PETITIONER SUMMARY DATA SHEET

("P-SDS")

SUBSTANCE : **Perfluoro acetic acid, a-substituted with the copolymer of perfluoro-1,2-propylene glycol and perfluoro-1,1-ethylene glycol, terminated with chlorohexafluoropropyloxy groups.**

USE OF SUBSTANCE : additive

REF No.: 71943

CAS. N.: 329238-24-6

SOCIETY: SOLVAY SOLEXIS

PERSON RESPONSIBLE FOR THE TECHNICAL DOSSIER: [redacted]

ADDRESS OF THE RESPONSIBLE: [redacted]

PHONE: [redacted] FAX: [redacted]

e-mail: [redacted]

Request for re-evaluation

A technical dossier for Perfluoro acetic acid, a-substituted with the copolymer of perfluoro-1,2-propylene glycol and perfluoro-1,1-ethylene glycol, terminated with chlorohexafluoropropyloxy groups has been submitted initially on March 1, 2007. Upon evaluation by the Working Group of the Scientific Panel on Food Additives, Flavourings, Processing Aids and Materials in Contact with Food (AFC) of EFSA, additional data were submitted in June 2007.

After evaluation of the additional data submitted on June 27, 2008, EFSA/AFC/FCM/1261, the Working Group of the Scientific Panel on food contact materials, enzymes, flavourings and processing aids (CEF) of the European Food Safety Authority (EFSA) has classified the substance in the SCF-list 7, because of the lack of data as set out below:

First of all a clarification is needed concerning results of the GC/MS measurements as described in 5.3.11. It is stated there that 'No additional peaks were found in the GC chromatograms of the sample extracts' from which was concluded that 'GC-MS gives no information on the migration …..'. On the other hand, in the corresponding GC-MS chromatograms attached in Annex 11 when comparing fig. 17 with fig. 18, 2 large and some further small additional peaks can be recognized. Unfortunately mass spectra are not

provided. This is in contradiction to the above statement made by the petitioner and it should be clarified and substantiated.
To clarify whether the substance is still present in the food contact material and whether it can migrate in the high temperature application, further experiments should be carried out such as:

1. P&T analysis (GC/MS and/or GC/AED on the fluorine wavelength) of the test sample (ground to small particles) where the sample should be heated up to 230°C for 90 min. Since it is a repeated use application, this experiment should be carried out 3 times in series on the same specimen. Eventually detected GC peaks related to the substance should be quantified and identified. To validate this procedure, recovery should be tested by spiking the substance into the ground test sample and repeating the experiment.

2. Depending on the results from 1, migration test using MPPO (modified polyphenylene oxide) at 230°C for 90 minutes (3 times in series using the same specimen) and either Head Space/ GC analysis of the MPPO or extraction with methanol (without concentration step and if with, then with appropriate recovery check) may be necessary.

With the present technical dossier, additional information as requested by EFSA is submitted as follows:

⇒ Explanation for the GC-MS results is given in item 5.3.11

⇒ Explanations for omitting the additional experiments as described in item 1 and 2 above by the EFSA Working Group are given 5.3.

Annexes 1-11 are not included in this technical dossier as they were provided already in the technical dossier submitted in 2007 and 2008.

Annex 12 is included in the present technical dossier, containing GC-MS chromatograms and spectra.

The additional information has been added to the P-SDS dated June 27, 2008 and can be recognized as text on a gray background.

REMARKS:

CONFIDENTIALITY

Certain sections of this filing contain confidential business information that should not be released to third parties. The information marked "Confidential" should be reviewed only by authorized members of EFSA and the European Commission in connection with its evaluation of the suitability of the petitioned substance for use in food contact materials.
More specifically the following information is considered confidential information:

- the proprietary manufacturing process *(see 1.3.6);*
- part of the information related to the substances formed *(see 1.3.7);*
- information on the purification process *(see 1.3.8);*
- information on the by-products formed *(see 1.3.9)*

Clearly, public disclosure of this information would harm Solvay Solexis' competitive position, as competitors could potentially improve their own process by learning the details of Solvay Solexis' process.
The confidential information has been removed from the "non-confidential" version of the filing, provided on CD-ROM.

The information considered to be treated as confidential can be recognized as text on a yellow background.

