

Environment and Health in Europe 37

**LEVELS OF PCBs, PCDDs AND PCDFs  
IN HUMAN MILK AND BLOOD**

**Second Round of Quality Control Studies**

Edited by

*Erkki J. Yrjänheikki*



Published on behalf of the World Health Organization  
Regional Office for Europe  
by FADL 1991

The views expressed in this publication are those of the author(s) and do not necessarily represent the decisions or the stated policy of the World Health Organization.

WHO Library Cataloguing in Publication Data

Levels of PCBs, PCDDs and PCDFs in human milk and blood: second round of quality control studies/edited by Erkki J. Yrjänheikki

(Environment and Health in Europe 37)

1. Milk, human - analysis 2. Polychlorobiphenyl compounds - analysis 3. Benzofurans - analysis 4. Dioxins - analysis 5. Blood chemical analysis 6. Quality control

I. Yrjänheikki, Erkki J. II. World Health Organization. Regional Office for Europe III. Series

(NLM Classification: QV 633)

ISBN 87-7749-063-0

ISSN 0904-8790

Series Editor:

Elaine C. Grandjean

Cover design by Patricia Christensen

Printed in Denmark by Villadsen & Christensen

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The Environment and Health in Europe Series is a continuation of the Environmental Health Series.

Available from your usual bookseller or from

FADL Publishers

Prinsesse Charlottesgade 29

DK-2200 Copenhagen N

Denmark

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## FOREWORD

Although the current levels of the common chemical contaminants in food in Europe generally fall well within established WHO guidelines for exposure limits, specific population groups may nonetheless be at risk. One such situation is that of infants exposed to contaminants in human milk.

Several years ago, a group of toxic chemicals, polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins (PCDDs), and polychlorinated dibenzofurans (PCDFs), was identified in human milk samples. This finding caused considerable public debate and concern, and raised doubts about the safety of breast-feeding.

In response to this debate, and in keeping with the WHO goal of health for all (HFA), the WHO Regional Office for Europe developed and is coordinating a project, in close collaboration with other international bodies as well as numerous national institutions, to provide guidance on prevention and control of emissions of these chemicals into the environment and to minimize human exposure from different sources. Special attention has been focused on infant exposure through breast milk and related adverse health effects on the developing child.

In 1987, a Working Group, organized jointly by the Regional Office and the Regional Government of Venice in Abano Terme, made a comprehensive evaluation of possible health risks in infants based on the available research data on exposure levels, health risks, and toxicity of these chemicals. The Working Group concluded that despite the levels found in human milk, breast-feeding should be continued and promoted. However, it stressed that additional information was needed to improve the interim risk assessment and recommended that epidemiological studies be developed to verify any possible health effects in infants exposed to the present levels of these chemicals in breast milk. A consultation, held in Bayreuth, Germany, in September 1990, agreed on the development of a protocol and identified possible populations for epidemiological studies.

The Abano Terme Working Group also recommended that more information should be obtained on exposure levels in breast milk to indicate possible trends in levels and to improve risk assessment. It recommended that comparability and reliability of the published research

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data on exposure levels be improved. The Regional Office has therefore been coordinating both analytical field studies and interlaboratory quality control studies on levels of the above chemicals in human milk. Several expert meetings were organized to design the protocols for these studies and to coordinate their implementation. The first round of the analytical field studies was completed at the end of 1987, and the results were discussed at a consultation held in Copenhagen in February 1988. The results of the first round of quality control studies were reviewed by a consultation in Umea, Sweden, in August 1987.

The reports of these two consultations were published in 1989 as No. 34 in the Environmental Health Series of the Regional Office. The Umea consultation recommended that quality control studies should be repeated every second year and that human blood should be included in addition to human milk. Based on this recommendation, the Regional Office has coordinated the second round of these studies. The results of analyses received from the participating laboratories were subjected to a comprehensive statistical treatment, and these data were reviewed by a consultation held in Rovaniemi, Finland, in June 1990.

This publication contains the report on this last consultation as well as the lists of laboratories that qualified, through these studies, to perform such analyses. It also includes detailed information on the statistical treatment of the analytical results from laboratories.

These results are a basic step towards reducing the sources of potential health risks to breast-feeding infants and as such help to carry out the overall Health for all strategy of the Regional Office, part of which concerns food safety.

The financial support provided by the Government of Finland to organize this consultation is gratefully acknowledged. Special thanks are extended to Dr Martin Nygren and his laboratory at the Swedish Defence Research Establishment, Umea, for his valuable work in the practical coordination of the studies, to Dr Erkki Yrjänheikki for his overall coordinating role, and to Ms Patricia Christensen for her administrative assistance.

S.Tarkowski  
Director, Environment and Health  
WHO Regional Office for Europe

## Introduction

The WHO Regional Office for Europe (WHO/EURO) is coordinating inter-laboratory quality control studies on levels of PCBs, PCDDs, and PCDFs in human milk and blood within its overall project on the adverse health effects of these chemicals. The aim is to ensure that reliable and comparable data on these compounds can be obtained. The results of the first round of studies, on human milk only, were evaluated by a consultation held in Umea, Sweden, in August 1987. That meeting recommended that the studies be continued and that a new round be organized every second year from 1988 onwards. Based on that recommendation, WHO/EURO planned the second round. A consultation held in Copenhagen in February 1988 designed the study protocol, which included analysis of both blood and milk. That meeting also agreed on the practical implementation of the studies, including coordination, laboratory work, reporting, and the timetable, the details of which were subsequently discussed at two informal meetings of laboratory representatives in Umea in August 1988 and Toronto in September 1989.

The Consultation on the Second Round of Quality Control Studies on Levels of PCBs, PCDDs and PCDFs in Human Milk and Blood, held in Rovaniemi, Finland, 5-6 June 1990, was organized with the financial assistance of the Government of Finland. Its main aim was to evaluate the results, received from the participating laboratories, based on a statistical analysis carried out by Ms Annette Ersboell on behalf of WHO/EURO. The participants were then asked to establish criteria to determine which of the laboratories could be considered as qualified to perform these analyses. In addition, the participants were requested to evaluate analytical procedures in order to identify any weak points and to advise on improvements, and to consider the necessity for further activities to ensure the continued reliability and comparability of results. The main output of the meeting was to be the identification of those laboratories considered qualified to perform these analyses. The meeting was attended by 22 experts from 15 countries, one observer, and two representatives of WHO/EURO (see Annex 6).

Dr J.S. Carlé chaired the meeting and Dr J.R. Startin agreed to act as Rapporteur.

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To avoid any misunderstandings it should be pointed out that not all laboratories represented at the consultation participated in the studies or submitted analytical results.

### Study design

The design of the second round of the studies was agreed at the consultation held in Copenhagen in February 1988 and was intended to facilitate the use of statistical methods of analysis, as recommended by Dr L. Pallesen after the first round. The studies have been coordinated by a team consisting of Professor B. Jansson (Swedish Environmental Protection Agency), Dr M. Nygren (Swedish Defence Research Establishment), Mr R. Vaz (Swedish National Food Administration), and Dr E.J. Yrjänheikki (WHO/EURO). The laboratory work of preparing samples for analysis has been carried out by Dr Nygren with the assistance of Dr Yrjänheikki.

The study was designed to take into account both the short-term and the long-term variation; in other words the repeatability and the reproducibility. For each of the two matrices, human milk and blood, a single homogeneous pool of material was made and divided into three subpools. Prior to this division a quantity of a  $^{14}\text{C}_{12}$ -labelled organic compound was added to enable the homogeneity to be checked. The radioactivity was measured in three different samples, and the standard deviation was within 10%. For each matrix, two of the subpools were fortified by the addition of certain of the PCDD and PCDF congeners having a substitution pattern including the 2-, 3-, 7-, and 8- positions. The third subpool remained unfortified. The reason for the fortification was to obtain more reliable results from the study. The exact fortification scheme had been agreed within the coordinating team in accordance with the requirements for a full statistical analysis, and was not known to the participating laboratories prior to this consultation. Details of the fortification levels are given in Table 1. Initially, samples from two of the subpools of each matrix were sent to participants. Samples of the third subpools were distributed after results for the first two had been submitted to WHO/EURO. Each participant was required to make three separate analyses of each subpool. All participating laboratories were to

use common  $^{12}\text{C}_{12}$  and  $^{13}\text{C}_{12}$  standards supplied by the coordinating laboratory. All samples were shipped in a frozen state.

For PCBs, fortification was not carried out and participants were required to complete the analysis in triplicate.

Table 1. Congeners used for fortification and their levels (in pg/mg)

Congener	Blood	
	Pool A	Pool C
2,3,7,8-tetraCDD	-	0.066
1,2,3,7,8-pentaCDD	0.195	-
1,2,3,4,7,8-hexaCDD	-	0.046
1,2,3,6,7,8-hexaCDD	-	0.337
1,2,3,4,6,7,8-heptaCDD	1.184	0.720
octaCDD	-	8.357
1,2,3,6,7,8-hexaCDF	-	0.035
2,3,4,6,7,8-hexaCDF	0.028	-
1,2,3,4,6,7,8-heptaCDF	0.157	-
Congener	Milk	
	Pool A	Pool C
2,3,7,8-tetraCDD	-	0.202
1,2,3,7,8-pentaCDD	-	0.359
1,2,3,4,7,8-hexaCDD	0.088	-
1,2,3,6,7,8-hexaCDD	0.535	0.664
1,2,3,4,6,7,8-heptaCDD	2.986	1.115
octaCDD	10.421	12.937
2,3,7,8-tetraCDF	0.171	-
2,3,4,7,8-pentaCDF	-	0.918
1,2,3,6,7,8-hexaCDF	0.081	0.101
1,2,3,4,6,7,8-heptaCDF	0.217	0.267
octaCDF	0.075	0.094

## Analytical methods for PCDDs and PCDFs

All of the procedures can be divided into three main steps: extraction, clean-up, and final analysis by gas chromatography/mass spectrometry (GS/MS). A summary of extraction and clean-up methods and GC/MS conditions, based on information supplied by participants, is contained in Tables 2 and 3, respectively.

