⟨223⟩ DIMETHYLANILINE

The following limit test is provided as a general procedure, when specified in the individual monographs for the gas chromatographic determination in compendial articles of traces of dimethylaniline, a hydrochloric acid scavenger that may have been carried over during processing.

PROCEDURE

Internal standard solution: Unless otherwise specified in the individual monograph, prepare a solution of naphthalene in cyclohexane containing about 50 µg per mL.

Standard preparation: Unless otherwise specified in the individual monograph, transfer 50.0 mg of *N*,*N*-dimethylaniline to a 50-mL volumetric flask, add 25 mL of 1 N hydrochloric acid, swirl to dissolve, dilute with water to volume, and mix. Transfer 5.0 mL of the resulting solution to a 250-mL volumetric flask, dilute with water to volume, and mix. To a suitable centrifuge tube add 1.0 mL of this solution, 5.0 mL of 1 N sodium hydroxide, and 1.0 mL of *Internal standard solution*, shake vigorously for 1 minute, and centrifuge. Use the clear supernatant as the *Standard preparation.*

Test preparation: Unless otherwise specified in the individual monograph, transfer 1.0 g of the substance to be tested to a suitable centrifuge tube, add 5 mL of 1 N sodium hydroxide, swirl to dissolve the specimen, add 1.0 mL of *Internal standard solution*, shake vigorously for 1 minute, and centrifuge. Use the clear supernatant as the *Test preparation.*

Chromatographic system

(See *Chromatography* ⟨621⟩.)

The gas chromatograph is equipped with a flame-ionization detector, maintained at about 250°, and a 0.53-mm × 30-m fused silica capillary column bonded with a 1.0-µm film of phase G42. The carrier gas is helium, with a linear velocity of about 30 cm per second and a split ratio of 10:1. The column temperature is maintained at 110° for the first 4 minutes after an injection is made, then increased from 110° to 200° at 8° per minute, and then held at 200° for 5 minutes. The injection port temperature is maintained at 250°. Chromatograph the *Standard preparation*, and record the responses as directed for *Procedure* identify the dimethylaniline and naphthalene peaks by their relative retention times, which are 1.0 and 1.3, respectively. The signal-to-noise ratio for the dimethylaniline peak is not less than 10.

Procedure: Inject equal volumes (about 1 µL) of the *Standard preparation* and the *Test preparation* into the chromatograph, record the chromatograms, and measure the areas for the major peaks. The ratio of the response of any dimethylaniline peak to the response of the naphthalene peak obtained from the *Test preparation* is not greater than that obtained from the *Standard preparation* (0.002%).

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