INTERNATIONAL STANDARD

ISO 3299

Second edition
1994-11-01

Photography — Processing chemicals — Specifications for 1-phenyl-3-pyrazolidinone

Photographie — Produits chimiques de traitement — Spécifications pour la phényl-1 pyrazolidinone-3

Reference number
ISO 3299:1994(E)

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 3299 was prepared by Technical Committee ISO/TC 42, *Photography.*

This second edition cancels and replaces the first edition (ISO 3299:1976) 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, 5 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.

Photography — Processing chemicals — Specifications for 1-phenyl-3-pyrazolidinone

1 Scope

This International Standard establishes criteria for the purity of photographic-grade 1-phenyl-3-pyrazolidinone (phenidone) and specifies the test methods to be used to determine the purity.

2 Normative references

The following International 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 565:1983, *Test sieves — Woven metal wire cloth, perforated 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 10349-5:1992, *Photography — Photographic-grade chemicals — Test methods — Part 5: Determination of heavy metals and iron content.*

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

3 General

3.1 Physical properties

1-Phenyl-3-pyrazolidinone, $C_9H_{10}N_2O$, exists as a pale cream to white powder, free from aggregates or large crystals. It has a relative molecular mass of 162,20.

3.2 Hazardous properties

1-Phenyl-3-pyrazolidinone is not hazardous when handled with normal precautions.

3.3 Handling and storage

1-Phenyl-3-pyrazolidinone shall be stored in a closed container at room temperature.

4 Requirements

A summary of the requirements is shown in table 1.

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.

Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay	98,5 % (*m/m*) min.	7.1	ISO 3299
Identity			
melting point	119 °C to 122 °C	7.2.1	ISO 3299
mixed melting point	Not lower than sample or standard	7.2.1	
infrared spectrum	Match reference spectrum	7.2.2	
Residue after ignition	0,10 % (*m/m*) max.	7.3	ISO 10349-4
Heavy metals (as Pb)	0,002 % (*m/m*) max.	7.4	ISO 10349-5
Iron (Fe)	0,005 % (*m/m*) max.	7.5	ISO 10349-5
Volatile matter	0,10 % (*m/m*) max.	7.6	ISO 10349-8
Solubility in alkaline sulfite solution	Clear and colourless or slight pink	7.7	ISO 3299
Matter insoluble in chloroform (optional)	0,10 % (*m/m*) max.	7.8	ISO 3299
NOTE — *m/m* = mass/mass			

7 Test methods

7.1 Assay

7.1.1. Specification

Content of 1-phenyl-3-pyrazolidinone shall be 98,5 % (*m/m*) min.

7.1.2 Reagents

7.1.2.1 Acetic acid, CH_3COOH, glacial (DANGER: <C><B>)[1].

7.1.2.2 Acetic acid, approximately 2 mol/l.

Dilute 120 g of glacial acetic acid (7.1.2.1) (<C><B>) to 1 000 ml.

7.1.2.3 Acetate buffer solution, pH 4.

Dissolve 55 g of sodium acetate trihydrate in about 200 ml of water. Then add 92 ml of the 2 mol/l acetic acid solution (7.1.2.2) and dilute to 1 litre with water.

7.1.2.4 Potassium iodide, KI.

7.1.2.5 Iodine solution, I_2, standard volumetric solution of 0,05 mol/l (12,7 g/l)[2].

7.1.2.6 Sodium thiosulfate, $Na_2S_2O_3$, standard volumetric solution of 0,100 mol/l (15,8 g/l)[2].

7.1.2.7 Salicylic acid, HOC_6H_4COOH, 1 % (10 g/l).

7.1.2.8 Starch indicator solution, 5 g/l.

Stir 5 g of soluble starch into 100 ml of the salicylic acid (7.1.2.7). Add 300 ml to 400 ml of boiling water until the starch dissolves and finally dilute to 1 litre with water.

