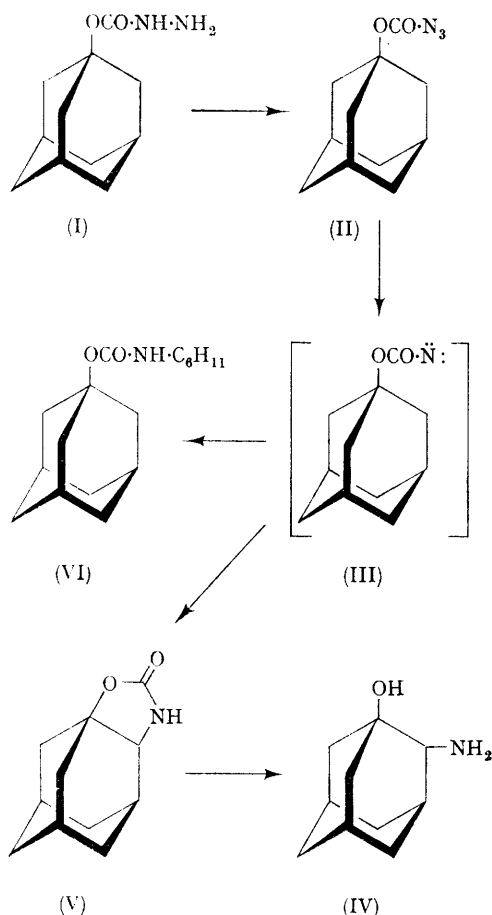


Synthesis of 2-Aminoadamantan-1-ol

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ALTHOUGH numerous 1-substituted and several 2-substituted adamantane derivatives have been reported,¹ simple 1,2-disubstituted adamantane compounds are rare. We report a route for the preparation of a member of this series, namely, 2-aminoadamantan-1-ol (IV).

1-Adamantyl carbazate² (I) treated with nitrous acid gave the azido-formate (II), which was extracted into cyclohexane and photolyzed in a quartz reaction vessel for 18 hr., using a Rayonet Photochemical Chamber.† Concentration of the solvent produced adamantanto[2,1-*d*]oxazolidin-2-one (V), in a 45% yield, m.p. 130–133°, $\lambda_{\max}(\text{KBr})$ 3.1, 5.7, and 5.8 μ , δ (CDCl_3) 6.2 (NH), 3.7 (1 H adjacent to NH).‡ Column chromatography of the filtrate also afforded a small amount (4%) of the cyclohexylcarbamate (VI), m.p. 84–87°, $\lambda_{\max}(\text{KBr})$ 2.9, 6.0 μ , resulting from insertion of the intermediate nitrene (III) into the solvent. Hydrolysis of the oxazolidinone (V) in refluxing 2*N*-hydrochloric acid for 1 hr. gave a good yield (60%) of 2-aminoadamantan-1-ol (IV) as a crystalline hydrochloride, $\lambda_{\max}(\text{KBr})$ 3.1, 5.05, 6.23, and 6.55 μ , δ (Me_2SO) 8.1 (NH_3^+), 5.2 (OH), 3.0 (1 H adjacent to NH_3^+).

Elemental analyses, which were satisfactory for all new compounds, were performed by Mr. L. Brancone and staff. I.r. and n.m.r. spectra were determined by Mr. W. Fulmor and staff.

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† The low-pressure mercury lamps which were used provided 84% of their emission at 2537 Å.

‡ A similar photolytic cyclization of *t*-butyl azidoformate has recently been reported by R. Kreher and G. H. Bockhorn, *Angew. Chem.*, 1964, 76, 681.

¹ R. C. Fort, jun., and P. von R. Schleyer, *Chem. Rev.*, 1964, 64, 277.

² W. L. Haas, E. V. Krumkalns, and K. Gerzon, *J. Amer. Chem. Soc.*, 1966, 88, 1988.