Determination of PCB in IRMM reference materials BCR-682 (mussel tissue) and BCR-718 (herring)

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Report number C038/11

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1. Introduction

The institute of Reference Materials and Measurements (IRMM) asked IMARES to analyse PCB in two different reference materials, LOT 1: BCR-682 (mussel tissue) and LOT 2: BCR-718 (herring).

2. Materials and Method

On the 15th of February 2011 IMARES received two different reference materials, four units each, from IRMM for the analyses of the following PCB:
LOT 1: BCR-682 (mussel tissue; units 15, 70, 115 and 339): CB28, 52, 118, 138+163, 149, 153, 170, 180.
LOT 2: BCR-718 (herring; units 3, 63, 150, 436): CB28, 52, 101, 105, 118, 128, 138+163, 149, 153, 156, 170, 180.
There was agreed upon that analyses should be performed in March 2011 (see e-mail d.d. 14th of January).
In each of the four units of a reference material three measurements were performed under repeatability conditions (12 determinations of each reference material), so 24 measurements of two different reference materials in one analytical run.

The PCB analyses were performed according to the following method (ISW 2.10.3.001):
Sample mixed with sodium sulphate (for removing moisture) is Soxhlet extracted for 6.5 hours with pentane/dichloromethane (1:1). After addition of 5 ml of iso-octane as a keeper, the extract is concentrated to about 5 ml in a rotary evaporator. Further clean-up and fractionation is then performed by putting the extract on top of a 15 g alumina · 9% H_2O column and elution with 170 ml n-pentane. After the addition of 2 ml of iso-octane as a keeper, the eluate is concentrated to about 2 ml in a rotary evaporator. The concentrate is transferred to a silica column (1.6 g silica · 2% H_2O) and eluted with 11 ml of iso-octane. Test determination by GC-ECD is then performed to estimate the concentration of the analytes in the eluate. If values are out of the range of the calibration curve, dilution or concentration of the eluate is performed. Final determination is then performed by gas chromatography (GC) coupled to electron capture detector (ECD) using two column system: Cp-Sil 8 (50 m long with internal diameter of 0.15 mm and film thickness of 0.20 µm) and Cp-Sil 19 (50 m long with internal diameter of 0.15 mm and film thickness of 0.20 µm).
All analyses of the requested PCB's are under accreditation by the Council for Accreditation in the matrix fish: CB28, 52, 101, 105, 118, 128, 138+163, 149, 153, 156, 170, 180 (Test laboratory code L097, Activity number 9). PCB138 will be reported as the sum of PCB138+163.
IMARES is registered as a Reference Lab at the Europese Commissie-Institute for Reference Materials and Measurements (IRMM) for the determination of PCB.

3. Results

The results given in this report apply only to the samples analyzed.

The results are given in appendix A. Concentrations are reported in µg/kg fresh material.
Decimal characters: Data is in derogation Dutch SI reported a decimal point (.) Instead of a comma (,).

The levels of the concentrations of the PCB in both reference materials BCR-682 and BCR-718 are very low in comparison of the levels of the PCB in our IRM cod liver 406 (see table 2).

4. Quality Assurance

IMARES utilises an ISO 9001:2008 certified quality management system (certificate number: 57846-2009-AQ-NLD-RvA). This certificate is valid until 15 December 2012. The organisation has been certified since 27 February 2001. The certification was issued by DNV Certification B.V. Furthermore, the chemical laboratory of the Environmental Division has NEN-AND-ISO/IEC 17025:2005 accreditation for test laboratories with number L097. This accreditation is valid until 27 March 2013 and was first issued on 27 March 1997. Accreditation was granted by the Council for Accreditation.

For every sequence of samples our results are tested on internal or certified reference materials (IRM/CRM). The results of the reference materials are collected in Quality Assurance Charts conform NPR 6603.

IMARES has a prominent role in Quasimeme preparing reference materials for certifying purposes and organizing international interlaboratory tests for contaminants in environmental samples.
The accuracy of our methods is tested by attendance to Performance Laboratory Studies (Quasimeme).

