भारतीय मानक

Indian Standard

IS 17370 : 2023

पी-ज़ाइलीन — विशिष्टि

(पहला पुनरीक्षण)

p-Xylene — Specification

(First Revision)

ICS 71.080.15

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

FOREWORD

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

Xylene are deemed as highly flammable and explosive materials, within the meaning of the *Indian Explosive Act*, 1884 and Rules framed thereunder. Their filling, possession, storage, transport, etc are governed by the *Petroleum Act*, 1934 and Rules framed thereunder with any alterations and additions made thereafter. IS 4644 Code of safety for benzene, toluene and xylene may be referred for details regarding its toxicity, its storage, handling etc.

The Standard was first published in 2020. In the preparation of the standard considerable assistance was derived from ASTM D5136-19 'High Purity *p*-Xylene'. This revision is taken up to incorporate indigenous test methods based on ASTM test methods as referred in the original standard for properties like purity and impurities present in *p*-xylene; colour etc. In the preparation of the indigenous test methods the assistance has been derived from the following ASTM test methods:

a) ASTM D 5917-15 Standard Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography and External Calibration;

b) ASTM D 7504-18 Standard Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography and Effective Carbon Number;

c) ASTM D 7183-18 Standard Test Method for Determination of Total Sulphur in Aromatic Hydrocarbons and Related Chemicals by Ultraviolet Fluorescence; and

d) ASTM E 2680-16 Standard Test Method for Appearance of Clear, Transparent Liquids (Visual Inspection Procedure).

The composition of the committee, responsible for the formulation of this standard is listed in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

p-XYLENE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and test methods for *p*-Xylene.

2 REFERENCES

The standards listed below contain provisions, which through reference in this text constitute the provisions of the 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 possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods
IS 1447 (Part 1) : 2021	Methods of sampling of petroleum and its products: Part 1 Manual sampling (*second revision*)
IS 1448	Methods of test for petroleum and its products:
(Part 160) : 2017/ISO 20846 : 2011	Determination of sulphur content of automotive fuels — Ultraviolet fluorescence method.
(Part 180) : 2020/ISO 16591: 2010	Petroleum products — Determination of sulphur content — Oxidative microcoulometry method
IS 8768 : 2000	Method of measurement of colour in liquid chemical products platinum — Cobalt scale (*second revision*)

3 REQUIREMENTS

3.1 Description

p-Xylene is a colorless liquid, free of haze, particulates or suspended matter particles.

3.2 The material shall also comply with the requirements given in Table 1, when tested as prescribed in col (4) and col (5) of Table 1.

3.2.1 Quality of Reagents

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

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

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in metallic or glass containers, when used for small applications. While for bulk transport, the material is transported through road tankers, rail wagon, etc. or as agreed between buyer or suppliers. All containers should be securely packed and sealed.

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear the following information:

a) Name of the material;
b) Name of the manufacturer and recognized trademark, if any;
c) Month and year of manufacture;
d) Lot or batch number;
e) Net weight and gross weight; and
f) Any other statutory requirement.

4.2.2 For supplies of material in bulk, a test report containing the following information shall be provided for each container:

a) Name of the material;
b) Name of the manufacturer and recognized trademark, if any; and
c) Date of analysis.

The test report shall be certified by authorized person of the manufacturer's organization.

4.2.3 All containers/consignments of *p*-xylene shall bear the label as given in Fig. 5 of IS 1260 (Part 1). The lower half of the label shall have the following words printed in red letters. Any other labels or warnings or other statements required by statutes, regulations or ordinances may also be used in combination or separately.

Warning

HIGHLY FLAMMABLE;

VAPOUR HARMFUL;

KEEP AWAY FROM HEAT, SPARK AND OPEN FLAME;

KEEP CONTAINER CLOSED;

USE ONLY WITH ADEQUATE VENTILATION;

AVOID PROLONGED BREATHING OF VAPOUR

4.2.4 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the Bureau of Indian Standards Act, 2016 and the Rulesand Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be in accordance with IS 1447 (Part 1).

