Products for derivatisation

Derivatisation reagents

The purpose of derivatisation

In gas chromatography it is often advantageous to derivatise polar functional groups (mainly active hydrogen atoms) with suitable reagents. Prerequisite for successful derivatisation is quantitative, rapid and reproducible formation of only one derivative. Aim of this reaction is an improved volatility, better thermal stability or a lower limit of detection due to improved peak symmetry. The halogen atoms introduced by derivatisation (e.g. trifluoroacetates) allow specific detection (ECD) with the advantage of high sensitivity. Elution orders and fragmentation patterns in mass spectroscopy can be influenced by a specific derivatisation.

The table on page 351 and the derivatisation examples are meant as a first orientation and have to be adjusted or optimised for special problems.

If you are in doubt, which type of derivatisation reaction best suits your needs, or which reagent is best suited for your sample, you can try different methods or different reagents by using the derivatisation method development kits described below.

We supply the following derivatisation reagents for acylation, alkylation (methylation) and silylation:

Reagents for acylation (see page 352)

HFBA	heptafluorobutyric acid anhydride
MBTFA	N-methyl-bis(trifluoroacetamide)
MBHFBA	N-methyl-bis(heptafluorobutyramide)
PFBC	pentafluorobenzoyl chloride
TFAA	trifluoroacetic acid

Reagents for methylation (see page 353)

DMF-DMA	N,N-dimethylformamide-dimethylacetal
TMSH	trimethylsulphonium hydroxide (in methanol)

Reagents for silylation (see page 355)

BSA	N,O-bis-trimethylsilyl-acetamide
BSTFA	N,O-bis-trimethylsilyl-trifluoroacetamide
DMCS	dimethylchlorosilane
HMDS	hexamethyldisilazane
MSHFBA	N-methyl-N-trimethylsilyl-heptafluorobutyramide
MSTFA	N-methyl-N-trimethylsilyl-trifluoroacetamide
MBDSTFA	N-methyl-N-*tert*-butyldimethylsilyl-trifluoroacetamide
TMCS	trimethylchlorosilane
TMSDEA	N-trimethylsilyl-diethylamine
TSIM	N-trimethylsilyl-imidazole

Additionally, we supply some mixtures of silylation reagents with or without pyridine.

Derivatisation method development kits

Designation	Contents of the kit	Cat. No.
Which type of derivatisation reaction is best suited for your sample (alkylation, acylation or silylation)?		
Derivatisation method development kit	2 x 1 ml each of TMSH, MSTFA, and MBTFA	**701952**
Which is the proper reagent for acylation?		
Acylation kit	2 x 1 ml each of MBTFA, TFAA, and MBHFBA	**701950**
Which is the proper reagent for methylation?		
Alkylation kit	3 x 1 ml each of TMSH, and DMF-DMA	**701951**
Which is the proper reagent for silylation?		
Silylation kit	2 x 1 ml each of MSTFA, BSTFA, TSIM, and MSHFBA	**701953**

Derivatisation reagents

Selection guide for derivatisation of important functional groups in GC

Function	method	derivative	recommended reagents
Alcohols, Phenols R'OH	silylation	R'O – TMS	BSA, MSTFA, MSHFBA, TSIM, SILYL-2110, SILYL-21, SILYL-1139
	acylation	R'O – C(=O) – R	TFAA, HFBA, MBTFA, MBHFBA
	alkylation	R'O – R	TMSH
sterically hindered	silylation	R'O – TMS	TSIM, BSTFA, SILYL-991
Amines primary, secondary R' – N(R") – H	silylation	R' – N(R") – TMS	BSA, MSTFA, MSHFBA, SILYL-991
	acylation	R' – N(R") – C(=O) – R	TFAA, HFBA, MBTFA, MBHFBA
hydrochlorides	silylation	R' – N(R") – TMS	MSTFA
Amides R' – C(=O) – NH_2	silylation	not stable	
	acylation	R' – C(=O) – N(H) – C(=O) – R	TFAA, MBTFA, HFBA, MBHFBA
Amino acids R' – CH(COOH) – NH_2	silylation	R' – CH(C(=O) – O – TMS) – HN – TMS	BSA, BSTFA, MSTFA, MSHFBA
	alkylation (a) + acylation (b)	R' – CH(C(=O) – O – R) – HN – C(=O) – R	a) MeOH/TMCS, TMSH b) TFAA, HFBA, MBTFA, MBHFBA
Carboxylic acids (fatty acids) R' – COOH	silylation	R' – C(=O) – O – TMS susceptible to hydrolysis	BSA, MSTFA, MSHFBA, TMCS, TSIM, SILYL-2110, SILYL-21
	alkylation	R' – C(=O) – O – R	DMF-DMA, MeOH/TMCS (1 M), TMSH
salts	silylation	R' – C(=O) – O – TMS susceptible to hydrolysis	TMCS
Carbohydrates	silylation acylation		MSTFA, TSIM, HMDS, SILYL-1139 TFAA, MBTFA
Steroids	silylation acylation		BSA, TSIM TFAA, MBTFA, HFBA, MBHFBA

Acylation

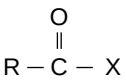
In principle three classes of compounds can be used for acylation: acyl halides, anhydrides and bisacylamides.

Acyl halides are very reactive, forming hydrohalic acids as by-products. Excess of reagent and acid have to be removed or trapped by a suitable base (e.g. pyridine).

Anhydrides are also very reactive, forming as by-products the corresponding acids. Excess reagent and the acid formed have to be removed.

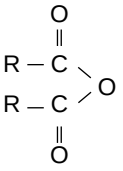
Bisacylamides (MBTFA, MBHFBA) are very reactive and form as by-products the corresponding **neutral** acylamides. Reagent and by-product can be easily removed due to their high volatility. Because of the neutral conditions and the favourable chromatographic properties often removal of the bisacylamide is not necessary. Thus sample preparation is much more convenient.

Acyl halides



PFBC: R = C_6F_5, X = Cl

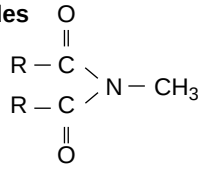
Anhydrides



TFAA: R = CF_3

HFBA: R = C_3F_7

Bisacylamides



MBTFA: R = CF_3

MBHFBA: R = C_3F_7

Reagents for acylation

Ordering information	**Cat. No.** for packing unit of			
code	10 x 1 ml	20 x 1 ml	1 x 10 ml	5 x 10 ml
Heptafluorobutyric acid anhydride m.w. 410.06, Bp 106 – 107 °C (760 mm Hg), density d20°/4° = 1.665				
HFBA		**701110.201**	**701110.110**	**701110.510**
N-methyl-bis(trifluoroacetamide) m.w. 223.08, Bp 123 – 124 °C (760 mm Hg), density d20°/4° = 1.55				
MBTFA		**701410.201**	**701410.110**	**701410.510**
N-methyl-bis(heptafluorobutyramide) m.w. 423.1, Bp 165 – 166 °C (760 mm Hg), density d20°/4° = 1.673				
MBHFBA	**701420.101**	**701420.201**		
Pentafluorobenzoyl chloride m.w. 230.52, Bp 158 – 159 °C (760 mm Hg), density d20°/4° = 1.601				
PFBC	**701120.101**			
Trifluoroacetic acid anhydride m.w. 210.04, Bp 39.5 – 40.5 °C (760 mm Hg), density d20°/4° = 1.490				
TFAA			**701130.110**	**701130.510**

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

Methods for acylation

1) Acylation with fluorinated acid anhydrides:
TFAA = trifluoroacetic acid anhydride
HFBA = heptafluorobutyric acid anhydride

Acylation with TFAA or HFBA 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.

Procedure:
Dissolve 0.1 to 1 mg of the sample in 0.1 ml solvent, add 0.1 ml of the respective anhydride and heat to 60 – 70 °C for 1 – 2 hours. If the sample need 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 acid in a stream of nitrogen. Dissolve the residue in 50 µl hexane, chloroform etc. and inject aliquot portions.

2) Acylation with fluorinated acid amides:
MBTFA = N-methyl-bis(trifluoroacetamide)
MBHFBA = N-methyl-bis(heptafluorobutyramide)

This method 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 [J. Sullivan and L. Schewe, J. Chromatogr. Sci. **15** (1977) 196 – 197].

Procedure:
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 which are 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 can be chromatographed directly.

Alkylation (methylation)

Except for some special cases (e.g. enantiomer separation of amino acids with PERMABOND® L-CHIRASIL-VAL) in GC generally methylation is the only type of alkylation used.

Reagents for methylation

Ordering information	**Cat. No.** for packing unit of			
code	10 x 1 ml	20 x 1 ml	1 x 10 ml	5 x 10 ml
N,N-dimethylformamidedimethylacetal m.w. 119.17, Bp 106 – 107 °C (760 mm Hg), density d20°/4° = 0.897				
DMF-DMA		**701430.201**	**701430.110**	
Trimethylsulphonium hydroxide (0.2 M in methanol) m.w. 94.06				
TMSH	**701520.101**	**701520.201**	**701520.110**	**701520.510**

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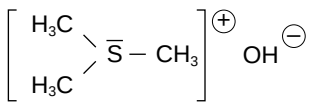
Methods for methylation

1) Methylation with TMSH
(0.2 M trimethylsulphonium hydroxide in methanol)

TMSH



Methylation with TMSH [1] is recommended for free acids [e.g. fatty acids [2], chlorophenoxycarboxylic acids [3]], their salts and derivatives as well as for phenols and chlorophenols, which can be detected in very small amounts. One great advantage is simplification of the sample preparation. Lipids or triglycerides can be converted to the corresponding fatty acid methyl esters (FAMEs) by a simple transesterification [2]. Isomerisations of multiple unsaturated fatty acids have not been observed.

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 dimethylsulphide will occur. Due to the high reactivity, complete derivatisation is often obtained at ambient temperature. However, heating (e.g. 10 min at 100 °C) in a closed sample vial may be necessary.

