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# International Standard



# 6401

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## Plastics — Homopolymer and copolymer resins of vinyl chloride — Determination of residual vinyl chloride monomer — Gas chromatographic method

*Plastiques — Résines d'homopolymères et de copolymères de 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6401 was prepared by Technical Committee ISO/TC 61, *Plastics*.

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# Plastics — Homopolymer and copolymer resins of vinyl chloride — Determination of residual vinyl chloride monomer — Gas chromatographic method

## 0 Introduction

The method specified in this International Standard is based on one that is described in the Directive of the European Economic Community (EEC) for *Materials and articles containing vinyl chloride monomer intended for use with foodstuffs*. (Commission Directive 80/776/EEC of 8th July 1980.)

## 1 Scope and field of application

This International Standard specifies a method for the determination of residual vinyl chloride monomer in homopolymer and copolymer resins of vinyl chloride.

For the purpose of this International Standard, the field of application is limited to these homopolymer and copolymer resins, but the method is also applicable to materials and articles made from the resins.

The lower limit of detection is dependent on the extent to which impurities in the solvent interfere. Use of the solvent specified ensures that 0,5 mg/kg can be detected but 0,2 mg/kg can be detected if interference is sufficiently low.

## 2 References

ISO 472, *Plastics — Vocabulary*.

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by interlaboratory tests*.

## 3 Principle

Dissolution or suspension of a test portion in a suitable solvent, and determination of the vinyl chloride content by gas chromatography, using the "head-space" method.

## 4 Reagents

All reagents shall be of recognized analytical grade and the water used shall be distilled water or water of equivalent purity.

**WARNING — Vinyl chloride is a hazardous substance and is a gas at ambient temperatures; the preparation of**

solutions must therefore be carried out only under a well-ventilated hood.

**4.1 Vinyl chloride**, of purity greater than 99,5 %.

**4.2 *N,N*-Dimethylacetamide**, or other suitable solvents such as *N,N*-dimethylformamide, tetrahydrofuran or dichloroethane, free of any impurity with the same chromatographic retention time as vinyl chloride or the internal standard (4.3), under the conditions of the test.

**4.3 Diethyl ether**, or other suitable reagent such as *cis*-but-2-ene, in the solvent (4.2), for use as the internal standard.

The internal standard shall not contain any impurity with the same chromatographic retention time as vinyl chloride under the conditions of the test.

The use of an internal standard is optional (see 6.3.1).

**4.4 Vinyl chloride**, standard solution corresponding to approximately 2 000 mg of vinyl chloride per kilogram.

Weigh, to the nearest 0,1 mg, a suitable glass vessel and place in it a suitable volume (for example 50 ml) of the solvent (4.2). Reweigh. Add a suitable mass (for example 0,1 g) of the vinyl chloride, either in liquid or gas form, injecting it slowly into the solvent.

The vinyl chloride may also be added by bubbling it through the solvent, provided that a device is used which will prevent loss of solvent.

Reweigh to the nearest 0,1 mg.

Leave for 2 h to allow equilibrium to be attained.

Store the solution in a refrigerator.

**4.5 Vinyl chloride**, dilute standard solution.

Dilute a weighed amount of the standard vinyl chloride solution (4.4) to a known volume or mass, using the solvent (4.2) or the internal standard (4.3).

Record the concentration of the dilute standard solution in milligrams per litre, or milligrams per kilogram, as appropriate.

## 5 Apparatus

Usual laboratory apparatus, and

**5.1 Gas chromatograph**, fitted with an automatic head-space sampler, or with facilities for manual sample injection.

**5.2 Flame ionization detector.**

In certain cases, it is possible to use other detectors, for example the micro-electrolytic conductivity detector described in the *Journal of Chromatographic Science*, **12**, March 1974, p. 152.

**5.3 Gas chromatographic column.**

The column shall be capable of completely separating the air peak, the vinyl chloride peak from the standard solution (4.5) and the internal standard peak, if used.

The signal obtained with a solution containing 0,02 mg of vinyl chloride per litre or kilogram shall be at least five times that of the background noise.

Examples of suitable columns are described in the annex.

**5.4 Recording potentiometer**, and **integrator**, if desired.

**5.5 Sample vials or flasks**, fitted with silicone or butyl rubber stoppers.

When using manual techniques, taking a sample from the head-space by means of a syringe may cause a partial vacuum to form inside the vial or flask. Hence, for manual techniques, if the containers are not pressurized before the sample is taken, the use of large sample containers (50 ml) is recommended.

**5.6 Micro-syringes.**

**5.7 Gas-tight syringes**, for manual head-space sampling.

**5.8 Analytical balance**, accurate to 0,1 mg.

## 6 Procedure

### 6.1 General

Take all the precautions necessary to ensure that no vinyl chloride or solvent is lost through volatilization.

If using manual sampling techniques, it is highly recommended that the internal standard (4.3) is used.

If the internal standard is used, the same solution shall be used throughout the procedure.

### 6.2 Test portion

Prepare three test portions by weighing, to the nearest 0,1 mg, not less than 200 mg of sample into each of three vials (5.5).

Try to ensure that the test portions have equal masses. Immediately close the vials.

