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STUDIES ON BIS(TRIMETHYLTIN) AZIDE HYDROXIDE AND HEXAMETHYL-1,5-DIAZIDOTRISTANNOXANE BY MÖSSBAUER AND VIBRATIONAL SPECTROSCOPY

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Summary

The solid state chemistry of bis(trimethyltin) azide hydroxide and hexamethyl-1,5-diazidotristannoxane has been investigated by Mössbauer, infrared and Raman spectroscopy. The structure of $[(CH_3)_3Sn]_2(OH)N_3$ may be described as polymeric trigonal bipyramidal, in which $(CH_3)_3Sn^{IV}$ moieties are bridged by OH and N_3 (through α-nitrogen) groups, and the chains are cross-linked by hydrogen bonding between oxygens and the γ-nitrogens of the azide groups. The structure of the tristannoxane is assumed to be that of a dimeric oligomer $[N_3(CH_3)_2Sn[OSn(CH_3)_2]_2N_3]_2$ containing five-coordinated trigonal bipyramidal Sn^{IV} arising from cross-linking through the oxygens and the two α-bridging azides.

Introduction

A research program involving Mössbauer, laser Raman and infrared spectroscopic studies of organotin(IV)—azide derivatives has been underway for some time in our laboratories and experimental results on neutral tri- and diorganotin(IV) azides have been reported [1]. A subsequent investigation was devoted to salts of complex organotin(IV)—azide and —azide thiocyanate anions [2]. The present investigation summarizes studies on two organotin(IV) azides containing Sn—O bonds (the title compounds), which were selected as representative of $(CH_3)_3Sn^{IV}$ and $(CH_3)_2Sn^{IV}$ derivatives resulting from the competition of N_3^- and H_2O ligands towards alkyltin(IV) moieties.

Triclinic crystals of bis(trimethyltin) azide hydroxide were identified in a solid sample of $(CH_3)_3SnN_3$ synthesized some years ago and presumably partially hydrolyzed by atmospheric moisture [3]. The unit cell dimensions of this sample were determined, suggesting a relationship between the configurations of $[(CH_3)_3Sn]_2(OH)N_3$ and $[(CH_3)_3Sn]_2(OH)NCO$, the latter showing a

TABLE 1

MÖSSBAUER PARAMETERS (mm · sec^{-1})

	I.S. [a]	Q.S.	Γ_+ [b]	Γ_- [b]	T(K)	R [c]
$[(CH_3)_3Sn]_2(OH)N_3$	1.291	3.254	1.010	0.985	81	0.896
$[N_3(CH_3)_2Sn\{OSn(CH_3)_2\}_2N_3]_2$	1.147	2.918	1.100	0.961	82.5	0.898
$(CH_3)_3SnN_3$	1.43	3.67			82 ± 4	
$(CH_3)_3SnOH$	1.202	2.944			78.2	1.07

[a] Relative to the centroid of a $BaSnO_3$—$BaSnO_3$ room temperature spectrum. [b] Full width at half height of the resonant peaks occurring at more positive and more negative velocity than the spectrum centroid resepctively. [c] Ratio of the line areas, $R = A_+/A_-$.

polymeric structure of the trigonal bipyramidal type [3]. Hexamethyl-1,5-diazidotristannoxane was obtained by reaction of NaN_3 and H_2O with $(CH_3)_2SnCl_2$ and $Cl(CH_3)_2SnOSn(CH_3)_2Cl$ [4]. The corresponding dibutyltin(IV) derivative was assumed to have a dimeric tristannoxane skeleton on the basis of the attempted X-ray structure determination [4].

The above studies did not yield definitive structural information, but led only to conjectures concerning plausible configurations for these molecules. The Mössbauer, infrared and Raman spectroscopic studies reported in the present study were carried out with the aim of further elucidating the solid state structures of these two interesting compounds.

Experimental

$(CH_3)_3SnN_3$ was prepared by ether extractions of aqueous HCl solutions of $(CH_3)_3SnCl$ (Fluka purum, Fluka AG, Buchs, Switzerland) and NaN_3 [5].

