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Sixth Round of an International Study

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Summary

In 2005, the sixth round of Interlaboratory Comparison on Dioxins in Food was conducted on the determination of the 2,3,7,8-chlorinated dibenzo-*p*-dioxins (PCDDs) and dibenzofurans (PCDFs) as well as dioxin-like non-ortho and mono-ortho chlorinated biphenyls (PCBs) in three different food items. In this round, also polybrominated diphenyl ethers (PBDEs) as well as the six indicator PCBs could voluntarily be determined and reported. The objectives were a) to offer a quality assurance instrument for the participating laboratories, b) to assess the between laboratory reproducibility and c) to assess the readiness of expert laboratories worldwide to determine levels of chlorinated and brominated persistent organic pollutants in regular foodstuffs.

The 2005 study was performed on sample homogenates of reindeer meat, herring filet and cod liver oil. In addition, five standard solutions were provided containing known concentrations of a) PCDDs/PCDFs, b) non-ortho PCBs, c) mono-ortho PCBs, d) PBDEs, and e) indicator PCBs. The testing materials were sent to 95 laboratories in February 2005, and results were returned from 87 laboratories in 28 different countries by the deadline in May. Most laboratories participated in all of the three food items. A draft report was made available on the Internet in July and was discussed among the participants at a consultation meeting during the Dioxin 2005 Symposium in Toronto, Canada.

This report presents all of the results reported from the participating laboratories for the 29 analytes assigned a toxic equivalency factor (TEF) by the WHO in 1998: all seventeen 2,3,7,8-substituted PCDDs and PCDFs, the non-ortho substituted PCBs #77, 81, 126 and 169, and the eight mono-ortho substituted PCBs #105, 114, 118, 123, 156, 157, 167, 189, in the three food items on a fresh weight basis. In addition, the results for eight PBDE congeners BDE #28, 47, 99, 100, 153, 154, 183 and 209 and six indicator PCB congeners PCB #28, 52, 101, 138, 153 and 180 were reported from those laboratories that voluntarily determined their concentrations. Assigned values for the analytes were determined by the participant consensus technique. Non-detected congeners were assigned a concentration corresponding to the reported detection limit except for PBDEs and indicator PCBs where non-detects were removed from the data set. The consensus concentration for each analyte in the three food samples was determined as follows: The median of all reported concentrations for each analyte was calculated. All values above two times the median were then removed from the calculation. The consensus median and consensus mean plus standard deviation were calculated from the remaining data. Toxic equivalents (TEQs) were calculated from the consensus values of individual congeners using the toxic equivalency factors derived by WHO in 1998. Z-scores for PCDD/PCDF TEQs were calculated for each laboratory using a deviation of ±20% of the consensus TEQs. Further, z-scores were calculated for the non-ortho PCB TEQ, the mono-ortho PCB TEQ, the total TEQ, the sum of six indicator PCBs, the sum of seven PBDEs and for each singel congener in all three matrixes.

The consensus values for the standard solutions were calculated in the same manner except that values outside ± 50% of the median of all values were removed prior to final calculation of the consensus median and mean. The consensus values for the lipid content were calculated by first excluding results deviating more than two standard deviations from the mean of all values and then re-calculating the median, mean and standard deviation.

For the determination of PCDDs/PCDF TEQs, z-scores within ±1 were obtained by 62-86% of the laboratories. The majority of the laboratories (88 to 95%) reported results for PCDD/PCDFs with an acceptable trueness (±40%) for all food samples. Also the RSD´s calculated for the total TEQ after removal of outliers are quite low (10 to 13%). It is therefore concluded that the performance of laboratories world-wide in determining dioxin-like compounds is generally good for food samples with elevated contamination levels.

Thirtyeight laboratories reported concentrations for the seven tetra- to hepta-BDEs and 62 laboratories reported results of the six indicator PCBs. The sum of the PBDE concentrations ranged from 37 pg/g fresh weight in reindeer to 14 ng/g fresh weight in cod liver oil. The RSD for PBDE concentrations on fresh weight basis was on average 29%, 31% and 44% for cod liver oil, herring and reindeer, respectively. The consensus concentrations calculated for BDE-209 are just indicative values as only few laboratories had reported this congener. The sum of concentrations for indicator PCBs were 1.1, 7.5 and 84 ng/g fresh weight for reindeer, herring and cod liver oil, respectively. The corresponding average RSDs were 36%, 28% and 27%.

