INTERNATIONAL STANDARD 3618

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Photographic grade benzotriazole — Specification

Benzotriazole de qualité photographique — Spécifications

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FOREWORD

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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.

International Standard ISO 3618 was drawn up by Technical Committee ISO/TC 42, *Photography*, and circulated to the Member Bodies in September 1974.

It has been approved by the Member Bodies of the following countries :

Australia	Germany	Sweden
Austria	Italy	Turkey
Belgium	Japan	United Kingdom
Bulgaria	Mexico	U.S.A.
Canada	South Africa, Rep. of	U.S.S.R.
France	Spain	Yugoslavia

No Member Body expressed disapproval of the document.

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Photographic grade benzotriazole — Specification

0 INTRODUCTION

This International Standard is one of a series of specifications for photographic grade chemicals which are commonly used in the processing of sensitized photographic materials. These specifications have been prepared to establish criteria of purity which will provide a practical and economical grade and prevent possible faulty processing which might be caused by chemicals of inferior quality, and to furnish manufacturers, suppliers, and processors with reliable and readily available specifications for photographic chemicals of satisfactory quality.

Photographic grade chemicals are those which meet the requirements specified in the appropriate International Standards. These specifications set out purity standards and state the limiting concentrations and test methods for certain inert or photographically harmful impurities that may be present.

Originally these specifications were based on known requirements for black-and-white photographic processing, but increased attention has been paid to the requirements of colour processing. Experience to date indicates that chemicals meeting these specifications are satisfactory for colour processes in general use.

0.1 Specification requirements

These specifications set out chemical and physical requirements. While it is recognized that the ultimate criterion of the quality of a photographic chemical is its successful performance in a photographic test, present knowledge indicates that, from a practical standpoint, chemical and physical methods of testing are generally adequate. The photographic industry has accumulated a comprehensive collection of such chemical tests for impurities. These tests, which correlate with objectionable photographic effects, have been drawn upon in the formulation of these specifications. Chemical tests are generally more sensitive, less variable, and less costly than photographic tests.

Purity requirements have been set as low as possible, consistent with the objectives mentioned. If, however, 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 higher-quality materials.

Every effort has been made to keep the number of requirements in each specification to a minimum. The requirements generally include only those photographically harmful impurities which, through experience, are likely to be present. Inert impurities are limited to amounts which will not unduly reduce the assay.

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. All assays are intended to be made on undried samples in view of the fact that photographic processing chemicals are normally used "as received".

Identity tests have been included in the specifications wherever a possibility exists that another chemical or a mixture of chemicals could pass the other tests.

All requirements listed in clause 3 of each specification are mandatory. The physical appearance of the material and any footnotes are for general information only and are not part of the requirements.

0.2 Selection of test methods

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.

While the test methods set out in the specifications are recommended, the use of other equally reliable methods is allowed. 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.

0.3 Reagents

An effort has been made to minimize the number of reagents employed in this series of specifications. The methods of preparation and of standardization have been included in all cases where these are not common, or where a preferred method is desirable.

Details of reagent preparation and standardization are included in each specification in which the reagent is called for so that each specification shall be self-sufficient.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the purity requirements of, and test methods for, photographic grade benzotriazole.

2 CHARACTERISTICS

Benzotriazole is in the form of white to light tan needles or powder, of chemical formula $C_6H_5N_3$ and relative molar mass 119,0.

3 REQUIREMENTS

3.1 Assay

The assay shall be not lower than 98 % (*m/m*) and not greater than 101 % (*m/m*), expressed as $C_6H_5N_3$, when determined by the method described in 4.1.

3.2 Appearance of solution

An aqueous solution shall be clear and colourless when prepared and examined by the method described in 4.2.

3.3 Volatile matter at 70 °C

The volatile matter at 70 °C shall be not greater than 0,5 % (*m/m*), when determined by the method described in 4.3.

