DETERMINATION OF PCDD AND PCB IN BAIKAL WATER BY USING NEGATIVE CHEMICAL IONIZATION WITHOUT SPECIAL SAMPLE PREPARATION.

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It is well known that determination of PCDD and PCB requires special and complex sample preparation .Meanwhile despite the complex and long-time process of sample preparation the sensitivity of MS by using EI mode is not sufficient to determine PCDD and PCB on background level.

The authors have developed the method of negative chemical ionization (NCI)[1] which have two distinctive peculiarities:the using of argon/methane(90:10)mixture as a reagent gas and high temperature of the ion source(up to 325°C).

EXPERIMENTAL

A quadrupole Hewlett-Packard mass-spectrometer "HP 5988" with gas chromatograph HP"5890" was used in this work.

Two capillary silica columns were used:Ultra 2 (25m, ID 0.32mm,temperature programmed 50-270°C,25°/min) and HP-1 (50m,ID 0,32mm,temperature programmed 50-250°C,15°/min and to 270°C at 30°/ min).

A temperature of injector was 270°C. The sample was injected in a split/splitless mode.The flowrate of reagent gas (He) was 1ml/min, Sample volume -2.The source temperature was 325°C; the ion sour e pressure was 0.75 torr.

Selected ions monitoring (SIM) mode was used.

From 1 to 6 litres of water from Baikal,some Baikal region rivers,and waste water of woodpulp and paper industries were extracted by 200 ml hexane and after that extract was evaporated to 2-4 ml.For GC/MS analysis 2 mkl of extact was used.

Besides the water examination,the samples of fish and plankton was collected and investigated.The initial purpose of this experiment was PCB determination and the sample preparation only for PCB [2]was realized.Therefore the preparated samples contended both PCB and PCDD fractions and determination of PCDD was possible only after elu tion PCB from the column.And so determination of more chlorinated dioxins (from penta-CDD to Octa-CDD) was possible.

RESULTS

1,2,3,4,7,8-hexa-CDD was founded both in fish tissue (1-2 pg/g) and in waste water (7.5 pg/l).This substance was detected also in the cellulose fiber found on the lake beach.

The PCB concentrations in winter 1992 in waters of south Baikal varied from 0.04 (0.5 km from Goloustnaya river) to 0.10 mkg/l (0.2 km from paper factory).

Maximum concentration of PCB in tributary rivers was detected in Utulic (0.12 mkg/l) and Selenga (0.13 mkg/l),minimum in Goloustnaya (0.02 mkg/l).

In snow cover of Baikal in march 1992 PCB concentration varied from 0.05 (Tompa river) to 0.38 mkg/l (10 km from Goryachie Kluchi).

In march 1992 in waste water of paper fabric 0.28 mkg/l PCB was found and tetra-PCB were prevailed.

In spring 1992 in phytoplankton up to 0.80 mkg/g PCB was detected,in zooplankton - up to 1.15 mkg/g.

In fish tissues PCB concentration was found from 0.003 to 0.1 mkg/g.

REFERENCES

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