peratures, but dissociates to adsorbed atoms at high temperatures.

It may be remarked that the total hydrogen adsorption remains practically the same in both cases. Any procedure which results in heating a reduced catalyst sample at high temperatures ($\sim 400^{\circ}$ C.) without hydrogen being present, for example, heating under nitrogen or even prolonged high-vacuum pumping at these temperatures, causes the rapid adsorption to decrease while at the same time some sintering is observed. Renewed reduction restores the rapid adsorption. Consequently, some process of surface oxidation occurs during this heat treatment. It may be that a diffusion of oxygen atoms from the interior of the metal to the surface occurs; but it is more probable that the silica carrier releases water which reacts with the metal :

$$Ni + H_2O \rightarrow NiO + H_2$$

either on the surface or in bulk or both.

To our mind the experiments described here fully confirm Roberts's hypothesis on the nature of the activated adsorption. Activated adsorption as de-fined by H. S. Taylor accordingly is adsorption on that part of a metal surface which is covered by oxygen atoms or molecules. The phenomenon hence cannot be considered as evidence for the heterogeneity of a pure metal surface.

The experiments reported here will be published in full in the Recueil des Travaux Chimiques des Pays-Bas.

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Koninklijke/Shell-Laboratorium, Amsterdam. Sept. 15.

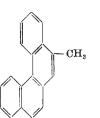
¹ Beeck, O., Ritchie, A. W., and Wheeler, A., J. Coll. Sci., 3, 505 (1948).

² Rideal, E. K., and Trapnell, B. M. W., J. Chim. Phys., 47, 126 (1950).

⁶ Benton, A. F., and White, T. A., J. Amer. Chem. Soc., **52**, 2325 (1930).
 ⁶ Sadek, H., and Taylor, H. S., J. Amer. Chem. Soc., **72**, 1168 (1950).
 ⁶ Roberts, J. K., Proc. Roy. Soc., A, 152, 445, 464, 477 (1935).

A Novel Synthesis of 2-Methyl-3:4-**Benzphenanthrene**

IT has been observed that 2-methyl-3: 4-benzphenanthrene^{1,2} is a potent carcinogenic hydrocarbon³. Although this hydrocarbon was synthesized previously by Hewett¹ and by Newman², their methods are rather laborious and involve numerous steps, and eventually make this hydrocarbon less accessible to synthetic chemists. However, the method developed in this laboratory for synthesis of polycyclics4,5 was extended to the synthesis of the above hydrocarbon. The synthesis of 9-methyl-phenanthrene⁶ served as a model for this synthesis, which gave 2-methyl-3: 4-benzphenanthrene in excellent over-all yield in four steps starting from 2-allyl-1-tetralone.



a-Tetralone was formylated to 2-formyl-1-tetralone (boiling point 136-138°/5 mm., lit.7 boiling point 154°) 10 mm.) following the conditions of Birch and Mukherji⁸. Allylation of this compound was effected with allyl iodide in presence of dry sodium ethoxide in ben-zene to give 2-allyl-2-formyl-1-tetralone (boiling point $134-138^{\circ}/1$ mm.). This substance was hydrolysed to 2-allyl-1-tetralone (boiling point $144-150^{\circ}/5$ mm.; semicarbazone, melting point $174-175^{\circ}$) by mechanical stirring with 5 per cent sodium hydroxide at 50-55° C. for 3 hr.⁹. 2-Allyl-1-tetralone was then subjected to aluminium chloride-catalysed reaction4,5 with benzene, when 2-(\beta-methyl-\beta-phenylethyl)-1-tetralone (boiling point 200-208°/4-5 mm.) was obtained in satis-

factory yield. Reduction of this substance with aluminium isopropoxide and isopropyl alcohol gave the corresponding carbinol (boiling point $205-210^\circ/4-5$ mm.) in almost quantitative yield. This carbinol was cyclized with concentrated sulphuric acid10. (1.84) to 2-methyl-1:2:9:10:11:12-hexahydro-3:4-benzphenanthrene, which distilled at 2 mm. from a bath at 195° C. This hexahydro derivative was smoothly dehydrogenated with palladium-charcoal (30 per cent) at 300-320° C. for 4 hr. to give 2-methyl-3: 4-benzphenanthrene, as a pale yellow viscous oil, which crystallized from acetone-alcohol in small, almost colourless needles (melting point 71-72° C.; cale. for $C_{19}H_{14}$: C, 94.2; H, 5.8; found: C, 94.07; H, 5.9 per cent; lit.¹, leaflets, melting point $69.5-70^{\circ}$ C.; lit.², almost colourless needles, melting point $70.4-71^{\circ}$ C.).

The hydrocarbon gave a vermilion picrate which crystallized from alcohol in needles (melting point cent).

The microanalyses were carried out by Drs. G. Weiler and F. B. Strauss (Oxford).

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¹ Hewett, J. Chem. Soc., 596 (1936).

- ^a Hewett, J. Chem. Soc., 596 (1936).
 ^a Newman and Joshel, J. Amer. Chem. Soc., 62, 972 (1940).
 ^a Bachmann, Cook, Dansi, de Worms, Haslewood, Hewett and Robinson, Proc. Roy. Soc., B, 123, 359 (1937).
 ^a Bhattacharyya and Mukherji, Science and Culture, 16, No. 8, 374
- (1951).
- ⁵ Cologne and Pichat, Bull. Soc. Chim., France, 853 (1949).
- ⁶ Bhattacharyya and Mukherji, Experientia (in the press).
- ⁷ Auwers and Wiegand, J. Prakt. Chem., 134, 82 (1932).
 ⁸ Birch and Mukherji, J. Chem. Soc., 2536 (1949).
 ⁹ Cf. Kloetzel, J. Amer. Chem. Soc., 62, 1171 (1940).
- ¹⁰ Cf. Bogert and Davidson, J. Amer. Che m. Soc., 56, 961 (1934).

Colour Reagents for the Paper Chromatography of Di- and **Tri-hydroxy** Phenols

VARIOUS colour-producing spraying reagents have been developed for the detection of di- and tri-hydroxy phenols on paper chromatograms. Excellent results are obtainable with the diazotized sulphanilic acid of Evans, Parr and Evans¹ and the phospho-molybdic acid reagent of Riley². Bate-Smith^{3,4} Bate-Smith^{3,4} applied Partridge's⁵ ammoniacal silver nitrate reagent for furnishing diagnostic evidence regarding the position and number of hydroxy groups and also used ultra-violet light, both in the presence and

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