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Paints and varnishes — Determination of preservatives in water-dilutable coating materials —
Part 3: Determination of in-can isothiazolinones with LC-UV and LC-MS

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Foreword

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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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This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 16, *Chemical analysis*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 139, *Paints and varnishes*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

A list of all parts in the ISO 7012 series can be found on the ISO website.

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.

Paints and varnishes — Determination of preservatives in water-dilutable coating materials — Part 3: Determination of in-can isothiazolinones with LC-UV and LC-MS

1 Scope

This document specifies the apparatus and the analytical ~~method~~ methods for determining the content of in-can isothiazolinone preservatives in water-dilutable coating materials or related products.

This document is also applicable ~~for~~to polymer dispersions. ~~This document is generally applicable for n-octyl 4-isothiazolin-3-one (OIT) and 4,5-dichloro-n-octyl 4-isothiazolin-3-one (DCOIT). However, the standard was not validated for these compounds in the interlaboratory test. There are national standards covering these compounds.~~

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 1513, *Paints and varnishes — Examination and preparation of test samples*

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

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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/>

3.1 ready for use

state of a product when it is mixed in accordance with the manufacturer's instructions in the correct proportions and thinned if required using the correct thinners so that it is ready for application by the approved method

[SOURCE: ISO 11890-2:2020, 3.7]

3.2

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water-dilutable coating material

water-reducible coating material

water-based coating material

water-borne coating material

water-thinnable coating material

DEPRECATED: water paint

coating material whose viscosity is reduced by the addition of water

[SOURCE: ISO 4618:2023, 3.272]

3.3

detection limit of instrument

three times the standard deviation of the result obtained in the blank test using a specific instrument

[SOURCE: ISO 8124-5: 2015, 3.5]

3.4

in-can preservative

biocide used to prevent growth of microorganisms during storage of a stock solution of a coating material or *water-based coating material* (3.2)

[SOURCE: ISO 4618:2023, 3.141]

4 Principle

The sample is extracted with suitable dilution solvent using an appropriate homogenization method, such as ultrasonic water bath. The extract is purified by centrifugation, after which the isothiazolinones are identified and quantified by liquid chromatography (LC) in combination with ~~UV/VIS~~ (ultraviolet/visible) (UV/VIS) detection or different mass spectrometry detectors. This includes liquid chromatography-tandem quadrupole mass spectrometry (LC-MS/MS) and liquid chromatography-single quadrupole mass spectrometry (LC-MS).

For the purposes of this document, LC also designates high-performance liquid chromatography (HPLC) or ultra-performance liquid chromatography (e.g. UPLC or UHPLC).

Atmospheric pressure chemical ~~ionisation~~ ionization (APCI) and electrospray ~~ionisation~~ ionization (ESI) have been found suitable. Other ionization techniques and mass analysers with sufficient performance can also be used.

NOTE The difference between the results is expected to be smaller than the reproducibility limits of the methods for a standard water-borne coating sample. In the interlaboratory test according to Annex E, the determination by the LC-UV/VIS method and the LC-MS or LC-MS/MS method turned out to deliver comparable results within the reproducibility limits of the methods, validating the above statement.

This document is generally applicable for n-octyl-4-isothiazolin-3-one (OIT) and 4,5-dichloro-n-octyl-4-isothiazolin-3-one (DCOIT). However, the document was not validated for OIT and DCOIT in the interlaboratory test. There are national standards covering these compounds.

5 Apparatus

5.1 General

The usual laboratory apparatus and, in particular, the following apparatus stated in 5.1 to 5.3 shall be used.

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5.15.2 Equipment for extraction and sample preparation

5.12.1 High speed centrifuge, with an acceleration at a suitable speed.

NOTE A rotation speed of (5_000 - 30_000) revolutions per minute (rpm/r/m) has been found suitable.

