The activities of the samples are corrected for self absorption of the soft β -radiation and are therefore comparable. These results indicate that under the above conditions only $\sim 25\%$ of the CO₃⁼ comes from the carboxyl group.⁹ It is apparent that the oxidation is complex and further work with the oxidation of various labelled derivatives of propionic acid (lactic, pyruvic, β -hydroxypropionic, etc.) under varying conditions is in progress so that the mechanism of these reactions can be understood more clearly.

The oxidation of propionic acid by $Cr_2O_7^{-}$ in concentrated sulfuric acid solution yields carbon dioxide and acetic acid.¹⁰ The reaction can be represented by the equation

 $C_2H_5COOH + Cr_2O_7^{=} + 8H^{+} =$

 $2Cr^{+++} + CO_2 + CH_3COOH + 5H_2O$ Control experiments showed that propionic acid in 18 N sulfuric acid is completely oxidized to CH₃COOH and carbon dioxide by $Cr_2O_7^{-}$ within three hours at 100°. No other products could be detected. $\sim 5 \times 10^{-4}$ mole of labelled propionic acid (containing 160 counts/min.¹¹) was refluxed with 1.5×10^{-3} mole of $K_2Cr_2O_7$ in 18 N sulfuric acid in a stream of nitrogen. The evolved carbon dioxide was trapped and determined as above. The acetic acid was separated by steam distillation and a measurement of the Duclaux constants showed only CH₃COOH to be present. The radioactivities of the carbon dioxide and acetic acid were measured and the results are tabulated in Table II.

	Tabl	εII	
Moles	Moles	Activity	
CH₃COOH	CO_2	CH3COOH	CO_2
$4.6 \cdot 10^{-4}$	$1.2 \cdot 10^{-3^a}$	0 ± 2	158 = 5

^a The value for carbon dioxide production is much too high (mainly due to absorption of atmospheric carbon dioxide) and was caused by a mishap in the analytical determination.

These results show that carbon dioxide is produced exclusively by rupture of the bond between the carboxyl and α -carbon. The results of further experiments now in progress will be reported soon.

CHEMICAL LABORATORY

UNIVERSITY OF CALIFORNIA BERKELEY, CALIFORNIA RECEIVED MARCH 31, 1941

2'-Nitro-2-aminobiphenyl

By Donald Purdie

In the course of a research project it became necessary to prepare 2'-nitro-2-aminobiphenyl; previous attempts at the preparation of this substance by Mascarelli and Gatti seem to have run into difficulties as it is described as a slowly crystallizing oil which could not be obtained in the pure state.¹ Nevertheless its formation was proved by the preparation of many derivatives.

A method of preparation for this substance, suitable for large scale work, is now described; starting from 2,2'-dinitrobiphenyl it differs from the method of Mascarelli in the use of aqueous alcoholic sodium polysulfide as a reducing agent in place of aqueous alcoholic ammonium sulfide. A pure product is obtained readily and the saving of time and manipulation is considerable. As a check on its identity the product was acetylated and the 2'-nitro-2-acetamidobiphenyl so formed was shown to have the same melting point as that observed by Mascarelli and co-workers; their observation that recrystallization from benzene is essential for a sharp-melting product was also confirmed.

Experimental

Ethanol (600 cc.) and 2,2'-dinitrobiphenyl (42 g.) were boiled under reflux condenser and a solution of sodium polysulfide (prepared by boiling 12 g. of sulfur with 48 g. of sodium sulfide crystals and 150 cc. of water until dissolved) slowly added. The solution turns green and then orange; boiling was continued for three hours and the whole allowed to stand overnight. Most of the ethanol was then removed by distillation and the residue poured into 1 liter of ice water. The product was extracted with ether and the ethereal solution washed with water and then dried over caustic soda pellets; this removes much impurity and is essential if a pure product is to be obtained. After removing most of the ether the rest was cooled and the 2'nitro-2-aminobiphenyl then separated as indistinct crystals. Thrice recrystallized from ethanol the final product had m. p. 94-94.5° and formed orange needles easily soluble in ether or benzene, sparingly in cold ethanol or petroleum ether.

Anal. Calcd. for $C_{12}H_{10}N_2O_2$: N, 13.1. Found: N, 13.4.

The acetyl derivative was prepared in the usual way and after recrystallization from ethanol and then from benzene had m. p. $159-60^{\circ}$ (Mascarelli gives 158°). Crystallization from ethanol alone always gave a low-melting product.

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⁽⁹⁾ Wood, et al., THIS JOURNAL, **63**, 2140 (1941), have found that in 0.1 N OH⁻, 37% of the carbonate arises from the carboxyl group. It would seem that the OH⁻ concentration plays a role in determining whether the bond between the α - and β -carbons or the α and carboxyl shall be ruptured. In a single experiment using 0.1 N OH⁻ we have repeated the permanganate oxidation of propionate and the results appear to bear this out; however, we are investigating this point in detail.

⁽¹⁰⁾ Polonovski and Lindenberg, Compt. rend., 209, 46 (1939).

⁽¹¹⁾ Prepared by the $C*O_2-C_2H_\delta MgBr$ method.

⁽¹⁾ Mascarelli and Gatti, Atti accad. Lincei, 10, 441 (1929); Gazz. chim. ital., 59, 861 (1929); Mascarelli, Gatti and Pirona, ibid., 61, 786 (1931).