

Australian/New Zealand Standard®

Methods of test for PVC pipes and fittings

Method 15: Method for determination of vinyl chloride monomer content

PREFACE

This Standard was prepared by the Joint Standards Australia/Standards New Zealand Committee PL/21 on Unplasticized PVC Pipe.

METHOD

1 SCOPE This Method sets out the procedure for determining the concentration of vinyl chloride monomer in PVC pipes and fittings.

2 REFERENCED DOCUMENTS The following documents are referred to in this Standard:

AS

2162 Code of practice for the use of volumetric glassware

2164 One-mark volumetric flasks

2166 One-mark pipettes

3 PRINCIPLE A sample of the PVC pipe or fitting immersed in NN-dimethylacetamide (DMA) contained in a sealed glass vial is equilibrated at 45°C. The vinyl chloride monomer (VCM) content of the sample is determined by the gas chromatographic analysis of a sample of the head space vapour (see Clause 10, Reference 2).

4 APPARATUS The following apparatus is required:

NOTE: Other apparatus which can be shown to provide an accuracy of the same or a higher degree may be used.

(a) *Gas chromatograph*—machines incorporating either manual or automatic headspace injections. All machines shall employ flame ionization to detect VCM.

(b) *Columns*—2 m × 2 mm I.D. stainless steel packed with Porapak Q80/100.

NOTE: A Carbowax or modified Porapak type column is recommended for the rare instances when Porapak Q shows unsatisfactory resolution of VCM from interfering substances.

(c) *Digital integrator*—alternatively, a recorder with 1 mV full scale deflection may be used to monitor peak heights.

(d) *Glass vials*—nominal 25 mL capacity equipped with butyl rubber septa and aluminium sealing caps, together with a manual crimping tool for sealing the vials.

NOTE: The dimensions of the vial are unimportant but it is necessary to select containers of equivalent volume.

- (e) *Gas tight syringe*
- (f) *Graduated Borosilicate flasks*—50 mL.
- (g) *Glassware*—glassware complying with AS 2162, AS 2164 or AS 2166, as appropriate.

5 REAGENTS The following reagents are required:

- (a) *NN-dimethylacetamide (DMA)*

NOTE: In practice new batches of DMA are screened for potential interferences. If DMA is of inferior purity, usually a single distillation (collect boiling range 163°C to 166°C at 760 mm) provides an acceptable fraction.

- (b) *Vinyl chloride monomer (VCM)*—pure, stored in a stainless steel sample cylinder of 500 mL, approximate capacity fitted with needle valves. From this pure VCM, $\pm 2\%$ accurately certified 100 $\mu\text{L/L}$ to 1000 $\mu\text{L/L}$ VCM in air or nitrogen samples are made for the preparation of calibration standards.

WARNING: VCM IS FLAMMABLE AND CARCINOGENIC; SPECIAL CARE SHOULD BE TAKEN TO AVOID THE INHALATION OF VCM VAPOURS.

6 PROCEDURE The procedure shall be as follows:

6.1 Sample preparation Weigh 0.500 ± 0.005 g cut from the pipe or fitting into a glass vial, add 5.9 mL DMA, seal the vial and equilibrate at $45 \pm 2^\circ\text{C}$ overnight. In the initial hour or two of heating, gently agitate the contents to ensure all the sample has completely dissolved or disintegrated.

One gram of polymer dissolved in 10 mL DMA gives the same VCM response as 0.5 g polymer dissolved in 5 mL DMA and in both cases, up to a 20% polymer solution, there is a linear relationship between VCM response and sample mass. Half a gram of polymer in 5 mL DMA is recommended as being the concentration of easiest dissolution of polymer.

6.2 Standards preparation Prepare an approximate 20 mg/mL solution of VCM in DMA as follows:

- (a) Fill a 50 mL graduated flask with DMA to within approximately 10 mL of the graduation.
- (b) Stopper flask with a rubber septum and weigh.
- (c) Slowly bubble VCM into the DMA through a syringe barrel and needle. Vent the flask with a second syringe needle.
- (d) After several minutes, remove both needles and reweigh.
- (e) Using a syringe, make up to 50 mL volume with DMA and calculate the actual VCM concentration of the standard solution (see Clause 10, Reference 1). Alternatively, a gas sampling bulb (at least 200 mL capacity), which is quantitatively filled at atmospheric pressure with a certified VCM gas standard, may be used. No more than 5% of the capacity of the bulb is removed for the preparation of standards.

After preparation, liquid standards should be stored under refrigerated conditions. When this is done, the standards are stable for up to one month.

6.3 Control preparation Select a 'control' sample of PVC so that the VCM content is below the detection limit (see Clause 7). Prepare a set of samples of this control as described in Clause 6.1. Use one of these samples for the 'blank' determination. Prepare calibration standards over an appropriate VCM concentration range by spiking the remainder of the set of solutions with varying amounts of the standard VCM mixture. Equilibrate overnight at $45 \pm 2^\circ\text{C}$.