Introduction

In order to ensure consumer protection and reduce human exposure to dioxins and dioxin-like PCBs through food consumption, many countries request frequent monitoring of the presence of these toxic pollutants in food and feed. There is therefore a large demand for chemical laboratories that are able to monitor these contaminants at low levels in food and feed. It is usually required by the authorities that laboratories performing such measurements are accredited according to ISO standards and prove their competence by successful participation in interlaboratory studies.

This study is the sixth round of a world-wide interlaboratory comparison study on dioxin-like compounds in food organised by the Department of Analytical Chemistry, Division of Environmental Medicine, Norwegian Institute of Public Health in Oslo, Norway.

The exercise took place from February 2005, when the samples were shipped to the laboratories for analysis, to the beginning of May 2005, when the last reports on the results were received. A draft report was made available to the participants on the Internet (http://www.fhi.no) in July and was discussed during a consultation meeting at the DIOXIN2005 Symposium in Toronto, Canada.

The main objective of this exercise was to assess the between laboratory reproducibility of dioxin-like compounds analyses in frequently consumed foods and provide a QA/QC instrument for each participating laboratory to contribute to its proficiency. Participants were also asked to voluntarily determine the concentrations of eight polybrominated diphenyl ethers and six indicator PCBs in the food samples in order to assess the readiness of laboratories to analyse these persistent organic pollutants.

All of the participants from previous rounds of this series of "Interlaboratory Comparisons on Dioxins in Food" were invited to participate. In addition, several other laboratories announced their participation. There was no limit to the total number of participating laboratories. The 87 laboratories that submitted results, and thereby contributed to the study results, are presented in Table 1.

Table 1. *Participants that reported results in the Sixth Round of Interlaboratory Comparison on Dioxins in Food 2005*

Agriculture and Food Science Centre
Belfast, Northern Ireland

AgriQuality Limited
Lower Hutt, Wellington, New Zealand

Alta Analytical Perspectives
Wilmington, NC, USA

AnalyCen Nordic AB
Lidköping, Sweden

ARPA Piemonte, Dipartimento di Alessandria Polo Microinquinanti
Alessandria, Italy

Axys Analytical Services Ltd.
Sidney, British Columbia, Canada

Bayerisches Landesamt für Umweltschutz
Augsburg, Germany

Canadian Food Inspection Agency Calgary Laboratory
Calgary, Alberta, Canada

CARSO-LSEHL
Lyon, France

CART-University of Liège
Liège, Belgium

Central Science Laboratory
York, UK

Centre d'expertise en analyse environnementale du Québec
Laval, Canada

CHELAB s.r.l.
Resana (TV), Italy

Chemisches Landes- und Staatliches Veterinäruntersuchungsamt
Münster, Germany

Chemisches und Veterinäruntersuchungsamt (CVUA) Freiburg, Dr. R. Malisch
Freiburg, Germany

Chemisches und Veterinäruntersuchungsamt (CVUA) Freiburg, Mr. A. Kotz
Freiburg, Germany

Chemisches und Veterinäruntersuchungsamt (CVUA) Freiburg, Dr. Karin Kypke
Freiburg, Germany

Consorzio Interuniversitario Nazionale la Chimica per l'Ambiente
Marghera, VE, Italy

Dalian Institute of Chemical Physics Chinese Academy of Science
Dalian, China

Danish Veterinary and Food Administration
Ringsted, Denmark

Danish Institute for Food and Veterinary Research Department of Food Chemistry
Søborg, Denmark

Department of Environmental and Occupational Health, National Cheng Kung University, College of Medicine
Tainan, Taiwan, R.O.C.

