

2 LAR File: Vinyl Chloride

Subpart W—Massachusetts

§ 52.1147 [Amended]

5. In § 52.1147-2 (5), subparagraph (a) is amended by revising the words "an average annual throughput of 5,000,000" to read "a daily throughput of 20,000".

Subpart FF—New Jersey

§ 52.1598 [Amended]

6. In § 52.1598-2 (3), subparagraph (a) is amended by revising the words "an average annual throughput of 5,000,000" to read "a daily throughput of 20,000".

Subpart NN—Pennsylvania

§ 52.3112 [Amended]

7. In § 52.3112-2 (3), subparagraph (a) is amended by revising the words "an average annual throughput of 5,000,000" to read "a daily throughput of 20,000".

Subpart SS—Texas

§ 52.3213 [Amended]

8. In § 52.3213-2 (3), subparagraph (a) is amended by revising the words "an average annual throughput of 5,000,000" to read "a daily throughput of 20,000".

Subpart VV—Virginia

9. In § 52.4513-2 (3), subparagraph (a) is amended by adding (iv), as follows:

§ 52.4513-2 (3)(iv) Gasoline transfer vapor control.

- (c)
- (3)

(iv) Facilities which have a daily throughput of 20,000 gallons or more of gasoline and which are engaged in the storage, distribution, or use of gasoline, and which are subject to the requirements of the Federal Clean Air Act, as amended, and the Federal Clean Air Act Regulations, as amended, shall be required to have a vapor recovery system in operation not later than May 31, 1977, and to be required to meet the provisions of this section not later than May 31, 1977.

A. General Vapor Recovery System. Every facility subject to a Federal Clean Air Act.

The Administrator announces the availability of and summarizes studies conducted by EPA regarding the availability of existing types of control technology for small bulk plants in compliance with the Federal Clean Air Act Regulations, as amended, as follows:

EPA initiated several studies in 1970 to determine if the requirements for bottom gasoline vapor recovery equipment would place severe economic burdens on the industry and whether the 90 percent control level was achievable by a vapor balance system, and to further determine the availability of control equipment which would satisfy the requirements of the existing regulations. Pacific Environmental Services, Inc., of Santa Monica, California, was chosen by EPA to perform these studies.

BACKGROUND

In 1970 and 1971, the Administrator promulgated Stage I Vapor Recovery regulations for 13 Air Quality Control Regions (AQCRs). The AQCRs, Federal Register references, and promulgation dates are listed below:

Metropolitan Los Angeles AQCR, 38 FR 31251, November 12, 1973
 Sacramento Valley AQCR, 38 FR 31251, November 12, 1973
 San Joaquin Valley Interstate AQCR, 38 FR 31251, November 12, 1973
 Metropolitan Denver Interstate AQCR, 38 FR 30822, November 7, 1973
 Marion County, Indiana, 38 FR 12349, April 5, 1974
 Maryland portion of National Capital Interstate AQCR, 38 FR 33719, December 6, 1973
 Metropolitan Baltimore Interstate AQCR, 38 FR 34253, December 12, 1973
 Boston Interstate AQCR, 38 FR 20033, November 3, 1973
 New Jersey portion of New Jersey-New York-Connecticut Interstate AQCR, 38 FR 31339, November 13, 1973
 New Jersey portion of Metropolitan Philadelphia Interstate AQCR, 38 FR 31339, November 13, 1973
 Allegheny County portion of Southern Tier-Tri-State AQCR, 38 FR 22205, November 22, 1973
 Houston-Galveston Interstate AQCR, 38 FR 32022, November 26, 1973
 San Francisco Bay Area AQCR, 38 FR 31540, November 26, 1973
 Virginia portion of National Capital Interstate AQCR, 38 FR 33719, December 6, 1973

The first study (EPA contract 68-01-3154, Task No. 5) evaluated the applicability and cost-effectiveness of bottom-loading vapor balance systems on small bulk plants. The second study (EPA contract 68-01-3154, Task No. 17) surveyed small bulk plants in and around Denver, San Diego, and the San Joaquin Valley, and included the source testing of the small bulk plants to determine if 90 percent control could be achieved for incoming and outgoing loads through the use of a vapor balance system and a refrigerant condensing system. A third study (EPA contract 68-01-3154, Task No. 20) surveyed small bulk plants in the Washington, D.C., Baltimore, and San Francisco areas. These studies were conducted to determine the applicability of the vapor balance system to the small bulk plants in the country. The results of the studies are being evaluated by bulk plant operators in these areas in compliance with vapor recovery regulations. A fourth study (EPA contract 68-01-4120, Task No. 9) provided for the evaluation of the applicability, availability, and the financial impact of installing and operating top-loading vapor balance systems at small bulk plants. In addition, Motion Corporation performed a study for EPA (EPA contract No. 68-01-1015, Task No. 2) in 1975 which evaluated vapor control methods for gasoline bulk plant operations, including small bulk plants.