1) W. Butte, J. Chromatogr. **261** (1983) 142

2) E. Schulte, K. Weber, Fat Sci. Technol. **91** (1989) 181; K. D. Müller et al., Chromatographia **30** (1990) 245

3) H. Färber et al., Vom Wasser **76** (1991) 13

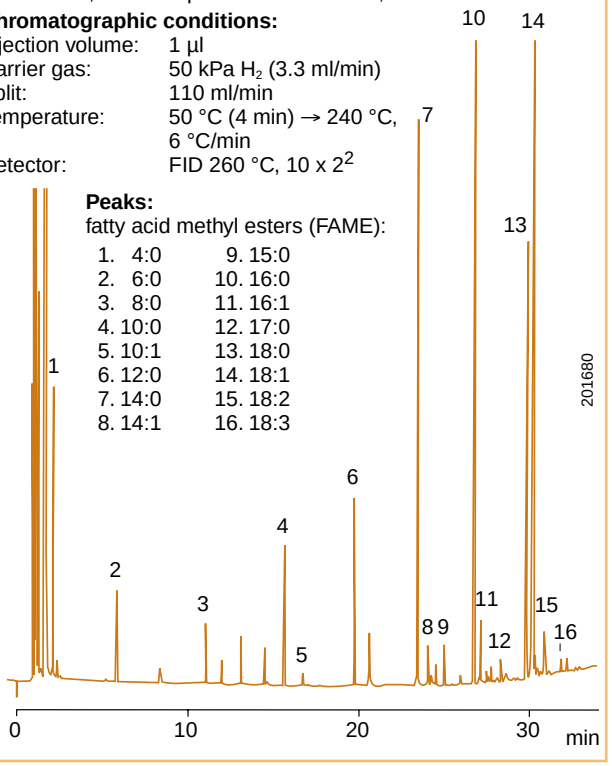
4) M. Syhre et al., GIT Fachz. Lab. 11/94

Procedure:
Dissolve 100 mg sample (e.g. butter) in 5 ml of a suitable 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 should be at least 250 °C.

Fatty acid methyl esters C4:0 – C18:3 from natural butter fat after derivatisation with TMSH

Capillary column: OPTIMA® FFAP, 0.25 µm film, 25 m x 0.32 mm ID, max. temperature 220/240 °C, Cat. No. 726341.25

Chromatographic conditions:
Injection volume: 1 µl
Carrier gas: 50 kPa H_2 (3.3 ml/min)
Split: 110 ml/min
Temperature: 50 °C (4 min) → 240 °C, 6 °C/min
Detector: FID 260 °C, 10 x 2^2

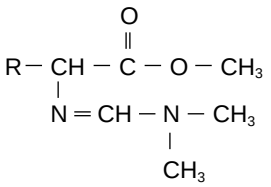


The figure above shows the chromatogram of a butter sample, which was derivatised with TMSH.

Alkylation (methylation)

2) Methylation with DMF-DMA

DMF-DMA derivative



Methylation with DMF-DMA can be applied for fatty acids, primary amines and (partially) amino acids forming N-dimethyl-aminomethylene amino acid methyl esters [Thenot et al., Anal. Letters **5** (1972) 217 – 223, 519 – 529]. DMF-DMA is a poor solvent, for this reason it is necessary to use a mixture of DMF-DMA with benzene, pyridine, THF, acetone (barbiturates) or another solvent.

Procedure:
Add 1 ml of a mixture of DMF-DMA and pyridine (1 : 1) to 1–50 mg fatty acids. As soon as a clear solution has formed, the sample can be injected. However, it is recommended to heat the solution to 60 – 100 °C for 10 – 15 minutes.

3) Methylation with methanol/TMCS

A 1molar solution of TMCS in methanol is suited for the esterification of free carboxylic acids and transesterification of glycerides. Formation of HCl catalyses the reaction. TMCS and silyl ether remove water and thus drive the reaction to completion. The mixture should be prepared fresh.

Procedure:
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.

Silylation

Usually the term silylation in gas chromatography 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 derivatisation. The trialkylsilyl group increases volatility and enhances thermal stability of the sample.

Silylation can be catalysed either acidic by addition of TMCS or basic by addition of pyridine or TSIM (e.g. for sterically hindered functionalities like tert. alcohols).

This chapter describes frequently used reagents and methods for silylation.

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

Reagents for silylation · Ordering information

				Cat. No. for a packing unit of				
10 x 1 ml	20 x 1 ml	1 x 10 ml	5 x 10 ml	1 x 50 ml	1 x 100 ml	6 x 50 ml	6 x 100 ml	12 x 100 ml
BSA = N,O-bis-trimethylsilyl-acetamide								
m.w. 203.4, Bp 71 – 73 °C (35 mm Hg), density d20°/4° = 0.832								
		701210.110	**701210.510**	**701210.150**				
BSTFA = N,O-bis-trimethylsilyl-trifluoroacetamide								
m.w. 257.4, Bp 40 °C (12 mm Hg), density d20°/4° = 0.961								
	701220.201	**701220.110**	**701220.510**					
DMCS = dimethyldichlorosilane								
m.w. 129.06, Bp 70 °C (760 mm Hg), density d20°/4° = 1.07								
						701230.650 *		
HMDS = hexamethyldisilazane								
m.w. 161.4, Bp 126 °C (760 mm Hg), density d20°/4° = 0.7742								
			701240.510			**701240.650 ***		
MSHFBA = N-methyl-N-trimethylsilyl-heptafluorobutyramide								
m.w. 299.1, Bp 148 °C (760 mm Hg),								
	701260.201	**701260.110**	**701260.510**		**701260.1100**		**701260.6100**	
MSTFA = N-methyl-N-trimethylsilyl-trifluoroacetamide								
m.w. 199.1, Bp 70 °C (75 mm Hg), density d20°/4° = 1.11								
	701270.201	**701270.110**	**701270.510**		**701270.1100**	**701270.650**	**701270.6100**	**701270.12100**
MBDSTFA = N-methyl-N-*tert*-butyldimethylsilyl-trifluoroacetamide								
m.w. 241.3, Bp 168 – 170 °C (760 mm Hg), density d20°/4° = 1.121								
701440.101	**701440.201**							
TMCS = trimethylchlorosilane								
m.w. 108.7, Bp 57 °C (760 mm Hg), density d20°/4° = 0.8580								
	701280.201 *					**701280.650 ***		
TMSDEA = N-trimethylsilyl-diethylamine								
m.w. 145.3, Bp 126 – 127 °C (760 mm Hg), density d20°/4° = 0.7627								
		701300.110	**701300.510**					
TSIM = N-trimethylsilyl-imidazole								
m.w. 140.3, Bp 94 – 96 °C (760 mm Hg), density d20°/4° = 0.961								
	701310.201	**701310.110**	**701310.510**					

Reagent mixtures for silylation

Code		20 x 1 ml	1 x 10 ml	5 x 10 ml	1 x 50 ml	1 x 100 ml
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-991	BSTFA-TMCS (99 : 1)	**701490.201**			**701490.150**	**701490.1100**

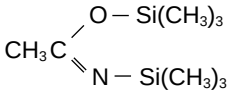
Due to their purpose, derivatisation reagents are very reactive substances. For this reason they should be stored cool and protected from moisture. Our derivatisation reagents are supplied in vials with crimp caps for easy access with a syringe. Vials with pierced sealing disks have limited stability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

* in vials with screw caps

Silylation

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

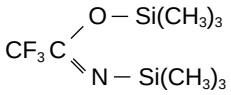
BSA



BSA is a strong silylation reagent, which can be used to form very stable TMS derivatives of a wide variety of compounds such as alcohols, amines, carboxylic acids, phenols, steroids, biogenic amines and alkaloids. However, it is not recommended for use with carbohydrates or very low molecular weight compounds. BSA is a good solvent for polar compounds, but frequently it is used in combination with a solvent (pyridine, DMF etc.) or together with other silylation reagents. When used with DMF, BSA is the reagent of choice for derivatising phenols.

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

BSTFA



BSTFA is a powerful trimethylsilyl donor with approximately the same donor strength as the unfluorinated analog BSA. Reactions of BSTFA are similar to those of BSA. The major advantage of BSTFA over BSA is the greater volatility of its reaction products. This property is particularly useful for the GC analysis of some lower boiling TMS amino acids. BSTFA is nonpolar (less polar than MSTFA), it can be mixed with acetonitrile for improved solubility. For silylating fatty acid amides, hindered hydroxyls and other compounds, which are difficult to silylate (like secondary alcohols and amines), we recommend BSTFA + 1% trimethylchlorosilane (TMCS), which is available under the designation SILYL-991 (Cat. No. 701490).

DMCS [dimethyldichlorosilane]

DMCS $(CH_3)_2SiCl_2$

DMCS is used to form dimethylsilyl (DMS) derivatives. DMS derivatives are much more susceptible to hydrolysis than TMS derivatives, therefore strictly anhydrous conditions during reaction are very important.

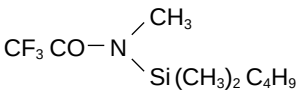
HMDS [hexamethyldisilazane]

HMDS $(CH_3)_3\,Si - NH - Si(CH_3)_3$

HMDS is a weak TMS donor. Used alone its action is slow and not very effective. However, after addition of catalytic quantities of TMCS (e.g. 1%) or as a mixture with TMCS (2:1, v/v; SILYL-21 and SILYL-2110) it becomes a fast and quantitative reagent for trimethylsilylation of organic compounds. Aprotic solvents like acetonitrile, pyridine, dimethylformamide, carbon disulphide and dimethylacetamide are recommended for use with HMDS. Basic work on silylation with HMDS was conducted by C. C. Sweeley et al. [J. Amer. Chem. Soc. **85** (1963) 2497 – 2507].

MBDSTFA [N-methyl-N-*tert*-butyldimethylsilyl-trifluoroacetamide]

MBDSTFA

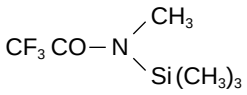


MBDSTFA is a silylation reagent which donates a *tert*-butyldimethylsilyl group (TBDMS) for derivatising active hydrogen atoms in hydroxyl, carboxyl and thiol groups as well as primary and secondary amines. Reactions proceed fast (typical reaction times of 5 to 20 minutes) with high yields (> 96%). The by-products are neutral and volatile. TBDMS ethers are 10^4 times more stable than the corresponding TMS ethers. Due to the large protecting group retention times are longer, which may improve some separations. The high molecular ion concentration at M^+-57 makes MBDSTFA useful for GC-MS applications [M. Donike, J. Zimmermann, J. Chromatogr. **202** (1980) 483].

Silylation

MSTFA [N-methyl-N-trimethylsilyl-trifluoroacetamide]

MSTFA

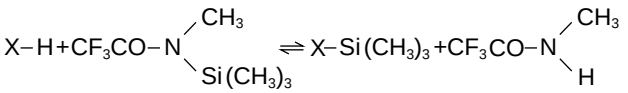


MSTFA is the most volatile trimethylsilyl amide available. BSA and BSTFA, which have been used most frequently in GC silylation, can often be replaced by MSTFA. MSTFA is a very strong TMS donor which does not cause any noticeable FID fouling even after long-time measuring series. The already good solution characteristics can be improved by addition of submolar quantities of protic solvents (e.g. trifluoroacetic acid for extremely polar compounds such as hydrochlorides) or pyridine (e.g. for carbohydrates).

MSTFA can be used for trimethylsilylation of e.g. carboxylic acids, hydroxy and ketocarboxylic acids, amino acids, amines, alcohols, polyalcohols, sugars, mercaptans and similar compounds with active hydrogen atoms. Even amine hydrochlorides can be silylated directly.