INFORMATION REQUESTED		**INFORMATION PROVIDED**
1.	**IDENTITY OF SUBSTANCE**	
1.1	**individual substance:**	NO
1.2	**defined mixture:**	NO
1.3	**Non-defined mixture:**	YES
1.3.1	**chemical name:**	1-propene,1,1,2,3,3,3,-hexafluoro-, telomers with chlorotrifluoroethene, oxidized, reduced, hydrolyzed
1.3.2	**synonym(s):**	Chloro-fluoropolyether acid
1.3.3	**trade name(s):**	FLUOROLINK 7800; FLUOROLINK 7850 Both products are covered by the same CASRN: the differences are in Molecular Weight (lower in 7800) and Molecular Weight Distribution (wider in 7850)
1.3.4	**CAS nr:**	329238-24-6
1.3.5	**starting substances:**	[redacted]
1.3.6	**manufacturing details:**	***THIS INFORMATION IS CONFIDENTIAL*** [redacted]

[redacted]

Ref: Annex 1

1.3.7 substances formed:

Fluorolink 7800 and 7850 are mixtures of perfluoro-acids with the following simplified notation:

species	n	m	Molecular weight
N1	0	0	296,5
N2	1	0	462,5
M3	1	1	578,5
N3	2	0	628,5
M4	2	1	744,5
N4	3	0	794,5
N5	4	0	960,5

Where n and m are the coefficients of the general formula:

C_3F_6ClO-[CF_2-$CF(CF_3)$-O]$_n$-[$CF(CF_3)$-O]$_m$-CF_2COOH

where n ranges from 1 to 4 and m from 0 to 2 in the active species.

The repeating unit –O-[CF_2-$CF(CF_3)$-O]- can also be present in 'reverse' shape:

-O-[$CF(CF_3)$-CF_2-O]-

The C_3F_6ClO- end group is present in both normal CF_3-$CFCl$-CF_2-O- and iso form: CF_2Cl-$CF(CF_3)$-O-.

Other end-groups in minor amount are CF_3O-, CF_2ClO- and the ketone hydrate –CF_2-$C(OH)_2$-CF_3.

Ref: Annex 1, 2

1.3.8 purification by:

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[redacted]

Ref: Annex 1

1.3.9 by-products:

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Main by-products are:

- ketones (in hydrate form) –CF_2-$C(OH)_2$-CF_3.
- -non-functional molecules (both end-groups like C_3F_6ClO-; CF_3O-; CF_2ClO-

Ref: Annex 1

1.3.10 molecular and structural formula:

The active ingredient is nominally

C_3F_6ClO-[CF_2-$CF(CF_3)$-O]$_n$-[$CF(CF_3)$-O]$_m$-CF_2COOH

where n ranges from 1 to 4 and m from 0 to 2.

Ref: Annex 2

1.3.11 molecular weight (M_W) and range:

	Typical Composition	
	FLK7800	**FLK7850**
Molecular Weight	500-550 D	500-650 D
MW<300 content	10% max	10% max
MW>750 content	5% max	-
MW>800 content	-	5% max

Ref: Annex 1,2

1.3.12 purity (%):

The typical active ingredient levels are:
-FLUOROLINK 7800= 80 ±5%
-FLUOROLINK 7850= 79 ±5%

Ref: Annex 2

1.3.13 impurities (%):

The total levels of impurities are:
-FLUOROLINK 7800= 20 ±5%
-FLUOROLINK 7850= 21 ±5%

Main impurities are:
- ketones ($–CF_2$-$C(OH)_2$-CF_3 (present only in hydrate form, in the acid-aqueous environment, 5-8%, acids and tensides titrations);
- H_2O (1-2%, determined by Karl Fisher titration)
- -non-functional molecules (both end-groups like C_3F_6ClO-; CF_3O-; CF_2ClO- (1-2%);
- residual HF (<0.1%, ion chromatography))
- MW < 300 max 10% (consisting of N1 (not detected at a detection limit of 100 ppm) and species like CF_3O-CF_2COOH or CF_2ClO-CF_2COOH, determined by GC);
- MW > 750 max. 5% (species N5 and higher, determined by GC)

Ref: Annex 2

1.3.14 spectroscopic data:

NMR spectrum is provided in the technical dossier

Ref: Annex 3

1.3.15 specifications:

None

1.3.16 other information:

-

1.4 polymer used as additive:

NO

2. PHYSICAL AND CHEMICAL PROPERTIES OF SUBSTANCE

2.1 physical properties

2.1.1 melting point (°C):

[redacted]

2.1.2 boiling point (°C):

[redacted]

2.1.3 decomposition temperature (°C):

Decomposition may start to occur at temperature exceeding 250 °C; however, under normal processing

conditions, evaporation will have preceded the possibility of decomposition.

Ref: Annex 4

2.1.4 solubility (g/l): The solubility in water is below 5% w/w at T = 25 °C. The substance is soluble in the following polar solvent: IPA, MEK, DPM (DiPropylenglycol monoMethylether)

2.1.5 octanol/water partition(log Po/w) Data not available; information not relevant (migration is < 0.05 mg/kg of food, substance is not requested to be subject to Fat Reduction Factor).

2.1.6 other information related to lipophilicity: No other information is submitted

2.2 chemical properties

2.2.1 nature: [redacted]

Ref: Annex 5

2.2.2 reactivity: The substance is not reactive under the conditions described in section 3.

2.2.3 stability: The substance is stable under the conditions described in section 3.

Ref: Annex 4

2.2.4 hydrolysis: The substance is not subject to hydrolysis

2.2.5 intentional decomposition/ transformation: None

2.2.6 unintentional decomposition/ transformation product(s): No decomposition/transformation has been observed under the conditions described in section 3.

