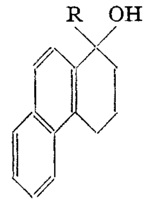
67 g. of the anilide. Reaction of the acid anilide with phosphorus pentachloride followed by reduction of the product by stannous chloride and hydrogen chloride in absolute ether yielded the new phenanthrene aldehyde 1-phenanthraldehyde (m. p. 110–111°; yield, 65%). These reactions now make it possible to prepare many 1-substituted phenanthrene derivatives hitherto difficult to obtain, and we are engaged in the preparation of such compounds. Reduction of the aldehyde to the carbinol (m. p. 165°), followed by a malonic ester condensation with the carbinol bromide (m. p. 97°) gave $\beta$-(1-phenanthryl)-propionic acid (m. p. 189–189.5°); experiments on the cyclization of this acid are not yet complete.

The excellent researches of Cook and coworkers [Cook, Dodds, Hewett and Lawson, *Proc. Roy. Soc.* (London), **114**, 278 (1934)] have shown that oestrogenic property is possessed by a variety of polycyclic compounds; and we are having the keto-cyclopentenophenanthrenes and derivatives tested for this property. We have also prepared three 1-hydroxy-1-alkyl-1,2,3,4-tetrahydrophenanthrenes (III) in crystalline state in which the alkyl group R is methyl (m. p. 86–86.5°), ethyl (m. p. 57–57.5°) and *n*-propyl (m. p. 86–87°); these compounds likewise are being tested for oestrogenic activity.



(III)

The physiological tests are being made by Dr. James Bradbury at the University of Michigan Hospital. The details of the experiments will be published later.

CHEMISTRY LABORATORY
UNIVERSITY OF MICHIGAN
ANN ARBOR, MICHIGAN
W. E. BACHMANN

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## THE PREPARATION OF MONOCHLOROACETOCATECHOL

*Sir:*

In the synthesis of adrenaline according to the method of Stolz [*Ber.*, **37**, 4149 (1904)] the preparation of monochloroacetocatechol is necessary. Chloroacetic acid, catechol and phosphorus oxychloride are mixed together and refluxed on the steam-bath. At the end of an hour the material in the flask is recrystallized from hot water. Using this method as it stands, repeated preparation shows large amounts of tars which obliterate much of the yield. Oxidation of the catechol must also be repressed for greater yields. To carry out this purpose the following method was used.

Fifty grams each of monochloroacetic acid, catechol, and *freshly distilled* phosphorus oxychloride were mixed together in a one-liter short-necked flask fitted with reflux condenser and a tube through which sulfur dioxide was passed. A tube was attached to the end of the reflux condenser to lead away hydrogen chloride gas evolved in the reaction, and the sulfur dioxide. After the flask was filled with the sulfur dioxide from a tank, heating was started, a steady stream of gas always flowing. At the end of an hour heating was stopped, the purple crystals of crude chloroacetocatechol crystallized from hot water, decolorized with acid, Norite and sodium sulfite, and again crystallized. The light violet tinged crystals were washed with absolute alcohol, and finally ether. The yield was 53.6 g., m. p. 173°. Without these modifications, namely, the sulfur dioxide atmosphere and freshly distilled phosphorus oxychloride, the yield was 8.3 g.

COLUMBIA UNIVERSITY
NEW YORK CITY
HENRY D. HOBERMAN

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## THE RAMAN SPECTRUM OF RUBBER

*Sir:*

It has been previously reported that the Raman effect is exhibited by rubber as a continuous scattering together with the presence of broad bands [Franklin and Laird, *Phys. Rev.*, **36**, 147 (1930); Busse, *J. Phys. Chem.*, **36**, 2862 (1932)]. Busse attributed this result to the viscosity of the solutions or to the possibility that the rubber groups respond to a wide range of frequencies. However, viscous solutions of polysterol in carbon tetrachloride give a line spectrum [Signer and Weiler, *Helv. Chim. Acta*, **15**, 649 (1932)]. As to the second explanation, this does not seem to be the nature of the Raman effect [Bär, *Helv. Phys. Acta*, **4**, 369 (1931); Bär, *Z. Physik*, **79**, 455 (1932)]. The Raman effect has been investigated extensively for terpenes other than rubber.

The bands and continuous background for rubber appear to be due to fluorescence of im-