MSTFA offers the following advantages:

1. For almost all compounds the reaction proceeds to the right side of the equation



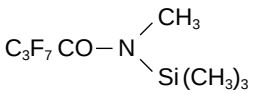
function to be silylated	MSTFA	trimethylsilyl derivative	MTFA

2. Even without a catalyst (1–2% TMCS or TSIM) the reaction rate is several times higher than with other TMS donors such as hexamethyldisilazane (HMDS).

3. As for BSTFA, the by-product of the silylation reaction (N-methyltrifluoroacetamide) features the advantage of high volatility and short retention time.

MSHFBA [N-methyl-N-trimethylsilyl-heptafluorobutyramide]

MSHFBA



MSHFBA is similar to MSTFA in reactivity and chromatography. It may be used for the general purpose trimethylsilylation of carboxylic acids, alcohols, phenols, primary and secondary amines and amino acids. MSHFBA is used either alone or in combination with a catalyst (TMCS, TSIM) or another silylation reagent with or without a solvent. The by-product N-methyl-heptafluorobutyric amide has a lower retention time than the silylating reagent itself. The large ratio of fluorine to silicon of 7 : 1 in MSHFBA is especially useful for flame ionisation detection, since degradation of the excess of MSHFBA does not produce SiO_2 but volatile, non-corrosive silicon compounds.

TMCS [trimethylchlorosilane]

TMCS $(CH_3)_3SiCl$

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

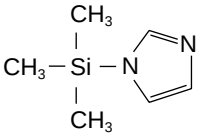
TMSDEA [N-trimethylsilyl-diethylamine]

TMSDEA $(CH_3)_3\,Si-N\,(C_2H_5)_2$

TMSDEA is a strongly basic silylating reagent which is particularly useful for derivatising amino acids, antibiotics, urea-formaldehyde condensates and steroids. The reaction by-product diethylamine is very volatile. TMSDEA is ideal for the synthesis of standards, as the reaction can be easily driven to completion by evaporating the diethylamine.

TSIM [N-trimethylsilyl-imidazole]

TSIM



TSIM is considered to be the strongest hydroxyl silylator and is the reagent of choice for carbohydrates and most steroids (even highly hindered steroids react). 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-derivatisation schemes for compounds with different functional groups, which are to be derivatised differently (e.g. -O-TMS / -N-HFB derivatives of catecholamines). TSIM is used in the trimethylsilylation of alcohols, phenols, organic acids, steroids, hormones, glycols, nucleotides and narcotics.

For ordering information of silylation reagents please see page 355.

Silylation

Methods for silylation:

Method 1
Silylation with BSA, BSTFA or SILYL-991
Procedure: Add 0.5 ml of the silylation reagent to 1–10 mg sample; if necessary, add some solvent (normally pyridine or DMF [dimethylformamide] are used). 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.

Silylation with BSA, BSTFA or SILYL-991
suitable for alcohols, phenols, aromatic acids, amino acids, steroids, nucleotides
Reagents:
BSA = N,O-bis-trimethylsilyl-acetamide
BSTFA = N,O-bis-trimethylsilyl-trifluoroacetamide
SILYL-991 = BSTFA : TMCS (99 : 1)

Method 2
Silylation with TSIM, SILYL-1139 or TMSDEA
Procedure: 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 analysed directly.
(When using SILYL-1139, the presence of water does not interfere.)

Silylation with TSIM, SILYL-1139 or TMSDEA
Reagents:
TSIM = N-trimethylsilyl-imidazole
SILYL-1139 = TSIM : pyridine (11 : 39)
suitable for sugars, glycols, alcohols
recommended solvent pyridine
TMSDEA = N-trimethylsilyl-diethylamine
suitable for carboxylic acids, amino acids, amines, hydroxyls in general
recommended solvent THF or acetonitrile

Method 3
Silylation with MSTFA, MSHFBA or MBDSTFA
Procedure: The general procedure is the same as for method 2; if TFA is used as a solvent, proceed as follows [M. Donike, J. Chromatogr. **85** (1973) 1–7]: 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.

Silylation with MSTFA, MSHFBA or MBDSTFA
suitable for alcohols, phenols, primary and secondary amines, carboxylic acids, amino acids
Reagents for trimethylsilylation:
MSTFA = N-methyl-N-trimethylsilyl-trifluoroacetamide
MSHFBA = N-methyl-N-trimethylsilyl-heptafluoro-butyramide
Reagent for tert.-butyldimethylsilylation:
MBDSTFA = N-methyl-tert.-butyldimethylsilyltrifluoro-acetamide

Method 4
Silylation with SILYL-21 or SILYL-2110
Procedure: Carefully add SILYL-21 or SILYL-2110 to 1–10 mg of the sample. A precipitate of ammonium chloride does not interfere. If the sample should not dissolve within 5 minutes, 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 derivatisation of 3-ketosteroids we recommend to use DMF (dimethylformamide).

Silylation with SILYL-21 or SILYL-2110
suitable for sugars, glycols, sterically unhindered alcohols, carboxylic acids, acids in urine, hydroxy fatty acids, nucleotides, steroids, vitamin D, xanthone derivatives
Reagents:
SILYL-21 = HMDS : TMCS (2 : 1)
SILYL-2110 = HMDS : TMCS : pyridine (2 : 1 : 10)
HMDS = hexamethyldisilazane
TMCS = trimethylchlorosilane

Method 5
Silylation with BSA in combination with other silylation reagents
Procedure: 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 hours at 60 °C). After addition of TSIM even strongly hindered hydroxyl groups will react (reaction time 6 – 24 hours at 60 °C).

Silylation with BSA in combination with other silylation reagents
suitable for steroids, primary and secondary amines
Reagents:
BSA : TMCS (5 : 1)
BSA : TMCS : TSIM (3 : 2 : 3)

Method 6
N-Trifluoroacetylation / O-trimethylsilylation with MSTFA and MBTFA
Procedure: Completely silylate 2 mg of the sample with 0.3 ml MSTFA e.g. according to method 3. After addition of 0.3 ml MBTFA the N-trimethylsilyl group is replaced by the N-trifluoroacetyl group. The mixture can be analysed directly.

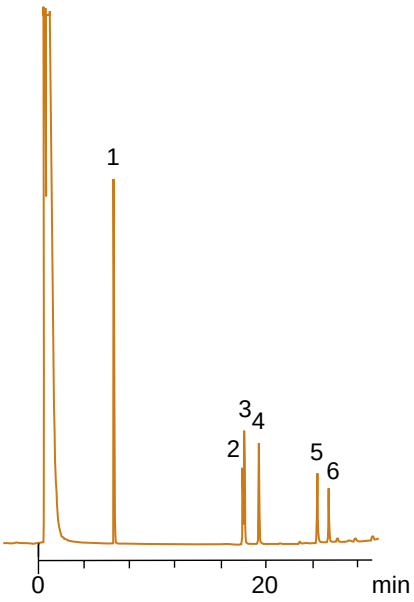
N-Trifluoroacetylation / O-trimethylsilylation with MSTFA and MBTFA
suitable for multifunctional compounds
Reagents:
MSTFA = N-methyl-N-trimethylsilyl-trifluoroacetamide
MBTFA = N-methyl-bis(trifluoroacetamide)

Environmental standards for chromatography

Ordering information

Designation	pack of	composition	Cat. No.
Haloform test mixture (quantitative standard) in n-pentane	1 ml	9 halogenated hydrocarbons acc. to German drinking water specifications (in ng/ml): dichloromethane (795), chloroform (75), 1,1,1-trichloroethane (67), carbon tetrachloride (80), trichloroethylene (73), bromodichloromethane (100), dibromochloromethane (122), tetrachloroethylene (81), bromoform (145)	**722311**
Haloform test mixture (quantitative standard) in methanol for headspace analyses	1 ml	9 halogenated hydrocarbons in increased concentration for calibration acc. to German Industrial Standard DIN 38407, part 5 (in µg/ml): dichloromethane (158.4), chloroform (14.9), 1,1,1-trichloroethane (13.4), carbon tetrachloride (15.9), trichloroethylene (14.6), bromodichloromethane (20), dibromochloromethane (24.5), tetrachloroethylene (16.2), bromoform (28.9)	**722371**
Haloform test kit	11 x 1 ml	1 ml each of 9 single undiluted halogenated hydrocarbons and 1 ml each of test mixtures Cat. Nos. 722311 and 722371	**722312**
Pesticide test mixture (quantitative standard) in n-hexane	1 ml	10 µg/ml each of α-BHC, HCB, β-BHC, γ-BHC, δ-BHC, heptachlor, aldrin, dieldrin, endrin, *p*,*p*'-DDT, mirex	**722313**
PAH test mixture acc. to EPA (quantitative standard) in toluene	1 ml	20 µg/ml each of naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benz[a]anthracene, chrysene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, indeno[1,2,3-cd]pyrene, dibenz[a,h]anthracene, benzo[ghi]perylene	**722314**
PAH test mixture acc. to German drinking water specifications (quantitative standard) in toluene	1 ml	20 µg/ml each of fluoranthene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, indeno[1,2,3-cd]pyrene, benzo[ghi]perylene	**722331**
Phenol test mixture (quantitative standard) in methanol	1 ml	0.1 mg/ml each of phenol, 2-chlorophenol, 2-nitrophenol, 2,4-dimethylphenol, 2,4-dichlorophenol, 4-chloro-3-methylphenol, 2,4,6-trichlorophenol, s2,4-dinitrophenol, 4-nitrophenol, 2-methyl-4,6-dinitrophenol, pentachlorophenol	**722315**
BTX standard (quantitative) in methanol	1 ml	10 ng/µl each of benzene, ethylbenzene, toluene, *m*-, *o*-, *p*-xylene	**722372**

PAH test mixture acc. to German drinking water specifications (Cat. No. 722331)



Capillary column: OPTIMA® 5, 0.25 µm film, 25 m x 0.32 mm ID, max. temp. 340/360 °C, Cat. No. 726314.25
Injection volume: 2 µl
Carrier gas: 0.6 bar H_2
Split: 1 : 10
Temperature: 80 °C ↑ 180 °C → 300 °C, 4 °C/min
Detector: FID 300 °C, 2^4

Peaks:
1. Fluoranthene
2. Benzo[b]fluoranthene
3. Benzo[k]fluoranthene
4. Benzo[a]pyrene
5. Indeno[1,2,3-cd]pyrene
6. Benzo[ghi]perylene

