

GfA mbH • Postfach 41 01 28 • D-48065 Münster-Roxel

Environmental Protection Agency (EPA)
Mr. Colman Concannon
McCumiskey House
Richview
Clonskeagh Road
Dublin 14

Ireland

October 30, 2007

Our ref.: 61243-005 P02-139-Kr

Please include in all correspondences

Your ref.: ./.

Project manager: Dr. R. Grümping / Dipl.-Ing. M. de Hoogd
Direct dial: -154 / -243

**Analysis of 5 composite cow's milk samples for brominated flame retardants (BFRs) and
PBDF/Ds;**

Your order PO 013517 dated July 19, 2007

Dear Mr. Concannon,

Enclosed please find our test report concerning the investigations mentioned above.

If you have any questions please don't hesitate to contact us.

Best regards


Dr. R. Grümping**GfA**Gesellschaft für Arbeitsplatz-
und Umweltanalytik mbHOtto-Hahn-Straße 22
D-48161 Münster-RoxelT | +49 (0) 25 34 / 807-0
F | +49 (0) 25 34 / 807-110Postfach 41 01 28
D-48065 Münster-Roxelwelcome@gfa-ms.de
www.gfa-ms.deNORD / LB
Konto 135 025 799
BLZ 250 500 00DEUTSCHES
AKKREDITIERUNGSSYSTEM
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Akkreditiert nach DIN EN ISO/IEC 17025:2005

Test report

61243-005 P02139

Client: Environmental Protection Agency (EPA)
McCumiskey House
Richview
Clonskeagh Road
Dublin 14
Ireland

Order dated: July 18, 2007

Sample: Cow's milk samples, details see table

Sample-No.	Client's sample characterization	GfA sample No.
Sample 1 (B1, B2, B14)	Cow milk	7N338501
Sample 2 (A5, A8, A9)	Cow milk	7N338502
Sample 3 (A7, A15, A24)	Cow milk	7N338503
Sample 4 (A3, A20, A23)	Cow milk	7N338504
Sample 5 (A11, A19, A25)	Cow milk	7N338505

Testing: Analysis for polybrominated Diphenylethers (PBDE), polybrominated Biphenyls (PBBs), Hexabromocyclododecane (HBCD), Tetrabromobisphenol A (TBBPA) and polybrominated Dibenzofurans and Dibenzodioxins (PBDF/D).

Sampling: The samples were sent to GfA by the client.

Sample entry: July 20, 2007

Test method: **Sample preparation (BFRs)**
Pooling of three individual milk samples to a composite sample; Freeze drying; Homogenisation; Addition of $^{13}\text{C}_{12}$ -labelled internal PBDE standards ($^{13}\text{C}_{12}$ -TriBDE, $^{13}\text{C}_{12}$ -TetraBDE, $^{13}\text{C}_{12}$ -PentaBDE, $^{13}\text{C}_{12}$ -HexaBDE, $^{13}\text{C}_{12}$ -HeptaBDE, $^{13}\text{C}_{12}$ -DecaBDE), a $^{13}\text{C}_{12}$ -HBCD standard (γ -HBCD) and a $^{13}\text{C}_{12}$ -TBBPA standard to the dried sample material; ASE extraction of a representative sample amount by means of Hexane/Dichloromethane/Methanol; Gravimetric determination of the fat content after evaporation of the solvents.

PBDE, PBB and HBCD analysis:

Clean-up by liquid/solid chromatography; HRGC/LRMS analysis; Quantitative determination of PBDEs and PBBs by means of the internal $^{13}\text{C}_{12}$ -labelled PBDE standards and of HBCD by means of the $^{13}\text{C}_{12}$ -HBCD standard (Isotope dilution method).

TBBPA analysis:

Treatment of an extract portion by means of sulphuric acid; Derivatisation; HRGC/LRMS analysis; Quantitative determination by means of the internal $^{13}\text{C}_{12}$ -labelled TBBPA standard (Isotope dilution method).

