
Textiles — Determination of certain benzotriazole compounds

Textiles — Détermination de certains composés benzotriazole

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Published in Switzerland

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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 documents 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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 38, *Textiles*.

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.

Introduction

Benzotriazole compounds, which have a phenolic group attached to benzotriazole structure, can absorb the full spectrum of UV light and are mainly used as UV-stabilizer for polymers such as textiles, plastics, polyurethanes and constituent in formulations used for coating of surfaces such as sun-protective clothing and parasols. Certain benzotriazole compounds including UV-320, UV-327, UV-328 and UV-350 are toxic. UV-320 and UV-328 have been identified as PBT-substances, while UV-327 and UV-350 have been identified as vPvB-substances.

Human exposure to certain benzotriazole compounds can occur through diverse sources such as skin contact. A series of studies have shown the occurrence of certain benzotriazole compounds in aquatic, terrestrial, atmospheric environment and as a result of anthropogenic activities. Considering their toxicity on several animal species, minimization of exposure to certain benzotriazole compounds is recognized as important to the preservation of human health. This document provides a method for the determination of certain benzotriazole compounds including UV-320, UV-327, UV-328 and UV-350 in textiles.

The method described in this document is based on the use of liquid chromatography-tandem mass spectrometry (LC-MS/MS), liquid chromatography-diode array detector (LC-DAD) or gas chromatography with mass spectrometry (GC-MS) for textile materials (fibres and fabrics). Further investigations regarding the extraction efficiency of solvents are needed in connection to accessories (for example, plastic buttons) on textile products.

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WARNING — This document calls for the use of substances and/or procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage. The execution of its provisions is entrusted to appropriately qualified and experienced people.

1 Scope

This document specifies a method for determining the amount of certain benzotriazole compounds (UV-320, UV-327, UV-328, UV-350) in textiles by using liquid chromatography-tandem mass spectrometry (LC-MS/MS) or liquid chromatography-diode array detector (LC-DAD) or gas chromatography with mass spectrometry (GC-MS).

The method is applicable to all kinds of textile materials (fibres and fabrics).

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological 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

Extract the benzotriazole compounds from textile specimen by ultrasonic generator with acetonitrile. Determine and identify the benzotriazole compounds in the specimen by liquid chromatography-tandem mass spectrometry (LC-MS/MS) or liquid chromatography-diode array detector (LC-DAD) or gas chromatography with mass spectrometry (GC-MS).

5 Reagents

Unless otherwise specified, use only reagents of recognized analytical grade.

5.1 2-(3-sec-butyl-5-tert-butyl-2-hydroxyphenyl) benzotriazole (UV-350), CAS No. 36437-37-3.

5.2 2-(3,5-di-tert-butyl-2-hydroxyphenyl) benzotriazole (UV-320), CAS No. 3846-71-7.

5.3 2-(3,5-di-tert-amyl-2-hydroxyphenyl) benzotriazole (UV-328), CAS No. 25973-55-1.

5.4 2-(3,5-di-tert-butyl-2-hydroxyphenyl)-5-chlorobenzotriazole (UV-327), CAS No. 3864-99-1.

5.5 Methanol, LC or LC-MS grade.

5.6 Acetonitrile, LC grade.

5.7 Ammonium formate, purity of not less than 97,0 % (mass fraction).

5.8 0,01 mol/l ammonium formate, 0,64 g of ammonium formate ([5.7](#)) is dissolved in 1 000 ml of water.

5.9 Grade 2 water, in accordance with ISO 3696.

6 Apparatus

6.1 Analytical balance, with a resolution of 0,000 1 g and 0,01 g.

6.2 Calibrated volumetric flasks, of capacity 25 ml.

6.3 Glass vials, approximately 25 ml, with tight closure.

6.4 Ultrasonic bath, at about 60 °C.

6.5 Round-bottom flask, 50 ml.

6.6 Water bath and rotary evaporator.

NOTE Other kinds of evaporation apparatus can be used, such as a water bath with a controlled flow of nitrogen or argon over the liquid.

