tural parameters do not suggest a marked attraction between the hydrogen and fluorines, or an increase in d(PH) compared to PH_3, or a decrease in $\angle$FPF compared to PF_3, all of which would be expected consequences of such an interaction. Admittedly, however, it is difficult to completely exclude this interaction.

Acknowledgments. The author thanks Dr. Ralph Rudolph and Mr. Louis Centofanti, who supplied the HPF_2 and DPF_2, and Dr. David Lide, Jr., in whose laboratory the initial measurements were made. The author also thanks Dr. Robert Parry, who suggested the problem to us.

Ring-Segment Exchange Reactions of Cyclic Germthioxanes at Equilibrium

Kurt Moedritzer and John R. Van Wazer

Contribution from the Central Research Department, Monsanto Company, St. Louis, Missouri 63166. *Received September 18, 1967*

Abstract: In mixtures of trimeric dimethylgermanium sulfide and trimeric and/or tetrameric dimethylgermanium oxide, interchange of bridging sulfur with oxygen atoms between the dimethylgermanium groups in the ring structures has been observed by proton nmr. The resulting equilibria between the four trimeric cyclic molecules—the two starting compounds and the two new dimethylcyclogermthioxanes (cyclic mixed oxide–sulfides of dimethylgermanium)—are determined by two equilibrium constants with a third constant relating one of these hexatomic ring structures to the octatomic ring structure of the dimethylgermanium oxide tetramer. Detectable amounts of other cyclic or linear molecules were not present at equilibrium. The reaction paths and the reasons for the observed values of the equilibrium constants are discussed.

Cyclic molecular structures based on germanium atoms alternating with heteroatoms, with the latter being all of one kind, have been known for some time. In the literature, such compounds have been described with the bridging heteroatoms being oxygen,[1,2] sulfur,[1,2] selenium,[3] or nitrogen.[4] However, there have been no reports of the synthesis of such ring compounds where the heteroatoms are of more than one kind. In this paper, by studying the exchange equilibria of bridging oxygen with bridging sulfur in mixtures of trimeric dimethylgermanium sulfide (hexamethylcyclotrigermanium trisulfide) with trimeric/tetrameric dimethylgermanium oxide (hexamethylcyclotrigermoxane and octamethylcyclotetragermoxane), the existence of cyclic molecular species having oxygen as well as sulfur atoms alternating with germanium (*i.e.*, cyclic germthioxanes) has been established. The work discussed herein is related to other investigations from this laboratory dealing with the equilibria in cyclic silthiazanes[5] and cyclic germanosilthianes.[6] Cyclic silthioxanes also have been reported recently.[7]

Experimental Section

Reagents. Trimeric dimethylgermanium sulfide and tetrameric[8] dimethylgermanium oxide were prepared according to the methods reported in the literature.[1,2] The recrystallized substances did not show any proton-containing impurities detectable by proton nuclear magnetic resonance (nmr) and the observed melting points agreed with literature values.[1,2]

Procedures. Seven samples made up of various proportions of trimeric dimethylgermanium sulfide and tetrameric dimethylgermanium oxide in five parts by volume of carbon tetrachloride were sealed in 5-mm o.d. precision nmr tubes. For the equilibrium at 120°, the samples were held at this temperature for various time intervals with the progress of the equilibration reaction checked at that temperature by proton nmr. No change in the spectra upon further heating was observed when the samples had been maintained at this temperature for *ca.* 3 days (high sulfide content) to *ca.* 20 days (high oxide content). To ensure complete equilibration, all samples were kept at 120° for another 24 days, and then analyzed by nmr, with the probe of the Varian A-60 spectrometer being at 120°.

Reequilibration of the 120° samples at room temperature appears to consist of several steps which vary by more than 100-fold in rate. Analyses showed a rapid increase in the amount of tetrameric dimethylgermanium oxide within the first few hours at 25° while reequilibration between the trimeric dimethylgermthioxanes was still underway after several weeks. The reequilibration experiment was terminated after a total of 47 days at room temperature. The nmr probe temperature for these room-temperature spectra was 35°.

