Odourless – yet dange

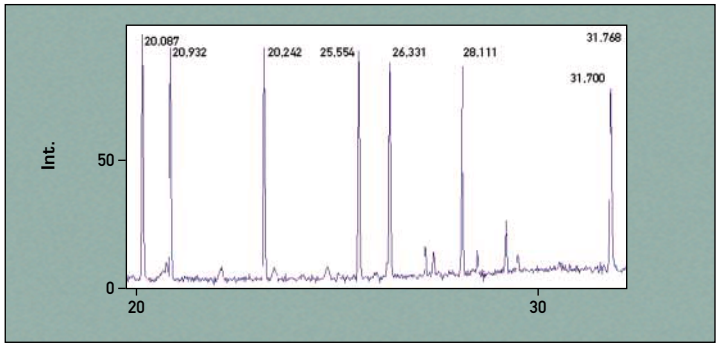

Figure 1: PCB standard analysed on a standard column (30 m x 0.25 mm x 0.25 µm)

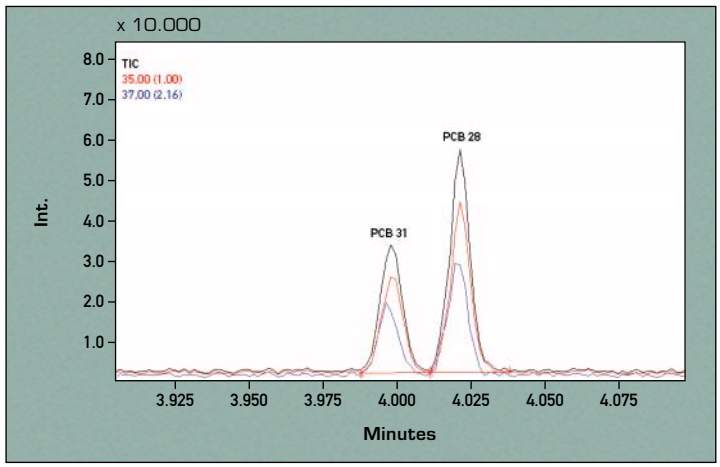

Figure 3: The separation of PCB congeners 28 and 31 on the HT-8 column

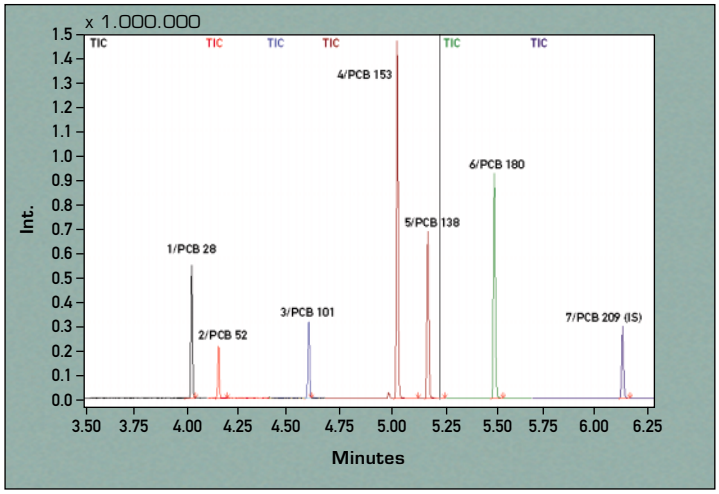

Figure 2: PCB standard analysed using negative chemical ionisation (NCI) in the SIM mode on a fast-capillary column

Polychlorinated biphenyls (PCBs) have been synthesised industrially since 1929. PCBs are mixtures of aromatic hydrocarbons that possess the same basic biphenyl structure with different degrees of chlorination.

A total of 209 so-called congeners exist today. PCBs have been extensively used in the past as insulators, flame-retardants and plasticizers. Due to their problematic persistence in the environment resulting their chemical stability, accumulation in the food chain and their toxicity to the liver, kidneys and spleen arising from continuous exposure, the use and the distribution of PCBs has been prohibited in Germany since 1989. The use of PCBs in so-called open systems has been banned since 1978.

PCBs have been released into the environment via water and the atmosphere. Human uptake of PCBs is primarily through foods of animal origin. PCBs, in particular those with a higher chlorine content, are concentrated in animal fats and can reach humans through the food chain. Continuous exposure via the atmosphere, however, also results in measurable toxicity for the body. In the mid-eighties attention was drawn to the presence of PCBs in buildings. The sources were attributed to leaking capacitors (for instance in fluorescence light tubes), sealing compounds (based on polysulphide rubber) as well as to flame retarding or sound-proofing paints for acoustic ceilings.

