130972

Attachment 4

13 Bulk Density[1]

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13–1 GENERAL INTRODUCTION

Soil *bulk density*, ρ_b, is the ratio of the mass of dry solids to the bulk volume of the soil. The bulk volume includes the volume of the solids and of the pore space. The mass is determined after drying to constant weight at 105 °C, and the volume is that of the sample as taken in the field.

Bulk density is a widely used value. It is needed for converting water percentage by weight to content by volume, for calculating porosity and void ratio when the particle density is known, and for estimating the weight of a volume of soil too large to weigh conveniently, such as the weight of a furrow slice or an acre-foot.

Bulk density is not an invariant quantity for a given soil. It varies with structural condition of the soil, particularly that related to packing. For this reason it is often used as a measure of soil structure. In swelling soils it varies with the water content (Hartge, 1965, 1968). In such soils, the bulk density obtained should be accompanied by the water content of the soil at the time of sampling.

The determination usually consists of weighing and drying a soil sample, the volume of which is known (core method) or must be determined (clod method and excavation method). These methods differ in the way the soil sample is obtained and its volume determined. A different principle is employed with the radiation method. Transmitted or scattered gamma radiation is measured; and with suitable calibration, the density of the combined gaseous-liquid-solid components of a soil mass is determined. Correction is then necessary to remove the components of density attributable to liquid and gas that are present. The radiation method is an in situ method.

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Clod and core methods have been used for many years. Excavation methods were developed in recent years, chiefly by soil engineers for bituminous and gravelly material. More recently the excavation method has found use in tillage research, or where surface soil is often too loose to allow core sampling, or where abundant stones preclude the use of core samplers. Radiation methods have been used since the 1950s, particularly in soil engineering.

Bulk density is expressed in SI units or units derived from them. The most straightforward would be kg m^{-3}. However, derived units such as tons m^{-3}, g cm^{-3}, or Mg m^{-3}, which are numerically equal to each other, may be more convenient, as they give values for soils which vary from about 1.2 to 1.7 (rather than from 1200 to 1700, as when units of kg m^{-3} are used). Obsolete terms such as "volume weight" (weight · $volume^{-1}$) and "bulk specific gravity" or "apparent specific gravity" are sometimes found in the older literature and in some foreign language literature. Specific gravity terms are relative densities, i.e. density of a substance with respect to water at 4 °C, and are nearly equal numerically to bulk density. At standard gravitation (g = 9.8 m s^{-2}), kilogram weight and kilogram mass are equal, and under this condition "volume weight" is numerically equal to bulk density. In many engineering and commercial applications, bulk density is expressed in lb ft^{-3}, which one may convert to g cm^{-3} by dividing by 62.4 (which is the mass, in pounds, of a cubic foot of a substance whose density is unity, i.e., water at 4 °C).

13–2 CORE METHOD

13–2.1 Introduction

With this method, a cylindrical metal sampler is pressed or driven into the soil to the desired depth and is carefully removed to preserve a known volume of sample as it existed in situ. The sample is dried to 105 °C and weighed. The core method is usually unsatisfactory if more than an occasional stone is present in the soil.

13–2.2 Method

Core samplers vary in design from a thin-walled metal cylinder to a cylindrical sleeve with removable sample cylinders that fit inside. Samplers are usually designed not only to remove a relatively undisturbed sample of soil from a profile, but also to hold the sample during transport and eventually during further measurements in the laboratory, such as pore-size distribution or hydraulic conductivity. For the latter measurement it is desirable to have core diameters not less than 75 mm and preferably 100 mm to minimize the effect of disturbed soil interfacing the cylinder wall. For the same reason it is desirable that the height of the cylinder not exceed the diameter.

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Fig. 13–1. Typical double-cyli...
for bulk density.

A widely used and very satisfactory sampler consists of two cylinders fitted one inside the other. The outer one extends above and below the inner to accept a hammer or press at the upper end and to form a cutting edge at the lower. The inside cylinder is the sample holder. The inside diameters of the two cylinders when nested are essentially the same at the lower end, the inner being fitted against a shoulder cut on the inner surface of the outside cylinder. Figure 13–1 shows such a sampler (available in slightly different design from the Utah State University Technical Services, UMC 12, Logan, UT 84322).

Where densities at various depths in a soil profile are to be determined, one can obtain samples with a hydraulically driven probe mounted on a pickup truck, tractor, or other vehicle. The probe is forced into the soil and removed hydraulically. The probe tube has a 2- to 3-cm wide slit running most of the length of the tube, through which one can insert a rounded knife or spatula to slice off segments of the soil as desired. Segments typically 10 cm in length are cut and removed from the tube and placed in containers for transport to the laboratory. Depending on the probe model, samples can be taken to about 1-m depth, though extensions for greater depths are available for many models. Probe samplers are available from Giddings Machine Co., P.O. Drawer 2024, 401 Pine St., Fort Collins, CO 80522; A. D. Bull Enterprizes, 1904 South 21st Street, Chickasha, OK 73018; or Soiltest Inc., 2205 Lee St., Evanston, IL 60202.

Fig. 13–1. Typical double-cylinder, hammer-driven core sampler, for obtaining soil samples for bulk density.

Numerous hand-driven samplers have been described in the literature. Some of the more accessible ones are described by Lutz (1947), Jamison et al. (1950), and U.S. Department of Agriculture (1954, p. 159). McIntyre (1974) describes types of core samples and their properties and gives additional references.

13-2.2.1 PROCEDURE

The exact procedure for obtaining the samples depends on the kind of sampler used. The following steps apply when the widely known double-cylinder sampler is used.

Drive or press the sampler into either a vertical or horizontal soil surface far enough to fill the sampler, but not so far as to compress the soil in the confined space of the sampler. Carefully remove the sampler and its contents so as to preserve the natural structure and packing of the soil as nearly as possible. A shovel, alongside and under the sampler, may be needed in some soils to remove the sample without disturbance. Separate the two cylinders, retaining the undisturbed soil in the inner cylinder. Trim the soil extending beyond each end of the sample holder (inner cylinder) flush with each end with a straight-edged knife or sharp spatula. The soil sample volume is thus established to be the same as the volume of the sample holder. In some sampler designs, the cutting edge of the sampler has an inside diameter slightly less than the sample holder, so as to reduce friction as the soil enters the holder. In these cases, determine the diameter of the cutting head and use this to calculate the sample holder volume. Transfer the soil to a container, place it in an oven at 105 °C until constant weight is reached, and weigh it. The bulk density is the oven-dry mass of the sample divided by the sample volume.

13-2.2.2 COMMENTS

It is not necessary that soil be kept undisturbed during transport to the laboratory and drying. A single sample cylinder can be reused if each sample is transferred to another container. It is often desired, however, to make other measurements such as pore-size distribution, conductivity, or water retention in addition to bulk density on the same samples. These require that they be kept undisturbed, each sample being transported in the sample cylinder in which it was taken. Thus one must provide for sufficient cylinders. Frequently other measurements to be made in the laboratory require that samples be kept at field water-content. In that case cylinders must be placed in containers that do not permit loss of water during transport. Waxed paper or plastic containers with lids are satisfactory for this purpose.

Core samples should be taken in soils of medium water content. In wet soils, friction along the sides of the sampler and vibrations due to hammering are likely to result in viscous flow of the soil and thus in compression of the sample. When this occurs the sample obtained is unrepresentative, being more dense than the body of the soil. Compres-

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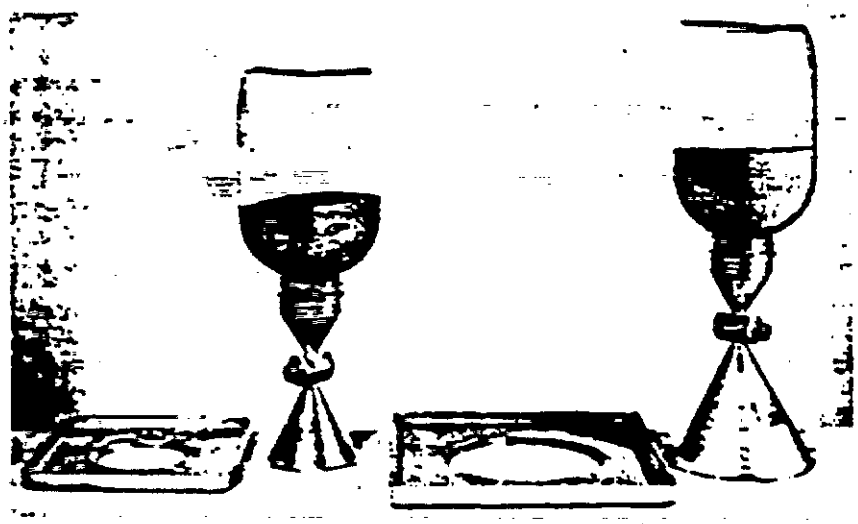

Fig. 13–2. Apparatus for sand-funnel technique of determining soil bulk density in place.

13–3.2.1.2. Rubber-Balloon Apparatus.

1. A thin-walled rubber balloon (may be purchased from Barr Inc., 1531 First Street, Sandusky, OH 44870, and the Anderson Rubber Co., 310-T N. Howard Street, P.O. Box 170 Akron, OH 44309).
2. A 1000-cm^3 graduated cylinder and a water container.
3. A template, described in section 13–3.2.1.1 (3).

Rubber-balloon density apparatus is available from several manufacturers supplying soil testing equipment. (One supplier is Soiltest, Inc., 2205 Lee Street, Evanston, IL 60202.) The apparatus made commercially has the convenience of a volumetrically calibrated water container-dispenser, with suction facilities for returning the water to the container for re-use (Fig. 13–3).

13–3.2.1.3 Mensuration Apparatus.

1. Tape measure marked in millimeters.
2. Flat metal plate approximately 50 cm square, with 30 to 40 evenly spaced holes forming a grid through which the tape measure can be inserted. Alternatively, 3 wooden beams 3 by 3 by 80 cm with 5-cm markings may be used.
3. Four wood stakes approximately 10 cm long, one end sharpened.
4. Scales to weigh to approximately 10 g.

13–3.2.2. PROCEDURE

13–3.2.2.1. Sand-Funnel Procedure. Level the soil surface and remove loose soil at the test site. Place the template on the soil. Excavate a soil sample through the center hole of the template, leaving a hole with a diameter of approximately 12 cm and a depth of approximately 12 cm,

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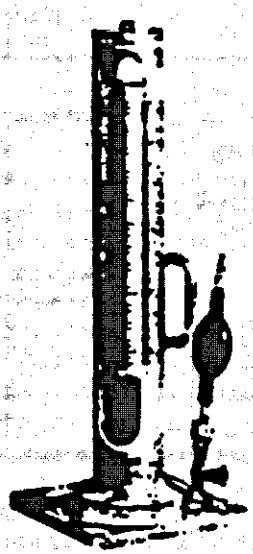

Fig. 13-3. Apparatus for determining soil bulk density in place by the rubber-balloon technique.

or other value as desired. A large spoon is convenient for excavating. Recover all evacuated soil in a container, being careful to include any loose soil that has fallen in from the sides of the excavation. Determine the oven-dry soil mass including stones by drying the soil to 105 °C and weighing it.

Determine the volume of the test hole by filling it with sand to the level of the bottom of the template. Level the sand with a spatula if necessary, but disturb it as little as possible to avoid packing the free-flowing sand. (Dispensing the sand through a funnel placed on the template, as is done with commercially available equipment, avoids the problem of leveling the sand. The excavation as well as the funnel is filled by free flow of sand, the predetermined weight required to fill the funnel being subtracted as a tare.)

Determine the weight of sand required to fill the test excavation by weighing it to the nearest 5 g. Precalibrate the mass-to-volume ratio of sand by letting sand fall from a similar height and at a similar rate of flow as in the test procedure. Using the calibration curve or values derived from it, determine the volume of the excavation from the measured mass of sand dispensed.

13-3.2.2.2. Rubber-Balloon Procedure. Level the soil surface, place the template on the surface, and excavate a soil sample as described in the preceding section. Place the rubber balloon in the test hole and fill the balloon with water to the bottom of the template. Determine the volume of water required to the nearest 2 cm^3. (A 1000-cm^3 graduate has markings to 10 cm^3, but one can estimate to 2 cm^3 if the graduate is placed on a horizontal surface.)

Table 13-1. Comparison of surface nuclear gauge and sand-cone method for determining soil bulk density (Mintzer, 1961).

Soil material	Number of comparisons	Mean difference	Extreme difference
		% †	
	Wet bulk density, ρ_w		
Brown sand, trace silt	9	2.03	−0.25–4.28
Brown silt and clay	4	0.89	−0.64–1.87
Brown sand and gravel, some silt, trace clay	6	1.01	−2.27–7.75
Brown till	4	−1.19	−3.97–1.60
	Dry bulk density, ρ_b		
Brown sand, trace silt	9	2.06	−1.21–4.96
Brown silt and clay	4	7.21	5.81–9.27
Brown sand and gravel, some silt, trace clay	6	1.48	−1.71–8.32
Brown till	4	−0.36	−4.83–3.21

† A positive value indicates nuclear method gave higher bulk density value.

in measuring water volumes or sand weights are insignificant. The disadvantage is the lack of discrimination to a localized horizon. An error of 5 cm^3 in liquid volume will give an error of 0.005 in a sample having a bulk density of 1.36 g cm^{-3}. Though one determines the water dispensed to perhaps 2 cm^3, a much greater source of error in water measurement arises in determining when the water in the excavation is level with the bottom of the template. Extreme care and judgment are required in this. The volume of the balloon itself, being of the order of 2 cm^3, will give a considerably smaller error than the volume measurement, and can be neglected.

An error of 7 g in weighing the sand gives an error of 0.005 if the bulk density is 1.36 g cm^{-3}. As in the balloon technique, greater error is likely to result in the precision with which one can determine the sand level at the bottom of the template. An error of 1 mm in this level will result in an error of 0.01 in the bulk density. Extreme care is therefore required to assure that the sand level is at the template bottom. The need for the dispensing funnel in reducing this error is obvious.

If one assumes an excavation of 30 by 30 by 10 cm in the mensuration method and measures depth 36 times on a 5-cm grid, assuming a cumulative error of 1 mm in each of the depth measurements the error in volume measurement is 1%.

A comparison of the sand-funnel and radiation methods was made by Mintzer (1961), and the results are summarized in Table 13–1.

13–4 CLOD METHOD

13–4.1 Introduction

The bulk density of clods, or coarse peds, can be calculated from their mass and volume. The volume may be determined by coating a

clod of known weight with a water-repellent substance and by weighing it first in air, then again while immersed in a liquid of known density, making use of Archimedes' principle. The clod or ped must be sufficiently stable to cohere during coating, weighing and handling.

13–4.2 Method

13–4.2.1 SPECIAL APPARATUS

1. A balance, modified to accept the clod suspended below the balance arm by means of a nylon thread or thin wire, to allow weighing the clod when it is suspended in a container of liquid.
2. A fine nylon thread or 28 to 30 gauge wire, to attach the clod to the balance. A fine nylon hairnet makes a good container for the clod.
3. Saran solution. Dissolve 1 part by weight of Saran resin (Dow Saran F-310; Dow Chemical Co., Suite 500/ Tower No. 2, 1701 West Golf Road, Rolling Meadows, IL 60008) in 7 parts by weight of methylethyl ketone in a 1-gallon container in sufficient quantities to fill the container about three-quarters full. Manufacturer's instructions for safe handling of solvents should be carefully followed. Dissolution requires about an hour, with vigorous stirring. Since the solvent is flammable and explosive when its vapors are mixed with air, either hand-stirring or use of an air-driven stirrer should be employed in a well ventilated hood. The solution can be stored for long periods if kept in a tightly closed container to prevent evaporation of the solvent.

13–4.2.2 PROCEDURE

Secure the clod with two loops of the thread or wire, loops being at right angles to one another, leaving sufficient thread or wire to connect to the balance arm. Weigh the clod and thread. Holding it by the thread, dip the clod into the saran solution. Suspend it in air under a hood for 15 to 30 min to allow the solvent to evaporate. Repeat dipping and drying one or more times as needed, to waterproof the clod. Weigh the clod, with its coating and the thread. Weigh it again when it is suspended in water and note the water temperature. Determine the tare weight of the thread or wire. To obtain a correction for water content of the soil, break open the clod, remove an aliquot of soil, and weigh the aliquot before and after oven-drying it at 105 °C.

Calculate the oven-dry mass of the soil sample W_{ods} as follows, from the water content of the aliquot removed from the clod after other weights are taken:

$$W_{ods} = W_w/(1 + \theta_g)$$

where θ = water content of the subsample in g/g and W_w = net weight of clod or ped in air at its original water content.

Calculate bulk density as follows:

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W_{sw} = net wei
W_m = weight
ρ_p = density

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$$\rho_b = \rho_w W_{ods}/[W_a - W_{spw} + W_{ps} - (W_{ps}\rho_w/\rho_p)]$$

where

ρ_w = density of water at temperature of determination,
W_{ods} = oven-dry weight of soil sample (clod or ped),
W_a = net weight of clod or ped in air,
W_{spw} = net weight of soil sample plus saran in water,
W_{ps} = weight of saran coating in air, and
ρ_p = density of saran.

13–4.2.3 COMMENTS

The clod method usually gives higher bulk-density values than do other methods (Tisdall, 1951). One reason is that the clod method does not take the interclod spaces into account.

Extreme care should be exercised to get naturally occurring masses of soil. Clods on or near the soil surface are likely to be unrepresentative, for these are often formed by packing with tillage implements. Natural soil masses, or coarse peds, that are more representative should be sought.

If bubbles appear on the saran when the sample is weighed in water or if the weight in water increases with time, water is penetrating the clod, and the sample must be discarded.

Brasher et al. (1966) first proposed use of saran coating. They suggested that for clods with large pores, a more viscous saran solution of 1 part resin to as little as 4 parts methylethyl ketone could be used. They gave the density of saran to be 1.3 g cm^{-3}.

Precision in calculating the bulk density would require a correction for the difference of the weight of the wire in air and in water. However, the error is negligible with thread or a 28-gauge wire.

Using clods as small as 40 g oven-dry weight and weighing to 10 mg gives a standard deviation in the bulk density with 25 replications of a single measurement of 0.07 g cm^{-3} (Hartge, 1965). This can be reduced by using larger clods or by weighing to 1 mg or both. Obviously, using a greater number of samples for a determination would also reduce the standard deviation.

Several other substances have been used to seal the clod against water, including paraffin, rubber, wax mixtures, and oils.

13–5 RADIATION METHODS

13–5.1 Introduction

The transmission of gamma radiation through soil or scattering within soil varies with soil properties, including bulk density. By suitable calibration, measurements of either transmission or scattering of gamma radiation can be used to estimate bulk density.

cording to the way it is handled in actual sampling. If there is doubt, the equipment should be checked for safety by a competent testing laboratory.

Since radiation transmitted from a source to a detector is dependent on probe spacing or sample thickness, care must be exercised with the two-probe sampler to assure that access holes are parallel and spaced exactly as in the calibration.

Mintzer (1961) reported comparisons of the surface-density probe and the sand-cone method on four engineering projects. He reported his comparisons on both the wet and dry bulk-density bases. He used a surface neutron meter for water content where the surface-density probe was used. His results are summarized in Table 13–1.

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Attachment 6

Designation: D 854 – 83

American Association State Highway and Transportation Officials Standard AASHTO No. T 100

Standard Test Method for Specific Gravity of Soils[1]

This standard is issued under the fixed designation D 854; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers determination of the specific gravity of soils by means of a pycnometer. When the soil is composed of particles larger than the No. 4 (4.75-mm) sieve, the method outlined in Test Method C 127 shall be followed. When the soil is composed of particles both larger and smaller than the No. 4 sieve, the sample shall be separated on the No. 4 sieve and the appropriate test method used on each portion. The specific gravity value for the soil shall be the weighted average of the two values (Note 1). When the specific gravity value is to be used in calculations in connection with the hydrometer portion of Method D 422, it is intended that the specific gravity test be made on that portion of the soil which passes the No. 10 (2.00-mm) sieve.

NOTE 1—The weighted average specific gravity should be calculated using the following equation:

$$G_{avg} = \frac{1}{\frac{R_1}{100G_1} + \frac{P_1}{100G_2}}$$

where:

G_{avg} = weighted average specific gravity of soils composed of particles larger and smaller than the No. 4 (4.75-mm) sieve,
R_1 = percent of soil particles retained on the No. 4 sieve,
P_1 = percent of soil particles passing the No. 4 sieve,
G_1 = apparent specific gravity of soil particles retained on the No. 4 sieve as determined by Test Method C 127, and
G_2 = specific gravity of soil particles passing the No. 4 sieve as determined by this test method.

1.2 The values stated in acceptable metric units are to be regarded as the standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 127 Test Method for Specific Gravity and Absorption of Coarse Aggregate[2]
C 670 Practice for Preparing Precision Statements for Test Methods for Construction Materials[2]
D 422 Method for Particle-Size Analysis of Soils[3]
E 12 Definitions of Terms Relating to Density and Specific Gravity of Solids, Liquids, and Gases[4]

3. Definition

3.1 *specific gravity*—the ratio of the mass of a unit volume of a material at a stated temperature to the mass in air of the same volume of gas-free distilled water at a stated temperature (per Definitions E 12).

4. Significance and Use

4.1 The specific gravity of a soil is used in almost every equation expressing the phase relationship of air, water, and solids in a given volume of material.

4.2 The term "solid particles," as used in geotechnical engineering, is typically assumed to mean naturally occurring mineral particles that are not very soluble in water. Therefore, the specific gravity of materials containing extraneous matter (such as cement, lime, etc.), water-soluble matter (such as sodium chloride), and soils containing matter with a specific gravity of less than one, typically require special treatment or a qualified definition of specific gravity.

5. Apparatus

5.1 *Pycnometer*—Either a volumetric flask having a capacity of at least 100 mL or a stoppered bottle having a capacity of at least 50 mL (Note 2). The stopper shall be of the same material as the bottle, and of such size and shape that it can be easily inserted to a fixed depth in the neck of the bottle, and shall have a small hole through its center to permit the emission of air and surplus water.

NOTE 2—The use of either the volumetric flask or the stoppered bottle is a matter of individual preference, but in general, the flask should be used when a larger sample than can be used in the stoppered bottle is needed due to maximum grain size of the sample.

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

Current edition approved Nov. 28, 1983. Published January 1984. Originally issued as D 854 – 45. Last previous edition D 854 – 58 (1979).

[2] *Annual Book of ASTM Standards*, Vol 04.02.
[3] *Annual Book of ASTM Standards*, Vol 04.08.
[4] *Annual Book of ASTM Standards*, Vol 15.05.

5.2 *Balance*—Either a balance sensitive to 0.01 g for use with the volumetric flask, or a balance sensitive to 0.001 g for use with the stoppered bottle.

6. Calibration of Pycnometer

6.1 The pycnometer shall be cleaned, dried, weighed, and the weight recorded. The pycnometer shall be filled with distilled water (Note 3) essentially at room temperature. The weight of the pycnometer and water, W'_a, shall be determined and recorded. A thermometer shall be inserted in the water and its temperature T, determined to the nearest whole degree.

NOTE 3—Kerosine is a better wetting agent than water for most soils and may be used in place of distilled water for oven-dried samples.

6.2 From the weight W'_a determined at the observed temperature T, a table of values of weights W'_a shall be prepared for a series of temperatures that are likely to prevail when weights W'_b are determined later (Note 4). These values of W'_a shall be calculated as follows:

$$W'_a \text{ (at } T_x) = (\text{density of water at } T_x/\text{density of water at } T) \times (W'_a(\text{at } T) - W_f) + W_f$$

where:

W'_a = weight of pycnometer and water, g,
W_f = weight of pycnometer, g,
T = observed temperature of water, °C, and
T_x = any other desired temperature, °C.

NOTE 4—This method provides a procedure that is most convenient for laboratories making many determinations with the same pycnometer. It is equally applicable to a single determination. Bringing the pycnometer and contents to some designated temperature when weights W_a and W_b are taken, requires considerable time. It is much more convenient to prepare a table of weights W'_a for various temperatures likely to prevail when weights W_b are taken. It is important that weights W'_a and W_b be based on water at the same temperature. Values for the relative density of water at temperatures from 18 to 30°C are given in Table 1.

7. Sampling

7.1 The soil to be used in specific gravity test may contain its natural moisture or be oven-dried. The weight of the test sample on an oven-dry basis shall be at least 25 g when the volumetric flask is to be used, and at least 10 g when the stoppered bottle is to be used.

7.2 *Samples Containing Natural Moisture*—When the sample contains its natural moisture, the weight of the soil, W_s, on an oven-dry basis shall be determined at the end of the test by evaporating the water in an oven maintained at 230 ±9°F (110 ±5°C) (Note 5). Samples of clay soils containing their natural moisture content shall be dispersed in distilled water before placing in the flask, using the dispersing equipment specified in Method D 422 (Note 6).

7.3 *Oven-Dried Samples*—When an oven-dried sample is to be used, the sample shall be dried for at least 12 h, or to constant weight, in an oven maintained at 230 ±9°F (110 ± 5°C) (Note 5), cooled in a desiccator, and weighed upon removal from the desiccator. The sample shall then be soaked in distilled water for at least 12 h.

NOTE 5—Drying of certain soils at 110°C may bring about loss of moisture of composition or hydration, and in such cases drying shall be done, if desired, in reduced air pressure and at a lower temperature.

NOTE 6—The minimum volume of slurry that can be prepared by the dispersing equipment specified in Method D 422 is such that a 500-mL flask is needed as the pycnometer.

TABLE 1 Relative Density of Water and Conversion Factor K For Various Temperatures

Temperature, °C	Relative Density of Water	Correction Factor K
18	0.9986244	1.0004
19	0.9984347	1.0002
20	0.9982343	1.0000
21	0.9980233	0.9998
22	0.9978019	0.9996
23	0.9975702	0.9993
24	0.9973286	0.9991
25	0.9970770	0.9989
26	0.9968156	0.9986
27	0.9965451	0.9983
28	0.9962652	0.9980
29	0.9959761	0.9977
30	0.9956780	0.9974

8. Procedure

8.1 Place the sample in the pycnometer, taking care not to lose any of the soil in case the weight of the sample has been determined. Add distilled water to fill the volumetric flask about three-fourths full or the stoppered bottle about half full.

8.2 Remove entrapped air by either of the following methods: (*1*) subject the contents to a partial vacuum (air pressure not exceeding 100 mm Hg) or (2) boil gently for at least 10 min while occasionally rolling the pycnometer to assist in the removal of the air. Subject the contents to reduced air pressure either by connecting the pycnometer directly to an aspirator or vacuum pump, or by use of a bell jar. Some soils boil violently when subjected to reduced air pressure. It will be necessary in those cases to reduce the air pressure at a slower rate or to use a larger flask. Cool samples that are heated to room temperature.

8.3 Fill the pycnometer with distilled water, clean the outside and dry with a clean, dry cloth. Determine the weight of the pycnometer and contents, W_b, and the temperature in degrees Celsius, T_x, of the contents as described in Section 6.

9. Calculation and Report

9.1 Calculate the specific gravity of the soil, based on water at a temperature T_x, as follows:

$$\text{Specific gravity, } T_x/T_x = W_s/[W_s + (W_a - W_b)]$$

where:

W_s = weight of sample of oven-dry soil, g,
W_a = weight of pycnometer filled with water at temperature T_x (Note 7), g,
W_b = weight of pycnometer filled with water and soil at temperature T_x, g, and
T_x = temperature of the contents of the pycnometer when weight W_b was determined, °C.

NOTE 7—This value shall be taken from the table of values of W'_a prepared in accordance with 6.2, for the temperature prevailing when weight W_b was taken.

9.2 Unless otherwise required, specific gravity values reported shall be based on water at 20°C. The value based on water at 20°C shall be calculated from the value based on water at the observed temperature T_x, as follows:

Specific gravity, $T_x/20\,C = K \times$ specific gravity, T_x/T_x

where:

K = a number found by dividing the relative density of water at temperature T_x by the relative density of water at 20°C. Values for a range of temperatures are given in Table 1.

9.3 When it is desired to report the specific gravity value based on water at 4°C, such a specific gravity value may be calculated by multiplying the specific gravity value at temperature T_x by the relative density of water at temperature T_x.

9.4 When any portion of the original sample of soil is eliminated in the preparation of the test sample, the portion on which the test has been made shall be reported.

10. Precision and Bias

10.1 Criteria for judging the acceptability of specific gravity test results obtained by this test method on material passing the No. 4 (4.75-mm) sieve are given as follows (Note 8):

Material and Type Index	Standard Deviation[A]	Acceptable Range of Two Results (percent of mean)[A]
Single-operator precision:		
Cohesive soils	0.021	0.06
Noncohesive soils	[B]	[B]
Multilaboratory precision:		
Cohesive soils	0.056	0.16
Noncohesive soils	[B]	[B]

[A] These numbers represent, respectively, the (1S) and (D2S) limits as described in Practice C 670.

[B] Criteria for assigning standard deviation values for non-cohesive soils are not available at the present time.

NOTE 8—The figures given in Column 2 are the standard deviations that have been found to be appropriate for the materials described in Column 1. The figures given in Column 3 are the limits that should not be exceeded by the difference between the results of two properly conducted tests.

U.S. Environmental Protection Agency
CLP Sample Management Office
209 Madison Street, Alexandria, VA 22313
Phone: (703) 557-2490 or FTS 557-2490

SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Client: EPA Region III/CH2M HILL

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Raymark Site, Montgomery County, PA

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. Description of analytical service requested:

   Analyze 4 soil samples for cation-exchange capacity (sodium acetate) using Test Method 9081 followed by CLP metals analysis for sodium acetate according to most recent CLP SOW. Duplicate samples and aqueous blank samples for cation-exchange capacity (sodium acetate) analysis are not applicable. Method is attached.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

   Analyze 4 low concentration soil samples for cation-exchange capacity (sodium acetate) using Test Method 9081 from EPA Test Methods for Evaluating Solid Waste (SW846), 3rd Ed., November, 1986 followed by CLP metals analysis for sodium according to the most recent CLP SOW for inorganics analysis.

3. Purpose of Analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

   Superfund RI/FS.

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment:

   Samples shipped by overnight courier.

6. Approximate number of days results required after lab receipt of samples:

   Results of analyses are requested forty (40) days after receipt of samples by the laboratory.

7. Analytical protocol required (attach copy if other than protocol currently used in this program):

   Analyze all samples using Test Method 9081 found on pages 9081-1 to 9081-4 in "EPA Test Methods for Evaluating Solid Waste (SW846)", 3rd Ed., November 1986 followed by CLP metals analysis for sodium acetate according to most recent CLP SOW for Inorganics Analysis. Method attached.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detention limits, etc.):

   Samples must be analyzed within 30 days from the date of collection.
   Standardize equipment according to manufacturer's instructions.
   Detection limit must be 5 ug/kg or less.

9. Analytical results (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

   Test procedures and specific insrument used must be clearly identified. Bench records tabulating order of calibration of standards, lab blanks, samples, lab control standards, spikes, duplicates, etc., with resulting output on concentration readout must be provided along with worksheets used to calculate results. Specify the organic compound used to prepare the standards and spikes. A photocopy of the instrument readout, i.e., stripcharts, printer, tapes, etc., must be included. Results are to be recorded in ug/kg dry weight. Records of analysis and calculations must be legible and sufficient to recalculate all concentrations. EPA QC reference samples, or any other reference sample or identical calibration verification, must be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analytical results must be provided for all reference samples used.

   Data package must include: all raw data, all instrument and/or equipment calibration results, calculations, duplicate results, chain-of-custody forms, SAS request forms, SAS packing lists, copy of airbill(s), and copies of analyst's logbooks (signed by

analyst) with date and time of sample preparation and analysis.

The cover page and all sample report forms MUST be labelled with the complete EPA sample number as it appears on the chain-of custody and CLP paperwork. The case narrative must document all problems encountered and the subsequent resolutions.

10. Other (use additional sheets or attached supplementary information, as needed):

The awarded laboratory is responsible for meeting all requirements as specified in this client request. Any changes in method(s) or other specifications must be approved by Region III prior to the award. If these stipulations are not met, Region III will recommend review for liquidation damages.

11. Name of sampling/shipping contact:

Tom McLaughlin Phone: (703) 471-6405

12. Data Requirements

Parameter	Detection Limit	Prescision Limits (+/- % or conc.)
Sodium Acetate	5 ug/kg	Not Applicable

13. QC Requirements

Audits Required	Frequency of Audits	Limits (+/- % or conc.)

All laboratory QA/QC requirements must be performed and reported as specified in the CLP SOW for Inorganics Analysis (10/86). The procedures, frequencies and acceptance criteria used shall be the same as specified in the SOW.

14. Action Required if Limits are Exceeded:

Contact Colleen K. Walling, USEPA and Tom McLaughlin, CH2M HILL.

15. Request Prepared by: Laura Gavin/CH2M HILL

Date: February 8, 1990

16. Request reviewed by:

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

METHOD 9081

CATION-EXCHANGE CAPACITY OF SOILS (SODIUM ACETATE)

1.0 SCOPE AND APPLICATION

1.1 Method 9081 is applicable to most soils, including calcareous and noncalcareous soils. The method of cation-exchange capacity by summation (Chapman, 1965, p. 900; see Paragraph 10.1) should be employed for distinctly acid soils.

2.0 SUMMARY OF METHOD

2.1 The soil sample is mixed with an excess of sodium acetate solution, resulting in an exchange of the added sodium cations for the matrix cations. Subsequently, the sample is washed with isopropyl alcohol. An ammonium acetate solution is then added, which replaces the adsorbed sodium with ammonium. The concentration of displaced sodium is then determined by atomic absorption, emission spectroscopy, or an equivalent means.

3.0 INTERFERENCES

3.1 Interferences can occur during analysis of the extract for sodium content. Thoroughly investigate the chosen analytical method for potential interferences.

4.0 APPARATUS AND MATERIALS

4.1 Centrifuge tube and stopper: 50-mL, round-bottom, narrow neck.

4.2 Mechanical shaker.

4.3 Volumetric flask: 100-mL.

5.0 REAGENTS

5.1 Sodium acetate (NaOAc), 1.0 N: Dissolve 136 g of $NaC_2H_2O_2 \cdot 3H_2O$ in water and dilute it to 1,000 mL. The pH of this solution should be 8.2. If needed, add a few drops of acetic acid or NaOH solution to bring the reaction of the solution to pH 8.2.

5.2 Ammonium acetate (NH_4OAc), 1 N: Dilute 114 mL of glacial acetic acid (99.5%) with water to a volume of approximately 1 liter. Then add 138 mL of concentrated ammonium hydroxide (NH_4OH) and add water to obtain a volume of about 1,980 mL. Check the pH of the resulting solution, add more NH_4OH, as needed, to obtain a pH of 7, and dilute the solution to a volume of 2 liters with water.

5.3 Isopropyl alcohol: 99%.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

7.0 PROCEDURE

7.1 Weigh 4 g of medium- or fine-textured soil or 6 g of coarse-textured soil and transfer the sample to a 50-mL, round-bottom, narrow-neck centrifuge tube. (A fine soil has >50% of the particles <0.074 mm, medium soil has >50% >0.425 mm, while a coarse soil has more than 50% of its particles >2 mm.

7.2 Add 33 mL of 1.0 N NaOAc solution, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear.

7.3 Decant the liquid, and repeat Paragraph 7.2 three more times.

7.4 Add 33 mL of 99% isopropyl alcohol, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear.

7.5 Repeat the procedure described in Paragraph 7.4 two more times.

7.6 Add 33 mL of NH_4OAc solution, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear. Decant the washing into a 100-mL volumetric flask.

7.7 Repeat the procedure described in Paragraph 7.6 two more times.

7.8 Dilute the combined washing to the 100-mL mark with ammonium acetate solution and determine the sodium concentration by atomic absorption, emission spectroscopy, or an equivalent method.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.3 Materials of known cation-exchange capacity must be routinely analyzed.

9.0 METHOD PERFORMANCE

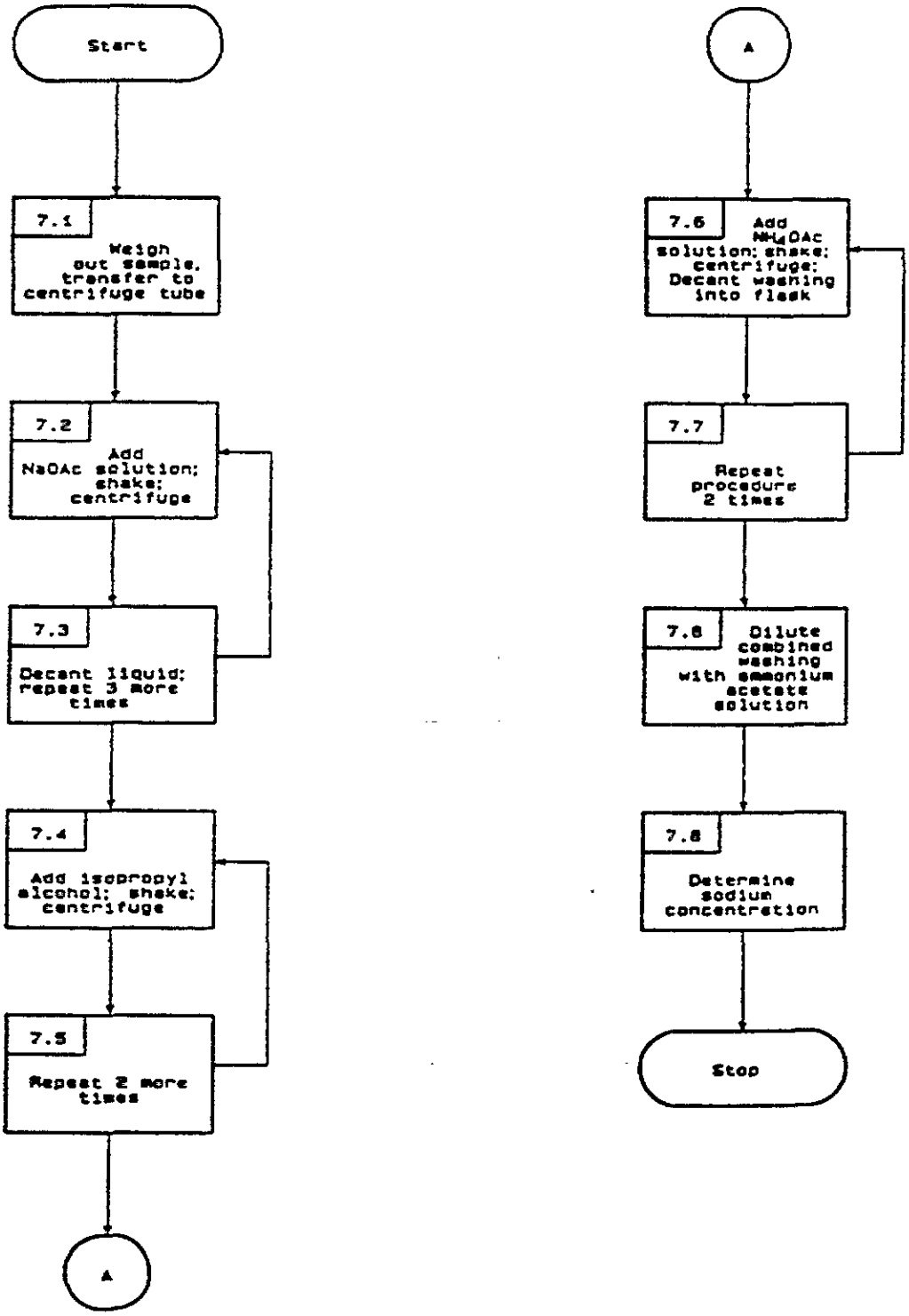
9.1 No data provided.

