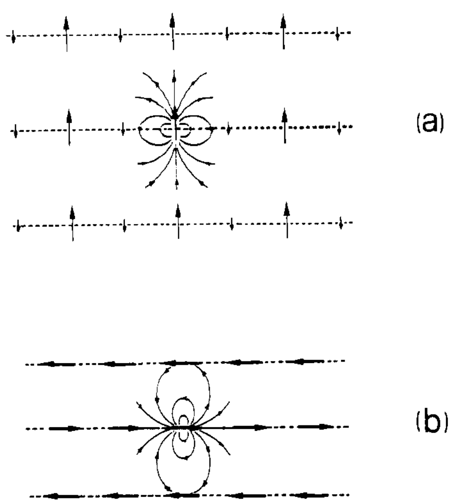

Figure 8. Schematic view of the dipolar field and preferred spin orientation in ideal 1-D ferrimagnets (a) and 1-D ferromagnets (b).

preferred spin orientation is orthogonal to the chain direction.[31,47] The dipolar field generated by a magnetic moment interacts with the moments of the neighboring parallel chains and orients the spins parallel to each other, as depicted in Figure 8a. On the other hand, if the coupling is ferromagnetic, the preferred spin orientation is parallel to the chain direction. In this case the dipolar interaction between parallel chains tends to keep the magnetic moments antiparallel to each other, as shown in Figure 8b.

Both $Cu(hfac)_2NITR$ compounds have shown a significant positive *g* shift along the chain, indicating this as the favorite spin orientation.[33] In the NITMe derivative II the chains are related by the unit translations and are parallel to each other and we observe a 3-D antiferromagnetic interaction, as predicted by our simple model. In the case of the compound I each chain is surrounded by four orthogonal chains, so complicating the pattern of interchain dipolar interactions. Our simple model, based on the symmetry of translations, cannot apply in this case, and detailed calculations would be needed. On the other hand, these cannot be performed unless more information on the ordered phase is available.

Due to the long distances between chains the interchain dipolar energy is fairly small in all these products. However, in the case of the manganese and nickel derivatives the magnetic correlation along the chain originates at low temperature large effective moments that enhance the effects of the weak dipolar interaction. The observed transition temperatures are in fact 8 and 5 K for the manganese[31,47] and nickel[30] compounds, respectively, while the copper compounds, which have much smaller magnetic correlation within the chains, remain paramagnetic down to 1 K.

As a conclusion, even if it is possible to obtain both antiferro- and ferromagnetic 3-D interactions by starting from ferromagnetic chains, the ferrimagnetic chains seem to be the most suitable building blocks in order to obtain bulk magnetic materials with desired properties.

Acknowledgment. The financial support of the CNR and of the MURST is gratefully acknowledged as well as that from the Solid State Chemistry Program, Division of Material Research, National Science Foundation (Grant DMR-8815798). C.I.C. acknowledges the support of the Comision De Investigaciones Cientificas PBA (Argentina).

Supplementary Material Available: Tables SI–SV, listing experimental and crystallographic parameters, anisotropic thermal factors, positional parameters of hydrogen atoms, and bond distances and angles, respectively (7 pages); a table of observed and calculated structure factors (11 pages). Ordering information is given on any current masthead page.

Contribution from Anorganische Chemie, Universität Dortmund, 4600 Dortmund, FRG,
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Synthesis of CF_3SSI, Vibrational Analyses for CF_3SSX (X = F, Cl, Br, I, and H), and Gas-Phase Structures of CF_3SSF and CF_3SSH[1]

