

A Novel Ion Detection System for a Magnetic Sector Mass Spectrometer

Rhys Jones¹, Robert Bateman¹

¹Waters MS Technologies Centre, Manchester

Introduction

Double focusing magnetic sector mass spectrometers are most often used for applications where high resolution and sensitivity are the primary requirements. The high resolution enables many chemical background masses to be eliminated and consequently allows a lower detection level to be achieved. The highest sensitivity can be achieved by performing selected ion recording as this provides a better duty cycle than scanning.

High resolution alone does not insure that all ions detected are of the target compound of interest. Background ions may be detected which are due to contaminants in the source or reference compound. Scattered ions from high abundance ion beams in close proximity to the selected mass may also be detected and which give rise to a ubiquitous background. Interference ions may be detected which are due to isobaric contaminants. If an interference compound is present in the sample, different in mass from the analyte by less than the current system resolution, then this can lead to false detections. This situation is typically remedied by increasing the system resolution, with a corresponding reduction in sensitivity.

A new detection system has been designed and constructed with the intention of automatically discriminating against such interference ions and background ions. Ions which have been detected but which are not ions of the target compound of interest may be suppressed or even discarded.

Methods and Materials

Instrumentation

The mass spectrometer used to develop the new detection system is an AutoSpec Ultima (Waters, Manchester, UK). The new detection system is mounted on the rear of the instrument, but does not require that the existing detector be removed. When the new detector is in use, the conventional detector is switched off which allows the ion beam to pass through to the new detection system.

The main components of the new detection system are an ion beam splitter and two separate high efficiency detectors. In the normal operation of the mass spectrometer, the ion beam has been focused at the collector slit and is then divergent beyond it. A focusing lens is placed immediately after the collector slit in order to bring the ion beam to a focus on the ion beam splitter. The ion beam splitter consists of a finely edge blade in line with, and parallel to, the length of the collector slit. Two flat reflection electrodes are placed either side of the blade.

A retarding potential is applied to the blade, equal to the accelerating voltage, such that ions are reflected away from the blade onto one of the two detectors. The ions directed towards the blade peel off to one side or the other, according to which side of the dead centre each ion is positioned, and reflect back to one of the two conversion dynodes. Secondary electrons emitted from the conversion dynodes are accelerated towards their two associated phosphors and the emitted light is detected using separate photo-multipliers.

Figure 1 gives an overview of the layout of the new detection system.

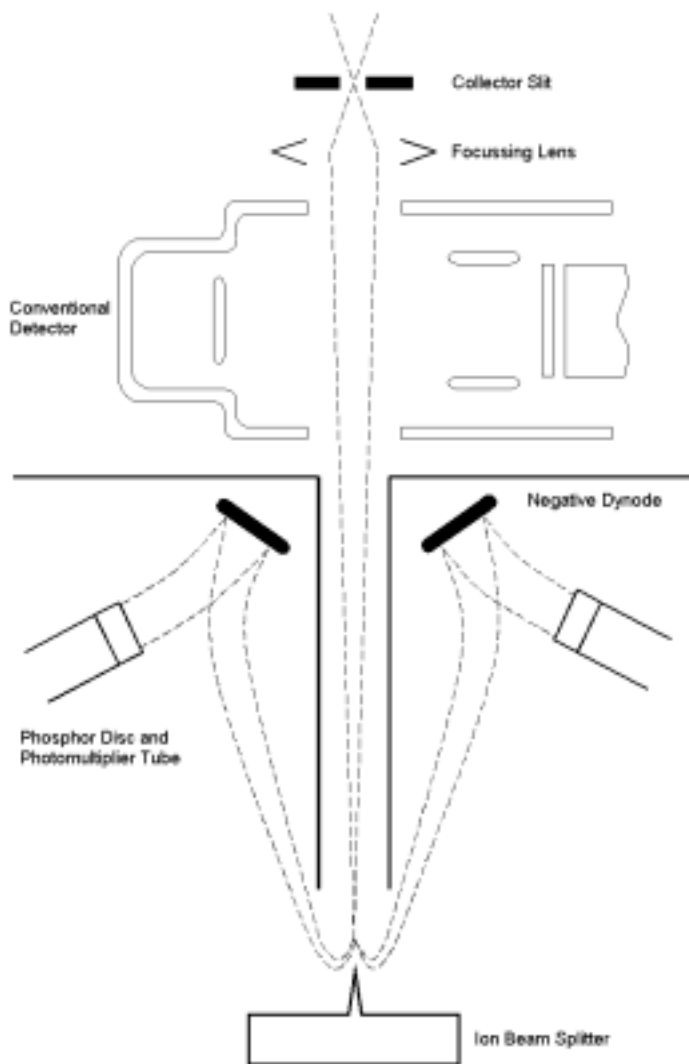


Figure 1. Overview of new detection system

The two output signals are compared. If the ions are symmetrically distributed onto the two detectors the two outputs will be approximately equal. This would be the case when an ion beam of the exact mass of interest is detected. If the ion beam is of a mass slightly higher or lower than that being monitored, but is still within the collector slit mass window, then the ions will be distributed asymmetrically between the two detectors. It is therefore possible to characterise an interference peak as such by merit of the relative intensities recorded by each detector.

Analysis

In order to evaluate the new detection system's ability to discriminate against unresolved mass interferences, an experiment has been devised whereby a sample of dioxin standard is spiked with a known interference compound. The mass that was monitored was for 1,2,3,7,8-PeCDD (357.8517 amu). The interference compound introduced was PCB169 (mass 357.8444 amu), contained within a WHO PCB standard (obtained from Wellington Laboratories Inc., Canada).

