

Tenneco Chemicals
A Tenneco Company

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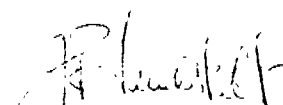
Mrs. Sonya Shashoua
New Jersey Department of Environmental Protection
Solid Waste Administration
32 East Hanover Street
Trenton, NJ 08625

Dear Mrs. Shashoua:

Per our recent conversation attached please find:

1. Copies of EPA Test Methods 106 and 107, for the determination of vinyl chloride monomer in air and residual monomer in slurry, wet cake and latex samples, respectively. As I indicated, these methods are selective to vinyl chloride, and were developed by EPA with regard to the NESHAPS Standard on Vinyl Chloride. The copies were taken from the EIC publication, but are available in 40CFR 61, Subpart F and the October 21, 1976 issue of the Federal Register.
2. A listing of the major raw materials used in the resin plant which could reach the landfill. Included are the materials for which analyses are run as part of Dr. Marwan Sadat's sludge quality assurance program.

Very truly yours,
TENNECO CHEMICALS, INC.



J. E. Sandstedt
Manager - Environmental Affairs

JPS:mm

cc: Mr. F. W. Kanzler - Burlington

COLORITE 017042

P = Production of grade G , resin represented by the sample, in kg
 G_i = Grade of resin, e.g., G_1 , G_2 , and G_3
 n = Total number of grades of resin produced during the 24-hour period.

(vi) The owner or operator shall retain at the source and make available for inspection by the Administrator for a minimum of 2 years records of all data needed to furnish the information required by paragraph (c) (2) (v) of this section: The records are to contain the following information:

(A) The vinyl chloride content found in all the samples required in paragraphs (c) (2) (i) and (c) (2) (ii) of this section, identified by the resin type and grade and the time and date of the sample, and
 (B) The corresponding quantity of polyvinyl chloride resin processed by the stripper(s), identified by the resin type and grade and the time and date it represents.

(3) The owner or operator shall include in the report a record of the emissions from each reactor opening for which an emission limit is prescribed in § 61.64(a) (2). Emissions are to be determined in accordance with § 61.67(g) (5), except that emissions for each reactor are to be determined. For a reactor that is also used as a stripper, the determination may be made immediately following the stripping operation.

§ 61.71 Recordkeeping.

(a) The owner or operator of any source to which this subpart applies shall retain the following information at the source and make it available for inspection by the Administrator for a minimum of two years:

(1) A record of the leaks detected by the vinyl chloride monitoring system, as required by § 61.65(b) (8), including the concentrations of vinyl chloride as measured, analyzed, and recorded by the vinyl chloride detector, the location of each measurement and the date and approximate time of each measurement.

(2) A record of the leaks detected during routine monitoring with the portable hydrocarbon detector and the action taken to repair the leaks, as required by § 61.65(b) (8), including a brief statement explaining the location and cause of each leak detected with the portable hydrocarbon detector, the date and time of the leak, and any action taken to eliminate that leak.

(3) A record of emissions measured in accordance with § 61.68.

(4) A daily operating record for each polyvinyl chloride reactor, including pressures and temperatures.

2. Appendix B is amended by adding Test Methods 106 and 107 as follows.

METHOD 106—DETERMINATION OF VINYL CHLORIDE FROM STATIONARY SOURCES

INTRODUCTION

Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph, nor by those who are unfamiliar with source sampling, as there are many details that are beyond the scope of this presentation. Care must be exercised to prevent exposure of

sampling personnel to vinyl chloride, a carcinogen.

1. Principle and Applicability.

1.1 An integrated bag sample of stack gas containing vinyl chloride (chloroethene) is subjected to chromatographic analysis, using a flame ionization detector.

1.2 The method is applicable to the measurement of vinyl chloride in stack gases from ethylene dichloride, vinyl chloride and polyvinyl chloride manufacturing processes, except where the vinyl chloride is contained in particulate matter.

2. Range and Sensitivity

The lower limit of detection will vary according to the chromatograph used. Values reported include 1×10^{-7} mg and 4×10^{-7} mg.

3. Interferences.

Acetaldehyde, which can occur in some vinyl chloride sources, will interfere with the vinyl chloride peak from the Chromasorb 102 column. See sections 4.3.2 and 6.4. If resolution of the vinyl chloride peak is still not satisfactory for a particular sample, then chromatograph parameters can be further altered with prior approval of the Administrator. If alteration of the chromatograph parameters fails to resolve the vinyl chloride peak, then supplemental confirmation of the vinyl chloride peak through an absolute analytical technique, such as mass spectroscopy, must be performed.

4. Apparatus

4.1 Sampling (Figure 106-1).

4.1.1 Probe—Stainless steel, Pyrex glass, or Teflon tubing according to stack temperature, each equipped with a glass wool plug to remove particulate matter.

4.1.2 Sample line—Teflon, 6.4 mm outside diameter, of sufficient length to connect probe to bag. A new unused piece is employed for each series of bag samples that constitutes an emission test.

4.1.3 Male (2) and female (2) stainless steel quick-connects, with ball checks (one pair without) located as shown in Figure 106-1.

4.1.4 Tedlar bags, 100 liter capacity—To contain sample. Teflon bags are not acceptable. Aluminized Mylar bags may be used, provided that the samples are analyzed within 24 hours of collection.

4.1.5 Rigid leakproof containers for 4.1.4, with covering to protect contents from sunlight.

4.1.6 Needle valve—To adjust sample flow rate.

4.1.7 Pump—Leak-free. Minimum capacity 2 liters per minute.

4.1.8 Charcoal tube—To prevent admission of vinyl chloride to atmosphere in vicinity of samplers.

4.1.9 Flow meter—For observing sample flow rate, capable of measuring a flow range from 0.10 to 1.00 liter per minute.

4.1.10 Connecting tubing—Teflon, 6.4 mm outside diameter, to assemble sample train (Figure 106-1).

4.1.11 Pitot tube—Type S (or equivalent), attached to the probe so that the sampling flow rate can be regulated proportional to the stack gas velocity.

