/U019
Carbon Disulfide	F005/P022
Carbon Tetrachloride	F001/U211
Chlorobenzene	F002/U037
Cyclohexane	U056
Cyclohexanone	F003/U057
Ethanol	D001
Ethyl Acetate	F003/U112
Ethyl Benzene	F003
Ethyl Ether	F003/U117
Hexane	D001
Isoamyl Alcohol	D001
Isobutyl Alcohol	F005/U140
Isooctane/2,2,4-Trimethylpentane	D001
Methanol	F003/U154
Methyl Ethyl Ketone	F005/U159
Methyl Isobutyl Ketone	F003/U161
Methyl Pyrrole	D001
Methylene Chloride	F002/U080
Naphthalene	U165
Nitrobenzene	F004/U169
Pyridine	F005/P075/U196
Tetrachloroethylene	F001/F002/U210
Toluene	F005/U220
Trichloroethylene	F001/F002/U228
Trichlorofluoromethane	F002/U121
Trichloromethane	U044/D001
m-Cresol	F004/U052
m-Xylene	F003/U239
n-Butyl Alcohol	F003/U031
o-Cresol	F004/U052
o-Xylene	F003/U239
p-Cresol	F004/U052
p-Xylene	F003/U239

GENERATOR
Waste

QUADREX HPS INC. INSTITUTIONAL WASTE  
Waste Stream Information and Management

GENERATOR
Waste Characterization And Profile

WASTE DISPOSAL
Generator / Broker
Contact QHPS Seeking "Approval For Disposal"
Include Anticipated Waste Stream Types and Quantity

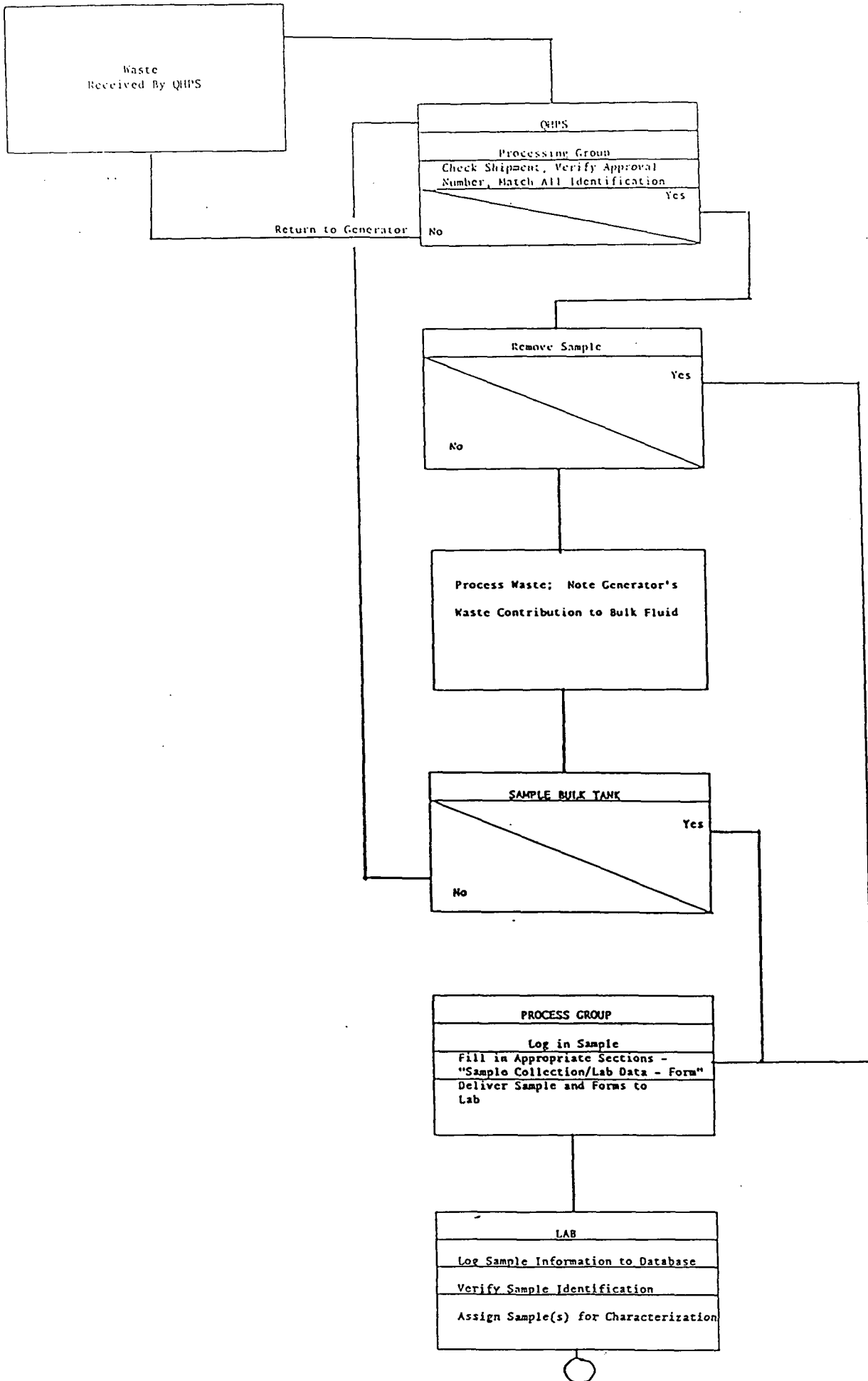
QHPS
Waste Approval
Lab
<div style="display: flex; justify-content: space-between;"> <span>No</span> <span>Is Waste "F" Series?</span> <span>Yes</span> </div>

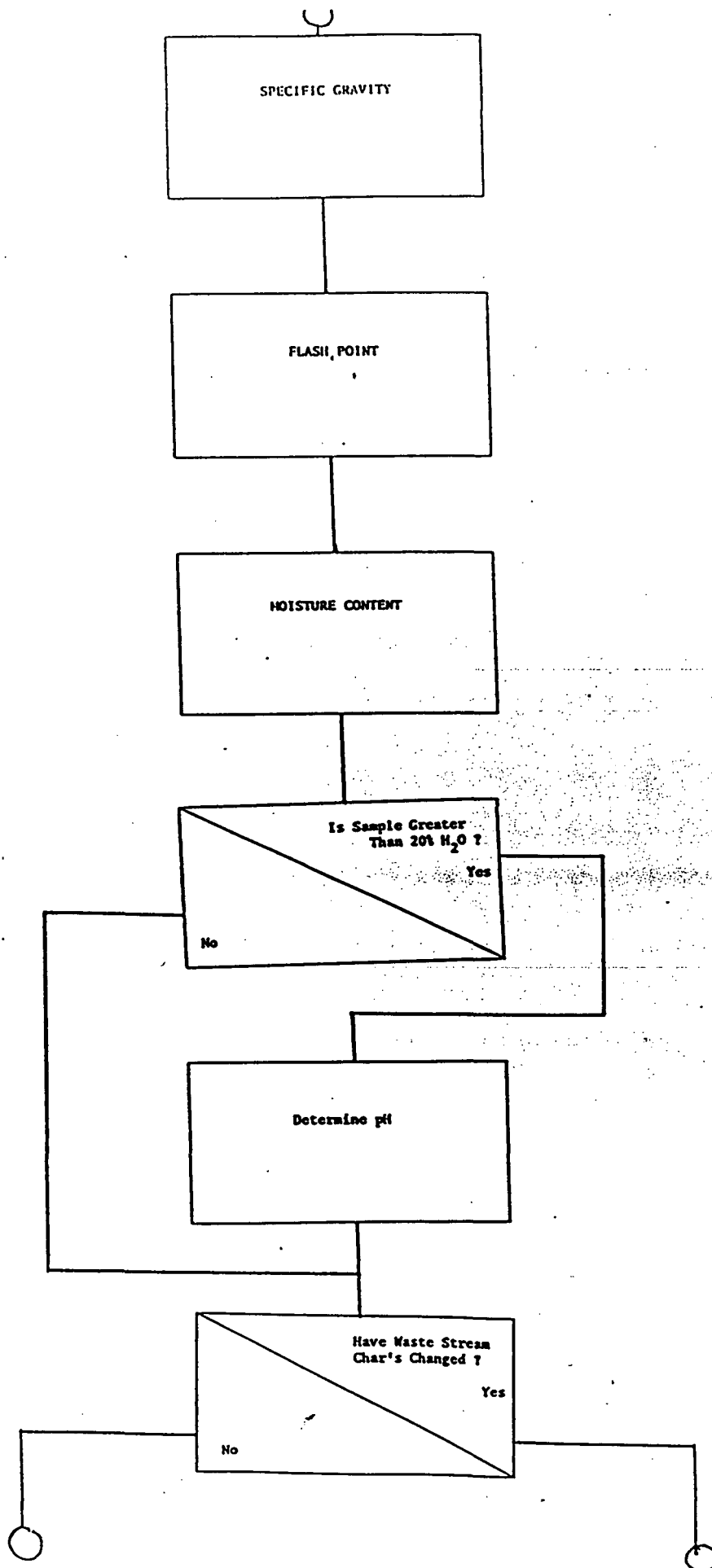
QHPS
Waste Disapproval
Lab
Generate Disapproval Number; Comment Reason(s). Notify Generator/Broker

