OSAZONES FROM PERFLUOROOLEFINS

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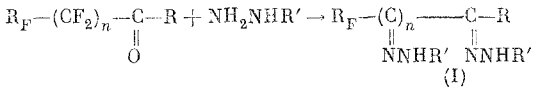
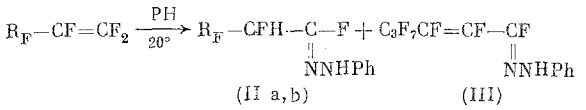
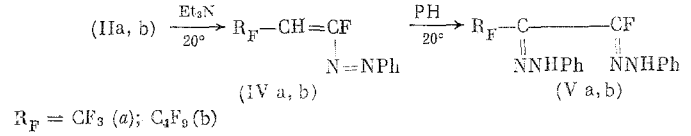
It was shown by us in [1] that polyfluoroketones are a convenient source of derivatives of polycarbonyl compounds (I)



It turned out that it is possible to use perfluoroolefins for this purpose. It is known [2] that perfluoropropene forms (IIa) with phenylhydrazine (PH). It was found by us that in the case of perfluorohex-1-ene up to 10% (III) was formed together with (IIb). The yield of (III) may be brought up to quantitative with an excess of PH.



Dehydrofluorination of (IIa, b) under the action of Et_3N led to the phenylazofluoroolefins (IVa, b) which readily added PH being converted into the osazones (Va, b).



$R_F = CF_3$ (a); C_4F_9 (b)

The structure of products was proved by data of ^1H and ^{19}F NMR, IR, and mass spectra.

TABLE 1

Compound	bp, °C (mm) mp	Yield, %	Found, %			Empirical formula	Calculated, %		
			C	H	N		C	H	N
(IIb)	125–127(8)	72	37,55	1,63	7,20	$C_{12}H_7F_{11}N_2$	37,11	1,80	7,21
(III)	83–84	73	39,21	1,57	7,89	$C_{12}H_6F_{10}N_2$	39,13	1,63	7,61
(IVa)	64–65(20)	95	49,31	2,75	12,82	$C_9H_6F_4N_2$	49,54	2,75	12,84
(IVb)	78–79(10)	91	39,12	1,71	7,85	$C_{12}H_6F_{10}N_2$	39,13	1,63	7,61
(Va)	126–127	50	54,37	3,74	16,91	$C_{15}H_{12}F_4N_4$	55,56	3,70	17,28

LITERATURE CITED

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2. R. A. Carboni and R. V. Lindsey, J. Am. Chem. Soc., 80, 5793 (1958).

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