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CF_3SSI has been synthesized by reacting CF_3SSH with *N*-iodosuccinimide at −120 °C. The compound decomposes at −100 °C. It was characterized by its Raman spectrum recorded at −196 °C and by ^{19}F NMR and UV/vis spectroscopy. For comparison Raman spectra of solid and IR spectra of gaseous or matrix-isolated samples were also recorded for the homologues CF_3SSX with X = F, Cl, Br, and H. Valence force fields for these (trifluoromethyl)disulfanes were derived from the spectral data. The S–S force constant for X = Cl, Br, and I (2.50×10^2 N m^{-1}) is equal to that in S_8; it is larger for X = F (2.59×10^2 N m^{-1}) and smaller for X = H (2.32×10^2 N m^{-1}). The S–X force constants decrease steadily with increasing size of the halogen from 3.49 $\times 10^2$ N m^{-1} for S–F to 1.74×10^2 N m^{-1} for S–I. Furthermore, the gas-phase structures of CF_3SSF and CF_3SSH were determined by electron diffraction. The S–S bond lengthens from 197.0 (3) pm in CF_3SSF to 203.8 (5) pm in CF_3SSH (r_g values; error limits are 3σ values and include a possible scale error of 0.1%). The CSSF dihedral angle is 91 (3)°.

Introduction

Until now, only a few iodosulfanes RSI with R = aryl or alkyl have been synthesized and fully characterized,[3-7] even though the S–I moiety is of considerable biochemical interest.[8] Despite the thermal instability, we were recently successful in determining the crystal structure of $(C_6H_5)_3CSI$.[9] The experimental S–I distance in this compound (240.6 (4) pm) is close to the sum of the covalent radii (237 pm). It is longer than the distances in the S_7I^+ cation and its derivatives (S–I between 231.8 (8) and 234.2 (3) pm[10,11]), where iodine is bonded to three-coordinate sulfur.

An alternative synthetic route to iodosulfanes such as CF_3SI[3] and $CF_3C(O)SI$[6] is iodination of thiols with *N*-iodosuccinimide. In continuation of these experiments, we report here on CF_3SSI. Because of its low stability, it is not possible to determine a

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Table I. Vibrational Frequencies (cm^{-1}) of the (Trifluoromethyl)disulfanes CF_3SSX with X = F, Cl, Br, I, and H

CF_3SSF		CF_3SSCl		CF_3SSBr		CF_3SSI	CF_3SSH	
IR (matrix)	Raman (solid)	IR (matrix)	Raman (solid)	IR (matrix)	Raman (solid)	Raman (solid)	Raman (solid)	assgnt
n.o.	1191 (4)	1180 vs	1175 (1)	1180 vs	n.o.	1180^a	1180 (2)	$\nu_{as}(CF_3)$
1168 vs	1170 (6)	1172 vs	1160 (1)	1172 vs	n.o.	1174^a	1170 (2)	$\nu_{as}(CF_3)$
1109 vs	n.o.	1114 vs	1100 (2)	1110 vs	n.o.	1109^a	1115 (3)	$\nu_s(CF_3)$
750 m	764 (52)	755 s	755 (35)	755 s	755 (5)	755 (3)	756 (92)	$\delta_s(CF_3)$
700 m	700 (100)	482 s	474 (100)	392 s	385 (100)	310 (100)	2540 (50)	$\nu(SX)$
566 w	576 (40)	563 w	563 (11)	563 m	561 (2)	560 (2)	575 (21)	$\delta_{as}(CF_3)$
n.o.	554 (51)	522 m	528 (41)	526 m	526 (4)	522 (8)	528 (100)	$\nu(SS)$
540 w	n.o.	540 w	n.o.	543 vw	n.o.	543^a	n.o.	$\delta_{as}(CF_3)$
450 m	452 (61)	450 m	450 (45)	450 m	450 (10)	456 (10)	458 (69)	$\nu(CS)$
349 vw	355 (24)	339 w	343 (10)	336 vw	337 (4)	337 (36)	n.o.	$\rho(CF_3)$
	332 (37)	322 w	321 (26)	320 vw	329 (11)	325 (43)	322 (80)	$\rho(CF_3)$
	261 (59)		200 (89)		155 (45)	140 (45)	895 (26)	$\delta(SSX)$
	194 (29)		167 (20)		170 (18)	165 (5)	163 (58)	$\delta(SSC)$
	127 (31)		92^b (17)		57 (4)	n.o.	n.o.	$\tau(SS)$
	87 (25)		92^b (17)		76 (18)	72 (2)	n.o.	$\tau(CF_3)$