Sample preparation (PBDF/Ds)

Pooling of three individual milk samples to a composite sample; Freeze drying; Homogenisation; ASE extraction of a representative sample amount by means of Hexane/Dichloromethane/Methanol. Addition of ten $^{13}\text{C}_{12}$ -labelled internal Tri- through HeptaBDF/D standards prior to extraction. Gravimetric determination of the fat content after evaporation of the solvents.

PBDF/D analysis:

For the PBDF/D analysis the solution was cleaned-up by multi-step liquid/solid chromatography. Prior to the gas chromatographic analysis, a further ^{13}C -labelled PCDF/D standard was added to the PBDF/D fraction for the determination of the recovery of the internal standards.

A capillary gas chromatograph (HRGC, HP 5890) equipped with a DB1 column coupled with a high resolution mass spectrometer (HRMS, VG-AutoSpec) was used for the PCDF/D analysis. The quantitative determination of native Tri- through HeptaBDF/Ds was achieved via the corresponding $^{13}\text{C}_{12}$ -labelled internal standards (Isotope dilution method; QMA504-171; DIN EN ISO/IEC 17025:2005 accredited method).

Start of testing: July 20, 2007

End of testing: October 26, 2007

Results: The results of the analysis of the samples are shown in the Tables 01 to 09.

Remarks: None

Tab. 01: Results of the analysis of composite milk samples for PBDEs; the results refer to the fat-weight and to the fresh-weight

Client's sample characterisation	Sample 1 (B1, B2, B14)		Sample 2 (A5, A8, A9)	
GfA Sample No.	7N338501		7N338502	
Unit	ng/kg fresh-weight	ng/kg fat-weight	ng/kg fresh-weight	ng/kg fat-weight
PBDEs				
2,2',4-TriBDE (BDE-17)	< 0,1	< 3,0	< 0,1	< 3,0
2,4,4'-TriBDE (BDE-28)	< 0,1	< 3,0	< 0,1	< 3,0
2,2',4,5'-TetraBDE (BDE-49)	< 0,2	< 5,0	< 0,2	< 5,0
2,3',4',6-TetraBDE (BDE-71)	< 0,2	< 5,0	< 0,2	< 5,0
2,2',4,4'-TetraBDE (BDE-47)	2,3	64,8	2,1	55,1
2,3',4,4'-TetraBDE (BDE-66)	< 0,2	< 5,0	< 0,2	< 5,0
3,3',4,4'-TetraBDE (BDE-77)	< 0,2	< 5,0	< 0,2	< 5,0
2,2',4,4',6-PentaBDE (BDE-100)	0,3	7,4	0,3	6,7
2,3',4,4',6-PentaBDE (BDE-119)	< 0,3	< 8,0	< 0,3	< 8,0
2,2',4,4',5-PentaBDE (BDE-99)	1,9	53,3	1,8	48,8
2,2',3,4,4'-PentaBDE (BDE-85)	< 0,3	< 8,0	< 0,3	< 8,0
3,3',4,4',5-PentaBDE (BDE-126)	< 0,3	< 8,0	< 0,3	< 8,0
2,2',4,4',5,6'-HexaBDE (BDE-154)	< 0,4	< 10,0	< 0,4	< 10,0
2,2',4,4',5,5'-HexaBDE (BDE-153)	0,4	12,4	0,5	12,7
2,2',3,4,4',5'-HexaBDE (BDE-138)	< 0,4	< 10,0	< 0,4	< 10,0
2,2',3',4,4',5,6'-HeptaBDE (BDE-183)	< 1,0	< 20,0	< 1,0	< 20,0
DecaBDE (BDE-209)	< 50	< 1000	< 50	< 1000
Sum of PBDEs (excl. LOQ)	4,9	137,9	4,7	123,3
HBCD (Sum of α -, β - and γ -HBCD)	< 20	< 500	< 20	< 500
TBBPA	< 20	< 500	< 20	< 500

< : Concentration below the indicated limit of quantification (LOQ)

