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Second edition

Plastics — Polymer dispersions — Determination of free formaldehyde

Plastiques — Dispersions de polymères — Dosage du formaldéhyde libre

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 15373:2001), which has been technically revised.

The main changes are as follows:

- text is revised according to the latest ISO Directive, Part 2.
- added Figure 1, as illustration of the principle of this method.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Plastics — Polymer dispersions — Determination of free formaldehyde

1 Scope

This document specifies two methods for the determination of free formaldehyde (HCHO) in polymer dispersions.

This document is applicable to acrylic, acrylonitrile butadiene, carboxylated styrene-butadiene and vinyl acetate polymer dispersions. It is also applicable to polymer dispersions of other compositions.

Method A (UV/Vis spectrometer method) is applicable to polymer dispersions with a free-formaldehyde content higher than 10 mg/kg.

Method B (HPLC method) is applicable for determining lower formaldehyde contents or carrying out arbitration analyses.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 2227, Formaldehyde solutions for industrial use — Determination of formaldehyde content

ISO 3696, Water for analytical laboratory use — Specification and test methods

3 Terms and definitions

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| https://standards.html.ai/catalog/standards/iso/9a1409c4-coaa-4a4/-baca-dd4bc1c9691b/iso-155/5-202

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The polymers dispersed in a polymer dispersion are separated from the aqueous phase by filtration, centrifuge or coagulation. The resulting aqueous sample solution containing formaldehyde reacts with 2,4-pentanedione to yield a lutidine derivative (see Figure 1), which can be analysed by UV detector at 410 nm.

Figure 1 — Reaction of formaldehyde with 2,4-pentanedione

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In method A, 2,4-pentanedione is added directly to the resulting aqueous solution. Absorption at 410 nm is measured with a UV/Vis spectrometer. The concentration of formaldehyde is determined using a calibration plot obtained by plotting the absorption at 410 nm of formaldehyde standards against the corresponding formaldehyde concentrations.

In method B, formaldehyde in the resulting aqueous solution is separated from other species by liquid chromatography on an octadecyldimethylsilyl (C18) reversed-phase column using an aqueous mobile phase. The detection system includes a post-column reactor which produces a lutidine derivative by reaction of formaldehyde with 2,4-pentanedione and a UV/Vis detector operating at 410 nm. The concentration of free formaldehyde in the resulting aqueous solution is determined using peak areas from the standard and sample chromatograms (calibration by external standard).

5 Interference

5.1 Method A

The following species have been identified as possible interferants in the method:

- acetaldehyde;
- glyoxylic acid.

However, interference by acetaldehyde and glyoxylic acid is to be expected only when the species concerned is present in excess amounts (100-fold and more) compared with the formaldehyde concentration.

5.2 Method B

This method is specific for formaldehyde because potential interferants such as acetaldehyde, acetone, benzaldehyde, formic acid, glyoxylic acid and propionaldehyde are either chromatographically separated from formaldehyde or do not react with the post-column reagent.

However, to adapt for various composition in different polymer dispersions, extended chromatography run time is recommended to allow for late-eluting compounds in Method B. Compounds which remain on the column after an analysis have potential interference with the formaldehyde peak in subsequent runs.

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6 Reagents (method A and B)

Unless otherwise stated, use only reagents of recognized analytical grade and only grade 1 water as defined in ISO 3696.

- **6.1** Acetic acid (CH₃CO₂H), glacial.
- 6.2 Ammonium acetate (CH₃CO₂NH₄).
- **6.3 Formaldehyde (HCHO),** mass fraction 37 % solution in water.
- 6.4 2,4-Pentanedione (acetyl acetone) (CH₃COCH₂COCH₃).
- **6.5 Phosphoric acid solution,** 33 mmol/l.

Dissolve 2,3 ml of mass fraction 85 % phosphoric acid (H_3PO_4) in water and dilute to 1 l with water.

6.6 Potassium ferrocyanide trihydrate solution, 36 g/l (Carrez solution I).

Dissolve 36 g of potassium ferrocyanide trihydrate (K_4 Fe(CN)₆·3H₂O) in water and dilute to 1 l with water.