dride. A −300 mesh sample of $VH_{0.87}$ was treated for several hours in a platinum dish with 10% HF. The HF solution then was removed and the product washed in water, alcohol, and finally in ether. An X-ray diffraction powder pattern of the product showed the presence of the f.c.c. vanadium dihydride. Faint lines in the low angle region, however, indicated a small amount of the monohydride. A trace of vanadium oxide also was indicated by faint lines in the diffraction pattern. The value of the lattice constant for the dihydride determined from this film was 4.271 ± 0.002 Å. The formula was found to be $VH_{1.77 \pm 0.05}$ by hydrogen loss *in vacuo*. Vanadium samples of 99.8+ per cent. purity were obtained from the Oregon Metallurgical Company, Albany, Oregon. Additional details of preparation, stability, and physical properties will be given in future publications.

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REDUCTION OF ISOLATED OLEFINIC BONDS BY MEANS OF *p*-TOLUENESULFONYLHYDRAZINE

Sir:

Prompted by Thiele's early work[1] on azodicarboxylic acid, and other considerations, we recently investigated the decomposition of this substance in the presence of olefinic compounds, with the discovery that reduction of isolated carbon–carbon double bonds occurred.[2] Of the various mechanistic interpretations which might be entertained (including reduction by azomonocarboxylic acid or a related species), involvement of the elusive H_2N_2 or an equivalent is the most direct. We have now found that saturation of isolated olefinic bonds also can be effected through thermal decomposition of another possible H_2N_2 (but not azomonocarboxylic acid) source, *p*-toluenesulfonylhydrazine.[3,4]

Reductions were carried out by heating under reflux a solution of the olefinic component and a 100% excess of *p*-toluenesulfonylhydrazine in diglyme for one hour under nitrogen.[5] As typical results, oleic and elaidic acids were reduced to stearic acid (73% and 70%, respectively, both by infrared analysis and bromine titration); in these runs the sulfur-containing by-products were removed by extraction or by oxidation to the water-soluble sulfonic acid. Reduction of allyl alcohol gave 1-propanol (99%), and cyclohexene gave cyclohexane (98%) (both by vapor phase chromatography). The thermal decomposition of *p*-toluene sulfonylhydrazine should give rise to *p*-toluenesulfinic acid as one of the initial products,[6] and confirmation of this presumption has been obtained by the isolation of the sulfinic acid, along with *p*-tolyl disulfide, from the pyrolysis of the sulfonhydrazide in diglyme.[7]

Thus, the azodicarboxylic acid and *p*-toluenesulfonylhydrazine reduction methods—insofar as they are compatible with the H_2N_2 hypothesis—involve preparation of a species which, although in itself too unstable to be isolated under ordinary conditions,[8] nevertheless can be utilized as a reagent in the presence of a substrate.

We wish to take this opportunity for drawing attention to the general possibilities of carrying out new reactions on organic molecules through the use of unstable neutral inorganic reagents, in the same sense that unstable, unisolated organic entities (such as carbenes) are utilized. This field of investigation would appear to be relatively virgin, in that incorporated into the entire body of organic chemistry are only few such examples—virtually all known reactions involving inorganic reagents are executed by means of "shelf" chemicals of normal stability. Further, within the confines of inorganic chemistry, this device may be useful in providing evidence for the existence of such unstable species.

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(1) J. Thiele, *Ann.*, **27**, 127 (1892). Thiele reported that in the decarboxylation of azodicarboxylic acid, carbon dioxide, nitrogen and hydrazine are formed; and he suggested that the hydrazine and nitrogen arise by disproportionation of the unstable diimide. The comment may be made that reduction of azodicarboxylic acid with H_2N_2 would also lead to hydrazine, *via* decarboxylation of the intermediate hydroazodicarboxylic acid.

(2) E. E. van Tamelen, R. S. Dewey and R. J. Timmons, *J. Am. Chem. Soc.*, **83**, 3725 (1961).

(3) No evidence is available to distinguish between $HN{=}NH$ and $H_2N{-}N \leftrightarrow H_2\overset{-}{N}{=}\overset{+}{N}$ in this decomposition.

(4) Commercially available from Aldrich Chemical Company, Milwaukee, Wis.

(5) The decomposition of *p*-tosylhydrazine can be accelerated by the addition of hydroxide ion, and to some extent by metal ions. Whether the decomposition is a radical or cyclic process, or involves an α- or β-elimination, is unknown.

(6) The loss of *p*-toluenesulfonylhydrazine by prolonged heating during recrystallization has been observed by C. H. DePuy and D. H. Froemsdorf, *J. Am. Chem. Soc.*, **82**, 636 (1960).

(7) *p*-Toluenesulfinic acid is converted to *p*-tolyl *p*-toluenethiosulfonate in hot aqueous solution (R. Otto and O. V. Gruber, *Ann.*, **145**, 13 (1868)), and the thioester has been converted to *p*-tolyl disulfide in hot aqueous sodium carbonate (E. Fromm, *Ber.*, **41**, 3409 (1908)).

(8) Some evidence for persistence of H_2N_2 at low temperatures has been presented, for example, by S. N. Foner and R. L. Hudson, *J. Chem. Phys.*, **28**, 719 (1958).

(9) National Institutes of Health Postdoctoral Fellow.

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THE STRUCTURE OF NYBOMYCIN

Sir:

The antibiotic nybomycin has been described in independent reports by Strelitz, Flon and Asheshov,[1] and by Eble, Boyack, Large and DeVries[2]; apart from strong *in vitro* biological activity, its chief characteristics are its thermal stability (m.p. 325–330°) and its extreme insolubility except in concentrated acids. The report by Eble[2] showed the molecular formula of nybomycin to be $C_{16}H_{14}N_2O_4$ (rather than $C_8H_7NO_2$)[1] and established the presence of an aliphatic hydroxyl group. The present report shows the structure of nybomycin to be represented by I.

On treatment with refluxing 47% hydriodic acid (in which nybomycin is soluble), I is converted to deoxynybomycin (II, $C_{16}H_{14}N_2O_3$,[3] dec. >335°), which precipitates from this medium. Deoxynybomycin differs from the parent I by replacement

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(1) F. Strelitz, H. Flon and I. N. Asheshov, *Proc. Natl. Acad. Sci. U. S.*, **41**, 620 (1955).

(2) T. E. Eble, G. A. Boyack, C. M. Large and W. H. DeVries, *Antibiotics and Chemotherapy*, **8**, 627 (1958).

(3) Microanalyses are within accepted limits.