5.2 Criteria for Conformity

For declaring the conformity of a lot to the requirements of all the other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

Table 1 Requirements for *p*-Xylene

(*Clause* 3.2 *and* 5.2)

Sl No.	Characteristics	Requirements	Methods of Test, Refer to	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Purity, mass percent, *Min*	99.7	A	—
ii)	*m*-Xylene, mass percent, *Max*	0.20	A	
iii)	*o*-Xylene, mass percent, *Max*	0.10	A	
iv)	Sulphur, mg/kg, *Max*	1.0	B	IS1448(Part 160)[1]/ IS1448(Part 180)
v)	Toluene, mass percent, *Max*	0.10	A	—
vi)	Ethylbenzene, mass percent, *Max*	0.20	A	—
vii)	Nonaromatic hydrocarbons, mass percent, *Max*	0.20	A	—
viii)	Appearance	Pass	C	—
ix)	Color, Pt-Co, *Max*	10	—	IS 8768

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[1] In case of disputes, IS 1448 (Part 160) shall be the referee method for determination of sulphur content.

ANNEX A

[*Table* 1, *Sl No*. (i), (ii), (iii), (v), (vi) *and* (vii)]

DETERMINATION OF PURITY OF *p*-XYLENE AND ITS IMPURITIES

A-1 GENERAL

Two methods namely, Method A Determination of purities and impurities in *p*-xylene by gas chromatographic (GC) and external calibration and Method B Determination of purities and impurities in *p*-xylene by gas chromatographic (GC) and effective carbon number have been prescribed. In case of disputes, method A shall be the referee method.

A-2 METHOD A

A-2.1 Outline of the Test Method

A-2.1.1 This method is applicable for the determination of purity and trace impurities in *p*-xylene by gas chromatographic (GC) technique. The instrument is calibrated using external standards. Aliphatic hydrocarbons up to C_{10} can be measured accurately as low as 0.001 0 mass percent [that is 10 ppm (mass)] up to 2.4 mass percent and individual mono cyclic aromatic hydrocarbons up to C_{10} can be measured accurately as low as 15 ppm (mass) up to 1 mass percent. Purity percent is calculated by subtracting total impurities from 100.

A-2.1.2 A known volume of sample is injected, manually or using auto sampler in the gas chromatographic instrument. The peak area of each component is measured, and concentration of each component is calculated using linear calibration curve.

NOTE — Small amount of Benzene may not be fully separated from nonaromatic peaks. Ethylbenzene and *m*-xylene elutes very close to *p*-xylene peak. Separation between *p*-xylene and an impurity peak can be accepted if height from valley to the base line is not more than half the height of the impurity peak.

A-2.2 Apparatus

A-2.2.1 *Gas Chromatograph*

Any gas chromatograph equipped with a flame ionization detector (FID), a split/splitless injector and a suitable electronic integrator/software may be used with following accessories and operating condition.

A-2.2.1.1 *Column*, fused silica column with a stationary phase of cross-linked polyethylene glycol (PEG); length 60 m; internal diameter 0.32 mm and 0.25 µm film thickness.

A-2.2.1.2 *Gas chromatographic condition parameters*

Injector

a) Temperature : 275 °C
b) Injection volume : 0.4 µl to 0.6 µl
c) Split ratio : 1 : 100

Oven Temperature

(a) Start : 50 °C for 10 min
(b) Ramping : 5 °C/min
(c) End : 150 °C for 10 min

Detector :

(a) Temperature : 300 °C

Carrier Gas

Carrier gas linear velocity : 20 cm/s for He and 45 cm/s for H_2

NOTES

1 Auto sampler for sample injection may also be used.

2 The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc), calibration technique (internal standard, external standard, area normalization, percent area etc), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

A-2.3 Reagents

A-2.3.1 *High purity p-xylene*

Take approximately 500 ml *p*-xylene and cool it to -10 °C to 5 °C, till approximately 3/4th portion is frozen. Decant the supernatant liquid and allow the frozen portion to liquify. The purity of *p*-xylene is checked as per this standard. The crystallization step is repeated until no ethylbenzene, *m*-xylene and *o*-xylene are detected. Purity of such purified *p*-xylene is approximately 99.999 mass percent. Four to five steps are required to get purified *p*-xylene.