4.2 Sample recovery.

4.2.1 Tubing—Teflon, 6.4 mm outside diameter, to connect bag to gas chromatograph sample loop. A new unused piece is employed for each series of bag samples that constitutes an emission test, and is to be discarded upon conclusion of analysis of those bags.

4.3 Analysis.

4.3.1 Gas chromatograph—With flame ionization detector, potentiometric strip chart recorder and 1.0 to 5.0 ml heated sampling loop in automatic sample valve.

1. Mention of trade names on specific products does not constitute endorsement by the Environmental Protection Agency.

4.3.2 Chromatographic column—Stainless steel, 2 m x 3.2 mm, containing 80/100 mesh Chromasorb 102. A secondary column of GE SF-96, 20 percent on 60/80 mesh AW Chromasorb P, stainless steel, 2 m x 3.2 mm or Porapak T, 80/100 mesh, stainless steel, 1 m x 3.2 mm is required if acetaldehyde is present. If used, a secondary column is placed after the Chromasorb 102 column. The combined columns should then be operated at 120° C.

4.3.3 Flow meters (2)—Rotameter (type, 0 to 100 ml/min capacity, with flow control valves).

4.3.4 Gas regulators—For required gas cylinders.

4.3.5 Thermometer—Accurate to one degree centigrade, to measure temperature of heated sample loop at time of sample injection.

4.3.6 Barometer—Accurate to 5 mm Hg, to measure atmospheric pressure around gas chromatograph during sample analysis.

4.3.7 Pump—Leak-free. Minimum capacity 100 ml/min.

4.4 Calibration

4.4.1 Tubing—Teflon, 6.4 mm outside diameter, separate pieces marked for each calibration concentration.

4.4.2 Tedlar bags—Sixteen-inch square size, separate bag marked for each calibration concentration.

4.4.3 Syringe—0.5 ml, gas tight.

4.4.4 Syringe—50 ml, gas tight.

4.4.5 Flow meter—Rotameter type, 0 to 100 ml/min range accurate to $\pm 1\%$, to meter nitrogen in preparation of standard gas mixture.

4.4.6 Stop watch—Of known accuracy, to time gas flow in preparation of standard gas mixtures.

5. Reagents. It is necessary that all reagents be of chromatographic grade.

5.1 Analytical

5.1.1 Helium gas or nitrogen gas—Zero grade, for chromatographic carrier gas.

5.1.2 Hydrogen gas—Zero grade.

5.1.3 Oxygen gas, or Air, as required by the method—Zero grade.

5.2 Calibration. Use one of the following options: either 5.2.1 and 5.2.2, or 5.2.3.

5.2.1 Vinyl chloride, 99.9+ percent. Pure vinyl chloride gas certified by the manufacturer to contain a minimum of 99.9 percent vinyl chloride for use in the preparation of standard gas mixtures in Section 7.1. If the gas manufacturer maintains a bulk cylinder supply of 99.9+ percent vinyl chloride, the certification analysis may have been performed on this supply rather than on each gas cylinder prepared from this bulk supply. The date of gas cylinder preparation and the certified analysis must have been affixed to the cylinder before shipment from the gas manufacturer to the buyer.

5.2.2 Nitrogen gas, Zero grade, for preparation of standard gas mixtures.

5.2.3 Cylinder standards (3). Gas mixture standards (50, 10, and 5 ppm vinyl chloride in nitrogen cylinders) for which the gas composition has been certified by the manufacturer. The manufacturer must have recommended a maximum shelf life for each cylinder so that the concentration does not change greater than ± 5 percent from the certified value. The date of gas cylinder preparation, certified vinyl chloride concentration and recommended maximum shelf life must have been affixed to the cylinder before shipment from the gas manufacturer to the buyer. These gas mixture standards may be directly used to prepare a chromatograph calibration curve as described in section 7.3.

5.2.3.1 Cylinder standards certification. The concentration of vinyl chloride in nitrogen in each cylinder must have been certified by the manufacturer by a direct analysis of each cylinder using an analytical procedure that the manufacturer had calibrated on the day of cylinder analysis. The calibration of the analytical procedure shall, as a minimum, have utilized a three-point calibration curve.

It is recommended that the manufacturer maintain two calibration standards and use these standards in the following way: (1) a high concentration standard (between 50 and 100 ppm) for preparation of a calibration curve by an appropriate dilution technique. (2) a low concentration standard (between 5 and 10 ppm) for verification of the dilution technique used.

5.2.3.2 Establishment and verification of calibration standards The concentration of each calibration standard must have been established by the manufacturer using reliable procedures. Additionally, each calibration standard must have been verified by the manufacturer by one of the following procedures, and the agreement between the initially determined concentration value and the verification concentration value must be within ± 5 percent: (1) verification value determined by comparison with a calibrated vinyl chloride permeation tube, (2) verification value determined by comparison with a gas mixture prepared in accordance with the procedure described in section 7.1 and using 99.9+ percent vinyl chloride, or (3) verification value obtained by having the calibration standard analyzed by the National Bureau of Standards. All calibration standards must be renewed on a time interval consistent with the shelf life of the cylinder standards sold.

6. Procedure

6.1 Sampling Assemble the sample train as in Figure 106-1. Perform a bag leak check according to Section 7.4. Observe that all connections between the bag and the probe are tight. Place the end of the probe at the centroid of the stack and start the pump with the needle valve adjusted to yield a flow of 0.5 lpm. After a period of time sufficient to purge the line several times has elapsed, connect the vacuum line to the bag and evacuate the bag until the rotameter indicates no flow. Then reposition the sample and vacuum lines and begin the actual sampling, keeping the rate proportional to the stack velocity. Direct the gas exiting the rotameter away from sampling personnel. At the end of the sample period, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container. Protect the bag container from sunlight.

