



Standard Test Method for Assay of *normal*-Propyl Bromide Content¹

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 reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

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- μ m film thickness AllTech AT-5, or equivalent, capillary column.

4.3 *Syringe*—5- μ L or 10- μ 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 ₂) at 1 mL/min split flow: 25 mL/min column head pressure: 7.5 psi
Make-up gas	He (or N ₂) at 30 mL/min
Injection volume	0.5 μ 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).

Component	ART	RRT	RRf
<i>n</i> -propyl bromide	13.30	0.83	0.3495
<i>n</i> -heptane (internal standard)	16.08	1.00	1.0000

¹ 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.

Current edition approved Jan. 10, 2000. Published April 2000.

8. Calculation

8.1 A calibrated internal standard weight percent method in the data system will yield the required weight percent data. Alternatively, the weight percent calculations may be performed manually as described as follows:

8.2 Calculate the relative response factor (RRf) for *n*-propyl bromide in the standard relative to the *n*-heptane internal standard. Average the peak areas of the three standard injections:

$$\text{RRf} = \frac{A_y \times W_{tl}}{W_{ty} \times A_l} \quad (1)$$

where:

- A_y = average area of *n*-propyl bromide peak in standard calibration mixture,
- A_l = average area of *n*-heptane (internal standard) peak in standard calibration mixture,
- W_{ty} = amount of *n*-propyl bromide in standard calibration mixture, and
- W_{tl} = amount of *n*-heptane (internal standard) in standard calibration mixture.

The assay of *n*-propyl bromide (weight % *y*) in the sample is calculated as follows from the sample injection:

$$\text{weight \% } y = \frac{A_y \times W_{tl} \times 100}{A_l \times \text{RRf} \times W_{tS}} \quad (2)$$

where:

- A_y = area of *n*-propyl bromide peak in sample mixture,

- A_l = area of *n*-heptane (internal standard) peak in sample mixture,
- W_{tl} = weight of internal standard in sample mixture,
- W_{tS} = weight of sample in sample mixture, and
- RRf = relative response factor from above.

9. Precision and Bias

9.1 *Precision*—The following criteria should be used for judging the acceptability of results:

9.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.037 % absolute at 22 df. The 95 % limit for the difference between 2 such runs is 0.10 % absolute.

9.1.2 *Laboratory Precision (Within-Laboratories, Between Days Variability, formerly called Repeatability)*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.62 % absolute at 8 df. The 95 % limit for the difference between 2 such runs is 1.72 % absolute.

9.1.3 *Reproducibility, Multilaboratory*—The standard deviation of results (each the average of duplicates) obtained by analysts at different laboratories, has been estimated to be 0.93 % absolute at 7 df. The 95 % limit for the difference between 2 such runs is 2.39 % absolute.

10. Keywords

10.1 assay; brominated; gas chromatography; halogenated; *normal*-propyl bromide; purity; solvent

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