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Derivatization
reagents for GC



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A guide to derivatization reagents for GC

Regarding derivatization

Derivatization is one of the most common ways to prepare compounds for GC that are otherwise difficult to separate. Through derivatization, it is possible to improve the separation by replacing active hydrogens from the analyte with various groups that are easier to handle. Derivatization generally improves the following GC parameters:

- Chromatographic behavior
- Peak shape
- Thermal and chemical stability
- Detectability
- Volatility

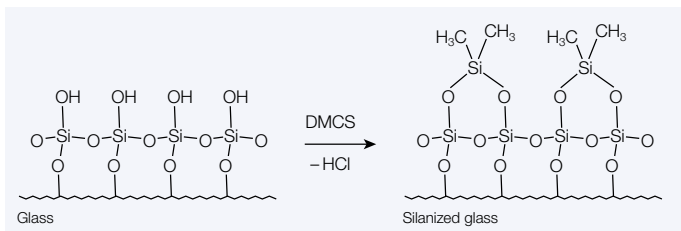
It is also important that all the instruments, e.g., laboratory glassware, will not interfere with the sample.

To make sure that no compounds containing -OH, -SH or -NH groups will be adsorbed by present Si-OH on the surface of the glass, a deactivation process may be necessary.

This is commonly achieved by rinsing the glass with a silylating agent, e.g., **DMCS** or **HMDS**, hence masking all silanols with non adsorptive methyl groups.

To achieve a satisfying rate of derivatization, it is essential to keep the following requirements in mind:

- The derivatization reaction needs to be complete $\approx 100\%$
- No loss of sample during derivatization
- The overall structure of the analyte will not be altered
- Produced derivative will be stable over time
- No interaction between the reagent and the chromatographic system



A guide to derivatization reagents for GC

Naturally, all other components of the sample preparation and handling process need to be contaminant-free and in top condition. Since water is, in most cases, a problem, it has to be removed from the derivatization process, e.g., by adding Na_2SO_4 to the reaction mixture. Like all reactions, derivatization takes time and a certain amount of heat to go to completion. As duration may vary greatly, dependent on the reactivity of the analyte, it is often necessary to screen several reagents for the best result. It is also important to realize that there is no such thing as the best derivatization method. There will always be several working solutions to a chromatographic problem with its own advantages and drawbacks, dependent on the equipment or on the approach of the chemist.

Derivatization reagents

There are many reagents in use today for derivatization. There are three categories they can be allocated to:

- Silylation
- Acylation
- Alkylation (Methylation)

Good to know

- Our derivatization reagents meet the highest demands of purity.



Silylation

Silylation is the most versatile method of derivatization in GC, i.e. more than 80 % of all derivatization reactions are actually silylations. Usually the term silylation in GC stands for replacement of active hydrogen atoms by a trimethylsilyl group (TMS derivative). Sometimes, however, trialkylsilyl groups or dimethylalkylsilyl groups with longer alkyl chains are used for derivatization. The trialkylsilyl group increases volatility and enhances thermal stability of the sample.

As with methylation, the replacement of an active hydrogen with a silyl group reduces the polarity of the compound, as well as hydrogen bonding.

Additionally, silylation improves volatility, so that many compounds that are normally considered nonvolatile or thermally unstable, can be chromatographed easily. Introducing a silyl group may also enhance the GC-MS properties of the derivative, either through characteristic ions or more favorable diagnostic patterns for structure investigations.

Good to know



- It is important to mention that silylated compounds should not be used with WAX or FFAP phases, as the OH groups of the stationary phase will definitely become derivatized by the silylating reagent, and this will irreversibly change selectivity of the column.

Silylation can be catalyzed either acidically by the addition of **TMCS** or basically by the addition of pyridine or **TSIM** (e.g., for sterically hindered molecules, such as tertiary alcohols).



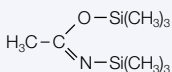
Amides

N,O-bis-trimethylsilyl-acetamide (BSA)

M: 203.4 g/mol

Bp: 71–73 °C (35 mm Hg)

density $d_{20}^{20} / 4^{\circ} = 0.83$



BSA is a strong silylation reagent that forms very stable TMS derivatives with a large variety of compounds, e.g., non-sterically hindered alcohols, carboxylic acids, phenols, enols, steroids, (biogenic) amines and alkaloids.

