
**Photography — Processing chemicals —
Specifications for benzotriazole**

*Photographie — Produits chimiques de traitement — Spécifications pour le
benzotriazole*



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.

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.

International Standard ISO 3618 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 3618:1976), which has been technically revised.

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Introduction

0.1 This International Standard is one of a series that establishes criteria of purity for chemicals used in processing photographic materials. General test methods and procedures cited in this International Standard are compiled in parts 1, 4 and 8 of ISO 10349.

This International Standard is intended for use by individuals with a working knowledge of analytical techniques which may not always be the case. Some of the procedures utilize caustic, toxic or otherwise hazardous chemicals. Safe laboratory practice for the handling of chemicals requires the use of safety glasses or goggles, rubber gloves and other protective apparel such as face masks or aprons where appropriate. Normal precautions required in the performance of any chemical procedure are to be exercised at all times but care has been taken to provide warnings for hazardous materials. Hazard warnings designated by a letter enclosed in angle brackets, <> are used as a reminder in those steps detailing handling operations and are defined in ISO 10349-1. More detailed information regarding hazards, handling and use of these chemicals may be available from the manufacturer.

0.2 This International Standard provides chemical and physical requirements for the suitability of a photographic-grade chemical. The tests correlate with undesirable photographic effects. Purity requirements are set as low as possible consistent with these photographic effects. These criteria are considered the minimum requirements necessary to assure sufficient purity for use in photographic processing solutions, except that if the purity of a commonly available grade of chemical exceeds photographic processing requirements and if there is no economic penalty in its use, the purity requirements have been set to take advantage of the availability of the higher-quality material. Every effort has been made to keep the number of requirements to a minimum. Inert impurities are limited to amounts which will not unduly reduce the assay. All tests are performed on samples "as received" to reflect the condition of materials furnished for use. Although the ultimate criterion for suitability of such a chemical is its successful performance in an appropriate use test, the shorter, more economical test methods described in this International Standard are generally adequate.

Assay procedures have been included in all cases where a satisfactory method is available. An effective assay requirement serves not only as a safeguard of chemical purity but also as a valuable complement to the identity test. Identity tests have been included whenever a possibility exists that another chemical or mixture of chemicals could pass the other tests.

All requirements listed in clause 4 are mandatory. The physical appearance of the material and any footnotes are for general information only and are not part of the requirements.

0.3 Efforts have been made to employ tests which are capable of being run in any normally equipped laboratory and, wherever possible, to avoid tests which require highly specialized equipment or techniques. Instrumental methods have been specified only as alternative methods or alone in those cases where no other satisfactory method is available.

Over the past few years, great improvements have been made in instrumentation for various analyses. Where such techniques have equivalent or greater precision, they may be used in place of the tests described in this International Standard. Correlation of such alternative procedures with the given method is the responsibility of the user. In case of disagreement in results, the method called for in the specification shall prevail. Where a requirement states "to pass test", however, alternative methods shall not be used.

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Photography — Processing chemicals — Specifications for benzotriazole

1 Scope

This International Standard establishes criteria for the purity of photographic-grade benzotriazole and specifies the tests to be used to determine the purity.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*.

ISO 10349-1:1992, *Photography — Photographic-grade chemicals — Test methods — Part 1: General*.

ISO 10349-4:1992, *Photography — Photographic-grade chemicals — Test methods — Part 4: Determination of residue after ignition*.

ISO 10349-8:1992, *Photography — Photographic-grade chemicals — Test methods — Part 8: Determination of volatile matter*.

3 General

3.1 Physical properties

Benzotriazole, $C_6H_5N_3$, comes in the form of white to light-tan needles or powder. It has a relative molecular mass of 119,13.

3.2 Hazardous properties

Benzotriazole is not hazardous when handled with normal precautions.

3.3 Storage

Benzotriazole requires no special storage conditions other than a cool, dry environment.

4 Requirements

A summary of the requirements is shown in table 1.

Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay	98 % (m/m) min. 101 % (m/m) max.	7.1	ISO 3618
Identity			ISO 3618
Melting point	94 °C to 99 °C	7.2.1	
Mixed melting point	Not lower than sample or standard	7.2.1	
Infrared spectrum	Match reference spectrum	7.2.2	
Residue after ignition	0,5 % (m/m) max.	7.3	ISO 10349-4
Volatile matter	0,5 % (m/m) max.	7.4	ISO 10349-8
Appearance of solution	Clear and colourless	7.5	ISO 3618
NOTE — m/m = mass/mass			

5 Reagents and glassware

All reagents, materials and glassware shall conform to the requirements specified in ISO 10349-1 unless otherwise noted. The hazard warning symbols used as a reminder in those steps detailing handling operations are defined in ISO 10349-1. These symbols are used to provide information to the user and are not meant to provide conformance with hazardous labelling requirements as these vary from country to country.

6 Sampling

See ISO 10349-1.

7 Test methods

7.1 Assay

7.1.1 Specification

Content of benzotriazole ($C_6H_5N_3$) shall be between 98,0 % (m/m) and 101 % (m/m).

7.1.2 Reagents

7.1.2.1 Ammonium hydroxide, NH_4OH (1 + 1) (DANGER: <C>)^{1) 2)}.

7.1.2.2 Silver nitrate, $AgNO_3$, 100 g/l³⁾.

7.1.3 Apparatus

7.1.3.1 Sintered glass crucible, porosity P40 (pore size index 16 μm to 40 μm).

7.1.4 Procedure

Weigh, to the nearest 0,000 1 g, a test portion of about 0,25 g and dissolve it in 10 ml of the ammonium hydroxide (7.1.2.1) (<C>). Add 50 ml of water and heat to 60 °C to 90 °C. Add slowly, with stirring, 10 ml of the silver nitrate (7.1.2.2). Digest at 60 °C for 15 min, cool and filter the solution through the previously dried and weighed sintered glass crucible (7.1.3.1). Wash the precipitate with six 10 ml portions of water and dry at 105 °C to constant mass.

7.1.5 Expression of results

The assay, expressed as a percentage by mass, for benzotriazole ($C_6H_5N_3$), is given by

$$52,70 \frac{m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the precipitate;

52,70 is the conversion factor for the relative molecular mass of benzotriazole (119) equivalent to the relative molecular mass of the benzotriazole silver salt (226) $\times 100$ (for percentage).

7.2 Identity tests

7.2.1 Melting point

7.2.1.1 Specifications

The melting point shall be from 94 °C to 99 °C.

A mixed melting point shall be from 94 °C to 99 °C and shall not be less than either that of the sample or the standard.

7.2.1.2 Apparatus

7.2.1.2.1 Capillary-tube melting point apparatus, complete with thermometer for the range 50 °C to 100 °C.

7.2.1.3 Procedure

Prepare three capillary tubes containing:

- the sample to be tested;
- a sample known to be benzotriazole;
- a finely ground mixture of a) and b) mixed in equal proportions.

Identify the tubes and attach them to the thermometer. Heat the apparatus (7.2.1.2.1) to about 85 °C. Insert the thermometer with the samples attached, and thereafter heat at a constant rate of 2 °C/min. Note the melting point of each sample as indicated by the first appearance of liquefaction.

7.2.2 Infrared spectrum

7.2.2.1 Specification

To pass test.

1) Hazard warning codes are defined in ISO 10349-1:1992, clause 4.

3) This solution can be prepared from concentrated ammonium hydroxide solution, $\rho \approx 0,91$ g/ml (DANGER: <C>).

3) This solution can be prepared from solid silver nitrate (DANGER: <C>).

