## Information on quality assurance

### International intercalibrations

The laboratories (NIVA and subcontractor Eurofins) have participated in the Quality Assurance of Information for Marine Environmental Monitoring in Europe (QUASIMEME), International Food Analysis Proficiency Testing Services (FAPAS), international intercalibration exercises and other proficiency testing relevant to chemical and imposex analyses. For chemical analyses, round 2018-1 apply to the 2018-samples. The results are acceptable. These QUASIMEME exercises included nearly all the contaminants as well as imposex analysed in this programme. The quality assurance programme is corresponding to the analyses of the 2017 samples (cf. Green *et al.* 2018 – M‑618|2018).

NIVA participated in the QUASIMEME Laboratory Performance Studies “imposex and intersex in Marine Snails BE1” in July-September 2017. Shell height, penis-length-male, penis-length-female, average-shell-height and female-male-ratio were measured. NIVA got the score satisfactory for all parameters except number of females for one sample, which got the score questionable. The score for VDSI was satisfactory for both samples tested.

### Analyses of certified reference materials

In addition to the QUASIMEME exercises, certified reference materials (CRM) and in-house reference materials are analysed routinely with the MILKYS samples. It should be noted that for biota, the type of tissue used in the CRMs does not always match the target tissue for analysis. Uncertain values identified by the analytical laboratory or the reporting institute are flagged in the database. The results are also “screened” during the import to the database at NIVA and ICES.

The laboratories used for the chemical testing are accredited according to ISO 17025:2005, except for the PFCs.

**Summary of quality control results**

Standard Reference Materials (SRM) as well as in-house reference materials were analysed regularly (***Table1***). Fish protein (DORM-4 and DOLT-5) was used as SRM for the control of the determination of metals. The reference material for determination of BDEs and HBCDDs in blue mussel was an internal reference (fish oil). For determination of PCBs, DDTs and PAHs in blue mussel, as well as HBCDDs, PCBs, DDTs and BDEs in liver, internal reference materials provided by EF GfA Lab services were used, these consisted of fish meal and feedingstuff. For TBBPA, spiked fish oil was used for quality assurance, and for chlorinated paraffines and octyl/nonylphenols, spiked fish meal was used. For organophosphorous flame retardants, spiked internal reference material was used.

**Table 1.** Summary of the quality control of results for the 2018 biota samples analysed in 2018-2019. The Standard Reference Materials (SRM) were DORM-4\* (fish protein) and DOLT-5\* for blue mussel, fish liver and fish fillet. The in-house reference materials were, spiked fish oil, spiked fish meal and spiked internal reference material (Pool 74, Pool 74 spiked with TBBPA, Pool 107, Pool 109 and Pool 122). For BPA, reference materials REFBP007 (olive oil) REFBP010 (apple purée) and REFBP005 (Liquor) were analysed and for tin organic compounds reference material ZRM 81 (mussel tissue) was used. The SRMs and in-house reference materials and quality assurance standards were analysed in series with the MILKYS samples and measured several times (N) over a number of weeks (W). The values are reported in the following units: metals (mg/kg), BDE (pg/g), PCB (µg/kg), DDTs (µg/kg), HBCDDs (ng/g), PAH (µg/kg), TBBPA (ng/sample), BPA (µg/kg), octyl/nonylphenol (ng/sample), organophosphorus flame retardants (pg/sample) and PFCs (% recovery). Tissue types were: mussel soft body (SB), fish liver (LI) and fish fillet (MU).

