none (I, R = C_6H_5). It was recrystallized from glacial acetic acid, xylene or butanol and melted at 158°.

Anal. Calcd. for $C_{25}H_{16}O$: C, 90.4; H, 4.8. Found: C, 90.2; H, 4.8.

The yellow **phenylhydrazone**, recrystallized from xylene, melted at 236°.

Anal. Calcd. for $C_{31}H_{22}N_2$: N, 6.6. Found: N, 7.0.

(d) **β-Acetylnaphthalene.**—Analogously, 3.4 g. of β-acetylnaphthalene gave an almost quantitative yield of the yellow α-(2-naphthoyl)-β-(3′-pyrenyl)-ethylene (I, R = β-naphthyl). Recrystallization from xylene or glacial acetic acid gave crystals, melting at 184°.

Anal. Calcd. for $C_{29}H_{18}O$: C, 91.1; H, 4.7. Found: C, 90.9; H, 4.6.

The orange-colored **phenylhydrazone** crystallized from xylene, and melted at 246°.

Anal. Calcd. for $C_{35}H_{24}N_2$: N, 5.9. Found: N, 6.2.

9-(3′-Pyrenylidene)-fluorene.—The condensation of 4.6 g. of the aldehyde with 3.3 g. of fluorene was carried out, using sodium ethoxide as catalyst.[3] The yellow-orange fulvene (II) separated spontaneously from the solution, and melted after recrystallization from xylene, butanol or glacial acetic acid at 210°.

Anal. Calcd. for $C_{30}H_{18}$: C, 95.2; H, 4.8. Found: C, 94.9; H, 5.0.

α-Phenyl-β-(3-pyrenyl)-acrylic Acid (III, R = C_6H_5).—One mole each of pyrene-3-aldehyde and sodium phenylacetate was heated in boiling acetic anhydride (1.5 to 2 moles) for two hours. After cooling, the reaction mixture was diluted with water, and the solid residue filtered and washed with alcohol. From glacial acetic acid, the acid was obtained in yellow needles of m. p. 259°; yield, 30%.

Anal. Calcd. for $C_{25}H_{16}O_2$: C, 86.2; H, 4.6. Found: C, 86.5; H, 4.9.

α-(1-Naphthyl)-β-(3′-pyrenyl)-acrylic acid (III, R = 1-naphthyl) was obtained analogously from pyrene-3-aldehyde and sodium 1-naphthylacetate; yellow needles of m. p. 290° (from nitrobenzene).

Anal. Calcd. for $C_{29}H_{18}O_2$: C, 87.4; H, 4.5. Found: C, 87.1; H, 4.7.

α-(3-Pyrenyl)-β-(2′-pyridyl)-ethylene Methiodide (IV).—To a solution of pyrene-3-aldehyde (4 g.) and α-picoline methiodide (4 g.) in a small quantity of absolute alcohol, 10 to 15 drops of piperidine was added at 180°. Heating was continued for thirty minutes; then the red precipitate was filtered, washed with alcohol, dried, and recrystallized from nitrobenzene: m. p. 295°; yield, 65%.

Anal. Calcd. for $C_{24}H_{18}IN$: C, 64.4; H, 4.0; N, 3.1. Found: C, 64.1; H, 4.0; N, 3.3.

α-(3-Pyrenyl)-β-(6′-methyl-pyridyl-2′)-ethylene methiodide was prepared in 70% yield from 2.3 g. of pyrene-3-aldehyde and 4 g. of 2,6-lutidine methiodide, as described above. It was recrystallized from nitrobenzene, and formed red shiny crystals of m. p. 275°.

Anal. Calcd. for $C_{25}H_{20}IN$: N, 3.0. Found: N, 3.1.

α-(3-Pyrenyl)-β-(2′-quinolyl)-ethylene methiodide was formed in 75% yield as a red-violet, insoluble substance from 2.3 g. of pyrene-3-aldehyde and 2.8 g. of quinaldine methiodide; m. p. 285°.

