



Designation: D 6505 – 00

## Standard Test Method for Assay of *normal*-Propyl Bromide Content<sup>1</sup>

This standard is issued under the fixed designation D 6505; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### 1. Scope

1.1 This test method provides a basis for the determination of the *normal*-propyl bromide (weight %) in the presence of stabilizers and impurities, in virgin or reclaimed *normal*-propyl bromide (*n*PB). The application range is from 50 to 100 wt %.

### 2. Summary of Test Method

2.1 This analytical test method describes the determination of the assay of *n*-propyl bromide by gas chromatography using an internal standard. The results are reported in weight percent.

### 3. Significance and Use

3.1 This test method provides an analysis in weight percent of the *normal*-propyl bromide concentration of virgin, formulated, or reclaimed *normal*-propyl bromide: compounds that co-elute with *normal*-propyl bromide or *normal*-heptane (internal standard) may interfere with this test method.

### 4. Apparatus

4.1 *Gas Chromatograph*—Hewlett Packard 5890 Series II equipped with a split/splitless injector, a flame ionization detector, and an autosampler, HP, or equivalent, if available.

4.2 *Column*—30 m by 0.25 mm, 1.0- $\mu$ m film thickness AllTech AT-5, or equivalent, capillary column.

4.3 *Syringe*—5- $\mu$ L or 10- $\mu$ L GC autosampler syringe.

4.4 *Data Acquisition and Analysis Device*—VAX MULTI-CHROM or equivalent chromatography data collection and processing system, or integrator.

4.5 *Crimp Top Vials*—Hewlett Packard 2-mL glass crimp top vials with TFE-fluorocarbon-lined silicone septa or equivalent.

4.6 *Volumetric Pipet*—25 mL.

4.7 *Glass Vials*—32-mL glass vials (25 by 95 mm), with TFE-fluorocarbon-lined screw caps.

4.8 *Transfer Pipets*—Borosilicate glass transfer pipets.

4.9 *Precision Balance*, accurate to 0.1 mg.

### 5. Required Chemicals

5.1 *n*-Propyl Bromide (1-Bromopropane), 99+ %, Aldrich Chemical Company

5.2 *n*-Heptane (internal standard), Reagent Grade, 99.5+ %, J.T. Baker (or equivalent)

5.3 *Methylene Chloride*, 99+ %, Aldrich Chemical Company (or equivalent)

### 6. Suggested Analysis Conditions

#### 6.1 Chromatographic Conditions:

Injector	150°C
Detector temperature	250°C
FID detector range	most sensitive setting
Temperature program	30°C for 10 min 30 to 250°C at 15°C/min hold at 250°C for 10 min
Carrier gas	He (or N <sub>2</sub> ) at 1 mL/min split flow: 25 mL/min column head pressure: 7.5 psi
Make-up gas	He (or N <sub>2</sub> ) at 30 mL/min
Injection volume	0.5 $\mu$ L

### 7. Analytical Procedure

7.1 *Internal Standard Calibration Mixture*—Carefully weigh about 0.25 g of *n*-heptane internal standard and about 0.5 g of *n*-propyl bromide into a 32-mL glass vial. Record the actual weights for each. Pipet 25 mL of methylene chloride into this vial and cap immediately. Mix well and then transfer approximately 1 mL of this standard solution into an autosampler vial. Cap the vial and analyze in accordance with the conditions specified in the chromatographic conditions section. Inject this standard at least three times and calibrate an internal standard method in the data system using the results from the standard analyses.

7.2 *Preparation of Samples for Analysis*—Weigh about 0.25 g of *n*-heptane and about 0.5 g of sample into a 32-mL glass vial. Record the actual weights for each. Pipet 25 mL of methylene chloride into this vial and cap immediately. Mix well and transfer approximately 1 mL of this solution to an autosampler vial. Cap the vial and analyze in accordance with the conditions specified in the chromatographic conditions section. The following table shows components of interest, their absolute retention time (ART) in minutes, their relative retention time (RRT) in relation to *n*-heptane, the internal standard, as well as their relative response factor (RRf).

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-26 on Halogenated Organic Solvents and Fire Extinguishing Agents and is the direct responsibility of Subcommittee D26.04 on Test Methods.

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