The proton nmr spectra were evaluated quantitatively by electronic integration of the peak areas or by cutting out and weighing of Xerox copies of the spectra. The chemical shifts of the resonances observed and their assignments to various segments of the cyclic molecules are presented in Table I. The equilibrium constants were calculated, using the computer programs described previously,[9] by formally treating the "cyclotrigermanium" assemblage as a trifunctional central moiety[5] and the bridging oxygen and sulfur atoms as the exchanging substituents. The mathematical treatment of the equilibria between cyclic trimeric and tetrameric forms of dimethylgermanium oxide has been described elsewhere.[8]

Results and Conclusions

The facile interchange of germanium–oxygen with germanium–sulfur bonds has been demonstrated earlier

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[8] K. Moedritzer, *J. Organometal. Chem.* (Amsterdam), 5, 254 (1966).

[9] L. C. D. Groenweghe, J. R. Van Wazer, and A. W. Dickinson, *Anal. Chem.*, 36, 303 (1964) [pp 305–307 only, with no corrections for sensitivity bias].

Table I. Proton Nmr Chemical Shifts[a] in Equilibrated Samples of the System $[(CH_3)_2GeO]$ *vs.* $[(CH_3)_2GeS]$

	S— / $(CH_3)_2Ge$ \ S—		S— / $(CH_3)_2Ge$ \ O—		O— / $(CH_3)_2Ge$ \ O—	
(SSS)[b,c]	A	−0.928				
(SSO)[d]	B	−0.892 (1)[e]	C	−0.748 (2)		
(SOO)[f]			D	−0.715 (2)	E	−0.523 (1)
(OOO)[g]					F	−0.491
(OOOO)[h]					G	−0.472

[a] Measured in ppm relative to internal tetramethylsilane in the 5:1 CCl_4 solution of an equilibrated sample having the over-all composition $R \equiv O/Ge = 0.632$. [b] Hexamethylcyclotrigermanium trisulfide. [c] The capital letters refer to the resonances in Figure 1. [d] Hexamethylcyclotrigermdithioxane. [e] The numbers in parentheses represent area ratios for the resonances of the compound given. [f] Hexamethylcyclotrigermthiodioxane. [g] Hexamethylcyclotrigermoxane. [h] Octamethylcyclotetragermoxane.

in a study of the equilibria involving exchange of the substituents OCH_3 and SCH_3 on the dimethylgermanium moiety in the system[10] $(CH_3)_2Ge(OCH_3)_2$ *vs.* $(CH_3)_2Ge(SCH_3)_2$. Therefore, the exchange of bridging oxygen with bridging sulfur on the dimethylgermanium moiety in the system $[(CH_3)_2GeO]$ *vs.* $[(CH_3)_2GeS]$ involving cyclic species was expected to proceed with ease.

Indeed, when mixtures of hexamethylcyclotrigermanium trisulfide (SSS) and octamethylcyclotetragermanium tetraoxide (OOOO) were heated at 120° for several days, exchange of bridging sulfur with bridging oxygen atoms was observed. As a result of this redistribution process, new cyclic structures were formed which contain sulfur as well as oxygen in the molecule. As determined quantitatively by proton nmr, the exchange processes proceed to an equilibrium state which is characterized by the presence of four trimeric and a tetrameric ring species. The assignment of the observed seven proton-nmr resonances at equilibrium to specific molecules or molecular segments in the mixed cyclic structures was based on the following reasoning.