5.12.2 Ultrasonic water bath, with a frequency from 35_kHz to 45_kHz.

5.12.3 Analytical balance, capable of weighing to an accuracy of 0,1mg/1 mg.

5.12.4 One-mark volumetric flask, of 10_ml, 25_ml, 50_ml and 100_ml nominal capacity.

5.12.5 Pipettes, suitable single-volume pipettes, graduated pipettes and Pasteur pipettes.

5.12.6 Centrifuge tubes, with a suitable snap/screw cap.

5.12.7 Glass autosampler vials, with a suitable snap-/screw pre-slit cap, 2_ml.

5.12.8 Syringe, minimum volume 2_ml.

5.12.9 Filtration membrane, about 0,22_µm pore size.

5.25.3 LC-UV/VIS system

5.23.1 LC system, equipped with a sample injection system, a solvent pumping system capable of mixing solvents, a sample compartment capable of maintaining required temperature and a temperature-controlled column compartment, a degassing system and data processing software. An LC system that can perform at the flows, pressures, controlled temperatures, sample volumes and other requirements of the document shall be used.

5.23.2 Analytical column; reverse phase C18 particle columns were used to develop this test method. Any column that achieves adequate resolution may be used. The retention times and order of elution may change depending on the column used and shall be monitored.

5.23.3 UV/VIS detector, a UV/VIS detector or optionally a diode array detector (DAD).

5.35.4 LC-MS/MS or LC-MS system

5.34.1 LC system, equipped with a sample injection system, a solvent pumping system capable of mixing solvents, a sample compartment capable of maintaining required temperature and a temperature-controlled column compartment, a degassing system and data processing software. An LC system that can perform at the flows, pressures, controlled temperatures, sample volumes, and other requirements of the document shall be used.

~~5.3.2 Analytical column, reverse phase C18 particle columns were used to develop this test method. Any column that achieves adequate resolution may be used. The retention times and order of elution can change depending on the column used and shall be monitored.~~

5.35.4.2 Analytical column, as specified in 5.3.2.

5.4.3 MS/MS or MS-system, equipped with APCI or ESI. Other ionization techniques and mass analysers with sufficient performance can also be used.

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6 Reagents and materials

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of grade 1 in accordance with ISO 3696. Solvents shall be of a suitable quality for LC-UV/VIS and LC-MS, i.e. LC-MS grade for LC-MS analysis.

6.1 Reference substances: methylisothiazolinone (MIT, CAS RN^{®1} 2682-20-4), 5-chloro-2-methyl-4-isothiazolin-3-one (CMIT, CAS RN[®] 26172-55-4), 1,2-benzisothiazolin-3-one (BIT, CAS RN[®] 2634-33-5).

6.2 Methanol (CAS RN[®] 67-56-1).

6.3 Acetonitrile (CAS RN[®] 75-05-8).

6.4 Formic acid (CAS RN[®] 64-18-6).

6.5 Acetic acid (CAS RN[®] 64-19-7), $\geq 99\%$ mass fraction, optional for better recovery rates.

NOTE The acetic acid is found useful to improve the recovery rate by loosening up the structure of the emulsion paint.

6.6 Ammonium acetate (CAS RN[®] 631-61-8).

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6.7 Precipitating agent (optional for better precipitation when using LC-UV/VIS), e.g. aluminium sulfate $\geq 98\%$, Carrez reagent.

A precipitating agent (e.g. aluminium sulfate, Carrez reagent) can be used to clean up the sample solution after the extraction. In this case, tests shall be carried out to ensure that the use of the precipitating agent does not reduce the recovery rate as compared to the analysis without precipitating agent.

6.8 Dilution solvent, suitable for diluting the sample. It shall have a purity of at least 99% by mass or shall be of known purity, and it shall not contain any substances which interfere with the determination, e.g. causing overlapping peaks in the chromatogram. Methanol and mixture of water and methanol (1:1 by volume) has been found suitable.

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Alternatively, other suitable solvents may also be used. However, it should be ensured that the sample does not agglomerate, which can lead to the inclusion of preservatives and thus to analytical errors.

6.9 Internal Standard (ISTD) for LC-MS method, which should be added at the extraction step and to standard working solutions.

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Isotope labelled internal standards should be used as much as possible.

6.10 Calibration standard solutions

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6.10.1 General

The calibration standard solutions should be prepared in the same dilution solvent as the final sample solution. All standard solutions used in this method shall be prepared as described in 6.10.

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Commercially available standard stock solutions with known content may also be used. If necessary, the content of these solutions shall be determined using certified/traceable standards (see 6.1).

NOTE If commercially available standard stock solution is used, standard working solutions are prepared in one-mark volumetric flask (5.4.4) by diluting the stock solution with dilution solvent (6.8).

