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Harmonizing methods for the determination of dioxins in food

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Content

Preface

The project no 653040 – 30412 on “Harmonizing methods for sampling, sample preparation, co-ordination of investigations and method development of dioxins and dioxin-like PCBs in food” was initiated in 2004 with the participation of all the Nordic countries including Denmark – as project manager – and the Faroe Islands, Finland, Iceland, Norway and Sweden. The project was finalised by the end of 2005 although some minor follow up activities and this report were completed in 2006.

In 2004, the current regulation of sampling and analytical methodology with respect to dioxins was the EU directive 2002/69/EC that was laying down the sampling methods and the methods of analysis for the official control of dioxins and the determination of dioxin-like PCBs in foodstuffs. However, the directive was not sufficiently precise and detailed which resulted in different interpretations in the Nordic countries. Furthermore, guidelines for sample preparation were not included in the directive. It is important for the comparison of analytical results from different laboratories that sample preparation is performed in a comparable way. It is quite clear that the Nordic countries would benefit by having a harmonised way to assess the analytical results and the analytical uncertainty. It should be possible by an active exchange of information to coordinate the national investigations of dioxin and dioxin-like PCBs in food and food products so that information on the dioxins in food is obtained as complete as possible. This is especially important since the EU limit values for dioxin and dioxin-like PCBs were going to be revised in 2006.

Since the introduction of harmonised EU limit values for dioxins and the coming limit values for dioxin-like PCBs the need for analysis of dioxins and PCBs in the Nordic countries as well as in other European countries has increased markedly. In spite of the increasing demand for analysis, it is still only a limited number of laboratories in the Nordic countries that are able to undertake the analytical challenge.

Summary

Methods for sampling and sample treatment as well as the analytical methodology for determination of dioxin and dioxin-like PCBs in food have been discussed and evaluated in 2 workshops in Oslo and Copenhagen respectively with participation from all Nordic countries. The first workshop was mainly focused on sampling problems but did also include the analytical methodology. However, both issues were included and discussed rather intensively during the second workshop that took place in Copenhagen one year later in November 2005.

The workshops found that recent amendments of the EU-regulation and guidelines for sampling have solved some of the problems that gave raise to difficulties in the interpretation and application of the guidelines. It was concluded that in general the guidelines appears to fit their purpose. However, the specific provisions in the draft directive for sampling of lots containing fishes of different size or weight seem to be rather complicated and difficult to apply in a standardised and uniform way. The workshops agreed that it is difficult since the definition of different fish size and weight probably has to be linked to the individual species and possibly even to the different catching areas.

Information on the equipment and analytical methods used by the Nordic dioxin laboratories were compiled through a questionnaire. The replies that are included in an annex to this report are an original and important result of the project. They are describing the analytical capacity and possibilities in the Nordic countries and they reflect the status and quality of the analytical work. The questionnaires also indicate that the analytical methodology used by the Nordic dioxin laboratories is in good agreement with EU guidelines and other international standards and recommendations. An evaluation of the results from the Interlaboratory Comparison on Dioxin in food 2005 was taken as a further indication that the analytical performance and quality of the Nordic dioxin laboratories are at a high international level and in general fully acceptable. It was therefore concluded that there is no need for further harmonisation of the methodology.

As a final observation by the workshops, it was pointed out that a direct and more binding cooperation between the Nordic countries on dioxin monitoring and control projects was inappropriate for scientific, administrative and financial as well as other reasons. However, it was equally pointed out that an improvement of the dialogue and exchange of information concerning dioxin and dioxin-like PCBs would be of interest for all the Nordic countries as well as the countries around the Baltic Sea.

Sammendrag
(Summary in Danish)

Metoder til prøveudtagelse og analysemetodik til bestemmelse af dioxin og dioxin-lignende PCBer er diskuteret og evalueret ved to workshops i henholdsvis Oslo og København med deltagelse fra alle de nordiske lande. Den første workshop var primært fokuseret på problemer knyttet til prøveindsamling og omfattede i mindre grad analysemetodik. Til gengæld blev begge emner gennemgået og diskuteret intensivt ved den anden workshop.

Der var ved de to workshops enighed om, at de senere forbedringer af EU's regulering og guidelines for prøveudtagelse havde løst nogle af de problemer, der har givet anledning til vanskeligheder ved fortolkning og anvendelse af regelsættet. Det blev konkluderet, at de nugældende guidelines generelt set syntes at være »fit for the purpose«. Samtidig blev det dog påpeget, at de særlige bestemmelser i udkast til direktiv vedr. prøveudtagelse fra partier af fisk af forskellig størrelse og vægt virker komplicerede og vanskelige at anvende på en ensartet måde. Det var imidlertid ikke muligt at udarbejde en alternativ procedure i løbet af de to workshops, hvor man indså vanskelighederne, der bl.a. skyldes at karakterisering af de enkelte partier i form af størrelse og vægt sandsynligvis skal variere fra art til art og sandsynligvis også vil afhænge af fangstområderne.

Oplysninger om udstyr og analysemetodik i de nordiske dioxinlaboratorier blev indsamlet ved hjælpe af et spørgeskema. Svarerne, der er inkluderet i et anneks til denne rapport, er et originalt og vigtigt resultat af projektet. De beskriver kapaciteten og de analytiske muligheder i de nordiske lande og afspejler kvaliteten af det analytiske arbejde. Spørgeskemaerne viser også, at den analysemetodik, der anvendes i de nordiske lande, er i god overensstemmelse med EU-guidelines og andre internationale standarder og anbefalinger. En evaluering af resultaterne fra en større interkalibrering af dioxin i fødevarer 2005 blev vurderet som en yderligere indikation af, at kvaliteten af det analytiske arbejde i de nordiske dioxin-laboratorier er på et internationalt niveau og generelt set fuldt acceptabelt. Det blev derfor konkluderet, at der ikke er behov for yderligere harmonisering af metodikken.

Som en sidste observation fra de to workshops blev det påpeget, at et mere direkte og forpligtende samarbejde mellem de nordiske lande om dioxinovervågning og -kontrolprojekter næppe ville være hensigtsmæssigt af faglige, administrative og økonomiske grunde. Imidlertid blev det samtidig understreget, at en udbygning af dialogen og udveksling af op-

lysninger om dioxin og dioxin-lignende PCB'er vil være af interesse for alle de nordiske lande så vel som for landene omkring Østersøen og den Botniske bugt.

1. Introduction

The present project has been undertaken as one of three projects that are initiated and financed by the Nordic Council of Ministers. The projects are focused on food safety aspects of dioxin covering different parts of the problem. One of the projects entitled “Dioxin and dioxin-like PCBs in the Nordic countries – intersectorial network for coordination of actions” is coordinated by Sweden. It is aiming to further develop and improve the Nordic dioxin network and to form an Internet based platform for exchanging information of common interest to the countries. The other project is coordinated by Norway and entitled “Review of maximum levels for dioxins and dioxin-like PCBs, impact on the consumer exposure and the food supply”. It is focused on exposure and risk assessment aiming – if possible – to develop a common Nordic position to the setting of EU maximum limits for dioxin in food. The two projects will be reported separately and will not be discussed further in this report.

The objective of the project to be reported here is to harmonise the interpretation of EU directive 2002/69 laying down the sampling methods and the methods of analysis for the official control of dioxins and the determination of dioxin-like PCBs in foodstuffs. Evaluation and – if needed – establishing procedures for treatment of samples are an integrated part of the project, as well as the coordination of on-going dioxin investigations in the Nordic countries. Furthermore, the objective of the project is to identify methods currently used in the Nordic countries to measure dioxin and dioxin-like PCBs in foodstuffs. The information and experiences from the project together with the coordination of the analytical methodology aim to form a basis for an improvement of the analytical investigations and to assure the reliability and comparability of the analytical data.

2. Activities

The main activities of the project included 2 workshops. The first workshop was arranged in November 2004 in Oslo by the Norwegian coordinator of the project on maximum levels for dioxins, as a joint workshop between the 2 projects. The second workshop was held in Copenhagen one year later in November 2005 and only dealt with the project on sampling and analytical methodology. The contents and results of the 2 workshops with respect to the project on sampling and analytical methodology are summarised in the following paragraphs:

2.1 Oslo workshop, 3–4 November 2004

18 participants including 2 from Denmark, 1 from the Faroe Islands, 3 from Finland, 1 from Iceland, 7 from Norway and 4 from Sweden attended the meeting. The list of participation is included in the annex 1.

The scientific content of the workshop included:

- An introductory presentation by Tommy Cederberg, Denmark, to the EU directive 2002/69/EC on sampling methods and methods of analysis for the official control of dioxins and the determination of dioxin-like PCBs in foodstuffs. The discussions following the presentation were focused on the understanding and interpretation of the definitions in the directive. In relationship to this, the workshop went through and discussed the latest EU document SANCO/0074/2004 aiming to clarify the interpretations.
- Presentations by the participating countries of the analytical methodology in use for their national control of dioxins in food were followed by discussions on the advantages and disadvantages of the methods. Furthermore, various problems in relation to sample treatment and work-up procedures were discussed and evaluated as to their influence on the comparability between analytical results from different laboratories.
- Presentations by the participating countries of their national control and monitoring programmes were followed by a discussion of the scientific possibilities for a comparison of the results.
- Discussion of the possibilities for improving the exchange of information about on-going dioxin-investigations in the Nordic countries, and furthermore discussion of the need and possibilities from a technical and administrative point of view for a coordination of the investigations.

- The future project activities.

The main conclusions from the workshop can be summarised as follows:

- The uncertainty as to an unambiguous interpretation and common understanding of the sampling directive 2002/69/EC is mainly connected to the sampling of fish. However, minor problems as to the sampling of milk, meat and eggs need to be clarified, and there is a strong need for a better specification of the method for determining of the fat content of meat. Problems with respect to sampling and analyses of fruit and vegetables are estimated to be rather limited, taking into account the low contribution from this type of food to the total human intake of dioxins.
- The document SANCO/0074/2004 is a step in the right direction, although far from all problems are solved by the present version of the draft directive.
- The workshop agreed, in accordance with the EU-guidelines, that the use of high resolution gas chromatography combined with high resolution mass spectrometry (HR-GC/MS) is mandatory for a reliable and safe determination of dioxins for the control of compliance with the existing EU limit values. However, the workshop agreed as well that less specific methods with a higher analytical uncertainty, like the so-called CALUX method or low resolution GC/MS, can be used as preliminary screening methods provided that compliance of results close to limit values are verified by use of HR-GC/MS.
- The workshop was pleased to conclude that Denmark, Finland, Norway and Sweden have laboratories that are well equipped with HR-GC/MS. The Faroe Islands and Iceland are using well-recognized foreign laboratories having the same equipment at their disposal.
- It was decided to carry out an additional survey of the analytical possibilities in the Nordic countries in terms of an enquiry in 2005 to the Nordic dioxin laboratories. A questionnaire should be worked out asking for information on the analytical methodology, status for ISO17025 accreditation, detection limits, congener profile etc.. It was further decided to include questions on the status for the analytical determination of Brominated Flame Retardants (BRFs).
- Evaluation of the various national investigations up to now has demonstrated that a comparison of the data is rather difficult without more detailed information about differences in the analytical parameters of the investigations. Especially the analytical uncertainty is very important.
- The workshop agreed that a more direct and binding cooperation between the Nordic countries on future dioxin investigations would be rather impossible for scientific, administrative and financial reasons. However, it was equally pointed out that an improvement of the dialog

and exchange of information concerning dioxin and dioxin-like PCBs would be of interest for all the Nordic countries.

