the hydrocarbon was recrystallized from benzene-methanol to a melting point of 106.4–107.0°.

*Anal.** Calcd. for $C_{22}H_{20}$: C, 92.91; H, 7.09. Found: C, 92.89, 92.96; H, 7.00, 6.92.

Summary

A new synthesis for hydrocarbons containing the 3,4-benzphenanthrene nucleus is described. The method is based on a double ring closure of β-benzohydrylglutaric acid to yield 2,9-diketo-1,2,9,10,11,12-hexahydro-3,4-benzphenanthrene. By reduction and dehydrogenation this diketone is converted into 3,4-benzphenanthrene, whereas by reaction with methyl- or ethylmagesium bromide followed by dehydration and dehydrogenation, the diketone is converted into 2,9-dimethyl- or 2,9-diethyl-3,4-benzphenanthrene, respectively.

COLUMBUS, OHIO RECEIVED DECEMBER 23, 1937

NOTES

An Obscure Reaction of Phosphorus Trichloride

BY ROBERT D. COGHILL

Two explosions occurring in the elementary organic laboratory this past Fall have prompted me to write this note as a warning to other teachers. The accidents occurred during the preparation of acetyl chloride from phosphorus trichloride and acetic acid. In each case the student was ignoring the printed directions and attempting to distil the acetyl chloride from the phosphorous acid residue with a free flame rather than with a hot water-bath. The phosphorous acid was thus locally overheated and exploded with sparks and a yellow flame, copious white fumes (probably phosphorus pentoxide), and the odor of phosphorus or phosphine.

In attempting to find an explanation for the phenomenon it was found that phosphorus trichloride, when evaporated in an open breaker on a steam-bath, caught fire spontaneously and burned with a yellow flame, leaving a large residue of phosphorus in the beaker. When the evaporation was carried out on an electric hot-plate the material did not burn and only a small residue of phosphorus was obtained. When the phosphorus trichloride was distilled from a distilling flask in a system protected from atmospheric moisture with a calcium chloride tube, only a faint trace of phosphorus remained.

It was thus evident that the flame and the phosphorus residue result from the reaction of phosphorus trichloride and water. Mellor[1] states that when phosphorous acid is heated, phosphine is produced. The latter is then alleged to react with phosphorus trichloride to form free phosphorus and hydrogen chloride. These reactions would serve to explain both the observed reaction of phosphorus trichloride and steam, and the laboratory explosions. In the latter cases, overheating of the phosphorous acid produced phosphine in sufficient quantity to blow out the stoppers, the gas subsequently igniting spontaneously.

(1) J. W. Mellor, "Inorganic and Theoretical Chemistry," Longmans, Green and Co., 1928, pp. 806, 1002–1003.

STERLING CHEMISTRY LABORATORY
YALE UNIVERSITY
NEW HAVEN, CONN. RECEIVED DECEMBER 23, 1937

2,2,3,4-Tetramethylhexane and 3,3,5-Trimethylheptane[1]

BY NATHAN L. DRAKE AND L. H. WELSH

2,2,3,4-Tetramethylhexane (I) and 3,3,5-trimethylheptane (II) have been prepared by the hydrogenation of corresponding olefins. The olefins were obtained by the action of sulfuric acid on methylisopropylcarbinol,[2] and were hydrogenated during passage in a stream of hydrogen over a copper chromite catalyst. Attempts to hydrogenate the olefins in the liquid phase using Adams platinum black catalyst were unsuccessful; a rapid initial absorption of hydrogen was observed but hydrogenation ceased after a few minutes. However, on distilling the olefins over copper chromite[3] with excess hydrogen, saturation proceeded smoothly, and after two such

(1) From the master's thesis of L. H. Welsh, University of Maryland, 1935.

(2) Drake, Kline and Rose, THIS JOURNAL, **56**, 2076 (1934).

(3) Adkins and Connor, *ibid.*, **53**, 1092 (1931).