Test report

61243-011-10G8016

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

Order dated: July 28, 2010

Sample: Cow's milk samples, details see table

Sample-No.	GfA sample No.	Client's sample characterization
Sample 1 (B1, B2, B14)	10G8016.1	Cow milk
Sample 2 (A5, A8, A9)	10G8016.2	Cow milk
Sample 3 (A7, A15, A24)	10G8016.3	Cow milk
Sample 4 (A3, A20, A23)	10G8016.4	Cow milk
Sample 5 (A11, A19, A25)	10G8016.5	Cow milk

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

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

Sample entry: July 30, 2010

Test method:

Sample preparation (BFRs)
Pooling of three individual milk samples to a composite sample; Freeze drying; Homogenisation; Addition of $^{13}C_{12}$-labelled internal PBDE standards ($^{13}C_{12}$-TriBDE, $^{13}C_{12}$-TetraBDE, $^{13}C_{12}$-PentaBDE, $^{13}C_{12}$-HexaBDE, $^{13}C_{12}$-HeptaBDE, $^{13}C_{12}$-OctaBDE, $^{13}C_{12}$-NonaBDE, $^{13}C_{12}$-DecaBDE), a $^{13}C_{12}$-HBCD standard (γ-HBCD) and a $^{13}C_{12}$-TBBPA standard to the dried sample material; soxhlet extraction of a representative sample amount by means of hexane/acetone (4:1); Gravimetrical 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 $^{13}C_{12}$-labelled PBDE standards and of HBCD by means of the $^{13}C_{12}$-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 $^{13}C_{12}$-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 $^{13}C_{12}$-labelled internal Tri- through HeptaBDF/D standards prior to extraction. Gravimetrical 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 $^{13}C_{12}$-labelled internal standards (Isotope dilution method; QMA504-341; DIN EN ISO/IEC 17025:2005 accredited method).