Testing for the presence of PCBs in indoor air in public buildings

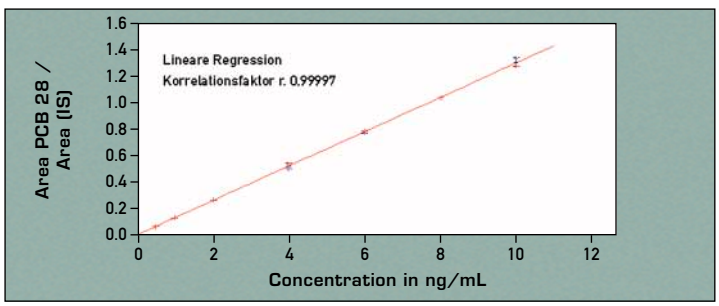

Figure 4: Calibration for the PCB 28 congener of 0.5 – 10 ng/mL

erous PCB in indoor air – Fast and selective analysis using GCMS

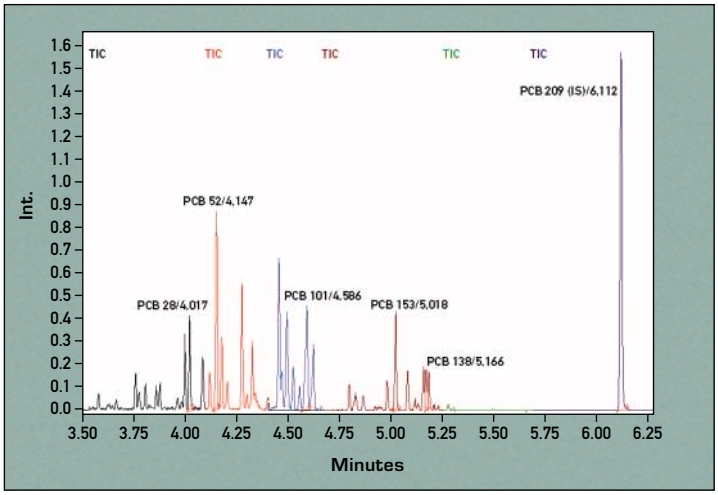

Figure 5: Real sample detected with NCI in the SIM mode on a fast-capillary

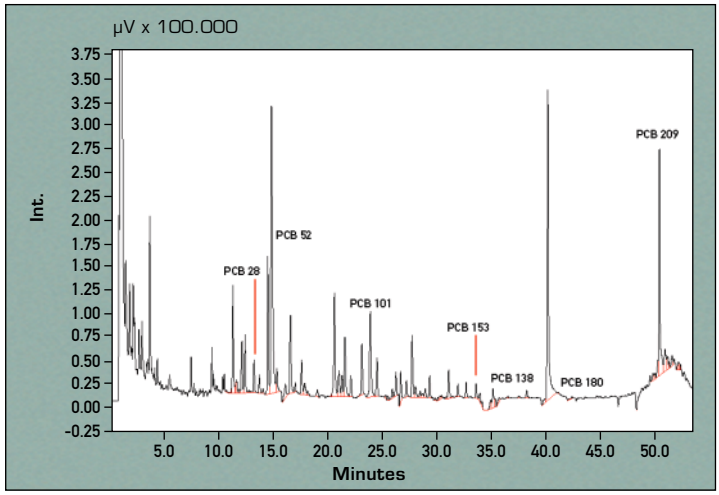

Figure 6: Real sample determined with GC-ECD

such as schools, kindergardens and administrative offices has been an important issue for many years. When suspected polluted buildings, especially those built in the 1960s or 1970s were tested for PCBs, occasionally extremely high PCB levels were measured. A PCB concentration of less than 300 ng/m³ in indoor air can be considered as decontamination goal and as long-term tolerance level.

A GCMS method for the analysis of PCBs in indoor air that is particularly suitable for high sample throughput, is presented below. This method was developed at the Central Laboratory of Analytis (Gesellschaft für Laboruntersuchungen GmbH in Wesseling, Germany) and will be used there for continuous routine analysis.

Six different PCBs that enable an estimation of the pollution level, are determined according to the PCB guidelines. The PCBs concerned are congeners 28, 52, 101, 138, 153 and 180. In the method described here, PCB congener 209 is used as internal standard.

After sampling onto polyurethane-foam and Soxhlett extraction over several hours, the sample is analysed using a Shimadzu QP2010 mass spectrometer in NCI mode. Negative chemical ionisation (NCI) is a selective analysis method for compounds that, based on their structure, are able to capture electrons.

In order to enable high sample throughput, a fast capillary column was used for the separation of the PCB congeners. This enables a drastic reduction in analysis time by a factor of five and more, compared to separation on a standard capillary column (30 m x 0.25 mm x 0.25 µm; analysis time approximately 30 – 50 minutes). The analysis of the standard solution shown in Figure 1 takes approximately 30 minutes. When the separation is carried out on a fast-capillary column instead (SGE HT-8, 10 m x 0.1 mm x 0.1 µm), the last peak – the internal standard (PCB 209) – will already elute after 6.1 minutes (Figure 2). In spite of the distinctly short analysis time, the separation of both critical PCBs 31 and 28 will proceed without any problems, as shown in Figure 3.

All six PCBs exhibit excellent linearity over a concentration range of 0.5 – 10 ng/mL. As an example, the calibration curve of the PCB congener 28 is shown in Figure 4. The analytical limiting values were determined according to DIN 32645 and a detection limit of 0.09 ng/mL and a determination limit of 0.3 ng/mL was found for the PCB congener. The detection- and determination limits for the other components are listed in Table 1.

Figure 5 and 6 show the chromatograms of a real sample (GCMS/NCI; SGE HT-8, 10 m x 0.1 mm x 0.1 µm, respectively and GC-ECD; SGE HT-8, 30 m x 0.25 mm x 0.25 µm).

For comparative measurements of the positive results no significant differences were found between the values obtained via ECD (2 columns of different polarity) and GCMS/NCI detection. The data shown clarify the suitability of fast NCI-GCMS for the determination of PCBs in indoor air with high sensitivity and excellent precision. In addition, the application presented here is another impressive example of how the use of fast chromatography can save valuable time in routine analysis.

[1] PCB Guideline NRW, guideline for the assessment and decontamination of PCB polluted construction materials and components in buildings.

Acknowledgement

Shimadzu thanks Mr. Michael Kolloch of Analytis for his help and cooperation as well as for providing the data.

	Detection limit [ng/mL]	Determination limit [ng/mL]
PCB 28	0.09	0.3
PCB 52	0.3	0.98
PCB 101	0.4	1.2
PCB 138	0.3	1.2
PCB 153	0.2	0.5
PCB 180	0.2	0.5

Table 1: Detection and determination limits for the six PCB congeners according to the PCB guidelines