ECOTOXICITY PROFILE OF 1H,1H,2H,2H-PERFLUORODECANOL

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ABSTRACT

1H,1H,2H,2H-Perfluorodecanol, CAS No. 678-39-7 (8-2 Telomer alcohol) is an important speciality intermediate. More than 80% of the produced amount of telomer alcohols are used for the preparation of high molecular weight polymers containing perfluorinated alcohols. These so-called Telomer-based Polymeric Products (TBPPs) are used e.g. for the coating of textiles and carpets to achieve resistance against water, oil and stains. The producers of telomer alcohols are currently investigating in detail the environmental fate and effects properties of telomer alcohols and especially of 1H,1H,2H,2H-Perfluorodecanol within the Telomer Research Program (TRP).

Telomer alcohols have in comparison to their hydrogenated counterparts some exceptional physico-chemical properties which are related to the fluorine atoms. But these properties (e.g. very low water solubility, high volatility) are considerable challenges for ecotoxicity testing. Within TRP testing strategies for this substance which is difficult to test were developed based on latest methodology.
The poster presents basic physico-chemical and fate properties of 1H,1H,2H,2H-Perfluorodecanol, applied design for acute and chronic aquatic tests as well as terrestrial / sediment tests and the results for the different species tested.

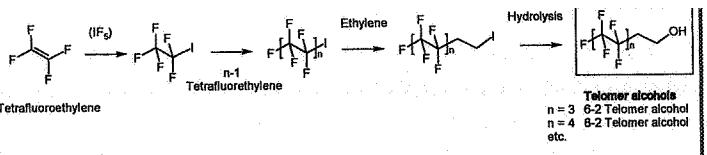
INTRODUCTION

1H,1H,2H,2H-Perfluorodecanol, CAS No. 678-39-7 (8-2 Telomer alcohol) is an important speciality intermediate. More than 80% of the produced amount of telomer alcohols are used for the preparation of high molecular weight polymers containing perfluorinated alcohols. These so-called Telomer-based Polymeric Products (TBPPs) are used e.g. for the coating of textiles and carpets to achieve resistance against water, oil and stains.

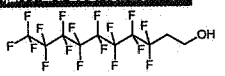
Ashai Glass, Clariant, Daikin and DuPont as major producer of these chemicals have investigated basic physico-chemical, fate and ecotoxicity properties of 8-2 Telomer alcohol which is the homologue with the highest content (50-60% w/w) in commercial Telomer alcohols. Most of the data presented here were established within the Telomer Research Program (TRP) funded by the companies mentioned above.
These data will be used in a future Environmental Risk Assessment.

8-2 Telomer alcohol differs in some physico-chemical and fate properties from the unfluorinated 1-Decanol (s. table on right hand side on Physico-chemical and Fate Properties of 1-Decanol & 8-2 Telomer alcohol). Whereas these properties are responsible for the desired effect (hydrophobicity and oleophobicity) in articles (e.g textiles and carpets) they are posing a considerable challenge for testing the ecotoxicity especially in the aquatic compartment.
It is also quite common to estimate physico-chemical and fate properties using property-property estimation algorithms / programs. It is important to note that these programs currently fail to deliver reliable estimates for telomer alcohols in most cases and measurements need to be carried out instead (s. table on right hand side on Physico-chemical and Fate Properties of 1-Decanol & 8-2 Telomer Alcohol) in order to avoid that wrong conclusions were drawn.

SYNTHESIS OF PERFLUOROALKANOLS (TELOMER ALCOHOLS)



STRUCTURE OF 1H,1H,2H,2H-PERFLUORODECANOL



1H, 1H,2H,2H-Perfluorodecanol

8-2 Telomer alcohol

ECOTOXICITY DATA OF 8-2 TELOMER ALCOHOL

TEST SETTINGS & TOXICITY RESULTS

Aquatic test settings

Test solution

The **acute** aquatic ecotoxicity tests were all carried out as limit tests with a saturated solution of 8-2 Telomer alcohol. The test solution was prepared by slow stirring of 8-2 Telomer alcohol in water to achieve a saturated solution. The solution was centrifuged and transfered into test vessel which were precoated with the test substance. Analytical monitoring was carried out at the beginning and at the end of the exposure period. For the **chronic** daphnia test the stock solution was prepared with methanol as solvent.

Aeration

In tests where aeration is required (e.g. acute fish test and chronic daphnia test) loss of 8-2 alcohol is very likely and was observed.

Static, semistatic and flow through conditions

Static conditions were successfully applied for the Acute Daphnia test, whereas for fish the substance concentration at the end of the test was below LOD. But despite considerable efforts it was not possible to establish a reliable and still cost-effective ***flow through system*** which would ensure constant concentration during the whole exposure period and which would be the test system of choice for acute fish and chronic daphnia testing. For the chronic daphnia test a semi-static test design was used and substance loss due to volatility was carefully monitored in 3 testing intervalls (see graph below).

