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.

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

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

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[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).

treatments only a slight amount of unsaturation could be detected by use of tetranitromethane. Silica gel[4] was used to remove this unsaturated material.

After passing the olefins twice over the chromite catalyst at 270°, the products were allowed to percolate through silica gel packed in a vertical tube of approximately 20 mm. diameter. One and one-half grams of gel was employed for each gram of hydrogenation product. The effluent was collected in 5-ml. portions, and each portion tested for unsaturation. By repeated treatment it was possible to obtain products which give no color with tetranitromethane. (I) and (II) thus freed of olefins were dried by refluxing them over sodium, and were then distilled from the sodium.

PROPERTIES AND ANALYSES

		I	II
Calcd. for $C_{10}H_{22}$	C, 84.39 Found	84.24 84.35	84.40 84.22
	H, 15.61 Found	15.58 15.60	15.62 15.64
$n^{25}D$		1.4202	1.4208
d^{25}		0.7511	0.7516
MR (calcd., 48.13)		47.90	47.97
B. p. (763 mm.), °C.		156.6	159.2

The absorption spectra of these substances in the near infrared is discussed in a recent publication.[5]

[4] B. J. Mair and J. D. White, *Bur. Standards J. Research*, **15**, 51 (1935), have shown that silica gel effectively removes olefins from paraffins and naphthenes.

[5] F. W. Rose, Jr., *ibid.*, **19**, 143 (1937), R. P. 1017.

CONTRIBUTION FROM THE
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A Study on the Parachor of Hexamethylenetetramine (Urotropine)

BY T. C. HUANG, M. Y. PENG, K. S. HU AND P. P. T. SAH

The method of Hammick and Andrew[1] for finding the parachor of a solute in a solution is particularly valuable for some substances (like urotropine) whose direct investigation in the fused state is not possible. However, these authors reported that, using water as solvent, anomalous results were obtained for the parachors of the solutes, though they gave neither examples nor data. On the other hand, Ray[2] determined the parachors of sugars in aqueous solutions and obtained normal results. Since water is the most important solvent, we have investigated the parachor of hexamethylenetetramine in aqueous solutions, and found it also to be normal.

"Urotropine," (U. S. P., 30 mesh, Heyden Chemical Corporation), was purified by recrystalling it twice from warm absolute alcohol according to Butlerow.[3] Its different concentrations in conductivity water were made up accurately. The surface tensions, γ, were measured at four different temperatures, 20, 25, 35, and 45°, by the method of capillary rise using first a cathetometer graduated to 0.05 mm. and later one graduated to 0.01 mm. The capillary, made from the stem of a broken thermometer, was calibrated against pure water and benzene at 25°, and found to have a radius of 0.1729 cm. The thermostat used was of the toluene–mercury type keeping the temperature constant within 0.1°. The density, D, of the solution at definite temperature was determined by the regular pycnometer method, correction being made for the buoyancy of the air. The parachor of the solution, P_m, and that of hexamethylenetetramine, P, were calculated according to the equations

$$P_m = M_m \gamma^{1/4}/D$$
$$M_m = (1 - x)M_0 + xM$$
$$P_m = (1 - x)P_0 + xP$$

in which M_0 and P_0 are the molecular weight and the parachor of water and M and x the molecular weight and the mole fraction of hexamethylenetetramine. The values of P_0 were calculated from the surface tensions of water, which are 72.75, 71.97, 70.38, and 68.74 dynes/cm. at 20, 25, 35, and 45°, respectively.[4] The results are summarized in the table.

PARACHOR OF HEXAMETHYLENETETRAMINE

t, °C.	x	D	γ	P_0	P_m	P	Av. P
20	0.02926	1.0422	72.19	52.67	60.38	316.1	315.5
	.03351	1.0482	72.11	52.67	61.46	314.8	
25	.02601	1.0353	71.22	52.64	59.48	315.4	314.8
	.04233	1.0557	70.77	52.64	63.71	314.2	
35	.008174	1.0070	70.07	52.51	54.64	313.1	314.9
	.02142	1.0261	69.98	52.51	58.17	316.7	
45	.01381	1.0115	68.40	52.40	56.04	315.6	315.4
	.02964	1.0324	67.97	52.40	60.19	315.1	

The average parachor of hexamethylenetetramine for four temperatures is 315.2. Evidently the result is normal though water is used as a solvent in the solution method.

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[1] Hammick and Andrew, *J. Chem. Soc.*, 754 (1929).

[2] Ray, *J. Indian Chem. Soc.*, **11**, 843 (1934).

[3] Butlerow, *Ann. Chem. Pharm.*, **115**, 322 (1860).

[4] "International Critical Tables," Vol. IV, 1927, p. 447.