Selective Separation of 2,3,7,8-substituted Tetra- to HexaCDD/CDF from other PCDD/PCDF by Fractionation on Alumina B Super 1 for Dioxin Analysis

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1. Introduction

In 1986 we described the selective separation of 2,3,7,8-TCDD from all other PCDD/PCDF by fractionation on Alumina B Super I produced by Woelm, Eschwege (now ICN Biomedicals) [1]. In 1987 Swerev and Ballschmiter [2] reported that under the same conditions not only 2,3,7,8-TCDD but also 2,3,7,8-TCDF could be separated from other PCDD/PCDF. When we checked the report by Swerev and Ballschmiter, we found that some lots of Alumina B Super I would elicit this property, but in general 2,3,7,8-TCDF was lost during the described fractionation procedure. In 1991 ICN Biomedicals improved their production process for the highly active Alumina B Super I and labeled the resulting product "Alumina B Super I for Dioxin Analysis". When we tested this product for the selective separation of 2,3,7,8-TCDD under the conditions described in [1], we found that this material could not only separate 2,3,7,8-TCDD and 2,3,7,8-TCDF from other PCDD/PCDF but under slightly different conditions also 2,3,7,8-substituted penta- and hexaCDD/CDF from most of the other PCDD/PCDF.

2. Experimental

The sample is first processed by the usual clean-up procedures for PCDD and PCDF using ^{13}C-labeled standards for all 2,3,7,8-substituted PCDD/PCDF as internal standards. The column used for the fractionation consists of 2.5 g of Alumina B Super I and 2 g of Na_2SO_4, prewashed with 20 ml heptane.

Selective separation of 2,3,7,8-TCDD and 2,3,7,8-TCDF

The sample is applied to the column and eluted first with 30 ml of heptane/dichloromethane (8:2). This fractions contains all PCDDs and PCDFs except 2,3,7,8-TCDD and 2,3,7,8-TCDF, which are subsequently eluted with 40 ml of heptane/dichloromethane (1:1).

Selective separation of 2,3,7,8-substituted tetra- to hexaCDD/CDF

The sample is applied to the column and eluted first with 40 ml of heptane/dichloromethane (87.5:12.5). Then the column is eluted with 40 ml of heptane/dichloromethane (1:1). This fraction contains all 2,3,7,8-substituted tetra- to hexaCDD/CDF and small number of other congeners.

3. Results

The result of such a fractionation of tetra- to hexaCDD/CDF and the percent recovery for the various congeners is shown in Figure 1. The percent recovery of the various congeners is somewhat dependent on the lot of Alumina B Super I. The separation of 2,3,7,8-TCDD and 2,3,7,8-TCDF can be used in cases were separation of these congeners from other isomers is insufficient and the separation of the 2,3,7,8-substituted tetra- to hexaCDD/CDF can be applied to great advantage for the unequivocal identification and quantification of 2,3,7,8-substituted PCDD/PCDF in difficult to analyze samples, especially in those, were non-2,3,7,8-substituted congeners are present in a large excess like in some industrial samples.

4. References

[1] Hagenmaier H., Brunner H., Haag R., Kraft M., (1986) Fresenius Z. Anal. Chem 323: 24-28.
[2] Swerev M., Ballschmiter K., (1987) Anal. Chem. 59: 2536-2538.

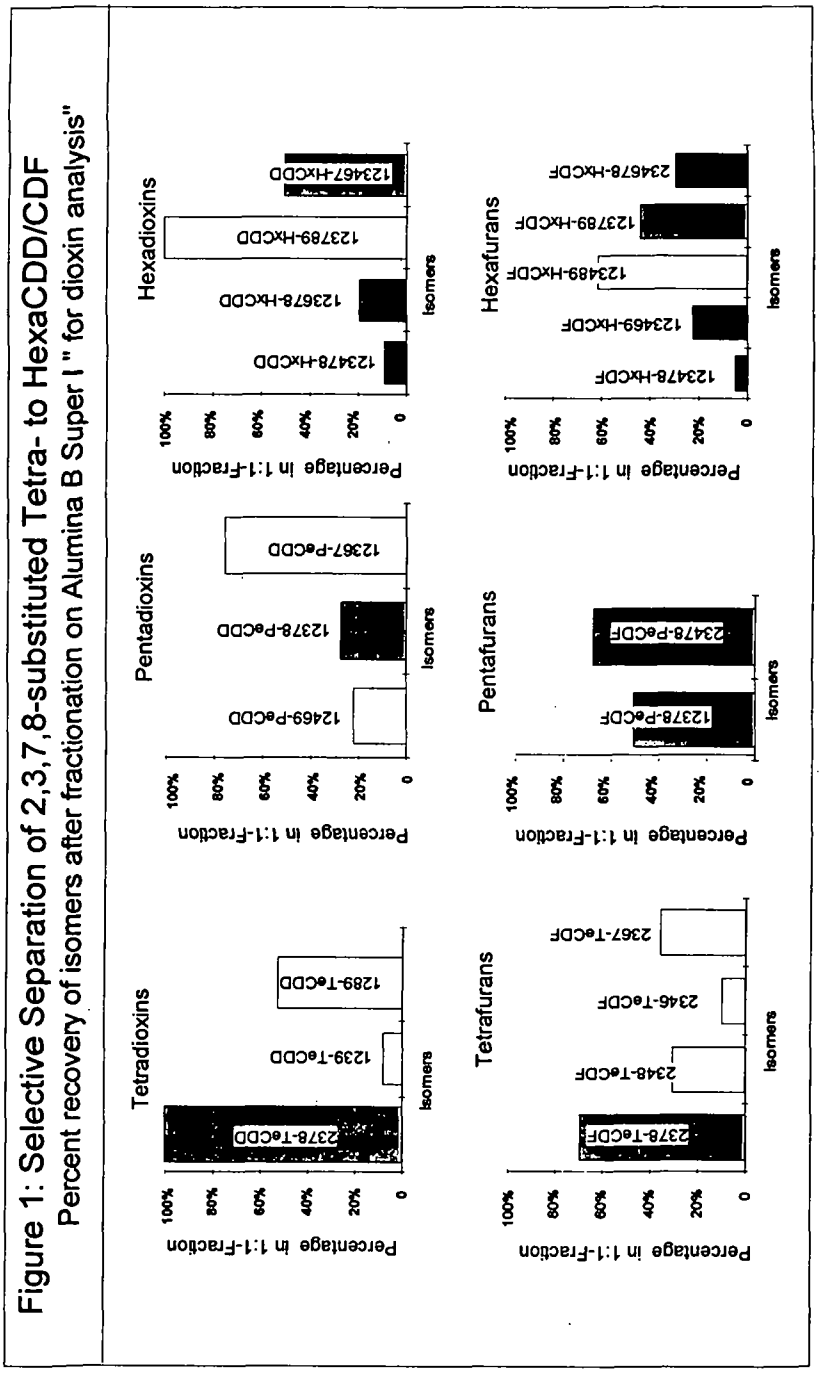

Figure 1: Selective Separation of 2,3,7,8-substituted Tetra- to HexaCDD/CDF
Percent recovery of isomers after fractionation on Alumina B Super I " for dioxin analysis"