COMMUNICATIONS TO THE EDITOR

TETRAFLUOROHYDRAZINE

Sir:

To date there have been reported and verified only four binary compounds composed of nitrogen and fluorine: nitrogen trifluoride,[1] *cis-* and *trans*-difluorodiazine[2] and fluorine azide.[2] We wish to report the preparation and properties of a fifth such compound, tetrafluorohydrazine.

Tetrafluorohydrazine is made by the thermal reaction of nitrogen trifluoride with various metals such as stainless steel, copper, arsenic, antimony and bismuth according to the equation $2NF_3 + M \rightarrow N_2F_4 + MF$. In a flow reactor packed with copper turnings at 375° with a residence time of 13 minutes tetrafluorohydrazine was produced from nitrogen trifluoride in 42–62% conversion and a yield of 62–71%.

Tetrafluorohydrazine was purified by distillation of the residual nitrogen trifluoride from a *n*-pentane slush bath (−131.5°) which retained the tetrafluorohydrazine. Further purification was carried out by gas phase adsorption chromatography using a column of Linde Molecular Sieve 13X.[3]

The identity of tetrafluorohydrazine has been established by elemental analysis and molecular weight determination: F, calcd. 73.06%, found 71.92%; mol. wt. calcd. 104, found 107. The molecular weight differentiates this compound from the NF_2 reported by Ruff,[4] as do the reported physical properties.

The vapor pressure of tetrafluorohydrazine was measured and can be expressed by a Clausius–Clapeyron equation

$$\log P_{(\mathrm{mm})} = -692/T + 6.33$$

The boiling point of tetrafluorohydrazine is calculated to be −73° and the heat of vaporization is estimated to be 3170 cal./mole. The critical temperature of tetrafluorohydrazine by the Cagniard de la Tour tube method is 36°. From an extrapolation of the vapor pressure data a critical pressure of 77 atmospheres is estimated.

The infrared absorption spectrum of tetrafluorohydrazine consists of a very strong complex band between 9.75 and 10.75 μ and a strong broad band at 13.60 μ.

The mass spectrum of tetrafluorohydrazine given in Table I obtained on a Consolidated Model 620 Mass Spectrometer is consistent with the proposed structure

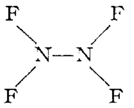

TABLE I

FRAGMENTATION PATTERN OF N_2F_4

m/e	Ion	Pattern
52	NF_2^+	90.6%
33	NF^+	100.0%
28	N_2^+	7.7%
19	F^+	4.7%
14	N^+	8.5%

Sensitivity div./μ 19.0

Instrument sensitivitv 100 div./μ for *m/e* 43 of *n*-butane

The F^{19} nuclear magnetic resonance spectrum of tetrafluorohydrazine consisted of a single broad unresolved band at a field of approximately 75 p.p.m. lower than that of the F^{19} nuclei of trifluoroacetic acid. Resolution of the expected triplet was not observed, probably because of the low symmetry of tetrafluorohydrazine.

The mechanism of formation of this compound and its chemical and physical properties are under investigation; results will be prepared at a later date.

The authors wish to express their appreciation to Dr. Keith S. McCallum for the analyses and n.m.r. spectrum of tetrafluorohydrazine.

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[1] O. Ruff, J. Fischer and F. Luft, *Z. anorg. allgem. Chem.*, **172**, 417-428 (1928).

[2] J. F. Haller, Ph.D. Thesis, Cornell University, 1942.

[3] Trade Mark of Union Carbide and Carbon Corporation, New York, N. Y.

[4] O. Ruff and L. Staub, *Z. anorg. allgem. Chem.*, **198**, 32-38 (1931).

MICROBIOLOGICAL TRANSFORMATIONS. II. THE MICROBIOLOGICAL AROMATIZATION OF STEROIDS[1]

Sir:

Although the transformation of steroids with microörganisms has been studied[1,2] extensively, no microbiological conversion of alicyclic steroids possessing an angular group at C_{10} to aromatic materials has as yet been reported. During our studies of the fermentation of 4-androstene-3,17-dione with a species of *Pseudomonas* isolated from cotton we obtained a phenolic compound (I), m.p. 123.5–125°; $\lambda_{\max}^{\text{methanol}}$ 280 mμ (ε 2,320); $[\alpha]_D$ +100.5° ($CHCl_3$); $\lambda_{\max}^{\text{KBr}}$ 2.92 μ (OH), 5.78 μ (17C=O), 5.92 μ (C=O), 6.23 μ, 6.66 μ (aromatic ring), 11.48 μ, 12.225 μ; (found: C, 76.14; H, 8.19). The phenolic compound (I) was prepared by the fermentation and extraction techniques previously described.[3] It was isolated by extraction with aqueous sodium hydroxide from a benzene–ether solution of the crude fermentation products, and was precipitated from the basic solution with either carbon dioxide or acetic acid. Acetylation of I

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[1] Previous paper: R. M. Dodson, A. H. Goldkamp and R. D. Muir, THIS JOURNAL, **79**, 3921 (1957). For excellent reviews on the microbiological transformation of steroids, see reference 1 of this paper.

[2] P. Talalay, *Physiol. Revs.*, **37**, 362 (1957).

[3] D. H. Peterson, H. C. Murray, S. H. Eppstein, L. M. Reineke, A. Weintraub, P. D. Meister and H. M. Leigh, THIS JOURNAL, **74**, 5933 (1952).