ND: Not determined since none of the corresponding congeners was above the LOQ

Tab. 02: Results of the analysis of composite milk samples for PBDEs; the results refer to the fat-weight and to the fresh-weight

Client's sample characterisation	Sample 3 (A7, A15, A24)		Sample 4 (A3, A20, A23)	
GfA Sample No.	7N338503		7N338504	
Unit	ng/kg fresh-weight	ng/kg fat-weight	ng/kg fresh-weight	ng/kg fat-weight
PBDEs				
2,2',4'-TriBDE (BDE-17)	< 0,1	< 3,0	< 0,1	< 3,0
2,4,4'-TriBDE (BDE-28)	< 0,1	< 3,0	< 0,1	< 3,0
2,2',4,5'-TetraBDE (BDE-49)	< 0,2	< 5,0	< 0,2	< 5,0
2,3',4',6'-TetraBDE (BDE-71)	< 0,2	< 5,0	< 0,2	< 5,0
2,2',4,4'-TetraBDE (BDE-47)	1,7	44,8	2,5	63,2
2,3',4,4'-TetraBDE (BDE-66)	< 0,2	< 5,0	< 0,2	< 5,0
3,3',4,4'-TetraBDE (BDE-77)	< 0,2	< 5,0	< 0,2	< 5,0
2,2',4,4',6'-PentaBDE (BDE-100)	< 0,3	< 8,0	< 0,3	< 8,0
2,3',4,4',6'-PentaBDE (BDE-119)	< 0,3	< 8,0	< 0,3	< 8,0
2,2',4,4',5'-PentaBDE (BDE-99)	1,5	39,4	2,0	48,9
2,2',3,4,4'-PentaBDE (BDE-85)	< 0,3	< 8,0	< 0,3	< 8,0
3,3',4,4',5'-PentaBDE (BDE-126)	< 0,3	< 8,0	< 0,3	< 8,0
2,2',4,4',5,6'-HexaBDE (BDE-154)	< 0,4	< 10,0	< 0,4	< 10,0
2,2',4,4',5,5'-HexaBDE (BDE-153)	0,4	10,3	0,6	14,4
2,2',3,4,4',5'-HexaBDE (BDE-138)	< 0,4	< 10,0	< 0,4	< 10,0
2,2',3',4,4',5,6'-HeptaBDE (BDE-183)	< 1,0	< 20,0	< 1,0	< 20,0
DecaBDE (BDE-209)	< 50,0	< 1000	< 50,0	< 1000
Sum of PBDEs (excl. LOQ)	3,6	94,5	5,1	126,5
HBCD (Sum of α -, β - and γ -HBCD)	< 20	< 500	< 20	< 500
TBBPA	< 20	< 500	< 20	< 500

< : Concentration below the indicated limit of quantification (LOQ)

ND: Not determined since none of the corresponding congeners was above the LOQ

Tab. 03: Results of the analysis of a composite milk sample for PBDEs; the results refer to the fat-weight and to the fresh-weight

Client's sample characterisation	Sample 5 (A11, A19, A25)	
GfA Sample No.	7N338505	
Unit	ng/kg fresh-weight	ng/kg fat-weight
PBDEs		
2,2',4-TriBDE (BDE-17)	< 0,1	< 3,0
2,4,4'-TriBDE (BDE-28)	< 0,1	< 3,0
2,2',4,5'-TetraBDE (BDE-49)	< 0,2	< 5,0
2,3',4',6-TetraBDE (BDE-71)	< 0,2	< 5,0
2,2',4,4'-TetraBDE (BDE-47)	3,8	99,7
2,3',4,4'-TetraBDE (BDE-66)	< 0,2	< 5,0
3,3',4,4'-TetraBDE (BDE-77)	< 0,2	< 5,0
2,2',4,4',6-PentaBDE (BDE-100)	0,6	14,7
2,3',4,4',6-PentaBDE (BDE-119)	< 0,3	< 8,0
2,2',4,4',5-PentaBDE (BDE-99)	5,2	136
2,2',3,4,4'-PentaBDE (BDE-85)	< 0,3	< 8,0
3,3',4,4',5-PentaBDE (BDE-126)	< 0,3	< 8,0
2,2',4,4',5,6'-HexaBDE (BDE-154)	< 0,4	< 10,0
2,2',4,4',5,5'-HexaBDE (BDE-153)	1,1	28,8
2,2',3,4,4',5'-HexaBDE (BDE-138)	< 0,4	< 10,0
2,2',3',4,4',5,6'-HeptaBDE (BDE-183)	< 1,0	< 20,0
DecaBDE (BDE-209)	< 50,0	< 1000
Sum of PBDEs (excl. LOQ)	10,7	279,2
HBCD (Sum of α -, β - and γ -HBCD)	< 20	< 500
TBBPA	< 20	< 500

