

violet absorption bands^{3,11} at 230 and 292 m μ which are not seen in B₁₀H₁₀⁻² itself. In addition, the absence of B-H-B bands in the infrared spectrum of B₂₀H₁₈⁻² is now rationalized.

Acknowledgments.—This research was generously supported by the Advanced Research Projects Agency through the Army Research Office (Durham) and by Public Health Service Research Grant No. CA-06773-02 from the National Cancer Institute. The use of the Varian Associates experimental nuclear magnetic spectrometer is gratefully acknowledged.

(11) A. R. Pitochelli, W. N. Lipscomb, and M. F. Hawthorne, *J. Am. Chem. Soc.*, **84**, 3026 (1962).

(12) Alfred P. Sloan Research Fellow.

DEPARTMENT OF CHEMISTRY
THE UNIVERSITY OF CALIFORNIA
RIVERSIDE, CALIFORNIA
VARIAN ASSOCIATES
PALO ALTO, CALIFORNIA

RICHARD L. PILLING
M. FREDERICK HAWTHORNE¹²

EUGENE A. PIER

RECEIVED JUNE 25, 1964

A 2,3-Diazacyclopropanone (Di-*t*-butyldiaziridinone)

Sir:

We wish to report the preparation of a new small-ring heterocycle, a diaziridinone (I, R = *t*-butyl) possessing a number of properties of special interest and of considerable potential as a synthetic intermediate.

Treatment of N,N'-di-*t*-butyl-N-chlorourea,¹ m.p. 30–31°, with potassium in pentane or with potassium *t*-butoxide in *t*-butyl alcohol² affords di-*t*-butyldiaziridinone (I, 40–80% yield), m.p. 0–1° (*Anal.* Found: C, 63.38; H, 10.74; N, 16.47), mol. wt. 178 (cryoscopic in cyclohexane), n.m.r. (in CCl₄), single peak at τ 8.80, infrared absorption in the carbonyl region (in CCl₄), maximum at 1880 and 1862 cm.⁻¹ with shoulders at 1926 and 1800 cm.⁻¹. Chemical evidence in support of structure I³ is found in: (1) cleavage of the N–N bond by hydrogenation (palladium on carbon) with formation of N,N'-di-*t*-butylurea in 96% yield; (2) cleavage of the C–N bond by sodium borohydride with formation of N,N'-di-*t*-butyl-N-formylhydrazine (60% yield), m.p. 42–43°, n.m.r. (in CCl₄), τ 8.92 (singlet, 9 H's), 8.63 (singlet, 9 H's), 5.80 (broad, 1 H), and 1.43 (singlet, 1 H).

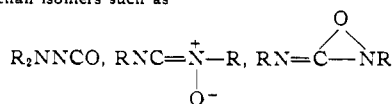
Compound I is reactive toward acids. Treatment of I with dry hydrogen chloride followed by water gives 2,3-di-*t*-butylcarbazyl chloride, II. Treatment of II with potassium *t*-butoxide in *t*-butyl alcohol yields a mixture of I (45%) and *t*-butyl 2,3-di-*t*-butylcarbazate (III, 25%).

Among the unusual properties of I are the high thermal stability (only slight decomposition in 2 hr. at 175°)

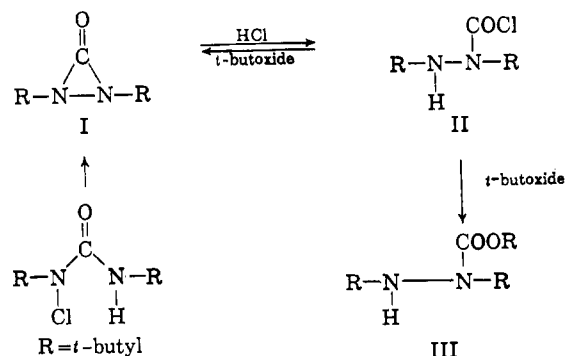
(1) Satisfactory combustion data and proof of structure have been obtained for new compounds.

