

ether, 2.8 g. of an almost colorless gum whose structure was not investigated. The partly crystalline dithiol acid was purified by vacuum sublimation followed by recrystallization from petroleum ether; yield 2.13 g. (41% from the gum), m.p. 59–61°. A mixture melting point with a sample isolated from asparagus juice showed no depression.

β,β' -Disothiuroniumisobutyric Acid Dihydroiodide.—A solution of 1.70 g. (0.005 mole) of β,β' -diiodoisobutyric acid was added to a solution of 0.76 g. (0.01 mole) of thiourea in 30 ml. of acetone and refluxed for 30 minutes. About half of the acetone was distilled off and benzene was added to precipitate an oil which readily crystallized; yield, quantitative. For analysis, it was recrystallized from water, m.p. 160–163° dec.

Anal. Calcd. for $C_6H_{14}I_2N_2O_2S_2$: C, 14.6; H, 2.87; N, 11.4. Found: C, 15.0; H, 3.02; N, 11.3.

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The Structures of the Diterpenoid Alkaloids Laurifoline and Cuauchichicine. Nomenclature Alteration

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We have recently reported¹ the isolation and structure elucidation of a new diterpenoid alkaloid, named laurifoline, from the Mexican tree *Garrya laurifolia* Hartw. It has now been called to our attention by Prof. M. Tomita of the University of Kyoto that the name "laurifoline" has been used already by him for an aporphine-like alkaloid isolated² from *Cocculus laurifolius* DC. In view of the priority of the Japanese workers and in order to avoid any confusion in the alkaloid literature, we propose that the name of our alkaloid¹ be changed to "garryfoline."

(1) C. Djerassi, C. R. Smith, A. E. Lippman, S. K. Figdor and J. Herran, *THIS JOURNAL*, **77**, 4801 (1955).

(2) M. Tomita and F. Kusuda, *Pharm. Bull. (Japan)*, **1**, 1 (1953).

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The Pyrolysis of Trifluoromethyl Sulfur Pentafluoride and its Reaction with Perfluoropropylene

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The pyrolysis of CF_3SF_5 , trifluoromethyl sulfur pentafluoride, and its reaction with C_3F_6 , perfluoropropylene, have been investigated. The experiments were carried out at temperatures between 425 and 518°. The only products found when CF_3SF_5 was heated, under the conditions outlined, were C_2F_6 , perfluoroethane, and SF_4 , sulfur tetrafluoride. When CF_3SF_5 and C_3F_6 were heated together at elevated temperatures, a series of fluorocarbons was isolated and identified.

Experimental

Apparatus.—The experiments were carried out in a closed system at approximately atmospheric pressure. The gases to be pyrolyzed or reacted were condensed air-free into a valved 1600-cc. stainless steel container. They were allowed to come to equilibrium at room temperature. The flow rate of the gases through the system was observed by their passage through a small bubbler containing a few cc. of dibutyl phthalate. The gas flow was controlled by the settings on the container valve and the stopcock on a liquid air cooled condenser-collector at the end of the system. The reactor was made from a 0.5" i.d. nickel tube 18" long and filled with extruded nickel packing. The reaction zone was wrapped with 0.25" copper wire and heated in a Hoskins furnace. The reaction temperatures were noted with a 22-gauge chromel-alumel thermocouple placed at the center of the reaction zone. The temperatures at the surface of the reactor at equilibrium were 2 to 5° higher than those observed inside the unpacked tube.

Conditions.—All experiments were carried out at approximately atmospheric pressure. The average mass flow rates were held between 0.20 and 0.40 g./min. Temperatures were maintained within $\pm 3^\circ$ of the reported values.

Materials.—Trifluoromethyl sulfur pentafluoride: this material was prepared by the electrochemical process.¹ The starting materials were a solution of $(CH_3)_2S$, dimethyl sulfide, in anhydrous HF .² The CF_3SF_5 was purified by fractionation and had a b.p. of -20.5° and a molecular weight of not less than 193 and not more than 196.

Perfluoropropylene.—This material was obtained from the Peninsular Chemical Research Co., Gainesville, Fla. It had a molecular weight of 150, b.p. -30° .

Results

Two trials were made to pyrolyze CF_3SF_5 . The first was made at 450°, a flow rate of 0.40 g./min., and an average pressure of 760 mm. No products were recovered that were characterized as different from the starting material. The second trial was made at 500°, a flow rate of 0.20 g./min. and the same pressure as in trial 1. Two grams of C_2F_6 and 2 g. of SF_4 were isolated and identified and some 16 g. of starting material was recovered out of 21 g. of CF_3SF_5 passed through the system. The nickel packing was very slightly attacked during the second trial.

Five trials were made with CF_3SF_5 and C_3F_6 . Generally, the unreacted starting materials were recovered from each trial and reused in the succeeding trial. The conditions and results of these trials are presented in Table I.

TABLE I

SUMMARY OF REACTIONS OF CF_3SF_5 AND C_3F_6 IN A NICKEL-PACKED REACTOR

| C_3F_6 , g. | CF_3SF_5 , g. | Flow, g./m. | Temp., °C. | Mm. | Products in g. | | | |
|------------------|--------------------|----------------|---------------|-----|----------------|--------|-----------------|---------------------|
| | | | | | C_2F_6 | SF_4 | SM ^a | b. above -19° |
| 17 | 21 | 0.29 | 425 | 735 | Tr. | Tr. | 37 | 1 |
| 16 | 20 | .32 | 485 | 740 | 1 | 1 | 28 | 4 |
| 26 | 33 | .28 | 512 | 760 | 1 | 13 | 15 | 17 |
| 60 | 75 | .40 | 515 | 760 | 2 | 27 | 70 | 38 |
| 45 | 55 | .28 | 518 | 740 | 1 | 27 | 25 | 47 |
| | | | | | 5 | 68 | | 107 |

^a Starting materials.

The products boiling above -19° were collected and fractionated in appropriate columns. The main fractions isolated and identified were 17 g. C_4F_{10} , 7 g. C_6F_{12} , 15 g. C_6F_{14} and 9 g. C_7F_{16} .

(1) J. H. Simons and co-workers, *J. Electrochem. Soc.*, **95**, 47 (1949).

(2) A. F. Clifford, H. K. El-Shamy, H. J. Emelús and R. N. Haszeldine, *J. Chem. Soc.*, 2372 (1953).