< : Concentration below the indicated limit of quantification (LOQ)

ND: Not determined since none of the corresponding congeners was above the LOQ

Tab. 04: Results of the analysis of composite milk samples for PBBs; the results refer to the fat-weight and to the fresh-weight

Client's sample characterisation	Sample 1 (B1, B2, B14)		Sample 2 (A5, A8, A9)	
GfA Sample No.	7N338501		7N338502	
Unit	ng/kg fresh-weight	ng/kg fat-weight	ng/kg fresh-weight	ng/kg fat-weight
PBBs				
2,2',5,5'-TetraBB	< 0,2	< 5,0	< 0,2	< 5,0
Total other TetraBB	ND	ND	ND	ND
Total TetraBB	ND	ND	ND	ND
2,2',4,5,5'-PentaBB	< 0,3	< 8,0	< 0,3	< 8,0
Total other PentaBB	ND	ND	ND	ND
Total PentaBB	ND	ND	ND	ND
2,2',4,4',5,5'-HexaBB	< 0,4	< 10,0	< 0,4	< 10,0
Total other HexaBB	ND	ND	ND	ND
Total HexaBB	ND	ND	ND	ND
HeptaBB	< 1,0	< 25,0	< 1,0	< 25,0
Total other HeptaBB	ND	ND	ND	ND
Total HeptaBB	ND	ND	ND	ND
OctaBB	< 2,0	< 50,0	< 2,0	< 50,0
Total OctaBB	ND	ND	ND	ND
NonaBB	< 2,0	< 50,0	< 2,0	< 50,0
Total NonaBB	ND	ND	ND	ND
DecaBB	< 3,0	< 60,0	< 3,0	< 60,0

< : Concentration below the indicated limit of quantification (LOQ)

ND: Not determined since none of the corresponding congeners was above the LOQ

Tab. 05: Results of the analysis of composite milk samples for PBBs; the results refer to the fat-weight and to the fresh-weight

Client's sample characterisation	Sample 3 (A7, A15, A24)		Sample 4 (A3, A20, A23)	
GfA Sample No.	7N338503		7N338504	
Unit	ng/kg fresh-weight	ng/kg fat-weight	ng/kg fresh-weight	ng/kg fat-weight
PBBs				
2,2',5,5'-TetraBB	< 0,2	< 5,0	< 0,2	< 5,0
Total other TetraBB	ND	ND	ND	ND
Total TetraBB	ND	ND	ND	ND
2,2',4,5,5'-PentaBB	< 0,3	< 8,0	< 0,3	< 8,0
Total other PentaBB	ND	ND	ND	ND
Total PentaBB	ND	ND	ND	ND
2,2',4,4',5,5'-HexaBB	< 0,4	< 10,0	< 0,4	< 10,0
Total other HexaBB	ND	ND	ND	ND
Total HexaBB	ND	ND	ND	ND
HeptaBB	< 1,0	< 25,0	< 1,0	< 25,0
Total other HeptaBB	ND	ND	ND	ND
Total HeptaBB	ND	ND	ND	ND
OctaBB	< 2,0	< 50,0	< 2,0	< 50,0
Total OctaBB	ND	ND	ND	ND
NonaBB	< 2,0	< 50,0	< 2,0	< 50,0
Total NonaBB	ND	ND	ND	ND
DecaBB	< 3,0	< 60,0	< 3,0	< 60,0

