

soluble inorganic material by gravity filtration, was added 0.4 g. (0.0038 mole) of *o*-phenylenediamine in 20 ml. of ethyl acetate. The pink solution was heated on the steam bath for 30 min., cooled, and on evaporation to dryness under an air stream left an amber residue which recrystallized from benzene as red microcrystalline 1-aminophenazine, 0.41 g. (67.7%) m.p. 183–184°.⁴

This and other phenazines prepared in a similar manner are described in Table I. Lead dioxide could be substituted for silver oxide in each example.

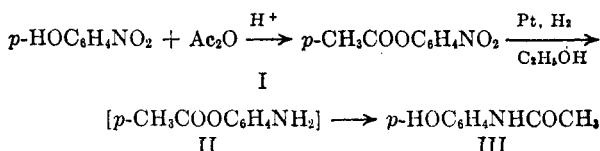
CHEMISTRY DEPARTMENT
TULANE UNIVERSITY
NEW ORLEANS 18, LA.

Acetyl Transfer during Hydrogenation of *p*-Nitrophenyl Acetate¹

RONALD FELDSTEIN, MARY H. ALDRIDGE,
AND B. H. ALEXANDER²

Received September 1, 1960

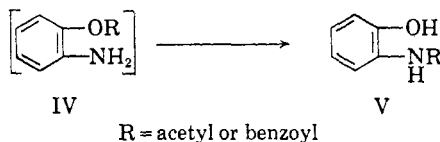
In connection with other work, *p*-aminophenyl acetate (II) was needed. Since the literature preparation of II³ is difficult and attended with low



yields, its preparation was attempted by the schematic representation shown above. The acetylation of *p*-nitrophenol went smoothly with acetic anhydride and a catalytic amount of sulfuric acid; however, the reduction of *p*-nitrophenyl acetate (I) at 850 p.s.i. and 120° in the presence of a platinum catalyst, gave not the desired product (II) but rather an isomer, *p*-hydroxyacetanilide (III) in high yield. To our knowledge, this is the first reported example of a *para* acetyl transfer during the reduction of I to yield probably II, then immediately III.

Many investigators,⁴ though, have reported *ortho* acetyl and benzoyl transfers. Others⁵ have

shown that these transfers were, in some instances, intramolecular migrations that occurred because the *ortho* esters of aminophenyl acetate (isomer of II) and aminophenyl benzoate were very unstable. Thus, when formed, they rearranged quickly to the corresponding *N*-acetyl or -benzoyl derivatives; i.e., the acetyl or benzoyl group migrated from the oxygen to the nitrogen atom.



A satisfactory explanation for the isolation of *p*-hydroxyacetanilide (III) from *p*-nitrophenyl acetate (I) in high yield is still premature; however, our results indicate that the reaction is a reduction of a nitro group followed by an intermolecular aminolysis of an ester.

EXPERIMENTAL

p-Nitrophenyl acetate (I) was prepared from *p*-nitrophenol (30 g.), acetic anhydride (61 ml.), and 4 drops of concd. sulfuric acid in the usual way; m.p. 79–80°; yield 95%.

p-Hydroxyacetanilide (III) was prepared as follows: *p*-nitrophenyl acetate (290 g.), absolute ethanol (500 ml.), and platinum oxide catalyst (2.5 g.) were kept at 850 p.s.i. of hydrogen and at approximately 120° for 6 hr. The mixture cooled on standing overnight and was filtered. The solvent was removed and crude III crystallized; crude yield was quantitative and the yield after recrystallization from hot water was 77%; m.p. 163–166°; ethyl acetate was later found to be a better solvent than water for the recrystallization of III, so III was then recrystallized from ethyl acetate; m.p. 168–169° (mixed m.p. with known III 168–169°).

DEPARTMENT OF ORGANIC CHEMISTRY
AMERICAN UNIVERSITY
WASHINGTON 16, D. C.

(5) W. Böttcher, *Ber.*, **16**, 629 (1883); J. H. Ransom, *Ber.*, **31**, 1055 (1898); **33**, 199 (1900); *Am. Chem. J.*, **23**, 1 (1900); A. Einhorn and B. Pfl, *Ann.*, **311**, 34 (1900); K. Auwers, *Ber.*, **33**, 1923 (1900).

A Convenient Method for the Preparation of 3-Cyano-6-methyl-2(1H)pyridone

LOUIS J. BINOVI AND HERBERT G. ARLT, JR.

Received October 5, 1960

There has been a continuing interest in this compound since 1944¹ as an intermediate in syn-

(1) This note is based partly upon the thesis submitted by Ronald Feldstein to the Graduate School of American University in partial fulfillment of the requirements for the Master of Science degree.

(2) Entomology Research Division, Agricultural Research Service, U. S. Department of Agriculture, Beltsville, Md.

(3) L. Galatis, *Ber.*, **59**, 850 (1926); S. E. Hazlet and C. A. Dornfeld, *J. Am. Chem. Soc.*, **66**, 1781 (1944).

(4) L. C. Raiford, *J. Am. Chem. Soc.*, **41**, 2068 (1919); L. C. Raiford and J. R. Couture, *J. Am. Chem. Soc.*, **44**, 1792 (1922); **46**, 2305 (1924); L. C. Raiford and H. A. Iddles, *J. Am. Chem. Soc.*, **45**, 469 (1923); L. C. Raiford and H. P. Lankelma, *J. Am. Chem. Soc.*, **47**, 1111 (1925); A. L. LeRosen and E. D. Smith, *J. Am. Chem. Soc.*, **70**, 2708 (1948); **71**, 2815 (1949).

(1) T. Matsukawa, and T. Matsuno, *J. Pharm. Soc. Japan* **64**, 145 (1944), *Chem. Abstr.*, **45**, 4724f (1951).

(2) L. A. Perez-Medina, R. P. Mariella, and S. M. McElvain, *J. Am. Chem. Soc.*, **69**, 2574 (1947).