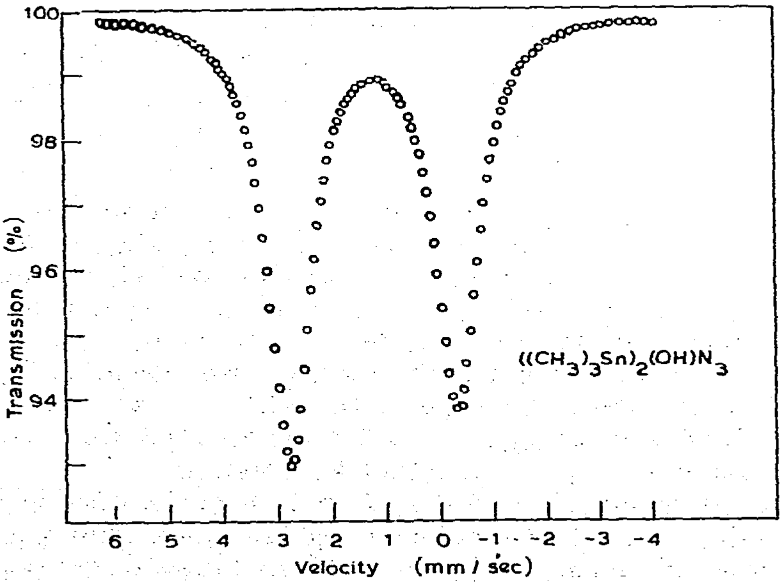

Fig. 1. Mössbauer spectrum of $[(CH_3)_3Sn]_2(OH)N_3$ at 81K. The isomer shift refers to the centroid of a room temperature $BaSnO_3$—$BaSnO_3$ spectrum taken using the same ^{119}Sn source. The intensity asymmetry in the two components of the resonance doublet may be due in part to crystal orientation effects.

Bis(trimethyltin) azide hydroxide was obtained by refluxing for a few minutes a 1/1 solution (1.3 mmol of each compound) of $(CH_3)_3SnN_3$ and $(CH_3)_3SnOH$ (Fluka) in 40 ml of 1/1 n-hexane/benzene; on cooling, 0.4 g of a white crystalline solid precipitated, m.p. 141 - 142° (lit. [3] 140 - 142°). This compound is also obtainable as a by-product of the ether extraction of aqueous $(CH_3)_3SnCl + NaN_3$, and isolated by repeated recrystallizations from benzene. Analysis for a typical sample: found: C, 18.67; H, 5.00; N, 11.02; O, 4.06; Sn, 60.90. $[(CH_3)_3Sn)]_2(OH)N_3$ calcd.: C, 18.64; H, 4.95; N, 10.87; O, 4.14; Sn, 61.40%.

Hexamethyl-1,5-diazidotristannoxane precipitated as a white powder from 20 ml of an aqueous solution of 2 mmol of $(CH_3)_2SnCl_2$ (Fluka) and 10 mmol of NaN_3. Although decomposition may occur earlier, no melting point could be found below 350°. Analysis found: C, 13.03; H, 3.10; N, 14.90; O, 5.70; Sn, 62.50. $N_3(CH_3)_2Sn[OSn(CH_3)_2]_2N_3$ calcd.: C, 12.81; H, 3.23; N, 14.95; O, 5.69; Sn, 63.32%. This compound corresponds to that prepared by Matsuda et al. [4] from water/methanol solution.

Mössbauer spectra were determined at liquid N_2 temperature using the apparatus and techniques described earlier [6,2]. Data reduction was effected using a matrix inversion program on the Rutgers IBM 360/67 computer. In this program, line width, effect magnitude and line position are allowed to vary as independent parameters using a successive iteration algorithm. Replicate spec-

TABLE 2

RELEVANT VIBRATIONAL BANDS [a] (cm^{-1})

$[(CH_3)_3Sn]_2(OH)N_3$		$[N_3(CH_3)_2Sn\{OSn(CH_3)_2\}_2N_3]_2$		Assignments
IR	Raman	IR	Raman	
3460 m (br)	3460 vw (br)			ν (OH)
2060 s	2055 w	2060 s	2080 vw	ν_{as} (N_3)
1337 m	1342 w	1330 m	1330 vw	2 × δ (N_3)
1295 m	1297 w	1280 m		ν_s (N_3)
1197 m	1195 m	1200 m	1197 m	
1065 m (br)				δ (OH)
		720 m		ν (SnOSn) ?
660 m		650 w		δ (N_3)
545 s	550 m		572 m	$\nu_{as}(SnC_n)$ [b]
		565 s (br)		ν (SnOSn) + $\nu_{as}(SnC_n)$ [b]
			540 s 530 s	ν (SnOSn)
519 m	519 s	525 m	512 m	$\nu_s(SnC_n)$ [b]
465 s (br)		480 w		ν (SnN_3) ?
		275 m (br)	800 vw (br)	
345 w (br)				
270 m (br)			220 m	

[a] Absorptions related to ν, δ and ρ modes of CH_3 groups are omitted. [b] $n = 3$ and 2, respectively.

tra gave parameters reproducible to better than ± 0.020 mm·sec^{-1}. The Mössbauer parameters are summarized in Table 1. A typical spectrum is shown in Fig. 1.

