steam distillation). The residue was dissolved in 750 ml. petroleum ether at room temperature and washed with 300 ml. 50% ethyl alcohol to remove traces of soap, after which the petroleum ether solution was dried and filtered. The solvent was removed, the residue distilled at 10 mm. pressure, and the fraction boiling at 161–165° collected. The yield of crude alcohol was 11 g. (40%).

After two crystallizations from acetone at −60° and vacuum distillation (b. p. 134°, 2 mm.) the 2-methyltetradecanol-1 melted at 32.0–32.2°. The alcohol is soluble in all common organic solvents.

Anal. Calcd. for $C_{15}H_{32}O$: C, 78.86; H, 14.13. Found: C, 78.90; H, 14.21.

INSTITUTE OF MEDICAL CHEMISTRY
UNIVERSITY OF UPPSALA, SWEDEN
KARIN LINDBLAD
EINAR STENHAGEN

RECEIVED OCTOBER 6, 1941

COMMUNICATIONS TO THE EDITOR

THE DECOMPOSITION OF OZONIDES WITH RANEY NICKEL

Sir:

We find that ozonides react with Raney nickel to give aldehydes or ketones and nickel oxide. The reaction is vigorous at 35° when a pentane solution of ozonide is slowly added to a suspension of the nickel in pentane. It appears to be the only reaction taking place and the yield of aldehydes and ketones is comparable with that obtained in the less convenient method of F. G. H. Fischer [Long, *Chem. Rev.*, **27**, 437 (1940)], and at least twice that obtained by the earlier procedures. The simple manipulation and the absence of water improve the value of the ozonolysis reaction as an analytical method.

A solution of 200 cc. of pentane and 75 g. (0.6 mole) of the nonenes from the dehydration of methylethylneopentyl carbinol was ozonized during fifteen hours with about 5% ozone at salt and ice temperature. The ozonide was then slowly added to 48 g. (0.8 g. atom, *i. e.*, 100% excess) of Raney nickel suspended in 100 cc. of pentane in a 500-cc. 3-necked flask. The flask was equipped with a mercury-seal stirrer, condenser and dropping funnel. The reaction was vigorous at first, boiling the pentane as the ozonide was added, but slowed down considerably before all the ozonide was added. Then a further 20 g. of Raney nickel was added. The mixture showed no test for ozonide after stirring overnight with the nickel. To be sure of the decomposition of the ozonide, the pentane was stripped and the mixture was refluxed for an hour over the nickel at 115–120°.

The nickel was allowed to settle and the product was decanted. The residue was extracted three times with 50-cc. portions of pentane and finally centrifuged to remove the last solid.

The pentane strippings from the decomposition which had been caught in a dry-ice trap were fractionated through a small 15-plate column using a dry-ice trap to catch most of the low-boiling products. About 5 g. of acetaldehyde was collected in the first pentane fractions. The dry-ice trap contents weighed 20 g. This was acetaldehyde with a trace of formaldehyde. These were identified as usual. The pentane extract was then fractionated and found to contain 46 g. of a mixture of methyl neopentyl ketone, ethyl neopentyl ketone and traces of trimethylacetaldehyde and methyl ethyl ketone. There was about three times as much of the first ketone as of the second. About 10 g. of higher boiling material was present which has not been identified. The yield of aldehydes and ketones from the reaction was about 75%. This new method is being studied intensively in all of its ramifications.

It is not as yet known that all ozonides in various solvents will react with Raney nickel or that the reaction is complete. Consequently the usual care and precautions should be used to avoid the danger of explosions. In this Laboratory wire glass screens are used in front of all apparatus even during the final distillation of the probably ozonide-free products.

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NEWELL C. COOK
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RECEIVED NOVEMBER 21, 1941

IN REFERENCE TO THE CATALYTIC REDUCTION OF ESTERS USING NICKEL ALONE AS A CATALYST

Sir:

In February, 1940, there appeared an article by Peter L. de Benneville and Ralph Connor,[1] concerning which I wish to make several comments.

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[1] De Benneville and Connor, THIS JOURNAL, **62**, 283 (1940).