

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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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° (MacHoul² 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 ₄ NS	192.0-193.0	3.58	3.57 3.52
Propyl (I, Br)	C ₂₃ H ₁₉ O ₄ NS	206.5	3.46	3.46 3.38
Isopropyl (I, Br)	C ₂₃ H ₁₉ O ₄ NS	256.0	3.46	3.43 3.47
Butyl (Br)	C ₂₄ H ₂₁ O ₄ NS	172.5-173.0	3.34	3.36 3.24
Isobutyl (Br)	C ₂₄ H ₂₁ O ₄ NS	210.5-211.0	3.34	3.38 3.44
<i>s</i> -Butyl (Br)	C ₂₄ H ₂₁ O ₄ NS	214.5-215.5	3.34	3.25 3.22
Amyl (Br)	C ₂₅ H ₂₃ O ₄ NS	153.0-154.0	3.23	3.20 3.17
Isoamyl (Br)	C ₂₅ H ₂₃ O ₄ NS	172.0-173.0	3.23	3.23 3.21
Hexyl (Br)	C ₂₆ H ₂₅ O ₄ NS	145.5-146.0	3.13	3.13 3.17
Heptyl (Br)	C ₂₇ H ₂₇ O ₄ NS	141.0-141.5	3.04	2.98 2.94
Benzyl (Cl)	C ₂₇ H ₁₉ O ₄ NS	194.0-195.0	3.09	3.08 3.01
Allyl (Br)	C ₂₅ H ₁₇ O ₄ NS	194.0-194.5	3.47	3.39 3.49

^a Halide used in parentheses.

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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 C₂₀H₂₂O₃: 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

(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).

(1) Microanalysis by Mr. L. G. Fauble and Miss Mary S. Kreger.