10.0 REFERENCES

10.1 This method is based on Chapman, H.D., "Cation-exchange Capacity," pp. 891-900, in C.A. Black (ed.), Method of Soil Analysis, Part 2: Chemical and Microbiological Properties, Am. Soc. Agron., Madison, Wisconsin (1965).

METHOD 9081

CATION-EXCHANGE CAPACITY OF SOILS (SODIUM ACETATE)



Revision 0
Date September 1986

U.S. Environmental Protection Agency
CLP Sample Management Office
209 Madison Street, Alexandria, VA 22313
Phone: (703) 557-2490 or FTS 557-2490

SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Client: EPA Region III/CH2M HILL

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Raymark Site, Montgomery County, PA

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. Description of analytical service requested:

   Analyze 5 soil samples for leachability of metals in soil by "ASTM Shake Extraction of Solid Waste With Water", Method Designation D 3987-85 followed by CLP metals analysis according to most recent CLP SOW. Aqueous blank samples for soil leachability analysis are not applicable. Method is attached.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

   Analyze 4 soil samples and 1 duplicate sample for leachability using ASTM Method Designation D 3987 - 85 followed by CLP metals analysis according to the most recent CLP SOW. Samples will be low level concentration. Method is attached.

3. Purpose of Analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

   Superfund RI/FS.

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment:

   Samples shipped by overnight courier.

6. Approximate number of days results required after lab receipt of samples:

   Results of analyses are requested forty (40) days after receipt of samples by the laboratory.

7. Analytical protocol required (attach copy if other than protocol currently used in this program):

   Analyze all samples using ASTM Method Designation D 3987 - 85 followed by CLP metals analysis according to most recent SOW. Method attached.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detention limits, etc.):

   Samples must be analyzed within 6 months days from the date of collection.
   Standardize equipment according to manufacturer's instructions.
   Detection limit according to most recent SOW or less.

9. Analytical results (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

   Test procedures and specific insrument used must be clearly identified. Bench records tabulating order of calibration of standards, lab blanks, samples, lab control standards, spikes, duplicates, etc., with resulting output on concentration readout must be provided along with worksheets used to calculate results. Specify the organic compound used to prepare the standards and spikes. A photocopy of the instrument readout, i.e., stripcharts, printer, tapes, etc., must be included. Results are to be recorded in ug/kg dry weight. Records of analysis and calculations must be legible and sufficient to recalculate all concentrations. EPA QC reference samples, or any other reference sample or identical calibration verification, must be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analytical results must be provided for all reference samples used.

   Data package must include: all raw data, all instrument and/or equipment calibration results, calculations, duplicate results, chain-of-custody forms, SAS request forms, SAS packing lists, copy of airbill(s), and copies of analyst's logbooks (signed by analyst) with date and time of sample preparation and analysis.

   The cover page and all sample report forms MUST be labelled with the complete EPA sample number as it appears on the chain-of

custody and CLP paperwork. The case narrative must document all problems encountered and the subsequent resolutions.

10. Other (use additional sheets or attached supplementary information, as needed):

The awarded laboratory is responsible for meeting all requirements as specified in this client request. Any changes in method(s) or other specifications must be approved by Region III prior to the award. If these stipulations are not met, Region III will recommend review for liquidation damages.

11. Name of sampling/shipping contact:

Tom McLaughlin Phone: (703) 471-6405

12. Data Requirements

Parameter	Detection Limit	Prescision Limits (+/- % or conc.)
Metals	CLP SOW (10/86)	+/- 20 %

13. QC Requirements

Audits Required	Frequency of Audits	Limits (+/- % or conc.)

All laboratory QA/QC requirements must be performed and reported as specified in the CLP SOW for Inorganics Analysis (10/86). The procedures, frequencies and acceptance criteria used shall be the same as specified in the SOW.

14. Action Required if Limits are Exceeded:

Contact Colleen K. Walling, USEPA and Tom McLaughlin, CH2M HILL.

15. Request Prepared by: Laura Gavin/CH2M HILL

Date: February 8, 1990

16. Request reviewed by:

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

Designation: D 3987 – 85

Standard Test Method for Shake Extraction of Solid Waste with Water[1]

This standard is issued under the fixed designation D 3987; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure for leaching of solid waste to obtain an aqueous solution to be used to determine the materials leached under the specified testing conditions.

1.2 This test method provides for the shaking of a known weight of waste with water of specified composition and the separation of the aqueous phase for analysis.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 471 Method for Chemical Analysis of Gypsum and Gypsum Products[2]

D 75 Practice for Sampling Aggregates[3]

D 420 Practice for Investigating and Sampling Soil and Rock for Engineering Purposes[3]

D 1129 Definitions of Terms Relating to Water[4]

D 1193 Specification for Reagent Water[4]

D 2216 Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures[3]

D 2234 Method for Collection of a Gross Sample of Coal[5]

D 3370 Practices for Sampling Water[4]

E 122 Practice for Choice of Sample Size to Estimate the Average Quality of a Lot or Process[6]

3. Definitions

3.1 For definitions of terms used in this test method, see Definitions D 1129.

4. Significance and Use

4.1 This test method is intended as a rapid means for obtaining an extract of solid waste. The extract may be used to estimate the release of certain constituents of the solid waste under the laboratory conditions described in this procedure.

4.2 This test method is not intended to provide an extract that is representative of the actual leachate produced from a solid waste in the field or to produce extracts to be used as the sole basis of engineering design.

4.3 This test method is not intended to simulate site-specific leaching conditions. It has not been demonstrated to simulate actual disposal site leaching conditions.

4.4 The intent of this test method is that the final pH of the extract reflect the interaction of the extractant with the buffering capacity of the solid waste.

4.5 The intent of this test method is that the water extraction simulate conditions where the solid waste is the dominant factor in determining the pH of the extract.

4.6 The test method produces an extract that is amenable to the determination of both major and minor constituents. When minor constituents are being determined, it is especially important that precautions are taken in sample storage and handling to avoid possible contamination of the samples.

4.7 This test method has been tested to determine its applicability to certain inorganic components in the solid waste. The test method has not been tested for applicability to organic substances and volatile matter (see 5.3).

4.8 The agitation technique, rate, and liquid-to-solid ratio specified in the procedure may not be suitable for extracting all types of solid wastes. (See Sections 7, 8, and the discussion in Appendix X1.)

5. Apparatus

5.1 *Agitation Equipment*, of any type that rotates about a central axis at a rate of 29 r/min, Fig. 1. (See discussion of agitation in Appendix X1.)

5.2 *Membrane Filter Assembly*—A borosilicate glass or stainless steel funnel with a flat, fritted base of the same material and membrane filters.

5.3 *Containers*, round, wide-mouth, of a composition suitable to the nature of the solid waste and the analyses to be performed, and constructed of materials that will not allow sorption of constituents of interest. One-gallon (or 4-L) containers should be used with 140-g samples and ½-gallon (or 2-L) containers with 70-g samples. Multiples of these sizes may be used for larger samples. The containers should be of the same approximate geometry as the 2-L and 4-L bottles. These sizes were selected to establish suitable geometry and provide that the sample plus liquid would occupy approximately 80 to 90 % of the container. Containers must have a watertight closure. Containers for samples where gas may be released should be provided with a venting mechanism. (Note that the venting of the container has the

[1] This test method is under the jurisdiction of Committee D-34 on Waste Disposal and is the direct responsibility of Subcommittee D34.02 on Extraction and Leachate Testing.

Current edition approved Oct. 21, 1985. Published March 1986. Originally published as D 3987 – 81. Last previous edition D 3987 – 81.

[2] *Annual Book of ASTM Standards*, Vol 04.01.

[3] *Annual Book of ASTM Standards*, Vol 04.08.

[4] *Annual Book of ASTM Standards*, Vol 11.01.

[5] *Annual Book of ASTM Standards*, Vol 05.05.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

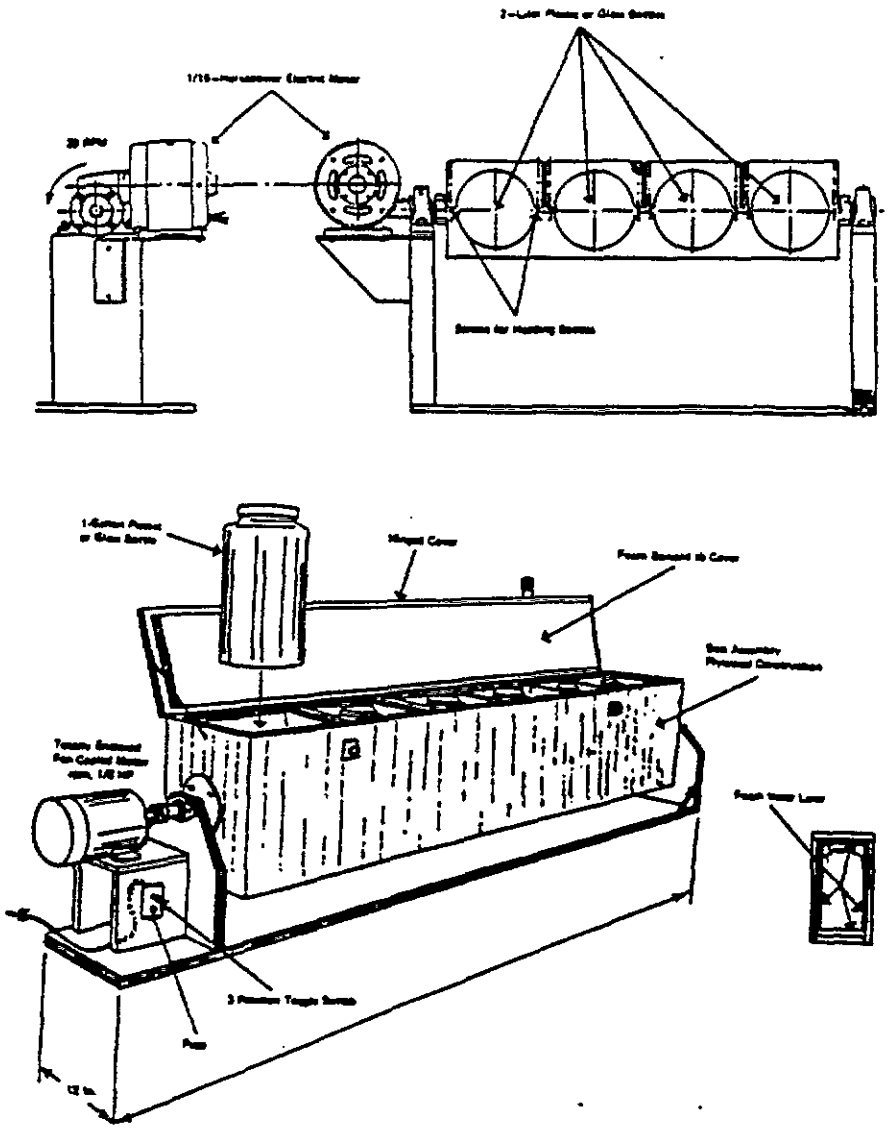


FIG. 1 Extractor

potential to affect the concentration of volatile extracts in the extract.) Containers should be cleaned in a manner consistent with the analyses to be performed.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the American Chemical Society, where such specifications are available.[7] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type IV reagent water at 18 to 27°C (Specification D 1193). The method by which the Type IV water is prepared, that is, distillation, ion exchange, reverse osmosis, electrodialysis, should remain constant throughout testing.

7. Sampling

7.1 Obtain a representative sample of the solid waste to be tested using ASTM sample methods developed for the specific industry where available. (See Practices D 75 and D 420 and Method D 2234.)

7.2 Where no specific methods are available, sampling methodology for materials of similar physical form shall be used.

[7] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

7.3 A minimum sample of 5000 g shall be sent to the laboratory (see Practice E 122).

7.4 It is important that the sample of the solid waste be representative with respect to surface area, as variations in surface area would directly affect the leaching characteristics of the sample. Solid waste samples should contain a representative distribution of particles sizes.

7.5 Keep samples in closed containers appropriate to the sample type prior to the extraction in order to prevent sample contamination or constituent loss. Where it is desired to extract biologically or chemically active samples in their existing state, store the samples at 4°C (Practices D 3370) and start the extraction within 8 h. Where it is desired to extract such samples in a state representative of the results of biological or chemical activities, the samples may be specifically handled to simulate such activities. Record the storage conditions and handling procedures in the report.

8. Sample Preparation

8.1 For free-flowing particulate solid wastes, obtain a sample of the approximate size required in the test by quartering the sample (Section 7) received for testing on an impermeable sheet of glazed paper, oil cloth, or other flexible material as follows:

8.1.1 Empty the sample container into the center of the sheet.

8.1.2 Flatten out the sample gently with a suitable straightedge until it is spread uniformly to a depth at least twice the maximum particle diameter particle size.

8.1.3 Remix the sample by lifting a corner of the sheet and drawing it across, low down, to the opposite corner in a manner that the material is made to roll over and over and does not merely slide along. Continue operation with each corner, proceeding in a clockwise direction. Repeat this operation ten times.

8.1.4 Lift all four corners of the sheet towards the center and holding all four corners together, raise the entire sheet into the air to form a pocket for the sample.

8.1.5 Repeat 8.1.2.

8.1.6 With a straightedge at least as long as the flattened mound of sample (such as a thin-edged yard stick), gently divide the sample into quarters. An effort should be made to avoid using pressure on the straightedge sufficient to cause damage to the particles.

8.1.7 Discard alternate quarters.

8.1.8 If further reduction of sample size is necessary, repeat 8.1.3 through 8.1.7. A minimum sample size of 70 g is recommended for each extraction. Additional samples should be provided for determination of solids content. If smaller samples are used in the test, report this fact.

8.2 For field-cored solid wastes or castings produced in the laboratory, cut a representative section weighing approximately 70 or 140 g for testing, plus samples for determination of solids content. Shape the sample so that the leaching solution will cover the material to be leached.

8.3 For fluid solid wastes, mix thoroughly in a manner that does not incorporate air to assure uniformity before withdrawing a 70 or 140 g sample for test. Take samples for determination of solids content at the same time as the test samples.

9. Procedure

9.1 Record the physical description of the sample to tested including particle size so far as it is known.

9.2 *Solids Content*—Determine the solids content of separate portions of the sample as follows:

9.2.1 Dry to constant weight two dishes or pans of size suitable to the solid waste being tested at 104 ± 2°C. Cool in a desiccator and weigh. Record the value to ± 0.1 g.

9.2.2 Put an appropriately sized portion of sample of the solid waste to be tested into each pan. Scale the weight used to the physical form of the solid waste tested. Use a minimum of 50 g but use larger samples where particles larger than 10-mm in average diameter are being tested. Weigh. Record the weight to ± 0.1 g.

9.2.3 Dry 16 to 20 h at 104 ± 2°C. Certain solid wastes, such as scrubber sludges, may contain compounds that are subject to calcination at the specified drying temperature. Dry these compounds at lower temperatures. For example, gypsum may be successfully dried at 45°C (Method C 471) and $CaSO_3 \cdot 1/2H_2O$ wastes at 85°C. Record the actual temperature and time of the drying period.

9.2.4 Cool to room temperature in a desiccator and reweigh. Record the weight to ± 0.1 g.

9.3 *Shake Procedure*—Weigh or tare the container to be used in the shake test to the nearest or within 1 g.

9.4 Add to the container approximately 70 g of solid waste (Section 8) and determine and record the weight of sample used to ½ g. If weights other than 70 g are used, note in the report.

9.5 Add to the container a volume of test water (6.2) equ in millilitres to 20 times the weight in grams of the sample used in 9.4. See discussion of dilution ratio in Appendix (Example: 70 g sample = 1400 mL water).

9.6 Agitate continuously for 18 ± 0.25 h at 18 to 27°C.

9.7 Open the container. Observe and record any physical changes in the sample and leaching solution.

9.8 Let the sample settle for 5 min; then separate the bulk of the aqueous phase from the solid phase by decantation, centrifugation, or filtration through a coarse filter paper as appropriate. Then vacuum or pressure filter the liquid through a 0.45-μm filter. If the separation method results in prolonged filtering time, an 8-μm filter or other device may be used. Record any deviations in the report.

9.9 The filtrate obtained in 9.8 is the extract mentioned elsewhere in this test method. Measure the pH of the extract immediately, then preserve the extract in a manner consistent with the chemical analysis or biological testing procedures to be performed (Practices D 3370). If sufficient liquid phase is not available for the analyses, so indicate in the report and do not continue the procedure; or alternatively, perform the extraction procedure on additional samples of the solid waste to obtain sufficient liquid phase. Where phase separation occurs during the storage of the extract, appropriate mixing should be used to ensure the homogeneity of the extract prior to its use in such analysis or testing.

9.10 Analyze the extract for specific constituents or properties or use the extract for biological testing procedures a desired using appropriate ASTM standard methods. Where no appropriate ASTM methods exist, other methods may be used and recorded in the report.

10. Calculation

10.1 Calculate the solids content of the individual samples from the data obtained in 9.2 as follows:

$$S = A/B$$

where:

A = weight in grams of sample after drying,
B = original weight in grams of sample, and
S = solid content, g/g.

Average the two values obtained. Record as the solids content.

11. Report

11.1 The report shall include the following:

11.1.1 Source of the solid waste, date of sampling, and sample preservation used,

11.1.2 Description of the solid waste including physical characteristics and particle size, if known (9.1),

11.1.3 Solids content (9.2) (see Method D 2216),

11.1.4 Sample weight if other than 70 g,

11.1.5 Drying time and temperature if other than 16 to 20 h at 104 ± 2°C,

11.1.6 pH and results of specific analyses calculated in appropriate units. State analytical procedures used, and filter used if other than 0.45 µm,

11.1.7 Observation of changes in test material or leaching solution recorded in 9.8.

11.1.8 Date leach testing started, preservation used for extract, and date of analysis.

12. Precision and Bias

12.1 No information is presently available as to the precision or bias of the analysis of specific constituents in the extract. It is recommended that users of this test method validate the applicability of their chosen methods of detection by spiking portions of the extract, before using these test methods for the analysis of the extract.

12.2 Based on a collaborative series of tests on five solid wastes including fly ash, cutting waste, aluminum industry waste, foundry waste, and non-ferrous foundry waste, the overall precision of this test method was improved. The above data was presented at the second ASTM D-34 Solid Waste Symposium.[8]

12.3 The precision of this test method may vary depending on the solid waste being tested and on the element being extracted.

12.4 Determination of the bias of this test method is not possible, as no standard reference material exists.

[8] A copy of this paper "Statistical Analysis and Description of Factors Affecting the ASTM Leaching Test" by Dr. Robert Paule can be obtained from ASTM Headquarters.

APPENDIX

X1. AGITATION TECHNIQUES AND RATE, AND LIQUID/SOLID RATIOS

X1.1 While a major effort relative to development of the test method has been undertaken to determine the optimum agitation rate, equipment, and liquid/solid ratios specified in the test method, it is recognized that these variables may significantly influence the results on certain solid wastes, and that they may not be adequate for certain solid wastes, such as monolithic, solidified, or organic wastes.

X1.2 The possible effects of varying the agitation technique and rate include degree of mixing, rate of release of constituents, and particle abrasion effects. The precision of the test method may also be influenced.

X1.3 The possible effects of varying the dilution ratio include degree of mixing, rate of release of constituents (and possible concentration effects, depending on availability), and particle abrasion effects.

X1.4 Efforts are underway to develop test methods that are more suitable for use with organic, monolithic, and solidified wastes.

U.S. Environmental Protection Agency SAS Number
CLP Sample Management Office [ ]
209 Madison Street, Alexandria, VA 22313
Phone: (703) 557-2490 or FTS 557-2490

SPECIAL ANALYTICAL SERVICES
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Client: EPA Region III/CH2M HILL

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Raymark Site, Montgomery County, PA

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. Description of analytical service requested:

Analyze 2 surface water samples and 1 blank samples for 5-day biological oxygen demand (BOD5) using EPA Method 405.1, chemical oxygen demand (COD) using EPA method 410.2, total suspended solids (TSS) using EPA Method 160.3, total dissolved solids (TDS) using EPA Method 160.1, alkalinity using EPA Method 310.1, hardness using EPA Method 130.2 and color using EPA Method 110.1. If COD concentrations are mid or high range, use EPA Method 410.1 or 410.3, respectively. (All Methods are attached).

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

Analyze 1 surface water samples, 1 surface water duplicate sample and 1 field handling blank for 5-day biological oxygen demand (BOD5) using EPA Method 405.1, chemical oxygen demand (COD) using EPA method 410.2, total dissolved solids (TDS) using EPA Method 160.1, alkalinity using EPA Method 310.1, hardness using EPA Method 130.2 and color using EPA Method 110.1. Concentrations are low for all parameters. If COD concentrations are mid or high range, use EPA Method 410.1 or 410.3, respectively. EPA Methods are found in the "EPA Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983".

3. Purpose of Analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund RI/FS

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment:

Samples shipped by overnight courier.

6. Approximate number of days results required after lab receipt of samples:

Results of analyses are requested forty (40) days after receipt of samples by the laboratory.

7. Analytical protocol required (attach copy if other than protocol currently used in this program):

See attachments.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detention limits, etc.):

Samples must be analyzed within the following holding times:

BOD5 - 48 hours from date and time of collection

COD - 28 days from date of collection

TSS - 7 days from date of collection

TDS - 7 days from date of collection

Alkalinity - 14 days from date of collection

Hardness - 6 months from date of collection

Color - 48 hours from the date and time of collection

Perform duplicate analysis on one in every 20 samples or fraction thereof. Standardize equipment according to manufacturer's instructions. Check pH with wide range pH paper. If pH > 2 for COD and hardness samples, contact CRL for instructions. Detection limits for all compounds must be as specified in # 12 of this SAS Request, or lower. Qualify results where suspended solids content may affect accuracy.

9. Analytical results (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedures and specific instrument used must be clearly identified. Bench records tabulating order of calibration of standards, lab blanks, samples, lab control standards, spikes, duplicates, etc., with resulting output on concentration readout must be provided along with worksheets used to calculate results. Specify the organic compound used to prepare the standards and spikes. A photocopy of the instrument readout, i.e., stripcharts, printer, tapes, etc., must be included. Results are to be recorded in mg/l. Records of analysis and calculations must be legible and sufficient to recalculate all concentrations. EPA QC reference samples, or any other reference sample or identical calibration verification, must be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analytical results must be provided for all reference samples used.

Data package must include: all raw data, all instrument and/or equipmentcalibrationresults,calcutlations,blankresults, duplicate results chain-of-custody forms, SAS request forms, SAS logbooks (signed by analyst) with date and time of sample preparation and analysis.

The cover page and all sample report forms MUST be labelled with the complete EPA sample number as it appears on the chain-of-custody and CLP paperwork.

Laboratory spike samples are not applicable for BOD5, COD, TDS, alkalinity, hardness, and color analyses.

10. Other (use additional sheets or attached supplementary information, as needed):

The awarded laboratory is responsible for meeting all requirements as specified in this client request. Any changes in method(s) or other specifications must be approved by Region III prior to the award. If these stipulations are not met, Region III will recommend review for liquidation damages.

11. Name of sampling/shipping contact:

Tom McLaughlin Phone: (703) 471-6405

12. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
BOD5	5 mg/l	+/- 20 %
COD	5 mg/l	+/- 20 %
TSS	10 mg/l	+/- 20 %
TDS	10 mg/l	+/- 20 %
Alkalinity	10 mg/l	+/- 20 %

Hardness	10 mg/l	+/- 20 %
Color	10 mg/l	Not applicable

13. QC Requirements

Audits Required	Frequency of Audits	Limits (+/- % or conc.)

All QA/QC requirements must be performed and reported as specified in the CLP SOW for Organics Analysis (10/86). The procedures, frequencies and acceptance criteria used shall be the same as specified in the SOW.

14. Action Required if Limits are Exceeded:

Contact Colleen K. Walling, USEPA and Tom McLaughlin, CH2M HILL.

15. Request Prepared by: Laura Gavin/CH2M HILL

Date: February 7, 1990

16. Request reviewed by:

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

BIOCHEMICAL OXYGEN DEMAND

Method 405.1 (5 Days, 20°C)

STORET NO. 00310
Carbonaceous 80082

1. Scope and Application
   1.1 The biochemical oxygen demand (BOD) test is used for determining the relative oxygen requirements of municipal and industrial wastewaters. Application of the test to organic waste discharges allows calculation of the effect of the discharges on the oxygen resources of the receiving water. Data from BOD tests are used for the development of engineering criteria for the design of wastewater treatment plants.
   1.2 The BOD test is an empirical bioassay-type procedure which measures the dissolved oxygen consumed by microbial life while assimilating and oxidizing the organic matter present. The standard test conditions include dark incubation at 20°C for a specified time period (often 5 days). The actual environmental conditions of temperature, biological population, water movement, sunlight, and oxygen concentration cannot be accurately reproduced in the laboratory. Results obtained must take into account the above factors when relating BOD results to stream oxygen demands.
2. Summary of Method
   2.1 The sample of waste, or an appropriate dilution, is incubated for 5 days at 20°C in the dark. The reduction in dissolved oxygen concentration during the incubation period yields a measure of the biochemical oxygen demand.
3. Comments
   3.1 Determination of dissolved oxygen in the BOD test may be made by use of either the Modified Winkler with Full-Bottle Technique or the Probe Method in this manual.
   3.2 Additional information relating to oxygen demanding characteristics of wastewaters can be gained by applying the Total Organic Carbon and Chemical Oxygen Demand tests (also found in this manual).
   3.3 The use of 60 ml incubation bottles in place of the usual 300 ml incubation bottles, in conjunction with the probe, is often convenient.
4. Precision and Accuracy
   4.1 Eighty-six analysts in fifty-eight laboratories analyzed natural water samples plus an exact increment of biodegradable organic compounds. At a mean value of 2.1 and 175 mg/1 BOD, the standard deviation was ±0.7 and ±26 mg/1, respectively (EPA Method Research Study 3).
   4.2 There is no acceptable procedure for determining the accuracy of the BOD test.

Approved for NPDES CBOD: pending approval for Section 304(h), CWA
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5. References

5.1 The procedure to be used for this determination is found in: Standard Methods for the Examination of Water and Wastewater, 15th Edition, p. 483, Method 507 (1980).

5.2 Young, J. C., "Chemical Methods for Nitrification Control," J. Water Poll. Control Fed., 45, p. 637 (1973).

CHEMICAL OXYGEN DEMAND

Method 410.1 (Titrimetric, Mid-Level)

STORET NO. 00340

1. Scope and Application
   - 1.1 The Chemical Oxygen Demand (COD) method determines the quantity of oxygen required to oxidize the organic matter in a waste sample, under specific conditions of oxidizing agent, temperature, and time.
   - 1.2 Since the test utilizes a specific chemical oxidation the result has no definite relationship to the Biochemical Oxygen Demand (BOD) of the waste or to the Total Organic Carbon (TOC) level. The test result should be considered as an independent measurement of organic matter in the sample, rather than as a substitute for the BOD or TOC test.
   - 1.3 The method can be applied to domestic and industrial waste samples having an organic carbon concentration greater than 50 mg/1. For lower concentrations of carbon such as in surface water samples, the Low Level Modification should be used. When the chloride concentration of the sample exceeds 2000 mg/1, the modification for saline waters is required.
2. Summary of Method
   - 2.1 Organic and oxidizable inorganic substances in the sample are oxidized by potassium dichromate in 50% sulfuric acid solution at reflux temperature. Silver sulfate is used as a catalyst and mercuric sulfate is added to remove chloride interference. The excess dichromate is titrated with standard ferrous ammonium sulfate, using orthophenanthroline ferrous complex as an indicator.
3. Sampling and Preservation
   - 3.1 Collect the samples in glass bottles, if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.
   - 3.2 Biologically active samples should be tested as soon as possible. Samples containing settleable material should be well mixed, preferably homogenized, to permit removal of representative aliquots.
   - 3.3 Samples should be preserved with sulfuric acid to a pH <2 and maintained at 4°C until analysis.
4. Interferences
   - 4.1 Traces of organic material either from the glassware or atmosphere may cause a gross, positive error.
     - 4.1.1 Extreme care should be exercised to avoid inclusion of organic materials in the distilled water used for reagent preparation or sample dilution.
     - 4.1.2 Glassware used in the test should be conditioned by running blank procedures to eliminate traces of organic material.

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4.2 Volatile materials may be lost when the sample temperature rises during the sulfuric acid addition step. To minimize this loss the flask should be cooled during addition of the sulfuric acid solution.

4.3 Chlorides are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate is added to the digestion flask to complex the chlorides, thereby effectively eliminating the interference on all but brine and estuarine samples.

5. Apparatus

5.1 Reflux apparatus: Glassware should consist of a 500 ml Erlenmeyer flask or a 300 ml round bottom flask made of heat-resistant glass connected to a 12 inch Allihn condenser by means of a ground glass joint. Any equivalent reflex apparatus may be substituted provided that a ground-glass connection is used between the flask and the condenser.

6. Reagents

6.1 Distilled water: Special precautions should be taken to insure that distilled water used in this test be low in organic matter.

6.2 Standard potassium dichromate solution (0.250 N): Dissolve 12.259 g $K_2Cr_2O_7$, primary standard grade, previously dried at 103°C for two hours, in distilled water and dilute to 1000 ml.

6.3 Sulfuric acid reagent: Conc. H_2SO_4 containing 23.5g silver sulfate, Ag_2SO_4, per 4.09kg bottle. With continuous stirring, the silver sulfate may be dissolved in about 30 minutes.

6.4 Standard ferrous ammonium sulfate (0.25 N): Dissolve 98.0 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in distilled water. Add 20 ml of conc. H_2SO_4 (6.8), cool and dilute to 1 liter. This solution must be standardized daily against standard $K_2Cr_2O_7$ solution (6.2).

6.4.1 Standardization: To approximately 200 ml of distilled water add 25.0 ml of 0.25 N $K_2Cr_2O_7$ (6.2) solution. Add 20 ml of H_2SO_4 (6.8) and cool. Titrate with ferrous ammonium sulfate (6.4) using 3 drops of ferroin indicator (6.6). The color change is sharp, going from blue-green to reddish-brown.

$$\text{Normality} = \frac{(\text{ml } K_2Cr_2O_7)(0.25)}{\text{ml } Fe(NH_4)_2(SO_4)_2}$$

6.5 Mercuric sulfate: Powdered $HgSO_4$.

6.6 Phenanthroline ferrous sulfate (ferroin) indicator solution: Dissolve 1.48 g of 1–10 (ortho) phenanthroline monohydrate, together with 0.70 g of $FeSO_4 \cdot 7H_2O$ in 100 ml of water. This indicator may be purchased already prepared.

6.7 Silver sulfate: Powdered Ag_2SO_4.

6.8 Sulfuric acid (sp. gr. 1.84): Concentrated H_2SO_4.

7. Procedure

7.1 Place several boiling stones in the reflux flask, followed by 50.0 ml of sample or an aliquot diluted to 50.0 ml and 1 g of $HgSO_4$ (6.5). Add 5.0 ml conc. H_2SO_4 (6.8); swirl until the mercuric sulfate has dissolved. Place reflux flask in an ice bath and slowly add, with swirling, 25.0 ml of 0.25 N $K_2Cr_2O_7$ (6.2). Now add 70 ml of sulfuric acid-silver

sulfate solution (6.3) to the cooled reflux flask, again using slow addition with swirling motion.

Caution: Care must be taken to assure that the contents of the flask are well mixed. If not, superheating may result, and the mixture may be blown out of the open end of the condenser.

7.1.1 If volatile organics are present in the sample, use an allihn condenser and add the sulfuric acid-silver sulfate solution through the condenser, while cooling the flask, to reduce loss by volatilization.

7.2 Apply heat to the flask and reflux for 2 hours. For some waste waters, the 2-hour reflux period is not necessary. The time required to give the maximum oxidation for a wastewater of constant or known composition may be determined and a shorter period of refluxing may be permissible.

7.3 Allow the flask to cool and wash down the condenser with about 25 ml of distilled water. If a round bottom flask has been used, transfer the mixture to a 500 ml Erlenmeyer flask, washing out the reflux flask 3 or 4 times with distilled water. Dilute the acid solution to about 300 ml with distilled water and allow the solution to cool to about room temperature. Add 8 to 10 drops of ferroin indicator (6.6) to the solution and titrate the excess dichromate with 0.25 N ferrous ammonium sulfate (6.4) solution to the end point. The color change will be sharp, changing from a blue-green to a reddish hue.

7.4 Blank–Simultaneously run a blank determination following the details given in (7.1) and (7.2), but using low COD water in place of sample.

8. Calculation

8.1 Calculate the COD in the sample in mg/l as follows:

$$\text{COD, mg/liter} = \frac{(A - B)N \times 8{,}000}{S}$$

where:

A = milliliters of $Fe(NH_4)_2(SO_4)_2$ solution required for titration of the blank,

B = milliliters of $Fe(NH_4)_2(SO_4)_2$ solution required for titration of the sample,

N = normality of the $Fe(NH_4)_2(SO_4)_2$ solution, and

S = milliliters of sample used for the test.

9. Precision and Accuracy

9.1 Eighty-six analysts in fifty-eight laboratories analyzed a distilled water solution containing oxidizable organic material equivalent to 270 mg/l COD. The standard deviation was ±17.76 mg/l COD with an accuracy as percent relative error (bias) of -4.7%. (EPA Method Research Study 3).

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 550, Method 508 (1975).
2. Annual Book of ASTM Standards, Part 31, "Water", Standard D1252–67, p 473 (1976).

CHEMICAL OXYGEN DEMAND

Method 410.2 (Titrimetric, Low Level)

STORET NO. 00335

1. Scope and Application
   - 1.1 The scope of this modification of the Chemical Oxygen Demand (COD) test is the same as for the high level test. It is applicable to the analysis of surface waters, domestic and industrial wastes with low demand characteristics.
   - 1.2 This method (low level) is applicable for samples having a COD in the range of 5–50 mg/l COD.
2. Summary of Method
   - 2.1 Organic and oxidizable inorganic substances in an aqueous sample are oxidized by potassium dichromate solution in 50 percent (by volume) sulfuric acid in solution. The excess dichromate is titrated with standard ferrous ammonium sulfate using orthophenanthroline ferrous complex (ferroin) as an indicator.
3. Sampling and Preservation
   - 3.1 Collect the samples in glass bottles, if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.
   - 3.2 Biologically active samples should be tested as soon as possible. Samples containing settleable material should be well mixed, preferably homogenized, to permit removal of representative aliquots.
   - 3.3 Samples should be preserved with sulfuric acid to a pH < 2 and maintained at 4°C until analysis.
4. Interferences
   - 4.1 Traces of organic material either from the glassware or atmosphere may cause a gross, positive error.
     - 4.1.1 Extreme care should be exercised to avoid inclusion of organic materials in the distilled water used for reagent preparation or sample dilution.
     - 4.1.2 Glassware used in the test should be conditioned by running blank procedures to eliminate traces of organic material.
   - 4.2 Volatile materials may be lost when the sample temperature rises during the sulfuric acid addition step.
   - 4.3 Chlorides are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate is added to the digestion flask to complex the chlorides, thereby effectively eliminating the interference on all but brine and estuarine samples.
5. Apparatus
   - 5.1 Reflux apparatus: Glassware should consist of a 500 ml Erlenmeyer flask or a 300 ml round bottom flask made of heat-resistant glass connected to a 12 inch Allihn condenser

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by means of a ground glass joint. Any equivalent reflux apparatus may be substituted provided that a ground-glass connection is used between the flask and the condenser.

6. Reagents

6.1 Distilled water: Special precautions should be taken to insure that distilled water used in this test be low in organic matter.

6.2 Standard potassium dichromate solution (0.025 N): Dissolve 12.259 g $K_2Cr_2O_7$, primary standard grade, previously dried at 103°C for two hours, in distilled water and dilute to 1000 ml. Mix this solution thoroughly then dilute 100.0 ml to 1000 ml with distilled water.

6.3 Sulfuric acid reagent: Conc. H_2SO_4 containing 23.5g silver sulfate, Ag_2SO_4, per 4.09kg bottle. (With continuous stirring, the silver sulfate may be dissolved in about 30 minutes.)

6.4 Standard ferrous ammonium sulfate (0.025 N): Dissolve 98 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in distilled water. Add 20 ml of conc. H_2SO_4 (6.8), cool and dilute to 1 liter. Dilute 100 ml of this solution to 1 liter with distilled water. This solution must be standardized daily against $K_2Cr_2O_7$ solution.

6.4.1 Standardization: To approximately 200 ml of distilled water add 25.0 ml of 0.025 N $K_2Cr_2O_7$ (6.2) solution. Add 20 ml of H_2SO_4 (6.8) and cool. Titrate with ferrous ammonium sulfate (6.4) using 3 drops of ferroin indicator (6.6). The color change is sharp, going from blue-green to reddish-brown.

$$\text{Normality} = \frac{(\text{ml } K_2Cr_2O_7)(0.025)}{\text{ml } Fe(NH_4)_2(SO_4)_2}$$

6.5 Mercuric sulfate : Powdered $HgSO_4$.

6.6 Phenanthroline ferrous sulfate (ferroin) indicator solution: Dissolve 1.48 g of 1–10 (ortho)phenanthroline monohydrate, together with 0.70 g of $FeSO_4 \cdot 7H_2O$ in 100 ml of water. This indicator may be purchased already prepared.

6.7 Silver sulfate : Powdered Ag_2SO_4.

6.8 Sulfuric acid (sp. gr. 1.84) : Concentrated H_2SO_4.

7. Procedure

7.1 Place several boiling stones in the reflux flask, followed by 50.0 ml of sample or an aliquot diluted to 50.0 ml and 1 g of $HgSO_4$ (6.5). Add 5.0 ml conc. H_2SO_4 (6.8); swirl until the mercuric sulfate has dissolved. Place reflux flask in an ice bath and slowly add, with swirling, 25.0 ml of 0.025 N $K_2Cr_2O_7$ (6.2). Now add 70 ml of sulfuric acid-silver sulfate solution (6.3) to the cooled reflux flask, again using slow addition with swirling motion.

Caution: Care must be taken to assure that the contents of the flask are well mixed. If not, superheating may result, and the mixture may be blown out of the open end of the condenser.

7.1.1 If volatile organics are present in the sample, use an Allihn condenser and add the sulfuric acid-silver sulfate solution through the condenser, while cooling the flask, to reduce loss by volatilization.

7.2 Apply heat to the flask and reflux for 2 hours. For some waste waters, the 2-hour reflux period is not necessary. The time required to give the maximum oxidation for a wastewater of constant or known composition may be determined and a shorter period of refluxing may be permissible.

7.3 Allow the flask to cool and wash down the condenser with about 25 ml of distilled water. If a round bottom flask has been used, transfer the mixture to a 500 ml Erlenmeyer flask, washing out the reflux flask 3 or 4 times with distilled water. Dilute the acid solution to about 300 ml with distilled water and allow the solution to cool to about room temperature. Add 8 to 10 drops of ferroin indicator (6.6) to the solution and titrate the excess dichromate with 0.025 N ferrous ammonium sulfate (6.4) solution to the end point. The color change will be sharp, changing from a blue-green to a reddish hue.

7.4 Blank—Simultaneously run a blank determination following the details given in (7.1) and (7.2), but using low COD water in place of sample.

8. Calculation

8.1 Calculate the COD in the sample in mg/l as follows:

$$\text{COD, mg/l} = \frac{(A - B)N \times 8{,}000}{S}$$

where:

A = milliliters of $Fe(NH_4)_2(SO_4)_2$ solution required for titration of the blank,
B = milliliters of $Fe(NH_4)_2(SO_4)_2$ solution required for titration of the sample,
N = normality of the $Fe(NH_4)_2(SO_4)_2$ solution, and
S = milliliters of sample used for the test.

