ABSTRACT

Low p.p.q Level PCDDs and Hexachlorobenzene Determination in Water using Low Resolution MS and CI in Negative Ion mode

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The analysis of publications has shown that in most cases the detection limits of PCDDs and related compounds determination is not less then 1 - 0,1 p.p.t. It greatly depends on sample preparation method , sample volume and detection limit of the MS being used. The best results have been obtained in EI mode HRMS but much more expensive then LRMS.

The aim of our research was to get lower detection limits then commonly known using LRMS. For these purposes we have made more detailed research of detection limit of LRMS using CI in negative ion mode and as reagent gas Argon-methane mixture (95:5). We have used in this research as mass selective detector a LRMS manufactured by Fisons ("TRIO-1000" model).

This LRMS contrary to other ones uses Fotomultiplier as ion detector instead of secondary electron multiplier. Thanks to it and to quadrupole lens used at the inlet of quadrupole mass analyzer this instrument has according to the manafactorer detection limit then other. Our investigations has shown that in negative ion CI mode we were able to detect as low as 2 fg of hexachlorobenzene (used as model compound) and of 2,3,7,8-TCDD. Signal to noise ratio in this case was about 7:1 and sample size 1 μl (on column).

For determination of minimum detectable concentrations (without preconcentration) we analyzed different volumes of hexane solutions containing analytes. Using 20 m long empty precolumn (i.d. 0,53 mm) and T-piece press-fit capillary connector between it and separating column we were able to analyse as

large as 10, 100 and 200 µl samples. It has been shown, that reproducibility of injection of such analyte quantity is rather high using different sample volumes and that detection limits of about 10 p.p.q. were achieved in our experiments without preconcentration.

We studied the use micro liquid extraction from water samples too. For these research we used special J & U Scientific flasks (40 ml volume), which allowed more accurately to collect small volumes of organic extract. The volume of the last one was about 0,2 ml. Efficiency of extraction (measured for hexachlorobenzene) was about 40% (but these results are preliminary - we intend to make them better). All extract were analysed using our GC/MS with precolumn. The minimum detectable concentration in this case was found to be about 0,1 p.p.q.