2.2.7 interaction with food substances: There are no known interactions with food substances.

2.2.8 other information: No other information is submitted.

3. INTENDED APPLICATION OF SUBSTANCE

3.1 food contact material: Fluorolink 7800 and 7850 are used in the manufacture of fluorinated polymers like:

- [redacted]
- [redacted]
- [redacted]
- [redacted]

These fluoropolymers are processed to produce a wide range of articles, like the following:

- Parts for food processing equipment (fitting, valves);
- Tubes, sheets, pipes;
- Tapes;
- Antistick coatings on cooking utensils

All of these articles manufactured from these polymers and copolymers are for repeated use.

Fluorolink 7800 [redacted]; 7850 [redacted].

EFSA question no. 4: A clear differentiation is needed between intended applications: which ones are for elastomers and which for non-elastomers?

The petition does not relate to fluoroelastomers but only to food contact materials that are covered by the definition of plastics.

Melt processable fluoropolymers and coating fluoropolymers mentioned in the present petition belong to the non-elastomer family.
They are processed in order to produce a wide range of articles;

- tubing, fittings, pipes and valves
- films and antistick coatings on various food contact articles.

Finished articles are intended for repeated use.

[redacted]

Ref: Annex 10

3.2 technological function:

The substance is used as an emulsifier/dispersing agent during the polymerization process of fluoropolymers. Its presence ensures that the polymer to be synthesised forms a stable suspension of particles.

3.3 maximum process temperature (°C):

The typical temperatures in tubing extrusion and compression moulding are in the range 340÷380 °C, up to 400 °C. The maximum process temperature is encountered in coating applications; the coating is dried and then baked at temperatures up to 420 °C for up to 10 minutes.

EFSA question no. 1: Information supporting the statement that the sample tested corresponds to a worst case situation: Can the substance quantitatively evaporate during compression moulding?

The sample tested for migration was [redacted], a melt processable TFE copolymer. Melt processable TFE grades are considered to represent the worst case, i.e. contain the highest amount of residual surfactant, if any. Due to the processing temperature (lower) and the

thickness (higher) of melt processable grades, reduced evaporation of the surfactant in melt processable grades is expected when compared to coating grades.
Furthermore, compression moulded articles can be considered the worst case vs the injection molded articles, the latter being processed at higher temperatures. Again, the lower molding temperature could result in a reduced evaporation of the surfactant.

Ref: Annex 10

3.4 maximum percentage in formulation:

The maximum content level, in polymerization recipe, is 0.5 %.

[redacted] the content of the surfactant is about 0.2% w/w of the full recipe; this content is equivalent to 0.5% w/w of the "solid" content.
The level of the surfactant in the sample tested for migration is 0.5% w/w of the solid content

The resulting emulsion can be :

- coagulated and the wet polymer is dried in a rotary kiln for ~ 1h at 130-150 °C; [redacted]

In order to manufacture the final articles, the polymer has to be sintered at high temperature; this process results in a further reduction in the level of residual substance.

3.5 conditions of contact in practice

3.5.1 contact food: All types of foodstuffs

3.5.2 time and temperature: The maximum temperature of contact is experienced by coated cookware, which is used at temperature up to 230 °C for usually up to 1 or 2 hours; there may be excursions up to 250 °C for 15 minutes.

3.5.3 surface to volume ratio: As a worst case the conventional surface to volume ratio of 6 dm2/kg is applicable. However many application will have lower surface to volume ratios.

3.5.4 other information: All the articles manufactured from these fluoropolymers are for repeated use.

3.6 treatment of food contact material prior to use: Cookware and bakeware are usually washed in hot soapy water before use; other articles, such as components of food processing machinery, are rinsed with hot water.

3.7	**other uses:**	Lubricant powder grades containing Fluorolink may be used as additives in a variety of polymers, which may be extruded and moulded into articles for contact with all types of foodstuffs, at all temperatures for repeated use. Lubricant powders are used to reduce friction and wear.
3.8	**other information:**	

4. AUTHORISATION OF SUBSTANCE

4.1	**EU countries**	
4.1.1	**in Member States:**	NO
4.1.2	**notified as "new substance" in the context of 6th Amendment of Directive 67/548/EEC:**	Substance is a polymer according to 92/32/EC
4.1.3	**other information:**	-
4.2	**non-EU countries**	
4.2.1	**in USA:**	NO
4.2.2	**in Japan:**	NO
4.2.3	**in other countries:**	NO
4.2.4	**other information:**	-
4.3	**other information:**	-