Dioxin Analysis Unit, National Measurement Institute
Sydney, Australia

Dr. Wessling Laboratorien GmbH
Altenberge, Germany

Ecochem a.s.
Pardubice, Czech Republic

Eco Research
Bolzano, Italy

Environmental Research and Protection Centre
Ufa, Russian Federation

Enviro-Test Laboratories
Edmonton, Alberta, Canada

ERGO Forschungsgesellschaft mbH
Hamburg, Germany

Eurofins/GfA mbH
Münster-Roxel, Germany

Eurofins Oekometric GmbH
Bayreuth, Germany

Food GmbH Jena
Jena, Germany

GEOTAIX GmbH
Wuerselen, Germany

GMLab, Department of Chemistry, National Tsing Hua University, Taiwan, ROC
Hsinchu, Taiwan, ROC

GSF National Research Center for Environment and Health, Institute of Ecological Chemistry
Neuherberg, Germany

Hong Kong Government Laboratory Dr. W.O. Lee
Kowloon, Hong Kong

Hong Kong Government Laboratory Dr. W.C. Chung
Kowloon, Hong Kong

Institut d'Investigacions Quimiques i Ambientals, CSIC
Barcelona, Spain

Institute of Aquaculture
Stirling, UK

Institute of Public Health Ostrava
Ostrava, Czech Republic

Institute of Organic Chemistry
Department of Instrumental Analysis
and Environmental Chemistry
Madrid, Spain

Instituto Nacional de Engenharia Industrial, INETI
Lisboa, Portugal

Istituto Superiore di Sanità
Dr. Luigi Turrio-Baldasarri
Roma, Italy

Istituto Superiore di Sanità
Toxicological Chemistry Unit
Roma, Italy

Istituto zooprofilattico sperimentale dell'abruzzo
del molise "G. Caporale"
Teramo, Italy

Institut Químic de Sarrià, Environmental Laboratory
Barcelona, Spain

Japan Food Research Laboratories
Tokyo, Japan

Korea Food and Drug Administration
Seoul, Korea

LABERCA
Nantes, France

Laboratory of Vendee
La Roche Sur Yon, France

Landesamt für Umweltschutz Sachsen-Anhalt
Halle, Germany

Landeslabor Brandenburg , Standort Potsdam
Potsdam, Germany

Landwirtschaftliche Untersuchungs-
und Forschungsanstalt
Rostock, Germany

Landwirtschaftliche Untersuchungs-
und Forschungsanstalt
Speyer, Germany

LEM S.A.
Illkirch, France

LUFA GmbH
Kiel, Germany

Marchwood Scientific Services
Southampton, UK

Mass Spectrometry and Dioxin Analysis Lab,
NCSR "Demokritos"
Athens, Greece

Maxxam Analytics Inc.
Waterloo, Ontario, Canada

MicroPolluants Technologie
Thionville, France

Nab Labs Oy
Espoo, Finland

National Food Administration
Uppsala, Sweden

National Institute of Nutrition and Food Safety
Chinese Center for Disease Control and Prevention
Beijing, China

National Institute of Nutrition and Seafood Research
Bergen, Norway

National Public Health Institute – KTL
Kuopio, Finland

Niedersächsisches Landesamt für Verbraucherschutz
und Lebensmittelsicherheit
Oldenburg, Germany

Norwegian Instiute for Air Research - NILU
Kjeller, Norway

Norwegian Institute of Public Health
Oslo, Norway

Public Health Institute
Environmental protection institute
Maribor, Slovenia

Research and Productivity Council
Fredericton, New Brunswick, Canada

Research Center for Eco-Environmetal Science
Chinese Academy of Science
Beijing, China

RIKILT institute for food safety
Wageningen, The netherlands

Scientific Institute of Public Health
Bruxelles, Belgium

Sea Fisheries Institute in Gdynia
Gdynia, Poland

Shenzhen Center for Disease Control and Prevention,
Shenzhen POPs Lab
Shenzhen, China

SGS Belgium NV
Antwerp, Belgium

SGS Institut Fresenius GmbH Bayreuth
Bayreuth, Germany

Shanghai Municipal Ceter for Disease Control & Prevention
Shanghai, P.R.China

TNO Environment and Geosciences Environmental quality and analysis
Apeldoorn, The Netherlands