A fifth study (EPA contract 68-01-1015, Task No. 1) was conducted to determine the applicability of existing types of vapor recovery systems and the availability of achieving the 90 percent control level of gasoline vapor recovery. They further concluded that the equipment and technology for achieving these levels does exist in sufficient supply, and has been satisfactorily applied on gasoline transfer operations for a number of years.

These studies also concluded that certain types of vapor control systems (e.g., a bottom-loading vapor balance system) are more desirable from the viewpoint of the owner and operator because of the more efficient marketing capability that the systems provide. It must be emphasized that there is no requirement in the Federal regulations that bottom-loading systems be

installed. These bottom-loading systems, because of the additional modifications and equipment required for installation on bulk plants, are consistently much more expensive to install than the alternative top-loading vapor balance systems.

The recent EPA studies on small bulk plants reaffirm the earlier conclusion that controls are available at a reasonable cost. Specifically, EPA has determined that the top-loading vapor balance system, which is readily available and the least expensive alternative, will meet the 90 percent control level established in the existing regulations at a reasonable cost. However, due to the Administrator's concern regarding the possible economic impacts of these regulations on the very small bulk plants (less than 4,000 gal./day of gasoline throughput), EPA will continue to study such impacts. EPA will report on its findings at the end of four to six weeks.

For further information or copies of the studies contact: Janet Field, Policy Planning Section (EPA-311), Division of Stationary Source Enforcement, U.S. Environmental Protection Agency, 401 M Street, S.W., Washington, D.C. 20460.

Dated: May 31, 1977.

Don R. Goodman,
 Administrator.

[EPA Doc. 77-12071-1 and 2-0-77-540-2A]

[EPA 740-7]

PART 61—NATIONAL EMISSION STANDARDS FOR HAZARDOUS AIR POLLUTANTS

Standard for Vinyl Chloride, Corrections and Amendments

AGENCY: Environmental Protection Agency.

ACTION: Final rule.

SUMMARY: These amendments are being made to the vinyl chloride standard which was promulgated under the authority of the Clean Air Act on October 21, 1970. The standard contains some typographical errors and needs clarification in some parts. These amendments are intended to correct the typographical errors and clarify the standard.

EFFECTIVE DATE: June 7, 1977.

FOR FURTHER INFORMATION CONTACT:

Don R. Goodman, Federal Standards and Engineering Division, Environmental Protection Agency, 401 M Street, N.W., Washington, D.C. 20460. Telephone: 202-343-2111, ext. 271.

STATUS OF THE STANDARD: On October 21, 1970, under authority of the Clean Air Act, as amended (42 U.S.C. 1857), the Environmental Protection Agency (EPA) promulgated a national emission standard for vinyl chloride (41 FR 40820). The standard covers plants which manufacture ethylene dichloride, vinyl chloride, and/or polyvinyl chloride. Since that time, it has become apparent that a few sections of the standard and Test Methods 106 and 107 are unclear. The purpose of the amendments being made at this time is to clarify these sections and to correct typographical errors. These corrections are in addition to those published on December 3, 1976 (41 FR 53017). The Administrator finds that

good cause exists for omitting prior notice and public comment on these amendments as unnecessary and for making them immediately effective because they simply clarify and correct the existing regulations and impose no additional substantive requirements.

The most significant amendment involves clarification of the requirements for certification of the analysis of gas cylinders which may be used to calibrate testing and monitoring equipment. The standard, as promulgated on October 21, 1976, requires that an analysis of the gas used for calibration purposes, " . . . be accessible to the National Bureau of Standards or to a governmentally certified calibration lab." Comments were received indicating that the term "accessible" was unclear.

