weighed 6.3 g. (85% yield). Crystallization from benzene-m.p. 72–73.5°.

Anal. Calcd. for $C_{12}H_{13}N_3O_2$: C, 62.32; H, 5.67; N, 18.17; neut. equiv., 231. Found: C, 62.43; H, 5.84; N, 18.31; neut. equiv., 234.

Methyl γ-2-phenyl-1,2,3,2H-triazol-4-ylbutyrate (XV). Six tenths of a gram (0.002 mole) of the acid (XIV) was dissolved in 25 ml. of absolute methyl alcohol which had been saturated with dry hydrogen chloride in the cold and the solution was refluxed overnight. After removal of the solvent under reduced pressure, the residue was dissolved in ether and washed thoroughly with 5% sodium carbonate solution, followed by water. The ether solution was dried over anhydrous sodium sulfate and the ether was evaporated on the steam bath, leaving an analytical sample of XV as an almost colorless oil. The sample weighed 0.4 g. (80% yield), too small an amount for the determination of the boiling point.

Anal. Calcd. for $C_{13}H_{15}N_3O_2$: C, 63.65; H, 6.16; N, 17.13. Found: C, 63.98; H, 6.52; N, 17.32.

γ-2-Phenyl-1,2,3,2H-triazol-4-ylbutyramide (XVI). Into a test tube was placed 0.1 g. (0.0004 mole) of XV and 5 ml. of concd. ammonium hydroxide. The contents of the test tube were shaken periodically for several days, after which time crystals formed. The crystals were filtered with suction, washed with water, and air-dried, yielding 0.075 g. (83% yield) of XVI. Crystallization from benzene afforded fine colorless needles, m.p. 105–106°.

Anal. Calcd. for $C_{12}H_{14}N_4O$: C, 62.59; H, 6.13; N, 24.33. Found: C, 62.21; H, 5.89; N, 23.79.

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Hydrogenation of the Imidazole Ring. Formation of Diacetylimidazolidine and of Diacetylbenzimidazoline[1]

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The remarkable resistance of the imidazole ring to catalytic hydrogenation is well established.[2] The only exception that has been described is the hydrogenation of lophine and of amarine which was catalyzed by a suspension of platinum black in glacial acetic acid.[3] In the work reported here it was found that hydrogenation of the imidazole ring proceeded smoothly at room temperature and at atmospheric pressure when acetic anhydride was used as the solvent and platinum oxide as the catalyst. In this way, two hitherto unknown compounds were prepared: diacetylimidazolidine was formed by the hydrogenation of imidazole; diacetylbenzimidazoline was formed by the hydrogenation of benzimidazole, leaving the benzene ring unchanged.

EXPERIMENTAL

Acetic anhydride (150 ml., 1.47 moles) was shaken with hydrogen and 1.0 g. of platinum oxide at room temperature and ordinary pressure until the uptake of hydrogen stopped. About 400–470 ml. of hydrogen (corr.) were absorbed.[4] To this pretreated suspension of platinum black in acetic anhydride, 2.04 g. (0.03 mole) of imidazole were added and the shaking continued until no further hydrogen was absorbed. The calculated amount of hydrogen (0.06 mole of hydrogen) was consumed in about 7 hr. The acetic anhydride solution was decomposed with ice water and evaporated to dryness *in vacuo* (20 mm.). The remaining oil was mixed with a small amount of absolute ethanol and ether. Upon storage in the cold room beautiful crystals of diacetylimidazolidine were formed; the yield was 3.77 g. (80%), m.p. 95°.

Anal. Calcd. for $C_7H_{12}N_2O_2$: C, 53.83; H, 7.75; N, 17.94. Found: C, 53.85; H, 7.50; N, 17.88.

Diacetylimidazolidine is very soluble in water and in ethanol, soluble in acetone and in hot benzene, and sparingly soluble in ether. It can be recrystallized from ethanol-ether. Determination of the acetyl groups: Calcd. as CH_3COOH: 76.89. Found: 76.97.

Boiling with 2*N* hydrochloric acid for 45 min. split the imidazolidine ring with the formation of formaldehyde (detected by the fuchsin-sulfurous acid reaction) and ethylenediamine (detected with ninhydrin spray after chromatography in *n*-propyl alcohol, 75, 28% NH_4OH, 1.5, water, 23.5). At room temperature, at the other hand, only a trace of formaldehyde was detected after 18 hr. in 2*N* hydrochloric acid.

Benzimidazole (2.36 g., 0.02 mole) was hydrogenated the same way in acetic anhydride and consumed the calculated amount of hydrogen (0.02 mole) in about 7 hr. After recrystallization from absolute ethanol the yield of diacetylbenzimidazoline was 3.51 g. (86%). The melting point was 168–170°.

Anal. Calcd. for $C_{11}H_{12}N_2O_2$: C, 64.69; H, 5.92; N, 13.72. Found: C, 64.60; H, 6.10; N, 13.60.

Diacetylbenzimidazoline is soluble in ethanol, sparingly in cold water, soluble in hot water, soluble in acetone and in benzene; it is almost insoluble in ether.

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[2] K. Hofmann, *Imidazole and Its Derivatives*, Interscience Publishers, New York, 1953, p. 16.

[3] E. Waser and A. Gratsos, *Helv. Chim. Acta*, 11, 944 (1928).

[4] This amount of hydrogen represents approximately 220 ml. (0.01 mole) more than that calculated for the reduction of platinum oxide to platinum black. The mechanism of this partial reduction of the acetic anhydride is unclear. Similar findings have been reported by C. Mannich and A. H. Nadelmann, *Ber.*, 63B, 796 (1930), who found that at room temperature and ordinary pressure, in the presence of palladium black as the catalyst, acetic anhydride consumed a small amount of hydrogen with the formation of aldehyde and a little ethyl acetate.

Preparation of 5(6)-Iodobenzimidazole and 4(7)-Iodobenzimidazole

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During the course of some work concerning a comparative study of the physical properties of