AR 226-1287

TELOMER B ALCOHOL, $C_8F_{17}CH_2CH_2OH$: WATER SOLUBILITY

DUPONT REPORT NUMBER 12718

Test Guideline(s)
OECD 105 (adopted 12 May 1981).

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16 July 2002
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Sponsor
Telomer Research Panel
c/o Rand Corporation

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1.0 SUMMARY

Test System:

The water solubility of 8-2 Telomer B Alcohol, $C_8F_{17}CH_2CH_2OH$, was determined in aqueous solution at 12° C, 25° C, 37° C and 60° C using a

variation of OECD method 105. The experimental mixture was shaken for at least three days and the test substance concentration determined at various intervals via gas chromatography mass spectrometry.

Findings:

The solubility in water was 134 µg/L (+/-29 µg/L) at 12° C ; 137 (+/-53 µg/L) at 25° C; 318 µg/L b (+/-176 µg/L) at 37° C, and 225 µg/L (+/- 51 µg/L) at 60° C.

Conclusions:

The solubility of 8-2 Telomer B Alcohol, $C_8F_{17}CH_2CH_2OH$, in water from 12° C to 60° C is very low (134 to 318 µg/L).

2.0 GENERAL STUDY INFORMATION

Study Objectives

The objectives of this study were to:

- Determine the water solubility between 12° C, 25° C, 37°C, and 60° C in pure water.
- Determine the effect of pH on water solubility at 12° C, 25° C, 37°C and 60° C.

Test System Justification

The test system was selected by the sponsor.

Study Personnel

Test Facility Name

DuPont Corporate Center for Analytical Sciences

Building 300, Glasgow, Delaware, 19714-6101 USA

Management:	Marianne Marsi, Ph.D.
Study Director:	Mary A. Kaiser, Ph.D.
	DuPont Company

Principle Investigator:	Daryl P. Cobranchi, Ph.D.
	DuPont Company
	Corporate Center for Analytical Sciences
Technical Personnel:	Barry W. Wolstenholme

Study Execution Dates

Study Initiation Date:	15-Jun-2001
Experimental Start Date:	15-Jun-2001
Experimental Completion Date:	28-June 2002
Study Completion Date:	12-July 2002

3.0 MATERIALS AND METHODS

3.1 Test Guidelines

The solubility in water of a substance is specified by the saturation mass concentration of the substance in water and is a function of temperature. The solubility in water is specified in units of weight per volume of solution. The SI-unit is kg/m^3; g/l may also be used.

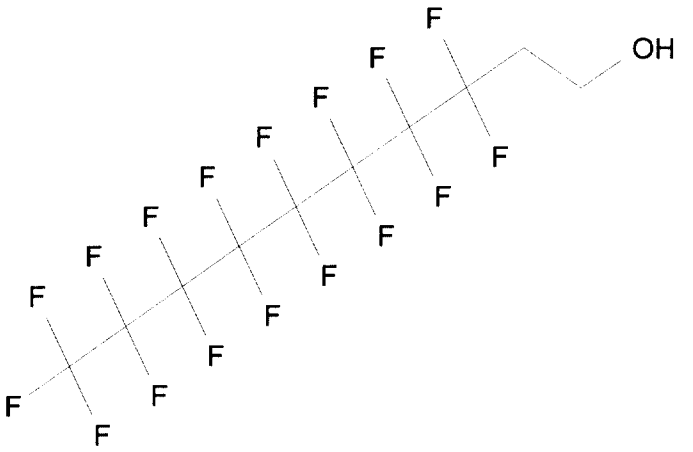
3.2 Chemical System

3.2.1 Test Substance

The test substance was obtained from Clariant Gmbh (Germany) and was 99.2% pure via gas chromatography. The major impurity (0.8% by area) was tentatively identified as $C_7F_{15}CF{=}CH_2OH$ based on the mass spectral fragmentation pattern.

Name:	8-2 Telomer B Alcohol
Synonym:	(Perfluorooctyl)ethanol
Active substance(s)	8-2 Telomer B Alcohol, 99.2%
CAS Name:	1-Decanol, 3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluoro-
CAS Number(s):	678-39-7

Structure:



H Number	H-24691
Lot Number:	P.00/001
EMSE Sample Number:	E93386-63
Concentration, nominal:	99%
Concentration, analyzed:	99.2%
Certificate of Analysis Date:	03-Apr. 2001
Date Received:	15-June 2001
Vapor pressure:	3 Pa at 25°C
Stability:	Stable at ambient room temperature
Appearance/Color:	White solid
Storage Conditions:	Room temperature; keep tightly closed

3.2.2 Test Vehicle

The test substance was dissolved in ultrapure water (17.6MΩ-cm).

