spectrometer. Only one sharp resonance line was observed: 4.42 p.p.m. referenced to internal tetramethylsilane (TMS). White (*21*) had predicted a chemical shift value of 4.44 p.p.m. (from TMS) for I. Isomeric 1,1,2,2,3,3-hexachloropropane exhibits a chemical shift of 6.35 p.p.m. (from TMS) (*21*).

**IR.** NaCl plates (neat) using a Perkin-Elmer Model 137 spectrophotometer showed absorptions ($\mu$) at: 3.32 (vw); 7.00 (m); 7.72 (vw); 8.25 (w); 9.45 (w); 9.80 (vw); 10.10 (vw); 10.30 (s); 11.78 (s); 12.30 (vs); 12.90 (vs); 13.35 (m); 13.70 (vs); and 14.85 (vs). The characteristic frequencies of the Raman spectrum of I have been reported (*2*).

**VPC.** F and M Chromatograph, Model 300: 1 meter, 0.25-inch O.D. aluminum column, packed with 5% Igepal on Chromosorb ABS (80- to 100-mesh); injection port temp., 250° C.; detector block temp., 280° C.; helium flow rate, 75 ml. per minute; column temp. programmed 9° C. per minute from 75° to 225° C. Retention time of I, 7.0 minutes.

The distillation residue (0.95 gram) consisted of a viscous liquid ($n_D^{26}$ 1.575) which was not characterized.

When the above reaction was conducted in a glass-lined rocking autoclave—75° C., 24 to 60 hours; 5 moles of methylene chloride, 1 mole of tetrachloroethylene, and 0.2 mole of aluminum chloride—the yield of I decreased to 2.5%. Mass spectral and infrared analysis indicated that a low-boiling fraction, b.p. 42–7° C., 4.5 mm. of Hg, consisted of II and/or III and IV. The distillation residue (maximum temp., 183° C., 4 mm. of Hg) was not identified.

Tetrachloroethylene (1.79 moles), methylene chloride (5.37 moles), and gallium chloride (0.057 mole; Alfa Inorganics) were heated at reflux for 22 hours. VPC indicated the presence of starting materials only.


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# ANNOUNCEMENT

# Infrared Spectra of Monosaccharides Related to Glycolipids in the Range 800–200 Cm.$^{-1}$


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INFORMATION is available on the infrared spectra of the following monosaccharides in the range of 800 to 200 cm.$^{-1}$: α-D-Galactose, α-D-glucose, α-D-mannose, α-D-fucose, D-ribose, β-lactose, N-acetylneuraminic acid, D-galactosamine-HCl, D-glucosamine-HCl, D-mannosamine-HCl, N-acetyl-D-galactosamine, N-acetyl-D-glucosamine, and N-acetyl-D-mannosamine. The majority of the spectra were recorded as suspension of the compounds in Nujol injected into a disposable or demountable polyethylene cell. Some spectra were recorded using pellets made from cesium iodide powder, which was obtained by lyophilization. The spectra were recorded with 2 to 20 mg. of the saccharides, using a Perkin-Elmer 621 Spectrophotometer. Expanded frequency scale and ordinate expansion was used. The spectra are quite different and can be used for the identification of these compounds.

The complete spectral data and techniques for sample preparation employing cesium iodide pellets of Nujol suspensions in polyethylene cells may be obtained as NAPS Document NAPS-00427 from ASIS National Auxiliary Publications Service, c/o CCM Information Sciences, Inc., 22 West 34th St., New York, New York 10001; remit $1.00 for microfiche or $3.00 for photocopies.


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