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

Research Report D16-1050

Intralaboratory Study to Establish Precision Statements for ASTM D7882-2013 Standard Test Method for Determination of 4-Carboxybenzaldehyde and p-Toluic Acid in Purified Terephthalic Acid by Capillary Electrophoresis with Normal Voltage Mode

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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 capillary electrophoresis (CE) with mormal voltage mode. This method is applicable for 4-CBA from 5 to 400 mg/kg and for p-TOL from 10 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. 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 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 electropherogram resembles peaks shown in D7882 Fig.3.

5.3 Inject appropriate amount of sample into the instrument.

5.4 Review the electrophoretic data system result of the test.

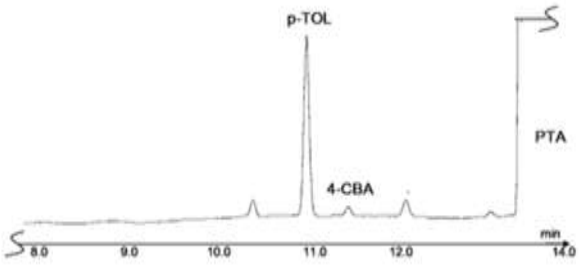

FIG. 3 Electropherogram of a PTA Sample in the Normal Voltage Mode

6. Description of Equipment/Apparatus[1]:

6.1 *Capillary Electrophoresis System*, the system consists of the following components, as shown in D7882 Fig.2, or equivalent:

[1] The equipment listed was used to develop a precision statement for D7882-13. This listing is not an endorsement or certification by ASTM International.