low-boiling petroleum ether; m. p. 151–153°. A mixed melting point with an authentic sample of mesitoic acid was not depressed. The yield appeared to be practically quantitative.

Reaction with Mesitylmagnesium Bromide.—The Grignard reagent, made from 1 g. of magnesium and 8.8 g. of bromomesitylene in 25 cc. of ether, was added dropwise to 1 g. of mesitoic anhydride dissolved in 10 cc. of ether and 25 cc. of benzene. As the drops of Grignard reagent struck the nitrile solution, a dark red coloration was produced. After being stirred for ten minutes, the reaction mixture was decomposed with ice and acetic acid. The organic layer was separated and washed with water and dilute hydrochloric acid. It was then steam distilled; the residual material was recrystallized from alcohol. It melted at 136–137° and proved to be dimesityl ketone

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2-Methyl-5-hydroxybenzimidazole

Five grams of 3,4-di-(acetylamino)-1-acetoxybenzene in a mixture of 100 cc. of alcohol and 7.5 cc. of concentrated hydrochloric acid was refluxed for two hours. The alcohol was removed by distillation, the residue dissolved in 50 cc. of water, neutralized with sodium carbonate, and cooled. The solid (2.3 g.) was crystallized from 1% sodium hydrosulfite, using decolorizing charcoal. One and three-tenths grams of white crystals was obtained. The dried compound (monohydrate of 2-methyl-5-hydroxybenzimidazole) lost water at 110–115°, resolidified and melted at 187–188°. When dried to constant weight *in vacuo*, at 100°, the anhydrous compound melted at 187.5–188.5°.

Anal. Calcd. for $C_8H_8ON_2 \cdot H_2O$: N, 16.86; H_2O, 10.84. Found: N, 17.03; H_2O, 11.06. Calcd. for $C_8H_8ON_2$: N, 18.91. Found: N, 18.77.

This compound was also prepared from 1-acetylamino-2-amino-4-acetoxybenzene by refluxing 10 g. in a mixture of 20 cc. of concentrated hydrochloric acid and 10 cc. of water for two hours.

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Ethyl γ-(2-Carboethoxy-2-cyclohexanone)-butyrate and Some Related Compounds

Alkylation of 2-carboethoxycyclohexanone with ethyl γ-bromobutyrate by the standard procedure for β-ketonic esters[1] gave ethyl γ-(2-carboethoxy-2-cyclohexanone)-butyrate in 48.5% yield; b. p. 166–168° at 2 mm.; $n^{20}{}_D$ 1.4630.

Anal. Calcd. for $C_{15}H_{24}O_5$: C, 63.40; H, 8.45. Found: C, 62.99; H, 8.64.

This keto-ester gave a 2,4-dinitrophenylhydrazone derivative which after two recrystallizations from 60% alcohol melted at 84–85°.

Anal. Calcd. for $C_{21}H_{28}O_8N_4$: C, 54.30; H, 6.03. Found: C, 54.15; H, 6.04.

Hydrolysis of this ester with dilute sodium hydroxide followed by treatment with acid and loss of carbon dioxide gave the known[2] γ-(2-ketocyclohexanyl)-butyric acid, m. p. 59–60° in 61% yield.

Reduction of 55 g. of the keto-ester in 75 cc. of absolute alcohol over Raney nickel[3] at 125° and 2500 lb. pressure gave 40.5 g. of the corresponding hydroxy ester; b. p. 164–166° at 2 mm.; $n^{20}{}_D$ 1.4672.

Anal. Calcd. for $C_{15}H_{26}O_5$: C, 63.00; H, 9.10. Found: C, 62.93; H, 9.01.

A Zerewitinoff determination showed 0.982 active hydrogens per mole.

2,4-Dinitrophenylhydrazone of Cyclopentylidenecyclopentanone.—Cyclopentylidenecyclopentanone prepared by the method of Wallach[4] gave a brick-red 2,4-dinitrophenylhydrazone which after crystallization from alcohol melted at 228–229°.

Anal. Calcd. for $C_{16}H_{18}N_4O_4$: C, 58.25; H, 5.45. Found: C, 58.33; H, 5.53.

1-(Hydrindylidene)-4-pentene.—Condensation of 66 g. of α-hydrindone and the Grignard reagent from 1-bromo-4-pentene[5] gave 53 g. of the corresponding tertiary alcohol which could not be distilled without loss of water. Distillation gave 48.5 g. of the diene, b. p. 119–121° at 3 mm.; $n^{22}{}_D$ 1.5518.

Anal. Calcd. for $C_{14}H_{16}$: C, 91.30; H, 8.70. Found: C, 90.65; H, 9.19.

1-(3-Phenylhydrindylidene)-4-pentene.—Similarly from 3-phenylindanone there was obtained 1-(3-phenylhydrindylidene)-4-pentene; b. p. 190–191° at 5 mm.; $n^{20}{}_D$ 1.6110.

Anal. Calcd. for $C_{20}H_{20}$: C, 92.40; H, 7.60. Found: C, 92.40; H, 7.77.

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[1] See "Organic Syntheses," Coll. Vol. I, John Wiley and Sons, Inc., New York, N. Y., 1932, p. 243.

[2] Hückel and Naab, *Ann.*, **502**, 136 (1933).

[3] Covert and Adkins, THIS JOURNAL, **54**, 4116 (1932).

[4] Wallach, *Ber.*, **29**, 2963 (1896).

[5] Gaubert, Linstead and Rydon, *J. Chem. Soc.*, 1972 (1937).

RECEIVED JULY 18, 1941

Hexacyclohexoxydisiloxane, $(C_6H_{11}O)_3Si\text{-}O\text{-}Si(C_6H_{11}O)_3$

In connection with the preparation of the ethyl esters derived from some of the silicon oxyhalides, described by us elsewhere in THIS JOURNAL,[1] the extension of the work to include other silicon esters has been undertaken in this Laboratory. The first of these esters obtained was prepared from cyclohexanol and Si_2OCl_6.

To 11 g. of Si_2OCl_6 (b. p. 137°) dissolved in 50 cc. of anhydrous ether in a round-bottomed flask, 27 g. of cyclohexanol was added slowly and, after complete addition, the

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[1] Schumb and Holloway, THIS JOURNAL, **63**, 2753 (1941).