ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 61

[AD-FRL 1643-6]

National Emission Standards for Hazardous Air Pollutants; Alternative Test Method 107A (Vinyl Chloride)

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed rule and notice of public hearing.

SUMMARY: The proposed method would apply to the measurement of the vinyl chloride content of solvents, resin-solvent solution, polyvinyl chloride resin, resin-slurry, wet resin, and latex samples. The proposed method was derived from a test method submitted to EPA by Union Carbide. The intent of this proposed method is to provide this alternative analytical procedure because it may be preferred over Method 107 in some circumstances.

A public hearing will be held to provide interested persons an opportunity for oral presentation of data views, or arguments concerning the proposed method.

DATES: *Comments.* Comments must be received on or before April 13, 1981.

Public Hearing. A public hearing will be held on March 26, 1981 (about 30 days after proposal) beginning at 9 a.m.

Request to Speak at Hearing. Persons wishing to present oral testimony must contact EPA by March 19 (1 week before hearing).

ADDRESSES: *Comments.* Comments should be submitted (in duplicate if possible) to: Central Docket Section (A-130), Attention: Docket A-80-37, U.S. Environmental Protection Agency, 401 M Street, SW., Washington, D.C. 20460.

Public Hearing. The public hearing will be held at Emissions Measurement Laboratory Building, Page Road and Interstate 40, R.T.P. North Carolina 27711. Persons wishing to present oral testimony should notify Ms. Deanna Tilley, Standards Development Branch (MD-13), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone number (919) 541-5421.

Docket. Docket No. A-80-37, containing material relevant to this rulemaking, is available for public inspection and copying between 8:00 a.m. and 4:00 p.m., Monday through Friday, at EPA's Central Docket Section, West Tower Lobby, Gallery 1, Waterside Mall, 401 M Street, SW., Washington, D.C. 20460. A reasonable fee may be charged for copying.

FOR FURTHER INFORMATION CONTACT: Mr. Roger T. Shigehara (MD-19), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone number (919) 541-2237.

SUPPLEMENTARY INFORMATION: On October 21, 1976 (41 FR 46560) and on June 7, 1977 (42 FR 29005) the Environmental Protection Agency promulgated Method 107—Determination of Vinyl Chloride Content of Inprocess Wastewater Samples and Vinyl Chloride Content of Polyvinyl Chloride Resin, Slurry, Wet Cake, and Latex Samples. Since that time, Union Carbide has submitted comparative supporting data to EPA on a test method that embodies a less sophisticated, but also technically satisfactory, analytical approach. Because practical considerations would favor its use in some instances, this alternative method is being considered for adoption as an EPA method. If adopted, this alternative test method would be available to determine compliance with the national emission standard for vinyl chloride, 40 CFR, Part 61 Subpart F.

(Secs. 112, 114, and 301(a) of the Clean Air Act as amended (42 U.S.C. 7412, 7414, and 760.(a)))

Dated: February 4, 1981.

Walter C. Barber,

Acting Administrator.

It is proposed to amend 40 CFR Part 61 by adding Method 107A to Appendix B as follows:

Appendix B—Test Methods

* * * * *

Method 107A—Determination of Vinyl Chloride Content of Solvents, Resin-Solvent Solution, Polyvinyl Chloride Resin, Resin Slurry, Wet Resin, and Latex Samples[1]

Introduction

Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph or by those who are unfamiliar with source sampling because knowledge beyond the scope of this presentation is required. Care must be exercised to prevent exposure of sampling personnel to vinyl chloride, a carcinogen.

1. *Applicability and Principle.*

1.1 Applicability. This is an alternative method and applies to the measurement of the vinyl chloride content of solvents, resin solvent solutions, PVC resin, wet cake slurries, latex, and fabricated resin samples. This method is not acceptable where methods from Section 304(h) of the Clean Water Act, 33 U.S.C. 1251 et seq. (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977) are required.

1.2 Principle. The basis for this method lies in the direct injection of a liquid sample into a chromatograph and the subsequent evaporation of all volatile material into the carrier gas stream of the chromatograph, thus permitting analysis of all volatile material including vinyl chloride.

