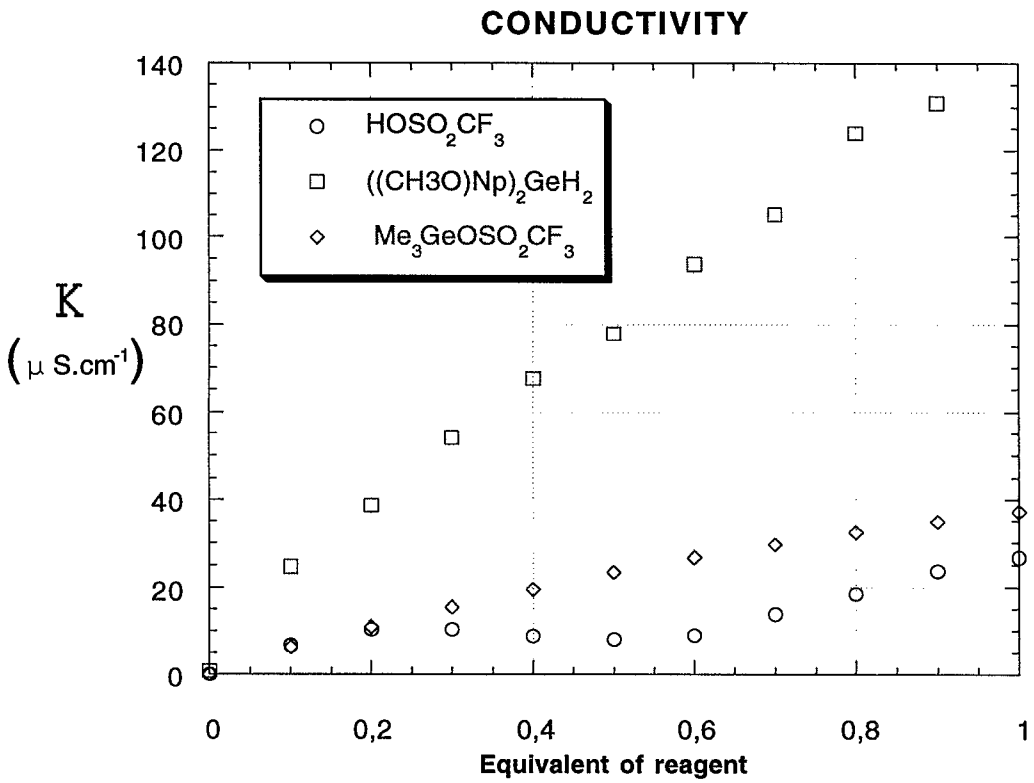
ORGANOMETALLICS
Organometallics, 1998, 17(11), 2222-2227, DOI:10.1021/om970963f



The conductivity (K) as a function of the amount of electrophilic reagent added to a solution of 0.146 mol.l^{-1} of germane **3** in CH_2Cl_2.

Table 1. Crystal data ,data collection and structure refinement for **1**

Empirical formula	$C_{23}H_{23}F_3GeO_5S$
Formula weight	541.06
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	a = 10.209(2) Å α = 90° b = 17.049(3) Å β = 95.15(3)° c = 12.918(3) Å γ = 90°
Volume	2239.3(8) $Å^3$
Z	4
Density (calculated)	1.605 Mg/m^3
Absorption coefficient	1.520 mm^{-1}
F(000)	1104
Crystal size	0.2 x 0.12 x 0.08 mm
Diffractometer used	Stoe IPDS
Temperature	293(2) K
Wavelength	0.71073 Å
Monochromator	graphite
Scan type	N/A
Control reflections	50 to 200 (I>6sigma(I)) on each image
θ range for data collection	1.98 to 24.13°
Index ranges	$-11 \leq h \leq 11$, $-19 \leq k \leq 19$, $-14 \leq \ell \leq 14$
Reflections collected	11832
Independent reflections	3348 (R_{int} = 0.0944)
Observed refection	2378 (I>2sigma(I))

Programs used	SHELXS-97 (Sheldrick, 1990), SHELXL-97 (Sheldrick, 1997)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3348 / 0 / 302
Goodness-of-fit on F^2	0.906
Final R indices [$I>2\sigma(I)$]	R1 = 0.0394, wR2 = 0.0881
R indices (all data)	R1 = 0.0611, wR2 = 0.0951
Hydrogen atoms	geom
Largest diff. peak and hole	0.534 and -0.366 $e\text{Å}^{-3}$

