of *o*-nitrobenzoyl chloride. Our experience was similar. Small quantities appeared to distil satisfactorily, but this trouble was encountered in an 8-mole run. The volatile products ($POCl_3$, PCl_5) were removed at 100° (20 mm.), then the flask was equipped for vacuum distillation with an oil pump and a glas-col heating mantle. Before any distillate was collected the material detonated violently. Fortunately, however, there was a warning period of a few seconds.

Reasonably pure *o*-nitrobenzoyl chloride may be made without distillation by use of thionyl chloride instead of phosphorus pentachloride. The gaseous reaction products and any unused thionyl chloride were removed by aspirating with a stream of dry illuminating gas for a few hours.

DEPARTMENT OF CHEMISTRY
NORTHWESTERN UNIVERSITY W. A. BONNER
EVANSTON, ILLINOIS CHARLES D. HURD

RECEIVED JANUARY 9, 1946

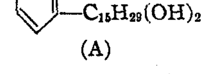
CASHEW NUT SHELL LIQUID. II. AN APPLICATION OF THE PREVOST REAGENT TO ALKENYL PHENOLS[1]

Sir:

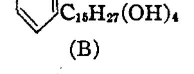
The noxious saps of numerous members of the *anacardiaceae* family, such as Poison Ivy, Cashew Nut Tree, Japanese Lac Tree, *etc.*, contain phenolic mixtures made up of saturated, mono-, di- and probably polyolefinic components. With the purpose of developing a method for separating and establishing the structures of such olefinic components, we have investigated the use of silver iodobenzoate.[2,3]

The monophenol, obtained by direct vacuum distillation of raw commercial cashew nut shell liquid, and possessing an average of two aliphatic double bonds,[1] was redistilled several times, methylated and then distilled. The resulting methyl ether possessed 1.56 double bonds. Treatment with silver iodobenzoate, and subsequent hydrolysis of the benzoates, yielded, on fractional crystallization from aqueous methanol, two crystalline glycols (A and B), both of which contained iodine. Both glycols showed no discoloration of bromine in carbon tetrachloride.

Glycol (A), m. p. 92–93°, analyzed correctly for an iodinated monoglycol.



(A)



(B)

Anal. Calcd. for $C_{22}H_{37}O_3I$: C, 55.35; H, 7.74; mol. wt., 476. Found: C, 55.55; H, 7.51; mol. wt. (ebulliometric in CCl_4), 478.

Glycol (B) m. p. 112–113° and analyzed correctly for an iodinated diglycol.

Anal. Calcd. for $C_{22}H_{37}O_5I$: C, 51.96; H, 7.28. Found: C, 51.92, 52.01; H,7.50, 7.43.

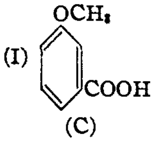
Oxidation of the mono glycol (A) with periodic acid in aqueous methanol gave *n*-heptaldehyde which was identified as the 2,4-dinitrophenylhydrazone, m. p. 106.5–107°, by mixed m. p. and analysis.

Anal. Calcd. for $C_{13}H_{18}O_4N_4$: C, 53.06; H, 6.12; N, 19.05. Found: C, 53.23; H, 6.21; N, 19.10.

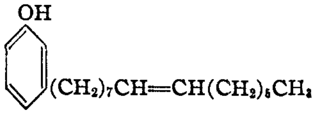
Oxidation of the mono glycol (A) with alkaline aqueous permanganate gave an aromatic acid containing iodine which analyzed correctly for (C).

Anal. Calcd. for $C_8H_7O_3I$: C, 34.54; H, 2.51. Found: C, 34.71; H, 2.77.

Catalytic hydrogenation of the mono glycol (A) using 5% Pd–$BaCO_3$ in ethanol yielded an iodine-free solid which is now under investigation.



(C)



Mono-olefinic component

The experiments described above establish that the mono phenol of commercial cashew nut shell liquid is a mixture of at least two different olefinic components: one a mono-olefin and the other a di-olefin. It appears likely that a more highly unsaturated component is also present. The mono-olefinic component is unsaturated in the 8–9 position of the side chain as established by the isolation of *n*-heptaldehyde after periodic acid oxidation of the glycol.

Further studies on the identification of the position of the iodine in the ring and the application of the Prevost reagent to the separation and identification of other unsaturated natural phenolic bodies, such as are found in poison ivy, are now in progress. Details of all this work will appear in forthcoming publications.

DEPARTMENT OF CHEMISTRY
COLUMBIA UNIVERSITY MEYER SLETZINGER
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RECEIVED JANUARY 16, 1946

SELECTIVE DEMETHYLATION OF PARAFFIN HYDROCARBONS

Sir:

Paraffin hydrocarbons, as for example, pentane and hexane, are known to react in the presence of active hydrogenation catalysts, such as nickel, to produce primarily methane and carbon. Mixtures of hexane and hydrogen give methane as the major product at 400°.

We wish to record a new reaction, **the selective** demethylation of branched-chain paraffin **hydro-**

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[1] For the first article in this series, see Wasserman and Dawson, *Ind. Eng. Chem.*, **37**, 396 (1945).

[2] Prevost, *Comp. rend.*, **196**, 1129 (1933); **197**, 1661 (1933); **198**, 2264 (1934).

[3] Hershberg, *Helv. Chim. Acta.*, **17**, 351 (1934).