(2) These methods are analogous to those used to prepare α -lactams: H. E. Baumgarten, J. F. Fuerholzer, R. D. Clark, and R. D. Thompson, *J. Am. Chem. Soc.*, **86**, 3303 (1963); J. C. Sheehan and I. Lengyel, *ibid.*, **86**, 1356 (1964).

(3) The physical and chemical evidence appears in strong favor of structure I rather than isomers such as



X-Ray evidence on the structure and particularly on the interesting question of the location of the substituents relative to the plane of the ring will be forthcoming.



and the stability toward nucleophiles⁴ (50% conversion of I to III by potassium *t*-butoxide (0.3 M) in *t*-butyl alcohol in 16 hr. at reflux; no reaction of I with aniline in ether for 6 days at 25°).

The *t*-butyl carbazate, III, is readily cleaved by hydrogen chloride; the resulting N,N'-di-*t*-butylhydrazine may be directly oxidized to azo-*t*-butane (one of several procedures for the conversion of I to the azo compound). Thus the work described here represents a method for the formation from ureas of substituted⁵ hydrazines with like or unlike substituents and of symmetrical and unsymmetrical azo compounds.

A full account of the synthetic aspects and of the physical and chemical properties of compounds of type I will be reported at a later date.

(4) These properties are more reminiscent of the cyclopropanones [R¹ Breslow and R. Peterson, *J. Am. Chem. Soc.*, **82**, 4426 (1960); R. Breslow, J. Posner, and A. Krebs, *ibid.*, **85**, 234 (1963)] than of the α -lactams (ref. 2).

(5) E.g., *t*-alkyl; primary and secondary N,N'-disubstituted hydrazines are accessible from the corresponding azines.

(6) National Science Foundation Predoctoral Fellow, 1961–1964.

DEPARTMENT OF CHEMISTRY
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
CAMBRIDGE, MASSACHUSETTS 02139

FREDERICK D. GREENE

JOHN C. STOWELL⁶

RECEIVED JUNE 15, 1964

Potassium Perxenate Nonahydrate¹

Sir:

Several salts of the unstable perxenic acid have been isolated and identified. Sodium perxenate^{2,3} exists in several crystalline forms, and the structures of Na₄XeO₆·8H₂O^{4,5} and Na₄XeO₆·6H₂O⁶ have been determined. Perxenate salts of barium^{7,8} and of calcium, lithium, and cesium⁸ have been prepared, but their structures are unknown. We have now prepared a potassium salt and characterized it as K₄XeO₆·9H₂O by determination of its crystal structure.

The solubility of potassium perxenate appears to be much greater than that of the sodium perxenates at a given concentration of OH⁻. The disproportionation reaction of xenic acid in potassium hydroxide solution failed to give crystals under conditions analogous to those at which Na₄XeO₆·8H₂O was easily crystallized.

(1) Work done in part under the auspices of the U. S. Atomic Energy Commission.

(2) J. G. Malm, B. D. Holt, and R. W. Bane, "Noble Gas Compounds," H. H. Hyman, Ed., University of Chicago Press, Chicago, Ill., 1963, p. 167.

(3) S. Siegel and E. Gebert, ref. 2, p. 193.

(4) W. C. Hamilton, J. A. Ibers, and D. R. Mackenzie, *Science*, **141**, 532 (1963).

(5) J. A. Ibers, W. C. Hamilton, and D. R. Mackenzie, *Inorg. Chem.*, to be published.

(6) A. Zalkin, J. D. Forrester, D. H. Templeton, S. M. Williamson, and C. W. Koch, *Science*, **142**, 501 (1963).

(7) E. H. Appelman and J. G. Malm, *J. Am. Chem. Soc.*, **86**, 2141 (1964).

(8) C. W. Koch and S. M. Williamson, unpublished work.