6.7 Organic phase filtration membrane, with 0,22 µm pore size, PTFE.

6.8 Chromatographic equipment

6.8.1 Liquid chromatograph (LC), with tandem mass spectrometer (MS/MS) and electro spray ionization (ESI), or

6.8.2 Liquid chromatograph (LC), with diode array detector (DAD), or

6.8.3 Gas chromatograph (GC), with mass spectrometer (MS).

7 Procedure

7.1 Preparation of standard solutions

7.1.1 Preparation of stock standard solutions

1 000 µg/ml stock standard solutions of benzotriazole compounds ([5.1](#) to [5.4](#)) are separately prepared in methanol ([5.5](#)).

EXAMPLE Weigh 25,0 mg of each benzotriazole compound in a 25 ml volumetric flask ([6.2](#)) and fill the volumetric flask up to the mark with methanol ([5.5](#)) and mix thoroughly to dissolve completely the substance.

The stock standards can be made in a mixed stock. This saves time and effort when preparing calibration solutions.

7.1.2 Preparation of the calibration solutions

From the stock standard solutions, prepare at least five appropriate calibration solutions to establish the calibration curve.

EXAMPLE Calibration solutions of 0,5 µg/ml, 0,8 µg/ml, 1 µg/ml, 1,5 µg/ml, 2 µg/ml are applicable for LC-MS/MS. Calibration solutions of 5 µg/ml, 8 µg/ml, 10 µg/ml, 15 µg/ml, 20 µg/ml are applicable for LC-DAD or GC-MS.

7.2 Preparation of test specimen

Prepare a representative test specimen of the sample. Cut it into small pieces with a maximum dimension less than 5 mm and mix them homogeneously. Weigh $(1,00 \pm 0,01)$ g of the pieces with a balance (6.1) and place them into a glass vial with a tight closure (6.3).

7.3 Extraction procedure

Add 15 ml of acetonitrile (5.6) and extract the pieces at about 60 °C in an ultrasonic bath (6.4) for (15 ± 1) min. Filter and transfer the extract into a 50 ml round-bottom flask (6.5). Add 15 ml of acetonitrile (5.6) to the residue in the glass vial and extract the residue at about 60 °C in an ultrasonic bath for (15 ± 1) min. Filter and merge the extract into the 50 ml round-bottom flask.

The extract obtained is concentrated near to dryness by the evaporator (6.6) in the water bath at 40 °C, and 2 ml of methanol (5.5) is added to dissolve the residue. Filter the solution through an organic phase filtration membrane (6.7). The filtrate is ready for determination of benzotriazole compounds.

In parallel, run a blank to control contaminations.

8 Chromatographic determination

Determine the benzotriazole compounds in the solution (7.3) by LC-MS/MS, LC-DAD or GC-MS (6.8).

Examples of test parameters for LC-MS/MS, LC-DAD and GC-MS are shown in Annexes A, B and C.

When the benzotriazole compounds level is beyond the linear detector response range of the equipment, it is necessary to dilute the specimen liquid properly.

9 Calculation

Calibration curves including the concentration range of each benzotriazole compounds are prepared with at least five calibration points. The calibration curves are plotted by the responses against the concentrations of calibration solutions. Quantify the concentration of each benzotriazole compound by using the calibration curve. The content of each benzotriazole compound is expressed by the mass ratio of benzotriazole compound to test specimen, in % or mg/kg. Calculate the result by using Formula (1) or (2).

$$X_i = \frac{(c_i - c_0) \times V}{m \times 10\,000} \quad (1)$$

where

X_i is the content of the benzotriazole compound, i , in the textile specimen, in %;

C_i is the concentration of the benzotriazole compound, i , in the specimen solution, in µg/ml;

C_0 is the concentration of the benzotriazole compound, i , in the blank solution, in $\mu\text{g/ml}$;

V is the final volume of the specimen solution, in ml;

m is the mass of the test specimen, in g.

$$X_i = \frac{(c_i - c_0) \times V}{m} \quad (2)$$

where

X_i is the content of the benzotriazole compound, i , in the textile specimen, in mg/kg;

C_i is the concentration of the benzotriazole compound, i , in the specimen solution, in $\mu\text{g/ml}$;

C_0 is the concentration of the benzotriazole compound, i , in the blank solution, in $\mu\text{g/ml}$;

V is the final volume of the specimen solution, in ml;

m is the mass of the test specimen, in g.