< : Concentration below the indicated limit of quantification (LOQ)

ND: Not determined since none of the corresponding congeners was above the LOQ

Tab. 06: Results of the analysis of a composite milk sample for PBBs; the results refer to the fat-weight and to the fresh-weight

Client's sample characterisation	Sample 5 (A11, A19, A25)	
GfA Sample No.	7N338505	
Unit	ng/kg fresh-weight	ng/kg fat-weight
PBBs		
2,2',5,5'-TetraBB	< 0,2	< 5,0
Total other TetraBB	ND	ND
Total TetraBB	ND	ND
2,2',4,5,5'-PentaBB	< 0,3	< 8,0
Total other PentaBB	ND	ND
Total PentaBB	ND	ND
2,2',4,4',5,5'-HexaBB	< 0,4	< 10,0
Total other HexaBB	ND	ND
Total HexaBB	ND	ND
HeptaBB	< 1,0	< 25,0
Total other HeptaBB	ND	ND
Total HeptaBB	ND	ND
OctaBB	< 2,0	< 50,0
Total OctaBB	ND	ND
NonaBB	< 2,0	< 50,0
Total NonaBB	ND	ND
DecaBB	< 3,0	< 60,0

< : Concentration below the indicated limit of quantification (LOQ)

ND: Not determined since none of the corresponding congeners was above the LOQ

Tab. 07: Results of the analysis of composite milk samples for PBDF/Ds; the results refer to the fat-weight and to the fresh-weight

Client's sample characterisation	Sample 1 (B1, B2, B14)		Sample 2 (A5, A8, A9)	
GfA Sample No.	7N338501		7N338502	
Fat content [%]	3,6		3,8	
Unit	pg/g fresh-weight	pg/g fat-weight	pg/g fresh-weight	pg/g fat-weight
PBDF				
238-TriBDF ^b	< 0,003	< 0,08	< 0,003	< 0,08
2378-TetraBDF ^b	< 0,002	< 0,05	< 0,001	< 0,03
12378-PentaBDF ^b	< 0,004	< 0,10	< 0,001	< 0,03
23478-PentaBDF ^b	< 0,006	< 0,16	< 0,001	< 0,03
123478-/123678-HexaBDF ^{a, b}	< 0,001	< 0,03	< 0,001	< 0,03
1234678-HeptaBDF ^b	< 0,13 ^e	< 3,64 ^e	< 0,008	< 0,22
PBDD				
237-TriBDD ^b	< 0,003	< 0,08	< 0,004	< 0,09
2378-TetraBDD ^b	< 0,001	< 0,04	< 0,001	< 0,03
12378-PentaBDD ^b	< 0,001	< 0,03	< 0,001	< 0,03
123478/123678-HexaBDD ^{a, b}	< 0,03	< 0,85	< 0,01	< 0,30
123789-HexaBDD ^b	< 0,01	< 0,32	< 0,01	< 0,30
WHO-PBDD/F-TEQ excl. LOQ [c]	ND	ND	ND	ND
WHO-PBDD/F-TEQ incl. LOQ [d]	0,011	0,296	0,005	0,133

< : Concentration below the indicated limit of quantification (LOQ)

ND: Not determined since none of the corresponding congeners was above the LOQ

[a] : Not separated on DB1 as GC stationary phase

[b] : Maximum value, coelution with other isomers cannot be excluded

[c] : TEQ-value calculated by including the quantified congeners only

[d] : TEQ-value calculated by including the non-quantified congeners by taking the full value of their LOQ

[e] : Higher detection limit due to interferences

Tab. 08: Results of the analysis of composite milk samples for PBDF/Ds; the results refer to the fat-weight and to the fresh-weight