6.2 Sample storage. Sample bags must be kept out of direct sunlight. When at all possible analysis is to be performed within 24 hours, but in no case in excess of 72 hours of sample collection.

6.4 Analysis Set the column temperature to 100° C, the detector temperature to 150° C, and the sample loop temperature to 70° C. When optimum hydrogen and oxygen flow rates have been determined, verify and maintain these flow rates during all chromatograph operations. Using zero helium or nitrogen as the carrier gas, establish a flow rate in the range consistent with the manufacturer's requirements for satisfactory detector operation. A flow rate of approximately 40 ml/min should produce adequate separations. Observe the base line periodically and determine that the noise level has stabilized and that base line drift has ceased. Purge the sample loop for thirty seconds at the rate of 100 ml/min, then activate the sample valve. Record the injection time (the position of the pen on the chart at the time of sample injection), the sample number, the sample loop temperature, the column temperature, carrier gas flow rate, chart speed and the attenuator setting. Record the laboratory pressure. From the chart, select the peak having the retention time corresponding to vinyl chloride, as determined in Sec-

tion 7.2. Measure the peak area, A_m , by use of H_m and a disc integrator or a planimeter. Measure the peak height H_m . Record A_m and the retention time. Repeat the injection at least two times or until two consecutive vinyl chloride peaks do not vary in area more than 5%. The average value for these two areas will be used to compute the bag concentration.

Compare the ratio of H_m to A_m for the vinyl chloride sample with the same ratio for the standard peak which is closest in height. As a guideline, if these ratios differ by more than 10%, the vinyl chloride peak may not be pure (possibly acetaldehyde is present) and the secondary column should be employed (see Section 4.3.2).

6.5 Measure the ambient temperature and barometric pressure near the bag. (Assume the relative humidity to be 100 percent.) From a water saturation vapor pressure table, determine the record and water vapor content of the bag.

7. Calibration and Standards

7.1 Preparation of vinyl chloride standard gas mixtures. Evacuate a sixteen-inch square Tedlar bag that has passed a leak check (described in Section 7.4) and meter in 5 liters of nitrogen. While the bag is filling, use the 0.5 ml syringe to inject 250 μ l of 99.9+ percent vinyl chloride through the wall of the bag. Upon withdrawing the syringe needle, immediately cover the resulting hole with a piece of adhesive tape. The bag now contains a vinyl chloride concentration of 50 ppm. In a like manner use the other syringe to prepare gas mixtures having 10 and 5 ppm vinyl chloride concentrations. Place each bag on a smooth surface and alternately depress opposite sides of the bag 50 times to further mix the gases. These gas mixture standards may be used for 10 days from the date of preparation, after which time preparation of new gas mixtures is required. (CAUTION—Contamination may be a problem when a bag is reused if the new gas mixture standard contains a lower concentration than the previous gas mixture standard did.)

7.2 Determination of vinyl chloride retention time. This section can be performed simultaneously with Section 7.3. Establish chromatograph conditions identical with those in Section 6.3, above. Set attenuator to X 1 position. Flush the sampling loop with zero helium or nitrogen and activate the sample valve. Record the injection time, the sample loop temperature, the column temperature, the carrier gas flow rate, the chart speed and the attenuator setting. Record peaks and detector responses that occur in the absence of vinyl chloride. Maintain conditions. With the equipment plumbing arranged identically to Section 6.3, flush the sample loop for 30 seconds at the rate of 100 ml/min with one of the vinyl chloride calibration mixtures and activate the sample valve. Record the injection time. Select the peak that corresponds to vinyl chloride. Measure the distance on the chart from the injection time to the time at which the peak maximum occurs. This quantity, divided by the chart speed, is defined as the retention time. Record.

7.3 Preparation of chromatograph calibration curve. Make a gas chromatographic measurement of each gas mixture standard (described in section 5.2.2 or 7.1) using conditions identical with those listed in sections 6.3 and 6.4. Flush the sampling loop for 30 seconds at the rate of 100 ml/min with each standard gas mixture and activate the sample valve. Record C_s , the concentration of

vinyl chloride injected, the attenuator setting, chart speed, peak area, sample loop temperature, column temperature, carrier gas flow rate, and retention time. Record the laboratory pressure. Calculate A_s , the peak area multiplied by the attenuator setting. Repeat until two injection areas are within 5 percent, then plot these points v. C_s . When the other concentrations have been plotted, draw a smooth curve through the points. Perform calibration daily, or before and after each set of bag samples, whichever is more frequent.

7.4 Bag leak checks. While performance of this section is required subsequent to bag use, it is also advised that it be performed prior to bag use. After each use, make sure a bag did not develop leaks as follows: To leak check, connect a water manometer and pressurize the bag to 5-10 cm H_2O (2-4 in. H_2O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. Also check the rigid container for leaks in this manner.

(Note: An alternative leak check method is to pressurize the bag to 5-10 cm H_2O or 2-4 in. H_2O and allow to stand overnight. A deflated bag indicates a leak.) For each sample bag in its rigid container, place a rotameter in-line between the bag and the pump inlet. Evacuate the bag. Failure of the rotameter to register zero flow when the bag appears to be empty indicates a leak.

8. Calculations

8.1 Determine the sample peak area as follows:

$$A_s = A_m A_r$$

Equation 106-1

where:

A_s = The sample peak area.
 A_m = The measured peak area.
 A_r = The attenuation factor.