3.2.3.1 Test Units

Test vessels were 2 mL (nominal) borosilicate glass vials, 12 mm outer diameter by 32 mm length. Vial caps were blue caps with Teflon® fluoropolymer resin and red-rubber septum caps.

3.2.3.2 **Test Conditions**

Test solutions were kept closed during the dissolution and shaking process.

3.3 Test Conduct

The water solubility of the 8-2 Telomer B Alcohol study material was determined at 12°C, 25°, 37°C and 60°C [1]. Excess test substance was placed in a 2-mL gas chromatography sample vials and shaken at constant temperature for 24 hours on a shaker table. The test substance concentration was measured at various time intervals by gas chromatography with mass spectrometric detection ((Electron Impact (EI) detection, m/z =463). In the second method used to determine water solubility, approximately 100 mg of the 8-2 Telomer B Alcohol was added to a standard screw-cap 2-mL glass gas chromatography vial. The vial was coated with excess 8-2 Telomer B Alcohol by placing the open vial on a warm surface to melt the test substance and condense the study substance onto the cooler upper surfaces of the vial. The vial was then removed from the warm surface and allowed to come to room temperature. Ultrapure (conductivity of 17.6 MΩ-cm) water was added to the vial so that no headspace was available. The silicone-rubber side of the vial cap was turned to cover the vial before sealing. The vial was placed on a thermostated, insulated shaker table at the temperature of interest. The table rotation rate was set at approximately 100 rpm for at least four days. A sample was removed to a temperature-chromatographic autosampler tray and the test substance was determined by GC/MS. This method is much faster than the first method and gives the same result.

Details of the GC/MS experiment

- Vials: Target DP (dual purpose) Vials, 1.8 mL capacity, borosilicate glass, 12 mm outer diameter by 32 mm length, National Scientific, Inc., 205 East Paletown Road, Quakertown, PA 18951 USA, catalog number C4000-1.
- Vial caps: Target DP (dual purpose) blue caps with Teflon® and red-rubber septum, National Scientific, Inc., 205 East Paletown Road, Quakertown, PA 18951 USA, catalog number C4000-51B
- Helium: 99.999% pure, plumbed with stainless steel and copper tubing, MG Industries, Newark, DE USA.
- Gas Chromatograph and Mass Spectrometer: HP (now Agilent) Series II Plus gas chromatograph and HP (now Agilent) 5972 mass selective detector (MSD) with a 100-sample autosampler tray and HP (now Agilent) 7673 GC injector tower, Agilent Technologies, 2850 Centerville Road, Wilmington, DE 19808, USA.
- Gas chromatograph's septa: Merlin-Microseal, Merlin Instrument Company, Half Bay, CA 94019 USA, part number 310.
- VWR cooling unit, 1310 Goshen Parkway, West Chester, PA 19380 USA, model 1160A.

- Nalgene polypropylene volumetric flasks, from VWR, 1310 Goshen Parkway, West Chester, PA 19380 USA.
- Thermolyne Bigger Bill orbital shaker set at 300 rpm, VWR, 1310 Goshen Parkway, West Chester, PA 19380 USA, with temperature-controlled table and tray. Shaker connected to a Brinkmann RM-6 cooling unit, Brinkmann Instruments, Inc., One Cantiague Road, PO Box 1019 Westbury, NY 11590 USA.

GC/MS conditions:

- Oven temperature: 80°C for one minute, increased at 20°C/minute to 300°C and held for 10 minutes. Run time 22 minutes.
- Injector: splitless, 225°C, purge flow 50.0 mL/minute, purge time 0.70 minutes, saver flow 20 mL/minute, 3 sample washes, no pump (quiescent), 1µL injection size, (10 µL Agilent syringe), three post injection washes with JT Baker ultraresi-analyzed cat. # 9263-03) methanol, sampling depth 10.0 mm above the nominal position.
- Solvent delay: 5.0 minutes (i.e., detector turned on after five minutes)
- Ions: 463.0, 131.0, 95.0, 69.0, 31.0 (preferred)
- EI at 70 eV
- Column: Restek RTX-200 (trifluoropropylmethyl polysiloxane), 60 meter, 250 µm diameter, 1.00 µm film thickness, catalog number 15056, Restek Corporation, 110 Benner Circle, Bellefonte, PA 16823 USA. Initial flow set at 1.4mL/minute, constant flow, initial inlet pressure 25.89 psi, average velocity 31 cm/sec.
- Limit of Detection (LOD) approximately 20 ppb; limit of quantification (LOQ) approximately 50 ppb.