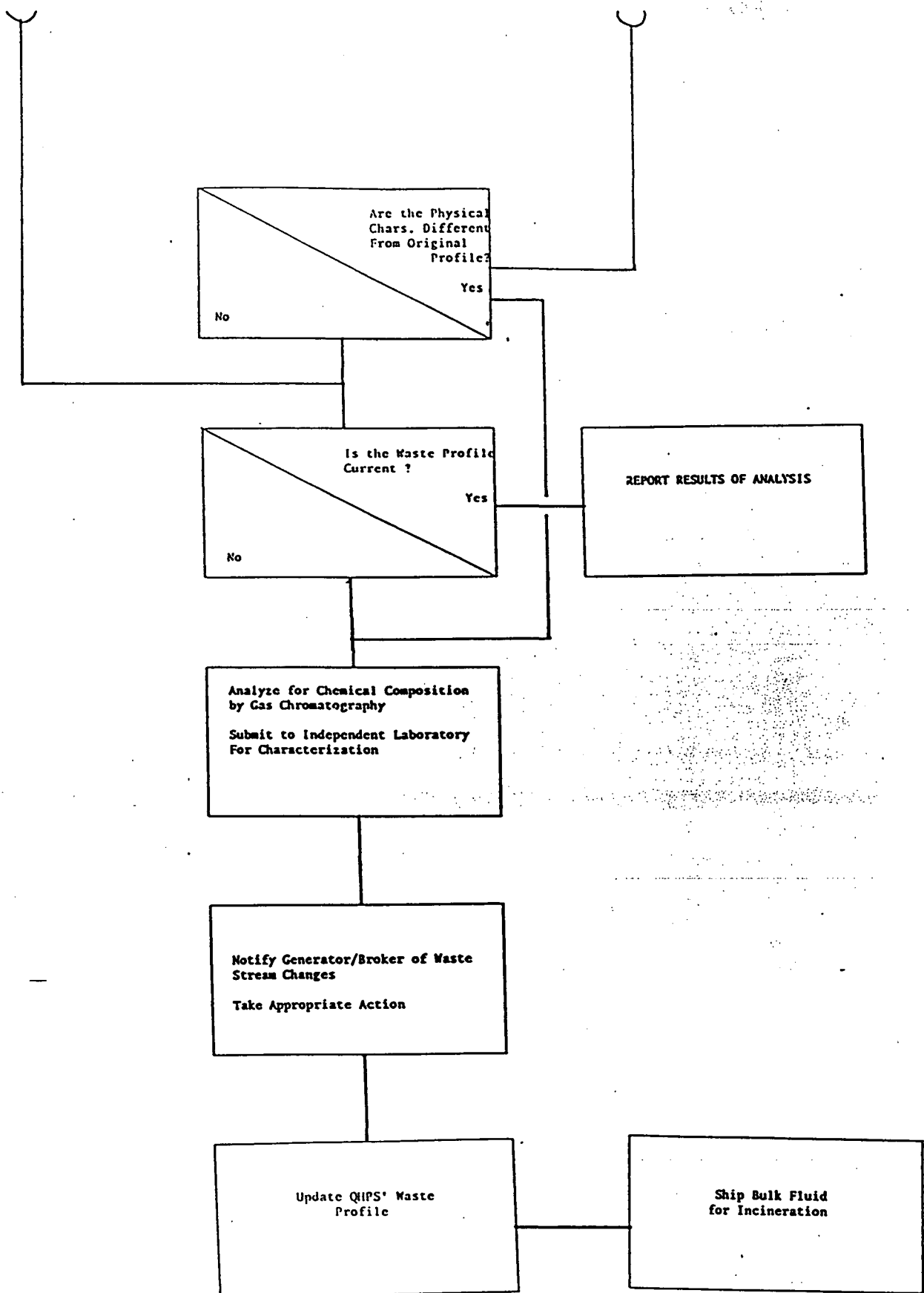
QHPS
Waste Approval
Lab
<div style="display: flex; justify-content: space-between;"> <span>No</span> <span>Is Waste "D001" ?</span> <span>Yes</span> </div>

QHPS
Waste Approval
Lab
Generate Approval Number; Notify Generator/Broker
Log Information to Database; Notify Processing Group of Valid Approvals

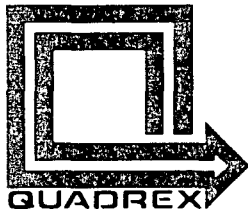












## Quadrex HPS Inc.

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1940 N.W. 67th Place, Gainesville, Florida 32606-1649  
904-373-6066 TELEX 53-5429 TELECOPY 904-373-0040

### Form Instructions:

Please fill in the Lab Pack Inventory Form as completely and accurately as possible. Include the chemical name of the container being placed in the drum for disposal. If the waste name is a trade name, please include a material safety data sheet. If the material safety data sheet is not available, please list all constituents of the product and the manufacturer's name and phone number.

List the number of containers of each waste present in the drum and the amount of the waste contained in each container. Also note the size of container the waste is contained in i.e. 500 ml/ quart.

Note the physical form the waste is in (solid or liquid).

Note the relative location of the container(s) in the drum i.e. "top", "middle", or "bottom".

Your cooperation is greatly appreciated.



LAB PACK INVENTORY FORM

Generator Name: \_\_\_\_\_

EPA ID#: \_\_\_\_\_

Drum ID#: \_\_\_\_\_

CHEMICAL NAME	Number of Cont.	Amount in Cont(s).	Physical Character Solid or Liquid	Container Location in Drum	Received (QHPS use)

Return to: Quadrex HPS Inc., 1940 N.W. 67th Place, Gainesville, FL 32606-1649



**QUADREX HPS INC. INSTITUTIONAL WASTE**

**Sample Collection/Request For Analysis/Lab Data-Form**

Directions: Sections A - L to be filled out at time of Sample Collection

Sections M - U to be filled out by the laboratory

A: Approval Number \_\_\_\_\_

B: Generator Name \_\_\_\_\_

C: I.D. \_\_\_\_\_

D: Broker \_\_\_\_\_

E: Shipment # \_\_\_\_\_

F: Waste Profile Code \_\_\_\_\_

G: QHPS Sample Code \_\_\_\_\_

H: Collection Date \_\_\_\_\_

I: Collection Time \_\_\_\_\_

J: Responsible: \_\_\_\_\_

K: Location: \_\_\_\_\_

L: Comments: \_\_\_\_\_

**Physical Characteristics of Waste**

M: Color \_\_\_\_\_

N: Odor: \_\_\_\_\_

O: Layers ? \_\_\_\_\_

P: Moisture Content: \_\_\_\_\_%

Q: Specific Gravity \_\_\_\_\_ g/ml

R: Flash Point \_\_\_\_\_ °C

S: pH: \_\_\_\_\_

T: Other \_\_\_\_\_

U: Chemical Composition: \_\_\_\_\_

_____	_____%
_____	_____%
_____	_____%
_____	_____%
_____	_____%
_____	_____%
_____	_____%
_____	_____%



QUADREX HPS INC. INSTITUTIONAL WASTE

Sample Collection/Request For Analysis/Lab Data-Form

Chemical Composition Continued:

_____	_____%
_____	_____%
_____	_____%
_____	_____%
_____	_____%
_____	_____%
_____	_____%
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_____	_____%

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

# STANDARD LABORATORY PROCEDURE

**PROCEDURE NUMBER:** 4000-001

**TITLE:** METHOD OF ANALYSIS FOR ORGANIC ANALYSIS OF WASTE STREAM  
SAMPLES BY GAS CHROMATOGRAPHY

**REVISION NUMBER:** 2

**PREPARED BY:**

Signature

Date

**APPROVED BY:**

Signature

Date

## 1.0 PURPOSE

Method 4000-001 is used to quantitatively determine the concentrations of the various permitted organic compounds present in Hazardous Waste waste streams. Method 4000-001 combines individual methods as published in EPA SW-846 Second Edition. The combined methods are: Method 8010 Halogenated Volatile Organics, Method 8015 Nonhalogenated Volatile Organics, Method 8020 Aromatic Volatile Organics, Method 8030 Acrolein, Acrylonitrile, Acetonitrile. The methods as published by EPA are very similar in scope and procedure. By combining chemical functional groups into one method, a rapid and accurate method of analysis may be performed on the waste sample that may contain any or all listed chemicals.

## 2.0 DEFINITIONS

Hazardous Waste - As defined in 40 CFR 261 Subpart A.

Hazardous Waste Characteristics - As defined in 40 CFR 261 Subpart C.

## 3.0 PRECAUTIONS

The following is a description of the "Toxicity Rating System" as described in: Haley, Thomas J., PhD, "Toxicology", Section 1 in Dangerous Properties of Industrial Materials, N. Irving Sax, Sixth Ed., (New York, NY: Van Nostrand Reinhold Company Inc., 1984), p. 1-2.

The appropriate toxicity rating (if available) for each specific chemical can be found in the procedure section, Table 1.

U = unknown

This rating covers chemicals for which insufficient data is available to enable a valid assessment of toxic hazards. Such chemicals usually are in one of the following categories:

# STANDARD LABORATORY PROCEDURE

April 5, 1988

1. No information has been discovered in the literature, and none is known to the authors.
2. Limited information from animal experiments is available, but it is not possible to relate it to human exposure. The data is mentioned to indicate to the reader that animal experiments have been done.
3. The validity of published toxicity data was questioned by the authors.

None = no toxicity (0)

This designation is for chemicals that fall into the following categories:

1. Chemicals that produce no toxic effects under any conditions of normal usage.
2. Chemicals that require overwhelming dosage to produce any toxic effects in humans.

Low = slight toxicity (1)

1. Acute local. Chemicals that on a single exposure lasting seconds, minutes, or hours cause only slight effects on the skin or mucous membranes or eyes, regardless of the extent of exposure.
2. Acute systemic. Chemicals that can enter the body by inhalation, ingestion, or dermal contact and produce only slight toxic effects, regardless of the duration of exposure or following the ingestion of a single dose, regardless of the amount absorbed or the extent of the exposure.
3. Chronic local. Chemicals that on repeated or continuous exposure covering days, months, or years cause only slight and reversible damage to the skin or mucous membranes. The extent of the exposure can be great or small.
4. Chronic systemic. Chemicals that on repeated or continuous exposure covering days, months, or years cause slight and usually reversible toxic effects on the skin, mucous membranes, or eyes. The exposure can be by ingestion, inhalation, or skin contact and may be great or small. Slightly toxic chemicals produce changes readily reversible once the exposure ceases with or without medical intervention.

## STANDARD LABORATORY PROCEDURE

Mod = moderate toxicity (2)

1. Acute local. Chemicals that on a single exposure lasting seconds, minutes, or hours produce moderate toxicity to the skin, mucous membranes, or eyes. The effects can be the result of an intense exposure for seconds or a moderate exposure for hours.
2. Acute systemic. Chemicals that after being absorbed by inhalation, ingestion, or skin contact produce moderate toxicity after a single exposure lasting seconds, minutes, or hours or after the ingestion of a single dose.
3. Chronic local. Chemicals that on continuous or repeated exposure over days, months, or years cause moderate toxicity to the skin, mucous membranes, or eyes.
4. Chronic systemic. Chemicals that on absorbed by ingestion, inhalation, or skin contact cause moderate toxicity after continuous or repeated exposures over days, months, or years.

Under the moderate classification are chemicals that can cause reversible or irreversible changes in the human body not necessarily severe enough to cause serious physical impairment or threaten life.

High = severe toxicity (3)

1. Acute local. Chemicals that on a single exposure covering seconds or minutes can cause injury to the skin, mucous membranes, or eyes of sufficient severity to threaten life, cause permanent physical impairment or disfigurement.
2. Acute systemic. Chemicals that after a single exposure by inhalation, ingestion, or skin contact cause injury of sufficient severity to threaten life. The exposure may last seconds, minutes, or hours or may be a single ingestion.
3. Chronic local. Chemicals that on continuous or repeated exposures covering days, months, or years can cause injury to the skin, mucous membranes, or eyes of sufficient severity to threaten life or produce permanent impairment, disfigurement, or irreversible change.

# STANDARD LABORATORY PROCEDURE

4. Chronic systemic. Chemicals that on continuous or repeated exposures by inhalation, ingestion, or dermal contact of small amounts for days, months, or years can produce death or serious physical impairment.

## 4.0 EQUIPMENT AND MATERIALS

Hewlett-Packard 5890 Gas Chromatograph with FI Detector and EC Detector.