8.2 Vinyl chloride concentrations. From the calibration curve described in Section 7.3, above, select the value of C_s that corresponds to A_s , the sample peak area. Calculate C_b as follows:

$$C_b = \frac{C_s P_s T_s}{P_r T_r (1 - B_{w_s})}$$

Equation 106-2

Where:

B_{w_s} = The water vapor content of the bag sample, as analyzed.
 C_s = The concentration of vinyl chloride in the bag sample in ppm.
 C_b = The concentration of vinyl chloride indicated by the gas chromatograph, in ppm.
 P_s = The reference pressure, the laboratory pressure recorded during calibration, mm Hg.
 T_s = The sample loop temperature on the absolute scale at the time of analysis, °K.
 P_r = The laboratory pressure at time of analysis, mm Hg.
 T_r = The reference temperature, the sample loop temperature recorded during calibration, °K.

9. References

1. Brown, D. W., Loy, E. W. and Stephenson, M. H. "Vinyl Chloride Monitoring Near the B. F. Goodrich Chemical Company in Louisville, Kentucky." Region IV, U.S. Environmental Protection Agency, Surveillance and Analysis Division, Athens, Georgia, June 24, 1974.
2. "Evaluation of A Collection and Analytical Procedure for Vinyl Chloride in Air," by G. D. Clayton and Associates, December 13, 1974. EPA Contract No. 68-02-1408, Task Order No. 2, EPA Report on 75-VCL-1.
3. "Standardization of Stationary Source Emission Method for Vinyl Chloride," by Midwest Research Institute, 1976. EPA Contract No. 68-02-1098, Task Order No. 7.

electrical tape around the cap and bottle to prevent the top from loosening. Place an identifying label on each bottle, and record the date, time, and sample location both on the bottles and in a log book.

7.1.2 Water sampling—Prior to use, the 60 ml vials (without the discs) must be capped with aluminum foil and mudged at 400°C for at least one hour to destroy or remove any organic matter that could interfere with analysis. At the sampling location fill the vials bubble-free, to overflowing so that a convex meniscus forms at the top. The excess water is displaced as the sealing disc is carefully placed, Teflon side down, on the opening of the vial. Place the aluminum seal over the disc and the neck of the vial and crimp into place. Affix an identifying label on the bottle, and record the date, time, and sample location both on the vials and in a log book. All samples must be kept refrigerated until analyzed.

7.2 Sample recovery Samples must be run within 24 hours.

7.2.1 Resin samples—The weight of the resin used must be between 0.1 and 4.5 grams. An exact weight must be obtained (± 0.001 gram) for each sample. In the case of suspension resins a volumetric cup can be prepared which will hold the required amount of sample. The sample bottle is opened, and the cup volume of resin is added to the tared sample vial (including septum and aluminum cap). The vial is immediately sealed and the exact sample weight is then obtained. Report this value on the data sheet as it is required for calculation of RVC. In the case of relatively dry resin samples (water content < 0.3 weight %), 100 μ l of distilled water must be injected into the vial after sealing and weighing, using a 100 μ l syringe. In the case of dispersion resins, the cup cannot be used. The sample is instead weighed approximately in an aluminum dish, transferred to the tared vial and weighed accurately in the vial. The sample is then placed in the Perkin-Elmer head space analyzer (or equivalent) and conditioned for one hour at 90°C.

Note: Some aluminum vial caps have a center section which must be removed prior to placing into sample tray. If not removed, serious damage to the injection needle will occur.

7.2.2 Suspension resin slurry and wet cake samples—Slurry must be filtered using a small Buchner funnel with vacuum to yield wet cake. The filtering process must be continued only as long as a steady stream of water is exiting from the funnel. Excessive filtration time could result in some loss of VCM. The wet cake sample (0.10 to 4.5 grams) is added to a tared vial (including septum and aluminum cap) and immediately sealed. Sample weight is then determined to 3 decimal places. The sample is then placed in the Perkin-Elmer head space analyzer (or equivalent) and conditioned for one hour at 90°C. A sample of wet cake is used to determine TS (total solids). This is required for calculating the RVC.

7.2.3 Dispersion resin slurry samples—This material should not be filtered. Sample must be thoroughly mixed. Using a tared vial (including septum and aluminum cap) add approximately 8 drops (0.25 to 0.35 grams) of slurry or latex using a medicine dropper. This should be done immediately after mixing. Seal the vial as soon as possible. Determine sample weight accurate to 0.001 grams. Total sample weight must not exceed 0.50 grams. Condition the vial for one hour at 90°C in the analyzer. Determine the TS on the slurry sample (Section 7.3.6).

7.2.4 Inprocess wastewater samples—Using a tared vial (including septum and aluminum cap) quickly add approximately 1 cc of water using a medicine dropper. Seal the vial as soon as possible. Determine sample weight accurate to 0.001 gram. Condition the vial for two hours at 90°C in the

analyzer.

7.3 Analysis

7.3.1 Preparation of gas chromatograph—Install the chromatographic column and condition overnight at 180°C. Do not connect the exit end of the column to the detector while conditioning.

7.3.1.1 Flow rate adjustments—Adjust flow rates as follows:

a. Nitrogen carrier gas—Set regulator on cylinder to read 50 psig. Set regulator on chromatograph to 1.3 kg/cm². Normal flows at this pressure should be 25 to 40 cc/minute. Check with bubble flow meter.

b. Burner air supply—Set regulator on cylinder to read 50 psig. Set regulator on chromatograph to supply air to burner at a rate between 260 and 300 cc/minute. Check with bubble flow meter.

c. Hydrogen supply—Set regulator on cylinder to read 30 psig. Set regulator on chromatograph to supply approximately 35–5 cc/minute. Optimize hydrogen flow to yield the most sensitive detector response without extinguishing the flame. Check flow with bubble meter and record this flow.

7.3.1.2 Temperature adjustments—Set temperatures as follows:

a. Oven (chromatographic column), 50°C.

b. Dosing line, 140°C.

c. Injection block, 140°C.

d. Sample chamber, water temperature, 90°C $\pm 1.0^\circ$ C.

