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Plastics — Poly(vinyl chloride) — Determination of residual vinyl chloride monomer — Gas-chromatographic method

Plastiques — Poly(chlorure de vinyle) — Détermination du chlorure de vinyle monomère résiduel — Méthode par chromatographie en phase gazeuse

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 6401 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 6401:1985), which has been technically revised.

Plastics — Poly(vinyl chloride) — Determination of residual vinyl chloride monomer — Gas-chromatographic method

SAFETY PRECAUTIONS — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory requirements.

1 Scope

This International Standard specifies a method for the determination of vinyl chloride monomer in homopolymer and copolymer resins of vinyl chloride and compounded materials. The method is based on sample dissolution and headspace gas chromatography. Concentrations of vinyl chloride in the range 0,1 mg/kg to 3,0 mg/kg can be determined.

A “dry method”, suitable for PVC resins but not compounded materials, is widely used within the industry for in-house determinations. A separate International Standard based on this methodology is under development.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 apply.

4 Principle

The level of vinyl chloride monomer is determined by headspace gas chromatography of the polymer test sample dissolved/swollen in *N*,*N*′-dimethylacetamide.

5 Sampling

A representative sample shall be taken.

A concentration gradient may form in stored resin samples due to the volatility of vinyl chloride. Cooling of the sample prior to sampling is advisable but condensation of humidity must be avoided. Sample preparation shall be carried out as quickly as possible to minimize losses of residual monomer. When exchanging samples between laboratories or when storage is necessary, samples should be sealed in completely filled and sealed glass bottles or vials (e.g. 6.5, 6.6).

6 Apparatus

Standard laboratory apparatus and the following:

6.1 Gas chromatograph (GC), fitted with an automatic static headspace sampler.

6.2 Flame ionization detector (FID).

6.3 Gas-chromatographic column.

The signal obtained with a solution containing 0,01 mg of vinyl chloride per litre shall be at least three times that of the baseline noise. The limit of detection of the method is 0,1 mg/kg vinyl chloride in test samples. Examples of suitable columns are described in Annex A, Table A.1.

6.4 Data-processing system, for data acquisition and evaluation of GC runs.

6.5 Glass bottles, capacity 30 ml, with polytetrafluoroethylene (PTFE) faced silicone septa and aluminium caps.

6.6 Glass vials, capacity 22,5 ml, with polytetrafluoroethylene (PTFE) faced silicone septa and aluminium caps.

6.7 Crimping and decapping tools, for sealing and uncapping the vials.

6.8 Glass pipettes, capacity 25 ml and 10 ml.

6.9 Microsyringes, capacity 500 µl and 100 µl.

6.10 Gastight glass syringe, capacity 10 ml, with lock valve.

6.11 Analytical balance, capable of weighing to 0,1 mg.

7 Reagents and materials

All reagents shall be of recognized analytical grade.

WARNING — Vinyl chloride is a hazardous substance which is a gas at ambient temperature. The preparation of solutions should therefore be carried out only under a well-ventilated fume hood.

7.1 Vinyl chloride, of purity greater than 99,5 %. The vinyl chloride gas cylinder shall be fitted with a syringe adapter.

7.2 *N,N′*-dimethylacetamide, density $\rho = 0{,}937$ g/ml. The solvent shall not contain any impurity with the same chromatographic retention time as vinyl chloride under the conditions of the test.

WARNING — *N,N′*-dimethylacetamide is also a hazardous substance.

7.3 Detector gases and carrier gas: High-purity gases shall be used to achieve the required low limits of quantification.

7.4 Vinyl chloride, standard solution, with a vinyl chloride concentration of approximately 1 600 mg/l.

To a 30 ml glass bottle (6.5), add, using a glass pipette (6.8), 25 ml of *N,N′*-dimethylacetamide (7.2) and cap the bottle with a PTFE-lined silicone septum. Weigh (to 0,1 mg) the bottle containing the *N,N′*-dimethylacetamide. Introduce 10 ml of vinyl chloride gas through the septum into the *N,N′*-dimethylacetamide, using a pre-flushed 10 ml gastight syringe (6.10) and holding the end of the syringe needle below the surface of the liquid. Avoid the contents of the bottle becoming contaminated by air. Identify this solution as solution A.

Repeat the procedure with a second 30 ml glass bottle and identify the resultant solution as solution B.