Chromatographic column: HP "PONA" Capillary Column, 6'x 1/4" glass, packed methyl silicone column.

## 5.0 DOCUMENTATION

All raw data is to be maintained in designated laboratory notebooks. The final chromatogram(s) and report of analysis are to be maintained indefinitely.

## 6.0 PROCEDURE

### 6.1 SAMPLE PREPARATION (LIQUID SAMPLE)

1. Liquid samples must be analyzed immediately if stored in a plastic container.
2. Remove an aliquot of the sample and secure in a glass vial with a Teflon-lined closure.
3. Dilute the sample if necessary, generally the sample is injected directly onto the column with no dilution.
4. Inject between 0.1 ul - 0.5 ul sample onto the capillary column. Compare FID mixture standard with sample chromatogram. "Confirm" halogenated compounds by injecting sample onto the packed column, detecting by electron capture.

### 6.2 ANALYTICAL PROCEDURE

#### GAS CHROMATOGRAPH CONDITIONS (FID)

##### Run Parameters

Zero - 10  
Att. - 2  
Chart Speed - 0.5  
PK. Width - 0.02  
Thresh. - 2  
Ar. Rej. - 0



# STANDARD LABORATORY PROCEDURE

## Temperatures

Initial Temp. - 45° C  
Initial Time - 10 min.  
Rate - 1.5° C/min.  
Final Temp. - 70° C  
Final Time - 0  
Ramp A Rate - 3.0° C/min.  
Final Temp. - 225° C/min.  
Final Time - 10 min.

Injection Port Temp. - 225° C/min.

Detector Temp. - 350° C/min.

## Gas Flows

Carrier (He) -  $u = 12$  cm/sec. or 2.3 cm<sup>3</sup>/min.  
Makeup (He) - 30 cm<sup>3</sup>/min.  
Hydrogen - 40 cm<sup>3</sup>/min.  
Air - 400 cm<sup>3</sup>/min.  
Split Ratio - 1:25

## GAS CHROMATOGRAPH CONDITIONS (ECD)

The following GC parameters are for "confirmation" of halogenated compounds using the electron capture detector.

### Run Parameters

Zero - 10  
Att. - 7  
Chart Speed - 1.0  
PK. Width - 0.04  
Thresh. - 5  
Ar. Rej. - 0

## Temperatures

Initial Temp. - 30° C  
Initial Time - 10 min.  
Rate - 3.0° C/min.  
Final Temp. - 70° C  
Final Time - 0  
Ramp A Rate - 5.0° C/min.  
Final Temp. - 225° C/min.  
Final Time - 10 min.

Injection Port Temp. - 225° C/min.

Detector Temp. - 350° C/min.

## Gas Flows

Carrier (Ag/Me) - 35 ml/min.

## STANDARD LABORATORY PROCEDURE

CALIBRATION

A calibration curve for each chemical of interest must be established prior to the analysis of a waste sample. The calibration concentrations should be inclusive of the concentration of the samples of interest. A minimum of 3 concentration levels should be prepared. The minimum concentration should be close, but slightly above the minimum level of detection (MLD) for that compound. If the response factor ( $R_f$  = weight of analyte/area of integration) for the calibration standards are  $\leq 10$  relative standard deviations ( $RSD = [Std. Dev./Avg. R_f] \times 100$ ), a single point calibration factor may be used.

The calibration will be performed by external calibration. It will be at the analyst discretion to calibrate using the complete list of chemicals; or based on the waste stream, prepare an "unique" calibration standard. Regardless of the chemicals chosen for analysis, reference peaks must be designated through out the chromatographic run. This will compensate for any retention time shifts occurring during the analytical run.

Criterion for selecting reference peaks are as follows: the reference peaks should be prominent and isolated; multiple reference peaks are preferable to a single one. If a single reference peak is chosen, it should occur later than the middle of the run; reference peaks (if in multiples) should be spaced evenly throughout the run.

The working calibration curve or  $R_f$  must be validated each day by measuring one or more of the calibration standards. If the response varies more than 10%, the calibration must be repeated using a fresh calibration standard, or a new calibration curve or  $R_f$  must be generated.

Table 1 contains the current list of chemicals designated on Attachment A of the operating Permit/Certification No. HT-01-136540. This list contains the chemical compounds QHPS is currently permitted to store and process on site. The table lists the approximate retention time for detection. Also listed is the chemical's physical characteristics and toxicity rating.

CONCENTRATION DETERMINATION

1. Determine the response factor for each of the compounds present in the standard chromatogram.

$$R_f = \frac{\text{ng. injected from the std}}{\text{area of integration}}$$

$$\text{Concentration (ug/ml)} = \frac{\text{Area from sample} \times R_f}{\text{ul injected}}$$

# STANDARD LABORATORY PROCEDURE

## 7.0 QUALITY CONTROL

### 7.1 BLIND SUBMITTAL

Ongoing performance checks must be performed on a routine basis. The check will consist of submitting to the analyst "blind" QC spiked samples containing all or part of the compounds of interest. The percent recovery (R) and the standard deviation of the percent recovery (s) noted at each concentration level.

### 7.2 INSTRUMENT MODIFICATIONS

All instrument changes or modifications must be noted in the appropriate instrument log.

### 7.3 INSTRUMENT PERFORMANCE

All raw and processed data must be maintained in its appropriate location. The data is used to perform periodical instrument performance audits.

### 7.4 DATA REPORTING

Analytical results will be reported in a timely manner to the person or group originating the sample (unless otherwise stated).

The data will be reported generally to two significant figures. The report will also indicate the MLD for that set of results.

### 7.5 SAMPLE DISPOSITION

The samples will generally be maintained for a period of three months from the time of reporting. At this time they will be disposed appropriately.

A detailed discussion QA/QC measures may be found in QHPS Laboratory Quality Control Manual.

# STANDARD LABORATORY PROCEDURE

PAGE 1

TABLE 1

REV. APRIL 15, 1988

## CHARACTERISTICS OF ORGANIC COMPOUNDS CURRENTLY PERMITTED TO BLEND

CHEMICAL NAME	RT	COLUMN TEMP. (C)	ACTUAL BP (C)	TOXICITY CODE	DENSITY (g./ml.)	FLASH POINT (C)	SOLUBILITY IN WATER
1,1,1-TRICHLOROETHANE	16.2	54.3	74.1	2-1	1.339	NONE	INSOLUBLE
1,1,2-TRICHLOROETHANE	26.7	70.1	114	3	1.4397	NONE	INSOLUBLE
1,1,2-TRICHLOROTRIFLUOROETHANE	10.3	45	47.7		1.5635	NONE	@25, .017%
1,2-DICHLOROBENZENE	52.1	146.5	180.5	3	1.3048	66	INSOLUBLE
1,3-DICHLOROBENZENE	49.7	139.3	173.4	3	1.2884	63.3	INSOLUBLE
2-ETHOXYETHANOL	21.9	62.9	135.1	2	0.9297	44	MISC
2-NITROPROPANE	19.1	58.7	120	3-2	0.9876	88	SLIGHT
ACETONE	8.7	45	56.29	2-1	0.79	-18	MISC
BENZENE	17.4	56	80.1	3	0.8787	-11	@25, .18%
CARBON DISULFIDE	10.5	45.8	46.5	3	1.2632	-30	MISC
CARBON TETRACHLORIDE	18	57	76.8	3	1.594	NONE	@20, .08%
CHLOROBENZENE	36	98.2	131.69		1.1058	30	@20, .1%
CYCLOHEXANONE	39.3	108.1	115.6	2	0.9478	43.9	@20, 15%
ETHANOL	8.1	45	78.32	3	0.7893	13.1	MISC
ETHYL ACETATE	13.7	50.6	77.15	2-1	0.9006	-4	@20, 8.7%
ETHYL BENZENE	37.8	103.6	132.6	2-1	0.8669	15	@20, .015%
ETHYL ETHER	9.4	45	34.6	2	0.7135	-45	@20, 6.9%
HEXANE	12.8	49.2	69	2-1	0.6603	-26	INSOLUBLE
HEXANE	15.6	53.4	69	2-1	0.6603	-26	INSOLUBLE
HEXANE	13.8	50.7	69	2-1	0.6603	-26	INSOLUBLE
ISOMYL ALCOHOL	24.2	66.3	107.9	3	0.813	42.8	SLIGHTLY
ISOBUTYL ALCOHOL	14.7	52.1	99.2		0.7982	28	@20, 8.7%
ISOOCTANE	20.9	61.4	132	3	0.698	-12	INSOLUBLE
METHANOL	7.2	45	64.7	3-2-1	0.698	12	MISC
METHYL ETHYL KETONE	12.4	48.6	79.57	3	0.8049	-1	@ 20, 24%
METHYL ISOBUTYL KETONE	24.2	66.3	118	3-2	0.8008	23	@20, 1.9%
METHYL PYRROLE	24	66	112		0.9	16	INSOLUBLE
METHYLENE CHLORIDE	9.9	45	39.8	3	1.326	NONE	@20, 1.6%
1,4-DIOXANE			101.0	3	1.035	12.2	SOLUABLE
CYCLOHEXANE			115.6	2	0.948	-27.0	@25, .006%
ETHYLENE GLYCOL			124.5	2	0.966	46.1	MISC
m-CRESOL	53.6	151	108.5	3	1.034	94.4	SLIGHT
m-XYLENE	38.7	106.3	139	3-2	0.8642	27.2	INSOLUBLE
NAPHTHALENE			219.9	3	1.162	79.0	INSOLUABLE
NITROBENZENE	54.5	153.7	210	3-2	1.205	87.8	SLIGHT
n-BUTYL ALCOHOL	17.3	56	117.5	3-2	0.8097	23.9	@20, 12.5
o-CRESOL	52.3	147.1	108.5	3	1.047	81.1	SOLUABLE
o-XYLENE	40.7	112.3	144.4	3-2	0.8802	32.2	INSOLUBLE
PYRIDINE	24	66	115.3	2	0.9832	20	@20, 100%
p-CRESOL			201.8	3	1.034	94.4	SLIGHT
p-XYLENE	38.6	106	138.3	3-2	0.8611	27.2	INSOLUBLE
TETRACHLOROETHYLENE	33.2	89.8	121.2	3	1.6311	NONE	@20, .015%

