The Physical Constants of Pentanol-3

BY FRANK C. WHITMORE AND J. D. SURMATIS

Pure pentanol-3 was required in large quantity for work in progress in this Laboratory. Since the physical constants for the carbinol reported in the literature[1] show wide disagreement, a study was made on the purity of material synthesized in the laboratory and that obtained from Sharples Solvents Corporation.

Propionaldehyde, b. p. 48.0° at 736 mm., $n^{20}D$ 1.3636, was prepared by dehydrogenation of *n*-propyl alcohol with a copper catalyst. It was treated in four 8-mole lots with ethylmagnesium chloride in anhydrous ethyl ether. The crude product obtained in 67% yield after distillation through a column of approximately 25 theoretical plates, was refractionated through a column, 2 × 260 cm. of the total condensation partial take-off type, having approximately 85 theoretical plates. From this distillation a yield of 90% of constant boiling and constant index material resulted. The boiling point was determined in a laboratory Cottrell apparatus, with a thermometer calibrated against one checked by the Bureau of Standards; the refractive index was determined by a Valentine refractometer: b. p. 114.4° at 740 mm., $n^{20}D$ 1.4104, d^{20}_4 0.8218.

Approximately 2800 g. of Sharples pentanol-3 was distilled through a column of approximately 16 theoretical plates, and then refractionated twice through the 85-plate column described above. Of the starting material 27% was obtained with the physical constants: b. p. 114.3–114.5° at 741.5 mm., $n^{20}D$ 1.4102–1.4104, d^{20}_4 0.8203.

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STATE COLLEGE, PENNA. RECEIVED DECEMBER 26, 1939

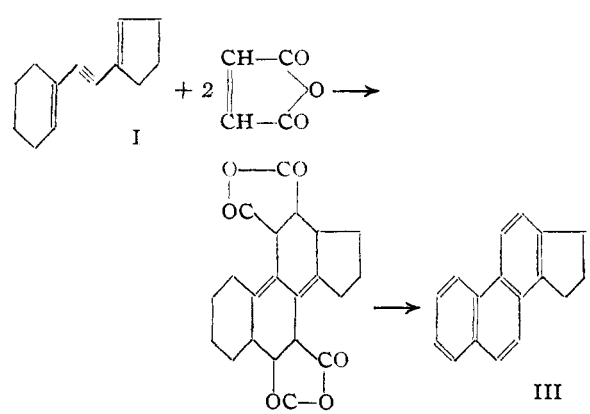
(1) Brunel, THIS JOURNAL, **45**, 1334 (1923); Lucas and Moyse, *ibid.*, **47**, 1460 (1925); Morris and Cortese, *ibid.*, **49**, 2644 (1927); Sherrill Otto and Pickett, *ibid.*, **51**, 3027 (1929); Timmermans and Hennaut-Roland, *J. chim. phys.*, **29**, 529 (1932); Clark and Hallonquist, *Trans. Roy. Soc. Can.*, [3] **24**, 115 (1930); Lauer and Stodola, THIS JOURNAL, **56**, 1216 (1934); Brooks, *ibid.*, **56**, 1998 (1934); Packendorff, *Ber.*, **67**, 905 (1934).

COMMUNICATIONS TO THE EDITOR

THE TOTAL SYNTHESIS OF A NON-BENZENOID STEROID[1]

Sir:

We reported in the last paper[2] that a derivative of hexahydronaphthalene results from the addition of maleic anhydride to 2,5-dimethyl-1,5-hexadiene-3-yne. It has now been found that an analogous reaction occurs when the hydrocarbon I[3] is heated with one mole of maleic anhydride at 130° without solvent. The crystalline product, from ethyl acetate or benzene, has m. p. 249–251° (cor.) with decomposition, and is converted in low yield to 15,16-dihydro-17-cyclopenta[a]phenanthrene (III), m. p. 132–133° (cor.), by heating with palladium–charcoal. This hydrocarbon did not depress the m. p. of an authentic specimen[4] kindly furnished by Dr. Erich Mosettig. *Anal.*[5]



15,16-Dihydro-17-cyclopenta[a]phenanthrene

Calcd. for $C_{21}H_{20}O_6$: C, 68.5; H, 5.5. Found: C, 68.7; H, 5.6. Calcd. for $C_{17}H_{14}$: C, 93.5; H, 6.5. Found: C, 93.5; H, 6.5. Structure II is tentatively assigned to the compound $C_{21}H_{20}O_6$ on the basis of analogy with the hexahydronaphthalene previously[2] described and the absorption curve of the solution in ethanol, λ max. 2555 Å., ε 19,000. It is suggested that a compound of this

(1) This work is supported by Bankhead-Jones funds. (Not subject to copyright.)

(2) Butz, Gaddis, Butz and Davis, *J. Org. Chem.* (recently submitted for publication). The present communication is the fourth paper in the series "Synthesis of Condensed Ring Compounds."

(3) Pinkney, Nesty, Wiley and Marvel, THIS JOURNAL, **58**, 972 (1936).

(4) Burger and Mosettig, *ibid.*, **59**, 1307 (1937).

(5) By Arlington Laboratories, Arlington, Virginia.