Leave both bottles for 2 h at room temperature to allow complete adsorption of the vinyl chloride. Reweigh to the nearest 0,1 mg to determine the mass of monomer which has been added. The mass of vinyl chloride in each standard solution will be about 40 mg, depending on cylinder pressure. Record the concentration of vinyl chloride in solutions A and B in milligrams per litre.

Store the solutions in a refrigerator.

7.5 Vinyl chloride, working calibrant stock solutions, with a vinyl chloride concentration of approximately 32 mg/l.

To a 30 ml glass bottle, add, using a glass pipette, 25 ml of *N*,*N'*-dimethylacetamide (7.2) and seal with a PTFE-lined silicone septum and cap. Transfer 500 µl of solution A through the septum into the bottle using a suitable syringe.

Repeat for solution B and label the two diluted calibrant solutions as solution C and solution D.

Record the concentration of vinyl chloride in the working calibrant stock solutions in milligrams per litre.

7.6 Vinyl chloride calibration solutions, with vinyl chloride concentrations between 0 mg/l and approximately 0,3 mg/l.

Take seven 22,5 ml headspace vials (6.6) and add, using a glass pipette, 10 ml of *N*,*N'*-dimethylacetamide (7.2) to each. Using a 100 µl syringe, transfer 0 µl, 20 µl, 40 µl, 50 µl, 60 µl, 80 µl and 100 µl of solution C into the individual vials and seal with silicone/PTFE septa and caps. Take two more 22,5 ml headspace vials and add 10 ml of *N*,*N'*-dimethylacetamide. To these add 20 µl of solution D (giving a final concentration of 0,06 mg/l) and seal with a septum and cap. These last two solutions are used as check solutions.

8 Procedure

8.1 Preparation of test solutions

Weigh 1 g of sample (to 0,1 mg) into a 22,5 ml headspace vial (cut compounded materials into small pieces) and add 10 ml of dimethylacetamide. Seal with a silicone/PTFE septum and then cap the vial. Repeat this to produce triplicate test solutions for each sample.

8.2 Gas chromatography

Depending on the type of gas chromatograph and column used for the determination, establish the appropriate GC and FID parameters.

NOTE For guidance, the transfer line temperature and column oven temperature profile established for a GC equipped with column 2 described in Annex A are:

Transfer line temperature: 150 °C.

Column oven temperature profile: Isothermal at 80 °C for 2 min, from 80 °C to 170 °C at 5 °C/min, then from 170 °C to 230 °C at 20 °C/min. Under these conditions, vinyl chloride elutes at 8,4 min.

8.3 Determination

Transfer the test solutions, calibrant solutions and two check solutions to the static headspace sampler. Equilibrate them at 70 °C for 1 h prior to analysis.

Suggested operating parameters for the headspace sampler are:

needle temperature: 150 °C;

pressurizing time: 1,0 min;

injection time: 0,1 min;

withdrawal time: 0,5 min.

8.4 Preparation of the calibration graph

Plot a graph of the vinyl chloride contents of the calibration solutions, in milligrams per litre, against the corresponding peak areas.

9 Calculation

Determine the vinyl chloride content, in milligrams per litre, of the three test solutions and two check solutions from the calibration graph.

The vinyl chloride content of the sample, expressed in milligrams per kilogram, is given by the formula

$$c \times 10/m$$

where

c is the vinyl chloride content, in milligrams per litre, of the test solution, determined from the calibration graph;

m is the mass, in grams, of vinyl chloride in the test solution.

Confirm that the data obtained on the check solutions gives the concentration expected (within the repeatability criterion for the method — to be determined). If this is the case, report the results for each of the three test solutions individually, as well as their arithmetic mean and the standard deviation from the mean.

10 Precision

To be determined by interlaboratory trial.

11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for complete identification of the material tested;

c) the individual results for the test solutions, their arithmetic mean and the standard deviation;

d) the expected and measured vinyl chloride monomer content of the two check solutions;

e) any deviations from this International Standard;

f) the date of the test.

Annex A
(informative)

Suitable GC columns for the determination of vinyl chloride monomer

The PLOT (porous layer open tubular) columns described in Table A.1 have been found to be suitable for the determination.

Table A.1 — Suitable columns

Column	Length m	Diameter mm	Column type
1	15,00	0,53	Bonded polystyrene-divinylbenzene
2	30,00	0,53	Porous divinylbenzene homopolymer