200450

Environmental standards for chromatography

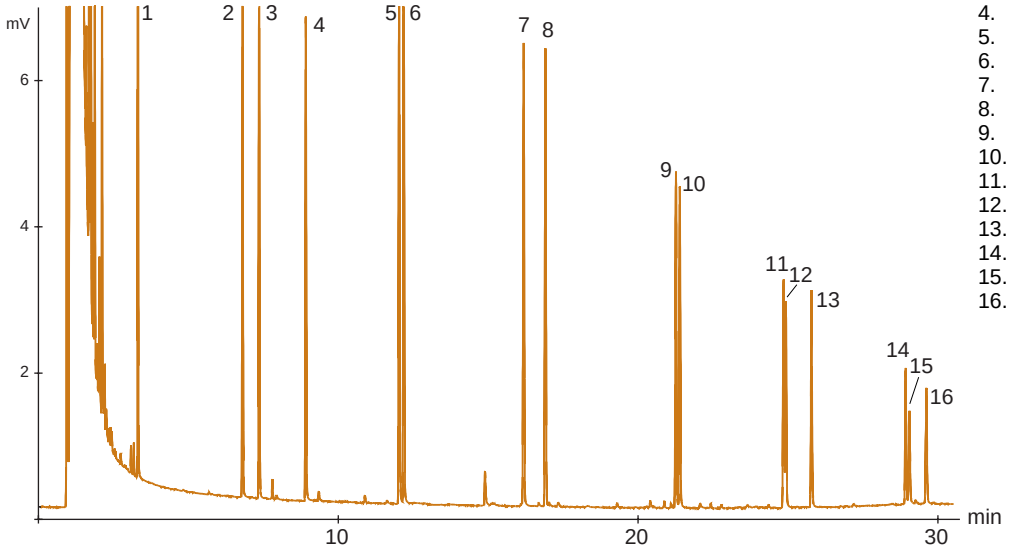
PAH test mixture acc. to EPA for GC (Cat. No. 722314)

200510

Capillary column: OPTIMA® 5, 0.25 µm film, 30 m x 0.32 mm ID, max. temperature 340/360° C, Cat. No. 726314.30

Chromatographic conditions:

Sample:	PAH test mixture according to EPA (20 µg/ml each in toluene)
Injection volume:	1.0 µl
Carrier gas:	H_2, 70 KPa
Split:	1 : 15
Temperature:	100° C, 7° C/min → 300° C
Detector:	FID, 300 °C, 2^4



Peaks:

1. Naphthalene
2. Acenaphthalene
3. Acenaphthene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Fluoranthrene
8. Pyrene
9. Benz[a]anthracene
10. Chrysene
11. Benzo[b]fluoranthene
12. Benzo[k]fluoranthene
13. Benzo[a]pyrene
14. Indeno[1,2,3-cd]pyrene
15. Dibenz[ah]anthracene
16. Benzo[ghi]perylene

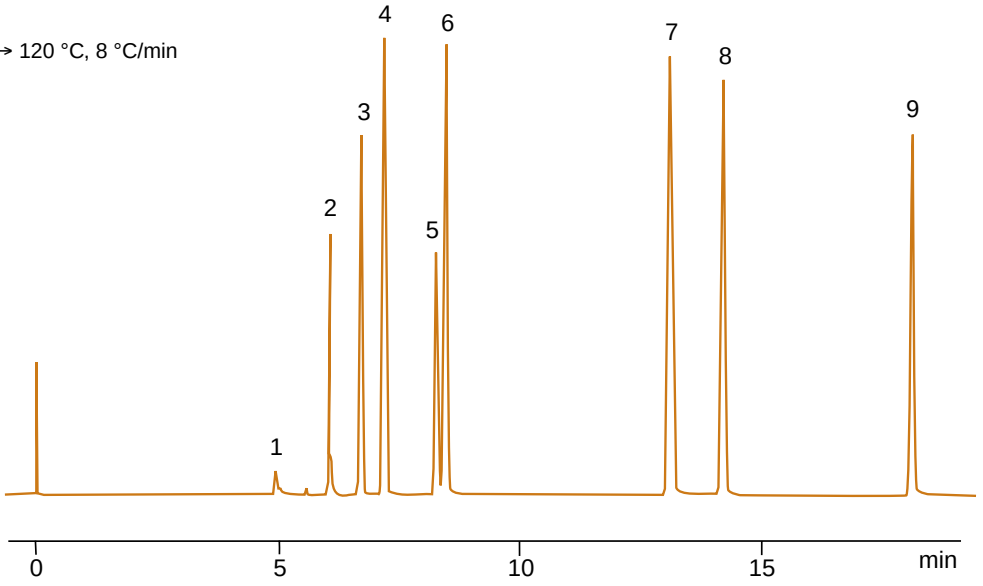
Haloform test mixture (Cat. No. 722311)

211190

Capillary column:	FS-SE-54, 0.35 µm film, 50 m x 0.25 mm ID, max. temperature 300 °C, Cat. No. 733623.50
Injection volume:	1 µl
Carrier gas:	1 bar N_2
Split:	about 1 : 30
Temperature:	45 °C (10 min) → 120 °C, 8 °C/min
Detector:	ECD 260 °C, 2^8

Peaks:

1. Dichloromethane
2. Chloroform
3. 1,1,1-Trichloroethane
4. Carbon tetrachloride
5. Trichloroethylene
6. Bromodichloromethane
7. Dibromochloromethane
8. Tetrachloroethylene
9. Bromoform



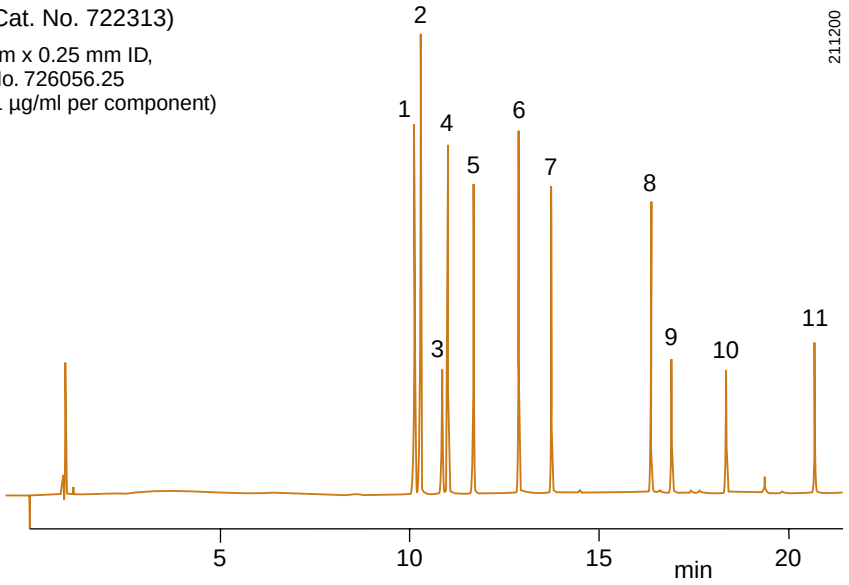
Environmental standards for chromatography

Pesticide test mixture (Cat. No. 722313)

Capillary column:	OPTIMA® 5, 0.25 µm film, 25 m x 0.25 mm ID, max. temp. 340/360 °C, Cat. No. 726056.25
Injection volume:	0.5 µl (test mixture diluted to 1 µg/ml per component) in *n*-hexane
Carrier gas:	100 kPa H_2
Split:	330 ml/min
Temperature:	100 °C → 270 °C, 8 °C/min
Detector:	ECD 300 °C, 2^{10}

Peaks:

1. α-BHC
2. HCB
3. β-BHC
4. γ-BHC
5. δ-BHC
6. Heptachlor
7. Aldrin
8. Dieldrin
9. Endrin
10. *p,p'*-DDT
11. Mirex

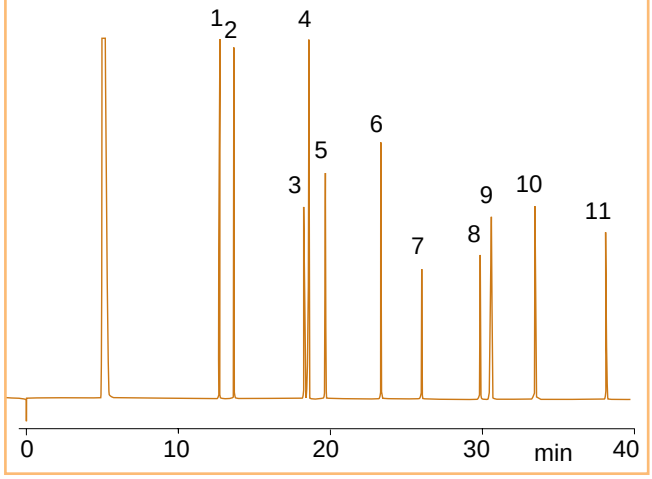


Phenol test mixture (Cat. No. 722315)

Capillary column:	FS-SE-54, 0.35 µm film, 50 m x 0.25 mm ID, max. temperature 300 °C, Cat. No. 733623.50
Injection volume:	1 µl
Carrier gas:	0.95 bar N_2
Split:	about 1 : 40
Temperature:	80 °C → 260 °C, 5 °C/min
Detector:	FID 300 °C, 2^4

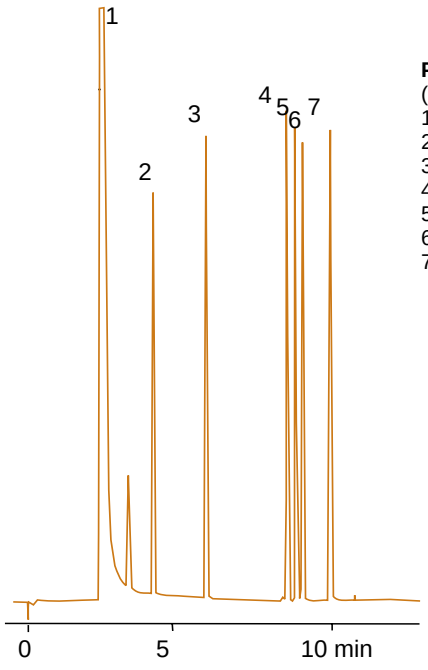
Peaks:

1. Phenol
2. 2-Chlorophenol
3. 2-Nitrophenol
4. 2,4-Dimethylphenol
5. 2,4-Dichlorophenol
6. 4-Chloro-3-methylphenol
7. 2,4,6-Trichlorophenol
8. 2,4-Dinitrophenol
9. 4-Nitrophenol
10. 2-Methyl-4,6-dinitrophenol
11. Pentachlorophenol



BTX standard (Cat. No. 722372)

Capillary column:	HYDRODEX® β-PM, 50 m x 0.25 mm ID, max. temperature 250 °C Cat. No. 723370.50
Injection volume:	2 µl
Carrier gas:	120 kPa H_2 (2.45 ml/min)
Split:	40 ml/min
Temperature:	60 °C → 100 °C, 2 °C/min
Detector:	FID 250 °C, 2^4



Peaks:
(10 ng/µl each)

1. Methanol
2. Benzene
3. Toluene
4. *p*-Xylene
5. *m*-Xylene
6. Ethylbenzene
7. *o*-Xylene

For ordering information of environmental standards please see page 359.