Start of testing: July 30, 2010

End of testing: October 28, 2010

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

Remarks: no remarks

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 GfA Sample No.	Sample 1 (B1, B2, B14) 10G8016.1		Sample 2 (A5, A8, A9) 10G8016.2	
Unit	ng/kg fresh-weight	ng/kg fat-weight	ng/kg fresh-weight	ng/kg fat-weight
PBDEs				
2,2',4-TriBDE (BDE-17)	< 0,2	< 4,0	< 0,2	< 4,0
2,4,4'-TriBDE (BDE-28)	< 0,2	< 4,0	< 0,2	< 4,0
2,2',4,5'-TetraBDE (BDE-49)	< 0,6	< 20,0	< 0,6	< 20,0
2,3',4',6-TetraBDE (BDE-71)	< 0,6	< 20,0	< 0,6	< 20,0
2,2',4,4'-TetraBDE (BDE-47)	11,2	266,7	2,2	53,2
2,3',4,4'-TetraBDE (BDE-66)	< 0,6	< 20,0	< 0,6	< 20,0
3,3',4,4'-TetraBDE (BDE-77)	< 0,6	< 20,0	< 0,6	< 20,0
2,2',4,4',6-PentaBDE (BDE-100)	< 0,8	< 20,0	< 0,8	< 20,0
2,3',4,4',6-PentaBDE (BDE-119)	< 0,8	< 20,0	< 0,8	< 20,0
2,2',4,4',5-PentaBDE (BDE-99)	12,2	291,2	3,0	73,6
2,2',3,4,4'-PentaBDE (BDE-85)	< 0,8	< 20,0	< 0,8	< 20,0
3,3',4,4',5-PentaBDE (BDE-126)	< 0,8	< 20,0	< 0,8	< 20,0
2,2',4,4',5,6'-HexaBDE (BDE-154)	< 0,8	< 20,0	< 0,8	< 20,0
2,2',4,4',5,5'-HexaBDE (BDE-153)	3,72	88,5	1,2	29,2
2,2',3,4,4',5'-HexaBDE (BDE-138)	< 0,8	< 20,0	< 0,8	< 20,0
2,2',3',4,4',5,6'-HeptaBDE (BDE-183)	< 2,0	< 40,0	< 2,0	< 40,0
DecaBDE (BDE-209)	< 25,0	< 500	< 25,0	< 500
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 GfA Sample No.	Sample 3 (A7, A15, A24) 10G8016.3		Sample 4 (A3, A20, A23) 10G8016.4	
Unit	ng/kg fresh-weight	ng/kg fat-weight	ng/kg fresh-weight	ng/kg fat-weight
PBDEs				
2,2',4-TriBDE (BDE-17)	< 0,2	< 4,0	< 0,3	< 9,0
2,4,4'-TriBDE (BDE-28)	< 0,2	< 4,0	< 0,3	< 9,0
2,2',4,5'-TetraBDE (BDE-49)	< 0,6	< 20,0	< 0,9	< 20,0
2,3',4',6-TetraBDE (BDE-71)	< 0,6	< 20,0	< 0,9	< 20,0
2,2',4,4'-TetraBDE (BDE-47)	1,1	26,9	1,8	46,0
2,3',4,4'-TetraBDE (BDE-66)	< 0,6	< 20,0	< 0,9	< 20,0
3,3',4,4'-TetraBDE (BDE-77)	< 0,6	< 20,0	< 0,9	< 20,0
2,2',4,4',6-PentaBDE (BDE-100)	< 0,8	< 20,0	< 0,8	< 20,0
2,3',4,4',6-PentaBDE (BDE-119)	< 0,8	< 20,0	< 0,9	< 20,0
2,2',4,4',5-PentaBDE (BDE-99)	1,0	24,7	1,6	41,2
2,2',3,4,4'-PentaBDE (BDE-85)	< 0,8	< 20,0	< 0,9	< 20,0
3,3',4,4',5-PentaBDE (BDE-126)	< 0,8	< 20,0	< 0,9	< 20,0
2,2',4,4',5,6'-HexaBDE (BDE-154)	< 1,0	< 30,0	< 1,0	< 30,0
2,2',4,4',5,5'-HexaBDE (BDE-153)	< 1,0	< 30,0	< 1,0	< 30,0
2,2',3,4,4',5'-HexaBDE (BDE-138)	< 1,0	< 30,0	< 1,0	< 30,0
2,2',3',4,4',5,6'-HeptaBDE (BDE-183)	< 2,0	< 40,0	< 2,0	< 40,0
DecaBDE (BDE-209)	< 25,0	< 500	< 25,0	< 500
HBCD (Sum of α-, β- and γ-HBCD)	**28,0**	**678**	**< 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 GfA Sample No.	Sample 5 (A11, A19, A25) 10G8016.5	
Unit	ng/kg fresh-weight	ng/kg fat-weight
PBDEs		
2,2',4-TriBDE (BDE-17)	< 0,3	< 6,0
2,4,4'-TriBDE (BDE-28)	< 0,3	< 6,0
2,2',4,5'-TetraBDE (BDE-49)	< 0,3	< 6,0
2,3',4',6-TetraBDE (BDE-71)	< 0,3	< 6,0
2,2',4,4'-TetraBDE (BDE-47)	1,3	29,1
2,3',4,4'-TetraBDE (BDE-66)	< 0,3	< 6,0
3,3',4,4'-TetraBDE (BDE-77)	< 0,3	< 6,0
2,2',4,4',6-PentaBDE (BDE-100)	< 0,8	< 20,0
2,3',4,4',6-PentaBDE (BDE-119)	< 0,8	< 20,0
2,2',4,4',5-PentaBDE (BDE-99)	1,7	40,1
2,2',3,4,4'-PentaBDE (BDE-85)	< 0,8	< 20,0
3,3',4,4',5-PentaBDE (BDE-126)	< 0,8	< 20,0
2,2',4,4',5,6'-HexaBDE (BDE-154)	< 1,0	< 30,0
2,2',4,4',5,5'-HexaBDE (BDE-153)	< 1,0	< 30,0
2,2',3,4,4',5'-HexaBDE (BDE-138)	< 1,0	< 30,0
2,2',3',4,4',5,6'-HeptaBDE (BDE-183)	< 2,0	< 40,0
DecaBDE (BDE-209)	< 25,0	< 500
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 GfA Sample No.	Sample 1 (B1, B2, B14) 10G8016.1		Sample 2 (A5, A8, A9) 10G8016.2	
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 GfA Sample No.	Sample 3 (A7, A15, A24) 10G8016.3		Sample 4 (A3, A20, A23) 10G8016.4	
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 GfA Sample No.	Sample 5 (A11, A19, A25) 10G8016.5	
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.	10G8016.1		10G8016.2	
Fat content [%]	4,2		4,1	
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	< 0,002	< 0,04
12378-PentaBDF [b]	< 0,006	< 0,15	< 0,006	< 0,14
23478-PentaBDF [b]	< 0,006	< 0,14	< 0,006	< 0,14
123478-/123678-HexaBDF [a, b]	< 0,02	< 0,52	< 0,01	< 0,26
1234678-HeptaBDF [b]	< 0,08	< 1,90	< 0,08	< 1,95
PBDD				
237-TriBDD [b]	< 0,01	< 0,23	< 0,01	< 0,29
2378-TetraBDD [b]	< 0,001	< 0,03	< 0,001	< 0,03
12378-PentaBDD [b]	< 0,003	< 0,08	< 0,009	< 0,22
123478/123678-HexaBDD [a,b]	< 0,01	< 0,28	< 0,01	< 0,29
123789-HexaBDD [b]	< 0,02	< 0,50	< 0,02	< 0,46
WHO-PBDD/F-TEQ excl. LOQ [c]	ND	ND	ND	ND
WHO-PBDD/F-TEQ incl. LOQ [d]	0,01	0,33	0,02	0,45