As for the coming activities, it was recommended by the workshop to continue the evaluations and discussions on sampling and methodology in 2005, whereas the need for improving the dialog and exchange of information between the Nordic countries should be addressed by the parallel ongoing project on "Dioxin and dioxin-like PCBs in the Nordic countries – intersectorial network for coordination of actions".

2.2 Copenhagen workshop, 14–15 November 2005

The continuation of the project in 2005 was unfortunately delayed due to administrative problems. It was the intention at the Oslo workshop that it should have been followed up by two other workshops in 2005, one dealing with sampling and one dealing with analytical methodology. However, the delay made it difficult to assemble qualified participants for two meetings. Competitive meetings arranged in the 2 parallel dioxin projects did not make it easier to find suitable dates for the workshops. It was therefore decided by the project manager to arrange only one workshop as a 2-day meeting dealing with sampling on the first day and analytical methodology on the second.

As decided at the Oslo workshop questionnaires on the analytical methodologies including sample treatment and analytical measurements were prepared and sent by the project manager to the Nordic dioxin laboratories in order to qualify the discussions at the workshop. The questionnaires were forwarded to the national contact points of the project and distributed from here to the laboratories. Later on, the replies were submitted to the project manager via the contact points.

In total 15 participants including 6 from Denmark, 1 from the Faroe Islands, 1 from Finland, 2 from Island, 2 from Norway and 1 from Sweden attended the meeting. In the light of the political intention in the Nordic countries to improve the cooperation with the Baltic countries, Estonia, Lithuania and Latvia were invited to participate in the workshop, but only Estonia with 2 persons accepted the invitation. The complete list of participants is included in the annex 1.

2.2.1 Sampling and sample preparation

The first day of the workshop dealt with sampling and sample preparation and included the following topics:

- Tommy Cederberg, Denmark, acting as chairperson of the day, began the workshop with a summary of the project work up to now followed by a specification of the objectives of the workshop.
- As an introduction to the work, Mette B. Hansen, Denmark, gave a presentation of the status for the regulatory work in the EU Commission on dioxin and dioxin-like PCBs. She particularly dealt with the revision of the EU dioxin maximum levels and the establishment of new maximum levels for the sum of dioxin and dioxin-like PCB in food. Furthermore, she did mention that together with the revision of the maximum levels, sampling methods and methods of analysis have been discussed. She underlined, however, that the draft Commission Directive amending Directive 2002/69/EC (the SANCO/002698/2005) was not yet up for a vote. The draft contains specifications with regards to sampling and sample preparation of fish in order to ensure a harmonised approach throughout the Community (changes to Annex 1 and 2). The draft will be discussed at future working group meetings in the Commission. In addition, adjustments to the EU monitoring plan for dioxin and PCB in food will also be on the agenda.
- Tommy Cederberg gave an overview of the sampling methods for the official control or monitoring of dioxins in foodstuffs. Starting from the directive 2002/69/EC on sampling that was debated at the 2004 workshop, he summarised the principles behind the rules and focused further on the experiences that were obtained since the adoption of the directive. I addition, he discussed the amendments in Directive 2004/44/EC, which is to be considered as an updating of the former 2002-directive. Finally, he summarised and discussed the recommendation in the draft directive SANCO/002698/2005.

The presentations by Mette B. Hansen and Tommy Cederberg were followed by a long and lively round table discussion of sampling and the comparability of monitoring and control data. Based on the debate, it was noted that:

- With the exception of fish, the sampling procedures described in the Directive generally appear to fit the purpose. However, for homogenous food items as mentioned in the Directive e.g. milk or oils, it seems to be inconsequent to still require three incremental samples to form the aggregate sample, and that the minimum amount should be 1 kg.
- When it comes to the comparability between countries of the results from food monitoring and control analyses, the situation is more complex than looking at the sampling methods described in Directive 2002/69. Other Directives e.g. Directive 96/23 and certain national legislation overrule Directive 2002/69. This leads to different

sampling strategies, where e.g. some countries take samples from slaughterhouses but are pooling samples from different animals and from different farms. Other countries take samples from only one animal. Obviously these types of results are difficult to compare, as the range of concentrations can be expected to be quite different. At the workshop it was not possible to get a full overview with regards to the actual sampling strategy in the Nordic countries. It was put forward as an option to send out a questionnaire, so each country could gather information from relevant people and institutions.

- The comments to the draft Directive concerning fish sampling and sample preparation gave a general acceptance of the proposed definition of the middle part of the fish. It was also agreed that from an analytical point of view the described procedure for the removal of the skin and the scraping of remaining fat and tissue is the best way to assure representative sample preparation.
- The comments were quite negative to the additions to point 4 of the draft directive concerning "sampling plans" with specific provisions for sampling of lots containing whole fishes. The provision tries to define when a lot contains fish of different size or weight, and how to determine when a specific size or weight of fish is representative for the entire lot.
- Although it is easy to criticise the draft proposals and to find the weaknesses, it is not straightforward to come up with a better solution. The problem is that the definition of different size and weight probably has to be linked to the individual fish species and possibly even to the different catching areas. Another difficulty is that an actual control of maximum limits of an entire "lot" of fish is a rare occasion. Most countries are conducting surveys of fish for content of dioxin and PCB, and if violations are observed, certain fish, fish sizes or catching areas are banned. If surveys of fish could be incorporated in the Directive as a mean of controlling maximum limits, this could solve the problems with the sampling of fish.

As the last part of the programme on the first day, Ott Roots, Estonia, gave a presentation entitled "Dioxins in the Baltic herring and sprat in Estonian costal waters". He described an investigation of dioxins in fish on the Estonian marked that was initiated by the food authorities in Estonia and carried out using a qualified dioxin laboratory in Germany for the analytical work. Besides the discussion of the investigation and the comparability of the data with data from the Nordic countries, the workshop agreed that the dioxin contamination of the Baltic Sea is a common problem for all the countries around the sea and that it is an obvious example of a problem that should be solved in a close cooperation between all the countries.

2.2.2 Analytical methodology

Arne Büchert, Denmark, was acting as chairperson on the second day of the workshop that was dealing with analytical methodology and included the following topics:

- An opening presentation by Kirsten H. Lund and Søren Sørensen, Denmark, on the "Analysis of dioxin – experiences from a Danish Laboratory". The background and work for the establishment of the Danish dioxin laboratory at the Regional Veterinary and Food Administration in Ringsted. The considerations for the analytical methodology to be used and the experiences up to now with training in and validation of the selected methodology, including the achievement of the ISO17025 accreditation by the Danish accreditation body, were described. Advantages and disadvantages of the selected methodology were discussed
- A presentation by Arne Büchert, Denmark, of the results of the enquiries for more detailed information on the analytical possibilities in the Nordic countries. In total 10 replies to the questionnaires were received at the time of the workshop. This included 2 from Denmark, 1 from Finland, 2 from Norway and 4 from Sweden. Iceland had communicated a reply from the German laboratory ERGO in Hamburg. The ERGO laboratory – today a part of the Eurofins laboratory chain – is used by Iceland to carry out the analytical work in connection with the official control and monitoring of dioxins in Iceland. The Faroe Islands also use the ERGO-laboratory for their control and monitoring work. (An additional reply from another Norwegian laboratory was received after the workshop). During the following discussion of the presentation, the workshop pointed at the following conclusions that seems to characterise the present status for the analytical competence and capability of the Nordic laboratories with respect to the analyses of dioxin and dioxin-like PCBs in foodstuffs:
    - The combined questionnaires are in all a valuable result of the project, reflecting the analytical status and capability of the Nordic dioxin laboratories and to some extent also the capacity and possibility for the food authorities to perform the official control and monitoring programmes as well as other specific directed dioxin investigations.
    - Denmark, Finland, Norway and Sweden have laboratories holding an ISO17025 accreditation for the determination of dioxin and dioxin-like PCBs in food and food products including milk, dairy products, fish, egg and products of animal origin.
    - Only Finland and Norway have laboratories that have received an ISO17025 accreditation for determination of Brominated Flame Retardants in food and food products.

- Most of the laboratories are using analytical methods that are based on method no. 1613 that is developed and validated by the American EPA and defined as the reference method of choice in the EU directives.
- The questionnaires reflect a certain variation between the laboratories as to the amount of sample, extraction solvents and clean-up procedures:
  - Depending of sample type, the amount of sample varies between 0,5 to 150 g pr analysis, as the amount normally corresponds to 0,5 to 3 g of fat.
  - Also depending on sample type, there is a variation between the laboratories in their use of solvents for extraction of the samples. This raises the question of the need for harmonisation. However, the opinion of the workshop was that the type and amount of solvent is not important as long as the efficiency of the extraction is acceptable.
  - The clean-up procedures were in all laboratories based on column-techniques. The variation between the laboratories was the degree of automation of the procedures, as some of the laboratories were using Automatic Solvent Extraction (ASE) and Power Prep techniques. The workshop agreed that automation on its own does not necessarily improve the analytical quality, as compared to the older manual clean-up procedures. It was concluded that there is no need for harmonisation, since automation is a question of the number of samples and the economy of the individual laboratory.
- The determination of dioxin and dioxin-like PCBs is in all laboratories performed using combined gas chromatography – mass spectrometry with Electron Impact (EI) ionisation at a high resolving power (usually > 10.000), for which reason the determination step is in fact harmonised.
- All laboratories are using C13-marked standards and peak areas for the quantification of the GC-signal. The workshop emphasized the need for quality assurance of the standards, since impurities in them can cause errors in the determination of the individual congeners.
- The procedure for the calibration of the system varies between the laboratories. Some laboratories use single-point calibration, whereas others use multi-point calibration. However, the workshop agreed that this difference is not crucial for the quantification, and for which reason there is no need for harmonising the procedures.
- The workshop noted that detection limits (LODs), analytical uncertainty, repeatability and reproducibility are at comparable levels in the individual laboratories:

  - Detection limits are usually at a level of 0.1 to 1.0 pg WHO-TEQ/g fat.
  - Analytical uncertainty is by most of the laboratories stated as 15–25%
  - Repeatability is usually in the area of 10–20%, whereas the reproducibility is at a 5 to 10% higher level.

