PENTAFLUOROPHENYLIMINOTHIODIHALIDES AND THEIR CONVERSIONS IN ACID MEDIA

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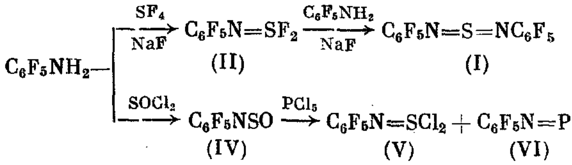
UDC 542.91:547.551.51:546.22'161

Organoiminothiodihalides, $RN{=}SHal_2$, are convenient starting substances in the synthesis of a series of compounds containing sulfur and nitrogen. Organoiminothiodifluorides are obtained by the action of SF_4 on the appropriate nitriles, isocyanates [1], primary amines [2, 3], and also upon the reaction of alkyliminothiodichlorides with sulfur dialkylaminotrifluoride [4]. Organoiminothiodichlorides are synthesized by the reaction of N-sulfonylarylamines with PCl_5 [5] and arylamines with SCl_2 [6]. The goal of the present work is to prepare pentafluorophenyliminothiodihalides (fluoride and chloride) and to study their behavior in acid media.

Earlier [7], the reaction of pentafluoroaniline with SF_4 was studied, which was conducted in a glass apparatus at 20°C, and even with an excess of SF_4 gave only bis(pentafluorophenyl)thiodiimide (I). It is found by us that pentafluorophenyliminothiodifluoride (II) is obtained by reacting pentafluoroaniline with SF_4 in the presence of NaF at 0-3°C in an apparatus made of stainless steel or polytetrafluoroethylene. Under these conditions, thiodifluoride derivatives can also be obtained from other amines, including heterocyclic amines. For example, pyrimidyl-2-iminothiodifluoride (III) is obtained by the action of SF_4 on 2-aminopyrimidine.

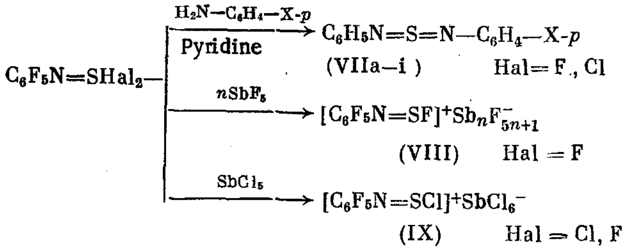
N-Sulfonylpentafluoroaniline (IV), obtained by the action of $SOCl_2$ on pentafluoroaniline, upon action with PCl_5 forms pentafluorophenyliminothiodichloride (V) with a high yield. One must note that upon the presence of traces of HCl in the PCl_5 the appearance of trichlorophosphazapentafluorophenyl (VI) is observed. Such a reaction course was observed earlier for N-sulfonyl-m-nitroaniline and N-sulfonyl-p-bromoaniline [5].



$$C_6F_5NH_2 \xrightarrow[NaF]{SF_4} \underset{(II)}{C_6F_5N{=}SF_2} \xrightarrow[NaF]{C_6F_5NH_2} \underset{(I)}{C_6F_5N{=}S{=}NC_6F_5}$$

$$C_6F_5NH_2 \xrightarrow{SOCl_2} \underset{(IV)}{C_6F_5NSO} \xrightarrow{PCl_5} \underset{(V)}{C_6F_5N{=}SCl_2} + \underset{(VI)}{C_6F_5N{=}PCl_3}$$

Compounds (II) and (V) are easily hydrolyzed by moist air. Their structures are verified by chemical conversions and by spectral data. So, (I) is formed with a practically quantitative yield upon reaction of (II) with pentafluoroaniline in the presence of NaF. N-Pentafluorophenyl-N'-arylthiodiimides (VIIa-i) are obtained in high yields upon the reaction of (II) and (V) with aromatic amines (Table 1). The reaction of 4-amino-N,N-dimethylaniline with (V) even at −40°C in a Me_3N medium proceeds with intense tarring of the reaction mixture, from which individual products could not be isolated.



