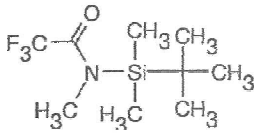
Produktinformation MBDS-TFA

N-tert.-Butyl-dimethylsilyl-N-methyl-trifluoracetamid

Art.-Nr.: **6.370550**

Lieferbare Einheiten: 20 x 1 ml, 1 x 10 ml
(andere Packungseinheiten auf Anfrage)

Physikalische Daten: M = 241,33 g/mol
Kp = 172-175°C
$d_{20/4}$ = 1,036 g/cm³



N-tert-Butyldimethylsilyl-N-methyltrifluoroacetamide, MTBSTFA

MTBSTFA is probably the most important tert-butyldimethylsilylating agent for analytical purposes today. First described in 1975 [1] and later used by M. Donike [2] for the silylation of steroids, its potential and its advantages over TBDMSCl have been published in 1982 [3, 4]. The main advantages of MTBSTFA over TBDMSCl are:

- the enhanced reactivity (reaction proceeds rapidly at room temperature, primary amines and thiols are silylated quantitatively)
- the easier work-up (no aqueous extraction necessary; for GC separations the reaction mixture can be injected directly)
- neutral reaction conditions (MTBSTFA and its by-product are neutral compounds).

Analytical applications

MTBSTFA is normally used in DMF or acetonitrile as solvent. The addition of TBDMSCl as catalyst (ca. 1%) increases its silylating potential [4–6]. Ketosteroids can be converted to their TBDMS enol ethers with tert-butyldimethyliodosilane as catalyst [2]. A mixture of MTBSTFA with TBDMSIM and catalytical amounts of TBDMSCl in acetonitrile (50:5:0.5:100) has been employed for the silylation of hydroxy fatty acid esters [26].

MTBSTFA readily silylates hydroxyl groups [1, 3, 4, 37], thiols [4, 33, 37], primary amines [1, 4, 37, 39] amides [1, 37], carboxyl groups [1, 4, 37, 40] and enolisable carbonyl groups [2, 8]. Secondary amines react more slowly [1, 4] and β-hydroxyl groups in secondary amines can be silylated selectively due to the additional steric hinderance [7] of the introduced TBDMS group. Many inorganic acids (e.g. sulfuric acid, sulfurous acid, phosphoric acid etc.) and their ammonium salts can be converted with MTBSTFA into their TBDMS derivatives, which are useful for GC-MS measurements [9]. Several different papers have been published on the silylation of amino acids with MTBSTFA di-silylation) and their determination with GC-MS [6, 8, 10–15, 32].

Furthermore, the following substances have been silylated with MTBSTFA: prostaglandins [3], propranolols [7], an oxime [16], α-keto acids [8], hydroxy acids [8, 17], monohydroxy fatty acid p-nitrobenzylesters (for HPLC) [26], carboxylic acids [8, 18, 25, 30, 39], N-(carbamoyl)-amino acids [35], phosphonic acids [27], alkylphosphonic acid and alkyl methyl phosphonic acids [31], aliphatic and aromatic sulfonic acids [38], adenine [5], porphyrins (via the dihydroxy silicon (IV) porphyrins) [19, 20, 21], guanine derivatives [34], leukotrienes [22], Z-oxylysine [23], dipeptides [28], urea [8], 5α-esterane-3β,17α-diol [36] and moniliformin [24] (3-hydroxycyclobut-3-ene1,2-dione) which forms a bis-silylated N-methyl-trifluoroacetamide adduct.

Synthetic applications

Preparative details of silylations with MTBSTFA have been published in the important paper of T. P. Mawhinney [4]. MTBSTFA has been reported for the silylation of a sulfoximine [29].

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Typical procedure

For silylation of substances, which contain hydroxyl, carboxyl, thiol, primary and secondary amine functional groups [40]:

1. Combine 1–10 mg of sample, 100 µl of MTBSTFA, and 100 µl of acetonitrile in a 1 ml Reacti-Vial™ Miniature Reaction Vial.
2. Cap vial and allow to stand at room temperature for 5–20 min.
3. Analyse by gas chromatography.

Note: Other aprotic solvents (such as DMF, pyridine, THF) may be used in place of CH_3CN. DMF is not recommended for primary or secondary amines.

Solid-phase extraction and TBDMS derivatisation of carboxylic acids [41]:
Evaporate 1.5 ml of ether solution containing 20 µl of triethylamine (TEA) to ca 50 µl in a stream of nitrogen, in a Reacti-Vial™. Add 20 µl of MTBSTFA and 60 µl of isooctane to the vial, cap and heat at 60°C, for derivatisation of TEA salts of carboxylic acids. Under these conditions the hindered hydroxyl and carboxylic groups of tartaric acid and γ-resorcylic acid, as their TEA salts, can be quantitatively silylated in 4 h and 8 h respectively. N-Silylation of p-aminobenzoic acid and hippuric acid does not occur, even after prolonged heating with excess of MTBSTFA.

Peptide hydrolysis and TBDMS derivatisation of amino acids [41]:
Weigh bovine insulin β-chain (100 µg) into a 1 ml derivatisation vial and add 500 µl of 6M hydrochloric acid. Heat the closed vial at 110°C for 24 h. After cooling to room temperature, evaporate the solvent under a stream of nitrogen and dry the residue over phosphorus pentoxide in vacuo. Derivatise the dry residue directly by the addition of MTBSTFA (50 µl), acetonitrile (50 µl) and ethanethiol (5 µl), then sonicate for 30 min at room temperature followed by heating at 150°C for 2.5 h. After cooling, aliquots of the reaction mixture can be used directly for capillary GC-MS analysis employing selected ion monitoring. Detection limits are at the picomole level. This derivatisation method is readily applied to the analysis of amino acids isolated from serum by elution through a column of Dowex 50W-X8 [10].

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