- As an introduction to further discussions of the questionnaires, Claudette Bethune, Norway, presented a draft report of the so-called DIFFERENCE-project on dioxins in food and foodstuffs. This project has been carried out under the 6th EU Framework programme in collaboration between several European dioxin laboratories including a few Nordic laboratories. The project is among other issues dealing with the quality of the analytical methodology. The workshop concluded that the standards of the Nordic dioxin laboratories were in good agreement with the observations and recommendations of the DIFFERENCE-project.
- The presentations of the questionnaires and the DIFFERENCE project were followed by a round table discussion of the advantages and disadvantages of the analytical methodology used by the Nordic dioxin laboratories. The workshop concluded that all the laboratories are using methods of acceptable quality with proper quality assurance and quality control in agreement with international recommendations and standards, including the requirements given in the EU directives. As a consequence, it was concluded that the need for (further) harmonisation of the analytical methodology is rather limited. It was suggested that an evaluation of data from the latest intercalibration study – the Interlaboratory Comparison on Dioxins in Food 2005 arranged by the Norwegian Institute of Public Health – might give further information about possible differences between the analytical methodologies used by the Nordic laboratories. Line Småstuen Haug, from the Norwegian institute, accepted to look for data for such an evaluation. The workshop furthermore agreed that the questionnaires being an individual and valuable result of the project should be made available to the laboratories and the Nordic food authorities by publication on the Internet or by other means provided that the laboratories can accept such a publication.

2.3 Follow-up of the workshops

It was concluded at the workshop in Copenhagen that there was no need for further meetings or workshops. However, it was important to follow-up on some of the conclusions from the workshop before a report could be made and the project finalised. This follow-up included an evaluation

of data from the Interlaboratory Comparison of Dioxins in Food and an acceptance from the laboratories to publish the questionnaires on the Internet.

As agreed upon in Copenhagen, Line Småstuen Haug performed the evaluation of data from the interlaboratory study. However, comparing the results of the Nordic laboratories from analysis of three types of samples (herring, reindeer and cod liver oil) with the results from all participating laboratories and the results from all European laboratories respectively did not show any significant differences although the Nordic results for reindeer are slightly better – but still not significant – in terms of a higher percentage of the results with a Z-score between ±1 than the results from the other laboratories.

The evaluation included an assessment of the determination of the fat content of reindeer and herring. The results from the Nordic laboratories were also here found to be better in terms of a lower relative standard deviation (RSD) of the results. However, it should be taken into account that a better fat determination does not necessarily result in a better determination of the dioxins.

Since the evaluation of the data from the interlaboratory study did not reveal any significant differences between the laboratories, it was concluded that it could be interpreted as additionally evidence that the performance of the Nordic dioxin laboratories is at the international level with an acceptable quality.

Acceptance from the laboratories to publish the questionnaires at the Internet were asked for by letter via the national contact points to all the laboratories. Positive commitments were obtained from all laboratories including 2 questionnaires from Denmark, 1 from Finland, 3 from Norway, 5 from Sweden and 1 from the German laboratory that has been used by Iceland and the Faroe Islands. The completed questionnaires are included in the annex 2.

3. Conclusions

Methods for sampling and sample treatment as well as the analytical methodology for determination of dioxin and dioxin-like PCBs in food have been discussed and evaluated in 2 workshops. The first that was arranged in November 2004 in Oslo was mainly focused on sampling problems but did also include the analytical methodology. However, both issues were included and discussed rather intensively during the second workshop that took place in Copenhagen one year later in November 2005.

As an important part of the project, information on the equipment and analytical methods used by the Nordic dioxin laboratories to measure dioxin and dioxin-like PCB in foodstuffs were compiled through a questionnaire. The replies that are attached in the annex 2 to this report are reflecting the analytical possibilities in the Nordic countries and the status and quality of their analytical potential.

Based on the evaluations and discussions during the workshops the main conclusions are summarised as follows:

- The need for harmonisation of the interpretation and understanding of the “sampling directive” 2002/69/EC is mainly connected to sampling of fish. Minor problems with the fat content determination of the sample exist for milk and meat products, whereas problems with respect to samples of fruit and vegetables are considered less important in the light of their limited contribution to the total dioxin intake.
- It was agreed that the amendment directive 2004/44/EC and the draft directive SANCO/002698/2005 have solved some of the problems and generally appears to fit their purpose. However, the specific provisions in the draft directive for sampling of lots containing fishes of different size or weight seem to be rather complicated and difficult to apply in a standardised and uniform way.
- It was not possible during the workshops to work out an alternatively procedure for sampling of lots of fishes. The workshops agreed that it is difficult since the definition of different fish size and weight probably has to be linked to the individual species and possibly even to the different catching areas. It was recommended that the EU-Commission should discuss the problem and aim to develop a better procedure.
- Comparison of results from national dioxin monitoring programmes and control analysis from the different Nordic countries (and other countries) should be based on detailed information of the sampling

strategies used for the investigations. It was not possible during the workshops to get a full overview of the actual strategies in the Nordic countries, for which reason it was recommended to compile such information at a later stage.

- In accordance with the EU-guidelines the workshops agreed that sample treatment and work-up procedures should be made with a quality similar to the quality described in EPA method no. 1613, which is noted as reference method in the EU-guidelines. The questionnaires reflect that most of the Nordic laboratories are using such methods.
- It was agreed at the first workshop that compliance with EU limit values for dioxin and dioxin-like PCBs should be based on measurements using High Performance Gas Chromatography combined with High Resolution Mass Spectrometry (HR-GC/MS) in accordance with established EU-guidelines. However, it was also accepted that less specific methods, like the so called CALUX method or Low Resolution GC/MS, can be used as screening methods provided that results close to the limit value are verified by HR-GC/MS.
- It was pointed out that a better specification of the method for the determination of the fat content of meat is needed.
- Denmark, Finland, Norway and Sweden have laboratories that are equipped with HR-GC/MS, while Iceland and the Faroe Islands are using foreign laboratories having the same instruments at their disposal.
- The information on the equipment and analytical methods used by the Nordic dioxin laboratories to measure dioxin and dioxin-like PCB in foodstuffs that were compiled through the questionnaire is an original and important result of the project, reflecting the analytical possibilities in the Nordic countries and the status and quality of their analytical potential. All the laboratories that have returned the questionnaires have accepted that the information can be published at the Internet.
- Although the questionnaires are focused on dioxin, they also show that at present only Finland and Norway have laboratories that possess an ISO17025 accreditation for the determination of Brominated Flame Retardants (BFR) in foodstuffs.
- It is strongly indicated by the questionnaires, that the analytical methodology used by the Nordic dioxin laboratories is in good agreement with the observations and recommendations from the so-called DIFFERENCE project on dioxins in food and feed. The DIFFERENCE project was financed by the EU and carried out in cooperation with several European dioxin expert laboratories. The recommendations from this project have had a strong impact on the

formulation of the EU-guidelines for the determination of dioxins and dioxin-like PCBs.

- Based on the questionnaires and the observations and recommendations in the DIFFERENCE project, it was concluded by the workshop that all Nordic dioxin laboratories are using proper analytical methods in good agreement with international standards, including the EU-guidelines. It was therefore further concluded that there is no need for further harmonisation of the methodology between the Nordic laboratories.
- An attempt was made to compare the analytical performance of the Nordic laboratories by evaluating the results from the Interlaboratory Comparison on Dioxin in food 2005 that was arranged by the Norwegian Institute of Public Health. However, it was not possible to find any significant differences between the Nordic results compared to all the other results or to the results from the European laboratories that were participating in the study. This finding was taken as a further indication that the analytical performance and quality of the Nordic dioxin laboratories in general are fully acceptable at the international level and that there is no need for further harmonisation of the methodology.
- As a final observation by the workshops, it was pointed out that a direct and more binding cooperation between the countries on dioxin monitoring and control projects was unrealistic for scientific, administrative and financial as well as other reasons. However, it was equally pointed out that an improvement of the dialogue and exchange of information concerning dioxin and dioxin-like PCBs would be of interest for all the Nordic countries as well as the countries around the Baltic Sea.

4. Recommendations

Based on the conclusions, the following topics are pointed out as the main recommendations for future work to improve the investigations of dioxin and dioxin-like PCBs in the Nordic countries:

- At the Nordic level, a compilation of more detailed information on the sampling strategies in the Nordic countries should be considered as an aim to better evaluate the comparability of data from the individual Nordic dioxin laboratories.
- At the European level, a revision of the procedure for sampling of lots of fish of different size and weight should be discussed and – if possible – developed and adopted by the EU-Commission.
- The need for further harmonisation of the analytical methodology is at present rather limited. However, a better specification of the method for the determination of the content of fat in meat samples would be an advantage.
- The dialogue on dioxins and the exchange of information between the Nordic countries should be improved and extended to include the Baltic countries.
- The number of laboratories with an ISO17025 accreditation for the determination of Brominated Flame Retardants (BFR) in food is at present rather low and should be increased.

References

EU directive 2002/69 laying down the sampling methods and the methods of analysis for the official control of dioxins and the determination of dioxin-like PCBs in foodstuffs

EU directive 2004/44/EC amending Directive 2002/69/EC

SANCO/0074/2004, draft conclusion of meeting on 28 September of the working group “Sampling and Sample Preparation of Fish for the Control on the Presence of Dioxins and PCBs”

Commission regulation (EC) No 199/2006 amending regulation (EC) No 466/2001 setting maximum levels for certain contaminants in foodstuffs as regards dioxin and dioxin-like PCBs

Interlaboratory Comparison on Dioxins in Food 2005 arranged by the Norwegian Institute for Public Health (www.fhi.no).

DIFFERENCE: Optimisation and validation of screening methods for the analysis of dioxins and dioxin-like PCBs in food and feed and the production of certified reference materials
S.P.J. van Leeuwen, J. Van Loco, J. de Boer, Organohalogen Compounds 60 (2003), 267–270

The international validation of bio- and chemical-analytical screening methods for dioxins and dioxin-like PCBs: the DIFFERENCE projects rounds 1 and 2
J.Van Loco, S.P.J. Van Leeuwen, P. Roos, S. Carbonnelle, J. de Boer, L. Goeyens, H. Beernaert
Talanta 63 (2004) 1169–1182

Annex 1: Participation in the workshops

Oslo workshop 4–5 November 2004

Country	Name	Affiliation
Denmark	Arne Büchert	Danish Institute for Food and Veterinary Research
	Tommy Cederberg	Danish Institute for Food and Veterinary Research
Faroe Islands	Maria Dam	Food, Veterinary and Environmental Agency of The Faroe Islands
Finland	Anja Hallikainen	Finnish Food Safety Authority Evira
	Liisa Rajakangas	Ministry of Trade and Industry of Finland
	Panu Rantakokko	National Public Health Institute of Finland
Iceland	Kristín Ólafsdóttir	Environment and Food Agency of Iceland
Norway	Are Sletta	Norwegian Food Safety Authority
	Christina Bergsten	Norwegian Food Safety Authority
	Marie Louise Wiborg	Norwegian Scientific Committee for Food Safety
	Line Småstuen Haug	Norwegian Institute of Public Health
	Helle K. Knutsen	Norwegian Institute of Public Health
	Martin Schlabach	Norwegian Institute for Air Research
	Claudette R. Bethune	National Institute for Nutrition and Seafood Research
Sweden	Östen Anderson	National Food Administration of Sweden
	Per Ola Danerud	National Food Administration of Sweden
	Marie Aune	National Food Administration of Sweden
	Petra Bergkvist	National Food Administration of Sweden