# STANDARD LABORATORY PROCEDURE

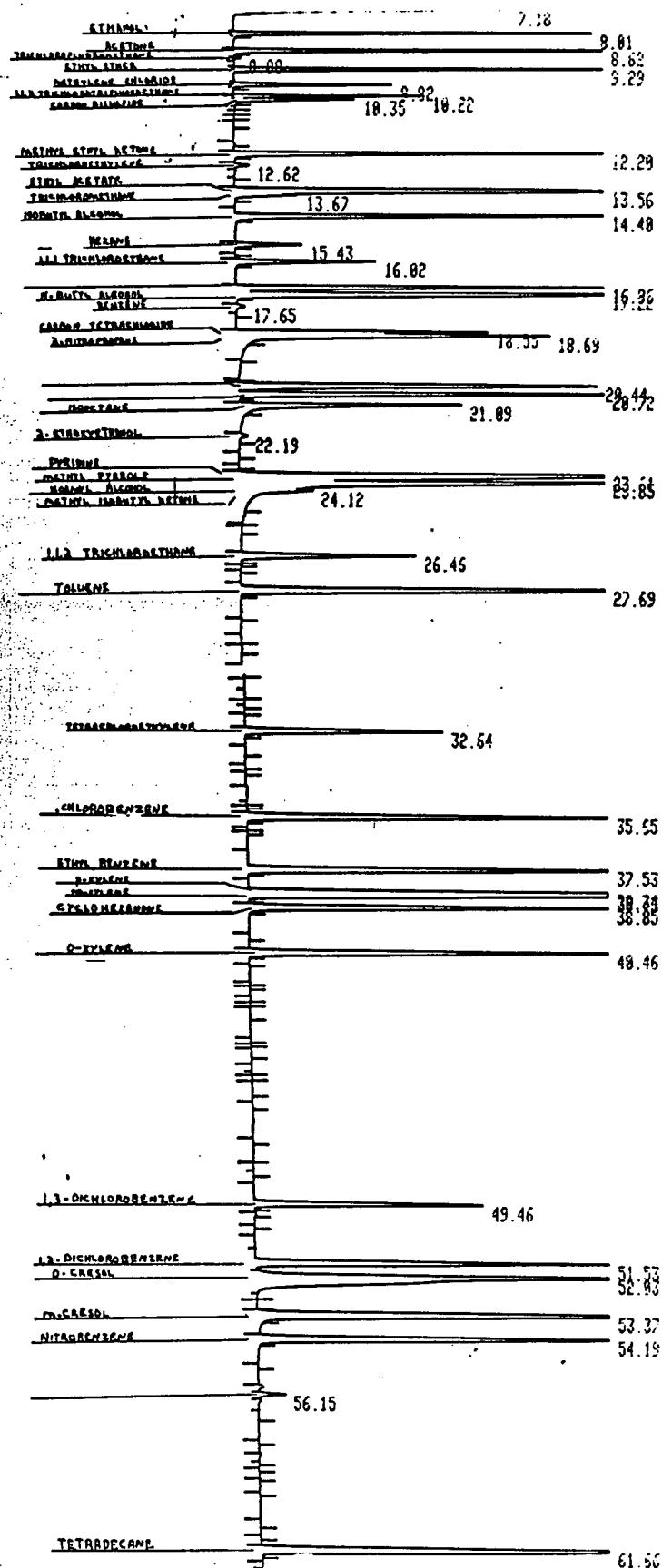
PAGE 2

TABLE 1

REV. APRIL 15, 1988

## CHARACTERISTICS OF ORGANIC COMPOUNDS CURRENTLY PERMITTED TO BLEND

CHEMICAL NAME	RT	COLUMN TEMP. (C)	ACTUAL BP (C)	TOXICITY CODE	DENSITY (g./ml.)	FLASH POINT (C)	SOLUBILITY IN WATER
TETRADECANE	59.1	167.5	253	3	0.7628	99	INSOLUABLE
TOLUENE	27.9	73.9	110.4	3	0.8669	6	020, .05%
TRICHLOROETHYLENE	12.7	49.1	86.7	3	1.4762	NONE	025, .1%
TRICHLOROFLUOROMETHANE	9	45	24.1	2-1	1.484	NONE	2.5?
TRICHLOROMETHANE			61.3	3	1.483	-63.5	INSOLUABLE
TRICHLOROMETHANE, (chloroform)	13.8	50.7	61.26	3	1.4832	NONE	020, .8%



# STANDARD LABORATORY <sup>AP-1088</sup>PROCEDURE

PROCEDURE NUMBER: 4000-005

TITLE: THE HYDROMETER METHOD FOR SPECIFIC GRAVITY  
DETERMINATION

REVISION NUMBER:

PREPARED BY:

Signature

Date

APPROVED BY:

Signature

Date

## 1.0 PURPOSE

This method uses the ERTCO hydrometer to determine the specific gravity in various organic solvents or wastes.

## 2.0 DEFINITIONS

None

## 3.0 PRECAUTIONS

Standard Lab Practice

DO NOT drop hydrometer into graduated cylinder.

## 4.0 EQUIPMENT AND MATERIALS

ERTCO hydrometers (ranges;0.7-1.0, 1.0-2.0, 1.40-1.66)

250ml graduated cylinder

## 5.0 DOCUMENTATION

Record data for each analysis on the Quadrex HPS Inc.  
Institutional Waste, Sample Collection/Request For Analysis/Lab  
Data-Form.

# STANDARD LABORATORY PROCEDURE

Apr 21, 1988

## 6.0 PROCEDURE

Fill 250 ml graduated cylinder with sample to approximately 180 ml. Place the lowest range hydrometer into the sample, replace with the next higher range if the level of the liquid does not all within the parameters of the hydrometer. When the level of the sample falls within the range of the hydrometer then read the specific gravity (g/ml) directly off the hydrometer.



# STANDARD LABORATORY PROCEDURE

December 9, 1987

PROCEDURE NUMBER: 4000-002

TITLE: PENSKY-MARTENS CLOSED-CUP METHOD FOR FLASH POINT-  
DETERMINATION<sup>1</sup>

REVISION NUMBER:

PREPARED BY:

Signature

Date

APPROVED BY:

Signature

Date

## 1.0 PURPOSE

This method uses the Pensky-Martens closed-cup tester to determine the flash point of fuel oils, lube oils, suspensions of solids, liquids that tend to form a surface film under test conditions, and other liquids.

## 2.0 DEFINITIONS

None

## 3.0 PRECAUTIONS

Standard Lab Practice

## 4.0 EQUIPMENT AND MATERIALS

Pensky-Martens Closed Flash Tester (as in Annex A1 of ASTM Method D93-77)

Standard Thermometers -  $-5^{\circ}$  -  $110^{\circ}$  C.

Calcium chloride

p-Xylene reference standard

## 5.0 DOCUMENTATION

Record data for each analysis in log book assigned to instrument.

# STANDARD LABORATORY PROCEDURE

December 9, 1987

## 6.0 PROCEDURE

Preparation of samples: Samples that do not contain volatile contaminants shall be prepared in the following manner:

Samples of very viscous materials may be warmed until they are reasonably fluid before they are tested. However, no sample should be heated more than is absolutely necessary, and no sample should ever be heated to a temperature that exceeds  $17^{\circ}\text{C}$  below the sample's expected flash point.

Samples containing dissolved or free water may be dehydrated with calcium chloride or by filtering through a qualitative filter paper or a loose plug or dry adsorbent cotton. Warming the sample is permitted, but it shall not be heated for prolonged periods or above a temperature of  $17^{\circ}\text{C}$  below the sample's expected flash point.

### Routine procedure

Thoroughly clean and dry all parts of the cup and its accessories before starting the test. Be sure to remove any solvent that was used to clean the apparatus. Fill the cup with the sample to be tested to the level indicated by the filling mark. Place the lid on the cup and set the latter in the stove. Be sure to properly engage the locating device. Insert the thermometer. Light the test flame and adjust it to a diameter of 4 mm. Supply the heat at such a rate that the temperature as indicated by the thermometer increased  $5^{\circ} - 6^{\circ}\text{C/min}$ . Turn on the stirrer.

If the sample is expected to have a flash point of  $110^{\circ}\text{C}$ , or below, apply the test flame when the temperature of the sample is from  $17^{\circ} - 28^{\circ}\text{C}$  below the expected flash point and thereafter at a temperature reading that is a multiple of  $1^{\circ}\text{C}$ . Apply the test flame by operating the mechanism on the cover which controls the shutter and test flame burner so that the flame is lowered into the vapor space of the cup in 0.5 sec, left in its lowered position for 1 sec, and quickly raised to its high position. Do not stir the sample while applying the test flame.

Record as the flash point the temperature read on the thermometer at the time the test flame application caused a distinct flash in the interior of the cup. Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at application preceding the one that causes the actual flash. The actual flash will have occurred when a large flame propagates itself over the surface of the sample.

# STANDARD LABORATORY PROCEDURE

December 9, 1987

Determination of flash point of suspensions of solids and highly viscous materials

Bring the material to be tested and the tester to a temperature of  $15^{\circ} \pm 5^{\circ} \text{ C}$  ( $60^{\circ} \pm 10^{\circ} \text{ F}$ ) or  $11^{\circ} \text{ C}$  ( $20^{\circ} \text{ F}$ ) lower than the estimated flash point, whichever is lower. Turn the stirrer  $250 \pm 10 \text{ rpm}$ , stirring in a downward direction. Raise the temperature throughout the duration of the test at a rate of not less than  $1^{\circ}$  nor more than  $1.5^{\circ} \text{ F}$  ( $2 \text{ to } 3^{\circ} \text{ F}$ )/min. With the exception of these requirements for rates of stirring and heating, proceed as prescribed in above section.

Calculation and report

Observe and record the ambient barometric pressure at the time of the test. When the pressure differs from 760 mm Hg (101.3 kPa), correct the flash point as follows:

- A. Corrected flash point =  $C + 0.25 (101.3 - p)$
- B. Corrected flash point =  $F + 0.06 (760 - P)$
- C. Corrected flash point =  $C + 0.333 (760 - P)$

where:

F = observed flash point,  $^{\circ} \text{ F}$   
C = observed flash point,  $^{\circ} \text{ C}$   
P = ambient barometric pressure, mm Hg  
p = ambient barometric pressure, kPa

NOTE: The barometric pressure used in this calculation must be ambient pressure for the laboratory at the time of test. Many aneroid barometers, such as those used at weather stations and airports, are precorrected to give sea level readings. These must not be used.

Record the corrected flash point to the nearest  $0.5^{\circ} \text{ C}$  (or  $1^{\circ} \text{ F}$ ).

Report the recorded flash point as the Pensky-Martens Closed Cup Flash Point ASTM D93 - IP 34, of the sample tested.

Refer to Method ASTM D93 77 for more details and background on the Pensky-Martens method.

## 7.0 QUALITY CONTROL

All quality control data should be available for review.

Duplicates and standard reference materials should be routinely analyzed.

# STANDARD LABORATORY PROCEDURE

December 9, 1987

The flash point of the p-Xylene reference standard must be determined in duplicate at least once per sample patch. The average of the two analyses should be  $27^{\circ} \pm 0.8^{\circ} \text{C}$  ( $81^{\circ} \pm 1.5^{\circ} \text{F}$ ).

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<sup>1</sup> U.S. Environmental Protection Agency, Method 1010 in "Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, 2nd Edition, Revised", Office of Solid Waste and Emergency Response, Washington, DC (April 1984a).