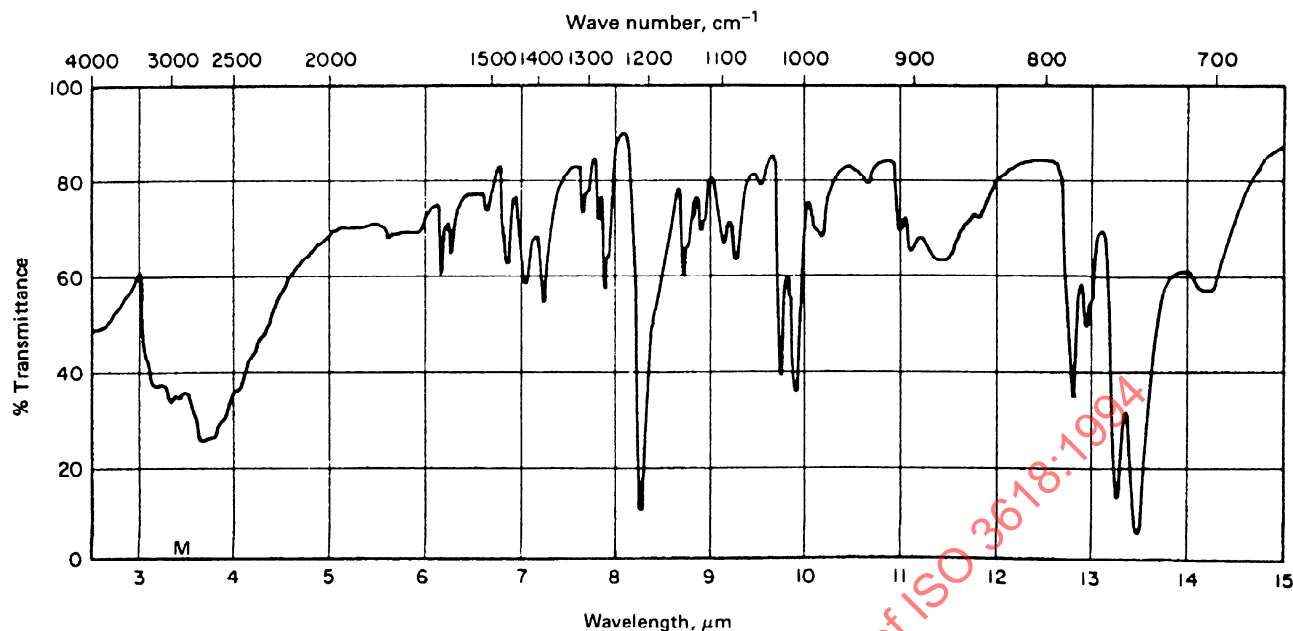


Figure 1 — Reference infrared spectrum of benzotriazole (KBr plate)

The infrared absorption curve shall be essentially the same as that of the reference spectrum (figure 1).

This optional recommendation is supplementary to that of 7.2.1.

7.2.2.2 Apparatus

7.2.2.2.1 Test sieve, of 63 μm aperture size, conforming to ISO 565.

7.2.2.2.2 Infrared spectrometer, equipped for the 2 μm to 16 μm regions, and accessory equipment for using potassium bromide plates or mineral oil mull.

7.2.2.3 Procedure

Grind about 1 g of the sample to a homogeneous fine powder and prepare a 0,5 % (*m/m*) mixture of the sample in finely ground potassium bromide. Grind together thoroughly to pass through the test sieve (7.2.2.2.1). Prepare a pressed plate of the mixture containing 0,13 g to 0,16 g of the mixture per square centimetre. Record the infrared spectrum from 2 μm to 16 μm . Compare with the reference spectrum given in figure 1.

NOTE 1 As an alternative procedure, the sample may be ground and dispersed in mineral oil. It will then be necessary to take into account the absorption bands of the oil.

7.3 Residue after ignition

7.3.1 Specification

Maximum residue after ignition shall be 0,5 % (*m/m*).

7.3.2 Procedure

Determine the percentage residue after ignition in accordance with ISO 10349-4. Weigh 2,0 g of the test sample and ignite in a platinum crucible (600 $^{\circ}\text{C} \pm 50$ $^{\circ}\text{C}$, 4 h, 0,000 1 g)⁴.

7.4 Volatile matter

7.4.1 Specification

Maximum percentage of volatile matter shall be 0,5 % (*m/m*).

7.4.2 Procedure

Determine the percentage of volatile matter at 70 $^{\circ}\text{C}$ in accordance with ISO 10349-8. Weigh 2,0 g of the test sample and dry in a weighing bottle (70 $^{\circ}\text{C}$, 1 h, 0,000 1 g)⁴.

7.5 Appearance of solution

7.5.1 Specification

The solution shall be clear and colourless.

7.5.2 Procedure

Prepare a solution of 10 g $\pm$ 0,5 g of the sample in 1 litre of water. Examine at 50 $^{\circ}\text{C}$ for clarity and colour.

4) The notation system used for the drying procedure is described in ISO 10349-1:1992, clause 1.

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