2. *Range and Sensitivity.*

The lower limit of detection of vinyl chloride in dry PVC resin is 0.2 ppm. For resin solutions, latexes, and wet resin, this limit rises inversely as the nonvolatile (resin) content decreases.

With proper calibration the upper limit may be extended as needed.

3. *Interferences.*

The chromatograph columns and the corresponding operating parameters herein described normally provide an adequate resolution of vinyl chloride. In cases where resolution interferences are encountered, the chromatograph operator shall select the column and operating parameters best suited to his particular analysis problem, subject to the approval of the Administrator. Approval is automatic, provided that the tester produces confirming data through an adequate supplemental analytical technique, such as analysis with a different column or GC/mass spectroscopy, and has the data available for review by the Administrator.

4. *Precision and Reproducibility.*

A standard sample of latex containing 181.8 ppm vinyl chloride analyzed 10 times by the alternative method showed a standard deviation of 7.5 percent and a mean error of 0.21 percent.

A sample of vinyl chloride copolymer resin solution was analyzed 10 times by the alternative method and showed a standard deviation of 6.6 percent at a level of 35 ppm.

5. *Safety.*

Do not release vinyl chloride to the laboratory atmosphere during preparation of standards. Venting or purging with vinyl chloride monomer (VCM) air mixtures must be held to minimum. When purging is required, the vapor must be routed to outside air. Vinyl chloride, even at low-ppm levels, must never be vented inside the laboratory.

6. *Apparatus.*

6.1 Sampling. The following equipment is required:

6.1.1 Glass Bottles, 16-oz wide mouth with polyethylene-lined, screw-on tops.

[1] Mention of trade names or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.

6.1.2 Adhesive Tape. To prevent loosening of bottle tops.

6.2 Sample Recovery. The following equipment is required:

6.2.1 Glass Vials. 20-ml capacity with polycone screw caps.

6.2.2 Analytical Balance. Capable of weighing to ±0.01 gram.

6.2.3 Syringe. 50-microliter size, with removable needle.

6.2.4 Fritted Glass Sparger. Fine porosity.

6.2.5 Aluminum Weighing Dishes.

6.2.6 Sample Roller or Shaker. To help dissolve sample.

6.3 Analysis. The following equipment is required:

6.3.1 Gas Chromatograph. Hewlett Packard Model 5720A or equivalent.

6.3.2 Chromatograph Column. Stainless steel, 6.1 m by 3.2 mm, packed with 20 percent Tergitol E-35 on Chromosorb W AW 60/80 mesh. The analyst may use other columns provided that the precision and accuracy of the analysis of vinyl chloride standards are not impaired and that he has available for review information confirming that there is adequate resolution of the vinyl chloride peak. (Adequate resolution is defined as an area overlap of not more than 10 percent of the vinyl chloride peak by an interferent peak. Calculation of area overlap is explained in Appendix C, Supplement A: "Determination of Adequate Chromatographic Peak Resolution.")

6.3.3 Valco Instrument Six-Port Rotary Valve. For column back flush.

6.3.4 Septa. For chromatograph injection port.

6.3.5 Injection Port Liners. For chromatograph used.

6.3.6 Regulators. For required gas cylinders.

6.3.7 Soap Film Flow Meter. Hewlett Packard No. 0101-0113 or equivalent.

6.4 Calibration. The following equipment is required:

6.4.1 Analytical Balance. Capable of weighing to ±0.0001 g.

6.4.2 Erlenmeyer Flask With Glass Stopper. 125 ml.

6.4.3 Pipets. 0.1, 0.5, 1, 5, 10, and 50 ml.

6.4.4 Volumetric Flasks. 10 and 100 ml.

7. *Reagents.*

Use only reagents that are of chromatograph grade.