Regarding milk, initial extraction was most frequently performed by the pesticide residues method proposed by the Association of Official Analytical Chemists (AOAC) (1), although some alternatives, such as extraction with acetone/hexane or solvent extraction following freeze drying, were used. The same methods were also applied to blood, although the single, most widely used method was a two-phase extraction with chloroform/methanol/water. For clean-up, most laboratories used a sequence of column chromatographic steps aided in some cases by liquid-liquid partition. Many of the procedures were similar to that of Smith et al. (2), but variations and alternatives were also used. None of the participants used high performance liquid chromatography (HPLC) in their clean-up. There was no evidence that any of these procedures was inherently better in terms of accuracy, precision, or sensitivity.

In the GC/MS analysis almost an equal number of participants used the on-column and splitless injection techniques. Most participants used nonpolar GC columns (DB5 and similar), but a few used polar columns (such as CPSIL88 or SP2330) in addition or exclusively. Most participants used double-focusing mass spectrometers at resolutions between 2000 and 10 000 running in electron ionization mode. A small proportion of participants used quadrupole instruments with negative ion chemical ionization, and one participant used a quadrupole mass selective detector with electron ionization for part of the study.

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*Table 2. Summary of extraction and clean-up methods for PCDDs and PCDFs*

Laboratory code no.	Sample	Fat extraction	Determination method	Clean-up procedure
1	Milk	Ethanol, ether/hexane	G,I	Column chromatography with silica, KOH/silica, carbon/glass fibre, CsOH/silica, H <sub>2</sub> SO <sub>4</sub> /silica, alumina
	Blood	Chloroform/methanol /water	G	Column chromatography with H <sub>2</sub> SO <sub>4</sub> /silica, NaOH/silica, silica, silica, alumina, Carbopak C
2	Milk	Acetone/hexane	I	Column chromatography on KOH/silica, carbon/silica, H <sub>2</sub> SO <sub>4</sub> /silica, CsOH/silica, alumina
3	Milk	Acetone/hexane	G	Hexane/conc. H <sub>2</sub> SO <sub>4</sub> partition, column chromatography on H <sub>2</sub> SO <sub>4</sub> /SiO <sub>2</sub> , SiO <sub>2</sub> , basic Al <sub>2</sub> O <sub>3</sub> , 18% Carbopak C/Celite
	Blood	Chloroform/methanol/water	G	" " "
4	Milk	Sodium oxalate, methanol, hexane/ether	G	Column chromatography with silica, KOH/silica, carbon/glass fibre, H <sub>2</sub> SO <sub>4</sub> /silica, alumina
5	Milk	NH <sub>4</sub> OH, ethanol, ether/pentane	G	Column chromatography on silica, KOH/silica, carbon/glass fibre, H <sub>2</sub> SO <sub>4</sub> /silica, Florisil

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Table 2. Continued

Laboratory code no.	Sample	Fat extraction	Determination method	Clean-up procedure
	Blood	Chloroform/methanol/ water	G	" " "
6	Milk	Potassium oxalate, ethanol, ether/pentane	G	Column chromatography Florisil (1% water), Carbopak C/ethanol, ether/pentane Celite
	Blood	" "		Column chromatography on H <sub>2</sub> SO <sub>4</sub> /silica, Florisil (1% water), Carbopak C/Celite
7	Milk	Centrifugation, sodium sulfate, hexane/ acetone or oxalate, ethanol, ether/pentane	G	H <sub>2</sub> SO <sub>4</sub> /silica, NaOH/silica eluted with hexane, Carbopak C/Celite eluted with dichloromethane/ cyclohexane, toluene, AgNO <sub>3</sub> /silica, basic alumina eluted with hexane
	Blood	Oxalate, ethanol, ether/pentane	G	" " "
8	Milk	Acetone/hexane	G	Hexane/H <sub>2</sub> SO <sub>4</sub> partition, column chromatography on acid/base silica, Florisil, carbon
	Blood	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> , ethanol/hexane	G	" " "

Analytical methods for PCDDs and PCDFs 13

Table 2. Continued

Laboratory code no.	Sample	Fat extraction	Determination method	Clean-up procedure
9	Milk	Sodium oxalate, ethanol, ether, hexane	G	H <sub>2</sub> SO <sub>4</sub> silica, neutral alumina, carbon, potassium silicate, acid/alumina
	Blood	Ammonium sulfate, ethanol, hexane	G	" " "
10	Blood	Dichloromethane/methanol/water	G	Column chromatography on KOH/silica, silica, carbon, CsOH/silica, H <sub>2</sub> SO <sub>4</sub> /silica, alumina
11	Milk	Oxalate, ethanol, ether/hexane	G	Column chromatography with H <sub>2</sub> SO <sub>4</sub> /silica, H <sub>2</sub> SO <sub>4</sub> , carbon/Celite eluted with dichloromethane/cyclohexane, toluene, basic alumina eluted with dichloromethane/hexane
	Blood	Chloroform/methanol/water	G	" " "
12	Milk, blood	Methanol/chloroform	G	Hexane/H <sub>2</sub> SO <sub>4</sub> partition, column chromatography on alumina with hexane, CCl <sub>4</sub> , CH <sub>2</sub> Cl <sub>2</sub>
13	Milk	Ethanol/ethyl ether	G	Column chromatography with silica, alumina, carbon/silica

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Table 2. Continued

Laboratory code no.	Sample	Fat extraction	Determination method	Clean-up procedure
14	Blood	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> , ethanol/hexane	J	KOH/silica, H <sub>2</sub> SO <sub>4</sub> /silica, silica, KOH/silica, silica, carbon/glass fibre, CsOH/silica, H <sub>2</sub> SO <sub>4</sub> /silica, acid/alumina
15	Blood	Solid phase extraction	G	Silica, KOH/silica, H <sub>2</sub> SO <sub>4</sub> /silica, carbon/glass fibre, CsOH/silica, H <sub>2</sub> SO <sub>4</sub> /silica, alumina
16	Milk	Sodium oxalate, methanol, hexane/ether	G	Column chromatography with silica, KOH/silica, carbon/Celite, H <sub>2</sub> SO <sub>4</sub> /silica, CsOH/silica, alumina
17	Milk, blood	Sodium oxalate, methanol, ether/petroleum, ether	G	Adsorbed on Carbosphere, rinsed with dichloromethane, extracted with toluene, column chromatography on alumina with hexane/CH <sub>2</sub> Cl <sub>2</sub>
18	Milk	Freeze drying, Soxlet with toluene	G	Column chromatography with carbon/glass fibre, acid and basic silica, silver nitrate silica, alumina
	Blood	Chloroform/methanol/water	G	Column chromatography with carbon/glass fibre, acid and basic silica, silver nitrate silica, alumina

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*Table 2. Continued*

Laboratory code no.	Sample	Fat extraction	Determination method	Clean-up procedure
19	Milk	Sodium oxalate, ethanol, ethyl ether/hexane	G	Column chromatography on carbon/glass fibre with hexane/ethyl ether then toluene, basic alumina
	Blood	Formic acid, solid phase extraction	G	" " "

G = Gravimetric; I = Infra-red; J = Summation of individual lipids.

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Table 3. Summary of analytical equipment and methods for PCDDs and PCDFs

Lab- oratory code no.	GC equipment		MS equipment		
	Injection technique	Columns	Instrument	Ionization mode	Resolution
1	Splitless	SP2330; 60x0.25; 0.25	VG 70-250S	EI	10 000
2	Splitless	DB5; 30x0.25; 0.25 or FFAP; 15x0.25; 0.25	VG 7070E	EI	2 000
3	Splitless	HP Ultra 2; 25x0.2; 0.33	VG 70-250S	EI; 30eV	10 000
4	Splitless	HP5; 25x0.2; 0.33 or; RTX233030x0.25; 0.1	HP 5987	EI (TCDD) or NICI	Low
5	Splitless	CPSIL8; 50x0.32; 0.12	VG 7070H	EI; 30eV	5 000
6	Splitless	DB5; 30x0.25; 0.1	VG Autospec	EI	10 000
7	On-column	Ultra 2; 50x0.32; 0.17	Finnigan MAT8230	EI	5 000
8	On-column	DB5; 25x0.25; 0.25	VG 7070	EI	2 000
9	Splitless	DB5; 60x0.25; 0.25	VG 70-250S	EI	10 000
10	On-column	OV2250H; 20x0.3; 0.2	Finnigan MAT4510	NICI	Low

Table 3. Continued

Lab- oratory code no.	GC equipment		MS equipment		
	Injection technique	Columns	Instrument	Ionization mode	Resolution
11	Splitless	HP5; 25x0.20; 0.11	VG 70SE	EI; 30eV	10 000
12	Splitless	DB5 and CPSil88; 50x0.25; 0.25	VG 70SQ	EI	10 000
13	On-column	DB5; 60x0.32; 0.1	Kratos MS-50	EI	Mass pro- file >5 000
14	Splitless	SP2331; 60x0.25; 0.25	VG 70S	EI; 30eV	10 000
15	On-column	SE54; 60x0.25; 0.2 or DB5; 30x0.25; 0.1	VG 7035	EI; 30eV	1 500-2 000
16	On-column	DB5; 60x0.32; 0.1	VG 7070E	EI; 30eV	10 000
17	Falling needle	CPSIL 19CB; 25x0.25; 0.21	VG 70SQ	EI	3 000
18	On-column	DB5; 60x0.32; 0.1	HP MSD (milk) VG 70SQ (blood)	EI	Low (milk) 3 000 (blood)
19	Splitless	DB5; 60x0.25; 0.1	Finnigan 4500	NICI	Low

## Analytical problems with PCDDs and PCDFs

Practical difficulties were encountered during analysis. Some participants reported that analytical standards shipped by the coordinating laboratory had evaporated or leaked; replacement standards were subsequently supplied by the coordinators and these problems did not affect the outcome of the study. In future studies a larger volume of standard solution should be shipped and the ampoule should be weighed before and after shipping.