3.4 Residue after ignition

The residue after ignition shall be not greater than 0,5 % (*m/m*), when determined by the method described in 4.4.

3.5 Identity

3.5.1 *Melting point*

The melting point shall be not lower than 94 °C and not higher than 99 °C, when determined by the method described in 4.5.1.

3.5.2 *Mixed melting point*

The melting point of the mixture of sample and standard shall be not lower than the melting point of either the sample or the standard, when determined by the method described in 4.5.1.

3.5.3 *Infra-red spectrum*

The infra-red absorption curve shall be essentially the same as that of the reference spectrum (see the figure), when determined by the method described in 4.5.2.

This requirement is an optional identity requirement supplementary to those of 3.5.1 and 3.5.2.

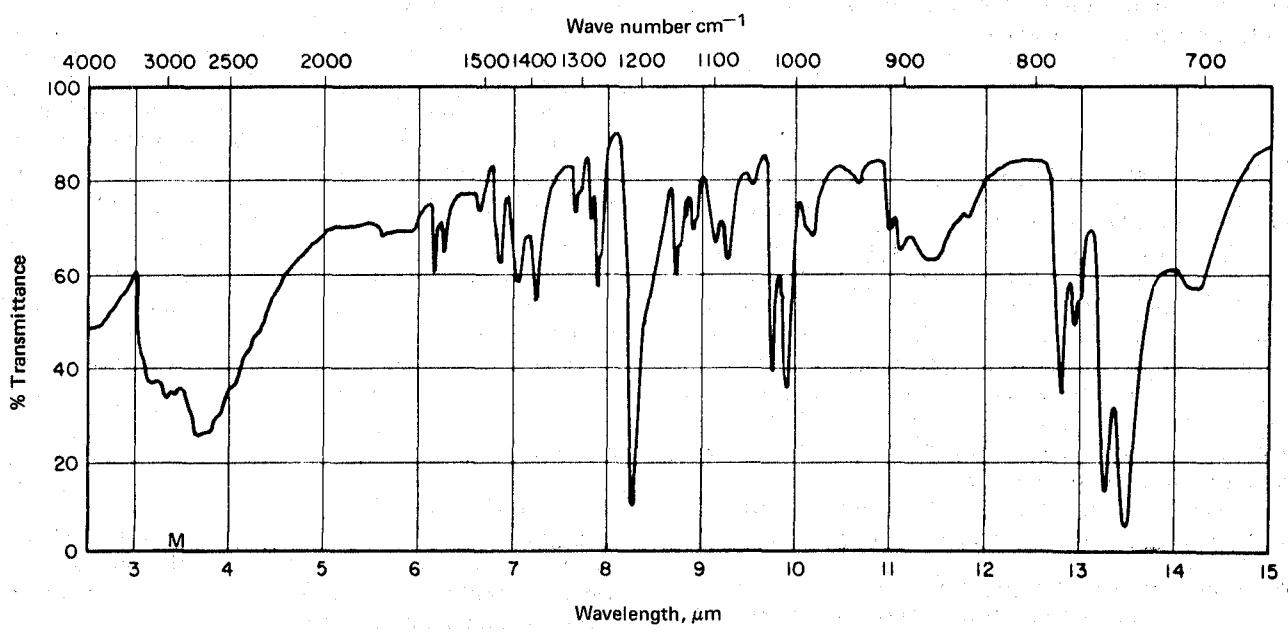

FIGURE — **Reference infra-red spectrum of benzotriazole**
(KBr plate)

4 TEST METHODS

Reagents used in the tests shall be recognized reagent grade chemicals normally used for careful analytical work. In all the directions the acids and ammonia solution referred to shall be of full strength unless dilution is specified. Dilution is specified in terms of molar concentration (molarity)[1] when standardization of the reagent is required. When dilution is indicated as $(1 + x)$, it means that 1 volume of the reagent or strong solution is added to x volumes of distilled water.

