Determination of TEQ in Chimney Soot by PCDD/PCDF Enzymeimmunoassay– a Comparison with GC/MS data

M. Zennegg and P. Schmid

Swiss Federal Laboratories for Materials Testing and Research, Überlandstrasse 129, CH-8600 Dübendorf, Switzerland

Introduction

Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in residues from incineration processes such as waste incineration or house heating are always present as mixtures of isomers and homologues. The toxicological assessment of the exposure to such emissions is based on the 2,3,7,8-TCDD toxicity equivalents (TEQ). Therefore, calculation of these values requires the analytical determination of all 2,3,7,8-chlorosubstituted PCDDs and PCDFs, which is time consuming and expensive if conventional methods, are used. Rapid methods such as enzyme immunoassays (EIA) could be helpful for the screening of large numbers of samples. Depending on the material used as fuel chimney soot can contain high amounts of PCDD/PCDF and therefore cause a significant exposure for the chimney sweepers [1]. For the fast monitoring of the workplace conditions of chimneysweepers EIA may be a suitable and helpful tool.

In previous work on fly ash from waste incinerators a good correlation of the EIA response with TEQ determined by GC/MS [2, 3] was demonstrated. In the present study, 10 chimney soot samples from house heating systems fed with different fuels were analyzed by EIA after a rapid oxidation step performed on the crude toluene extracts and simple chromatographic purification. In parallel, TEQ values were determined using the classical GC/MS method with samples subjected to the full clean-up procedure. The reliability of the EIA method was evaluated by comparing the two data sets.

Materials and Methods

The clean-up of the chimney soot for GC/MS was based on an in-house routine method [5]. Calculation of TEQ values was based on I-TEFs [5].

Sample preparation for the EIA (High Performance Dioxin/Furan Immunoassay Kit DF-1 60 from Cape Technologies, 3 Adams Street, South Portland ME 04106, USA) included 3 h acid leaching of the soot with 1 M hydrochloric acid followed by Soxhlet extraction with toluene. An aliquot of this extract was reduced in volume and treated with concentrated sulphuric acid containing ca. 7 % (w/w) sulphur trioxide. The mixture was extracted with n-hexane and the extract was further purified by active carbon chromatography (PX-21). The sample was redissolved in methanol containing 0.1 ‰ (w/w) Triton X-100 and an aliquot of 10 μ L was used for the EIA.

ORGANOHALOGEN COMPOUNDS 317 Vol.40 (1999)

Results and Discussion

Figure 1 shows the EIA response values plotted against the TEQ values as calculated from single congener concentrations measured by GC/MS. The Figure shows that the sensitivity of the EIA covers the whole concentration range of the 10 samples. The specified sensitivity of the EIA used in this work was 14 pg 2,3,7,8-TCDD/tube (50 % inhibition) and the detection limit was 3 pg 2,3,7,8-TCDD/tube (85 % inhibition). The discrepancies between EIA and GC/MS data can be attributed the fact that the cross reactivities of the 2,3,7,8-chlorosubstituted PCDDs and PCDFs in the EIA and the I-TEFs are slightly different [6].

The comparison confirms that this EIA is sufficiently reliable for the determination of TEQ in chimney soot. As the EIA does not require any time consuming steps in the sample preparation (e.g. chromatographic methods) it is suitable as a simple and rapid screening method for large sample numbers in order to select critical samples for detailed investigation by GC/MS analysis.

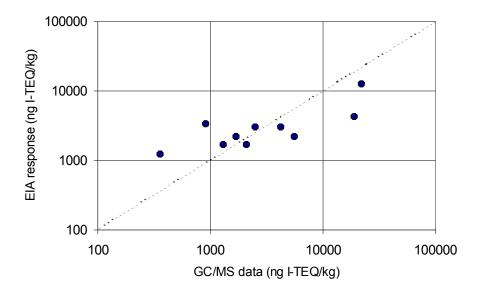


Figure 1: Comparison of TEQ in chimney soot determined by EIA and calculated by application of I-TEFs [5] to single PCDD and PCDF congener concentrations from GC/MS analysis (bisector: EIA response = TEQ calculated from GC/MS data).

ORGANOHALOGEN COMPOUNDS 318 Vol.40 (1999)

References

- 1. Thoma H., Dumler-Gradl R., Beyer B., and Wrbitzky R., Organohalogen Compounds **1998**, 38, 25-28.
- 2. Harrison R.O. and Carlson R.E., Organohalogen Compounds 1998, 35, 43-46.
- 3. Zennegg M., Harrison R.O., and Schmid P., Organohalogen Compounds 1998, 35, 213-215.
- 4. Wunderli S., Zennegg M., Dolezal I.S., Noger D., and Hasler Ph., *Organohalogen Compounds* **1996**, 27, 231-236.
- 5. Kutz F.W., Barnes D.G., Bottimore D.P., Greim H., and Bretthauer E.W., Chemosphere **1990**, 20, 751-757.
- 6. Harrison R.O. and Carlson R.E., Organohalogen Compounds 1998, 35, 97-100.

ORGANOHALOGEN COMPOUNDS 320 Vol.40 (1999)