IR spectra were taken with Perkin - Elmer model 457 and 225 instruments in the range 4000 - 250 cm^{-1}. Solids were investigated as Nujol mulls, using CsI windows, or in KBr ($\approx$ 0.5%) pellets. Comparison of the mull and KBr spectra showed no evidence for azide - bromide exchange, although the possibility of such a process cannot be conclusively excluded by the available data. Relevant bands are summarized in Table 2.

Raman spectra were obtained at room temperature using a Cary Model 82 instrument employing an Ar laser at 5145 Å. Precision scans were made at speeds of 1 to 2 cm^{-1}·min^{-1} at a slit-width of 2 cm^{-1} and a power level of 20 to 100 mW. The absence of plasma lines in the low frequency region was established by independent scans under experimental conditions. Relevant bands are also summarized in Table 2.

Discussion

A preliminary structural study of $(CH_3)_3SnOH$ has been reported [4], although the full positional parameters have not as yet been discussed in the literature. The monoclinic solid consists of chains of planar trimethyltin groups bridged by oxygen atoms in a linear O—Sn—O array, with the $(CH_3)_3Sn$ planes inclined at about 15° to the long chain axis of the polymer. Each layer has its threefold symmetry axis rotated by 45° from those above and below so that there is an eight layer repeat distance in the unit cell, with $Z = 2$ and 16 for the primitive and superlattice structures. The crystal structure data for the corresponding azide have not been reported, but the extensive earlier Mössbauer, infrared and NMR data have been interpreted on the basis of a linear array of planar $(CH_3)_3SnN_3$ moieties bridged by bidentate azide groups [1]. For a trigonal bipyramidal array of the type $AM(R)_3B$ in which the A and B ligands occupy the polar (z axis) positions and the R groups define the three-fold rotational plane through the metal atom M, the quadrupole splitting (Q.S.) parameter of the M atom can be expressed [8,9] as Q.S. $= \frac{1}{2}e^2|Q|\cdot\{2[A] + 2[B] - 3[R]\}$, where Q is the quadrupole moment of M (-0.05 ± 0.02 b for the 3/2 state of ^{119}Sn), and the terms in square brackets are the contributions to the field gradient of the ligands A, B and R, respectively. From the above, it follows [if the tilt angle [7] of the $(CH_3)_3Sn$ group in the hydroxide is ignored for the time being] that the Q.S. parameter for $[(CH_3)_3Sn]_2(OH)N_3$ should be just the arithmetic mean of the Q.S. for $(CH_3)_3SnN_3$ and $(CH_3)_3SnOH$, since the contribution from the equatorial ligands is presumably the same in each case. This prediction is completely confirmed by experiment since the liquid nitrogen temperature values for the azide [1], hydroxide [10,11], and azide hydroxide are 3.67, 2.94 and 3.25 mm·sec^{-1} respectively, with the latter value in good agreement with the calculated value of 3.30 mm·sec^{-1}. On the basis of this agreement the most probable structure for bis(trimethyltin) hydroxide azide is that of a linear chain polymer in which OH and N_3 moieties bridge adjacent trimethyltin(IV) units.

This suggested alternation of OH and N_3 bridging groups is also supported by the isomer shift (I.S.) data summarized in Table 1. This parameter is related to the electron density at the tin atom nucleus and is commonly referred to that observed in a reference compound, in the present case $BaSnO_3$ [9]. With respect to this standard, the I.S. of $(CH_3)_3SnOH$ is $+1.15$ mm·sec^{-1} [10,11], while that of $(CH_3)_3SnN_3$ has been reported [1] as $+1.43$ mm·sec^{-1}, giving a mean of 1.29 mm·sec^{-1} in good agreement with the value observed for $[(CH_3)_3Sn]_2(OH)N_3$ (Table 1). Again it is assumed in this context that the trimethyltin(IV) moiety in each case makes the same contribution to the total molecular isomer shift.

The vibrational spectra lend further support for the postulated structure of $[(CH_3)_3Sn]_2(OH)N_3$. Significant bands are reported in Table 2. Other IR absorptions, due to modes insensitive to configurational changes, correspond to those observed in $(CH_3)_3SnOH$ [12,13] and $(CH_3)_3SnN_3$ [5], and are not reported in Table 2. Assignments are made in consonance with the results of previous studies [5,11-14].

