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On the Synthesis of and Magnetic Measurements on Xenon Tetrafluoride

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Recently the synthesis of XeF_4 was reported from the Argonne National Laboratory, and some of its physical properties were given¹. In order to obtain further information on this extraordinarily interesting compound XeF_4 was prepared in our laboratory. Since the details of the synthesis were not available the direct fluorination was performed under pressure at 400°C. The basic idea was to obtain the maximum possible amount of the product in a small volume. The product was identified by mass spectroscopic analysis². The X-ray pattern was taken and the magnetic susceptibility was also measured. At present the NMR spectra are also under investigation.

Xenon was supplied by *Société L'air Liquide*. Fluorine was generated in the electrolytic cell described elsewhere^{3,4} and purified in the essembly developed for filling fluorine into cylinders⁵. The 850 ccm reaction vessel was made of monel metal and was equipped with a valve of own construction withstanding a pressure of 150 atmospheres and being sufficiently tight even under vacuum. The evacuated and prefluorinated reaction vessel was filled with xenon (1 atm) and fluorine (3 atm) at room temperature (i.e. 50% excess of fluorine). The pressure was measured by means of a manometer, connected to the system via a diaphragm. The vessel was heated in an electric resistance furnace for 3 hours at 400°C, which corresponds to 9 atm pressure at the beginning of the reaction. After the completion of the reaction the pressure in the vessel was only about 1 atm., showing a quantitative consumption of xenon. The unreacted fluorine was pumped off and the product purified by vacuum sublimation in a glass apparatus. The same technique was used for filling the sample bulbs. After remaining for some time in glass bulbs, the colorless crystals have grown up to a size of 3 mm.

Using Gouy's method, the magnetic susceptibility was measured. At room temperature, XeF_4 is diamagnetic with a susceptibility per g-mol. $\chi_M = -50.6 \times 10^{-6}$. However, the susceptibility becomes temperature dependent below 240°K as can be seen from the diagram, constructed from the preliminary data.

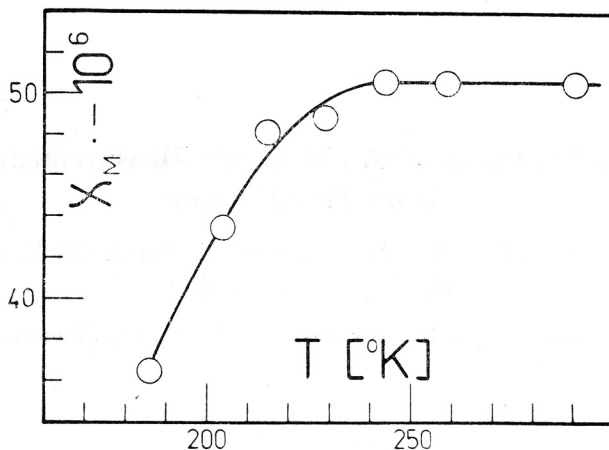


Fig. 1. The temperature dependence of the susceptibility of XeF₄.

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Note added in proof. — A letter to the Editor of *J. Am. Chem. Soc.* **84** (1962) 3593 by H. H. Claassen, H. Selig, and J. G. Malm, describing the synthesis and properties of XeF₄ appeared while this communication was in print.

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IZVOD

O sintezi i magnetskim mjerenjima na ksenonovu tetrafluoridu

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Potvrđena je sinteza XeF₄ i dani su detalji preparativne tehnike. Ksenon pod 1 atm i fluor pod 3 atm uvedeni su u reakcionu posudu od monel metala i zapremine 850 ccm. Upotrebljen je specijalno konstruirani pipac nepropusan pod vakuumom, koji izdrži 150 atm. Reakcija je bila kvantitativna nakon trosatnog zagrijavanja kod 400°C. Mjerenja magnetske susceptibilnosti pokazuju da postoji temperaturno zavisni »dijamagnetizam« ispod 240°K.

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