| **Code** | **Contaminant** | **Tissue type** | **SRM type** | **SRM value confidence interval** | **N** | **W** | **Mean value** | **Standard deviation** |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Ag** | **Silver** | SB/LI | DOLT-5 | 2.05 ± 0.08 | 53 | 17 | 1,49 | 0,11 |
| **As** | **Arsenic** | SB/LI | DORM-4 | 6,80±0,64 | 55 | 17 | 6,34 | 0,250 |
| **Cd** | **Cadmium** | SB/LI | DORM-4 | 0,306±0,015 | 54 | 16 | 0,304 | 0,012 |
| **Cr** | **Chromium** | SB/LI | DORM-4 | 1,87±0,16 | 54 | 17 | 1,745 | 0,16 |
| **Co** | **Cobalt** | SB/LI | DOLT-5 | 0.267 ± 0.026 | 52 | 16 | 0,225 | 0,019 |
| **Cu** | **Copper** | SB/LI | DORM-4 | 15,9±0,9 | 55 | 16 | 14,21 | 0,87 |
| **Hg** | **Mercury** | SB/MU | DORM-4 | 0,41±0,055 | 64 | 17 | 0,41 | 0,029 |
| **Ni** | **Nickel** | SB/LI | DORM-4 | 1,36±0,22 | 51 | 17 | 1,18 | 0,081 |
| **Pb** | **Lead** | SB/LI | DORM-4 | 0,416±0,053 | 55 | 17 | 0,37 | 0,024 |
| **Zn** | **Zinc** | SB/LI | DORM-4 | 52,2±3,2 | 54 | 16 | 50,33 | 2,99 |
| **Sn** | **Tin** | SB/LI | DOLT-5 | 0.069 ± 0.036 | 53 | 17 | 0,095 | 0,025 |
| **BDE-28** | **2,2,4’ Tribromodiphenylether** | SB/LI | Pool 74 |  | 47 | 26 | 86,4411 | 5,049 |
| **BDE-47** | **2,2',4,4',-Tetrabromodiphenylether** | SB/LI | Pool 74 |  | 47 | 26 | 1605,988 | 39,309 |
| **BDE-100** | **2,2',4,4',6-Pentabromodiphenylether** | SB/LI | Pool 74 |  | 47 | 26 | 323,1465 | 14,186 |
| **BDE-99** | **2,2',4,4',5-Pentabromodiphenylether** | SB/LI | Pool 74 |  | 47 | 26 | 250,6408 | 6,815 |
| **BDE-154** | **2,2',4,4',5,6'-Hexabromodiphenylether** | SB/LI | Pool 74 |  | 47 | 26 | 202,1234 | 20,735 |
| **BDE-153** | **2,2’,4,4’5,5’- Hexabromodiphenylether** | SB/LI | Pool 74 |  | 47 | 26 | 61,4060 | 3,622 |
| **BDE-209** | **Decabromodiphenylether** | SB/LI | Pool 74 |  | 8 | 26 | 551,2245 | 325,006 |
| **BDE-49** | **2,2',4,5'-tetrabromodiphenyleter** | SB/LI | Pool 74 |  | 47 | 26 | 434,4130 | 22,258 |
| **BDE-66** | **2,3',4,4'-Tetrabromodiphenyleter** | SB/LI | Pool 74 |  | 47 | 26 | 58,4452 | 8,118 |
| **BDE-119** | **2,3',4,4',6-Pentabromodiphenyl ether** | SB/LI | Pool 74 |  | 47 | 26 | 34,5222 | 3,744 |
| **PCB 77** | **PCB congener CB-77** | SB/LI | Pool 109 |  | 58 | 25 | 9,68 | 2,19 |
| **PCB 52** | **PCB congener CB-52** | SB/LI | Pool 109 |  | 59 | 25 | 269,38 | 15,11 |
| **PCB 28** | **PCB congener CB-28** | SB/LI | Pool 109 |  | 59 | 25 | 104,58 | 15,11 |
| **PCB 189** | **PCB congener CB-189** | SB/LI | Pool 109 |  | 59 | 25 | 6,28 | 0,33 |
| **PCB 180** | **PCB congener CB-180** | SB/LI | Pool 109 |  | 59 | 25 | 480,06 | 28,13 |
| **PCB 169** | **PCB congener CB-169** | SB/LI | Pool 109 |  | 58 | 25 | 0,73 | 0,08 |