3.4 Parameters Observed

The peak area or height was measured for the test substance saturated in the aqueous solution at specific temperatures.

3.5 Result Analysis

The peak height or peak area obtained for the test substance was compared to the same parameter of a standard carefully preparred in methanol.

3.6 Validity Criteria of the Study

The test results were considered valid if the peak areas of the standards run before and after the test substance were within 5% of the parameter measured.

3.7 Guideline Deviations

Deviation 1	Concerning:	Addition of the test substance to the vessel
	Deviation:	The test substance was coated on the wall of the test vessel.
	Reason:	The "excess" test substance formed needle-like structures, which interfered with the

		chromatographic injection.
	Impact on Study:	None
Deviation 2	Concerning:	Choice of test method
	Deviation:	The "flask" method recommended for compounds having solubility > 10 ppm was used rather than the "column elution method" which is recommended for compounds having solubility < 10 ppm.
	Reason:	The "column elution method" is less reliable that the "flask" method for volatile compounds.
	Impact on Study:	None

4.0 RESULTS AND DISCUSSION

Table 1 summarizes the solubility data versus temperature.

The solubility at pH 3, pH 11 and autogeneous pH (5.8) at 12°C were measured with no significant difference in the solubility for 8-2 Telomer B Alcohol over the pH range. *Table 2* ***through 7 summarize the measured water solubility versus pH determinations.***

The solubility in water at 12° C is 134 µg/L (+/-29 µg/L); 137 µg/L (+/-53 µg/L) at 25° C; 318 µg/L (+/-176 µg/L) at 37° C, and 225 µg/L (+/- 51 µg/L) at 60° C. Varying pH from 3 to 11 had no impact on the solubility measurement.

5.0 CONCLUSIONS

The solubility of 8-2 Telomer B Alcohol is in the 100 to 250 µg/l range from 12° C to 60° C.

6.0 RETENTION OF RECORDS

Study documents and materials will be stored in the archives of the DuPont Experimental Station including but not limited to:

- study protocol;
- any protocol and/or report amendments or addenda or deviations;
- all raw data;
- one original signed copy of the final report;
- laboratory-specific or site-specific raw data such as personnel files, instrument, equipment, refrigerator, and/or freezer raw data.

7.0 DISPOSAL OF TEST ITEM

After issuance of the final report, the remaining test substance will be stored at the DuPont Glasgow site until its expiration date and then destroyed by burning, unless other arrangements are made between the sponsor and the Test Facility.

8.0 REFERENCE

1. OECD Guidelines for the Testing of Chemicals / Section 1: Physical-Chemical properties Test No. 105: Water Solubility, adopted 12 May 1981. (published July 1995)

Table 1

Water Solubility versus Temperature (°C) at Autogeneous pH

12	134	29	8
25	137	53	17
37	318	176	10
60	225	51	10

n = number of tests

Table 2

Water Solubility versus pH at 12°C

pH	Solubility (ng/mL)	Std. Dev. (ng/mL)	n
3	227	109	6
autogeneous pH = 5.8	104	57	6
11	74	29	5

n = number of tests

Table 3

Water Solubility Raw Data : Autogeneous pH 5.8, 12°C

Slope	238.61
Intercept	6555.5

Std Dev	29
n	8
t	2.3646
95% UCL	158
95% LCL	109

Average 134 ppb

Sample	Area	Conc. (ppb)
1	36,712	126
2	32,865	110
3	38,141	132
4	28,037	90
5	44,510	159
6	37,270	129
7	0	0
8	51,133	187
9	38,660	135
10	0	0

Peak Area used for quantitation
n = number of determinations
UCL = Upper Confidence Level (ppb)
LCL = Lower Confidence Level (ppb)

Table 4

Water Solubility Raw Data : Autogeneous pH 5.8, 25°C

Slope	2.9948
Intercept	0

Std Dev	53
n	17
t	2.1199
95% UCL	164
95% LCL	109

Average	**137 ppb**

Sample	Set 1 Peak Height	Set 2 Peak Height	Conc (ppb)
1	511	595	185
2	470	543	169
3	*	*	
4	414	320	123
5	640	405	174
6	397	282	113
7	459	608	178
8	170	209	63
9	203	180	64
10	*	544	182

Peak Height used for quantitation
*Peak too high to measure. Solid probably included in injection.
n = number of determinations
UCL = Upper Confidence Level (ppb)
LCL = Lower Confidence Level (ppb)