A-2.4 Calibration and Standardization

To calibrate the instrument, use of certified reference material is recommended. Alternately, calibration blend may be prepared by the following procedure.

A-2.4.1 *Preparation of Calibration Blend*

All chemicals including n-nonane, benzene, toluene, ethylbenzene, o-xylene, m-xylene, p-xylene (high purity), cumene, paradiethylbenzene (PDEB) are cooled to room temperature and the blend is prepared by mixing the chemicals, as prescribed in table given below:

	Volume	*Concentration (volume percent)*	*Density at 20 °C*	*Concentration (mass percent)*
n-Nonane	20 µl	0.020	0.718	0.017
Benzene	10 µl	0.010	0.879	0.010
Toluene	20 µl	0.020	0.867	0.020
Ethyl Benzene	100 µl	0.100	0.867	0.101
p-Xylene	99.70 ml	99.70	0.861	99.70
m-Xylene	100 µl	0.100	0.864	0.100
Cumene	10 µl	0.010	0.862	0.010
o-Xylene	20 µl	0.020	0.88	0.020
Para-diethyl Benzene (PDEB)	20 µl	0.020	0.866	0.020

NOTE — Alternately, calibration blend can be prepared gravimetrically also.

A-2.4.2 The instrument is set as per **A-2.2.1.2** or as per manufacturer's recommendation. Inject the calibration blend once it attains room temperature. Calculate the response factor for each component as:

Response factor of component Y (RF)

$$= \frac{\text{Concentration of component,Y}}{\text{Area of component, Y}}$$

NOTE — RF of *n*-nonane is used for non-aromatic fraction.

While RF of cumene is used for all aromatics hydrocarbon with C_9 or greater except *o*-xylene, ethylbenzene and *m*-xylene, whose RF are calculated and used independently. RF of para-diethyl benzene is also calculated and used independently.

Take average of minimum three analyses and calculate average response factor.

A-2.5 Procedure

A-2.5.1 Ensure that GC is set as recommended before injecting the sample. Inject the sample in GC once it attains room temperature.

A-2.5.2 All the peaks before o-xylene excluding benzene, toluene, ethyl benzene, p-xylene, m-xylene and cumene are considered as non-aromatics. All peaks eluted after o-xylene plus cumene are considered as C9 plus aromatics.

A-2.5.3 C9 plus aromatics are grouped together and reported. If required, cumene or para-diethyl benzene (PDEB) are reported separately, and remaining components are reported as C9 plus aromatics.

A-2.6 Calculation

A-2.6.1 Calculations are performed by the instrument to display results in desired format. For manual calculation, the mass percent concentration of each impurity is calculated by the formula as given:

Concentration of component Y, percent =

$$\frac{A_y \,\mathrm{x}\, RF_y \,\mathrm{x}\, D}{d}$$

where

RF_y = Response factor of Y;
A_y = Area of component Y;
D = Density of calibration solution (p-Xylene) as per **A-2.4**; and
d = Density of sample at 20 °C.

A-2.6.2 Purity, percent = 100 – sum of percent of all impurities

A-2.7 Report

Report all the purity of p-xylene, individual impurities, non-aromatics hydrocarbons and total C_9 plus aromatics nearest to 0.01 mass percent.

A-2.8 Precision and Bias

A-2.8.1 *Repeatability*

Difference between two results obtained in the same laboratory, same operator, and same instrument on same sample shall not differ more than the values shown in the Table 2.