5. DATA ON MIGRATION OF SUBSTANCE

5.1	**specific migration (SM):**	SM not determined; instead, the actual content of Fluorolink 7850 was determined. ***Ref: Annex 6***
5.2	**overall migration (OM):**	Overall migration determined. ***Ref: Annex 6***
5.2.1	**test sample:**	Sheets of fluoropolymer [redacted] ([redacted] = propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene and trifluoro(trifluoromethoxy)ethene) The level of the surfactant used in the manufacture of the test sample is 0.5% w/w of the solid content. In the migration test report the substance name is indicated as Fluorolink 7800:7850, while actually Fluorolink 7850 was tested. Fluorolink 7800 is a "sub-set" of Fluorolink 7850, therefore this last grade should be the "worst-case".
5.2.2	**treatment of sample**	none

prior to testing:

5.2.3 food simulant(s): Iso-octane and 95% ethanol

5.2.4 contact mode: Total immersion

5.2.5 contact time and temperature: 95% ethanol: 6 hr 60°C
Iso-octane: 4 hr 60°C

5.2.6 surface to volume ratio: Per test two sheets of approximately 13.5x13.5x0.04 cm were immersed in 100 ml food simulant resulting in a surface to volume ration of 3.484 dm^2/100 ml, taking one side into account.

5.2.7 test method: After incubation period the samples were removed and after evaporating off the solvents under controlled conditions, the residues are estimated gravimetrically. The test was performed in duplicate. From the results the overall migration in mg/6 dm^2 was determined.

5.2.8 other information: -

5.2.9 results: Results obtained for the overall migration are presented in table below. The total or overall migration in 95% ethanol and iso-octane are < 1 mg/6 dm^2. Individual values are well below the established analytical error. Residues determined may be caused by some migration of substances or they may be just the result of the weighing error in the analytical procedure.

Overall migration from Fluoropolymer [redacted]

Simulant	Test condition	Overall migration (mg/ 6 dm^2)
95% ethanol	6 hr 60°C	0.27
		0.68
		Mean 0.48
Iso-octane	4 hr 60°C	0.45
		0.95
		Mean 0.7

5.3 Quantification and identification of: a) migrating oligomers and b) reaction products derived from monomers and starting substances and additives

Determined.
The residues obtained from the overall migration experiments extracts with 95% ethanol and iso-octane were characterized using NMR.

Ref: Annex 6

EFSA question no. 2: Clarification on the procedure used for identification and quantification of migrating oligomers. - What in the procedure ensured that there is no loss of the substances to be analyzed during solvent evaporation?
The residue obtained under item 5.2.9 (overall migration) was characterized and was obtained by concentrating the migration extract under controlled conditions, i.e. by solvent evaporation on a boiling water bath. This means

that no precautions were taken to ensure that no loss of the substances to be analyzed during solvent evaporation took place, and “only the non-volatile” oligomers/reaction products, if any, were taken into consideration.

In order to obtain additional data on the identification and quantification of migrating reaction product(s) or oligomer(s) of Fluorolink 7800:7850, including migration of any volatile substances, the migration study was repeated in 95% ethanol. The migration extract was analysed directly (i.e. without any concentration step) and after evaporation to dryness under milder conditions, for the presence of any Fluorolink 7800:7850 reaction product(s) and/or oligomer(s) using GC-MS and LC-MS.

Ref: Annex 11

- Why was 1H-NMR used, rather than 19F-NMR?
LC-MS was used to determine the residual content of Fluorolink 7800:7850, [redacted], while 1H-NMR was used to find out if any other (non-fluoro containing) oligomer or reaction product of Fluorolink could be detected in the migration extracts.

The Working Group of the Scientific Panel on food contact materials, enzymes, flavourings and processing aids (CEF) of EFSA has asked to clarify whether the substance is still present in the food contact material and whether it can migrate in the high temperature application.

Additional tests as proposed by EFSA have not been performed because the data submitted in the past are considered to represent the worst case migration of Fluorolink 7800:7850, including any possible migration at the high temperature application, as explained below:

- worst case migration <1 µg/kg food
The residual content of Fluorolink 7800:7850 in [redacted] has been determined by extracting the polymer test sample for 8 hr reflux in methanol/ water (80/20) (see item 6). Where 6 hr 60°C in 95% ethanol could possibly result in incomplete extraction of residual Fluorolink 7800:7850, if any, *8 hr reflux in methanol/water is considered to represent a worst case migration condition.*
From the result obtained it can be calculated that for [redacted] or tubings used up to 100°C for short time, any migration of Fluorolink 7800:7850 is < 1 µg/kg food (see 6.7), *while toxicity data are provided covering a migration level up to 50 µg/kg food.*

- worst case polymer
The migration tests proposed by EFSA applying temperatures up to 230°C cover baking conditions are applicable to coatings manufactured using Fluorolink 7800:7850. The proposed tests are not applicable to the sample tested for this dossier.
The sample tested for migration and residual content was [redacted], a melt processable TFE copolymer. These polymers are not used for baking at high temperatures.

However, melt processable TFE grades are considered to represent the worst case, i.e. contain the highest amount of residual surfactant, if any, taking into account the thickness of the polymer test sample (0.04 cm) and the temperature during manufacture (see 3.3).

