

Product Information

394912 Heptafluorobutyric anhydride

for GC derivatization, LiChropur®

Storage temperature: room temperature

Pentafluoroacetic acid anhydride (HFBA), a perfluoroacylated acylation reagent, forms stable, volatile derivatives with alcohols, amines, and phenols. The major use for HFBA is to prepare electroncapturing derivatives for GC/electron capture detection. This detector provides greatly enhanced responses for halogenated derivatives. Although the anhydride may be used on its own, the acylation reactions go most smoothly and quickly in a solvent and with a catalyst. Bases such as triethylamine (TEA) and trimethylamine (TMA) often are added to promote reactivity. HFBA should be used with an acid scavenger, to help drive the reaction to completion and to prevent column damage from acidic by-products of the derivatization reaction.

Features/Benefits

- Produces stable, volatile derivatives of alcohols, amines, and phenols for electron capture or flame ionization detection.
- Frequently used in confirmation testing for drugs of abuse by GC/ MS. HFBA is used to identify amphetamines and phencyclidine.

Typical Procedure

This procedure is intended to be a guideline and may be adapted as necessary to meet the needs of a specific application. Always take proper safety precautions when using a acetylating reagent.

Prepare a reagent blank (all components, solvents except sample), following the same procedure as used for the sample.

- 1. Dissolve 50 μg sample (250 μg for FID) in 0.5 mL benzene.
- 2. Add 0.1 mL 0.05 M TMA (acid scavenger) in benzene, followed by 10 µL HFBA.
- 3. Cap the vial and heat at 50°C for 15 min.
- 4. Cool the mix and add 1 mL 5% ammonia in water.
- 5. Shake for 5 min., allow the layers to separate and inject an aliquot of the benzene (upper) layer onto the chromatograph.

Derivatization times vary widely, depending upon the specific compound(s) being derivatized. If derivatization is not complete, evaluate the addition of a catalyst, use of an appropriate solvent, higher temperature, longer time and/or higher reagent concentration.

Mechanism¹⁻²

Acylation involves the introduction of an acyl group into a molecule that has a replaceable hydrogen atom (OH, NH or SH group). Except for TFAA, anhydride acylating reagents form acidic byproducts that must be removed prior to GC analysis, to prevent destructive effects on the phase in the column. Consequently, acylations with anhydride reagents normally are performed in pyridine, tetrahydrofuran, or another solvent capable of accepting the acid byproducts.

$$R^{O}H \xrightarrow{C_3F_7} O \xrightarrow{C_3F_7} R^{O} C_3F_7 + C_3F_7 O H$$

Storage/Stability

Recommended storage conditions for the unopened product are stated on the label. Store in a bottle or ampule at room temperature in a dry, well ventilated area. Use only in a well ventilated area. Keep away from ignition sources. **Before reuse, validate that your storage conditions adequately protected the reagent.**

References

- 1. K. Blau and J. Halket, *Handbook of Derivatives* for *Chromatography* (2nd ed.), John Wiley & Sons, New York, 1993.
- 2. D.R. Knapp, *Handbook of Analytical Derivatization Reactions,* John Wiley & Sons, New York, 1979.

Precautions and Disclaimer

This product is for R&D use only, not for drug, household, or other uses.

Please consult the Safety Data Sheet for information regarding hazards and safe handling practices.