A-2.8.2 *Reproducibility*

Difference between two results obtained in the different laboratories, different operator and different instrument of the sample shall not differ more than the values shown in the Table 2. Atleast one or all the condition mentioned above is considered for reproducibility calculations.

A-3 METHOD B

A-3.1 Outline of the Test Method

A-3.1.1 This method is applicable for the determination of purity of p-xylene and its impurities by gas chromatographic (GC) technique. This test method is based on effective carbon number. The individual impurities can be quantified as low as 0.001 mass percent (that is 10 ppm or 10 mg/kg). Impurities like non-aromatic hydrocarbons up to C_{10}, aromatic compounds of C_6 to C_{10} are normally found in high purity p-xylene and can be determined using this method. Percent purity of the p-xylene is calculated by subtracting total impurities from 100.

A-3.1.2 A known volume of sample is injected, manually or using auto sampler in the gas chromatographic instrument. The peak area of each component is then calculated, adjusted against effective carbon number correction factor, amount is calculated against relative percent of total adjusted area and normalized to 100 percent by the integration software. It is important to identify all the components. Since results are normalized, presence of an unknown peak can affect the final result.

NOTE — Ethylbenzene and m-xylene elutes very close to p-xylene peak. Separation between p-xylene and an impurity peak can be accepted if height from valley to the base line is not more than half the height of the impurity peak.

A-3.2 Apparatus

A-3.2.1 *Gas Chromatograph*

Any gas chromatograph equipped with a flame ionization detector (FID), a split/splitless injector and a suitable electronic integrator/software may be used with following accessories and operating condition.

A-3.2.1.1 *Column*, fused silica column with a stationary phase of cross-linked polyethylene glycol (PEG); length 60 m; internal diameter 0.32 mm and 0.25 µm film thickness.

A-3.2.1.2 *Gas chromatographic condition parameters*

Injector		
a) Temperature	:	275 °C
b) Injection volume	:	0.4 µl to 0.6 µl
c) Split ratio	:	1 : 100
Oven Temperature		
a) Start	:	50 °C for 10 min
b) Ramping	:	5 °C/min
c) End	:	150 °C for 10 min
Detector	:	
(a) Temperature	:	300 °C
Carrier Gas		
Carrier gas linear velocity	:	20 cm/s for He and 45 cm/s for H_2

NOTES

1 Auto sampler for sample injection may also be used.

2 The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc), calibration technique (internal standard, external standard, area normalization, percent area etc), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

A-3.3 Reagent

A-3.3.1 *High Purity p-Xylene*

Take approximately 500 ml *p*-xylene and cool it to -10 °C to 5 °C, till approximately 3/4th portion is frozen. Decant the supernatant liquid and allow the frozen portion to liquify. The purity of *p*-xylene is checked. The crystallization step is repeated until no ethylbenzene, *m*-xylene and *o*-xylene are detected. Purity of such purified *p*-xylene is approximately 99.999 mass percent. Four to five steps are required to get purified *p*-xylene.

A-3.4 Calibration and Standardization

As this method is based on effective carbon number, hence external calibration is not required.

A-3.5 Retention Time Check

A-3.5.1 *Retention Time Check Solution*

Take 75 ml high purity *p*-xylene (**A-3.3.1**) in 100 ml volumetric flask. To it add 0.1 ml *m*-xylene, 0.01 ml each of toluene, ethyl benzene, cumene, *o*-xylene, styrene, phenyl acetylene and alpha methyl styrene and 0.001 ml each of benzene and 1,4-dioxane. Now dilute it up to the mark with high purity *p*-xylene.

A-3.5.2 *Performance Check Sample*

Prepare 10 mg/kg toluene standard using the high purity *p*-xylene (*see* **A-3.3.1**).

