

[54] PERFLUOROALKYL ALCOHOLS

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Related U.S. Application Data

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abandoned, which is a continuation of Ser. No.
281,084, Aug. 16, 1972, abandoned.

[52] U.S. Cl. **260/481 R**; 204/158 HE;
260/78.3 R; 260/78.3 UA; 260/455 R;
260/485 F; 260/561 S

[51] Int. Cl.² **C07C 149/20**

[58] Field of Search 260/481 R

[56] **References Cited**

UNITED STATES PATENTS

3,172,910 3/1965 Brace 260/481 R
3,706,789 12/1972 Bonner 260/481 R

Primary Examiner—Vivian Garner
Attorney, Agent, or Firm—Joseph G. Kolodny; Edward
McC. Roberts; Prabodh I. Almula

[57] **ABSTRACT**

Perfluoroalkyl group containing alcohols are disclosed with utility as intermediates for the synthesis of oil and water repellent fluorochemicals. The alcohols are obtained by the addition of mercaptoalcohols to perfluoroalkyl group containing esters of fumaric, maleic, citraconic, mesaconic, itaconic, methylene malonic or aconitic acids.

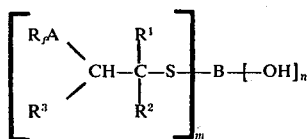
6 Claims, No Drawings

PERFLUOROALKYL ALCOHOLS

This application is a continuation of application Ser. No. 493,362, filed July 31, 1974, now abandoned, which is a continuation of application Ser. No. 281,084, filed Aug. 16, 1972, now abandoned.

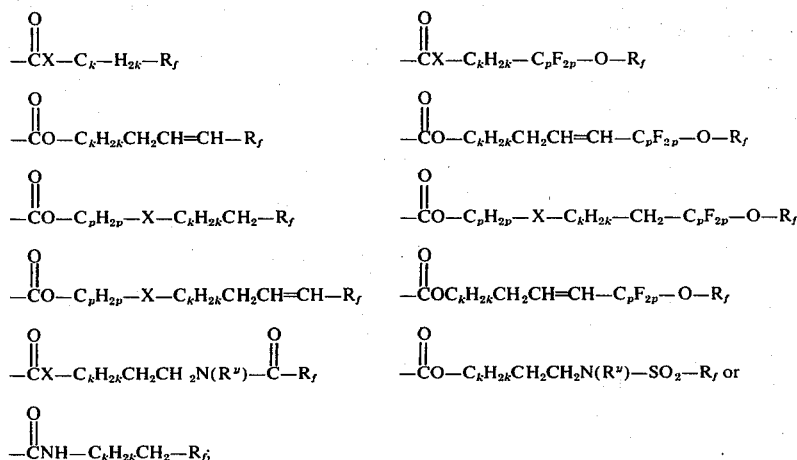
This invention is directed to novel perfluoroalkyl group containing alcohols. These alcohols are useful in the direct preparation of compounds which possess low free surface energies with oil and water repellancy.

The novel perfluoroalkyl group containing alcohols have the following general structure:



wherein R^1 , R^2 , R^3 are hydrogen, methyl, $R_f A$ or $R_f ACH_2$ with the requirement at least one of R^1 , R^2 , R^3 represent $R_f A$ or $R_f ACH_2$;

R_f is a perfluoroalkyl group of 1 to 18 carbon atoms; AR_f is



X is oxygen or sulfur;

k is zero to 10;

p is 2 to 12;

R^y is hydrogen or alkyl of 1 to 4 carbon atoms;

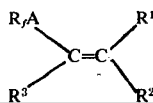
m is 1 to 6;

n is 1 to 6;

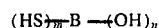
and B is an inert linkage group.

In the above definition B is required to be an inert linkage group. Inert in the present context is employed in its normal definition so that B remains as a discrete unreacted group in the starting and final compound in the reaction procedure to produce the novel perfluoroalkyl (R_f) alcohol of formula I.

