

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 ₁₁ H ₁₃ ON	8.00	7.83
	<i>p</i> -Toluide	96-96.2	C ₁₇ H ₁₇ ON	5.57	5.36
	<i>o</i> -Bromo- anilide	129-131	C ₁₄ H ₁₃ ON	5.28	4.98
1-Phenyl- cyclo- pentane-1- carboxylic	Amide	82-83	C ₁₇ H ₁₉ ONBr	4.23	4.24
	<i>p</i> -Toluide	98-99	C ₁₈ H ₁₉ ON	5.28	5.54
	<i>o</i> -Bromo- anilide	145-146	C ₁₉ H ₂₁ ON	5.02	4.95
1-Phenyl- cyclohex- ane-1- carboxylic	Amide	75-76	C ₁₈ H ₁₉ ONBr	4.07	4.13
	<i>p</i> -Toluide	85-86	C ₁₉ H ₂₁ ON	5.03	5.01
	<i>o</i> -Bromo- anilide	165-166	C ₂₀ H ₂₃ ON	4.78	4.89
		167-169	C ₁₉ H ₂₀ ONBr	3.91	3.93

CHEMISTRY DEPARTMENT
UNIVERSITY OF CHICAGO
CHICAGO, ILLINOIS

RICHARD D. KLEENE

RECEIVED SEPTEMBER 25, 1941

α -Naphthylisopropylpotassium

This compound was prepared according to the method of Ziegler¹ for the preparation of the corresponding phenyl compound.

Methyl Ether of α -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}^{20} , 1.0422, n_D^{20} , 1.5867, M_D (calcd.) 63.76, M_D (obs.) 64.49.

Anal. Calcd. for C₁₄H₁₆O: OCH₃, 15.5; C, 84.00; H, 8.00. Found: OCH₃, 15.24; C, 84.14; H, 7.98.

α -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.

α -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 C₁₄H₁₄O₂: C, 78.50; H, 6.55; neut. equiv., 214. Found: C, 78.88; H, 6.63; neut. equiv., 215.

CHEMISTRY DEPARTMENT
UNIVERSITY OF CHICAGO
CHICAGO, ILLINOIS

RICHARD D. KLEENE

RECEIVED SEPTEMBER 25, 1941

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 C₈H₁₃OCl: Cl, 21.82. Found: Cl, 22.04.

2211 BURLING STREET
CHICAGO, ILLINOIS

RICHARD D. KLEENE

RECEIVED SEPTEMBER 25, 1941

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 alcohol¹ 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).