A-3.6 Instrument Performance Verification

A-3.6.1 The instrument is set as per **A-3.2.1.2** or as per manufacturer's recommendation. Inject the solution so prepared at **A-3.5.1** into GC**,** to determine retention time of each component and there should be sufficient separation of *p*-xylene peak from ethyl benzene and *m*-xylene. Poor resolution of these peaks may sometimes require tangent skimming from the nearby peak. For performance verification of the instrument, set the instrument as per **A-3.2.1.2** or as per manufacturer's recommendation and inject the solution prepared at **A-3.5.2** into GC. Calculate the concentration of toluene by using effective carbon number and density value (*see* Table 3). It shall be in the range of (10 ± 3) mg/kg. Instrument performance shall be periodically verified as per this procedure.

Table 2 Repeatability and Reproducibility
(*Clauses* A-2.8.1 *and* A-2.8.2)

Sl No. (1)	Components (2)	Average Concentration, Mass Percent (3)	Repeatability (4)	Reproducibility (5)
i)	Non-aromatics	0.012 0	0.018 8	0.027 4
ii)	Benzene	0.000 2	0.000 2	0.000 3
iii)	Toluene	0.015 6	0.005 4	0.007 1
iv)	Ethyl Benzene	0.040 8	0.002 6	0.008 7
v)	*p*-Xylene	99.823 2	0.031 5	0.042 4
vi)	*m*-Xylene	0.077 0	0.016 5	0.025 1
vii)	*o*-Xylene	0.023 3	0.001 0	0.029 8
viii)	C_9 plus Aromatics	0.007 8	0.001 4	0.016 8

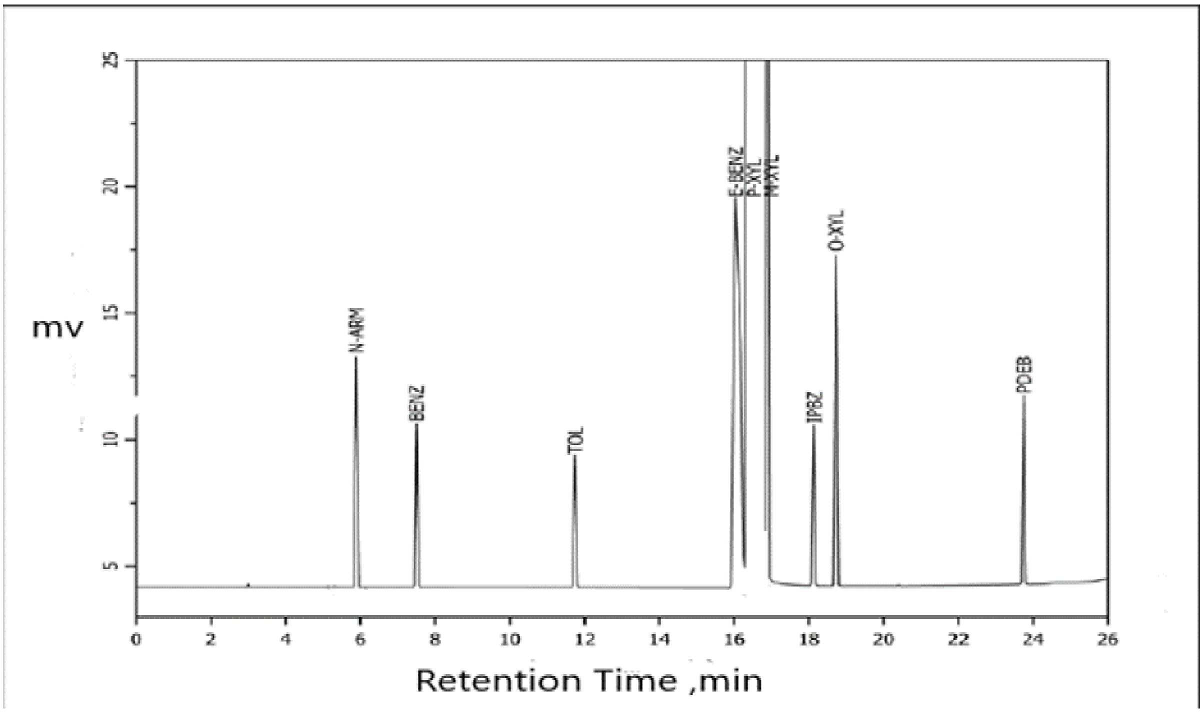

FIG. 1 TYPICAL CHROMATOGRAM OF P-XYLENE

Table 3 Effective Carbon Number and Density
(*Clause* A-3.6.1)