^{a}Calculated from force field (see text). ^{b}Coincidence of $\tau(SS)$ and $\tau(CF_3)$.

gas-phase structure of this compound by either gas-phase electron diffraction (GED) or microwave spectroscopy. Therefore, vibrational spectra were recorded to characterize its physical properties. To secure a systematic characterization, the vibrational spectra of the homologues CF_3SSX with X = F, Cl, Br, and H were analyzed for comparison. In addition, the gas-phase structures of the two compounds in this series with X at the extremes of the electronegativity scale, CF_3SSF and CF_3SSH, were determined by GED. The structures of the CF_3SS moiety in the compounds with X = Cl, Br, and I can be estimated by interpolation.

Experimental Section

Preparation of CF_3SSI. A 3-mmol sample of CF_3SSH was condensed onto a suspension of 3.3 mmol of *N*-iodosuccinimide (NIS) in 15 mL of CF_2Cl_2, and the reaction mixture was stirred for 10 h at −120 °C. After filtration of the claret-colored solution and vacuum evaporation of the solvent at −120 °C, CF_3SSI remained as a red solid. (^{19}F NMR: −49.02 ppm; internal standard $CFCl_3$; $T = -30$ °C. UV/vis: $\lambda_{max} = 290$, 380 nm (sh); solvent *n*-hexane.) CF_3SSH[12] and NIS[13] were prepared according to literature methods and purified by vacuum fractionation and crystallization, respectively. The purity was checked by NMR and Raman spectroscopy.

The iododisulfane CF_3SSI is less stable than the iodomonosulfane CF_3SI[4] and cannot be sublimed under vacuum. Particularly under the influence of light, CF_3SSI decomposes above −100 °C to $(CF_3SS)_2$ and I_2. $(CF_3)_2S_3$ is also observed. Thus, preparation and handling of this compound should be carried out in the dark. CF_3SSI is more stable in 10^{-4} *m* solution of *n*-hexane than in the solid state. In solution it can still be detected after remaining at −30 °C for several hours.

Preparation of CF_3SSCl, CF_3SSBr, and CF_3SSF. The chlorine derivative was prepared by low-temperature (−78 °C) chlorination of CF_3SSH.[14] CF_3SSBr is formed in analogy with CF_3SBr[15] by halide exchange according to

$$CF_3SSCl + HBr \rightarrow CF_3SSBr + HCl$$

and CF_3SSF is prepared by flowing gaseous CF_3SSCl through activated KF at +120 °C. The halogeno(trifluoromethyl)disulfanes were purified by fractional condensation over a series of U-traps.

Vibrational Spectra. The Raman spectra of all pure compounds were recorded at −196 °C with a Coderg T 800 spectrometer. For excitation of CF_3SSF, CF_3SSCl, and CF_3SSH, an Ar^+ ion laser (514.5 nm and 400 mW) was used. Because of their light sensitivity, CF_3SSBr and CF_3SSI were excited by a Kr^+ ion laser (647.1 nm and 300 mW). All Raman spectra were recorded in the range 50–4000 cm^{-1} with a maximum resolution of 1.5 cm^{-1}. Furthermore, N_2 matrix IR spectra at −258 °C were measured for all compounds except the iodine compound (Perkin-Elmer 580B, 270–4000 cm^{-1} and maximum resolution 2.3 cm^{-1}) and the gas-phase IR spectrum of CF_3SSH was recorded with a Bruker IFS 113V spectrometer.