$$C_6F_5N{=}SHal_2 \xrightarrow[\text{Pyridine}]{H_2N-C_6H_4-X\text{-}p} \underset{(VIIa-i)}{C_6H_5N{=}S{=}N-C_6H_4-X\text{-}p} \quad Hal = F, Cl$$

$$C_6F_5N{=}SHal_2 \xrightarrow{nSbF_5} \underset{(VIII)}{[C_6F_5N{=}SF]^+Sb_nF_{5n+1}^-} \quad Hal = F$$

$$C_6F_5N{=}SHal_2 \xrightarrow{SbCl_5} \underset{(IX)}{[C_6F_5N{=}SCl]^+SbCl_6^-} \quad Hal = Cl, F$$

X = H(VIIa), Me(VIIb); MeO(VIIc), F(VIId), Cl(VIIe), Br(VIIf), I(VIIg), COMe(VIIh), NO_2(VIIi)

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0568-5230/79/2811-2352$07.50

TABLE 1. N-Pentafluorophenyl-N'-arylthiodiimides

Compound	mp, °C	Yield, %	Found, %						Empirical formula	Calculated, %						UV spectrum: λ_{max}, nm (log ε) of long-wave band
			C	H	F	N	S	Hal		C	H	F	N	S	Hal	
(VIIa)	87–88	87	47,4	1,48	31,3	9,30	10,4		$C_{12}H_5F_5N_2S$	47,4	1,64	31,3	9,21	10,5		406 (3,97)
(VIIb)	103–104	84	49,3	2,34	30,0	8,62	10,2		$C_{13}H_7F_5N_2S$	49,1	2,20	29,9	8,80	10,1		413 (4,11)
(VIIc)	117–118	80	46,7	1,89	28,0	8,49	9,89		$C_{13}H_7F_5N_2OS$	46,7	2,10	28,4	8,38	9,58		417 (4,30)
(VIId)	90–91	85	45,0	1,60	35,3	8,38	9,59		$C_{12}H_4F_6N_2S$	44,7	1,24	35,4	8,70	9,94		408 (4,11)
(VIIe)	92–93	82	42,3	1,40	28,2	8,45	9,63	10,4	$C_{12}H_4F_5N_2ClS$	42,5	1,18	28,1	8,27	9,45	10,5	413 (4,16)
(VIIf)	98–99	83	37,7	1,12	24,6	7,14	8,82	20,5	$C_{12}H_4BrF_5N_2S$	37,6	1,04	24,8	7,31	8,36	20,9	414 (4,09)
(VIIg)	97–98	77	33,5	1,10	21,7	6,28	7,80	29,1	$C_{12}H_4F_5IN_2S$	33,5	0,93	22,1	6,51	7,44	29,5	414 (4,16)
(VIIh)	106–107	74	48,8	2,51	27,3	8,27	9,52		$C_{14}H_7F_5N_2OS$	48,6	2,02	27,5	8,09	9,25		413 (4.03)
(VIIi)	96–97	76	41,6	1,18	27,6	11,9	9,42		$C_{12}H_4F_5N_3O_2S$	41,2	1,15	27,2[illegible]	12,0	9,17		414 (4,07)

