stopcock lubricant described by Herrington and Starr[3] was a satisfactory lubricant for the stopcocks and ground joints used in the distillation apparatus. The following fractions were obtained:

Fraction	Volume, ml.	Temp., °C.	n^{29}_D	α^{29}_D
1	20	30	1.4677	+84.17°
2	60	31	1.4683	+96.67°
3	10	32	1.4690	+92.00°
Residue	510	..	1.5173	− 0.50°

Fractions 1, 2 and 3 account for 15% of the oil. The physical properties of fraction 2 are similar to those of *d*-limonene. The presence of *d*-limonene was confirmed by preparing the β-nitrol anilide, m. p. 153°, and the tetrabromide, m. p. 103.5°, $[\alpha]^{30}_D$ +72.0° (0.1785 g. of *d*-limonene tetrabromide in 5 ml. of carbon tetrachloride solution).

A 1500-ml. portion of the fresh oil was subsequently fractionated, as above, at a pressure of 22 mm., with a reflux ratio of 0.9 (90% of the condensate returned to the column). The first fraction, 3.1 ml., boiling at 55°, had a refractive index of 1.4628^{25}, and a rotation of −37.67°. Calculated for *l*-α-pinene this would be a specific rotation of −43.70°, which is near the reported −48.63°.[4] The presence of *l*-α-pinene was confirmed by the preparation of α-pinene nitrolpiperidine, m. p. 118.5°. The quantity found by distillation represents 0.2% of the oil.

The specific rotation of *d*-limonene as calculated from the rotation of fraction 2 would be +114°, as compared to a specific rotation for pure *d*-limonene of +126°.[5] This plus the refractive index of fraction 2 indicates the probable presence of dipentene. No direct evidence for the presence of dipentene or other terpenes was obtained. The negative rotation of the terpeneless residue indicates that the borneol is probably *l*-borneol. Methylchavicol, an isomer of anethol, is responsible for the anise-like odor and flavor. The odor and flavor of the oil are decidedly improved by the removal of the terpenes.

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(4) Gildemeister and Hoffmann, "The Volatile Oils," second edition, John Wiley and Sons, Inc., New York, N. Y., 1913, Vol. I, p. 293.

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Preparation of Tetraacetyl-α-D-glucopyranosyl Bromide

BY C. G. JEREMIAS, G. B. LUCAS AND C. A. MACKENZIE

There are many references in the literature pertaining to the preparation of tetraacetyl-α-D-glucopyranosyl bromide. Most authors recommend the use of an acetic acid solution of hydrogen bromide with pentaacetyl-β-D-glucose. They differ in their experimental details, particularly with respect to the use of vacuum distillations, solvents and purifications methods. The description given below eliminates vacuum distillations, simplifies the purification process and gives a pure product in good yield with a minimum of effort.

Experimental

A mixture of 20 g. of pentaacetyl-β-D-glucose and 20 ml. of hydrobromic acid–acetic acid solution (Eastman Kodak Co. 30–32% hydrobromic acid–acetic acid) is stirred at room temperature for two hours in a flask protected from moisture. The clear, yellow solution is poured, in a thin stream, into 400 ml. of vigorously stirred ice water. A finely divided, crystalline material is obtained. If the addition is made too rapidly the product solidifies in the form of large lumps. The crude acetobromoglucose is filtered with suction and then transferred to a small separatory funnel. Fifty ml. of carbon tetrachloride is added to put the solid into solution. The water layer formed is drawn off with a suction pipet and the product remaining is washed once with 20 ml. of ice water and then with a few ml. of cold, saturated sodium bicarbonate solution until all free acid has been removed. The solution is finally washed with two 20-ml. portions of ice water. The carbon tetrachloride layer is filtered through glass wool into an erlenmeyer flask and dried over calcium chloride. The dry solution is poured slowly, with stirring, into 200 ml. of petroleum ether (35–75° boiling range was used; a good quality reagent is necessary or a yellowing of the product may occur at this point). When crystallization at room temperature is complete, an ice-salt-bath is placed around the container and an additional crop of crystals form. The crystals are filtered with suction and air-dried or dried in a vacuum desiccator. The product at this point has a m. p. 88–89°; yield, about 18 g. (80–85%).

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Heats of Mixing of Some Fluorinated Ethers with Chloroform

BY J. R. LACHER, J. J. MCKINLEY AND J. D. PARK

It is well known that chloroform and monofluorodichloromethane will form hydrogen bonds with solvents containing donor atoms such as nitrogen and oxygen.[1] Diethyl ether[2] and polyethylene glycol ethers[3] show a considerable heat evolution when mixed with chloroform or monofluorodichloromethane. The substitution of chlorine in an aliphatic ether[4] or the replacing of an alkyl by an aryl group[5] reduces considerably the tendency for bonding. Recently[6] a number of polyfluoro alkyl ethers have been prepared in this Laboratory. If one interprets hydrogen bonds as the result of an interaction between dipoles,[7,8,9] one might expect that these fluorinated ethers would also show only a slight tendency for hydrogen bonding. The moments are not known for these molecules. However, one can calculate,

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