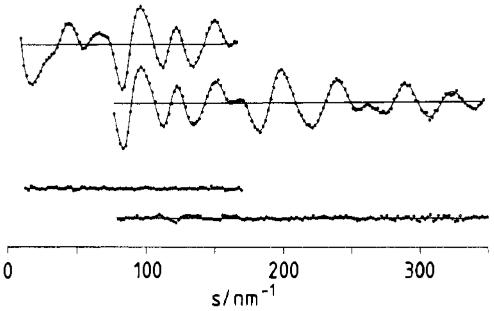

Figure 1. Experimental (dots) and calculated (full line) electron diffraction intensities and differences for CF_3SSF.

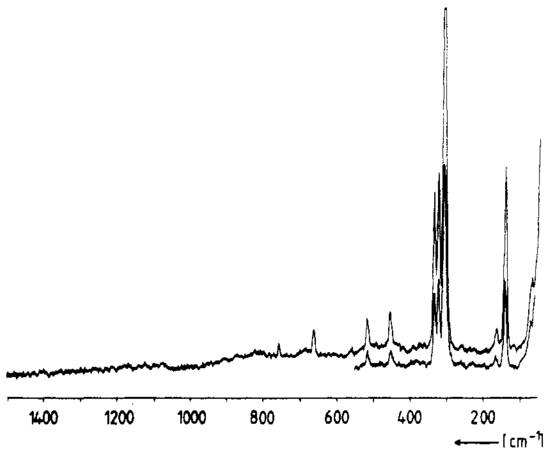

Figure 2. Raman spectrum of solid CF_3SSI.

Electron Diffraction. The scattering intensities were recorded with a KD-G2 gas diffractograph[16] at two camera distances (25 and 50 cm) and with an accelerating voltage of ca. 60 kV. The electron wavelength was determined from ZnO diffraction patterns in each experiment, which in turn were calibrated by CO_2 gas diffraction in the experiment for CF_3SSH. The samples were kept at −52 °C (CF_3SSF) and −46 °C (CF_3SSH), respectively. The camera pressure did not exceed 2.10^{-5} Torr during the experiments. Two plates for each compound and camera distance were analyzed by the usual procedures.[17] The averaged molecular intensities for CF_3SSF are shown in Figure 1, and numerical values for the intensities of both compounds are available as supplementary data.

Results and Discussion

Vibrational Spectra. The Raman spectrum of solid CF_3SSI is presented in Figure 2. The vibrational frequencies of the entire

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Table II. Diagonal Force Constants (10^2 N m^{-1}) of the (Trifluoromethyl)disulfanes CF_3SSX with X = F, Cl, Br, I, and H[a]

	CF_3SSF	CF_3SSCl	CF_3SSBr	CF_3SSI	CF_3SSH
S–S	2.59	2.50	2.50	2.50	2.32
S–X	3.49	2.66	2.25	1.74	3.66
S–C	3.59	3.61	3.61	3.61	3.61
C–F	5.85	5.88	5.89	5.89	5.83
SSX	1.22	0.92	0.77	0.70	0.96
SSC	1.26	1.13	1.11	1.11	1.19
SCF	0.98	0.95	1.01	1.05	0.87
FCF	2.22	2.13	2.20	2.10	2.24
τ(S–S)	0.55	0.26	0.22		
τ(S–C)	0.25	0.26	0.26	0.26	

[a] Values for off-diagonal force constants can be obtained upon request from R.M.

series are summarized in Table I with their assignment on the basis of C_1 symmetry. Polarization measurements on liquid CF_3SSCl demonstrated that all observed bands are polarized.

To simplify the assignment, the nine CF_3 vibrations (three stretchings, three deformations, two rockings, and one torsion) are separated from the remaining vibrations (C–S, S–S, and S–X stretchings, C–S–S and S–S–X deformations, and S–S torsion). The frequencies of the CF_3 group in CF_3SSX with X = Cl, Br, I, and H are nearly independent of the substituent, and only slight deviations occur for CF_3SSF. Considerable differences, however, are observed for the band intensities of the CF_3 rocking vibrations within this series. Because of their weak Raman intensities, $\nu_s(CF_3)$ and $\nu_{as}(CF_3)$ are not observed for CF_3SSI.