A suitable method for forming the novel R_f -alcohols of formula I, involves a base or free radical catalyzed addition reaction of α , β -unsaturated di- or triesters of the formula:



with mono- or polymercaptoalcohols of formula:



III

In formulas II and III the substituents have been defined previously in relationship to formula I.

The type of esters of formula II which may be employed are derived from fumaric, maleic, citraconic, mesaconic, itaconic, methylene malonic and aconitic acids. The formula II esters are defined to be:

Type Ester	R^1	R^2	R^3
15 Fumarate	-H	- AR_f	-H
Maleate	- AR_f	-H	-H
Mesaconate	-H	- AR_f	- CH_3
Citraconate	- AR_f	-H	- CH_3
Itaconate	-H	-H	- CH_2AR_f
Methylene malonate	-H	-H	- AR_f
cis-Aconitate	-H	- AR_f	- CH_2AR_f
20 trans-Aconitate	- AR_f	-H	- CH_2AR_f

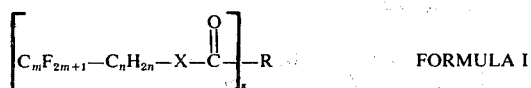
The preparation of perfluoroalkyl containing esters disclosed herein and especially useful in preparing the novel compounds or adducts of the present invention are described in the following copending applications

assigned to the assignee of the present invention: Ser. No. 720,370 filed Apr. 10, 1968 now abandoned, in the names of **Eduard K. Kleiner and Martin Knell**; Ser. No. 732,040 filed May 27, 1968, in the names of Eduard K. Kleiner, Martin Knell and Pier Luigi Pacini now U.S. Pat. 3,658,857; Ser. No. 812,439, filed Apr. 1, 1969, in the name of Eduard K. Kleiner now U.S. Pat. No. 3,636,085; Ser. No. 820,647, filed Apr. 30, 1969, in the name of Eduard K. Kleiner now U.S. Pat. No. 3,658,843; and Ser. No. 833,706, filed June 16, 1969, in the names of Eduard K. Kleiner and Pier Luigi Pacini now U.S. Pat. No. 3,645,985; Ser. No. 199,793 filed on Nov. 11, 1971, in the names of Eduard K. Kleiner and Martin Knell which, as CIP application Ser. No. 299,487 issued as U.S. Pat. No. 3,763,116; and Ser. No. 199,794 filed on Nov. 11, 1971, in the names of Eduard K. Kleiner and Martin Knell now U.S. Pat. No. 3,794,623.

The disclosure of these copending applications for the preparation of the starting esters and the related subject matter is incorporated by reference herein.

Specifically disclosed from copending application

pounds of the present invention are of the following formula:



wherein

m is an integer of 3 to 18, preferably 6 to 12, and most preferably 7 to 10;

n is an integer of 0 to 10, preferably 1 or 2;

X is oxygen or sulfur, preferably oxygen;

R is an ethylenically unsaturated hydrocarbon radical derived from fumaric, maleic, citraconic, mesaconic, itaconic, aconitic, or methylene malonic acid, preferably fumaric; and

s is an integer of 2 or 3 and is equal to the number of carboxyl groups of the acid from which R is derived, preferably being 2 to correspond to fumaric acid.

The ester monomers of this invention can generally be prepared by well known esterification reactions between: acids and perfluorinated alcohols, alkyl esters and perfluorinated alcohols, acid chloride and perfluorinated alcohols, acid salts and perfluorinated halogenides, and acid chlorides and perfluorinated alkoxides. Of course, it is understood that the corresponding mercaptans may be used in lieu of the alcohols.

Generally the reaction of acid chlorides with the perfluorinated alcohol or mercaptan is preferred since the acid chlorides are easily available and the esterification proceeds readily. An exception, of course, is the case of maleyl chloride and chloromaleyl chloride which do not exist.

