Oxidative Cleavage of Nitrilotriacetic Acid to Iminodiacetic Acid

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Summary The oxygenation of nitrilotriacetic acid solution at pH 8·5 and 90° in the presence of 5% Pd/C gives almost quantitative yields of iminodiacetic acid.

NITRILOTRIACETIC ACID (I) is a possible replacement for phosphates in detergents, therefore, a knowledge of its decomposition products under oxidizing conditions is important. Cerium(IV) salts have been reported[1] to produce $NHMe_2$ and CO_2, while biodegradation led to CO_2, H_2O, and N_2.[2]

We have now found that the oxygenation of an aqueous solution of (I) in the presence of 5% Pd/C at pH 8·5 (90° for 10 h) formed CO_2† and iminodiacetic acid‡ (II) almost quantitatively.§ Although we used O_2 gas, related work indicates that this reaction will also occur under these conditions using air at atmospheric pressure. No CO was detected and small amounts of oxalic acid could be isolated as sodium oxalate. These data support the following scheme:

$$\underset{(I)}{N(CH_2CO_2H)_3} \xrightarrow[Pd/C]{O_2,\ H_2O} \underset{(II)}{HN(CH_2CO_2H)_2} + (CO_2H)_2 \xrightarrow{O_2} CO_2 + H_2O$$

In the absence of catalyst under the same conditions, (I) was recovered unchanged. The only precedent for selective fragmentation is the reported hydrolytic cleavage of (I) with sulphuric acid at 130—135°[3] and with hydrochloric acid at 190—200°[4] to give iminodiacetic acid and glycollic acid.

(*Received, October 22nd*, 1970; *Com.* 1819.)

† CO and CO_2 detection was by Fisher Gas Partitioner.

‡ I.r., n.m.r., m.s., and t.l.c. data were all in agreement with those of an authentic sample of iminodiacetic acid.

§ A 92·5% yield of monoammonium iminodiacetate was obtained by absorbing the filtered reaction solution on an acid resin (Rexyn 101H R-203 Fisher) and eluting with 1 : 1 NH_4OH.

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