10 Test report

The test report shall include at least the following information:

- a) a reference to this document, i.e. ISO 24040:2022;
- b) the date of the test;
- c) all details necessary for complete identification of the sample tested;
- d) detection method used by LC-MS/MS, LC-DAD or GC-MS;
- e) the content of each benzotriazole compound;
- f) any deviation from the procedure specified.

Annex A (informative)

Test parameters by LC-MS/MS

As the instrumental equipment of the laboratories can vary, no generally applicable parameters can be provided for chromatographic analyses. The following parameters have been found suitable.

Stationary phase: Zorbax® Eclipse Plus C18¹⁾, 3,5 µm, 2,1 mm × 50 mm;

Eluent 1: Methanol;

Eluent 2: 0,01 mol/l ammonium formate;

Flow rate: 0,3 ml/min;

Gradient:

Time (min)	Eluent 1 (% vol- ume fraction)	Eluent 2 (% vol- ume fraction)
0	88	12
7	90	10
8	100	0
10	100	0
10,5	88	12
12	88	12

Column temperature: 20 °C;

Injection volume: 10 µl;

Detection mode: Multiple reaction monitoring (MRM);

Spray gas: Nitrogen;

Ionization: Electrospray ion source (ESI), positive;

Capillary voltage: 4 000 V;

Gas flow: 12 l/min;

Dry gas temperature: 350 °C;

Nebulizer pressure: 241 kPa;

Typical ions: see [Table A.1](#).

1) Zorbax® Eclipse Plus C18 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

Table A.1 — Typical ions for LC-MS/MS

Benzotriazole compound	MRM, m/z		Declustering potential V	Collision energy eV
	Precursor ion	Product ion		
UV-350 ^b	324	268 ^a	120	19
		212	120	27
UV-320 ^b	324	268 ^a	140	23
		212	140	27
UV-328	352	282 ^a	120	23
		212	120	31
UV-327	358	302 ^a	136	23
		246	136	31

^a Quantification ion.

^b UV-329 cannot be confused with UV-320 and UV-350 (based on different typical ions), although they have the same molecular weight.

The detection limits of LC-MS/MS method for benzotriazole compounds are 1 mg/kg.

Under the given analysis conditions, the chromatograms of benzotriazole compounds are shown in [Figures A.1 to A.3](#).

