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Environmental Protection Agency (EPA)

Mr. Colman Concannon

McCumiskey House

Richview

Clonskeagh Road

Dublin 14

Ireland

March 28, 2007

Our ref.:

61243-004

P04-139-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 008889 dated July 27, 2006**

Dear Mr. Concannon,

Enclosed please find our test report including the revised data for BFRs and PBDF/Ds.

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

Best regards



Dr. R. Grümping

**GfA**

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DEUTSCHES  
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DAP-PL-1053.99

**DAP**

Akkreditiert nach DIN EN ISO/IEC 17025:2005

## Test report 61243-004 P04 139

### Correction of test report 61243-004 P03 139 dated December 15, 2006

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

**Order dated:** July 27, 2006

**Sample:** Cow's milk samples, details see table

Sample-No.	Client's sample characterization	GfA sample No.
Composite sample 1 (B1, B2, B14)	Cow milk	6N267101
Composite sample 2 (A5, A8, A9)	Cow milk	6N267102
Composite sample 3 (A7, A15, A24)	Cow milk	6N267103
Composite sample 4 (A3, A20, A23)	Cow milk	6N267104
Composite sample 5 (A11, A19, A25)	Cow milk	6N267105

**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 19, 2006

**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 ( $\gamma$ -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. 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  $^{13}\text{C}$ -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 27, 2006

**End of testing:** March 28, 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	Composite sample 1 (B1, B2, B14)		Composite sample 2 (A5, A8, A9)	
GfA Sample No.	6N267101		6N267102	
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	< 6,0	< 0,2	< 6,0
2,3',4',6-TetraBDE (BDE-71)	< 0,2	< 6,0	< 0,2	< 6,0
2,2',4,4'-TetraBDE (BDE-47)	3,1	81,9	2,9	80,4
2,3',4,4'-TetraBDE (BDE-66)	< 0,2	< 6,0	< 0,2	< 6,0
3,3',4,4'-TetraBDE (BDE-77)	< 0,2	< 6,0	< 0,2	< 6,0
2,2',4,4',6-PentaBDE (BDE-100)	0,4	9,3	0,4	9,7
2,3',4,4',6-PentaBDE (BDE-119)	< 0,3	< 9,0	< 0,3	< 9,0
2,2',4,4',5-PentaBDE (BDE-99)	3,7	97,9	3,3	90,4
2,2',3,4,4'-PentaBDE (BDE-85)	< 0,3	< 9,0	< 0,3	< 9,0
3,3',4,4',5-PentaBDE (BDE-126)	< 0,3	< 9,0	< 0,3	< 9,0
2,2',4,4',5,6'-HexaBDE (BDE-154)	< 0,4	< 12,0	< 0,4	< 12,0
2,2',4,4',5,5'-HexaBDE (BDE-153)	1,4	35,3	0,7	19,8
2,2',3,4,4',5'-HexaBDE (BDE-138)	< 0,4	< 12,0	< 0,4	< 12,0
2,2',3',4,4',5,6'-HeptaBDE (BDE-183)	< 1,0	< 30,0	< 1,0	< 30,0
DecaBDE (BDE-209)	< 50	< 1500	< 50	< 1500
Sum of PBDEs (excl. LOQ)	8,6	224	7,0	200
HBCD (Sum of $\alpha$ -, $\beta$ - and $\gamma$ -HBCD)	< 50	< 1500	< 50	< 1500
TBBPA	< 200	< 6000	< 200	< 6000