7.1 Analysis. The following items are required:

7.1.1 Hydrogen Gas. Zero grade.

7.1.2 Nitrogen Gas. Zero grade.

7.1.3 Air. Zero grade.

7.1.4 Tetrahydrofuran (THF). Reagent grade.

Analyze the THF by injecting 10 microliters into the prepared gas chromatograph. Compare the THF chromatogram with that shown in Figure 107A-1. If the chromatogram is comparable to A, the THF should be sparged with pure nitrogen for approximately 2 hours using the fritted glass sparger to attempt to remove the interfering peak. Reanalyze the sparged THF to determine whether the THF is acceptable for use. If the scan is comparable to B, the THF should be acceptable for use in the analysis.

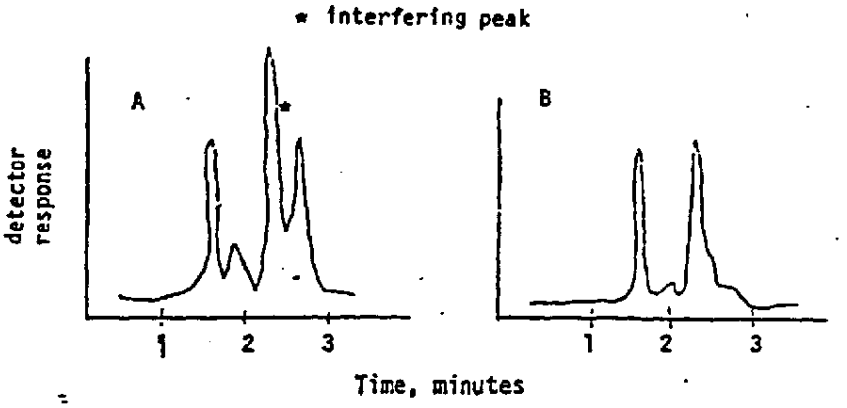

Figure 107A-1

7.1.5 N, N-Dimethylacetamide (DMAC). Spectrographic grade. For use in place in THF.

7.2 Calibration. The following item is required:

7.2.1 Vinyl Chloride 99.9 Percent. Ideal Gas Products lecture bottle, or equivalent. For preparation of standard solutions.

8. *Procedure.*

8.1 Sampling. Allow the liquid or dried resin to flow from a tap on the tank, silo, or pipeline until the tap has been purged. Fill a wide-mouth pint bottle, and immediately tightly cap the bottle. Place an identifying label on each bottle and record the date, time, sample location, and material.

8.2 Sample Treatment. Samples must be run within 24 hours.

8.2.1 Resin Samples. Weight 9.00 ± 0.01 g of THF or DMAC in a tared 20-ml vial. Add 1.00 ± 0.01 g or resin to the tared vial containing the THF or DMAC. Close the vial tightly with the screw cap, and shake or otherwise agitate the vial until complete solution of the resin is obtained. Shaking may require several minutes to several hours, depending on the nature of the resin.

8.2.2 Suspension Resin Slurry and Wet Resin Samples. Slurry must be filtered using a small Buchner funnel with vacuum to yield a wet resin sample. 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 resin sample is weighed into a tared 20-ml vial with THF or DMAC as described earlier for resin samples (8.2.1) and treated the same as the resin sample. A sample of the wet resin is used to determine total solids as required for calculating the RVCM (Section 8.3.4).

8.2.3 Latex and Resin Solvent Solutions. Samples must be thoroughly mixed. Weigh 1.00 ± 0.01 g of the latex or resin-solvent solution into a 20-ml vial containing 9.00 ± 0.01 g of THF or DMAC as for the resin samples (8.2.1). Cap and shake until complete solution is obtained. Determine the total solids of the latex or resin solution sample (Section 8.3.4).

8.2.4 Solvents and Non-viscous Liquid Samples. No preparation of these samples is required. The neat samples are injected directly into the gas chromatograph.

8.3 Analysis.

8.3.1 Preparation of Gas Chromatograph. Install the chromatographic column, and condition overnight at 70°C. Do not connect the exit end of the column to the detector while conditioning.