Not recommended for use with carbohydrates or very low molecular weight compounds.

Good solvent for polar compounds, but frequently used in combination with a solvent (pyridine, DMF etc.), with other silylation reagents or catalysts such as TFA, HCl or TMBS.

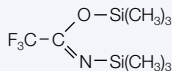
Used in combination with DMF, **BSA** is the reagent of choice for derivatizing phenols.

N,O-bis-trimethylsilyl-trifluoroacetamide (BSTFA)

M: 257.4 g/mol

Bp: 40 °C (12 mm Hg)

density $d_{20}^{20} / 4^{\circ} = 0.96$



BSTFA is a powerful trimethylsilyl donor with approximately the same donor strength as the nonfluorinated analog **BSA**.

Advantage of BSTFA over BSA

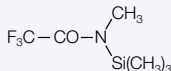
Greater volatility of its derivatives (particularly useful for GC of some lower boiling TMS amino acids). **BSTFA** will generally react with all organic material present, but may not react with some amides, secondary amines and hindered hydroxyl groups. However, adding 1% **TMCS (SILYL-991)** will solve that problem.



Silylation

N-methyl-*N*-trimethylsilyl-trifluoroacetamide (MSTFA)

M: 199.1 g/mol
Bp: 70 °C (75 mm Hg)
density $d_{20}^{20} = 1.11$



MSTFA is the most volatile trimethylsilyl amide available.

MSTFA is a very strong TMS donor that does not cause any noticeable FID contamination even after long-time measuring series. It is one of the most important silylating reagents. It can be used, to silylate the hydrochloride salts of amines or amino acids directly.

The already good solution characteristics can be improved by adding submolar quantities of protic solvents (e.g., TFA for extremely polar compounds such as hydrochlorides) or pyridine (e.g., for carbohydrates).

Advantages

- Complete reaction with high reaction rates, even without a catalyst (1–2 % TMCS or TSM)
- By-product of the reaction (*N*-methyltrifluoroacetamide) features high volatility and short retention time.

Reactivity of silylation reagents (acc. to M. Donike)

TMS amides (e.g., BSA, MSTFA) >
TMS amine = TSIM > Enol-O-TMS ether >
S-TMS ether > O-TMS ether > TMS-O-TMS

Stability of the TMS derivatives

O-TMS ether > S-TMS ether > Enol-O-TMS
ether > TMS amine > TMS amide

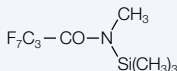


Silylation

N-methyl-*N*-trimethylsilyl-heptafluorobutyramide (MSHFBA)

M: 299.1 g/mol

Bp: 148 °C (760 mm Hg)



MSHFBA is similar to MSTFA in reactivity and chromatography.

Used either alone or in combination with a catalyst (TMCS, TSIM) or another silylation reagent with or without solvent.

By-product *N*-methylheptafluorobutyric amide has a lower retention time than the silylating reagent.

Especially useful for FID, because, due to the large 7:1 ratio of fluorine to silicon, the degradation of excess MSHFBA does not produce SiO₂ but volatile, non-corrosive silicon compounds.

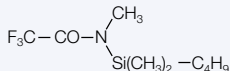


N-methyl-*N*-tert-butyl(dimethylsilyl)-trifluoroacetamide (MBDSTFA)

M: 241.3 g/mol

Bp: 168–170 °C (760 mm Hg)

density d₂₀ °/4 ° = 1.12



MBDSTFA is a silylation reagent that donates a tert-butyl(dimethylsilyl) group (TBDMS) for derivatizing active hydrogens in hydroxyl, carboxyl and thiol groups, primary and secondary amines, as well as in amino acids.

Fast reactions (typically 5–20 min) with high yields (> 96 %). By-products are neutral and volatile.

TBDMS ethers are 10⁴ times more stable than the corresponding TMS ethers. Chromatographic retention times are longer due to the large protecting group, which may improve some separations.

Very useful for GC-MS applications, because of a high molecular ion concentration at M⁺-57 applications.



Silanes / silazanes

Dimethyldichlorosilane (DMCS)

M: 129.06 g/mol

Bp: 70 °C (760 mm Hg)

density $d_{20}^{20} / 4^{\circ} = 1.07$



DMCS is used to form dimethylsilyl (DMS) derivatives.