9. Precision and Accuracy

9.1 Eighty-six analysts in fifty-eight laboratories analyzed a distilled water solution containing oxidizable organic material equivalent to 12.3 mg/l COD. The standard deviation was ±4.15 mg/l COD with an accuracy as percent relative error (bias) of 0.3%. (EPA Method Research Study 3.)

CHEMICAL OXYGEN DEMAND

Method 410.3 (Titrimetric, High Level for Saline Waters)

STORET NO. 00340

1. Scope and Application
   1.1 When the chloride level exceeds 1000 mg/1 the minimum accepted value for the COD will be 250 mg/1. COD levels which fall below this value are highly questionable because of the high chloride correction which must be made.
2. Summary of Method
   2.1 Organic and oxidizable inorganic substances in an aqueous sample are oxidized by potassium dichromate solution in 50 percent (by volume) sulfuric acid solution. The excess dichromate is titrated with standard ferrous ammonium sulfate using orthophenanthroline ferrous complex (ferroin) as an indicator.
3. Sample Handling and Preservation
   3.1 Collect the samples in glass bottles, if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.
   3.2 Biologically active samples should be tested as soon as possible. Samples containing settleable material should be well mixed, preferably homogenized, to permit removal of representative aliquots.
   3.3 Samples should be preserved with sulfuric acid to a pH < 2 and maintained at 4°C until analysis.
4. Interferences
   4.1 Traces of organic material either from the glassware or atmosphere may cause a gross, positive error.
      4.1.1 Extreme care should be exercised to avoid inclusion of organic materials in the distilled water used for reagent preparation or sample dilution.
      4.1.2 Glassware used in the test should be conditioned by running blank procedures to eliminate traces of organic material.
   4.2 Volatile materials may be lost when the sample temperature rises during the sulfuric acid addition step.
   4.3 Chlorides are quantitatively oxidized by dichromate and represent a positive interference. A chloride correction is made using the procedure outlined in 7.7 of this method.
5. Apparatus
   5.1 Reflux apparatus: Glassware should consist of a 500 ml Erlenmeyer flask or a 300 ml round bottom flask made of heat-resistant glass connected to a 12 inch Allihn condenser by means of a ground glass joint. Any equivalent reflux apparatus may be substituted provided that a ground-glass connection is used between the flask and the condenser.

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6. Reagents

6.1 Standard potassium dichromate solution, (0.25 N): Dissolve 12.2588 g of $K_2Cr_2O_7$, primary standard grade, previously dried for 2 hours at 103°C in water and dilute to 1000 ml.

6.2 Sulfuric acid reagent: Conc. H_2SO_4 containing 23.5 g silver sulfate, Ag_2SO_4, per 4.09kg. bottle. (With continuous stirring, the silver sulfate may be dissolved in about 30 minutes.)

6.3 Standard ferrous ammonium sulfate, 0.250 N: Dissolve 98 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in distilled water. Add 20 ml of conc. H_2SO_4, (6.7), cool and dilute to 1 liter. This solution must be standardized against the standard potassium dichromate solution (6.1) daily.

6.3.1 Standardization: Dilute 25.0 ml of standard dichromate solution (6.1) to about 250 ml with distilled water. Add 20 ml conc. sulfuric acid (6.7). Cool, then titrate with ferrous ammonium sulfate titrant (6.3), using 10 drops of ferroin indicator (6.5).

$$\text{Normality} = \frac{(\text{ml } K_2Cr_2O_7)(0.25)}{\text{ml } Fe(NH_4)_2(SO_4)_2}$$

6.4 Mercuric sulfate : Powdered $HgSO_4$.

6.5 Phenanthroline ferrous sulfate (ferroin) indicator solution: Dissolve 1.48 g of 1–10-(ortho) phenanthroline monohydrate, together with 0.70 g of $FeSO_4 \cdot 7H_2O$ in 100 ml of water. This indicator may be purchased already prepared.

6.6 Silver sulfate : Powdered Ag_2SO_4.

6.7 Sulfuric acid (sp. gr. 1.84) : Concentrated H_2SO_4.

7. Procedure

7.1 Pipet a 50.0 ml aliquot of sample not to exceed 800 mg/l of COD into a 500 ml, flat bottom, Erlenmeyer flask. Add $HgSO_4$ (6.4) in the ratio of 10 mg to 1 mg chloride, based upon the mg of chloride in the sample aliquot and 5 ml of sulfuric acid (6.7). Swirl until all the mercuric sulfate has dissolved. Add 25.0 ml of 0.25N $K_2Cr_2O_7$ (6.1). Carefully add 70 ml of sulfuric acid-silver sulfate solution (6.2) and gently swirl until the solution is thoroughly mixed. Glass beads should be added to the reflux mixture to prevent bumping, which can be severe and dangerous.

Caution: The reflux mixture must be thoroughly mixed before heat is applied. If this is not done, local heating occurs in the bottom of the flask, and the mixture may be blown out of the condenser.

7.1.1 If volatile organics are present in the sample, use an Allihn condenser and add the sulfuric acid-silver sulfate solution through the condenser, while cooling the flask, to reduce loss by volatilization.

7.2 Attach the flask to the condenser and reflux the mixture for two hours.

7.3 Cool, and wash down the interior of the condenser with 25 ml of distilled water. Disconnect the condenser and wash the flask and condenser joint with 25 ml of distilled water so that the total volume is 350 ml. Cool to room temperature.

7.4 Titrate with standard ferrous ammonium sulfate (6.3) using 10 drops of ferroin (6.5) indicator. (This amount must not vary from blank, sample and standardization.) The color change is sharp, going from blue-green to reddish-brown and should be taken as the end point although the blue-green color may reappear within minutes.

7.5 Run a blank, using 50 ml of distilled water in place of the sample together with all reagents and subsequent treatment.

7.6 For COD values greater than 800 mg/1, a smaller aliquot of sample should be taken; however, the volume should be readjusted to 50 ml with distilled water having a chloride concentration equal to the sample.

7.7 Chloride correction[1]: Prepare a standard curve of COD versus mg/1 of chloride, using sodium chloride solutions of varying concentrations following exactly the procedure outlined. The chloride interval, as a minimum should be 4000 mg/1 up to 20,000 mg/1 chloride. Lesser intervals of greater concentrations must be run as per the requirements of the data, but in no case must extrapolation be used.

8. Calculation

$$\text{mg/l COD} = \frac{[(A - B)C \times 8{,}000] - 50D}{\text{ml of sample}} \times 1.2$$

where:

A = ml $Fe(NH_4)_2(SO_4)_2$ for blank;
B = ml $Fe(NH_4)_2(SO_4)_2$ for sample;
C = normality of $Fe(NH_4)_2(SO_4)_2$;
D = chloride correction from curve (step 7.7)
1.2 = compensation factor to account for the extent of chloride oxidation which is dissimilar in systems containing organic and non-organic material.

9. Precision and Accuracy

9.1 Precision and accuracy data are not available at this time.

Bibliography

1. Burns, E. R., Marshall, C., Journal WPCF, Vol. 37, p 1716–1721 (1965).

RESIDUE, FILTERABLE

Method 160.1 (Gravimetric, Dried at 180°C)

STORET NO. 70300

1. Scope and Application
   - 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   - 1.2 The practical range of the determination is 10 mg/1 to 20,000 mg/1.
2. Summary of Method
   - 2.1 A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C.
   - 2.2 If Residue, Non-Filterable is being determined, the filtrate from that method may be used for Residue, Filterable.
3. Definitions
   - 3.1 Filterable residue is defined as those solids capable of passing through a glass fiber filter and dried to constant weight at 180°C.
4. Sample Handling and Preservation
   - 4.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.
5. Interferences
   - 5.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.
   - 5.2 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate.
   - 5.3 Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.
6. Apparatus
   - 6.1 Glass fiber filter discs, 4.7 cm or 2.1 cm, without organic binder, Reeve Angel type 934-AH, Gelman type A/E, or equivalent.
   - 6.2 Filter holder, membrane filter funnel or Gooch crucible adapter.
   - 6.3 Suction flask, 500 ml.
   - 6.4 Gooch crucibles, 25 ml (if 2.1 cm filter is used).
   - 6.5 Evaporating dishes, porcelain, 100 ml volume. (Vycor or platinum dishes may be substituted).
   - 6.6 Steam bath.
   - 6.7 Drying oven, 180°C ±2°C.
   - 6.8 Desiccator.

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6.9 Analytical balance, capable of weighing to 0.1 mg.

7. Procedure

7.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus and insert into bottom of a suitable Gooch crucible. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Discard washings.

7.2 Preparation of evaporating dishes: If Volatile Residue is also to be measured heat the clean dish to 550 ±50°C for one hour in a muffle furnace. If only Filterable Residue is to be measured heat the clean dish to 180 ±2°C for one hour. Cool in desiccator and store until needed. Weigh immediately before use.

7.3 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 ml to the funnel by means of a 100 ml graduated cylinder. If total filterable residue is low, a larger volume may be filtered.

7.4 Filter the sample through the glass fiber filter, rinse with three 10 ml portions of distilled water and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

7.5 Transfer 100 ml (or a larger volume) of the filtrate to a weighed evaporating dish and evaporate to dryness on a steam bath.

7.6 Dry the evaporated sample for at least one hour at 180 ±2°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5 mg.

8. Calculation

8.1 Calculate filterable residue as follows:

$$\text{Filterable residue, mg/l} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = weight of dried residue + dish in mg
B = weight of dish in mg
C = volume of sample used in ml

9. Precision and Accuracy

9.1 Precision and accuracy are not available at this time.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 92, Method 208B, (1975).

RESIDUE, NON-FILTERABLE

Method 160.2 (Gravimetric, Dried at 103–105°C)

STORET NO. 00530

1. Scope and Application
   1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   1.2 The practical range of the determination is 4 mg/1 to 20,000 mg/1.
2. Summary of Method
   2.1 A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103–105°C.
   2.2 The filtrate from this method may be used for Residue, Filterable.
3. Definitions
   3.1 Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103–105°C.
4. Sample Handling and Preservation
   4.1 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
   4.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.
5. Interferences
   5.1 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.
   5.2 Samples high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter (7.5) minimizes this potential interference.
6. Apparatus
   6.1 Glass fiber filter discs, without organic binder, such as Millipore AP-40, Reeves Angel 934-AH, Gelman type A/E, or equivalent.
   NOTE: Because of the physical nature of glass fiber filters, the absolute pore size cannot be controlled or measured. Terms such as "pore size", collection efficiencies and effective retention are used to define this property in glass fiber filters. Values for these parameters vary for the filters listed above.
   6.2 Filter support: filtering apparatus with reservoir and a coarse (40–60 microns) fritted disc as a filter support.

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NOTE: Many funnel designs are available in glass or porcelain. Some of the most common are Hirsch or Buchner funnels, membrane filter holders and Gooch crucibles. All are available with coarse fritted disc.

6.3 Suction flask.

6.4 Drying oven, 103–105°C.

6.5 Desiccator.

6.6 Analytical balance, capable of weighing to 0.1 mg.

7. Procedure

7.1 Preparation of glass fiber filter disc: Place the glass fiber filter on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible with wrinkled surface up. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus or both crucible and filter if Gooch crucible is used, and dry in an oven at 103–105°C for one hour. Remove to desiccator and store until needed. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg). Weigh immediately before use. After weighing, handle the filter or crucible/filter with forceps or tongs only.

7.2 Selection of Sample Volume

For a 4.7 cm diameter filter, filter 100 ml of sample. If weight of captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 1.0 mg of residue. If other filter diameters are used, start with a sample volume equal to 7 ml/cm^2 of filter area and collect at least a weight of residue proportional to the 1.0 mg stated above.

NOTE: If during filtration of this initial volume the filtration rate drops rapidly, or if filtration time exceeds 5 to 10 minutes, the following scheme is recommended: Use an unweighed glass fiber filter of choice affixed in the filter assembly. Add a known volume of sample to the filter funnel and record the time elapsed after selected volumes have passed through the filter. Twenty-five ml increments for timing are suggested. Continue to record the time and volume increments until fitration rate drops rapidly. Add additional sample if the filter funnel volume is inadequate to reach a reduced rate. Plot the observed time versus volume filtered. Select the proper filtration volume as that just short of the time a significant change in filtration rate occurred.

7.3 Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support.

7.4 Shake the sample vigorously and quantitatively transfer the predetermined sample volume selected in 7.2 to the filter using a graduated cylinder. Remove all traces of water by continuing to apply vacuum after sample has passed through.

7.5 With suction on, wash the graduated cylinder, filter, non-filterable residue and filter funnel wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through.

NOTE: Total volume of wash water used should equal approximately 2 ml per cm^2. For a 4.7 cm filter the total volume is 30 ml.

7.6 Carefully remove the filter from the filter support. Alternatively, remove crucible and filter from crucible adapter. Dry at least one hour at 103–105°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg).

8. Calculations

8.1 Calculate non-filterable residue as follows:

$$\text{Non-filterable residue, mg/l} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = weight of filter (or filter and crucible) + residue in mg
B = weight of filter (or filter and crucible) in mg
C = ml of sample filtered

9. Precision and Accuracy

9.1 Precision data are not available at this time.

9.2 Accuracy data on actual samples cannot be obtained.

Bibliography

1. NCASI Technical Bulletin No. 291, March 1977. National Council of the Paper Industry for Air and Stream Improvement, Inc., 260 Madison Ave., NY.

ALKALINITY

Method 310.1 (Titrimetric, pH 4.5)

STORET NO. 00410

1. Scope and Application
   - 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   - 1.2 The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 50 ml.
   - 1.3 Automated titrimetric analysis is equivalent.
2. Summary of Method
   - 2.1 An unaltered sample is titrated to an electrometrically determined end point of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way.
3. Comments
   - 3.1 The sample should be refrigerated at 4°C and run as soon as practical. Do not open sample bottle before analysis.
   - 3.2 Substances, such as salts of weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.
   - 3.3 For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoint of pH 3.9, using the procedure in:
     Annual Book of ASTM Standards, Part 31, "Water", p 115, D-1067, Method D, (1976).
   - 3.4 Oil and grease, by coating the pH electrode, may also interfere, causing sluggish response.
4. Apparatus
   - 4.1 pH meter or electrically operated titrator that uses a glass electrode and can be read to 0.05 pH units. Standardize and calibrate according to manufacturer's instructions. If automatic temperature compensation is not provided, make titration at 25 ±2° C.
   - 4.2 Use an appropriate sized vessel to keep the air space above the solution at a minimum. Use a rubber stopper fitted with holes for the glass electrode, reference electrode (or combination electrode) and buret.
   - 4.3 Magnetic stirrer, pipets, flasks and other standard laboratory equipment.
   - 4.4 Burets, Pyrex 50, 25 and 10 ml.
5. Reagents
   - 5.1 Sodium carbonate solution, approximately 0.05 N: Place 2.5 ±0.2 g (to nearest mg) Na_2CO_3 (dried at 250°C for 4 hours and cooled in desiccator) into a 1 liter volumetric flask and dilute to the mark.

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5.2 Standard acid (sulfuric or hydrochloric), 0.1 N: Dilute 3.0 ml conc H_2SO_4 or 8.3 ml conc HCl to 1 liter with distilled water. Standardize versus 40.0 ml of 0.05 N Na_2CO_3 solution with about 60 ml distilled water by titrating potentiometrically to pH of about 5. Lift electrode and rinse into beaker. Boil solution gently for 3–5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH inflection point. Calculate normality using:

$$N = \frac{A \times B}{53.00 \times C}$$

where:
A = g Na_2CO_3 weighed into 1 liter
B = ml Na_2CO_3 solution
C = ml acid used to inflection point

5.3 Standard acid (sulfuric or hydrochloric), 0.02 N: Dilute 200.0 ml of 0.1000 N standard acid to 1 liter with distilled water. Standardize by potentiometric titration of 15.0 ml 0.05 N Na_2CO_3 solution as above.

6. Procedure

6.1 Sample size

6.1.1 Use a sufficiently large volume of titrant (>20 ml in a 50 ml buret) to obtain good precision while keeping volume low enough to permit sharp end point.

6.1.2 For < 1000 mg $CaCO_3$/1 use 0.02 N titrant

6.1.3 For > 1000 mg $CaCO_3$/1 use 0.1 N titrant

6.1.4 A preliminary titration is helpful.

6.2 Potentiometric titration

6.2.1 Place sample in flask by pipetting with pipet tip near bottom of flask

6.2.2 Measure pH of sample

6.2.3 Add standard acid (5.2 or 5.3), being careful to stir thoroughly but gently to allow needle to obtain equilibrium.

6.2.4 Titrate to pH 4.5. Record volume of titrant.

6.3 Potentiometric titration of low alkalinity

6.3.1 For alkalinity of <20 mg/1 titrate 100–200 ml as above (6.2) using a 10 ml microburet and 0.02 N acid solution (5.3).

6.3.2 Stop titration at pH in range of 4.3–4.7, record volume and exact pH. Very carefully add titrant to lower pH exactly 0.3 pH units and record volume.

7. Calculations

7.1 Potentiometric titration to pH 4.5

$$\text{Alkalinity, mg/1 } CaCO_3 = \frac{A \times N \times 50{,}000}{\text{ml of sample}}$$

HARDNESS, Total (mg/1 as $CaCO_3$)
Method 130.2 (Titrimetric, EDTA)

STORET NO. 00900

1. Scope and Application
   - 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   - 1.2 The method is suitable for all concentration ranges of hardness; however, in order to avoid large titration volumes, use a sample aliquot containing not more than 25 mg $CaCO_3$.
   - 1.3 Automated titration may be used.
2. Summary of Method
   - 2.1 Calcium and magnesium ions in the sample are sequestered upon the addition of disodium ethylenediamine tetraacetate (Na_2EDTA). The end point of the reaction is detected by means of Eriochrome Black T indicator, which has a red color in the presence of calcium and magnesium and a blue color when the cations are sequestered.
3. Sample Handling and Preservation
   - 3.1 Cool to 4°C, HNO_3 to pH < 2.
4. Comments
   - 4.1 Excessive amounts of heavy metals can interfere. This is usually overcome by complexing the metals with cyanide.
     - 4.1.1 Routine addition of sodium cyanide solution (Caution: deadly poison) to prevent potential metallic interference is recommended.
5. Apparatus
   - 5.1 Standard laboratory titrimetric equipment.
6. Reagents
   - 6.1 Buffer solution
     - 6.1.1 If magnesium EDTA is available: Dissolve 16.9 g NH_4Cl in 143 ml conc. NH_4OH in a 250 ml volumetric, add 1.25 g of magnesium salt of EDTA and dilute to the mark with distilled water. Then go to 6.1.3.
     - 6.1.2 If magnesium EDTA is unavailable: Dissolve 1.179 g disodium EDTA (analytical reagent grade) and 780 mg $MgSO_4 \cdot 7H_2O$ (or 644 mg $MgCl_2 \cdot 6H_2O$) in 50 ml distilled water. Add this solution to a 250 ml volumetric flask containing 16.9 g NH_4Cl and 143 ml conc. NH_4OH with mixing and dilute to the mark with distilled water.
     - 6.1.3 Store in a tightly stoppered plastic bottle; stable for approximately one month. Dispense with bulb operated pipet. Discard when 1 or 2 ml added to sample fails to produce a pH of 10.0 ±0.1 at end point of titration.

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6.1.4 Commercially available "odorless buffers" which are more stable, may be used.

6.2 Inhibitors: For most waters inhibitors are not necessary. If interfering ions are present use one of the following:

6.2.1 Inhibitor I: NaCN powder. (Caution: extremely poisonous). Flush solutions or sample containing this down drain using large quantities of water. Make sure no acids are present which might liberate HCN gas.

6.2.2 Inhibitor II: Dissolve 5.0 g $Na_2S \cdot 9\ H_2O$ or 3.7 g $Na_2S \cdot 5\ H_2O$ in 100 ml distilled water. Exclude air with tightly fitted rubber stopper. This gives sulfide precipitates which may obscure the end point if large quantities of heavy metals are present. Deteriorates rapidly through air oxidation.

6.2.3 Inhibitor III: Dissolve 4.5 g hydroxylamine hydrochloride in 100 ml of 95% ethanol or isopropanol.

6.3 Indicator: Use a commercially available indicator such as Calmagite indicator (Mallinckrodt) or one of the formulations described below (6.3.1–6.3.3)

6.3.1 Mix 0.5 g Eriochrome Black T with 4.5 g hydroxylamine hydrochloride. Dissolve in 100 ml of 95% ethanol or isopropanol.

6.3.2 Dissolve 0.5 to 1.0 g Eriochrome Black T in an appropriate solvent such as triethanolamine or 2-methoxyethanol. Stable approximately one week.

6.3.3 Mix together 0.5 g Eriochrome Black T and 100 g NaCl.

6.4 Standard EDTA titrant, 0.02 N: Place 3.723 g analytical reagent grade disodium ethylenediamine tetraacetate dihydrate, $Na_2H_2C_{10}H_{12}O_8N_2 \cdot 2\ H_2O$ in a 1 liter volumetric flask and dilute to the mark with distilled water. Check with standard calcium solution (6.4.1) by titration (6.4.5). Store in polyethylene. Check periodically because of gradual deterioration.

6.4.1 Standard calcium solution 0.02 N: Place 1.000 g anhydrous calcium carbonate (primary standard low in metals) in a 500 ml flask. Add, a little at a time, 1 + 1 HCL (6.4.2) until all of the $CaCO_3$ has dissolved. Add 200 ml distilled water. Boil for a few minutes to expel CO_2. Cool. Add a few drops of methyl red indicator (6.4.3) and adjust to intermediate orange color by adding 3N NH_4OH (6.4.4) or 1 + 1 HCl (6.4.2) as required. Quantitatively transfer to a 1 liter volumetric flask and dilute to mark with distilled water.

6.4.2 Hydrochloric acid solution, 1 + 1.

6.4.3 Methyl red indicator: Dissolve 0.10 g methyl red in distilled water in a 100 ml volumetric flask and dilute to the mark.

6.4.4 Ammonium hydroxide solution, 3 N: Dilute 210 ml of conc. NH_4OH to 1 liter with distilled water.

6.4.5 Standardization titration procedure: Place 10.0 ml standard calcium solution (6.4.1) in vessel containing about 50 ml distilled water. Add 1 ml buffer solution (6.1). Add 1–2 drops indicator (6.3) or small scoop of dry indicator (6.3.3). Titrate slowly with continuous stirring until the last reddish tinge disappears, adding last

few drops at 3–5 second intervals. At end point the color is blue. Total titration duration should be 5 minutes from the time of buffer addition.

$$N \text{ of EDTA} = \frac{0.2}{\text{ml of EDTA}}$$

6.5 Ammonium Hydroxide, 1N: Dilute 70 ml of conc. NH_4OH to 1 liter with distilled water.

7. Procedure

7.1 Pretreatment

7.1.1 For drinking waters, surface waters, saline waters, and dilutions thereof, no pretreatment steps are necessary. Proceed to 7.2.

7.1.2 For most wastewaters, and highly polluted waters, the sample must be digested as given in the Atomic Absorption Methods section of this manual, paragraphs 4.1.3 and 4.1.4. Following this digestion, proceed to 7.2.

7.2 Titration of sample-normal to high hardness:

7.2.1 Sample should require < 15 ml EDTA titrant (6.4) and titration should be completed within 5 minutes of buffer addition.

7.2.2 Place 25.0 ml sample in titration vessels, neutralize with 1N ammonium hydroxide (6.5) and dilute to about 50 ml.

7.2.3 Add 1 to 2 ml buffer solution (6.1).

7.2.4 If end point is not sharp (as determined by practice run) add inhibitor at this point (see 7.4).

7.2.5 Add 1 to 2 drops indicator solution (6.3.1 or 6.3.2) or small scoop of dried powder indicator formulation (6.3.3).

7.2.6 Titrate slowly with continuous stirring with standard EDTA titrant (6.4) until last reddish tint disappears. Solution is normally blue at end point.

7.3 Titration of sample–low hardness (less than 5 mg/1)

7.3.1 Use a larger sample (100 ml)

7.3.2 Use proportionately larger amounts of buffer, inhibitor and indicator

7.3.3 Use a microburet and run a blank using redistilled, distilled or deionized water.

7.4 To correct for interferences:

7.4.1 Some metal ions interfere by causing fading or indistinct end points. Inhibitors reduce this in accord with the scheme below for 25.0 ml samples diluted to 50 ml.

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COLOR

Method 110.1 (Colorimetric, ADMI)

STORET NOS.
00082 at pH 7.6
00083 at ORIGINAL SAMPLE pH

1. Scope and Application
   - 1.1 This method is applicable to colored waters and waste that have color characteristics significantly different from the yellow platinum-cobalt standard.
   - 1.2 A working range of 25 to 250 color units is recommended. Sample values above 250 units may be determined by quantitative dilution.
2. Summary of Method
   - 2.1 This method is an extension of the Tristimulus Filter Method[1]. Tristimulus values are converted to an ADMI single number color difference, of the same magnitude assigned to platinum-cobalt standards, using the Adams Nickerson Color Difference (DE).
   - 2.2 Tristimulus values obtained by Spectrophotometric Method 204B[1] may be used to calculate ADMI values as outlined in this procedure under Calculation 9.2.
3. Interferences
   - 3.1 Since very slight amounts of turbidity interfere with the determination, turbid samples must be filtered prior to analysis. The optimum filter media to remove turbidity without removing color has not been found. Membrane and glass fiber filters with functional pore sizes of approximately 0.45 *u* are convenient to use. Other techniques such as centrifuging and/or filter aids may be used.
4. Sample Handling and Preservation
   - 4.1 Since biological activity may change the color characteristics of a sample, the determination should be made as soon as possible. Refrigeration at 4°C is recommended.
5. Calibration
   - 5.1 Standard curves must be established (as outlined in Procedure 8.3) for each photometer used, and are not interchangeable. For color values less than 250, a 5 cm cell path is recommended. Less than 5 cm cell paths may be used if calibration is performed with the shorter cell.
6. Apparatus
   - 6.1 Spectrophotometer or filter photometer capable of transmission measurements using tristimulus filters listed below:

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Filter number	Wavelength of maximum transmittance in nm	Corning designation
1	590	CS 3-107
2	540	CS 4-98
3	438	CS 5-70

*Available from Corning Glass Works, Optical Products Department, Corning, NY 14830.

7. Reagents

7.1 Standard chloroplatinate solution: Dissolve 1.246 g potassium chloroplatinate, K_2PtCl_6, (equivalent to 0.500 g metallic Pt) and 1 g crystalline cobaltous chloride, $CoCl_2 \cdot 6H_2O$, in distilled water containing 100 ml of conc. HCl. Dilute to 1000 ml with distilled water. This standard solution is defined as 500 ADMI color units.

7.2 Sulfuric acid, concentrated.

7.3 Sodium hydroxide, 10 N: Dissolve 40 g of sodium hydroxide in 80 ml of distilled water. Cool to room temperature and dilute to 100 ml with distilled water.

8. Procedure

8.1 Prepare two 100 ml volumes of sample by maintaining the original pH of one aliquot and adjusting the second aliquot as necessary to pH 7.6 with sulfuric acid (7.2) or sodium hydroxide (7.3).

8.2 Filter samples to remove turbidity through a 0.45 u membrane filter, glass fiber filter or other suitable media (see interferences 3.1).

8.3 Use distilled water to set the transmittance at 100% and then determine the transmittance of the clarified sample or standard with each of the three tristimulus filters. Calibration standards from 25 to 250 units are recommended.

9. Calculations

9.1 Calculate intermediate tristimulus values for samples and standards from the transmittance data in 8.3 using the following equations:

$$X_s = (T_3 \times 0.1899) + (T_1 \times 0.791)$$

$$Y_s = T_2$$

$$Z_s = T_3 \times 1.1835$$

where:

T_1 = transmittance value in % using filter number 1

T_2 = transmittance value in % using filter number 2

T_3 = transmittance value in % using filter number 3

9.2 Convert tristimulus values to the corresponding Munsell values V_x, V_y and V_z by the use of published tables[2,3,4], or the equation suggested by Bridgeman[5].

9.3 Calculate DE values for samples and standards, construct a calibration curve by plotting DE against ADMI units of standards and determine ADMI color units of samples from the calibration curve.

$$DE = \sqrt{[0.23 \times (V_{x_s} - V_{x_c})]^2 + [(V_{y_s} - V_{y_c}) - (V_{z_s} - V_{z_c})]^2 + [0.4[(V_{y_s} - V_{y_c}) - (V_{z_s} - V_{z_c})]]^2}$$

where V_{x_s}, V_{y_s} and V_{z_s} = the Munsell values for X_s, Y_s and Z_s respectively and V_{x_c}, V_{y_c} and V_{z_c} = the Munsell values for a blank solution whose tristimulus values are X_c, Y_c and Z_c.

NOTE 1: If the photometer used is set at 100% transmittance with distilled water, the tristimulus values for the blank are 98.09, 100.00 and 118.35 for X_c, Y_c and Z_c respectively. If necessary, tristimulus values for the blank are determined as in calculation (9.1).

9.4 Report ADMI color values at pH 7.6 and at the original pH.

NOTE 2: The intermediate tristimulus values calculated under 9.1, using the three tristimulus filters, are used only to calculate the ADMI color value. They should not be reported as tristimulus values or used to determine dominant wavelength, luminance and purity.

10. Precision and Accuracy

10.1 Accuracy data on actual samples cannot be obtained.

10.2 Precision data are not available at this time.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition (1975), p 64.
2. J. Soc. Dyers and Colorists, 86, No. 8, 354 (1970).
3. Wyszecki and Stiles, Color Science, Wiley, N.Y., 1967, Tables 6.4 A, B and C.
4. Judd and Wyszecki, Color in Business, Science and Industry, 2nd Edition, Wiley, N.Y. (1963) Tables A, B and C in Appendix.
5. J. Opt. Soc. Am., Volume 53, page 499, April 1963.
6. Dyes and the Environment–Report on Selected Dyes and Their Effects, Volume 1, Sept. 1973 Appendix; American Dye Manufacturers Institute, Inc.

U.S. Environmental Protection Agency SAS Number
CLP Sample Management Office [ ]
209 Madison Street, Alexandria, VA 22313
Phone: (703) 557-2490 or FTS 557-2490

SPECIAL ANALYTICAL SERVICES
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Client: EPA Region III/CH2M HILL

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Raymark Site, Montgomery County, PA

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. Description of analytical service requested:

   Analyze 24 groundwater samples and 12 aqueous blanks for volatile organics as specified in EPA Method 524.2 -- Revision 3.0 plus acetone, carbon disulfide, 2-butanone, vinyl acetate, 4-methyl-2-pentanone, and 2-hexanone.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

   Analyze 20 groundwater samples, 4 duplicate groundwater samples 6 aqueous field handling blanks, and 6 aqueous trip blanks for volatile organics as specified in EPA Method 524.2 -- Revision 3.0 plus acetone, carbon disulfide, 2-butanone, vinyl acetate, 4-methyl-2-pentanone, and 2-hexanone. Samples will be low concentration. EPA Method 524.2 -- Revision 3.0 is found in the "EPA Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", September 1986. (Method attached).

3. Purpose of Analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

   Superfund RI/FS.

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment:

Samples shipped by overnight courier.

6. Approximate number of days results required after lab receipt of samples:

Normal turnaround (35 days)

Holding time: To meet the holding time requirements, the volatile organic samples MUST be analyzed within 10 days from date of collection. A written report (Data package) MUST be submitted within 35 days after receipt of the last sample within the case.

7. Analytical protocol required (attach copy if other than protocol currently used in this program):

Aqueous samples submitted for volatile organic analysis must be analyzed by EPA Method 524.2 -- Revision 3.0 (see attached). The samples must be analyzed for the compounds in item 1 of this SAS request IN ADDITION to the compounds listed in item 1 of Method 524.2

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detention limits, etc.):

The maximum number of samples in a sample delivery group (SDG) is 20. The detection limit must be 1 ug/l or less for all analytes. A minimum of five calibration standards is required at concentrations of 4, 10, 20, 30 and 40 ug/l. The initial five point calibration must be analyzed within the 2 days preceding sample analysis. A continuing calibration must be performed prior to sample analysis and once per 12 hours. In addition, a Laboratory Fortified Blank at a concentration of 1 ug/l must be analyzed immediately following each continuing calibration. Verify the MS tune and the intial calibration at the beginning of each 12 hour period of analyses. Before any samples are analyzed, it must be demonstrated that a laboratroy reagent blank is reasonably free of contamination that would prevent the determination of any analyte of concern at 1 ug/l. Each sample, blank, duplicate, and standard must be spiked with a surrogate spiking solution of 4-bromofluorobenzene and 1,2-dichlorobenzene-d4 at a concentration equal to that of the internal standard. Results are to be reported on CLP data forms modified to include all volatile organic compounds listed in EPA Method 524.2 -- Revision 3.0 plus those compounds listed in item 1 above. Include a laboratory generated mass spectrum for all compounds detected above and below the MDL. All samples mus be analyzed within 12 hours of a GC/MS tune. The laboratory selected for this analysis must provide, as part of their written

narrative, their initial demonstration of laboratory accuracy and precision as described in section 10.3 of method 524.2. All calibration and analytical procedures given in method 524.2, whether presented as optional or required, MUST be followed. All samples are to be analyzed within the specified holding times and reported on results summary sheets. Up to ten volatile compounds (not listed in method 524.4 and item 1 above) of greatest apparent concentration shall be tentatively identified via a forward search of the NBS mass spectral library as described in the current SOW for Organics (2/88). Only tentatively identified compounds (TICs) with a peak area > 40% of the nearest internal standard are to be reported.

9. Analytical results (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

The SAS package must be equivalent to the CLP RAS format, modified to include all compounds listed in Method 524.2 -- Revision 3.0 and in item 1 of this SAS request. The laboratory must submit all documentation including the SAS packing lists, chain-of-custody forms, and analytical results on standard CLP forms, as described above for all samples submitted to the labaratory (the data sheets should binclude the dates the samples were collected, received, and analyzed by the laboratory). Laboratory duplicated, reagent blanks, and laboratory fortified blanks must also be reported on standard CLP RAS forms. All QA/QC information, standards information, raw data including laboratory generated standards and sample mass spectra for compounds detected both above and below the MDL, and initial and continuing calibration results must be provided. Initial calibrations, continuing calibrations, surrogate recoveries, internal standards performance, etc. must be reported on standard CLP forms modified for this SAS request. A written narrative describing problems encountered in receipt or during analysis and corrective actions taken (including telephone logs, etc.) must be provided. The report should describe the actual methods used from preparation to analysis. The laboratory shall also provide as part of the written report their initial demonstration of laboratory accuracy and precision. The report shall paginated. Results shall be reported in ug/l.

The cover page and all sample report forms MUST be labelled with the complete EPA sample number as it appears on the chain-of custody and CLP paperwork. The case narrative must document all problems encountered and the subsequent resolutions.

10. Other (use additional sheets or attached supplementary information, as needed):

The laboratory must supply a detailed example calculation that clearly demonstrates the manner in which the initial and final result was derived. Where applicable, each component of the calculation must be explained (e.g.. if the calculation includes a dilution factor, it must be clear where, why, and how each dilution occurred). The laboratory must supply any and all information required to reproduce, during independent data review, all results reported by the laboratory.

The awarded laboratory is responsible for meeting all requirements as specified in this client request. Any changes in method(s) or other specifications must be approved by Region III prior to the award. If these stipulations are not met, Region III will recommend review for liquidation damages.

11. Name of sampling/shipping contact:

Tom McLaughlin Phone: (703) 471-6405

12. Data Requirements

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
TCL VOA's and NYMCLs principal organic contaminants	1 ug/l	+/- 20 %
Vinyl chloride	0.5 ug/l	+/- 20 %

13. QC Requirements

Audits Required	Frequency of Audits	Limits (+/- % or conc.)
Method Blank	one per 12 hr.	less than 1 ug/l
Lab Duplicate	one per 20 sample	+/- 20 % (RPD)
Surrogate Spike	each analysis	Per the CLP SOW for Organics (2/88)
Laboratory Fortified Blank (1 ug/l)	every 12 hrs.	Accuracy 80-120% recovery Precision 20% (RSD)

- To ensure the 1 ug/l and 0.5 ug/l detection limit for vinyl chloride can be achieved, a Laboratory Fortified Blank MUST be prepared at 1 ug/l and 0.5 ug/l. The area of the analyte at this concentration must be measured but it does not have to be included in the calculations of relative standard deviation for the calibration. Report RF and concentration detected.

14. Action Required if Limits are Exceeded:

Failure to obtain method blank values less than 1 ug/l requires that all samples prepared with that method blank be re-prepared and re-analyzed for the affected parameters. All blank data are to be reported and the necessity for re-analysis must be included in the narrative summary. If duplicate results exceed control limits, re-analyze the samples and duplicate. If re-analysis fails to produce results in control, contact the RSCC. For the fortified blank, if the accuracy, precision, and method detection limits cannot

be acheived, the problem must be located and corrected before additional samples are analyzed. If the problem cannot be identified, contact the RSCC. Re-analysis is required when one or more surrogate percent recoveries are out of QC limits. If the 1 ug/l check standard does not meet control limits the instrument must be re-calibrated. The laboratory must demonstrate the ability to reach the detection limit.

15. Request Prepared by: Laura Gavin/CH2M HILL

Date: February 8, 1990

16. Request reviewed by:

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

METHOD 524.2. VOLATILE ORGANIC COMPOUNDS IN WATER BY PURGE AND TRAP CAPILLARY COLUMN GAS CHROMATOGRAPHY/MASS SPECTROMETR
(August, 1986)

1. SCOPE AND APPLICATION

1.1 This method is applicable for the determination of various vola organic compounds in finished drinking water, raw source water, drinking water in any treatment stage. (1) The following compo can be determined by this method:

Analyte	Chemical Abstract Servic Registry Number
Benzene	71-43-2
Bromobenzene	108-86-1
Bromochloromethane	74-97-5
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane	74-83-9
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroethane	75-00-3
Chloroform	67-66-3
Chloromethane	74-87-3
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
Dibromochloromethane	124-48-1
1,2-Dibromo-3-chloropropane	96-12-8
1,2-Dibromoethane	106-93-4
Dibromomethane	74-95-3
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Dichlorodifluoromethane	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
cis-1,2-Dichloroethene	156-59-4

Analyte	Chemical Abstract Services Registry Number
trans-1,2-Dichloroethene	156-60-5
1,2-Dichloropropane	78-87-5
1,3-Dichloropropane	142-28-9
2,2-Dichloropropane	590-20-7
1,1-Dichloropropene	563-58-6
Ethylbenzene	100-41-4
Hexachlorobutadiene	87-68-3
Isopropylbenzene	98-82-8
p-Isopropyltoluene	99-87-6
Methylene chloride	75-09-2
Naphthalene	91-20-3
n-Propylbenzene	103-65-1
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene	79-01-6
Trichlorofluoromethane	75-69-4
1,2,3-Trichloropropane	96-18-4
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl chloride	75-01-4
o-Xylene	95-47-6
m-Xylene	108-38-3
p-Xylene	106-42-3

1.2 Method detection limits (MDLs) (2) are compound dependent and vary with purging efficiency and concentration. The MDLs for selected analytes are presented in Table 1. The applicable concentration range of this method is compound and instrument dependent but is approximately 0.1 to 200 ug/L. Analytes that are inefficiently purged from water will not be detected when present at low concentrations, but they can be measured with acceptable accuracy and precision when present in sufficient amounts. Determination of some geometrical isomers (i.e., xylenes) may be hampered by coelution.

1.3 This method is recommended for use only by analysts experienced the measurement of purgeable organics at the low ug/L level or experienced technicians under the close supervision of a qualif analyst.

2. SUMMARY OF METHOD

2.1 Highly volatile organic compounds with low water solubility are extracted (purged) from the sample matrix by bubbling an inert through a 25 mL aqueous sample. Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb trapped sample components. The analytes desorbed directly to a large bore capillary or cryofocussed on capillary precolumn before being flash evaporated to a narrow b capillary for analysis. The column is temperature programmed t separate the method analytes which are then detected with a mas spectrometer (MS) interfaced to the gas chromatograph.

