

It now appears that Professor Chichibabin did not as a matter of fact report the diazotate reaction as a method for the preparation of alpha-phenoxy pyridine, its isolation being merely incidental in the reaction which he was investigating. Unfortunately, we had only abstracts of the Russian article at our disposal.

The designation of 2-phenoxy pyridine as the only alpha-substituted pyridine reported in the literature (page 297) is a very patent error, since a large number of alpha-substituted pyridines have been known for many years and the writer had dealt with derivatives of such compounds in two earlier publications, as well as in the paper under discussion. (Note reference to Grave's article page 298.) On examination it was found that this error was due to the accidental omission of *aryloxy-* after *alpha-* in the term "alpha-substituted pyridine," in the final typing of the manuscript.

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RECEIVED MARCH 31, 1937

FLUOROCARBONS

Sir:

From a reaction mixture of carbon and fluorine the following fluorocarbons have been isolated and identified by their molecular weights: profluorane, C_3F_8 ; bufluorane, C_4F_{10} ; pentafluorane, C_5F_{12} ; and hexfluorane, C_6F_{14} . The well-known compounds methfluorane, CF_4 , and ethfluorane, C_2F_6 , were also formed in the reaction. The separation was made by distillation and the molecular weights were determined with an all-glass density balance. Seven to fifteen grams of each compound was obtained. The boiling and freezing points have been determined approximately and are: profluorane f. p. -183° , b. p. -36° ; bufluorane f. p. -84.5° , b. p. $+4^\circ$; pentafluorane f. p. -10° , b. p. $+30^\circ$; and hexfluorane f. p. -4° , b. p. $+60^\circ$.

The distillation was performed in a low temperature column packed with glass helices. It was carried out at 200 mm. and the temperature remained constant within 1° during the collection of each fraction. The molecular weights as obtained from the density balance and determined at short intervals during the distillation remained constant so long as the temperature remained constant. Each fraction was redistilled and the same molecular weights obtained: C_3F_8 184,

188; C_4F_{10} 234, 238; C_5F_{12} 277, 288; and C_6F_{14} 328, 338. The precision of the density balance decreased as the molecular weight increased, and the increased adsorption of the higher boiling substances resulted in low values. The balance was calibrated with oxygen and carbon dioxide and was checked with CF_4 and C_2F_6 , which gave the theoretical values of 88 and 138. The redistilled fractions had relatively sharp melting points when observed visually.

The existence of these compounds has been previously postulated from the properties of small quantities of mixtures of fluorocarbons [Ruff and Keim, *Z. anorg. allgem. Chem.*, **192**, 249 (1930); Ruff and Bretschneider, *ibid.*, **210**, 173 (1933); **217**, 1 (1934); **217**, 19 (1934)]. Their separation and individual identification however, has not been reported previously. A greater quantity will be made and the chemical and physical properties determined.

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RECEIVED MAY 28, 1937

A CONCENTRATION OF N^{15} BY A CHEMICAL EXCHANGE REACTION

Sir:

Using the exchange reaction between ammonia gas and ammonium sulfate solution suggested by Urey and Greiff [THIS JOURNAL **57**, 321 (1935)] and investigated by Aten and Urey [*Phys. Rev.*, **50**, 575 (1936)] and using the apparatus devised by Huffman and Urey [*Ind. Eng. Chem.*, **29**, 531 (1937)] we have attempted to concentrate the heavy isotope of nitrogen for use in biochemical and chemical investigations. The method consists of pumping a 30% solution of ammonium sulfate at a constant rate of 15 cc. per minute into the top of the fractionation column under low pressure, liberating the ammonia from the salt in solution at the bottom by the addition of sodium hydroxide and stripping the ammonia from the solution by a packed stripping column. This ammonia was fed back to the bottom of the column and escaped from the top. As the result of a thirteen-day run of this kind we have produced nitrogen containing 2.54% of N^{15} , a $6\frac{1}{2}$ fold increase in concentration.

The transport of heavy ammonia is equal to the total flow multiplied by the difference in concentration of heavy ammonia in the solution and gas