TÜV Industrie Service GmbH TÜV Süd Gruppe
Donzdorf, Germany

UEG GmbH – Institut für Umweltanalytik und Geotechnik
Wetzlar, Germany

Umeå University, Environmental Chemistry
Umeå, Sweden

Unilever SEAC
Bedfordshire, UK

USDA ARS, Biosciences Research Laboratory
Fargo, ND, USA

US Environmental Protection Agency, Environmental Chemistry Laboratory
Stennis Space Center, MS, USA

Wellington Laboratories Inc.
Guelph, Ontario, Canada

Zhejiang Center for Disease Control and Prevention, Zhejiang Dioxin Lab
Hanzhou, China

Design and practical implementation

Study design

As in the previous rounds of this interlaboratory comparison, the test material chosen represented naturally contaminated food samples. The analytes to be determined by each participating laboratory were all seventeen 2,3,7,8-substituted PCDDs and PCDFs, the four non-ortho substituted PCBs #77, 81, 126 and 169, and the eight mono-ortho substituted PCBs #105, 114, 118, 123, 156, 157, 167, 189.

In addition, laboratories were asked to determine on a voluntary basis eight polybrominated diphenyl ethers, namely PBDEs #28, #47, #99, #100, #153; #154, #183 and #209 and six indicator PCB congeners PCB #28, 52, 101, 138, 153 and 180. These six PCB congeners belong together with the mono-ortho PCB #118 to the selection of PCBs commonly referred to as ICES-7. These PCBs have been selected for inclusion in the legislation of several countries.

Analysis should be performed using the laboratory's own methods for sample preparation and instrumental analysis, their own standards and quantification procedures, and their own method for lipid determination.

It was recommended that laboratories determine as many as possible of all 2,3,7,8-substituted PCDDs/PCDFs and dioxin-like PCBs, PBDEs and indicator PCBs. The report was to include the determined lipid percent for the test samples. Also the actual sample and lipid intake (g) for each determination should have been reported. For each sample, laboratories were to report one concentration on fresh and lipid weight basis for each congener which was detected (S/N ≥3) as well as the level of determination (LOD, e.g., S/N =3). Non-detected congeners (S/N <3) were to be marked "ND" in the Comments column of the Report form.

In addition, five standard solutions containing known concentrations of a) seventeen 2,3,7,8-substituted PCDDs/PCDFs, b) four non-ortho PCBs, c) eight mono-ortho PCBs d) eight PBDEs, and e) six indicator PCBS were to be analysed using the laboratory's own quantification standards and methods. The results were to be reported on a separate form.

The test material consisted of reindeer meat, filet of herring and cod liver oil. The laboratory could choose to participate in analysing one, two or all three of the food samples

Collection, preparation, and distribution of samples

- Reindeer meat from Norway (~90g)
- Herring from the Baltic Sea (~75g)
- Cod liver oil (~15g)

The test materials consisted of three natural products not fortified with standards. Reindeer meat was purchased from Aage Pedersen AS in the province of Finmark, Northeast Norway. Baltic herring was obtained form the National Food Administration in Sweden. Raw and refined cod liver oil was a gift from the company Peter Möller, Oslo, Norway.

Homogenisation of the reindeer meat and herring sample was performed by repeatedly grinding portions of the food item in a grinder and homogenising these portions in a mixer. The cod liver oil sample was prepared by thoroughly mixing raw and refined cod liver oil (1:2 ratio v/v). Sub-samples of at least 90 g of reindeer meat (R), 75 g of herring filet (H) were placed into carefully cleaned screw-cap glass bottles and 15 g of cod liver oil (C) were placed into crimp-cap ampoules. All samples were stored at –20 °C until shipment. The frozen samples were shipped to the participating laboratories marked as test material R, H and C.

Reporting and handling of data

Detailed instructions for participants and Excel report forms were sent out to the participants together with the samples in February 2005. For each analyte in each sample, participants were requested to report a single value for the concentration or indicate non-detected congeners by "ND". In addition, detection limits had to be given for each analyte. Concentrations were to be reported on fresh weight basis including the lipid content of the sample. Additionally, the concentrations of each analyte in the five standard solutions, determined by the laboratories' own quantification standards and methods, had to be reported.

Each participating laboratory was given a code number by the co-ordinators. Participants had access to their own code only and laboratory codes were not revealed to third parties.