Wide-bore capillary columns generally require a jet separator, whereas narrow-bore capillaries can be directly interfaced to ion source.

2.2 Tentative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing resul mass spectra and GC retention times. Each identified component measured by relating the MS response for an appropriate selecte ion produced by that compound to the MS response for another i produced by a compound that is used as an internal standard.

3. INTERFERENCES

3.1 During analysis, major contaminant sources are volatile materi in the laboratory and impurities in the inert purging gas and the sorbent trap. The use of non-polytetrafluoroethylene (PTF plastic tubing, non-PTFE thread sealants, or flow controllers rubber components in the purging device should be avoided sinc such materials out-gas organic compounds which will be concent in the trap during the purge operation. Analyses of laborator reagent blanks (Sect. 9.1.3) provide information about the pre of contaminants. When potential interfering peaks are noted i laboratory reagent blanks, the analyst should change the purge source and regenerate the molecular sieve purge gas filter (Fi 1). Subtracting blank values from sample results is not permi

3.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing relatively high concentrations of volatile organic compounds. A preventive technique is between-sample rinsing of the purging apparatus and sample syringes with two portions of reagent water. After analysis of a sample containing high concentrations of volatile organic compounds, one or more laboratory reagent blanks should be analyzed to check for cross contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high levels of compounds being determined, it may be necessary to wash out the purging device with a soap solution, rinse it with reagent water, and then dry it in an oven at 105°C between analyses.

3.3 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean since clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination.

4. SAFETY

4.1 The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each laboratory is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method. Additional references to laboratory safety are available (3-5) for the information of the analyst.

4.2 The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. APPARATUS AND EQUIPMENT

5.1 SAMPLE CONTAINERS - 60-mL to 120-mL screw cap vials (Pierce #13(or equivalent) each equipped with a PTFE-faced silicone septum (Pierce #12718 or equivalent). Prior to use, wash vials and se: with detergent and rinse with tap and distilled water. Allow t: vials and septa to air dry at room temperature, place in a 105° oven for one hour, then remove and allow to cool in an area kno to be free of organics.

5.2 PURGE AND TRAP SYSTEM - The purge and trap system consists of t. separate pieces of equipment: purging device, trap, and desorb Systems are commercially available from several sources that me all of the following specifications.

5.2.1 The all glass purging device (Fig. 1) must be designed t accept 25-mL samples with a water column at least 5 cm deep. Gaseous volumes above the sample must be kept to minimum (< 15 mL) to eliminate dead volume effects. A g frit should be installed at the base of the sample chamb so the purge gas passes through the water column as fine divided bubbles with a diameter of < 3 mm at the origin. Needle spargers may be used, however, the purge gas must introduced at a point ≤ 5 mm from the base of the water column.

5.2.2 The trap (Fig. 2) must be at least 25 cm long and have a inside diameter of at least 0.105 in. Starting from the inlet, the trap should contain 1.0 cm of methyl silicone coated packing and the following amounts of adsorbents: of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and of coconut charcoal. Before initial use, the trap shou conditioned overnight at 180°C by backflushing with an gas flow of at least 20 mL/min. Vent the trap effluent the room, not to the analytical column. Prior to daily the trap should be conditioned for 10 minutes at 180°C backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column m be run through the temperature program prior to analysi samples.

5.2.3 The use of the methyl silicone coated packing is recommended, but not mandatory. The packing serves a d purpose of protecting the adsorbent from aerosols, and of insuring that the adsorbent is fully enclosed within heated zone of the trap thus eliminating potential cold spots. Alternatively, silanized glass wool may be used spacer at the trap inlet.

5.2.4 The desorber must be capable of rapidly heating the trap to 180°C. The polymer section of the trap should not be heated higher than 200°C or the life expectancy of the trap will decrease. Trap failure is characterized by a pressure drop in excess of 3 pounds per square inch across the trap during purging or by poor bromoform sensitivities. The desorber design illustrated in Fig. 2 meets these criteria.

5.2.5 Figures 3 and 4 show typical flow patterns for the purge-sorb and desorb modes.

5.3 GAS CHROMATOGRAPHY/MASS SPECTROMETER/DATA SYSTEM (GC/MS/DS)

5.3.1 The GC must be capable of temperature programming and should be equipped with variable-constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation. The column oven must be cooled to <30°C, therefore, a subambient oven controller is required. The GC usually is interfaced to the MS with an all-glass enrichment device and an all-glass transfer line, but any enrichment device or transfer line can be used if the performance specifications described in Sect. 9.2.5 can be achieved.

5.3.2 Gas Chromatographic Column 1 - 60m long x 0.75mm ID VOCOL (Supelco, Inc.) wide-bore capillary column with 1.5 µm film thickness. The flow rate of helium carrier gas is established at 15 mL/min. The column temperature is held for 5 minutes at 10°C, then programmed to 160°C at 6°C/min, and held until all expected compounds have eluted. A sample chromatogram obtained with this column is presented in Fig. 5. This column was used to develop the method performance statements in Section 13.

5.3.3 Gas Chromatographic Column 2 - 30m long x 0.53mm ID DB-624 Mega-Bore (J&W Scientific, Inc.) column with 3 µm film thickness. Helium carrier gas flow is 15 mL/min. The column temperature is held for 5 minutes at 10°C, then programmed to 160°C at 6°C/min. A sample chromatogram obtained with this column is presented in Fig. 6.

5.3.4 Gas Chromatographic Column 3 - 30 meter long x 0.32mm ID fused silica capillary column coated with Durabond DB-5 (J&W Scientific, Inc.) with a 1µm film thickness. Helium carrier gas flow is 4.0 mL/min. The column is maintained at 10°C for 5 mins, then programmed at 6°/min for 10 min than 15°/min for 5 min to 145°C. A sample chromatogram obtained with this column is presented in Fig. 7.

5.3.5 Mass spectral data are obtained with electron-impact ionization at a nominal electron energy of 70 eV. The spectrometer must be capable of scanning from 35 to 300 every 2s or less and must produce a mass spectrum that all criteria in Table 3 when 50 ng or less of 4-bromofluorobenzene is introduced into the GC. To ensu sufficient precision of mass spectral data, the desirab scan rate allows acquisition of at least five spectra w a sample component elutes from the GC.

5.3.6 An interfaced data system (DS) is required to acquire, store, reduce and output mass spectral data. The comput software must allow searching any GC/MS data file for i of a specific mass and plotting ion abundances versus t or scan number. This type of plot is defined as an extracted ion current profile (EICP). Software must al allow integrating the abundance in any EICP between specified time or scan number limits.

5.4 CAPILLARY INTERFACE - The device interfaces the purge and trap concentrator to the capillary gas chromatograph. The interfac condenses the desorbed sample components and focuses them into narrow band on an uncoated fused silica capillary pre-column. the interface is flash heated the sample is transferred to the analytical capillary column.

5.4.1 Under a stream of liquid nitrogen, the temperature of t fused silica in the interface is maintained at -150°C d the cryofocusing step. After the desorption period, th interface must be capable of rapid heating to +250°C in sec. or less to complete the transfer of analytes.

5.5 SYRINGE AND SYRINGE VALVES

5.5.1 Two 25-mL glass hypodermic syringes with Luer-Lok tip.

5.5.2 Three 2-way syringe valves with Luer ends.

5.5.3 Micro syringes - 10, 25, 100 µL.

5.5.4 Syringes - 0.5, 1.0, and 5-mL, gas tight with shut-off

5.6 MISCELLANEOUS

5.6.1 Standard solution storage containers - 15-mL bottles wi PTFE-lined screw caps.

6. REAGENTS AND CONSUMABLE MATERIALS

6.1 TRAP PACKING MATERIALS

6.1.1 2,6-Diphenylene oxide polymer, 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

6.1.2 Methyl silicone packing (optional)— OV-1 (3%) on Chromosorb W, 60/80 mesh, or equivalent.

6.1.3 Silica gel - 35/60 mesh, Davison, grade 15 or equivalent.

6.1.4 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26 lot #M-2549 by crushing through 26 mesh screen.

6.2 REAGENTS

6.2.1 Methanol - Demonstrated to be free of analytes.

6.2.2 Reagent water - Prepare reagent water by passing tap water through a filter bed containing about 0.5 kg of activated carbon, by using a water purification system, or by boiling distilled water for 15 min followed by a 1-h purge with inert gas while the water temperature is held at 90°C. Store in clean, narrow-mouth bottles with PTFE-lined septa and screw caps.

6.2.3 Hydrochloric acid (1+1) - Carefully add measured volume of conc. HCl to equal volume of reagent water.

6.2.4 Vinyl chloride - 99.9% pure vinyl chloride is available from Ideal Gas Products, Inc., Edison, New Jersey and from Matheson, East Rutherford, New Jersey. Certified mixtures of vinyl chloride in nitrogen at 1.0 and 10.0 ppm (v/v) are available from several sources.

6.3 STANDARD STOCK SOLUTIONS

- These solutions may be purchased as certified solutions or prepared from pure standard materials using the following procedures:

6.3.1 Place about 9.8 mL of methanol into a 10-mL ground-glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol-wetted surfaces have dried and weigh to the nearest 0.1 mg.

6.3.2 If the analyte is a liquid at room temperature, use a 1
syringe and immediately add two or more drops of refere
standard to the flask. Be sure that the reference stan
falls directly into the alcohol without contacting the
of the flask. If the analyte is a gas at room temperat
fill a 5-mL valved gas-tight syringe with the standard
the 5.0-mL mark, lower the needle to 5 mm above the met
meniscus, and slowly inject the standard into the neck
of the flask. The gas will rapidly dissolve in the met

6.3.3 Reweigh, dilute to volume, stopper, then mix by inverti
the flask several times. Calculate the concentration i
micrograms per microliter from the net gain in weight.
compound purity is certified at 96% or greater, the wei
can be used without correction to calculate the
concentration of the stock standard.

6.3.4 Store stock standard solutions in 15-mL bottles equippe
with PTFE-lined screw caps. Methanol solutions prepare
from liquid analytes are stable for at least four weeks
stored at 4°C. Methanol solutions prepared from gaseou
analytes are not stable for more than one week when sto
at <0°C; at room temperature, they must be discarded af
one day.

6.4 SECONDARY DILUTION STANDARDS - Use standard stock solutions to
prepare secondary dilution standard solutions that contain the
analytes in methanol. The secondary dilution standards should
prepared at concentrations that can be easily diluted to prepa
aqueous calibration solutions (Sect. 8.1) that will bracket th
working concentration range. Store the secondary dilution sta
solutions with minimal headspace and check frequently for sign
deterioration or evaporation, especially just before preparing
calibration solutions from them. Storage times described for
standard solutions in Sect. 6.3.4 also apply to secondary dilu
standard solutions.

6.5 INTERNAL STANDARD SPIKING SOLUTION — Prepare a spiking soluti
containing fluorobenzene, and 1,2-dichlorobenzene-d_4 in methar
using the procedures described in Sect. 6.3 and 6.4. It is
recommended that the secondary dilution standard be prepared a
concentration of 25 ug/mL of each internal standard compound.
addition of 10 uL of such a standard to 25.0 mL of sample or
calibration standard would be equivalent to 10 ug/L.

6.6 BFB STANDARD — Prepare a 25-ug/mL solution of bromofluoroben
in methanol.

6.7 LABORATORY QUALITY CONTROL STANDARD CONCENTRATE - Using standard stock solutions, prepare a solution containing each analyte of interest at a concentration of 500 times the MCL in methanol.

7. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

7.1 SAMPLE COLLECTION

7.1.1 Collect all samples in duplicate. Fill sample bottles to overflowing. No air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed.

7.1.2 When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 10 min). Adjust the flow to about 500 mL/min and collect duplicate samples from the flowing stream.

7.1.3 When sampling from an open body of water, fill a 1-quart wide-mouth bottle or 1-liter beaker with sample from a representative area, and carefully fill duplicate sample bottles from the container.

7.2 SAMPLE PRESERVATION

7.2.1 Adjust the pH of the duplicate samples to <2 by carefully adding one drop of 1:1 HCl for each 20 mL of sample volume.(6) Seal the sample bottles, PFTE-face down, and shake vigorously for one minute.

7.2.2 The samples must be chilled to 4°C on the day of collection and maintained at that temperature until analysis. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure that they will be at 4°C on arrival at the laboratory.

7.3 SAMPLE STORAGE

7.3.1 Store samples at 4°C until analysis. The sample storage area must be free of organic solvent vapors.

7.3.2 Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and replaced.

8. CALIBRATION AND STANDARDIZATION

8.1 PREPARATION OF CALIBRATION STANDARDS

8.1.1 A set of at least five calibration standards containing method analytes is needed. One calibration standard sh contain each analyte at a concentration approaching but greater than the method detection limit (Table 1) for t compound; the other calibration standards should contai analytes at concentrations that define the range of the method.

8.1.2 To prepare a calibration standard, add an appropriate v of a secondary dilution standard solution to an aliquot reagent water in a volumetric flask. Use a microsyring rapidly inject the alcoholic standard into the expanded of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting flask three times only. Discard the contents contained the neck of the flask. Aqueous standards are not stabl should be discarded after one hour unless sealed and st as described in Sect. 7.2.

8.2 CALIBRATION

8.2.1 After meeting the BFB criteria in Sect. 10.1, analyze e calibration standard according to Sect. 10, adding 10 internal standard spiking solution directly to the syri For each analyte, select a significant characteristic when feasible, use the most intense ion in the mass spectrum; when a less intense ion is more characteristi sufficiently intense to provide necessary sensitivity, that ion to avoid possible interferences. Tabulate are response of the characteristic m/z versus the concentra for each analyte and internal standard. Calculate resp factors (RF) for each analyte using Equation 1:

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)} \qquad \text{Equation 1}$$

where:

A_s = Area of the characteristic m/z for the analyte measured;

A_{is} = Area of the characteristic m/z for the internal standard;

C_{is} = Concentration of the internal standard, in ug/L.
C_s = Concentration of the analyte to be measured, in ug/L.

The choice of which internal standard to use for an analyte is left to the analyst.

8.2.2 Prepare a calibration curve for each analyte. Alternatively, if the RF for an analyte is constant (less than 10% RSD) over the working range, the average RF can be used for that analyte.

8.2.3 The working calibration curve or average response factor must be verified on each working day by the measurement of one or more calibration standards. If the quantitation ion area for any analyte varies from the response determined for that standard concentration from the calibration curve or average RF established in Sect. 8.2.2 by more than ±20%, repeat steps 8.2.1 and 8.2.2.

8.2.4 Calibration for vinyl chloride using a certified gaseous mixture of vinyl chloride in nitrogen can be accomplished by the following steps.

8.2.4.1 Fill the purging device with 25.0 mL of reagent water or aqueous calibration standard.

8.2.4.2 Start to purge the aqueous mixture. Inject a known volume (between 100 and 2000 uL) of the calibration gas (at room temperature) directly into the purging device with a gas tight syringe. Slowly inject the gaseous sample through a septum seal at the top of the purging device at 2000 uL/min. Do not inject the standard through the aqueous sample inlet needle. Inject the gaseous standard before five min of the 11-min purge time have elapsed.

8.2.4.3 Determine the aqueous equivalent concentration of vinyl chloride standard, in ug/L, injected with the equation:

$$S = 0.102\ (C)(V) \qquad \text{Equation 2}$$

where S = Aqueous equivalent concentration of vinyl chloride standard in ug/L;
C = Concentration of gaseous standard in ppm;
V = Volume of standard injected in milliliters.

9. QUALITY CONTROL

9.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of thi program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate a document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criter to determine if the results of analyses meet the performance characteristics of the method. A quality control check standar must be analyzed to confirm that the measurements were perform an in-control mode of operation.

9.1.1 The analyst must make an initial, one-time, demonstratic the ability to generate acceptable accuracy and precisic with this method. This ability is established as descr in Section 9.2.

9.1.2 In recognition of advances that are occurring in chroma graphy, the analyst is permitted certain options (detai in Section 10.2.2) to improve the separations or lower cost of measurements. Each time such a modification is to the method, the analyst is required to repeat the procedure in Section 9.2.

9.1.3 Each day, the analyst must analyze a reagent water blan demonstrate that interferences from the analytical syst are under control.

9.1.4 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards the operation of the measurement system is in control. procedure is described in Section 9.3. The frequency o check standard analyses is equivalent to 10% of all sam analyzed but at least two samples per month.

9.1.5 On a weekly basis, the laboratory must demonstrate the ability to analyze low level samples. A procedure for level check samples is described in Section 9.4.

9.1.6 The laboratory must maintain performance records to doc the quality of data that is generated. This procedure described in Section 9.5.

9.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

9.2.1 A quality control (QC) check sample concentrate is required containing each regulated analyte, and any additional analyte which is to be reported, at a concentration of 500 times the MCL or 5 ug/mL, whichever is smaller, in methanol. The QC check sample must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

9.2.2 Analyze seven 25-mL QC check samples at 1/5 MCL or 2 ug/L according to the method beginning in Sect. 10. Each sample is produced by injecting 10 uL of QC check sample concentrate into 25 mL of reagent water in a glass syringe through the syringe valve.

9.2.3 Calculate the average recovery ($\bar{X}$) in ug/L, and the standard deviation of the recovery (s) in ug/L for each analyte using the seven results. Calculate the MDL for each analyte as specified in Ref. 2. The calculated MDL must be less than the spike level.

9.2.4 For each analyte, ($\bar{X}$) must be between 90% and 110% of the true value. Additionally, s must be $\leq$ 35% of $\bar{X}$. If s and $\bar{X}$ for all analytes meet the criteria, the system performance is acceptable and analysis of actual samples can begin. If any s exceeds the precision limit or any $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that analyte.
NOTE: The large number of analytes present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes are analyzed.

9.2.5 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 9.2.2 only for the analytes which failed the test.

9.3 The laboratory must demonstrate on a regular basis as outlined in Section 9.1.4 that the measurement system is in control by analyzing a quality control check sample for all analytes of interest at the MCL or 10 ug/L, whichever is smaller.

9.3.1 Prepare a QC check standard by adding 50 uL of QC check sample concentrate to 25 mL of regent water in a glass syringe.

9.3.2 Analyze the QC check according to Section 10, and calculate the recovery for each analyte. The recovery must be between 60% and 140% of the expected value.

9.3.3 If the recovery for any analyte falls outside the desig range, the analyte has failed the acceptance criteria. check standard containing each analyte that failed must re-analyzed.

9.4 On a weekly basis, the laboratory must demonstrate the ability analyze low level samples.

9.4.1 Prepare a low level check sample by spiking 10 uL of QC check sample concentrate to 25 mL of reagent water and analyze according to the method in Sect. 10.

9.4.2 For each analyte, the recovery must be between 60% and of the expected value.

9.4.3 When one or more analytes fail the test, the analyst m repeat the test only for those analytes which failed to the criteria. Repeated failure, however, will confirm general problem with the measurement system. If this occurs, locate and correct the source of the problem a repeat the test for all compounds of interest beginnin 9.4.1.

9.5 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of t laboratory and the nature of the samples. Field duplicates m analyzed to assess the precision of the environmental measure Whenever possible, the laboratory should analyze standard ref materials and participate in relevant performance evaluation studies.

10. PROCEDURE

10.1 DAILY GC/MS PERFORMANCE TESTS

10.1.1 At the beginning of each day that analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria are achieved for BFB (The performance test must be passed before any samples blanks, or standards are analyzed.

10.1.2 At the beginning of each day, inject 2 uL (50 ng) of B solution directly on the column. Alternatively, add 2 BFB solution to 25.0 mL of reagent water or calibratio standard and analyze the solution according to Sect. 1 Obtain a background-corrected mass spectrum of BFB and confirm that all the key m/z criteria in Table 3 are achieved. If all the criteria are not achieved, the a must retune the mass spectrometer and repeat the test all criteria are achieved.

10.2 INITIAL CONDITIONS

10.2.1 Acquire GC/MS data for performance tests, standards and samples using the following instrumental analytes:

Electron Energy:	70 V (nominal)
Mass Range:	35 to 300 amu
Scan Time:	To give at least 5 scans per peak but not to exceed 2 s per scan.

10.2.2 The operating conditions for the gas chromatograph are summarized under Sections 5.3.2 through 5.3.4.. Tables 1 and 2 list the retention times and MDL that can be achieved under these conditions. Examples of separations achieved with this method are shown in Figures 5–7. Other columns or chromatographic conditions may be used if the requirements of Section 9 are met.

10.3 SAMPLE INTRODUCTION AND PURGING

10.3.1 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device and open the syringe valve on the purging device.

10.3.2 Remove the plungers from two 25-mL syringes and attach a closed syringe valve to each. Warm the sample to room temperature, open the sample (or standard) bottle, and carefully pour the sample into one of the syringe barrels to just short of overflowing. Replace the syringe plunger, invert the syringe, and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 25.0 mL. Add 10 uL of the internal standard spiking solution (Section 6.5) to the sample through the syringe valve. Close the valve. Fill the second syringe in an identical manner from the same sample bottle. Reserve this second syringe for a reanalysis if necessary.

10.3.3 Attach the sample syringe valve to the syringe valve on the purging device. Be sure that the trap is cooler than 25°C, then open the sample syringe valve and inject the sample into the purging chamber. Close both valves and initiate purging. Purge the sample for 11.0 ± 0.1 min at ambient temperature (Fig. 3).

10.4 SAMPLE DESORPTION - The mode of sample desorption is determined by the type of capillary column employed for the analysis. When using a wide-bore capillary column, follow the desorption conditions of Sect. 10.4.1. The conditions for using narrow bore columns is described in Sect. 10.4.2.

10.4.1 Sample Desorption for Wide-Bore Capillary Column

Under most conditions, this type of column must be interfaced to the MS through an all-glass jet separator.

10.4.1.1 After the 11-min purge, attach the trap to the chromatograph, adjust the purge and trap system the desorb mode (Fig. 4) and initiate the temperature program sequence of the gas chromatograph and start data acquisition. Introduce the trapped materials to the GC colum rapidly heating the trap to 180°C while backflushing the trap with an inert gas at 15 mL/min for 4.0 ± 0.1 min. While the extracted sample is being introduced into the gas chromatograph, empty the purging device using t sample syringe and wash the chamber with two 25 flushes of reagent water. After the purging de has been emptied, leave the syringe valve open allow the purge gas to vent through the sample introduction needle.

10.4.1.2 Gas Chromatography - Hold the column temperatur 10°C for 5 min, then program at 6°C/min to 160° and hold until all analytes elute.

10.4.1.3 Trap Reconditioning - After desorbing the sampl for 4 min, recondition the trap by returning th purge and trap system to the purge mode. Wait s, then close the syringe valve on the purging device to begin gas flow through the trap. Maintain the trap temperature at 180°C. After approximately 7 min, turn off the trap heater a open the syringe valve to stop the gas flow thr the trap. When the trap is cool, the next sam can be analyzed.

10.4.2 Sample Desorption for Narrow-Bore Capillary Column

Under normal operating conditions, most narrow-bore capillary columns can be interfaced directly to the MS without a jet separator.

10.4.2.1 Sample Desorption - After the 11 min purge, at the trap to the cryogenically cooled interface -150°C and adjust the purge and trap system to desorb mode (Fig. 4). Introduce the trapped materials to the interface by rapidly heating trap to 180°C while backflushing the trap with

inert gas at 4 mL/min for 5.0 ± 0.1 min. While the extracted sample is being introduced into the interface, empty the purging device using the sample syringe and rinse the chamber with two 25-mL flushes of reagent water. After the purging device has been emptied, leave the syringe valve open to allow the purge gas to vent through the sample introduction needle. After desorbing for 5 min, flash heat the interface to 250°C and quickly introduce the sample on the chromatographic column. Start the temperature program sequence, and initiate data acquisition.

10.4.2.2 Gas Chromatography - Hold the column temperature at 10°C for 5 min, then program at 6°C/min to 70°C and then at 15°/min to 145°C.

10.3.2.3 Trap Reconditioning - After desorbing the sample for 5 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s, then close the syringe valve on the purging device to begin gas flow through the trap. Maintain the trap temperature at 180°C. After approximately 15 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

10.5 TERMINATION OF DATA ACQUISITION - When sample components have eluted from the GC, terminate MS data acquisition and store data files on the data system storage device. Use appropriate data output software to display full range mass spectra and appropriate EICPs. If any ion abundance exceeds the system working range, dilute the sample aliquot in the second syringe with reagent water and analyze the diluted aliquot.

11. QUALITATIVE IDENTIFICATION

11.1 IDENTIFICATION PROCEDURES CRITERIA — Tentatively identify a sample component by comparison of its mass spectrum (after background subtraction) to a reference spectrum in a collection. Use the following criteria to confirm a tentative identification:

11.1.1 The GC retention time of the sample component must be within 10 s of the time observed for that same compound when a calibration solution was analyzed.

11.1.2 All ions that are present above 10% relative abundance in the mass spectrum of the standard must be present in the

mass spectrum of the sample component and should agree within absolute 10%. For example, if an ion has a relati abundance of 30% in the standard spectrum, its abundance the sample spectrum should be in the range of 20 to 40%.

11.1.3 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. Because purgeable organic compounds are relatively small molecules and produce comparatively simple mass spectra, this is not a significant problem for most method analyt When GC peaks obviously represent more than one sample c ponent (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra background spectra can be selected by examining EICPs of characteristic ions for tentatively identified component When analytes coelute (i.e., only one GC peak is apparen the identification criteria described in Section 11.1.2 be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

11.1.4 Structural isomers that produce very similar mass spectr can be explicitly identified only if they have sufficien different GC retention times. Acceptable resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak height Otherwise, structural isomers are identified as isomeric pairs.

12. CALCULATIONS

12.1 When an analyte has been identified, the quantitation of that analyte should be based on the integrated abundance from the E of the primary characteristic m/z given in Table 6. If the sa produces an interference for the primary m/z, use a secondary characteristic m/z to quantitate. Instrument calibration for secondary ions is performed, as necessary, using the data and procedures described in Sect. 8.2.

12.2 Calculate the concentration in the sample using the calibratio curve or average response factor (RF) determined in Sect. 8.2. Equation 3:

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})}{(A_{is})(RF)} \qquad \text{Equation 3.}$$

where:

A_s = Area of the characteristic m/z for the analyte to be measured;
A_{is} = Area of the characteristic m/z for the internal standard;
C_{is} = Concentration of the internal standard, in µg/L.

12.3 Report results in µg/L. All QC data obtained should be reported with the sample results.

13. ACCURACY AND PRECISION

13.1 This method has been tested in a single laboratory using spiked reagent water. Using a wide-bore capillary column, water was spiked at concentrations between 0.5 and 10 µg/L (8). Single laboratory accuracy and precision data are presented for the method analytes in Table 4. Calculated MDLs are presented in Table 1.

13.2 The method was tested using reagent water spike at 0.1 to 0.5 µg/L and analyzed on a cryofocused narrow-bore column. The accuracy and precision data for these compounds are presented in Table 5 (9). MDL values were also calculated from these data and are presented in Table 2.

14. REFERENCES

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Table 1. CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC COMPOUNDS ON WIDE BORE CAPILLARY COLUMNS

ANALYTE	RETENTION TIME (mins)		MDL (µg/L)
	Column 1[a]	Column 2[b]	
Dichlorodifluoromethane	1.55	0.70	0.10
Chloromethane	1.63	0.73	0.13
Vinyl chloride	1.71	0.79	0.17
Bromomethane	2.01	0.96	0.11
Chloroethane	2.09	1.02	0.10
Trichlorofluoromethane	2.27	1.19	0.08
1,1-Dichloroethene	2.89	1.57	0.12
Methylene Chloride	3.60	2.06	0.03
trans-1,2-Dichloroethene	3.98	2.36	0.06
1,1-Dichloroethane	4.85	2.93	0.04
2,2-Dichloropropane	6.01	3.80	0.35
cis-1,2-Dichloroethene	6.19	3.90	0.12
Chloroform	6.40	4.80	0.03
Bromochloromethane	6.74	4.38	0.04
1,1,1-Trichloroethane	7.27	4.84	0.08
Carbon Tetrachloride	7.61	5.26	0.21
1,1-Dichloropropene	7.68	5.29	0.10
Benzene	8.23	5.67	0.04
1,2-Dichloroethane	8.40	5.83	0.06
Trichloroethene	9.59	7.27	0.19
1,2-Dichloropropane	10.09	7.66	0.04
Bromodichloromethane	10.59	8.49	0.08
Dibromomethane	10.65	7.93	0.24
Toluene	12.43	10.00	0.11
1,1,2-Trichloroethane	13.41	11.05	0.10
Tetrachloroethene	13.74	11.15	0.14
1,3-Dichloropropane	14.04	11.31	0.04
Dibromochloromethane	14.39	11.85	0.05
1,2-Dibromoethane	14.73	11.83	0.06
1-Chlorohexane	15.46	13.29	0.05
Chlorobenzene	15.76	13.01	0.04
1,1,1,2-Tetrachloroethane	15.94	13.33	0.05
Ethylbenzene	15.99	13.39	0.06
p-Xylene	16.12	13.69	0.13
m-Xylene	16.17	13.68	0.05
o-Xylene	17.11	14.52	0.11
Styrene	17.31	14.60	0.04
Bromoform	17.93	14.88	0.12
Isopropylbenzene	18.06	15.46	0.15
1,1,2,2-Tetrachloroethane	18.72	16.35	0.04
Bromobenzene	18.95	15.86	0.03

Table 1. (Continued)

ANALYTE	RETENTION TIME (mins)		MDL (µg/L)
	Column 1[a]	Column 2[b]	
1,2,3-Trichloropropane	19.02	16.23	0.32
n-Propylbenzene	19.06	16.41	0.04
2-Chlorotoluene	19.34	16.42	0.04
1,3,5-Trimethylbenzene	19.47	16.90	0.05
4-Chlorotoluene	19.50	16.72	0.06
tert-Butylbenzene	20.28	17.57	0.14
1,2,4-Trimethylbenzene	20.34	17.70	0.13
sec-Butylbenzene	20.79	18.09	0.13
p-Isopropyltoluene	21.20	18.52	0.12
1,3-Dichlorobenzene	21.22	18.14	0.12
1,4-Dichlorobenzene	21.55	18.39	0.03
n-Butylbenzene	22.22	19.49	0.11
1,2-Dichlorobenzene	22.52	19.17	0.03
1,2-Dibromo-3-Chloropropane	24.53	21.08	0.26
1,2,4-Trichlorobenzene	26.55	23.08	0.04
Hexachlorobutadiene	26.99	23.68	0.11
Naphthalene	27.17	23.52	0.04
1,2,3-Trichlorobenzene	27.78	24.18	0.03
INTERNAL STANDARDS/SURROGATES			
Fluorobenzene	8.81	6.45	
p-Bromofluorobenzene	18.63	15.71	
1,2-Dichlorobenzene-d_4	22.26	19.14	

[a]Column 1 - 60 meter x 0.75mm ID VOCOL capillary. Hold at 10°C for 5 m then program to 160°C at 6°/min.

[b]Column 2 - 30 meter x 0.53mm ID DB-624 mega-bore capillary. Hold at 1 for 5 min, then program to 160°C at 6°/min.

Table 2. CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC COMPOUNDS ON NARROW BORE CAPILLARY COLUMN

ANALYTE	RETENTION TIME (mins) Column 3	MDL (ug/L)
Dichlorodifluoromethane	0.88	0.11
Chloromethane	0.97	0.05
Vinyl chloride	1.04	0.04
Bromomethane	1.29	0.06
Chloroethane	1.45	0.02
Trichlorofluoromethane	1.77	0.07
1,1-Dichloroethene	2.33	0.05
Methylene Chloride	2.66	0.09
trans-1,2-Dichloroethene	3.54	0.03
1,1-Dichloroethane	4.03	0.03
cis-1,2-Dichloroethene	5.07	0.06
2,2-Dichloropropane	5.31	0.08
Chloroform	5.55	0.04
Bromochloromethane	5.63	0.09
1,1,1-Trichloroethane	6.76	0.04
1,2-Dichloroethane	7.00	0.02
1,1-Dichloropropene	7.16	0.12
Carbon Tetrachloride	7.41	0.02
Benzene	7.41	0.03
1,2-Dichloropropane	8.94	0.02
Trichloroethene	9.02	0.02
Dibromomethane	9.09	0.10
Bromodichloromethane	9.34	0.03
Toluene	11.51	0.08
1,1,2-Trichloroethane	11.99	0.08
1,3-Dichloropropane	12.49	0.06
Dibromochloromethane	12.80	0.07
Tetrachloroethene	13.20	0.05
1,2-Dibromoethane	13.60	0.10
Chlorobenzene	14.33	0.03
1,1,1,2-Tetrachloroethane	14.73	0.07
Ethylbenzene	14.73	0.03
p-Xylene	15.30	0.06
m-Xylene	15.30	0.03
Bromoform	15.70	0.20
o-Xylene	15.78	0.06
Styrene	15.78	0.27
1,1,2,2-Tetrachloroethane	15.78	0.20
1,2,3-Trichloropropane	16.26	0.09
Isopropylbenzene	16.42	0.10

Table 2. (Continued)

ANALYTE	RETENTION TIME (mins)	MDL (µg/L)
Bromobenzene	16.42	0.11
2-Chlorotoluene	16.74	0.08
n-Propylbenzene	16.82	0.10
4-Chlorotoluene	16.82	0.06
1,3,5-Trimethylbenzene	16.99	0.06
tert-Butylbenzene	17.31	0.33
1,2,4-Trimethylbenzene	17.31	0.09
sec-Butylbenzene	17.47	0.12
1,3-Dichlorobenzene	17.47	0.05
p-Isopropyltoluene	17.63	0.26
1,4-Dichlorobenzene	17.63	0.04
1,2-Dichlorobenzene	17.79	0.05
n-Butylbenzene	17.95	0.10
1,2-Dibromo-3-Chloropropane	18.03	0.50
1,2,4-Trichlorobenzene	18.84	0.20
Naphthalene	19.07	0.10
Hexachlorobutadiene	19.24	0.10
1,2,3-Trichlorobenzene	19.24	0.14
INTERNAL STANDARD		
Fluorobenzene	8.81	6.45

Column - 30 meter x 0.32mm ID DB-5 capillary with µm film thickness

Table 3. BFB KEY m/z ABUNDANCE CRITERIA

Mass	m/z Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5 to 9% of mass 174
176	> 95% but < 101% of mass 174
177	5 to 9% of mass 176

Table 4. SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR VOLATILE ORGANIC COMPOUNDS IN REAGENT WATER DETERMINED WITH A WIDE BORE CAPILLARY COLUMN

Analyte	Conc. Range, ug/L	Number of Samples	Recovery,[a] %	Standard Deviation of Recovery[b]
Benzene	0.1 - 10	31	97	5.5
Bromobenzene	0.1 - 10	30	100	5.5
Bromochloromethane	0.5 - 10	24	90	5.7
Bromodichloromethane	0.1 - 10	30	95	5.7
Bromoform	0.5 - 10	18	101	6.4
Bromomethane	0.5 - 10	18	95	7.8
n-Butylbenzene	0.5 - 10	18	100	7.6
sec-Butylbenzene	0.5 - 10	16	100	7.6
tert-Butylbenzene	0.5 - 10	18	102	7.4
Carbon tetrachloride	0.5 - 10	24	84	7.4
Chlorobenzene	0.1 - 10	31	98	5.8
Chloroethane	0.5 - 10	24	89	8.0
Chloroform	0.5 - 10	24	90	5.5
Chloromethane	0.5 - 10	23	93	8.3
2-Chlorotoluene	0.1 - 10	31	90	5.6
4-Chlorotoluene	0.1 - 10	31	99	8.2
1,2-Dibromo-3-chloropropane	0.5 - 10	24	83	16.6
Dibromochloromethane	0.1 - 10	31	92	6.5
1,2-Dibromoethane	0.5 - 10	24	102	4.0
Dibromomethane	0.5 - 10	24	100	5.6
1,2-Dichlorobenzene	0.1 - 10	31	93	5.8
1,3-Dichlorobenzene	0.5 - 10	24	99	6.8
1,4-Dichlorobenzene	0.2 - 20	31	103	6.6
Dichlorodifluoromethane	0.5 - 10	18	90	6.9
1,1-Dichloroethane	0.5 - 10	24	96	5.1
1,2-Dichloroethane	0.1 - 10	31	95	5.1
1,1-Dichloroethene	0.1 - 10	34	94	6.3
cis-1,2 Dichloroethene	0.5 - 10	18	101	6.7
trans-1,2-Dichloroethene	0.1 - 10	30	93	5.2
1,2-Dichloropropane	0.1 - 10	30	97	5.9
1,3-Dichloropropane	0.1 - 10	31	96	5.7
2,2-Dichloropropane	0.5 - 10	12	86	14.6
1,1-Dichloropropene	0.5 - 10	18	98	8.7
Ethylbenzene	0.1 - 10	31	99	8.4
Hexachlorobutadiene	0.5 - 10	18	100	6.8
Isopropylbenzene	0.5 - 10	16	101	7.7
p-Isopropyltoluene	0.1 - 10	23	99	6.7

Table 4. (Continued)

Analyte	Conc. Range, ug/L	Number of Samples	Recovery,[a] %	Standard Deviation of Recovery[b]	Percent Rel. Std. Dev.
Methylene chloride	0.1 - 10	30	95	5.0	5.3
Naphthalene	0.1 - 100	31	104	8.6	8.2
n-Propylbenzene	0.1 - 10	31	100	5.8	5.8
Styrene	0.1 - 100	39	102	7.3	7.2
1,1,1,2-Tetrachloroethane	0.5 - 10	24	90	6.1	6.8
1,1,2,2-Tetrachloroethane	0.1 - 10	30	91	5.7	6.3
Tetrachloroethene	0.5 - 10	24	89	6.0	6.8
Toluene	0.5 - 10	18	102	8.1	8.0
1,2,3-Trichlorobenzene	0.5 - 10	18	109	9.4	8.6
1,2,4-Trichlorobenzene	0.5 - 10	18	108	9.0	8.3
1,1,1-Trichloroethane	0.5 - 10	18	98	7.9	8.1
1,1,2-Trichloroethane	0.5 - 10	18	104	7.6	7.3
Trichloroethene	0.5 - 10	24	90	6.5	7.3
Trichlorofluoromethane	0.5 - 10	24	89	7.2	8.1
1,2,3-Trichloropropane	0.5 - 10	16	108	15.6	14.4
1,2,4-Trimethylbenzene	0.5 - 10	18	99	8.0	8.1
1,3,5-Trimethylbenzene	0.5 - 10	23	92	6.8	7.4
Vinyl chloride	0.5 - 10	18	98	6.5	6.7
o-Xylene	0.1 - 31	18	103	7.4	7.2
m-Xylene	0.1 - 10	31	97	6.3	6.5
p-Xylene	0.5 - 10	18	104	8.0	7.7

a. Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.

b. Standard deviation was calculated by pooling data from three levels.