Sl No. (1)	Component (2)	Density at 20 °C (3)	Effective Carbon Number Correction Factor (4)
i)	Non-aromatics	0.725 5 (average density)	1.000 0
ii)	Benzene	0.878 0	0.909 5
iii)	Toluene	0.865 8	0.919 5
iv)	Ethyl Benzene	0.865 8	0.927 1
v)	*p*-Xylene	0.859 7	0.927 1
vi)	*m*-Xylene	0.863 0	0.927 1
vii)	Cumene	0.860 5	0.932 9
viii)	*o*-Xylene	0.878 6	0.927 1
ix)	1,4-Dioxane	1.032 9	3.077 4
x)	C_9 plus Aromatics	0.871 5 (average density)	0.932 9
xi)	Styrene	0.904 8	0.921 0
xii)	C_{10} Aromatics	0.869 4 (average density)	0.937 6
xiii)	PDEB	0.862 0	0.937 6
xiv)	Phenyl acetylene	0.930 0	0.829 6
xv)	Alpha Methyl Styrene	0.907 7	0.927 6

A-3.7 Procedure

Ensure that GC is set as recommended before injecting the sample. Inject the sample in GC once it attains room temperature and at the end of the run, calculate the results.

A-3.8 Calculation

A-3.8.1 Calculations are performed by the instrument to display results in desired format. For manual calculation:

A-3.8.1.1 The mass percent concentration of each impurity is calculated by the formula as given:

Concentration of component Y (C_y), mass percent

$$= \frac{A_y \text{x } R_y \text{x } 100}{\Sigma(A_y \text{ x } R_y)}$$

where

A_y = area of component Y; and

R_y = effective carbon number correction factor of component Y.

A-3.8.1.2 The volume percent concentration of each impurity is calculated by the formula as given:

Concentration of component Y (V_y), volume percent

$$= \frac{\frac{C_y}{D_y} \text{ x } 100}{\Sigma(\frac{C_y}{D_y})}$$

where

C_y = concentration of Y calculated at **A-3.8.1.1**, mass percent; and

A-3.9 Report

Report all the purity of *p*-xylene, individual impurities, non-aromatics hydrocarbons and total C_9 plus aromatics nearest to 0.01 mass percent.

A-3.10 Precision and Bias

A-3.10.1 *Repeatability*

Difference between two results obtained in the same laboratory, same operator, and same instrument on same sample shall not differ more than the values shown in the Table 4.

A-3.10.2 *Reproducibility*

Difference between two results obtained in the different laboratories, different operator, and different instrument of the sample shall not differ more than the values shown in the Table 4. Atleast one or all the condition mentioned above is considered for reproducibility calculations.

Table 4 Repeatability and Reproducibility

(*Clauses* A-3.10.1 *and* A-3.10.2)

Sl No.	Components	Average Concentration, Percent (*wt/wt*)	Repeatability	Reproducibility
(1)	(2)	(3)	(4)	(5)
i)	Non-aromatics	0.012 5	0.013 0	0.019 7
ii)	Benzene	0.002 0	0.000 7	0.001 8
iii)	Toluene	0.020 0	0.000 5	0.001 5
iv)	Ethyl Benzene	0.041 2	0.002 7	0.004 7
v)	*p*-Xylene	99.806 6	0.004 0	0.017 0
vi)	*m*-Xylene	0.104 5	0.004 0	0.013 7
vii)	*o*-Xylene	0.031 6	0.000 3	0.002 4
viii)	C_9 + Aromatics	0.006 7	0.000 7	0.002 1

ANNEX B

[*Table 1, Sl No.* (iv)]

DETERMINATION OF TOTAL SULPHUR IN *p*-XYLENE

B-1 GENERAL

Three methods namely, IS 1448 (Part 160) determination of sulphur content by Ultraviolet fluorescence method; IS 1448 (Part 180) determination of sulphur content by oxidative micro-coulometry method and method A determination of sulphur content by oxidative combustion and UV fluorescence detection technique have been prescribed.

