

METHOD FOR THE DETERMINATION OF DIOXINS, PLANAR AND OTHER PCB'S IN HUMAN MILK

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INTRODUCTION

Seven institutes in the Netherlands: Academisch Ziekenhuis (Groningen); Landbouwwuniversiteit (Wageningen); MBL-TNO (Rijswijk); IBC-TNO (Zeist); Sophia Kinderziekenhuis (Rotterdam); ITV-TNO (Zeist) and RIKILT-DLO (Wageningen) are working on a project: "Long term effects of early (foetal/neonatal) exposure to toxic substances (polychlorinated biphenyls (PCB's) and dibenzo-p-dioxins (PCDD's) and dibenzofurans (PCDF's))". The project is subsidised by "Stimuleringsprogramma Gezondheidsonderzoek" (SGO) and "Programma Commissie Toxicologisch Onderzoek" (PCT).

In the frame work of this project RIKILT-DLO determines the contents of these compounds in human milk.

Now we report on a streamlined, integrated method of analysis (see Fig. 1) in which depending on the goal, only normal PCB's with GC-ECD can be determined after GPC and alumina clean-up or dioxins, planar PCB's on the one hand and normal PCB's on the other hand after GPC and alumina clean-up and fractionation on porous graphitised carbon for the separation in a group containing normal PCB's for the determination with GC-ECD and a group containing dioxin and planar PCB's for the determination with GC-HRMS. In the different steps highly automated procedures were used.

The method used in this study is a combination of methods (1,2,3).

EXPERIMENTAL

Fig. 1 shows schematically the analytical procedure. The sample is spiked with ^{13}C labelled compounds and two PCB congeners (# 3 and # 198) (4). Up to 3 g of isolated fat (5) is cleaned on a 60 x 2.5 cm ID GPC column filled with BioBeads SX3 and eluted with ethyl acetate/cyclohexane (1:1) at a flow of 5 ml/min.

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The concentrated GPC fraction (containing 50 μ l dodecane) of 1 ml was applied to a 3 ml disposable SPE extraction column containing 1 g deactivated (7% H₂O) basic alumina and eluted with 3 ml hexane.

In the eluate immediately a determination of non planar PCB's (including mono ortho PCB's) can be carried out with GC-ECD (A route). However in case that also dioxin and planar PCB's have to be determined (B route) the eluate is concentrated to 2 ml and a separation on porous graphized carbon is carried out using forward and backward flush resulting in two fractions. One contains the non planar PCB's and can be analysed with GC-ECD, the second fraction is analysed with GC HRMS at 10.000 mass resolution.

RESULTS

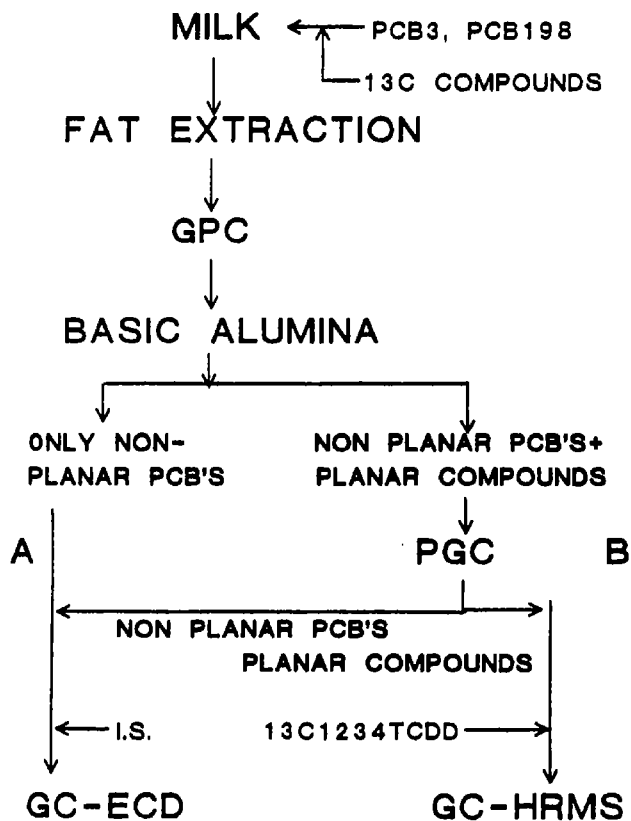
During the analysis of human milk samples all kind of checks were performed to control the reliability of the procedure.

Attention has been paid to blanc chemicals, homemade blanc fats; recoveries of spiked chemicals and samples, reproducibility of control samples, comparison of results obtained in the same sample after analysis according to route A and B. Results will be presented at the symposium.

LITERATURE

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Fig. 1



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