< : 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	Composite sample 3 (A7, A15, A24)		Composite sample 4 (A3, A20, A23)	
GfA Sample No.	6N267103		6N267104	
Unit	ng/kg fresh-weight	ng/kg fat-weight	ng/kg fresh-weight	ng/kg fat-weight
<b>PBDEs</b>				
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	< 6,0	< 0,2	< 6,0
2,3',4',6-TetraBDE (BDE-71)	< 0,2	< 6,0	< 0,2	< 6,0
2,2',4,4'-TetraBDE (BDE-47)	2,2	63,2	2,9	84,8
2,3',4,4'-TetraBDE (BDE-66)	< 0,2	< 6,0	< 0,2	< 6,0
3,3',4,4'-TetraBDE (BDE-77)	< 0,2	< 6,0	< 0,2	< 6,0
2,2',4,4',6-PentaBDE (BDE-100)	0,3	8,8	0,4	10,8
2,3',4,4',6-PentaBDE (BDE-119)	< 0,3	< 9,0	< 0,3	< 9,0
2,2',4,4',5-PentaBDE (BDE-99)	2,3	64,3	2,8	82,1
2,2',3,4,4'-PentaBDE (BDE-85)	< 0,3	< 9,0	< 0,3	< 9,0
3,3',4,4',5-PentaBDE (BDE-126)	< 0,3	< 9,0	< 0,3	< 9,0
2,2',4,4',5,6'-HexaBDE (BDE-154)	< 0,4	< 12,0	< 0,4	< 12,0
2,2',4,4',5,5'-HexaBDE (BDE-153)	0,5	13,9	0,7	17,5
2,2',3,4,4',5'-HexaBDE (BDE-138)	< 0,4	< 12,0	< 0,4	< 12,0
2,2',3',4,4',5,6'-HeptaBDE (BDE-183)	< 1,0	< 30,0	< 1,0	< 30,0
DecaBDE (BDE-209)	< 50	< 1500	< 50	< 1500
Sum of PBDEs (excl. LOQ)	5,3	150	6,8	195
HBCD (Sum of $\alpha$ -, $\beta$ - and $\gamma$ -HBCD)	< 50	< 1500	< 50	< 1500
TBBPA	< 200	< 6000	< 200	< 6000

< : 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	Composite sample 5 (A11, A19, A25)	
GfA Sample No.	6N267105	
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	< 6,0
2,3',4',6'-TetraBDE (BDE-71)	< 0,2	< 6,0
2,2',4,4'-TetraBDE (BDE-47)	3,6	93,0
2,3',4,4'-TetraBDE (BDE-66)	< 0,2	< 6,0
3,3',4,4'-TetraBDE (BDE-77)	< 0,2	< 6,0
2,2',4,4',6'-PentaBDE (BDE-100)	0,4	10,4
2,3',4,4',6'-PentaBDE (BDE-119)	< 0,3	< 9,0
2,2',4,4',5'-PentaBDE (BDE-99)	4,7	122
2,2',3,4,4'-PentaBDE (BDE-85)	< 0,3	< 9,0
3,3',4,4',5'-PentaBDE (BDE-126)	< 0,3	< 9,0
2,2',4,4',5,6'-HexaBDE (BDE-154)	< 0,4	< 12,0
2,2',4,4',5,5'-HexaBDE (BDE-153)	1,5	39,7
2,2',3,4,4',5'-HexaBDE (BDE-138)	< 0,4	< 12,0
2,2',3',4,4',5,6'-HeptaBDE (BDE-183)	< 1,0	< 30,0
DecaBDE (BDE-209)	< 50	< 1500
Sum of PBDEs (excl. LOQ)	10,2	265
HBCD (Sum of $\alpha$ -, $\beta$ - and $\gamma$ -HBCD)	< 50	< 1500
TBBPA	< 200	< 6000

< : 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	Composite sample 1 (B1, B2, B14)		Composite sample 2 (A5, A8, A9)	
GfA Sample No.	6N267101		6N267102	
Unit	ng/kg fresh-weight	ng/kg fat-weight	ng/kg fresh-weight	ng/kg fat-weight
<b>PBBs</b>				
2,2',5,5'-TetraBB	< 0,2	< 6,0	< 0,2	< 6,0
Total other TetraBB	ND	ND	ND	ND
<b>Total TetraBB</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>
2,2',4,5,5'-PentaBB	< 0,3	< 9,0	< 0,3	< 9,0
Total other PentaBB	ND	ND	ND	ND
<b>Total PentaBB</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>
2,2',4,4',5,5'-HexaBB	< 0,4	< 12	< 0,4	< 12
Total other HexaBB	ND	ND	ND	ND
<b>Total HexaBB</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>
HeptaBB	< 1,0	< 30	< 1,0	< 30
Total other HeptaBB	ND	ND	ND	ND
<b>Total HeptaBB</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>
OctaBB	< 1,0	< 30	< 1,0	< 30
<b>Total OctaBB</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>
NonaBB	< 2,0	< 60	< 2,0	< 60
<b>Total NonaBB</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>
<b>DecaBB</b>	<b>&lt; 3,0</b>	<b>&lt; 90</b>	<b>&lt; 3,0</b>	<b>&lt; 90</b>

