

**IS 16112 : 2013**

**(Reaffirmed 2018)**

**भारतीय मानक**

**बीटा पिकोलीन — विशिष्टि**

*Indian Standard*

**BETA PICOLINE — SPECIFICATION**

ICS 71.080.30

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**BUREAU OF INDIAN STANDARDS**  
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NEW DELHI 110002

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**Price Group 3**

Organic Chemicals, Alcohols and Allied Products and Dye Intermediates Sectional Committee, PCD 09

#### FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products and Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Beta Picoline is used in the manufacture of Agrochemicals, Vitamin B3, Thermoplastics and Polymers.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

## Indian Standard

# BETA PICOLINE — SPECIFICATION

### 1 SCOPE

This standard prescribes the requirements and methods of sampling and test for Beta Picoline.

### 2 REFERENCES

The following Indian Standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibilities of applying the most recent editions of the standards listed below:

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water — Specification ( <i>third revision</i> )
2362 : 1993	Determination of water by Karl Fischer method — Test method ( <i>second revision</i> )
4905 : 1968	Methods for random sampling

### 3 REQUIREMENTS

**3.1 Description** — The material shall be clear, colourless to yellow liquid with characteristic odour.

**3.2 Solubility** — The material shall be soluble in water in all proportions.

**3.3** The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in this standard.

### 4 PACKING AND MARKING

#### 4.1 Packing

The material shall be packed in GI Drums (Drums made of galvanized iron sheet, zinc coated) or HM-HDPE drums. The gaskets for the bungs shall be of high density polyethylene.

#### 4.2 Marking

**4.2.1** Each container shall be suitably marked with the following information:

- Name and grade of the material;
- Net mass of the material in the container;

- Name of the manufacturer and his recognized trade-mark, if any; and
- Batch number or lot number, in code or otherwise.

**Table 1 Requirements for Beta Picoline**  
(Clause 3.3)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex of this Standard
(1)	(2)	(3)	(4)
i)	Beta picoline content, percent by mass, <i>Min</i>	98.5	A
ii)	2-Ethyl pyridine content, percent by mass, <i>Max</i>	0.4	A
iii)	Alpha picoline content, percent by mass, <i>Max</i>	0.2	A
iv)	Gamma picoline content, percent by mass, <i>Max</i>	0.6	A
v)	Other unknown impurities, percent by mass, <i>Max</i>	0.2	A
vi)	Moisture content, percent by mass, <i>Max</i>	0.1	B

#### 4.2.2 BIS Certification Marking

The containers may also be marked with the Standard Mark.

**4.2.2.1** The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the license for the use of Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

### 5 SAMPLING

The procedure for sampling and the criteria for conformity of the material shall be as prescribed in Annex C.

### 6 TEST METHODS

**6.1** Tests shall be conducted as prescribed in col 4 of Table 1.

#### 6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see IS 1070*) shall be used in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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## ANNEX A

[Table 1, Sl No. (i) to (v)]

### DETERMINATION OF BETA PICOLINE CONTENT AND IMPURITIES

#### A-1 OUTLINE OF THE METHOD

The content of beta picoline along with other components is determined by gas chromatography. The chromatographic conditions given here are for guidance only. The typical chromatogram for beta picoline is shown in Fig. 1.

#### A-2 APPARATUS

**A-2.1 Gas Chromatograph** — A gas chromatograph equipped with a Flame-Ionization Detector using capillary column.

**A-2.2 Data Acquisition System** — A system capable of acquiring chromatographic data and integrating chromatographic peaks.

**A-2.3 Column** — CP-Sil-52CB Wax, 60 m long, 0.25 mm internal diameter, 0.25  $\mu$ m film thickness (df).

#### A-3 REAGENTS

**A-3.1 Certified Reference/Working Standard of 2-Ethylpyridine**

**A-3.2 Certified Reference/Working Standard of Alpha Picoline**

**A-3.3 Certified Reference/Working Standard of Beta Picoline**

**A-3.4 Certified Reference/Working Standard of Gamma Picoline**

**A-3.5 Internal Standard — Pentadecane** (Purity more than 99 percent).

NOTE — Working standard is prepared against Certified Reference Material.