Table 5. SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR VOLATILE ORGANIC COMPOUNDS IN REAGENT WATER DETERMINED ON A NARROW BORE CAPILLARY COLUMN

Analyte	Conc, ug/L	Number of Samples	Recovery, [a] %	Standard Deviation	Percent Rel. Std. Dev.
Benzene	0.1	7	99	6.2	6.3
Bromobenzene	0.5	7	97	7.4	7.6
Bromochloromethane	0.5	7	97	5.8	6.0
Bromodichloromethane	0.1	7	100	4.6	4.6
Bromoform	0.5	7	101	5.4	5.3
Bromomethane	0.5	7	99	7.1	7.2
n-Butylbenzene	0.5	7	94	6.0	6.4
sec-Butylbenzene	0.5	7	110	7.1	6.5
tert-Butylbenzene	0.5	7	110	2.5	2.3
Carbon tetrachloride	0.1	7	108	6.8	6.3
Chlorobenzene	0.1	7	91	5.8	6.4
Chloroethane	0.1	7	100	5.8	5.8
Chloroform	0.1	7	105	3.2	3.0
Chloromethane	0.5	7	101	4.7	4.7
2-Chlorotoluene	0.5	7	99	4.6	4.6
4-Chlorotoluene	0.5	7	96	7.0	7.3
1,2-Dibromo-3-chloropropane	0.5	7	92	10.0	10.9
Dibromochloromethane	0.1	7	99	5.6	5.7
1,2-Dibromoethane	0.5	7	97	5.6	5.8
Dibromomethane	0.5	7	93	5.6	6.0
1,2-Dichlorobenzene	0.1	7	97	3.5	3.6
1,3-Dichlorobenzene	0.1	7	101	6.0	5.9
1,4-Dichlorobenzene	0.1	7	106	6.5	6.1
Dichlorodifluoromethane	0.1	7	99	8.8	8.9
1,1-Dichloroethane	0.5	7	98	6.2	6.3
1,2-Dichloroethane	0.1	7	100	6.3	6.3
1,1-Dichloroethene	0.1	7	95	9.0	9.5
cis-1,2 Dichloroethene	0.1	7	100	3.7	3.7
trans-1,2-Dichloroethene	0.1	7	98	7.2	7.3
1,2-Dichloropropane	0.5	7	96	6.0	6.3
1,3-Dichloropropane	0.5	7	99	5.8	5.9
2,2-Dichloropropane	0.5	7	99	4.9	4.9
1,1-Dichloropropene	0.5	7	102	7.4	7.3
Ethylbenzene	0.5	7	99	5.2	5.3
Hexachlorobutadiene	0.5	7	100	6.7	6.7
Isopropylbenzene	0.5	7	102	6.4	6.3
p-Isopropyltoluene	0.5	7	113	13.0	11.5

Table 5. (Continued)

Analyte	Conc, ug/L	Number of Samples	Recovery,[a] %	Standard Deviation	Percent Rel. Std. Dev.
Methylene chloride	0.5	7	97	13.0	13.4
Naphthalene	0.5	7	98	7.2	7.3
n-Propylbenzene	0.5	7	99	6.6	6.7
Styrene	0.5	7	96	19.0	19.8
1,1,1,2-Tetrachloroethane	0.5	7	100	4.7	4.7
1,1,2,2-Tetrachloroethane	0.5	7	100	12.0	12.0
Tetrachloroethene	0.1	7	96	5.0	5.2
Toluene	0.5	7	100	5.9	5.9
1,2,3-Trichlorobenzene	0.5	7	102	8.9	8.7
1,2,4-Trichlorobenzene	0.5	7	91	16.0	17.6
1,1,1-Trichloroethane	0.5	7	100	4.0	4.0
1,1,2-Trichloroethane	0.5	7	102	4.9	4.8
Trichloroethene	0.1	7	104	2.0	1.9
Trichlorofluoromethane	0.1	7	97	4.6	4.7
1,2,3-Trichloropropane	0.5	7	96	6.5	6.8
1,2,4-Trimethylbenzene	0.5	7	96	6.5	6.8
1,3,5-Trimethylbenzene	0.5	7	101	4.2	4.2
Vinyl chloride	0.1	7	104	0.2	0.2
o-Xylene	0.5	7	106	7.5	7.1
m-Xylene	0.5	7	106	4.6	4.3
p-Xylene	0.5	7	97	6.1	6.3

a. Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.

Table 6. CHARACTERISTIC MASSES (m/z) FOR PURGEABLE ORGANICS COMPOUNDS

Analyte	Primary Characteristic Ion	Secondary Characteristic Ions
Benzene	78	-
Bromobenzene	156	77,158
Bromochloromethane	128	49,130
Bromodichloromethane	83	85,127
Bromoform	173	175,254
Bromomethane	94	96
n-Butylbenzene	91	92,134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91,134
Carbon tetrachloride	117	119
Chlorobenzene	112	77,114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-Chloropropane	75	155,157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109,188
Dibromomethane	93	95,174
1,2-Dichlorobenzene	146	111,148
1,3-Dichlorobenzene	146	111,148
1,4-Dichlorobenzene	146	111,148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65,83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61,63
cis-1,2-Dichloroethene	96	61,98
trans-1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110,77
Ethylbenzene	91	106
Hexachlorobutadiene	225	223,227
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134,91
Methylene chloride	84	86,49
Naphthalene	128	-
n-Propylbenzene	91	120
Styrene	104	78
1,1,1,2-Tetrachloroethane	131	133,119

Table 6. (Continued)

Analyte	Primary Ion	Secondary Ions
1,1,2,2-Tetrachloroethane	83	131,85
Tetrachloroethene	166	168,129
Toluene	92	91
1,2,3-Trichlorobenzene	180	182,145
1,2,4-Trichlorobenzene	180	182,145
1,1,1-Trichloroethane	97	99,61
1,1,2-Trichloroethane	83	97,85
Trichloroethene	95	130,132
Trichlorofluoromethane	101	103
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl Chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
INTERNAL STANDARDS/SURROGATES		
Fluorobenzene	96	70
1,2-Dichlorobenzene-d_4	150	115,152
p-Bromofluorobenzene	95	174,176

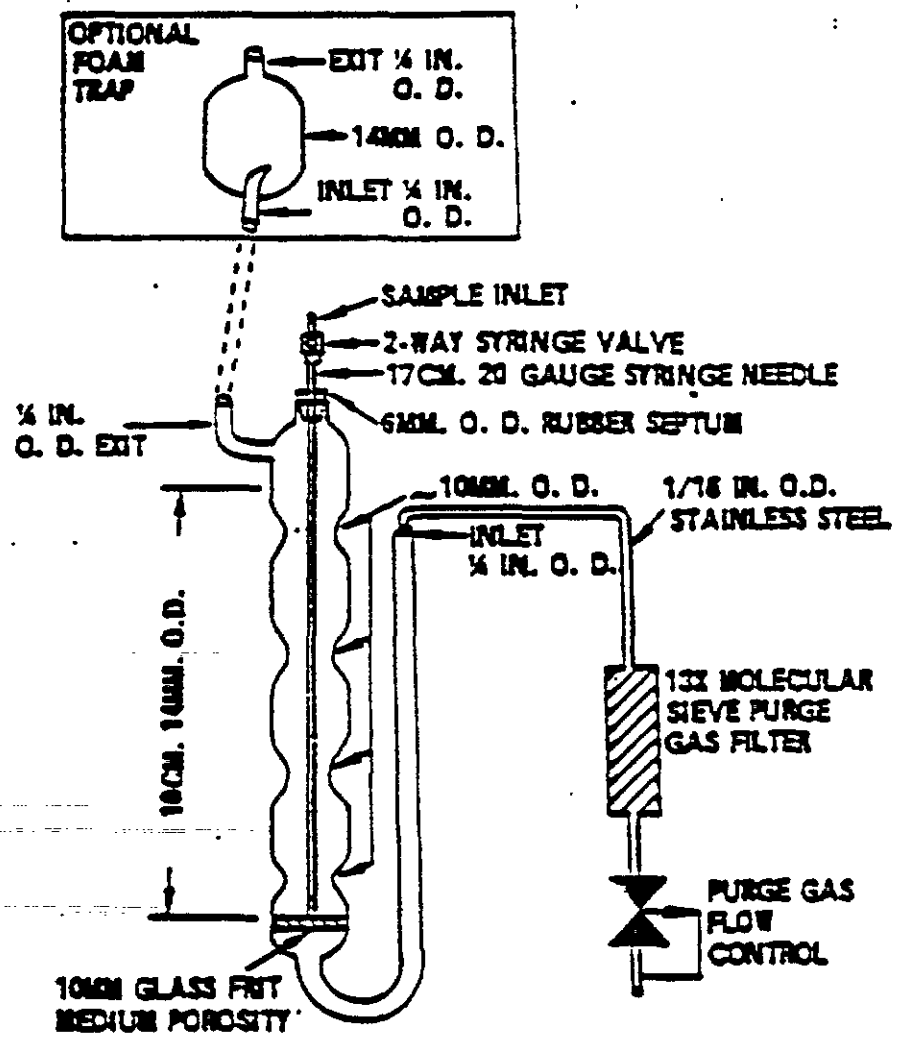

Figure 1. Purging device

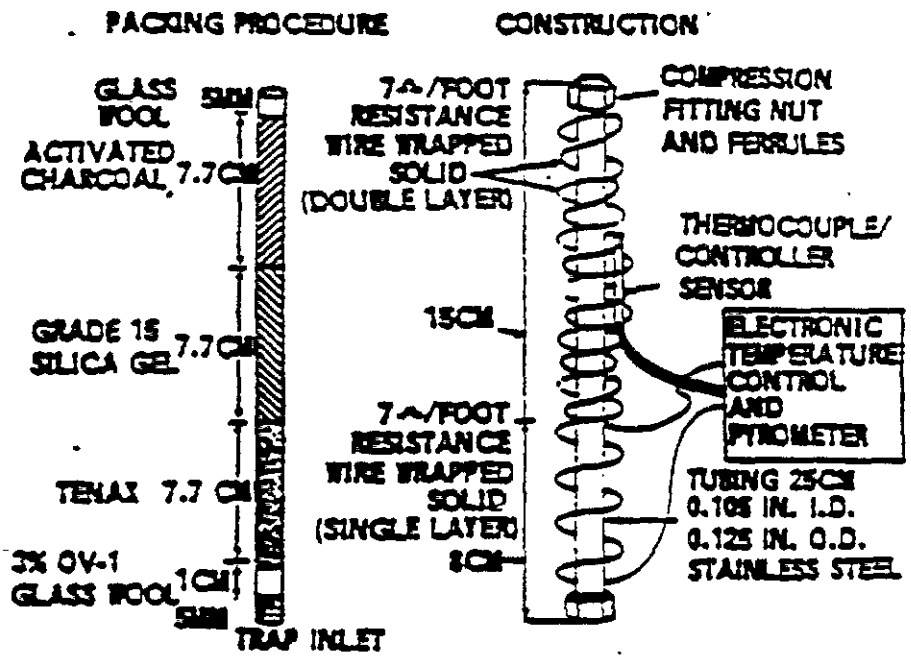

Figure 2. Trap packings and construction to include desorb capability

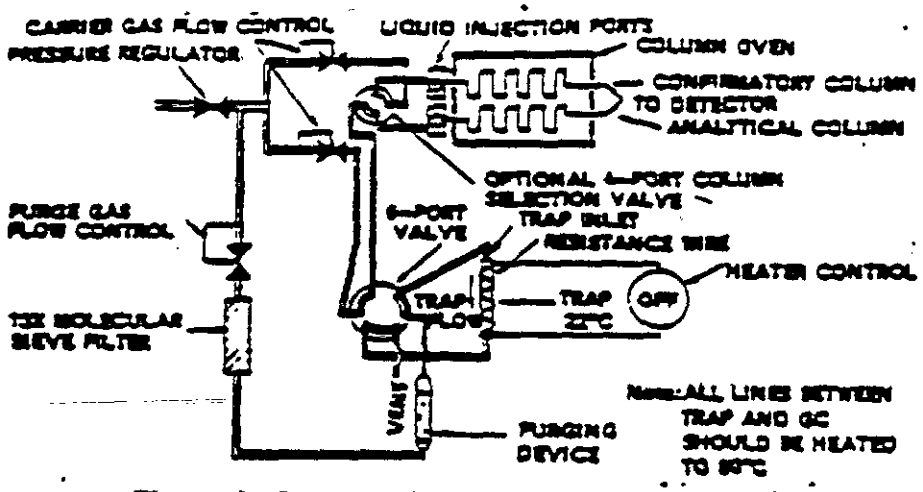

Figure 3. Purge and trap system - purge mode.

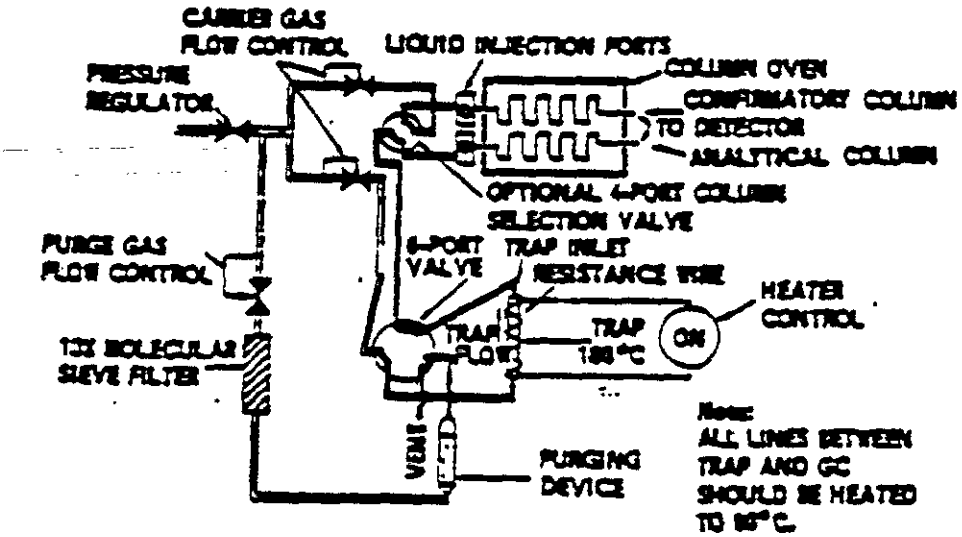

Figure 4. Schematic of purge and trap device - desorb mode

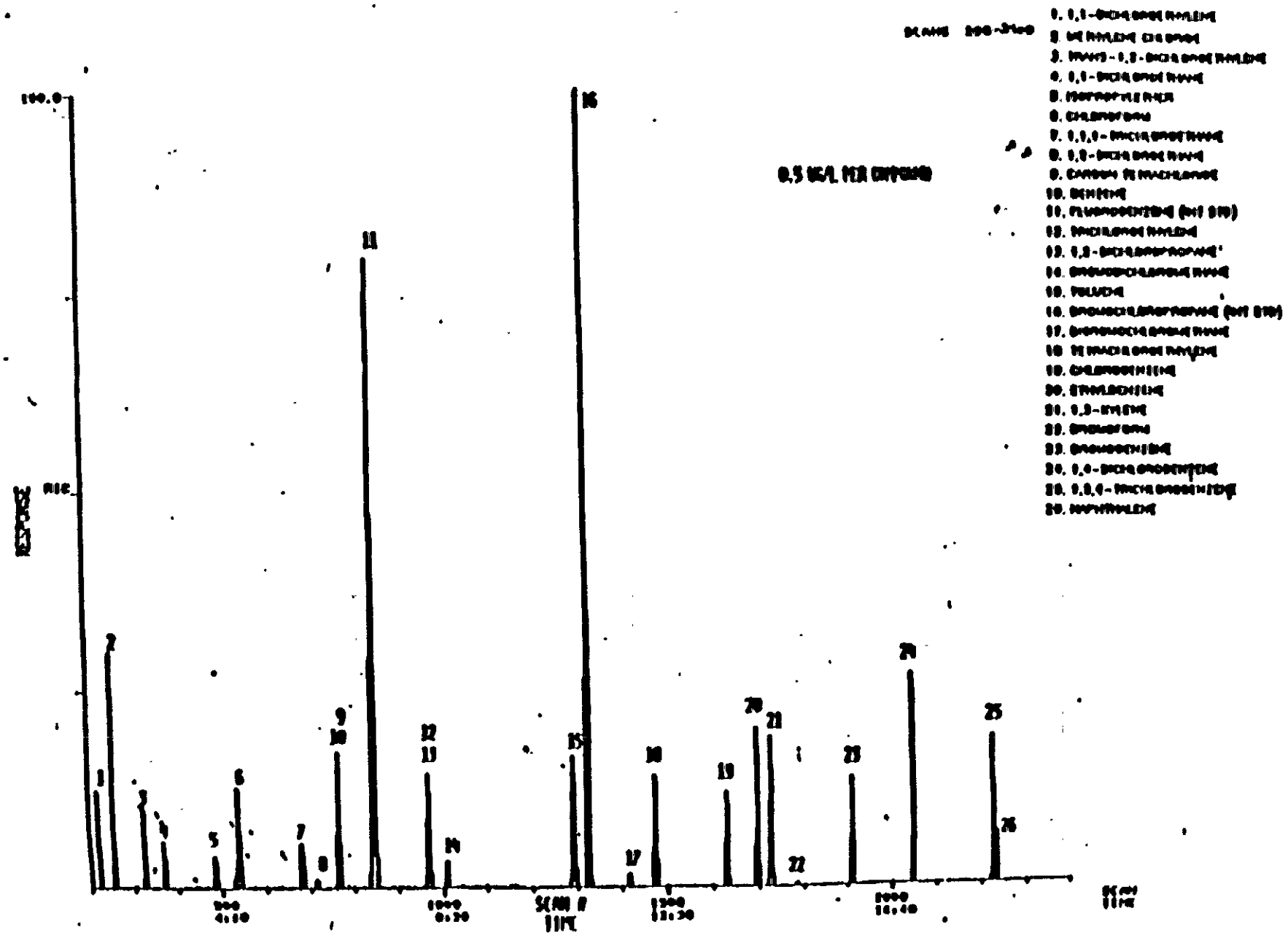

Figure 7. CHROMATOGRAM OF TEST MIXTURE

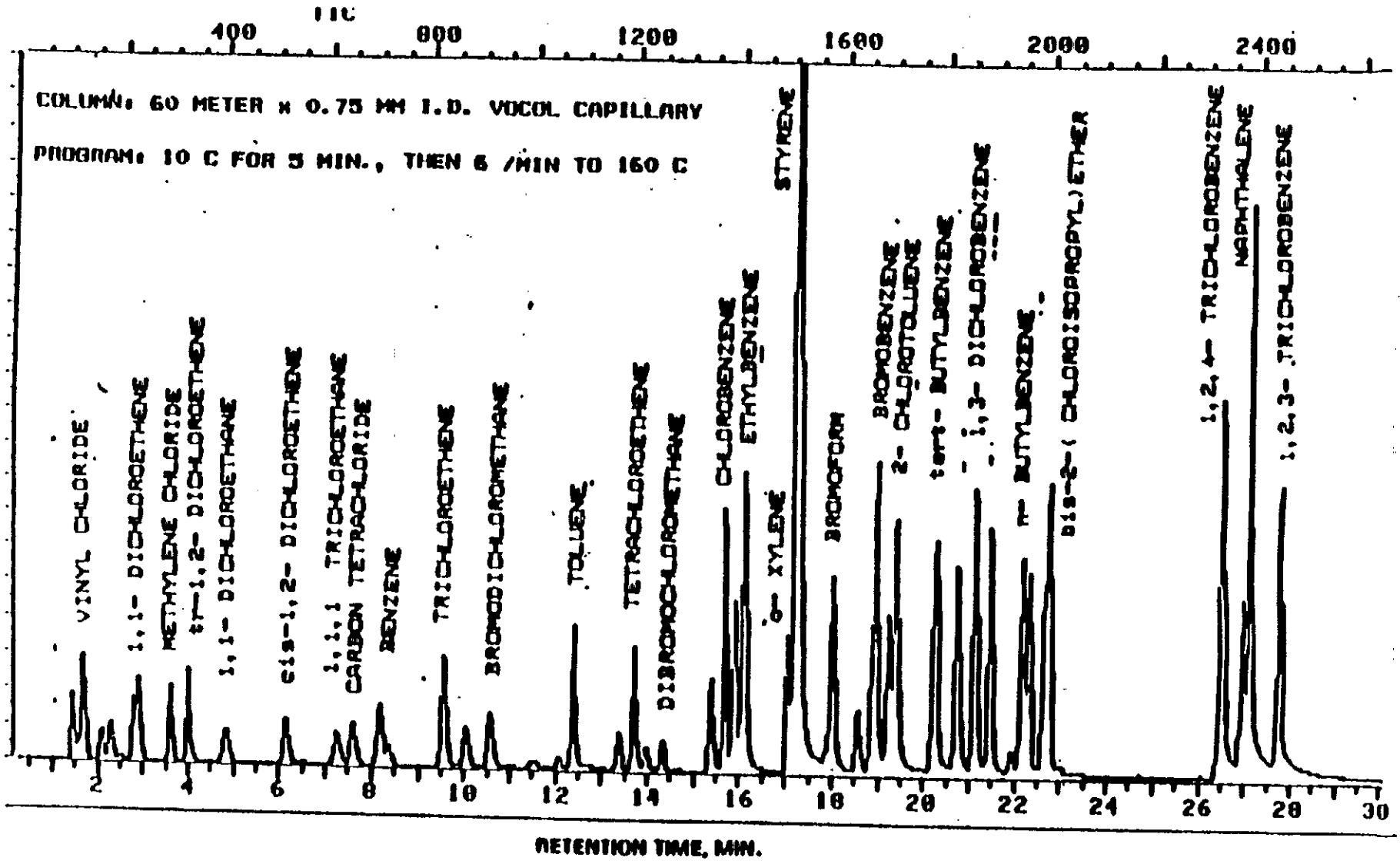

Figure 3. Gas chromatogram of volatile organics.

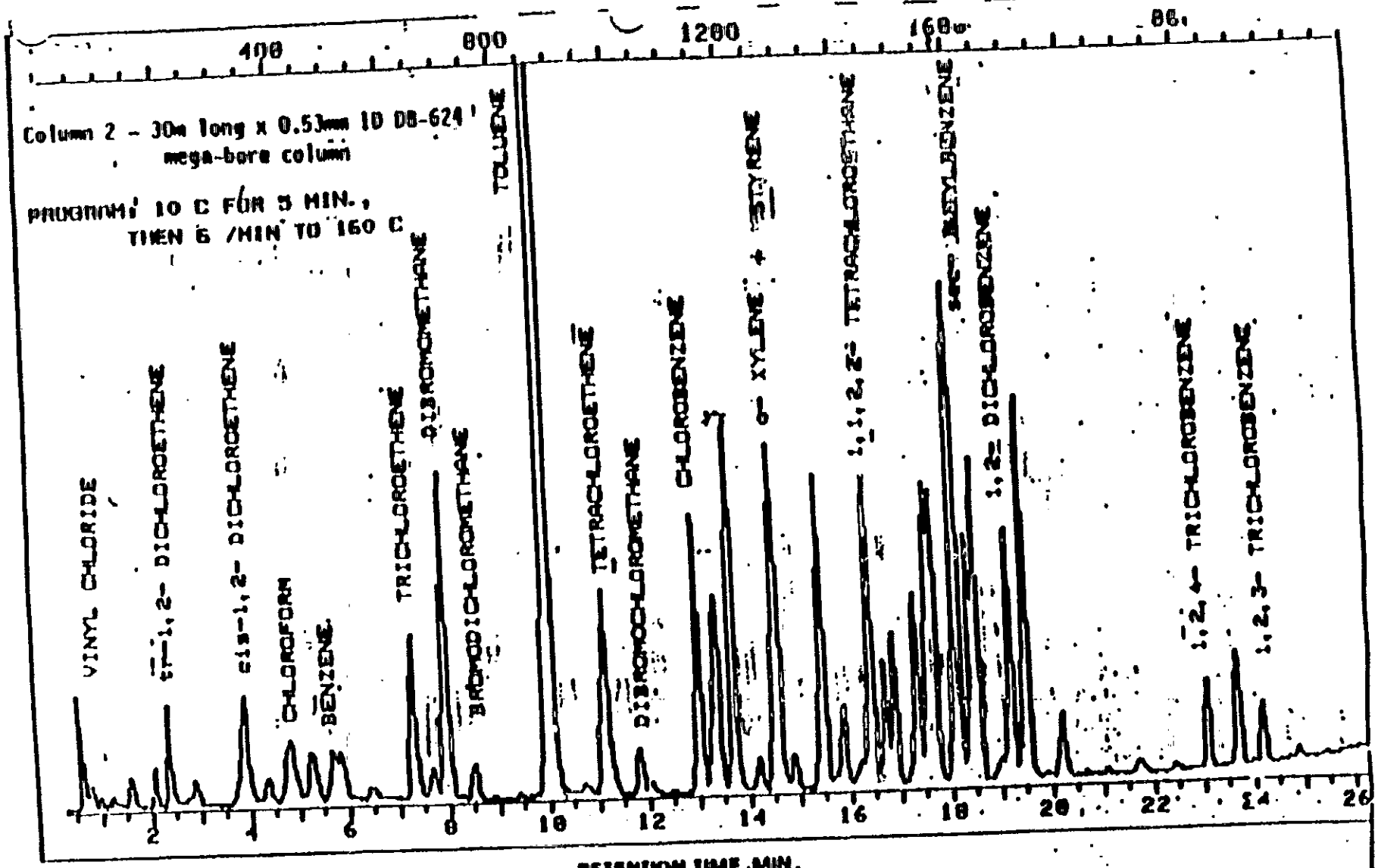

Figure 6. Gas chromatogram of volatile organics.

U.S. Environmental Protection Agency SAS Number
CLP Sample Management Office [ ]
209 Madison Street, Alexandria, VA 22313
Phone: (703) 557-2490 or FTS 557-2490

SPECIAL ANALYTICAL SERVICES
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Client: EPA Region III/CH2M HILL

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Raymark Site, Montgomery County, PA

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. Description of analytical service requested:

Analyze 6 soil samples and 2 sediment samples for Total Organic Carbon (TOC) using Method S1D, S3 - from EPA/CE-81-1 page 3-73 to 3-76 (Attachment 1). Aqueous blank samples for TOC analysis are not applicable.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

Analyze 4 soil samples, 2 soil duplicate samples, 1 sediment sample, and 1 sediment duplicate sample for TOC using Method S1D,S3 from - EPA/CE-81-1, page 3-73 to 3-76.

3. Purpose of Analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund RI/FS.

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment:

Samples shipped by overnight courier.

6. Approximate number of days results required after lab receipt of samples:

Results of analyses are requested forty (40) days after receipt of samples by the laboratory.

7. Analytical protocol required (attach copy if other than protocol currently used in this program):

Analyze all samples using Method S1D,S3 found on pages 3-73 to 3-76 in "Procedures for Handling and Chemical Analysis of Sediment and Water Samples", Technical Report EPA/CE-81-1, R.H. Plumb, Jr., 1981. Method attached.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detention limits, etc.):

Samples must be analyzed within 28 days from the date of collection.
Perform duplicate analysis on one in every 10 samples, per week or fraction thereof. Standardize equipment according to manufacturer's instructions.
Detection limit must be 10 mg/kg or less.

9. Analytical results (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedures and specific insrument used must be clearly identified. Bench records tabulating order of calibration of standards, lab blanks, samples, lab control standards, spikes, duplicates, etc., with resulting output on concentration readout must be provided along with worksheets used to calculate results. Specify the organic compound used to prepare the standards and spikes. A photocopy of the instrument readout, i.e., stripcharts, printer, tapes, etc., must be included. Results are to be recorded in mg/kg dry weight. Records of analysis and calculations must be legible and sufficient to recalculate all concentrations. EPA QC reference samples, or any other reference sample or identical calibration verification, must be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analytical results must be provided for all reference samples used.

Data package must include: all raw data, all instrument and/or equipment calibration results, calculations, duplicate results, chain-of-custody forms, SAS request forms, SAS packing lists, copy of airbill(s), and copies of analyst's logbooks (signed by analyst) with date and time of sample preparation and analysis.

The cover page and all sample report forms MUST be labelled with the complete EPA sample number as it appears on the chain-of custody and CLP paperwork. The case narrative must document all problems encountered and the subsequent resolutions.

10. Other (use additional sheets or attached supplementary information, as needed):

The awarded laboratory is responsible for meeting all requirements as specified in this client request. Any changes in method(s) or other specifications must be approved by Region III prior to the award. If these stipulations are not met, Region III will recommend review for liquidation damages.

11. Name of sampling/shipping contact:

Tom McLaughlin Phone: (703) 471-6405

12. Data Requirements

Parameter	Detection Limit (+/- % or conc.)	Precision Desired
TOC	10 mg/kg	+/- 20 %

13. QC Requirements

Audits Required	Frequency of Audits	Limits (+/- % or conc.)

All QA/QC requirements must be performed and reported as specified in the CLP SOW for Organics Analysis (10/86). The procedures, frequencies and acceptance criteria used shall be the same as specified in the SOW.

14. Action Required if Limits are Exceeded:

Contact Colleen K. Walling, USEPA and Tom McLaughlin, CH2M HILL.

15. Request Prepared by: Laura Gavin/CH2M HILL

Date: February 7, 1990

16. Request reviewed by:

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

ENVIRONMENTAL PROTECTION AGENCY/
CORPS OF ENGINEERS
TECHNICAL COMMITTEE ON CRITERIA
FOR DREDGED AND FILL MATERIAL

PROCEDURES FOR HANDLING AND CHEMICAL ANALYSIS OF SEDIMENT AND WATER SAMPLES

by

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May 1981

Prepared for U.S. Environmental Protection Agency/Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material

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CARBON, TOTAL ORGANIC AND INORGANIC

Carbon may exist in sediment and water samples as either inorganic or organic compounds. Inorganic carbon is present as carbonates, bicarbonates, and possibly free carbon dioxide. Specific types of compounds that are considered to be included in the organic carbon fraction are nonvolatile organic compounds (sugars), volatile organic compounds (mercaptans), partially volatile compounds (oils), and particulate carbonaceous materials (cellulose).[1,2*]

The basis of the method is the catalytic or chemical oxidation of carbon in carbon-containing compounds to carbon dioxide followed by the quantification of the carbon dioxide produced. Alternately, the carbon may be reduced to methane and appropriately quantified. It follows, then, that the distinction between inorganic carbon and organic carbon is the method of sample pretreatment. There are presently two procedures for defining this separation. One method is based on sample treatment with a strong acid. Analysis of an untreated sample is a measure of total carbon while analysis of the acid-treated fraction is a measure of organic carbon. Inorganic carbon is calculated by subtraction. The second method of separation is based on differential thermal combustion with organic compounds being converted to carbon dioxide at 500°C to 650°C[3,4] and inorganic carbon being converted to carbon dioxide at 950°C to 1300°C.[4,5]

Sample Handling and Storage

Flowcharts for the handling of samples intended for organic carbon and inorganic carbon analysis are presented in Figure 3-6 and Figure 3-7. Water and sediment samples to be analyzed for inorganic carbon may be stored in glass or plastic containers. There is no effective preservative because of the carbon dioxide reserve in the atmosphere. The only precaution that can be taken for inorganic

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* References for this procedure can be found on page 3-76.

carbon is to completely fill the sample container at the time of sampling (exclude all air bubbles), tightly seal the container, and complete the analysis immediately (Figure 3-6).

Water samples for organic carbon analysis should be stored in glass containers unless substitute containers have been shown not to affect total organic carbon (TOC) analyses. Samples should be processed as soon as possible (within 24 hr if possible) to minimize change due to chemical or biological oxidation. Atmospheric uptake of carbon dioxide is less critical since it would be evolved when the sample is acidified prior to analysis. Sediment samples for organic carbon analysis may be stored in either plastic or glass containers (Figure 3-7). Air drying of sediments (S2) may lead to low TOC results due to oxidation or volatilization. Therefore, moist storage (S1) or frozen storage (S3) would be the preferred method of storage. If samples are frozen, excessive temperatures should not be used to thaw the samples.

Procedures for Sediment Samples (S1D, S3)

Method 1: Sample Ignition (Use this method)

Apparatus

Induction furnace such as the Leco WR-12, Dohrmann DC-50, Coleman CH analyzer, or Perkin Elmer 240 elemental analyzer

Combustion boats

Microbalance

Desiccator

Reagents

10 percent hydrochloric acid: mix 100 ml concentrated HCl with 900 ml distilled water.

Copper oxide fines.

Benzoic acid.

Procedure

Dry at 70°C and grind the sediment sample.

Weigh a combustion boat and record the weight. Place 0.2 to 0.5 g homogenized sediment in the combustion boat and reweigh. Combustion boats should not be handled with the bare hand during this process.

If total carbon or inorganic carbon is to be determined, Cupric oxide fines may be added to the sample to assist in combustion. Combust the sample in an induction furnace. Record the result as total carbon.

If organic carbon is to be determined, treat a known weight of dried sediment with several drops of 10 percent HCl. Wait until the effervescing is completed and add more acid. Continue this process until the incremental addition of acid causes no further effervescence. Do not add too much acid at one time as this may cause loss of sample due to frothing.

Dry the sample at 70°C and place in a desiccator. Add Cupric oxide fines, combust the sample in an induction furnace, and record the result as organic carbon.

Reweigh sample & crucible & document weight.

Calculations

The carbon content of the sample can be calculated as:

$$\%C = \frac{\text{weight of tube (after-before)}}{\text{sample weight}} \times 27.29$$

Derivation of factor:

$$27.29 = \frac{12.011 \text{ (molecular weight carbon)}}{44.011 \text{ (molecular weight carbon dioxide)}} \times 100\%$$

When the total sample results are used, the result is percent carbon in the sample. When acid-treated samples are used, the result is percent organic carbon. Inorganic carbon is calculated as total carbon minus organic carbon.

U.S. ENVIRONMENTAL PROTECTION AGENCY
Sample Management Office
P.O. Box 8181 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SPECIAL ANALYTICAL SERVICES
Regional Request

X Regional Transmittal _ Telephone Request

A. EPA Region/Client: Region III/CH2M HILL

B. RSCC Representatives: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request: May 9, 1990

E. Site Name: Raymark, Montgomery County, Pennsylvania

Please provide below description of your recent request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for our request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

   Preparation of 500ml amber glass jars with methanol to preserve soil samples.

   Analysis of soil for EPA ICL volatiles using the medium level methodology given in CLP SOW 2/88.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentrations):

   Preparation of 120 500ml amber glass jars with methanol and surrogate compounds.

   Analyze 12 soil samples and 20 QC samples for TCL volatiles (i.e., samples will be preserved with methanol in the field. The laboratory will receive 12 soil/methanol mixtures and 20 methanol QC samples).

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3. Purpose of analysis [specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.]:

   Superfund Remedial

4. Estimated date(s) of collection:

   May 30 - June 15, 1990

5. Estimated date(s) and method of shipment:

   Dailiy by Overnight Carrier

6. Approximate number of days results required after lab receipt of samples:

   Analysis within 10 days of sample collection.

   Results of analyses are requested forty (40) days after receipt of last samples by laboratory.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

   Protocol as per Statement of Work for Organics, SOW No. WA-87J001, WA-87J002, WA-87J003 (2/88 or most recent.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

   See Attachment 1 for sample bottle preparation and sample analytical procedures.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

   ALL deliverables included in the SOW (2/88) required including instrument sensitivity determinations.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Tom McLaughlin or Laura Gavin
    Phone: (703) 471-1441 (201) 316-9300

12. Data Requirements

Parameter	Detection Limit	(+/- % of Concentration
As per SOW (2/88) Medium Level	As per SOW (2/88) Medium Level	As per SOW (2/88) Medium Level

13. Quality Control Requirements

Audits Required	Frequency of Audits	Limits (+/- % or Concentration)
As per SOW (2/88) Medium Level	As per SOW (2/88) Medium Level	As per SOW (2/88) Medium Level

14. Action Required if Limits are Exceeded:

Contact Colleen K. Walling at EPA Region III, (301) 266-9180

15. Request prepared by: Laura Gavin/CH2M HILL
Date: March 15, 1990

16. Request reviewed by:
Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

ATTACHMENT 1

The following procedures are based upon the "Methanol Preservative VOC Method", presented at the "EPA 5th Annual Waste Testing and Quality Assurance Symposium, July 24-28, 1989, Omni Shorum Hotel, Washington, D.C.".

SAMPLE BOTTLE PREPARATION

The laboratory will be provided with 80 empty 500ml amber glass jars with Teflon-lined lids. These bottles must be prepared according to the following procedures:

1. Fill each sample bottle with 250ml of HPLC grade methanol.

2. Add 625µg each of the three surrogate compounds listed below, by syringe to the methanol in each sample bottle:

Surrogate Compounds

Surrogate	% Acceptable Recoveries
1,2-Dichloroethane-d_4	70-121%
Toluene-d_8	81-117%
p-Bromoflurobenzene (BFB)	74-121%

3. Cap each bottle tightly and gently swirl.

4. Weigh each sample bottle containing the 250ml of methanol and the surrogates to the nearest gram, and record the weight on the bottle label, in the laboratory logbook and on the chain-of-custody record (to be shipped with the sample bottles). All sample bottles containing methanol and surrogate compounds must be shipped in paint cans with vermiculite, according to DOT regulations.

5. The empty sample bottles will be shipped to the laboratory by May 15, 1990, 1990. The laboratory must prepare the sample bottles and ship them to the following address by May 23, 1990:

CH2M HILL
99 Cherry Hill Road, Suite 304
Parsippany, New Jersey 07540
Attention: Laura Gavin

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ANALYTICAL PROCEDURES

1. When the sample bottles containing the soil/methanol mixtures arrive at the laboratory, they must be weighed to the nearest gram in order to determine the amount of soil placed into the bottle. Subtracting the weight of the bottle, methanol, and surrogates (weighed by laboratory prior to shipping the bottles in the field) from the total weight of the sample bottles with the soil sample (as returned to the laboratory) will give the wet weights of the soil being methanol extracted for VOC analysis.

2. Samples must be stored at 4°C until analysis.

3. Use the CLP medium level soil method starting with the injection of a quantity of the methanol from the sample bottle into 5 ml of organic-free water in the purge device.

4. The matrix spike and matrix spike duplicate will be accomplished by adding 600 µg each of the required spike compounds listed below:

Matrix Spike and Matrix Spike Duplicate Compounds

Spike Compounds	% Acceptable Recoveries
1,1-dichloroethane	59-172%
Trichloroethene	62-137%
Chlorobenzene	60-133%
Toluene	59-139%
Benzene	66-142%

5. The medium level soil method is based on extracting the soil sample with methanol. An aliquot of the methanol extract is added to reagent water containing the surrogate and internal standards. This is purged at ambient temperature.

6. The GC/MS system should be set up as in Sections 7.12 through 7.14 of the U.S. EPA CLP Statement of Work for Organic Analysis, Revision 2/88. This should be done before adding the methanol extract to reagent water. Initial and continuing calibrations (Section 5.3) are performed by adding standards in methanol to reagent water and purging at ambient temperature. For all other purge and

trap GC/MS method details, please follow the U.S. EPA CLP Statement of Work for Organic Analysis, Revision 2/88, exactly.

7. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of linear range of the curve.

8. The following table can be used to determine the volume of methanol extract to add to the 5 ml of reagent water for analysis.

Concentration Range	Take this Volume of Methanol Extract
500 - 10,00 µg/kg	100 µL
1,000-20,000 µg/kg	50 µL
5,000-100,000 µg/kg	10 µL
25,000-500,000 µg/kg	100 of 1/50 dilution

(Note: Actual concentration ranges could be 10 to 20 times higher than this if the compounds are halogenated and the estimates are from GC/FID).

Calculate the appropriate dilution factor for concentrations exceeding the table.

9. Remove the plunger from 5 ml "Luerlock" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 ml. Pull the plunger back to 5 ml to allow volume for the addition of sample and standards. Add 10 µl of the internal standard solution. Also add the volume of methanol extract determined previously and a volume of methanol solvent to total 100 µL (excluding methanol in standards).

10. Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

11. Proceed with the analysis as outlined in Section 7.1.9 through 7.1.13 of CLP SOW 2/88. Analyze all reagent blanks on the same instrument as the samples. The standard should also contain 100 µl of methanol to simulate the sample conditions.

12. A separate sample aliquot (unpreserved with methanol) will be shipped to the laboratory to be used to calculate the percent moisture for each sample.