First, the proton chemical shift of the single resonances corresponding to each of the starting materials as well as to that of the hexamethylcyclotrigermanium trioxide were known[8,11] and were found to agree well with three of the shifts seen for the mixtures. Furthermore, addition of small amounts of each one of these substances to an equilibrated mixture caused an increase of the area of its assigned resonance as expected (*i.e.*, peaks A, F, and G, respectively, in Table I). Secondly, pairs of the four remaining resonances were found to occur in a constant-area ratio throughout the series of spectra of equilibrated mixtures of different over-all composition, regardless of whether or not the samples were at equilibrium. As exemplified by the sample spectrum of Figure 1, the resonances B and C maintain (within experimental error) the exact area ratio 1:2 and therefore were assigned to the hexamethylcyclotrigermdithioxane (SSO) in which one dimethylgermanium moiety is linked through two sulfur bridges to the other two germanium atoms which in turn are bonded to an oxygen as well as a sulfur bridge. Similarly from the constant 2:1-area relationship of the resonances D and E, these were assigned to the hexamethylcyclotrigermthiodioxane molecule (SOO). The above assignments are confirmed by material-balance calculations.

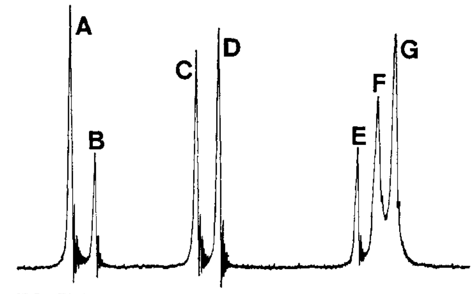
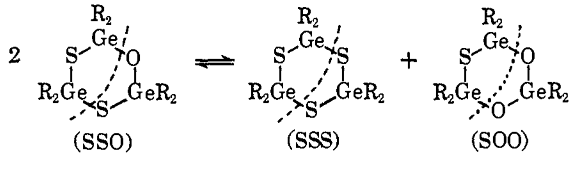
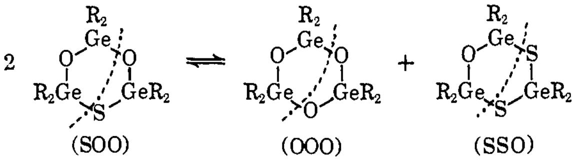


Figure 1. Proton nmr spectrum of an equilibrated sample in the system $[(CH_3)_2GeS]$ *vs.* $[(CH_3)_2GeO]$ of the composition $R \equiv O/Ge = 0.632$ in CCl_4 solution (5:1) referenced *vs.* internal tetramethylsilane as standard. The assignments of the resonances are presented in Table I.

The equilibria between the hexatomic ring molecules are fully described by the reactions of eq 1 and 2, with



(1)



(2)

the methyl groups being denoted by R. In addition to the hexatomic ring molecules at equilibrium, there is also present octamethylcyclotetragermoxane which therefore may be included in the representation of the over-all equilibria by the equation

$$4[(CH_3)_2GeO]_3 \rightleftarrows 3[(CH_3)_2GeO]_4 \qquad (3)$$

No evidence has been found in the nmr spectra for the presence at equilibrium of other possible octatomic or larger sized ring structures, *i.e.*, species containing oxygen as well as sulfur bridges.

A complete quantitative description of the over-all equilibria in this system requires three equilibrium constants which we have chosen to be derived from the reactions of eq 1–3.

$$K^{\circ\circ}{}_{SSO} = [SSS][SOO]/[SSO]^2 \qquad (4)$$

$$K^{\circ\circ}{}_{SOO} = [OOO][SSO]/[SOO]^2 \qquad (5)$$

$$K^{\circ\circ}{}_{40,30} = [OOOO]^3/[OOO]^4 \qquad (6)$$

At 120°, the values of these equilibrium constants were found to be the following: $K^{\circ\circ}{}_{SSO} = 0.87 \pm 0.05$; $K^{\circ\circ}{}_{SOO} = 0.59 \pm 0.03$; $K^{\circ\circ}{}_{40,30} = 0.24 \pm 0.03$ l./mole. The experimental data on which these constants are based are given in Table II, along with their respective values calculated from the constants given in the preceding sentence. It should be noted that the agreement between the experimental and calculated values is good.