Copenhagen workshop 14–15 November 2005

Country	Name	Affiliation
Denmark	Arne Büchert	Danish Institute for Food and Veterinary Research
	Tommy Cederberg	Danish Institute for Food and Veterinary Research
	Rikke L. Bille	Danish Institute for Food and Veterinary Research
	Kirsten H. Lund	Danish Veterinary and Food Administration - Regional Control Center Ringsted
	Søren Sørensen	Danish Veterinary and Food Administration - Regional Control Center Ringsted
	Mette B. Hansen	Danish Veterinary and Food Administration
Estonia	Siret Dreyersdorff	Estonian Ministry of Agriculture
	Ott Roots	Estonian Environmental Research Center
Faroe Islands	Elsba Danielsen	Food, Veterinary and Environmental Agency of The Faroe Islands
Finland	Hannu Kiviranta	National Public Health Institute of Finland
Iceland	Ingólfur Gissurarson	Environment and Food Agency of Iceland
	Kristín Ólafsdóttir	Environment and Food Agency of Iceland
Norway	Line Småstuen Haug	Norwegian Institute of Public Health
	Claudette R. Bethune	National Institute for Nutrition and Seafood Research
Sweden	Marie Aune	National Food Administration of Sweden

Annex 2: Questionnaires

The enquiry to the Nordic dioxin laboratories for information on the analytical methodology that is being used for the determination of dioxins and dioxin-like PCBs as well as Brominated Flame Retardants resulted in 12 replies including 2 from Denmark, 1 from Finland, 3 from Norway, 5 from Sweden and 1 from the laboratory in Germany that has been used by Iceland and the Faroe Islands. All laboratories have accepted the publication of the questionnaires.

1. Danish Institute for Food and Veterinary Research

Country: Denmark

Dioxin, dioxin-like PCBs

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		Yes	
Sample preparation			
	Type of samples analysed	Food, human milk	
	Sample amount	Corresponding to 3 g of fat if possible	
	Extraction solvent	Pentane/acetone 88/12 v/v	Fat extraction by soxhlet
	Clean-up technique	Multilayer column (silica, sulphuric acid treated silica, sodium sulphate), HPLC clean-up and fractionation on silica and PYE columns	n-pentane is used as eluent for multilayer column. The HPLC eluent is isooctane for PCB and 1:1 isooctane/ethylacetate for PCDD/F
Analysis			
	GC-column type	DB5-MS 60m, 0.25 id, 0.25 um film thickness	
	Injection mode	Splitless	
	Injection volume	2 ul	
	Injection temperature	280	
	MS Detection	SIR	PCDD/F and non-ortho PCB are analysed in one run using 5 SIR-functions. Mono- and di-ortho PCB in another run using 3 SIR-functions
	Ionization mode	EI	
	MS resolution	10.000	
	Calibrations	Multi level (5 point plus zero)	

		Answer	Comments
	Internal standards (injection and calibration standards)	PCDD/F and non-ortho PCB: C-13 labelled internal standards for each congener. C-13 labelled 1,2,3,4-TCDD and 1,2,3,7,8,9-HxCDD as syringe (volume) standards. Mono- and di-ortho PCB: C-13 labelled internal standards for PCB 28, 118,180 and 189. C-13 labelled PCB 105 as syringe (volume) standard.	
	Area/height quantification	Area (from 2 ion fragments for each congener)	
Compounds analysed (specify congeners)		17 PCDD/F (WHO), 12 PCB (WHO) plus PCB 28, 52, 101, 138, 153, 170, 180	
Limit of detection (TEQ, and congener if possible)		TEQ: 0.15 pg TEQ/g fat (PCDD/F) 0.006 pg TEQ/g fat (non-ortho PCB) 0.02 pg TEQ/g fat (mono- and di-ortho PCB) Congener: 0.04 pg/fat (PCDD/F) 0.05 pg/g fat (non-ortho PCB) 0.01 ng/g fat (mono- and di-ortho PCB)	LOD is calculated for each sample based on S/N 3. The listed figures are the typical LOD obtained for 3 g of fat analysed.
Analytical uncertainty (TEQ, and congener if possible)		Expanded uncertainty with a coverage factor of 2: TEQ: 30%	At approximately 1 pg TEQ
Repeatability (TEQ, and congener if possible)		CV: TEQ: <5%, congener: <10%	
In-lab reproducibility (TEQ, and congener if possible)		CV: TEQ: 15%	At approximately 1 pg TEQ
Has the method or part of it been published? If yes, where?		EPA 1613 and 8290	
Additional information can be written here			

Brominated flame retardants

		Answer	Comments
Are the laboratory accredited according to ISO 17025?			
Sample preparation			
	Type of samples analysed		
	Sample amount		
	Extraction solvent		
	Clean-up technique		
Analysis			
	GC-column type		
	Injection mode		
	Injection volume		
	Injection temperature		
	MS Detection		
	Ionization mode		
	MS resolution		
	Calibrations		
	Internal standards (injection and calibration standards)		
	Area/height quantification		
Compounds analysed (specify congeners)			
Limit of detection (TEQ, and congener if possible)			
Analytical uncertainty (TEQ, and congener if possible)			
Repeatability (TEQ, and congener if possible)			
In-lab reproducibility (TEQ, and congener if possible)			
Has the method or part of it been published? If yes, where?			
Additional information can be written here		Analytical methods for PBDE, HBCD and TBBPA are under development (for food and human milk).	

Danish Veterinary and Food Administration in Ringsted

Country: Denmark

Dioxin, dioxin-like PCBs

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		Yes	
Sample preparation		ASE extraction and PowerPrep clean up	
	Type of samples analysed	Food and feed. Not environmental.	
	Sample amount	1-40 g depending on sample type	
	Extraction solvent	Pentane/acetone	
	Clean-up technique	ASE and PowerPrep	
Analysis			
	GC-column type	60 m DB-5MS-DG (10m)	
	Injection mode	Splitless	
	Injection volume	2.5 µl (dioxins and non-ortho-PCB) 1 µl (mono- and diortho-PCB)	
	Injection temperature	260 °C	
	MS Detection	HR-MS	
	Ionization mode	EI	
	MS resolution	Min. 10000	
	Calibrations	Solvent (5 point)	
	Internal standards (injection and calibration standards)	C13-IS for all PCDD/F and non-ortho PCB congeners, except 123789-HxCDD, which are used as syringe standard together with 1234-TCDD. C13-IS for PCB 28, 52, 118 and 180. C13-PCB 105 is used as syringe standard.	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		17 PCDD/Fs (WHO), 4 non-ortho-PCBs (77, 81, 126 and 169) and 15 mono and di-ortho PCB (28, 52, 101, 105, 114, 118, 123, 138, 153, 156, 157, 167, 170, 180 and 189)	
Limit of detection (TEQ, and congener if possible)		PCDD/F: 0.1 pg TEQ/g fat PCB: 0.02 pg TEQ/g fat Congeners: PCDD/F: 0.02-0.06 pg/g fat Non-ortho PCB: 0.05-0.1 pg/g fat Mono and di ortho PCB: 0.003-0.01 ng/g fat	LOD is calculated for each sample. The given numbers are typical LOD for 3 g of fat.
Analytical uncertainty (TEQ, and congener if possible)		+/- 15%, except for OCDF +/- 25 %	
Repeatability (TEQ, and congener if possible)		5-15 %, except OCDF 22 %	
In-lab reproducibility (TEQ, and congener if possible)		15-20 %	
Has the method or part of it been published? If yes, where?		No	
Additional information can be written here			

National Public Health Institute, Chemistry Laboratory

Country: Finland

Dioxin, dioxin-like PCBs

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		Yes	Since year 1996
Sample preparation			
	Type of samples analysed	All kinds of liquid, solid and semisolid food samples	
	Sample amount	Depends on the sample type, for solid samples typically 10 – 100 g. With fat containing samples up to 3 g of fat can be handled after fat extraction.	
	Extraction solvent	Toluene or ethanol-toluene azeotrope (Soxhlet), 35 % acetone in hexane (ASE)	Previously only toluene was used with soxhlet, nowadays ethanol-toluene and even more ASE solvents.
	Clean-up technique	SPE (briefly: sulfuric acid silica for fat removal, activated carbon to separate planar compounds from non-planars, alumina for final clean-up) When ASE is used to extract fat, fat removal is also done with ASE in a similar fashion as in *Journal of Chromatography A 1040 (2004)155*.	
Analysis			
	GC-column type	DB-Dioxin	60 m, 0.25 mm, 0.15 µm
	Injection mode	Splitless	
	Injection volume	2 µl	
	Injection temperature	270 °C	
	MS Detection	Double focusing high resolution instrument	
	Ionization mode	EI+	
	MS resolution	10 000	
	Calibrations	Solvent, single level	HRMS has a very wide linear area allowing one point calibration curves
	Internal standards (injection and calibration standards)	**Calibration standards**: 16 ^{13}C labelled compounds for 17 PCDD/Fs (^{13}C-123789-HeCDD used as injection standard); 8 ^{13}C labelled compounds for 12 DL-PCBs (13C-PCBs 77, 81, 126, 169, 105, 118, 156, 157) **Injection standards:** 1234-TeCDD and 123789 HeCDD for PCDD/Fs; 13C-PCB-60 for non-ortho PCBs; 12C-PCB159 for other DL-PCBs;	
	Area/height quantification	Sum of primary and secondary ion areas	
Compounds analysed (specify congeners)		All 2378-substituted PCDD/Fs (17 congeners), all DL-PCBs plus 25 other PCB congeners.	
Limit of detection (TEQ, and congener if possible)		About 0.25 pg WHO-PCDD/F-TEQ/g fat and 0.03 pg WHO-	

	PCB-TEQ/g fat when 2 g of fat is analysed.	
Analytical uncertainty (TEQ, and congener if possible)	On TEQ-basis MU is for either PCDD/Fs or DL-PCBs when sample amount is 1 g: < 1 pg/g 50 % 1 – 5 pg/g 30 % > 5 pg/g 20 %	
Repeatability (TEQ, and congener if possible)	Not determined	
In-lab reproducibility (TEQ, and congener if possible)	**On TEQ-basis in-lab reproducibility is for** PCDD/Fs: 6 % DL-PCBs: 7 %	
Has the method or part of it been published? If yes, where?	Soxhlet method is an in-house method and has been used in numerous publications and described briefly therein, i.e. Chemosphere 32 (1999)311, Chemosphere 50 (2003)1201.	In-house ASE-method has not been published.
Additional information can be written here		