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¹ CAS Registry Number[®] is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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6.10.2 Standard stock solutions

Weigh each of the target reference substances (6.1) into a 100-ml one-mark volumetric flask (5.4.2.4) at concentrations that are sufficiently high to allow the preparation of standard working solutions. Fill up to the mark with the dilution solvent (6.8) and let them dissolve completely. Transfer the standard stock solutions to amber-glass flask and seal with glass cap or PTFE-coated cap, ~~seal~~, Seal the bottle neck again with flexible laboratory sealing film. The preparation of standard stock solutions shall be repeated after a time period that shall be specified by the laboratory, after which a degradation can occur.

NOTE Normally, a stock solution can be kept in the refrigerator at 2 °C to 6 °C in the dark for up to 1 month, when stability has been proven, or according to the ~~expire~~expiry date of the producer.

Given the broad applicability of this method and the partly divergent pH-stability of preservatives, analyte mixtures of different composition can be necessary.

6.10.3 Standard working solutions

Prepare standard working solutions of a minimum of five concentration levels from the standard stock solution (6.10.2) by diluting with dilution solvent (6.8). It is possible that they are stable for only a few days. The preparation of standard working solutions shall be repeated after a time period that shall be specified by the laboratory, after which a degradation can occur.

If internal standard is used, a fixed volume of internal standard solution shall be added to the standard working solutions before dilution.

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7 Sampling

Take a representative sample of the product to be tested (or of each product in the case of a multi-coat system) as specified in ISO 15528.

Examine and prepare each sample for testing, as specified in ISO 1513, preparing the final sample for testing in the "ready for use" state.

8 Procedure

8.1 General

Carry out all tests by taking duplicate coating samples as described in Clause 7.

8.2 LC-UV/VIS method

8.2.1 Extraction of LC-UV/VIS

Weigh, to the nearest 0,1-mg, 2-g ~~± ±~~ 0,2-g of the test sample into a 25-ml one-mark volumetric flask (5.4.2.4). Add about 10-ml dilution solvent (6.8) to the one-mark volumetric flasks, shake it in the ultrasonic water bath (5.4.2.2) until it is homogenized (typically 3-min of manual shaking is sufficient), fill up to the mark with dilution solvent (6.8) and shake again until it is homogenized (typically 30-s of manual shaking is sufficient).

~~Another sample mass may also be used, as long as the mass of the sample and the volume of the one-mark volumetric flask change proportionally. Another sample mass may also be used, as long as the mass of the sample and the volume of the one-mark volumetric flask change proportionally.~~ For example, if 4-g ~~± ±~~ 0,4-g of the test sample is used, the volume of the one-mark volumetric flask shall be 50-ml and the dilution solvent for the first ~~homogenisation~~homogenization should be about 20 ml. However, the mass shall not be smaller than 0,5-g.

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NOTE-1 Dead volume is not taken into account in the extraction procedure and further calculations.

Put the one-mark volumetric flask (5.4.2.4) into the ultrasonic water bath (5.4.2.2) and extract at (25±5) °C for 10 min.

Other suitable homogenization and extraction method proved to be equally suitable may also be used. An example of mechanical method for homogenization and extraction is given in Annex D.

Then a suitable aliquot of the sample solution is transferred into a centrifuge tube (5.4.2.6) and centrifuged at a suitable speed (typically 25-000 ~~rpm~~ r/m) for at least 10-min to obtain a transparent solution. Using a filtration membrane (5.4.2.9), withdraw and transfer a suitable aliquot of the solution into a glass autosampler vial (5.4.2.7) for LC analysis.

NOTE-2 Direct injection into LC of suspended matter solution or cloudy solution can contaminate the equipment. The use of a precipitating agent (6.7) is found to be helpful for cleaning up the sample solution after extraction.

NOTE-3 Centrifugation at -5 °C to -1 °C is found helpful to clean the sample and make the filtration easier.

If necessary, prepare a further diluted solution using an appropriate volume of dilution solvent (6.8).

8.2.2 Operation conditions of LC-UV/VIS

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Optimize the operating conditions of the LC-UV/VIS in accordance with the manufacturer's instructions. The appropriate LC-UV/VIS gradient programme for the mobile phase is determined experimentally during method development and validation. An example of typical operating conditions is given in [Annex A-Annex A](#).

