lines of cupric oxychloride, and of atacamite, but noticeably broad, were found for these cements, indicating that cupric oxychloride had been formed in appreciable amounts, but in very small, almost colloidal, particles. The amount of this compound is probably somewhat greater in the aged cement than in the unaged material. On the other hand, it is evident that cupric oxychloride has not formed a complex compound with magnesium oxychloride nor entered appreciably into solid solution in it.

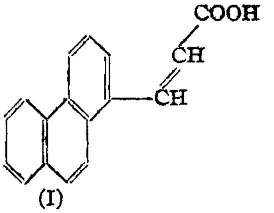
It has been observed that the formation of the new phase takes place at the expense of the copper particles and that it cannot proceed in the absence of oxygen or of water vapor. Exposure to sunlight appears to retard and strong ultraviolet light prevents its formation.

MELLON INSTITUTE
PITTSBURGH, PENNA.
RECEIVED NOVEMBER 2, 1936

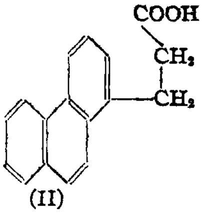
Rapid Synthesis of β-(1-Phenanthryl)-propionic Acid

BY SAMUEL NATELSON AND SIDNEY P. GOTTFRIED

1-Phenanthraldehyde, prepared by the method of Bachmann,[1] was converted rapidly in good yield to β-(1-phenanthryl)-propionic acid after the method described by the authors for a similar series.[2] 1-Phenanthraldehyde, which condenses with acetic anhydride and sodium acetate in extremely poor yield after the usual Perkin reaction, condenses almost quantitatively with malonic acid, with the elimination of carbon dioxide. The β-(1-phenanthryl)-acrylic acid formed (I) may be reduced to yield β-(1-phenanthryl)-propionic acid (II).[3] This acid has been prepared before by Bachmann[1] by a more tedious route in an attempt to synthesize 3′-keto-1,2-cyclopentenophenanthrene.



(I)



(II)

Ten grams of 1-phenanthraldehyde is mixed with 4 g. of malonic acid (excess) and 0.5 cc. of pyridine. The mixture is heated on a water-bath for thirty minutes, when effervescence ceases and the whole mass solidifies. The β-(1-phenanthryl)-acrylic acid, formed in almost quantitative yield, is washed with dilute acid to remove the pyridine and is recrystallized from acetone, m. p. 259°. This acid dissolved in dilute potassium hydroxide, is reduced with an excess of 3% sodium amalgam to yield 9–11 g. of β-(1-phenanthryl)-propionic acid (II), m. p. 187–188° (from acetone). It seems curious to the authors that reduction at the 9,10-positions of the phenanthrene nucleus was not brought about by the sodium amalgam.

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(1) Bachmann, THIS JOURNAL, **57**, 1383 (1935); **58**, 2097 (1936).

(2) Natelson and Gottfried, *ibid.*, **58**, 1432 (1936).

(3) In a private communication, the authors have been informed by Bachmann that he corroborates these observations and has also successfully applied this series of reactions to several phenanthryl aldehydes.

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COMMUNICATION TO THE EDITOR

SYNTHETIC VITAMIN B_1

Sir:

In our last communication [THIS JOURNAL, **58**, 1504 (1936)] we reported synthetic vitamin B_1 chloride melting at 232–234° rather than at 246–250° as previously given for the natural product. We have, however, using our previous method but by a different choice of solvents for purification, obtained, in good yield, synthetic vitamin B_1 chloride which agrees in every particular with the natural vitamin. We owe thanks to Dr. G. A. Stein of the Merck Laboratories for his collaboration in this matter.

A low melting point has also been observed in the bromide. In both chloride and bromide the low melting point is associated with a much greater solubility in alcoholic solvents. Both low melting salts are obtained from methanol solutions by addition of ether. However, when recrystallized from methanol by addition of ethanol or from water by addition of ethanol, the higher melting forms have been obtained. The discrepancies in melting