Client's sample characterisation	Sample 3 (A7, A15, A24)		Sample 4 (A3, A20, A23)	
GfA Sample No.	7N338503		7N338504	
Fat content [%]	3,9		3,9	
Unit	pg/g fresh-weight	pg/g fat-weight	pg/g fresh-weight	pg/g fat-weight
PBDF				
238-TriBDF ^b	< 0,003	< 0,08	< 0,003	< 0,08
2378-TetraBDF ^b	< 0,002	< 0,04	< 0,001	< 0,03
12378-PentaBDF ^b	< 0,001	< 0,03	< 0,001	< 0,03
23478-PentaBDF ^b	< 0,005	< 0,13	< 0,001	< 0,04
123478-/123678-HexaBDF ^{a, b}	< 0,001	< 0,03	< 0,001	< 0,03
1234678-HeptaBDF ^b	< 0,02	< 0,48	< 0,008	< 0,19
PBDD				
237-TriBDD ^b	< 0,003	< 0,08	< 0,003	< 0,08
2378-TetraBDD ^b	< 0,001	< 0,03	< 0,001	< 0,03
12378-PentaBDD ^b	< 0,001	< 0,03	< 0,001	< 0,03
123478/123678-HexaBDD ^{a, b}	< 0,01	< 0,29	< 0,01	< 0,29
123789-HexaBDD ^b	< 0,01	< 0,29	< 0,01	< 0,29
WHO-PBDD/F-TEQ excl. LOQ [c]	ND	ND	ND	ND
WHO-PBDD/F-TEQ incl. LOQ [d]	0,007	0,183	0,005	0,132

< : Concentration below the indicated limit of quantification (LOQ)

ND: Not determined since none of the corresponding congeners was above the LOQ

[a] : Not separated on DB1 as GC stationary phase

[b] : Maximum value, coelution with other isomers cannot be excluded

[c] : TEQ-value calculated by including the quantified congeners only

[d] : TEQ-value calculated by including the non-quantified congeners by taking the full value of their LOQ

Tab. 09: Results of the analysis of a composite milk sample for PBDF/Ds; the results refer to the fat-weight and to the fresh-weight

Client's sample characterisation	Sample 5 (A11, A19, A25)	
GfA Sample No.	7N338505	
Fat content [%]	3,8	
Unit	pg/g fresh-weight	pg/g fat-weight
PBDF		
238-TriBDF ^b	< 0,003	< 0,08
2378-TetraBDF ^b	< 0,001	< 0,03
12378-PentaBDF ^b	< 0,001	< 0,03
23478-PentaBDF ^b	< 0,002	< 0,05
123478-/123678-HexaBDF ^{a, b}	< 0,001	< 0,03
1234678-HeptaBDF ^b	< 0,02	< 0,60
PBDD		
237-TriBDD ^b	< 0,003	< 0,08
2378-TetraBDD ^b	< 0,001	< 0,03
12378-PentaBDD ^b	< 0,001	< 0,03
123478/123678-HexaBDD ^{a, b}	< 0,01	< 0,29
123789-HexaBDD ^b	< 0,01	< 0,29
WHO-PBDD/F-TEQ excl. LOQ [c]	ND	ND
WHO-PBDD/F-TEQ incl. LOQ [d]	0,006	0,145

< : Concentration below the indicated limit of quantification (LOQ)

ND: Not determined since none of the corresponding congeners was above the LOQ

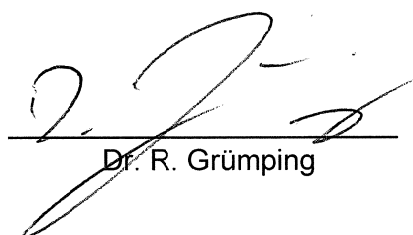
[a] : Not separated on DB1 as GC stationary phase

[b] : Maximum value, coelution with other isomers cannot be excluded

[c] : TEQ-value calculated by including the quantified congeners only

[d] : TEQ-value calculated by including the non-quantified congeners by taking the full value of their LOQ

October 30, 2007



Dr. R. Grümping

Remark: The test results relate only to the items tested. Extracts of the report shall not be reproduced without written approval of the GfA mbH.