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(10) J. R. Van Wazer, K. Moedritzer, and L. C. D. Groenweghe, *J. Organometal. Chem.* (Amsterdam), **5**, 420 (1966).

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Table II. Experimental and Calculated Equilibrium Concentrations in Mole Per Cent for the System [$(CH_3)_2GeS$] *vs.* [$(CH_3)_2GeO$] at 120°

$R \equiv$ O/Ge	(SSS)	(SSO)	(SOO)	(OOO)	(OOOO)
0.143[a] (0.138)[c]	70.6[b] (69.5)[d]	20.0 (22.8)	7.2 (6.5)	1.5 (1.1)	0.8 (0.2)
0.411 (0.404)	32.7 (32.0)	28.6 (29.7)	23.5 (24.0)	12.0 (11.4)	3.2 (3.0)
0.487 (0.462)	26.2 (26.8)	29.4 (28.5)	23.9 (26.3)	14.9 (14.3)	5.6 (4.2)
0.632 (0.615)	14.2 (13.8)	20.9 (22.1)	31.2 (30.5)	24.5 (24.8)	9.2 (8.8)
0.735 (0.706)	8.7 (8.1)	16.0 (16.7)	30.2 (30.0)	32.3 (31.7)	12.8 (13.5)
0.862 (0.841)	2.5 (2.2)	7.2 (7.8)	21.1 (23.9)	42.8 (42.9)	26.4 (23.1)

[a] From the ingredients of the samples. [b] From the nmr peak areas. [c] From the nmr data. [d] Calculated from $K^{\circ\circ}_{SSO} = 0.87$, $K^{\circ\circ}_{SOO} = 0.59$, and $K^{\circ\circ}_{40,30} = 0.24$ l./mole.

The experimental data obtained after reequilibration of these samples at 25° for 47 days is shown in Table III. If equilibrium constants are fitted to these room-temperature data, the following values are found: $K^{\circ\circ}_{SSO} = 0.80 \pm 0.02$; $K^{\circ\circ}_{SOO} = 0.79 \pm 0.02$; and $K^{\circ\circ}_{40,30} = 5.3 \pm 0.8$ l./mole. Since lowering the temperature should cause the equilibrium constants of eq 4 and 5 to deviate increasingly from the random value of 0.333, it is apparent that the value of $K^{\circ\circ}_{SSO}$ is incorrect. On the assumption that the entropy of the reactions of eq 1 and 2 is completely established by the scrambling of the bridging oxygen and sulfur atoms, a value of the enthalpy of 0.6 kcal for eq 1 and 0.5 kcal for eq 2 was calculated from the relationship $\Delta H = -(RT/2) \ln (K/0.333)$. From this, we conclude that at room temperature $K^{\circ\circ}_{SSO} = 1.07$ and $K^{\circ\circ}_{SOO} = 0.72$. These two values and the experimental $K^{\circ\circ}_{40,30} = 5.3$ have been used to calculate the theoretical concentrations shown in parentheses in Table III.

In previous studies[8,12] involving the equilibrium between trimeric and tetrameric dimethylgermanium oxides, it was found that the equilibrium between these two species is established within several hours at room temperature. It is apparent that the reequilibration observed on going from 120 to 25° first involves a fairly rapid shift of eq 3 to the right. The resulting diminution in the amount of trimeric dimethylgermanium oxide leads to a slower shift in the equilibrium represented by eq 2, which in turn is followed by an extremely slow shift in the equilibrium of reaction 2. Thus from Table III we see that, after the 49-day period of reequilibration at room temperature, the 25° equilibrium represented by eq 3 is established and that of eq 2 is nearly established while the reaction of eq 1 has not yet reached the 25° equilibrium position. Nmr measurements during the reequilibration period bear out this rationale. Even at 1 hr after quenching to room temperature, a pronounced shift of the OOO/OOOO equilibrium (corresponding to eq 3) was observed, with no change in the relative amounts of OOS, OSS, and SSS.