Our **Quasimeme results** for the requested PCB are given in table 1.

Table 1. Quasimeme results over several years

Component	Z-score			
	satisfied	unsatisfied	questionable	total
CB28	38	2	2	42
CB52	48	2	4	54
CB101	43	7	5	55
CB105	37	2	11	50
CB118	45	4	5	54
CB128	*not specified*			
CB138+163	42	2	2	46
CB149	*not specified*			
CB153	51	2	1	54
CB156	34	3	0	37
CB170	*not specified*			
CB180	46	3	2	51

The **repeatability** and **trueness** of the requested PCB in an internal reference material (IRM) cod liver 406 are given in table 2.

Table 2. Repeatability and trueness

Component	repeatability	trueness*	
	CV(%),n=9	J(%)	n
CB28	2.8 at a level of 45.9 µg/kg	99.9	8
CB52	2.1 at a level of 160 µg/kg	91.0	8
CB101	2.1 at a level of 410 µg/kg	109.8	8
CB105	5.8 at a level of 138 µg/kg	90.4	8
CB118	4.0 at a level of 461 µg/kg	98.4	10
CB128	2.3 at a level of 88 µg/kg	*not specified*	
CB138+163	3.1 at a level of 808 µg/kg	*because of sum of CB138+163 no trueness specified*	
CB149	2.5 at a level of 220 µg/kg	89.4	6
CB153	2.0 at a level of 1128 µg/kg	97.6	10
CB156	2.7 at a level of 60 µg/kg	100.6	9
CB170	2.9 at a level of 140 µg/kg	*not specified*	
CB180	3.6 at a level of 309 µg/kg	96.4	8

The **standard uncertainty** and the **expanded uncertainty** according to NEN 7779 are mentioned in table 3.

Table 3. Standard and expanded uncertainty

Component	standard uncertainty (%) (%)	expanded uncertainty (%)	n	Accreditation
PCB28	20.5	40.9	36	Q
PCB52	17.3	34.6	45	Q
PCB101	16.3	32.6	42	Q
PCB105	24.6	49.2	43	Q
PCB118	16.8	33.6	44	Q
PCB128	not defined	not defined		Q
PCB138+163	16.7	33.4	45	Q
PCB149	not defined	not defined		Q
PCB153	9.9	19.8	45	Q
PCB156	19.1	38.2	30	Q
PCB170	not defined	not defined		Q
PCB180	18.1	36.5	43	Q

Justification

Rapport C038/11
Project Number: 4305107601

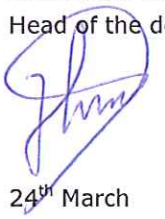
The scientific quality of this report has been peer reviewed by the a colleague scientist and the head of the department of IMARES.

Approved: Dr. Ir. M.J.J. Kotterman
Project leader

Signature: 

Date: 24Th March

Approved: Drs. J.H.M. Schobben
Head of the department Environment

Signature: 

Date: 24th March

Appendix A. Results of PCB in reference materials BCR-682 and BCR-718 in µg/kg fresh material

