



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Air Quality Planning and Standards
Research Triangle Park, North Carolina 27711

December 20, 1976

Dr. Armond T. Perretta
Precision Gas Products, Inc.
681 Mill Street
Rahway, New Jersey 07065

Dear Dr. Perretta:

This is in response to your letter of November 9, 1976, to Dr. Steigerwald concerning vinyl chloride calibration gas mixtures. We appreciated receiving your letter and comments, and we agree that clarification of the methods is needed.

As a result of your inquiry we have evaluated the provisions in Methods 106 and 107 concerning calibration gases, and have prepared clarifying revisions to the methods. These revisions are consistent with the intent of the current regulation, and are consistent with our position that accurate calibration materials are a key element necessary to assure quality analytical results. The revisions are, we believe, clear and are workable. Although traceability to NBS is not necessary, the cylinder certification procedures do require analysis of each cylinder standard (gas mixture standard) and prescribe procedures which involve verification against independently prepared standards. Gravimetric procedures are acceptable for preparation of cylinder standards. However, such preparation is not considered an acceptable basis for certification of the cylinder standards.

These revisions are considered to be clarifications which offer more options and which are not more restrictive than the present requirements. For this reason, and because these clarifications need to be made available as soon as possible, we are planning to promulgate these revisions in January. I am enclosing a draft copy of the revisions and would be happy to review any comments which you may have on these.

Sincerely yours,

Robert L. Ajax/rs

Robert L. Ajax
Chief

Emission Measurement Branch
Emission Standards and
Engineering Division

Enclosure

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METHOD 106

Recommended Revision to Section 5.2 (Use in toto)

5.2 CALIBRATION. Use one of the following options: either 5.2.1 or 5.2.2.

5.2.1 Vinyl chloride, 99.9+% - pure vinyl chloride gas certified by the manufacturer to contain a minimum of 99.9% 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+% vinyl chloride, the certification analysis may be 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 be affixed to the cylinder before shipment from the gas manufacturer to the buyer.

5.2.2 Cylinder standards (3) - gas mixture standards (50, 10, and 5 ppm vinyl chloride in nitrogen cylinders) prepared and certified by a gas manufacturer. The gas manufacturer must recommend 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 be 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.

5.2.2.1 Cylinder standards certification - The concentration of each 50, 10, and 5 ppm vinyl chloride in nitrogen cylinder must be certified by the gas manufacturer. The certified concentration must have been determined by a direct analysis of each cylinder using an analytical procedure that the gas manufacturer had calibrated on the day of cylinder analysis. The calibration of the analytical procedure shall, as a minimum, utilize a three-point calibration curve. It is recommended that the gas 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.

5.2.2.2 Establishment and verification of calibration standards. The concentration of each calibration standard must be established by the gas manufacturer using reliable procedures. Additionally, each calibration standard must be verified by one of the following procedures, and the agreement between the initially determined concentration value and the verification concentration value must be within $\pm 5\%$: (1) verification value determined by comparison with a calibrated vinyl chloride permeation tube, (2) verification value determined by comparison with the procedure described in Section 7.1 for preparing standard gas mixtures using 99.9+% 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.

Recommended Revision to Section 7.1

Add the following to the end of the current paragraph. "These gas mixture standards may be used for 10 days from the date of preparation, after which time preparation of new gas mixture standards is required. (Caution - contamination may be a problem when a bag is reused if the new gas mixture standard is a lower concentration than that of the previous gas mixture standard."

Recommended Revision to Section 7.3

Change the second sentence to read: "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."

METHOD 107

Recommended Revision to Section 6.2

6.2 CALIBRATION.

6.2.1 Cylinder standards (4) - gas mixture standards (50, 500, 2000 and 4000 ppm vinyl chloride in nitrogen cylinders) prepared and certified by a gas manufacturer. The gas manufacturer must recommend 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 be affixed to the cylinder before shipment from the gas manufacturer to the buyer.

6.2.1.1 Cylinder standard certification - The concentration of each 50, 500, 2000 and 4000 ppm vinyl chloride in nitrogen cylinder must be certified by the gas manufacturer. The certified concentration must have been determined by a direct analysis of each cylinder using an analytical procedure that the gas manufacturer had calibrated on the day of cylinder analysis. The calibration of the analytical procedure shall, as a minimum, utilize a three-point calibration curve. It is recommended that the gas manufacturer maintain two calibration standards and use these standards in the following way: (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 500 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 be established by the gas manufacturer using reliable procedures. Additionally, each calibration standard must be verified by one of the following procedures, and the agreement between the initially determined concentration value and the verification concentration value

must be within $\pm 5\%$: (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 is inserted for preparing gas mixture standards using 99.9+% 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.