# STANDARD LABORATORY PROCEDURE

January 6, 1988

PROCEDURE NUMBER: 4000-003

TITLE: Moisture Determination by Karl Fischer Method

REVISION NUMBER:

PREPARED BY:

Signature

Date

APPROVED BY:

Signature

Date

## 1.0 PURPOSE

To determine the water content in various organic solvents or wastes.

## 2.0 DEFINITIONS

Karl Fischer Reagent (KFR), consisting of a mixture of pyridine, iodine, sulphur dioxide, and methanol, reacts stoichiometrically with water. A given volume of KFR will react with an exact volume of water. In an excess of KFR, as the reaction mixture changes from light yellow to brown, the electrical resistance of the solution drops to less than a thousand ohms; in an excess of water the electrical resistance remains high (over several thousand ohms), and the reaction changes from dark brown to light yellow.

## 3.0 PRECAUTIONS

Warning! Combustible, Causes Burns - Overexposure may cause male or female reproductive disorders. Exceptional health and contact hazards. Protective clothing must be worn. Work area must be adequately ventilated.

## 4.0 EQUIPMENT AND MATERIALS

Aquametry Apparatus and Reagents

Methanol

## 5.0 DOCUMENTATION

All data and results of standardization must be maintained in appropriate log for auditing purposes.

## 6.0 PROCEDURE

Set up apparatus as shown in the figure.

# STANDARD LABORATORY PROCEDURE

January 6, 1988

Make sure the drying tubes are in place prior to dispensing the reagent.

Transfer the KFR to the apparatus reagent bottle.

Add approximately 50 ml of solvent (methanol) to the reaction vessel through the sample port. And turn on the magnetic stirrer.

Remove the water in the solvent by adding KF reagent to just turn the solution color from yellow to dark brown. This is known as the KF end-point. Note the meter indication on the conductance meter. You may titrate to any point in the brown KF zone, provided that you always titrate to that end-point. Allow the solution to stabilize at the end-point for at least one minute before proceeding.

Standardize the water equivalence of the KF reagent. Normally this needs to be done only once a day, or when changing the KF reagent supply.

Into the reaction vessel, still at the end-point, pipet a weighed or accurately measured volume of pure water, usually 15 mg (15  $\mu$ l), through the sample port. For greatest accuracy, weigh the pipet before and after dispensing to determine the exact weight dispensed.

Titrate the water to the same end-point (meter reading) before the water was added. (1 ml of KF reagent is equivalent to approximately 5 mg water).

Compute the KF reagent water equivalence, F:  $F = \text{mg water added} / \text{ml KFR required to return to the same end-point}$ .

To the reaction vessel, still at the end-point, add a weighted amount of your sample which is of sufficient size to contain 20 to 50 mg water. A sufficiency of water is indicated by the solution color change to light yellow and/or the meter registering below the KF zone.

Titrate the water to the original end-point.

# STANDARD LABORATORY PROCEDURE

January 6, 1988

Calculation: Calculate the water content of the sample as follows:

Water, percent =  $(A * F * 0.1)/S$  where:

A = Milliliters of KFR required for titration of sample

F = Water equivalence of KFR as determined in Step 4 (expressed in mg of water per ml of KFR)

S = Grams of sample

# STANDARD LABORATORY PROCEDURE

December 9, 1987

PROCEDURE NUMBER: 4000-004

TITLE: pH MEASUREMENT OF AQUEOUS WASTES<sup>1</sup>

REVISION NUMBER:

PREPARED BY:

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date

APPROVED BY:

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date

## 1.0 PURPOSE

Procedure 4000-003 is used to measure the pH of aqueous wastes and those multiphasic wastes where the aqueous phase comprises at least 20% of the total volume of the waste.

## 2.0 DEFINITIONS

The pH of the sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode. The measuring device is calibrated using a series of solutions known pH.

## 3.0 PRECAUTIONS

The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or high salinity.

Sodium error at pH levels greater than 10 can be reduced or eliminated by using a "low sodium error" electrode.

Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by distilled water rinsing. An additional treatment with hydrochloric acid (1:9) may be necessary to remove any remaining film.

Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample-dependent and cannot be controlled. It should, therefore, be noted by reporting both the pH and temperature at the time of analysis.



# STANDARD LABORATORY PROCEDURE

December 9, 1987

## 4.0 EQUIPMENT AND MATERIALS

pH Meter: Fisher Accumet Model 825MP

Universal Glass Electrode

Calomel Reference Electrode

Magnetic stirrer and Teflon-coated stirring bar.

## 5.0 DOCUMENTATION

None

## 6.0 PROCEDURE

### Calibration

Primary standard buffer salts are available from the National Bureau of Standards and should be used in situations where extreme accuracy is necessary. Preparation of reference solutions from these salts requires some special precautions and handling such as low-conductivity dilution water, drying ovens, and carbon-dioxide-free purge gas. These solutions should be replaced at least once each month.

Secondary standard buffers may be prepared from NBS salts or purchased as a solution from commercial vendors. These commercially available solutions have been validated by comparison to NBS standards and are recommended for routine use.

Because of the wide variety of pH meters and accessories, detailed operation procedures cannot be incorporated into this method. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions. Special attention to care of the electrodes is recommended.

Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart.

Place the sample or buffer solution in a clean glass beaker using a sufficient volume to cover the sensing elements of the electrodes and to give adequate clearance for the magnetic stirring bar. If field measurements are being made, the electrodes may be immersed directly in the sample stream to an adequate depth and moved in a manner to ensure sufficient sample movement across the electrode-sensing element as indicated by drift-free (less than 0.1 pH) readings.

If the sample temperature differs by more than 2° C from the buffer solution, the measured pH values must be corrected. Instruments are equipped with automatic or manual compensators

# STANDARD LABORATORY PROCEDURE

December 9, 1987

that electronically adjust for temperature differences. Refer to manufacturer's instructions.

Thoroughly rinse and gently wipe the electrodes prior to measuring pH of samples. Immerse the electrodes into the sample beaker or sample stream and gently stir at a constant rate to provide homogeneity and suspension of solids. Note and record sample pH and temperature. Repeat measurement on successive volumes of sample until values differ by less than 0.1 pH units. Two or three volume changes are usually sufficient.

## 7.0 QUALITY CONTROL

Duplicate samples and check standards should be analyzed routinely.

Electrodes must be thoroughly rinsed between samples.

Refer to instrument operation manual or QA/QC manual for additional information.

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<sup>1</sup> U.S. Environmental Protection Agency, Method 9040 in "Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, 2nd Edition, Revised, "Office of Solid Waste and Emergency Response, Washington, DC (April 1984a).

# STANDARD LABORATORY P..OCEDURE

PROCEDURE NUMBER: 4000-006

TITLE: TOTAL CHLORINE IN WASTE OIL<sup>1</sup>

REVISION NUMBER:

PREPARED BY:

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date

APPROVED BY:

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date

\_\_\_\_\_  
\_\_\_\_\_  
  
This method describes a field test kit, developed by CHEMetrics, Incorporated, for the rapid field determination of total chlorine in waste oil.

# Technical Bulletin

1845-8

from CHEMetrics, Inc.  
Rt. 28, Calverton, VA 22016, (703) 788-9026  
Telex 292861 CHEM UR

## TOTAL CHLORINE IN WASTE OIL

### Quanti-Chlor<sup>™</sup> Test Kit

#### 1. Scope and Application

- 1.1 This method describes a field test kit, developed by CHEMetrics, Incorporated, for the rapid field determination of total chlorine in waste oil.
- 1.2 The method covers a range of 750 to 7,000 ppm (µg/g) Total Chlorine as Cl.

#### 2. Summary of Method

- 2.1 Organic chlorine is converted to inorganic chloride by reacting it with a mixture of diglyme, naphthalene and metallic sodium. An aqueous buffer is used to extract the inorganic chloride from the treated sample. Total chloride levels are then measured titrimetrically using mercuric nitrate as the titrant and diphenylcarbazone as the endpoint indicator.

#### 3. Comments

- 3.1 When the sodium ampoule is crushed (Step 6.4), waste oils that contain a high percentage of water will cause the sample to turn clear to light gray. Under these circumstances, the final test results will be low.
- 3.2 Other halogens (bromide and iodide) are measured as chloride.
- 3.3 The final calculation is based upon an average oil having a specific gravity of 0.9.

#### 4. Apparatus

- 4.1 Test kit components (see figure 1):
  - 4.1.1 Plastic reaction bottle, 1 ounce, with "flip top" dropper cap, containing a glass sodium ampoule.
  - 4.1.2 Plastic buffer bottle, containing 9.5 mls of an aqueous buffer solution.
  - 4.1.3 Titration vial, containing buffer bottle and indicator-impregnated paper.
  - 4.1.4 Glass vial, containing 2.0 mls of solvent.
  - 4.1.5 Micropipet and plunger.
  - 4.1.6 Activated carbon filtering column.
  - 4.1.7 Titret and valve assembly.
  - 4.1.8 Instructions.

1 Quanti-Chlor<sup>™</sup> is a trademark of CHEMetrics, Incorporated, Calverton, Virginia 22016.

# STANDARD LABORATORY PROCEDURE

Page 2 of 5

- 4.2 The procedure utilizes a Titret. Titrets® are hand-held, disposable cells for titrimetric analysis. A Titret is an evacuated glass ampoule (13 mm diameter) which contains an exact amount of a "standardized" liquid titrant. A flexible valve assembly is attached to the tip of the ampoule. Titrets® employ the principle of "reverse titration"; that is, small doses of sample are added to the titrant to the appearance of the endpoint color. The color change signals that the equivalency point has been reached. The flow of the sample into the Titret may be controlled by using an accessory called a Titrettor™.
- 4.3 The reagents needed for the test are packaged in disposable containers. No precautionary measures are required for safe disposal of the test components when the oil sample contains less than 1,000 ppm (µg/g) Cl. Samples containing higher levels are considered hazardous and must be treated accordingly.
- 4.4 No safety equipment is required to run this test. However, if the user suspects the oil sample contains hazardous levels of total chlorine, gloves should be worn when collecting the sample and while performing the test.

## 5. Reagents

- 5.1 The crushable glass ampoule, which is inside the reaction bottle, contains 85 mg of metallic sodium in a light oil dispersion.
- 5.2 The buffer bottle contains 0.44 g of  $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$  and 0.32 mls of  $\text{HNO}_3$  in distilled water.
- 5.3 The glass vial contains 770 mg Stoddard Solvent (CAS No. 8052-41-3), 260 mg toluene, 260 mg butyl ether, 260 mg diglyme, 130 mg naphthalene, and 70 mg demulsifier.
- 5.4 The Titret contains 1.12 mg mercuric nitrate in distilled water.
- 5.5 The indicator-impregnated paper contains approximately 0.3 mg of diphenylcarbazone and 0.2 mg of brilliant yellow.