Sediment and Terrestrial test settings

Spiking procedure

The test substance was dissolved in methanol and mixed thoroughly with quartz sand. The solvent was evaporated in a hood and the substance concentration verified by reextracting from the sand (96% recovery). The quartz sand was then mixed with the aritifical sediment and the soil

Volatilisation from Sediment and Soil

Sediment and soil samples were taken at the beginning and at the end of the exposure and an analysis carried out to determine the total fluorine content (Total Organic Fluorine TOF) in order to ensure that there was no considerable loss by volatilisation during the test period (3d, 14d or 21d).

Analytical methods

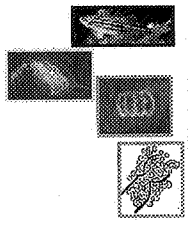
GC/MS for 8-2 Telomer alcohol (Aquatic compartment)

GC: CP-3800 Varion Fused Silica CP-SIL8 Capillary column
MS: Saturn 2000, Varian; m/z = 463 and 395
Calibration curve: r^2 = 0,9984; LOD = 9 µg/L, LOQ = 27 µg/L

Wickbold Torch Method for determination of Total Organic Fluorine (TOF) (Sediment and Soil compartment)

Combustion of the analyte in an oxyhydrogen flame (ca. 1200 °C) (details see [9]).

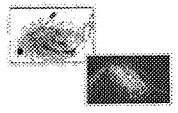
AQUATIC TOXICITY RESULTS



ACUTE	Species	Guideline	Exposure	Conc. at start (mg/L)	Conc. at end (mg/L)	Aeration	Result
Fish	Danio rerio	OECD 203	96 h	0,18	< LOD [a]	yes	no effect under test conditions
Invertebrate	Daphnia magna	OECD 202	48 h	0,18	0,18	no	no effect under test conditions
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

GC/MS LOQ 0,027 mg/L
LOD 0,009 mg/L

(a) Conc. dropped below LOD due to aeration most likely
flow through test was not possible due to volatility losses
(b) Conc. dropped below LOD due to sorption onto algal biomass most likely

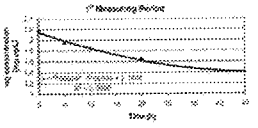


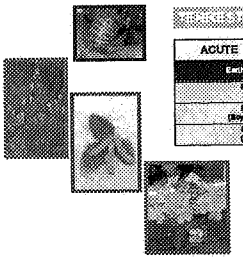
CHRONIC	Species	Guideline	Exposure	Conc. at day 0 (mg/L)	Conc. at day 3 (mg/L)	Aeration	Result
Invertebrate	Daphnia magna	OECD 211	21d semi-static (3 renewals)	0,18 [b]	0,012 [a,b]	yes	no effect under test conditions on all endpoints of the test. NOEC 0,09 µg/L (geometric mean of exposures)

GC/MS LOQ 0,027 mg/L
LOD 0,009 mg/L

(a) Conc. dropped due to aeration
flow through test was not possible due to volatility losses
(b) Conc. given for one of the 4 intervals which is typically for all

Test with Daphnia magna according to OECD 211 Semi-static 21d Daphnia Reproduction Test using 3x7-d Renewal Schedule

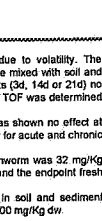




TERRESTRIAL TOXICITY RESULTS

ACUTE	Species	Guideline	Exposure	Nominal test conc. (mg/Kg soil dw.)	Result (most sensitive endpoint)
Earthworm	Eisenia foetida	OECD 207	14d	10-32-100-320-1000	NOEC 32 mg/Kg
Plants (Oat)	Avena sativa	OECD 208 (Seedling & Emergence Test)	21d	10-32-100-320-1000	All endpoints an effect at highest test conc. NOEC, EbIR, EC50 > 1000 mg/Kg
Plants (Soybean)	Glycine max		21d	10-32-100-320-1000	Fresh weight NOEC 320 mg/Kg, EC50 EbIR 60 > 1000 mg/Kg
Plants (Rape)	Brassica napus		21d	6,25-12,5-25-50-100	Fresh weight NOEC 25 mg/Kg, EC50 > 74 mg/Kg

SEDIMENT TOXICITY RESULTS



CHRONIC	Species	Guideline	Exposure	Nominal test conc. (mg/Kg sediment dw.) [a]	Result
Nematode	Caenorhabditis elegans	Transfer of 10 adults	3d	1000	No effects on adult growth and reproduction at 1000 mg/Kg dw. sediment