#### A-4 PROCEDURE

**A-4.1 Operating Parameters of Gas Chromatograph**

**A-4.1.1 Injector Temperature** : 240°C

**A-4.1.2 Detector Temperature** : 250°C

**A-4.1.3 Carrier Flow ( $N_2$ )** : 1.5 ml/min

**A-4.1.4 Split Ratio** : 1 : 100

**A-4.1.5 Make up Flow ( $N_2$ )** : 20 ml/min

**A-4.1.6 Oven Temperature** : 75°C for 18 min

**A-4.1.7 Oven Temperature** : 200°C for 6 min

**A-4.1.8 Programming Rate** : 10°C/min

**A-4.1.9 Total Run Time** : 36.5 min

**A-4.1.10 Injection Volume** : 1.0  $\mu$ l of calibration solution 0.2  $\mu$ l of standard and test sample

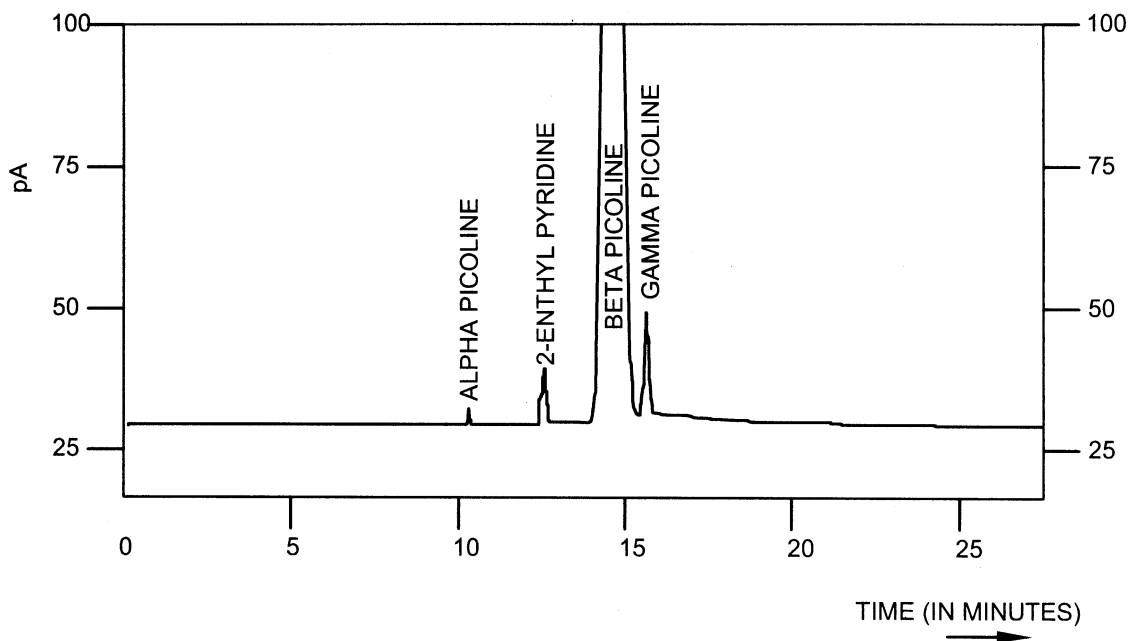


FIG. 1 A TYPICAL CHROMATOGRAM OF BETA PICOLINE — SAMPLE SOLUTION

#### A-4.2 Preparation of Internal Standard Solution

**A-4.2.1** Weigh and transfer accurately about 1 000 mg of pentadecane in a 100 ml volumetric flask. Add Methanol to dissolve and dilute the content to the volume. If required, sonicate or stir till stock solution becomes clear and preserve in a cool place.

**A-4.2.2** Pipette out 4.0 ml of the above solution into another 100 ml volumetric flask and dilute to the mark with Methanol. Final concentration of pentadecane is about 0.4 mg/ml.

#### A-4.3 Preparation of Standard Solution for Calibration

**A-4.3.1** Weigh accurately  $960 \pm 0.5$  mg standard of Beta picoline and 10 mg of each standard of 2-Ethylpyridine, Alpha picoline and Gamma Picoline in a clean and dry vial and mix thoroughly.

**A-4.3.2** Weigh accurately about  $400 \pm 0.5$  mg of the above synthetic mixture into another 10 ml volumetric flask and add accurately 10.0 ml of internal standard solution by pipette. Dissolve and mix the contents. The solution thus prepared is called Calibration Solution.

#### A-4.4 Method

**A- 4.4.1** Condition the column at 200°C for 30 min. Allow the gas chromatograph to equilibrate at 75°C and obtain a steady baseline before proceeding for analysis. Following is the recommended sequence for analysis and evaluate system suitability:

- a) Inject 1.0  $\mu$ l — Blank (Methanol) (single injection).
- b) Inject 1.0  $\mu$ l — Calibration solution (duplicate injection).

The system must meet suitability criteria as per Table 2 before proceeding further and determine the response factor from Calibration Standard chromatogram nearest to 0.001.

- c) Inject 0.2  $\mu$ l (neat) — Standard and each test sample (single injection).