7.3.1.3 Ignition of flame ionization detector—Ignite the detector according to the manufacturer's instructions.

7.3.1.4 Amplifier balance—Balance the amplifier according to the manufacturer's instructions.

7.3.2 Programming the chromatograph—Program the chromatograph as follows:

a. I—Dosing time—The normal setting is 2 seconds.

b. A—Analysis time—The normal setting is 8 minutes. Certain types of samples contain high boiling materials which can cause interference with the vinyl chloride peak on subsequent analyses. In these cases the analysis time must be adjusted to eliminate the interference. An automated backflush system can also be used to solve this problem.

c. B—Flushing—The normal setting is 0.2 minutes.

d. W—Stabilization time. The normal setting is 0.2 minutes.

e. X—Number of analyses per sample—The normal setting is 1.

7.3.3 Preparation of sample turntable—Before placing any sample into turntable, be certain that the center section of the aluminum cap has been removed. The numbered sample bottles should be placed in the corresponding numbered positions in the turntable. Insert samples in the following order:

Positions 1 & 2—Old 2000 ppm standards for conditioning. These are necessary only after the analyzer has not been used for 24 hours or longer.

Position 3—50 ppm standard, freshly prepared.

Position 4—500 ppm standard, freshly prepared.

Position 5—2000 ppm standard, freshly prepared.

Position 6—4000 ppm standard, freshly prepared.

Position 7—Sample No. 7 (This is the first sample of the day, but is given as 7 to be consistent with the turntable and the integrator printout.)

After all samples have been positioned, insert the second set of 50, 500, 2000, and 4000 ppm standards. Samples, including standards, must be conditioned in the bath of 90°C for 1 hour (not to exceed 6 hours).

7.3.4 Start chromatograph program—When all samples, including standards, have been conditioned at 90°C for 1 hour, start

the analysis program according to the manufacturer's instructions. These instructions must be carefully followed when starting and stopping program to prevent damage to the dosing assembly.

7.3.6 Determination of total solids (TS)—For wet cake slurry, resin solution, and PVC latex samples, determine TS for each sample by accurately weighing approximately 3 to 4 grams of sample in an aluminum pan before and after placing in a draft oven (105 to 110°C). Samples must be dried to constant weight. After first weighing return the pan to the oven for a short period of time and then reweigh to verify complete dryness. TS is then calculated as the final sample weight divided by initial sample weight.

8 Calibration

Calibration is to be performed each eight-hour period when the instrument is used. Each day, prior to running samples, the column should be conditioned by running two of the previous days 2000 ppm standards.

8.1 Preparation of Standards

Calibration standards are prepared by filling the vials with the vinyl chloride/nitrogen standards, rapidly seating the septum and sealing with the aluminum cap. Use a stainless steel line from the cylinder to the vial. Do not use rubber or tygon tubing. The sample line from the cylinder must be purged (into hood) for several minutes prior to filling vials. After purging, reduce the flow rate to approximately 500–1000 cc/min. Place end of tubing into vial (near bottom) and after one minute slowly remove tubing. Place septum in vial as soon as possible to minimize mixing air with sample. After the standard vials are sealed, inject 100 μ l of distilled water.

8.2 Preparation of chromatograph calibration curve

Prepare two 50 ppm, two 500 ppm, two 2000 ppm, and two 4000 ppm standard samples. Run the calibration samples in exactly the same manner as regular samples. Plot A_i , the integrator area counts for each standard sample vs C_i , the concentration of vinyl chloride in each standard sample. Draw a line of best fit through the points.

9. Calculations

9.1 Response factor

From the calibration curve described in Section 8.2, above, select the value of C_i that corresponds to A_i for each sample. Compute the response factor, R_i , for each sample, as follows:

$$R_i = \frac{A_i}{C_i} \quad \text{Equation 107-1}$$

9.2 Residual vinyl chloride monomer concentration, or vinyl chloride monomer concentration

Calculate C_{res} as follows:

$$C_{res} = \frac{A_s P_s}{R_i T_i} \left(\frac{M_s V_s}{m_s R} + K T_i \right)$$

Equation 107-2

where:

C_{res} = Concentration of vinyl chloride in the sample, in ppm.

P_s = Laboratory atmosphere pressure, mm Hg.

T_i = Room temperature, °K.

M_s = Molecular weight of VCM (62.5).

V_s = Volume of vapor phase (vial volume less sample volume).

m_s = Weight of sample, grams.

R = Gas constant [62,360 (cc-mm-mole-degree Kelvin)].

K = Henry's Law constant. For VCM in PVC at 90°C,

$K = 6.52 \times 10^{-4} = K_p$. For VCM in 1 cc (approximate)

wastewater sample at 90°C,

$K = 5.0 \times 10^{-4} = K_w$.

$$C_{\text{res}} = \frac{A_s}{R_s} \left(4.197 \times 10^{-3} + \frac{5.988 \times 10^{-3}}{m_i} \right) \quad \text{Equation 107-3}$$

The following general equation can be used for any sample which contains VCM, PVC and water.

$$C_{\text{res}} = \frac{A_s P_s}{R_s T_s} \times \left[\frac{M_s V_s}{R m_i} + K_s (TS) T_s - K_s (1 - TS) T_s \right] \quad \text{Equation 107-4}$$

where.

TS = Total solids.

Note: K_s must be determined for samples with a vapor volume to liquid volume ratio other than 22.5 to 1. This ratio can be obtained by adjusting the sample weight through giving consideration to the total solids and density of the PVC.

Results calculated using Equation 107-4 represent concentration based on the total sample. To obtain results based on dry PVC content, divide by TS .

