B140 Simultaneous GC-NPD-MSD System for Forensic Analyses

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After attending this presentation, attendees will understand new tech- niques to significantly improve their gas chromatographic analyses which use nitrogen specific and mass spectral detection. The techniques increase the speed of analysis by collecting the nitrogen signal; mass spectral (MS) scan data, and mass spectometry in the single ion monitoring mode (MS SIM) data simultaneously. Analysis speed is further increased by back- flushing heavy matrix components from the column.

This presentation will impact the forensic community and/or humanity by providing a faster and more reliable GC analyses of, for example, drugs of abuse and toxicology samples.

In forensic gas chromatography (GC) analyses, there are often three important types of data to collect. Nitrogen selective detection with an nitrogen phosphorus detector (NPD) is used because it is sensitive and selective for drugs and makes a convenient screening tool. Mass spec- trometry is used in the single ion monitoring (SIM) mode for trace detection of target analytes, and in scan mode for identity confirmation via spectral matching with libraries. In many cases, samples are run on separate GCs: one with the NPD and one with a mass selective detector (a mass spectometer) (MSD). The MS data may require two runs, one in scan mode and one in SIM. It would therefore be advantageous to collect all three types of data simultaneously in a single instrument run.

To accomplish the desired simultaneous collection of all three types of detection data, a gas chromatography interfaced simultaneously with a nitrogen phosphorus detector and mass spectral detection (GC-NPD-MSD) system was constructed with two post-column microfluidic devices in series. The first device is a Dean's switch, which is a fluidic component that directs the column effluent to either of two pathways. Effluent can be sent out a vent or to an effluent splitter. The effluent splitter divides the effluent equally between an NPD and MSD. The system uses deactivated fused silica tubing as restrictors and interconnects between devices. Metal fer- rules are used to obtain leak free seals that do not loosen with thermal cycling of the oven. Electronic pneumatic control is used to supply both devices with constant pressure makeup gas.

In practice, the Deans switch is used to vent the solvent peak, excess derivatization reagent, column bleed during post analysis bakeout, and any other unwanted peaks. The rest of the time the column effluent is sent to the splitter and thus to the NPD and MSD. The venting capability reduces the frequency of MS source cleaning and extends the life of the NPD bead. A significant advantage is the ability use solvents which would normally damage the NPD bead, like $CHCl_2$, CCl_4, $CHCl_3$, etc. The MSD is operated in a mode where SIM data and scan data are collected in alternate cycles. The data from a sample analysis consists of three signals: the NPD response, SIM MS data, and scan data and are processed accordingly.

After acquisition of the data, if high boiling matrix components are present in the sample, the column can be backflushed. This removes the heavies much faster than simple bakeout. Backflushing is done by time pro- gramming the Deans switch pressure to a high value while programming the inlet pressure to a low one, reversing the flow through the column. The heavy material is the carried out the split vent. For example, backflushing at 300 C for two minutes can remove heavies that would take 20 min at 320C to elute.

The described system provides a means to obtain nitrogen, SIM and scan data with decreased analysis time.

Gas Chromatography, Nitrogen, Mass Spectrometry

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