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

Research Report D16-1073

**Interlaboratory Study to Establish Precision Statements for ASTM D7884,
Determination of 4-Carboxybenzaldehyde and p-Toluic Acid
in Purified Terephthalic Acid by Reverse Phase 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 reverse phase 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 Reverse Phase HPLC Method- PTA sample is dissolved in ammonium hydroxide solution, and a fixed volume of this solution is injected into a high performance liquid chromatograph equipped with a UV detector. A C18 chemically bonded 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 I: Shanghai Research Institute of Petrochemical Technology
Contact: Yuhong Zhang
- 2) Lab J: Ningbo Mitsubishi Chemical
Contact: Yun Chen
- 3) Lab K: Agilent Technology
Contact: Bo Chen

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 3 laboratories analyzing 5 samples 2 times. The operation conditions are showed in Table 2.

Table 2. The Operation Conditions

Column	C18
stationary phase	Octadecylsilane chemically bonded silica
Particle size	5 μm
Material of column	Stainless steel
Length of column	150 mm
Inner diameter	4.6 mm
Mobile phase	0.06% H_3PO_4 solution : acetonitrile=82:18
Flow rate	1.0 mL/min
UV Detector	254 nm for 4-CBA, 240 nm for p-TOL
Injection amount	20 μL
Column temperature	40°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.

6.2 *Sample Injection System*- capable of injecting 1 to 25 μL , using either partial or full loop mode, with a repeatability of $\pm 1\%$.

6.3 *Detector*-Variable Wavelength Ultraviolet Photometric Detector (VWD), multi-wavelength detector, or Photometric Diode Array Detector (PDA), capable of operating at 240 and 254 nm.

6.4 *Column Oven*- Any suitable HPLC column oven (block heating or air circulating) capable of maintaining a constant temperature of $\pm 1^\circ\text{C}$ within the range of 20-70°C.

6.5 Chromatography Data system

6.6 HPLC Columns

¹ The equipment listed was used to develop a precision statement for [Standard's Designation with Date]. This listing is not an endorsement or certification by ASTM International.
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