The compound forms long, flexible, colorless needles which may be grouped in sheaves. It is easily soluble in cold chloroform, insoluble in aqueous acids or bases. It does not reduce permanganate and the usual sterol reactions are negative.

From other fluorescing zones five more colorless, crystallized substances were obtained in small quantities (1–3 mg. per 30 kilos of lignite). The melting points were: 261–264°, 273–274°, 238–240°, 297–298° and 249–251°.

We wish to express our sincere thanks to Professor L. C. Harrington of the University of North Dakota for the raw material he kindly placed at our disposal.

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RECEIVED JULY 5, 1941

NEW COMPOUNDS

N-Alkyl Anthraquinone-β-sulfonanilides

Anthraquinone-β-sulfonanilide.—This sulfonanilide was prepared essentially by the method of MacHoul.[1]

An intimate mixture of 2 g. of Eastman Kodak Co. technical sodium anthraquinone-β-sulfonate and 5 g. of phosphorus pentachloride was heated under reflux in an oil-bath at 150° for thirty minutes. The sulfonyl chloride formed was extracted with boiling toluene and the hot extract, after being filtered and cooled, was washed with several portions of water. After adding 3 cc. of aniline to the toluene solution and cooling, a gelatinous precipitate formed. This was filtered off and the filtrate was evaporated to about one-third of the original volume to give, upon cooling, 1.2 g. of yellowish-brown crystals of anthraquinone-β-sulfonanilide. Washing with several portions of a 1:1 mixture of methanol and water produced crystals sufficiently pure, in practically all cases, for the purpose desired. Greater purity, m. p. 192.5-193.5° (MacHoul[2] gives 193°), may be obtained by crystallization from toluene.

N-Alkyl-β-sulfonanilides.—These derivatives were prepared from the potassium salt of anthraquinone-β-sulfonanilide, readily produced by adding a slight excess of 5% alcoholic potassium hydroxide to a boiling solution of the sulfonanilide in toluene. The insoluble brownish-red salt was filtered while hot and washed with several portions of boiling toluene before use. To 1 g. of the dry salt in a round-bottomed flask was added 0.5 to 1 cc. of the alkyl halide (the larger quantity for the more volatile halides) and 1 cc. of water. Upon refluxing gently for an hour and cooling, the derivative separated out in plates or needles. The solid was washed with 10 cc. of aqueous 5% potassium hydroxide and then with water until the red color disappeared. Further purification was achieved by dissolving the dried compound in a minimum amount of boiling toluene and throwing it out of solution with about 10 cc. of ethanol. The solid derivative was finally washed with several portions of methanol and dried. Yields varied from about 0.05 to 0.1 g. for the iso and secondary to about 0.3 g. for the primary halides.

TABLE I

Derivative[a]	Formula	M. p., °C. cor.	Nitrogen, % Calcd.	Found	
Ethyl (I, Br)	$C_{22}H_{17}O_4NS$	192.0–193.0	3.58	3.57	3.52
Propyl (I, Br)	$C_{23}H_{19}O_4NS$	206.5	3.46	3.46	3.38
Isopropyl (I, Br)	$C_{23}H_{19}O_4NS$	256.0	3.46	3.43	3.47
Butyl (Br)	$C_{24}H_{21}O_4NS$	172.5–173.0	3.34	3.36	3.24
Isobutyl (Br)	$C_{24}H_{21}O_4NS$	210.5–211.0	3.34	3.38	3.44
s-Butyl (Br)	$C_{24}H_{21}O_4NS$	214.5–215.5	3.34	3.25	3.22
Amyl (Br)	$C_{25}H_{23}O_4NS$	153.0–154.0	3.23	3.20	3.17
Isoamyl (Br)	$C_{25}H_{23}O_4NS$	172.0–173.0	3.23	3.23	3.21
Hexyl (Br)	$C_{26}H_{25}O_4NS$	145.5–146.0	3.13	3.13	3.17
Heptyl (Br)	$C_{27}H_{27}O_4NS$	141.0–141.5	3.04	2.98	2.94
Benzyl (Cl)	$C_{27}H_{19}O_4NS$	194.0–195.0	3.09	3.08	3.01
Allyl (Br)	$C_{23}H_{17}O_4NS$	194.0–194.5	3.47	3.39	3.49

[a] Halide used in parentheses.

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RECEIVED AUGUST 16, 1941

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[1] MacHoul, *Ber.*, **13**, 692 (1880).

[2] Although MacHoul does not state which anthraquinone sulfonanilide he prepared, his compound was apparently the β since the α form melts at 216° (cor.), Ullmann and Kertesz, *ibid.*, **52**, 551 (1919).

Mesitoic Anhydride

In an attempt to prepare mesitoyl cyanide, mesitoyl chloride was heated with potassium cyanide in the presence of pyridine. A compound was isolated in good yield (73%) but was found to contain no nitrogen. Investigation showed this compound to be mesitoic anhydride. The same compound was obtained by the interaction of sodium mesitoate and mesitoyl chloride in the presence of pyridine.

Ten grams of sodium mesitoate, 10 g. of mesitoyl chloride, and 75 cc. of pyridine were heated in an oil-bath at 150°. The reaction mixture was stirred and refluxed for two hours. It was cooled, diluted with benzene and poured into water. The benzene solution was washed repeatedly with a dilute solution of hydrochloric acid. It was then washed successively with water, dilute potassium bicarbonate solution and water. The benzene solution was dried over calcium chloride, treated with norite and distilled under slightly reduced pressure. The residue solidified and was crystallized from a mixture of benzene and low-boiling petroleum ether; m. p. 106–107°; yield 9 g.

Anal.[1] Calcd. for $C_{20}H_{22}O_3$: C, 77.42; H, 7.10. Found: C, 77.69; H, 7.20.

Hydrolysis.—About 200 mg. of the compound was heated with 30 cc. of 10% sodium hydroxide solution. When the material did not appear to react readily, 20 cc. of ethanol was added to increase its solubility. After two hours of refluxing, all of the oily droplets had disappeared. The mixture was cooled, filtered and made acid with concentrated hydrochloric acid. A white solid separated from the solution and was recrystallized several times from

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[1] Microanalysis by Mr. L. G. Fauble and Miss Mary S. Kreger.

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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RECEIVED JULY 14, 1941

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.; $n^{20}\text{D}$ 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.; $n^{20}\text{D}$ 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.; $n^{22}\text{D}$ 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.; $n^{20}\text{D}$ 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).

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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).