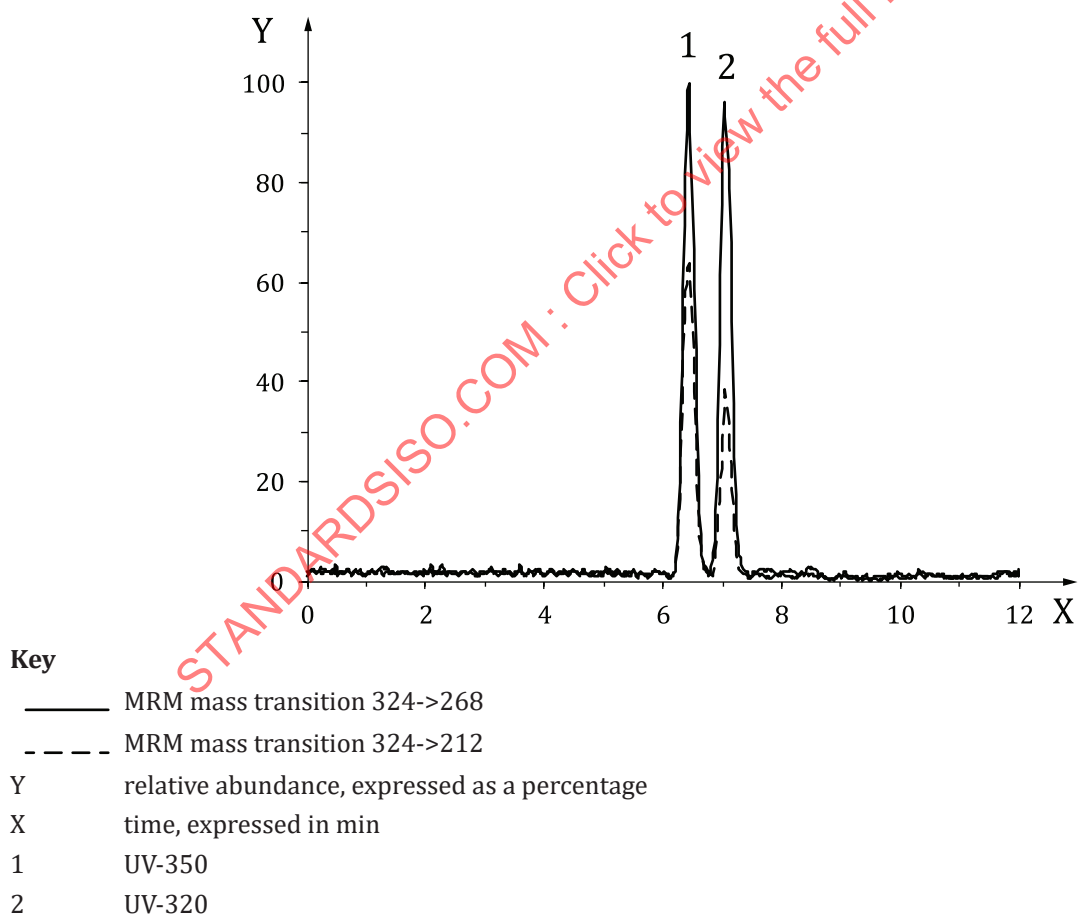


Figure A.1 — LC-MS/MS chromatogram for UV-350 and UV-320

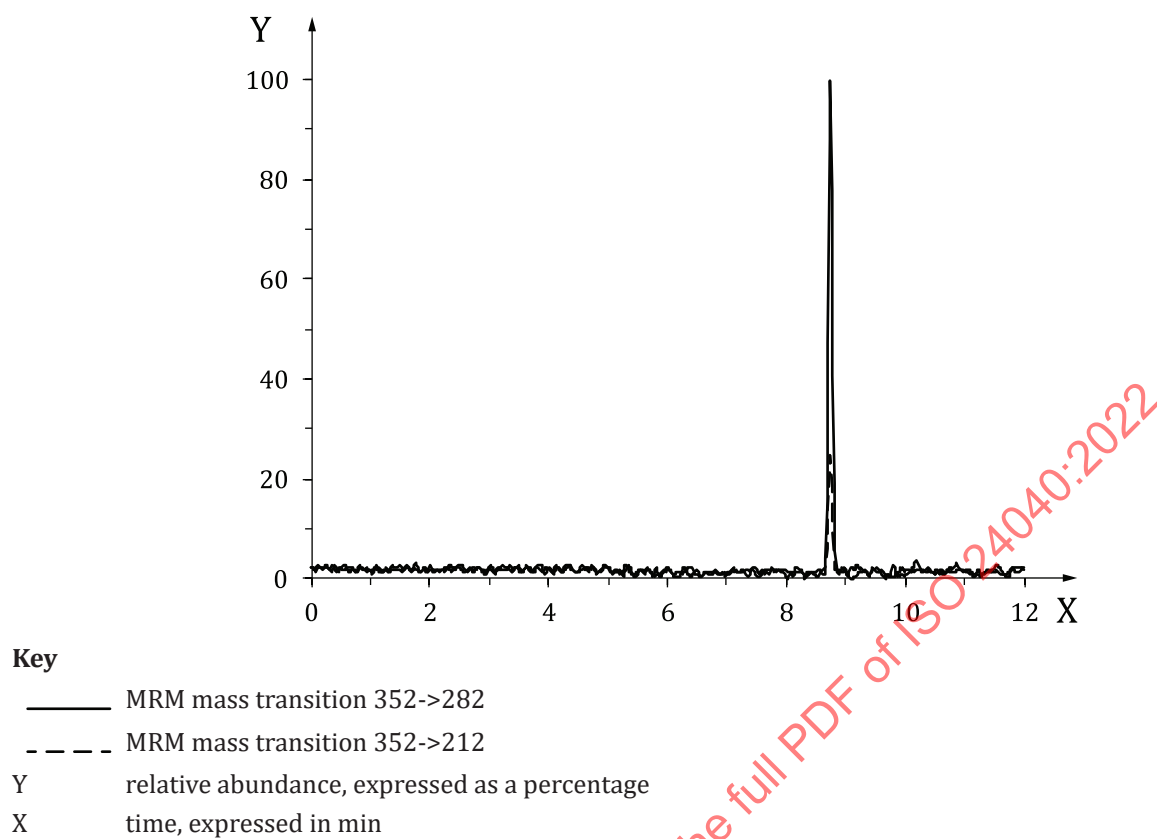


Figure A.2 — LC-MS/MS chromatogram for UV-328

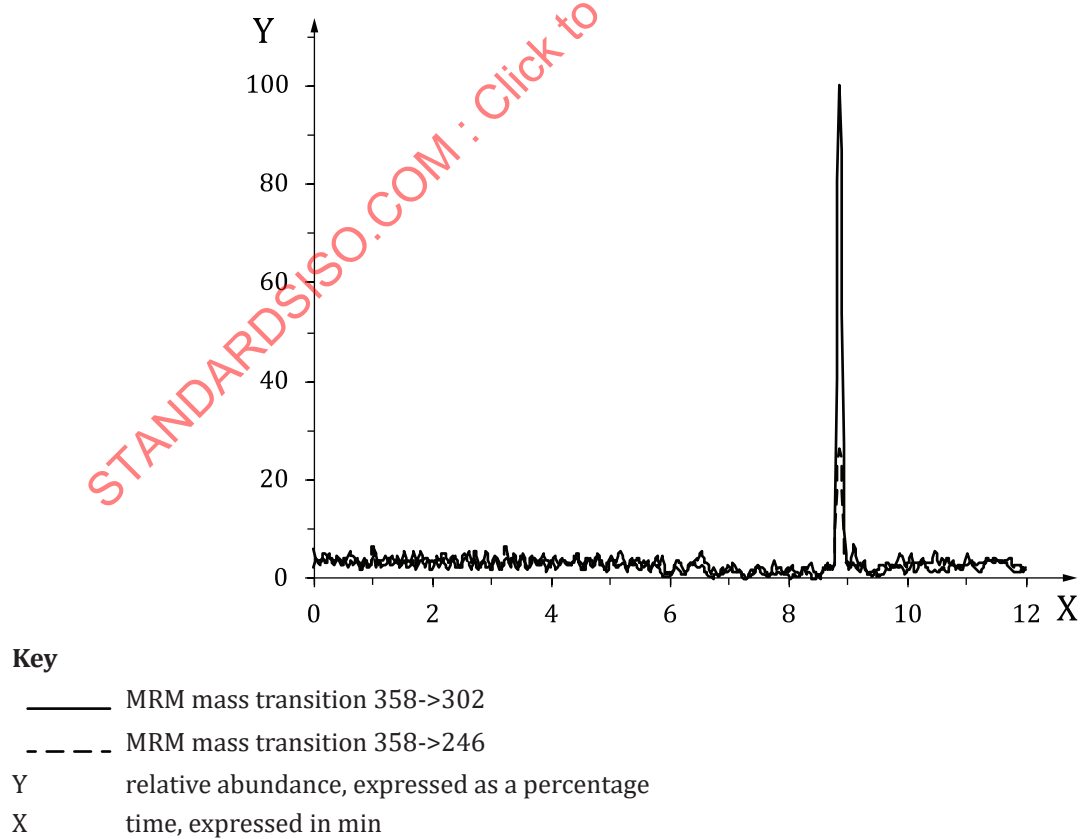


Figure A.3 — LC-MS/MS chromatogram for UV-327

Annex B (informative)

Test parameters by LC-DAD

As the instrumental equipment of the laboratories can vary, no generally applicable parameters can be provided for chromatographic analyses. The following parameters have been found suitable.

Stationary phase: Zorbax® Eclipse XDB C18²⁾, 5 µm, 4,6 mm × 250 mm;

Eluent 1: Methanol;

Eluent 2: 0,01 mol/l ammonium formate;

Gradient:

Time (min)	Eluent 1 (% vol- ume fraction)	Eluent 2 (% vol- ume fraction)
0	80	20
30	100	0
45	100	0
50	80	20
60	80	20

Flow rate: 1,0 ml/min;

Column temperature: 40 °C;

Injection volume: 20 µl;

Detection: DAD, spectrograph, 210 nm to 400 nm;

Quantification: at 305 nm and 345 nm.

The detection limits of LC-DAD method for benzotriazole compounds are 10 mg/kg.

Under the given analysis conditions, the chromatogram of benzotriazole compounds is shown in [Figure B.1](#).

2) Zorbax® Eclipse XDB C18 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.