None of the laboratories with extensive experience in analysing PCDDs and PCDFs in milk reported any unusual difficulties with these samples, although one participant reported that the requirement to measure recoveries meant that an HPLC clean-up step normally applied had been omitted and this had resulted in high background signals in the GC/MS measurement. Some laboratories with little previous experience had severe difficulties in obtaining sufficiently low blanks, adequate recoveries, and repeatable results.

In the case of blood, an excessively high fortification level of 2,3,4,7,8-pentaCDF had inadvertently been added to one of the subpools. This prevented meaningful data from being obtained for this congener and for 1,2,3,7,8-pentaCDF, and also introduced interference with 2,3,7,8-tetraCDF. Many of the laboratories had limited previous experience with blood analysis and this analysis was more difficult because of the lower fat content compared to milk.

The double-focusing mass spectrometer is advantageous in providing high sensitivity with a uniform response. Quadrupole instruments operating in electron ionization mode were considered suitable for milk analysis only in conjunction with a very effective clean-up, and not at all for blood. Most of the current methods of analysis are tedious and time consuming.

## Analytical methods for PCBs

The most frequently used method for fat extraction was that proposed by AOAC, as was generally used for PCDD and PCDF analyses. Some laboratories also used solvent extractions such as acetone/petroleum ether or ethanol/acetonitrile. Column chromatographic techniques with alumina

and silica packings were used for clean-up, a slight modification of those used for PCDD and PCDF analyses.

For identification and quantification of different isomers, many laboratories used normal capillary chromatographic instrumentation equipped with electron capture detection. Some laboratories completed the analyses using GC/MS instrumentation. Nonpolar GC columns, such as DP5 and HP5, were most frequently used for identification although some laboratories also used polar columns, such as CPSIL18.

A summary of extraction and clean-up methods and of analytical instrumentation, based on information supplied by participants, is presented in Tables 4 and 5, respectively.

Table 4. Summary of extraction and clean-up methods for PCBs

Laboratory code no.	Sample	Fat extraction	Determination method	Clean-up procedure
1	Milk	Na-oxalate, ethanol, diethylether/n-hexane	G	Florisil
2	Milk	Sodium oxalate, ethanol, ether, hexane	G	H <sub>2</sub> SO <sub>4</sub> silica gel
	Blood	Ammonium sulfate, ethanol, hexane	G	" " "
3	Milk, blood	Acetone/petroleum ether	G	Basic alumina
4	Milk	Oxalate, ethanol, ether/hexane	G	Column chromatography with H <sub>2</sub> SO <sub>4</sub> /silica, H <sub>2</sub> SO <sub>4</sub> , carbon/Celite eluted with 50% dichloromethane/cyclohexane, basic alumina eluted with 2% dichloromethane/hexane
	Blood	Chloroform/methanol/water	G	" " "

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Table 4. Continued

Laboratory code no.	Sample	Fat extraction	Determination method	Clean-up procedure
5	Milk, blood	Na-oxalate, methanol, diethylether/petroleum ether	G	Alumina with hexane/dichloromethane, silica with hexane
6	Milk	Ethanol-acetonitrile	I	Extraction with hexane
7	Milk	GERBER-method (centrifugation with H <sub>2</sub> SO <sub>4</sub> and amyl alcohol	I	Extraction with hexane
8	Milk, blood	Potassium oxalate, ethanol, ether, pentane	G	Column chromatography on silica (1.5% water)
9	Milk	Na-oxalate, ethanol, diethylether hexane	G	CH <sub>3</sub> CN partition, Florisil column
10	Milk	Centrifugation, NaSO <sub>4</sub> , hexane/acetone	G	H <sub>2</sub> SO <sub>4</sub> /silica, NaOH/silica eluted with hexane, Carboapak C/Celite eluted with dichloromethane/cyclohexane
	Blood	Oxalate, ethanol, ether/pentane	G	" " "

G = Gravimetric; I = Infra-red.

Table 5. Summary of analytical equipment and methods for PCBs

Lab- oratory code no.	GC equipment		MS equipment		
	Injection technique	Columns	Instrument	Ionization mode	Resolution
1	Splitless	HP PONA; 25x0.2; 0.50	HP 5970B	EI	Low
2	Splitless	DB5; 60x0.25; 0.25	Varian 3700	ECD	-
3	Splitless	CPSIL8CB; 50x0.32; 0.13	Varian 3500	-	-
4	Splitless	HP; 25x0.20; 0.11	VG 70SE	EI; 30eV	10 000
5	Splitless	HP Ultra 2; 50x0.20; 0.33	HP 5880A	-	-
6	Splitless	DB5; 30x0.25; 0.1	HP 5880	-	-
7	Splitless	HP5; 40x0.2; 0.33	HP 5970	EI	Low
8	Split	DB5; 60x0.25; 0.1; DB 1701; 30x0.25; 0.1	Varian 3700/ECD	-	-
9	Splitless	HP Ultra 2; 25x0.2; 0.33	VG 70-250S	EI; 30eV	6 000
10	Splitless	HP Ultra 2; 25x0.2; 0.11	Varian 3400	-	-

## Analytical problems with PCBs

Laboratories met fewer difficulties in analysing PCBs compared to PCDDs and PCDFs. PCB congeners analysed within this study were those with International Union of Pure and Applied Chemistry (IUPAC) numbers 28, 52, 101, 138, 153, and 180. Further interlaboratory quality studies should also include congeners that are more important from a toxicological point of view and found in higher concentrations in human milk and blood samples. These congeners have IUPAC numbers 77, 118, 126, and 169. Other congeners might also be considered.

## Reporting of results from laboratories

The informal meeting held in Toronto in September 1989 had agreed on a deadline of 28 February 1990 for the reporting of final results. In practice, results were accepted until 20 March, allowing nine additional sets of results to be considered. Within the revised timeframe the number of laboratories reporting results were:

	Milk	Blood
PCDDs and PCDFs	16	15
PCBs	10	6

Results, sent to WHO/EURO on standard reporting forms, were entered into a database by a person without any knowledge of the identities of the participating laboratories. When "not detected" was reported, half of the detection limit has been used rather than a "missing value". However, if the detection limit was not reported, the result was treated as a missing value. Independent verification of the accuracy of the data entered was made but due to time constraints, the participating laboratories were unable to proofread their own data.

## Determination of fat content

The fat content determined in association with each analysis for PCDDs and PCDFs appears in Figure 1 for milk and Figure 2 for blood. The variability was considered to be unsatisfactory, especially so in the case of blood, and therefore all further treatment of the data was made on a whole sample basis. A possible contribution to this variability was the variety of methods used, some of which might discriminate against certain components of fat to different degrees. Further efforts to improve the reliability and comparability of fat determinations were recommended.