8.3.1.1 Flow Rate Adjustments. Adjust the flow rates as follows:

a. Nitrogen Carrier Gas. Set regulator on cylinder to read 60 psig. Set column flow controller on the chromatograph using the soap film flow meter to yield a flow rate of 40 cc/min.

b. Burner Air Supply. Set regulator on the cylinder at 40 psig. Set regulator on the chromatograph to supply air to the burner to yield a flow rate of 250 to 300 cc/min using the flow meter.

c. Hydrogen. Set regulator on cylinder to read 60 psig. Set regulator on the chromatograph to supply 30 to 40 cc/min using the flow meter. Optimize hydrogen flow to yield the most sensitive detector response without extinguishing the

flame. Check flow with flow meter and record this flow.

d. Nitrogen Back Flush Gas. Set regulator on the chromatograph using the soap film flow meter to yield a flow rate of 40 cc/min.

8.3.1.2 Temperature Adjustments. Set temperature as follows:

a. Oven (chromatographic column) at 70°C.

b. Injection Port at 100°C.

c. Detector at 300°C.

8.3.1.3 Ignition of Flame Ionization Detector. Ignite the detector according to the manufacturer's instructions. Allow system to stabilize approximately 1 hour.

8.3.1.4 Recorder. Set pen at zero and start chart drive.

8.3.1.5 Attenuation. Set attenuation to yield desired peak height depending on sample VCM content.

8.3.2. Chromatographic Analyses.

a. Sample Injection. Remove needle from 50-microliter syringe. Open sample vial and draw 50-microliters of THF or DMAC sample recovery solution into the syringe. Recap sample vial. Attach needle to the syringe and while holding the syringe vertically (needle uppermost), eject 40 microliters into an absorbent tissue. Wipe needle with tissue. Now inject 10 microliters into chromatograph system. Repeat the injection until two consecutive values for the height of the vinyl chloride peak do not vary more than 5 percent. Use the average value for these two peak heights to compute the sample concentration.

b. Back Flush. After 4 minutes has elapsed after sample injection, actuate the back flush valve to purge the first 4 feet of the chromatographic column of solvent and other high boilers.

c. Sample Data. Record on the chromatograph strip chart the data from the sample label.

d. Elution Time. Vinyl chloride elutes at 2.8 minutes. Acetaldehyde elutes at 3.7 minutes. Analysis is considered complete when chart pen becomes stable. After 5 minutes, reset back flush valve and inject next sample.

8.3.3 Chromatograph Servicing.

a. Septum. Replace after five sample injections.

b. Sample Port Liner. Replace the sample port liner with a clean spare after five sample injections.

c. Chromatograph Shut Down. If the chromatograph has been shut down overnight, rerun one or more samples from the preceding day to test stability and precision prior to starting on the current day's work.

8.3.4 Determination of Total Solids (T.S.). For wet resin, resin solution, and PVC latex samples, determine the T.S. for each sample by accurately weighing approximately 3 to 5 grams of sample into a tared aluminum pan. The initial procedure is as follows:

a. Where water is the major volatile component: Tare the weighing dish, and add 3 to 5 grams of sample to the dish. Weigh to the nearest milligram.

b. Where volatile solvent is the major volatile component: Transfer a portion of the sample to a 20-ml screw cap vial and cap immediately. Weigh the vial to the nearest milligram. Uncap the vial and transfer a 3- to 5-gram portion of the sample to a tared aluminum weighing dish. Recap the vial and reweigh to the nearest milligram. The vial weight loss is the sample weight.

To continue, now place the weighing pan in a 130°C oven for 1 hour. Remove the dish and allow to cool to room temperature in a desiccator. Weigh the pan to the nearest 0.1 mg. Total solids is the weight of material in the aluminum pan after heating divided by the net weight of sample added to the pan originally times 100.

9. *Calibration of the Chromatograph.*

9.1 Preparation of Standards. Prepare a 1 percent by weight (approximate) solution of vinyl chloride in THF or DMAC by bubbling vinyl chloride gas from a cylinder into a tared 125-ml glass-stoppered flask containing THF or DMAC. The weight of vinyl chloride to be added should be calculated prior to this operation, i.e., 1 percent of the weight of THF or DMAC contained in the tared flask. This must be carried out in a laboratory hood. Adjust the vinyl chloride flow from the cylinder so that the vinyl chloride dissolves essentially completely in the THF or DMAC and is not blown to the atmosphere. Take particular care not to volatize any of the solution. Stopper the flask and swirl the solution to effect complete mixing. Weigh the stoppered flask to nearest 0.1 mg to determine the exact amount of vinyl chloride added.

