

Table 2. DETERMINATION OF \bar{M}_n OF AN IONIC POLYSTYRENE USING CELLULOSE ACETATE MEMBRANES CA3/4; BENZENE SOLUTIONS AT 30° C.

Concentration C per cent (w/v)	Equilibrium osmotic pressures					Number average molecular weight \bar{M}_n
	Rising hr (cm.)	Time (hr.)	Falling hr (cm.)	Time (hr.)	h (cm.)	
0.100	7.64	40	7.54	31	7.64	} 3,800 ± 200
0.080	6.15	50	6.33	112.5	6.24	
0.060	4.56	49	4.59	28	4.57	
0.040	3.39	89	3.36	44.5	3.38	
0.020	1.60	70	1.54	50	1.60	

higher solvent permeabilities. The very selective membranes can be used for determining quite low molecular weights and in Table 2 are shown the results obtained using membrane CA3/4 with an ionic polystyrene having $\bar{M}_n = 3,800$. Unfortunately, while it is comparatively easy to make membranes such as CA2/58, only a few membranes like CA1/68 and CA3/4 have so far been prepared. In these experiments benzene was used as solvent, but successful osmotic measurements have also been made in toluene and water.

The cellulose acetate used in the experiments was obtained from the British Drug Houses, Ltd.

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Liquid/Solid Content of Fats

THE consistency of margarine and other commercial fats depends to a large extent on the relative amounts of liquid and solid fats present. The usual method for determining these values is by dilatation measurements and the dilatometer is used fairly considerably for this work. There are, however, certain inherent errors in this type of measurement. It is stated that the calculation of liquid/solid content from dilatometric data is either not possible with any degree of accuracy¹ or even that it is impossible² with complex mixtures of glycerides. This arises from the fact that a prior knowledge of the glycerides present is required and that mathematical adjustment is needed to take account of the different melting dilatations. Such prior knowledge is not always available.

We have recently found that the use of low-resolution nuclear magnetic resonance spectroscopy provides a new means for such determinations which eliminates many of the inherent errors of the dilatation method. In effect, use is made of the inherent disparity in the nuclear magnetic line-widths observed with liquids and solids. It is known that the shape of the hydrogen resonance line is influenced by the chemical and physical state of the sample and that the width is related to the mobility of the compound containing the hydrogen and to the field homogeneity. With solids the line-width is of the order of several gauss, while with liquids the line-width is very narrow and is determined by the inhomogeneity of the applied magnetic field³.

An example of the method adopted is illustrated by the determination of the liquid content of simple mixtures of known amounts of tristearin and triolein (tristearin does not form a solid solution in the triolein phase). A bridged-T nuclear magnetic resonance spectrometer was used⁴. The magnetic field was modulated at a sinusoidal frequency of about 40 c/s. with a magnitude of about 30 milligauss, peak to peak. The line-width was determined by the inhomogeneity of the magnetic field and was about 100 milligauss at half-height. Under these conditions the derivative curve shows no signal due to the broad band of the solid, and a measure of the peak-to-peak height of the recorded derivative curve is a direct measure of the amount of liquid present. By comparing the peak-height for any of the mixtures with that for 100 per cent liquid, the liquid content can therefore be determined. Typical results are shown in Table 1. Similar work has also been carried out with other fat mixtures.

Table 1

Known percentage of liquid glyceride	Average percentage found
75.1	74.0
50.2	51.2
25.1	25.4

The new technique has the following advantages:
(a) Fats or mixtures may be examined in the state in which they are received, that is, without altering the structure by melting or crystallization.

(b) The results are independent of the particular composition of the fat, that is, no prior knowledge of the glycerides present is required.

(c) The results are independent of the polymorphic form of the solid present.

A full account of this work will be reported elsewhere.

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Determination of Galactosediethylmercaptal and 3,6 Anhydrogalactosediethylmercaptal with the Anthrone and Seliwanoff Reagents

SOME pure 3,6 anhydro-L-galactose was required as standard to determine the concentration of this sugar in several algal polysaccharides. Efforts to prepare it in a crystalline form were unsuccessful. Therefore the standard solution was made up from the 3,6 anhydro-L-galactosediethylmercaptal. Unfortunately the ethylmercaptan liberated on acid hydrolysis of the mercaptal interfered with colour formation in the anthrone and Seliwanoff tests. A similar interference was obtained with the diphenylamine reagent¹. A method was developed to obtain