7.1.3 Apparatus

7.1.3.1 Burette, of 50 ml capacity.

7.1.3.2 Pipette, of 25 ml capacity.

7.1.4 Procedure

Weigh, to the nearest 0,001 g, a test portion of about 0,15 g and transfer to a conical flask. Add about 70 ml of warm water (50 °C to 60 °C) and dissolve. Cool to room temperature and add 25 ml of the acetate buffer solution (7.1.2.3). Then using a pipette (7.1.3.2), deliver 25 ml of the iodine solution (7.1.2.5) into a flask and back titrate the excess iodine at once with the sodium thiosulfate (7.1.2.6) using the starch

1) Hazard warning codes are defined in ISO 10349-1.

2) Commercially available analysed reagent solutions are recommended. If solutions are to be prepared, see any quantitative analytical chemistry text.

indicator (7.1.2.8). Carry out a blank titration in the same manner omitting the sample. The total elapsed time from the start of the addition of iodine solution to the completion of the titration should not exceed 3 min. Undue delay will result in erroneously high assay values.

7.1.5 Expression of results

The assay, expressed as a percentage by mass, for 1-phenyl-3-pyrazolidinone, is given by

$$8{,}11\ c(V_1-V_2)/m$$

where

c is the actual concentration, expressed in moles per litre, of the sodium thiosulfate (7.1.2.6);

V_1 is the volume, in millilitres, of the sodium thiosulfate used for the sample titration (7.1.4);

V_2 is the volume, in millilitres, of the sodium thiosulfate solution used for the blank titration (7.1.4);

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

8,11 is the conversion factor obtained from the mass of 1-phenyl-3-pyrazolidinone equivalent to 1 mole of iodine (i.e. 81,1) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

7.2 Identity tests

7.2.1 Melting point

7.2.1.1 Specification

The melting point shall be from 119 °C to 122 °C.

A mixed melting point shall be from 119 °C to 122 °C and shall not be lower than either 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 100 °C to 200 °C.

7.2.1.3 Procedure

Prepare three capillary tubes containing:

a) the sample to be tested;

b) a sample known to be 1-phenyl-3-pyrazolidinone;

c) 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 100 °C. Insert the thermometer with the samples attached, and thereafter heat at a constant rate of 1 °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

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, 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 0,10 % (*m/m*).

7.3.2 Procedure

Determine the percentage residue after ignition in accordance with ISO 10349-4. Use a test portion of about 5,0 g and ignite in a platinum crucible (600 °C ± 50 °C, 4 h, 0,000 1 g)[3]. Retain this residue for the heavy metals and iron content tests in 7.4 and 7.5.

3) The notation system used for the drying process procedure is given in ISO 10349-1.

7.4 Heavy metals content

7.4.1 Specification

Maximum content of heavy metals shall be 0,002 % (*m/m*).

7.4.2 Procedure

NOTE 2 The standard for the iron test (7.5) is prepared in the same way as the heavy metals standard.

Determine the percentage of heavy metals in accordance with ISO 10349-5. Use a test portion of the residue from the ignition test (7.3.2) corresponding to 2 g of the sample prepared in accordance with ISO 10349-5:1992, 7.1 (i.e. 10 ml of the 25 ml residue solution). Use 4 ml of heavy metals standard prepared in accordance with ISO 10349-5:1992, 8.1.1.

7.5 Iron content

7.5.1 Specification

Maximum content of iron shall be 0,005 % (*m/m*).

7.5.2 Procedure

Determine the percentage of iron in accordance with ISO 10349-5. Use a test portion of the residue from the ignition test (7.3.2) corresponding to 2 g of the sample prepared in accordance with ISO 10349-5:1992, 7.1 (i.e. 10 ml of the 25 ml residue solution). Use 10 ml of iron standard prepared in accordance with ISO 10349-5:1992, 8.1.1.

7.6 Volatile matter

7.6.1 Specification

Maximum percentage of volatile matter shall be 0,10 % (*m/m*).