B-2 METHOD A

B-2.1 Outline of the Test Method

B-2.1.1 This method is used for the determination of total sulphur in high purity *p*-xylene by oxidative combustion and UV fluorescence detection technique. This method is applicable for total sulphur in the range of 0.1 ppm to 10 ppm (*wt/wt*).

B-2.1.2 A known volume of sample is injected, manually or using auto sampler, into the combustion tube which is maintained at high temperature.

Sample vapour is combusted by oxygen which produces CO_2, SO_2 and water vapour. The Sample vapour is swept into the combustion zone by inert gas like argon. Inside the combustion tube, combustion gases then passed through a drier to remove water vapour, which then enters detector, where it is exposed to UV light. SO_2 gas absorbs the specific frequency of UV light and enters into the excited state. Upon returning from excited state to normal state, energy is released (known as fluorescence) in form of UV light. Intensity of UV light emitted is determined by a photo multiplier tube and converted into an electrical signal, which is a measure of amount of sulphur present in the sample.

NOTES

1 Halide's concentration in excess of 10 percent and nitrogen content in excess of 1 500 ppm may cause interference in determination of total sulphur.

2 One mg/kg is equal to 1 ppm.

B-2.2 Apparatus

B-2.2.1 *Total Sulphur Analyzer*, equipped with combustion furnace; combustion tube, capable to withstand temperature of about 900 °C to 1 200 °C; automatic sample charger; flow controllers; gas driers; UV reaction cell; detector and a software to control the operating parameters.

B-2.2.2 *Syringe*

B-2.3 Reagents

B-2.3.1 *Dibenzothiophene* (purity 98 percent, minimum) or Dibutyl Sulfide (purity 98 percent, minimum)

B-2.3.2 *Inert Gas or Air*, purity 99.99 percent, minimum.

B-2.3.3 *Oxygen Gas,* purity 99.99 percent, minimum.

B-2.3.4 *Solvent*

Any suitable solvents capable of dissolving sulphur compound, like iso-octane, *p*-xylene etc. can be used.

B-2.3.5 *Oxidation Reagent***,** if required.

B-2.4 Calibration and Standardisation

B-2.4.1 For calibration, standards may be prepared using pure chemicals like dibenzothiophene or dibutyl sulfide or ready-made certified reference material may also be used.

B-2.4.2 *Stock Solution Preparation,* approximately 1 000 mg/kg.

B-2.4.2.1 Prepare a stock solution by accurately weighing W_1 g of standard chemical (that is dibenzothiophene or dibutyl sulfide) to the nearest 0.1 mg into 100 ml volumetric flask. Dilute it with relevant solvent like iso-octane, *p*-xylene etc. Concentration of the total sulphur is calculated by the formula as given:

$$\text{Total Sulphur (mg/kg)} = \frac{W_1 \times P \times S \times 10^2}{W_2}$$

where

W_1 = weight of standard chemical, in g;

P = purity of standard chemical, percent;

S = percentage of sulphur in the standard chemical; and

W_2 = total weight (standard + solvent), in g.

B-2.4.3 Prepare standards of varying concentration by diluting the stock solution (**B-2.4.2**) with the same solvent used while preparing stock solution.

B-2.4.4 *Calibration Curve*

Prepare two separate calibration curves for range of 0.1 mg/kg to 1 mg/kg and 1 mg/kg to 10 mg/kg. To perform the calibration, follow manufacturer's recommendation. Solvent used for preparation of standards shall be run along with standards and its value shall be considered as blank while plotting the calibration curve. The calibration curve (µgS vs area) should be linear with minimum 0.99 correlation coefficient.