Compound (II) is dissolved in an excess of SbF_5 at 40°C with the formation of a colored solution. Examination of the UV and the ^{19}F NMR spectra and measurement of the electric conductivity of this solution indicate the generation of cation (VIII). Thus, a band appears in the UV spectrum at 373 nm [UV spectrum of (II) in heptane: λ_{max}, nm (log ε): 223 (4.19) and 243 (4.12)]. The specific conductance is higher by a factor of 10^3 in comparison with SbF_5 ($\varkappa_{SbF_5} = 1.2 \cdot 10^{-8}$ $cm^{-1} \cdot cm^{-1}$ [8]). The absence of signals of the original compound in the ^{19}F NMR spectrum of a solution of compound (II) in SbF_5 (Table 2) indicates the practically complete conversion of (II) to the cation of salt (VIII). The stability of salt (VIII) is guaranteed by the delocalization of the positive charge on the polyfluorinated aromatic ring and by the stability of the SbF_6^- anion. The signals of F atoms in the o ($\Delta\delta_F = 31.7$ ppm) and especially in the p positions ($\Delta\delta_F = 57.8$ ppm) are more strongly shifted in the weak field to the cation center in comparison with the original compound (II), which is in accordance with data obtained earlier for polyfluorinated arylsulfonyl cations [9]. During the conversion of (II) to its cation, significant changes in the spin–spin coupling constants (SSCC) are also observed (here and throughout SSCC values are obtained from analysis of spectra in approximations of the first order). For compound (II), $J_{F\beta-F^2} = 12.5$, $J_{F^2-F^4} = 2$, and $J_{F^3-F^4} = 20.5$ Hz; for the cation of salt (VIII), $J_{F\beta-F^2} = 25$, $J_{F^3-F^4} = 23$, $J_{F^2-F^4} = 26$, and $J_{F\beta-F^4} = 33$ Hz; the latter constant is not observed in the spectrum of (II). The SSCC $J_{F^2-F^4}$ ($\Delta J = 24$ Hz) and $J_{F\beta-F^2}$ ($\Delta J = 12.5$ Hz) undergo the greatest change, which also conforms to data obtained for the perfluorophenylsulfonyl cation [9]. The shift of the signal of the F atom connected to S in the cation of salt (VIII) in the weak field ($\Delta\delta_{F\beta} = 53.5$ ppm) in comparison with the original compound (II) is noted. Such changes in the ^{19}F NMR spectrum upon the conversion from the precursor to its cation are in agreement with data concerning the distribution of charge, obtained from the quantum-chemical calculation by the CNDO/2 method on the BÉSM-6 electronic computer at the Computer Center of the Siberian Branch of the Academy of Sciences of the USSR (we obtained the geometry of the NSF_2 group from [10], the geometry of the pentafluorophenyl radical from [11], the length of the N–Ar bond from [12], the minimum energy of bonding is obtained for a conformation with the dihedral angle between the plane of the pentafluorophenyl ring and the C^1NS plane equal to 90°, to which also all data presented refer) (Tables 3 and 4).

A splitting of signals of F atoms in the o and m positions (see Table 2) and a change in SSCC are observed in the ^{19}F NMR spectrum of a solution of compound (II) in SbF_5–SO_2 at –80°C. This is connected with the fact that the most stable conformation, determined by the rotation around the Ar–N bond, is realized at a low temperature. A neutralization of the signals of F atoms in the o and m positions is observed with an increase in temperature (see Table 2).

In SO_2, BF_3 reacts analogously on compound (II). However, only signals from F atoms of the aromatic ring are observed in the ^{19}F NMR spectrum of a solution of compound (II) in $SbCl_5$–SO_2 at –20°C and the constant $J_{F\beta-F^4}$ is reduced to zero. The exchange of F atoms, bonded with S, by Cl atoms and the formation of the salt of the sulfonyl cation (IX) under the action of $SbCl_5$ are assumed by us. Actually, a solution of (V) in $SbCl_5$–SO_2 at –20°C gives an identical ^{19}F NMR spectrum. In this case a splitting of the signals of F atoms in the o and m positions is also observed upon a decrease in temperature. Besides this, the action of BF_3 on (V) in SO_2 at –20 to –60°C does not lead to the generation of the cation of salt (IX).

It was noted that iminothiodifluorides form the corresponding N-sulfonyl derivatives upon contact with glass [13]. According to our data, holding compound (II) for 24 h in a glass ampul leads to a mixture of pentafluoroaniline, compound (I), and the fluorohydrate of pentafluoroaniline. The formation of compound (IV) is not observed. In a polytetrafluoroethylene vessel the decomposition of pentafluorophenyliminothiodifluoride occurs only in the presence of traces of HF. One can assume that the role of the acid results in the formation of pentafluoroaniline from compound (II) according to the scheme