Test mixtures for GC capillary columns

Ordering information

Designation	pack of	composition	Cat. No.
Polarity mixture POL_5 (qualitative standard) in *n*-pentane	1 ml	1-butanol, benzene, methyl butyrate, toluene, cyclopentanone, 1-octene, dibutyl ether	**722306**
Activity test mixture (FA-TMS test according to Donike) in MSTFA/*n*-hexane (1 + 4)	1 ml	1 mg/ml each of TMS capric acid (C_{10}), TMS myristic acid (C_{14}), TMS stearic acid (C_{18}), TMS behenic acid (C_{22}), hexadecane (C_{16}), eicosane (C_{20}), tetracosane (C_{24}), octacosane (C_{28})	**722307**
Grob test mixture (modified) in *n*-hexane	1 ml	(in mg/ml) *n*-decane (2.83), *n*-undecane (2.87), *n*-octanol (3.55), 2,6-dimethylphenol (3.20), 2,6-dimethylaniline (3.20), methyl decanoate (4.23), dicyclohexylamine (3.13), methyl undecanoate (4.19), methyl dodecanoate (4.13)	**722310**
MN OPTIMA® test mixture in pentane	1 ml	0.1% each of undecane, dodecane, octanol, dimethylaniline, decylamine, methyl decanoate, methyl undecanoate, henicosane, docosane, tricosane (chromatograms see page 311)	**722316**
MN OPTIMA® amine test mixture in methanol	1 ml	0.2% diisobutylamine, 1% diethanolamine, 0.2% 2,6-dimethylaniline, 0.2% *o*-propanol-pyridine, 0.2% dicyclohexylamine, 0.2% dibenzylamine	**722317**
FAME test mixture in hexane	1 ml	0.1% each of FAMEs C4, C6, C8, C10, C12, C14, C16, C18, C18:1 cis, C18:1 trans, C18:2, C18:3, C20, C22, C22:1, C24 (chromatogram see page 329)	**722320**

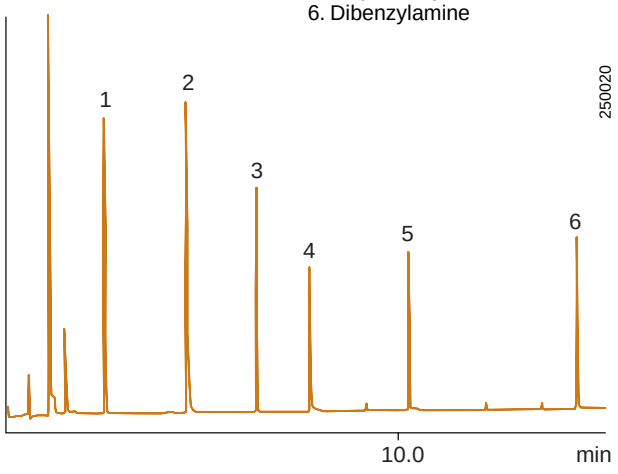
Separation of the test mixture OPTIMA® Amine
(Cat. No. 722317)

Capillary column: OPTIMA® 5 Amine, 1.0 µm film, 30 m x 0.32 mm ID, max. temp. 300/320 °C, Cat. No. 726353.30

Chromatographic conditions:

Sample: OPTIMA® Amine test mixture (Cat. No. 722317)
Injection volume: 1 µl
Carrier gas: 0.6 bar H_2
Split: 1:50
Temperature: 100 °C → 290 °C, 10 °C/min
Detector: FID, 280 °C, 2^6

Peaks:
1. Diisobutylamine
2. Diethanolamine
3. 2,6-Dimethylaniline
4. *o*-Propanol-pyridine
5. Dicyclohexylamine
6. Dibenzylamine

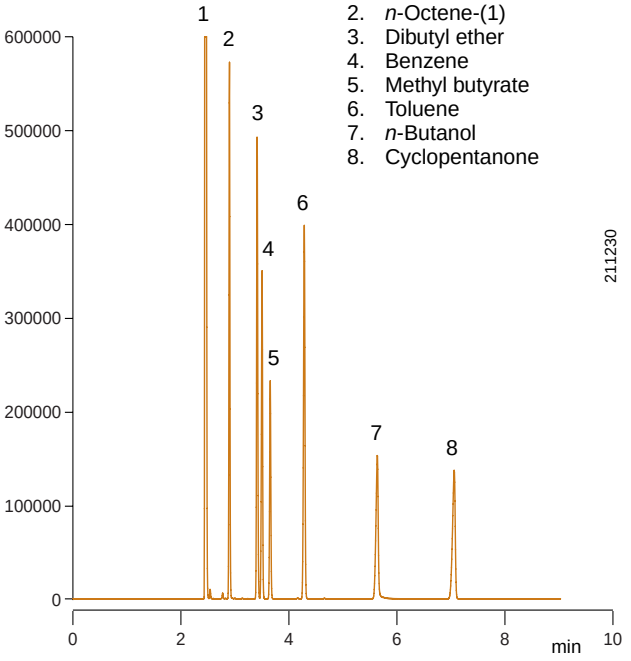


Polarity mixture POL_5 (qualitative)
(Cat. No. 722306)

Capillary column: OPTIMA® Wax, 0.25 µm film, 25 m x 0.25 mm ID, max. temp. 250/260 °C, Cat. No. 726600.25

Injection: 1 µl, split: 1: 50, 220 °C
Carrier gas: 0.75 bar N_2
Temperature: 80 °C isothermal
Detector: FID, 220 °C, 10^1, 2^7

Peaks:
1. *n*-Pentane
2. *n*-Octene-(1)
3. Dibutyl ether
4. Benzene
5. Methyl butyrate
6. Toluene
7. *n*-Butanol
8. Cyclopentanone



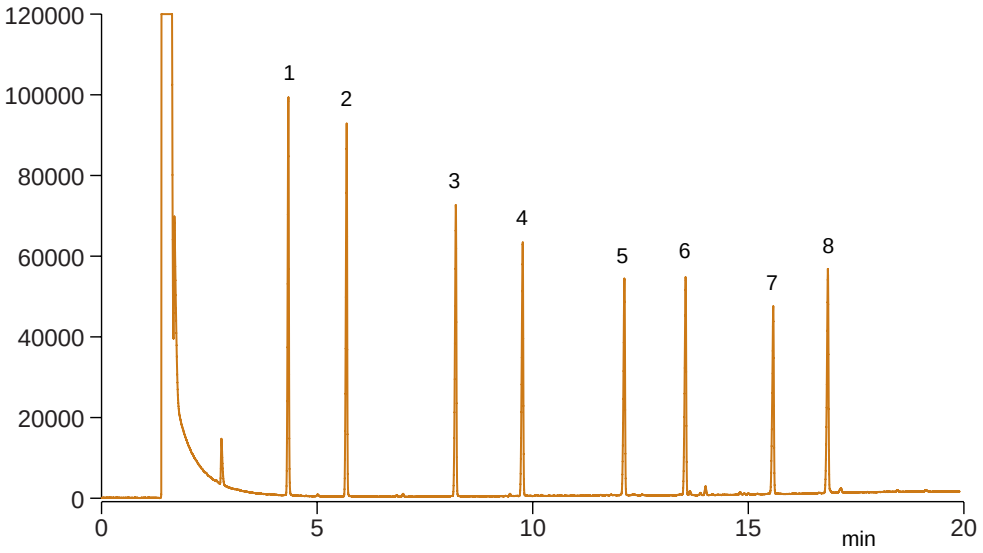
Test mixtures for GC capillary columns

Activity test mixture (Cat. No. 722307)

Capillary column:	OPTIMA® 5, 1.0 µm film, 25 m x 0.32 mm ID, max. temp. 340/360 °C, Cat. No. 726316.25
Injection:	1 µl, split: 1: 40, 300 °C
Carrier gas:	0.6 bar H_2
Temperature:	150 °C → 300 °C (8 min), 10 °C/min
Detector:	FID, 300 °C, 10^1, 2^3

Peaks:

1. TMS capric acid (C_{10})
2. Hexadecane (C_{16})
3. TMS myristic acid (C_{14})
4. Eicosane (C_{20})
5. TMS stearic acid (C_{18})
6. Tetracosane (C_{24})
7. TMS behenic acid (C_{22})
8. Octacosane (C_{28})



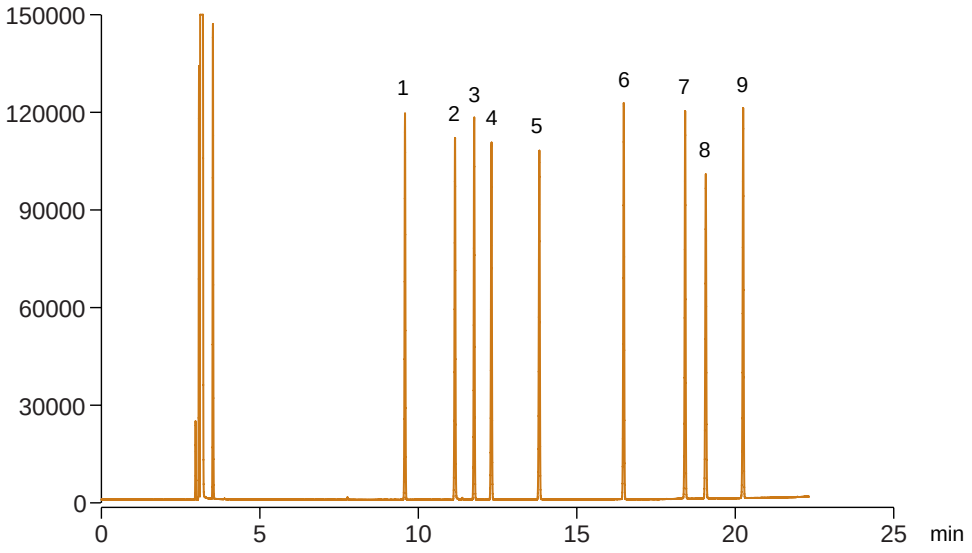
211240

Grob test mixture (Cat. No. 722310)

Capillary column:	OPTIMA® 5, 1.0 µm film, 50 m x 0.25 mm ID, max. temp. 340/360 °C, Cat. No. 726807.50
Injection:	1 µl, split 1: 40, 280 °C
Carrier gas:	1.5 bar H_2
Temperature:	80 °C → 280 °C (10 min), 8 °C/min
Detector:	FID, 280 °C, 10^1, 2^6

Peaks:

1. *n*-Decane
2. 1-Octanol
3. *n*-Undecane
4. 2,6-Dimethylphenol
5. 2,6-Dimethylaniline
6. Methyl decanoate
7. Methyl undecanoate
8. Dicyclohexylamine
9. Methyl dodecanoate



211250

Accessories for gas chromatography

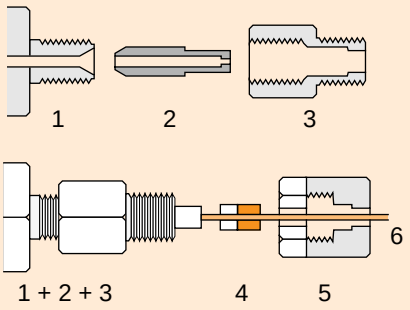
In this chapter you can find

- **special accessories for capillary columns**
- **Valco fittings for capillary GC**
- **special accessories for packed columns and for packing GC columns**
- **products for gas flow measurement and gas purification**
- **ferrules**
- **septa**

For other fittings and valves for gas chromatography please ask for our Valco programme, which is available on request.

