of an alcohol containing one more carbon atom, as described by Wojcik and Adkins.[3] No other hydrocarbon was isolated in quantity sufficient for identification, although there was some evidence that a fraction boiling at 130–50° contained *n*-nonane. However, inasmuch as dodecyl and tetradecyl alcohols are the principal constituents of "Lorol," the amount of hydrocarbon formed from any of the other alcohols present should be small.

[3] Wojcik and Adkins, This Journal, **55**, 1293 (1933).

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COMMUNICATIONS TO THE EDITOR

PHOSPHOROUS FLUOROCHLORIDES

Sir:

We have found that the fluorination of PCl_3 by SbF_3 in the presence of $SbCl_5$[1] yields three gases, PF_3 in large amounts and two new gases, PF_2Cl boiling at approximately −48° and melting at approximately −166°, and small amounts of a $PFCl_2$ boiling a little below room temperature. These new substances are colorless both as gas and liquid, and fume in moist air. Variations of the experimental conditions indicate that the best yields are obtained when 450 parts of PCl_3 and 10 parts of $SbCl_5$ are vigorously stirred while sublimed SbF_3 is slowly added at room temperature.

The low yields by the above method caused us to try converting the PF_3 to the fluorochlorides. It was found that by passing an equimolecular gaseous mixture of PCl_3 and PF_3 through a glass tube filled with broken porcelain heated by an electric furnace so that the temperature of the exit gases was 200°, 50% of the mixture was converted to PF_2Cl and $PFCl_2$ in one pass. We are trying this same general method on mixtures of other non-polar chlorides and fluorides. The complete results of these investigations will be reported later.

[1] Booth and Swinehart, This Journal, **54**, 4751 (1932).

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AN ATTEMPT TO PREPARE A CHLORIDE OR FLUORIDE OF XENON

Sir:

Frequent attempts have been made to prepare compounds of the noble gases with more electronegative elements, but the results have not been satisfyingly definite. Von Antropoff[1] has described briefly some experiments which indicate that krypton chloride and bromide exist, but no determinations of their composition and physical properties have been published. Both von Antropoff[1] and Pauling[2] have suggested that combination might possibly result with krypton or, more probably, with the heavier xenon. We have carried out experiments in which xenon was mixed with both chlorine and fluorine in an attempt to obtain these halides of xenon.

A mixture of xenon (70 mm.) and chlorine (225 mm.) contained in a quartz tube was irradiated with the light from a mercury arc in Pyrex and in quartz, and from a carbon arc. The source of light in all cases was placed close to the container. The pressure was measured by means of a Pyrex click gage attached through a graded seal to the quartz vessel. In no case was any pressure change observed during or at the end of a twelve-hour exposure. When the gas mixture was condensed by means of liquid air, a small amount of red solid was observed whose color disappeared shortly after removing the liquid air. The mixture was next transferred to a Pyrex bulb equipped with tungsten electrodes. After sparking for varying periods of time and then condensing the contents of the bulb with liquid air, larger amounts of the red solid noted above appeared. A number of tests showed that, in all probability, the red substance was the colored modification of hydrogen chloride described by Giauque.[3] Small amounts of hydrogen chloride are difficult to remove from chlorine and it was doubtless present as an impurity; some may have also been formed by the action of the highly activated chlorine on small amounts of water adsorbed on the walls of the vessel. No definite evidence for the presence of a xenon chloride was found.

It was found that fluorine, carefully freed from hydrogen fluoride, could be kept for an indefinite period in quartz bulbs which had been baked out under vacuum.[4] The small amount of silicon tetrafluoride and oxygen formed as the result of sealing the bulbs at long narrow capillary constrictions did not appear to promote any etching action. By means of quartz "break-offs" a mixture of some 600 mm. of fluorine and 30 mm. of xenon was prepared in an all-quartz apparatus provided with copper electrodes. On condensing the mixture in a side tube at −210° no visual evidence of the existence of anything except liquid fluorine and solid xenon was observed. When the refrigerant was removed the fluorine boiled away rapidly, leaving a volatile white solid with all the properties of xenon. A high voltage (30 k.v.) discharge was then passed through the mixed gases for varying intervals of time. The contents of the apparatus were con-

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(1) Von Antropoff, Weil and Frauenhof, *Naturwissenschaften*, **20**, 688 (1932).

(2) Private communication.

(3) Giauque, THIS JOURNAL, **50**, 101 (1928).

(4) See also von Wartenberg, Sprenger and Taylor, *Z. physik. Chem.*, Bodenstein Festband, 61 (1931).

densed out frequently, but the appearance and properties were no different from those observed before applying the discharge. It was noted that the side tube was appreciably attacked after some time, and this might be due to the action of a reactive xenon fluoride (compare rhenium hexafluoride) or to the presence of a small amount of moisture in the xenon. It cannot be said that definite evidence for compound formation was found. It does not follow, of course, that xenon fluoride is incapable of existing. It is known, for example, that nitrogen and fluorine do not combine in an electrical discharge, but when prepared indirectly nitrogen trifluoride is a very stable compound.

We are greatly indebted to Dr. F. J. Allen, who kindly supplied the xenon used in the experiments.

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PREPARATION OF PURE 1-BUTENE AND ISOBUTENE

Sir:

Recently [*Compt. rend.*, **196**, 973 (1933)] Matignon, Moureu and Dode reported that pure aluminum oxide yields only 85% of 1-butene, the remainder being 2-butene.

This is not in agreement with the results previously obtained in our laboratories where it has been found that 1-butene without a trace of 2-butene can be prepared by the dehydration of *n*-butyl alcohol with aluminum oxide. The procedure is as follows: *n*-butyl alcohol is passed with a speed of 75 g. per hour through a glass tube (length, 50 cm.; inner diameter, 2 cm.), filled with 6–8 mesh aluminum oxide and maintained at a temperature of 375–425°. The butenes formed are collected and subjected to distillation in a low-temperature Podbielniak precision distilling apparatus [*Ind. Eng. Chem., Anal. Ed.*, **5**, 172 (1933)].

The aluminum oxide was prepared by the precipitation of aluminum nitrate with ammonium hydroxide; activated alumina received from Alcoa Ore Company, St. Louis, Missouri, gives 1-butene 99.6% pure.

The discrepancy between these results and those of Matignon, Moureu and Dode lies probably in the fact that these authors analyzed the product by converting it into dibromide and subjecting the dibromide to distillation. This method of analysis is not a very reliable one, since isomerization of dibromide can readily take place during the distillation.

V. N. Ipatiev [*Ber.*, **36**, 2011 (1903)] obtained pure isobutene by the dehydration of isobutyl alcohol using aluminum oxide. This work has