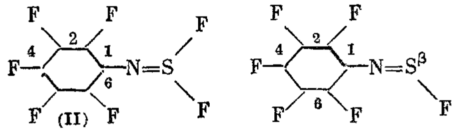
$$C_6F_5NSF_2 \xrightarrow{H^+} [C_6F_5NHSF_2]^+ \xrightarrow{F^-} [C_6H_5NHSF_3] \xrightarrow{H^+} C_6F_5NH_2 + [SF_3]^+$$

In addition, pentafluoroaniline can react with (II), giving (I). Actually, the formation of the salt of pentafluoroaniline is observed upon solution of (II) in an excess of HSO_3F at

TABLE 2. ^{19}F NMR Spectra of Pentafluorophenyliminothiodihalides in Various Media

Compound	System	T., °C	Chemical shift, ppm from C_6F_6 (relative intensity of the signal)			
			F^2, F^6	F^4	F^3, F^5	F at S
(II)	CCl_4	40	−17,0 (2)	−5,0 (1)	−0,5 (2)	−230,5 (2)
	SbF_5	40	−48,7 (2)	−53,7 (1)	−6,5 (2)	−284 (1)
	SbF_5-SO_2	−80	−56,8, −49,3	−60,3	−11,4, −10,0	−284
	BF_3-SO_2	−80	−51,1, −44,8	−53,7	−7,5, −6,5	−284
	HSO_3F-SO_2ClF	−40	−52,4, −47,3	−58,6	−10,8, −9,8	−284
(V)	CCl_4	40	−18,1 (2)	−9,4 (1)	−2,6 (2)	
	$SbCl_5-SO_2$	−20	−54,5 (2)	−61,6 (1)	−11,0 (2)	
	$SbCl_5-SO_2$	−60	−58,2, −51,0	−62,1	−11,9, −10,8	

TABLE 3. Quantum-Chemical Characteristics of Compound (II) and the Cation of Salt (VIII)



Compound	Charges on atoms									
	C^1	C^2, C^6	C^3, C^5	C^4	F^{β}	F^2, F^6	F^3, F^5	F^4	N	S
(II)	0,1015	0,1597	0,1500	0,1514	−0,2352	−0,1648	−0,1519	−0,1586	−0,0823	0,4696
Cation of salt (VIII)	0,0876	0,2007	0,1573	0,1854	−0,0806	−0,1457	−0,1216	−0,1182	−0,1780	0,5664

40°C. Three signals with an intensity ratio of 2:1:2 are observed in the ^{19}F NMR spectrum of this solution, corresponding to −17.7, −15.8, and −5.7 ppm (a solution of pentafluoroaniline in HSO_3F has an analogous ^{19}F NMR spectrum). Upon decantation of this solution into an aqueous solution of $NaHCO_3$, pentafluoroaniline (for ^{19}F NMR spectrum, see [14]) is produced with an almost quantitative yield. The formation of the salt prevents the reaction of pentafluoroaniline with (II). In the case of catalytic amounts of acid such a reaction proceeds and leads to compound (I).

Upon the action of a protic acid on compound (II), a S atom can be affected along with a N atom. The possibility of splitting off HF in this case can lead to the generation of a cation of pentafluorophenyliminothiofluoride of salt $C_6H_5NSF^+SO_3F^-$ (X). A similar situation was observed earlier during the solution of sulfur pentafluorophenyltrifluoride in HSO_3F [15]. In this case the perfluorophenylsulfonyl cation was generated. Actually, during the solution of (II) in HSO_3F at −40°C the cation of salt (X) is generated along with the formation of the salt of pentafluoroaniline (in a ratio of 2:1). Three signals are observed in the ^{19}F NMR spectrum with an intensity ratio of 2:1:2 at −17.7, −15.8, and −5.7 ppm, respectively, which belong to the salt of pentafluoroaniline, and six signals of equal intensity are observed, which belong to the cation of salt (X) (see Table 2).

EXPERIMENTAL

The ^{19}F NMR spectra are obtained on a Varian A-56/60A spectrometer (56.4 MHz) (internal standard — C_6F_6), the UV spectra for pentane solutions are taken on a Specord UV-VIS spectrophotometer, and the IR spectra are taken on a UR-20 instrument.