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 1 . U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor

	x	y	z	U(eq)
Ge	1263(1)	2109(1)	5373(1)	45(1)
S	4331(1)	1853(1)	5899(1)	49(1)
C(1)	554(4)	1209(2)	6062(3)	45(1)
C(2)	1238(5)	812(2)	6869(3)	61(1)
C(3)	695(6)	142(3)	7299(4)	68(1)
C(4)	-492(5)	-126(2)	6928(4)	62(1)
C(5)	-1240(4)	243(2)	6110(3)	51(1)
C(6)	-2475(5)	-28(2)	5687(4)	61(1)
C(7)	-3140(5)	352(3)	4888(4)	70(1)
C(8)	-2643(5)	1030(3)	4455(4)	59(1)
C(9)	-1462(4)	1303(2)	4846(3)	47(1)
C(10)	-697(4)	926(2)	5674(3)	44(1)
C(11)	738(4)	3100(2)	5941(3)	44(1)
C(12)	-78(5)	3028(2)	6721(3)	58(1)
C(13)	-456(5)	3673(3)	7294(3)	66(1)
C(14)	-3(5)	4399(3)	7087(3)	60(1)
C(15)	829(5)	4519(2)	6289(3)	52(1)
C(16)	1265(5)	5280(3)	6053(4)	66(1)
C(17)	2044(5)	5386(3)	5285(4)	72(1)
C(18)	2424(5)	4756(2)	4679(4)	65(1)
C(19)	1997(4)	4018(2)	4879(3)	52(1)
C(20)	1198(4)	3863(2)	5695(3)	45(1)
C(21)	-1553(6)	2438(3)	3720(4)	78(2)
C(22)	2929(6)	3463(3)	3395(4)	87(2)
C(23)	5347(6)	2729(3)	6122(4)	73(1)
O(1)	-850(3)	1960(2)	4499(2)	61(1)
O(2)	2271(3)	3371(2)	4309(2)	65(1)
O(3)	3028(3)	2139(2)	6158(2)	53(1)
O(4)	4360(3)	1671(2)	4840(2)	74(1)
O(5)	4894(4)	1324(2)	6660(3)	78(1)
F(1)	6534(3)	2593(2)	5938(3)	118(1)
F(2)	5399(5)	2956(2)	7084(3)	146(2)
F(3)	4905(5)	3306(2)	5585(5)	197(3)

Table 3. Bond lengths [Å] and angles [°] for 1 .

Ge-C(11)	1.938(4)	Ge-C(1)	1.945(4)
Ge-O(3)	1.988(3)	Ge-O(1)	2.357(3)
Ge-O(2)	2.799(3)	Ge-H(1)	1.33(3)
S-O(4)	1.406(3)	S-O(5)	1.417(3)
S-O(3)	1.483(3)	S-C(23)	1.827(5)
C(1)-C(2)	1.379(6)	C(1)-C(10)	1.414(5)
C(2)-C(3)	1.405(6)	C(2)-H(2)	0.9300
C(3)-C(4)	1.342(7)	C(3)-H(3)	0.9300
C(4)-C(5)	1.398(6)	C(4)-H(4)	0.9300
C(5)-C(6)	1.406(6)	C(5)-C(10)	1.427(5)
C(6)-C(7)	1.349(7)	C(6)-H(6)	0.9300
C(7)-C(8)	1.400(6)	C(7)-H(7)	0.9300
C(8)-C(9)	1.348(6)	C(8)-H(8)	0.9300
C(9)-O(1)	1.376(4)	C(9)-C(10)	1.420(5)
C(11)-C(12)	1.369(5)	C(11)-C(20)	1.428(5)
C(12)-C(13)	1.400(6)	C(12)-H(12)	0.9300
C(13)-C(14)	1.356(6)	C(13)-H(13)	0.9300
C(14)-C(15)	1.408(6)	C(14)-H(14)	0.9300
C(15)-C(16)	1.413(6)	C(15)-C(20)	1.426(5)
C(16)-C(17)	1.339(7)	C(16)-H(16)	0.9300
C(17)-C(18)	1.403(7)	C(17)-H(17)	0.9300
C(18)-C(19)	1.364(6)	C(18)-H(18)	0.9300
C(19)-O(2)	1.369(5)	C(19)-C(20)	1.415(5)
C(21)-O(1)	1.436(5)	C(21)-H(21A)	0.9600
C(21)-H(21B)	0.9600	C(21)-H(21C)	0.9600
C(22)-O(2)	1.418(5)	C(22)-H(22A)	0.9600
C(22)-H(22B)	0.9600	C(22)-H(22C)	0.9600
C(23)-F(3)	1.263(6)	C(23)-F(1)	1.277(6)
C(23)-F(2)	1.298(6)	O(2)-H(1)	2.46(3)
O(4)-H(1)	2.78(3)		