13. The standard CLP data package and data forms for all QA and sample results must be used.

Appendix C

CH2M HILL
SITE SAFETY PLAN

I. GENERAL INFORMATION

CLIENT: USEPA Region III (ARCS) JOB NO: NJO63109.PP.PP
PROJECT MANAGER: Joseph Cleary
SITE NAME: Raymark
SITE LOCATION: Hatboro, Pennsylvania
DATE OF VISIT(S): April through July, 1990
BACKGROUND INFORMATION: Complete _____ Preliminary X
INFORMATION AVAILABLE FROM: NJO
OVERALL HAZARD SUMMARY: Serious _____ Moderate _____
Low X Unknown _____
PURPOSE OF FIELD VISIT(S): Field investigation activities for the RI/FS including subsurface sampling, surface water/sediment sampling, well installation, groundwater sampling, and septic sampling. Figures have been included to show the subsurface boring locations, the septic tank location, the monitoring well installation locations and the existing onsite well.

II. SITE CHARACTERISTICS

FACILITY DESCRIPTION (site map attached)

The site is located on Jacksonville Road in Hatboro Borough, Montgomery County, Pennsylvania. The site covers 7 acres, and consists of a manufacturing building and four filled-in lagoons (see Principal Disposal Method below). The site was used to manufacture rivets since approximately 1948. The building is currently leased and operated by Penn Fasteners, a manufacturer of rivets and fasteners.

Principal Disposal Method (type and location):

During 1948-72, treated wastes and untreated wastewater from plating and degreasing operations were disposed of in four lagoons onsite behind the manufacturing plant. In 1972, the lagoons were excavated and filled with clean fill.

Trichloroethylene (TCE) was the solvent used to degrease non-porous ferrous and non-ferrous parts. The TCE was stored in outdoor aboveground storage tanks, which have been removed. One round of soil sampling at the site where the tanks had been located indicated high TCE concentrations at 3 feet below the surface.

Building drains are also a suspected major source of existing soil contamination.

Status (active, inactive, unknown): Active

History (worker or non-worker injury; complaints from public; previous agency action):

Hatboro drinking water wells near the Raymark site were discovered to be contaminated with TCE in 1979. They have since been taken out of service or equipped with air strippers. The Raymark site has been identified as a source of contamination of the Stockton Aquifer, which supplies drinking water to approximately 900,000 people via public and private wells within 3 miles of the site. During EPA's 1985 sampling, 3.1 million micrograms per kilogram of TCE were found in onsite soil. Wells within 250 feet of the site are contaminated with TCE ranging from 14 to 8,600 micrograms per liter. Up to 900 micrograms per liter of 1,2-dichloroethylene have been detected in monitoring wells in the vicinity of the site.

In 1985, the USEPA brought suit against present and past owners of the site (Raymark Industries, Inc., Raymark Formed Products Co., and Penn Fasteners, Inc.) under Section 7003 of RCRA and Section 106 of CERCLA.

III. WASTE CHARACTERISTICS

WASTE TYPE(S)

Liquid __X__ Solid __X__ Sludge __X__ Gas _____

CHARACTERISTIC(S)

Corrosive ____ Ignitable ____ Radioactive ____ Volatile __X__

Toxic __X__ Reactive ____ Unknown ____ Other(Name) ____

IV. HAZARD EVALUATION

Overall Hazard Level:

Low to Moderate. Inhalation is the most significant route of exposure to TCE. Continuous HNu monitoring will be conducted by CH2M HILL personnel during all field activities. Due to the potential for vinyl chloride (a breakdown product of TCE) to be present, Draeger tube air sampling will also be required. If HNU readings >3 ppm are observed, a Draeger tube air sample will be taken. If the draeger tube reads 1 ppm vinyl chloride or higher, field personnel will back off of the sampling area and sampling task will be reevaluated. Venting will probably reduce the vinyl chloride levels.

Chemical Hazards:

Chemical exposure hazards are expected to be relatively low for these sampling events. Inhalation is the most significant route of exposure to TCE. The predominant physiologic response to TCE exposure is central nervous system (CNS) depression. TCE is irritating to the eyes and should be washed out promptly if eye exposure occurs. It is only mildly irritating to the skin if allowed to evaporate.

The threshold limit value for TCE recommended by the ACGIH is 50 ppm (260 mg/m3) and the OSHA PEL is 25 ppm.

TCE has a typical odor that has been characterized as ethereal or chloroform like. It could not, however, be considered an effective warning. TCE is a suspect carcinogen. It affects the central nervous system (euphoria, anaesthesia), liver, kidney, lung, heart, skin, and bone marrow. Alcohol potentiates the nervous system effects of TCE. The following response to various concentrations is based on industrial experience and human studies.

100 ppm	Odor threshold, barely perceptible to unacclimated individuals.
200 ppm	Odor apparent, not unpleasant. Transient mild eye irritation disappearing upon cessation of exposure. Slight fatigue, sleepiness, headache, alcohol intolerance when chronically exposed.
400 ppm	Odor very definite, not unpleasant. Slight eye irritation. Minimal light-headedness after 3 hours.
1000 to 1200 ppm	Odor very strong, unpleasant. Definite eye and nasal irritation. Definite light-headedness, dizziness after 6 minutes.
2000 ppm	Odor very strong, not likely to be tolerated. Markedly irritating to the eyes and respiratory tract. Drowsiness, dizziness, nausea in 5 minutes.

Contaminant avoidance is important in preventing potential exposure. Do not come in contact with soil or groundwater. Remain upwind during site activities involving soil disturbances, sampling, and well venting and purging. Monitor with the HNu during all field activities.

1,2-Dichloroethylene has a TLV of 200 ppm. Overexposure effects include narcosis and irritation of the central nervous system. It has been used as a human anaesthetic with no side effects.

Vinyl chloride is a confirmed human carcinogen with a TLV of 5 ppm. The OSHA is 1 ppm. Overexposure effects to this colorless gas include central nervous system effects of euphoria, followed by inebriation and narcosis. Chronic overexposures may also produce liver and gastrointestinal damage, as well as allergic dermatitis.

Hazards Posed by Site Activities:

Drilling:

Drilling poses safety hazards to personnel in the immediate vicinity of the drill rig. To protect personnel from overhead falling objects (i.e., bolts, wrenches, pieces of pipe), hard hats must be worn in the immediate vicinity of the drilling. Safety glasses are also required to protect against flying projectiles that could be caused by hammering fittings/ connections and driving retaining clips. Continuous monitoring with an HNu will be performed to identify potential inhalation hazards.

Underground utilities located at the site present a hazard during drilling activities. Therefore, prior to conducting any drilling activity, all underground utilities will be identified and properly marked. Although drilling activities near overhead electrical lines are not anticipated, the driller shall maintain a distance of 10 feet from the drill rig mast to all overhead electrical lines. The driller is responsible for safety of his personnel around the drill rig during drilling activities.

Septic Sampling:

Septic sampling poses the hazards of exposure to hepatitis and of explosion due to potentially high levels of methane. Personnel must wear Tyvek coveralls over cotton clothing, chemical safety goggles, neoprene gloves over disposable surgical gloves, and latex booties over steel toe, steel shank neoprene boots. Boots and gloves must be taped to Tyvek. Odor will be a nuisance to personnel in the immediate vicinity of the open septic tank. Personnel not directly involved in the sampling should stay upwind of the open tank. HNu monitoring must be performed upon opening the septic tank and continued throughout the sampling event. No CH2M HILL personnel will enter the septic tank.

Combustible gases may accumulate in the septic tank if the vents become clogged. Care must be taken to not create a spark. The lower explosive limit will be monitored before and after opening the septic tank inlet.

Groundwater Sampling:

Groundwater samples will be collected from eleven onsite wells, ten of which will be installed as part of the RI/FS. The one existing onsite well will be

drilled to sample a deeper aquifer. Due to the potential inhalation hazard, HNu monitoring will be conducted and logged every 30 minutes. To the extent possible, remain upwind during well venting and purging. If HNu readings are greater than 3 ppm and the draeger tube readings for vinyl chloride are greater than 1 ppm, field personnel will back off of the sampling area. In addition, if HNu readings exceed 5 ppm, field personnel are to back off of the sampling area. Venting the well will probably reduce the vinyl chloride and HNu readings.

Subsurface Soil Sampling:

Soil boring samples will be collected from eleven boring locations onsite. Continuous split spoon samples will be collected from the borings. HNu monitoring will be conducted for each split spoon sample and recorded in the log books, as well as at the breathing zone during drilling activities.

Surface Water/Sediment Sampling:

A surface water and sediment sample may be collected if a hydraulic pathway from the site to Pennypack Creek northwest of the site via surface runoff is determined to exist. The surface runoff sample will be collected using remote handling sampling equipment which does not present a splash hazard to personnel. If the sampling equipment cannot be used as designed and field personnel must approach within three feet of the surface water, face shields must be worn to protect from potential splash hazard.

Hazards Posed by Chemical Substances Provided by CH2M HILL:

In accordance with Pennsylvania regulations for hazard communication, Material Safety Data Sheets are provided for the following chemicals:

HNU/MSA explosion meter calibration gas
MSA Sanitizer
Acetone
Nitric Acid
Methanol
Hydrochloric Acid
Sodium Hydroxide

V. PROCEDURES

SITE ORGANIZATION

Map/Sketch Attached X
Site Secured No
Perimeter Identified X
Zone(s) of Contamination Identified No

SITE PERSONNEL

Team Member	Responsibility
Joe Cleary (1,2)	Visitor, no entry onsite (lacks current medical and refresher training)
Jim Burd	Visitor, sampler
Cliff Bell (1,2)	Hydrogeologist/Field Manager
Mary Kate Dwyer	Hydrogeologist, Level D SSC
Jolecia Mitchell (1)	Hydrogeologist
Dawn DeBiasse	Environmental Engineer Alternate Level D SSC
Laura Gavin (1)	Environmental Scientist

1 - No record of current CPR certification.
2 - No record of current first aid certification

The above named team members meet the medical and training requirements of 29 CFR 1910.120 and of CH2M HILL. Unless otherwise stated, each is currently certified in both CPR and first aid. CH2M HILL requires that at least 2 onsite team members be currently certified in both CPR and first aid. The SCC must complete Form 533 weekly and mail it to Ann West/WDC.

Level of Protection

A ____ B ____ C ____ D X

Modifications: Level D; steel toe, steel shank neoprene boots, latex booties, Tyvek, neoprene gloves with surgical inner gloves, and safety glasses. Chemical safety goggles required for septic sampling and other sampling involving a splash hazard. Hard hats are required if overhead equipment is in use, including drill rig, back hoe. Have face shield available for use when there is a splash hazard.

Safety Equipment and Materials:

First aid kit and eye wash bottle with sufficient supplies of clean water to deliver a 15-minute eye wash. Potable water and paper cups will be available for field personnel. CH2M HILL personnel to wear TLD badge if issued to them.

Monitoring Equipment and Procedures:

Carefully inspect each piece of monitoring equipment prior to work startup. Failure of any of the equipment listed below to work properly must be reported to the Project Manager immediately.

HNu (with 10.2 eV lamp):

Calibrate prior to each day's activities, according to manufacturer's instructions. Record calibration in the SSC log book. Take background readings in an upwind position. Background is NOT taken in the area you suspect to be contaminated. Take readings in the breathing zone upon initiating each field activity. Recalibrate after cleaning the lamp or when background levels drift. This instrument is sensitive to humidity and may require periodic lamp cleaning if it is humid. Monitor continuously during sampling and record measured levels in the log books every 30 minutes.

Action Levels:

0-3 ppm:	continue work
3-5 ppm:	monitor with Draeger tube for vinyl chloride
Greater than 5 ppm:	leave the area

Draeger Tube for Vinyl Chloride (6728061):

Leak test pump daily and follow manufacturer's directions for use.

Action Levels:

0-1 ppm:	continue work
Greater than 1 ppm:	leave the area

Explosimeter/O2 Meter:

Calibrate prior to each day's activities, according to manufacturer's instructions. Recharge at the end of each day. Monitor before and after opening the septic tank inlet and during drilling and record measured levels in the log book.

Action Levels:

$\leq 19.5 0_2$:	Evacuate.
0-10% LEL:	Continue. Monitor septic tank inlet valve or manhole prior to opening and continuously after opening and while sampling.
10-20% LEL:	Monitor continuously, be prepared to shut down sampling if LEL approaches 20 percent.
Greater than 20% LEL:	Shutdown operations and evacuate area. Allow vapors to dissipate to below 10 percent LEL before continuing operations.

SITE ENTRY PROCEDURES

- Sign in with facility representative and review facility's contingency plan and communications.
- Locate nearest available telephone.
- Confirm and post emergency telephone numbers and route to hospital.
- Designate at least one vehicle for emergency use. Be sure that each team member knows where the car keys are and that the directions to the hospital are by the driver's seat.
- If toilet and handwashing facilities are not located within a 3 minute walk from the decontamination facilities, either provide chemical toilet and handwashing facilities or have a vehicle available (not the emergency vehicle) for transport to nearby facilities.
- The SSC will conduct and document a site safety briefing for all team members prior to work start-up.
- The SSC shall brief team members on the SSP and emergency procedures and record agenda and attendees in the SSC log book.
- Determine wind direction, and set up decontamination facilities prior to walking around the site.
- Set up exclusion zone(s) and mark the boundaries. Do not allow unauthorized entry into exclusion zones. Inspect for spark sources and eliminate them.

WORK LIMITATIONS (Time of day, etc.)

- All work must be done during daylight hours only.
- No eating, drinking, or smoking onsite.
- No contact lenses onsite.
- Buddy system at all times.
- CH2M HILL employees to wear TLD badges (if issued to them by CH2M HILL) at all times when on or near the site.
- Two persons onsite must be certified in both CPR and first aid.
- Avoid walking or kneeling in areas of obvious contamination.
- No onsite work during electrical storms or heavy rainfall.
- If visible dust arises from the site, wet the soil with water to control dust emissions.
- If temperatures are above 75°F, schedule a beverage break every 2 hours and review heat stress symptoms and prevention in a safety briefing.

DECONTAMINATION PROCEDURES

Personnel:

- Remove disposable booties and place in plastic bag for disposal.
- Wash outer gloves in detergent solution and rinse in clean water. Remove outer gloves and place into plastic bag for disposal or retain for subsequent reuse.
- Remove safety glasses or face shield (if worn). Wash in detergent solution and rinse in clean water.
- Remove Tyvek or Saranex coveralls. Take care to prevent the release and dispersion of dusts that may have accumulated on the coveralls during onsite operations and place coveralls into disposable plastic bag.
- Remove and dispose of inner surgical gloves.
- Thoroughly wash hands and face.

- As soon as possible at the end of work shift, shower and shampoo. Launder clothing worn onsite before rewearing.

Sampling Equipment: Sampling equipment will be decontaminated as outlined in the Field Sampling Plan.

Samples: Follow Field Sampling Plan.

Documentation: It is the responsibility of the SSC to make sure that all equipment coming offsite is properly decontaminated, according to the procedures outlined above. The equipment and samples must be clean, dry and free from stains, deposits, etc. A suitable tag is to be placed on each piece of equipment or package of equipment that is returned to CH2M HILL stating the date of decontamination and initialed by the SSC.

VI. CONTINGENCY PLAN

If an injury occurs, take the following steps:

- Prevent further injury and notify SSC.
- Initiate first aid and get medical attention for the injured immediately.
- Depending upon the type and severity of the injury, call the medical consultant and/or occupational physician.
- Notify the Health and Safety Manager.
- Notify the injured person's personnel office.
- Prepare an incident report. The SSC is responsible for ensuring its preparation and submittal to the Health and Safety Director and CH2M HILL corporate personnel office within 48 hours.
- The SSC will assume charge during a medical emergency.

In the event of a fire, explosion, or chemical release, team members are to immediately evacuate upwind. The SSC assumes control for CH2M HILL personnel and directs a team member to call the fire department and to notify the facility. The SSC may direct control measures if these measures do not endanger team members and such controls will not increase the hazard to others. The SSC briefs the fire department when they arrive. Work shall not continue until the fire department declares the incident controlled.

LOCAL:

(CH2M HILL Form 311, Emergency Phone Numbers, to be posted)

Ambulance: 215-441-6600

Hospital: 215-441-6600 - Warminster General

Poison Control Center: 1-800-962-1253

Police: 215-675-4400

Fire: 215-672-1221

EMERGENCY ROUTES (map attached to plan)

Follow Jacksonville Road north to County Line Road (approximately 1/2 mile). Turn right. After two traffic lights, come to Newtown Road. Turn left. Follow Newtown Road for 3 blocks (approximately 1/2 mile). Hospital is on the right at 225 Newtown Road.

EMERGENCY CONTACTS

- CH2M HILL Medical Consultant

  Name: Dr. Kenneth Chase, Washington Occupational Health Associates, Inc.
  Phone: 202-463-6698 (8-5 EST)
  202-463-6440 (after hours answering service; physician will return call within 30 minutes)

- CH2M HILL Health and Safety Manager

  Name: Mary Anne Chillingworth/WDC
  Phone: 703-471-1441 (O)
  703-476-0882 (H)

- Occupational Physician

  Name: Envirocare
  Phone: 201-225-5454
  Address: 135 Raritan Center Parkway
  Edison, NJ

  Team members under his care:
  All NJO personnel

- CH2M HILL Project Manager

  Name: Joseph Cleary
  Phone: 201-316-9300

- Client Contact

  Name: Michael Towle
  Phone: 215-597-3166

- CH2M HILL Regional Manager

  Name: Steve Guttenplan
  Phone: 201-316-9300

- CH2M HILL Personnel Office

  Name: Sharon Springer
  Phone: 201-316-9300

If an injury occurs, notify the injured person's personnel office as soon as possible after obtaining medical attention for the injured. Notification MUST be made within 24 hours of the injury.

- CH2M HILL Director of Health and Safety

  Name: David Lincoln/SEA (Acting)
  Phone: 206-453-5000

- CH2M HILL Corporate Personnel Office

  Name: Sharon Robinson/CVO
  Phone: 503-752-4271
  Address: CH2M HILL
  2300 N.W. Walnut Blvd.
  Corvallis, OR 97330

VII. PLAN APPROVAL

This site safety plan has been written for the use of CH2M HILL, its employees and subcontractors. CH2M HILL claims no responsibility for its use by others. The plan is written for the specific site conditions, purposes, dates and personnel specified and must be amended if these conditions change.

PLAN PREPARED BY: Dawn M. DeBiasse Date: Jan. 15, 1990

APPROVED BY: Mary Anne Chillingworth Date: 2/2/90
Mary Anne Chillingworth

Attachments

- Site Map
- Form 311, Emergency Phone Numbers
- Form 533, Record of Hazardous Waste Field Activity
- MSDS for:
  - Pentane
  - Isobutylene
  - Acetone
  - Nitric Acid
  - Methanol
  - Hydrochloric Acid
  - Sodium Hydroxide

Distribution of approved plan:

Site Manager (responsible for distribution to team members and client.

Health and Safety Manager

WDCR471/003.50

EMERGENCY PHONE NUMBERS

	phone	address	contact
POLICE DEPARTMENT	675-4400		
			attn:
FIRE DEPARTMENT	672-1221		
			attn:
PARAMEDIC			
			attn:
FIRE REPORT			
			attn:
AMBULANCE SERVICE	441-6600		
			attn:
WATER DEPARTMENT			
			attn:
GAS UTILITY			
			attn:
ELECTRIC UTILITY			
			attn:
TELEPHONE UTILITY			
			attn:
LOCAL SANITARIAN			
			attn:
HOSPITAL	Warminster General	225 Newtown Rd	441-6600
			attn:
OWNER			
			attn:
			attn:
			attn:
			attn:

this notice is located at: _____ WDCHS6/003.50

AR301402

FORM 533

RECORD OF HAZARDOUS WASTE FIELD ACTIVITY

SITE NAME: Raymark
SITE SAFETY COORDINATOR:
PROJECT NUMBER: NJO63109.PP.PP
RECORD OF ACTIVITIES FOR (DATES):

Employee Name	Total Days Onsite	Days at the Site in Level B	Days at the Site in Level C	Days at the Site in Level D	or	Number of days as SSC Level B	Number of days as SSC Level C	Number of days as SSC Level D	Activities Employees Performed While Onsite
05943 Jim Burd									
07699 Cliff Bell									
08137 Mary Kate Dwyer									
05795 Jolecia Mitchell									
10241 Dawn DiBiasse									
08780 Laura Gavin									

Signature of SSC: _____

WDCM19/101.50

chemists helping chemists in research & industry
aldrich chemical co.
© P.O. Box 355, Milwaukee, Wisconsin 53201 USA

Telephone: (414) 273-3850
TWX: (910) 262-3052 Aldrichem MI
Telex: 26 843 Aldrich MI
FAX: (414) 273-4979

ATTN: SAFETY DIRECTOR
CH2M HILL INC
PO BOX 4400
RESTON VA 22090
KIRK THOMSPON

DATE: 11/06/87
CUST # 924476 P.O. # W6530

MATERIAL SAFETY DATA SHEET

PAGE:

IDENTIFICATION

PRODUCT # 15495-4 NAME: PENTANE, 99+%, SPECTROPHOTOMETRIC GRADE
CAS # 109-66-0

TOXICITY HAZARDS

RTECS # RZ9450000
PENTANE
TOXICITY DATA
IVN-MUS LD50:446 MG/KG JPMSAE 67,566,78
REVIEWS, STANDARDS, AND REGULATIONS
ACGIH TLV-TWA 600 PPM; STEL 750 PPM 85INA8 5,463,86
MSHA STANDARD-AIR:TWA 500 PPM (1475 MG/M3) DTLVS# 3,200,71
OSHA STANDARD-AIR:TWA 1000 PPM FEREAC 39,23540,74
NIOSH REL TO ALKANES-AIR:TWA 350 MG/M3 MMWR## 34(1S),6S,85
EPA TSCA CHEMICAL INVENTORY, 1986
EPA TSCA TEST SUBMISSION (TSCATS) DATA BASE, DECEMBER 1986
NIOSH ANALYTICAL METHODS: SEE HYDROCARBONS, BP 36-126 C, 1500

ONLY SELECTED REGISTRY OF TOXIC EFFECTS OF CHEMICAL SUBSTANCES (RTECS) DATA IS PRESENTED HERE. SEE ACTUAL ENTRY IN RTECS FOR COMPLETE INFORMATI

HEALTH HAZARD DATA

ACUTE EFFECTS
HARMFUL IF INHALED OR SWALLOWED.
VAPOR OR MIST IS IRRITATING TO THE EYES, MUCOUS MEMBRANES AND UPPER RESPIRATORY TRACT.
CAUSES SKIN IRRITATION.
EXPOSURE CAN CAUSE:
DAMAGE TO THE LUNGS
FIRST AID
IN CASE OF CONTACT, IMMEDIATELY FLUSH EYES OR SKIN WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MINUTES WHILE REMOVING CONTAMINATED CLOTHING AND SHOES.
IF INHALED, REMOVE TO FRESH AIR. IF NOT BREATHING GIVE ARTIFICIAL RESPIRATION. IF BREATHING IS DIFFICULT, GIVE OXYGEN.
CALL A PHYSICIAN.
WASH CONTAMINATED CLOTHING BEFORE REUSE.

PHYSICAL DATA

MELTING POINT: -130 C
BOILING POINT: 35 C TO 36 C
SPECIFIC GRAVITY: 0.626
VAPOR DENSITY: 2.48
VAPOR PRESSURE: 776.0 MM @ 37.8 C

FIRE AND EXPLOSION HAZARD DATA

LOWER EXPLOSION LEVEL: 1.5%
UPPER EXPLOSION LEVEL: 7.8%
FLASH POINT: -57 F

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M A T E R I A L S A F E T Y D A T A S H E E T

CATALOG # 15495-4 NAME: PENTANE, 99+%, SPECTROPHOTOMETRIC GRADE

EXTINGUISHING MEDIA
CARBON DIOXIDE, DRY CHEMICAL POWDER, ALCOHOL OR POLYMER FOAM.
DO NOT USE WATER.
SPECIAL FIRE FIGHTING PROCEDURES
WEAR SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING TO PREVENT CONTACT WITH SKIN AND EYES.
USE WATER SPRAY TO COOL FIRE-EXPOSED CONTAINERS.
UNUSUAL FIRE AND EXPLOSION HAZARDS
EXTREMELY FLAMMABLE.
VAPOR MAY TRAVEL CONSIDERABLE DISTANCE TO SOURCE OF IGNITION AND FLASH BACK.
CONTAINER EXPLOSION MAY OCCUR UNDER FIRE CONDITIONS.

REACTIVITY DATA

INCOMPATIBILITIES
OXIDIZING AGENTS
HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS
TOXIC FUMES OF:
CARBON MONOXIDE, CARBON DIOXIDE

SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED
EVACUATE AREA.
SHUT OFF ALL SOURCES OF IGNITION.
WEAR SELF-CONTAINED BREATHING APPARATUS, RUBBER BOOTS AND HEAVY RUBBER GLOVES.
COVER WITH AN ACTIVATED CARBON ADSORBENT, TAKE UP AND PLACE IN CLOSED CONTAINERS. TRANSPORT OUTDOORS.
VENTILATE AREA AND WASH SPILL SITE AFTER MATERIAL PICKUP IS COMPLETE.
WASTE DISPOSAL METHOD
BURN IN A CHEMICAL INCINERATOR EQUIPPED WITH AN AFTERBURNER AND SCRUBBER BUT EXERT EXTRA CARE IN IGNITING AS THIS MATERIAL IS HIGHLY FLAMMABLE.

OBSERVE ALL FEDERAL, STATE & LOCAL LAWS.

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE

CHEMICAL SAFETY GOGGLES.
SAFETY SHOWER AND EYE BATH.
MECHANICAL EXHAUST REQUIRED.
RUBBER GLOVES.
NIOSH/MSHA-APPROVED RESPIRATOR.
DO NOT BREATHE VAPOR.
AVOID CONTACT WITH EYES, SKIN AND CLOTHING.
WASH THOROUGHLY AFTER HANDLING.
IRRITANT.
KEEP TIGHTLY CLOSED.
KEEP AWAY FROM HEAT, SPARKS, AND OPEN FLAME.
OPEN CAREFULLY.
MAY DEVELOP PRESSURE.
HANDLE AND STORE UNDER NITROGEN.
REFRIGERATE.

ADDITIONAL PRECAUTIONS AND COMMENTS

NOT APPLICABLE

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MATERIAL SAFETY DATA SHEET

PAGE:

CATALOG #: I5495-4 NAME: PENTANE, 99+%, SPECTROPHOTOMETRIC GRADE

THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. ALDRICH SHALL NOT BE HELD LIABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT. SEE REVERSE SIDE OF INVOICE OR PACKING SLIP FOR ADDITIONAL TERMS AND CONDITIONS OF SALE.

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AR301406

4495

AIR PRODUCTS

BYRNE SPECIALTY GASES

Byrne Specialty Gases, Inc.
514 S. River Street
Seattle, Washington 98108 (206) 764-4633

Specialty Ga Material Saf Data Shee

EMERGENCY PHONE (800) 523-9374 IN PENNSYLVANIA (800) 322-9092	PRODUCT NAME ISOBUTYLENE	CAS #115-11-7
AIR PRODUCTS AND CHEMICALS, INC. BOX 538 ALLENTOWN, PA 18105 (215) 481-8257	TRADE NAME AND SYNONYMS Isobutylene	
	CHEMICAL NAME AND SYNONYMS Isobutylene, Isobutene, 2-Methylpropene	
ISSUE DATE AND REVISIONS 04/78, 06/85	FORMULA (iso) C_4H_8	CHEMICAL FAMILY Alkene

HEALTH HAZARD DATA

TIME WEIGHTED AVERAGE EXPOSURE LIMIT

See last page.

SYMPTOMS OF EXPOSURE

Inhalation: Moderate concentrations which exclude an adequate supply of oxygen to the lungs cause dizziness, drowsiness and eventual unconsciousness. It also has a very mild anesthetic effect which might cause lack of coordination or lessened mental alertness.

Skin and Eye Contact: It is mildly irritating to mucous membranes. Due to its rapid rate of evaporation, isobutylene can cause tissue freezing or frostbite on contact.

TOXICOLOGICAL PROPERTIES

Isobutylene has a very mild anesthetic effect, however, the major health hazard is the exclusion of an adequate supply of oxygen to the lungs.

Frostbite effects are a change in color of the skin to gray or white possibly followed by blistering.

RECOMMENDED FIRST AID TREATMENT

PROMPT MEDICAL ATTENTION IS REQUIRED IN ALL CASES OF OVEREXPOSURE TO ISOBUTYLENE. RESCUE PERSONNEL SHOULD BE EQUIPPED WITH SELF-CONTAINED BREATHING APPARATUS AND MUST BE AWARE OF EXTREME FIRE AND EXPLOSION HAZARD.

Inhalation: Move exposed personnel to an uncontaminated area. If not breathing, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen. Medical assistance should be sought immediately.

Skin Contact or Frostbite: Remove contaminated clothing and flush affected areas with lukewarm water. DO NOT USE HOT WATER. A physician should see the patient promptly if the cryogenic "burn" has caused blistering of the skin or deep tissue freezing.

AR301407

HAZARDOUS MIXTURES OF OTHER LIQUIDS, SOLIDS, OR GASES
Isobutylene is flammable over a wide range in air.

PHYSICAL DATA

BOILING POINT 19.6°F (−6.9°C)	**LIQUID DENSITY AT BOILING POINT** 39.1 lb/ft³ (626 kg/m³)
VAPOR PRESSURE @ 70°F (21.1°C) = 39 psia (269 kPa)	**GAS DENSITY AT 70°F, 1 atm** 0.148 lb/ft³ (2.37 kg/m³)
SOLUBILITY IN WATER Insoluble	**FREEZING POINT** −220.6°F (−140.3°C)
APPEARANCE AND ODOR Colorless gas with an unpleasant odor similar to that which is emitted when burning anthracite coal.	

FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (Method used) See last page.	**AUTO IGNITION TEMPERATURE** 869°F (465°C)	**FLAMMABLE LIMITS % BY VOLUME** LEL 1.8 UEL 9.6
EXTINGUISHING MEDIA Water, carbon dioxide, dry chemical		**ELECTRICAL CLASSIFICATION** Class 1, Group not specified

SPECIAL FIRE FIGHTING PROCEDURES
Keep cylinder(s) cool with water spray from a distance. If possible without risk, move cylinder(s) away from fire area. If possible without risk, stop the flow of gas to a fire. Allow gas fire to burn itself out. (Continued on last page.)

UNUSUAL FIRE AND EXPLOSION HAZARDS
Isobutylene is denser than air and can travel considerable distances to an ignition source and flash back. Cylinder(s) may explode or vent when exposed to fire.

REACTIVITY DATA

STABILITY Unstable		**CONDITIONS TO AVOID**
Stable	X	

INCOMPATIBILITY (Materials to avoid)
Oxidizers

HAZARDOUS DECOMPOSITION PRODUCTS
None

HAZARDOUS POLYMERIZATION May Occur		**CONDITIONS TO AVOID**
Will Not Occur	X	

SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED
Evacuate all personnel from affected area. Use appropriate protective equipment. If leak is in user's equipment, be certain to purge piping with an inert gas prior to attempting repairs. If leak is in container or container valve, call the "800" emergency phone number listed herein.

WASTE DISPOSAL METHOD
All Federal, State and Local regulations regarding health and pollution should be followed in waste disposal. Contact Air Products for specific recommendations. Do not dispose of unused quantities.

(Continued on last page.)

SPECIAL PROTECTION INFORMATION

RESPIRATORY PROTECTION (Specify type) Positive pressure air line with mask or self-contained breathing apparatus should be available for emergency use.

VENTILATION	LOCAL EXHAUST	SPECIAL
Hood with forced ventilation	To prevent accumulation above the LEL	
	MECHANICAL (Gen.) In accordance with electrical codes	**OTHER**

PROTECTIVE GLOVES
Plastic or rubber

EYE PROTECTION
Safety goggles or glasses

OTHER PROTECTIVE EQUIPMENT
Safety shoes, safety shower, eyewash "fountain."

SPECIAL PRECAUTIONS*

SPECIAL LABELING INFORMATION
DOT Shipping Name: Liquified petroleum gas DOT Hazard Class: Flammable gas
DOT Shipping Label: Flammable gas ID No.: UN 1075

SPECIAL HANDLING RECOMMENDATIONS
Use only in well-ventilated areas. Valve protection caps must remain in place unless container is secured with valve outlet piped to use point. Do not drag, slide or roll cylinders. Use a suitable hand truck for cylinder movement. Use a pressure reducing regulator when connecting cylinder to lower pressure (< 250 psig) piping or systems. Do not heat cylinder by any means to increase the discharge rate of product from the cylinder. Use a check valve or trap in the discharge line to prevent hazardous back flow into the cylinder.

For additional recommendations consult the Air Products Specialty Gas Catalog Safety and Technical Information Section or Compressed Gas Association Pamphlet P-1.

SPECIAL STORAGE RECOMMENDATIONS
Protect cylinders from physical damage. Store in cool, dry, well-ventilated area of non-combustible construction away from heavily trafficked areas and emergency exits. Do not allow the temperature where cylinders are stored to exceed 130°F (54°C). Cylinders should be stored upright and firmly secured to prevent falling or being knocked over. Full and empty cylinders should be segregated. Use a "first in-first out" inventory system to prevent full cylinders being stored for excessive periods of time. Post "No Smoking or Open Flames" signs in the storage or use area. There should be no sources of ignition in the storage or use area.

For additional recommendations consult the Air Products Specialty Gas Catalog Safety and Technical Information Section or Compressed Gas Association Pamphlet P-1.

SPECIAL PACKAGING RECOMMENDATIONS
Isobutylene is noncorrosive and may be used with any common structural material.

OTHER RECOMMENDATIONS OR PRECAUTIONS
Earth-ground and bond all lines and equipment associated with the isobutylene system. Electrical equipment should be non-sparking or explosion proof. Compressed gas cylinders should not be refilled except by qualified producers of compressed gases. Shipment of a compressed gas cylinder which has not been filled by the owner or with his (written) consent is a violation of Federal Law (49CFR).

*Various Government agencies (i.e., Department of Transportation, Occupational Safety and Health Administration, Food and Drug Administration and others) may have specific regulations concerning the transportation, handling, storage or use of this product which will not be reflected in this data sheet. The customer should review these regulations to ensure that he is in full compliance.

Specialty Gas Department
Air Products and Chemicals, Inc.
Box 538, Allentown, PA 18105
(215) 481-8257

AIR PRODUCTS

TIME WEIGHTED AVERAGE EXPOSURE LIMIT (Continued)

Isobutylene is defined as a simple asphyxiant. Oxygen levels should be maintained at greater than 18 molar percent at normal atmospheric pressure which is equivalent to a partial pressure of 135 mm Hg. (ACGIH 1984–85)

FLASH POINT (Method Used) (Continued)

– 105°F (– 76°C) Closed Cup

SPECIAL FIRE FIGHTING PROCEDURES (Continued)

Ventilate low areas where flammable or explosive mixtures may form.

WASTE DISPOSAL METHOD (Continued)

Return the properly labeled shipping container to Air Products for disposal with valve(s) tightly closed, outlet seal(s) secured and valve protection cap in place. For emergency disposal assistance, call the "800" emergency phone number listed herein.

Printed in U.S.A.

320-548

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ATTN: SAFETY DIRECTOR
CH2M HILL INC
PO BOX 4400
RESTON VA 22090

DATE: 02/22/88
CUST # 924476 P.O. # WO795

MATERIAL SAFETY DATA SHEET

PAGE:

IDENTIFICATION

PRODUCT # 15459-8 NAME: ACETONE, 99.5+%, SPECTROPHOTOMETRIC GRADE
CAS # 67-64-1

TOXICITY HAZARDS

RTECS # AL3150000
ACETONE
IRRITATION DATA

EYE-HMN 500 PPM	JIHTAB 25,282,43
SKN-RBT 395 MG OPEN MLD	UCDS** 5/7/70
SKN-RBT 500 MG/24H MLD	28ZPAK -,42,72
EYE-RBT 3950 UG SEV	AJOPAA 29,1363,46
EYE-RBT 100 MG/24H MOD	28ZPAK -,42,72

TOXICITY DATA

UNR-MAN LDLO:1159 MG/KG	85DCAI 2,73,70
ORL-RAT LD50:5800 MG/KG	JTEHD6 15,609,85
IVN-RAT LD50:5500 MG/KG	NPIRI* 1,1,74
ORL-MUS LD50:3000 MG/KG	PCJOAU 14,162,80
IPR-MUS LD50:1297 MG/KG	SCCUR* -,1,61
SKN-RBT LD50:20 GM/KG	UCDS** 5/7/70

REVIEWS, STANDARDS, AND REGULATIONS
ACGIH TLV-TWA 750 PPM; STEL 1000 PPM 85INA8 5,6,86
MSHA STANDARD-AIR:TWA 1000 PPM (2400 MG/M3) DTLVS* 3,3,71
OSHA STANDARD-AIR:TWA 1000 PPM FEREAC 39,23540,74
NIOSH REL TO KETONES-AIR:TWA 590 MG/M3 MMWR** 34(1S),20S,85
EPA GENETOX PROGRAM 1986, NEGATIVE: SHE-CLONAL ASSAY; CELL TRANSFORM.-MOUSE EMBRYO
EPA GENETOX PROGRAM 1986, NEGATIVE: CELL TRANSFORM.-RLV F344 RAT EMBRYO
EPA GENETOX PROGRAM 1986, NEGATIVE: IN VITRO CYTOGENETICS-NONHUMAN
EPA GENETOX PROGRAM 1986, NEGATIVE: HISTIDINE REVERSION-AMES TEST; IN VITRO SCE-NONHUMAN
EPA TSCA CHEMICAL INVENTORY, 1986
EPA TSCA TEST SUBMISSION (TSCATS) DATA BASE, JUNE 1987
NIOSH ANALYTICAL METHODS: SEE KETONES I, 1300
NTP CARCINOGENESIS STUDIES;ON TEST (PRECHRONIC STUDIES), JANUARY 1987
MEETS CRITERIA FOR PROPOSED OSHA MEDICAL RECORDS RULE FEREAC 47,30420, 82

ONLY SELECTED REGISTRY OF TOXIC EFFECTS OF CHEMICAL SUBSTANCES (RTECS) DATA IS PRESENTED HERE. SEE ACTUAL ENTRY IN RTECS FOR COMPLETE INFORMATI

HEALTH HAZARD DATA

ACUTE EFFECTS
MAY BE HARMFUL BY INHALATION, INGESTION, OR SKIN ABSORPTION.
CAUSES SEVERE EYE IRRITATION.
CAUSES SKIN IRRITATION.
MATERIAL IS IRRITATING TO MUCOUS MEMBRANES AND UPPER RESPIRATORY TRACT.
FIRST AID
IN CASE OF CONTACT, IMMEDIATELY FLUSH EYES WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MINUTES.

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CATALOG # 15459-8 NAME: ACETONE, 99.5+%, SPECTROPHOTOMETRIC GRADE

ASSURE ADEQUATE FLUSHING OF THE EYES BY SEPARATING THE EYELIDS WITH FINGERS.
FLUSH SKIN WITH WATER.
IF INHALED, REMOVE TO FRESH AIR. IF NOT BREATHING GIVE ARTIFICIAL RESPIRATION. IF BREATHING IS DIFFICULT, GIVE OXYGEN.
CALL A PHYSICIAN.
REMOVE AND WASH CONTAMINATED CLOTHING PROMPTLY.

