Reactions (9), (11), (13) and (15).—There does not seem to be any experimental evidence available for these reactions and in these cases also we have assigned values which are in agreement with the free radical mechanism.

THE JOHNS HOPKINS UNIVERSITY BALTIMORE, MD. RECEIVED NOVEMBER 29, 1933

Ammonolysis of Fluorenone and Fluorenone Anil

BY LOUIS A. PINCK AND GUIDO E. HILBERT

Strain¹ has shown that ketones, such as acetophenone, on heating with ammonia at 180° with aluminum chloride as a dehydrating agent, yield the corresponding ketimines. It has now been found that fluorenone imide can be prepared in excellent yield by the interaction of fluorenone and ammonia at room temperature. This method is superior to that of Kliegl² and is recommended for preparative purposes. Benzophenone, 9,9dichlorofluorene and 1,2-dichlorodibiphenyleneethane under similar conditions, however, do not react.

Additional information on the behavior of Schiff bases in liquid ammonia³ was obtained in a study of the ammonolysis of fluorenone anil. This reaction leading to the formation of fluore-

(1) Strain, THIS JOURNAL, 52, 820 (1930).

(2) Kliegl, Ber., 43, 2488 (1910); see also Goldschmidt and Beuschel, Ann., 447, 203 (1926).

(3) Strain, THIS JOURNAL, 50, 2218 (1928).

none imide and aniline was catalyzed by ammonium chloride and did not go to completion; apparently an equilibrium was attained. Fluorenone anil was readily prepared by heating fluorenone imide and aniline.

Experimental Procedure

Ammonolyis of Fluorenone.—Ten grams of fluorenone⁴ was treated with 15 cc. of sodium-dried ammonia and allowed to stand at room temperature for several weeks. After the removal of ammonia the fluorenone imide was recrystallized from ligroin; m. p. 124°, yield practically quantitative.

Fluorenone Anil.—A mixture of 0.2 g. of fluorenone imide and 0.2 cc. of aniline was heated at 120° for ninety minutes; ammonia was evolved. The yellow sirupy reaction product solidified on cooling and was recrystallized from petroleum ether; m. p. $87^{\circ}.^{5}$

Ammonolysis of Fluorenone Anil.—A solution of 0.5 g. of fluorenone anil and 0.25 g. of ammonium chloride in dry liquid ammonia was heated at 60° for four days. The ammonia was removed and the reaction product dissolved in boiling ligroin. On concentration of the solution 0.22 g. of fluorenone imide separated. From the mother liquor was isolated 0.15 g. of unchanged anil.

No fluorenone imide was isolated in an experiment in which a solution of fluorenone anil in dry ammonia was heated at 60° for twenty hours.

(5) Reddelien, Ber., 43, 2479 (1910); Schlenk and Bergmann,, Ann., 463, 292 (1928).

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U. S. DEPARTMENT OF AGRICULTURE

WASHINGTON, D. C.

COMMUNICATIONS TO THE EDITOR

TWO TYPES OF ACTIVATED ADSORPTION OF HYDROGEN ON THE SURFACE OF A PROMOTED IRON SYNTHETIC AMMONIA CATALYST

Sir:

By making adsorption measurements in conjunction with our study of the catalytic conversion of ortho to para hydrogen we have obtained data that seem to establish definitely the existence of two types [see Benton, *Trans. Faraday Soc.*, **28**, 202 (1932), and Langmuir, *Chem. Rev.*, **13**, 188 (1933)] of activated and one type of physical adsorption of hydrogen on the surface of an iron catalyst promoted with 1.3% Al₂O₃ and 1.59% K_2O . The characteristics of the three types of adsorption on the promoted iron catalyst are as follows.

Physical Adsorption. The adsorption of hydrogen at 760 mm. pressure by a 10-cc. (about 22-g.) sample of catalyst that had been reduced by hydrogen at 450° and degassed at the same temperature, was 5.0, 2.8 and 0.25 cc. at -196, -183 and -144° , respectively. The heat of adsorption calculated from isotherms was about 2000 calories.

Type A Activated Adsorption.—Between -78and 0° apparent equilibrium could be obtained in

DEPARTMENT OF CHEMISTRY

⁽⁴⁾ This was prepared according to the method of Schmidt and Wagner, Ber., 43, 1796 (1910), (3 kilos of technical fluorene yielded 2450 g. of pure fluorenone), which is practically identical with that recently published by Huntress, Hershberg and Cliff, THIS JOURNAL, 53, 2720 (1931).