C(11)-Ge-C(1)	112.81(15)	C(11)-Ge-O(3)	93.04(14)
C(1)-Ge-O(3)	98.29(14)	C(11)-Ge-O(1)	90.19(14)
C(1)-Ge-O(1)	76.84(14)	O(3)-Ge-O(1)	174.93(10)
C(11)-Ge-O(2)	68.99(12)	C(1)-Ge-O(2)	177.79(12)
O(3)-Ge-O(2)	82.79(10)	O(1)-Ge-O(2)	102.03(9)
C(11)-Ge-H(1)	127.8(12)	C(1)-Ge-H(1)	116.4(12)
O(3)-Ge-H(1)	96.1(13)	O(1)-Ge-H(1)	84.9(13)
O(2)-Ge-H(1)	61.5(12)	O(4)-S-O(5)	119.5(2)
O(4)-S-O(3)	112.9(2)	O(5)-S-O(3)	111.64(19)
O(4)-S-C(23)	105.8(2)	O(5)-S-C(23)	102.9(3)
O(3)-S-C(23)	101.7(2)	C(2)-C(1)-C(10)	118.7(4)
C(2)-C(1)-Ge	123.3(3)	C(10)-C(1)-Ge	117.9(3)
C(1)-C(2)-C(3)	120.4(5)	C(1)-C(2)-H(2)	119.8
C(3)-C(2)-H(2)	119.8	C(4)-C(3)-C(2)	120.8(4)
C(4)-C(3)-H(3)	119.6	C(2)-C(3)-H(3)	119.6
C(3)-C(4)-C(5)	122.1(4)	C(3)-C(4)-H(4)	119.0
C(5)-C(4)-H(4)	119.0	C(4)-C(5)-C(6)	123.7(4)
C(4)-C(5)-C(10)	117.4(4)	C(6)-C(5)-C(10)	118.8(4)
C(7)-C(6)-C(5)	120.7(4)	C(7)-C(6)-H(6)	119.6
C(5)-C(6)-H(6)	119.6	C(6)-C(7)-C(8)	121.8(5)
C(6)-C(7)-H(7)	119.1	C(8)-C(7)-H(7)	119.1
C(9)-C(8)-C(7)	118.7(4)	C(9)-C(8)-H(8)	120.7
C(7)-C(8)-H(8)	120.7	C(8)-C(9)-O(1)	125.1(4)