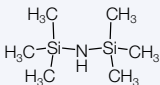
DMS derivatives are much more susceptible to hydrolysis than TMS derivatives. Therefore, strictly anhydrous conditions during the reaction are very important.

Hexamethyldisilazane (HMDS)

M: 161.4 g/mol

Bp: 126 °C (760 mm Hg)

density $d_{20}^{20} / 4^{\circ} = 0.77$



HMDS is a weak TMS donor. If used as sole reagent, it is slow and not very effective. After addition of catalytic quantities (e.g., 1 %) of **TMCS** or as a mixture with **TMCS**, it is a fast and quantitative reagent for trimethylsilylation of organic compounds.

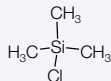
Aprotic solvents, e.g., acetonitrile, pyridine, dimethylformamide, carbon disulfide and dimethylacetamide are recommended for use with **HMDS**.

Trimethylchlorosilane (TMCS)

M: 108.7 g/mol

Bp: 57 °C (760 mm Hg)

density $d_{20}^{20} / 4^{\circ} = 0.86$



TMCS is often used as a catalyst with other trimethylsilyl reagents. Without additives it can be used for preparing TMS derivatives of organic acids.

Together with methanol, **TMCS** can be used for Methylation.

Silylation reagent mixtures

SILYL-271 BSA – HMDS – TSIM (2:7:1)

SILYL-271 will derivatize all hydroxyl groups in any position. Useful in multidervivatization schemes involving hydroxyl or amine groups.

SILYL-1139 TSIM – pyridine (11:39)

Recommended application: alcohols, phenols, organic acids, steroids, hormones, glycols, nucleotides and narcotics.

SILYL-21 HMDS – TMCS (2:1)

SILYL-21 will derivatize amides and many secondary amines and hindered hydroxyls that would not be completely derivatized by HMDS alone. It can be used without solvent.

SILYL-2110 HMDS – TMCS – pyridine (2:1:10)

SILYL-319 HMDS – TMCS – pyridine (3:1:9)

SILYL-2110 and SILYL-319 will derivatize alcohols, bile acids, phenols, most steroids, sterols, and sugars that would not be completely derivatized by HMDS alone. SILYL-2110 and SILYL-319 are fast and easy to use, and can be used without solvent.

SILYL-991 BSTFA – TMCS (99:1)

BSTFA is a powerful trimethylsilyl donor. For silylating of fatty acid amides, hindered hydroxyls and other compounds that are difficult to silylate, e.g., secondary alcohols and amines, we recommend BSTFA + 1 % TMCS, available under the designation SILYL-991.

Good to know



- Most derivatives are susceptible to water and hydrolysis
- Reactions only in aprotic solvents possible
- The presence of water does not interfere with SILYL-1139.

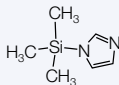
Imidazoles

N-Trimethylsilyl-imidazole (TSIM)

M: 140.3 g/mol

Bp: 94–96 °C (760 mm Hg)

density d₂₀ °/4 ° = 0.96



TSIM is the strongest hydroxyl silylator, the reagent of choice for carbohydrates and most steroids (even highly hindered steroids).

The reagent is unique in that it reacts quickly and smooth with hydroxyl (even tert. OH) and carboxyl groups, but not with amines. This characteristic makes TSIM particularly useful in multi-derivatization schemes for compounds with different functional groups that are to be derivatized differently, e.g., -O-TMS/-N-HFB derivatives of catecholamines.

Summary silylation

- Silylation can be applied on many compounds
- Silylating reagents are easily prepared
- Large variety of reagents available

Acylation / Benzoylation

Generally, acylation involves the introduction of an acyl group into a molecule with a replaceable hydrogen, or across a double bond. Acylation is used to convert compounds like alcohols, amines and thiols into their respective esters, amides and thioesters. Additionally, they enhance the detectability of the compounds by adding halogenated carbon to the compounds. This is achieved through the reaction with fluorinated acyl halides, anhydrides or bisacylamides. While the corresponding acidic by-products of the reactions with acyl halides and anhydrides need to be removed from the system by a suited base, e.g., pyridine, to prevent column damage. By-products of bisacylamides are not acidic and normally do not interfere with the subsequent analysis. Hence, they are favorable reagents for acylations.