The esterifications are carried out in the absence of a base.

The esters of methylene malonic acid generally require a two step synthesis. The intermediate malonesters are made using one of the above esterification techniques and then the methylene malonic ester is formed for example by condensation of the malonester with formaldehyde. See E. Haworth and W. H. Perkin, J. Chem. Soc. 73, 339 - 345 (1898).

Representative preparations of examples of alcohols and mercaptans useful as starting materials in the practice of this invention are disclosed in: U.S. Pat. Nos. 2,666,797; 3,283,012; 3,171,861; 3,285,975; 2,884,991; and French Pat. No. 1,221,415.

Other alcohols which may form esters with the above acids or derivatives thereof are those such as $\text{HO}-(\text{CH}_2)_n\text{CH}=\text{CH}-\text{R}_f$, disclosed in U.S. Pat. No. 3,285,975; $\text{HO}-\text{CH}_2\text{CH}_2\text{CF}_2-\text{O}-\text{R}_f$ disclosed in U.S. Pat. No. 2,826,564; and $\text{HO}-(\text{CH}_2)_n\text{O}(\text{CH}_2)_m\text{R}_f$ and $\text{HO}(\text{CH}_2)_n\text{S}(\text{CH}_2)_m\text{R}_f$ which are obtained by reduction of the corresponding $\text{CH}_3\text{OCO}(\text{CH}_2)_{n-6}\text{O}(\text{CH}_2)_m\text{R}_f$ esters described in U.S. Pat. No. 3,172,910.

The following examples describing certain representative embodiments of this invention will serve to further illustrate the nature of this invention. It is to be understood that the examples are merely illustrative and intended to enable those skilled in the art to practice the invention in all of the embodiments flowing therefrom and do not in anyway limit the scope of the invention defined in the claims. Unless otherwise specified, the relationship of parts by weight to parts by volume is that of grams to cubic centimeters, and tem-

EXAMPLE 1

a. BIS(1,1-DIHYDROPERFLUOROBUTYL) FUMARATE

A mixture of 15.3 parts of fumaryl chloride and 40 parts of 1,1-dihydroperfluorobutyl alcohol is heated with stirring at $90^\circ - 5^\circ$ until no further hydrogen chloride is liberated. After a total reaction time of 120 hours, the product is isolated by distillation. In this manner, 18.0 parts of bis (1,1-dihydroperfluorobutyl) fumarate boiling at $106^\circ - 110^\circ$ at 5 mm pressure and melting at $45^\circ - 45.5^\circ$ is obtained.

Analysis for $\text{C}_{12}\text{H}_6\text{F}_{14}\text{O}_4$

Calculated: C, 30.01; H, 1.25

Found: C, 29.92; H, 1.28

b. MIXED BIS (1,1,2,2-TETRAHYDROPERFLUOROALKYL) FUMARATE

The procedure of a) is repeated, using 120 parts of a mixture containing 42.0% $\text{C}_6\text{F}_{13}\text{CH}_2\text{CH}_2\text{OH}$, 35.8% $\text{C}_6\text{F}_{17}\text{CH}_2\text{CH}_2\text{OH}$, 21.0% $\text{C}_{10}\text{F}_{21}\text{CH}_2\text{CH}_2\text{OH}$, 1.0% $\text{C}_{12}\text{F}_{25}\text{CH}_2\text{CH}_2\text{OH}$ and 0.2% $\text{C}_{14}\text{F}_{29}\text{CH}_2\text{CH}_2\text{OH}$ instead of the 1,1-dihydroperfluorobutyl alcohol. After 5 hours of heating at $95^\circ - 100^\circ$, VPC indicated that the reaction is essentially complete. Distillation of the product gives 93 parts of mixed fumarate diesters boiling at $145^\circ - 207^\circ$ at 0.020 mm. pressure and having the following microanalysis: C, 28.69; H, 1.05; F, 63.54.