< : 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	Composite sample 3 (A7, A15, A24)		Composite sample 4 (A3, A20, A23)	
GfA Sample No.	6N267103		6N267104	
Unit	ng/kg fresh-weight	ng/kg fat-weight	ng/kg fresh-weight	ng/kg fat-weight
<b>PBBs</b>				
2,2',5,5'-TetraBB	< 0,2	< 6,0	< 0,2	< 6,0
Total other TetraBB	ND	ND	ND	ND
<b>Total TetraBB</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>
2,2',4,5,5'-PentaBB	< 0,3	< 9,0	< 0,3	< 9,0
Total other PentaBB	ND	ND	ND	ND
<b>Total PentaBB</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>
2,2',4,4',5,5'-HexaBB	< 0,4	< 12	< 0,4	< 12
Total other HexaBB	ND	ND	ND	ND
<b>Total HexaBB</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>
HeptaBB	< 1,0	< 30	< 1,0	< 30
Total other HeptaBB	ND	ND	ND	ND
<b>Total HeptaBB</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>
OctaBB	< 1,0	< 30	< 1,0	< 30
<b>Total OctaBB</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>
NonaBB	< 2,0	< 60	< 2,0	< 60
<b>Total NonaBB</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>	<b>ND</b>
<b>DecaBB</b>	<b>&lt; 3,0</b>	<b>&lt; 90</b>	<b>&lt; 3,0</b>	<b>&lt; 90</b>

< : 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	Composite sample 5 (A11, A19, A25)	
GfA Sample No.	6N267105	
Unit	ng/kg fresh-weight	ng/kg fat-weight
<b>PBBs</b>		
2,2',5,5'-TetraBB	< 0,2	< 6,0
Total other TetraBB	ND	ND
<b>Total TetraBB</b>	<b>ND</b>	<b>ND</b>
2,2',4,5,5'-PentaBB	< 0,3	< 9,0
Total other PentaBB	ND	ND
<b>Total PentaBB</b>	<b>ND</b>	<b>ND</b>
2,2',4,4',5,5'-HexaBB	< 0,4	< 12
Total other HexaBB	ND	ND
<b>Total HexaBB</b>	<b>ND</b>	<b>ND</b>
HeptaBB	< 1,0	< 30
Total other HeptaBB	ND	ND
<b>Total HeptaBB</b>	<b>ND</b>	<b>ND</b>
OctaBB	< 1,0	< 30
<b>Total OctaBB</b>	<b>ND</b>	<b>ND</b>
NonaBB	< 2,0	< 60
<b>Total NonaBB</b>	<b>ND</b>	<b>ND</b>
<b>DecaBB</b>	<b>&lt; 3,0</b>	<b>&lt; 90</b>

< : 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	Composite sample 1 (B1, B2, B14)		Composite sample 2 (A5, A8, A9)	
GfA Sample No.	6N267101		6N267102	
Fat content [%]	3,7		3,6	
Unit	pg/g fresh-weight	pg/g fat-weight	pg/g fresh-weight	pg/g fat-weight
PBDF				
238-TriBDF <sup>b</sup>	< 0,0030	< 0,080	0,0243	0,682
2378-TetraBDF <sup>b</sup>	< 0,0007	< 0,020	0,0124	0,348
12378-PentaBDF <sup>b</sup>	< 0,0011	< 0,030	0,0507	1,42
23478-PentaBDF <sup>b</sup>	< 0,0011	< 0,030	0,0456	1,279
123478-/123678-HexaBDF <sup>a, b</sup>	0,0045	0,121	0,0451	1,263
1234678-HeptaBDF <sup>b</sup>	< 0,0074	< 0,200	0,1451	4,066
PBDD				
237-TriBDD <sup>b</sup>	< 0,0030	< 0,080	0,0202	0,567
2378-TetraBDD <sup>b</sup>	< 0,0007	< 0,020	0,0014	0,039
12378-PentaBDD <sup>b</sup>	0,0015	0,039	0,0198	0,554
123478/123678-HexaBDD <sup>a, b</sup>	< 0,0112	< 0,300	0,0463	1,297
123789-HexaBDD <sup>b</sup>	< 0,0112	< 0,300	< 0,0112	< 0,315
WHO-TEQ PBDF/D excl. LOQ	0,0019	0,051	0,0583	1,635
WHO-TEQ PBDF/D incl. LOQ	0,0056	0,152	0,0595	1,667
I-TEQ PBDF/D excl. LOQ	0,0012	0,032	0,0485	1,358
I-TEQ PBDF/D incl. LOQ	0,0049	0,132	0,0496	1,390

