

methyltestosterone acetate (If acetate) (m.p. 171–173°, $[\alpha]_D +44^\circ$, λ_{\max} . 240 $m\mu$, $\log \epsilon$ 4.19. Found: C, 77.23; H, 9.81).

While anti-tumor screening of the above described 2-methyl hormones is still in progress, Ia and IIa have already been shown to be very effective tumor inhibitors.

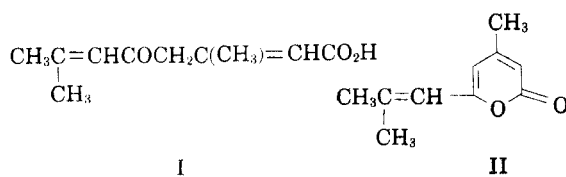
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2-Pyrones. XXIII. 4-Methyl-6-(2'-methylpropenyl)-2-pyrone

Sir:

We wish to report the synthesis of a new ten carbon isoprenoid lactone which is of interest as a simple multiple of senecioic acid in studies of the biosynthesis of cholesterol from acetate.¹⁻⁷ 4-Methyl-6-(2'-methylpropenyl)-2-pyrone (II), the lactone of the enol form of γ -seneciolsenecioic acid (I) has been prepared by the acylation of β -methylglu-



taconic anhydride with senecieryl chloride followed by decarboxylative rearrangement. This is a modification of a synthetic route previously described,⁸ but successfully applied here for the first time to an aliphatic acid chloride having over four carbon atoms.

A solution of β -methylglutaconic anhydride in pyridine and ether was treated with senecieryl chloride. Ether extraction of the acidified reaction mixture gave a red oil which was decarboxylated by flash distillation and refractionated to give 12% yield of 4-methyl-6-(2'-methylpropenyl)-2-pyrone, m.p. 46.5–47.5°, (*Anal.* Calc'd for $\text{C}_{10}\text{H}_{12}\text{O}_2$: C, 73.14; H, 7.37. Found: C, 73.08; H, 7.37) showing the 2-pyrone carbonyl absorption band at 1730 cm^{-1} and the trisubstituted ethylenic absorption band at 840 cm^{-1} . Reaction with bromine gave 3-bromo-4-methyl-6-(2'-methyl-2',3'-dibromopro-

pyl)-2-pyrone, m.p. 119–120°. (*Anal.* Calc'd for $\text{C}_{10}\text{H}_{11}\text{Br}_3\text{O}_2$: C, 29.80; H, 2.75. Found: C, 29.88; H, 3.10) showing the 2-pyrone carbonyl absorption band at 1724 cm^{-1} shifted slightly as with other 3-substituted types.⁹

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Ozonolysis of Phenanthrene in Chloroform

Sir:

Schmitt, Moriconi, and O'Connor¹ recently claimed the preparation of the first stable monomeric ozonide of an aromatic hydrocarbon. The material was obtained by the ozonolysis of phenanthrene in either chloroform or acetic acid. It melted at 65–90°. It was assigned a monoozonide structure on the basis of elementary analyses, a Rast molecular weight determination, catalytic hydrogenation to 2,2'-biphenyldicarboxaldehyde, and infrared spectra which showed strong bands in the region 5.7–5.9 μ , which Briner² had originally ascribed to ozonides.

Criegee³ has shown that pure simple ozonides, such as the monoozonide of phenanthrene would be, do not absorb in the 5.6–6.2 μ region, which is the carbonyl region. Briner⁴ has recently acknowledged the findings of Criegee and ascribed his results to the formation of aldehydes or ketones during the passage of ozone through the reaction mixture.

We have ozonized phenanthrene (5.9 g.) in chloroform (60 ml.) at -60° and have immediately precipitated the product (7.3 g., 98% yield, m.p. 129–130°) by addition of either ligroin or methanol. Several recrystallizations from benzene by addition of ligroin gave an 80% recovery of material melting at 139–141° (*Anal.* Calcd. for $\text{C}_{14}\text{H}_{10}\text{O}_3$: C,⁵ 74.33; H, 4.46; Active O, 7.07. Found: C, 74.58; H, 4.80; Active O, 6.95). The material was

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(4) Briner and Dallwigk, *Compt. rend.*, **243**, 630 (1956); *Helv. Chim. Acta*, **39**, 1446 (1956).

(5) Schmitt, Moriconi, and O'Connor¹ erred in this calculation. Their product, therefore, analyzed 1% low in carbon.