PHYSICAL DATA

MELTING POINT: -94 C
BOILING POINT: 56 C
SPECIFIC GRAVITY: 0.791
VAPOR DENSITY: 2
VAPOR PRESSURE: 184.0 MM @ 20 C
400.0 MM @ 39.5 C

FIRE AND EXPLOSION HAZARD DATA

AUTO IGNITION TEMP.: 869 F
LOWER EXPLOSION LEVEL: 2%
UPPER EXPLOSION LEVEL: 13%
FLASH POINT: 1 F

EXTINGUISHING MEDIA
CARBON DIOXIDE, DRY CHEMICAL POWDER, ALCOHOL OR POLYMER FOAM.
WATER MAY BE EFFECTIVE FOR COOLING, BUT MAY NOT EFFECT EXTINGUISHMENT.

SPECIAL FIRE FIGHTING PROCEDURES
WEAR SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING TO PREVENT CONTACT WITH SKIN AND EYES.

UNUSUAL FIRE AND EXPLOSION HAZARDS
EXTREMELY FLAMMABLE.
VAPOR MAY TRAVEL CONSIDERABLE DISTANCE TO SOURCE OF IGNITION AND FLASH BACK.

REACTIVITY DATA

INCOMPATIBILITIES
BASES
OXIDIZING AGENTS
REDUCING AGENTS
PROTECT FROM MOISTURE.

HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS
TOXIC FUMES OF:
CARBON MONOXIDE, CARBON DIOXIDE

SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED
SHUT OFF ALL SOURCES OF IGNITION.
EVACUATE AREA.
WEAR SELF-CONTAINED BREATHING APPARATUS, RUBBER BOOTS AND HEAVY RUBBER GLOVES.
COVER WITH AN ACTIVATED CARBON ADSORBENT, TAKE UP AND PLACE IN CLOSED CONTAINERS. TRANSPORT OUTDOORS.
VENTILATE AREA AND WASH SPILL SITE AFTER MATERIAL PICKUP IS COMPLETE.

WASTE DISPOSAL METHOD
BURN IN A CHEMICAL INCINERATOR EQUIPPED WITH AN AFTERBURNER AND SCRUBBER BUT EXERT EXTRA CARE IN IGNITING AS THIS MATERIAL IS HIGHLY FLAMMABLE.

OBSERVE ALL FEDERAL, STATE & LOCAL LAWS.

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MATERIAL SAFETY DATA SHEET

CATALOG # 15459-8 NAME: ACETONE, 99.5+%, SPECTROPHOTOMETRIC GRADE

--- PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE ----

CHEMICAL SAFETY GOGGLES.
MECHANICAL EXHAUST REQUIRED.
SAFETY SHOWER AND EYE BATH.
COMPATIBLE CHEMICAL-RESISTANT GLOVES.
NIOSH/MSHA-APPROVED RESPIRATOR.
FACESHIELD (8-INCH MINIMUM).
DO NOT BREATHE VAPOR.
DO NOT GET IN EYES, ON SKIN, ON CLOTHING.
AVOID PROLONGED OR REPEATED EXPOSURE.
WASH THOROUGHLY AFTER HANDLING.
SEVERE EYE IRRITANT.
KEEP TIGHTLY CLOSED.
KEEP AWAY FROM HEAT, SPARKS, AND OPEN FLAME.
STORE IN A COOL DRY PLACE.

---------- ADDITIONAL PRECAUTIONS AND COMMENTS ---------

NOT APPLICABLE

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ATTN: SAFETY DIRECTOR
CH2M HILL INC
PO BOX 4400
RESTON VA 22090

DATE: 02/22/88
CUST # 924476 P.O. # W6795

TO: SAFETY DIRECTOR
SUBJECT: MATERIAL SAFETY DATA SHEETS (MSDS'S)

FEBRUARY 22, 1988

ENCLOSED ARE MATERIAL SAFETY DATA SHEETS FOR PRODUCTS YOU HAVE RECENTLY PURCHASE THESE ARE BEING SENT TO YOUR ATTENTION SO THAT YOU CAN DIRECT THE INFORMATION TO THOSE IN YOUR ORGANIZATION WHO REQUIRE IT.

ALDRICH'S POLICY IS TO PROVIDE MSDS'S WITH THE FIRST ORDER FOR PRODUCTS WHICH AR CONSIDERED HAZARDOUS. ON SUBSEQUENT ORDERS, NO ADDITIONAL MSDS'S WILL BE PROVIDE UNLESS A SIGNIFICANT CHANGE IN INFORMATION OCCURS.

THANK YOU FOR YOUR CONTINUED USE OF ALDRICH PRODUCTS.

ALDRICH CHEMICAL COMPANY
MSDS DEPARTMENT

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SUBSTANCE IDENTIFICATION

CAS-NUMBER 7697-37-2

SUBSTANCE: **NITRIC ACID**

TRADE NAMES/SYNONYMS: AQUA FORTIS; HYDROGEN NITRATE; AZOTIC ACID; NITRYL HYDROXIDE; A-200; A-200C; A-200S; A-198; A-202; A-206-C

CHEMICAL FAMILY:
INORGANIC ACID

MOLECULAR FORMULA: H-N-03 MOL WT 63.02

CERCLA RATINGS (SCALE 0-3): HEALTH=3 FIRE=0 REACTIVITY=0 PERSISTENCE=0

COMPONENTS AND CONTAMINANTS

PERCENT: 70 COMPONENT: HYDROGEN NITRATE

PERCENT: 30 COMPONENT: WATER

OTHER CONTAMINANTS: NONE

EXPOSURE LIMITS:
2 PPM (5 MG/M3) OSHA TWA; 2 PPM NIOSH RECOMMENDED TWA;
2 PPM ACGIH TWA; 4 PPM ACGIH STEL

PHYSICAL DATA

DESCRIPTION: COLORLESS FUMING LIQUID WITH AN ACRID ODOR; SUFFOCATING FUMES. THE ODOR IS NOT CONSIDERED AN ADEQUATE WARNING PROPERTY.

BOILING POINT: 181 F (83 C) MELTING POINT: -44 F (-42 C)

SPECIFIC GRAVITY: 1.5 VAPOR PRESSURE: 62 MMHG @ 25 C

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EVAPORATION RATE: NOT AVAILABLE SOLUBILITY IN WATER: MISCIBLE

SOLVENT SOLUBILITY: ETHER ODOR THRESHOLD: <5.0 PPM VAPOR DENSITY: 2.2

FIRE AND EXPLOSION DATA

FIRE AND EXPLOSION HAZARD:
INCREASES THE FLAMMABILITY OF COMBUSTIBLES, ORGANIC MATERIAL, AND READILY OXIDIZABLE MATERIALS, CAUSING IGNITION OF SOME. SEVERE EXPLOSION HAZARD BY REACTION WITH MANY INCOMPATIBLES, INCLUDING METALLIC POWDERS, CARBIDES, HYDROGEN SULFIDE, AND TURPENTINE. IN OR NEAR FIRE, MATERIAL EMITS TOXIC AND REACTIVE NITROGEN OXIDES AS GASES.

FLASH POINT: NONCOMBUSTIBLE

FIREFIGHTING MEDIA:
WATER SPRAY

FIREFIGHTING:
MOVE CONTAINER FROM FIRE AREA IF POSSIBLE. COOL CONTAINERS EXPOSED TO FLAMES WITH WATER FROM SIDE UNTIL WELL AFTER FIRE IS OUT. FOR MASSIVE FIRE IN STORAGE AREA, USE UNMANNED HOSE HOLDER OR MONITOR NOZZLES; ELSE WITHDRAW FROM AREA AND LET FIRE BURN (1984 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.3).

EXTINGUISH USING AGENTS INDICATED. IF LARGE AMOUNTS OF COMBUSTIBLE MATERIALS ARE INVOLVED, USE WATER SPRAY OR FOG IN FLOODING AMOUNTS. USE WATER SPRAY TO ABSORB CORROSIVE VAPORS. COOL CONTAINERS WITH FLOODING AMOUNTS OF WATER FROM AS FAR A DISTANCE AS POSSIBLE. AVOID BREATHING CORROSIVE VAPORS; KEEP UPWIND

TOXICITY

430 MG/KG ORAL-HUMAN LDLO; 110 MG/KG UNKNOWN-MAN LDLO;
CARCINOGEN STATUS: NONE.
NITRIC ACID IS A SEVERE EYE, MUCOUS MEMBRANE, AND SKIN IRRITANT.

HEALTH EFFECTS AND FIRST AID

INHALATION:
CORROSIVE. 100 PPM IS IMMEDIATELY DANGEROUS TO LIFE AND HEALTH.
ACUTE EXPOSURE- MAY CAUSE COUGHING, HEADACHE, DIZZINESS, AND WEAKNESS. DELAYED SYMPTOMS MAY INCLUDE DRYNESS OF THE THROAT AND NOSE, CHEST PAIN OR TIGHTNESS, DYSPHEA, FROTHY SPUTUM, HYPOTENSTION AND CYANOSIS FOLLOWED BY PNEUMONITIS AND PULMONARY EDEMA, WHICH MAY BE FATAL. IF PATIENT RECOVERS, SCAR TISSUE MAY CAUSE STRICTURE OF THE PYLORUS OR ESOPHAGUS.

CHRONIC EXPOSURE- REPEATED OR PROLONGED EXPOSURE CAUSES DENTAL EROSION FOLLOWED BY JAW NECROSIS, CHRONIC COUGH AND BRONCHITIS OR CHEMICAL PNEUMONITIS AND GASTROINTESTINAL DISTURBANCES.

FIRST AID- REMOVE FROM EXPOSURE AREA TO FRESH AIR IMMEDIATELY. IF BREATHING HAS STOPPED, GIVE ARTIFICIAL RESPIRATION. MAINTAIN AIRWAY AND ADMINISTER OXYGEN IF AVAILABLE. KEEP AFFECTED PERSON WARM AND AT REST.

SKIN CONTACT:
CORROSIVE.
ACUTE EXPOSURE- DIRECT CONTACT WITH LIQUID OR CONCENTRATED VAPOR CAUSES IMMEDIATE SEVERE AND PENETRATING BURNS, STAINING THE SKIN YELLOW OR YELLOWISH-BROWN.

CHRONIC EXPOSURE- PROLONGED OR REPEATED EXPOSURE MAY CAUSE DERMATITIS.

FIRST AID- REMOVE CONTAMINATED CLOTHING AND SHOES IMMEDIATELY. WASH AFFECTED AREA WITH SOAP OR MILD DETERGENT AND LARGE AMOUNTS OF WATER UNTIL NO EVIDENCE OF CHEMICAL REMAINS (APPROXIMATELY 15-20 MINUTES). IN CASE OF CHEMICAL BURNS, COVER AREA WITH STERILE, DRY DRESSING. BANDAGE SECURELY, BUT NOT TOO TIGHTLY. GET MEDICAL ATTENTION.

EYE CONTACT:
CORROSIVE.
ACUTE EXPOSURE- DIRECT CONTACT WITH THE LIQUID MAY CAUSE PAIN, PHOTOPHOBIA, TEARING, EDEMA, CORNEAL ULCERATION, SEVERE BURNS, AND NECROSIS OF THE DEEPER TISSUES WITH PERMANENT DAMAGE AND BLINDNESS IS POSSIBLE.

CHRONIC EXPOSURE- REPEATED OR PROLONGED EXPOSURE MAY CAUSE CONJUNCTIVITIS.

FIRST AID- WASH EYES IMMEDIATELY WITH LARGE AMOUNTS OF WATER, OCCASIONALLY LIFTING UPPER AND LOWER LIDS, UNTIL NO EVIDENCE OF CHEMICAL REMAINS (APPROXIMATELY 15-20 MINUTES). IN PRESENCE OF BURNS, APPLY STERILE BANDAGES LOOSELY WITHOUT MEDICATION. GET MEDICAL ATTENTION.

INGESTION:
CORROSIVE.
ACUTE EXPOSURE- IMMEDIATE PAIN IN THE MOUTH, THROAT, AND STOMACH MAY BE FOLLOWED BY VOMITING, AND DIARRHEA OF DARK PRECIPATED BLOOD. HYPOTENSION, OLIGURIA, ANURIA, SEVERE, POSSIBLY FATAL, CIRCULATORY COLLAPSE, AND ASPHYXIA FROM EDEMA OF THE GLOTTIS ARE POSSIBLE. BURNS OF THE GASTROINTESTINAL TRACT MAY BE SEVERE ENOUGH TO CAUSE PERFORATION OF THE ESOPHAGUS AND STOMACH WHICH MAY BE FOLLOWED BY MEDIASTINITUS OR PERITONITIS, INDICATED BY FEVER.

FIRST AID- IF VICTIM IS CONSCIOUS, GIVE HIM LARGE QUANTITIES OF WATER IMMEDIATELY TO DILUTE THE ACID. DO NOT INDUCE VOMITING. GIVE PATIENT 1 OUNCE (30 ML) OF MILK OF MAGNESIA. GET MEDICAL ATTENTION IMMEDIATELY.

REACTIVITY

REACTIVITY:
STABLE UNDER NORMAL TEMPERATURES AND PRESSURES. HOWEVER NITRIC VAPOR AND/OR NITRIC OXIDES ARE QUIETLY EVOLVED. ALSO SUNLIGHT CATLAYSES THE FORMATION OF THE OXIDES AND THIS GIVES A YELLOW COLOR TO THE CONCENTRATED ACID.

INCOMPATIBILITIES:
EASILY OXIDIZED SUBSTANCES, EXAMPLES FOLLOW:

EXPLOSION: ACETONITRILE, CESIUM CARBIDE, CUPRIC NITRIDE, CYANIDES, 1,2-DIAMINOETHANE BISTRIMETHYL GOLD, DINITROTOLUENE, EPICHLOROHYDRIN, 5-ETHYL-2-METHYLPYRIDINE, CYCLOPENTADIENE, BENZENE, TOLUENE, METALS, METAL CARBIDES, 4-METHYLCYCLOHEXANONE, NITROBENZENE AND WATER, NITROMETHANE, POLYDIBROMOSILANES, PHOSPHORUS TRICHLORIDE, POTASSIUM HYPOPHOSPHITE (ON EVAPORATION), RUBIDIUM CARBIDE, SELENIUM IODOPHOSPHIDE, SULFUR DIOXIDES, THIOCYANATES,

THIOCYANIC ACID METAL SALTS, THIOPHENES, TETRABORANE, TRICADMIUM DIPHOSPHIDE, TRITHIOACETONE.
PROBABLE EXPLOSION: ACETONE AND ACETIC ACID, SULFURIC ACID AND GLYCERIDES, TRIAZINE AND TRIFLUOROACETIC ANHYDRIDE @ 36 C.
POSSIBLE EXPLOSION: ACETIC ACID, I-AMINOTHIAZOLE AND SULFURIC ACID, CYANATES, 1,3-CYCLOPENTADIENE, FLURORINE, LACTIC ACID AND HYDROGEN FLUORIDE, MESITYLENE, ORGANIC SUBSTANCES AND SULFURIC ACID, ORGANIC SUBSTANCES AND PERCHLORATES, PHTHALIC ACID OR PHTHALIC ANHYDRIDE AND SULFURIC ACID, REDUCING AGENTS, SULFURIC ACID, TITANIUM ALLOY.
EXPLOSION BY FRICTION OR IMPACT: ACETIC ANHYDRIDE.
EXPLOSIVE OXIDATION: NON-METAL OXIDES- ARSINE, PHOSPHINE, OR TETRABORANE, DIPHENYLDISTIBENE.
POSSIBLE EXPLOSION BY IMPACT : TITANIUM-MAGNESIUM ALLOY.
VIOLENT REACTION: ACRYLONITRILE, ALCOHOLS, ARSINE, CARBON (PULVERIZED), CHLORINE TRIFLUORIDE, CUPROUS NITRIDE, CYCLIC KETONES, CYCLOHEXANOL, ETHANOL, GERMANIUM, HYDRAZINE, SULFUR HALIDES, SULFURIC ACID AND TEREPHTHALIC ACID, THIOALDEHYDES OR THIOKETONES, URANIUM, URANIUN ALLOYS.
VIOLENT OXIDATION: ACETONE AND SULFURIC ACID, SULFAMIC ACID.
VIOLENT DECOMPOSITION: BUTANETHIOL, PHOSPHINE.
VIOLENT DECOMPOSITION RESULTING IN IGNITION: CROTONALDEHYDE, TETRAPHOSPHORUS TRIIODIDE.
POSSIBLE VIOLENT REACTION: ANTIMONY.
POSSIBLE VIOLENT EXOTHERMIC REACTION: ANION EXCHANGE RESIN.
INTENSE EXOTHERMIC REACTION: ACROLEIN, ALLYL ALCOHOL, ALLYL CHLORIDE, 2-AMINOETHANOL, AMMONIUM HYDROXIDE, BISMUTH, N-BUTYRALDEHYDE, CHLOROSULFONIC ACID, CRESOL, CUMENE, DIISOPROPYL ETHER, ETHYLENEDIAMINE, POLYALKENES, GLYOXAL, ISOPRENE, MESITYL OXIDE, 2-METHYL-5-ETHYLPYRIDINE, OLEUM, PROPYLENE OXIDE, PROPIOLACTONE (BETA-), PYRIDENE, SODIUM HYDROXIDE, VINYL ACETATE, VINYLDENE CHLORIDE,.
INTENSE REACTION: DIETHYLETHER, HYDRAZOIC ACID, P-XYLENE IN THE PRESENCE OF SULFURIC ACID, SELENIUM, SODIUM AZIDE, TOLUENE, TRIMETHYLTRIOXANE.
IGNITION WITH POSSIBLE EXPLOSION: HYDROGEN TELLURIDE.
IGNITION: ANILINE, BORON PHOSPHIDE, BROMINE PENTAFLUORIDE, N-BUTYLMERCAPTAN, CALCIUM HYPOPHOSPHITE, DIBORANE, DIPHENYL TIN, M-ETHYL ANILINE, ETHYL PHOSPHINE, FURFURYL ALCOHOL, HALOGEN PHOSPHIDES, HYDROGEN IODIDES, LITHIUM, METALS, PHOSPHONIUM IODIDE, PHOPHORUS, SELENIUM HYDRIDE, SODIUM, TERPENES, TOLUIDINE, TRIETHYLGALLIUM MONOETHYL ETHER COMPLEX, UNS-DIMETHYLHYDRAZINE.
POSSIBLE IGNITION: AMMONIA, ANION EXCHANGE RESIN AND CHROMITES OR DICHROMATE, AROMATIC AMINES, DIVINYL ETHER, DIENE OR ACETYLENE DERIVATIVES, LITHIUM, REDUCING AGENTS.
INCANDESCENT REACTION: BORON, FERROUS OXIDE (POWDER), HYDROGEN SULFIDE, LITHIUM SILICIDE, SELENIUM HYDRIDE, MAGNESIUM PHOSPHIDE, MANGANESE, ZINC.
FORMATION OF HIGHLY EXPLOSIVE PRODUCTS: NITROAROMATIC HYDROCARBONS.
FORMATION OF EXPLOSIVE PRODUCTS: ACETYLENE, 4-CHLORO-2-NITROANILINE, CYCLOHEXANE, CYCLOHEXYLAMINE, 2,6-DI-T-BUTYL PHENOL, DICHLOROMETHANE, ETHANOL AND SILVER, 5-ETHYL-2-PICOLINE, HYDROGEN PEROXIDE AND KETONES, HYDROGEN PEROXIDE AND MERCURIC OXIDE, HYDROGEN PEROXIDE AND THIOUREA, INDANE AND SULFURIC ACID, METAL SALICYLATES, PHENYLORTHOPHOSPHORIC ACID DISODIUM SALT, TITANIUM.
FORMATION OF POSSIBLY EXPLOSIVE PRODUCTS: BENZOTHIOPHENE DERIVATIVES.
FORMATION OF EASILY COMBUSTIBLE ESTER: CELLULOSE.
DETONATABLE MIXTURE (DEPENDING ON AMOUNT OF WATER PRESENT): NITROBENZENE.

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DECOMPOSITION:
DECOMPOSES ON EXPOSURE TO AIR OR ORGANIC MATTER, OR WITH HEAT, TO RELEASE HIGHLY TOXIC FUMES OR OXIDES OF NITROGEN, INCLUDING NITRIC OXIDE AND NITROGEN DIOXIDE, AND HYDROGEN NITRATE. REACTS WITH THE FOLLOWING TO RELEASE TOXIC GASES: SULFIDES, CARBONATES, CYANIDES. VIOLENT REACTION WITH ALL CARBIDES, GAS MIXTURE EVOLVED (N2O4) REACTS STRONGLY WITH HYDROCARBONS, FLUORINE, OR

POLYMERIZATION:
WILL NOT OCCUR.

****
CONDITIONS TO AVOID

MAY IGNITE COMBUSTIBLE MATERIALS (WOOD, PAPER, OIL, ETC.). REACTS VIOLENTLY WITH WATER AND FUELS. FLAMMABLE, POISONOUS GASES MAY ACCUMULATE IN TANKS AND HOPPER CARS. RUNOFF TO SEWER MAY CREATE FIRE OR EXPLOSION HAZARD. AVOID CONTACT WITH OR STORAGE WITH INCOMPATIBLE MATERIALS, INCLUDING THOSE MATERIALS AND CLASSES OF MATERIALS LISTED IN THE REACTIVITY SECTION. HEATING MAY INCREASE THE EVOLUTION OF NITRIC ACID VAPOR AND/OR NITROGEN OXIDES (GASES) BEYOND AN ACCEPTABLE LEVEL.

****
SPILL AND LEAK PROCEDURES

OCCUPATIONAL SPILL:
KEEP COMBUSTIBLES (WOOD, PAPER, OIL AND OTHER EASILY OXIDIZABLE MATERIALS) AWAY FROM SPILLED MATERIAL. WEAR PERSONAL PROTECTIVE EQUIPMENT. DO NOT TOUCH SPILLED MATERIAL. STOP LEAK IF YOU CAN DO IT WITHOUT RISK. USE WATER SPRAY TO REDUCE VAPORS. DILUTE SPILLS OR LEAKS WITH PLENTY OF WATER. NEUTRALIZE RESIDUE WITH (A) ALKALI, SUCH AS SOLDA ASH, LIME, LIMESTONE; OR (B) OTHER SUITABLE NEUTRALIZATION MATERIALS. ADEQUATE VENTILATION IS REQUIRED TO ELIMINATE ANY NITROGEN OXIDES RELEASED AND, IF SODA ASH OR LIMESTONE IS USED, CO2. ABSORB WITH EXCESS SODA ASH, SCOOP UP AND PLACE IN A SUITABLE E.G. GLASS OR PLASTIC CONTAINER AND CLOSE. LABEL 'OXIDIZER'. KEEP OUT OF SEWERS AND WATER SOURCES. KEEP UNNECESSARY PEOPLE AWAY. ISOLATE HAZARD AREA AND DENY ENTRY. VENTILATE CLOSED SPACES BEFORE ENTERING.

PROTECTIVE EQUIPMENT

VENTILATION:
PROVIDE LOCAL EXHAUST VENTILATION, PROCESS ENCLOSURE OR GENERAL DILUTION VENTILATION TO MEET PERMISSABLE EXPOSURE LIMITS REQUIREMENTS. EQUIPMENT MUST BE CORROSION-RESISTANT.

RESPIRATOR:
EXPOSURE LIMIT TO 100 MG/M3-
SELF-CONTAINED BREATHING APPARATUS WITH A FULL FACEPIECE OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE-PRESSURE MODE.
TYPE 'C' SUPPLIED-AIR RESPIRATOR OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE-PRESSURE OR CONTINUOUS FLOW MODE.

-> 100 MG/M3, INCLUDING THE IDLH LEVEL, 250 MG/M3-
SELF-CONTAINED BREATHING APPARATUS WITH A FULL FACEPIECE OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE-PRESSURE MODE, OR USE EQUIVALENT RESPIRATOR.

FIREFIGHTING- SELF-CONTAINED BREATHING APPARATUS WITH A FULL FACEPIECE OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE PRESSURE MODE.

CLOTHING:
EMPLOYEE MUST WEAR IMPERVIOUS CLOTHING AS NECESSARY TO AVOID ANY POSSIBILITY

OF CONTACT WITH CHEMICAL.

GLOVES:
WEAR IMPERVIOUS GLOVES AS NECESSARY TO AVOID ANY POSSIBLILITY OF CONTACT WITH SUBSTANCE. PREFERRED MATERIALS: VITON OR SARANEX.

EYE PROTECTION:
WEAR FACESHIELD (8 INCH MINIMUM) AND VENTED SAFETY GOGGLES. DO NOT WEAR CONTACT LENSES WHEN WORKING WITH CHEMICALS.

AUTHORIZED - ALLIED FISHER SCIENTIFIC
CREATION DATE: 02/10/85 REVISION DATE: 10/21/85

-ADDITIONAL INFORMATION-

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MATERIAL SAFETY DATA SHEET

PAGE:

IDENTIFICATION

PRODUCT # 15490-3 NAME: METHYL ALCOHOL, 99.9%, SPECTROPHOTOMETRIC GRADE

CAS # 67-56-1

TOXICITY HAZARDS

RTECS # PC1400000
METHANOL

IRRITATION DATA
SKN-RBT 500 MG/24H MOD — 28ZPAK -,33,72
EYE-RBT 40 MG MOD — UCDS** 3/24/70

TOXICITY DATA

ORL-HMN LDLO:428 MG/KG	NPIRI* 1,74,74
ORL-HMN LDLO:143 MG/KG	34ZIAG -,382,69
UNR-MAN LDLO:857 MG/KG	BSDCAI 2,73,70
ORL-RAT LD50:5628 MG/KG	GTPZAB 19(11),27,75
INL-RAT LC50:64000 PPM/4H	NPIRI* 1,74,74
IPR-RAT LD50:7529 MG/KG	EVHPAZ 61,321,85
IVN-RAT LD50:2131 MG/KG	EVHPAZ 61,321,85
OSL-MUS LD50:7300 MG/KG	TXCYAC 25,271,82
IPR-MUS LD50:10765 MG/KG	EVHPAZ 61,321,85
SCU-MUS LD50:9800 MG/KG	TXAPA9 16,165,71
IVN-MUS LD50:4710 MG/KG	EVHPAZ 61,321,85
SKN-RBT LD50:15800 MG/KG	NPIRI* 1,74,74
IPR-RBT LD50:1328 MG/KG	EVHPAZ 61,321,85
IVN-RBT LD50:8907 MG/KG	EVHPAZ 61,321,85
IPR-GPG LD50:3559 MG/KG	EVHPAZ 61,321,85
IPR-HAM LD50:3555 MG/KG	EVHPAZ 61,321,85

REVIEWS, STANDARDS, AND REGULATIONS
ACGIH TLV-TWA 200 PPM; STEL 250 PPM (SKIN) 85INA8 5,372,86
MSHA STANDARD-AIR:TWA 200 PPM (260 MG/M3) (SKIN) DTLVS* 3,155,71
OSHA STANDARD-AIR:TWA 200 PPM FEREAC 39,23540,74
NIOSH REL TO METHYL ALCOHOL-AIR:TWA 200 PPM;CL 800 PPM/15M MMWR** 34(1S),21S,85
EPA GENETOX PROGRAM 1986, NEGATIVE: SHE-CLONAL ASSAY; CELL TRANSFORM.-SA7/SHE
EPA GENETOX PROGRAM 1986, NEGATIVE: N CRASSA-ANEUPLOIDY; IN VITRO SCE-NONHUMAN
EPA TSCA CHEMICAL INVENTORY, 1986
EPA TSCA SECTION 8(E) STATUS REPORT 8EHQ-0378-0108
EPA TSCA TEST SUBMISSION (TSCATS) DATA BASE, DECEMBER 1986
NIOSH ANALYTICAL METHODS: SEE METHANOL, 2000
MEETS CRITERIA FOR PROPOSED OSHA MEDICAL RECORDS RULE FEREAC 47,30420, P2

ONLY SELECTED REGISTRY OF TOXIC EFFECTS OF CHEMICAL SUBSTANCES (RTECS) DATA IS PRESENTED HERE. SEE ACTUAL ENTRY IN RTECS FOR COMPLETE INFORMAT

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MATERIAL SAFETY DATA SHEET

CATALOG # 15490-3 NAME: METHYL ALCOHOL, 99.9%, SPECTROPHOTOMETRIC GRADE

HEALTH HAZARD DATA

ACUTE EFFECTS
MAY BE FATAL IF SWALLOWED.
HARMFUL IF INHALED OR ABSORBED THROUGH SKIN.
SYMPTOMS OF EXPOSURE MAY INCLUDE BURNING SENSATION, COUGHING, WHEEZING, LARYNGITIS, SHORTNESS OF BREATH, HEADACHE, NAUSEA AND VOMITING.
EXPOSURE CAN CAUSE:
DAMAGE TO THE EYES
DAMAGE TO THE LIVER
DAMAGE TO THE HEART
DAMAGE TO THE KIDNEYS
GASTROINTESTINAL DISTURBANCES
MAY CAUSE CONVULSIONS.
FIRST AID
IN CASE OF CONTACT, IMMEDIATELY FLUSH EYES OR SKIN WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MINUTES WHILE REMOVING CONTAMINATED CLOTHING AND SHOES.
ASSURE ADEQUATE FLUSHING OF THE EYES BY SEPARATING THE EYELIDS WITH FINGERS.
IF INHALED, REMOVE TO FRESH AIR. IF NOT BREATHING GIVE ARTIFICIAL RESPIRATION. IF BREATHING IS DIFFICULT, GIVE OXYGEN.
CALL A PHYSICIAN.
DISCARD CONTAMINATED CLOTHING AND SHOES.
ADDITIONAL INFORMATION
METHYL ALCOHOL MAY BE FATAL OR CAUSE BLINDNESS IF SWALLOWED. CANNOT BE MADE NON-POISONOUS.

PHYSICAL DATA

MELTING POINT: -98 C
BOILING POINT: 64.6 C
SPECIFIC GRAVITY: 0.791
VAPOR DENSITY: 1.1
VAPOR PRESSURE: 97.66 MM @ 20 C

FIRE AND EXPLOSION HAZARD DATA

AUTO IGNITION TEMP.: 725 F
LOWER EXPLOSION LEVEL: 6.0%
UPPER EXPLOSION LEVEL: 36.0%
FLASH POINT: 52 F
EXTINGUISHING MEDIA
CARBON DIOXIDE, DRY CHEMICAL POWDER, ALCOHOL OR POLYMER FOAM.
SPECIAL FIRE FIGHTING PROCEDURES
WEAR SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING TO PREVENT CONTACT WITH SKIN AND EYES.
UNUSUAL FIRE AND EXPLOSION HAZARDS
EXTREMELY FLAMMABLE.
VAPOR MAY TRAVEL CONSIDERABLE DISTANCE TO SOURCE OF IGNITION AND FLASH BACK.

REACTIVITY DATA

INCOMPATIBILITIES
ACIDS
ACID CHLORIDES
ACID ANHYDRIDES
OXIDIZING AGENTS
REDUCING AGENTS
ALKALI METALS

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CATALOG # 15490-3 NAME: METHYL ALCOHOL, 99.9%, SPECTROPHOTOMETRIC GRADE

HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS
TOXIC FUMES OF:
CARBON MONOXIDE, CARBON DIOXIDE

---------- SPILL OR LEAK PROCEDURES ----------

STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED
EVACUATE AREA.
SHUT OFF ALL SOURCES OF IGNITION.
WEAR SELF-CONTAINED BREATHING APPARATUS, RUBBER BOOTS AND HEAVY RUBBER GLOVES.
COVER WITH DRY-LIME, SAND, OR SODA ASH. PLACE IN COVERED CONTAINERS USING NON-SPARKING TOOLS AND TRANSPORT OUTDOORS.
VENTILATE AREA AND WASH SPILL SITE AFTER MATERIAL PICKUP IS COMPLETE.
WASTE DISPOSAL METHOD
BURN IN A CHEMICAL INCINERATOR EQUIPPED WITH AN AFTERBURNER AND SCRUBBER BUT EXERT EXTRA CARE IN IGNITING AS THIS MATERIAL IS HIGHLY FLAMMABLE.

OBSERVE ALL FEDERAL, STATE & LOCAL LAWS.

--- PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE ---

WEAR APPROPRIATE NIOSH/MSHA-APPROVED RESPIRATOR, CHEMICAL-RESISTANT GLOVES, SAFETY GOGGLES, OTHER PROTECTIVE CLOTHING.
MECHANICAL EXHAUST REQUIRED.
SAFETY SHOWER AND EYE BATH.
DO NOT BREATHE VAPOR.
AVOID CONTACT WITH EYES, SKIN AND CLOTHING.
AVOID PROLONGED OR REPEATED EXPOSURE.
DO NOT USE IF SKIN IS CUT OR SCRATCHED. WASH THOROUGHLY AFTER HANDLING.
POISON.
KEEP TIGHTLY CLOSED.
KEEP AWAY FROM HEAT, SPARKS, AND OPEN FLAME.
HYGROSCOPIC.
STORE IN A COOL DRY PLACE.

----------ADDITIONAL PRECAUTIONS AND COMMENTS ---------

NOT APPLICABLE

THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. ALDRICH SHALL NOT BE HELD LIABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT. SEE REVERSE SIDE OF INVOICE OR PACKING SLIP FOR ADDITIONAL TERMS AND CONDITIONS OF SALE.

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HYDROCHLORIC ACID, CONCENTRATED (36-37%)
HYDROCHLORIC ACID, CONCENTRATED (36-37%)
HYDROCHLORIC ACID, CONCENTRATED (36-37%)

MATERIAL SAFETY DATA SHEET

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CHEMICAL DIVISION
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SUBSTANCE IDENTIFICATION

CAS-NUMBER 7647-01-0

SUBSTANCE: **HYDROCHLORIC ACID, CONCENTRATED (36-37%)**

TRADE NAMES/SYNONYMS:
CHLOROHYDRIC ACID; HYDROCHLORIDE; MURIATIC ACID; SPIRITS OF SALT; HYDROCHLORIC ACID, CONCENTRATED; HYDROGEN CHLORIDE, 23 EB; UN 1789; A-142; A-144; A-508; A-966; ACC1115S

CHEMICAL FAMILY:
INORGANIC ACID

MOLECULAR FORMULA: H-CL

MOLECULAR WEIGHT: 36.46

CERCLA RATINGS (SCALE 0-3): HEALTH=3 FIRE=0 REACTIVITY=1 PERSISTENCE=0
NFPA RATINGS (SCALE 0-4): HEALTH=3 FIRE=0 REACTIVITY=0

COMPONENTS AND CONTAMINANTS

COMPONENT: HYDROGEN CHLORIDE PERCENT: 37

COMPONENT: WATER PERCENT: 63

OTHER CONTAMINANTS: NONE

EXPOSURE LIMITS:
HYDROGEN CHLORIDE (HYDROCHLORIC ACID):
5 PPM OSHA CEILING
5 PPM ACGIH CEILING

500 POUNDS SARA SECTION 302 THRESHOLD PLANNING QUANTITY (GAS)
1 POUND SARA SECTION 304 REPORTABLE QUANTITY (GAS)
5000 POUNDS CERCLA SECTION 103 REPORTABLE QUANTITY (LIQUID)
SUBJECT TO SARA SECTION 313 ANNUAL TOXIC CHEMICAL RELEASE REPORTING

PHYSICAL DATA

DESCRIPTION: COLORLESS OR SLIGHTLY YELLOW FUMING LIQUID WITH A PUNGENT ODOR. BOILING POINT: 384 F (196 C) SPECIFIC GRAVITY: 1.2

VAPOR PRESSURE: NOT AVAILABLE PH: 1.1 (0.1 N)

SOLUBILITY IN WATER: SOLUBLE VAPOR DENSITY: 1.3

FIRE AND EXPLOSION DATA

FIRE AND EXPLOSION HAZARD:
NEGLIGIBLE FIRE HAZARD WHEN EXPOSED TO HEAT OR FLAME.

FIREFIGHTING MEDIA:
DRY CHEMICAL, CARBON DIOXIDE, HALON, WATER SPRAY OR STANDARD FOAM (1987 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.4).

FOR LARGER FIRES, USE WATER SPRAY, FOG OR STANDARD FOAM (1987 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.4).

FIREFIGHTING:
MOVE CONTAINERS FROM FIRE AREA IF POSSIBLE. COOL CONTAINERS EXPOSED TO FLAMES WITH WATER FROM SIDE UNTIL WELL AFTER FIRE IS OUT. STAY AWAY FROM STORAGE TANK [illegible]

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EXTINGUISH USING AGENTS SUITABLE FOR TYPE OF FIRE. USE FLOODING AMOUNTS OF WATER AS FOG. COOL CONTAINERS WITH FLOODING AMOUNTS OF WATER. APPLY FROM AS FAR A DISTANCE AS POSSIBLE. AVOID BREATHING CORROSIVE VAPORS, KEEP UPWIND.

TRANSPORTATION DATA

DEPARTMENT OF TRANSPORTATION HAZARD CLASSIFICATION 49CFR172.101:
CORROSIVE MATERIAL

DEPARTMENT OF TRANSPORTATION LABELING REQUIREMENTS 49CFR172.101 AND 172.402:
CORROSIVE

DEPARTMENT OF TRANSPORTATION PACKAGING REQUIREMENTS: 49CFR173.263
EXCEPTIONS: 49CFR173.244

TOXICITY

HYDROGEN CHLORIDE (HYDROCHLORIC ACID):
IRRITATION DATA:
ANHYDROUS: 100 MG RINSED EYE-RABBIT MILD.
TOXICITY DATA:
HYDROGEN CHLORIDE (ANHYDROUS GAS): 4701 PPM/30 MINUTES INHALATION-RAT LC50; 2644 PPM/30 MINUTES INHALATION-MOUSE LC50.
MONOHYDRATE: NO DATA AVAILABLE.
DIHYDRATE: NO DATA AVAILABLE.
TRIHYDRATE: NO DATA AVAILABLE.
HEXAHYDRATE: NO DATA AVAILABLE.
HYDROGEN CHLORIDE (AEROSOL): 5666 PPM/30 MINUTES INHALATION-RAT LC50; 2142 PPM/30 MINUTES INHALATION-MOUSE LC50.
HYDROCHLORIC ACID: 1300 PPM/30 MINUTES INHALATION-HUMAN LCLO; 3000 PPM/5 MINUTES INHALATION-HUMAN LCLO; 81 MG/KG UNREPORTED MAN LDLO; 3124 PPM/1 HOUR INHALATION-RAT LC50; 1108 PPM/1 HOUR INHALATION-MOUSE LC50; 1449 MG/KG INTRAPERITONEAL-MOUSE LD50; 900 MG/KG ORAL-RABBIT LD50; 4416 PPM/30 MINUTES INHALATION-RABBIT LCLO; 4416 PPM/30 MINUTES INHALATION-GUINEA PIG LCLO; 1000 MG/M3/2 HOURS INHALATION-MAMMAL LCLO; MUTAGENIC DATA (RTECS); REPRODUCTIVE EFFECTS DATA (RTECS).
CARCINOGEN STATUS: NONE.
LOCAL EFFECTS: CORROSIVE- INHALATION, SKIN, EYE AND INGESTION.
ACUTE TOXICITY LEVEL: MODERATELY TOXIC BY INHALATION.
TARGET EFFECTS: NO DATA AVAILABLE.

