trans-2-**Chloroindanol-1 (28) and** *cis*-**2-Chloroindanol-1 (31)**.—These chlorohydrins were prepared according to the procedure of Suter and Lutz[25] by addition of chlorine to indene, treatment with aqueous ethanol and fractional crystallization of the resulting mixture. Deposited from ether the *cis* isomer had m.p. 112–113°, and the *trans* isomer (from benzene) had m.p. 124–126°. Both of these m.p.'s are corrected.

trans-**2-Bromoindanol-1 (29)**.—The action of aqueous N-bromosuccinimide on indene gave this bromohydrin, m.p. 130–131° (cor.), when crystallized from benzene-petroleum ether (b.p. 60–75°).[21]

trans-**2-Iodoindanol-1 (30)**.—The literature procedure[26] was duplicated to give the iodohydrin, which was purified from benzene-petroleum ether (b.p. 60–75°); m.p. 124–126° dec.

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[25] C. M. Suter and G. A. Lutz, THIS JOURNAL, **60**, 1360 (1938).

[26] M. Tiffeneau and O. Orekhoff, *Bull. soc. chim.*, **27**, 782 (1920).

COMMUNICATIONS TO THE EDITOR

THE SYNTHESIS OF FLUOROCARBON PHOSPHORUS IODIDES[1]

Sir:

A news report[2] of our work on fluorocarbon-phosphorus compounds produced from CF_3PI_2 or $(CF_3)_2PI$ led to inquiries concerning the synthesis of these iodides by the CF_3I–P_4 reaction. Hence it appears timely to describe the conditions under which we obtained them in yields as good as originally reported,[3] and with far higher utilization of CF_3I per single run. A convenient short-cut method also is described.

In the work at Corona (Table I) most of the runs were done in a liter-size stainless-steel bomb, which had been thoroughly cleaned, and dried at 200°, before loading with the thoroughly-dried reagents in a dry argon atmosphere. In all cases the CF_3I had been made from iodine and silver trifluoroacetate[4] and purified by distillation. Each heating was for 48 hr. at 200°. For comparison, the first line of Table I summarizes one of the experiments by the earlier workers,[3] showing their results after twice-repeated heatings of the nonvolatile part with the $(CF_3)_3P$ and the recovered CF_3I.

From this table it appears that equally good results can be obtained with red or white phosphorus, except that the presence of iodine seems to be more important for the latter. The improvement over previously reported results is attributed to the rigorous exclusion of atmospheric contaminants.

For the work at the University it was considered that the reaction $AgCO_2CF_3 + I_2 \rightarrow CO_2 + AgI + CF_3I$ might well occur in the presence of phosphorus, which then could react to form the CF_3–P–I compounds. Thus a convenient one-step procedure was devised: the three well-dried solid reagents were loaded into a one-liter mild-steel bomb in such a way that the silver trifluoroacetate formed a layer between the red phosphorus and the iodine; or in another case the premature I_2–P_x reaction was avoided by cooling the bomb to −78° for the loading process. A heating of 88 g. of $AgCO_2CF_3$, 50 g. of P_x, and 100 g. of I_2, for 120 hr. at 195°, produced 9 g. of $(CF_3)_3P$, 13 g. of $(CF_3)_2PI$, and 10 g. of CF_3PI_2; all representing 58% of the CF_3 groups. Other products included 4 g. of CF_3I, 3 g. of POF_3, and 5 g. of a new compound of yet-uncertain composition (m.p. 0°; mol. wt. 196; vapor tension 19 mm. at 0°), which was separated from the $(CF_3)_2PI$ fraction (44 mm. at 0°) by a fairly tedious process of high-vacuum distillation. The presence of this new substance does no harm when the $(CF_3)_2PI$ is used for making $P_2(CF_3)_4$[3] or $(CF_3)_2PCl$, for the former can be purified by treatment with water and the latter by distillation. If pure $(CF_3)_3P$ is desired, most of the POF_3 can be removed by distillation at atmospheric pressure, and the remainder by hydrolysis.

TABLE I

Run	Reactants, g.	% Conversion to $(CF_3)_3P$	$(CF_3)_2PI$	CF_3PI_2	% CF_3I utilized
(BEH)	60 CF_3I; 50 P_4; 50 I_2	13	19	18	50
1	50 CF_3I; 50 P_4; 50 I_2	22	60	11	93
2	342 CF_3I; 175 P_x (red)	30	50	16	96
3	406 CF_3I; P_x residue of 2	49	32	14	95
4	60 CF_3I; 55 P_4	5	10		12

The use of the fluorocarbon-phosphorus iodides for making a considerable variety of new compounds will be reported in detail in later papers.

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[2] *Chem. and Eng. News*, **34**, 2124 (1956).

[3] F. W. Bennett, H. J. Emeléus and R. N. Haszeldine, *J. Chem. Soc.*, 1568 (1953).

[4] A. L. Henne and W. G. Finnegan, THIS JOURNAL, **72**, 3806 (1950).