Pipet 10 ml of the approximately 1 percent solution into a 100-ml glass-stoppered volumetric flask, and add THF or DMAC to fill to the mark. Cap the flask and invert 10 to 20 times. This solution contains approximately 1,000 ppm by weight of vinyl chloride (note the exact concentration).

Pipet 50-, 10-, 5-, 1-, 0.5-, and 0.1-ml aliquots of the approximately 1,000 ppm solution into 100 ml glass stoppered volumetric flasks. Dilute to the mark with THF or DMAC, cap the flasks and invert each 10 to 20 times. These solutions contain approximately 500, 100, 50, 10, 5, and 1 ppm vinyl chloride. Note the exact concentration of each one. These standards are to be kept under refrigeration in stoppered bottles, and must be renewed every 3 months.

9.2 Preparation of Chromatograph Calibration Curve.

Obtain the gas chromatograph for each of the six final solutions prepared in Section 9.1 by using the procedure in Section 8.3.2. Prepare a chart plotting peak height obtained from the chromatogram of each solution versus the known concentration. Draw a straight line through the points derived by the least squares method.

10. *Calculations.*

10.1 Response Factor. From the calibration curve described in Section 9.2, select the value of C_c that corresponds to H_c for each sample. Compute the response factor, R_f, for each sample as follows:

$$R_f = \frac{C_c}{H_c} \qquad \text{Eq. 107A-1}$$

10.2 Residual vinyl chloride monomer concentration (C_{rvc}) or vinyl chloride monomer concentration in resin:

$$C_{rvc} = 10 H_s R_f \qquad \text{Eq. 107A-2}$$

Where:

H_s = Peak height of sample, mm.

R_f = Chromatograph response factor.

10.3 Samples containing volatile material, i.e., resin solutions, wet resin, and latexes:

$$C_{rvc} = \frac{H_s R_f (1{,}000)}{T.S.} \qquad \text{Eq. 107A-3}$$

10.4 Samples of solvents and inprocess waste water:

$$C_{vc} = \frac{H_s R_f}{0.888} \qquad \text{Eq. 107A-4}$$

Where:

0.888 = Specific gravity of THF.

11. *Bibliography.*

1. Communication from R.N. Wheeler, Jr.; Union Carbide Corporation. Part 61 National Emissions Standards for Hazardous Air Pollutants Appendix B, Method 107—Alternate Method, September 19, 1977.

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the overall emission level from that existing plant would be reduced.

Emission Offset

Because the present vinyl chloride standard focuses on reducing emissions rather than attaining a particular ambient air quality concentration, there is no provision for limiting the size of plants or the clustering of plants in a geographical area. The doubling of the size of an existing plant or the construction of a new plant beside an existing plant would considerably increase the ambient air concentrations of vinyl chloride in the vicinity of the plant(s) even if the vinyl chloride standard was met. EPA determined at the time of promulgation of the current standard that the costs of prohibiting the production of vinyl chloride and polyvinyl chloride were too high and the continued operation of existing plants should be allowed. EPA believes, however, that the standard should include a mechanism for prohibiting an increase in ambient concentrations of vinyl chloride due to new construction in areas where existing sources are already located.

Accordingly, EPA is proposing an amendment which would prohibit an increase in emissions within 8 kilometers (km) (approximately five miles) of an existing source due to the construction of a new emission source. This means that if a new source were added to an existing plant, the increase in emissions due to that new source would have to be offset by a reduction in emissions from other existing sources within that plant or at other plants within 8 km of the construction site of the new source. Similarly, a new plant could not be constructed within 8 km of an existing plant(s) unless the emission increase due to the new plant were offset by an emission reduction at the existing plant or plants. This provision may result in few existing plants being expanded and few new plants being constructed in the vicinity of existing plants. However, the proposed amendment does not preclude this possibility.

