

BSA + TMCS

Product Specification

The combination of **BSA (N,O-bis(trimethylsilyl)acetamide)** and **TMCS (trimethylchlorosilane)** is one of the most commonly used silylating reagents. The reactivity of BSA is similar to that of BSTFA, readily silylating a wide range of functional groups such as non-sterically hindered alcohols, amides, amines, amino acids, carboxylic acids, and enols. TMCS is a silylation catalyst, rarely used alone in analytical applications but typically mixed with other silylation reagents to increase their reactivity (e.g., HMDS+TMCS+pyridine, BSTFA+TMCS, BSA+TMCS) in derivatization of alcohols, alkaloids, amines, biogenic amines, carboxylic acids, phenols, and steroids.

Features/Benefits

Under mild reaction conditions, BSA forms highly stable products with most organic functional groups. BSA will silylate unhindered hydroxyl groups. Reactions are generally fast and quantitative. TMS derivatives are thermally stable but more susceptible to hydrolysis than their parent compounds.

BSA and its byproducts are volatile, causing less chromatographic interference than many other silylating reagents.

BSA has good solvent properties and usually can function as an efficient silylating reagent without additional solvents. (DMF is the solvent most frequently used to improve efficiency.)

TMCS increases the reactivity of BSA (or other silylation reagents). Amides and many secondary amines and hindered hydroxyls, incompletely derivatized by BSA alone, can be derivatized by adding 1-20% TMCS to BSA.

BSA+TMCS has good solvent properties and can function as a silylation reagent without additional solvents. Alternatively, the mixture is very soluble in most commonly used silylation solvents.

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 silylating reagent – consult MSDS for specific handling information. BSA+TMCS is extremely sensitive to moisture and should be handled under dry conditions.

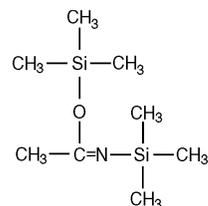
Prepare a reagent blank (all components, solvents, etc., *except sample*), following the same procedure as used for the sample.

1. Weigh 1-10mg of sample into a 5mL reaction vessel. If appropriate, dissolve sample in solvent (see below). If sample is in aqueous solution, evaporate to dryness, then use neat or add solvent.
2. Add excess silylating reagent (BSA+TMCS, 5:1). The reagent can be used at full strength or with a solvent.* In most applications it is advisable to use an excess of the silylating reagent – at least a 2:1 molar ratio of reagent to active hydrogen. In most cases BSA+TMCS, 5:1 is sufficient to achieve the desired derivatization.
3. Allow the mixture to stand until silylation is complete. 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.

Derivatization times vary widely, depending upon the specific compound(s) being derivatized. Many compounds are completely derivatized as soon as they dissolve in the reagent. Compounds with poor solubility may require warming. A few compounds will require heating at 70°C for 20-30 minutes. Under extreme conditions compounds may require heating for up to 16 hours to drive the reaction to completion. Amino

Properties

BSA Structure:



CAS Number: 10416-59-8

Molecular Formula: $\text{CH}_3\text{C}=\text{NSi}(\text{CH}_3)_3\text{OSi}(\text{CH}_3)_3$

Formula Weight: 203.43

Boiling Point: 71-73°/35mm

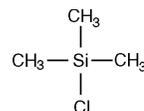
Flash Point: 53°F (11°C)

d: 0.823

n_D: 1.4170 at 20°C

Appearance: clear, colorless liquid
moisture sensitive

TMCS Structure:



CAS Number: 75-77-4

Molecular Formula: $\text{ClSi}(\text{CH}_3)_3$

Formula Weight: 108.66

Boiling Point: 57°C

Flash Point: -18°F (-27°C)

d: 0.856

n_D: 1.3870 at 20°C

Appearance: clear, colorless liquid with a pungent odor
moisture sensitive

796-0258, 0259

acids may require reaction in a sealed tube or vial. Heat samples cautiously, near the boiling point of the mixture, until a clear solution is obtained.

If derivatization is not complete, the addition of a catalyst, use of an appropriate solvent, higher temperature, longer time and/or higher reagent concentration should be evaluated.

Use a glass injection port liner or direct on-column injection when working with silylating reagents. Erratic and irreproducible results are more common when stainless steel injection ports are used.

TMS derivatives and silylating reagents react with and are sensitive to active hydrogen atoms. Do not analyze BSA+TMCS derivatives on stationary phases with these functional groups (e.g., polyethylene glycol phases). Silicones are the most useful phases for TMS derivatives – they combine inertness and stability with excellent separating characteristics for these derivatives. Nonpolar silicone phases include SPB™-1 and SPB-5. Normal hydrocarbons (carbon-hydrogen analytes with single bonds) are separated by these

*Nonpolar organic solvents such as hexane, ether, benzene, and toluene are excellent solvents for the reagent and the reaction products; they do not accelerate the rate of reaction. Polar solvents such as pyridine, dimethylformamide (DMF), dimethylsulfoxide (DMSO), tetrahydrofuran (THF), and acetonitrile are more often used because they can facilitate the reaction. Pyridine is an especially useful solvent because it can act as an HCl acceptor in silylation reactions involving organochlorosilanes.