Table 5

Water Solubility Raw Data : Autogeneous pH, 37°C

Slope	10.0332
Intercept	642.42

Std Dev	176
n	10
t	2.2622
95% UCL	444
95% LCL	192

Average 318 ppb

Sample	Area	Conc. (ppb)
1	4207	355
2	4624	397
3	2498	185
4	3205	255
5	2055	141
6	2660	201
7	1828	118
8	3991	334
9	5870	521
10	7366	670

Peak Area used for quantitation
n = number of determinations
UCL = Upper Confidence Level (ppb)
LCL = Lower Confidence Level (ppb)

Table 6

Water Solubility Raw Data : Autogeneous pH, 60°C

Standard run before and after each sample.

	Set 1	Set 2	
Sample	**Area**	**Area**	**Conc. (ppb)**
1	486500	366225	297
2	289001	266224	196
3	319066	273785	219
4	225266	226886	175
5	275743	263277	214
6	335225	240121	236
7	206398	190197	171
8	180304	326588	228
9	301712	343656	288
10	285294	212958	227

Std Dev	51
n	20
t	2.09302
95% UCL	249
95% LCL	201

Average 225 ppb

Peak Area used for quantitation
n = number of determinations
UCL = Upper Confidence Level (ppb)
LCL = Lower Confidence Level (ppb)

Table 7

Water Solubility vs. pH Raw Data at 12°C

		Sample pH	Peak Height	Conc. (ppb)
Std Dev	109	3	30	284
n	6	3	18	170
		3	12	114
95% UCL	234	3	23	217
95% LCL	219	3	44	416
		3	17	161
pH 3 average	**227 ppb**			

		Sample pH	Peak Height	Conc. (ppb)
Std Dev	57	5.81	11	104
n	6	5.81	23	217
		5.81	9	85
95% UCL	107	5.81	7	66
95% LCL	100	5.81	8	76
		5.81	8	76
pH 5.81 average	**104 ppb**			

		Sample pH	Peak Height	Conc. (ppb)
Std Dev	29	11	6	57
n	5	11	8	76
		11	4	38
95% UCL	76	11	1087*	*
95% LCL	71	11	12	114
		11	9	85
		*rejected due to probable solid introduction		
pH 11 average	**74 ppb**			

Peak Area used for quantitation
n = number of determinations
UCL = Upper Confidence Level (ppb)
LCL = Lower Confidence Level (ppb)

Appendix A

Chemical Analysis Summary
2-Perfluorooctyl ethanol : $C_8F_{17}CH_2CH_2OH$
H-24691

Analysis Summary

The subject material was analyzed by gas chromatography using a standard method to assess its composition. This analysis showed that the material comprised 99.2% of the subject material. The balance, 0.8%, was not another Telomer B alcohol, or Telomer B Iodide at a limit of quantitation of 0.02%. Further analysis by GC/MS suggests that the 0.8% is a single impurity, whose chemical structure is $C_7F_{15}CF{=}CHCH_2OH$.

Finally, the subject material was evaluated by High Performance Liquid Chromatograpy - Tandem Mass Spectrometry (LC/MS/MS) for the presence of perfluorooctyl sulfonic acid and perfluorooctanoic acid at a detection limit of 0.1ppm. Neither material was observed to be present.

Based upon these analyses, we conclude that methods are in hand to determine and confirm the composition of H-24691 to be as follows:

Material	Wt. % (±0.3)
$C_8F_{17}CH_2CH_2OH$ 2-Perfluorooctyl ethanol	99.2
$C_7F_{15}CF{=}CHCH_2OH$	0.8

Appendix B

Solubility Determination- Modified ASTM Procedure

1. A concentrated (~50% w/w) solution of 8-2A in GC grade methanol was prepared.
2. Approximately 200 µL were placed into a standard screw-cap GC vial.
3. The **open** vial is placed in a warm location to evaporate the solvent.
4. After the methanol had evaporated, the 8-2A was observed to collect on the upper surfaces of the GC vial.
5. The vial was then removed and allowed to cool to room temperature.
6. Ultrapure18 MΩ water was carefully added to the vial, filling it all the way to the top (little or no headspace).
7. The septum was turned upside down so that the silicone rubber side was facing towards the solution.
8. The vials were then capped and placed in a temperature controlled shaker table operating at ~100 rpm.
9. The vials were shaken for a minimum of 4 days prior to analysis.
10. The vials were then removed to the GC/MS auto-sampler tray, temperature controlled.
11. The GC/MS was calibrated against 8-2A samples prepared in methanol.