< : 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

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	Composite sample 3 (A7, A15, A24)		Composite sample 4 (A3, A20, A23)	
GfA Sample No.	6N267103		6N267104	
Fat content [%]	3,5		3,4	
Unit	pg/g fresh-weight	pg/g fat-weight	pg/g fresh-weight	pg/g fat-weight
PBDF				
238-TriBDF <sup>b</sup>	0,0055	0,155	0,0044	0,129
2378-TetraBDF <sup>b</sup>	0,0008	0,024	< 0,0007	< 0,021
12378-PentaBDF <sup>b</sup>	0,0019	0,055	< 0,0011	< 0,032
23478-PentaBDF <sup>b</sup>	< 0,0011	< 0,031	0,0021	0,062
123478-/123678-HexaBDF <sup>a, b</sup>	0,0084	0,237	0,0044	0,128
1234678-HeptaBDF <sup>b</sup>	0,0571	1,615	0,0330	0,962
PBDD				
237-TriBDD <sup>b</sup>	< 0,0029	< 0,081	< 0,0029	< 0,085
2378-TetraBDD <sup>b</sup>	< 0,0007	< 0,020	< 0,0007	< 0,021
12378-PentaBDD <sup>b</sup>	< 0,0011	< 0,031	0,0012	0,036
123478/123678-HexaBDD <sup>a, b</sup>	< 0,0108	< 0,305	< 0,0110	< 0,319
123789-HexaBDD <sup>b</sup>	< 0,0108	< 0,305	< 0,0110	< 0,319
WHO-TEQ PBDF/D excl. LOQ	0,0016	0,045	0,0031	0,090
WHO-TEQ PBDF/D incl. LOQ	0,0061	0,172	0,0061	0,179
I-TEQ PBDF/D excl. LOQ	0,0016	0,045	0,0025	0,072
I-TEQ PBDF/D incl. LOQ	0,0055	0,157	0,0055	0,161

< : 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

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	Composite sample 5 (A11, A19, A25)	
GfA Sample No.	6N267105	
Fat content [%]	3,8	
Unit	pg/g fresh-weight	pg/g fat-weight
PBDF		
238-TriBDF <sup>b</sup>	0,0057	0,148
2378-TetraBDF <sup>b</sup>	0,0008	0,021
12378-PentaBDF <sup>b</sup>	< 0,0011	< 0,029
23478-PentaBDF <sup>b</sup>	< 0,0011	< 0,029
123478-/123678-HexaBDF <sup>a, b</sup>	0,0068	0,178
1234678-HeptaBDF <sup>b</sup>	0,1770	4,618
PBDD		
237-TriBDD <sup>b</sup>	< 0,0030	< 0,078
2378-TetraBDD <sup>b</sup>	0,0052	0,136
12378-PentaBDD <sup>b</sup>	0,0113	0,294
123478/123678-HexaBDD <sup>a, b</sup>	< 0,0113	< 0,294
123789-HexaBDD <sup>b</sup>	< 0,0113	< 0,294
WHO-TEQ PBDF/D excl. LOQ	0,0190	0,496
WHO-TEQ PBDF/D incl. LOQ	0,0219	0,571
I-TEQ PBDF/D excl. LOQ	0,0134	0,349
I-TEQ PBDF/D incl. LOQ	0,0163	0,424

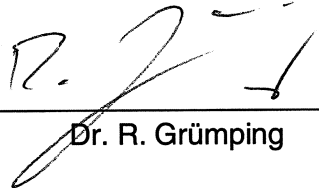
< : Concentration below the indicated limit of quantification (LOQ)

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

March 28, 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.