Brominated flame retardants

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		Yes	Since year 1996
Sample preparation			
	Type of samples analysed	All kinds of liquid, solid and semisolid food samples	
	Sample amount	Depends on the sample type, for solid samples typically 10 – 100g. With fat containing samples up to 3 g of fat can be handled after fat extraction.	
	Extraction solvent	Toluene or ethanol-toluene azeotrope (Soxhlet), 35 % acetone in hexane (ASE)	
	Clean-up technique	SPE (briefly: sulfuric acid silica for fat removal, acticated carbon to separate planar compounds from non-planars, alumina for further clean-up) When ASE is used to extract fat, fat removal is also done with ASE in a similar fashion as in *Journal of Chromatography A 1040 (2004)155*.	
Analysis			
	GC-column type	DB-5MS	-60 m, 0.25 mm, 0.25 µm for bulk PBDEs -6 m, 0.25 mm, 0.25 µm for BDE-209
	Injection mode	Splitless PTV, splitless for BDE 209	
	Injection volume	2 µl	
	Injection temperature	300 °C 130 °C -> 700 °C/min -> 300 °C for BDE 209	
	MS Detection	Double focusing high resolution instrument	
	Ionization mode	EI+	
	MS resolution	10 000	
	Calibrations	Solvent, single level	
	Internal standards (injection and calibration standards)	7 ^{13}C labelled compounds for 15 PBDEs.	
	Area/height quantification	Sum of primary and secondary ion areas	
Compounds analysed (specify congeners)		BDE-28, 71, 75, 47, 66,77, 85, 99, 100, 119, 138, 153, 154, 183, and 209 (209 from separate injection to a shorter column)	
Limit of detection (TEQ, and congener if possible)		0.1 – 0.5 ng/g depending on the congener for sample amount of about 2 g (of fat).	
Analytical uncertainty (TEQ, and congener if possible)		MU for the sum of PBDEs when sample amount is 1 g: < 5 ng/g 70 % 5 – 50 ng/g 50 % > 50 ng/g 20 %	This is a very conservative estimate especially at lower concentrations. It is based on results from few interlaboratory comparisons and in-lab reproducibility, where the sum of PBDEs was about 50 ng/g.
Repeatability (TEQ, and congener if possible)		Not determined	

	Answer	**Comments**
In-lab reproducibility (TEQ, and congener if possible)	In-lab reproducibility is 5 %.	
Has the method or part of it been published? If yes, where?	PBDEs are analysed with GC-MS from the same fraction as mono-ortho and di-ortho-PCBs, i.e. same publications apply here as for PCDD/Fs and DL-PCBs.	In-house ASE-method has not been published.
Additional information can be written here		

4. Norwegian Institute of Public Health

Country: Norway

Dioxin, dioxin-like PCBs

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		Yes/no	Yes: PCDDs/PCDFs and PCB 77, PCB 126, PCB 169(1) No: mono-ortho PCBs (2)
Sample preparation			
	Type of samples analysed	Food (oils, fish, eggs, diary products, meat, animal fat) breast milk, blood	Blood is not accreditated
	Sample amount	1) 10g -150g 2) 0.5g – 20g	
	Extraction solvent	1) <u>Food</u> Cyclohexane:dichloromethane 1:1 <u>Breast milk</u> Metanol: heptane:diethylether 1:1:1 <u>Blood</u> Isopropanol:heptane 2:3 2) <u>Food</u> Cyclohexane:dichloromethane 1:1 <u>Breast milk</u> Metanol: heptane:diethylether 1:1:1 <u>Blood</u> Methanol:dichloromethane 30:70	
	Clean-up technique	Food Column extraction Breast milk Liquid/liquid extraction Blood 1) Column extraction 2) SPE	
Analysis			
	GC-column type	1) RTX-5MS 60m, 0.25mm, 0.1 µm 2) DB-5 MS 60m, 0.25mm, 0.25 µm	
	Injection mode	splitless	
	Injection volume	1µl	
	Injection temperature	1) 280ºC 2) 290 ºC	
	MS Detection	1) HRMS 2) LRMS	
	Ionization mode	1) EI 2) NCI and EI	
	MS resolution	1) 10000 for PCDDs/PCDFs 8000 for non-ortho PCBs 2) 1000	
	Calibrations	Solvent, multi level	

		Answer	Comments
	Internal standards (injection and calibration standards)	1) Internal standards: PCDDs/PCDFs 13C 2,3,7,8 tetra chloro dibenzo-para-dioxin 13C 1,2,3,7,8 penta chloro dibenzo-para-dioxin 13C 1,2,3,4,7,8 hexa chloro dibenzo-para-dioxin 13C 1,2,3,6,7,8 hexa chloro dibenzo-para-dioxin 13C 1,2,3,4,6,7,8 hepta chloro dibenzo-para-dioxin 13C 1,2,3,4,6,7,8,9 octa chloro dibenzo-para-dioxin 13C 2,3,7,8 tetra chloro dibenzofuran 13C 1,2,3,7,8 penta chloro dibenzofuran 13C 2,3,4,7,8 penta chloro dibenzofuran 13C 1,2,3,4,7,8 hexa chloro dibenzofuran 13C 1,2,3,6,7,8 hexa chloro dibenzofuran 13C 1,2,3,7,8,9 hexa chloro dibenzofuran 13C 2,3,4,6,7,8 hexa chloro dibenzofuran 13C 1,2,3,4,6,7,8 hepta chloro dibenzofuran 13C 1,2,3,4,7,8,9 hepta chloro dibenzofuran PCB 77, PCB 126, PCB 169 13C PCB 77 13C PCB 126, 13C PCB 169 Syringe standards PCDDs/PCDFs 13C 1,2,3,4 tetra chloro dibenzo-para-dioxin 13C 1,2,3,7,8,9 hexa chloro dibenzo-para-dioxin PCB 77, PCB 126, PCB 169 PCB 189	
		2) Internal standards: 13C PCB 101 13C PCB 105, 13C PCB 114, 13C PCB 118, 13C PCB 123, 13C PCB 156, 13C PCB 157, 13C PCB 167, 13C PCB 189 Syringe standards PCB 207	
	Area/height quantification	Area	

	Answer	Comments
Compounds analysed (specify congeners)	PCDDs/PCDFs 2,3,7,8 tetra chloro dibenzo-para-dioxin 1,2,3,7,8 penta chloro dibenzo-para-dioxin 1,2,3,4,7,8 hexa chloro dibenzo-para-dioxin 1,2,3,6,7,8 hexa chloro dibenzo-para-dioxin 1,2,3,7,8,9 hexa chloro dibenzo-para-dioxin 1,2,3,4,6,7,8 hepta chloro dibenzo-para-dioxin 1,2,3,4,6,7,8,9 octa chloro dibenzo-para-dioxin 2,3,7,8 tetra chloro dibenzofuran 1,2,3,7,8 penta chloro dibenzofuran 2,3,4,7,8 penta chloro dibenzofuran 1,2,3,4,7,8 hexa chloro dibenzofuran 1,2,3,6,7,8 hexa chloro dibenzofuran 1,2,3,7,8,9 hexa chloro dibenzofuran 2,3,4,6,7,8 hexa chloro dibenzofuran 1,2,3,4,6,7,8 hepta chloro dibenzofuran 1,2,3,4,7,8,9 hepta chloro dibenzofuran 1,2,3,4,6,7,8,9 octa chloro dibenzofuran Dioxinlike PCBs PCB 77,PCB 126,PCB 169 PCB 81, PCB 105, PCB 114, PCB 118, PCB 123, PCB 156, PCB 157, PCB 167, PCB 189	
Limit of detection (TEQ, and congener if possible)	1) Determined for each congener in each sample. Typical 5fg/g lipid to 50fg/g lipid 2) Typical 1pg/g lipid to 10 pg/g lipid	
Analytical uncertainty (TEQ, and congener if possible)	**1) Food and breast milk** PCDDs/PCDFs For samples with sum TEQ (PCDD/F)-upperbound < 0.05pg/g fresh weight: ±50 % for sum TEQ (PCDD/PCDF)- upperbound For samples with sum TEQ (PCDD/F)-upperbound > 0.05pg/g fresh weight: ±20 % for sum TEQ (PCDD/PCDF)- upperbound PCB 77, PCB 126, PCB 169 For samples with sum TEQ (non-ortho PCB)-upperbound < 0.05pg/g fresh weight : ±50 % for sum TEQ (non-ortho PCB)- upperbound For samples with sum TEQ (non-ortho PCB)-upperbound > 0.05pg/g fresh weight : ±20 % for sum TEQ (non-ortho PCB)- upperbound **2)** Estimated to be 20-25% for each congener	
Repeatability (TEQ, and congener if possible)	Se above	
In-lab reproducibility (TEQ, and congener if possible)	Se above	
Has the method or part of it been published? If yes, where?	1) Based on “Smith, L.M., Stalling, D.L., Johnson, J.L.; "Determination of Part-per-Trillion Levels of Polychlorinated Dibenzofurans and Dioxins in Enviromental Samples", Analytical Chemistry, vol. 56, No. 11, september 1984 • 1833” and EPA 1613	
Additional information can be written here		

Brominated flame retardants

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		No	
Sample preparation			
	Type of samples analysed	Food (oils, fish, eggs, diary products, meat, animal fat) breast milk, blood	
	Sample amount	0.5 – 20g	
	Extraction solvent	Food Cyclohexane:dichloromethane 1:1 Breast milk Metanol: heptane:diethylether 1:1:1 Blood Methanol:dichloromethane 30:70	
	Clean-up technique	Food Column extraction Breast milk Liquid-liquid extraction Blood SPE	
Analysis			
	GC-column type	DB-5 MS 30m, 0.25mm, 0.25 µm DB-5 MS 15m, 0.25mm, 0.1 µm	
	Injection mode	splitless	
	Injection volume	2µl	
	Injection temperature	290ºC	
	MS Detection	LRMS	
	Ionization mode	NCI	
	MS resolution	1000	
	Calibrations	Solvent, multi level,	
	Internal standards (injection and calibration standards)	BDE 18 BDE 51 13C BDE 77 BDE 103 BDE 156 BDE 181 13C BDE 209 CtriBBP-A TBCr	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		BDE 28 BDE 37 BDE 47 BDE 85 BDE 99 BDE 100 BDE 119 BDE 138 BDE 153 BDE 154 BDE 183 BDE 209 TBBP-A HBCD PeBP	
Limit of detection (TEQ, and congener if possible)		Typical 1pg/g lipid to 10 pg/g lipid	
Analytical uncertainty (TEQ, and congener if possible)		Estimated to be 20-25% for each congener	
Repeatability (TEQ, and congener if possible)		See above	

	Answer	**Comments**
In-lab reproducibility (TEQ, and congener if possible)	See above	
Has the method or part of it been published? If yes, where?	No	
Additional information can be written here		

