

The unsaturated hydrocarbon (5.9 g.) is heated with sulfur (2 g.) at 250° until no more hydrogen sulfide is evolved (one hour) and then distilled. There is obtained 4.5 g. of *o,m'*-ditolyl boiling at 265–267°.

1-Benzoylfluorenone.—The *o,m'*-ditolyl (4 g.) on oxidation with potassium permanganate gives 2.3 g. of *iso*-diphenic acid (m. p. 215–216°) and this is converted into fluorenone-1-carboxylic acid (m. p. 191–193°) by treatment with sulfuric acid.⁹

Fluorenone-1-carboxylic acid (0.35 g.) is refluxed with an excess of thionyl chloride for one hour and then warmed under reduced pressure. The crystalline acid chloride remaining is dissolved in 10 ml. of benzene, and 0.5 g. of aluminum chloride is added. After it has been warmed on a water-bath for ten minutes, the mixture is hydrolyzed with ice and hydrochloric acid. The benzene solution is separated and the solvent is distilled under reduced pressure. The residue is crystallized from alcohol, when there is obtained 0.2 g. of 1-benzoylfluorenone in the form of yellow needles that melt at 114–116°. The compound gives a red-brown color with sulfuric acid.

Anal. Calcd. for C₂₀H₁₂O₂: C, 83.8; H, 4.2. Found: C, 84.2; H, 4.3.

Summary

The halochromic sulfate of α,γ -bisdiphenylene- β -phenylallyl alcohol decomposes in acetic acid solution to give 1-diphenylene-2-phenyl-3,4-phenyleneindene, a hydrocarbon which contains a ring system hitherto considered impossible.

The structure of this hydrocarbon is proved by its degradation through 1-diphenyleneoxymethylfluorenone and *o*-(fluorenone-1-carbonyl)-benzoic acid to 1-benzoylfluorenone.

An independent synthesis of 1-benzoylfluorenone is described.

⁹ Mayer and Freitag, *Ber.*, **54**, 354 (1921).

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COMMUNICATIONS TO THE EDITOR

THE PREPARATION OF *l*-ARABINOSE AND *l*-RIBOSE BY THE OXIDATION OF *l*-ARABINAL WITH BENZOIC PERACID

Sir:

By the reactions *l*-arabinose \longrightarrow acetobromo-*l*-arabinose \longrightarrow diacetyl-*l*-arabinal \longrightarrow *l*-arabinal, employed by Gehrke and Aicher [*Ber.*, **60**, 918 (1927)] and by Meisenheimer and Jung [*ibid.*, **60**, 1462 (1927)] we have prepared 75 g. of crystalline *l*-arabinal, with melting point 78–81° and $[\alpha]_D^{20-25} -192^\circ$, in chloroform. An aqueous solution of 68 g. of this *l*-arabinal was stirred with a solution of benzoic peracid in ethyl acetate according to the general procedure of Levene and Tipson [*J. Biol. Chem.*, **93**, 631 (1931)]. The titration with iodine by the directions of Goebel [*ibid.*, **72**, 801 (1927)] was then equivalent to the oxidation of 78% of the *l*-arabinal to aldopentose. The observed rotation of the solution gave, on the basis of the above yield of aldopentose, the value $[\alpha]_D^{20-25} +49.77^\circ$. This rotation has now been shown to be due largely to the presence of both *l*-arabinose and *l*-ribose in the reaction product.

The solution of the above reaction products was concentrated to a sirup, thinned with absolute alcohol and ether, and yielded 7 g. of crystalline *l*-arabinose. This sugar was recrystallized to 5.5 g. of pure *l*-arabinose, with m. p. 159–160° and $[\alpha]_D^{20-25} +104.6^\circ$, in water. The solution containing the remaining larger portion of the reaction products was again concentrated to a sirup, from which no more *l*-arabinose could be crystallized. After three weeks crystal nuclei appeared spontaneously in this sirup. These were stirred into the sirup after the addition of absolute alcohol. Refrigeration overnight resulted in the formation of 15 g. of crystalline *l*-ribose, with m. p. 81–84° and $[\alpha]_D^{20-25} +23.85^\circ$ (seventeen hours after solution in water). One recrystallization from absolute alcohol has raised its melting point to 85–87°. From the mother liquors an additional 13 g. of *l*-ribose has been secured by direct crystallization, and 8.7 g. more by way of the *p*-bromophenylhydrazone in the method of van Ekenstein and Blanksma [*Chem. Weekblad*, **22**, 373 (1909)]. The purified *p*-bromophenylhydrazone of *l*-ribose melted at 171–172°, while the value given by Levene and Jacobs [*Ber.*, **42**, 2474 (1909)] for the same derivative of *d*-ribose was 170°.

The synthesis of both *d*- and *l*-ribose may now be accomplished by the above reactions more quickly and cheaply than by preparations from any other sources. Our studies, which were supported in part by a Grant-in-aid from the National Research Council, will be more fully described in a future publication.

From the *l*-ribose which has thus been formed we shall undertake the preparation, by the cyanhydrin reaction, of the remaining unknown aldohexoses, *l*-allose and *l*-altrose.

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THE FLUOROCHLORIDES OF SILICON

Sir:

Since 1915 this Laboratory has been engaged in the investigation of the fluorides, especially in Groups III and IV of the periodic table.¹ Unaware of any other investigators working on the problem, we have continued the studies begun in this Laboratory some time ago on the fluorination of

¹ Germann and Booth, *J. Phys. Chem.*, **21**, 92 (1917); Germann and Cleaveland, *Science*, [2] **53**, 582 (1921); Germann and Torrey, *ibid.*, **54**, 16 (1921); Germann and Booth, *J. Phys. Chem.*, **30**, 369 (1926); Booth and Starrs, *ibid.*, **35**, 3553 (1931); Booth and Carter, *ibid.*, **36**, 1359 (1932); Booth, Mong and Burchfield, *Ind. Eng. Chem.*, **24**, 328 (1932); Booth and Bixby, *ibid.*, **24**, 637 (1932); Booth, Swinehart and Morris, *THIS JOURNAL*, **54**, 2561 (1932).