When using Fluorolink 7800:7850 in antistick coatings on cooking utensils, the maximum process temperature during manufacture of a coating is higher when compared to [redacted] melt processable TFE copolymer. The coating is dried and then sintered or baked at temperatures up to 420 °C for up to 10 minutes. Therefore, residual Fluorolink 7800:7850, if any would be lower in coatings then in a melt processable TFE copolymer like [redacted]. In addition, the thickness of the coating is much lower than the thickness of the [redacted] melt processable TFE copolymer sample. Furthermore, cookware and bakeware are usually washed in hot soapy water before use (see 3.6), leading to further decrease of surfactant content, if any at all.

The thickness of a coating is in the range of 0.025 mm to 0.25 mm (coating weight ~ 50 g/m2 , which corresponds to a coating thickness of 25 µm), *see Annex 11.*
The detection limit for the determination of Fluorolink 7800:7850 in samples of fluoropolymer was 20 µg/kg (*see 6.5.1).*
As a result, worst case migration of Fluorolink 7800:7850 is calculated to be 0.06 µg/kg food from a coating with a thickness of 0.025 mm or 25 µm.
In the unrealistic worst case, from a coating with a thickness of 0.25 mm or 250 µm, worst case migration would be 0.6 µg/kg food while, again, *toxicity data are provided covering a migration level up to 50 µg/kg food*.

5.3.1 test sample: Sheets of fluoropolymer [redacted]

Sheets of fluoropolymer [redacted]

Ref: Annex 11

5.3.1.1 chemical composition: [redacted] = propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene and trifluoro(trifluoromethoxy)ethene

The level of the surfactant, Fluorolink 7850, in the test sample is 0.5% w/w of the solid content.

The sample tested was manufactured according the same process of the samples tested previously (TNO report V7054, October 2006) [redacted] the content of the surfactant is about 0.2% w/w of the full recipe; this content is equivalent to 0.5% w/w of the solid content.

Ref: Annex 11

5.3.1.2 physical composition: Homogeneous material

5.3.1.3 density, melt flow index of polymer:	Density: 2.12-2.17 Melt Flow Index = 2-5 g/10 m Measured at 372 °C. with a load of 5 kg (ASTM D1238)
5.3.1.4 dimensions of test sample:	Sheets of approximately 13.5x13.5x0.04cm Sheets of 13 x13 cm; thickness ca. 0.04 cm ***Ref: Annex 11***
5.3.1.5 dimensions of test specimen	Per test two sheets of approximately 13.5x13.5x0.04 cm were immersed in 100 ml food simulant. Per test two sheets of 13 x13 x 0.04 cm were immersed in 100 ml food simulant ***Ref: Annex 11***
5.3.2 treatment of test sample prior to testing:	-
5.3.3 test food(s)/food simulant(s)/extraction solvent(s):	95% ethanol and iso-octane 95% ethanol ***Ref: Annex 11***
5.3.4 contact mode:	Total immersion Total immersion ***Ref: Annex 11***
5.3.5 contact time and temperature:	95% ethanol: 6 hr 60°C Iso-octane: 4 hr 60°C 95% ethanol: 6 hr 60°C ***Ref: Annex 11***
5.3.6 surface to volume ratio in migration tests:	3.484 dm^2/100 ml, taking one side into account. 3.38 dm^2 /100 ml, taking one side into account ***Ref: Annex 11***
5.3.7 analytical method:	The residue obtained under item 5.2.9 (overall migration) was characterized by NMR. Prior to NMR spectroscopy, the residue was dissolved in 1 ml deuterated chloroform ($CDCl_3$) containing 0.05 % tetramethylsilane (TMS). The spectra were acquired by accumulation of 256 FIDs. Spectra were recorded using a spectral width of 8000 Hz. Chemical shifts of all spectra are expressed in ppm by reference to TMS (0.00 ppm). Resolution enhancement and noise reduction of the spectra was performed using an exponential window function with a line broadening of 0.3 Hz and a manual baseline correction was applied. Per test, two sheets of fluoropolymer (one sheet is 13 cm x 13 cm, i.e. 1.69 dm^2 per sheet or 3.38 dm^2 for two sheets, taking one side into account) were cut into pieces

of ca. 2x2 cm and transferred into a 250 ml Scott flask. An amount of 100 ml 95% ethanol (preheated at 60°C) was added to the polymer sample. The flask was closed with a gas-tight cap and placed in an oven for 6 hours at 60°C. After this period, the migration solution was cooled down and an aliquot of 0.5 ml of the 95% migration solution was injected directly for GC-MS and LC-MS analysis.

The remaining migration solution was gently evaporated to dryness at 60°C under a nitrogen stream, redissolved in 0.5 ml 95% ethanol and injected for GC and LC-MS analysis.

For reference purposes, standard solutions of Fluorolink 7800:7850 were prepared in 95% ethanol at a level of 10 µg/ml and 50 µg/ml.

