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# INTERNATIONAL STANDARD



# 3299

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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## Photographic grade 1-phenyl-3-pyrazolidinone (Phenidone) — Specification

*Phényl-1 pyrazolidinone-3 (Phénidone) de qualité photographique — Spécifications*

First edition — 1976-05-01

**iTeh STANDARD PREVIEW**  
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ISO 3299:1976

<https://standards.iteh.ai/catalog/standards/sist/efddc579-73e-45a7-952a-66a268055949/iso-3299-1976>

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UDC 771.7 : 547.775.004.11

Ref. No. ISO 3299-1976 (E)

**Descriptors** : photographic materials, phenidone, materials specifications, tests.

Price based on 5 pages

## FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

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 3299 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	Spain
Austria	Italy	Turkey
Belgium	Japan	United Kingdom
Bulgaria	Mexico	U.S.A.
Canada	Netherlands	U.S.S.R.
France	South Africa, Rep. of	Yugoslavia

No Member Body expressed disapproval of the document.

# Photographic grade 1-phenyl-3-pyrazolidinone (Phenidone) — 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 1-phenyl-3-pyrazolidinone.

NOTE—These test methods are generally adequate to ensure satisfactory materials but, under certain conditions, some samples may give high fog. In such cases, a photographic test should be made under conditions of intended use.

## 2 CHARACTERISTICS

1-Phenyl-3-pyrazolidinone is a pale cream to white powder, free from aggregates or large crystals, of chemical formula  $C_9H_{10}N_2O$  and relative molar mass 162.

## 3 REQUIREMENTS

### 3.1 Assay

The assay shall be not less than 98,5% (*m/m*), when determined by the method described in 4.1.

### 3.2 Alkaline sulphite solution test

The test solution shall be clear, but a pink coloration is permissible, when examined by the procedure described in 4.2.

### 3.3 Matter insoluble in chloroform

The matter insoluble in chloroform shall be not greater than 0,10% (*m/m*), when determined by the method described in 4.3.

### 3.4 Volatile matter at 65 °C

The volatile matter at 65 °C shall be not greater than 0,10% (*m/m*), when determined by the method described in 4.4.

### 3.5 Residue after ignition

The residue after ignition shall be not greater than 0,10% (*m/m*), when determined by the method described in 4.5.

### 3.6 Heavy metals content

The heavy metals content, expressed as lead (Pb), shall be not greater than 20 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.6, when the colour produced in the test solution shall be not greater than that produced in the control solution.

### 3.7 Iron content

The iron content, expressed as iron (Fe), shall be not greater than 50 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.7, when the colour produced in the test solution shall be not greater than that produced in the control solution.

### 3.8 Identity

#### 3.8.1 Melting point

The melting point shall be not lower than 119 °C and not higher than 122 °C, when determined by the method described in 4.8.1.

#### 3.8.2 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 4.8.1.

#### 3.8.3 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 4.8.2.

This requirement is an optional identity requirement supplementary to those of 3.8.1 and 3.8.2.

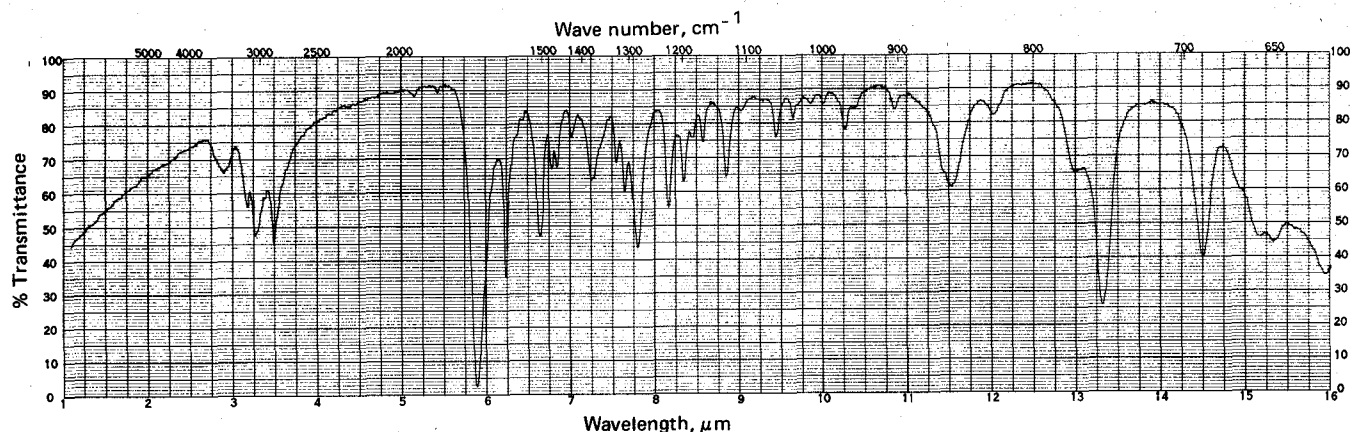


FIGURE — Reference infra-red spectrum of 1-Phenyl-3-pyrazolidinone (KBr plate)

## 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)<sup>1)</sup> 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 Iodine solution.