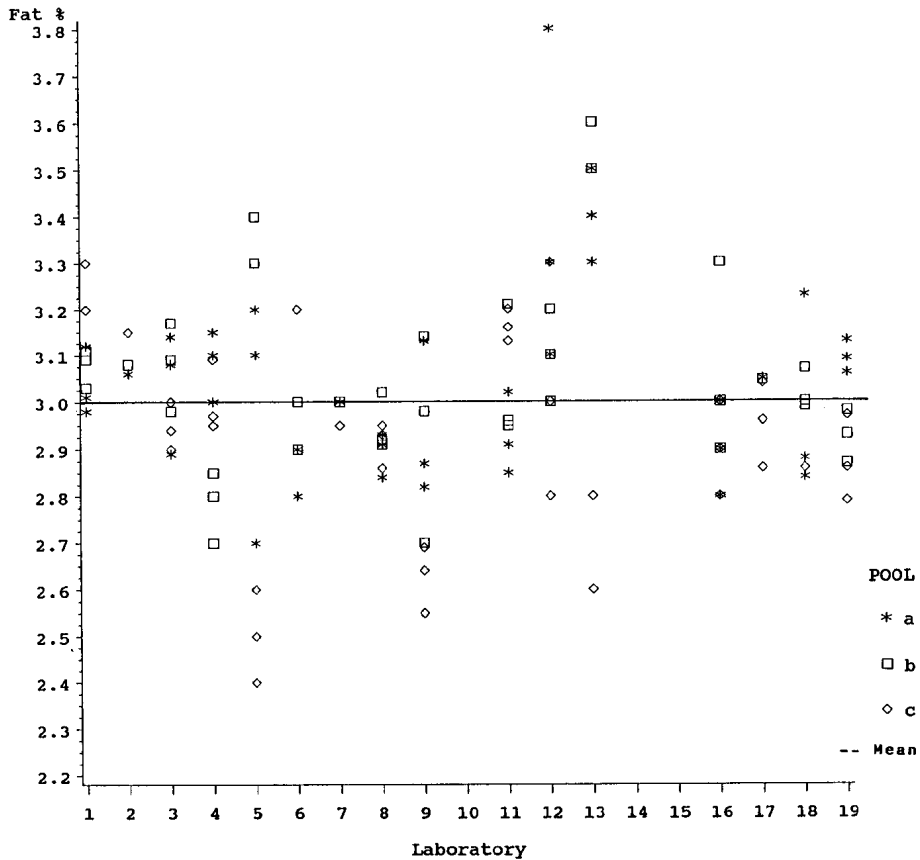


Figure 1. Fat content (%) of PCDDs and PCDFs in milk

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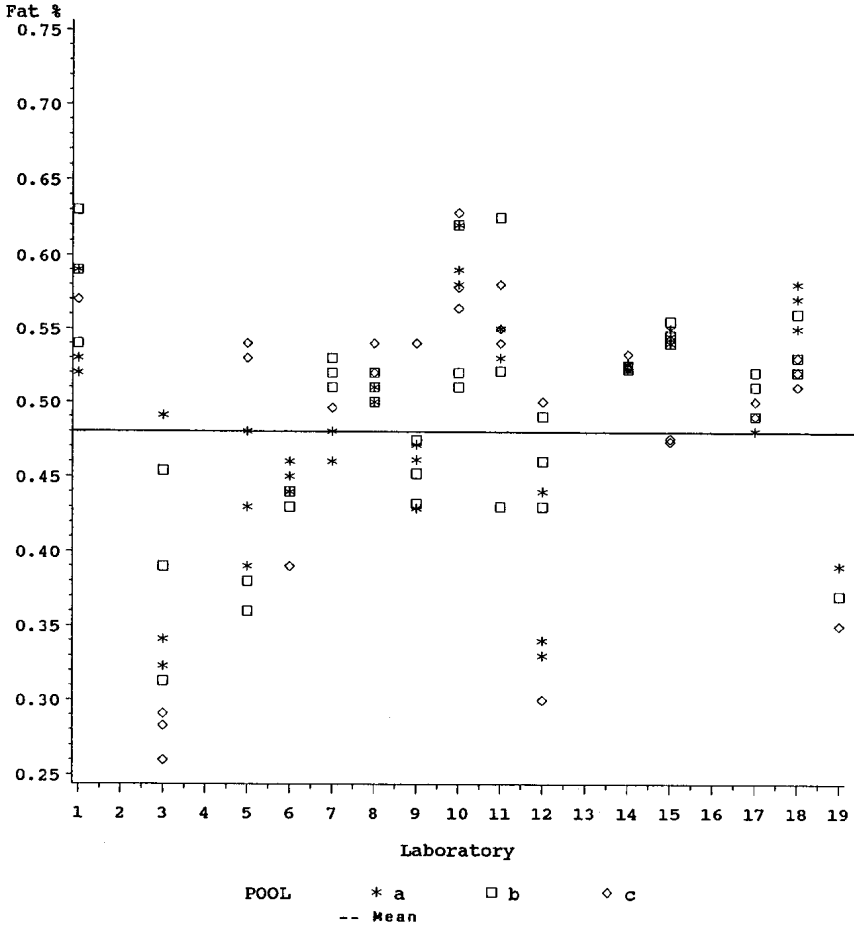


Figure 2. Fat content (%) of PCDDs and PCDFs in blood

### Statistical analysis of data

The coordinating committee had met on 20 March 1990 to review the statistical treatment of the results, which had been prepared for presentation at the consultation. PCDDs and PCDFs, and PCBs were treated as two separate categories, as were the two matrices, milk and blood. The most important output of this treatment was a score for each laboratory. The reproducibility and repeatability are defined as follows (3,4):

**Reproducibility:** Closeness of agreement between individual results obtained with the same method on identical test material but under different conditions (e.g. time).

**Repeatability:** Closeness of agreement between successive results obtained with the same method on identical test material and under the same conditions (e.g. time).

In this study the reproducibility for each laboratory was based on the absolute deviation from the median value for each pool and congener. To obtain the final score for each laboratory, the following calculations were made:

- the median value for each pool and congener;
- the average of the three determinations for each laboratory, pool, and congener;
- the absolute deviation (from the average) for each laboratory from the median for each pool and congener;
- the ranks of the absolute deviations for each pool and congener; if the laboratory had a missing value for this combination, the laboratory was given the same rank as that of the worst deviation;
- the ranks for the three pools were averaged for each laboratory and congener;
- the average ranks were (weighted and) summed over all congeners using toxic equivalent factors according to the Nordic equivalent model;
- in the final score, a weight factor of 1.0 was given for reproducibility.

The repeatability for each laboratory was based on the standard deviations between the three determinations. To obtain the final score, the following calculations were made:

- the standard deviation of the three determinations for each laboratory, pool, and congener;

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- the ranks of the standard deviations for each pool and congener; if the laboratory had a missing value for this combination, the laboratory was given the same rank as that of the worst standard deviation;
- the ranks for the three pools were averaged for each laboratory and congener;
- the average ranks were (weighted and) summed over all congeners using toxic equivalent factors according to the Nordic equivalent model;
- in the final score, a weight factor of 0.8 was given for repeatability.

The scores for reproducibility and repeatability, as well as the final score for each laboratory, are presented in Annex 1. This also includes the calculated mean values, standard deviations, and median values for each congener.

The coefficient of variation (CV) was calculated for both reproducibility and repeatability. The CV value for reproducibility for each laboratory was computed as the absolute deviation from the median for each laboratory, congener, and pool divided by the median for each congener and pool. To obtain a more robust estimate of the CV for reproducibility, this measure was used instead of the method where the standard deviation is divided by the mean. The CV value for repeatability for each laboratory was computed as the standard deviation between the three determinations for each laboratory, congener, and pool divided by the mean for each laboratory, congener, and pool. The average CV for each laboratory was calculated from the CV values for reproducibility and repeatability. These values are presented in Annex 1.

Some of the PCDD and PCDF congeners had been spiked with certain amounts in two of the three pools. The PCBs were not spiked. Since the spiked levels were known, the recoveries (in %) of the spiked amounts for each laboratory could be computed. In the milk samples, pools A and C were spiked and in the blood samples, pools A and B. The spiked congeners and spiking levels are presented in Table 1. The recovery was computed as the difference between the spiked and unspiked pool for each

laboratory, congener, and determination. It was calculated as a percentage of the spiking level (e.g. 100% means that the whole added amount was detected in the congener in question). The calculated recoveries for each laboratory are presented in Annex 1.

In the statistical calculations the following points should be noted:

1. In summarizing the results for all congeners for each laboratory, ranks were used instead of the measured values since this gives a much more robust analysis (5).
2. The data were replaced by ranks based on the analytical results reported to WHO/EURO. The ranks were calculated for every combination of congener and pool. They were standardized to lie between 0 (smallest and best) and 1 (largest and worst), which is equivalent to the usual ordinal scale.
3. Missing measurements were replaced by the worst rank, which is 1. In this way, missing values were given a penalty in the analysis by using the worst case results.
4. The total score for each laboratory was measured as the (weighted) sum of rankings for all congeners and pools.
5. The score for repeatability and reproducibility for each laboratory was calculated by using a toxic equivalent factor according to the Nordic equivalent model. Using the international toxic equivalent factors (I-TEF) would not have significantly altered the outcome.
6. In calculating the final score for each laboratory, the reproducibility was weighted by a factor of 1.0 and the repeatability by a factor of 0.8.

All of the results were presented at the consultation using coded laboratory numbers, the coding remaining unknown to participants and to the expert performing the statistical analysis until the discussion at the consultation was completed. The actual analytical results were not made available and only the statistical parameters were considered. In this way

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participants were unable to identify the scores associated with their own results.

In order that alternative statistical examinations of the data can be made, WHO/EURO will arrange to supply the raw data from laboratories, identified by code number, upon request by any of the participating laboratories.

### Criteria for qualification of laboratories

The criteria for qualification of the laboratories were based on the final scores. The coefficients of variation for repeatability and reproducibility for individual congeners averaged over all congeners, were also calculated to verify the validity of the final scores. Moreover, the ratio between theoretical and observed fortification levels ("recovery") was discussed. Other possible criteria were also discussed, including the detection limits and percentage recovery of the internal standards. However, as some laboratories had not reported these data, they could not be included when setting the quality criteria. Furthermore, their inclusion would not have changed the number of laboratories that qualified.

#### *PCDDs and PCDFs in milk*

Based on the final scores, the results of laboratories with code numbers 2, 11, 12, and 18 were clearly of a significantly lower standard than those of the remaining laboratories. This finding corresponds, in terms of an average CV for reproducibility and repeatability (based on median values), to about 30%. The above four laboratories were therefore not regarded as qualified. The qualified laboratories are listed in Annex 2.

#### *PCDDs and PCDFs in blood*

Based on the final scores, the results of laboratories with code numbers 10, 11, and 12 were clearly of a significantly lower standard than those of the remaining laboratories. This finding corresponds, in terms of an average

CV for reproducibility and repeatability (based on median values), to about 45%. The above three laboratories were therefore not regarded as qualified. The qualified laboratories are listed in Annex 3.

In the case of blood, the analytical difficulties are greater and the CVs and deviations in the observed fortification levels tend to be larger than for milk, as reflected in the larger variations in the ranges. The overall quality of blood analysis needs to be improved, and not only in the case of the laboratories that did not qualify.

#### *PCBs in milk*

Based on the final scores, the results of laboratories with code numbers 1, 3, 4, and 6 were clearly of a significantly lower standard than those of the remaining laboratories. This finding corresponds, in terms of an average CV for reproducibility and repeatability (based on median values), to about 20%. The above four laboratories were therefore not regarded as qualified. The qualified laboratories are listed in Annex 4.