Pentafluorophenyliminothiodifluoride (II). We placed a mixture of 7.4 g (40 mmole) of pentafluoroaniline and 15 g of NaF, which had been ground to uniformity, into a stainless-steel autoclave of 20-ml capacity and introduced 8.7 g (85 mmole) of SF_4 at −60°C. After 2 h at 0-3°C we opened the autoclave, transferred the solid, bright-yellow mass to a polytetrafluoroethylene flask filled with Ar, and extracted with several portions of 1,1,2-trichloro-1,2,2-trifluoroethane. We concentrated the extract in a polytetrafluoroethylene apparatus and distilled the residue in vacuum. We obtained 3.3 g of (II) (33% based on the amine)

TABLE 4. Energetic Characteristics (CNDO/2 calculation) of Compound (II) and the Cation of Salt (VIII)

Characteristics	(II)	(VIII)
Bonding energy, eV	−140,87	−124,27
First ionization potential, eV	12,27	17,75
Electron affinity, eV	−0,97	−8,97

in the form of a yellow liquid with a bp of 42-43°C (5 mm). IR spectrum (ν, cm^{-1}, in CCl_4): 1530 (polyfluorinated aromatic ring), 1370 (ν NSF_2 [1, 16]), 1290 (ν_{as} SN [3]), 725 (ν_{as} SF [3, 16]). We sublimed (at 50°C and 0.5 mm) the residue from the distillation flask after recrystallization from petroleum ether (40-60°C). We obtained 3.0 g of compound (I) (38%), mp 52.5°C (see [7]), identical, according to IR, UV, and ^{19}F NMR spectra, to the sample obtained according to [7].

Pyrimidyl-2-iminothiodifluoride (III). Analogously from 3.3 g of 2-aminopyridine, 15 g of NaF, and 8.7 g of SF_4, 1.0 g of (III) (15%) was obtained, which was a yellow liquid with a bp of 108-109°C (13 mm). ^{19}F NMR spectrum: a singlet at −221 ppm. IR spectrum (ν, cm^{-1}): 1415 s, 1385 s (ν NSF_2 [1, 16]), 1270 s (ν_{as} SN [3]), 1075 s (ν_s SN [17]), 715 s (ν_{as} SF [3, 16]), 817 s (ν_s SF [16]); λ_{max} 233 nm.

N-Sulfonylpentafluoroaniline (IV). We boiled a mixture of 36.6 g (200 mmole) pentafluoroaniline, 40 ml dry benzene, and 35.7 g (300 mmole) $SOCl_2$ until complete discontinuance of the evolution of HCl. The solvent was removed by aspirator; the residue was distilled in vacuum on a 25-cm Vigreux column. We obtained 43.5 g (95%) of a yellow liquid with a bp of 38-40°C (4 mm). IR spectrum (ν, cm^{-1}): 1260 (ν_{as} NSO) and 1175 (ν_s NSO) [18]. ^{19}F NMR spectrum (δ, ppm, relative intensity): −22.2 (2), −7.2 (1), and 0.0 (2). UV spectrum[λ_{max}, nm (log ε)]: 238 (3.66), 286 (3.53), 345 (3.41). Found: C 31.4; F 41.2; N 6.30; S 14.1%. C_6F_5NOS. Calculated: C 31.4; F 41.4; N 6.12; F 14.0%.

Pentafluorophenyliminothiodichloride (V). a) We heated a mixture of 9.16 g (40 mmole) of (IV), 8.32 g (40 mmole) of finely ground PCl_5, and 10 ml of CCl_4 at 55-60°C with mixing until complete solution of the PCl_5. We removed the $POCl_3$ and CCl_4 by aspirator and distilled the residue in vacuum. We obtained 9.52 g of (V) (84%) with a bp of 78-80°C (4-5 mm).

b) We mixed 4.5 g of (IV), 4 g of PCl_5, 0.5 ml H_2O, and 10 ml CCl_4 at 60°C for 30 min. The solvent was removed by aspirator and the residue was distilled in vacuum at 78-80°C (4-5 mm). We cooled the yellow oil and filtered off the crystals. We obtained 0.5 g of (VI) with a mp of 102-103°C (see [19]), which was identical to the known sample according to IR and ^{19}F NMR spectra. Repeated distillation of the filtrate gave 3 g of (V).