7.6.2 Procedure

Determine the percentage of volatile matter at 65 °C in accordance with ISO 10349-8. Use a test portion of 10 g, weighed to the nearest 0,1 g into a dry glass-stoppered weighing bottle (65 °C, 4 h, 0,000 1 g)[3].

7.7 Solubility in alkaline sulfite solution

7.7.1 Specification

To pass test.

7.7.2 Reagents

7.7.2.1 Sodium carbonate, anhydrous, Na_2CO_3.

7.7.2.2 Sodium sulfite, anhydrous, Na_2CO_3.

7.7.3 Procedure

Prepare a solution of 25 g of sodium carbonate (7.7.2.1) and 25 g of sodium sulfite (7.7.2.2) in 1 litre of water. Dissolve a test portion of 0,5 g in this solution. Observe the solution for colour and clarity. The solution shall be clear and colourless or slightly pink.

7.8 Matter insoluble in chloroform (optional)[4]

7.8.1 Specification

Maximum content of matter insoluble in chloroform shall be 0,10 % (*m/m*)

7.8.2 Reagents

7.8.2.1 Chloroform, $CHCl_3$ (DANGER:<B><S>).

7.8.3 Apparatus

7.8.3.1 Sintered glass crucible, medium porosity.

7.8.4 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 5 g and dissolve it in 100 ml of chloroform (<B><S>). If any undissolved material remains, filter through a weighed crucible (7.8.3.1) and wash with 25 ml of chloroform. Dry at 105 °C for 4 h. Cool in a desiccator and weigh to 0,001 g.

7.8.5 Expression of results

The matter insoluble in chloroform, expressed as a percentage by mass, is given by

$$100\,(m_1 - m_2)/m_0$$

where

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

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

m_2 is the mass, in grams, of the crucible;

100 is the factor for percentage.

4) Concern over the use of halogenated hydrocarbons as solvents has resulted in a request for alternatives to the use of chloroform in this test procedure. Other solvents have not yet been evaluated and there is no information available on the applicability of other solvents in this test procedure. This is why this part of the specification is optional.

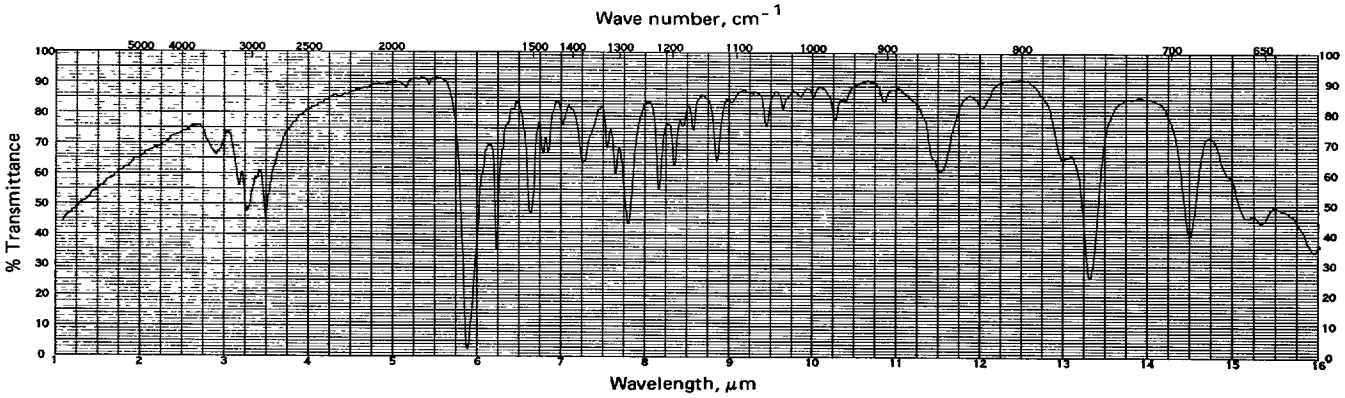

Figure 1 — Reference infrared spectrum of 1-phenyl-3-pyrazolidinone (KBr plate)

ICS 37.040.30

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