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Report No. ARF-C6001-5
(Progress Report)

PREPARATION AND EVALUATION OF NEW HYDRAULIC FLUIDS

Bureau of Ships
Washington 25, D. C.

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Report No. ARF-C6001-5
(9) (Progress Report) 1

(6) PREPARATION AND EVALUATION OF NEW HYDRAULIC FLUIDS

March 28 through April 27, 1963

Bureau of Ships
Washington 25, D. C.

(15) Contract No. NObs-88249
ARF Project C6001

The purpose of this project is to develop new fire-resistant hydraulic fluids based on fluorinated, sulfur-containing compounds. The compounds will be synthesized specifically to meet the critical property requirements. Various derivatives of sulfur hexafluoride and other fluorinated materials are being investigated.

The program consists of four phases: Simon's cell fluorinations, fluorinations with metallic fluorides, addition of sulfur-chloride pentafluoride to olefins, and determination of physical and chemical properties.

Simon's Cell Fluorinations

The apparatus used in these experiments was fully explained in Report No. ARF-C6001-3. For the preparation of bis-heptafluoropropylsulfur tetrafluoride, $(C_3F_7)_2SF_4$, a 10% solution of propylsulfide was electrolyzed for about 5 hours. A sufficient amount of $(C_3F_7)_2SF_4$ is now available for determining the necessary physical constants.

The corresponding propyl derivative, $(C_4F_9)_2SF_4$, is currently being prepared from butyl sulfide. Work has also begun on the preparation of perfluoro-s-trithiane dodecafluoride, $(CF_2SF_4)_3$, by the electrolysis of s-trithiane. It is anticipated that this cyclic material may have a desirable viscosity-temperature relationship. Dresdner and Young[1] prepared this compound by the method described above, but since the yield is only about 1% it is necessary to use a large amount of starting material in order to isolate a few grams of product. The cell presently being used has a capacity of only 150 ml; therefore, a larger cell is being constructed.

A few preliminary reactions were carried out in the small cell in order to define the operating conditions. The first run, in which the condenser was maintained at -78°C, proceeded very slowly (as evidenced by the rate of gas evolution). Subsequent warming to -30°C increased the rate of the reaction, but the current was only 0.2 amps when the potential difference was set at the recommended 4.3 volts. At this rate it would take about 60 hours to complete the reaction.

Sodium fluoride, which acts as a current carrier, was added to the next run. The reaction proceeded at a faster rate, but after about 1-1/2 hours a short circuit developed. It was found

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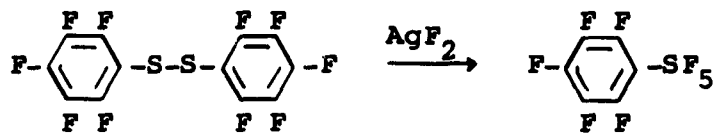
[1]Dresdner, R. D. and Young, A., J. Am. Chem. Soc. 81, 574, 1959.

that the paraffin wax which was used to insulate the electrodes from the cell wall had dissolved. This problem was corrected by replacing the paraffin with Kel-F wax. Then, another run was carried out. The products are currently being investigated.

Fluorinations with Metallic Fluoride

Work on the preparation of pentafluorosulfur pentafluorobenzene, $C_6F_5SF_5$, is proceeding, Equation 1.



(1)

As pointed out in Report No. ARF-C6001-4, the main product formed in this reaction was the intermediate $C_6F_5SF_3$ rather than $C_6F_5SF_5$. The reaction was repeated with a 6:1 mole ratio of silver difluoride to decafluorodiphenyldisulfide.

The trifluoride, $C_6F_5SF_3$, is easily hydrolyzed, whereas the pentafluoride, $C_6F_5SF_5$, should be stable to hydrolysis. Hence hydrolysis appeared to be a convenient way of disposing of the trifluoride and obtaining pure pentafluoride. Therefore, the sample was hydrolyzed, washed with sodium bicarbonate, and extracted with diethyl ether. The products from the hydrolysis should remain in the water extract. Chromatographic analysis of the product remaining in the ether extract showed one major peak and only small amounts of impurities. The observed molecular

weight of the product was 308 compared with 294 calculated for $C_6F_5SF_5$. The NMR analysis of the product is now being determined.

Respectfully submitted,

ARMOUR RESEARCH FOUNDATION
of Illinois Institute of Technology

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