EXAMPLE 2

BIS (1,1-DIHYDROPERFLUOROOCYL) FUMARATE

A mixture of 57.5 parts of fumaryl chloride and 300 parts of 1,1-dihydroperfluorooctyl alcohol is heated with stirring at $80^\circ - 5^\circ$ until no further hydrogen chloride is liberated. The total reaction time is 141 hours. After two recrystallizations from isopropyl alcohol, 169.2 parts of bis (1,1-dihydroperfluorooctyl) fumarate, melting at $74^\circ - 7^\circ$ is obtained. Further purification yields material melting at $80^\circ - 82.5^\circ$ which is identified by microanalysis, IR and NMR spectroscopy.

EXAMPLE 3

BIS (1,1,2,2-TETRAHYDROPERFLUOROOCYL) FUMARATE

A mixture of 9.46 parts of 1,1,2,2-tetrahydroperfluorooctanol (prepared as described in U.S. Pat. No. 3,283,012), 1.87 parts of dimethyl fumarate, 0.55 parts of concentrated sulfuric acid, and 100 parts by volume of toluene is placed in a pear shaped flask fitted with a short Vigreux distilling column. Toluene is distilled until no further methanol is detected in the distillate. Additional toluene is added as needed to the reaction flask during the reaction. On cooling, the product crystallizes from the toluene, and is filtered, and washed with water until the washings are neutral. After two recrystallizations from toluene and one from hexane, 1.4 parts of bis (1,1,2,2-tetrahydroperfluorooctyl) fumarate melting at $56^\circ - 72^\circ$ is obtained. Although the compound does not melt sharp, VPC, IR, NMR and microanalysis indicate that it is pure and has the desired structure.

Analysis for $\text{C}_{20}\text{H}_{10}\text{F}_{26}\text{O}_4$ -

Calculated: C, 29.72; H, 1.25; F, 61.12

Found: C, 29.53; H, 1.40; F, 60.76

EXAMPLE 4

BIS (1,1,2,2-TETRAHYDROPERFLUOROOCYL) FUMARATE

5

A solution of 2.68 parts of fumaryl chloride and 14.5 parts of 1,1,2,2-tetrahydroperfluorononyl alcohol (prepared as described in U.S. Pat. No. 3,283,021) in 45.0 parts by volume of acetonitrile is allowed to stand overnight at room temperature. The reaction mixture is then heated with stirring at 80° for 3 hours. After cooling in an ice bath, the product is separated by filtration, washed with cold acetonitrile and air dried. 8.9 Parts of crude product are obtained. After two recrystallizations from isopropyl alcohol the melting point is 81° - 83.5° and the IR, NMR and microanalysis conform with the expected structure.

Analysis for $C_{22}H_{10}F_{30}O_4$ -

Calculated: C, 29.09, H, 1.11; F, 62.75

Found: C, 28.91; H, 1.24; F, 62.94

EXAMPLE 5

BIS (HEXAFLUOROISOPROPYL) FUMARATE

To a slurry of 2.4 parts of sodium hydride, as a 50% dispersion in mineral oil, in 45 parts of toluene, cooled to 15° is added with stirring 25.2 parts of hexafluoroisopropyl alcohol keeping the temperature below 30°. A solution of 7.7 parts of fumaryl chloride in 10 parts of toluene is then added dropwise with stirring keeping the temperature below 30°. The reaction mixture is stirred for 1½ hours at room temperature and heated to boiling. After cooling to room temperature, 100 parts of water is added. The toluene layer is separated, washed two times with 50 parts of water, dried over anhydrous magnesium sulfate, and distilled through an 18 inch spinning band column.

The product fraction, which boils at 66° at 4 mm pressure and melts at 53° - 5°, amounts to 13.6 parts and is identified by IR, NMR and microanalysis as bis (hexafluoroisopropyl) fumarate.