The offset provision would apply only to new construction which results in an increase in production rate. Replacing or adding equipment such as pumps, compressors, agitators, sampling equipment and unloading hoses is a routine practice at existing plants. Additions of equipment of this nature would, in and of itself, be expected to result in little, if any, increase in emissions. In EPA's judgment, a plant should not be required to prove this fact each time one of these pieces of equipment is added. The addition of this type of equipment in conjunction with major process equipment, however, is likely to result in both an increase in emissions as well as an increase in production rate, and is therefore covered by the offset provision.

If the offset provision were adopted, the reduction in emissions could be achieved in the production rate of an existing source or sources. The baseline emission rate would be determined based on the maximum production rate which had been attained by each existing source. The allowable emission rate for each source would be based on the maximum production rate at which that source would be operated in the future.

Also, if the emissions from an existing source were already below the emission limit applicable to it, the proposed amendment would give the source credit for the difference between the emission limit and the actual emission level. That is the baseline emission rate would be based on the standard rather than on an emission test. It is EPA's judgment that this is a more equitable approach than penalizing a source which has already taken measures to reduce emissions below the standard. Such a source would have less room for further reducing emissions.

The emission limits applicable to both the existing and new sources involved in the offset arrangement would be contained in the approval of new construction granted by the Administrator under 40 CFR 61.08.

EPA believes that a policy of no net increase in emissions due to new construction is justified because of the hazardous nature of vinyl chloride. However, EPA recognizes the potential difficulties in implementing such a policy and interested persons are urged to submit comments and factual information relating to this policy.

Review of Standard

EPA plans to undertake a full-scale review of Subpart F of 40 CFR Part 61 beginning three years from the promulgation of any amendments. In the study EPA will review information concerning technological advances in the control of vinyl chloride emissions to determine what further changes might then be appropriate to move toward the goal of zero vinyl chloride emissions. EPA will also consider recent health data to determine whether the approach for regulating vinyl chloride should be altered.

Environmental Impact

The proposed amendment, in contrast to the current standard, would encourage the development of new technology and improvements in existing technology and would have the following three positive environmental impacts: (1) further reduction of emissions at existing plants, (2) no increase in emissions within 8 km of an existing source, and (3) lower emissions from new sources than would be accomplished through the current standard regardless of the construction site. These environmental impacts would provide progress toward the ultimate goal of zero emissions without banning vinyl chloride, and in the process would provide additional protection of public health by further minimizing the health risks to the people living in the vicinity of existing plants and to any additional people who are exposed as a result of new construction.

Specifically, for those existing sources which are currently subject to a 10 ppm emission limit, emissions would be reduced by half within three years after the promulgation date of these amendments. At both an existing average-sized ethylene dichloride-vinyl chloride plant and an existing average-sized polyvinyl chloride plant, which contain other sources than the ones required to meet a 5 ppm emission limit, it is estimated this will have the effect of reducing total emissions by less than one percent. Emissions at existing plants would be further reduced as existing oxychlorination reactors are replaced with new oxychlorination reactors and as new polyvinyl chloride resins are produced to replace existing ones.

Under the proposed amendment, emissions from new plants would be considerably lower than they would be under the current standard. For a typical new average-sized ethylene dichloride-vinyl chloride plant (318×10^6 kg/yr or 700×10^6 lb/yr produced), the hourly emissions would be 5.1 kg (11.5 lb) instead of 10.3 kg (23.1 lb). For a typical new average-sized dispersion polyvinyl chloride plant (46×10^6 kg/yr or 100×10^6 lb/yr production), the emissions would be about 9 kg/hr (20 lb/hr) instead of 17.5 kg/hr (39 lb/hr) and for a typical new average-sized suspension polyvinyl chloride (68×10^6 kg/yr or 150×10^6 lb/yr production) the emissions would be 13.5 kg/hr (30 lb/hr) instead of 16 kg/hr (36 lb/hr). These emissions are calculated based on the emission factors published in the documentation for the existing standard. (*1*) Ambient air concentrations are expected to be reduced proportionately.