C(8)-C(9)-C(10)	122.6(4)	O(1)-C(9)-C(10)	112.3(3)
C(1)-C(10)-C(9)	122.0(3)	C(1)-C(10)-C(5)	120.6(4)
C(9)-C(10)-C(5)	117.4(4)	C(12)-C(11)-C(20)	118.7(3)
C(12)-C(11)-Ge	114.0(3)	C(20)-C(11)-Ge	127.1(3)
C(11)-C(12)-C(13)	122.2(4)	C(11)-C(12)-H(12)	118.9
C(13)-C(12)-H(12)	118.9	C(14)-C(13)-C(12)	119.9(4)
C(14)-C(13)-H(13)	120.1	C(12)-C(13)-H(13)	120.1
C(13)-C(14)-C(15)	121.1(4)	C(13)-C(14)-H(14)	119.5
C(15)-C(14)-H(14)	119.5	C(14)-C(15)-C(16)	120.9(4)
C(14)-C(15)-C(20)	119.0(4)	C(16)-C(15)-C(20)	120.0(4)
C(17)-C(16)-C(15)	120.2(4)	C(17)-C(16)-H(16)	119.9
C(15)-C(16)-H(16)	119.9	C(16)-C(17)-C(18)	121.5(4)
C(16)-C(17)-H(17)	119.2	C(18)-C(17)-H(17)	119.2
C(19)-C(18)-C(17)	119.4(4)	C(19)-C(18)-H(18)	120.3
C(17)-C(18)-H(18)	120.3	C(18)-C(19)-O(2)	123.9(4)
C(18)-C(19)-C(20)	121.9(4)	O(2)-C(19)-C(20)	114.2(3)
C(19)-C(20)-C(15)	116.9(3)	C(19)-C(20)-C(11)	124.0(3)
C(15)-C(20)-C(11)	119.1(3)	O(1)-C(21)-H(21A)	109.5
O(1)-C(21)-H(21B)	109.5	H(21A)-C(21)-H(21B)	109.5
O(1)-C(21)-H(21C)	109.5	H(21A)-C(21)-H(21C)	109.5
H(21B)-C(21)-H(21C)	109.5	O(2)-C(22)-H(22A)	109.5
O(2)-C(22)-H(22B)	109.5	H(22A)-C(22)-H(22B)	109.5
O(2)-C(22)-H(22C)	109.5	H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5	F(3)-C(23)-F(1)	109.7(5)
F(3)-C(23)-F(2)	106.0(5)	F(1)-C(23)-F(2)	105.9(5)
F(3)-C(23)-S	112.4(4)	F(1)-C(23)-S	110.9(4)
F(2)-C(23)-S	111.6(4)	C(9)-O(1)-C(21)	118.5(4)
C(9)-O(1)-Ge	110.7(2)	C(21)-O(1)-Ge	130.7(3)
C(19)-O(2)-C(22)	119.6(3)	C(19)-O(2)-H(1)	132.7(7)
C(22)-O(2)-H(1)	107.5(7)	C(19)-O(2)-Ge	104.7(2)
C(22)-O(2)-Ge	135.7(3)	H(1)-O(2)-Ge	28.4(6)
S-O(3)-Ge	131.84(16)	S-O(4)-H(1)	91.8(6)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for **1** .
The anisotropic displacement factor exponent takes the form: $-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^*b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
Ge	52(1)	37(1)	46(1)	-1(1)	11(1)	-4(1)
S	50(1)	42(1)	56(1)	-9(1)	8(1)	-4(1)
C(1)	48(3)	39(2)	47(2)	-2(2)	5(2)	2(2)
C(2)	64(3)	55(3)	64(3)	10(2)	3(3)	-1(2)
C(3)	85(4)	50(3)	70(3)	24(2)	9(3)	9(2)
C(4)	77(4)	35(2)	79(3)	8(2)	26(3)	1(2)
C(5)	60(3)	30(2)	66(3)	-3(2)	24(2)	-3(2)
C(6)	62(3)	41(2)	85(3)	-5(2)	26(3)	-11(2)
C(7)	58(3)	59(3)	95(4)	-17(3)	17(3)	-16(2)
C(8)	54(3)	57(3)	66(3)	-5(2)	-1(3)	-2(2)
C(9)	49(3)	40(2)	51(2)	-1(2)	7(2)	-6(2)
C(10)	49(3)	32(2)	52(2)	-7(2)	16(2)	1(2)
C(11)	48(2)	42(2)	44(2)	-1(2)	7(2)	-4(2)
C(12)	67(3)	55(2)	54(2)	2(2)	23(2)	2(2)
C(13)	78(4)	72(3)	51(2)	-1(2)	25(3)	11(3)
C(14)	70(3)	61(3)	47(2)	-10(2)	-3(2)	16(2)
C(15)	61(3)	42(2)	49(2)	-6(2)	-13(2)	5(2)
C(16)	85(4)	40(2)	70(3)	-4(2)	-12(3)	-2(2)
C(17)	85(4)	40(2)	89(4)	7(2)	-11(3)	-15(2)
C(18)	66(3)	54(3)	73(3)	15(2)	4(3)	-12(2)
C(19)	56(3)	45(2)	55(2)	8(2)	4(2)	-1(2)
C(20)	46(3)	42(2)	47(2)	7(2)	-2(2)	0(2)
C(21)	91(4)	71(3)	72(3)	22(3)	1(3)	3(3)
C(22)	108(5)	80(3)	79(3)	3(3)	46(3)	-12(3)
C(23)	71(4)	61(3)	88(4)	-14(3)	16(3)	-15(3)
O(1)	68(2)	49(2)	62(2)	16(1)	-7(2)	-11(1)
O(2)	86(2)	49(2)	66(2)	4(1)	41(2)	-4(2)
O(3)	52(2)	55(2)	54(2)	-11(1)	12(1)	-4(1)
O(4)	67(2)	93(2)	65(2)	-32(2)	15(2)	-3(2)
O(5)	75(2)	63(2)	93(2)	21(2)	-8(2)	5(2)
F(1)	70(2)	118(3)	170(3)	-27(3)	34(2)	-37(2)
F(2)	163(4)	136(3)	143(3)	-82(3)	41(3)	-75(3)
F(3)	142(4)	83(2)	342(7)	95(4)	-103(4)	-57(3)