B-2.5 PROCEDURE

B-2.5.1 The instrument and operating parameters are set as per as per manufacturer's recommendation. Inject the sample size as recommended by the instrument manufacturer, once the sample attains room temperature. Determine the total sulphur value as calculated by the software.

B-2.5.2 Take average of atleast three determination to calculate the total sulphur value. Density of the considered for reproducibility calculations sample value can be used for the unit conversion from mass to volume and vice versa.

B-2.6 Report

Report all sulphur results nearest to 0.1 mg/kg.

B-2.7 Precision and Bias

B-2.7.1 *Repeatability*

Difference between two results obtained in the same laboratory, same operator, and same instrument on same sample shall not differ more than the values shown in the Table 5.

B-2.7.2 *Reproducibility*

Difference between two results obtained in the different laboratories, different operator, and different instrument on different sample shall not differ more than the values shown in the Table 5. Atleast one or all the condition mentioned above is

Table 5 Repeatability and Reproducibility
(*Clauses* B-2.7.1 *and* B-2.7.2)

Sl No.	Amount of Sulphur	Repeatability	Reproducibility
(1)	(2)	(3)	(4)
i)	0.5 mg/kg	0.04	0.22
ii)	1.4 mg/kg	0.11	0.59
iii)	5.3 mg/kg	0.20	0.64

ANNEX C

[*Table 1, Sl No*. (viii)]

DETERMINATION OF APPEARANCE (VISUAL APPEARANCE)

C-1 OUTLINE OF THE TEST METHOD

This method is used to check the visual appearance of the high purity *p*-xylene. This test is carried out at room temperature. A known volume of sample is transferred into a clear transparent glass bottle from the sample container. The bottle is held upright against the bright white light and inspected for the presence of the foreign material.

C-2 PROCEDURE

C-2.1 Shake vigorously the sample container for atleast 2 min, once the sample attain room temperature. Now immediately transfer 750 ml of sample into a clean and dust free 1 litre transparent clear glass bottle.

C-2.2 Allow the liquid to settle. Hold the bottle against the bright white light and visually examine for presence of free water, haziness, cloudiness and particles of size greater than 1 mm. Gently swirl the liquid to create a vertex and observe for any particles that might settled at bottom. The sample shall not contain more than six particles of size greater than or equal to 1 mm.

C-3 REPORTING

C-3.1 Samples found free of any of the contaminants (**C-2.2**) shall be reported as “Pass”. Samples not passing the test shall be reported as “Fail” along with the type of contaminant observed.

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

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

Organization	*Representative(s)*
National Chemical Laboratory (NCL), Pune	Dr C. V. Rode **(*Chairperson*)**
Alkyl Amines Chemicals Limited, Mumbai	Shri S. V. Nikumbhe Shri Sameer Katdare (*Alternate*)
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	Shri K. L. Raphael Shri Kirti Gajjar (*Alternate*)
All India Distillers Association (AIDA), New Delhi	Shri V. N. Raina
BASF India Limited, Mumbai	Shri Kiran Bhat Shri Hemal (*Alternate*)
CSIR-Central Drug Research Institute (CDRI), Lucknow	Dr Sanjeev Kanojiya
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Department of Chemicals and Petrochemicals, Ministry of Chemicals and Fertilizers, New Delhi	Shri O. P. Sharma Shri Varun Singh Poonia (*Alternate*)
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Reliance India Limited (RIL), Mumbai	Shri Sreeramachandran Kartha Shri Vasant Warke (*Alternate*)

Organization	*Representative(s)*
United Phosphorus Limited (UPL), Mumbai	SHRI M. D. VACHHANI
In Personal Capacity (*37 Nandanvan Society, Near GNFC Township, Narmada Nagar, Bharuch, 392015*)	DR MAYUR J. KAPADIA
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