The only negative environmental impact would be an increase in hydrogen chloride emissions at ethylene dichloride-vinyl chloride plants if incineration were used to control emissions from new oxychlorination reactors. However, due to the corrosion problems which would otherwise occur on plant property and in the community, plants are expected to use scrubbers to control the hydrogen chloride emissions. The proposed amendment is not expected to have a significant impact on energy consumption.

Economic Impact

The potential economic impacts of the proposed standard are:

(1) Costs for research and development of improved methodology for operation of existing control technology so that it can be used to meet the 5 ppm emission limit.

(2) Costs for research and development of improved stripping techniques to meet the standard for new polyvinyl chloride resins.

(3) Cost of research and development or licensing for converting over to the oxygen system for a new oxychlorination reactor.

(4) Possibly increased transportation costs of raw materials in the case that the offset policy results in the construction of a new plant farther from an existing plant than it otherwise would have been.

(5) Costs of building a new plant more than 8 km from an existing plant in the event that the offset requirement precluded the expansion of an existing plant.

Group D

Liquid organic compounds including organic acids but excluding organic bases. (e.g. acetone, xylene, butyric acids.) Note: Peroxide forming materials must be stabilized prior to shipment.

Group E

Inorganic oxiding agents (e.g. bromine, chromerge, potassium & sodium perchlorates, chlorates, chlorites, dichromates, hypochlorites permanganate, persulfates, etc.)

Group F

Solid pesticides, insecticides, fungicides, etc. Note: Each pesticide & insecticide PLC will have to be reviewed on an individual basis.

Group G

Known and suspect carcinogenic materials.

Note: In case of a compound which might fit into two(2) different groups put it into the group based on the part of the molecule of higher toxicity. For example, lead acetate, lead would be Group B, the acetate portion is solid organic, Group C. It would go in Group B.

Group H

Waste oils,including oil from vacuum pumps, oil baths, mineral oils, lubricating oils, etc. all with flash points at or above 100°F.

EXCEPTIONS:

The following exceptions will not be allowed,(cannot be disposed of by this Section VII-A.)

1) Shock sensitive materials (e.g. mercury fulminate, sodium azide, azide, azo-compounds, perchloric acid etc.)

4. MEASUREMENT TECHNIQUES

4.1. ENVIRONMENTAL AIR

In selecting methods suitable for measuring VCM in ambient air, two factors must be considered. The method employed must be capable of measuring in the part per million to the part per billion range, and, because emissions are discontinuous, the method must be capable of responding to high concentration peaks as well as low level backgrounds.

4.1.1 Spectrophotometry

To design or describe a useful measurement technique or analytical method for a particular purpose requires that three major criteria be satisfied: sensitivity, accuracy and specifity. In addition practicality and economics are important considerations in the development of new analytical methods. As a general rule, it is most desirable to measure a pollutant or chemical specie directly in the matrix or phase--gas, liquid or solid--in which the material is generally encountered. This rule precludes any loss or transformation of the analyte to a non-detectable form. Whenever possible, in the following descriptions of analytical techniques, the above criteria will be addressed.

VCM absorbs infra red (IR) radiation in the gas phase. The absorption bands at 941 or 917 cm^{-1} have been commonly used to quantify VC. However, the method is not entirely specific for VCM as interfering substances (Table 4.1) are encountered in ambient air.[1] Multiband measurement and data processing techniques are available to correct for these interferences, but additional instrumentation is required. The Fourier transformation system is an excellent example of a refinement in this technique. The cost, however, of this type of system would be prohibitive for routine

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Grab samples are expected to lose quantities of VCM due to continuing reaction in the sampling container, wall loses, leaks, etc. Indication of VCM losses in these containers range from 0 to 10 percent per day. Losses of VCM while using charcoal are expected to be larger than in the containers. Collection efficiency and recoveries have not been definitively established. Preliminary data indicate recoveries of 71 to 76 percent when extraction by CS_2 is used to recover the VCM from charcoal. For 7 data points in which samples were collected in parallel and analyzed by two different laboratories, the values disagreed markedly. The relative standard deviation about the mean ranged from 5 to 140 percent.