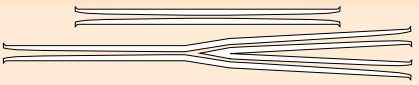
Accessories for capillary columns

Ordering information				Product specification
Description		pack of	Cat. No.	application
Empty cages for GC capillary columns				
Cage 160	160 mm dia.	1	**723721**	for all column lengths up to 50 m with 0.25 and 0.32 mm ID
Cage 190/32	190 mm dia.	1	**723722**	for column lengths up to 25 m with 0.53 mm ID / 60 m x 0.32 mm ID
Cage 190/58	190 mm dia.	1	**723734**	recommended for 50 and 60 m columns with 0.53 mm ID
Flushing unit for capillaries				
Flushing unit		1	**708317**	for capillary columns with immobilised phases
Spare solvent tubes		2	**708316**	replacement for flushing unit
Special products for connecting capillary columns				
Graphseal® connecting system for capillary columns				
Graphseal adaptor		1	**708320**	
Graphseal nut, slotted		2	**708321**	
Graphseal ferrule, **0.4 mm** bore		10	**708337**	
Graphseal ferrule, **0.5 mm** bore		10	**708318**	
Graphseal ferrule, **0.8 mm** bore		10	**708319**	
Universal capillary glass connectors for fused silica capillaries from 0.2 to 0.53 mm ID				
linear		5	**707971**	
linear		10	**707972**	
Y splitter		1	**707973**	

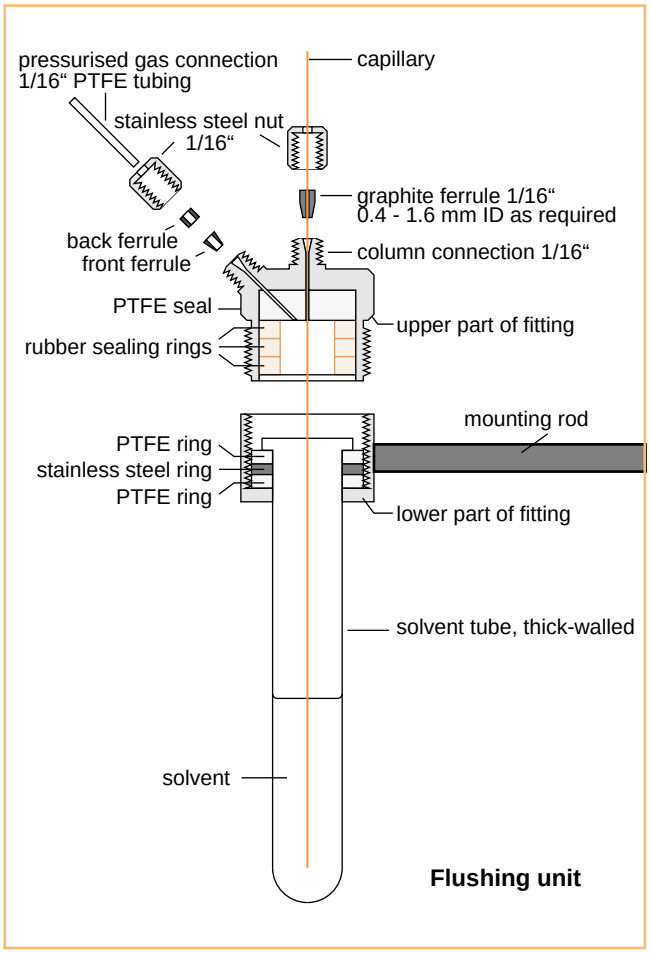


1 1/16“ exit, injector or detector
2 Grapseal insert, 0.8 mm bore
3 Graphseal reducing unit
4 Graphseal ferrule
5 slotted nut
6 capillary

2 + 3 + 5 Graphseal adaptor Cat. No. 708320



Accessories for gas chromatography



Flushing unit for GC capillary columns with chemically bonded (immobilised) phases

Capillary columns with immobilised phases can be flushed with solvents to remove contaminations (also see page 332). For this purpose we offer a flushing unit consisting of a thick-walled glass tube and a stainless steel head with fittings. The glass tube holds about 20 ml of solvent. The head has two $^{1}/_{16}$” fittings: one for the capillary to be cleaned, and one for the pressurizing gas (usually nitrogen). The figure above shows the design of the complete flushing unit. Spare glass tubes are available separately.

Special products for connecting capillary columns in GC

This chapter describes products, which have been especially developed for fused silica capillaries. Other fittings for GC can be found in our Valco programme, which is available on request.

Graphseal® connecting system for capillaries

Basic component of the Graphseal system is the Graphseal ferrule: a stainless steel ferrule filled with graphite – the ideal sealing material for capillaries.

The capillary is mounted on a $^{1}/_{16}$" exit (detector, injector etc.) with the appropriate ferrule, a Graphseal nut (with slit) and an adaptor.

The design of the system can be seen from the ordering information table on the previous page.

Glass connectors for GC capillary columns

These capillary connectors are manufactured from deactivated glass with slightly tapered inner diameter and allow to join two fused silica capillaries of equal or different diameters. Advantages compared to stainless steel fittings are

- easy connection without tools
- optical control during connection
- negligible heat capacity
- no dead volume

For use, scratch the capillaries to be connected with a diamond file (see below), break them to give a square and clean end and under slight pressure and a 90° turn push them into the glass connector. You can control the proper fit of the capillary by a dark ring formed at the column end. During operation of the column in the gas chromatograph at elevated temperatures a mechanically stable connection is formed.

For connection of capillary columns we also recommend the Valco capillary fittings and unions on the following pages.

Valco fittings for capillary GC

Valco manufactures two different types of fused silica adaptors for capillaries: one-piece FS adaptors and two-part removable FSR adaptors.

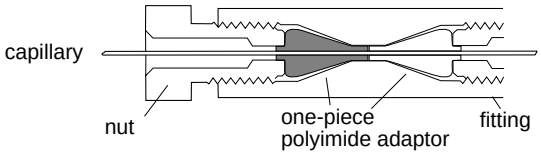
For other fittings and valves for GC please ask for our Valco programme, which is available on request.

Ordering information					Product specification
Valco code	description		pack of	Cat. No.	
One-piece fused silica adaptors					
		for capillary OD			
FS.25	1/32”	< 0.25 mm	5	**724405**	
FS.4	1/32”	0.25 – 0.4 mm	5	**724243**	
FS.5	1/32”	0.4 – 0.5 mm	5	**724244**	
FS1-.4	1/16”	< 0.4 mm	5	**724406**	
FS1-.5	1/16”	0.4 – 0.5 mm	5	**724407**	
FS1-.8	1/16”	0.6 – 0.8 mm	5	**724408**	
Removable fused silica adaptors (incl. nuts)					
FSR.25	1/32”	< 0.25 mm	5	**724409**	
FSR.4	1/32”	0.25 – 0.4 mm	5	**724410**	
FSR.5	1/32”	0.4 – 0.5 mm	5	**724411**	
FS1R.5	1/16”	< 0.5 mm	5	**724335**	
FS1R.8	1/16”	0.5 – 0.8 mm	5	**724334**	
Replacement liners					
FSL.25	1/32”	< 0.25 mm	5	**724412**	
FSL.4	1/32”	0.25 – 0.4 mm	5	**724413**	
FSL.5	1/32”	0.4 – 0.5 mm	5	**724414**	
FS1L.5	1/16”	< 0.5 mm	5	**724415**	
FS1L.8	1/16”	0.5 – 0.8 mm	5	**724416**	
Special nut for fused silica adaptors					
ZCN1	1/16”	counterbored	1	**724417**	
For standard Vespel ferrules as well as standard nuts please see the Valco programme, which is available on request.					
Unions, Tees and crosses for fused silica adaptors (without ferrules, but incl. standard nuts)					
ZU.5TJ	1/32”– 1/32”	for butt connection	1	**724418**	
ZU1TJ	1/16”– 1/16”	for butt connection	1	**724333**	
ZT.5J	1/32”	Tee	1	**724421**	
ZT1CJ	1/16”	Tee, capillary bore	1	**724336**	
ZX.5J	1/32”	cross	1	**724422**	
ZX1CJ	1/16”	cross, capillary bore	1	**724337**	
Tools for Valco fused silica adaptors					
OEW	open end wrench (3/16” x 1/4”)		1	**724423**	for use with 1/32” fittings
PV	pin vise and drill index (0.34 to 1.0 mm)		1	**724424**	for removing ferrules from Tees and crosses and for enlarging the interior diameter of fused silica adaptors

Valco fittings for capillary GC

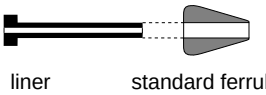
FS adaptor, one piece

Union with FS adaptor

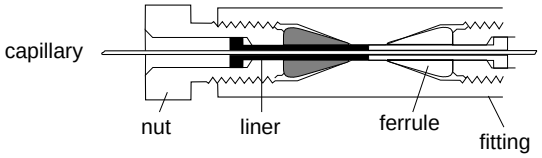


The one-piece FS adaptor is recommended for use in fittings where the polyimide ferrule need not be removed.

FSR adaptor, two-part

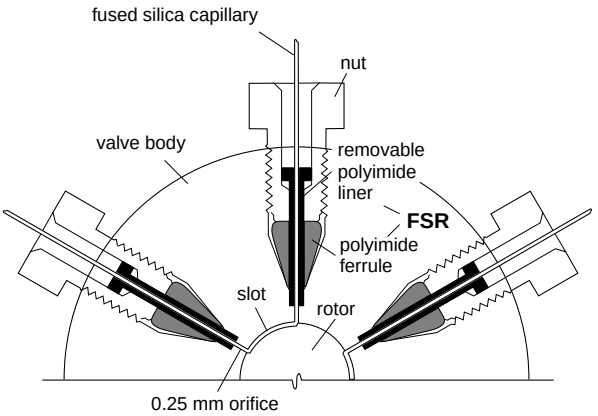


Union with FSR adaptor



The FSR adaptor is recommended for use in Valco valves. It consists of two components: a liner which slides over the fused silica tubing, and a ferrule. Both components are made of high temperature polyimide alloys. The liner has an enlarged diameter at one end which fits within the nut. This design ensures that the liner and the tube within are removed as the nut is unscrewed from the valve (see figure). The $^1/_{16}$" FSR adaptor comes with a special counterbored $^1/_{16}$" nut (ZCN1) to receive the liner. The $^1/_{32}$" adaptor works with standard Valco $^1/_{32}$" nuts.