Analysis for $C_{10}H_4F_{12}O_4$ -

Calculated: C, 28.86; H, 0.97;

Found: C, 28.59; H, 0.96;

EXAMPLE 6

BIS (1,1,2,2-TETRAHYDROPERFLUORONONYLTHIOL) FUMARATE

a. 1,1,2,2-Tetrahydroperfluorononyl mercaptan

A mixture of 30.0 parts of 1,1,2,2-tetrahydroperfluorononyl iodide, 5.2 parts of thiourea and 150 parts by volume of ethanol is refluxed for 6 hours after which 12 parts of 25% sodium hydroxide solution is added dropwise with stirring and the resulting mixture refluxed for 1 hour. After cooling to room temperature, the reaction mixture is acidified with dilute sulfuric acid, 150 parts of water is added and the yellow organic layer is separated and dried over anhydrous magnesium sulfate. The product is then distilled through a short Vigreux column. In this manner, 13.7 parts of product boiling at 72° - 4° at 17 mm pressure is obtained. The product is identified as 1,1,2,2-tetrahydroperfluorononyl mercaptan by IR, NMR and microanalysis.

Analysis for $C_9H_5F_{15}S$ -

Calculated: C, 25.13; H, 1.17;

Found: C, 24.68; H, 1.29;

b. Bis (1,1,2,2-Tetrahydroperfluorononylthio) Fumarate

A mixture of 3.7 parts of 1,1,2,2-tetrahydroperfluorononyl mercaptan and 0.65 parts of fumaryl chloride is placed in a 10 ml. flask and heated at 80°C for 18 hours while passing a slow stream of nitrogen through the reaction flask. The resulting solid is dissolved in 7.0

6

80° for an additional 3 hours. An additional 10 parts by volume of trifluorotoluene is added, the hot solution filtered, and the product allowed to crystallize slowly. After cooling, filtering, washing with cold trifluorotoluene, and drying, 0.85 parts of bis (1,1,2,2-tetrahydroperfluorononylthio) fumarate melting at 137.5° - 139° is obtained.

Analysis for $C_{22}H_{10}F_{30}S_2O_2$ -

Calculated: C, 28.03; H, 1.07;

Found: C, 27.98; H, 1.12;

EXAMPLE 7

BIS (1,1,2,2-TETRAHYDROPERFLUOROHEXYL) FUMARATE

A mixture of 5.05 parts of fumaryl chloride and 17.0 parts of 1,1,2,2-tetrahydroperfluorohexanol, prepared as described in U.S. Pat. No. 3,283,012, is heated with stirring at 80° - 90°C under a nitrogen atmosphere until the liberation of hydrogen chloride ceases and VPC indicates that the starting materials are no longer present. The crude reaction mixture is recrystallized from hot hexane yielding 12.3 parts of a colorless semi-solid. Further purification is carried out by dissolving in 1,1,2-trichloro-trifluoroethane, passing through a neutral aluminum oxide column and evaporating the solvent. The product is still a semi-solid at room temperature, but IR, NMR and VPC indicates that it is pure.

Analysis for $C_{16}H_{10}F_{18}O_4$ -

Calculated: C, 31.60; H, 1.66; F, 56.24

Found: C, 31.57; H, 1.65; F, 56.02

EXAMPLE 8

BIS (1.1-DIHYDROPERFLUOROOCYL) ITACONATE

A mixture of 12.52 parts of itaconyl chloride and 72.52 parts of 1.1-dihydroperfluorooctyl alcohol and 50 parts of benzotrifluoride is refluxed for 72 hours. The benzotrifluoride is then removed on a rotary evaporator and the residue is distilled twice under high vacuum. 34.6 Parts (yield 47.4%) of pure bis (1.1-dihydroperfluorooctyl) itaconate, boiling point 120° - 131°C at 0.1 mm Hg. pressure is obtained. Identification is made by microanalysis, IR and NMR.