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LIMS number	IRMM Unit ID-number	replicate	reference material	date of analysis	CB28 µg/kg	CB52 µg/kg	CB101 µg/kg	CB105 µg/kg	CB118 µg/kg	CB128 µg/kg	CB138+163 µg/kg	CB149 µg/kg	CB153 µg/kg	CB156 µg/kg	CB170 µg/kg	CB180 µg/kg
2011/0693	0682/15	1	BCR-682	2th of March 2011	0.36	0.86	n.a.	n.a.	2.27	n.a.	6.00	5.05	8.86	n.a	0.21	0.96
2011/0694		2	BCR-682	2th of March 2011	0.35	0.81	n.a.	n.a.	2.27	n.a.	5.83	5.06	8.88	n.a	0.21	0.92
2011/0695		3	BCR-682	2th of March 2011	0.40	0.80	n.a.	n.a.	2.29	n.a.	5.77	4.98	8.83	n.a	0.21	0.97
2011/0696	0682/70	1	BCR-682	2th of March 2011	0.38	0.92	n.a.	n.a.	2.49	n.a.	6.45	5.43	9.79	n.a	0.21	0.99
2011/0697		2	BCR-682	2th of March 2011	0.36	0.87	n.a.	n.a.	2.41	n.a.	6.33	5.26	9.51	n.a	0.20	0.95
2011/0698		3	BCR-682	2th of March 2011	0.38	0.89	n.a.	n.a.	2.47	n.a.	6.58	5.24	9.46	n.a	0.21	0.98
2011/0699	0682/115	1	BCR-682	2th of March 2011	0.33	0.77	n.a.	n.a.	2.28	n.a.	5.80	4.75	8.60	n.a	0.21	0.91
2011/0700		2	BCR-682	2th of March 2011	0.35	0.82	n.a.	n.a.	2.23	n.a.	5.87	4.86	8.88	n.a	0.22	0.90
2011/0701		3	BCR-682	2th of March 2011	0.39	0.83	n.a.	n.a.	2.32	n.a.	6.05	4.85	8.64	n.a	0.22	0.91
2011/0702	0682/339	1	BCR-682	2th of March 2011	0.38	0.93	n.a.	n.a.	2.27	n.a.	6.16	5.20	9.24	n.a	0.20	0.93
2011/0703		2	BCR-682	2th of March 2011	0.37	0.90	n.a.	n.a.	2.43	n.a.	6.18	5.22	9.39	n.a	0.20	0.96
2011/0704		3	BCR-682	2th of March 2011	0.39	0.98	n.a.	n.a.	2.41	n.a.	6.25	5.23	9.19	n.a	0.20	0.89
2011/0705	0718/3	1	BCR-718	2th of March 2011	0.50	1.21	2.08	0.67	2.06	0.68	3.21	2.61	4.41	0.20	0.33	0.86
2011/0706		2	BCR-718	2th of March 2011	0.46	1.07	2.01	0.64	1.97	0.64	2.95	2.52	4.38	0.21	0.33	0.85
2011/0707		3	BCR-718	2th of March 2011	0.47	1.16	2.07	0.66	2.05	0.68	3.28	2.53	4.41	0.22	0.28	0.86
2011/0708	0718/63	1	BCR-718	2th of March 2011	0.62	1.27	2.07	0.59	1.91	0.60	3.04	2.63	4.18	0.22	0.30	0.78
2011/0709		2	BCR-718	2th of March 2011	0.43	1.01	1.78	0.61	1.70	0.58	2.80	2.42	3.88	0.20	0.29	0.76
2011/0710		3	BCR-718	2th of March 2011	0.45	1.04	1.84	0.60	1.77	0.58	2.91	2.44	3.94	0.21	0.29	0.76
2011/0711	0718/150	1	BCR-718	2th of March 2011	0.42	1.00	1.72	0.57	1.67	0.55	2.64	2.34	3.80	0.18	0.27	0.69
2011/0712		2	BCR-718	2th of March 2011	0.47	1.12	2.05	0.64	1.90	0.67	3.16	2.66	4.28	0.22	0.31	0.81
2011/0713		3	BCR-718	2th of March 2011	0.43	1.02	1.90	0.63	1.91	0.61	2.91	2.51	4.26	0.21	0.31	0.81
2011/0714	0718/436	1	BCR-718	2th of March 2011	0.43	1.08	1.92	0.66	1.91	0.61	3.00	2.54	4.25	0.23	0.31	0.79
2011/0715		2	BCR-718	2th of March 2011	0.50	1.06	1.89	0.64	1.91	0.63	3.09	2.57	4.37	0.24	0.31	0.85
2011/0716		3	BCR-718	2th of March 2011	0.46	1.02	1.89	0.63	1.89	0.61	3.07	2.50	4.14	0.22	0.31	0.82

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