Dissolve about 13 g of iodine in a solution of 120 g of potassium iodide in 200 ml of water. When dissolution is complete, dilute to 1 000 ml.

##### 4.1.1.2 Acetate buffer solution, pH 4.

Dissolve 55 g of sodium acetate trihydrate ( $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$ ) in about 200 ml of water. Then add 92 ml of 2 M acetic acid solution and dilute to 1 000 ml with water.

##### 4.1.1.3 Sodium thiosulphate, 0,1 M standard volumetric solution.

##### 4.1.1.4 Starch indicator solution.

Stir 5 g of soluble starch with 100 ml of 10 g/l salicylic acid solution. Then add 300 to 400 ml of boiling water and boil until the starch dissolves, finally diluting to 1 000 ml with water.

### 4.1.2 Apparatus

Ordinary laboratory apparatus and

#### 4.1.2.1 Pipette, 25 ml capacity, conforming to class A of ISO/R 648.

#### 4.1.2.2 Burette, 50 ml capacity, conforming to class A of ISO/R 385.

### 4.1.3 Procedure

Weigh, to the nearest 0,001 g, a test portion of about 0,15 g of the laboratory sample and transfer to a conical flask. Add about 70 ml of warm water (50 to 60 °C) and dissolve.

Cool to room temperature and add 25 ml of the acetate buffer solution (4.1.1.2). Then add, from the pipette (4.1.2.1), 25 ml of the iodine solution (4.1.1.1) and back-titrate the excess iodine at once with the 0,1 M sodium thiosulphate solution (4.1.1.3) using the starch indicator (4.1.1.4). Carry out a blank titration in the same manner. The total elapsed time from the start of the addition of the iodine solution to the completion of the titration should not exceed 3 min. Undue delay will result in erroneously high assay values.

### 4.1.4 Calculation

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

$$\frac{8,11 (V_1 - V_2) T}{m}$$

where

$V_1$  is the volume, in millilitres, of sodium thiosulphate solution used for the blank titration;

$V_2$  is the volume, in millilitres, of sodium thiosulphate solution used for the determination;

$T$  is the molarity of the sodium thiosulphate solution;

$m$  is the mass, in grams, of the test portion.

## 4.2 Alkaline sulphite solution test

Dissolve 0,5 g of the sample in 1 000 ml of an aqueous solution of 25 g of sodium sulphite, anhydrous, and 25 g of sodium carbonate, anhydrous. Examine the solution for clarity and colour.

## 4.3 Determination of matter insoluble in chloroform

### 4.3.1 Apparatus

Ordinary laboratory apparatus and

#### 4.3.1.1 Sintered glass crucible of medium porosity.

### 4.3.2 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 5 g of the laboratory sample and dissolve in 100 ml of chloroform. If any undissolved material remains, filter through a weighed crucible (4.3.1.1) and wash with 25 ml of chloroform. Dry at 105 °C for 4 h. Cool in a desiccator and weigh to the nearest 1 mg.

1) 1 mol/l = 1 kmol/m<sup>3</sup> = 1 mol/dm<sup>3</sup> = 1 M

#### 4.3.3 Calculation

Matter insoluble in chloroform is given, as a percentage by mass, by the formula

$$\frac{m_1 - m_2}{m_0} \times 100$$

where

- $m_0$  is the mass, in grams, of the test portion;
- $m_1$  is the mass, in grams, of crucible and residue;
- $m_2$  is the mass, in grams, of the crucible.

#### 4.4 Determination of volatile matter at 65 °C

##### 4.4.1 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 10 g of the laboratory sample into a previously weighed squat-form, glass-stoppered weighing bottle. Dry at 65 °C for 4 h. Cool in a desiccator and weigh to the nearest 1 mg.

##### 4.4.2 Calculation

Volatile matter at 65 °C, as a percentage by mass, is given 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 test portion before drying;
- $m_3$  is the mass, in grams, of the bottle and test portion after drying.

#### 4.5 Determination of residue after ignition

##### 4.5.1 Apparatus

Ordinary laboratory apparatus and

##### 4.5.1.1 Platinum crucible.

4.5.1.2 Muffle furnace, capable of being controlled at  $600 \pm 50$  °C.

##### 4.5.2 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 5 g of the laboratory sample into the previously weighed platinum crucible (4.5.1.1) and incinerate. Heat the residue in the furnace (4.5.1.2), controlled at  $600 \pm 50$  °C, for 4 h. Cool in a desiccator and weigh to the nearest 1 mg. Retain this residue for the heavy metals and iron tests under 4.6.3 and 4.7.3.

#### 4.5.3 Calculation

Residue after ignition, 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.