The comments require that the analysis of gas cylinders which may be used for calibration of testing and monitoring equipment be certified by the National Bureau of Standards or by a governmentally certified calibration lab. The standard is of this gas contained in each cylinder of the gas used in a analytical procedure the manufacturer had calibrated on the day the analysis was performed. Calibration of the analytical procedure was to have been done using gas for which the concentrations have been certified: (1) by comparison with a certified ethylene dichloride reference gas, (2) by comparison with a gas mixture certified in accordance with the procedure described in § 7.1 of Test Method 106 and using 99.9 percent vinyl chloride, or (3) by direct analysis by the National Bureau of Standards. These amendments are being made to § 61.65(b)(2)(iii) and § 61.67, which contain the provisions for gas cylinders, and to § 61.62 and § 61.63, which contain the provisions for gas cylinders, respectively.

There are several other changes in the standard for gas cylinders. For example, § 61.60 is being amended to require that the gas cylinder used for calibration of testing and monitoring equipment be certified by the National Bureau of Standards or by a governmentally certified calibration lab. The standard is of this gas contained in each cylinder of the gas used in a analytical procedure the manufacturer had calibrated on the day the analysis was performed. Calibration of the analytical procedure was to have been done using gas for which the concentrations have been certified: (1) by comparison with a certified ethylene dichloride reference gas, (2) by comparison with a gas mixture certified in accordance with the procedure described in § 7.1 of Test Method 106 and using 99.9 percent vinyl chloride, or (3) by direct analysis by the National Bureau of Standards. These amendments are being made to § 61.65(b)(2)(iii) and § 61.67, which contain the provisions for gas cylinders, and to § 61.62 and § 61.63, which contain the provisions for gas cylinders, respectively.

each batch of resin is to be measured for its vinyl chloride content. Section 61.71 (a) is being changed to correct typographical errors and to clarify that daily operating records for polyvinyl chloride reactors are required to be kept whether a relief valve discharges or not.

Section 4.3.2 of Test Method 106 is being revised to allow the option of using Poropak T as the column packing instead of GE SP-96 in a secondary gas chromatographic column if acetaldehyde is present. This packing has also been shown to produce adequate separation of vinyl chloride and acetaldehyde. Section 61.67(e) of the regulation and § 6.2 of Test Method 106 are being amended to include a limit on the amount of time a test sample can be kept before it is analyzed for vinyl chloride. Section 1.2 of Test Method 107 is being amended to clarify that chromatograph parameters can be altered if the precision and repeatability of analysis of vinyl chloride is not impaired. Section 5.2.2 of Test Method 107 is being amended to allow the use of a pair of Poropak Q columns if methanol or acetaldehyde is present in the sample. Also in Test Method 107 a clarification for the term K_w has been added to § 9.2.

The remaining changes are corrections of typographical errors or are self-explanatory.

These amendments are issued under the authority of section 112 of the Clean Air Act, sec. 4(a) of Pub. L. 91-604, 84 Stat. 1855 (42 U.S.C. 1857c-7) and section 301(a) of the Clean Air Act, sec. 2 of Pub. L. No. 90-148, 81 Stat. 504, as amended by sec. (15)(c)(2) of Pub. L. 91-604, 84 Stat. 1713 (42 U.S.C. 1857c-6). The amendments to §§ 61.67 and 61.63 are also issued under the authority of section 114 of the Clean Air Act, as added by sec. 4(a) of Pub. L. 91-604, 84 Stat. 1867 and amended by Pub. L. 92-619, sec. 6(a)(4), 93 Stat. 379 (42 U.S.C. 1857c-9).

The amendments are being made to the following sections of the Code of Federal Regulations: 40 CFR 61.60, 61.61, 61.62, 61.63, 61.65, 61.67, 61.68, 61.69, 61.70, and 61.71.

Dated: May 16, 1977.

Robert F. Trow,
Acting Assistant Administrator
for Air and Waste Management.

Part 61 of Chapter I, Title 40 of the Code of Federal Regulations is amended as follows:

1. In § 61.60, paragraph (c) is amended as follows:

§ 61.60 Applicability.

(c) Sections of this subpart other than §§ 61.61; 61.64 (a) (1), (b), (c), and (d); 61.67; 61.68; 61.69; 61.70; and 61.71 . . .