The spiked dioxin standard was introduced to the mass spectrometer through an Agilent 6890Plus GC. A splitless injector was used in conjunction with a Restek Rtx-5MS 60m column (0.25mm ID). A chromatogram showing the elution of the two components can be seen in figure 2. This acquisition was a voltage SIR experiment monitoring a number of analytical masses, including the 357.8517 amu mass of interest and a reference mass (which was used for lockmass corrections), and was performed at a system resolution of 10,000 (10% valley definition) on the normal detection system.

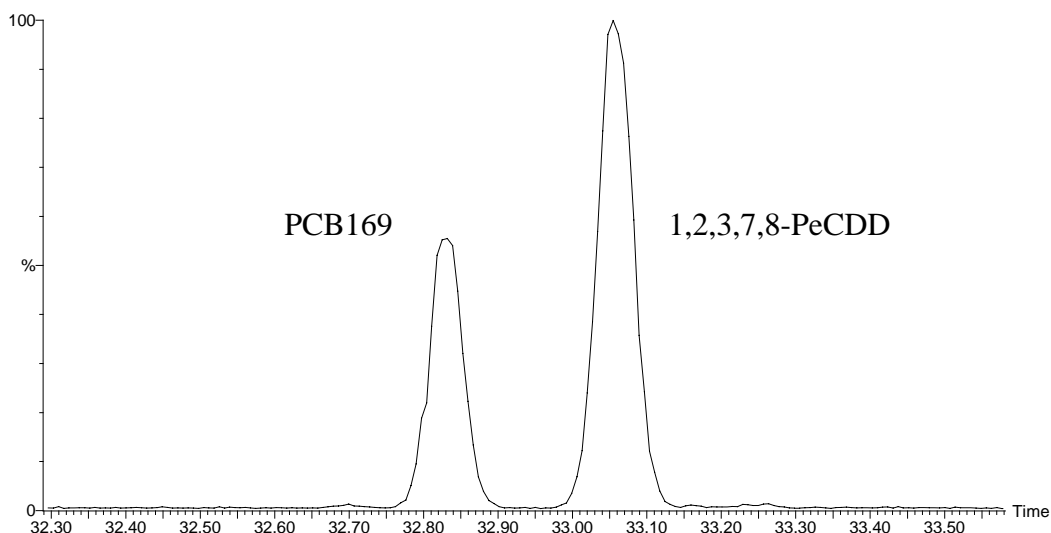


Figure 2. Chromatogram of mass 357.8517 amu

As can be seen, the PCB169 is detected whilst monitoring the 1,2,3,7,8-PeCDD mass even though it has a mass of 357.8444 amu, 20 ppm lower. This is because with the system resolution set to 10,000, a fraction of ions with masses within ± 50 ppm of the selected mass may still be transmitted. In order to fully resolve out the interference, the system resolution would have to be set to 24,500.

This experiment was run using the same parameters on the new detection system.

Results and Discussion

With the new detection system, two chromatographs are produced, one per detector. Figure 3 shows the same two components as in figure 2, as detected by the new detection system. There are three overlapping chromatographic traces shown. The largest trace is the sum of the two outputs, and the two smaller traces are the raw data from the individual detectors.

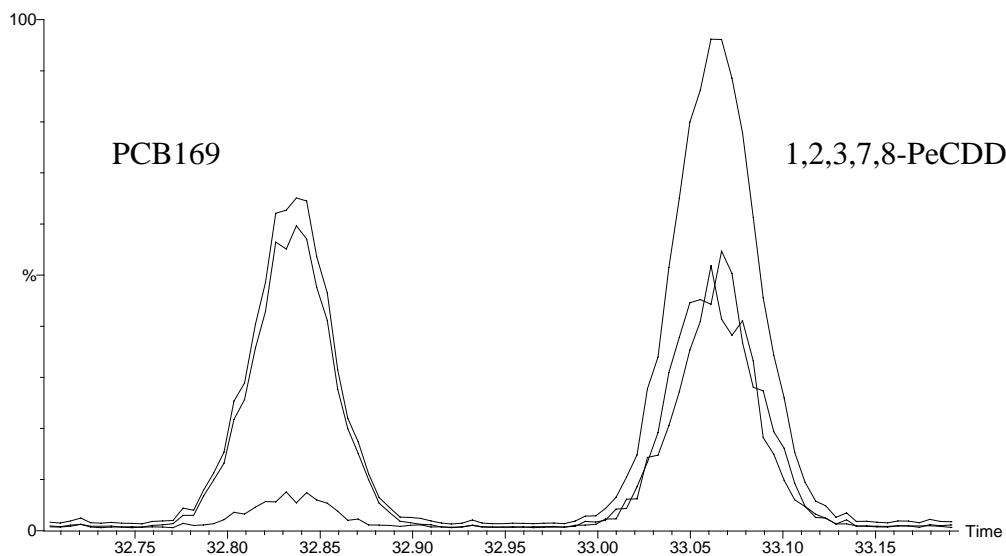


Figure 3. Chromatogram of mass 357.8517 amu on new detection system

As can be seen, when the 1,2,3,7,8-PeCDD elutes, it is detected with a very similar intensity on both detectors. The two smaller traces are both approximately 50% of the summed trace for this chromatographic peak.

The PCB component, by contrast, was detected with a noticeable difference between the signals recorded on the two detectors. The signal on the detector monitoring the lower mass side of the collector slit recorded an intensity of around ten times greater than that of the other detector. This demonstrates how an unresolved mass interference can be characterised as such by the new detection system.