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1 August 2013

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

Research Report: D16-1047

**Intralaboratory Study to Establish Precision Statements for ASTM
D7883-2013 Standard Test Method for 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:

The presence of 4-CBA and p-TOL in PTA used for the production of polyester is undesirable because they can slow down the polymerization process, and 4-CBA is also imparting coloration to the polymer due to thermal instability. Determining the amount of 4-CBA and p-TOL remaining from the manufacture of PTA is often required.

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.

This test method is suitable for setting specifications and for use as an internal quality control tool where these products are produced or are used.

2. Test 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.

The Test Method used for this ILS is [Standard's Designation with Date]. To obtain a copy of [Standard Designation], go to ASTM's website, www.astm.org, or contact ASTM Customer Service by phone at 610-832-9585 (8:30 a.m. - 4:30 p.m. Eastern U.S. Standard Time, Monday through Friday) or by email at service@astm.org.

3. Participating Laboratory:

The following laboratory participated in this study:

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4. Description of Samples:

There were 1 PTA sample and 1 QTA (Qualified Terephthalic Acid) sample used for this study. Both of them were provided by Mitsubishi Chemical Corporation.

5. Interlaboratory Study Instructions

- 5.1 Dissolve PTA/QTA sample in ammonium hydroxide solution.
- 5.2 Make sure the chromatogram resembles peaks shown in D7883 Fig.1.
- 5.3 Inject appropriate amount of sample into the instrument.
- 5.4 Review the result of the test.

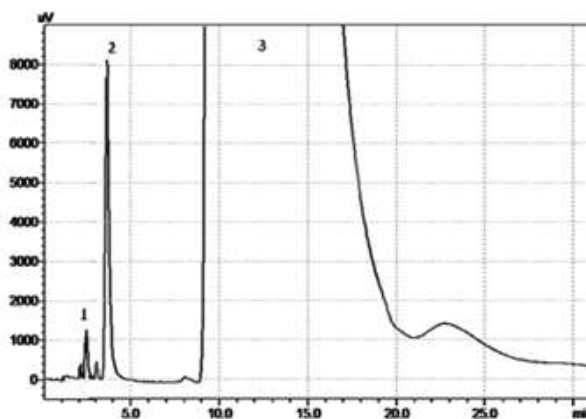


FIG. 1. Chromatogram of a PTA Sample (weak anion exchange HPLC) 1-4-CBA, 2-*p*-TOL, 3-PTA

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 D7883 Table 1. 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 μ L, using either partial or full loop mode, with a repeatability of $\pm 1\%$.

¹ The equipment listed was used to develop a precision statement for D7883-13. This listing is not an endorsement or certification by ASTM International.
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