On receipt by the co-ordinators, the raw data from the laboratories were entered into a database. The

draft final report was generated and made available to all participants on the Internet in July 2005. The draft of the final report was discussed at a consultation meeting at DIOXIN2005 in August in Toronto, Canada.

Statistical analysis

Based on experiences from previous rounds, we have chosen the following approach for the calculation of the consensus concentrations for each of the congeners:

For PCDDs/PCDFs and dioxin-like PCBs congener-by-congener medians were calculated from food sample data of all reporting laboratories using the detection limit as concentration for non-detected congeners (upperbound concentration). For PBDEs and indicator PCBs, non-detected congeners were removed from the data set prior to consensus calculation. Outliers were defined as those values above two times the median of all values and were removed from the data set. The consensus values were defined as the median of the remaining data for each congener. In addition, the consensus mean and standard deviation (SD) were calculated from this data set for each congener. Those congener data which had been removed prior to consensus calculation are marked in the tables presenting the individual results.

For the standard solutions, outliers were defined as those values outside ± 50% of the median of all reported values. Consensus median, mean and SD were calculated from the remaining data. The consensus of the lipid content was calculated as the mean after removal of values outside ± 2SD.

Toxic equivalents (TEQ) were calculated from the consensus values for PCDDs/PCDFs, non-ortho PCBs, and mono-ortho PCBs, using the toxic equivalency factors derived by WHO in 1998. As the detection limit was used for the concentration of non-detects, these TEQs represent upper bound concentrations.

Z-scores for PCDD/PCDF TEQs as well as for the non-ortho PCB TEQ, the mono-ortho PCB TEQ, the total TEQ, the sum of six indicator PCBs, the sum of seven PBDEs and for each singel congener were calculated for each laboratory according to the following equation:

$$z = (x - X)/\sigma$$

where x = reported value; X = assigned value (consensus); σ = target value for standard deviation. A σ of 20% of the consensus was used, i.e. z-scores between +1 and -1 reflect a deviation of ± 20% from the consensus value.

The final report and certificate

The draft of the final report was prepared by the co-ordination group and published on the Internet in July 2005. The draft was discussed at the consultation meeting at the DIOXIN2005 Symposium in August in Toronto, Canada.

In the present report, the participants are presented in the tables and figures by their laboratory codes. Each laboratory has access to its own code only and the codes are not revealed to third parties. A certificate, stating the participant's code, will be sent to each participant contributing to the results together with the printed report in autumn 2005. Further copies of the report may then be ordered from the co-ordinators for a fee covering printing and mailing costs.

Co-ordination

The study was initiated and carried out by the Department of Analytical Chemistry, Division of Environmental Medicine, Norwegian Institute of Public Health, Oslo, Norway. Members of the co-ordination committee were:

Line Småstuen Haug, Senior Engineer
line.smastuen.haug@fhi.no

Georg Becher, PhD, Department Director and Professor
georg.becher@fhi.no

Results

Results are presented in the chapters below. A participating laboratory will be able to compare its performance congener by congener with the other laboratories. Since variations in performances are based on several factors, it is recommended that each laboratory carefully evaluates the factors that, favourably or unfavourably, have contributed to its performance. A general reader of the report, who has no access to the laboratory codes, will be able to get a picture of the analytical performance of laboratories world-wide for determining dioxins, dioxin-like PCBs, indicator PCBs and PBDEs in regular foods.

Presentation in the report

87 laboratories from 28 different countries have submitted results. A summary of the results is presented in the chapter below. In Appendix C are given the consensus statistics on fresh and lipid weight basis for concentrations and TEQ values of individual congeners, a summary of TEQ values for each food item, and the z-score plots. In order to be consistent, a target deviation of ±20% was choosen for both total TEQ and all individual congener consentrations. For serveral congeners appearing at low levels in the samples, this target deviation might be to stringent. Therfore, higher Z-scores might be accepted for these congeners when evaluating the laboratories performance for their analytical uncertainty. Further, the results of the lipid determinations are presented. Finally, individual results reported by the laboratories for concentrations for each congener are given for reindeer meat, herring filet and cod liver oil in Appendix 2, 3 and 4.

