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Committee D16 on Aromatic Hydrocarbons and Related Chemicals Subcommittee D16.02 on Oxygenated Aromatics

Research Report: D16-2003

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/ Background:

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 The Test Method used for this ILS is D7884-20. To obtain a copy of D7884, go to ASTM's website, www.astm.org, or contact ASTM Customer Service by phone at 610-832-9585 (8:30 a.m. - 6:00 p.m. Eastern U.S. Standard Time, Monday through Friday) or by email at service@astm.org.

2.2 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:

The following laboratories participated in this interlaboratory study:

Shanghai Research Institute of Petrochemical Technology

Contact: Yuhong Zhang

Ningbo Mitsubishi Chemical Contact: Yun Chen

Agilent Technology Contact: Bo Chen

4. Description of Samples:

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