Gas Chromatography in the Fat and Oil Industry

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PERHAPS NO analytical method has captured the imagination of chemists and their colleagues in other scientific fields as Gas Liquid Chromatography (GLC) has is the past decade. Except for the petroleum industry, no industry or technology has been affected by gas chromatography as much as that concerned with fats and oils. Those of us who have been so closely associated with the development of GLC methodology are often surprised to find many of our colleagues are quite baffled by the meaning of the little wiggling lines. Now this really isn't too surprising on introspect when one considers the specialization of our modern technologies. It is our present aim to familiarize those who would like to know a little more about this powerful separating tool. How does it work? How is it applied to specific fat and oil problems? Where is it going? These questions we will consider in a popular sense. In order to find the answer, it is best to start at the beginning with some definitions and then some history.

Chromatography has been with us a long time. Its beginning goes well back in this century. T. Swett gave the separating process we call chromatography its name. The materials he separated were highly colored; therefore, he concluded that separation was a function of the color. Hence, the name was coined from chromos, the Greek word for color.

What is chromatography? Chromatography is a separating process in which the materials to be separated are placed on a column which contains two phases. One phase is stationary and has a large surface area; the second phase is a moving liquid or gas which percolates through the stationary phase. The substances to be separated will move through the column at different rates of speed depending on their affinity for the stationary phase or the moving phase. This definition describes all chromatography, whether it is column, paper, or gas chromatography.

Actually, gas chromatography was first described by Martin and Synge in 1941 in their Nobel Prize Paper on liquid-liquid partition chromatography. However, nothing was done on this idea until Martin and James published their now classic paper on separating fatty acids in 1952. The roof didn't exactly fall in on the fat and oil industry, but this was the beginning of the avalanche of papers on gas chromatography that has descended upon us in recent years. It is interesting to note that Dr. Martin probably cheated himself out of a second Nobel prize with that one paragraph in the 1941 paper.

The gas chromatograph is a comparatively simple apparatus. It consists of a column for separating the components, a carrier gas supply, a detector for the column effluent, and some means of recording the detector signal. Since we are mainly concerned with high boiling lipid materials, provision must be made for heating the column and the detector. Just about any material can be gas chromatographed, providing it has 15 mm of vapor pressure at the temperature at which it is to be separated. The time it takes a compound to emerge from the column is called its retention time and is a qualitative measure. The size of the peak is a quantitative measure.

As an analytical tool, gas chromatography has a number of great advantages. GLC has a wide range of applications to many compounds. It has great sensitivity with a wide choice of detection systems. Most analyses take a comparatively short time. It is capable of both qualitative and quantitative analyses of very complex mixtures. The gas chromatograph is cheap compared to most modern analytical instrumentation. The apparatus, indeed, begins to approach the ideal analytical machine.

The column is the heart of gas liquid partition chromatography. The column packing is usually a liquid adsorbed on an inert support material such as Celite or powdered firebrick. The separation achieved on a given column is dependent of a number of factors. These include the amount and nature of the liquid phase, the solid support, the length of the column, and the operating temperature. Thousands of liquid phases have been used in gas chromatography. However, only a relatively few can be used in fat and oil analysis. The reason for this is because of the high boiling nature of the fatty acids and their related compounds. Another variation of the column is the capillary or open tube column. This is usually a long, small diameter tube, with the liquid phase coated on the inside of the column. Very long columns of this type have enormous separating ability.

There is a wide choice of reliable GLC detection systems. These include thermal conductivity, beta ray ionization, flame ionization, gas density balance, ionization cross section, and radio frequency detection system, to name a few. Each of these systems has its advantage, but the first three named are currently the most widely used. Carrier gases that are used include helium, nitrogen, argon, and hydrogen. There are other useful techniques available in the instrumentation such as temperature programming. With this technique, one starts the chromatograph at a low temperature and gradually increases the temperature throughout the run. It is most useful for separating wide boiling mixtures.

We are now ready to discuss the applications of gas chromatography. As mentioned earlier, James and Martin first separated fatty acids up to dodecanoic acids. They soon found that the methyl esters were easier to work

(Continued on page 16)

THE COVER

The gas chromatograph on the cover is a separation of the methyl esters of the fatty acids from human hair oil. This separation of an 8-μg sample was made on a capillary column. Human hair oil is a fine example of the complex nature of some natural occurring fats. It is a rich source of unusual acids mentioned in the feature article. The tentative identifications of some of the fatty acids were made from literature values of relative retenion time.

Gas Chromatographic Conditions

Sample:	Methyl ester of fatty acids of human hair oil
Instrument:	Barber Colman Model 20
Column:	100-ft stainless steel 0.01 in. capillary coated with Apiezon L

Injection temperature	255C
Column temperature	185C
Cell temperature	203C
Gas flow	4 ml/min
Scavanger gas flow	60 ml/min
Sample size	10 nanoliters