## 6. Procedure

- 6.1 Shake the glass vial and pour its contents into the reaction bottle.
- 6.2 Fill the micropipet with a well-shaken oil sample by pulling the plunger until its top edge is even with the top edge of the micropipet. Wipe off the excess oil and transfer the sample into the reaction bottle (see fig. 2.1).
- 6.3 Gently squeeze most of the air out of the reaction bottle (see figure 2.2). Cap the bottle securely and shake vigorously for 30 seconds.
- 6.4 Crush the sodium ampoule by pressing against the outside wall of the reaction bottle (see figure 2.3).

**Caution:** Samples containing a high percentage of water will generate heat and gas, causing the reaction bottle walls to expand. To release the gas, briefly loosen the cap. Under these circumstances, the contents of the reaction bottle may be clear to light gray (rather than black), and consequently, the final test results will be low.

2 Titrets® is a registered trademark of CHEMetrics, Incorporated, Calverton, Virginia 22016

# STANDARD LABORATORY PROCEDURE

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- 6.5 Shake the reaction bottle vigorously for 30 seconds.
- 6.6 Wait 1 minute. Shake the reaction bottle occasionally during this minute.
- 6.7 Remove the buffer bottle from the titration vial, and slowly pour its contents into the reaction bottle (see figure 2.4).
- 6.8 Cap the reaction bottle and shake gently for a few seconds. As soon as the foam subsides, release the gas by loosening the cap. Tighten the cap and shake vigorously for 30 seconds. As before, release any gas that has formed, then turn the reaction bottle upside down (see figure 2.5).
- 6.9 Wait 1 minute.
- 6.10 While holding the filtering column in a vertical position, remove the plug. Gently tap the column to settle the carbon particles.
- 6.11 Keeping the reaction bottle upside down, insert the flip top into the end of the filtering column and position the column over the titration vial (see figure 2.6). Slowly squeeze the lower aqueous layer out of the reaction bottle and into the filtering column. Keep squeezing until the first drop of oil is squeezed out.

Note: The aqueous layer should flow through the filtering column into the titration vial in about one minute. In rare cases, it may be necessary to gently tap the column in order to begin the flow. The indicator paper should remain in the titration vial.

- 6.12 Cap the titration vial and shake it vigorously for ten seconds.
- 6.13 Slide the flexible end of the valve assembly over the tapered tip of the Titret so that it fits snugly (see figure 3.1).
- 6.14 Lift the control bar and insert the assembled Titret into the Titrettor™ (see figure 3.2).

- 6.15 Hold the Titrettor™ with the sample pipe in the sample and press the control bar to snap the pre-scored tip of the Titret (see figure 3.3).

Note: Because the Titret is sealed under vacuum, the fluid inside may be agitated when the tip snaps.

- 6.16 With the tip of the sample pipe in the sample, briefly press the control bar to pull in a SMALL amount of sample (see figure 3.3). The contents of the Titret will turn a purple color.

Note: During the titration there will be some undissolved powder inside the Titret, this does not interfere with the accuracy of the test.

- 6.17 Wait 30 seconds.
- 6.18 Gently press the control bar again to allow another SMALL amount of the sample to be drawn into the Titret.

Caution: Do not press the control bar unless the sample pipe is immersed in the sample. This prevents air from being drawn into the Titret.

# STANDARD LABORATORY P.O.CEDURE

Page 4 of 5

6.19 After each addition, rock the entire assembly to mix the contents of the Titret. Watch for a color change from purple to very pale yellow.

6.20 Repeat steps 6.18 and 6.19 until the color change occurs.

Note: The endpoint color change (from purple to pale yellow) actually goes through an intermediate gray color. During this intermediate stage, extra caution should be taken to bring in SMALL amounts of sample and to mix the Titret contents well.

6.21 When the color of the liquid in the Titret changes to PALE YELLOW, remove the Titret from the Titrettor™. Hold the Titret in a vertical position and carefully read the test result on the scale opposite the liquid level.

## 7. Calculation

7.1 To obtain readings in PPM ( $\mu\text{g}/\text{gram}$ ) Total Chlorine as Cl, multiply scale units on the Titret by 1.3 and then subtract 200.

## 8. Precision and Accuracy

8.1 In a single laboratory, ten different oil samples that had been independently prepared and standardized by Research Triangle Institute were analyzed for total chlorine. Unless otherwise specified, all results below are reported in PPM ( $\mu\text{g}/\text{gram}$ ) Chlorine as Cl.

Sample	Expected Result	Trials	Average	Precision as Standard Deviation	Accuracy as Bias	%Bias
No. 6 Fuel Oil	354	3	<775	--	N/A	N/A
No. 6 Fuel Oil	1568	3	1243	117	-325	-20.7
Used Crankcase Oil <sup>1</sup>	485	1	<775	--	N/A	N/A
No. 6 Fuel Oil	3155	4	2408	184	-747	-23.7
Metalworking Oil	2011	3	<775	--	>-1236	N/A
Metalworking Oil	27636	3	>8900	--	N/A	N/A
Metalworking Oil	303	4	<775	--	N/A	N/A
Used Crankcase Oil	1560	3	1317	75	-243	-15.6
Used Crankcase Oil	3148	3	2703	75	-445	-14.1
Used Crankcase Oil	890	3	905	57	15	1.7

<sup>1</sup> There was only enough sample in this case, to perform one test.

## BIBLIOGRAPHY

1. Brown, Jr., J.F.; Lynch, M.E.; Carnahan, J.C.; Singleton, J. "Chemical Destruction of PCB's in Transformer Oil"; Paper presented at the symposium on Detoxication of Hazardous Waste. 186th National Meeting of the American Chemical Society, New York, 26-28 August 1981.
2. APHA Standard Methods, 15th ed., p.271, method 4078(1980).
3. 1979 Annual Book of ASTM Standards, Part 31, method D-512, p.356.
4. EPA Methods for Analysis of Waters & Wastes, p.325.3-1, method 325.3(1979).



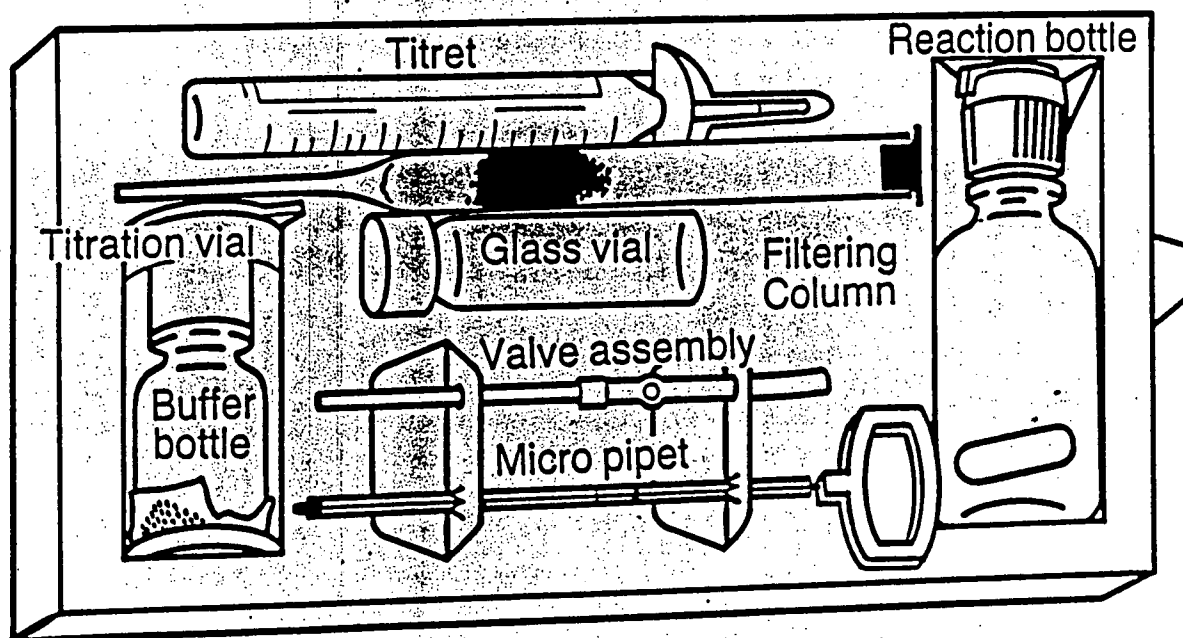


FIG.1 COMPONENTS OF CHEMetrics TOTAL CHLORINE IN WASTE OIL TEST KIT  
(Cat. No. K-2610)

(1973-3)