GC-MS conditions
Samples were injected on an Alltech AT 5ms column (30 m x 0.25 mm; film thickness 0.25 µm); MS data were collected in the scan mode ([redacted]).

LC-MS conditions:
Blank, migration and standard solutions were analysed by Ion Trap- Mass Spectrometry (LC-IT-MS) in the ESI(+) and in the ESI (-) mode. 10 µl of the solutions were directly injected (flow injection) in the system, using MeOH : ammonium acetate (5 mM) 1:1 (v/v) at a flow rate 300 µl/min. [redacted].

5.3.8 detection/ determination limit:

Not relevant

GC-MS: not established

The detection limit of the LC-MS method applied is set at 10 µg/ml ([redacted] Fluorolink 7800:7850 [redacted]).
Taking a surface to volume ratio of 3.38 dm²/ 100 ml [redacted] this corresponds to 9 µg/6 dm² or 9 µg/kg food.
For the non-concentrated extract the detection limit is 1.8 mg/kg, see our comments in 5.3.11.

5.3.9 recovery:

Not relevant

-

5.3.10 other information:

-

5.3.11 results:

It appeared that the signals present in the ^{1}H-NMR spectra of both, the fluoropolymer extracts and in their blanks, originate from compounds introduced during the extraction procedure and the NMR sample preparation; i.e. residual solvent (7.26 ppm), acetic acid (2.10 ppm_water (1.56 ppm), TMS (0.00 ppm). Due to the absence of protons in the fluoropolymer and monomers

these are not visible for ^{1}H-NMR. The signals present in the extract samples and absent in their blanks are assigned as proton containing migrants. Due to the low intensities en pure resolution of the signals further assignment was not possible.
Final conclusion is that no significant peaks are detected that can be ascribed to reaction products of the fluoropolymer tested.

GC-MS analysis ([redacted])

[redacted] Fluorolink 7800:7850 [redacted] Fluorolink 7800:7850.

LC-MS analysis

[redacted] Fluorolink 7800:7850 [redacted]

[redacted] Fluorolink 7800:7850 [redacted] Fluorolink 7800:7850 [redacted] Fluorolink 7800:7850 [redacted]

[redacted] Fluorolink [redacted]

[redacted] Fluorolink 7800:7850 [redacted]

[redacted] Fluorolink 7800:7850 [redacted].

Taking a surface to volume ratio of 3.38 dm²/ 100 ml [redacted] Fluorolink 7800:7850 [redacted] is considered not detectable, i.e. less than 9 µg/6 dm² or less than 9 µg/kg food.

The detection limit for direct injections is too high to detect small amounts of “volatile” components. However, in item 6 the loss of volatile components by using alkalized/non-alkalized extraction solvent was investigated. The extracts were directly injected, i.e. without any concentration step. No differences were found for the alkalized/not alkalized extracts, which means that no loss of migratable substances during extraction was observed. The detection level was 20 µg/kg polymer, which results in a worst case migration of less than 1 µg/kg food.
The experiments described in item 6 were measured in the SIM mode, with significant lower detection limit, while the 95% ethanol migration extracts were measured in the scan mode [redacted].
It is considered that the alkalization will avoid evaporation of substance containing the functional carboxylic group. Substances, like decomposition products, that do not contain the carboxylic group will not be influenced by the alkalization. However decomposition products -if any- that might have lost the polar carboxylic group are very volatile. They will disappear, even faster than the Fluorolink, during manufacturing of the polymer and/or the final article.
As no differences were found in the spectra for the non-concentrated and the concentrated ethanol migration extract, it is concluded that concentrating the 95% ethanol extract under the conditions applied does not cause a loss of extracted substances as it was demonstrated (see item 6) that no Fluorolink related substances remain in the final FCM.

Ref: Annex 11

Clarification of GC-MS results
- standard solution 10 µg/ml and 50µg/ml.

For the standard solutions containing Fluorolink at a level of 10 µg/ml and 50 µg/ml, no differences were found in the GC-chromatograms, i.e. *not any elevated signal was detected for the high standard solution when compared to the lower standard solution.*
[redacted] *Fluorolink.*

- concentrated sample extract versus concentrated blank extract
It is concluded that for the concentrated sample extracts, the GC chromatogram showed an increased noise level

and an increased signal for the peaks already detected in the blank extracts.

[redacted] Fluorolink.

[redacted] *Fluorolink 7800:7850*.

