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

Research Report D16-1070

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

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1. Introduction:

1.1 This test method covers the determination of 4-carboxybenzaldehyde (4-CBA) and p-toluic acid (p-TOL) in purified terephthalic acid (PTA) by capillary electrophoresis (CE) with reverse voltage mode and UV detection. It is applicable for 4-CBA from 3 to 400 mg/kg and for p-TOL from 8 to 400 mg/kg, respectively.

2. Test Method:

2.1 A PTA sample is dissolved in ammonium hydroxide. The 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:

3.1 The following laboratory participated in this study:

- 1) Lab A: SINOPEC Yangzi Petrochemical Company
Contact: Daxi Ding
- 2) Lab B: Agilent
Contact: Bo Chen
- 3) Lab C: Zhejiang Yisheng Petrochemical Company
Contact: Houjun Ke
- 4) Lab D: Dalian Yisheng Petrochemical Company
Contact: Yongming Jiang
- 5) Lab E: Xianglu Petrochemicals
Contact: Zhihong Li
- 6) Lab F: BP(Zhuhai)
Contact: Yubo Guo
- 7) Lab G: Chongqing Pengwei Petrochemical Company
Contact: Shiyun Wang
- 8) Lab H: SINOPEC Yizheng Chemical Fiber Co. LTD
Contact: Liuliu Gong
- 9) Lab I: Zhejiang Asia Petrochemical Company
Contact: Jichi Wang
- 10) Lab J: Beckman
Contact: Peng Zhang

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 10 laboratories involved in the ILS. 5 laboratories tested the samples with mode 1 and the others tested with mode 2. Each sample was analyzed twice. The operation conditions are showed in Table 2.

Table 2. The Operation Conditions of Capillary Electrophoresis

Mode 1	Electrolyte: 50 mM sodium 1-hexanesulfonate, 10 mM disodium hydrogen phosphate and 1 mM OFM (pH = 10.5–11.0) Capillary: 60 cm × 75 μm (Effective length: 50cm) Applied voltage: -20 kV Detector: UV 200 nm Capillary temperature: 25 °C Injection technique: Electrokinetic sampling –10 kv × 90 s Capillary purge program: NaOH solution 1 min; water 2 min; electrolyte 3 min
Mode 2	Electrolyte: 50 mM sodium 1-hexanesulfonate, 5 mM CAPS and 1 mM OFM (pH = 10.5–11.0) Capillary: 60 cm × 75 μm (Effective length: 50cm) Applied voltage: -20 kV Detector: UV 200 nm Capillary temperature: 25 °C Injection technique: Electrokinetic sampling –10 kv × 90 s Capillary purge program: NaOH solution 1 min; water 2 min; electrolyte 3 min

6. Description of Equipment/Apparatus1:

6.1 Capillary Electrophoresis System—the system consists of the following components, as shown in Fig. 1 or equivalent:

¹ The equipment listed was used to develop a precision statement for D7881-19. This listing is not an endorsement or certification by ASTM International.

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