A simple pretreatment technique for PCB analysis

A key to solving the PCB-waste issue: fast and accurate analysis

One of the persistent organic pollutants, polychlorinated biphenyl (PCB) was once widely used as dielectric fluids and other applications. Fast, simple and accurate determination of PCB concentration is essential to properly evaluate the risks of vast numbers of PCB-wastes.

We synthesized a sulfoxide residue and ammonium ion-bonded silica stationary phase for liquid chromatography, and investigated its ability to separate PCB from mineral oil matrices. After separation with the stationary phase, major PCB congeners in insulating oil samples containing Japanese legal regulation level of PCB (0.5 mg total PCB/kg) were determined with a gas chromatograph/quadrupole mass spectrometry system. The established method is much faster and requires less hazardous chemicals compared with the Japanese official method for PCB analysis. Because of the effectiveness, the stationary phase has been commercialized by a private company.

The potentials of the stationary phase for separation of other pollutants, such as polyaromatic hydrocarbons, dioxins, brominated frame retardants, and heavy metals are shown by other laboratories.

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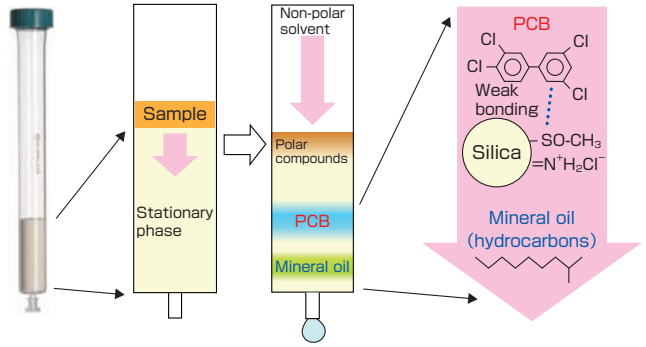

The novel stationary phase, and principle of separation

Beyond the limit of mass spectrometry

Kinetic-energy-sensitive mass spectrometry for separation of different ions with the same *m/z* value

The double-focusing mass spectrometer equipped with a superconducting-tunnel-junction (STJ) detector has been applied to measure relative ionization cross-sections for production of ions that are accompanied by different ionic species with the same mass-to-charge ratio (*m/z*). The STJ detector fabricated for this study enables kinetic energy (*E*) measurement of incoming individual ions at an energy resolution (*ΔE/E*) of 15 % with the assistance of an infrared-blocking filter which prevents detector-performance degradation due to infrared radiation illuminating the detector surface at 0.3 K. The high energy resolution is necessary to independently determine both *m* and *z* values. One of the limits of conventional mass spectrometry is that it measures the *m/z* values and thus different ions with the same *m/z* cannot be analyzed. The unconventional discrimination capability allows direct determination of relative ionization cross-section of the homonuclear diatomic ions $^{14}N_2^{2+}/^{14}N_2^{+}$ and $^{16}O_2^{2+}/^{16}O_2^{+}$, which are difficult to measure due to the strong interference by the signals of their dissociated atomic ions $^{14}N^{+}$ and $^{16}O^{+}$ with noticeably large ionization cross-sections. A kinetic-energy-sensitive mass spectrometer is useful for a wide range of analytical chemistry such as ionization processes of multiply ionized molecules and fragmentation of immunoglobulins.

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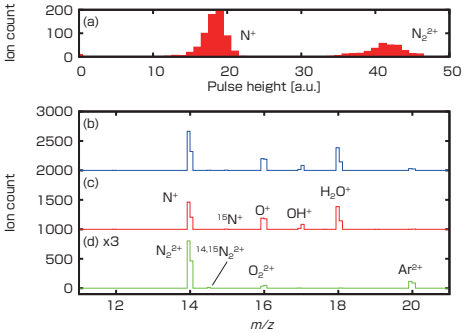

Mass spectra of the air ionized with electron impact at 70 eV
The superconducting tunnel junction detector enables us to distinguish different charge states and re-construct corresponding mass spectra; (a) pulse height spectrum for the ions with *m/z* of 14, (b) mass spectrum for all ions, (c) singly charged ions, (d) doubly charged ions.