Ref: Annex 12

6. DATA ON RESIDUAL CONTENT OF SUBSTANCE IN THE FOOD CONTACT MATERIAL

6.1	**actual content:**	Actual content determined ***Ref: Annex 6***
6.2	**substance:**	In the report the substance name is indicated as Fluorolink 7800:7850, while Fluorolink 7850 has been used; Fluorolink 7800 is a "sub-set" of Fluorolink 7850, therefore this last grade should be the "worst-case".
6.3	**test sample:**	Sheets of fluoropolymer [redacted]
6.3.1	**chemical composition**	[redacted] = propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene and trifluoro(trifluoromethoxy)ethene The level of the surfactant used in the manufacture of the test sample is 0.5% w/w of the solid content.
6.3.2	**physical composition**	Homogeneous material
6.3.3	**density, melt flow index of polymer**	Density 2.12-2.17 g/cm^3
6.3.4	**dimensions of test sample**	Sheets of approximately 13.5x13.5x0.04cm
6.3.5	**dimensions of test specimen:**	The sheets of fluoropolymer were cut into pieces of ca. 1x3 cm. A test-sample of approximately 27 g is transferred to a conical flask of 100 ml.
6.4	**treatment of sample:**	The sample is refluxed for 8 hours with methanol/water

(80/20; v/v). After the extraction an aliquot of the extract was transferred to a HPLC vial. The remaining extraction solvent was decanted and the extraction was repeated with a fresh amount of 50 ml methanol/water (80/20;v/v). From each of the first and second extracts 50 µl was injected into the HPLC and analysed for containing Fluorolink 7800:7850 as described in 6.5.
Using a new test specimen the extraction was repeated by using methanol/water (80/20) containing 0.1% ammonia. By addition of ammonia the less volatile Fluorolink 7800:7850 ammonium salt was formed and so the possibility of evaporation of the Fluorolink 7800:7850 during the extraction procedure was reduced.

The rationale of the procedure to determine the residual concentration of the substance.
- Is methanol a swelling solvent for the polymer?
- Why was an extraction solution like that not used for identification and quantification of oligomers?

Methanol/water (80/20) was used as extraction solvent for the determination of residual Fluorolink because Fluorolink is good soluble in the methanol/water mixture. In addition the test sample has a thickness of only 0.4 mm. Although swelling was not checked an extraction for 8 h at ± 80ºC was considered an exhaustive extraction. To check exhaustive extraction, the test sample was extracted a second time. In the first extract some small peaks (below the LOD) were detected, but in the second extract no peaks were found, it can be concluded that the conditions of extraction applied were adequate for the intended purpose, i.e. determination of the residual content of Fluorolink 7800:7850 in the polymer sample.

For the identification and quantification of oligomers, it was considered more appropriate to perform a migration experiment in a food simulant, i.e. 95% ethanol, instead of extraction with methanol/water. Performing a migration test is more close to any release in real foods, although it was noticed that condition of 6 h at 60ºC with 95% ethanol and extraction for 8 h at ± 80ºC with methanol/water most likely would not make a great difference in extraction quantities.

6.5 test method:

The extracts are analysed for the content of chlorofluoropolyether acids by LC-MS in the negative electrospray ionisation mode.
Quantification is done by means of an external standard calibration.

6.5.1 detection/ determination limit:

10,68 ng/ml, based on lowest calibration point. This corresponds to a residual content of 20 µg/per kg fluoropolymer (= 10 ng/ml x 50 ml / 2.7 g).

6.5.2 precision of test method:

Recovery and repeatability (SD of the recovery; n=3) of the method were for the standard addition to the sample material at the concentration level of 42.7 ng/ml: 99 ± 7% for the extraction with methanol/water (80/20; v/v) and 101 ± 8% for the extraction with methanol/water

containing 0.1% of ammonia.

6.5.3 recovery: 99-101%, see 6.5.2.

6.5.4 other information: -

6.6 results: [redacted] Fluorolink 7800:7850 [redacted] Fluorolink 7800:7850 :

[redacted]

[redacted]

[redacted]

[redacted]

[redacted]

[redacted]

[redacted]

[redacted]
Fluorolink 7800:7850 [redacted] Fluorolink 7800:7850 [redacted]

[redacted] Fluorolink 7800:7850 [redacted]

Conclusion: The residual content of Fluorolink 7800:7850 in the sample tested was not-detectable or <20 µg/kg fluoropolymer.

6.7 calculated migration (worst case): The detection limit for the determination of Fluorolink 7800:7850 in samples of fluoropolymer was 20 µg/kg. From this result the worst case migration (M) of Fluorolink

7800:7850 to foodstuff can be calculated.
Therefore the following formula has to be applied:

M=(Q*A*Ld*D)/1000

Assuming that:

Q= conc in mg/kg in fluoropolymer= 0.020 mg/kg,
A= area of the food contact material in dm^2,
conventionally set at 600 cm^2,
Ld= thickness of food contact material = 0.04 cm and
D=fluoropolymer density is 2.17 g/cm3

The worst case migration into foodstuff is
M=(0.020*600*0.04*2.17)/1000=0.001 mg/kg or <1 µg/kg into food.