Anal. Calcd. for $C_{28}H_{20}IN$: N, 2.8. Found: N, 2.8.

sym-**Di-(3-pyrenyl)-ethylene** (V).—(a) 10 g. of pyrene-3-aldehyde was dissolved in 100 ml. of dry benzene. Dry hydrogen chloride and hydrogen sulfide were passed simultaneously through the cold solution. The yellow precipitate was filtered and washed with water, alcohol and ether. It could not be recrystallized, as it decomposed on heating in high-boiling solvents. In its crude state the polymeric pyrene-3-thioaldehyde melted at 260–270°.

Anal. Calcd. for $(C_{17}H_{10}S)_x$: C, 82.9; H, 4.1. Found: C, 82.5; H, 4.4.

(b) A mixture of 2 g. of the thioaldehyde, 1 g. of Raney nickel and 20 g. of boiling naphthalene was heated for one hour. After removal of the naphthalene *in vacuo*, a brown-red residue was obtained, which was washed with alcohol and recrystallized from ethyl benzoate: m. p. 315°; yield, 75% (calculated on pyrene-3-aldehyde).

Anal. Calcd. for $C_{34}H_{20}$: C, 95.3; H, 4.7. Found: C, 95.1; H, 4.8.

DEPARTMENT OF ORGANIC CHEMISTRY
THE HEBREW UNIVERSITY, JERUSALEM

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(3) Schlenk and Bergmann, *Ann.*, **479**, 56 (1930).

NEW COMPOUNDS

Hexahydrojulolidine

To a solution of julolidine,[1] 1,2,3,5,6,7-hexahydrobenzo-(i,j)-quinolizine (20 g., 0.115 mole), in alcohol (20 ml.) there was added Raney nickel (2 g.) and the mixture was shaken at 200° under a pressure of 2000 lb. of hydrogen. After removal of solvent and catalyst, the residue was distilled yielding 5 g. (low yield due to accidental loss) of a colorless oil: b. p. 76–80° at 3 mm.

Anal. Calcd. for $C_{12}H_{21}N$: C, 80.44; H, 11.73. Found: C, 80.55; H, 11.45.

The **picrate** of hexahydrojulolidine was readily prepared using an ethereal solution of picric acid. After recrystallization from hot water the picrate was obtained as an amorphous yellow solid, m. p. 165–167°.

Anal. Calcd. for $C_{18}H_{24}N_4O_7$: C, 52.94; H, 5.88. Found: C, 52.82; H, 5.69.

The **methiodide** of hexahydrojulolidine formed readily and could be recrystallized from a solution of alcohol and ether. It was obtained as an amorphous white solid, m. p. 294–298° with decomposition.

Anal. Calcd. for $C_{13}H_{24}IN$: C, 48.50; H, 7.47. Found: C, 48.51; H, 7.45.

Hexahydrojulolidine hydrochloride and also the quaternary methiodide derivative were found to be convulsants when tested intravenously in rabbits.

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(1) Glass and Weissberger, "Organic Syntheses," **26**, 40 (1946).

(2) Aided by a grant from the National Foundation for Infantile Paralysis, Inc.

Some Derivatives of Levulinic Acid

Levulinic acid diethylamide was prepared (a) by heating 50 g. of levulinic acid with 50 g. of diethylamine at 250° for ten hours (autoclave), yield 60%; and (b) by adding 13.4 g. of γ-chlorovaleroactone[1] to a cold solution of 15 g. of diethylamine in 50 cc. of ether; the reaction was completed on the water-bath and the solution filtered from diethylamine hydrochloride and distilled, yield 70%. The amide is soluble in water and boils at 108° (0.9 mm.); n^{22}_D 1.457.

Anal. Calcd. for $C_9H_{17}O_2N$: C, 63.2; H, 10.0; N, 8.2. Found: C, 63.3; H, 10.2; N, 8.2.

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(1) Clemo and Ramage, *J. Chem. Soc.*, 54 (1931).