2. In § 61.61 paragraphs (t) and (u) are added as follows:

§ 61.61 Definitions.

(t) "Standard temperature" means a temperature of 20° C (69° F).
(u) "Standard pressure" means a pressure of 760 mm of Hg (29.92 in. of Hg).

3. Section 61.62 is corrected as follows:

§ 61.62 Emission standard for ethylene dichloride plants.

(a) Ethylene dichloride purification: The concentration of vinyl chloride in all exhaust gases discharged to the atmosphere from any equipment used in ethylene dichloride purification is not to exceed 10 ppm, except as provided in § 61.65(a). This requirement does not apply to equipment that has been repaired, is out of operation, and met the requirement in § 61.65(b) (6) (i) before being repaired.

(b) Oxychlorination reactor: Except as provided in § 61.65(a), emissions of vinyl chloride to the atmosphere from each oxychlorination reactor are not to exceed 0.2 g/kg (0.0002 lb/lb) of the 100 percent ethylene dichloride product from the oxychlorination process.

4. In § 61.65, paragraphs (b) (1), (b) (8) (ii) (A), and (b) (3) (ii) (B) are amended as follows:

§ 61.65 Emission standard for ethylene dichloride, vinyl chloride and polyvinyl chloride plants.

(b) . . .

(1) Loading and unloading lines: Vinyl chloride emissions from loading and unloading lines in vinyl chloride service which are opened to the atmosphere after each loading or unloading operation are to be minimized as follows:

(b) . . .

(iii) . . .

(A) A calibration gas mixture prepared from the gases specified in sections 5.2.1 and 5.2.2 of Test Method 106 and in accordance with section 7.1 of Test Method 106, or

(B) A calibration gas cylinder containing the appropriate concentration of vinyl chloride. The gas composition of the calibration gas cylinder standard is to have been certified by the National Bureau of Standards. 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 manufacturer to the buyer. If a gas chromatograph is used as the vinyl chloride monitoring system, these gas mixtures may be directly used to prepare a chromatograph calibration curve as described in section 7.3 of Test Method 106. The requirements in section 5.2.3.1 and 5.2.3.2 of Test Method 106 for certification of cylinder standards and for establishment and verification of calibration standards are to be followed.

(Secs. 112 and 301(a), Clean Air Act (42 U.S.C. 1857c-7 and 1857c-9(a)).)

5. Section 61.67 is amended by deleting and reserving paragraph (d), revising paragraphs (e), (g)(1)(ii) and (g)(1)(iii), and by adding paragraph (g)(1)(iv) as follows:

§ 61.67 Emission tests.

(d) (Reserved)

(e) When at all possible, each sample is to be analyzed within 24 hours, but in no case in excess of 72 hours of sample collection. Vinyl chloride emissions are to be determined within 30 days after the emission test. The owner or operator shall report the determinations to the Administrator by a registered letter dated before the close of the next business day following the determination.

(g)

(1)

(ii) Each analysis is to be a test of ten runs. For the purpose of determining emissions, the sum of the results of all runs is to be used. The results of the first two runs are to be discarded.

(iii) For gas streams containing more than 10 percent oxygen the concentration of vinyl chloride as determined by Test Method 106 is to be corrected to 10 percent oxygen (dry basis) for determination of each leak by using the following equation:

$$C_{\text{corrected}} = \frac{C_{\text{measured}} \times 200}{200 - P_{\text{O}_2}}$$

where:

$C_{\text{corrected}}$ = The concentration of vinyl chloride in the gas stream, in percent.

C_{measured} = The concentration of vinyl chloride in the gas stream, in percent, as measured by Test Method 106.

200 = Percent oxygen in the gas stream at standard conditions, in percent.

P_{O_2} = Percent oxygen in the gas stream at standard conditions, in percent.

(iv) For those emission sources where the emission limit is prescribed in terms of mass rather than concentration, mass emissions in kg/100 kg product are to be calculated by using the following equation:

$$C_{\text{EPR}} = \frac{C_{\text{ax}}(2.60)Q}{Z}$$

where:

C_{ax} = kg vinyl chloride/100 kg product.

C_{EPR} = The concentration of vinyl chloride as measured by Test Method 106.

2.60 = Density of vinyl chloride at one atmosphere and 20° C in kg/m³.

Q = Volumetric flow rate in m³/hr as determined by Reference Method 2 of Appendix A to Part 60 of this chapter.