Table 5. Crystal data ,data collection and structure refinement for 2

Empirical formula	$C_{23}H_{21}F_3GeO_6S$
Formula weight	555.05
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	a = 10.286(2) Å α = 67.55(3)°
	b = 11.052(2) Å β = 67.69(3)°
	c = 12.449(2) Å γ = 68.76(3)°
Volume	1170.3(4) $Å^3$
Z	2
Density (calculated)	1.575 Mg/m^3
Absorption coefficient	1.459 mm^{-1}
F(000)	564
Crystal size	0.3 x 0.2 x 0.15 mm
Diffractometer used	Stoe IPDS
Temperature	293(2) K
Wavelength	0.71073 Å
Monochromator	graphite
Scan type	N/A
Control reflections	50 to 200 (I>6sigma(I)) on each image
θ range for data collection	1.84 to 22.39°
Index ranges	$-10 \leq h \leq 10$, $-11 \leq k \leq 11$,
	$-13 \leq \ell \leq 13$
Reflections collected	7545
Independent reflections	2818 (R_{int} = 0.0730)
Observed refection	1995 (I>2sigma(I))
Absorption correction	N/A

Programs used	SHELXS-97 (Sheldrick, 1990), SHELXL-97 (Sheldrick, 1997)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2818 / 4 / 319
Goodness-of-fit on F^2	1.060
Final R indices [$I>2\sigma(I)$]	R1 = 0.0591, wR2 = 0.1526
R indices (all data)	R1 = 0.0869, wR2 = 0.1609
Hydrogen atoms	geom
Largest diff. peak and hole	0.627 and -0.322 $e\text{Å}^{-3}$

Table 6. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for **2** . U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor

	x	y	z	U(eq)
Ge	7019(1)	3043(1)	7064(1)	39(1)
S	6413(3)	-1869(2)	8552(2)	62(1)
O(3)	5893(8)	1851(6)	8398(5)	52(2)
O(1)	7944(7)	4802(5)	5345(5)	52(2)
C(1)	7584(9)	4870(7)	4357(7)	43(2)
C(2)	7966(10)	5742(8)	3213(8)	61(3)
C(3)	7593(11)	5668(10)	2283(8)	67(3)
C(4)	6872(10)	4772(10)	2467(7)	59(3)
C(5)	6447(9)	3880(8)	3629(7)	43(2)
C(6)	5673(10)	2942(9)	3873(8)	54(2)
C(7)	5277(10)	2098(9)	5000(9)	57(2)
C(8)	5651(9)	2137(8)	5938(7)	47(2)
C(9)	6386(9)	3034(7)	5794(7)	41(2)
C(10)	6814(8)	3920(7)	4590(6)	37(2)
C(11)	8246(11)	5972(8)	5352(9)	67(3)
O(2)	7865(7)	2855(6)	9010(5)	65(2)
C(12)	9267(11)	2097(9)	8821(8)	55(2)
C(13)	10105(14)	1781(10)	9555(8)	72(3)
C(14)	11544(15)	986(12)	9280(12)	83(4)
C(15)	12100(13)	539(10)	8328(12)	79(3)
C(16)	11281(10)	812(8)	7543(9)	54(2)
C(17)	11858(11)	344(8)	6532(11)	68(3)
C(18)	11093(11)	602(9)	5754(10)	70(3)
C(19)	9636(9)	1432(8)	6002(7)	49(2)
C(20)	9009(9)	1935(7)	6969(7)	38(2)
C(21)	9806(9)	1635(7)	7779(8)	46(2)
C(22)	7239(13)	3418(11)	10002(8)	87(4)
O(4)	5825(9)	-2707(7)	9712(6)	106(3)
O(5)	5913(10)	-1681(11)	7581(8)	139(4)
O(6)	6526(12)	-676(7)	8645(8)	131(4)
C(23)	8314(13)	-2765(13)	8121(10)	82(3)
F(1)	8388(10)	-3904(9)	7979(7)	148(3)
F(2)	9057(9)	-2161(10)	7125(8)	180(5)
F(3)	8886(9)	-3165(9)	8982(8)	149(3)