The S–X stretchings (X = F, Cl, Br, I) are assigned to the intensive Raman signals at ν(SF) = 700 cm^{-1}, ν(SCl) = 474 cm^{-1}, ν(SBr) = 385 cm^{-1}, and ν(SI) = 310 cm^{-1}. All these vibrations occur at higher wavenumbers relative to the mean frequencies in the corresponding dihalogenodisulfanes S_2X_2: $\bar{\nu}$(SF) = 693 cm^{-1},[18] $\bar{\nu}$(SCl) = 442 cm^{-1},[19] $\bar{\nu}$(SBr) = 357 cm^{-1},[20] and $\bar{\nu}$(SI) = 300 cm^{-1}.[3] The S–S vibrations in this series occur around 526 cm^{-1}, except for that of CF_3SSF, which is shifted to higher frequencies (554 cm^{-1}).

Our Raman spectrum of CF_3SSH is in total agreement with that of Gombler and Seel,[12] except for the S–H stretch. This vibration has a high intensity in our spectrum, at variance with ref 12, where it was not observed. The intensities of the CF stretchings do not allow for a unique identification, and therefore the gas IR frequencies were transferred.

Normal-Coordinate Analyses. Valence force fields of the (trifluoromethyl)disulfanes CF_3SSX and X = F, Cl, Br, I, and H were derived from the vibrational frequencies by using the programs NORKO[21] and NORKOR.[22] Thereby, the experimental structures for CF_3SSF and CF_3SSH (see below) were used and those for the chloro, bromo, and iodo compounds were estimated by interpolation. The sulfur-halogen distances were set to S–Cl = 206 pm, S–Br = 224 pm, and S–I = 240 pm, on the basis of data for S_2Cl_2,[23] S_2Br_2,[24] and $(C_6H_5)_3CSI$.[9] The force constants in Table II reproduce the experimental frequencies with mean deviations better than 5 cm^{-1}. Values for the CF_3 group and for the S–C stretch are nearly equal for the entire series. The value for the C–F force constant in CF_3SSI has been transferred from the bromine compound. The S–S constants for the chloro-, bromo-, and iododisulfanes are equal to the value in S_8.[25] This constant is slightly larger for X = F and smaller for X = H. The S–X stretch and SSX deformation constants decrease steadily with increasing size of the halogen. To our knowledge, no S–I force constant for any other compound is known for comparison. According to Siebert,[26] the constants for X = Cl, Br, and I correspond to S–X bond orders of 1.0, whereas the S–F constant indicates a reduced bond order of 0.8. This observation is in accordance with the bond length (161.1 (3) pm) which is longer than that in SF_2 (159.2 pm).[27] Variations in the S–X force constants (X = F, Cl, Br) of XSSX and CF_3SSX compounds (Table III) indicate that introduction of one CF_3 group strengthens

Table III. S–X Force Constants (10^2 N m^{-1}) in CF_3SSX and XSSX for X = F, Cl, Br, I, and H

X	CF_3SSX	XSSX
F	3.49	3.41,[a] 3.21,[b] 2.90[c]
Cl	2.66	2.02,[d] 1.985,[e] 1.98[f]
Br	2.25	1.75,[d] 1.542[c]
I	1.74	
H	3.66	4.08[g]

[a] Reference 18. [b] Reference 28. [c] Reference 29. [d] Reference 30. [e] Reference 31. [f] Reference 32. [g] Reference 33.