Summarising comments on results

PCDDs/PCDFs

Analyte solution

Concentrations for PCDDs/PCDFs were reported by 77 laboratories. The average relative standard deviation (RSD) for the 17 congeners was 9.5% ranging from 8.3% for 1,2,3,4,6,7,8-HpCDD to 12% for 1,2,3,4,6,7,8,9-OCDF. The calculation of z-scores for the TEQs (target 13.6 pg TEQ/µl) of the PCDD/PCDF standard solution shows that 96% of the labs are within the range of ±20% of the assigned value. This demonstrates a good quality for analysis of the calibration solutions.

Reindeer meat

For the reindeer meat sample, PCDD/PCDF results from 73 laboratories were received. The consensus TEQ was 0.42pg/g fresh weight. Z-scores within ±1 were obtained by 62% of the laboratories and 88% of the laboratories had z-scores within ±2. The PCDD/PCDF TEQ level of this sample on lipid basis was 3.4 pg/g lipid thereby exceeding EU's maximum limit of 2.0 pg/g lipid for game .

Herring filet

PCDD/PCDF concentrations in the herring sample were reported by 80 laboratories. The consensus TEQ was 0.81 pg/g fresh weight. The contamination level is about 20% of EU´s maximum limit of 4 pg TEQ/g fresh weight. Z-scores were within ±1 for 84% of the laboratories and within ±2 for 95% of the laboratories. More than 90% of the PCDD/PCDF TEQ is made up by the four congeners 2,3,7,8-TCDD, 1,2,3,7,8-PeCDD, 2,3,7,8-TCDF and 2,3,4,7,8-PeCDF.

Cod liver oil

For cod liver oil 69 laboratories have determined PCDD/PCDF concentrations. The consensus TEQ was 2 pg/g both on fresh weight and lipid weight basis assuming a lipid content of 100%. This concentration is exactly at the maximum limit in force in the EU for fish oil intended for human consumption. The average RSD was quite high at 44% ranging from 17% to 88% for 2,3,7,8 TCDF and 1,2,3,4,7,8,9 HpCDF respectively. Z-scores for PCDD/PCDF TEQ within ±1 were obtained by 86% of the laboratories and 94% had z-scores within ±2.

Dioxin-like PCBs

Analyte solution

The 12 dioxin-like PCBs in the analyte solution were analysed and reported by 73 to 76 laboratories. The relative standard deviations for the different congeners were 7.8% to 10.3% with an average of 8.9%.

Reindeer meat

Dioxin-like PCB concentrations were reported from 67 to 72 laboratories. The concentrations of the 12 congeners varied between 0.48 pg/g fresh weight

(CB-169) and 402 pg/g fresh weight (CB-118). The average standard deviation for concentrations of individual dioxin-like PCB congeners on fresh weight basis was 32% ranging from 23% for CB-126 to 39% for CB-123.

Herring filet

Between 72 and 77 laboratories have measured and reported dioxin-like PCB concentrations in herring. The concentration ranged from 1.1pg/g fresh weight to 1.0 ng/g fresh weight. The dioxin-like PCBs contributes to about 50% of the total TEQ in the sample. The main contributor is PCB 126 which makesup 76% of the PCB TEQ.

Cod liver oil

Dioxin-like PCBs were reported by 63 to 68 laboratories. Levels were relatively high ranging from 4.3 pg/g fresh weight to 17 ng/g fresh weight. RSDs were correspondingly low with an average of 23%. The contribution of the dioxin-like PCBs to the total TEQ was 85%. This may be explained by the fact that the sample partly contained refined cod liver. In the refining process, PCDDs/PCDFs are removed to a larger extent than PCBs thereby increasing the PCBs contribution to the TEQ.

Total TEQ

In Figure 1, the contribution of the three groups of dioxin-like compounds is depicted. For all three sample types, dioxin-like PCBs contributed to more than 50% of the total TEQs demonstrating the importance of PCBs for the determination of the total 2,3,7,8-TCDD related toxic potency of food samples.

