hydroquinone) showed only slight decomposition. It does not react in ether with ordinary or activated magnesium. A vigorous reaction takes place with sodium, and this is being investigated. From other studies it appears that alkyl groups labilize furan nuclear halides as, for example, the β-iodine in 2,5-dimethyl-3-iodofuran.

In the reduction of 5-nitro-2-furfural, copper bronze in quinoline in place of potassium hydroxide was used for the decomposition of the hydrazone. The 2-methyl-5-nitrofuran was shown to be identical, by the method of mixed melting points, with an authentic specimen.[5] The copper bronze–quinoline procedure for removal of nitrogen is novel, we believe, and its application was indicated because of the marked sensitivity of α-nitrofurans to strong alkalies.

CONTRIBUTION FROM THE
CHEMICAL LABORATORY OF
IOWA STATE COLLEGE
AMES, IOWA

RECEIVED JUNE 22, 1932
PUBLISHED OCTOBER 5, 1932

Phenanthrene-1-carboxylic Acid

BY LOUIS F. FIESER

Following the request of Dr. E. Mosettig for a sample of the above substance with which to compare an isomer isolated in the Virginia Laboratory,[1] this new phenanthrene derivative was prepared from the 1-sulfonic acid,[2] a small sample of which was still available.

Phenanthrene-1-nitrile, prepared from 1 g. of potassium phenanthrene-1-sulfonate and 2 g. of potassium ferrocyanide, was obtained in a pure condition (yield 0.4 g.) by distillation *in vacuo* and crystallization from alcohol. It formed stout, colorless needles melting at 128°.

Anal. Calcd. for $C_{15}H_9N$: C, 88.64; H, 4.47. Found: C, 88.50; H, 4.58.

Phenanthrenequinone-1-nitrile resulted from the oxidation of the phenanthrene derivative with chromic acid at 90°. It is sparingly soluble in glacial acetic acid and separates as fine, orange needles melting at 339°.

Anal. Calcd. for $C_{15}H_7O_2N$: C, 77.24; H, 3.03. Found: C, 77.30; H, 3.25.

Phenanthrene-1-carboxylic acid amide was obtained by boiling the corresponding nitrile with alcoholic alkali for two hours. It formed colorless plates, m. p. 284°, from dilute acetic acid.

Anal. Calcd. for $C_{15}H_{11}ON$: C, 81.41; H, 5.01. Found: C, 81.50; H, 5.26.

Phenanthrene-1-carboxylic Acid.—The amide is hydrolyzed only with difficulty. The reaction with alcoholic alkali was not complete even after heating for several hours at 160°. The acid is readily soluble in alcohol or benzene and crystallizes well from the former solvent as colorless needles melting at 232–233°.

Anal. Calcd. for $C_{15}H_{10}O_2$: C, 81.06; H, 4.54. Found: C, 81.22; H, 4.56.

The methyl ester, prepared with diazomethane, is very readily soluble even in cold organic solvents. Purified by distillation, it melted at 57°.

CHEMICAL LABORATORY
HARVARD UNIVERSITY
CAMBRIDGE, MASSACHUSETTS

RECEIVED JUNE 21, 1932
PUBLISHED OCTOBER 5, 1932

---

[1] Mosettig and van de Kamp, THIS JOURNAL, **54**, 3328 (1932).

[2] Fieser, *ibid.*, **51**, 2460 (1929).