#### *PCBs in blood*

Based on the final scores, the results of laboratories with code numbers 3 and 4 were clearly of a significantly lower standard than those of the remaining laboratories. This finding corresponds, in terms of an average CV for reproducibility and repeatability (based on median values), to about 20%.

The above two laboratories were therefore not regarded as qualified. The qualified laboratories are listed in Annex 5.

Regarding the CV values for reproducibility and repeatability for PCDDs, PCDFs, and PCBs, the results show that the CV values for reproducibility are higher, reflecting difficulties in analysing samples over a longer period of time. Regarding the recovery values, these are much worse in the case of those laboratories that did not qualify as compared with the laboratories that qualified.

## Need for further studies

The agreement reached at the Copenhagen consultation in 1988 that quality control studies should be repeated every 2 years was endorsed. The same study design should be used in principle and a design committee should decide the necessary details. In particular the committee should specify the exact method for fat determination. The information to be reported should be considered by the committee but should include details of detection limits and recoveries. The study design should also require the analysis of method blanks at specified intervals to identify any cross contamination. As noted above, standards should be diluted so that a larger volume is shipped and ampoules should be weighed before and after shipping.

## Conclusions and Recommendations

### *Conclusions*

1. The variability of fat measurements, especially for blood, was unsatisfactory, possibly because of the variety of methods used. Because of this, the results from this study were interpreted on a whole-sample basis.
2. After consideration of both the accuracy and the precision of the analyses of PCDDs and PCDFs in milk, 12 laboratories were judged qualified out of 16 reporting results.
3. For the analysis of PCDDs and PCDFs in blood, the difficulties are greater and a wider range of variation should, for the time being, be regarded as acceptable. This wide tolerance was regarded as necessary in order not to restrict unreasonably the number of laboratories producing new data for health risk assessment; however, all except a very few of these laboratories are urgently requested to seek ways of improving the quality of their results. Twelve laboratories qualified out of 15 submitting results.

4. For the analysis of PCBs in milk, six laboratories qualified out of 10 submitting results.
5. For the analysis of PCBs in blood, four laboratories qualified out of six submitting results.
6. Simple methods for the analysis of PCBs might provide useful results and would promote the gathering of data from the widest possible area, including developing countries. Congener-specific analysis is essential in order to provide comparable results.

### *Recommendations*

1. Only results from laboratories that had qualified in this or some other appropriate study should be used by the WHO Regional Office for Europe for further health risk assessment.
2. In future studies a design committee should specify the exact methods for fat determination to be followed by all laboratories. In general, laboratories are encouraged to improve the reliability of their fat determinations, noting that simple and easily applied methods are preferred.
3. Those laboratories that did not fulfil the criteria for qualification, should take steps to improve their methodology before performing further analyses. Furthermore, they are strongly requested to take part in future interlaboratory quality control studies.
4. A further interlaboratory quality control study should be conducted in 2 years for both PCDDs and PCDFs, and PCBs. The determination of PCBs should include at least the congeners with IUPAC numbers 28, 52, 77, 101, 118, 126, 138, 153, 169, and 180. Other congeners might also be considered.
5. In future studies the design committee should specify the information to be reported, and this should include details of detection limits and

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recoveries. The exact method by which these are to be determined should be specified by the committee. The design of the study should also require analysis of blanks at specified intervals. Failure to report all the information requested may result in penalties or rejection of the results.

6. A European programme for the determination of PCDDs and PCDFs in food should be included in the UNEP/WHO Global Environmental Monitoring System (GEMS) food programme. The monitoring programme should include the three major commodity groups: cow's milk and milk products; fish and fish products; and meat and meat products. Special attention should be paid to cow's milk. [Note: All of the laboratories represented at this consultation have provisionally indicated their willingness to take part.]
7. Considering the need to gather data on PCBs from the widest possible area, including developing countries, and to use the most appropriate methods of analysis, the methodology, as a minimum, should be based on capillary column GC with electron capture detection and should provide for the measurement of congeners with IUPAC numbers 28, 52, 101, 138, 153, and 180.

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## ANNEX 1

# Statistical analysis of results from laboratories participating in the second round of quality control studies on levels of PCBs, PCDDs, and PCDFs in human milk and blood

Prepared by Annette Ersboell, statistical expert, Lyngby, Denmark

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The measurements can be described by the variable  $Y_{ijkl}$  where

Congener	: $i = 1, 2, \dots, n_i$
Pool	: $j = 1, 2, 3$
Laboratory	: $k = 1, 2, \dots, n_k$
Determination	: $l = 1, 2, 3$

### 1. Formula for computation of reproducibility

Rankings are measured according to the standardized average  $\bar{Z}_{ijk}$ .

Let  $Z_{ijkl}$  be the absolute value of the observation standardized with the median.

$$Z_{ijkl} = |Y_{ijkl} - Y_{ij..}^*| \quad \text{where } Y_{ijkl} \text{ is the observed value and}$$

$Y_{ij..}^*$  is the median for congener  $i$  and pool  $j$ .

The standardized average  $\bar{Z}_{ijk}$  is then  $\bar{Z}_{ijk} = \frac{1}{n_1} \sum_1 Z_{jkl}$  where  $n_1$

is the number of replications.

### 2. Formula for computation of repeatability

Rankings are measured according to the standard deviation  $s_{ijk}$  between the three determinations.

The standard deviation between the three determinations  $s_{ijk}$  is the

square root of the variation  $s_{ijk}^2$  between the three determinations.

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$$s_{ijk}^2 = \frac{1}{n_1 - 1} \sum_1 (Y_{ijk} - \bar{Y}_{ijk})^2 \quad \text{where } Y_{ijk} \text{ is the observed value, } \bar{Y}_{ijk}$$

is the mean value of the replications 1 and  $n_1$  is the number of replications.

The standard deviation is then  $s_{ijk} = \sqrt{s_{ijk}^2}$

3. *Formula for computation of coefficient of variation for reproducibility*

The coefficient of variation is the standard deviation divided by the mean value.

To get a more robust estimate of the coefficient of variation for the reproducibility, the absolute deviation from the median for each laboratory, congener, and pool are used instead of the standard deviation and the median value for each laboratory, congener, and pool is used instead of the mean value (see 1. above).

This means that the coefficient of variation for the reproducibility is (using the results from 1. above)

$$CV_{reproducibility} = \frac{\bar{Z}_{ijk}}{Y_{ijk}^*}$$

4. *Formula for computation of coefficient of variation for repeatability*

The coefficient of variation for repeatability is computed as the standard deviation between the three determinations for each laboratory, congener, and pool divided by the mean value of the three determinations for each laboratory, congener, and pool.

This means, that the coefficient of variation for the repeatability

is (using the results from 2. above)  $CV_{repeatability} = \frac{s_{ijk}}{\bar{Y}_{ijk}}$

5. *Formula for computation of recovery of spiked amounts*

For each laboratory the recovery of the spiked level in pool s compared

with the unspiked pool t is  $R_{st} = \frac{Y_{istl} - Y_{itkl}}{X_{is}} \times 100\%$  where  $Y_{istl}$  is the

observed value for congener i, pool s, laboratory k, and determination l where pool s is spiked;  $Y_{itkl}$  is the observed value for congener i, pool t, laboratory k, and determination l where pool t is unspiked; and  $X_{is}$  is the spiking value for congener i and pool s.

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Table 1. List of congeners

Congener no.	Congener name
PCDDs and PCDFs	
1	2,3,7,8-tetradoxin
2	1,2,3,7,8-pentadoxin
3	1,2,3,4,7,8-hexadoxin
4	1,2,3,6,7,8-hexadoxin
5	1,2,3,7,8,9-hexadoxin
6	1,2,3,4,6,7,8-heptadoxin
7	1,2,3,4,6,7,8,9-octadoxin
8	2,3,7,8-tetrafuraxin
9	1,2,3,7,8-pentafuraxin
10	2,3,4,7,8-pentafuraxin
11	1,2,3,4,7,8-hexafuraxin
12	1,2,3,6,7,8-hexafuraxin
13	1,2,3,7,8,9-hexafuraxin
14	2,3,4,6,7,8-hexafuraxin
15	1,2,3,4,6,7,8-heptafuraxin
16	1,2,3,4,7,8,9-heptafuraxin
17	1,2,3,4,6,7,8,9-octafuraxin
PCBs	
1	IUPAC Number 28
2	IUPAC Number 52
3	IUPAC Number 101
4	IUPAC Number 138
5	IUPAC Number 153
6	IUPAC Number 180

*Table 2.* PCDDs and PCDFs in milk (on volume basis/parts-per-trillion): mean, standard deviation (SD), median, and range

Congener		Pool		
		A	B	C
1	Mean	0.07	0.06	0.26
	SD	0.04	0.02	0.13
	Median	0.05	0.06	0.22
	Range	0.17	0.09	0.44
2	Mean	0.12	0.12	0.48
	SD	0.04	0.03	0.13
	Median	0.12	0.11	0.45
	Range	0.18	0.17	0.76
3	Mean	0.14	0.07	0.10
	SD	0.05	0.03	0.14
	Median	0.15	0.07	0.07
	Range	0.24	0.15	0.90
4	Mean	1.04	0.54	1.14
	SD	0.24	0.17	0.25
	Median	1.00	0.53	1.10
	Range	1.10	0.85	1.32
5	Mean	0.16	0.13	0.13
	SD	0.07	0.04	0.05
	Median	0.15	0.13	0.13
	Range	0.44	0.20	0.27
6	Mean	4.12	1.32	2.62
	SD	0.86	0.23	0.60
	Median	4.04	1.30	2.49
	Range	3.80	1.17	2.30
7	Mean	14.25	6.34	17.95
	SD	4.46	2.70	8.53
	Median	13.60	5.64	15.90
	Range	18.19	13.03	41.90