# STANDARD LABORATORY P..OCEDURE

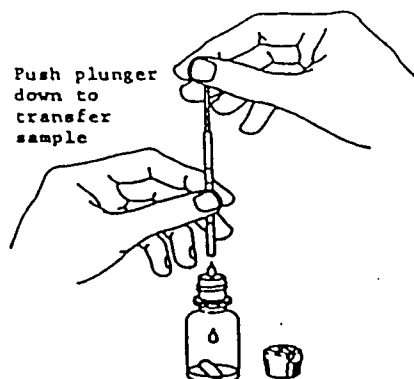


FIGURE 2.1

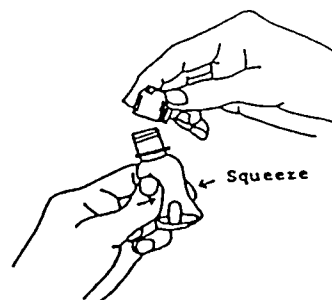


FIGURE 2.2

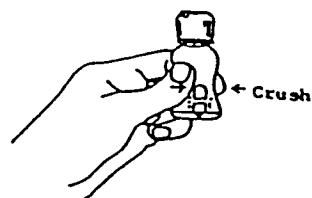


FIGURE 2.3

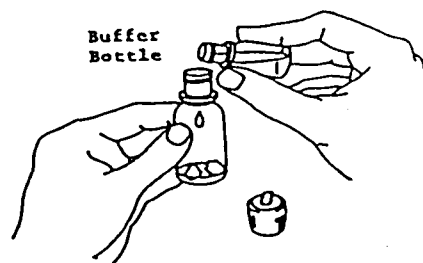


FIGURE 2.4

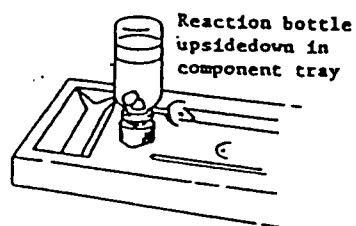


FIGURE 2.5

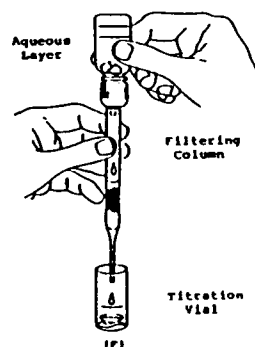


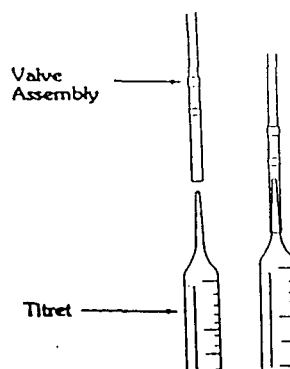
FIGURE 2.6

FIGS. 2.1 - 2.6 REACTION-EXTRACTION PROCEDURE

# STANDARD LABORATORY P.O.CEDURE

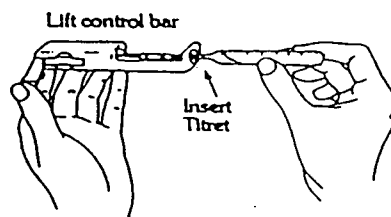
## Attaching the Valve Assembly

FIGURE 3.1



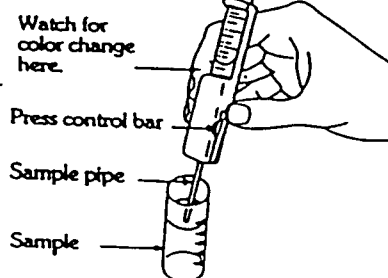
## Snapping the Tip

FIGURE 3.2



## Performing the Analysis

FIGURE 3.3



## Reading the Result

FIGURE 3.4

Read  
scale units  
when color  
changes  
permanently



FIGS. 3.1 - 3.4 TITRATION PROCEDURE

# Product Data Sheet

from CHEMetrics, Inc.  
Rt. 28, Calverton, VA 22016, (703) 788-9026  
Telex 292861 CHEM UR

CSP/987-2/QC

Test:	Quanti-Chlor™ Total Chlorine in Waste Oil
References:	Brown Jr., J.F.; et al. "Chemical Destruction of PCB's in Transformer Oil"; Presented at the American Chemical Society, New York, August 1981. APHA Standard Methods, 15th ed., p. 271, method 4078 (1980). ASTM Part 31 on Water, p. 356, method D-512 part A (1979). EPA Methods for Analysis of Waters & Wastes, p.325.3-1, method 325.2 (1979).
Description:	<p>Waste oils are often burned as the fuel source in non-industrial boilers. To combat potential health risks associated with the burning of contaminated oils in urban areas, the EPA promulgated regulations which establish safe levels of certain contaminants. As a result, the total chlorine content of waste oils which will ultimately be burned as fuel is limited to 1000 µg/g (ppm). With the Quanti-Chlor™ test, the analyst can readily determine whether the total chlorine content of the oil sample exceeds the established limit.</p> <p>The Quanti-Chlor™ test employs a chemistry in which organic chlorine is converted to inorganic chloride by reaction with a mixture of diglyme, naphthalene, and metallic sodium. An aqueous buffer is used to extract the inorganic chloride from the treated sample. Total chloride levels are then measured titrimetrically using mercuric nitrate as the titrant and diphenylcarbazone as the end point indicator. Disposable titration cells (U.S. Pat. No. 4,332,769) are used to simplify the titration procedure. The method covers the range of 750 to 7000 µg/g (ppm) Cl.</p>
Applications:	This test is applicable to the monitoring of total chlorine levels in light to heavy waste oils. Those involved in the regulation, collection, transport, recycling, and marketing of waste oils will find it an excellent screening device.
Advantages:	The Quanti-Chlor™ kit is the only quantitative, disposable, field portable test available for this application. It is simple to use, enabling non-technical personnel to obtain reliable results in less than 5 minutes.
Known Interferences:	Waste oils containing as much as 70% water give less than -10% bias. Higher water levels cause a greater negative interference.

# STANDARD LABORATORY PROCEDURE

## Shelf Life:

Item	Shelf-life	Conditions
Titret	virtually unlimited	none
Naphthalene	one year*	none
Diglyme	one year*	none
Iso-octane	one year*	none
Metallic Sodium	virtually unlimited	none
Buffer solution	one year*	none
Indicator	one year*	none

\*Based on currently available data. Actual shelf-life may be longer.

## Ordering Information:

To place an order, call or write:

CHEMetrics, Inc.  
Route 28  
Calverton, VA 22016  
(703) 788-9026  
Telex 292861 CHEM UR

### Quanti-Chlor™ Shelf-Packs\*

K-2610S  
(12 kits each)\*\*

Quantity	Price/Pack
1 - 3	\$87.00
4 - 15	78.00
16 - 31	74.00
32 +	70.00

### Quanti-Chlor™ Cases

K-2610C  
(48 kits each)\*\*

Quantity	Price/Case
1 - 3	\$312.00
4 - 7	296.00
8 +	280.00

\* For convenient stocking, kits are sold in shelf-packs and cases. Minimum order is one shelf-pack. Each shelf-pack contains a Free Titrettor™. Additional Titrettors may be purchased as required. Catalog Number A-0053, price \$3.00 each.

\*\* One test may be performed with each test kit.

## Safety Information:

To obtain safety information applicable to this product, request a copy of the following Material Safety Data Sheets:

The above information is believed to be accurate and represents the best information currently available to us. However, we make no warranty of merchantability or any other warranty express or implied.

Quadrex HPS Inc.

# STANDARD LABORATORY PROCEDURE

December 9, 1987

PROCEDURE NUMBER: 4000-001

TITLE: DETERMINATION OF SELECTED ORGANIC COMPOUNDS PRESENT IN  
HAZARDOUS INSTITUTIONAL MIXED WASTE

REVISION NUMBER:

PREPARED BY:

*Gary Boeth*  
Signature

*12/9/87*  
Date

APPROVED BY:

*Eugene T. McQuinn*  
Signature

*12.9.87*  
Date

## 1.0 PURPOSE

The purpose of this method is to quantitatively determine the organic compounds present in the mixed institutional waste as received from generators.

## 2.0 DEFINITIONS

Mixed Waste - any organic solvent (or mixture of solvents) listed under the "Industry and EPA Hazardous Waste"; numbers F001-F005, or characterized as a D001 ignitable solvent.

## 3.0 PRECAUTIONS

The following is a description of the "Toxicity Rating System" as described in: Haley, Thomas J., PhD, "Toxicology", Section 1 in Dangerous Properties of Industrial Materials, N. Irving Sax, Sixth Ed., (New York, NY: Van Nostrand Reinhold Company Inc., 1984), p. 1-2.

The appropriate toxicity rating (if available) for each specific chemical can be found in the procedure section, Table 1.

U = unknown

This rating covers chemicals for which insufficient data is available to enable a valid assessment of toxic hazard to be made. Such chemicals usually are in one of the following categories:

1. No information has been discovered in the literature, and none is known to the authors.
2. Limited information from animal experiments is available, but it is not possible to relate it to human exposure. The data is mentioned to indicate to the reader that animal experiments have been done.

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3. The validity of published to toxicity data was questioned by the authors.

None = no toxicity (0)

This designation is for chemicals that fall into the following categories:

1. Chemicals that produce no toxic effects under any conditions of normal usage.
2. Chemicals that require overwhelming dosage to produce any toxic effects in humans.

Low = slight toxicity (1)

1. Acute local. Chemicals that on a single exposure lasting seconds, minutes, or hours cause only slight effects on the skin or mucous membranes or eyes, regardless of the extent of exposure.
2. Acute systemic. Chemicals that can enter the body by inhalation, ingestion, or dermal contact and produce only slight toxic effects, regardless of the duration of exposure or following the ingestion of a single dose, regardless of the amount absorbed or the extent of the exposure.
3. Chronic local. Chemicals that on repeated or continuous exposure covering days, months, or years cause only slight and reversible damage to the skin or mucous membranes. The extent of the exposure can be great or small.
4. Chronic systemic. Chemicals that on repeated or continuous exposure covering days, months, or years cause slight and usually reversible toxic effects on the skin, mucous membranes, or eyes. The exposure can be by ingestion, inhalation, or skin contact and may be great or small. Slightly toxic chemicals produce changes readily reversible once the exposure ceases with or without medical intervention.

Mod = moderate toxicity (2)

1. Acute local. Chemicals that on a single exposure lasting seconds, minutes, or hours produce moderate toxicity to the skin, mucous membranes, or eyes. The effects can be the result of an intense exposure for seconds or a moderate exposure for hours.

# STANDARD LABORATORY PROCEDURE

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2. Acute systemic. Chemicals that after being absorbed by inhalation, ingestion, or skin contact produce moderate toxicity after a single exposure lasting seconds, minutes, or hours or after the ingestion of a single dose.
3. Chronic local. Chemicals that on continuous or repeated exposure over days, months, or years cause moderate toxicity to the skin, mucous membranes, or eyes.
4. Chronic systemic. Chemicals that on absorbed by ingestion, inhalation, or skin contact cause moderate toxicity after continuous or repeated exposures over days, months, or years.

Under the moderate classification are chemicals that can cause reversible or irreversible changes in the human body not necessarily severe enough to cause serious physical impairment or threaten life.

High = severe toxicity (3)

1. Acute local. Chemicals that on a single exposure covering seconds or minutes can cause injury to the skin, mucous membranes, or eyes of sufficient severity to threaten life, cause permanent physical impairment or disfigurement.
2. Acute systemic. Chemicals that after a single exposure by inhalation, ingestion, or skin contact cause injury of sufficient severity to threaten life. The exposure may last seconds, minutes, or hours or may be a single ingestion.
3. Chronic local. Chemicals that on continuous or repeated exposures covering days, months, or years can cause injury to the skin, mucous membranes, or eyes of sufficient severity to threaten life or produce permanent impairment, disfigurement, or irreversible change.
4. Chronic systemic. Chemicals that on continuous or repeated exposures by inhalation, ingestion, or dermal contact of small amounts for days, months, or years can produce death or serious physical impairment.

## 4.0 EQUIPMENT AND MATERIALS

Hewlett-Packard Gas Chromatograph with FI Detector or equivalent.

Chromatographic column: HP "PONA" Capillary Column



# STANDARD LABORATORY PROCEDURE

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## 5.0 DOCUMENTATION

None

## 6.0 PROCEDURE

### 6.1 SAMPLE PREPARATION

1. Transfer an aliquot of the solvent waste to a crimp top vial.
2. Label vial and analyze.

### 6.2 ANALYTICAL PROCEDURE

#### GAS CHROMATOGRAPH CONDITIONS

##### Run Parameters

Zero - 10  
Att. - 2  
Chart Speed - 0.5  
PK. Width - 0.02  
Thresh. - 2  
Ar. Rej. - 0

##### Temperatures

Initial Temp. - 45° C  
Initial Time - 10 min.  
Rate - 1.5° C/min.  
Final Temp. - 70° C  
Final Time - 0  
Ramp A Rate - 3.0° C/min.  
Final Temp. - 175° C/min.  
Final Time - 10 min.