Table 7. Bond lengths [Å] and angles [°] for **2** .

Ge-C(9)	1.928(8)	Ge-C(20)	1.953(8)
Ge-O(3)	1.951(6)	Ge-O(1)	2.425(5)
Ge-O(2)	2.785(6)	Ge-H(1)	1.51(6)
S-O(5)	1.404(8)	S-O(6)	1.415(8)
S-O(4)	1.425(7)	S-C(23)	1.816(12)
O(3)-O(6)	2.550(9)	O(3)-O(4)	4.684(9)
O(3)-H(3A)	0.849(5)	O(3)-H(3B)	0.848(5)
O(1)-C(1)	1.382(9)	O(1)-C(11)	1.439(9)
C(1)-C(2)	1.382(11)	C(1)-C(10)	1.418(11)
C(2)-C(3)	1.388(13)	C(2)-H(2)	0.9300
C(3)-C(4)	1.347(13)	C(3)-H(3)	0.9300
C(4)-C(5)	1.414(11)	C(4)-H(4)	0.9300
C(5)-C(10)	1.404(11)	C(5)-C(6)	1.405(12)
C(6)-C(7)	1.358(12)	C(6)-H(6)	0.9300
C(7)-C(8)	1.381(12)	C(7)-H(7)	0.9300
C(8)-C(9)	1.375(11)	C(8)-H(8)	0.9300
C(9)-C(10)	1.445(10)	C(11)-H(11A)	0.9600
C(11)-H(11B)	0.9600	C(11)-H(11C)	0.9600
O(2)-C(12)	1.362(11)	O(2)-C(22)	1.421(10)
C(12)-C(13)	1.354(13)	C(12)-C(21)	1.417(12)
C(13)-C(14)	1.409(15)	C(13)-H(13)	0.9300
C(14)-C(15)	1.305(15)	C(14)-H(14)	0.9300
C(15)-C(16)	1.404(14)	C(15)-H(15)	0.9300
C(16)-C(17)	1.383(13)	C(16)-C(21)	1.442(12)
C(17)-C(18)	1.357(13)	C(17)-H(17)	0.9300
C(18)-C(19)	1.432(12)	C(18)-H(18)	0.9300
C(19)-C(20)	1.355(11)	C(19)-H(19)	0.9300
C(20)-C(21)	1.405(11)	C(22)-H(22A)	0.9600
C(22)-H(22B)	0.9600	C(22)-H(22C)	0.9600
C(23)-F(2)	1.260(12)	C(23)-F(3)	1.272(12)
C(23)-F(1)	1.309(14)		