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Table 2. Continued

Congener		Pool		
		A	B	C
8	Mean	0.18	0.04	0.05
	SD	0.04	0.01	0.03
	Median	0.19	0.04	0.04
	Range	0.21	0.08	0.12
9	Mean	0.02	0.02	0.02
	SD	0.02	0.01	0.01
	Median	0.02	0.02	0.01
	Range	0.08	0.05	0.07
10	Mean	0.28	0.27	1.11
	SD	0.08	0.08	0.21
	Median	0.28	0.26	1.10
	Range	0.54	0.49	0.89
11	Mean	0.13	0.09	0.09
	SD	0.06	0.02	0.04
	Median	0.13	0.09	0.08
	Range	0.32	0.13	0.19
12	Mean	0.13	0.08	0.15
	SD	0.04	0.09	0.04
	Median	0.13	0.07	0.14
	Range	0.22	0.67	0.25
13	Mean	0.03	0.02	0.02
	SD	0.02	0.01	0.01
	Median	0.02	0.02	0.01
	Range	0.10	0.05	0.05
14	Mean	0.05	0.04	0.05
	SD	0.04	0.01	0.10
	Median	0.04	0.03	0.03
	Range	0.24	0.07	0.70

Table 2. Continued

Congener		Pool		
		A	B	C
15	Mean	0.31	0.16	0.38
	SD	0.10	0.14	0.27
	Median	0.30	0.13	0.32
	Range	0.71	0.76	1.72
16	Mean	0.03	0.03	0.02
	SD	0.03	0.03	0.02
	Median	0.02	0.02	0.01
	Range	0.15	0.15	0.06
17	Mean	0.12	0.16	0.19
	SD	0.07	0.32	0.23
	Median	0.10	0.04	0.11
	Range	0.37	1.81	0.96

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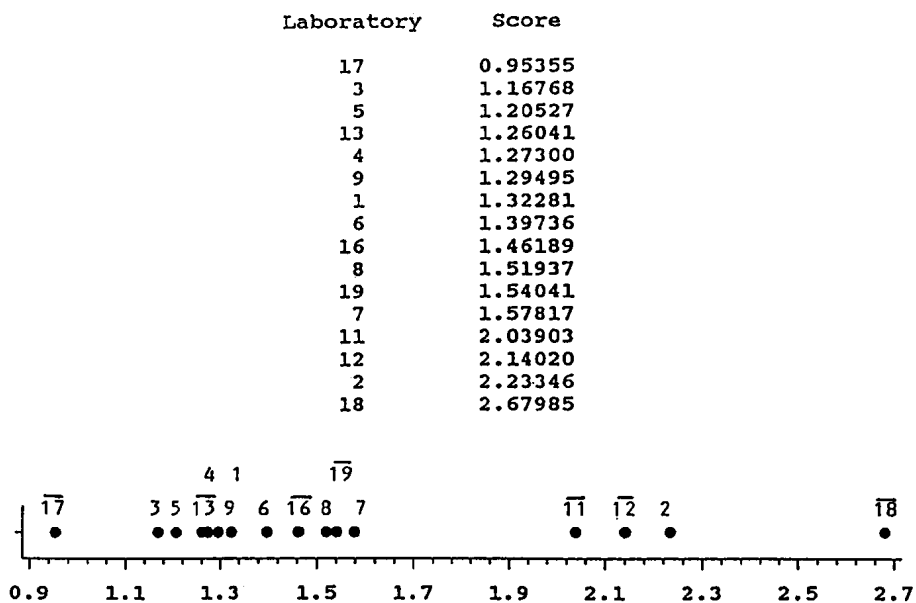


Figure 1. PCDDs and PCDFs in milk: score for reproducibility (with Nordic equivalent model)

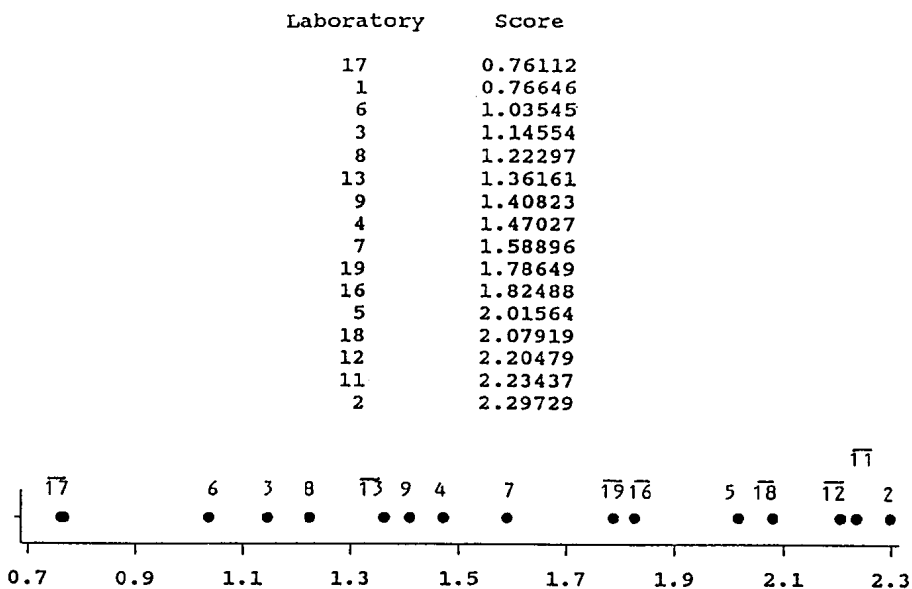


Figure 2. PCDDs and PCDFs in milk: score for repeatability (with Nordic equivalent model)

Laboratory	Score
17	1.56244
1	1.93598
3	2.08411
6	2.22572
13	2.34970
9	2.42153
4	2.44922
8	2.49775
5	2.81778
7	2.84934
16	2.92179
19	2.96960
11	3.82653
12	3.90403
2	4.07129
18	4.34320

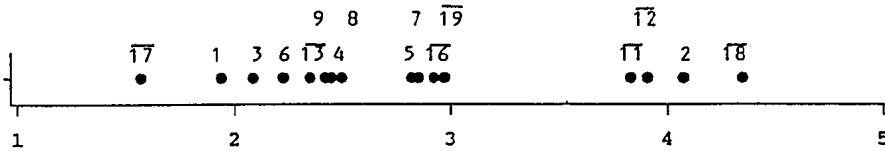


Figure 3. PCDDs and PCDFs in milk: final score (Nordic equivalent model) based on reproducibility and repeatability (weight factor 1.0 and 0.8)

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*Table 3.* PCDDs and PCDFs in milk: median coefficient of variation (CV) for reproducibility

Laboratory	CV	Range
1	14.04	488.63
2	38.89	2300.84
3	26.10	477.78
4	8.18	116.20
5	7.67	145.00
6	17.24	88.10
7	19.84	178.57
8	20.51	324.10
9	12.82	64.29
11	34.72	789.75
12	30.77	370.56
13	15.60	216.67
16	18.00	446.67
17	8.75	59.83
18	58.51	925.28
19	10.14	444.44

*Table 4.* PCDDs and PCDFs in milk: median coefficient of variation (CV) for repeatability

Laboratory	CV	Range
1	5.97	137.78
2	22.35	241.16
3	13.13	1175.40
4	11.76	154.00
5	15.80	116.36
6	8.33	108.39
7	12.37	257.60
8	8.18	170.45
9	8.33	464.20
11	24.89	1190.76
12	32.29	589.76
13	15.34	303.31
16	15.56	778.85
17	8.66	46.81
18	13.32	126.45
19	8.19	525.15

*Table 5.* PCDDs and PCDFs in milk: average coefficient of variation (CV) between reproducibility and repeatability

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Laboratory	Mean CV
1	10.00
2	30.62
3	19.62
4	9.97
5	11.74
6	12.79
7	16.11
8	14.35
9	10.58
11	29.81
12	31.53
13	15.47
16	16.78
17	8.70
18	35.92
19	9.16

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Table 6. PCDDs and PCDFs in milk: average recovery (%) of spiked amount (Pool A)

Laboratory	Mean							
	Congener							
	3	4	6	7	8	12	15	17
1	74.62	76.64	75.91	61.41	68.81	77.78	58.37	-261.78
2	-24.62	0.62	107.61	13.95	46.78	-16.46	15.36	-1108.89
3	67.80	87.85	103.82	143.30	91.62	114.81	84.33	31.11
4	114.02	90.97	93.99	82.21	93.18	101.23	93.24	95.11
5	-	-	89.98	81.92	88.11	99.59	82.33	124.89
6	98.86	122.18	108.28	99.80	69.59	77.37	67.59	69.78
7	81.06	137.69	108.73	50.28	91.62	55.56	97.85	33.33
8	132.20	148.91	141.77	131.79	124.17	-146.91	89.09	205.33
9	64.77	91.59	93.77	56.62	92.20	103.70	109.06	173.33
11	34.05	72.83	66.88	61.41	64.52	34.16	-33.49	-195.56
12	77.65	119.00	107.06	115.79	103.31	139.92	99.85	-37.78
13	62.50	96.26	85.29	63.94	74.07	61.73	89.86	173.33
16	65.53	72.90	74.79	88.92	67.84	90.12	57.76	56.00
17	85.61	83.49	79.93	73.25	73.88	83.95	69.12	102.56
18	128.79	104.67	103.59	60.45	101.36	139.92	86.02	-284.44
19	57.58	90.97	59.05	29.56	56.92	89.30	56.84	52.00

Table 7. PCDDs and PCDFs in milk: average recovery (%) of spiked amount (Pool C)

