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

Research Report: D16-1067

Intralaboratory Study to Establish Precision Statements for ASTM D8207, Test Method for Determination of Metals in Purified Terephthalic Acid (PTA) by Atomic Absorption (AA) Spectrometry

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1. Introduction:

The presence of metals in PTA used for the production of polyester is undesirable because they may speed up or slow down the reaction and be impurities in the final product.

Determination of metals in PTA is often required. This test method is suitable for setting specifications and for use as an internal quality control tool where these products are produced or used.

This test method covers the determination of aluminum, chromium, cobalt, titanium, iron, manganese, molybdenum, sodium and nickel in purified terephthalic acid (PAT) by graphite furnace atomic absorption (GFAA) spectrometry. It is applicable to PAT samples containing sodium, chromium, cobalt, titanium, manganese, molybdenum and nickel over 0.004 mg/kg, and iron and aluminum over 0.054 mg/kg.

2. Test Method:

PTA sample in a platinum crucible is ignited and burned, then ashed at $750\,^{\circ}\mathrm{C}$ approximately for 45min in a muffle fumace, and finally dissolved with sulfuric acid solution. The sample solution is analyzed by graphite fumace atomic absorption spectrophotometer. The external standard calibration is used for quantification.

3. Participating Laboratory:

The following laboratory participated in this study:

China Petrochemical Corporation, Yangzi Petrochemical Company Limited

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4. Description of Samples:

There was 1 PTA sample used for this study. The sample was provided by China Petrochemical Corporation.

5. Interlaboratory Study Instructions

Prepare the graphite furnace atomic absorption spectrometer according to the manufacturer's instructions.

Ignite, burn and ash PTA sample at 750 °C, and then dissolve in sulfuric acid solution.

Freshly prepare multi-element standard stock solution, sodium standard solution, mixed working standards, sodium working standards and work standard daily when samples are to be analyzed.

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