### 6.3 Determination

#### 6.3.1 Preparation of test solutions

To the test portions (6.2) in the vials, add, in the proportion 10 ml per gram of test portion, the solvent (4.2), containing, if it is considered useful, the internal standard (4.3). Close the vials.

#### 6.3.2 Preparation of calibration solutions

Into two series of seven vials (5.5), introduce volumes of the dilute standard vinyl chloride solution (4.5), and of the solvent (4.2) or internal standard (4.3), such that duplicate series of solutions are obtained, having vinyl chloride concentrations of approximately 0 — 0,050 — 0,075 — 0,100 — 0,125 — 0,150 and 0,200 mg/l and such that all the vials contain the same volume of solvent as the test solutions (see 6.3.1). Close the vials.

#### 6.3.3 Preparation of calibration check solutions

Prepare a new standard vinyl chloride solution by repeating the procedure specified in 4.4, and subsequently prepare a dilute standard vinyl chloride solution, having a vinyl chloride concentration of 0,1 mg/l, using the procedure described in 4.5. Use duplicate aliquot portions for gas chromatography.

#### 6.3.4 Gas chromatography

**6.3.4.1** Place the vials containing the test solutions (6.3.1), the calibration solutions (6.3.2), and the calibration check solutions (6.3.3) in a water bath controlled at  $60 \pm 1$  °C and leave them for 2 h to allow equilibrium to be attained. Agitate the vials, avoiding contact between the liquid and stopper, to obtain, in the vials containing the test solutions, as homogeneous a suspension of resin as possible.

**6.3.4.2** Take samples from the head space of each vial.

If using manual sampling techniques, take care to obtain a reproducible sample (see 5.5); in particular, warm the syringe to the same temperature as the sample.

**6.3.4.3** Inject the samples, in turn, into the column and record the chromatograms.

If necessary, remove excess solvent from the column, using appropriate methods, as soon as solvent peaks appear on the chromatogram.

### 6.4 Preparation of the calibration graph

**6.4.1** Plot a graph having, for example, the vinyl chloride contents of the calibration solutions, in milligrams per litre or per kilogram, as abscissae and the corresponding peak areas (or heights) or these areas (or heights) relative to the internal standard as ordinates.

**6.4.2** The following requirements shall be carefully observed in the calculation of the calibration graph.

The graph shall be based on at least seven pairs of points.

The repeatability of the responses, as defined in ISO 472 and ISO 5725, shall be better than 0,02 mg of vinyl chloride per litre or kilogram.

The graph shall be calculated from the points by the method of least squares.

The graph shall be linear, i.e. the standard deviation of the responses around the regression line divided by the mean value of all responses shall not exceed 0,07.

**6.4.3** The average of the results of the determinations on the calibration check solutions shall not differ by more than 7 % from the corresponding point on the calibration graph. If the difference is greater than 7 %, reject the standard vinyl chloride solution (4.4) and subsequent dilutions, together with the standard vinyl chloride solution used to prepare the calibration check solutions, and repeat the test from the beginning.

## 7 Expression of results

### 7.1 Method of calculation

If the same quantities of sample, and of solvent of known and constant concentration of internal standard, were used in the determinations, read the vinyl chloride content, in milligrams per litre or kilogram, of the test solutions directly from the calibration graph.

Otherwise, determine the vinyl chloride content, in milligrams per litre or kilogram, of the test solutions by interpolation from the calibration graph.

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

$$\frac{C V}{m} \times 1\,000$$

where

$C$  is the vinyl chloride content, in milligrams per litre or kilogram, of the test solution, determined from the calibration graph;

$V$  is the volume, in millilitres, of solvent used<sup>1)</sup>;

$m$  is the mass, in milligrams, of the test portion.

Take as the result the arithmetic mean of the three determinations.

### 7.2 Precision

Round-robin tests conducted on homogeneous samples of a film have given the following results for a vinyl chloride monomer content of about 1 mg/kg :

- repeatability : 0,2 mg/kg
- reproducibility : 0,4 mg/kg.

## 8 Test report

The test report shall include the following information :

- a) a reference to this International Standard;
- b) complete identification of the material tested, including manufacturer's code number, grade, and form;
- c) description of the chromatographic column used;
- d) the type of detector;
- e) the calibration graph;
- f) the individual results for the three test portions and their arithmetic mean.

1) In the case of solvents measured by mass, this should be used in the formula instead of the volume.

## Annex

## Suitable columns for the determination of vinyl chloride monomer in homopolymer and copolymer resins

(This annex is given for information only.)

Column	Length, m	Diameter, mm	Stationary phase	Support	Operating temperature range, °C
1	2,00	2	Carbowax 1 500 20 % (m/m)	Carbopack B 180 to 150 µm	50 to 70
2 <sup>1)</sup>	1,00	2		Porapak Q or QS 180 to 150 µm	120 to 140
3	3,00	2	Di-isodecylphthalate 25 % (m/m)	Chromosorb WHP 180 to 150 µm	50 to 70
4	3,00	3	1,2,3-Tri(2 cyanoethoxy) propane 20 % (m/m)	Chromosorb W AW-DMCS 180 to 150 µm	35 to 55
5	3,60	2	Polypropylene-glycol 3 000 15 % (m/m)	Kieselguhr 250 to 180 µm	40 to 60

1) This column may not be suitable for determinations on copolymers of vinyl chloride and vinyl acetate, VC/VAC.

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