Laboratory	1	2	4	6	7	10	12	15	17
1	76.24	77.99	62.25	101.64	98.43	84.97	62.38	72.41	-109.93
2	201.32	200.56	125.50	173.39	219.01	107.84	141.91	76.15	-843.97
3	72.11	89.14	88.35	212.26	197.88	90.78	35.97	119.73	-143.62
4	45.54	86.82	90.86	114.20	77.81	84.97	91.09	65.79	78.72
5	82.67	92.20	-	111.81	75.78	89.65	79.21	69.91	64.18
6	103.30	119.78	103.46	137.52	90.70	109.30	88.45	77.40	148.58
7	88.45	112.81	114.96	163.23	44.57	100.58	94.06	74.53	15.96
8	56.60	73.35	57.23	59.79	62.35	79.16	-147.52	46.19	77.66
9	53.14	61.28	-	44.84	28.60	86.60	53.47	54.93	45.74
11	217.66	74.09	124.05	77.16	62.28	83.12	70.63	247.82	590.43
12	51.16	68.71	70.78	87.59	71.63	78.07	118.81	-14.98	-44.33
13	84.16	113.28	77.56	89.09	70.29	64.27	49.50	54.31	184.40
16	85.31	108.64	75.80	112.41	82.19	68.26	88.78	93.13	72.70
17	87.62	100.28	86.85	98.65	69.31	104.21	103.63	79.90	107.45
18	168.32	109.56	107.93	179.97	121.43	135.44	108.91	154.81	202.13
19	-	95.91	76.81	98.06	64.41	86.78	143.23	61.30	-11.70

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*Table 8.* PCDDs and PCDFs in blood (on volume basis/parts-per-trillion): mean, standard deviation (SD), median, and range

Congener		Pool		
		A	B	C
1	Mean	0.10	0.20	0.04
	SD	0.28	0.46	0.05
	Median	0.02	0.07	0.02
	Range	1.29	2.18	0.20
2	Mean	0.19	0.04	0.04
	SD	0.07	0.02	0.04
	Median	0.21	0.04	0.03
	Range	0.35	0.12	0.19
3	Mean	0.03	0.06	0.04
	SD	0.02	0.02	0.07
	Median	0.02	0.05	0.02
	Range	0.07	0.07	0.34
4	Mean	0.16	1.02	0.17
	SD	0.06	2.37	0.06
	Median	0.16	0.47	0.16
	Range	0.28	12.49	0.32
5	Mean	0.04	0.06	0.05
	SD	0.02	0.12	0.06
	Median	0.04	0.04	0.03
	Range	0.09	0.83	0.33
6	Mean	1.40	2.03	0.41
	SD	0.27	3.14	0.16
	Median	1.40	1.25	0.40
	Range	1.30	14.33	0.81
7	Mean	4.90	15.29	4.20
	SD	3.59	16.09	2.94
	Median	3.15	10.47	3.10
	Range	11.76	88.76	10.49

Table 8. Continued

Congener		Pool		
		A	B	C
8	Mean	-	-	0.02
	SD	-	-	0.02
	Median	-	-	0.01
	Range	-	-	0.10
9	Mean	-	-	0.01
	SD	-	-	0.02
	Median	-	-	0.00
	Range	-	-	0.12
10	Mean	-	-	0.24
	SD	-	-	0.39
	Median	-	-	0.12
	Range	-	-	1.58
11	Mean	0.04	0.04	0.05
	SD	0.04	0.02	0.07
	Median	0.03	0.03	0.03
	Range	0.26	0.10	0.32
12	Mean	0.03	0.06	0.04
	SD	0.01	0.02	0.07
	Median	0.03	0.05	0.02
	Range	0.08	0.10	0.33
13	Mean	0.01	0.01	0.03
	SD	0.02	0.01	0.08
	Median	0.00	0.00	0.01
	Range	0.09	0.03	0.33
14	Mean	0.03	0.01	0.03
	SD	0.01	0.01	0.08
	Median	0.03	0.01	0.01
	Range	0.06	0.05	0.33

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Table 8. Continued

Congener		Pool		
		A	B	C
15	Mean	0.20	0.11	0.11
	SD	0.07	0.05	0.04
	Median	0.20	0.10	0.10
	Range	0.32	0.28	0.24
16	Mean	0.01	0.01	0.02
	SD	0.02	0.01	0.05
	Median	0.01	0.01	0.01
	Range	0.06	0.07	0.28
17	Mean	0.06	0.06	0.08
	SD	0.06	0.06	0.12
	Median	0.03	0.05	0.03
	Range	0.22	0.27	0.46

Laboratory	Score
1	0.76666
6	0.91115
17	1.07310
8	1.22420
9	1.27615
5	1.34829
7	1.51898
14	1.68681
3	1.75112
18	1.80414
15	1.81668
19	1.82197
12	2.23064
10	2.45916
11	2.58119

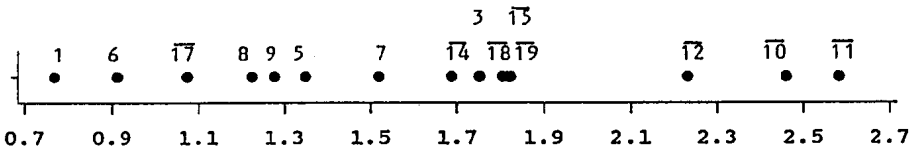


Figure 4. PCDDs and PCDFs in blood: score for reproducibility (with Nordic equivalent model)

Laboratory	Score
14	0.75812
8	0.89272
17	1.16606
1	1.18748
6	1.38727
3	1.48063
7	1.51635
9	1.57466
15	1.67168
18	1.73941
5	1.97166
19	1.99207
10	2.11145
12	2.33061
11	2.42088

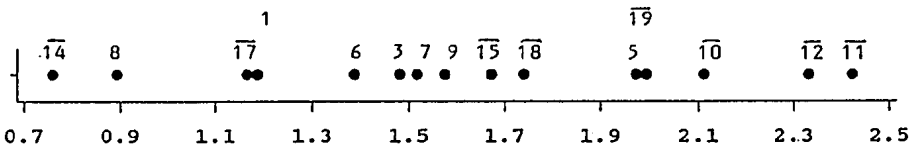


Figure 5. PCDDs and PCDFs in blood: score for repeatability (with Nordic equivalent model)

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Laboratory	Score
1	1.71664
8	1.93838
17	2.00595
6	2.02097
14	2.29331
9	2.53588
7	2.73206
5	2.92562
3	2.93562
15	3.15402
18	3.19567
19	3.41563
12	4.09513
10	4.14832
11	4.51790

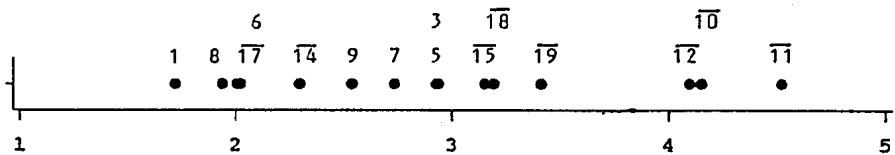


Figure 6. PCDDs and PCDFs in blood: final score (Nordic equivalent model) based on reproducibility and repeatability (weight factor 1.0 and 0.8)

Table 9. PCDDs and PCDFs in blood: median coefficient of variation (CV) for reproducibility

Laboratory	CV	Range
1	8.33	150.00
3	31.65	185.17
5	15.08	415.15
6	12.80	179.07
7	25.49	744.44
8	16.67	70.59
9	15.38	78.81
10	87.03	1514.35
11	114.58	6035.63
12	55.72	6066.13
14	21.39	158.33
15	21.21	163.33
17	12.96	758.33
18	48.98	1459.39
19	13.40	98.79

*Table 10.* PCDDs and PCDFs in blood: median coefficient of variation (CV) for repeatability

Laboratory	CV	Range
1	5.16	964.37
3	23.26	415.73
5	15.04	322.92
6	7.77	366.61
7	15.95	158.25
8	6.03	195.12
9	12.37	124.35
10	33.88	156.31
11	18.18	377.07
12	35.78	1028.39
14	7.87	2442.92
15	14.93	399.50
17	9.67	241.48
18	14.97	142.00
19	12.84	82.81

*Table 11.* PCDDs and PCDFs in blood: average coefficient of variation (CV) between reproducibility and repeatability

Laboratory	Mean CV
1	6.74
3	27.46
5	15.06
6	10.28
7	20.72
8	11.35
9	13.88
10	10.46
11	66.38
12	45.75
14	14.63
15	18.07
17	11.31
18	31.97
19	13.12

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Table 12. PCDDs and PCDFs in blood: average recovery (%) of spiked amount (Pool A)

Laboratory	Mean			
	Congener			
	2	6	14	15
1	88.38	82.49	111.90	66.45
3	87.86	82.21	70.83	76.43
5	88.89	102.17	90.48	82.17
6	88.03	81.08	99.52	53.50
7	94.10	61.06	85.71	60.19
8	93.50	85.30	-2.38	83.44
9	105.30	83.05	92.86	77.71
10	2.23	60.18	-17.42	-42.82
11	2.56	46.66	91.07	58.92
12	-	75.17	-1041.67	-20.17
14	67.91	77.30	74.29	69.28
15	138.80	104.73	66.67	118.90
17	89.06	78.55	78.45	59.02
18	101.37	122.72	111.90	113.80
19	114.70	95.16	114.29	91.72

Table 13. PCDDs and PCDFs in blood: average recovery (%) of spiked amount (Pool B)