#### 4.6 Limit test for heavy metals

##### 4.6.1 Reagents

4.6.1.1 Hydrochloric acid solution, dilute (1 + 99).

4.6.1.2 Ammonia solution, dilute (1 + 9).

4.6.1.3 Heavy metals, standard solution.

Dissolve a soluble lead salt in water to give a solution containing 10 mg of lead per 1 000 ml.

4.6.1.4 Water, saturated at room temperature with hydrogen sulphide.

4.6.1.5 *p*-Nitrophenol indicator, aqueous solution, 2,5 g/l.

##### 4.6.2 Apparatus

Ordinary laboratory apparatus and

4.6.2.1 Two one-mark volumetric flasks, 50 ml capacity, conforming to class A of ISO 1042.

4.6.2.2 Pipette, 20 ml capacity, conforming to class A of ISO/R 648.

4.6.2.3 Two matched Nessler cylinders, 50 ml capacity.

##### 4.6.3 Procedure

Dissolve the residue retained from 4.5.2 in 0,5 ml of the hydrochloric acid solution (4.6.1.1) and transfer this solution (with washing) to one of the one-mark volumetric flasks (4.6.2.1). To the other volumetric flask, transfer 25 ml of the standard heavy metals solution (4.6.1.3). To each, add 2 drops of the *p*-nitrophenol indicator solution (4.6.1.5) followed by the ammonia solution (4.6.1.2), drop by drop, until the solutions turn yellow. Add the hydrochloric acid solution (4.6.1.1), drop by drop, until the solutions become colourless and then add 2,5 ml in excess. Dilute each to the mark and mix well.

Using the pipette (4.6.2.2), transfer a 20 ml aliquot of each solution to separate Nessler cylinders (4.6.2.3), retaining the balance of the test solution for the iron test under 4.7.3. Add 5 ml of the hydrogen sulphide water (4.6.1.4) to each, dilute to 50 ml and mix well.

Compare, in the Nessler cylinders, the colours produced in the test and control solutions.

#### 4.7 Limit test for iron

##### 4.7.1 Reagents

4.7.1.1 Hydrochloric acid solution, dilute (1 + 99).

4.7.1.2 Ammonia solution, dilute (1 + 9).

4.7.1.3 Acetate buffer solution, pH 5,0.

Dissolve 23 g of anhydrous sodium acetate in 58 ml of 2 M acetic acid and dilute to 1 000 ml. Adjust the final pH of the solution to  $5,0 \pm 0,1$  with glacial acetic acid or 100 g/l sodium hydroxide solution.

4.7.1.4 Iron, standard solution.

Dissolve a soluble iron(III) salt in water to give a solution containing 10 mg of iron(III) per 1 000 ml.

4.7.1.5 1,10-Phenanthroline reagent solution.

Thoroughly mix equal volumes of a 1 g/l aqueous solution of 1,10-phenanthroline, a 100 g/l aqueous solution of hydroxylammonium hydrochloride and the acetate buffer solution (4.7.1.3).

4.7.1.6 *p*-Nitrophenol indicator, aqueous solution, 2,5 g/l.

##### 4.7.2 Apparatus

Ordinary laboratory apparatus and

4.7.2.1 One-mark volumetric flask, 50 ml capacity, conforming to class A of ISO 1042.

4.7.2.2 Pipette, 20 ml capacity, conforming to class A of ISO/R 648.

4.7.2.3 Two matched Nessler cylinders, 50 ml capacity.

##### 4.7.3 Procedure

To 25 ml of the standard iron solution (4.7.1.4) in the one-mark volumetric flask (4.7.2.1), add 2 drops of the *p*-nitrophenol indicator solution (4.7.1.6) and then add the ammonia solution (4.7.1.2), drop by drop, until the solution turns yellow. Add the hydrochloric acid solution (4.7.1.1), drop by drop, until the solution becomes colourless and then add 2,5 ml in excess. Dilute to the mark and mix well.

Using the pipette (4.7.2.2), transfer a 20 ml aliquot of this treated standard iron solution to one of the Nessler cylinders (4.7.2.3) and 20 ml of the balance of the treated test solution from 4.6.3 to the other Nessler cylinder. Add 5 ml of the 1,10-phenanthroline reagent solution (4.7.1.5) to each, mix and allow to stand for 10 min. Dilute each to 50 ml and mix well.

Compare, in the Nessler cylinders, the colours produced in the test and control solutions.

#### 4.8 Identity tests

##### 4.8.1 Melting point tests

###### 4.8.1.1 APPARATUS

Capillary-tube melting point apparatus, complete with thermometer including the range 100 to 200 °C.

###### 4.8.1.2 PROCEDURE

Prepare three capillary tubes, containing

- 1) the sample to be tested;
- 2) a sample known to be 1-phenyl-3-pyrazolidinone;
- 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 (4.8.1.1) 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.

##### 4.8.2 Infra-red identity test

###### 4.8.2.1 APPARATUS

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

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

###### 4.8.2.2 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 thoroughly together to pass the test sieve (4.8.2.1.1). Prepare a pressed plate of the mixture to contain 0,13 to 0,16 g of mixture per square centimetre of die area. 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.

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