C(9)-Ge-C(20)	110.4(3)	C(9)-Ge-O(3)	97.2(3)
C(20)-Ge-O(3)	102.0(3)	C(9)-Ge-O(1)	76.7(3)
C(20)-Ge-O(1)	89.4(3)	O(3)-Ge-O(1)	168.4(2)
C(9)-Ge-O(2)	175.2(3)	C(20)-Ge-O(2)	69.2(3)
O(3)-Ge-O(2)	78.4(2)	O(1)-Ge-O(2)	107.96(19)
C(9)-Ge-H(1)	111(2)	C(20)-Ge-H(1)	131(2)
O(3)-Ge-H(1)	97(2)	O(1)-Ge-H(1)	77(2)
O(2)-Ge-H(1)	71(2)	O(5)-S-O(6)	115.0(6)
O(5)-S-O(4)	118.7(6)	O(6)-S-O(4)	111.2(5)
O(5)-S-C(23)	103.8(6)	O(6)-S-C(23)	101.2(6)
O(4)-S-C(23)	104.3(5)	Ge-O(3)-O(6)	122.1(3)
Ge-O(3)-O(4)	138.1(2)	O(6)-O(3)-O(4)	16.1(3)
Ge-O(3)-H(3A)	141(5)	O(6)-O(3)-H(3A)	69(3)
O(4)-O(3)-H(3A)	56(3)	Ge-O(3)-H(3B)	92(5)
O(6)-O(3)-H(3B)	82(5)	O(4)-O(3)-H(3B)	85(5)
H(3A)-O(3)-H(3B)	127(7)	C(1)-O(1)-C(11)	118.4(6)
C(1)-O(1)-Ge	108.7(4)	C(11)-O(1)-Ge	128.2(5)
O(1)-C(1)-C(2)	124.5(8)	O(1)-C(1)-C(10)	114.3(6)
C(2)-C(1)-C(10)	121.2(8)	C(1)-C(2)-C(3)	118.9(9)
C(1)-C(2)-H(2)	120.6	C(3)-C(2)-H(2)	120.6
C(4)-C(3)-C(2)	121.8(9)	C(4)-C(3)-H(3)	119.1
C(2)-C(3)-H(3)	119.1	C(3)-C(4)-C(5)	120.7(9)

C(3)-C(4)-H(4)	119.6	C(5)-C(4)-H(4)	119.6
C(10)-C(5)-C(6)	117.9(7)	C(10)-C(5)-C(4)	119.1(8)
C(6)-C(5)-C(4)	123.0(8)	C(7)-C(6)-C(5)	121.8(8)
C(7)-C(6)-H(6)	119.1	C(5)-C(6)-H(6)	119.1
C(6)-C(7)-C(8)	119.9(8)	C(6)-C(7)-H(7)	120.1
C(8)-C(7)-H(7)	120.1	C(9)-C(8)-C(7)	122.6(8)
C(9)-C(8)-H(8)	118.7	C(7)-C(8)-H(8)	118.7
C(8)-C(9)-C(10)	117.0(7)	C(8)-C(9)-Ge	123.4(6)
C(10)-C(9)-Ge	119.2(6)	C(5)-C(10)-C(1)	118.4(7)
C(5)-C(10)-C(9)	120.7(7)	C(1)-C(10)-C(9)	120.9(7)
O(1)-C(11)-H(11A)	109.5	O(1)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5	O(1)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5	H(11B)-C(11)-H(11C)	109.5
C(12)-O(2)-C(22)	119.0(8)	C(12)-O(2)-Ge	104.9(5)
C(22)-O(2)-Ge	135.9(6)	C(13)-C(12)-O(2)	124.3(9)
C(13)-C(12)-C(21)	121.3(10)	O(2)-C(12)-C(21)	114.5(8)
C(12)-C(13)-C(14)	119.8(10)	C(12)-C(13)-H(13)	120.1
C(14)-C(13)-H(13)	120.1	C(15)-C(14)-C(13)	121.5(10)
C(15)-C(14)-H(14)	119.2	C(13)-C(14)-H(14)	119.2
C(14)-C(15)-C(16)	121.4(11)	C(14)-C(15)-H(15)	119.3
C(16)-C(15)-H(15)	119.3	C(17)-C(16)-C(15)	122.0(10)
C(17)-C(16)-C(21)	118.9(9)	C(15)-C(16)-C(21)	119.1(10)
C(18)-C(17)-C(16)	123.1(9)	C(18)-C(17)-H(17)	118.5
C(16)-C(17)-H(17)	118.5	C(17)-C(18)-C(19)	116.9(9)
C(17)-C(18)-H(18)	121.6	C(19)-C(18)-H(18)	121.6
C(20)-C(19)-C(18)	123.1(9)	C(20)-C(19)-H(19)	118.5
C(18)-C(19)-H(19)	118.5	C(19)-C(20)-C(21)	119.3(8)
C(19)-C(20)-Ge	113.8(6)	C(21)-C(20)-Ge	126.9(6)
C(20)-C(21)-C(12)	124.4(8)	C(20)-C(21)-C(16)	118.7(8)
C(12)-C(21)-C(16)	116.8(9)	O(2)-C(22)-H(22A)	109.5
O(2)-C(22)-H(22B)	109.5	H(22A)-C(22)-H(22B)	109.5
O(2)-C(22)-H(22C)	109.5	H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5	S-O(4)-O(3)	48.4(3)
S-O(6)-O(3)	156.3(7)	F(2)-C(23)-F(3)	113.4(13)
F(2)-C(23)-F(1)	105.1(11)	F(3)-C(23)-F(1)	102.6(10)
F(2)-C(23)-S	114.7(8)	F(3)-C(23)-S	111.6(9)
F(1)-C(23)-S	108.4(10)		