Acyl halides

Pentafluorobenzoyl chloride (PFBC)

R= C₆F₅, X=Cl

M: 230.52 g/mol

Bp: 158–159 °C (760 mm Hg)

density d₂₀^{°/4°} = 1.60



PFBC will react with hydroxyls, primary and secondary amines, amides and thiols.

Anhydrides

Trifluoroacetic acid anhydride (TFAA)

R=CF₃

M: 210.04 g/mol

Bp: 39.5–40.5 °C (760 mm Hg)

density d₂₀^{°/4°} = 1.49

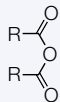
Heptafluorobutyric acid anhydride (HFBA)

R=C₃F₇

M: 410.06 g/mol

Bp: 106–107 °C (760 mm Hg)

density d₂₀^{°/4°} = 1.665



Acylation with fluorinated acid anhydrides can be used for alcohols, phenols, carboxylic acids, amines, amino acids and steroids forming volatile, stable derivatives suited for FID as well as for ECD detection.

Bisacylamides

N-methyl-bis(trifluoroacetamide) (MBTFA)



M: 223.08 g/mol

Bp: 123–124 °C (760 mm Hg)

density $d_{20}^{20} / 4^\circ = 1.55$

N-methyl-bis(heptafluorobutyramide) (MBHFBA)



M: 423.1 g/mol

Bp: 165–166 °C (760 mm Hg)

density $d_{20}^{20} / 4^\circ = 1.67$



Acylation with fluorinated acid amides is recommended for alcohols, primary and secondary amines as well as for thiols under mild, neutral conditions. **MBTFA** also forms very volatile derivatives with carbohydrates.

Good to know



- Acylation reagents are moisture sensitive
- Reaction products (acidic by-products) often have to be removed before analysis

Summary acylation

- Addition of halogenated carbons enhances detectability by ECD
- Derivatives are hydrolytically stable
- Increased sensitivity by adding molecular weight

Alkylation

Alkylation (methylation)/ esterification

Alkylation is a derivatization method used to replace an acidic hydrogen with an alkyl or methyl group. It is generally restricted to amines or hydroxy groups like in amino or carboxylic acids. The resulting derivatives are ethers, esters, methylamines or -amides and less polar than the original compounds. Therefore, less hydrogen bonding occurs. The acidity of the hydrogen to be replaced significantly determines the conditions needed to perform the alkylation. The less acidic, the more vigorous the conditions.

Methylation reagents

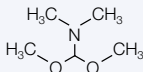
Dialkylacetals

N,N-dimethylformamide dimethylacetal
(DMF-DMA)

M: 119.17 g/mol

Bp: 106–107 °C (760 mm Hg)

density $d_{20}^{20} / 4^{\circ} = 0.89$

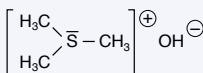


DMF-DMA is recommended for sterically hindered carboxylic acids, aldehydes, phenols and amines.

Trimethylsulfonium compounds

Trimethylsulfonium hydroxide (TMSH, 0.2 M
in methanol)

M: 94.06 g/mol



Methylation with TMSH is recommended for free acids, chlorophenoxy-carboxylic acids, their salts and derivatives as well as for phenols and chlorophenols. Lipids or triglycerides can be converted to the corresponding fatty acid methyl esters (FAMES) by a simple transesterification.

This reaction is very elegant and convenient, because it is just necessary to add the reagent (0.2 M in methanol) to the sample solution. Removal of excess reagent is not required, since in the injector of the gas chromatograph, at 250 °C, pyrolysis to volatile methanol and dimethylsulfide will occur. Due to the high reactivity, complete derivatization is often obtained at ambient temperature. However, heating (e.g., 10 min at 100 °C) in a closed sample vial may be necessary to complete the reaction.



Alkylation

Esterification reagents

Methylation with methanol / TMCS

An 1M solution of **TMCS** in methanol is suited for the esterification of free carboxylic acids and transesterification of glycerides. Formation of HCl catalyzes the reaction. **TMCS** and silyl ether remove water and thus drive the reaction to completion. The mixture should be freshly prepared.