Table III. Experimental and Calculated Equilibrium Concentrations in Mole Per Cent for the System [$(CH_3)_2GeS$] *vs.* [$(CH_3)_2GeO$] at Room Temperature

$R \equiv$ O/Ge	(SSS)	(SSO)	(SOO)	(OOO)	(OOOO)
0.143[a] (0.127)[c]	69.5[b] (71.6)[d]	24.1 (20.5)	5.3 (6.3)	0.8 (1.4)	0.3 (0.4)
0.307 (0.263)	49.5 (51.7)	29.8 (26.1)	13.4 (14.1)	4.8 (5.5)	2.6 (2.6)
0.411 (0.390)	37.8 (38.6)	25.7 (26.1)	18.2 (18.9)	9.8 (9.9)	8.5 (6.6)
0.487 (0.467)	28.3 (28.5)	27.2 (27.6)	20.7 (21.3)	12.8 (12.7)	11.0 (10.0)
0.632 (0.617)	16.4 (18.2)	21.9 (20.0)	22.2 (23.6)	19.1 (20.0)	20.5 (18.3)
0.737 (0.731)	8.5 (10.2)	16.0 (14.7)	23.2 (22.6)	23.0 (25.0)	29.3 (27.6)
0.862 (0.858)	3.5 (3.5)	7.7 (7.7)	17.0 (18.0)	30.2 (30.6)	41.7 (40.3)

[a] From the ingredients of the mixture. [b] From the nmr peak areas. [c] From the nmr data. [d] Calculated from $K^{\circ\circ}_{SSO} = 1.07$, $K^{\circ\circ}_{SOO} = 0.72$, and $K^{\circ\circ}_{40,30} = 5.3$ l./mole.

Discussion

Reaction Paths. The reason that eq 1–3 completely account for the kinds and amounts of products obtained by mixing dimethylgermanium sulfide with its oxide is due to the fact that the ring–chain equilibria[12] are sufficiently shifted toward the ring structures so that the trace amounts of chain-terminating impurities (perhaps Cl or OH groups) introduced with the reagents are insufficient to give a detectable amount of noncyclics. For example, the equilibrium constants obtained for the chlorine-terminated polydimethylgermoxanes[12] (or polydimethylgermthians[11]) lead to a calculated amount of 0.05% (or 0.007%) of the total germanium in chains averaging 10.4 (or 2.5) Ge atoms per molecule on the reasonable assumption of 0.01 mole % of chain-terminating impurities in the reagents.

It seems reasonable to postulate that the various reactions making up the dynamic equilibria represented by eq 1–3 proceed *via* a small amount of chain species (*vide supra*) as intermediates. Thus eq 1 and 2 are thought to be based on reactions involving (1) opening of a reactant ring as it is incorporated into a chain, (2) exchange of parts between the chains, followed by (3) the twisting off of product rings from the chains. In view of the lesser amount of chains and their shorter average length at the high sulfur end of this system, the observed faster rate with increasing sulfur content surely means that the intrinsic rate of exchange involving Ge–S bond scission is faster than that involving Ge–O bond scission.

Equilibrium Constants. The small deviations from randomness observed for $K^{\circ\circ}_{SOO}$ and $K^{\circ\circ}_{SSO}$ seem to be due in the main to the fact that sulfur atoms are being sorted with oxygen atoms, regardless of any complications due to the ring structure. This can be seen quite clearly by reference to eq 1 and 2, where it should be noted that for each equation the -Ge-S-Ge- or -Ge-O-Ge- bridge within the dashed semicircle remains the same so that, for the purpose of illustration, it may be neglected. If this is done, we see that both eq 1 and

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(12) K. Moedritzer and J. R. Van Wazer, *Inorg. Chem.*, 4, 1753 (1965).

