

peaks indicated approximately 500 theoretical plates. Daidzin, 7-glucoside of daidzein and emodin glucoside isolated from Leguminosae have got well-defined peaks. Amygdalin, one of the cyanogenetic glycosides, appears to be most stable to heat. Monoterpene glycosides, such as aucubin from Cornaceae (*Aucuba*), monotropein from Pirolaceae (*Monotropa*), catalposide from Bignoniaceae (*Catalpa*) and paeoniflorin from Paeoniaceae (*Paeonia*) gave good peaks whose retention times increase in proportion as the number of C-atoms in the compounds increases as shown below: aucubin (C_{15} , $t_R = 1.7$), monotropein (C_{16} , $t_R = 2.6$) and catalposide (C_{22} , $t_R = 11.1$). The gas chromatogram of sinigrin showed a major peak with an additional minor peak. At a column temperature of 175° three peaks, namely $t_R = 6.35$ (main), 4.8 (minor), 3.1 (minor), were observed.

Because of its great sensitivity and resolving power, it is suggested that this method will be useful for the analysis of medicinal plants and their constituents.

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The importance of column material in the gas chromatography of isocyanides

In gas chromatography, interferences caused by the material of the tube itself have received little notice until recently. For the analysis of chlorinated pesticides, quartz¹ or glass² tubes have been recommended instead of metal; glass is preferred for phosphorus compounds³. Glass is also safer for steroid analysis although metal may be used in a properly designed system⁴. On the other hand, all-glass is the only way to handle some pyrrolizidine alkaloids⁵. In these cases it appears that the metal surface catalyses decomposition.

We have observed that with aliphatic isocyanides glass tubing is satisfactory

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but metal is not. Our results appear to explain the difficulty experienced by LIFSHITZ, CARROLL AND BAUER⁶ when attempting quantitative analyses of methyl cyanide-isocyanide mixtures.

Using a Perkin-Elmer model 154 gas chromatograph with 1 m metal columns (carrier gas, helium; stationary phase, "Reoplex 400"; temperature, 90°), we obtained unsatisfactory chromatograms for methyl, ethyl, propyl and butyl isocyanides which varied with the use of different inert supports.

On acid-washed but otherwise untreated material (such as "Embacel"), no peak at all was seen for 0.5 μ l samples. Many such samples (up to twenty) were injected at intervals equal to the expected retention time without observing a measurable peak. Injection of 2-5 μ l samples gave badly tailed peaks at varying times after the expected retention time.

On "Chromosorb W-HMDS" (a hexamethyldisilazane-treated support) 0.5 μ l samples produced small peaks at the correct retention time. When further samples were injected, the peak height increased. A total of approximately 4 μ l (introduced in small or large injections) was required before the peak height of a 0.5 μ l sample became constant. If a column subjected to such a series of samples were left with carrier gas passing through it overnight, a repetition of this behaviour was observed again next day. This suggests that the effect is due to a reversible adsorption. Similar effects were observed with both stainless steel and copper tubes.

Satisfactory chromatograms for sub-microgram quantities of alkyl isocyanides had been previously obtained using a Pye Argon chromatograph (4 ft. glass tube). When a glass tube was used in the Perkin Elmer instrument with either "Embacel" or "Chromosorb W-HMDS" as support, normal peaks were obtained with one 0.5 μ l injection.

The improvement in performance of the metal tubes using a silanized support could be due to the partial coating of the metal wall by HMDS from the treated support. This could mean that metal tubes, if internally precoated with a suitable material, would give chromatograms comparable with those obtained using glass.

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