5. National Institute for Nutrition and Seafood Research

Country: Norway

Dioxin, dioxin-like PCBs

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		Yes	
Sample preparation			
	Type of samples analysed	Fish, shellfish, feed, oil, canned seafood	
	Sample amount	15-30 g fish, 3-5 g oil, 3-10 g feed	To about 3 g fat is extracted
	Extraction solvent	Hexane (100%) or Hexane:dichloromethane (80:20)	Method is under optimization now
	Clean-up technique	Power-Prep (FMS)	Can load up to 3 g fat for clean up
Analysis			
	GC-column type	RTX-5 Sil MS	25 m x 0.25 mm i.d. x 0.25 um film thickness
	Injection mode	Splitless w/ surge	
	Injection volume	2 uL	
	Injection temperature	230 C	
	MS Detection	SIM	
	Ionization mode	EI	
	MS resolution	10000	
	Calibrations	single point in nonane	
	Internal standards (injection and calibration standards)	EDF-8999, EC-4937 Recovery stds: EDF 5999, EC-4979	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		WHO, 1998: 17 PCDD/Fs, 4 non-ortho-PCB congeners (77, 81, 126 and 169) and 8 mono-ortho PCB congeners (105, 114, 118, 123, 156, 157, 167, and 189). TEQs are calculated with TEF factors by WHO	
Limit of detection (TEQ, and congener if possible)		3 fold signal to noise ratio, varies and is calculated with each run	
Analytical uncertainty (TEQ, and congener if possible)		< 20% CV	
Repeatability (TEQ, and congener if possible)		< 10% CV	
In-lab reproducibility (TEQ, and congener if possible)		Within 2 SD	
Has the method or part of it been published? If yes, where?		U.S. EPA method 1613	
Additional information can be written here			

Brominated flame retardants

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		Yes	
Sample preparation			
	Type of samples analysed	Fish, shellfish, feed, oil, canned seafood	
	Sample amount	5 -10g fatty and 25 g for lean fish, 2.5 g feed and fish meal, 1-2.5 g oil	
	Extraction solvent	Dichloromethane:hexane (80:20) 40 deg C	Under optimization (dcm:hex 20:80) 75-100 deg C
	Clean-up technique	H2SO4 acid wash, 2 times in 8 ml hexane	
Analysis			
	GC-column type	HP-5MS, 30 m x 0.25 mm i.d. x 0.25 um film thickness	
	Injection mode	Splitless w/ surge	
	Injection volume	1 uL	
	Injection temperature	225 deg C	GC oven at 45 deg C for HBCD
	MS Detection	SIM	
	Ionization mode	NCI	
	MS resolution	Br ion m/z 79 and 81	
	Calibrations	Multilevel (5) in nonane	
	Internal standards (injection and calibration standards)	PBDE 139, 119	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		PBDE 28, 47, 66, 99, 100, 119, 138,153, 154, 183, and HBCD a	
Limit of detection (TEQ, and congener if possible)		30 pg/g for PBDEs, 5 ng/g for HBCD a	
Analytical uncertainty (TEQ, and congener if possible)		< 20 % CV	
Repeatability (TEQ, and congener if possible)		< 20 % CV	
In-lab reproducibility (TEQ, and congener if possible)		Within 2 SD	
Has the method or part of it been published? If yes, where?		deBoer J, Allchin C, Law R, Zegers B, Boon JP. Method for the Analysis of Polybrominated Diphenylethers in Sediments and Biota. *TrAC* 2001;20: 591-99.	
Additional information can be written here			

6. Norwegian Institute for Air Research (NILU)

Country: Norway

Dioxin, dioxin-like PCBs

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		Yes	
Sample preparation			
	Type of samples analysed	Food, feed, fats and oils (and others)	
	Sample amount	5-50 g	Typical sample amount
	Extraction solvent	Toluene, cyklohex-ane/dichlormethane	
	Clean-up technique	”3 column system”, in house method	
Analysis			
	GC-column type	RTX 2330	
	Injection mode	Splitless	
	Injection volume	1 ul	
	Injection temperature	265	
	MS Detection	HRMS Autospec	
	Ionization mode	EI	
	MS resolution	R ~10000	
	Calibrations	Solvent, multi level	
	Internal standards (injection and calibration standards)	13C-labelled 2,3,7,8-chlorinate isomers	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		See attached file	
Limit of detection (TEQ, and congener if possible)		See attached file	
Analytical uncertainty (TEQ, and congener if possible)		25-30 %	
Repeatability (TEQ, and congener if possible)		-	
In-lab reproducibility (TEQ, and congener if possible)		-	
Has the method or part of it been published? If yes, where?		The Science of the Total Envi-ronment 162, pp 75-91 Analytical Methods and Instru-mentation 1, pp. 153-163	
Additional information can be written here			

Brominated flame retardants

		Answer	Comments
Are the laboratory accredited according to ISO 17025?			
Sample preparation			
	Type of samples analysed	Food, feed, fats and oils (and others)	
	Sample amount	0,3-20	Typical sample amounts
	Extraction solvent	Cyclohexane/ethylacetate, hexane	
	Clean-up technique	Adsorption chromatograpy and GPC	
Analysis			
	GC-column type	ZB-1 Zebron	
	Injection mode	Splittless	
	Injection volume	1 ul	
	Injection temperature	300	
	MS Detection	HRMS Autospec	
	Ionization mode	EI	
	MS resolution	R ~10000	
	Calibrations	Solvent, single level	
	Internal standards (injection and calibration standards)	13C labelled	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		17 PBDE congeners, α, β and γ-HBCD, BPA, TBBPA	
Limit of detection (TEQ, and congener if possible)		See attached file	
Analytical uncertainty (TEQ, and congener if possible)		25-30% (PBDE, HBCD) Other 40-50%	
Repeatability (TEQ, and congener if possible)		-	
In-lab reproducibility (TEQ, and congener if possible)		-	
Has the method or part of it been published? If yes, where?		No	
Additional information can be written here			

7. Lantmännen Analycen AB (AnalyCen Nordic AB)

Country: Sweden

Dioxin, dioxin-like PCBs

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		Yes	
Sample preparation			
	Type of samples analysed	Meat, fish, oil, egg, milk, food, feed	
	Sample amount	50g; 3-10g extracted	
	Extraction solvent	Toluene	
	Clean-up technique	Silica, Alumina, carbon	
Analysis			
	GC-column type	Restek RTX-dioxin2 60m X 0.25 mm i.d., df 0.25 µm	
	Injection mode	PTV	
	Injection volume	2 µl	
	Injection temperature	300°C	
	MS Detection	High resolution mass spectrometry	
	Ionization mode	EI+	
	MS resolution	>10 000	
	Calibrations	Solvent, multi level	
	Internal standards (injection and calibration standards)	Yes ^{13}C-labelled, congener-specific.	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		2378-TCDF, 12378-PeCDF, 23478-PeCDF, 123478-HxCDF, 123678-HxCDF, 234678-HxCDF, 123789-HxCDF, 1234678-HpCDF, 1234789-HpCDF, OCDF, 2378-TCDD, 12378-PeCDD, 123478-HxCDD, 123678-HxCDD, 123789-HxCDD, 1234678-HpCDD, OCDD, T-PCB-81, T-PCB-77, P-PCB-123, P-PCB-118, P-PCB-114, P-PCB-105, P-PCB126, Hx-PCB-167, Hx-PCB-156, Hx-PCB-157, Hx-PCB-169, Hp-PCB-189, ,	
Limit of detection (TEQ, and congener if possible)		Dioxins: 0.1 ng/kg WHO-TEQ PCB: 0.1 ng/kg WHO-TEQ	
Analytical uncertainty (TEQ, and congener if possible)		10-20% TEQ-WHO	
Repeatability (TEQ, and congener if possible)		3%	
In-lab reproducibility (TEQ, and congener if possible)		10%	
Has the method or part of it been published? If yes, where?		EPA 1613, EPA 1668	
Additional information can be written here			

Brominated flame retardants

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		No	
Sample preparation			
	Type of samples analysed	Fish, oil	
	Sample amount	10g	
	Extraction solvent	hexane	
	Clean-up technique	Silica, Alumina	
Analysis			
	GC-column type	DB-5 15m X 0.18 mm i.d. df 0.18 µm	
	Injection mode	PTV	
	Injection volume	1 µl	
	Injection temperature	70->300°C	
	MS Detection	Single quadropole	
	Ionization mode	NCI	
	MS resolution	unit	
	Calibrations	Solvent, multi level,	
	Internal standards (injection and calibration standards)	BDE77, BDE181 ^{13}C-PBDE 209	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		BDE 17, BDE 28, BDE 47, BDE 66, BDE 71, BDE 85, BDE 99, BDE 100, BDE 138, BDE 153, BDE 154, BDE 183, BDE 190, BDE 203 och BDE 209	
Limit of detection (TEQ, and congener if possible)		1 µg/kg/congener	
Analytical uncertainty (TEQ, and congener if possible)		20-30%	
Repeatability (TEQ, and congener if possible)			
In-lab reproducibility (TEQ, and congener if possible)			
Has the method or part of it been published? If yes, where?		No	
Additional information can be written here			

8. National Food Administration

Country: Sweden

Dioxin, dioxin-like PCBs

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		No	The laboratory is accredited for other methods but not yet for dioxin-like PCBs
Sample preparation			
	Type of samples analysed	Fish, meat, eggs, milk, fish oil, veg. oil, human milk, blood serum	
	Sample amount	5-50 g	
	Extraction solvent	n-hexane, acetone, diethyl ether	
	Clean-up technique	Silica column, HPLC with carbon column	
Analysis			
	GC-column type	Ultra-2 (50mx0,2mmx0,33µm)	
	Injection mode	PTV	
	Injection volume	3 µl	
	Injection temperature	105 °C, 700 °C/min to 320 °C (1 min)	
	MS Detection	Quadrupole	
	Ionization mode	ECNI	
	MS resolution	Unit	
	Calibrations	Solvent n-hexane, multi level	
	Internal standards (injection and calibration standards)	^{13}C-labelled standards for each congener	
	Area/height quantification	Height	
Compounds analysed (specify congeners)		PCB#77,81,105,114,118,123,126,156,157,167,169,189	We do not analyse dioxins
Limit of detection (TEQ, and congener if possible)		Depends on sample size 0,6-10 pg/g f.w. on congener basis (not TEQ)	
Analytical uncertainty (TEQ, and congener if possible)		Expanded measurement uncertainty 25-35 % on congener basis.	
Repeatability (TEQ, and congener if possible)		6 % on congener basis	
In-lab reproducibility (TEQ, and congener if possible)		9 % on congener basis	
Has the method or part of it been published? If yes, where?		No	
Additional information can be written here			

Brominated flame retardants

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		No	The laboratory is accredited for other methods but yet not for dioxin-like PCBs
Sample preparation			
	Type of samples analysed	Mainly fish and human milk	
	Sample amount	10-35 g	
	Extraction solvent	n-hexane, acetone, diethyl ether	
	Clean-up technique	Silica gel column	
Analysis			
	GC-column type	DB-17MS (30mx0,25mmX0,15µm)	
	Injection mode	PTV	
	Injection volume	3 µl	
	Injection temperature	95 °C, 700 °C/min to 320 °C (2 min)	
	MS Detection	Quadrupole	
	Ionization mode	ECNI	
	MS resolution	Unit	
	Calibrations	Solvent n-hexane, multi level	
	Internal standards (injection and calibration standards)	BDE-85	
	Area/height quantification	Height	
Compounds analysed (specify congeners)		BDE28,47,66,99,100,138,153,154,183	
Limit of detection (TEQ, and congener if possible)		3-10 pg/g f.w. on congener basis	
Analytical uncertainty (TEQ, and congener if possible)			
Repeatability (TEQ, and congener if possible)			
In-lab reproducibility (TEQ, and congener if possible)		10-25 % on congener basis (also depending on level in sample)	
Has the method or part of it been published? If yes, where?		No	
Additional information can be written here			