2 refer to the scrambling of a sulfur and an oxygen atom on a dimethylgermanium group. The equilibrium constants, $K = [(CH_3)_2Ge(S-)_2][(CH_3)_2Ge(O-)_2]/[(CH_3)_2Ge(O-)(S-)]^2$, calculated on the basis of such scrambling are 0.87 ± 0.05 for eq 1 and 0.59 ± 0.03 for eq 2 at 120°. These may be compared with the scrambling[10] of methoxyl and methylthio groups on the dimethylgermanium moiety in terms of the deviation of the free energy from the random value. For the latter case, this deviation corresponds to +0.9 kcal, whereas for the equilibrium constants of 0.87 and 0.59 the respective values are +0.5 and +0.3 kcal. The sign of the deviation is the same in all three cases and the size is not much different.

In the three cases (ref 5 and 6 and the data reported here) where equilibrium constants have been obtained for the stepwise substitution of a bridging moiety in hexatomic rings, the equilibrium constants of the general type of eq 4 and 5 are close to random, and an argument (such as the one given in the preceding paragraph) which ignores cyclization can be used for accounting for the deviations from randomness. At first glance, one would expect the wide range of observed values of the ring–chain[13] equilibrium constants for hexatomic rings would lead to correspondingly large deviations from randomness in the scrambling of bridging atoms in this sized ring. This apparent problem is resolved by requiring that there be a linear change, with increasing substitution of one bridging group by another, in the free energy (exclusive of the entropy contribution due to simple scrambling) of formation of the rings.

A statistical-mechanical study[13] has shown that the primary contribution to the differences between the ring–chain constants is the torsional freedom within the rings. Likewise, the torsional freedom must also play a key role in establishing the values of the equilibrium constants of the type of eq 4 and 5 of this paper. This means that the stepwise replacement of pairs of adjacent bonds by either weaker or stronger bonds must lead to a nearly fixed incremental change in the over-all torsional energy of the ring in order to have these equilibrium constants be approximately random. Presumably such behavior results from the strong coupling of the torsional vibrations within any hexatomic ring.

Acknowledgment. We wish to thank Dr. Richard M. Levy for helpful discussion of ring-equilibrium theory and Mr. Raymond E. Miller for experimental assistance.

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Derivatives of Bis(trifluoromethyl)ketene Which Contain Fluorosulfato, Difluoramino, Fluorimino, and/or Fluoroxy Groups

Dayaldas T. Meshri and Jean'ne M. Shreeve

Contribution from the Department of Chemistry, University of Idaho, Moscow, Idaho 83843. *Received September 23, 1967*

Abstract: Addition reactions across the carbon–carbon double bond in bis(trifluoromethyl)ketene with peroxodisulfuryl difluoride, tetrafluorohydrazine, difluoraminofluorosulfate, and fluorine fluorosulfate yield $(CF_3)_2C(OSO_2F)C(O)OSO_2F$, $(CF_3)_2C(NF_2)CFO$, $(CF_3)_2C(NF_2)C(O)OSO_2F$, $(CF_3)_2C(OSO_2F)CFO$, and $(CF_3)_2CFC(O)OSO_2F$, respectively. While in the presence of CsF only, $(CF_3)_2C(NF_2)CFO$ is converted to $(CF_3)_2C{=}NF$. With CsF and F_2, the former and $(CF_3)_2C{=}C{=}O$ give rise to $(CF_3)_2C(NF_2)CF_2OF$ and $(CF_3)_2CFCF_2OF$, respectively. These new compounds have been characterized and structures confirmed by nmr, mass, and infrared spectra.

Numerous investigations dealing with the interaction of bis(trifluoromethyl)ketene($(CF_3)_2C{=}C{=}O$)and organic materials have been reported; *e.g.*, cycloaddition to both the C=C and C=O groups occurs with vinyl benzoate at 100°, and simple alkenes also add to form cyclobutanones and linear adducts.[1] Knunyants and coworkers have published extensively on the reaction chemistry of $(CF_3)_2C{=}C{=}O$.[2-11] However, the reactions of $(CF_3)_2C{=}C{=}O$ with typical inorganic fluorine-containing free-radical sources had not been

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