

The separation of phenylhydrazones of volatile carbonyl compounds by vapour phase chromatography

The fractionation of carbonyl compounds and their identification generally involve very complex operations. We are particularly referring to the analysis of odoriferous constituents, where carbonyl compounds are present in very limited quantities, and to the study of ozonolysis fragments of complex organic substances.

The problem is even more complex when the compounds in the mixtures have different volatilities. In this case separation involves the formation of non-volatile derivatives and their resolution.

The problem is therefore now shifted to the separation of the derivatives. Among these, the 2,4-dinitrophenylhydrazones (2,4-DNPh) are generally preferred for known reasons. Chromatographic techniques are very useful in the fractionation of 2,4-DNPh mixtures into single components but the use of gas-liquid chromatography (GLC) for identification purposes is impossible, unless the carbonyl compounds can be regenerated and subsequently analysed by gas chromatography^{1, 2}. Obviously, a direct method of GLC-analysis for carbonyl compound derivatives would greatly assist in the study of such complex mixtures in small amounts.

The literature on this subject, as far as we know, is very scarce: CASON AND HARRIS³ have examined mixtures of dioximes of volatile aldehydes on celite-di-2-ethylhexyl phthalate columns, and LOHR AND WARREN⁴ have examined the oximes of benzaldehyde and salicylaldehyde on a celite-silicone column.

We have examined some aldehyde phenylhydrazones by GLC and here some preliminary data are presented for the gas chromatography of phenylhydrazones of normal aldehydes from C₁ to C₁₂.

Columns of SE 30 (2%) on Chromosorb W were used with a flame ionization detector. Nitrogen (flow rate 25 c.c./min) was used as carrier gas, and the column temperatures were between 120° and 190°. The peaks obtained were well shaped and no decomposition products were noted. A plot of the log of retention times of the *n*-aldehyde phenylhydrazones against the number of carbon atoms indicates a straight line relationship.

We intend to investigate this further and to expand these initial data.

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¹ J. W. RALLS, *J. Agr. Food Chem.*, 8 (1960) 141.

² S. W. RALLS, *Anal. Chem.*, 32 (1960) 332.

³ J. CASON AND E. R. HARRIS, *J. Org. Chem.*, 24 (1959) 676.

⁴ L. J. LOHR AND R. W. WARREN, *J. Chromatog.*, 8 (1962) 127.

Received February 14th, 1964