Summary alkylation (methylation)

- Methylation derivatives are generally stable
- Wide range of reaction conditions (from strongly acidic to strongly basic)
- Some reactions can be achieved with water present

Good to know



- Reactions are limited to acidic hydrogens or amines
- Reaction conditions may be extreme



Derivatization procedures

Silylation

with BSA, BSTFA or SILYL-991 (BSTFA + 1 % TMCS)
BSA MN Appl. No. 213091 · BSTFA MN Appl. No. 213092 · SILYL-991
MN Appl. No. 213093

Add 0.5 mL of the silylation reagent to 1–10 mg sample; if necessary, add some solvent (normally pyridine or DMF [dimethylformamide]). Heat to 60–80 °C for 20 min to increase the reaction rate. 1–2 drops of **TMCS** (trimethylchlorosilane) or **TSIM** will also speed up the reaction.

with BSA in combination with other silylation reagents · MN Appl. No. 213100

BSA alone silylates all sterically unhindered hydroxyl groups of the steroid skeleton; addition of **TMCS** will enable reaction of moderately hindered OH groups (reaction time 3–6 h at 60 °C). After addition of **TSIM** even strongly hindered hydroxyl groups will react (reaction time 6–24 h at 60 °C).

with MSTFA, MSHFBA or MBDSTFA
MSTFA MN Appl. No. 213111 · MSHFBA MN Appl. No. 213112 · MBDSTFA
MN Appl. No. 213113

Dissolve 10–15 mg sample in 0.8 mL solvent, then add 0.2 mL of the silylation reagent. The reaction mixture can be heated to 60–70 °C for up to 1 h and can be analyzed directly. If TFA is used as a solvent, proceed as follows [20]: dissolve 1–2 mg sample in 100 µL TFA. Dropwise add 0.9 mL of the silylating reagent. After cooling the sample can be chromatographed directly.

with TSIM or SILYL-1139 (TSIM – pyridine 11:39)
TSIM MN Appl. No. 213121 SILYL-1139 MN Appl. No. 213122

Dissolve 10–15 mg sample in 0.8 mL solvent, then add 0.2 mL of the silylation reagent. The reaction mixture can be heated to 60–70 °C for up to 1 hour and can be analyzed directly. Recommended solvent is pyridine. When using SILYL-1139, the presence of water does not interfere.

Derivatization procedures

Silylation

with SILYL-21 or SILYL-2110 or SILYL-319 · SILYL-21 MN Appl. No. 213131 · SILYL-2110 · MN Appl. No. 213132

Carefully add **SILYL-21**, **SILYL-2110** or **SILYL-319** to 1–10 mg of the sample. Precipitated ammonium chloride does not interfere. If the sample should not dissolve within 5 min, heat to 75–85 °C. If no mutarotation is to be expected, you may dissolve the sugar in warm pyridine first and then add the silylation reagent. In some cases it may be advantageous to use a different solvent instead of pyridine. For derivatization of 3-ketosteroids we recommend to use DMF (dimethylformamide).

O-trimethylsilylation with MSTFA followed by N-trifluoroacetylation with MBTFA
MN Appl. No. 213140

Completely silylate 2 mg of the sample with 0.3 mL **MSTFA**. After addition of 0.3 mL **MBTFA** the *N*-trimethylsilyl group is replaced by the *N*-trifluoroacetyl group. The mixture can be analyzed directly.



Derivatization procedures

Acylation

with fluorinated acid anhydrides

TFAA MN Appl. No. 213041 · HFBA MN Appl. No. 213042

Dissolve 0.1 to 1 mg sample in 0.1 mL solvent, add 0.1 mL of the anhydride and heat to 60–70 °C for 1–2 h. If the sample needs not be concentrated prior to the analysis and if there is no danger of catalytically induced side reactions, pyridine is used as solvent. The reaction solution can be injected directly into the gas chromatograph. Otherwise, use a volatile solvent and evaporate solvent, excess reagent and free acid in a stream of nitrogen. Dissolve residue in 50 µL hexane, chloroform etc. and inject aliquot portions.

with fluorinated acid amides

MBTFA MN Appl. No. 213051 · MBHFBA MN Appl. No. 213052

Add 0.5 mL MBTFA or MBHFBA to about 2 mg sample. If there is no reaction at ambient temperature, heat the reaction mixture to 120 °C. Compounds difficult to dissolve, can be trifluoroacetylated in suitable solvent mixtures. It is recommended to use a ratio of solvent to MBTFA or MBHFBA of 4:1. The reaction mixture is chromatographed directly.



