Product Information

90257 2,3,4,5,6-Pentafluorobenzyl bromide
for GC derivatization, LiChropur®

80833 18-Crown-6
LiChropur®

Storage temperature: room temperature

Pentafluorobenzyl bromide (PFBBr) converts carboxylic acids, mercaptans, phenols, and sulfonamides to halogenated derivatives that are easily detected by electron capture. Electron capturing esters are popular for GC analyses of short chain fatty acids. The reagent can be used for identification and detection of trace amounts of carboxylic acids, mercaptans, and phenols in drinking water. PFBBr is used in extractive alkylation (simultaneous extraction and derivatization), in conjunction with tetrabutylammonium hydrogen sulfate as the counterion. This procedure, also called ion paired extraction, allows extraction/derivatization analyses of drugs from biological matrices. The analyte is removed as an ion (anion) through the use of a quaternary ammonium cation. The anion moves from the aqueous phase to the organic phase when a sample-specific pH is achieved. Once in the organic phase, the anion comes in contact with the alkylation reagent, PFBBr, and is easily derivatized.

1,4,7,10,13,16-Hexaoxacyclooctadecane (18-Crown-6) is an 18-membered crown ether ring with 6 oxygen atoms. 18-Crown-6 (and other crown ethers) is a phase transfer catalyst, forming complexes with many cations, particularly potassium, in nonpolar organic solvents. In the reaction of a crown ether with the potassium salt of an acid the potassium ion is complexed into the center of the ring, mainly through electrostatic forces. This makes the anionic portion of the analyte molecule very reactive to an alkyl halide, leading to alkylation under mild conditions. PFBBr and 18-Crown-6 are used in combination to prepare pentafluorobenzyl-phenol derivatives for US Environmental Protection Agency Method 604 (analyses of phenols in wastewater).

Features/Benefits
- PFBBr converts carboxylic acids, mercaptans, phenols, and sulfonamides to halogenated derivatives easily monitored by electron capture. The derivatives also are detectable by UV, for HPLC and TLC applications.
- Extraction alkylation with PFBBr allows simultaneous extraction and derivatization.
- 18 crown 6 is effective for analyses of small cations.

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.

Acids
1. Combine 0.8 mg acid and 100 mL acetone. Add 250 mg PFBBr and 50 mg potassium bicarbonate.*
2. Reflux for 3 hours.
3. Add 500 mL ethyl ether and 20 mL ethyl acetate.
4. Wash briefly with water, then dry over sodium sulfate and evaporate to dryness.
5. Dissolve residue in hexane containing 1% acetone and 1% ethanol.
6. Analyze 1 µL aliquot by GC

   Note: In evaporating the extract, expect some loss of the highly volatile derivatives of low molecular weight acids. Water in the reaction mixture will lead to production of artifacts.

* The crown ether confers high reactivity on the sample anion, eliminating the need for a large excess of reagent and subsequent removal of the excess before GC/ ECD. Use a strong base (*e.g.* K_2CO_3) to derivatize both carboxylic acids and phenols; a weaker base (e.g., KOAc, $KHCO_3$, KCNO) will give selective derivatization of carboxylic acids.

Extractive Alkylation

1. Combine 0.2 mg sample and 1 mL methylene chloride in a reaction vessel.
2. Add 1 mL 0.1 M tetrabutylammonium hydrogen sulfate, 1 mL 0.2 M sodium hydroxide and 25 µL PFBBr and cap.
3. Shake for 20-30 min at 25°C
4. Analyze aliquots by GC/FID.

   For GC/ECD analysis, evaporate to dryness to eliminate methylene chloride. Dissolve the residue in an appropriate solvent.

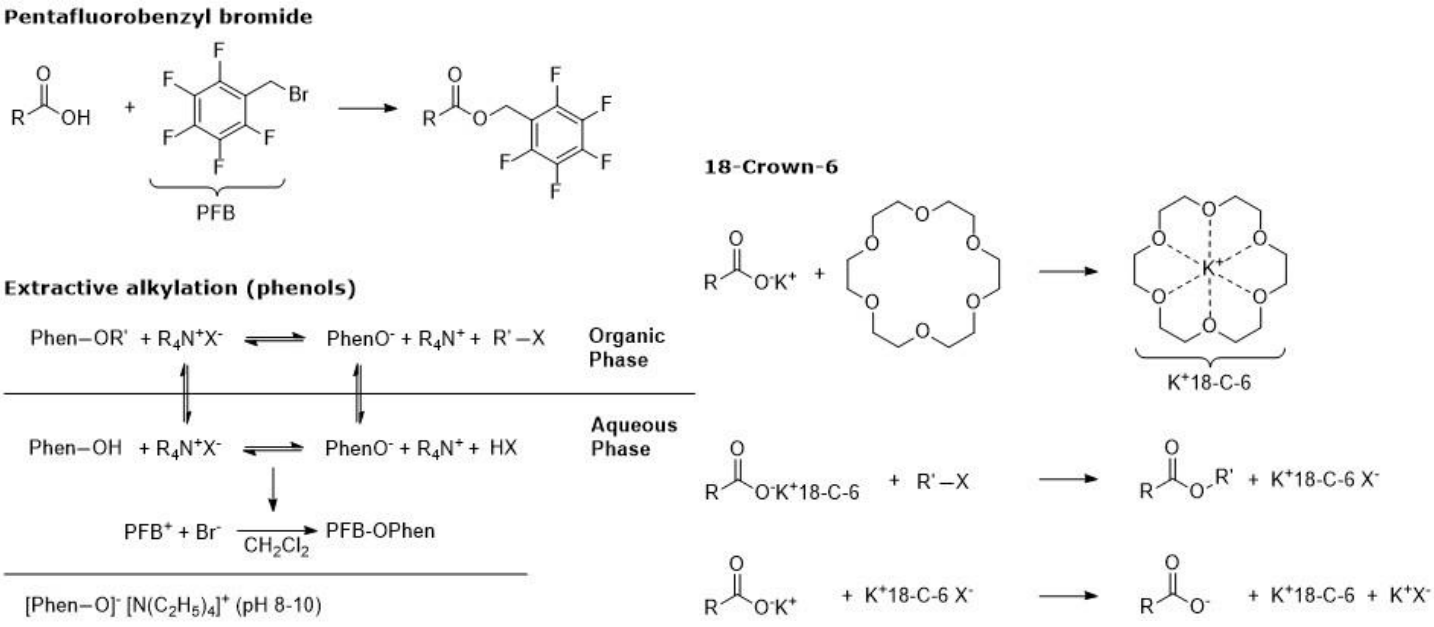
Reagent for Preparing Pentafluorobenzyl-Phenol Derivatives

Combine 1 mL PFBBr and 1 g 18-Crown-6. Dilute with 50 mL 2-propanol.

1 mL of this reagent will derivatize up to 0.3 mg of phenols. US EPA Method 604 describes how to add the reagent to the sample.

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.

Mechanism[1-2]



Storage/Stability

Recommended storage conditions for the unopened product are stated on the label. Store in a bottle or ampule in a cooler 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.