Analysis for $C_{21}H_6O_4F_{30}$ -

Calculated: C, 28.20; H, 0.90; F, 63.74

Found: C, 24.41; H, 1.18; F, 64.00

EXAMPLE 9

BIS (1.1-DIHYDROPERFLUOROOCYL) MALONATE

A mixture of 14.8 parts of malonyl chloride, 84.02 parts of 1.1-dihydroperfluorooctyl alcohol and 200 parts of dry toluene was refluxed for 45 hours. The toluene was removed in a rotary evaporator and the residue was distilled under high vacuum. 37.54 parts (yield 86.5%) of pure bis (1.1-dihydroperfluorooctyl) malonate, boiling point 122° - 125° at 0.2 mm Hg. pressure is obtained. It is soluble at room temperature and melts at 35.5° - 36.5°C. Purity test and identification is made by VPC, IR and NMR.

Analysis for $C_{19}H_6F_{30}O_4$ -

Calculated: C, 26.28; H, 0.70; F, 65.65

Found: C, 26.14; H, 0.78; F, 65.32

EXAMPLE 10

BIS (1-METHYL-1,2,2-TRIHYDROPERFLUORONONYL) FUMARATE

To a solution of 18.7 parts of silver nitrate in 60 parts of acetonitrile is added dropwise with stirring 53.8 parts of 1-methyl-1,2,2-trihydroperfluorononyl iodide. The addition takes 50 minutes and there is no visible exotherm, but after a short time, a yellow solid begins to separate. Stirring is continued at room temperature for 72 hours, after which time the solid is removed by filtration. Water (100 parts) is added to the filtrate, the lower layer separated, dried over anhydrous magnesium sulfate, and distilled through an 18 inch spinning band column. In this manner, 32.5 parts of the desired 1-methyl-1,2,2-trihydroperfluorononyl nitrate, boiling at 77° - 70° at 4 mm pressure is obtained.

Analysis for $C_{10}H_7F_{15}NO_3$ -

Calculated: C, 25.38; H, 1.28; N, 3.03; F, 60.25

Found: C, 25.19; H, 1.32; N, 3.34; F, 60.07

b. 1-Methyl-1,2,2-Trihydroperfluorononyl

To a slurry of 44.7 parts of 60% sodium sulfhydrylate in 100 parts by volume of ethanol is added dropwise in 1 hour 28.4 g of 1-methyl-1,2,2-trihydroperfluorononyl nitrate. The reaction is exothermic to 35°C. After stirring at room temperature for 1 hour, 250 parts of water is added, the bottom layer separated, washed again with water, and dried over anhydrous magnesium sulfate. Distillation through a short Vigreux column gives 20.5 parts of 1-methyl-1,2,2-trihydroperfluorononyl, boiling at 82°C at 100 mm. pressure.

Analysis for $C_{10}H_7F_{15}O$ -

Calculated: C, 28.05; H, 1.69; F, 66.56

Found: C, 28.15; H, 1.52; F, 66.35

c. Bis (1-Methyl-1,2,2-Trihydroperfluorononyl) Fumarate

A mixture of 15.0 parts of 1-methyl-1,2,2-trihydroperfluorononyl, 2.68 parts of fumaryl chloride and 15.0 parts of triglyme is heated with stirring at 95° - 100°C for 32 hours and at 125°C for 35 hours until the evolution of hydrogen chloride ceases. After cooling to room temperature, water is added and the product

extracted into ether. The ether solution is washed with 10% sodium bicarbonate, water and then dried over anhydrous magnesium sulfate. After evaporation of solvent, the product is distilled through a micro distillation apparatus. Six and one half parts of the fraction boiling at 140° - 160°C at 0.050 mm pressure is ob-

tained. This fraction analysed only 68% product by VPC, so it was recrystallized two times from hot ethanol. In this manner, 3.0 parts of bis (1-methyl-1,2,2-trihydroperfluorononyl) fumarate, which is 99+% pure by VPC, is obtained.

