

Product Information

91204 Acetic anhydride for GC derivatization, LiChropur®

Storage temperature: room temperature

Acetic anhydride is an acylation reagent. Acylation reduces the polarity of amino, hydroxy, and thiol groups. Acylation may improve the stability of a compound by protecting unstable groups, and may increase volatility. Acetic anhydride can be used with a basic catalyst, such as pyridine. This combination promotes smooth reactions and has great solvent power. Pyridine acts as an acceptor for the acid by-product formed in the reaction. Pyridine may also react with acetic anhydride, however, forming N-acetyl-1,2-dihydro-2-pyridylacetic acid.

Features/Benefits

 Acylation is an alternative to silylation, producing stable, volatile derivatives of alcohols, phenols, and amines for analysis by GC/ FID. Acetylated compounds are more stable than corresponding silylated compounds.

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.

General Procedure

- 1. Dissolve 5 mg sample in 5 mL chloroform.
- 2. Add 0.5 mL acetic anhydride and 1 mL acetic acid. Heat at 50°C for 2-16 hours.
- 3. Remove excess reagent by evaporating the mixture to dryness and redissolve the residue in chloroform for analysis by GC.

Alditol Formation

- 1. Dissolve 5 mg sample in 5 mL chloroform.
- 2. Add 1 mL acetic anhydride:pyridine, 1:1. Heat at 100°C for 20 min.
- 3. Remove excess reagent by evaporating the mixture to dryness and redissolve the residue in ethyl acetate for analysis by GC.

Derivatization times vary widely, depending upon the specific compound(s) being derivatized. To determine when derivatization is

complete, analyze aliquots of the sample at selected time intervals until no further increase in product peak(s) is observed. 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). Anhydride acylating reagents form acidic by-products 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 by-products

$$R$$
 H_3C O CH_3 CH_3 O CH_3

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. Before reuse, validate that your storage conditions adequately protected the reagent.

References

- 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.