8.2.3 Blank

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Treat the blank, i.e. dilution solvent (6.8), in exactly the same manner as the samples. Procedural blanks should preferably be analysed with each batch of samples (a maximum of 20 samples).

8.2.4 Interferences of LC-UV/VIS

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Substances with similar retention times and detection wavelengths can interfere during the LC-UV/VIS determination.

These interferences can lead to incompletely resolved signals or additional signals in the chromatographic pattern. Depending on their levels in the sample, such substances can affect the accuracy and precision of the results.

8.2.5 Calibration of LC-UV/VIS

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The calibration standard solutions are prepared as described in 6.10. Take care to inject the same volume for calibration as for sample measurement.

The initial calibration serves to establish the linear working range of the calibration curve. This calibration is performed when the method is used for the first time and after maintenance and/or repair of the equipment.

The conformity of the calibration shall be checked at the start and at the end of every sample series by a standard working solution (6.10.3). The conformity should additionally be proved after a maximum of 20 samples per series.

The recalibration shall cover the desired working range and be performed using a standard working solution whenever the validity checks show a deviation \rightarrow $\geq 10\%$ from the expected concentration.

The resulting working range for each analyte shall be within the linear domain of the detector. The linear domain of each analyte shall be determined during the evaluation or the validation of the method. The calibration curve shall be established with at least 5 concentration levels. The calibration range should be appropriate to the preservative concentration to be qualified. Thus, it can be necessary to construct more than one calibration graph from the results of calibration measurement.

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The pairs of varieties A_i and C_i obtained from the standard working solutions (6.10.3) are displayed graphically and visually tested for linear dependence. If this is fulfilled, the best-fit line is calculated through linear regression using Formula (1):

$$A_i = a \times C_i + b \quad (1)$$

where

- A_i is the measuring value (signal area) of the analyte i depending on c_i (unit depends on evaluation);
- C_i is the concentration of analyte i in the calibration solution, expressed in microgram per millilitre [$\mu\text{g}/\text{ml}$];
- a is the slope of the calibration curve for analyte i (expressed in $\text{ml}/\mu\text{g}$, if no internal standard is used);
- b is the intercept on the ordinate of the calibration curve, same unit as measuring value— b can have positive and negative values.

8.2.6 Determination by LC-UV/VIS

Set the instrumental parameters as specified in 8.2.2.

Determine isothiazolinones by LC-UV/VIS.

8.3 LC-MS method

8.3.1 Extraction of LC-MS

Weigh, to the nearest 0,1-mg, 2-g \pm 0,2-g of the test sample into a 25-ml one-mark volumetric flask (5.4.2.4), add internal standard (6.9) when needed, and add about 10-ml dilution solvent (6.8) to the one-mark volumetric flasks. Shake the solvent in the ultrasonic water bath until it is homogenized (typically 3-min of manual shaking is sufficient). Fill up to the mark with dilution solvent (6.8) and shake again until it is homogenized (typically 30-s of manual shaking are sufficient).

Another sample mass may also be used, as long as the mass of the sample and the volume of the one-mark volumetric flask change proportionally. Another sample mass may also be used, as long as the mass of the sample and the volume of the one-mark volumetric flask change proportionally. For example, if 4-g \pm 0,4-g of the test sample is used, the volume of the one-mark volumetric flask shall be 50-ml and the dilution solvent for the first homogenisation ~~homogenization~~ should be about 20 ml. However, the mass shall not be smaller than 0,5-g.

NOTE 1 Dead volume is not taken into account in the extraction procedure and further calculations.

Put the one-mark volumetric flask (5.4.2.4) into the ultrasonic water bath (5.4.2.2) and extract at (25 \pm 5) °C for 10 min.

Other suitable homogenization and extraction method proved to be equally suitable may also be used. An example of a mechanical method for homogenization and extraction is given in Annex D.

Then a suitable aliquot of the sample solution is transferred into a centrifuge tube (5.2.6) and centrifuged at a suitable speed (typically 25-000-rpm) for at least 10-min to obtain a transparent solution.

NOTE 2 Centrifugation at -5 °C to -1-°C is found helpful to clean the sample and make the filtration easier.

For most water-dilutable coating material samples, both the APCI combined with further dilution method (see 8.3.2) and the ESI combined with standard addition method (see Annex C) can solve the matrix interferences problem. However, if compounds in a sample are known to be strongly affected by matrix-

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