# Hydrolysis of Heptafluoropropylphosphonous Diiodide and Bisheptafluoropropylphosphinous Iodide. Formation of Bisheptafluoropropylphospine

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HEPTAFLUOROPROPYLPHOSPHONOUS diiodide and bisheptafluoropropylphosphinous iodide formed by the interaction of heptafluoropropyl iodide with phosphorus were characterized as the *N,N*-dimethyl amides. Treatment of the mixture of iodides with water gave the expected heptafluoropropylphosphinic acid, and heptafluoropropane, but unexpectedly gave bisheptafluoropropylphosphine. The quantity of phosphine formed increased on refluxing with aqueous alkali, and was greatest when the acid iodides were treated with solid alkali. These results contrasted with those of Emeleus and Smith (*1*) who observed the formation of heptafluoropropylphosphinic acid and heptafluoropropane, but did not observe the formation of bisheptafluoropropylphosphine under comparable experimental conditions.

## ACKNOWLEDGMENT

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## LITERATURE CITED

(1) Emeleus, H.J., Smith, J.D., *J. Chem. Soc.* **375**, (1959).

Table I. Summary of Reactions

| Reactants | Conditions | Products | Boiling range °C. | Yield, % | | Analysis[a] Calcd. | Found |
|---|---|---|---|---|---|---|---|
| HFPI + P | 190° C. 48 hours | $C_3F_7PI_2$, 67% | 27–29 | 47.5 | %C | 12.4 | 12.3 |
| | | $(C_3F_7)_2PI$, 33% | (for mixture) | 25.7 | %P | 6.4 | 6.4 |
| | | | | | %I | 35.2 | 36.3 |
| $HFPP_2$+ bis HFPPI + $(CH_3)_2NH$ | Acid iodides added to cooled Pet. ether soln. of amine | $C_3FP[N(CH_3)_2]_2$ | 27–29 | ... | %C | 29.2 | 28.7 |
| | | | | | %H | 4.2 | 4.6 |
| | | | | | %P | 10.8 | 10.5 |
| | | | | | %N | 9.7 | 9.4 |
| | | $(C_3F_7)_2PN(CH_3)_2$ | 23–25 | ... | %C | 23.1 | 24.6 |
| | | | | | %H | 1.5 | 2.2 |
| | | | | | %P | 7.5 | 7.9 |
| | | | | | %N | 3.4 | 4.9 |
| $HFPPI_2$ + bisHFPPI + $H_2O$ | 3-hour reflux | $C_3F_7H$ | < room temperature | 27 | %C | 21.2 | 22.2 |
| | | | | | %H | 0.6 | 0.8 |
| | | $C_3F_7P(O)H(OH)$[b] | 90 (1 mm.) | 7 | %C | 15.4 | 15.3 |
| | | | | | %H | 0.9 | 1.0 |
| | | | | | %P | 13.2 | 13.3 |
| $HFPPI_2$ + bis HFPPI + NaOH | Cooled | $(C_3F_7)_2PH$ | 30–32 | | %C | 19.5 | 19.7 |
| | | | | | %H | 0.3 | 0.4 |
| | | | | 31 | %P | 8.4 | 8.0 |
| $C_3F_7P(O)H(OH)$ $H_2O_2$ + NaOH | Neutralized to phenolphthalene | $C_3F_7P(O)(ONa)_2$ | solid | ... | %Na | 15.7 | 15.9 |
| | | | | | %C | 12.2 | 12.3 |
| | | | | | %P | 10.5 | 10.7 |

[a] Analyses are by Schwarzkopf Microanalytical Laboratories, Woodside 77, N. Y.
[b] A small amount of $(C_3F_7)_2PH$ was also isolated.