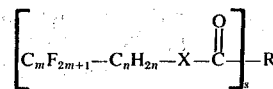
Analysis for $C_{24}H_{14}F_{30}O_4$ -

Calculated: C, 30.78; H, 1.51; F, 60.87

Found: C, 30.44; H, 1.81; F, 60.92

EXAMPLE 11

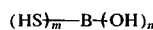
Using procedures of the foregoing examples employing stoichiometrically equivalent amounts of corre-



FORMULA I

	$C_m F_{2m+1}$	$C_n H_{2n}$	X	S	R
11a	$(CF_3)_2CF$	CH_2CH_2	O	2	from citraconic acid
11b	$CF_3(CF_2)_6$	$(CH_2)_{10}$	O	2	from fumaric acid
11c	$CF_3(CF_2)_6$	CH_2	S	3	from aconitic acid
11d	$CF_3(CF_2)_{17}$	CH_2CH_2	O	2	from fumaric acid
11e	$(CF_3)_2(CF_2)_4$	$(CH_2)_4$	S	2	from mesaconic acid
11f	$CF_3(CF_2)_8$	CH_2	O	2	from maleic acid

Mercaptoalcohols of type III



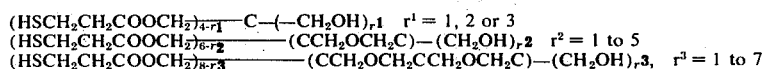
may contain from 1 to 6 hydroxy and mercapto groups and are either commercially available or can be easily prepared. Among the commercially available mercapto alcohols, the following are of special importance:

2-mercaptoethanol	$HSCH_2CH_2OH$
2,3-dimercapto-1-propanol	$HSCH_2CH(SH)CH_2OH$
3-mercapto-1,2-propanediol	$HSCH_2CH(OH)CH_2OH$
1,3-dimercapto-2-propanol	$HSCH_2CH(OH)CH_2SH$
1,4-dimercapto-2,3-butanediol	$HSCH_2CH(OH)CH(OH)CH_2SH$

Of importance also are mercaptoalcohols derived from polyols and thioglycolic acid or 3 mercaptopropionic acid, such as

hydroxyethyl thioglycolate	$HSCH_2COOCH_2CH_2OH$
hydroxyethyl mercaptopropionate	$HSCH_2CH_2COOCH_2CH_2OH$
hydroxyalkyl thioglycolate	$HSCH_2COOC_nH_{2n}OH_n$
hydroxyalkyl mercaptopropionate	$HSCH_2CH_2COOC_nH_{2n}OH_n$

Additionally suitable are thioglycolates and mercaptopropionates derived from mono, di- and trientaerythritol and thioglycolic or mercaptopropionic acid:

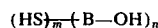


Of similar interest are mercaptoalcohols which are obtained by partially esterifying trimethylolethane, trimethylolpropane, polyhydroxy compounds such as sorbitol, with thioglycolic or mercaptopropionic acid.

Also suitable are mercapto alcohols known as Dion Polymercaptan Resins:

DPM 3-800LC	$R[O(C_3H_6O)_N CH_2CH(CH)CH_2SH]_3$	$N = 1-2$
DPM 1002	$R[O(C_3H_6O)_N CH_2CH(OH)CH_2SH]_{N-2}$	$N^1 = 20-25$
DPM 5-1300	$R[O(C_3H_6O)_N CH_2CH(OH)CH_2SH]_5$	$N^2 = 2-3$

Referring to the starting mono or poly-mercaptoalcohols of formula III,



B serves as an inert linkage only and connects the mercapto and the hydroxy groups. The sole criticality for B is that it remains intact throughout the reaction.

The addition of mercaptoalcohols of type III to α , β -unsaturated di- and triesters of type II is accomplished either by a base or free radical catalysis.

The base catalyzed addition reaction of mercaptans

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