

ANALYSIS OF POLYCHLORODIBENZO-DIOXINS (PCDDs) AND POLYCHLORODIBENZO-FURANS (PCDFs) IN DAIRY PRODUCTS IN FRANCE

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ABSTRACT

52 samples collected in different departments in France through the services of the Ministry of Agriculture, Fisheries and Food have been analysed for PCDDs and PCDFs :

- 12 milk samples,
- 8 butter samples,
- 20 cheese samples,
- 10 milk dessert samples,¹
- 2 cream samples.

The average I-TEQ content on milk fat basis amounts to 1.33 pg I-TEQ/g fat which is lower than the standard of the Netherlands (6 pg I-TEQ/g fat) and of the Germany reference value (5 pg I-TEQ/g fat).

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INTRODUCTION

In 1996, the French Ministry of Agriculture, Fisheries and Food decided to pursue the survey launched in 1994¹⁾ aimed at assessing the PCDDs and PCDFs content of the cow milk in France. In addition to cow milk samples, the new survey focused on milk based products such as butter, cheese, cream and milky desert samples. This survey is one step ahead of an assessment of the PCDDs/PCDFs daily intake in France.

EXPERIMENTAL METHOD : PROCEDURE FOR SAMPLING AND ANALYSIS

Sampling procedure

The sampling was performed by the local services of the Ministry of Agriculture, Fisheries and Food in several departments.

- Dairy products were selected to get samples from every French regions :
 - ⇒ For cheese samples, 20 departments were selected in regards with several different cheese types.
 - ⇒ For milk based desert and cream, 12 departments were selected.
 - ⇒ For butter, 8 departments were selected.
- Milk samples were selected from 2 departments through 12 pooled samples in order to get an average of PCDDs and PCDFs content in milk in these departments, whereas the previous survey performed in 1994-1995 focused on sampling in the area of industrial sites of these departments.

These milk and dairy products were sampled in the last quarter of 1996.

The samples have been frozen and transported to the analytical laboratory where the samples were stored at - 20° C until analysis.

Analytical procedure

Analyses of samples have been carried out according to the method described by Liem et Al.²⁾ Shortly, samples were fortified with known amounts of sixteen ¹³C₁₂-labelled PCDD and PCDF standards. The fat fraction was then extracted by the appropriate method using organic solvents (diethyl ether, petroleum ether...).

Fat content was dried and weighed.

LEVELS IN FOOD

The clean-up of the fat fraction, redissolved in dichloromethane was performed by carbosphere active carbon column chromatography followed by alumina column chromatography. The final purified extract was concentrated in 25 μl of dodecane containing two internal standards ($^{13}\text{C}_{12}$ -1,2,3,4-TCDD and $^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD). A volume of 1.5 μl was injected for HRGC-HRMS quantification. The resolution of the mass spectrometer (Ultima-Micromass) was set at 10,000 resolution.

RESULTS : SUMMARY OF PCDDs AND PCDFs CONTENT OF THE 52 SAMPLES

| Type of sample | Origin | Number of samples | Σ I-TEQ Total pg/g fat (mean) |
|-----------------------|----------------|-------------------|--|
| Cow milk | 2 departments | 12 | 1.91 |
| Cheese | 20 departments | 20 | 1.11 |
| Butter | 8 departments | 8 | 1.01 |
| Milk desert and cream | 12 departments | 12 | 1.34 |

LITERATURE CITED

¹ Fraisse, B. Schnepf, C. Mort-Bontemps, F. Le Querrec (1996)

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Levels of polychlorodibenzo-dioxins (PCDDs) and polychlorodibenzo-furans (PCDFs) in milk in France,

² Litterature : Liem A.K.D., De Jong A.P.J.M., Marsuman J.A., De Boer A.C., Groenemeijer G.S., Van der Heeft E., De Korte G.A.L., Hoogerbrug R., Den Hartag R.S., Kootstra P.R., Van't Klooster H.A., Chemosphere, 20, 7-9, 843-850, 1990a