

sively with small amounts of water, sodium hydroxide solution, dilute hydrochloric acid, and again with water. The benzene was then evaporated on a steam-bath and the residue recrystallized from water-alcohol mixtures. The method was found to work satisfactorily with quantities of acid as small as 50 mg. In some cases it was found necessary to use a small amount of decolorizing charcoal to render the product colorless.

The analyses were performed by Dr. T. S. Ma.

TABLE I

Acid	Derivative	M. p., °C. (uncor.)	Empirical formula	N analyses, %	
				calcd.	Found
1-Phenyl- cyclobu- tane-1-car- boxylic	Amide	75-76	C <sub>11</sub> H <sub>13</sub> ON	8.00	7.83
	<i>p</i> -Toluide	96-96.2	C <sub>17</sub> H <sub>17</sub> ON	5.57	5.36
	<i>o</i> -Bromo- anilide	129-131	C <sub>14</sub> H <sub>13</sub> ON	5.28	4.98
1-Phenyl- cyclo- pentane-1- carboxylic	Amide	82-83	C <sub>17</sub> H <sub>19</sub> ONBr	4.23	4.24
	<i>p</i> -Toluide	98-99	C <sub>18</sub> H <sub>19</sub> ON	5.28	5.54
	<i>o</i> -Bromo- anilide	145-146	C <sub>19</sub> H <sub>21</sub> ON	5.02	4.95
1-Phenyl- cyclohex- ane-1- carboxylic	Amide	75-76	C <sub>18</sub> H <sub>19</sub> ONBr	4.07	4.13
	<i>p</i> -Toluide	85-86	C <sub>19</sub> H <sub>21</sub> ON	5.03	5.01
	<i>o</i> -Bromo- anilide	165-166	C <sub>20</sub> H <sub>23</sub> ON	4.78	4.89
		167-169	C <sub>19</sub> H <sub>20</sub> ONBr	3.91	3.93

CHEMISTRY DEPARTMENT  
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RICHARD D. KLEENE

RECEIVED SEPTEMBER 25, 1941

### $\alpha$ -Naphthylisopropylpotassium

This compound was prepared according to the method of Ziegler<sup>1</sup> for the preparation of the corresponding phenyl compound.

**Methyl Ether of  $\alpha$ -Naphthylidimethylcarbinol.**—Twenty grams of the carbinol was refluxed with 8 g. of sodamide in 75 ml. of dioxane and then with 40 g. of methyl iodide. After removal of the solvent, and two vacuum distillations, 8 g. of a colorless, fragrant oil was obtained boiling at 100–101° at 3 mm. pressure. The substance displayed a light purple fluorescence:  $d_{20}^{25}$  1.0422,  $n_D^{20}$  1.5867,  $M_D$  (calcd.) 63.76,  $M_D$  (obs.) 64.49.

*Anal.* Calcd. for C<sub>14</sub>H<sub>16</sub>O: OCH<sub>3</sub>, 15.5; C, 84.00; H, 8.00. Found: OCH<sub>3</sub>, 15.24; C, 84.14; H, 7.98.

**$\alpha$ -Naphthylisopropylpotassium.**—Three grams of the methyl ether was sealed off in a flask under nitrogen with 100 ml. of absolute diethyl ether and excess sodium-potassium alloy. The cleavage started almost immediately and the solution turned a deep cherry-red color, showing the presence of an organometallic compound. The alkyl was identified by treatment with carbon dioxide, followed by isolation of the resulting carboxylic acid.

**$\alpha$ -Naphthylisobutyric Acid.**—The flask containing the alkyl solution was broken open, and gaseous carbon dioxide introduced. The acid was isolated as small white needles, which after recrystallization from alcohol melted at 121–122°. The yield was 32% based on the amount of methyl ether originally taken.

(1) Ziegler, *et al.*, *Ann.*, **473**, 1 (1929).

*Anal.* Calcd. for C<sub>14</sub>H<sub>14</sub>O<sub>2</sub>: C, 78.50; H, 6.55; neut. equiv., 214. Found: C, 78.88; H, 6.63; neut. equiv., 215.

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### Tetrahydrofuran Compounds. II. Preparation of 1-Tetrahydrofuryl-3-chlorobutane

This compound was prepared with the view of determining the effect of the tetrahydrofuryl group as a substituent in alkyl halides.

**1-Tetrahydrofurylbutanol-3.**—Thirty-four grams (0.25 mole) of Eastman Kodak Co. furfuralacetone was placed in a rotating-type steel bomb along with 3 g. of nickel oxide catalyst and 30 ml. of ethanol. The initial hydrogen pressure was 100 atmospheres. The contents were then heated at 125° for four hours. After the reaction was complete, the catalyst was separated and the desired product was obtained as a colorless liquid,  $n_D^{20}$  1.4512. The yield was 63%.

**1-Tetrahydrofuryl-3-chlorobutane.**—Forty grams of the alcohol with 28 g. of pyridine was placed in a 3-necked, 500-ml. flask with stirrer, dropping funnel and condenser. Forty grams of thionyl chloride was added dropwise during thirty minutes. The temperature was not allowed to rise above 50°. On standing overnight, a precipitate of pyridine hydrochloride settled out. The liquid was decanted, and the precipitate washed thoroughly with dry diethyl ether. The washings were combined with the main product, thoroughly washed with water, and then dried over anhydrous sodium sulfate. The ether was distilled off, and the residue distilled in vacuum. There was obtained 19.5 g. of a colorless liquid with a pungent odor, boiling at 58–60° at 3 mm. pressure:  $n_D^{20}$  1.4505,  $d_{20}^{20}$  0.9976,  $M_D$  (calcd.) 43.46,  $M_D$  (obs.) 43.82. The product was insoluble in water.

*Anal.* Calcd. for C<sub>5</sub>H<sub>10</sub>OCl: Cl, 21.82. Found: Cl, 22.04.

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### 2-Methyl-tetradecanol-1

Thirty-three grams (0.12 mole) of ethyl methyl-*n*-dodecylacetate (b. p. 166° (6 mm.),  $n_D^{20}$  1.4369, from methyl-*n*-dodecylacetic acid of setting point 36.50°) was dissolved in 330 ml. of petroleum ether (b. p. 70–80°), 40 g. of sodium added and the mixture refluxed; 200 ml. of butyl alcohol<sup>1</sup> was added in portions of 10–15 ml. and the mixture refluxed for eight hours; 410 ml. of 96% ethyl alcohol was then added and the mixture refluxed for another hour. After cooling, 300 ml. of water was added cautiously and the resulting alkaline bottom layer, which contained unreduced acid as sodium salt, removed. The top layer was washed with 600 ml. of hot water and the petroleum ether and butyl alcohol removed (the latter by

(1) Bleyberg and Ulrich, *Ber.*, **64**, 2506 (1931).

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-methyl-tetradecanol-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 KARIN LINDBLAD  
UNIVERSITY OF UPPSALA, SWEDEN EINAR STENHAGEN

RECEIVED OCTOBER 6, 1941

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## COMMUNICATIONS TO THE EDITOR

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### 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 ozone 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 ozone 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.

DEPARTMENT OF CHEMISTRY  
THE PENNSYLVANIA STATE COLLEGE NEWELL C. COOK  
STATE COLLEGE, PENNSYLVANIA FRANK C. WHITMORE

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,<sup>1</sup> concerning which I wish to make several comments.

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