For a 1-cc wastewater sample (that is, 22.5 to 1 vapor volume to liquid volume ratio) K_s is 5.0×10^{-4} . Thus, Equation 107-4 can be simplified to the following:

$$C_{\text{res}} = \frac{A_s}{R_s} \left[\frac{5.988 \times 10^{-3}}{m_i} + (2.066 \times 10^{-3}) \right] \quad \text{Equation 107-5}$$

(Secs. 112 and 301(a) of the Clean Air Act, 42 U.S.C. 1857c-7 and 1857g(a).)

T_s = Equilibration temperature, °K.

If the following conditions are met, Equation 107-2 can be simplified as follows:

1. $T_s = 22^\circ \text{C}$ (295°K)
2. $T_i = 90^\circ \text{C}$ (363°K)
3. $P_s = 750 \text{ mm Hg}$.

$$4. V_s = V_i - \frac{m_i}{1.4} = 23.5 - \frac{m_i}{1.4}$$

where

V_s = Vial volume, cc (23.5).

5. Sample contains less than 0.5 percent water

10. References.

1. Residual Vinyl Chloride Monomer Content of Polyvinyl Chloride Resins and Wet Cake Samples, B. F. Goodrich Chemical Co. Standard Test Procedure No. 1005-T, B. F. Goodrich Technical Center, Avon Lake, Ohio, January 30, 1976.
2. Berens, A. E., "The Solubility of Vinyl Chloride in Polyvinyl Chloride," ACS-Division of Polymer Chemistry, Polymer Preprints 15 (2): 197, 1974.
3. Berens, A. E., "The Diffusion of Vinyl Chloride in Polyvinyl Chloride," ACS-Division of Polymer Chemistry, Polymer Preprints 15 (2): 203, 1974.
4. Berens, A. E., L. B. Crider, C. J. Tomanek and J. M. Whitney, Analysis for Vinyl Chloride in PVC Powders by Head-Space Gas Chromatography, to be published.

[FR Doc. 77-15828 Filed 6-6-77; 2:45 am]

TENNECO CHEMICALS, INC.

BURLINGTON, NEW JERSEY

MAJOR RAW MATERIALS

methanol
tridecanol
vinyl chloride
vinyl acetate
trichloroethylene
ammonia
food grade gelatin
liquid caustic
liquid chloride
ferric sulfate
lime
sulfuric acid

MATERIALS REPORTED ON SLUDGE QUALITY REPORT

phenol
toluene
trichloroethylene
mercury
thiostop-N
methylene chloride
tetrachloroethylene
di(2-ethylhexyl) phthalate

COLORITE 017048



DATE

To

58752 phoned
108583 phone
74216 truckload of hay
7439776 nursery
125041 livestock
7872 wellington school
127134 truckload of hay
117517 ~~27~~ 27 D2E(1)7

From the office of: J. P. SANDSTEDT



B

LIST OF TOXIC SUBSTANCES
FROM APPENDICES B-1, B-2, B-3

CAS NO'S	RATE	U.S.D.		PERSONAL PROTECTIVE EQUIPMENT	
		PROCESS	ANALYST	PROTECTIVE EQUIPMENT	PROTECTIVE EQUIPMENT
1. 7440350	<.5Lbs/yr.		X		X
2. 7440352	<.5Lbs/yr.		X		X
3. 7440352	<.5Lbs/yr.		X		X
4. 7440439	<.5Lbs/yr.		X		X
5. 56235	25Lbs/yr.		X		X
6. 67663	25Lbs/yr.		X		X
7. 7440473	<.5Lbs/yr.		X		X
8. 7440508	<.5Lbs/yr.		X		X
9. 57125	<.5Lbs/yr.		X		X
10. 75343	<.5Lbs/yr.		X		X
11. 75092	36Lbs/yr.		X		X
12. 117817	2460 Lbs/yr.		X		X
13. 85687	93Lbs/yr.		X		X
14. 122394	<.5Lbs/yr.		X		X
15. 7439921	<.5Lbs/yr.		X		X
7439921	15Lbs/yr.	X		X	
16. 7439976	<.5Lbs/yr.		X		X
17. 7440020	<.5Lbs/yr.		X		X
7440020	1800Lbs/yr.	X		X	
18. 108952	<.5Lbs/yr.		X		X
19. 7440224	<.5Lbs/yr.		X		X
20. 127184	2067Lbs/yr.		X		X
127184	18 gal/yr.	X		X	

B

LIST OF TOXIC SUBSTANCES
FROM APPENDICES B-1, B-2, B-3

CAS NO.	GTY'S	USDO		D. SPANAL		X
		PROS	AG. 1M	1000	1000	
21. 105781	200Lbs/yr.		X			X
22. 79095	<.5Lbs/yr.		X			X
79095	775,1007, 21	X		X		
23. 79016	12Lbs/yr.		X			X
24. 7440666	<.5Lbs/yr.		X			X
25. 7440473	<.5Lbs/yr.		X			X
7440473	500Lbs/yr.	X		X		
26. 128041	12Lbs/yr.		X			X

C

SELECTED LIST OF TOXIC SUBSTANCES
AND
THE PROCEDURES FOR TESTING

CAS NO	NAME	PROCEDURE FOR TESTING
7439976	Mercury (total)	Standard Methods, 20116 Fluorescence absorption
100812	PCP	EPA Method 604- gas chromatograph, solvent extraction (methylene chloride) followed by analysis with flame ionization detector.
105883	Toluene	EPA Method 602- gas chromatograph with Purge and Trap device and Flame ionization detector.
79016	Trichloroethylene	EPA Method 601- gas chromatograph with purge and trap device and Hall or Electron Capture Detector.
128041	Sodium dimethyl dithiocarbamate	No EPA method has been published for the analysis of this compound. We have requested information on the specific procedures required but have not yet received it.