Injection Port Temp. - 225° C/min.

Detector Temp. - 350° C/min.

##### Gas Flows

Carrier (He) -  $u = 12$  cm/sec. or  $2.3 \text{ cm}^3/\text{min.}$   
Makeup (He) -  $30 \text{ cm}^3/\text{min.}$   
Hydrogen -  $40 \text{ cm}^3/\text{min.}$   
Air -  $400 \text{ cm}^3/\text{min.}$   
Split Ratio - 1:25

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## CALIBRATION

A calibration curve for each chemical of interest must be established prior to the analysis of a mixed waste sample. The calibration concentrations should be inclusive of the concentration of the samples of interest. A minimum of 3 concentration levels should be prepared. The minimum concentration should be close, but slightly above the minimum level of detection (MLD) for that compound. If the response factor ( $R_f = \text{weight of analyte/area of integration}$ ) for the calibration standards are  $< 10$  relative standard deviation ( $RSD = [\text{Std. Dev.}/\text{Avg. } R_f] * 100$ ), a single point calibration factor may be used.

The calibration will be performed by external calibration. It will be at the analyst discretion to calibrate using the current complete group of compounds; or based on the waste stream, prepare an "unique" calibration standard. Regardless of the chemicals chosen for analysis, reference peaks must be designated through out the chromatographic run.

Criterion for selecting reference peaks are as follows: the reference peaks should be prominent and isolated; multiple reference peaks are preferable to a single one. If a single reference peak is chosen, it should occur later than the middle of the run; reference peaks should be spaced evenly throughout the run, if multiples.

The working calibration curve or  $R_f$  must be validated each day by measuring one or more of the calibration standards. If the response varies more than 10%, the calibration must be repeated using a fresh calibration standard, or a new calibration curve or  $R_f$  must be generated.

Table 1 contains the current list of chemicals designated in the F001-F005 series of industrial waste solvents. The table lists the approximate retention time for chemical detection. The table also lists the oven temperature at which detection occurs. Also listed are the chemical's actual boiling point, density, flash point, and toxicity code.

Figure 1 is a typical chromatogram including most of the compounds listed in Table 1. Missing from the chromatogram is the following: Ethanol, n-Butyl Alcohol, Methyl Pyrrole, Isoamyl Alcohol, Nitrobenzene, and the isomers of Cresol.

# STANDARD LABORATORY PROCEDURE

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## CONCENTRATION DETERMINATION

1. Determine the response factor for each of the compounds present in the standard chromatogram.

$$Rf = \frac{\text{ng. injected from the std}}{\text{area of integration}}$$

$$\text{Concentration (ug/ml)} = \frac{\text{Area from sample}}{\text{ul injected}} \times Rf$$

## 7.0 QUALITY CONTROL

### 7.1 BLIND SUBMITTAL

Ongoing performance checks must be performed on a routine basis. The check will consist of submitting to the analyst "blind" QC spiked samples containing all or part of the compounds of interest. The percent recovery (R) and the standard deviation of the percent recovery (s) noted at each concentration level.

### 7.2 INSTRUMENT MODIFICATIONS

All instrument changes or modifications must be noted in the appropriate instrument log.

### 7.3 INSTRUMENT PERFORMANCE

All raw and processed data must be maintained in its appropriate location. The data is used to perform periodical instrument performance audits.

### 7.4 DATA REPORTING

Analytical results will be reported in a timely manner to the person or group originating the sample (unless otherwise stated).

The data will be reported generally to two significant figures. The report will also indicate the MLD for that set of results.

### 7.5 SAMPLE DISPOSITION

The samples will generally be maintained for a period of three months from the time of reporting. At this time they will be disposed appropriately.

Further QA/QC measures may be found in QHPS Laboratory Quality Control Manual.

# STANDARD LABORATORY PROCEDURE

TABLE 1

15-Oct-87

## CHARACTERISTICS OF CURRENT CHEMICALS OF INTEREST

\*\*\*\*\*

CHEMICAL NAME	RT	COLUMN TEMP. (C)	ACTUAL BP (C)	TOXICITY CODE	DENSITY (g./ml.)	FLASH POINT (C)	SOLUBILITY IN WATER
METHANOL	7.2	45	64.7	3-2-1	0.698	12	MISC
ETHANOL	8.1	45	78.32	3	0.7893	13.1	MISC
ACETONE	8.7	45	56.29	2-1	0.79	-18	MISC
TRICHLOROFLUOROMETHANE	9	45	24.1	2-1	1.484	NONE	2.5?
ETHYL ETHER	9.4	45	34.6	2	0.7135	-45	@20, 6.9%
METHYLENE CHLORIDE	9.9	45	39.8	3	1.326	NONE	@20, 1.6%
1,1,2-TRICHLOROTRIFLUOROETHANE	10.3	45	47.7		1.5635	NONE	@25, .017%
CARBON DISULFIDE	10.5	45.8	46.5	3	1.2632	-30	MISC
METHYL ETHYL KETONE	12.4	48.6	79.57	3	0.8049	-1	@ 20, 24%
TRICHLOROETHYLENE	12.7	49.1	86.7	3	1.4762	NONE	@25, .1%
HEXANE	12.8	49.2	69	2-1	0.6603	-26	INSOLUBLE
ETHYL ACETATE	13.7	50.6	77.15	2-1	0.9006	-4	@20, 8.7%
TRICHLOROMETHANE, (chloroform)	13.8	50.7	61.26	3	1.4832	NONE	@20, .8%
HEXANE	13.8	50.7	69	2-1	0.6603	-26	INSOLUBLE
ISOBUTYL ALCOHOL	14.7	52.1	99.2		0.7982	28	@20, 8.7%
HEXANE	15.6	53.4	69	2-1	0.6603	-26	INSOLUBLE
1,1,1-TRICHLOROETHANE	16.2	54.3	74.1	2-1	1.339	NONE	INSOLUBLE
n-BUTYL ALCOHOL	17.3	56	117.5	3-2	0.8097	23.9	@20, 12.5
BENZENE	17.4	56	80.1	3	0.8787	-11	@25, .18%
CARBON TETRACHLORIDE	18	57	76.8	3	1.594	NONE	@20, .08%
2-NITROPROPANE	19.1	58.7	120	3-2	0.9876	88	SLIGHT
ISOOCTANE	20.9	61.4	132	3	0.698	-12	INSOLUBLE
2-ETHOXYETHANOL	21.9	62.9	135.1	2	0.9297	44	MISC
METHYL PYRROLE	24	66	112		0.9	16	INSOLUBLE
PYRIDINE	24	66	115.3	2	0.9832	20	@20, 100%
METHYL ISOBUTYL KETONE	24.2	66.3	118	3-2	0.8008	23	@20, 1.9%
ISOAMYL ALCOHOL	24.2	66.3	107.9	3	0.813	42.8	SLIGHTLY
1,1,2-TRICHLOROETHANE	26.7	70.1	114	3	1.4397	NONE	INSOLUBLE
TOLUENE	27.9	73.9	110.4	3	0.8669	6	@20, .05%
TETRACHLOROETHYLENE	33.2	89.8	121.2	3	1.6311	NONE	@20, .015%
CHLOROBENZENE	36	98.2	131.69		1.1058	30	@20, .1%
ETHYL BENZENE	37.8	103.6	132.6	2-1	0.8669	15	@20, .015%
p-XYLENE	38.6	106	138.3	3-2	0.8611	27.2	INSOLUBLE
m-XYLENE	38.7	106.3	139	3-2	0.8642	27.2	INSOLUBLE
CYCLOHEXANONE	39.3	108.1	115.6	2	0.9478	43.9	@20, 15%
o-XYLENE	40.7	112.3	144.4	3-2	0.8802	32.2	INSOLUBLE
1,3-DICHLOROBENZENE	49.7	139.3	173.4	3	1.2884	63.3	INSOLUBLE
1,2-DICHLOROBENZENE	52.1	146.5	180.5	3	1.3048	66	INSOLUBLE
o-CRESOL	52.3	147.1	108.5	3	1.047	81.1	SOLUABLE
m-CRESOL	53.6	151	108.5	3	1.034	94.4	SLIGHT
NITROBENZENE	54.5	153.7	210	3-2	1.205	87.8	SLIGHT
TETRADECANE	59.1	167.5	253	3	0.7628	99	INSOLUABLE
p-CRESOL			201.8	3	1.034	94.4	SLIGHT



QUADREX HPS INC. INSTITUTIONAL WASTE

Sample Collection/Request For Analysis/Lab Data-Form

Directions: Sections A - I to be filled out at time of Sample Collection

Sections J - Q to be filled out by the laboratory

A: Generator Name\_\_\_\_\_

B: Waste Profile Code\_\_\_\_\_

C: QHPS Sample Code\_\_\_\_\_

D: Collection Date\_\_\_\_\_

E: Collection Time\_\_\_\_\_

F: Responsible:\_\_\_\_\_

G: Location:\_\_\_\_\_

H: Comments:\_\_\_\_\_

Physical Characteristics of Waste

I: Color\_\_\_\_\_

J: Odor:\_\_\_\_\_

K: Layers ?\_\_\_\_\_

L: Moisture Content:\_\_\_%

M: Specific Gravity\_\_\_\_\_g/ml

N: Flash Point\_\_\_\_\_°C

O: pH:\_\_\_\_\_

P: Other\_\_\_\_\_

Q: Chemical Composition:

_____	_____%
_____	_____%
_____	_____%
_____	_____%
_____	_____%
_____	_____%
_____	_____%



QUADREX HPS INC. INSTITUTIONAL WASTE

Sample Collection/Request For Analysis/Lab Data-Form

Chemical Composition Continued:

_____	_____ %
_____	_____ %
_____	_____ %
_____	_____ %
_____	_____ %
_____	_____ %
_____	_____ %
_____	_____ %
_____	_____ %
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_____	_____ %
_____	_____ %
_____	_____ %
_____	_____ %

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

GENERATOR
Waste

QUADREX HPS INC. INSTITUTIONAL WASTE  
Waste Stream Information and Management

GENERATOR
Waste Characterization And Profile

WASTE DISPOSAL
Generator / Broker
Contact QHPS Seeking "Approval For Disposal"
Include Anticipated Waste Stream Types and Quantity

QHPS
Waste Approval
Lab
<div style="display: flex; justify-content: space-between;"> <span>No</span> <span>Is Waste "F" Series?</span> <span>Yes</span> </div>

QHPS
Waste Disapproval
Lab
Generate Disapproval Number; Comment Reason(s). Notify Generator/Broker

QHPS
Waste Approval
Lab
<div style="display: flex; justify-content: space-between;"> <span>No</span> <span>Is Waste "D001" ?</span> <span>Yes</span> </div>

QHPS
Waste Approval
Lab
Generate Approval Number; Notify Generator/Broker
Log Information to Database; Notify Processing Group of Valid Approvals