9. MTM, Örebro University

Country: Sweden

Dioxin, dioxin-like PCBs

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		No	Working according to ISO 17025
Sample preparation			
	Type of samples analysed	Biological /Food/Feed	
	Sample amount	1-100 g	
	Extraction solvent	MeCl/hexane	
	Clean-up technique	Open column chromatography	Multilayer Silica, AlOx, Carbon
Analysis			
	GC-column type	DB-5MS (30m x 0.25 x 0.25)	Use confirmation column when needed (Sp2330, RT-dioxin 2)
	Injection mode	Splitless	
	Injection volume	(1-2 ul)	
	Injection temperature	250	
	MS Detection	High Resolution	
	Ionization mode	EI	
	MS resolution	> 10 000	
	Calibrations	5 point calibration curve using ^{13}C labelled IS and RS	
	Internal standards (injection and calibration standards)	5 point calibration curve using ^{13}C labelled IS and RS	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		All 17 2,3,7,8 substituted congeners and the 3 planar PCBs (#77, #126, #169)	
Limit of detection (TEQ, and congener if possible)		Depends on sample seize 0.003-0.011 pg/g / congener	
Analytical uncertainty (TEQ, and congener if possible)		25 %	Data from participating in inter-national interlaboratory studies
Repeatability (TEQ, and congener if possible)		15 %	
In-lab reproducibility (TEQ, and congener if possible)		25 %	Internal QA/QC samples within 25%, otherwise flagged
Has the method or part of it been published? If yes, where?		Based on EPA method 1613 and EU method EN 1948	
Additional information can be written here			

Brominated flame retardants

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		No	Working according to ISO 17025
Sample preparation			
	Type of samples analysed	Biological /Food/Feed	
	Sample amount	1-100 g	
	Extraction solvent	MeCl/hexane	
	Clean-up technique	Open column chromatography	Multilayer silica, GPC if needed
Analysis			
	GC-column type	DB-5MS (30m x 0.25 x 0.25)	
	Injection mode	Splitless	
	Injection volume	(1-2 ul)	
	Injection temperature	250	
	MS Detection	Quadrupole	
	Ionization mode	NCI	
	MS resolution	Mass resolution 500	
	Calibrations	5 point calibration curve using ^{13}C labelled IS and RS	
	Internal standards (injection and calibration standards)	5 point calibration curve using ^{13}C labelled IS and RS	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		All Tri- through Octa-BDEs. BTBPE, Deca-ethane	Deca-BDE #209 is run on a shorter 15 m DB-5 like column
Limit of detection (TEQ, and congener if possible)		Depends on sample seize 1-100 pg/g / congener	
Analytical uncertainty (TEQ, and congener if possible)		25%	Data from participating in inter-national interlaboratory studies
Repeatability (TEQ, and congener if possible)		15%	
In-lab reproducibility (TEQ, and congener if possible)		25%	Internal QA/QC samples within 25%, otherwise flagged
Has the method or part of it been published? If yes, where?		Arch. Environ. Contam. Toxi-col. 36, 355-363 (1999)	
Additional information can be written here		No TEQs for BDEs exist !!	

10. MTM, Örebro University

Country: Sweden

Dioxin, dioxin-like PCBs

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		No	Working according to ISO 17025
Sample preparation			
	Type of samples analysed	Biological /Food/Feed	
	Sample amount	1-5 g sample (1g fat)	
	Extraction solvent	SFE-LC	Combined extraction and clean up
	Clean-up technique	SFE-LC	using supercritical CO_2, and a special carbon (LC) column for clean up
Analysis			
	GC-column type	DB-5MS (30m x 0.25 x 0.25)	Use confirmation column when needed (Sp2330, RT-dioxin 2)
	Injection mode	Splitless	
	Injection volume	(1-2 ul)	
	Injection temperature	250	
	MS Detection	High Resolution	
	Ionization mode	EI	
	MS resolution	> 10 000	
	Calibrations	5 point calibration curve using ^{13}C labelled IS and RS	
	Internal standards (injection and calibration standards)	5 point calibration curve using ^{13}C labelled IS and RS	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		All 17 2,3,7,8 substituted congeners and the 3 planar PCBs (#77, #126, #169)	
Limit of detection (TEQ, and congener if possible)		Depends on sample seize 0.03-0.11 pg/g / congener	
Analytical uncertainty (TEQ, and congener if possible)		25%	Data from participating in international interlaboratory studies
Repeatability (TEQ, and congener if possible)		15%	
In-lab reproducibility (TEQ, and congener if possible)		25%	Internal QA/QC samples within 25%, otherwise flagged
Has the method or part of it been published? If yes, where?		Anal. Chem. 68 (1996) 1279-1283	

Brominated flame retardants

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		No	Working according to ISO 17025
Sample preparation			
	Type of samples analysed	Biological /Food/Feed	
	Sample amount	1-5 g sample (1g fat)	
	Extraction solvent	SFE-LC	Combined extraction and clean up
	Clean-up technique	SFE-LC	using supercritical CO_2, and a special carbon (LC) column for clean up
Analysis			
	GC-column type	DB-5MS (30m x 0.25 x 0.25)	
	Injection mode	Splitless	
	Injection volume	(1-2 ul)	
	Injection temperature	250	
	MS Detection	Quadrupole	
	Ionization mode	NCI	
	MS resolution	Mass resolution 500	
	Calibrations	5 point calibration curve using ^{13}C labelled IS and RS	
	Internal standards (injection and calibration standards)	5 point calibration curve using ^{13}C labelled IS and RS	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		All Tri- through Octa-BDEs. BTBPE, Deca-ethane	Deca-BDE #209 is run on a shorter 15 m DB-5 like column
Limit of detection (TEQ, and congener if possible)		Depends on sample size 1-100 pg/g / congener	
Analytical uncertainty (TEQ, and congener if possible)		25%	Data from participating in inter-national interlaboratory studies
Repeatability (TEQ, and congener if possible)		15%	
In-lab reproducibility (TEQ, and congener if possible)		25%	Internal QA/QC samples within 25%, otherwise flagged
Has the method or part of it been published? If yes, where?		Anal. Chem. 68 (1996) 1279-1283	
Additional information can be written here		No TEQs for BDEs exist	

11. Umeå University/Environmental Chemistry

Country: Sweden

Dioxin, dioxin-like PCBs

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		Yes	
Sample preparation			
	Type of samples analysed	Fish,fat,egg,milk,meat,vegetables,cereals	
	Sample amount	1-100g	
	Extraction solvent	Hexane,acetone,diethyl ether	
	Clean-up technique	SPE, carbon column	
Analysis			
	GC-column type	DB-5, DB-5MS	
	Injection mode	Splitless	
	Injection volume	3ul	
	Injection temperature	280˚C	
	MS Detection	SIR	
	Ionization mode	EI	
	MS resolution	7000-10000	
	Calibrations	Single level	
	Internal standards (injection and calibration standards)	^{13}C-isomers for all analytes. Quantitation standards for all analytes, except PCB#81.	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		All 2378-substituted PCDD/F. PCB#77,81,105,114,118,123,126,156,157,167,169,189	
Limit of detection (TEQ, and congener if possible)		0,2-5pg/g lipid 0,01-0,5 pg/g fish fresh weight	
Analytical uncertainty (TEQ, and congener if possible)		+/- 26% (95% confidence level)	
Repeatability (TEQ, and congener if possible)		+/- 10% TEQ	
In-lab reproducibility (TEQ, and congener if possible)			
Has the method or part of it been published? If yes, where?		Journal of Chromatography A, 1086(2005)61-70	
Additional information can be written here			

Brominated flame retardants

		Answer	Comments
Are the laboratory accredited according to ISO 17025?			
Sample preparation			
	Type of samples analysed		
	Sample amount		
	Extraction solvent		
	Clean-up technique	GPC, SPE, HPLC,…	
Analysis			
	GC-column type		
	Injection mode		
	Injection volume		
	Injection temperature		
	MS Detection		
	Ionization mode		
	MS resolution		
	Calibrations	Solvent, matrix matched, single or multi level, …	
	Internal standards (injection and calibration standards)		
	Area/height quantification		
Compounds analysed (specify congeners)			
Limit of detection (TEQ, and congener if possible)			
Analytical uncertainty (TEQ, and congener if possible)			
Repeatability (TEQ, and congener if possible)			
In-lab reproducibility (TEQ, and congener if possible)			
Has the method or part of it been published? If yes, where?			
Additional information can be written here			

12. Eurofins – ERGO Forschungsgesellschaft mbH

Country: Germany

Dioxin, dioxin-like PCBs

	Answer	Comments
Are the laboratory accredited according to ISO 17025?	Yes	
Sample preparation		
Type of samples analysed	Fish and fish products, meat and meat products, milk and milk products, eggs, diary products, vegetables	
Sample amount	According to fat content (approx. 1,5 g fat)	
Extraction solvent	Depending on sample matrix: - Meat/ fish: Cyclohexane and dichloromethane - Milk: diethyl ether and pentane - Eggs: acetone and pentane - Oils and fats: hexane	
Clean-up technique	Multicolumn system SPE	
Analysis		
GC-column type	DB 5 capillary column, 60m	
Injection mode	Split/Splitless	
Injection volume	2 µl	
Injection temperature	270°C	
MS Detection	Double focussing magnetic sector	
Ionization mode	Electron impact (EI)	
MS resolution	High	
Calibrations	Solvent, 5 level	
Internal standards (injection and calibration standards)	All compounds plus 1.2.3.4-$^{13}C_{12}$-TCDD, 1.2.3.4.6.7.8-$^{13}C_6$-HpCDF and 1.2.3.4-$^{13}C_6$-TCDD as Injection-STD	
Area/height quantification	Area	
Compounds analysed (specify congeners)	2.3.7.8-Tetra-CDD 1.2.3.7.8-Penta-CDD 1.2.3.4.7.8-Hexa-CDD 1.2.3.6.7.8-Hexa-CDD 1.2.3.7.8.9-Hexa-CDD 1.2.3.4.6.7.8-Hepta-CDD OCDD 2.3.7.8-Tetra-CDF 1.2.3.7.8-Penta-CDF 2.3.4.7.8-Penta-CDF 1.2.3.4.7.8-Hexa-CDF 1.2.3.6.7.8-Hexa-CDF 1.2.3.7.8.9-Hexa-CDF 2.3.4.6.7.8-Hexa-CDF 1.2.3.4.6.7.8-Hepta-CDF 1.2.3.4.7.8.9-Hepta-CDF OCDF 3,3',4,4'-Tetra-CB 77 3,4,4',5-Tetra-CB 81 3,3',4,4',5-Penta-CB 126 3,3',4,4',5,5'-Hexa-CB 169	All

