

solution was filtered; the filtrate was concentrated to half its original volume and stored overnight at 0° under carbon monoxide. The orange crystals were filtered and the filtrate was cooled in a bath of Dry Ice-acetone, whereupon a yellow precipitate was obtained. The crystals and the second crop of precipitate were recrystallized separately from *n*-pentane and proved to be identical. After four such recrystallizations, orange crystals melting at 93–94° with slight decomposition were obtained.

*Anal.* Calcd. for  $C_{11}H_5CoFeHgO_6$ : C, 24.13; H, 0.91; mol. wt., 547. Found<sup>7</sup>: C, 24.14; H, 1.14; mol. wt.,<sup>8</sup> 523.

The X-ray emission spectrum showed bands corresponding to Hg  $L\alpha_1$  at 35.90, Co  $K\alpha_1$  at 52.75, and Fe  $K\alpha_1$  at 57.47°.

I is soluble in most organic solvents but insoluble in water. It is air stable as a solid but decomposes slowly in solution on exposure to air. On treatment with an aqueous iodine solution (KI + I<sub>2</sub> in water) 4 moles of CO are evolved per mole of the compound, and  $\pi$ -C<sub>5</sub>H<sub>5</sub>Fe(CO)<sub>2</sub>I,<sup>9</sup> m.p. 118–120° dec., is recovered. I reacts with excess triphenylphosphine in hexane solution with the evolution of 1 mole of CO per mole of I to yield a bright yellow, insoluble, air-stable compound. This compound was difficult to purify because of its limited solubility.

The infrared spectra in the C–O stretching region of I and II in carbon tetrachloride solution showed the following bands: I (cm.<sup>-1</sup>): 1952 (m), 1972 (sh), 1983 (s), 1990 (s), 2008 (s), 2054 (sh, w), and 2066.6 (s); II (cm.<sup>-1</sup>): 1830 (m), 1961 (sh), 1985 (s), 2021 (s), 2063 (m), 2075 (s), and 2130 (w).

I probably has the geometry of two trigonal bipyramids connected at corners through mercury. It may be considered as being derived from half of Hg[ $\pi$ -C<sub>5</sub>H<sub>5</sub>Fe(CO)<sub>2</sub>]<sub>2</sub> and half of Hg[Co(CO)<sub>4</sub>]<sub>2</sub>, both of which are also probably trigonal bipyramids. However, the spectrum of I shows some interesting differences from the two moieties above and this will be the subject of a future paper.

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(7) Microanalyses were performed by Galbraith Laboratories, Knoxville, Tenn.

(8) Determined in benzene using a Mechrolab vapor pressure osmometer.

(9) T. S. Piper and G. Wilkinson, *J. Inorg. Nucl. Chem.*, **2**, 38 (1956).

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RECEIVED JULY 30, 1964

### Crystallographic Studies of Krypton Difluoride<sup>1</sup>

Sir:

The identification of KrF<sub>2</sub> has been established by Turner and Pimentel<sup>2</sup> and MacKenzie.<sup>3</sup> Samples of the difluoride, prepared by J. Malm and F. Schreiner in this laboratory, have been found to be sufficiently

(1) Based on work performed under the auspices of the U. S. Atomic Energy Commission.

(2) J. J. Turner and G. C. Pimentel, *Science*, **140**, 974 (1963).

(3) D. R. MacKenzie, *ibid.*, **141**, 3586 (1963).

stable to enable us to obtain some crystallographic information.

Single crystal oscillation and Weissenberg patterns indicate that the symmetry is tetragonal, with  $a = 6.533$  and  $c = 5.831$  Å. The calculated X-ray density, assuming four molecules in the cell, is 3.24 g./cm.<sup>3</sup>.

It is not possible to establish the space group on the basis of the available X-ray data. However, the cell appears to be primitive, and KrF<sub>2</sub> is therefore not isostructural with XeF<sub>2</sub>. The latter is also tetragonal, with  $a = 4.315$  and  $c = 6.990$  Å., but is based on a body-centered cell with space group I4/mmm.

The quality of the X-ray patterns is poor because of a tendency for the crystals to decompose upon irradiation. Hence, the assignment of the symmetry is tentative until further structural studies are completed.

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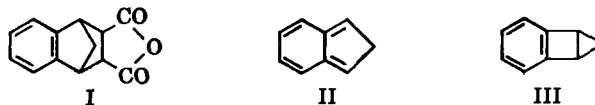
STANLEY SIGEL  
ELIZABETH GEBERT

RECEIVED JULY 20, 1964

### Formation and Capture of a Reactive Intermediate Related to Dimethylpseudoindene<sup>1</sup>

Sir:

The formation of benzenorbornene derivatives (*e.g.*, I) from the reaction of maleic anhydride with indene<sup>2–4</sup> (or from the zinc debromination of 1,3-dibromoindane in the presence of maleic anhydride<sup>5</sup>) and the thermal 1,5-hydrogen rearrangements of indene<sup>5</sup> are readily interpreted on the assumption that isoindene (II)



intervenes as an intermediate. The possibility that a valency tautomer, pseudoindene (III, benzobicyclo[2.1.0]pentene), may be capable of existence prompted the present study.

Pseudoindene (III) is a formal cycloaddition product of benzyne and cyclopropene. In attempting to realize this type of addition in practice, we observed that 1,2,3-triphenylcyclopropene<sup>6</sup> (IV) and benzyne<sup>7</sup> (V) did not react in this sense but instead gave the substitutive addition product 1,2,3,3-tetraphenylcyclopropene (VI), m.p. 177–178°, in 60–70% yield.<sup>10</sup> The structure of VI was assigned on the basis of its elemental composition. *Anal.* Calcd. for C<sub>27</sub>H<sub>20</sub>: C, 94.15; H, 5.85; mol. wt., 344. Found: C, 93.85; H, 5.97; mol. wt. 383, 325 (osmometric). Spectral peaks appeared at: ultraviolet  $\lambda_{\max}$  (ethanol) 229, 303, 316,

(1) The support of this work by the Directorate of Chemical Sciences, Air Force Office of Scientific Research, through Grant No. AF AFOSR-389-63, and by the National Science Foundation through a fellowship, is gratefully acknowledged.

(2) K. Alder, F. Pascher, and H. Vagt, *Ber.*, **75**, 1501 (1942).

(3) K. Alder and M. Fremery, *Tetrahedron*, **14**, 190 (1961).

(4) J. A. Berson and G. B. Aspin, *ibid.*, in press.

(5) W. R. Roth, *Tetrahedron Letters*, No. 17, 1009 (1964).

(6) R. Breslow and P. Dowd, *J. Am. Chem. Soc.*, **85**, 2729 (1963).

(7) Generated *in situ* from an ethylene chloride suspension of *o*-benzenediazonium carboxylate according to a modification<sup>8</sup> of the Stiles<sup>9</sup> procedure. We are indebted to Prof. L. Friedman for details.

(8) L. Friedman, private communication.

(9) M. Stiles, R. G. Miller, and U. Burckhardt, *J. Am. Chem. Soc.*, **85**, 1792 (1963).

(10) This kind of process seems to be analogous to the thermal dimerization of IV to VII.<sup>6</sup>