Distilled water, or water otherwise produced of at least equal purity, shall be used whenever water is required.

4.1 Assay

4.1.1 *Reagents*

4.1.1.1 Ammonia solution, approximately 100 g/l.

Dilute ammonia solution, ρ 0,91 g/ml (1 + 1).

4.1.1.2 Silver nitrate solution, 100 g/l.

4.1.2 *Apparatus*

Ordinary laboratory apparatus and

4.1.2.1 Sintered glass crucible, porosity P 40 (pore size index 16 – 40 μm).

4.1.3 *Procedure*

Weigh, to the nearest 0,001 g, a test portion of about 0,25 g of the laboratory sample and dissolve in 10 ml of the ammonia solution (4.1.1.1). Add 50 ml of water and heat to between 60 and 90 °C. Add slowly, with stirring, 10 ml of the silver nitrate solution (4.1.1.2), digest at 60 °C for 15 min, cool and filter through the previously dried and weighed sintered glass crucible (4.1.2.1). Wash the precipitate with six 10 ml portions of water, and dry at 105 °C to constant mass.

4.1.4 *Calculation*

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

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

where

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

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

4.2 Appearance of solution test

Prepare a 10 g/l solution of the laboratory sample and examine at 50 °C for clarity and colour.

4.3 Determination of volatile matter at 70 °C

4.3.1 *Procedure*

Weigh, to the nearest 0,01 g, a test portion of about 2 g of the laboratory sample into a previously weighed low-form, glass-stoppered weighing bottle. Dry at 70 °C for 5 h, cool in a desiccator and weigh to the nearest 1 mg.

4.3.2 *Calculation*

Volatile matter at 70 °C is given, as a percentage by mass, by the formula

$$\frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the weighing bottle;

m_2 is the mass, in grams, of the bottle and sample before drying;

m_3 is the mass, in grams, of the bottle and sample after drying.

4.4 Determination of residue after ignition

4.4.1 *Apparatus*

Ordinary laboratory apparatus and

4.4.1.1 Platinum crucible.

4.4.1.2 Muffle furnace, capable of being controlled at 600 ± 50 °C.

4.4.2 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of about 2 g of the laboratory sample into the previously weighed platinum crucible (4.4.1.1), and heat carefully. Finally, ignite the residue in the furnace (4.4.1.2), controlled at 600 ± 50 °C, for 4 h. Cool in a desiccator and weigh the crucible and contents to the nearest 1 mg.

4.4.3 *Calculation*

Residue after ignition, expressed as a percentage by mass, is given by the formula

$$\frac{m_3 - m_1}{m_2 - m_1} \times 100$$

where

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

m_2 is the mass, in grams, of the crucible and test portion;

m_3 is the mass, in grams, of the crucible and residue.

1) 1 mol/l = 1 kmol/m^3 = 1 mol/dm^3 = 1 M

4.5 Identity tests

4.5.1 *Melting point tests*

4.5.1.1 Apparatus

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

4.5.1.2 Procedure

Prepare three capillary tubes, containing :

1) the sample to be tested;

2) a sample known to be benzotriazole; and

3) a finely ground mixture of 1) and 2) mixed in the ratio 1 : 1.

Identify the tubes and attach them to the thermometer. Heat the apparatus (4.5.1.1) to about 85 °C, insert the thermometer with 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.

4.5.2 *Infra-red identity test*

4.5.2.1 Apparatus

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

4.5.2.1.2 Infra-red spectrophotometer, equipped for the 2 to 16 μm regions and accessory equipment for using potassium bromide plates or mineral oil mull.

4.5.2.2 Procedure

Grind about 1 g of the laboratory 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 the test sieve (4.5.2.1.1). Prepare a pressed plate of the mixture containing 0,13 to 0,16 g of the mixture per square centimetre. Record the infra-red spectrum from 2 to 16 μm. Compare with the reference spectrum given in the figure.

NOTE — *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.