Table IV. Vibrational Amplitudes for CF_3SSF and CF_3SSH from Electron Diffraction and Spectroscopic Data

		CF_3SSF		CF_3SSH	
		ED	spectr	ED	spectr
C–F	l_1	4.5 (2)	4.6	4.6 (4)	4.6
S–X	l_2	3.8 (4)	4.7		
S–C	l_3	6.2 (10)	5.0	6.1 (7)	6.0
S–S	l_4	4.9 (4)	4.9	6.1 (5)	6.1
$F_1 \cdots F_2$	l_5	5.9 (3)	5.6	7.3 (6)	7.5
$S_1 \cdots F_i$	l_6	6.0 (7)	6.6	7.0 8)	6.7
$S_1 \cdots X$	l_7	8.8 (12)	7.7		
$S_2 \cdots C$			8.9	9.7 (16)	9.0
$S_2 \cdots F_2$	l_8	12 (3)	13.6		14.2
$S_2 \cdots F_3$			13.8	15 (5)	13.8
$S_2 \cdots F_1$	l_9	10 (2)	7.9	10 (2)	9.8
$C \cdots X$		13[b]	13.3		
$F_2 \cdots X$	l_{10}	16 (4)	20.0		
$F_3 \cdots X$			18.2		
$F_1 \cdots X$		18[b]	18.2		

[a] Values in pm. Error limits refer to the last digit and are 3σ values. For atom numbering see Figures 3 and 4. For CF_3SSH all distances to hydrogen are omitted. [b] Not refined.

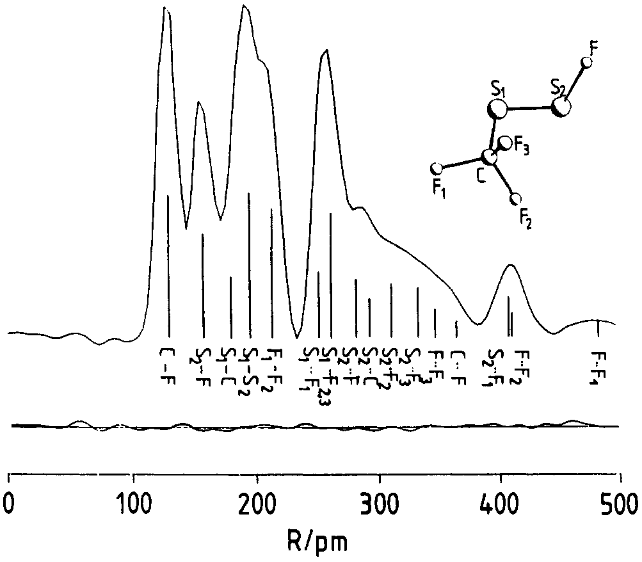

Figure 3. Experimental radial distribution curve for CF_3SSF.

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Table V. Geometric Parameters for CF_3SSF and CF_3SSH and Related Disulfanes[a]

		CF_3SSF[f]	CF_3SSH[f]	CF_3SSCF_3[g]	FSSF[h]	HSSH[i]
S–S	p_1	197.0 (3)	203.8 (5)	203.0 (5)	189.0 (2)	206.10 (3)
S–X	p_2	161.1 (3)	134[e]		163.5 (2)	134.21 (5)
S–C	p_3	182.9 (6)	180.6 (6)	183.5 (5)		
C–F	p_4	133.4 (2)	133.7 (2)	133.3 (2)		
∠SSX	p_5	105.7 (8)	98[e]		108.3 (2)	97.51 (5)
∠SSC	p_6	102.0 (6)	101.2 (6)	101.6 (6)		
∠FCF	p_7	108.0 (3)	107.7 (4)	108.3 (3)		
τ(S–S)[b]	p_8	91 (3)	91[e]	104 (4)	87.7 (4)	90.76 (6)
$\tau(CF_3)$[c]	p_9	11 (2)	9 (5)	1 (4)		
tilt[d]	p_{10}	4.6 (6)	4.6 (8)	5.0 (5)		

[a] Values in pm and deg; error limits refer to the last digit. [b] Dihedral angle. [c] Torsion of the CF_3 group around the S–C bond. For $\tau = 0°$, the CF_3 group staggers exactly the S–S bond and a positive value implies increasing $X\cdots F_3$ and decreasing $X\cdots F_2$ nonbonded distances. [d] Tilt angle of the CF_3 group away from the S–S bond. [e] Not refined. [f] r_g values from this study. Error limits are 3σ values and include a possible scale error of 0.1% for bond lengths. [g] r_g values from ref 35. [h] r_z values from ref 36. [i] r_z values from ref 34.