Removable FSR adaptors in a valve



For use of fused silica adaptors with Valco valves please order the number of adaptors (FSR required) when you order the valve, or when you want to use an existing valve with open tubular columns. Please note that for $^1/_{16}$" FSR adaptors you have to use the special counterbored nut ZCN1 which is supplied with the adaptors FS1R.5 and FS1R.8.

Examples:

1) For connecting a capillary with 0.32 mm ID (0.5 mm OD) to a valve with $^1/_{32}$" fittings we recommend the removable FSR adaptor FSR.5.
2) For connecting a capillary with 0.53 mm ID (0.8 mm OD) to a valve with $^1/_{16}$" fittings we recommend the removable FSR adaptor FS1R.8.

To order Valco fittings for use with fused silica adaptors (FS or FSR recommended), add suffix “J“ to the fitting code and specifiy the appropriate number of adaptors separately. The stainless steel ferrules normally provided with the fittings are omitted since they are replaced by the FS (or FSR) adaptors. Again, for $^1/_{16}$" FSR adaptors use the counterbored nut ZCN1 supplied with the adaptor.

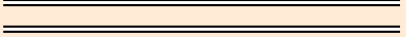
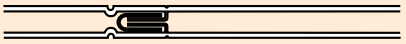
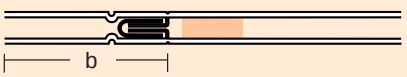
Examples:

1) Connection of 2 capillaries with 0.25mm ID and 0.4mm OD: either use a $^1/_{32}$" union ZU.5TJ and 2 FS adaptors FS.4 or a $^1/_{32}$" union ZU.5TJ and 2 removable FSR adaptors FSR.4
2) Connection of 2 capillaries with 0.53 mm ID and 0.8 mm OD: we recommend either a $^1/_{16}$" union ZU1TJ and 2 FS adaptors FS1-.8 or a $^1/_{16}$" union ZU1TJ and 2 removable FSR adaptors FS1R.8

Should a tube break in a straight-through union, remove the nuts and the tube opposite the broken one. Clear the fitting by passing a drill or wire of appropriate diameter into the unbroken side and through the center of the fitting. A pin vise and drill index are used for removing ferrules from Tee and cross fittings, and for enlarging the interior diameter of FS adaptors (Valco code PV).

Valco fittings for capillary GC

More accessories for capillary columns

Ordering information			Product specification			
Description	pack of	Cat. No.	drawing / application			
Glass injection liners protect samples from catalytic decomposition at active metal surfaces of the injector				length [mm]	OD [mm]	ID [mm]
for Hewlett-Packard instruments						
Liner with glass wool for split injection	1	**708380**		78	6.1	4
Liner for splitless injection	1	**708382**		78	6.1	4
Liner for splitless injection	1	**708381**		78	6.1	2
Liner with flow reversal b = 22 mm	1	**708383**		78	6.1	4
Liner with flow reversal packed with 10% OV-1 on Chromosorb W-HP 80/100	1	**708385**		78	6.1	4
for Carlo Erba / Fisons instruments						
Liner with flow reversal	1	**708384**	fig. see obove, b = 46 mm	98	6.1	4
Liner with flow reversal	1	**708387**	packed as 708385	98	6.1	4
Other accessories for capillary GC						
PTFE shrinking tube, thin-walled	1 m	**708305**	for connecting capillaries, min. ID expanded 1.17 mm, max. ID shrunk 0.40 mm			
Diamond file	1	**708300**	for cutting capillaries and straightening capillary ends			
Magnifying lens with scale	1	**706296**	magnification 7x			

Glass injection liners for gas chromatography

Glass injection liners protect the sample from catalytic decomposition at active metal surfaces in the injector. The programme comprises liners with glass wool for split injection, liners for splitless injection and liners with flow reversal for different gas chromatographs.

Procedure for silanising glass injection liners (inserts):
inserts are refluxed in a solution of 40% HMDS (hexamethyldisilazane, Cat. No. 710240.510) in toluene for two hours. After cooling they are rinsed first with methanol, then with dichloromethane and finally dried 1 hour at 100 °C in a drying cabinet. Silanised liners have to be handled with gloves.

PTFE shrinking tube

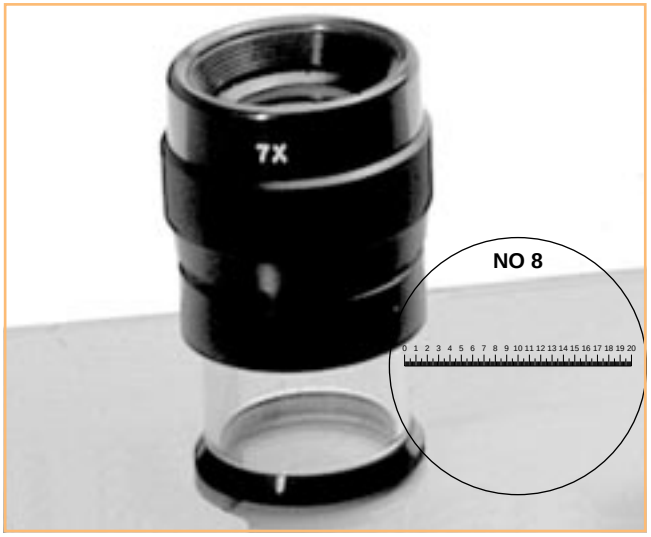
This material is also used for connecting capillaries. The minimum inner diameter expanded is 1.17 mm, the maximum ID shrunk is 0.40 mm. Shrinking occurs above 310 °C. Connections with PTFE shrinking tube are applicable up to 200 °C only. They should never be used above 250 °C.

Diamond file

The diamond file is a useful tool for cutting capillaries and smoothing ends of capillaries. Square capillary ends without protruding particles are especially important for butt connections (e.g. in Valco unions).

Magnifying lens for gas chromatography

A magnifying lens is a very versatile tool for any laboratory. In capillary GC it is often important to inspect column integrity or check cut ends of capillaries. When closing a column by melting the magnifying lens can be used to check whether the column is really closed or whether an open channel has formed in the sealed end. Our lens provides 7fold magnification and is supplied with a scale as pictured in the figure below. The space between lines corresponds to $^1/_{10}$ mm.



Accessories for packed GC columns

In this chapter we describe materials and tools which are useful or necessary for packing GC columns.

Ordering information			Product specification
Description	pack of	Cat. No.	
Stainless steel tubing for packed GC columns			
1/8” OD x 2 mm ID	1 m*	**706128**	* stainless steel tubing for GC columns is available in three diameters ($^1/_8$", $^1/_4$" or 6 mm OD), either in pieces from 1 to 10 meters length or in coils of 15 m. Please indicate the length required when ordering.
1/8” OD x 2 mm ID	15 m	**706124**	
1/4” OD x 5.33 mm ID	1 m*	**706239**	
1/4” OD x 5.33 mm ID	15 m	**706358**	
Glass wool			
Glass wool, long fibres, for packed GC columns	50 g	**706200**	for terminating the packing in a packed GC column you can choose from different types of glass wool and glass fibre wadding.
Glass wool, long fibres, DMCS treated, for packed GC columns	50 g	**706201**	
Glass fibre wadding silanised, very fine fibres	25 g	**718002**	
Quartz wool, very fine fibres	25 g	**718587**	
Glass wool inserter for GC columns	1	**706116**	
Glass wool extractor for GC columns	1	**706117**	

Devices for gas flow measurement

Soap film flowmeters are the primary standard for measuring gas flows. We offer three different sizes.

Leak check 734145 is an ideal residue-free solution to be used with the flowmeters described above.

Ordering information		
Description	Pack of	Cat. No.
Flowmeters and accessories		
1 ml soapfilm flowmeter	1	**734142**
10 ml soapfilm flowmeter	1	**734143**
25 ml soapfilm flowmeter	1	**734144**
Leak check in bottles	250 g	**734145**

Systems for in-line gas purification

Gas chromatography is quite demanding with respect to the purity of the carrier and burner gases used, especially if maximum column lifetime and interference-free detector operation are required. Since the gas supplies available in a laboratory often do not meet the quality requirements, installation of an in-line gas purification system can be generally recommended.

For this purpose we offer purification systems which use special absorber cartridges to reduce the concentration of contaminants in the gas flow almost to the detection limit. Depending on the substance to be removed, the method is based on physisorption or chemisorption. The programme comprises cartridges – packed under inert atmosphere – for the removal of oxygen and humidity as well as activated carbon cartridges for removal of hydrocarbons or oil traces. All cartridges can be fitted into tubing lines with $^1/_4$", $^1/_8$" or 6 mm outer diameter using the holders described below. The multiple adsorber can only be used in $^1/_8$" lines. Please remember to exchange the cartridges in regular intervals (e.g. whenever you change the steel gas cylinder), because exhausted purification cartridges are useless! Regeneration of the adsorber mass is uneconomical or not possible.

Oxygen absorber cartridges Oxisorb®

Oxygen removal from gases is recommended to improve column life of capillaries. Generally, oxygen-free carrier gas should be used for all types of capillary columns, however **removal of oxygen is of vital importance for the phases Carbowax 20M and FFAP**. For this reason always use carrier gas which is absolutely free of oxygen. We recommend that you use an Oxisorb® cartridge which is exchanged in regular intervals.

The combinded gas purification with Oxisorb® / molecular sieve guarantees:

Oxygen:	< 0.1 vpm
Water vapour:	< 0.5 vpm

Oxisorb® consists of a large surface support impregnated with chromium trioxide. Heating and treatment with different gases activates the absorber mass. Oxisorb® removes even minute traces of oxygen from gases by chemisorption. The absorption capacity is pressure-independent and between –190 and +300 °C almost temperature-independent. Oxisorb® can be used for cleaning noble gases, nitrogen, hydrogen, carbon monoxide, carbon dioxide and saturated hydrocarbons.

Oxisorb® can be supplied in aluminium or glass cartridges.

Aluminium cartridges are packed with Oxisorb® and additionally contain molecular sieve for simultaneous removal of water vapour and oxygen. One cartridge is sufficient to remove a maximum of 10 vpm each of oxygen and water vapour from the contents of a steel gas cylinder with max. 10 m³ gas or higher contents of impurities from correspondingly smaller volumes of gas.