Z = Production rate (kg/hr).

6. Section 61.63 is amended by revising paragraphs (c)(1) and (c)(2) as follows:

§ 61.63 Emission monitoring.

(c)

(1) A calibration gas mixture prepared from the gases specified in sections 5.2.1 and 5.2.2 of Test Method 106 and in accordance with section 7.1 of Test Method 106, or

(2) A calibration gas cylinder standard containing the appropriate concentration of vinyl chloride. The gas composition of the calibration gas cylinder standard is to have 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 ±0.5 percent from the certified value. The date of gas fill, expiration date, certified vinyl chloride concentration and recommended shelf life must have been printed on the cylinder before shipment to the user. The user must certify the date of receipt, the date of calibration, the date of use, and the date of expiration. The cylinder must be directly used to produce a check gas with calibration gases as described in section 7.3 of Test Method 106. The requirements in sections 5.2.3.1 and 5.2.3.2 of Test Method 106 for certification of cylinder standards and for establishment and verification of calibration standards are to be followed.

tion of calibration standards are to be followed.

(Secs. 112, 114, and 301(a), Clean Air Act (42 U.S.C. 1857c-7, 1857c-9, and 1857c-9(a)).)

7. In § 61.70 paragraphs (c)(2)(i) and (c)(2)(v) are amended as follows:

§ 61.70 Semiannual report.

(c)

(2)

(i) If batch stripping is used, one representative sample of polyvinyl chloride resin is to be taken from each batch of each grade of resin immediately following the completion of the stripping operation, and identified by resin type and grade and the date and time the batch is completed. The corresponding quantity of material processed in each stripper batch is to be recorded and identified by resin type and grade and the date and time the batch is completed.

(v) The report to the Administrator by the owner or operator is to include the vinyl chloride content found in each sample required by paragraphs (c)(2)(i) and (c)(2)(iv) of this section, averaged separately for each type of resin, over each calendar day and weighted according to the quantity of each grade of resin processed by the stripper(s) that calendar day, according to the following equation:

$$A_r = \frac{\sum_{i=1}^n P_i M_i}{Q_r} = \frac{P_1 M_1 + P_2 M_2 + \dots + P_n M_n}{Q_r}$$

where:

A_r = 24-hour average concentration of type r resin in ppm (dry weight basis).

Q_r = Total production of type r resin over the 24-hour period, in kg.

P_i = $T_{i,m}$ of resin i , 1, 2, . . . , m , where m is total number of resin types produced during the 24-hour period.

M = Concentration of vinyl chloride in one sample of grade G , resin, in ppm.

P = Production of grade G resin represented by the sample, in kg.

G = Grade of resin, e.g., G_1 , G_2 , and G_3 .

m = Total number of grades of resin produced during the 24-hour period.

8. Section 61.71 is amended by correcting paragraphs (a)(2) and (a)(3), and by adding paragraph (a)(4) as follows:

§ 61.71 Recordkeeping.

(a)

(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)(2), 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 each leak detected in accordance with § 61.65.

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

9. Section 1.1 of Test Method 106 is corrected as follows:

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

10. Section 3 of Test Method 106 is corrected as follows:

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 4.2 and 6.4. If resolution of the vinyl chloride peak is not satisfactory for a particular sample, then chromatographic 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.

11. Section 4.5 of Test Method 106 is corrected as follows:

4.1 Sampling (Figure 106-1).

12. Section 4.1.3 of Test Method 106 is corrected as follows:

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.

13. Section 4.1.10 of Test Method 106 is corrected as follows:

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

14. Section 4.3.2 of Test Method 106 is amended as follows:

4.3.2 Chromatographic column. Stainless steel, 2 m x 3.2 mm, containing 80/100 mesh Chromasorb 102. A secondary column of GE SP-96, 20 percent on 60/80 mesh AW Chromasorb P, stainless steel, 2 m x 3.2 mm or Porapak Q, 20/100 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 100° C.

15. Section 5.2 of Test Method 106 is amended as follows:

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

5.2.1 Vinyl chloride, 99.9+ percent. Pure vinyl chloride is used by the manufacturer to prepare a mixture of 99.9+ percent vinyl chloride in nitrogen. If the manufacturer has a bulk cylinder of 99.9+ percent vinyl chloride, the manufacturer may have been permitted on this supply rather than on each cylinder prepared from this bulk supply. The date of gas cylinder preparation and the manufacturer must have been affixed to the cylinder before shipment from the gas cylinder to the buyer.