| **PCB 167** | **PCB congener CB-167** | SB/LI | Pool 109 |  | 59 | 25 | 29,58 | 3,60 |
| **PCB 157** | **PCB congener CB-157** | SB/LI | Pool 109 |  | 59 | 25 | 13,58 | 0,41 |
| **PCB 156** | **PCB congener CB-156** | SB/LI | Pool 109 |  | 59 | 25 | 49,33 | 1,23 |
| **PCB 153** | **PCB congener CB-153** | SB/LI | Pool 109 |  | 59 | 25 | 1505,87 | 110,33 |
| **PCB 138** | **PCB congener CB-138** | SB/LI | Pool 109 |  | 59 | 25 | 909,37 | 54,39 |
| **PCB 126** | **PCB congener CB-126** | SB/LI | Pool 109 |  | 59 | 25 | 2,87 | 0,42 |
| **PCB 123** | **PCB congener CB-123** | SB/LI | Pool 109 |  | 59 | 25 | 4,80 | 0,89 |
| **PCB 118** | **PCB congener CB-118** | SB/LI | Pool 109 |  | 59 | 25 | 449,06 | 18,02 |
| **PCB 114** | **PCB congener CB-114** | SB/LI | Pool 109 |  | 59 | 25 | 7,76 | 1,01 |
| **PCB 105** | **PCB congener CB-105** | SB/LI | Pool 109 |  | 59 | 25 | 137,3 | 5,02 |
| **PCB 101** | **PCB congener CB-101** | SB/LI | Pool 109 |  | 59 | 25 | 613,61 | 49,92 |
| **DDEOP** | **o,p'-DDE** | SB/LI | Pool 122 |  | 72 | 29 | 0,116 | 0,0116 |
| **TDEOP** | **o,p'-DDD** | SB/LI | Pool 122 |  | 73 | 29 | 0,258 | 0,0276 |
| **DDTOP** | **o,p'-DDT** | SB/LI | Pool 122 |  | 73 | 29 | 0,228 | 0,0497 |
| **DDEPP** | **p,p'-DDE** | SB/LI | Pool 122 |  | 73 | 29 | 5,12 | 0,39 |
| **TDEPP** | **p,p'-DDD** | SB/LI | Pool 122 |  | 73 | 29 | 1,57 | 0,15 |
| **DDTPP** | **p,p'-DDT** | SB/LI | Pool 122 |  | 73 | 29 | 0,611 | 0,0443 |
| **α-HBCDD** | **α-Hexabromocyclododecane** | SB/LI | Pool 74 |  | 54 | 29 | 0,955 | 0,079 |
| **β-HBCDD** | **β- Hexabromocyclododecane** | SB/LI | Pool 74 |  | 54 | 29 | 0,056 | 0,010 |
| **γ-HBCDD** | **γ- Hexabromocyclododecane** | SB/LI | Pool 74 |  | 54 | 29 | 0,289 | 0,041 |
| **BGHIP** | **Benzo[ghi]perylene** | SB/LI | Pool 107 |  | 31 | 35 | 0,53 | 0,06 |
| **ICDP** | **Indeno[1,2,3-cd]pyrene** | SB/LI | Pool 107 |  | 31 | 35 | 0,44 | 0,05 |
| **BBJF** | **Benzo[b+j]fluoranthene** | SB/LI | Pool 107 |  | 31 | 35 | 0,32 | 0,04 |
| **DBA3A** | **Dibenzo[ac,ah]anthracene** | SB/LI | Pool 107 | - | 9 | 35 | 0,16 | 0,02 |
| **BKF** | **Benzo[k]fluoranthene** | SB/LI | Pool 107 |  | 31 | 35 | 1,12 | 0,15 |
| **ACNLE** | **Acenaphthylene** | SB/LI | Pool 107 |  | 29 | 35 | 1,62 | 0,32 |
| **ANT** | **Anthracene** | SB/LI | Pool 107 |  | 30 | 35 | 0,99 | 0,15 |
| **BAA** | **Benzo[a]anthracene** | SB/LI | Pool 107 |  | 31 | 35 | 1,14 | 0,15 |
| **BAP** | **Benzo[a]pyrene** | SB/LI | Pool 107 |  | 31 | 35 | 0,59 | 0,05 |
| **CHR** | **Chrysene** | SB/LI | Pool 107 |  | 31 | 35 | 1,10 | 0,17 |
| **FLU** | **Fluoranthene** | SB/LI | Pool 107 |  | 31 | 35 | 3,22 | 0,50 |