Derivatization procedures

Alkylation (Methylation)

with TMSH · MN Appl. No. 213060

Dissolve 100 mg sample (e.g., butter) in 5 mL of a solvent (e.g., tert.-butyl methyl ether). Add 50 μL reagent to 100 μL of this solution. The mixture is injected directly. The temperature of the injector must be at least 250 °C.

with DMF-DMA · MN Appl. No. 213070

Add 1 mL of a mixture of DMF-DMA and pyridine (1:1) to 1–50 mg fatty acids. The sample can be injected as soon as a clear solution has formed. It is recommended, however, to heat the solution to 60–100 °C for 10–15 min.

with methanol – TMCS · MN Appl. No. 213080

Add 1 mL methanol – TMCS to about 50 mg carboxylic acid or glyceride and heat. Then evaporate in a stream of nitrogen and dissolve again for injection in, e.g., *n*-heptane.



Overview of important functional groups

Functional Group	Silylation*	Acylation / Benzoylation	Alkylation
Primary alcohols	BSA, MSTFA, MSHFBA, TSIM, SILYL-2110, SILYL-319, SILYL-21, SILYL-1139	TFAA, HFBA, MBTFA, MBHFBA, PFBC	TMSH
Secondary alcohols	BSA, MSTFA, MSHFBA, TSIM, SILYL-2110, SILYL-319, SILYL-21, SILYL-1139	TFAA, HFBA, MBTFA, MBHFBA, PFBC	TMSH
Tertiary (and sterically hindered) alcohols	TSIM, BSTFA, SILYL-991	TFAA, HFBA, PFBC	
Thiols	BSA, MSTFA, MSHFBA, TSIM, SILYL-2110, SILYL-319, SILYL-21, SILYL-1139	MBTFA, MBHFBA, HFBA, TFAA	TMSH
Phenols	BSA, MSTFA, MSHFBA, TSIM, SILYL-2110, SILYL-319, SILYL-21, SILYL-1139	TFAA, HFBA, MBTFA, MBHFBA, PFBC	DMF-DMA, TMSH
Glycols	BSA, MSTFA, MSHFBA, TSIM, SILYL-2110, SILYL-319, SILYL-21, SILYL-1139	TFAA, HFBA, MBTFA, MBHFBA, PFBC	TMSH
Aldehydes	BSA, MSTFA, MSHFBA, TSIM, SILYL-2110, SILYL-319, SILYL-21, SILYL-1139	TFAA, HFBA, MBTFA, MBHFBA, PFBC	DMF-DMA, TMSH, MeOH/TMCS
Ketones	BSA, MSTFA, MSHFBA, TSIM, SILYL-2110, SILYL-319, SILYL-21, SILYL-1139	TFAA, HFBA, MBTFA, MBHFBA, PFBC	DMF-DMA, TMSH, MeOH/TMCS
Carboxylic acids	BSA, MSTFA, MSHFBA, TSIM, SILYL-2110, SILYL-319, SILYL-21, SILYL-1139	TFAA, HFBA, MBTFA, MBHFBA, PFBC	DMF-DMA, TMSH, MeOH/TMCS
Carbohydrates /Sugars	MSTFA, TSIM, SILYL-2110, SILYL-319, SILYL-991, HMDS	TFAA, MBTFA, PFBC	
Acid anhydrides			MeOH/TMCS
α -hydroxy acids	MSTFA	MBTFA	