6.8 residual content versus specific migration: -

7 MICROBIOLOGICAL PROPERTIES OF SUBSTANCE

7.1 Is the substance used as an anti-microbial agent? NO

8. TOXICOLOGICAL DATA

8.1 Genotoxicity

8.1.1 Gene mutation in bacteria:

Type of test: Ames
Bacteria: *S. typhimurium* TA1535, TA1537, TA98, TA100 & *E. Coli* WP2 uvrA
Year: 2006
GLP: YES
Test Substance: Fluorolink 7850
Dose Levels: Main Assay I & Main Assay II ranging from 1.53 ug/plate to 5000 ug/plate
Method: the study was designed in compliance with EEC Council Directive 2000/32, Annex 4D; OECD 471; ICH S2A
Results: The test item does not induce reverse mutation in both S. typhimurium and E. coli both in absence and presence of metabolic activation S9
Body responsible for the test: RTC – Research Toxicology Centre Rome
Report number: RTC52400

Ref: *Annex 7*

8.1.2	**In vitro mammalian cell gene mutation test:**	Type of test: Mutation in L5178Y TK+/- Mouse Lymphoma cells (Fluctuation method) Year: 2006 GLP: YES Test Substance: Fluorolink 7850 Dose Levels (ug/ml): Assay I (-S9) 125, 100, 80, 40, 20, 10, 5– Assay I (+S9) 150, 100, 75, 37.5, 18.8, 9.38 – Assay II (-S9) 160, 120, 80, 40, 20, 10 – Assay II (+S9) 120, 100, 83.3, 69.4, 57.9 Method: the study was designed in compliance with EEC Council Directive 2000/32, Annex 4E; OECD 476; ICH S2A, ICH S2B Results: Fluorolink 7850 does not induce mutation in mouse lymphoma L5178Y cells after *in vitro* treatment both in absence and presence of S9 metabolic activation Body responsible for the test: RTC – Research Toxicology Centre Rome Report number: RTC52410 Ref: *Annex 8*
8.1.3	**In vitro mammalian chromosome aberration test :**	Type of test: Chromosome aberration in Chinese Hamster Ovary cells in vitro Year: 2006 GLP: YES Test Substance: Fluorolink 7850 Dose Levels: Experiment I – 313, 156 and 78.1 ug/ml +/- S9; Experiment II – 300, 200, 133 ug/ml +/- S9 Method: the study was designed in compliance with EEC Council Directive 2000/32, Annex 4A; OECD 473 Results: Fluorolink 7850 does not induce chromosomal aberrations in CHO cells after *in vitro* treatment in the absence or presence of S9 metabolic activation Body responsible for the test: RTC – Research Toxicology Centre Rome Report number: RTC52420 Ref: *Annex 9*
8.1.4	**Other information:**	
8.2	**General toxicity**	
8.2.1	**Subchronic (90d) oral toxicity:**	
8.2.2	**Chronic toxicity/ carcinogenicity:**	
8.2.3	**Reproduction/teratogenicity:**	
8.2.4	**Other information:**	
8.3	**Metabolism**	

8.3.1 Absorption, distribution, biotransformation and excretion:

8.3.2 Accumulation in man:

8.3.3 Other information:

8.4 Miscellaneous

8.4.1 Effects on immune system:

8.4.2 Neurotoxicity:

8.4.3 Introduction on peroxisome proliferation:

8.4.4 Other information:

LIST OF ANNEXES

Annex 1: Production process for FLK 7850 – Confidential information, *P-SDS item 1.3.6, 1.3.8 1.3.9 and 1.3.11;*

Annex 2: Analytical and Structural Characterization – Analytical report No. 60712, Bollate 24/01/2007; Confidential information, - Method of Analysis PF 29/151- PF30/83 and PF30/82 - *P-SDS item 1.3.7, 1.3.11, 1.3.12 and 1.3.13;*

Annex 3: NMR spectrum, *P-SDS item 1.3.14;*

Annex 4: TGA curve Fluorolink 7850 – [redacted], *P-SDS item 2.1.3, 2.2.3;*

Annex 5: Determination of the K_a, *P-SDS item 2.2.1;*

Annex 6: TNO migration report V7054: Migration study of Fluorolink 7800:7850 – 6 October, 2006, *P-SDS item 5 and 6;*

Annex 7: RTC report no. 52400: Fluorolink 7850 – Bacterial Mutation Assay, 15 September 2006, *P-SDS item 8.1.1;*

Annex 8: RTC report no. 52410: Fluorolink 7850 – Mutations in L5178Y $TK^{+/-}$ mouse lymphoma cells (fluctuations method), 13 December 2006, *P-SDS item 8.1.2;*

Annex 9: RTC report no. 52420: Fluorolink 7850 – Chromosome aberrations in Chinese Hamster Ovary cells in vitro, September 2006, *P-SDS item 8.1.3.*

Annex 10: Additional information provided by Solvay Solexis – Bollate 23/05/2008

Annex 11: TNO migration report V8128: Characterization of migrating oligomers and reaction products of Fluorolink 7800:7850 from a fluoropolymer sample – June 2008

Annex 12: GC-MS chromatograms and mass spectra