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**Committee D16 on Aromatic Hydrocarbons and Related Chemicals
Subcommittee D16.02 on oxygenated aromatics**

Research Report D16-1072

**Interlaboratory Study to Establish Precision Statements for ASTM D7883,
Determination of 4-Carboxybenzaldehyde and p-Toluic Acid
in Purified Terephthalic Acid by Weak Anion Exchange High
Performance Liquid Chromatography**

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1. Introduction:

1.1 This test method covers the determination of the 4-Carboxybenzaldehyde (4-CBA) and p-Toluic acid (p-TOL) in purified terephthalic acid (PTA) by weak anion exchange high performance liquid chromatography (HPLC). This method is applicable for 4-CBA from 2 to 500 mg/kg and for p-TOL from 10 to 500 mg/kg, respectively.

2. Test Method:

2.1 Weak Anion Exchange HPLC Method- PTA sample is dissolved in ammonium hydroxide solution. After pH adjustment, a fixed volume of this solution is injected into a high performance liquid chromatograph equipped with a UV detector. An anion-exchange column is used to separate the impurities 4-CBA and p-TOL from PTA. The external standard calibration is used for quantification.

3. Participating Laboratories:

3.1 The following laboratory participated in this study:

- 1) Lab D: Shanghai Research Institute of Petrochemical Technology
Contact: Yuhong Zhang
- 2) Lab E: SINOPEC Luoyang Engineering Co. LTD
Contact: Zhenguo Yang
- 3) Lab F: PetroChina Wulumuqi Petrochemical Company
Contact: Guangshuo Yi
- 4) Lab G: SINOPEC Tianjin Petrochemical Company
Contact: Jianzhong Liu
- 5) Lab H: SINOPEC Yangzi Petrochemical Company
Contact: Daxi Ding

4. Description of Samples:

4.1 Five PTA samples with different concentrations of 4-CBA and p-TOL are tested in this study. The expected concentrations of the component interest are listed in Table 1.

Table 1 Expected Concentrations of 4-CBA and p-TOL in PTA (mg/kg)

	Level I	Level II	Level III	Level IV	Level V
4-CBA	11	17	25.1	4~5	2
p-TOL	210	100	127.3	210	63

5. Inter-laboratory Study Instructions:

5.1 An ILS was conducted which included 5 laboratories analyzing 5 samples 2 times. The operation conditions are showed in Table 2.

Table 2. The Operation Conditions

Column	Weak alkali anion exchange
stationary phase	(-NMe ₂) chemically bonded silica
Particle size	3 μm
Material of column	Stainless steel
Length of column	50 mm
Inner diameter	4 mm
Mobile phase	0.1 mol/L NH ₄ H ₂ PO ₄ solution:acetonitrile (or methanol) =9:1
Flow rate	1.0 mL/min
UV Detector	258 nm for 4-CBA 236 nm for p-TOL
Injection amount	20μL
Column temperature	35°C

6. Description of Equipment/Apparatus¹:

6.1 High Performance Liquid Chromatograph (HPLC)-Any HPLC capable of pumping the mobile phase at flow rates between 0.1 and 2.0 mL/min, with a pressure between 0 and 40 MPa and a pulsation of less than 1% full scale deflection under the test conditions described in Table 2. The S/N (signal to noise) ratio should be 3:1 or greater for 2 mg/kg 4-CBA and 10 mg/kg p-TOL.

¹ The equipment listed was used to develop a precision statement for D7883-19. This listing is not an endorsement or certification by ASTM International.

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