Symmetry transformations used to generate equivalent atoms:

Table 8. Anisotropic displacement parameters [$Å^2$ x 10^3] for **2** .
The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [$(ha^*)^2U_{11}$ + ... + $2hka^*b^*U_{12}$]

	U11	U22	U33	U23	U13	U12
Ge	40(1)	36(1)	37(1)	-10(1)	-10(1)	-7(1)
S	74(2)	60(2)	46(1)	-17(1)	-9(1)	-16(1)
O(3)	50(5)	55(4)	52(4)	-14(3)	-10(4)	-19(3)
O(1)	71(5)	56(4)	43(3)	-6(3)	-21(3)	-36(3)
C(1)	43(6)	38(5)	41(5)	-8(4)	-7(4)	-12(4)
C(2)	63(7)	47(5)	60(6)	-6(5)	-9(5)	-17(5)
C(3)	63(8)	75(7)	43(6)	-7(5)	-15(5)	-6(6)
C(4)	59(7)	72(7)	43(6)	-25(5)	-16(5)	-4(5)
C(5)	35(5)	48(5)	44(5)	-17(4)	-14(4)	0(4)
C(6)	55(7)	61(6)	58(6)	-35(5)	-29(5)	4(5)
C(7)	44(6)	64(6)	81(7)	-34(5)	-21(5)	-15(5)
C(8)	43(6)	46(5)	51(5)	-9(4)	-13(4)	-15(4)
C(9)	41(5)	38(4)	44(5)	-10(4)	-13(4)	-8(4)
C(10)	33(5)	40(4)	31(4)	-9(4)	-12(4)	-2(4)
C(11)	78(8)	53(6)	89(7)	-22(5)	-41(6)	-18(5)
O(2)	65(5)	82(4)	55(4)	-35(3)	-19(3)	-7(4)
C(12)	70(8)	53(5)	48(6)	-4(4)	-24(5)	-26(5)
C(13)	107(10)	78(7)	53(6)	-5(5)	-45(6)	-39(7)
C(14)	100(11)	78(8)	91(9)	19(7)	-74(8)	-39(8)
C(15)	65(8)	61(7)	115(10)	7(7)	-66(8)	-12(6)
C(16)	53(7)	33(5)	72(6)	-2(5)	-27(5)	-10(4)
C(17)	44(7)	40(5)	111(9)	-28(6)	-19(6)	3(4)
C(18)	55(7)	68(6)	88(8)	-40(6)	-10(6)	-11(6)
C(19)	43(6)	46(5)	56(5)	-26(4)	-14(4)	2(4)
C(20)	40(5)	39(4)	40(5)	-10(4)	-19(4)	-8(4)
C(21)	45(6)	28(4)	60(6)	-5(4)	-15(5)	-12(4)
C(22)	116(10)	106(8)	55(6)	-45(6)	-13(6)	-36(7)
O(4)	111(7)	81(5)	76(5)	-23(4)	29(4)	-25(5)
O(5)	97(7)	218(11)	110(7)	-71(7)	-52(6)	2(7)
O(6)	198(10)	54(5)	133(7)	-34(4)	-38(7)	-23(5)
C(23)	80(9)	88(8)	67(7)	-20(6)	-31(7)	-1(7)
F(1)	159(8)	129(6)	148(7)	-95(6)	-40(6)	27(6)
F(2)	93(6)	200(9)	127(7)	20(6)	28(5)	-37(6)
F(3)	115(7)	198(8)	135(7)	-73(6)	-70(6)	22(6)