**Table 2 Criteria for System Suitability**

Sl No. (1)	Method Performance Parameter (2)	Criteria (3)
i)	Tailing factor for all analyte	$\leq 2.5$
ii)	Resolution between all impurities	$\geq 1.5$

#### A-4.5 Calculation

**A-4.5.1** *Calculation of Calibration Factor or Response Factor* — Calculate the area/mass (A/M) ratio by dividing the area of each peak by its mass as under:

Component	Mass, Percent	Area	A/M
Pentadecane	1.0	$A_1$	$A_1/1.0 = K$
2-Ethylpyridine	1.0	$A_2$	$A_2/1.0 = L$
Alpha picoline	1.0	$A_3$	$A_3/1.0 = M$
Beta picoline	96.0	$A_4$	$A_4/96.0 = N$
Gamma picoline	1.0	$A_5$	$A_5/1.0 = O$

**A-4.5.2** *Calculation of Actual Mass Percent in Calibration Solution* — Set arbitrarily internal standard response factor to 1.0 and find response factor of other components as follows:

Component	Slope	Response Factor
Pentadecane	K/K	1.000 0
2-Ethylpyridine	K/L	Value obtained
Alpha picoline	K/M	do
Beta picoline	K/N	do
Gamma picoline	K/O	do

**A-4.5.3** Multiply the areas by their response factor to get the true areas of the peaks. Add up the other unspecified area with true area to get total true area and calculate as per following:

$$\text{Component 'n' in the sample, percent by mass} = \frac{A_n \times (100 - m)}{A_t}$$

where

- $A_n$  = true area of component 'n';  
 $m$  = percent of water in the sample; and  
 $A_t$  = total true peak area.

**A-4.5.3** Typical Retention Time of interest components in Beta picoline is given in Table 3.

**Table 3 Retention Time of Interest Components in Beta Picoline**

Sl No. (1)	Component Name (2)	Retention Time (in min) (3)
i)	Alpha picoline	11.9
ii)	2-Ethyl pyridine	16.1
iii)	Beta picoline	17.5
iv)	Gamma picoline	17.8
v)	Pentadecane	25.6

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## ANNEX B

[Table 1, Sl No. (vi)]

### DETERMINATION OF MOISTURE CONTENT

#### B-1 GENERAL

Moisture is determined by the Karl Fischer method.

#### B-2 PROCEDURE

**B-2.1** Weigh approximately 10 g of the material and determine the moisture content by the procedure given in IS 2362.

## ANNEX C

(Clause 5)

### SAMPLING OF BETA PICOLINE

#### C-1 GENERAL REQUIREMENTS FOR SAMPLING

**C-1.1** Samples shall be taken in a protected place not exposed to damp air, dust or soot.

**C-1.2** The sampling instrument shall be clean and dry.

**C-1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**C-1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**C-1.5** The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

**C-1.6** The sample containers shall be of such a size that they are almost three-fourth filled by the sample.

**C-1.7** Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and details given under 4.2.

#### C-2 SCALE OF SAMPLING

**C-2.1 Lot** — All the containers in a single consignment of the material of the same grade drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots.

**C-2.2** For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately.

**C-2.3** The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 4.

**Table 4 Number of Containers to be Selected from Lots of Different Sizes**

Lot Size 'N' (1)	Sample Size 'n' (2)
3 to 15	3
16 to 40	4
41 to 110	5
111 to 180	6
181 to 300	7
301 to 500	8
501 and above	9

**C-2.4** The containers shall be chosen at random from the lot with the help of a suitable random number table. Reference may be made to IS 4905 for guidance to random selection procedures.

#### C-3 TEST SAMPLE AND REFEREE SAMPLE

**C-3.1** From each of the containers selected as in C-2.3, draw with the help of a sampling bottle a representative portion of the material from different parts of the container. Out of this portion from each container equal quantity of the material shall be taken and thoroughly mixed to form a composite sample of about 1500 ml. This composite sample shall be thoroughly mixed and divided into three equal portions, one for the purchaser, another for the supplier and the third for the referee.

**C-3.2** The remaining portion corresponding to each of the selected containers as in C-2.3, shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers selected shall be for the purchaser, another for the supplier and the third for the referee.

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**C-3.3** All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **C-1.7**.

**C-3.4** The referee samples consisting of a composite sample and a set of  $n$  individual samples shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. These shall be used in case of any dispute between the two.

**C-4 TESTS**

**C-4.1** Tests for beta picoline content and moisture shall be conducted on individual samples.

**C-4.2** Tests for the remaining characteristics shall be conducted on the composite sample.

**C-5 CRITERIA FOR CONFORMITY**

**C-5.1 For Individual Samples** — The lot shall be declared as conforming to the requirements of beta picoline content and moisture if each of the test results on the individual samples satisfies the corresponding requirement of the test.

**C-5.2 For Composite Sample** — For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample, the test results shall satisfy the relevant requirements given in **3** and Table 1.



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## Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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