(a) Nematodes are the most abundant organisms in sediment and soil

CONCLUSIONS

The physico-chemical and fate properties of 1H,1H,2H,2H-Perfluorodecanol, CAS No. 678-39-7 (8-2 Telomer alcohol) require special test settings and careful analytical monotoring in order to derive reliable ecotoxicity data. The low water solubility and high volatility makes testing in the aquatic compartment difficult especially when aeration is requested for the test e.g. fish (acute test) and daphnia (chronic test). Testing is also difficult for algae as during the test the biomass increases considerably. Due to the sorptive properties of 8-2 Telomer alcohol the concentration in water drops in the algae test by sorption.
Testing 8-2 Telomer alcohol in the solid compartments (sediment and soil) was found less difficult as coating of quartz sand with a methanolic solution was effective observing almost no loss due to volatility. The quartz sand coated with 8-2 Telomer alcohol can be mixed with soil and sediment and during the exposure period of the tests (3d, 14d or 21d) no loss of Total Organic Fluorine (TOF) was observed. TOF was determined with the Wickbold Torch Method [9].
In the Aquatic compartment 8-2 Telomer alcohol has shown no effect at the concentrations which could be achieved in water for acute and chronic tests.
In the Terrestrial compartments the NOEC for earthworm was 32 mg/Kg dw. and for the most sensitive plants species rape and the endpoint fresh weight the NOEC was 25 mg/Kg dw.
The nematode Caenorhabditis elegans abundant in soil and sediment showed no effects on growth and reproduction at 1000 mg/Kg dw.

PHYSICO-CHEMICAL AND FATE PROPERTIES OF 1-DECANOL & 8-2 TELOMER ALCOHOL

The physico-chemical and fate properties of C8-2 Telomer alcohol differ in various aspects considerably from the properties of the unfluorinated 1-Decanol (e.g. density, water solubility, volatility).

It is important to note that property-property estimation methods deliver reasonable results for 1-Decanol but are in most cases not suitable to derive properties for 8-2 Telomer alcohol. This can be seen easily when comparing measured and estimated results for 8-2 Telomer alcohol (e.g. water solubility, vapour pressure, volatility, sorption constants, BCF).

The low water solubility, high volatility as well as sorptive properties of 8-2 alcohol makes testing especially of the Aquatic Ecotoxicity difficult.

	1-Decanol, CAS No. 112-30-1		1H,1H,2H,2H-Perfluorodecanol, CAS No. 678-39-7	
	estimated	measured	estimated	measured
MOLECULAR FORMULA				
Molecular weight	158,28 g/mol		-	446,12 g/mol
Formula	$C_{10}H_{22}O$		-	$C_{10}H_2F_{17}O$
PHYSICAL DATA				
Melting point [°C]	7 [1]	-	48 [3]	-
Density [g/cm³]	0,829 (20° C) [1]	-	1,7 (20° C) [3]	-
Refractive Index	1,4375 (20° C) [1]	-	1,3085 (20° C) [3]	-
Water solubility [mg/L]	37 (20° C) [1]	37 (20° C) [EPI] [5]	0,13 - 0,33 (21° C) [4] 0,19 - 0,48 (20° C) [4+17] [5]	0,009 (20° C) [2]
Vapor pressure [Pa]	1,11 [1]	1,06 (20° C) [EPI] [5]	1 (20° C) [3]	1,193 (20° C) [EPI] [5]
PARTITION DATA				
Air - Water Partitioning Coef. [-]	0,001 [5]	0,002 (20° C) [EPI] [5] 0,003 (20° C) [Bond] [5] 0,003 (20° C) [Group] [5]	Nonsensicals using static boilerplate method are correctly carried out	0,0 (20° C) [4+17] [5] 199 (EPI Direct) [5] 475 (EPI Direct) [5]
Air - Water Partitioning KOC (Adsorption)	2,3 [5]	3,8 (Bond) [EPI] [5] 3,6 (EPI Direct) [5] 7,2 (EPI Group) [5]		4,386 (20° C) [4+17] [5] 4,20 (20° C) [Bond] [5] 1,4270 (EPI Group) [5]
Octanol - Water Partitioning log Kow	4,57 [5]	3,73 (EPI) [5]	4,57 (EPI) [5] (OECD 117) [7]	1,81 (EPI) [5]
Organic carbon - Water Part. log Koc	not suitable	1,96 (EPI) [5]	3,98 (EPI) [5] (OECD 121) [7] 2,79 - 3,57 (Soils / sediment) (OECD 106, 142) [8]	2,28 (EPI) [5]
Bioconcentration Factor Fish BCF	not suitable	14 (EPI) [5]	320-1100 (Carp, 60d, 10 µg/L) 87 - 410 (Carp, 56d, 1 µg/L) [6]	1200 (EPI) [5]
BIODEGRADATION DATA				
Primary & Ultimate Biodegradation	readily biodegradable (OECD 301B) [1]	Test EPI - at ocean [5] Daly-weak (EPI) [5]	not readily biodegradable (OECD 301B) [6] not biocidally biodegradable (OECD 302B) [6]	not fast (EPI, at ocean) [5] recalcitrant (EPI) [5]
ABIOTIC DEGRADATION DATA				
Primary Photodegradation	not suitable	1 1/2 - 5,1 h (12h day, EPI) [5]	11 d (Atmospheric Hydroxyl Rate) [5] Measurements will be carried out in Summer 2004	1 1/2 - 30,7 h (12h day, EPI) [5]
Ultimate Photodegradation	not suitable	-		-

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