*NOTE: The above EPA Methods for GC analysis were published in the Federal Register on December 3, 1979. (Vol. 44, No. 233)

APPENDIX B-1HEAVY METALS AND TOXIC ORGANIC COMPOUNDSContamination Indicators

<u>Heavy Metals</u>	<u>ppm</u>	<u>Toxic Organic Compounds</u>	<u>ppm</u>
Arsenic (total)	10	Aldrin	0.10
		Chlordane	0.10
Cadmium (total)	10	Dieldrin	0.10
Chromium (total)	890	Endrin	0.10
Copper (total)	850	Heptachlor	0.10
Lead (total)	500	Heptachlor epoxide	0.10
Mercury (total)	5	Lindane	0.10
Nickel (total)	82	Methoxychlor	0.25
Zinc (total)	17740	p,p' - DDE (DDD)	0.25
		Mirex	0.25
		p,p' - DDT	0.25
		p,p' - DDE	0.25
		Toxaphene	1.0
		Polychlorinated biphenyls (PCB's)	0.50

*dry-weight basis

E

TESTING PROCEDURES
FOR
WASTE WATER SLODGE
APPENDIX A-4

Sudge Quality Assurance Equations using the following procedures:

COD- EPA Method 451.3 in "Methods for Chemical Analysis of water and wastes" EPA, March 1979. (attached)

Oil & Grease- Standard Methods, 14 ed Method 502D (attached)

pH- pH meter (electrode)

Total Solids- Standard Methods, 14 ed. Method 2080 determination of total residue in solid & semi solid samples (attached)

TOXIC POLLUTANTS

* Chemical Abstracts Service Number

APPENDIX B-2 (continued)

	CAS No.	
	88733	2-chloronitrobenzene
	100005	4 - chloronitrobenzene
	25167800	2 - chlorophenol
	7005723	4 - chlorophenyl phenyl ether
	126998	chloroprene
1/2	7440473	chromium (total)
1/2	218019	chrysene
	532821	chrysolidine
1/2	7440503	copper (total)
	98828	cumene
1/2	57125	cyanide (total)
	72548	p,p' - DDD (p,p' - TDE)
	72559	p,p' - DDE (p,p' - DDX)
	50293	p,p' - DDT
	53703	1,2:5,6 - dibenzanthracene
	608275	2,3 - dichloroaniline
	554007	2,4 - dichloroaniline
	95829	2,5 - dichloroaniline
	95761	3,4 - dichloroaniline
	626437	3,5 - dichloroaniline
	95501	1,2 - dichlorobenzene
	541731	1,3 - dichlorobenzene
	106467	1,4 - dichlorobenzene
	91941	3,3' - dichlorobenzidine
	75274	dichlorobromomethane
1/2	75343	1,1 - dichloroethane
1/2	107062	1,2 - dichloroethane
	156592	1,2 - dichloroethylene (trans)
36	75092	dichloromethane (methylene chloride) L
	120832	2,4 - dichlorophenol
	78875	1,2 - dichloropropane
	542756	1,3 - dichloropropene
	60571	dieldrin
	84662	diethyl phthalate
1460	117817	di (2 - ethylhexyl) phthalate L
93	85687	butyl benzyl phthalate L
	131113	dimethyl phthalate
	84742	di - n - butyl phthalate
	117840	di - n - octyl phthalate
	119904	3,3' - dimethoxybenzidine
	121697	dimethylaniline
	119937	3,3' - dimethylbenzidine
	57147	1,1 - dimethylhydrazine
	105679	2,4 - dimethylphenol
	534521	4,6 - dinitro - o - cresol
	51285	2,4 - dinitrophenol
	121142	2,4 - dinitrotoluene

APPENDIX B-2 (continued)

<u>lbs/yr</u>	<u>CAS No.</u>	
	606202	2,6 - dinitrotoluene
	123911	1,4 - dioxane (1,4 - diethylene dioxide)
< 1/2	122394	diphenylamine
	122667	1,2 - diphenylhydrazine
	959988	endosulfan - I
	333-13659	endosulfan - II
	1031078	endosulfan sulfate
	72208	endrin
	7421934	endrin aldehyde
	53494705	endrin ketone
	100414	ethylbenzene
	106934	ethylene dibromide
	151564	ethyleneimine (Aziridine)
	206440	fluoranthene
	86737	fluorene
	76448	heptachlor
	1024573	heptachlor epoxide
	118741	hexachlorobenzene
	87683	hexachlorobutadiene
	77474	hexachlorocyclopentadiene
	67721	hexachloroethane
	302012	hydrazine
	193395	indeno (1,2,3-C,D) pyrene
	78591	isophorone
< 1/2	7439921	lead (total)
< 1/2	7439976	mercury (total)
	101144	4,4' - methylene bis (2-chloraniline)
	101779	4,4' - methylene dianiline
	108101	methylisobutyl ketone
	91203	naphthalene
	134327	alpha naphthylamine
< 1/2	91598	beta naphthylamine
	7440020	nickel (total)
	98953	nitrobenzene
	88755	2 - nitrophenol
	100027	4 - nitrophenol
	1321126	nitrotoluene
	100618	N - methylaniline
	62759	N - nitrosodimethylamine
	621647	N - nitrosodi - n - propylamine
	86306	N - nitrosodiphenylamine
	59507	parachlorometacresol
		PCB - 1016 (Aroclor 1016)
		PCB - 1221 (Aroclor 1221)
11097691		PCB - 1232 (Aroclor 1232)
		PCB - 1242 (Aroclor 1242)
		PCB - 1248 (Aroclor 1248)
		PCB - 1254 (Aroclor 1254)
		PCB - 1260 (Aroclor 1260)

APPENDIX B-2 (continued)