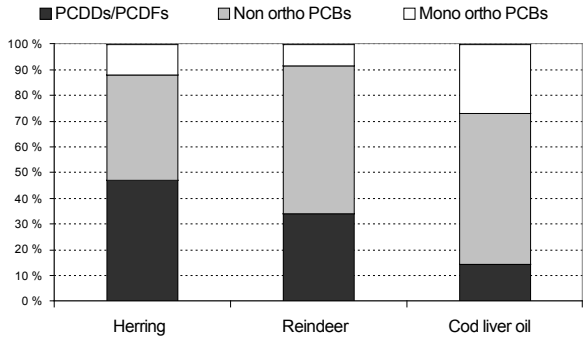

Figure 1. *Contribution of PCDDs/PCDFs, non-ortho PCBs and mono-ortho PCBs to the total TEQ in the food samples used in this interlaboratory comparison.*

The RSD for total TEQ on fresh weight basis as calculated from the RSD of individual congeners was 13.5% for reindeer, 10.1% for herring and 9.8% for cod liver oil.

Indicator PCBs

Analyte solution

In the analyte solution indicator PCBs were reported by 61 laboratories. The average relative standard deviation was 12% ranging from 9.5% to 13% which is slightly higher than for PCDD/PCDFs and the dioxin-like PCBs.

Reindeer meat

For the reindeer meat sample indicator PCB results were received from 57 laboratories. The concentrations were low, varying between 23 pg/g fresh weight and 549 pg/g fresh weight. The relative standard deviations were correspondingly high ranging from 31% to 45% .

Herring filet

Within the deadline 61 laboratories reported results of indicator PCBs in herring. The concentrations ranged form 0.28 ng/g fresh weight to 2.9 ng/g fresh weight. The average relative standard deviation was 28% ranging from 24% to 32%.

Cod liver oil

Reports were obtained from 56 laboratories. The concentrations of indicator PCBs in the cod liver sample was high, ranging from 2.5 ng/g fresh weight to 29 ng/g fresh weight. The average relative standard deviation was 27% ranging from 22% to 35% for CB-153 and CB-28, respectively.

PBDEs

Analyte solution

The PBDE standard solution was analysed by 31 to 33 laboratories for BDE-28 to BDE-183, but only 21 laboratories reported values for BDE-209. The RSD was between 8.0% to 13% for the first seven congeners while it was 18% for BDE-209.

Reindeer meat

PBDE concentrations were reported by 32 to 33 laboratories, except for BDE-209 for which only 18 results were received. The consensus concentrations (median with outliers and NDs removed) were quite low, varying between 1.6 pg/g fresh weight for BDE-28 and 85 pg/g fresh weight for BDE-209. The concentration for BDE-209 must be regarded as indicative. The sum of tri- to heptaBDEs was 37 pg/g fresh weight. The range of standard deviations on fresh weight was 22-70% with an average of 44%, excluding BDE-209.

Herring filet

Within the deadline 36-38 laboratories had reported results for tri- to hepta BDEs. The concentrations varied between 2.3 pg/g fresh weight and 395 pg/g

fresh weight. The sum of tri- to heptaBDEs was 642 pg/g fresh weight. The RSD calculated from the concentrations on fresh weight ranged from 28 to 34% with an average of 31% for the tri- to heptaBDEs.

Cod liver oil

Between 31 and 32 laboratories reported results for tri- to hepta BDEs and 18 reported results for BDE-209. The sum of tri to hepta-BDEs was 13.6 ng/g fresh weight. The RSDs for the individual congeners were ranging from 16 to 42% with an average of 29%, excluding BDE-209.

Lipid content

The mean and relative standard deviations (in parentheses) for the lipid contents were calculated to 12.4% (14.1%) for reindeer meat and 11.3% (7.59%) for herring. The lipid content of the cod liver oil was assumed to be 100%. The distribution of z-scores shows that several laboratories have reported a far too low lipid content for the reindeer which indicates that they have not been able to extract the lipids from the meat tissue efficiently.

Acknowledgements

The laboratories are acknowledged for their participation in this interlaboratory comparison and their interest in its overall objectives, thereby making it clear that they value good analytical performance. All the individual analysts are acknowledged for their contributions to the results.

We are grateful to Cambridge Isotope Laboratories, Inc. for providing the standard solutions for this interlaboratory study. We thank Marie Aune at the National Food Administration in Sweden for providing us the Baltic herring and the Oslo based company Peter Möller AS for the cod liver oil.