Laboratory	Mean					
	Congener					
	1	3	4	6	7	12
1	77.27	73.19	82.10	107.87	97.72	82.86
3	73.13	62.39	87.04	84.26	74.19	81.90
5	67.17	-	-	105.65	74.35	99.05
6	93.94	82.61	100.89	141.20	87.75	125.71
7	49.24	115.22	116.32	96.25	33.98	55.71
8	81.82	30.43	89.02	107.87	86.55	86.67
9	83.84	-	-	104.17	50.46	100.00
10	-	92.39	2176.06	1881.83	837.76	-25.29
11	2415.66	-97.83	24.73	61.57	34.30	133.33
12	-151.52	-326.09	34.12	68.52	82.25	-761.90
14	59.95	71.74	82.45	38.97	70.50	114.48
15	91.92	78.26	171.12	143.52	84.12	86.67
17	67.68	50.72	94.96	115.28	131.63	58.10
18	242.42	111.96	111.87	177.73	178.63	157.14
19	-	111.59	112.76	131.94	76.18	95.24

Table 14. PCBs in milk (on volume basis/parts-per-trillion): mean, standard deviation (SD), median, and range

Congener	Mean	SD	Median	Range
1	377.60	113.12	369.00	440.00
2	75.44	181.13	30.00	930.00
3	154.11	189.13	75.00	708.00
4	3302.57	940.07	3113.00	4080.00
5	4061.21	1167.87	3865.00	5950.00
6	1728.93	567.43	1657.50	2550.00

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Laboratory	Score
5	0.37778
2	0.38519
8	0.39630
10	0.45926
7	0.46667
1	0.63889
4	0.67593
9	0.69630
3	0.78333
6	0.82407

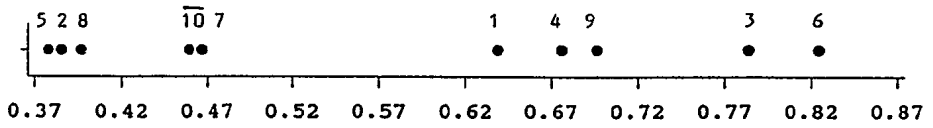


Figure 7. PCBs in milk: score for reproducibility

Laboratory	Score
5	0.40741
10	0.43056
8	0.48148
7	0.49074
9	0.53241
2	0.66898
3	0.68519
1	0.90509
4	0.95833
6	1.00000

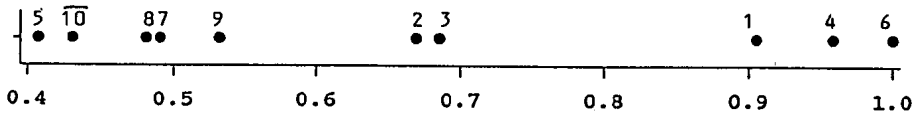


Figure 8. PCBs in milk: score for repeatability

Laboratory	Score
5	0.70370
8	0.78148
10	0.80370
7	0.85926
2	0.92037
9	1.12222
3	1.33148
1	1.36296
4	1.44259
6	1.62407

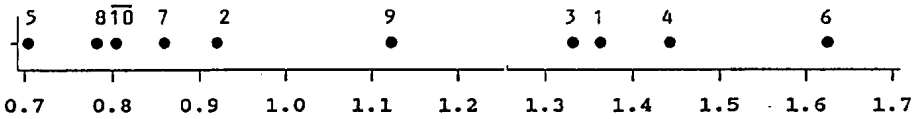


Figure 9. PCBs in milk: final score based on reproducibility and repeatability (weight factor 1.0 and 0.8)

Table 15. PCBs in milk: median coefficient of variation (CV) for reproducibility

Laboratory	CV	Range
1	36.63	54.91
2	12.58	63.32
3	39.95	432.20
4	31.97	666.00
5	21.79	38.46
6	46.68	3024.93
7	21.06	40.00
8	10.58	60.00
9	39.61	64.59
10	14.67	150.00

*Table 16.* PCBs in milk: median coefficient of variation (CV) for repeatability

Laboratory	CV	Range
1	24.33	31.88
2	9.30	11.33
3	3.17	4.00
4	34.29	30.21
5	3.08	16.50
6	-	-
7	0.71	24.74
8	3.33	6.24
9	6.66	9.56
10	1.31	13.91

*Table 17.* PCBs in milk: average coefficient of variation (CV) between reproducibility and repeatability

Laboratory	Mean CV
1	30.48
2	10.94
3	21.56
4	33.13
5	12.44
6	46.68
7	10.88
8	6.95
9	23.14
10	7.99

Table 18. PCBs in blood (on volume basis/parts-per-trillion): mean, standard deviation (SD), median, and range

Congener	Mean	SD	Median	Range
1	46.43	56.04	25.00	183.00
2	13.60	11.72	10.50	37.00
3	42.55	64.93	12.40	178.00
4	942.33	450.55	819.00	1628.00
5	1366.00	716.01	1135.00	2590.00
6	593.27	126.63	630.00	407.00

Laboratory	Score
5	0.28889
10	0.47222
2	0.48889
8	0.67222
3	0.94444
4	1.00000

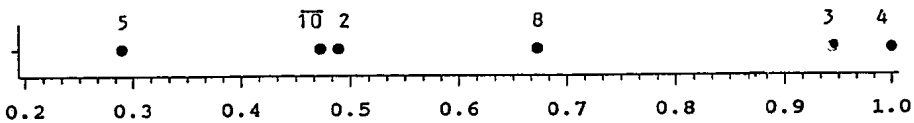


Figure 10. PCBs in blood: score for reproducibility

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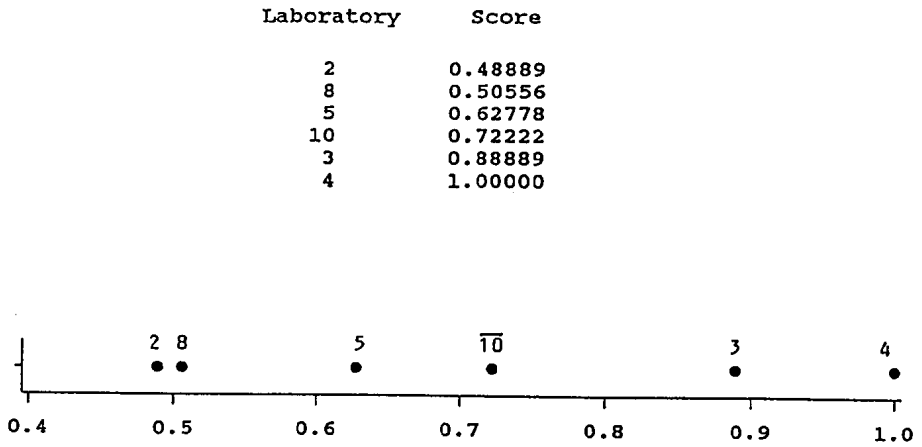


Figure 11. PCBs in blood: score for repeatability

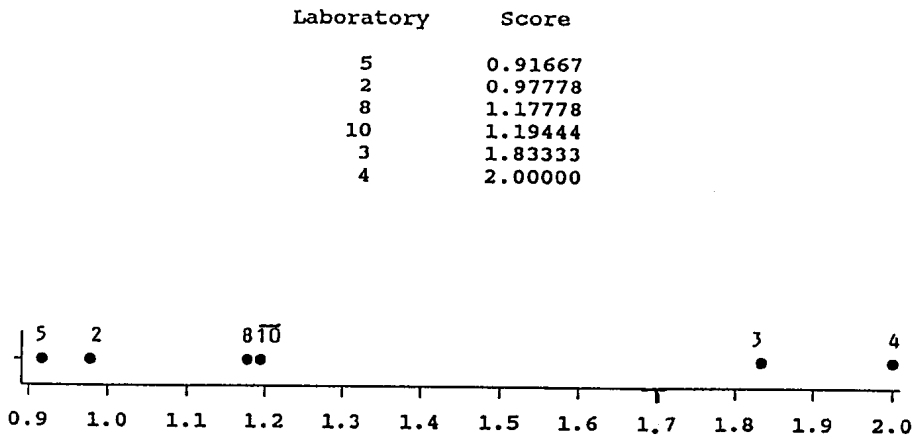


Figure 12. PCBs in blood: final score based on reproducibility and repeatability (weight factor 1.0 and 0.8)

*Table 19.* PCBs in blood: median coefficient of variation (CV) for reproducibility

Laboratory	CV	Range
2	11.70	19.05
3	36.30	3.03
4	333.33	1111.60
5	2.99	58.00
8	34.92	77.80
10	5.13	73.33

*Table 20.* PCBs in blood: median coefficient of variation (CV) for repeatability

Laboratory	CV	Range
2	0.00	6.45
3	10.87	8.44
4	14.35	11.00
5	3.58	119.19
8	0.96	3.25
10	6.48	9.30

*Table 21.* PCBs in blood: average coefficient of variation (CV) between reproducibility and repeatability

Laboratory	Mean CV
2	5.85
3	23.58
4	123.84
5	3.29
8	17.94
10	5.80

## ANNEX 2

## List of qualified laboratories for analysis of PCDDs and PCDFs in milk

(showing responsible persons - listed in alphabetical order of countries)

Dr J.J. Ryan	Telephone: 613-9570978
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*64 Levels of PCBs, PCDDs and PCDFs in human milk and blood*

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## ANNEX 3

## List of qualified laboratories for analysis of PCDDs and PCDFs in blood

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*66 Levels of PCBs, PCDDs and PCDFs in human milk and blood*

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## ANNEX 4

## List of qualified laboratories for analysis of PCBs in milk

(showing responsible persons - listed in alphabetical order of countries)

- |  |  |
|--|--|
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## ANNEX 5

**List of qualified laboratories for analysis of  
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- |  |  |
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