the Periodic Table, the "inert gases," because of their presumed nonreactivity, have occupied an isolated position, apart from the other groups of chemical elements. Discovery that at least two of these elements readily form stable compounds opens the door to investigations which should more closely integrate this group with the rest of the Periodic Table.

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References and Notes

1. See for example, Mellor's *Modern Inorganic Chemistry*, G. D. Parkes, Ed. (Longmans, Green, London, 1961).
2. Based on work performed under the auspices of the U.S. Atomic Energy Commission.
3. A. Van Antropoff, K. Weil, H. Fraüenhof, [*Naturwissenschaften* **20**, 688 (1932)] claimed the synthesis of a krypton chlorine compound with the aid of an electric discharge. O. Ruff and W. Menzel [*Z. Anorg. Allgem. Chem.* **213**, 206 (1933)] attempted to make the fluorine compound by an analogous technique and found no evidence of reaction. D. Yost and A. L. Kaye [*J. Am. Chem. Soc.* **55**, 3890 (1933)] found no reaction between krypton and chlorine under ultraviolet irradiation and between xenon and fluorine in an electric discharge. Yost and Kaye pointed out that Pauling and others had suggested the possibility of such reactions and that their results were only tentative. However, Antropoff [A. Van Antropoff, H. Frauenhof, K. H. Krüger, *Naturwissenschaften* **21**, 315 (1933)] conceded a partial misinterpretation of his earlier results, and there has been little subsequent research.
4. C. D. Cooper, G. C. Cobb, E. L. Tolnas, *J. Mol. Spectry.* **7**, 223 (1961).
5. N. Bartlett, *Proc. Chem. Soc.* **1962**, 218 (1962).
6. At Argonne National Laboratory the reaction with platinum hexafluoride was duplicated and xenon was shown to react at room temperature with ruthenium hexafluoride, but not with uranium, neptunium, or iridium hexafluorides. When the ruthenium hexafluoride system was studied quantitatively, a larger than equimolar consumption of the hexafluoride was observed, and some reduction of the ruthenium seemed to occur. This suggested the role of a hexafluoride as a fluorine carrier and led to the studies with xenon and fluorine. Xenon also reacts with plutonium hexafluoride. The course of this reaction, however, has not yet been elucidated (M. Steindler and J. Fischer, Argonne Chem. Eng. Div., private communication).
7. H. H. Claassen, H. Selig, J. G. Malm, *J. Am. Chem. Soc.* **84**, 3593 (1962).
8. M. H. Studier and E. N. Sloth, *J. Phys. Chem.*, in press.
9. Other species found (for example, Xe, O_2, H_2F_2) can be explained without reference to xenon compounds.
10. Xenon which had been irradiated with slow neutrons in a nuclear reactor was used to prepare the xenon tetrafluoride used in the hydrolysis experiment described. The behavior of the xenon was followed by observing the gamma activity associated with the resulting mixture of radioactive xenon isotopes.
11. P. R. Fields, L. Stein, M. H. Zirin, submitted for publication.

2 October 1962

Azeotropic Freeze-Drying

Abstract. Spheres of comminuted frozen meat were freeze-dried by boiling in toluene under reduced pressure. The dehydration was performed at 30°C and apparently took place without melting the ice. The dried product rehydrated readily and showed all the characteristics of freeze-dried defatted meat.

Freeze-drying is possible without vacuum, so long as the water vapor pressure of the medium surrounding a frozen specimen is below the vapor pressure of ice at that temperature. This process was demonstrated in the dry air freeze-drying experiments of Meryman (*1*) and Lewin and Mateles (*2*).

Azeotropic mixtures are composed of two or more compounds that distill together without decomposition or reaction. The boiling point of an azeotropic mixture is constant, and is lower than the boiling point of its constituents. The composition of the azeotropic vapor depends upon the molecular weight and vapor pressures of the mixture components at a given temperature. The purpose of our work was to test the feasibility of azeotropic freeze-drying.

At 0°C water and toluene form an azeotrope with a vapor pressure of 12.1 mm-Hg. Of this pressure 4.6 mm represents the vapor pressure of ice and 7.5 mm the vapor pressure of toluene. The composition of the azeotrope is 10.7 percent water and 89.3 percent toluene. If the temperature is increased, but the water is still frozen, the composition of the azeotrope changes. The vapor pressure of the solvent increases, while the vapor pressure of water depends on the temperature of ice. The azeotropic mixture becomes unsaturated. The degree of saturation depends on the rate of heat supply to sublimate the ice, and on the rate of vapor removal from the boiling flask. The vapor pressure at the solvent-ice contact is greater than the vapor pressure of solvent in other parts of the flask. Thus, violent boiling occurs around a frozen specimen. Due to the temperature difference between the condensor and the boiling flask, the azeotrope is rapidly removed. Heat of 620 cal is necessary to sublimate 1 g of ice at 0°C, and since the vapors are rapidly removed, it is not likely that the ice will melt. These postulates were tested experimentally.

Approximately 300 ml of toluene were placed in a 700-ml round bottom flask and heated to 30°C. The flask was fitted with an insulated Bidwell-Sterling receiver arm, a condensor, a manometer and a vacuum line. A 7-g sphere of frozen (−15°C) comminuted meat, about 1.5 cm in diameter, was placed in the flask and a vacuum was drawn immediately. When the pressure reached 33 mm-Hg (in approximately 60 seconds) the contents of the flask surrounding the meat sample came to a vigorous boil. Vapors rosè to the condensor, where they separated into water and toluene. Water settled in the Bidwell-Sterling receiver while toluene returned to the flask. At the end of 90 minutes, 4.8 ml of water had accumulated in the receiver, and the boiling subsided. At that time air was allowed to enter the system. The sample was withdrawn and placed for 45 minutes in an air oven at 101°C to dry off the solvent.

After drying in the oven, the sample had the typical porous structure of freeze-dried meat. The weight of the dry material was 1.5 g and its dimensions appeared unchanged. Upon immersion in water, the material promptly rehydrated to the consistency and appearance of very lean, raw, ground meat. Its taste was that of uncooked beef.

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2. L. M. Lewin and R. I. Mateles, *Food Technol.* **16**, 94 (1962).

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Simple Apparatus for Ultrafiltration

Abstract. A rapid and highly efficient ultrafiltration apparatus has been devised from readily available laboratory materials costing less than $5. Employing a fritted glass tube to support an evacuated dialysis bag, it accommodates as little as 1 milliliter and is ideally suited to dialysis of small volumes and concentration of macromolecules for chromatography and electrophoresis.

In our laboratory it is routinely necessary to concentrate protein solutions for electrophoretic analysis and to remove protein from an amino acid solution for chromatographic analysis. These two operations are carried out simultaneously with the apparatus shown in Fig. 1. The grommet (Walsco Electronics, No. 7034-F; inside diameter, ¼ in.; mounting hole, ⅜ in.) is