HEALTH EFFECTS AND FIRST AID

INHALATION:
HYDROGEN CHLORIDE (HYDROCHLORIC ACID):
CORROSIVE. 100 PPM IMMEDIATELY DANGEROUS TO LIFE OR HEALTH.
ACUTE EXPOSURE- INHALATION OF GAS OR FUMES AT LEVELS OF 5-35 PPM MAY CAUSE IRRITATION AND BURNING OF THE THROAT, COUGHING AND CHOKING; 50-100 PPM MAY BE BARELY TOLERABLE FOR 1 HOUR. HIGH LEVELS MAY CAUSE INFLAMMATION AND OCCASIONALLY ULCERATION OF THE NOSE, THROAT OR LARYNX, BRONCHITIS, PNEUMONIA, PALPITATIONS AND HEADACHE. HIGHER CONCENTRATIONS MAY CAUSE NECROSIS OF THE TRACHEAL AND BRONCHIAL EPITHELIUM, NASOSEPTAL PERFORATION, ATELECTASIS, EMPHYSEMA, DAMAGE TO PULMONARY BLOOD VESSELS AND LESIONS OF THE LIVER AND OTHER ORGANS. DEATH MAY BE DUE TO LARYNGEAL SPASM, BRONCHOPNEUMONIA OR PULMONARY EDEMA. 1300-2000 PPM MAY BE DANGEROUS, EVEN ON BRIEF EXPOSURES. REPRODUCTIVE EFFECTS HAVE BEEN REPORTED IN ANIMALS.
CHRONIC EXPOSURE- REPEATED OR PROLONGED EXPOSURE MAY CAUSE EROSION AND DISCOLORATION OF EXPOSED TEETH, CHRONIC BRONCHITIS AND GASTRITIS.

FIRST AID- REMOVE FROM EXPOSURE AREA TO FRESH AIR IMMEDIATELY. IF BREATHING HAS STOPPED, GIVE ARTIFICIAL RESPIRATION. MAINTAIN AIRWAY AND BLOOD PRESSURE AND ADMINISTER OXYGEN IF AVAILABLE. KEEP AFFECTED PERSON WARM AND AT REST. TREAT SYMPTOMATICALLY AND SUPPORTIVELY. ADMINISTRATION OF OXYGEN SHOULD BE PERFORMED BY QUALIFIED PERSONNEL. GET MEDICAL ATTENTION IMMEDIATELY.

SKIN CONTACT:
HYDROGEN CHLORIDE (HYDROCHLORIC ACID):
CORROSIVE.
ACUTE EXPOSURE- CONTACT MAY CAUSE SEVERE IRRITATION, INFLAMMATION, ULCERATION, NECROSIS AND CHEMICAL BURNS. SHOCK SYMPTOMS MAY DEVELOP INCLUDING RAPID PULSE, SWEATING AND COLLAPSE. PHOTOSENSITIZATION REACTIONS MAY OCCUR IN PERSONS PREVIOUSLY EXPOSED. CONTACT WITH A COMPRESSED GAS MAY CAUSE FROSTBITE.
CHRONIC EXPOSURE- REPEATED OR PROLONGED CONTACT WITH VAPORS OR DILUTE SOLUTIONS MAY CAUSE DERMATITIS. PHOTOSENSITIZATION MAY OCCUR.

FIRST AID- REMOVE CONTAMINATED CLOTHING AND SHOES IMMEDIATELY. WASH AFFECTED AREA WITH SOAP OR MILD DETERGENT AND LARGE AMOUNTS OF WATER UNTIL NO EVIDENCE OF CHEMICAL REMAINS (AT LEAST 15-20 MINUTES). IN CASE OF CHEMICAL BURNS, COVER AREA WITH STERILE, DRY DRESSING. BANDAGE SECURELY, BUT NOT TOO TIGHTLY. GET MEDICAL ATTENTION IMMEDIATELY.

EYE CONTACT:
HYDROGEN CHLORIDE (HYDROCHLORIC ACID):

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CORROSIVE.
ACUTE EXPOSURE- CONTACT MAY CAUSE SEVERE IRRITATION, CONJUNCTIVITIS, CORNEAL NECROSIS AND BURNS WITH IMPAIRMENT OR PERMANENT LOSS OF VISION. A DROP OF HYDROCHLORIC ACID SPLASHED IN THE EYE AND IMMEDIATELY WASHED OUT HAS PRODUCED A WHITE COAGULATION OF THE CORNEAL AND CONJUNCTIVAL EPITHELIUM. ANIMALS EXPOSED TO VAPOR CONCENTRATIONS OF 1350 PPM FOR ONE AND A HALF HOURS SHOWED CLOUDING OF THE CORNEA AND 300 PPM FOR 6 HOURS SHOWED SLIGHT EROSION OF THE CORNEAL EPITHELIUM. CONTACT WITH A COMPRESSED GAS MAY CAUSE FROSTBITE.
CHRONIC EXPOSURE- ANIMALS EXPOSED TO VAPOR AT 100 PPM FOR 6 HOURS DAILY FOR 50 DAYS SHOWED ONLY SLIGHT UNREST AND IRRITATION OF THE EYES, BUT NO OCULAR INJURY. EFFECTS ARE DEPENDENT UPON CONCENTRATION AND DURATION OF EXPOSURE. CONJUNCTIVITIS OR EFFECTS SIMILAR TO THOSE FOR ACUTE EXPOSURE MAY OCCUR.

FIRST AID- WASH EYES IMMEDIATELY WITH LARGE AMOUNTS OF WATER, OCCASIONALLY LIFTING UPPER AND LOWER LIDS, UNTIL NO EVIDENCE OF CHEMICAL REMAINS (AT LEAST 15-20 MINUTES). CONTINUE IRRIGATING WITH NORMAL SALINE UNTIL THE PH HAS RETURNED TO NORMAL (30-60 MINUTES). COVER WITH STERILE BANDAGES. GET MEDICAL ATTENTION IMMEDIATELY.

INGESTION:
HYDROGEN CHLORIDE (HYDROCHLORIC ACID):
CORROSIVE.
ACUTE EXPOSURE- INGESTION OF THE ACID MAY CAUSE BURNS OF THE MOUTH, THROAT, ESOPHAGUS AND STOMACH WITH CONSEQUENT PAIN, UNEASINESS, NAUSEA, SALIVATION, VOMITING, DIARRHEA, CHILLS, SHOCK AND INTENSE THIRST. NEPHRITIS, FEVER AND PERFORATION OF THE INTESTINAL TRACT, AND CIRCULATORY COLLAPSE MAY OCCUR. DEATH MAY BE DUE TO ESOPHAGEAL OR GASTRIC NECROSIS.
CHRONIC EXPOSURE- NO DATA AVAILABLE.

FIRST AID- DO NOT USE GASTRIC LAVAGE OR EMESIS. DILUTE THE ACID IMMEDIATELY BY DRINKING LARGE QUANTITIES OF WATER OR MILK. IF VOMITING PERSISTS, ADMINISTER FLUIDS REPEATEDLY. INGESTED ACID MUST BE DILUTED APPROXIMATELY 100 FOLD TO RENDER IT HARMLESS TO TISSUES. MAINTAIN AIRWAY AND TREAT SHOCK. (DREISBACH, HANDBOOK OF POISONING, 12TH ED.). GET MEDICAL ATTENTION IMMEDIATELY. IF VOMITING OCCURS, KEEP HEAD BELOW HIPS TO HELP PREVENT ASPIRATION.

ANTIDOTE:
NO SPECIFIC ANTIDOTE. TREAT SYMPTOMATICALLY AND SUPPORTIVELY.

REACTIVITY

REACTIVITY:
REACTS EXOTHERMICALLY WITH WATER OR STEAM TO PRODUCE TOXIC AND CORROSIVE FUMES.

CONTACT WITH COMMON METALS PRODUCES HYDROGEN WHICH MAY FORM EXPLOSIVE MIXTURES WITH AIR.

INCOMPATIBILITIES:
HYDROGEN CHLORIDE (HYDROCHLORIC ACID):
ACETIC ANHYDRIDE: VIOLENT REACTION.
ALCOHOLIC HYDROGEN CYANIDE: EXPLOSIVE REACTION.
ALUMINUM: EXPLOSION.
ALUMINUM-TITANIUM ALLOYS: IGNITES OR INCANDESCES WHEN HEATED.
2-AMINOETHANOL: VIOLENT REACTION.
AMMONIUM HYDROXIDE: VIOLENT REACTION.
BASES: VIOLENT REACTION.
BRASS: CORRODES.
BRONZE: CORRODES.
CALCIUM CARBIDE: REACTS WITH INCANDESCENCE.
CALCIUM HYPOCHLORITE: IGNITION.
CESIUM ACETYLIDE: IGNITES ON CONTACT.
CHLORINE + DINITROANILINES: VIGOROUS REACTION WITH RELEASE OF FLAMMABLE HYDROGEN GAS FUMES.
CHLOROSULFONIC ACID: VIOLENT REACTION.
1,1-DIFLUOROETHYLENE: EXTREMELY EXOTHERMIC DECOMPOSITION REACTION.
DOWICIL 100: DECOMPOSES.
ETHYLENE DIAMINE: VIOLENT REACTION.
ETHYLENE IMINE: VIOLENT REACTION.
FLUORINE: IGNITES ON CONTACT.
HEXALITHIUM DISILICIDE: INCANDESCES.
IRON: CORRODES WITH EVOLUTION OF FLAMMABLE HYDROGEN GAS.
MAGNESIUM BORIDE: PRODUCES A SPONTANEOUSLY FLAMMABLE GAS.
MERCURIC SULFATE: VIOLENT REACTION AT 125 C.
METAL ACETYLIDES: VIOLENT REACTION.
METALS: SEVERE CORROSION WITH EVOLUTION OF FLAMMABLE HYDROGEN GAS.
OLEUM: VIOLENT REACTION.
OXIDIZERS (STRONG): VIOLENT REACTION.
OXYGEN + PLATINUM: IGNITES ON CONTACT.
PERCHLORIC ACID: VIOLENT REACTION.
PLASTICS, RUBBER, COATINGS: ATTACKS.
POTASSIUM PERMANGANATE: EXPLOSION HAZARD.
BETA-PROPIOLACTONE: VIOLENT REACTION.
PROPYLENE OXIDE: VIOLENT REACTION.
SILICA (GEL): INCOMPATIBLE.

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SODIUM: VIGOROUS OR EXPLOSIVE REACTION.
SULFURIC ACID: EXPLOSIVE REACTION WITH RELEASE OF TOXIC HYDROGEN CHLORIDE GAS.
TETRASELENIUM TETRANITRIDE: EXPLODES ON CONTACT.
VINYL ACETATE: VIOLENT REACTION.

DECOMPOSITION:
THERMAL DECOMPOSITION MAY RELEASE CORROSIVE HYDROGEN CHLORIDE.

POLYMERIZATION:
HAZARDOUS POLYMERIZATION HAS NOT BEEN REPORTED TO OCCUR UNDER NORMAL TEMPERATURES AND PRESSURES.

STORAGE AND DISPOSAL

OBSERVE ALL FEDERAL, STATE AND LOCAL REGULATIONS WHEN STORING OR DISPOSING OF THIS SUBSTANCE. FOR ASSISTANCE, CONTACT THE DISTRICT DIRECTOR OF THE ENVIRONMENTAL PROTECTION AGENCY.

STORAGE

THRESHOLD PLANNING QUANTITY (TPQ):
THE SUPERFUND AMENDMENTS AND REAUTHORIZATION ACT (SARA) SECTION 302 REQUIRES THAT EACH FACILITY WHERE ANY EXTREMELY HAZARDOUS SUBSTANCE IS PRESENT IN A QUANTITY EQUAL TO OR GREATER THAN THE TPQ ESTABLISHED FOR THAT SUBSTANCE NOTIFY THE STATE EMERGENCY RESPONSE COMMISSION FOR THE STATE IN WHICH IT IS LOCATED. SECTION 303 OF SARA REQUIRES THESE FACILITIES TO PARTICIPATE IN LOCAL EMERGENCY RESPONSE PLANNING (40 CFR 355.30).

PROTECT AGAINST PHYSICAL DAMAGE. STORE IN COOL, WELL-VENTILATED PLACE, SEPARATED FROM ALL OXIDIZING MATERIALS (NFPA 49, HAZARDOUS CHEMICALS DATA, 1975).

****

CONDITIONS TO AVOID

MAY BURN BUT DOES NOT IGNITE READILY. FLAMMABLE. POISONOUS GASES MAY ACCUMULATE IN TANKS AND HOPPER CARS. MAY IGNITE COMBUSTIBLES (WOOD, PAPER, OIL, ETC.).

****

SPILL AND LEAK PROCEDURES

SOIL SPILL:
DIG HOLDING AREA SUCH AS LAGOON, POND OR PIT FOR CONTAINMENT.

DIKE FLOW OF SPILLED MATERIAL USING SOIL OR SANDBAGS OR FOAMED BARRIERS SUCH AS POLYURETHANE OR CONCRETE.

USE CEMENT POWDER OR FLY ASH TO ABSORB LIQUID MASS.

NEUTRALIZE SPILL WITH SLAKED LIME, SODIUM BICARBONATE OR CRUSHED LIMESTONE.

AIR SPILL:
KNOCK DOWN VAPORS WITH WATER SPRAY. KEEP UPWIND.

WATER USED TO KNOCK DOWN VAPORS MAY BECOME CORROSIVE OR TOXIC AND SHOULD BE CONTAINED PROPERLY FOR LATER DISPOSAL.

WATER SPILL:
NEUTRALIZE WITH AGRICULTURAL LIME, SLAKED LIME, CRUSHED LIMESTONE, OR SODIUM BICARBONATE.

OCCUPATIONAL SPILL:
DO NOT TOUCH SPILLED MATERIAL. STOP LEAK IF YOU CAN DO IT WITHOUT RISK. FOR SMALL SPILLS, TAKE UP WITH SAND OR OTHER ABSORBENT MATERIAL AND PLACE INTO CONTAINERS FOR LATER DISPOSAL. FOR SMALL DRY SPILLS, WITH CLEAN SHOVEL PLACE MATERIAL INTO CLEAN, DRY CONTAINER AND COVER. MOVE CONTAINERS FROM SPILL AREA. FOR LARGER SPILLS, DIKE FAR AHEAD OF SPILL FOR LATER DISPOSAL. KEEP UNNECESSARY PEOPLE AWAY. ISOLATE HAZARD AREA AND DENY ENTRY.

REPORTABLE QUANTITY (RQ): 1 POUND
THE SUPERFUND AMENDMENTS AND REAUTHORIZATION ACT (SARA) SECTION 304 REQUIRES THAT A RELEASE EQUAL TO OR GREATER THAN THE REPORTABLE QUANTITY FOR THIS SUBSTANCE BE IMMEDIATELY REPORTED TO THE LOCAL EMERGENCY PLANNING COMMITTEE AND THE STATE EMERGENCY RESPONSE COMMISSION (40 CFR 355.40). IF THE RELEASE OF THIS SUBSTANCE IS REPORTABLE UNDER CERCLA SECTION 103, THE NATIONAL RESPONSE CENTER MUST BE NOTIFIED IMMEDIATELY AT (800) 424-8802 OR (202) 426-2675 IN THE METROPOLITAN WASHINGTON, D.C. AREA (40 CFR 302.6).

PROTECTIVE EQUIPMENT

VENTILATION:
PROVIDE LOCAL EXHAUST OR PROCESS ENCLOSURE VENTILATION TO MEET PUBLISHED

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RESPIRATOR:
THE SPECIFIC RESPIRATOR SELECTED MUST BE BASED ON THE CONTAMINATION LEVELS FOUND IN THE WORK PLACE, MUST NOT EXCEED THE WORKING LIMITS OF THE RESPIRATOR AND BE JOINTLY APPROVED BY THE NATIONAL INSTITUTE FOR OCCUPATIONAL SAFETY AND HEALTH AND THE MINE SAFETY AND HEALTH ADMINISTRATION.
THE FOLLOWING RESPIRATORS ARE RECOMMENDED BASED ON THE DATA FOUND IN THE PHYSICAL DATA, HEALTH EFFECTS AND TOXICITY SECTIONS. THEY ARE RANKED IN ORDER FROM MINIMUM TO MAXIMUM RESPIRATORY PROTECTION:

HYDROGEN CHLORIDE (HYDROCHLORIC ACID):

50 PPM- ANY SUPPLIED-AIR RESPIRATOR.
ANY SELF-CONTAINED BREATHING APPARATUS.
ANY CHEMICAL CARTRIDGE RESPIRATOR WITH CARTRIDGE(S) PROVIDING PROTECTION AGAINST HYDROCHLORIC ACID.
ANY POWERED AIR-PURIFYING RESPIRATOR WITH CARTRIDGE(S) PROVIDING PROTECTION AGAINST HYDROCHLORIC ACID.

100 PPM- ANY SUPPLIED-AIR RESPIRATOR OPERATED IN A CONTINUOUS FLOW MODE.
ANY SUPPLIED-AIR RESPIRATOR WITH A FULL FACEPIECE.
ANY SELF-CONTAINED BREATHING APPARATUS WITH A FULL FACEPIECE.

ESCAPE- ANY AIR-PURIFYING FULL FACEPIECE RESPIRATOR (GAS MASK) WITH A CHIN-STYLE OR FRONT- OR BACK-MOUNTED ACID GAS CANISTER.
ANY APPROPRIATE ESCAPE-TYPE SELF-CONTAINED BREATHING APPARATUS.

FOR FIREFIGHTING AND OTHER IMMEDIATELY DANGEROUS TO LIFE OR HEALTH CONDITIONS:

SELF-CONTAINED BREATHING APPARATUS WITH FULL FACEPIECE OPERATED IN PRESSURE DEMAND OR OTHER POSITIVE PRESSURE MODE.

SUPPLIED-AIR RESPIRATOR WITH FULL FACEPIECE AND OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE PRESSURE MODE IN COMBINATION WITH AN AUXILIARY SELF-CONTAINED BREATHING APPARATUS OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE PRESSURE MODE.

CLOTHING:
EMPLOYEE MUST WEAR APPROPRIATE PROTECTIVE (IMPERVIOUS) CLOTHING AND EQUIPMENT TO PREVENT ANY POSSIBILITY OF SKIN CONTACT WITH THIS SUBSTANCE.

GLOVES:
EMPLOYEE MUST WEAR APPROPRIATE PROTECTIVE GLOVES TO PREVENT CONTACT WITH THIS SUBSTANCE.

EYE PROTECTION:
EMPLOYEE MUST WEAR SPLASH-PROOF OR DUST-RESISTANT SAFETY GOGGLES AND A FACESHIELD TO PREVENT CONTACT WITH THIS SUBSTANCE. CONTACT LENSES SHOULD NOT BE WORN.

EMERGENCY WASH FACILITIES:
WHERE THERE IS ANY POSSIBILITY THAT AN EMPLOYEE'S EYES AND/OR SKIN MAY BE EXPOSED TO THIS SUBSTANCE, THE EMPLOYER SHOULD PROVIDE AN EYE WASH FOUNTAIN AND QUICK DRENCH SHOWER WITHIN THE IMMEDIATE WORK AREA FOR EMERGENCY USE.

AUTHORIZED - FISHER SCIENTIFIC GROUP, INC.
CREATION DATE: 04/30/85 REVISION DATE: 03/30/89

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Fisher Scientific Company

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Fair Lawn, NJ 07410

MATERIAL SAFETY DATA SHEET (Adapted from USDL From LSD-005-4) (201) 796-710

SECTION I. IDENTIFICATION OF PRODUCT

CHEMICAL NAME	FORMULA
Sodium Hydroxide Solution N/100 to 1N	

SYNONYM OR CROSS REFERENCE
SO-S-266, 270, 272, 274, 276, 277, 278, 282, 283, 284, 441

SECTION II. HAZARDOUS INGREDIENTS

MATERIAL	NATURE OF HAZARD
Sodium Hydroxide Solution	Corrosive

SECTION III. PHYSICAL DATA

BOILING POINT approximately 100°C	MELTING POINT approximately 0°C
VAPOR PRESSURE(mm Hg) 14 (water)	SPECIFIC GRAVITY 1.0
OR DENSITY (AIR = 1) 0.7 (water)	PERCENT VOLATILE BY VOLUME (%) 96 - 100%
ATER SOLUBILITY complete	EVAPORATION RATE (ether = 1) greater than 1

APPEARANCE Colorless liquid

SECTION IV. FIRE AND EXPOSION HAZARD DATA

FLASH POINT (method used) (°F)	FLAMMABLE LIMITS	Uel	Lel
NA	NA		

FIRE EXTINGUISHING MEDIA NA

SPECIAL FIRE-FIGHTING PROCEDURES NA

UNUSUAL FIRE AND EXPLOSION HAZARD NA

SECTION V. HEALTH HAZARD

THRESHOLD LIMIT VALUE None listed

HEALTH HAZARDS May cause irritation to skin and eyes.

ST AID PROCEDURES Skin and eyes: Flush with water for at least 15 minutes. eyes; contact a physician.

SECTION VI. REACTIVITY DATA

STABILITY			CONDITIONS TO AVOID
	UNSTABLE		
	STABLE	X	

IMCOMBATIBILITY (material to avoid)

HAZARDOUS DECOMPOSITION PRODUCTS

HAZARDOUS POLYMERIZATION			CONDITIONS TO AVOID
	MAY OCCUR		
	WILL NOT OCCUR	X	

SECTION VII. SPILL AND DISPOSAL PROCEDURES

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED

Absorb on vermiculite. Scoop up and place in a suitable container.

WASTE DISPOSAL METHOD

DISPOSE OF BY MEANS AS TO COMPLY WITH ALL LOCAL, STATE, AND FEDERAL REGULATIONS OR CONTACT AN APPROVED AND LICENSED DISPOSAL AGENCY.

SECTION VIII. PROTECTION INFORMATION

RESPIRATORY PROTECTION (specify type)

none

VENTILATION	LOCAL	SPECIAL
none required	MECHANICAL (general)	OTHER

PROTECTIVE GLOVES	EYE PROTECTION
rubber	safety glasses

OTHER PROTECTIVE EQUIPMENT

SECTION IX. HANDLING AND STORAGE PRECAUTIONS

STORAGE AND HANDLING

SECTION X. MISCELLANEOUS INFORMATION

INFORMATION FURNISHED BY:	TITLE
Gaston L. Pillori	Manager of Quality Assurance

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REV. NO. 0 DATE September 29, 1980

Form No 75
11-79

SODIUM HYDROXIDE
SODIUM HYDROXIDE
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MATERIAL SAFETY DATA SHEET

FISHER SCIENTIFIC
CHEMICAL DIVISION
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INDEX: 15-8626-00286
CAT NO: S3183

SUBSTANCE IDENTIFICATION

CAS-NUMBER 1310-73-2

SUBSTANCE: **SODIUM HYDROXIDE**

TRADE NAMES/SYNONYMS: CAUSTIC SODA; SODA LYE; LYE; WHITE CAUSTIC; CAUSTIC ALKALI; CAUSTIC SODA, BEAD; CAUSTIC SODA, DRY; CAUSTIC SODA, FLAKE; CAUSTIC SODA, GRANULAR; CAUSTIC SODA, SOLID; SODIUM HYDRATE; SODIUM HYDROXIDE, BEAD; SODIUM HYDROXIDE, FLAKE; SODIUM HYDROXIDE, DRY; SODIUM HYDROXIDE, SOLID; S-613 ASCARITE; S-318; S-320; S-612

CHEMICAL FAMILY:
INORGANIC BASE

MOLECULAR FORMULA: NA-O-H MOL WT: 40.00

CERCLA RATINGS (SCALE 0-3): HEALTH=3 FIRE=0 REACTIVITY=1 PERSISTENCE=0

COMPONENTS AND CONTAMINANTS

PERCENT: 97 COMPONENT: SODIUM HYDROXIDE

PERCENT: 0.50 COMPONENT: SODIUM CARBONATE

PERCENT: .008 COMPONENT: SODIUM CHLORIDE

PERCENT: <0.1 COMPONENT: SODIUM SULFATE

PERCENT: 0.1 COMPONENT: POTASSIUM, CALCIUM, AND MAGNESIUM

OTHER CONTAMINANTS: SILICON DIOXIDE (0.03%) AND OTHER METALS (0.01%).

EXPOSURE LIMITS:
2 MG/M3 OSHA TWA; 2 MG/M3 ACGIH CEILING; 2 MG/M3 NIOSH RECOMMENDED 15 MINUTE CEILING.

PHYSICAL DATA

DESCRIPTION: ODORLESS, WHITE OR OFF-WHITE HYGROSCOPIC SOLID.

BOILING POINT: 2534 F (1390 C) MELTING POINT: 605 F (318 C)

SPECIFIC GRAVITY: 2.1 VAPOR PRESSURE: 42 MMHG @ 1000 C

PH: 14 FOR A 5% AQ SOLN SOLUBILITY IN WATER: 42%

SOLVENT SOLUBILITY: ALCOHOL, GLYCEROL.

FIRE AND EXPLOSION DATA

FIRE AND EXPLOSION HAZARD:
NEGLIGIBLE FIRE AND EXPLOSION HAZARD WHEN EXPOSED TO HEAT OR FLAME.

FLASH POINT: NON-FLAMMABLE

FIREFIGHTING MEDIA:
DRY CHEMICAL, CARBON DIOXIDE, WATER SPRAY OR FOAM
(1984 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.3).

FOR LARGER FIRES, USE FLOODING QUANTITIES OF WATER.

FIREFIGHTING:
MOVE CONTAINERS FROM FIRE AREA IF POSSIBLE. COOL CONTAINERS EXPOSED TO FLAMES WITH WATER FROM SIDE UNTIL WELL AFTER FIRE IS OUT (1984 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.3).

TOXICITY

1%/24 HOURS EYE-MONKEY SEVERE IRRITATION; 50 MG/24 HOURS SKIN-RABBIT SEVERE IRRITATION; 1% EYE-RABBIT SEVERE IRRITATION; 50 UG/24 HOURS EYE-RABBIT SEVERE IRRITATION; 1 MG/24 HOURS EYE-RABBIT SEVERE IRRITATION;
CARCINOGEN STATUS: NONE.
SODIUM HYDROXIDE IS AN EYE AND MUCOUS MEMBRANE IRRITANT AND SEVERE SKIN IRRITANT.

HEALTH EFFECTS AND FIRST AID

INHALATION:
CORROSIVE. 200 MG/M3 IS IMMEDIATELY DANGEROUS TO LIFE AND HEALTH.
ACUTE EXPOSURE- THE EFFECTS OF THE DUST OR MIST WILL VARY FROM MILD IRRITATION OF THE NOSE @ 2 MG/M3 TO SEVERE PNEUMONITIS DEPENDING ON THE SEVERITY OF EXPOSURE. LOW CONCENTRATIONS MAY CAUSE SORE THROAT, COUGHING, AND LABORED BREATHING. INTENSE EXPOSURES MAY RESULT IN DELAYED PULMONARY EDEMA.

CHRONIC EXPOSURE- PROLONGED EXPOSURE MAY CAUSE BRONCHIAL IRRITATION, COUGHING, BRONCHIAL PNEUMONIA, AND GASTROINTESTINAL DISTURBANCES.

FIRST AID- REMOVE FROM EXPOSURE AREA TO FRESH AIR IMMEDIATELY. IF BREATHING

HAS STOPPED, GIVE ARTIFICIAL RESPIRATION. KEEP AFFECTED PERSON WARM AND AT REST. GET MEDICAL ATTENTION AS SOON AS POSSIBLE.

SKIN CONTACT:
CORROSIVE.
ACUTE EXPOSURE- ON THE SKIN, SOLUTIONS OF 25 TO 50% MAY CAUSE THE SENSATION OF IRRITATION WITHIN ABOUT 3 MINUTES. WITH SOLUTIONS OF 4% THIS DOES NOT OCCUR UNTIL AFTER SEVERAL HOURS. IF NOT REMOVED FROM THE SKIN, SEVERE BURNS WITH DEEP ULCERATION MAY OCCUR. EXPOSURE TO THE DUST OR MIST MAY CAUSE MULTIPLE SMALL BURNS AND TEMPORARY LOSS OF HAIR.

CHRONIC EXPOSURE- REPEATED EXPOSURE MAY RESULT IN DERMATITIS.

FIRST AID- REMOVE CONTAMINATED CLOTHING WHILE RUNNING STREAMS OF WATER UNDER CLOTHING. WASH AFFECTED AREA WITH SOAP OR MILD DETERGENT AND LARGE AMOUNTS OF WATER (APPROXIMATELY 15-20 MINUTES) UNTIL NO EVIDENCE OF CHEMICAL REMAINS. FOR CHEMICAL BURNS, APPLY STERILE BANDAGE SECURELY, BUT NOT TOO TIGHTLY. GET MEDICAL ATTENTION.

EYE CONTACT:
CORROSIVE.
ACUTE EXPOSURE- CONTACT MAY CAUSE DISINTEGRATION AND SLOUGHING OF CONJUNCTIVAL AND CORNEAL EPITHELIUM, CORNEAL OPACIFICATION, MARKED EDEMA AND ULCERATION; AFTER 7 TO 13 DAYS EITHER GRADUAL RECOVERY BEGINS OR THERE IS PROGRESSION OF ULCERATION AND CORNEAL OPACIFICATION. COMPLICATIONS OF SEVERE EYE BURNS ARE SYMBLEPHARON WITH OVERGROWTH OF THE CORNEA BY A VASCULARIZED MEMBRANE, PROGRESSIVE OR RECURRENT CORNEAL ULCERATION AND PERMANENT CORNEAL OPACIFICATION.

CHRONIC EXPOSURE- REPEATED OR PROLONGED VAPOR CONTACT AT LOW LEVELS OF EXPOSURE MAY CAUSE CONJUNCTIVITIS.

FIRST AID- WASH EYES IMMEDIATELY WITH LARGE AMOUNTS OF WATER, OCCASIONALLY LIFTING THE UPPER AND LOWER LIDS, UNTIL NO EVIDENCE OF CHEMICAL REMAINS (APPROXIMATELY 15-20 MINUTES). GET MEDICAL ATTENTION.

INGESTION:
CORROSIVE.
ACUTE EXPOSURE- SEVERE ABDOMINAL PAIN, CORROSION OF THE LIPS, MOUTH, TONGUE, AND PHARYNX, AND VOMITING OF LARGE PIECES OF MUCOSA. ASPHYXIA CAN OCCUR FROM SWELLING OF THE THROAT. PERFORATION OF THE ESOPHAGUS AND STOMACH CAN OCCUR. CASES OF SQUAMOUS CELL CARCINOMA OF THE ESOPHAGUS HAVE OCCURRED WITH LATENT PERIODS OF 12 TO 42 YEARS AFTER INGESTION; A RESULT TISSUE DESTRUCTION AND POSSIBLY SCAR FORMATION RATHER THAN THE RESULT OF DIRECT CARCINOGENIC ACTION.

FIRST AID- IF VICTIM IS CONSCIOUS, GIVE HIM LARGE QUANTITIES OF WATER IMMEDIATELY TO DILUTE THE ALKALI. DO NOT INDUCE VOMITING. GET MEDICAL ATTENTION IMMEDIATELY.

REACTIVITY

--REACTIVITY:
THE SUBSTANCE IS A STRONG BASE. IT REACTS EXOTHERMICALLY WITH WATER RELEASING CORROSIVE FUMES OF SODIUM HYDROXIDE.

INCOMPATIBILITIES:

ACETALDEHYDE: RESULTS IN VIOLENT POLYMERIZATION OF ACETALDEHYDE.
ACETIC ACID: MIXING IN A CLOSED CONTAINER INCREASES TEMPERATURE AND PRESSURE
ACETIC ANHYDRIDE: MIXING IN A CLOSED CONTAINER INCREASES TEMPERATURE AND PRESSURE.
ACROLEIN: RESULTS IN AN EXTREMELY VIOLENT POLYMERIZATION OF ACROLEIN.
ACRYLONITRILE: VIOLENT POLYMERIZATION.
ALLYL ALCOHOL: AS A BENZENE EXTRACT OF ALLYL BENZENESULFONATE WAS PREPARED FROM ALLYL ALCOHOL AND BENZENE SULFONYL CHLORIDE IN THE PRESENCE OF AQUEOUS SODIUM HYDROXIDE, UNDER VACUM DISTILLATION TWO FRACTIONS CAME OFF, THEN THE TEMPERATURE ROSE TO 135 C, WHEN THE RESIDUE DARKENED AND EXPLODED.
ALLYL CHLORIDE: IN CONTACT WITH DRY CAUSTIC SODA BEADS, HYDROLYSIS MAY TAKE PLACE PRODUCING ALLYL ALCOHOL.
ALUMINUM: VIGOROUS REACTION WITH THE EVOLUTION OF FLAMMABLE HYDROGEN GAS.
CHLORINE TRIFLUORIDE: VIOLENT REACTION.
CHLOROFORM AND METHYL ALCOHOL: EXOTHERMIC REACTION.
CHLOROHYDRIN: MIXING IN A CLOSED CONTAINER CAUSES AN INCREASE IN TEMPERATURE AND PRESSURE.
CHLORONITROTOLUENES: POSSIBLE EXPLOSION.
CHLOROSULFONIC ACID: MIXING IN A CLOSED CONTAINER CAUSES AN INCREASE IN TEMPERATURE AND PRESSURE.
1,2-DICHLOROETHYLENE: MAY FORM SPONTANEOUSLY FLAMMABLE MONOCHLOROACETYLENE.
ETHYLENE CYANOHYDRIN: MIXING IN A CLOSED CONTAINER CAUSES AN INCREASE IN TEMPERATURE AND PRESSURE.
GLYOXAL: MIXING IN A CLOSED CONTAINER INCREASES TEMPERATURE AND PRESSURE.
HALOGENATED HYDROCARBONS: VIOLENT REACTION.
HYDROCHLORIC ACID: MIXING IN A CLOSED CONTAINER CAUSES AN INCREASE IN TEMPERATURE AND PRESSURE.
HYDROFLUORIC ACID: MIXING IN A CLOSED CONTAINER CAUSES AN INCREASE IN TEMPERATURE AND PRESSURE.
HYDROQUINONE: RAPID DECOMPOSITION OF HYDROQUINONE WITH EVOLUTION OF HEAT.
MALEIC ANHYDRIDE: EXPLOSIVE DECOMPOSITION.
METALS: CORRODES METALS, REACTING TO FORM FLAMMABLE HYDROGEN GAS.
NITRIC ACID: MIXING IN A CLOSED CONTAINER INCREASES TEMPERATURE AND PRESSURE
NITROETHANE: FORMS AN EXPLOSIVE SALT.
NITROMETHANE: FORMS AN EXPLOSIVE SALT.
NITROPARAFFINS: THE NITROPARAFFINS, IN THE PRESENCE OF WATER, FORM DRY SALTS WITH ORGANIC BASES. THE DRY SALTS ARE EXPLOSIVE.
NITROPROPANE: FORMS AN EXPLOSIVE SALT.
OLEUM: MIXING IN A CLOSED CONTAINER CAUSES AN INCREASE IN TEMPERATURE AND PRESSURE.
PENTOL (3-METHYL-2-PENTEN-4-YN-1-OL): POSSIBLE EXPLOSION.
PHOSPHORUS: PHOSPHORUS BOILED WITH ALKALINE HYDROXIDES YIELDS MIXED PHOSPHINES WHICH MAY IGNITE SPONTANEOUSLY IN AIR.
PHOSPHORUS PENTOXIDE: EXTREMELY VIOLENT REACTION WHEN INITIATED BY LOCAL HEATING.
B-PROPIOLACTONE: MIXING IN A CLOSED CONTAINER CAUSES AN INCREASE IN TEMPERATURE AND PRESSURE.
SULFURIC ACID: MIXING IN A CLOSED CONTAINER CAUSES AN INCREASE IN TEMPERAURE AND PRESSURE.
TETRACHLOROBENZENE AND METHYL ALCOHOL: POSSIBLE EXPLOSION.
TETRAHYDROFURAN: SERIOUS EXPLOSIONS CAN OCCUR.
TRICHLOROETHYLENE: FORMATION OF EXPLOSIVE MIXTURES OF DICHLOROACETYLENE.
WATER: CAUSTIC SODA BEADS IN CONTACT WITH WATER MAY GENERATE ENOUGH HEAT TO IGNITE ADJACENT COMBUSTIBLES.

DECOMPOSITION:
MAY RELEASE TOXIC FUMES OF SODIUM OXIDE, WHICH CAN REACT WITH WATER OR STEAM TO PRODUCE HEAT AND FLAMMABLE HYDROGEN VAPORS.

POLYMERIZATION:
NOT KNOWN TO OCCUR.

****
CONDITIONS TO AVOID

MAY BURN BUT DOES NOT IGNITE READILY. FLAMMABLE, POISONOUS GASES MAY ACCUMULATE IN TANKS AND HOPPER CARS. MAY IGNITE COMBUSTIBLES (WOOD, PAPER, OIL, ETC.).

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SPILL AND LEAK PROCEDURES

SOIL SPILL:
DIG HOLDING AREA SUCH AS LAGOON, POND OR PIT FOR CONTAINMENT.

USE PROTECTIVE COVER SUCH AS A PLASTIC SHEET TO PREVENT MATERIAL FROM DISSOLVING IN FIRE EXTINGUISHING WATER OR RAIN.

WATER SPILL:
ADD SUITABLE AGENT TO NEUTRALIZE SPILLED MATERIAL TO PH-7.

OCCUPATIONAL SPILL:
DO NOT TOUCH SPILLED MATERIAL. STOP LEAK IF YOU CAN DO IT WITHOUT RISK. FOR SMALL SPILLS, TAKE UP WITH SAND OR OTHER ABSORBENT MATERIAL AND PLACE INTO CONTAINERS FOR LATER DISPOSAL. FOR SMALL DRY SPILLS, WITH CLEAN SHOVEL PLACE MATERIAL INTO CLEAN, DRY CONTAINER AND COVER. MOVE CONTAINERS FROM SPILL AREA. FOR LARGER SPILLS, DIKE FAR AHEAD OF SPILL FOR LATER DISPOSAL. KEEP UNNECESSARY PEOPLE AWAY. ISOLATE HAZARD AREA AND DENY ENTRY.

PROTECTIVE EQUIPMENT

VENTILATION:
PROVIDE LOCAL EXHAUST VENTILATION SYSTEM TO MEET PERMISSIBLE EXPOSURE LIMITS.

RESPIRATOR:

100 MG/M3- HIGH-EFFICIENCY PARTICULATE RESPIRATOR WITH A FULL FACEPIECE.
SUPPLIED-AIR RESPIRATOR WITH A FULL FACEPIECE, HELMET, OR HOOD.
SELF-CONTAINED BREATHING APPARATUS WITH A FULL FACEPIECE.

200 MG/M3- POWERED AIR-PURIFYING RESPIRATOR WITH A HIGH-EFFICIENCY PARTICULATE FILTER AND A FULL FACEPIECE.
TYPE C SUPPLIED-AIR RESPIRATOR WITH A FULL FACEPIECE OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE PRESSURE MODE OR WITH A FULL FACEPIECE, HELMET, OR HOOD OPERATED IN CONTINUOUS-FLOW MODE.

ESCAPE- DUST MASK, EXCEPT SINGLE-USE AND QUARTER-MASK RESPIRATORS.
SELF-CONTAINED BREATHING APPARATUS.

FIREFIGHTING- SELF-CONTAINED BREATHING APPARATUS WITH A FULL FACEPIECE OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE PRESSURE MODE.

CLOTHING:
EMPLOYEE MUST WEAR APPROPRIATE PROTECTIVE CLOTHING AND EQUIPMENT TO PREVENT ANY POSSIBILITY OF SKIN CONTACT WITH THIS SUBSTANCE.

GLOVES:
EMPLOYEE MUST WEAR APPROPRIATE PROTECTIVE GLOVES TO PREVENT CONTACT WITH THIS SUBSTANCE.

EYE PROTECTION:
EMPLOYEE MUST WEAR SPLASH-PROOF OR DUST-RESISTANT SAFETY GOGGLES AND A FACESHIELD TO PREVENT CONTACT WITH THIS SUBSTANCE.

WHERE THERE IS ANY POSSIBILITY THAT AN EMPLOYEE'S EYES MAY BE EXPOSED TO THIS SUBSTANCE, THE EMPLOYER SHALL PROVIDE AN EYE-WASH FOUNTAIN WITHIN THE IMMEDIATE WORK AREA FOR EMERGENCY USE.

AUTHORIZED - ALLIED FISHER SCIENTIFIC
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