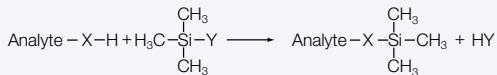
Overview of important functional groups

Functional Group	Silylation*	Acylation / Benzoylation	Alkylation
Primary amines	BSA, MSTFA, MSHFBA, SILYL-991	TFAA, HFBA, MBTFA, MBHFBA, PFBC	DMF-DMA
Secondary amines	BSA, MSTFA, MSHFBA, SILYL-991	TFAA, HFBA, MBTFA, MBHFBA, PFBC	DMF-DMA
Amides	Silylamides are not stable	TFAA, HFBA, MBTFA, MBHFBA, PFBC	
Amino acids	BSA, BSTFA, MSTFA, MSHFBA	TFAA, HFBA, MBTFA, MBHFBA, PFBC	MeOH/TMCS, TMSH
Amino sugars	BSA, MSTFA, MSHFBA, SILYL-991	TFAA, HFBA, MBTFA, MBHFBA, PFBC	
Imino acids	BSA, MSTFA, MSHFBA, SILYL-991	TFAA, HFBA, MBTFA, MBHFBA, PFBC	
Carbamides	Silylamides are not stable	TFAA, HFBA, MBTFA, MBHFBA	
Alkylamides	Silylamides are not stable	PFBC	
Amino alcohols	MSTFA	MBTFA	

* (Avoid polar stationary phases containing active protons, e.g., WAX or FFAP)

General reaction mechanisms

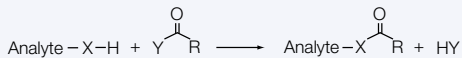
Silylation



X = e.g., O, S, COO, etc.

Y = rest of silylation reagents; structures see page 7-11

Acylation

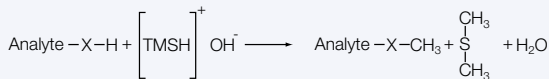


X = e.g., O, S, NH, etc.

Y = rest of acylation reagents; structures see page 12, 13

General reaction mechanisms

Alkylation (Methylation) • example TMSH



X = e.g., O, S, COO, etc.

Alkylation reagents; structures see page 14

References

1. MN Chromatography catalog
2. Blau, K., Halket, J., Handbook of Derivatives for Chromatography, Second Edition; John Wiley & Sons; Chichester, 1993
3. Knapp, D.R. Handbook of Analytical Derivatizations Reactions; John Wiley & Sons; New York, 1979
4. Butte, W., J. Chromatogr. A, 261, 1983

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Email: info@greyhoundchrom.com
Web: <https://www.greyhoundchrom.com>

Ordering information

Substance	Packing unit			
	10 x 1 mL	20 x 1 mL	1 x 10 mL	5 x 10 mL
Silylation reagents*				
BSA			701210.110	701210.510
BSTFA		701220.201	701220.110	701220.510
DMCS**				
HMDS				701240.510
TMCS**		701280.201		
TSIM		701310.201	701310.110	701310.510
MSHFBA		701260.201	701260.110	701260.510
MSTFA		701270.201	701270.110	701270.510
MBDSTFA	701440.101	701440.201		
SILYL-271 (BSA – HMDS – TSIM 2:7:1)		701450.201	701450.110	701450.510
SILYL-1139 (TSIM – pyridine 11:39)		701460.201		
SILYL-21 (HMDS – TMCS 2:1)		701470.201		
SILYL-2110 (HMDS – TMCS – pyridine 2:1:10)		701480.201		
SILYL-319 (HMDS – TMCS – pyridine 3:1:9)		701241.201		
SILYL-991 (BSTFA – TMCS 99:1)		701490.201		
Acylation reagents*				
HFBA		701110.201	701110.110	701110.510
MBTFA		701410.201	701410.110	701410.510
MBHFBA	701420.101	701420.201		
PFBC	701120.101			
TFAA			701130.110	701130.510
Alkylation reagents*				
DMF-DMA		701430.201	701430.110	
TMSH	701520.101	701520.201	701520.110	701520.510

Due to their purpose, derivatization reagents are very reactive chemicals. For this reason, they should be stored cool and protected from moisture. For easy access with a syringe, our derivatization reagents are supplied in vials with crimp caps. Vials with pierced sealing disks have limited stability and should be used up soon.

Ordering information

1 x 50 mL

6 x 50 mL

1 x 100 mL

6 x 100 mL

12 x 100 mL

701210.150

701230.650

701240.650

701280.650

701260.1100

701260.6100

701270.650

701270.1100

701270.6100

701270.12100

701490.150

701490.1100

* These products contain harmful chemicals which must be specially labeled as hazardous.
For detailed information please see SDS.

** Vials with screw caps. Further screw caps on request.

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