5.2.2 Nitrogen gas, 2 grade, for preparation of standard gas mixtures.

5.2.3 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 a shelf life for each cylinder that does not exceed 5 percent from the date of gas cylinder preparation, or 5 ppm vinyl chloride concentration and must have been affixed to the cylinder before shipment from the gas cylinder to the buyer. These gas mixture standards may be used to prepare a calibration curve by the manufacturer.

5.2.3.1 Preparation of calibration curve. The concentration of each gas mixture standard is determined by the manufacturer using a gas chromatograph calibrated with standards in sections 6.2 and 6.4. Each standard is prepared for 30 days at the rate of 100 ml/min with each standard gas mixture and analyze the sample. The concentration of vinyl chloride in the standard gas mixture, flow rate, and retention time should be recorded. Calculate the peak area multiplied by the detector setting. Repeat until two injection areas are within 5 percent, then plot the points v. C. 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.

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 certified 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.

16. Section 6.2 of Test Method 106 is amended as follows:

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.

17. Section 7.1 of Test Method 106 is amended as follows:

7.1 Preparation of vinyl chloride standard gas mixtures. Evacuate a sixteen-inch square Teflon 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 0.04 ml 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.)

18. Section 7.3 of Test Method 106 is amended as follows:

7.3 Preparation of chromatograph calibration curve. Make a gas chromatograph calibration curve of each gas mixture standard (described in section 5.2.2 or 7.1) using conditions identical with those listed in sections 6.2 and 6.4. Each standard is prepared for 30 days at the rate of 100 ml/min with each standard gas mixture and analyze the sample. The concentration of vinyl chloride in the standard gas mixture, flow rate, and retention time should be recorded. Calculate the peak area multiplied by the detector setting. Repeat until two injection areas are within 5 percent, then plot the points v. C. 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.

19. Section 1.2 of Test Method 107 is amended as follows:

1.2 This procedure is suitable for determining the vinyl chloride content (VCM) of residual in process waste streams and the residual vinyl chloride monomer (RVCM) content of polyvinyl chloride (PVC) resins, wet cake, slurry, and latex samples. It cannot be used for polymer in solid forms, such as sheet or cubes. If a reduction of the vinyl chloride peak is not satisfactory for a particular sample, then

chromatograph parameters may be altered provided that the precision and reproducibility of the analysis of vinyl chloride cylinder standards are not impaired. If there is reason to believe that some other hydrocarbon with an identical retention time is present in the sample, then supplemental confirmation of the vinyl chloride peak through an absolute analytical technique, such as mass spectroscopy, should be performed.

20. Section 3.3.2 is amended as follows:

3.3.2 Chromatographic column. Stainless steel, 2 m x 3.2 mm, containing 80/100 mesh Carbowax 1500 on Carbowax A, Perkin-Elmer Corporation No. 105-0133, or 10/100 mesh Carbowax C can be used in place of Carbowax A. If acetaldehyde or acetaldehyde is present in the sample, a pair of Porapak Q columns in series (1 m x 3.2 mm followed by 2 m x 3.2 mm) with provision for 50 ml of the first column has been added to provide adequate separation of vinyl chloride.

21. Section 6.2 of Test Method 107 is revised as follows:

6.2 Calibration.

6.2.1 Cylinder standards (4). Gas mixture standards (50, 10, 5, and 1 ppm vinyl chloride in nitrogen cylinders) for which the gas composition has been certified by the manufacturer. Lower concentration standards should be obtained if lower concentrations of vinyl chloride samples are expected, as the intent is to bracket the sample concentrations with standards. 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 manufacturer to the buyer.

6.2.1.1 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 a calibration standard and use these standards in the following ways: (1) a high concentration standard (between 4000 and 8000 ppm) for preparation of a calibration curve by an appropriate dilution technique; (2) a low concentration standard (between 50 and 100 ppm) for verification of the dilution technique used.

6.2.1.2 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 gas mixture standard generated in a similar manner to the procedure described in section 7.1 of Method 106 for preparing gas mixtures and standards using 99.9+ percent vinyl chloride, or (2) 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.

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