	Answer	**Comments**
	2,3,3',4,4'-Penta-CB 105 2,3,4,4',5-Penta-CB 114 2,3',4,4',5-Penta-CB 118 2',3,4,4',5-Penta-CB 123 2,3,3',4,4',5,-Hexa-CB 156 2,3,3',4,4',5'-Hexa-CB 157 2,3',4,4',5,5'-Hexa-CB 167 2,3,3',4,4',5,5'-Hepta-CB 189	
Limit of detection (TEQ, and congener if possible)	Congener 2,3,7,8 – TCDD 1,2,3,7,8 – PeCDD 1,2,3,4,7,8 – HxCDD 1,2,3,6,7,8 – HxCDD 1,2,3,7,8,9 – HxCDD 1,2,3,4,6,7,8 – HpCDD OCDD 2,3,7,8 – TCDF 1,2,3,7,8 – PeCDF 2,3,4,7,8 – PeCDF 1,2,3,4,7,8 – HxCDF 1,2,3,6,7,8 – HxCDF 1,2,3,7,8,9 – HxCDF 2,3,4,6,7,8 – HxCDF 1,2,3,4,6,7,8 – HpCDF 1,2,3,4,7,8,9 – HpCDF OCDF 3,3',4,4' – TeCB #77 3,4,4',5 – TeCB #81 3,3',4,4',5 – PeCB #126 3,3',4,4',5,5' – HxCB #169 2,3,3',4,4'- PeCB #105 2,3,4,4',5 – PeCB #114 2,3',4,4',5 – PeCB #118 2',3,4,4',5 – PeCB #123 2,3,3',4,4',5 – HxCB #156 2,3,3',4,4',5' – HxCB #157 2,3',4,4',5,5' – HxCB #167 2,3,3',4,4',5,5' – HpCB #189 2,4,4'-TriCB #28 2,2',5,5'-TeCB #52 2,2',4,5,5'-PeCB #101 2,2',3,4,4',5'- HxCB #138 2,2',4,4',5,5'- HxCB #153 2,2',3,4,4',5,5'- HpCB #180	Limit of detection (mean) 20-50 pg/kg 25-50 pg/kg 30-60 pg/kg 30-60 pg/kg 30-60 pg/kg 40-100 pg/kg 50-100 pg/kg 20-50 pg/kg 25-50 pg/kg 25-50 pg/kg 30-60 pg/kg 30-60 pg/kg 30-60 pg/kg 30-60 pg/kg 40-100 pg/kg 40-100 pg/kg 50-100 pg/kg 15-30 ng/kg 5-10 ng/kg 4-10 ng/kg 0,2-0,5 ng/kg 100-200 ng/kg 30-60 ng/kg 0,4-1,0 µg/kg 50-100 ng/kg 40-100 ng/kg 30-60 ng/kg 35-70 ng/kg 40-100 ng/kg 0,6-1,5 µg/kg 3-6 µg/kg 1-3 µg/kg 0,6-1,2 µg/kg 0,6-1,2 µg/kg 0,3-0,6 µg/kg

Analytical uncertainty (TEQ, and congener if possible)	Precision in series		
	Oil samples		
		Mean (lipid based)	Relative standard deviation (%)
	Sum 2.3.7.8-PCDD/PCDF (WHO TEQ), n = 4	7,8 ng/kg	2,4
	2.3.7.8–Tetra–CDD, n = 4	0,93 ng/kg	2,9
	1.2.3.6.7.8–Hexa–CDD, n = 4	0,13 ng/kg	8,4
	2.3.4.7.8–Penta-CDF, n = 4	6,2 ng/kg	1,9
	3.3'.4.4'.5–PeCB #126, n = 5	130 ng/kg	1,3
	2.3'.4.4'.5–PeCB #118, n = 5	18000 ng/kg	7,6
	2.2'.4.4'.5.5'-HxCB #153, n = 5	39 µg/kg	2,3
	Precision in series		
	Milk samples		
		Mean (lipid based)	Relative standard deviation (%)
	Sum 2.3.7.8-PCDD/PCDF (WHO TEQ), n = 5	2,6 ng/kg	2,6
	2.3.7.8–Tetra–CDD, n = 5	0,24 ng/kg	14,3
	1.2.3.6.7.8–Hexa–CDD, n = 5	0,85 ng/kg	5,4
	2.3.4.7.8–Penta-CDF, n = 5	2,3 ng/kg	2,1
	3.3'.4.4'.5–PeCB #126, n = 5	28 ng/kg	1,2
	2.3'.4.4'.5–PeCB #118, n = 5	2400 ng/kg	8,7
	2.2'.4.4'.5.5'-HxCB #153, n = 5	4,5 µg/kg	6,3
Repeatability (TEQ, and congener if possible)	Repeatability		
	Oil samples		
		Mean (lipid based)	Repeatability (%)
	Sum 2.3.7.8-PCDD/PCDF (WHO TEQ), n = 4	7,8 ng/kg	11
	2.3.7.8–Tetra–CDD, n = 4	0,93 ng/kg	13
	1.2.3.6.7.8–Hexa–CDD, n = 4	0,13 ng/kg	37
	2.3.4.7.8–Penta-CDF, n = 4	6,2 ng/kg	8,4
	3.3'.4.4'.5–PeCB #126, n = 5	130 ng/kg	4,8
	2.3'.4.4'.5–PeCB #118, n = 5	18000 ng/kg	31
	2.2'.4.4'.5.5'-HxCB #153, n = 5	39 µg/kg	9,3
	Reproducibility		
	Milk fat samples		
		Mean (lipid based)	Repeatability (%)
	Sum 2.3.7.8-PCDD/PCDF (WHO TEQ), n = 5	0,27 ng/kg	10
	2.3.7.8–Tetra–CDD, n = 5	0,14 ng/kg	56
	1.2.3.6.7.8–Hexa–CDD, n = 5	0,18 ng/kg	21
	2.3.4.7.8–Penta-CDF, n = 5	0,19 ng/kg	8,3
	3.3'.4.4'.5–PeCB #126, n = 5	1,3 ng/kg	4,6
	2.3'.4.4'.5–PeCB #118, n = 5	800 ng/kg	34
	2.2'.4.4'.5.5'-HxCB #153, n = 5	1,1 µg/kg	25
In-lab reproducibility (TEQ, and	Precision from day to day		

	Oil samples		
		Mean (lipid based)	Standard deviation (%)
	Sum 2.3.7.8-PCDD/PCDF (WHO TEQ), n = 211	8,0 ng/kg	6,9
	2.3.7.8–Tetra–CDD, n = 210	0,98 ng/kg	11
	1.2.3.6.7.8–Hexa–CDD, n = 210	1,2 ng/kg	11
	2.3.4.7.8–Penta–CDF, n = 211	6,2 ng/kg	8,3
	3.3'.4.4'.5–PeCB #126, n = 159	120 ng/kg	9,4
	2.3'.4.4'.5–PeCB #118, n = 165	18000 ng/kg	10
	2.2'.4.4'.5.5'-HxCB #153, n = 97	41 µg/kg	7,8
	Precision from day to day		
	Milk samples		
		Mean (lipid based)	Standard deviation (%)
	Sum 2.3.7.8-PCDD/PCDF (WHO TEQ), n = 28	2,4 ng/kg	8,1
	2.3.7.8–Tetra–CDD, n = 28	0,29 ng/kg	13
	1.2.3.6.7.8–Hexa–CDD, n = 28	0,85 ng/kg	8,1
	2.3.4.7.8–Penta–CDF, n = 28	2,0 ng/kg	8,6
	3.3'.4.4'.5–PeCB #126, n = 8	31 ng/kg	5,3
	2.3'.4.4'.5–PeCB #118, n = 9	1900 ng/kg	14
	2.2'.4.4'.5.5'-HxCB #153, n = 5	4,2 µg/kg	5,7
Has the method or part of it been published? If yes, where?	Parts of the method have been published in the scientific literature.		

Brominated flame retardants

		Answer	Comments
Are the laboratory accredited according to ISO 17025?		Yes	
Sample preparation			
	Type of samples analysed	Fish and fish products	
	Sample amount	According to fat content	
	Extraction solvent	Cyclohexane and dichloro-methane	
	Clean-up technique	Multicolumn system SPE	
Analysis			
	GC-column type	DB 1 capillary column, 10m	
	Injection mode	Split/Splitless	
	Injection volume	2 µl	
	Injection temperature	290°C	
	MS Detection	Double focussing magnetic sector	
	Ionization mode	Electron impact (EI)	
	MS resolution	High	
	Calibrations	Solvent, 5 level	
	Internal standards (injec-tion and calibration stan-dards)	BDE no.: 28, 47, 99, 100, 153, 154, 183, 209 and 139 as Injection-STD	
	Area/height quantification	Area	
Compounds analysed (specify congeners)		BDE no: 17, 28, 47, 66, 85, 99, 100, 138, 153, 154, 183, 209	
Limit of detection (TEQ, and congener if possible)		Congener	Limit of detection (mean)
		BDE-17 BDE-28 BDE-47 BDE-66 BDE-77 BDE-85 BDE-99 BDE-100 BDE-138 BDE-153 BDE-154 BDE-183 BDE-209	0,001-0,003 ng/g 0,003-0,006 ng/g 0,02-0,04ng/g 0,001-0,003 ng/g 0,001-0,003 ng/g 0,001-0,003 ng/g 0,001-0,003 ng/g 0,002-0,004 ng/g 0,001-0,003 ng/g 0,003-0,006 ng/g 0,007-0,01 ng/g 0,007-0,01 ng/g 0,04-0,08 ng/g
Analytical uncertainty (TEQ, and congener if possible)		Precision in series	
		Human milk samples	
			Mean (lipid based) / Relative standard deviation (%)
		BDE-47	0,73 ng/g / 1,2
		BDE-99	0,27 ng/g / 2,0
		BDE-153	0,38 ng/g / 1,5
		Precision in series	
		Oil samples	
			Mean (lipid based) / Relative standard deviation (%)
		BDE-47	19 ng/g / 2,9
		BDE-99	4,0 ng/g / 2,6
		BDE-153	0,34 ng/g / 2,5
Repeatability (TEQ, and congener if possible)		Repeatability	
		Human milk samples	
			Mean (lipid based) / Repeatability (%)
		BDE-47	0,73 ng/g / 4,9
		BDE-99	0,27 ng/g / 7,9

	BDE-153	0,38 ng/g	6,1
	Repeatability		
	Oil samples		
		Mean (lipid based)	Repeatability (%)
	BDE-47	19 ng/g	13
	BDE-99	4,0 ng/g	12
	BDE-153	0,34 ng/g	11
In-lab reproducibility (TEQ, and congener if possible)	Precision from day to day		
	Human milk samples		
		Mean (lipid based)	Standard deviation (%)
	BDE-47	0,76 ng/g	10
	BDE-99	0,31 ng/g	15
	BDE-153	0,40 ng/g	7,3
	Precision from day to day		
	Oil samples		
		Mean (lipid based)	Standard deviation (%)
	BDE-47	18 ng/g	9,2
	BDE-99	3,8 ng/g	9,9
	BDE-153	0,33 ng/g	8,9
Has the method or part of it been published? If yes, where?	Parts of the method have been published in the scientific literature.		