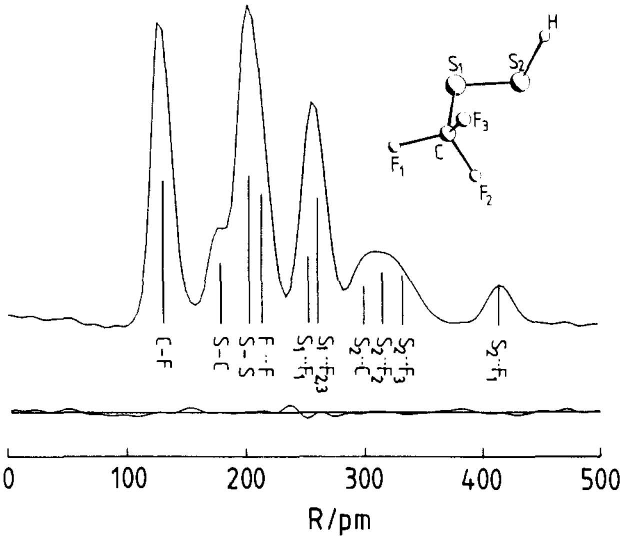

Figure 4. Experimental radial distribution curve for CF_3SSH.

the S–X bonds. The opposite trend is observed for the S–H constants.

Gas-Phase Structures of CF_3SSF and CF_3SSH. The radial distribution functions for both compounds are presented in Figures 3 and 4. Assuming local C_{3v} symmetry for the CF_3 groups with a possible tilt angle between the C_3 axis and the S–C bond direction, ten geometric parameters are required to describe the geometry of CF_3SSF. Only seven parameters were refined for CF_3SSH, and the three parameters locating the hydrogen atom, i.e., S–H, SSH, and the dihedral angle τ(S–S), were set to the corresponding values in H_2S_2.[34] Vibrational amplitudes were grouped according to their distances, and constraints are evident from Table IV. Least-squares analyses are based on the molecular intensities, and a diagonal weight matrix was applied to the intensities. With the above assumptions the following correlation coefficients had values larger than $|0.6|$. CF_3SSF: $p_3/p_4 = -0.67$, $p_3/p_7 = -0.82$, $p_4/p_7 = 0.69$, $p_9/l_8 = 0.80$, $p_{10}/l_6 = -0.88$. CF_3SSH: $p_3/p_7 = -0.82$, $p_1/l_5 = 0.75$, $p_7/l_4 = 0.65$, $p_9/l_8 = 0.88$, $p_{10}/l_6 = -0.90$, $l_4/l_5 = 0.71$.

The vibrational amplitudes derived from the electron diffraction intensities (Table IV) agree well with those calculated from the force field (Table II). Structural parameters in Table V are compared to those of related disulfanes. The S–S bond, which is extremely short in FSSF, lengthens with successive substitution of fluorines by CF_3 groups or by hydrogens. On the other hand, the S–F bond shortens in going from FSSF to CF_3SSF, and as was pointed out above, the force constants indicate similar trends for the S–X distances (X = Cl, Br, I) between XSSX and CF_3SSX. The –S–S– dihedral angles are close to 90° in all disulfanes, except for CF_3SSCF_3, where this angle is increased to about 100°.

Supplementary Material Available: Listings of numerical values for total scattering intensities of CF_3SSF and CF_3SSH (4 pages). Ordering information is given on any current masthead page.

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