The Oxisorb® cartridges made from Duran® glass allow a visual control of the degree of exhaustion of the absorber mass. Oxisorb® shows a characteristic colour change from blue to brown upon absorption of oxygen. Thus the remaining absorption capacity can be checked from the colour of the packing. Cartridges for visible control contain only Oxisorb® absorber mass, but no molecular sieve. Again < 0.1 vpm oxygen can be reached in the purified gas. The capacity of the cartridge is 100 ml O_2. Regeneration of used cartridges is not possible.

Gas purification cartridges Hydrosorb

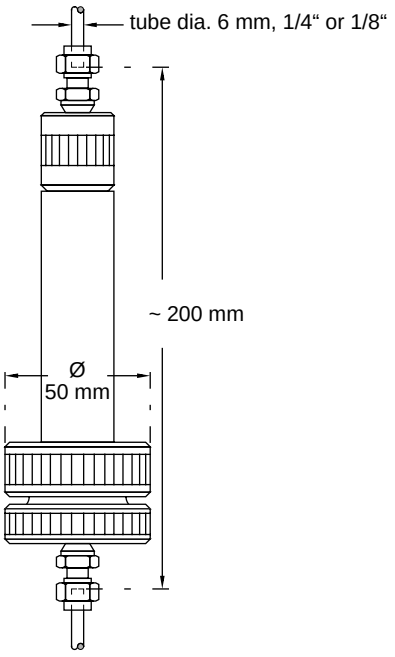
The requirements for minimum moisture content are the same for carrier gases as for the burner gases hydrogen and air.

For removal of moisture we recommend our Hydrosorb cartridges. Hydrosorb is a specially conditioned, highly reactive molecular sieve for adsorption of minute traces of water. The capacity increases with increasing pressure and decreasing temperature. A final purity of 0.5 vpm H_2O can be guaranteed. The capacity is about 10fold larger than the water capacity of the Oxisorb® aluminium cartridges and sufficient to purify 100 m³ gas with a maximum of 10 vpm water vapour.

Gas purification cartridges with activated carbon

Activated carbon cartridges are especially recommended to remove oil traces from in-house pressurised gas supplies. For this purpose we offer aluminium cartridges, which are packed with activated carbon under argon. The guaranteed final purity for hydrocarbons except methane is 0.2 vpm. The capacity is 1 mg for ethane and 180 mg for higher hydrocarbons.

Small absorbers for installation in tubing



Systems for in-line gas purification

Holders for in-line gas purification cartridges

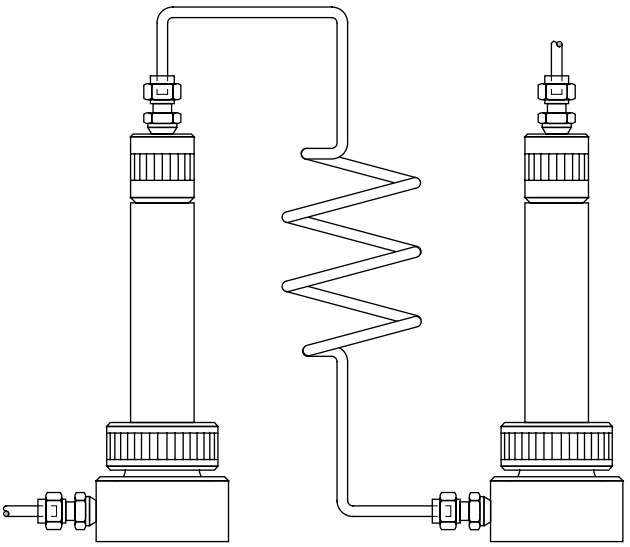
Small absorbers L are designed for installation of gas purification cartridges in low pressure tubing (max. 10 bar), e.g. for the gas supply of gas chromatographs. These absorbers consist of a Hostaform® housing with brass fittings. They are supplied complete with fittings.

Small absorbers L, PN 10 for cartridges with visible packing, too, are made from Hostaform® with brass fittings and suited for installation in low pressure tubings (max. 10 bar). For your safety these absorbers are supplied with a protective plexiglass jacket.

The **multiple absorber II** is designed for the combination of two absorber cartridges in series. Special fittings, which are self-closing at the pure gas side, allow a rapid exchange of cartridges. Materials used are Hostaform® and chrome-plated brass. Useful combinations are e.g. activated carbon and Hydrosorb for pressurised air or Hydrosorb and Oxisorb® for carrier gases.

The separate **protective plexiglass jacket PN 10** is required, if the multiple absorber is to be used with Oxisorb® glass cartridges.

Multiple absorber II



Ordering information

Description	pack of	Cat. No.
Small absorbers L (without cartridges)		
for 6 mm OD tubing	1	**734326**
for 1/4” OD tubing	1	**734327**
for 1/8” OD tubing	1	**734328**
Small absorbers L, PN 10, with protective jacket		
for 6 mm OD tubing	1	**734322**
for 1/4” OD tubing	1	**734323**
for 1/8” OD tubing	1	**734324**
Multiple absorber II		
Multiple absorber for 1/8” OD tubing (without cartridges)	1	**734361**
Protective plexiglas jacket PN 10	1	**734362**
Gas purification cartridges		
Oxisorb® cartridges, glass (with visible packing)	2	**734325**
Oxisorb® cartridges, aluminium, with molecular sieve	2	**734329**
Hydrosorb cartridges	2	**734363**
Activated carbon cartridges	2	**734364**

Ferrules (graphite, Teflon®, Vespel® and Vespel/graphite mixtures)

We supply several types of ferrules for different applications in GC. Graphite ferrules provide the highest temperature stability (up to 450 °C). They are reusable when handled with care. We also offer $^{1}/_{16}$" graphite ferrules specially designed for Carlo Erba gas chromatographs. Vespel ferrules come in three types: pure Vespel, Vespel with 15% graphite and Vespel with 40% graphite. All versions are stable up to 400 °C and reusable. Teflon ferrules can only be used up to 250 °C. They are not reusable and not recommended for temperature programming. However, they show the best chemical inertness of all ferrules.

Ordering information		**Cat. No.** (packing unit 10 ferrules)				
		Graphite	**Vespel**			**Teflon**
Bore (≙ column O D)	max. temp.	450 °C	plain 400 °C	+ 15% graphite 400 °C	+ 40% graphite 400 °C	250 °C
1/16” ferrules						
no bore		**708336**	**706187**	**706167**	**706197**	**706177**
0.4 mm		**708309**	**706270**	**–**	**706246**	**–**
0.5 mm		**708308**	**706271**	**–**	**706247**	**–**
0.8 mm		**708301**	**706272**	**–**	**706248**	**–**
1 mm		**708302**	**–**	**–**	**–**	**–**
1.2 mm		**708303**	**–**	**–**	**–**	**–**
1/16”		**706155**	**706180**	**706160**	**706190**	**706170**
1/16” Carlo Erba ferrules						
0.4 mm		**708338**	**–**	**–**	**–**	**–**
0.5 mm		**708339**	**–**	**–**	**–**	**–**
0.8 mm		**708340**	**–**	**–**	**–**	**–**
1/16” ferrules for HP instruments						
0.4 mm		**708353**	**–**	**–**	**–**	**–**
0.5 mm		**708354**	**–**	**–**	**–**	**–**
0.8 mm		**708355**	**–**	**–**	**–**	**–**
1/8” ferrules						
no bore		**708341**	**706188**	**706168**	**706198**	**706178**
0.4 mm		**708342**	**706266**	**706249**	**706240**	**–**
0.5 mm		**708343**	**706267**	**706250**	**706241**	**–**
0.8 mm		**708333**	**706268**	**706251**	**706242**	**–**
1/16”		**708158**	**706183**	**706163**	**706193**	**706173**
1/8”		**708156**	**706181**	**706161**	**706191**	**706171**
1/4” ferrules						
no bore		**708344**	**706189**	**706169**	**706199**	**706179**
0.4 mm		**708345**	**–**	**–**	**706243**	**–**
0.5 mm		**708346**	**–**	**–**	**706244**	**–**
0.8 mm		**708334**	**–**	**–**	**706245**	**–**
1/16”		**708159**	**706184**	**706164**	**706194**	**706174**
1/8”		**708347**	**706185**	**706165**	**706195**	**706175**
6.0 mm		**708348**	**706186**	**706166**	**706196**	**706176**
1/4”		**706157**	**706182**	**706162**	**706192**	**706172**
6 mm ferrules						
no bore		**708349**	**706252**	**–**	**–**	**–**
6.0 mm		**–**	**–**	**–**	**–**	**706259**

If you are in doubt about the correct size / Cat. No. please send us an old, used ferrule for the right selection.

Septa

Ordering information	Cat. No. for outer diameter of						
(packing unit 50 septa)	6 mm N 6	9 mm N 9	10 mm N 10	11 mm N 11	12 mm N 12	13 mm N 13	17 mm N 17
Standard septa beige							
Standard septa are 4 mm thick, made from beige silicone rubber, and have a hardness of 60 shore.							
Standard septa (ST)	**702608**	**702609**	**702610**	**702611**	**702612**	**702613**	**–**
High temperature septa red							
High temperature septa are 3 mm thick and made from red, especially pretreated, non-bleeding silicone rubber with a hardness of 60 shore. These septa can be used up to about **320 °C**. When used at considerably higher temperatures – and working without septum purge – interfering peaks can occur due to thermal decomposition of the material.							
High temperature septa (HT)	**–**	**702619**	**702620**	**702621**	**702622**	**702623**	**702632**
Silicone septa soft, transparent							
Silicone septa, soft are 3 mm thick, made from transparent silicone rubber, and have a hardness of only 45 shore.							
Silicone septa, soft	**702601**	**702602**	**702603**	**702604**	**702605**	**702606**	**–**
Silicone septa PTFE, white/grey							
Silicone septa PTFE are 3 mm thick and consist of white silicone rubber with one side coated with PTFE. The thermal stability is limited to about **200 °C** due to the use of PTFE.							
Silicone septa PTFE	**702633**	**702624**	**702625**	**702626**	**702627**	**702628**	**–**
Butyl rubber septa PTFE, red							
Butyl rubber septa PTFE are 3 mm thick and consist of red butyl rubber with one side coated with PTFE. The thermal stability is limited to about **200 °C** due to the use of PTFE.							
Butyl rubber septa PTFE	**–**	**702614**	**702615**	**702616**	**702617**	**702618**	**–**
Sandwich septa green/white/green							
Sandwich septa green/white/green are 4 mm thick and consist of three layers of silicone rubber. They feature a good thermal stability (near **300 °C**) and very good sealing properties even after frequent piercing.							
Sandwich septa green	**–**	**702634**	**702635**	**702636**	**702637**	**702638**	**–**
Septum remover							
Tool for removing septa which have become baked into the injection port of the gas chromatograph	**706141**