	CAS No.	
	87865	pentachlorophenol
	85018	phenanthrene
< 1/2	-108952	phenol
	95545	1,2 - phenylenediamine
	108452	1,3 - phenylenediamine
	106503	1,4 - phenylenediamine
	129000	pyrene
	7782492	selenium (total)
< 1/2	-7440224	silver (total)
	842079	sudan I (solvent yellow 14)
	1746016	2,3,7,8 - tetrachlorodibenzo-p-dioxin (TCDD)
	79345	1,1,2,2 - tetrachloroethane
2007	127184	tetrachloroethylene L
	7440280	thallium (total)
	62566	thiourea
2	-108883	toluene
	104154	toluene sulfonic acids
	26915128	toluidines
	8001352	toxaphene
	120821	1,2,4 - trichlorobenzene
< 1/2	-71556	1,1,1 - trichloroethane
< 1/2	-79005	1,1,2 - trichloroethane
< 1/2	-79016	trichloroethylene
	88062	2,4,6 - trichlorophenol
	1300716	xlenols
	1300738	xylidines
< 1/2	-7440666	zinc (total)

APPENDIX B-3

POLLUTANTS THAT ARE INHIBITORY TO
BIOLOGICAL TREATMENT PROCESSES

CAS No.

INORGANIC POLLUTANTS

100/1000
14213979 Borate (Boron)
7440473 Chromium
7439965 Manganese
7440622 Vanadium

ORGANIC POLLUTANTS

107186 Allyl alcohol
57067 Allyl isothiocyanate
141435 2-Aminoethanol (mono ethanolamine)
538283 Benzyl thiuronium chloride
4170303 Crotonaldehyde
97234 Dichlorophen
591355 3,5 - dichlorophenol
138896 Dimethylparanitrosoaniline
86566 N,N-dimethyl-1-naphthylamine
593851 Guanidine carbonate
124094 Hexamethylenediamine
148243 8 - hydroxyquinoline
556616 Methyl isothiocyanate
867447 Methyl thiuronium sulphate
107197 Propargyl alcohol
83341 Skatole
12 128041 x Sodium dimethyl dithiocarbamate - *Tram. N.A.*
137428 Sodium methyl dithiocarbamate
137268 Tetramethyl thiuram disulphide
97745 Tetramethyl thiuram monosulphide
79196 Thiosemicarbazide

475358-475364:Y:A:

APPENDIX B-2

TOXIC POLLUTANTS

CAS No.

83329	acetylcholine
208962	acetylnaphthylamine
107023	acetaldehyde
79051	acrylonitrile
107131	acrylonitrile
300332	aldrin
1321115	ammonium nitrate
61825	aniline
72125	aniline
62533	aniline
142041	aniline hydrochloride
100663	anisole
120127	anthracene
7440360	antimony (total)
7440382	arsenic (total)
2465272	auramine
56553	1,2 - benzanthracene
71432	benzene
92875	benzidine
205992	3,4 - benzofluoranthene
207089	11,12 - benzofluoranthene
191242	1,12 - benzoperylene
50328	3,4 - benzopyrene (benzo (a)pyrene)
98077	benzotrachloride
100469	benzylamine
7440417	beryllium (total)
319846	alpha BHC
319857	beta BHC
58899	gamma BHC (Lindane)
319868	delta BHC
111911	bis (2 - chloroethoxy) methane
111444	bis (2 - chloroethyl) ether
39638329	bis (2 - chloroisopropyl) ether
75252	bromoform (tribromomethane)
101553	4 - bromophenyl phenyl ether
7440439	cadmium (total)
56235	carbon tetrachloride (tetrachloromethane)
57749	chlordane (technical mixture and metabolites)
95512	2 - chloroaniline
108429	3 - chloroaniline
106478	4 - chloroaniline
108907	chlorobenzene
124461	chlorodibromomethane
110753	2 - chloroethyl vinyl ether (mixed)
67663	chloroform (trichloromethane)
91587	2 - chloronaphthalene

* Chemical Abstracts Service Number

RAW MATERIALS LIST

Process Related

AC, Celvatol 2090
Methocel, 15 cps 65 hg
Methocel, 50 cps 65 hg
Tributyl Phosphate
Lauroyl Peroxide
Sodium Bicarbonate
— Ionol
40% DOW-80
Avisol POL-1-S
Ninol 1281
Colloids 581-B
AF-60
Trisodium Phosphate
Sodium Carbonate
Tween 20
Tridecanol
Vinyl Chloride ()
Vinyl Acetate
Trichloroethylene — TCE
TATC
Epoxol 9-5
Sodium Bisulfite
Layrsol 223M
Ammonium Persulfate
Anhydrous Ammonia
Methocel, 100 cps 90 hg
Hormel Gelatin Blend
Colloids 991
Sodium Acetate
Aqueous Ammonia
Phenol (10% in MeOH) —
Shell Sol 71

Operating Supplies

Glycerine
Nitrogen
Thiostop N
Air, breathing

Boiler House & Effluent Plant

Phosphoric Acid 75%
Aqueous Ammonia 28%
Degreaser 701
#6 Fuel Oil 0.3% sulfur max.
#4 Fuel Oil
Muriatic Acid (128 lbs/13 gal)
Caustic Liquid
Liquid Chlorine
Ferric Sulfate
Calcium Hypochlorite, granular (65-70%)
Hydrated Lime, low mg
Solar Salt (rock)
Sink Flocc AZ-6
Sulfuric Acid 66%
Octafilm
#2 Fuel Oil
Polyfloc 1120
Defoamer 300-66
Liqui-Treat CL-2247
Betz Slimicide C-30
Betz Fuel-Solv FS-20
Betz Fuel-Solv FS-534
Pulverized Quick Lime
Corrogen
Ferrous Sulfate
Arco Chem RW-877

Miscellaneous

Ethylene Glycol
Methanol
Acetone
Solvesso 100
THF
Calcium Chloride
Sand
Perchloroethylene
Toluene

R.A. Iojewski
4/12/79