| **FLE** | **Fluorene** | SB/LI | Pool 107 |  | 31 | 35 | 12,7 | 2,1 |
| **NAP** | **Naphthalene** | SB/LI | Pool 107 |  | 23 | 35 | 28,9 | 16,0 |
| **PA** | **Phenanthrene** | SB/LI | Pool 107 |  | 31 | 35 | 9,55 | 1,62 |
| **PYR** | **Pyrene** | SB/LI | Pool 107 |  | 31 | 35 | 3,49 | 0,46 |
| **ACNE** | **Acenaphthene** | SB/LI | Pool 107 |  | 31 | 35 | 23,7 | 3,9 |
| **TBBPA** | **Tetrabromobisphenol-A** | SB/LI | Pool 74 (spiked) | - | 34 | 29 | 0,87 | 0,21 |
| **BPA** | **Bisphenol-A** | SB/LI | REFBP007 Olive oil | 40.0 ± 6.0 | 21 | 10 | 41,8 | 6,0 |
| **BPA** | **Bisphenol-A** | SB/LI | REFBP010 Apple puree | 40.0 ± 0.7 | 81 | 14 | 4,3 | 1,0 |
| **BPA** | **Bisphenol-A** | SB/LI | REFBP005 Rum | 22.9 ± 2.8 | 77 | 26 | 21,9 | 2,5 |
| **APO** | **4-tert-oktylfenol** | LI/SB | Internal RM (spiked blank) |  | 8 | 14 | 41206 | 9022 |
| **APO** | **4-n-oktylfenol** | LI/SB | Internal RM (spiked blank) |  | 8 | 14 | 39602 | 1274 |
| **APO** | **4-n-nonylfenol** | LI/SB | Internal RM (spiked blank) |  | 8 | 14 | 42383 | 871 |
| **MBT** | **Monobutyltinn (MBT)** | LI/SB | ZRM 81 |  | 41 | 27 | 2,02 | 0,22 |
| **DBT** | **Dibutyltinn (DBT)** | LI/SB | ZRM 81 |  | 41 | 27 | 1,12 | 0,16 |
| **TBT** | **Tributyltinn (TBT)** | LI/SB | ZRM 81 |  | 41 | 27 | 1,70 | 0,29 |
| **TPhT** | **Trifenyltinn (TPhT)** | LI/SB | ZRM 81 |  | 41 | 27 | 1,40 | 0,23 |
| **PFBS** | **Perfluorobutane sulphonate** | LI | In-house spiked liver | 100%1) | 8 |  | 93 | 6,3% |
| **PFHxA** | **Perfluorohexane acid** | LI | In-house spiked liver | 100%1) | 8 |  | 100 | 5,7% |
| **PFHpA** | **Perfluoroheptane acid** | LI | In-house spiked liver | 100%1) | 8 |  | 107 | 17,8% |
| **PFOA** | **Perfluorooctane acid** | LI | In-house spiked liver | 100%1) | 8 |  | 108 | 9,0% |
| **PFNA** | **Perfluorononane acid** | LI | In-house spiked liver | 100%1) | 8 |  | 101 | 10,7% |
| **PFOS** | **Perfluorooctane sulphonate** | LI | In-house spiked liver | 100%1) | 8 |  | 96 | 3,3% |
| **PFOSA** | **Perfluorooctane sulphone amide** | LI | In-house spiked liver | 100%1) | 8 |  | 94 | 8,2% |
| **PFHxS** | **Perfluorohexane sulphonate** | LI | In-house spiked liver | 100%1) | 8 |  | 89 | 5,4% |
| **PFDA** | **Perfluorodecanoic acid** | LI | In-house spiked liver | 100%1) | 8 |  | 98 | 9,5% |
| **PFUDA** | **Perfluoroundecanoic acid** | LI | In-house spiked liver | 100%1) | 8 |  | 95 | 5,2% |
| **PFDS** | **Perfluorodecanesulphonate** | LI | In-house spiked liver | 100%1) | 8 |  | 81 | 9,8% |

\* National Research Council Canada, Division of Chemistry, Marine Analytical Chemistry Standards.

1) Recovery of spiked control sample