N-Pentafluorophenyl-N'-arylthiodiimides (VIIa-i). A solution of 20 mmole of compound (II) or (V) in 10 ml of absolute ether was added dropwise with stirring to a solution of 20 mmole of the arylamine and 3.16 g (40 mmole) of pyridine in 30 ml of absolute ether at 10°C. The solution was mixed for 2 h at 20°C and filtered. The filtrate was evaporated at reduced pressure and the residue was recrystallized from MeCN. The characteristics of the products are presented in Table 1.

Bis(pentafluorophenyl)thiodiimide (I). A mixture of 1.85 g (10 mmole) of pentafluoroaniline, 7 g of NaF, and 2.51 g (10 mmole) of (II), ground to uniformity, was placed in a polytetrafluoroethylene vessel, which is filled with Ar. The mixture was heated for 3 h at 50°C, then it was extracted by 1,1,2-trichloro-1,2,2-trifluoroethane. After distilling the solvent in vacuum, the residue was sublimed at 50°C (0.5 mm). We obtained 3.74 g of (I) (95%) with a mp of 52.5°C. ^{19}F NMR spectrum (δ, ppm, relative intensity of the signal): −19.8 (2), −5.8 (1), and −0.2 (2). The IR and UV spectra are identical to the spectra of the known sample. UV spectrum [λ_{max}, nm (log ε)]: 250 (3.76) and 390 (3.76). IR spectrum (ν, cm^{-1}): 1320 m (ν_{as} NSN); 1065 s (ν_s NSN [17]); 510 w (ν NSN [17]).

Reaction of Compound (II) with Glass. We kept 3 g of (II) in an ampul of molybdenum glass for 24 h at 20°C. We poured the contents on ice and filtered and dried the residue. We obtained 0.8 g of (I) with a mp of 52.5°C which was identical to the known sample according to IR and ^{19}F NMR spectra. The filtrate was neutralized with $NaHCO_3$ and extracted with ether. The ether extract was dried over $MgSO_4$. After distilling the ether, we obtained 1 g of pentafluoroaniline, which was identified by IR and ^{19}F NMR spectra,

Reaction of Compounds (II) and (V) with SbF_5, BF_3, $SbCl_5$, and HSO_3F. a) We added 1 g of (II) to 5 ml of freshly distilled SbF_5 at 10°C with mixing. We took UV and ^{19}F NMR spectra and measured the specific conductivity according to method of [20] for this solution. We added the solution to SO_2 at −40°C and took ^{19}F NMR spectra at various temperatures.

b) We added 0.15 g of (II) or (V) to 1 ml of a mixture of HSO_3F-SO_2ClF (1:2) (SbF_5-SO_2, $SbCl_5-SO_2$, BF_3-SO_2) at −60°C. After 0.5 h of mixing we conducted a survey of the ^{19}F NMR spectra at various temperatures.

c) We added 1 g of (II) to 5 ml of HSO_3F at 20°C and conducted a survey of the ^{19}F NMR spectrum at 40°C. Then we poured the solution on ice, neutralized with $NaHCO_3$, cooled to 5°C, and filtered. We obtained 0.64 g of pentafluoroaniline (90%), which was identified according to IR and ^{19}F NMR spectra.

CONCLUSIONS

1. Pentafluorophenyliminothiodifluoride is formed upon the action of SF_4 on pentafluoroaniline in the presence of NaF. A new group of unsymmetrical thiodiimides, containing the pentafluorophenyl ring, is synthesized.

2. The pentafluorophenyliminothiofluoride cation is generated upon the reaction of Lewis acids with pentafluorophenyliminothiodifluorides.

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