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

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.	10G8016.3		10G8016.4	
Fat content [%]	4,1		4,0	
Unit	pg/g fresh-weight	pg/g fat-weight	pg/g fresh-weight	pg/g fat-weight
PBDF				
238-TriBDF [b]	< 0,004	< 0,1	< 0,004	< 0,10
2378-TetraBDF [b]	< 0,001	< 0,03	< 0,003	< 0,08
12378-PentaBDF [b]	< 0,007	< 0,17	< 0,007	< 0,17
23478-PentaBDF [b]	< 0,007	< 0,17	< 0,007	< 0,17
123478-/123678-HexaBDF [a, b]	< 0,01	< 0,34	< 0,01	< 0,28
1234678-HeptaBDF [b]	< 0,08	< 1,93	< 0,10	< 2,54
PBDD				
237-TriBDD [b]	< 0,006	< 0,14	< 0,004	< 0,10
2378-TetraBDD [b]	< 0,0005	< 0,01	< 0,001	< 0,03
12378-PentaBDD [b]	< 0,005	< 0,12	< 0,003	< 0,08
123478/123678-HexaBDD [a,b]	< 0,01	< 0,29	< 0,02	< 0,38
123789-HexaBDD [b]	< 0,02	< 0,43	< 0,03	< 0,65
WHO-PBDD/F-TEQ excl. LOQ [c]	ND	ND	ND	ND
WHO-PBDD/F-TEQ incl. LOQ [d]	0,01	0,35	0,01	0,36

< : 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 congeneres only
[d] : TEQ-value calculated by including the non-quantified congeneres 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 GfA Sample No.	Sample 5 (A11, A19, A25) 10G8016.5	
Fat content [%]	4,3	
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]	< 0,005	< 0,12
23478-PentaBDF [b]	< 0,005	< 0,12
123478-/123678-HexaBDF [a, b]	< 0,01	< 0,24
1234678-HeptaBDF [b]	< 0,08	< 1,91
PBDD		
237-TriBDD [b]	< 0,004	< 0,09
2378-TetraBDD [b]	< 0,0008	< 0,02
12378-PentaBDD [b]	< 0,002	< 0,05
123478/123678-HexaBDD [a,b]	< 0,01	< 0,29
123789-HexaBDD [b]	< 0,02	< 0,36
WHO-PBDD/F-TEQ excl. LOQ [c]	ND	ND
WHO-PBDD/F-TEQ incl. LOQ [d]	0,01	0,25

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

October 28, 2010

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Dr. Dieter Stegemann

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.

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Environmental Protection Agency (EPA)
Dr. Colman Concannon
McCumiskey House
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Ireland

October 28, 2010

Our ref.: 61243-011 P02-079-Kr
Please include in all correspondences

Your ref.: ./.
Project manager: Dr. D. Stegemann
Direct dial: -115

Analysis of 5 composite cow's milk samples for brominated flame retardants (BFRs) and PBDF/Ds;
Your order PO 030350 dated July 28, 2010

Dear Dr. Concannon,

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

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

Best regards

Dr. Dieter Stegemann