

This Research Report is issued under the fixed designation RR: D16-1049. You agree not to reproduce or circulate or quote, in whole or part, this document outside of ASTM International Committee/Society activities, or submit it to any other organization or standards body (whether national, international or other) except with the approval of the Chairman of the Committee having jurisdiction and the written authorization of the President of the Society. If you do not agree to these conditions, please immediately destroy all copies of this document. *Copyright ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. All rights reserved.*

15 August 2013

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

Research Report D16-1049

**Intralaboratory Study to Establish Precision Statements for ASTM
D7881-2013 Standard Test Method for Determination of 4-
Carboxybenzaldehyde and p-Toluic Acid in Purified Terephthalic Acid
by Capillary Electrophoresis with Reverse Voltage Mode**

Technical contact:

Analytical specialist
Yuhong Zhang
SINOPEC
Shanghai, 201208
China
zhangyh.sshy@sinopec.com

ASTM International
100 Barr Harbor Drive
West Conshohocken, PA 19428-2959

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 capillary electrophoresis (CE) with reverse voltage mode. This method is applicable for 4-CBA from 3 to 400 mg/kg and for p-TOL from 8 to 400 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:

A PTA sample is dissolved in ammonium hydroxide solution. 4-CBA, p-TOL and PTA dissociate and become homologous ions under basic conditions. A fixed amount of this solution is introduced into the capillary using hydrodynamic sampling or electrokinetic sampling. A voltage is applied to the capillary to separate the impurities, 4-CBA and p-TOL, from PTA. External standard calibration is used for quantification.

3. Participating Laboratories:

The following laboratory participated in this study:

China Petrochemical Corporation
1658 Pudong Beilu
SHANGHAI, SH
201208
CN
Yuhong Zhang
zhangyh.sshy@sinopec.com

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 electropherogram resembles peaks shown in D7881 Fig.3.
- 5.3 Inject appropriate amount of sample into the instrument.
- 5.4 Review the electrophoretic data system result of the test.

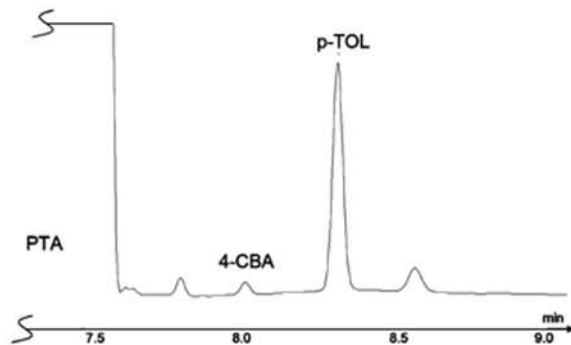


FIG. 3 Electropherogram of a PTA Sample in the Reverse Voltage Mode

6. Description of Equipment/Apparatus¹:

- 6.1 *Capillary Electrophoresis System*, the system consists of the following components, as shown in D7881 Fig.2, or equivalent:

¹ The equipment listed was used to develop a precision statement for D7881-13. This listing is not an endorsement or certification by ASTM International.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.