The vibrational modes involving the N_3 group and its bonds to Sn in $[(CH_3)_3Sn]_2(OH)N_3$ coincide with those observed in $(CH_3)_3SnN_3$ [1,5,14] (IR), which suggests a similar bridging behavior of N_3 in both compounds [1,5]. The modes associated with SnC_3 skeletal vibrations in $[(CH_3)_3Sn]_3\cdot(OH)(N_3)$ show that this group deviates appreciably from a planar configuration, in contrast with its structure in $(CH_3)_3SnOH$ [7,11-13] and the slightly distorted arrangement indicated in $(CH_3)_3SnN_3$ by a weak band at 505 cm^{-1}, attributable to $\nu_s(SnC_3)$ [5]. Bands associated with the OH group (Table 2) are very different from the corresponding bands in $(CH_3)_3SnOH$. The ν(OH) mode, occurring at 3620 cm^{-1} in $(CH_3)_3SnOH$, excludes hydrogen bonding in this compound [11-13], in consonance with X-ray structural studies [7], which show that the shortest interchain distance is 6.67 Å, much too long for such an interaction in the solid at room temperature. In $[(CH_3)_3Sn]_2(OH)N_3$ this mode is broadened and shifted to 3460 cm^{-1}, while the bending mode, δ(OH), increases in energy [920 cm^{-1} in $(CH_3)_3SnOH$] [11-13] and ν(SnO) presumably falls below 250 cm^{-1} [370 cm^{-1} in $(CH_3)_3SnOH$] [12]. These features are fully consistent with the occurrence of hydrogen bonding, which may be envisioned as taking place with the γ-nitrogen of N_3 of an adjacent chain, on the hypothesis that N_3 α-bridges Sn atoms as shown in Fig. 2a. This structure would correspond to that of $[(CH_3)_3Sn]_2(OH)NCO$ [3].

The Mössbauer parameters for $N_3(CH_3)_2Sn[OSn(CH_3)_2]_2N_3$, summarized in Table 1, are particularly useful in aiding the structural assignments for this molecule. The average line width of 1.031 mm·sec^{-1} indicates that except for the resolution of the spectrum into its two quadrupole split components, further hyperfine interactions are negligibly small or absent. Hence, the tin atoms in the molecule all have essentially the same environment and the same coordination geometry. In other words a structure in which both four-coordinate ($\approx T_d$) and six-coordinate (either $\approx$ *cis* or $\approx$ *trans* O_h) tin atoms are present, is clearly ruled out [8,15,16,17]. The most probable structural assignment is one in which the metal atoms occupy the central position in a trigonal bipyramidal arrangement of the ligands, with two of the four azide groups and all four oxygen atoms serving as bridging entities between adjacent tin atoms (Fig. 2b). For such a five-coordinate trigonal bipyramidal structure involving an

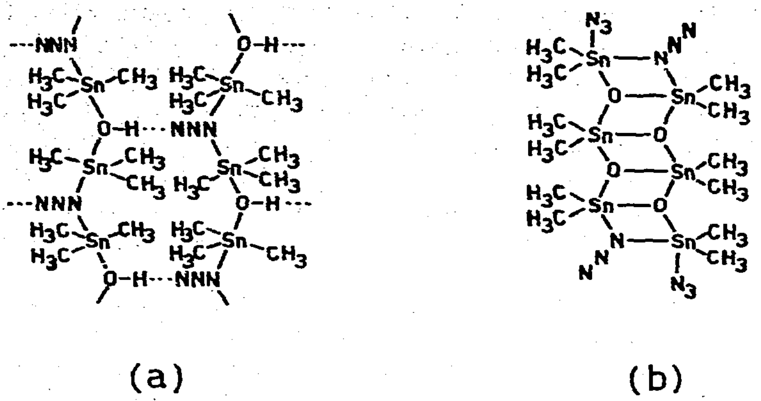

Fig. 2. Proposed structures for the compounds discussed in the text: (a) $[(CH_3)_3Sn]_2(OH)N_3$; (b) $[N_3(CH_3)_2Sn\{OSn(CH_3)_2\}_2N_3]_2$.

SnC_2 fragment, the two organic ligands normally occupy positions in the trigonal plane [18]. A point charge model calculation for such a configuration predicts Q.S. ≈ 2.90 to 3.00 mm·sec^{-1}, although this value is sensitive [8c] to departures of the C—Sn—C bond angle from 120°. In this context it should be noted that in an earlier study [19] of diorganotin(IV) compounds involving tridentate bonding to tin via O, N and S atoms, quadrupole splittings as small as 2.34 mm·sec^{-1} were reported. The experimental values reported in the present study are also in good agreement with the Mössbauer parameters observed for dialkyltin(IV) bis(mercaptoester) complexes of the type $R_2Sn(SCH_2COOC_8C_{17})_2$ in which the metal atom is coordinated to two sulfur and one oxygen atom as well as the two alkyl groups in a trigonal bipyramidal array such as that indicated in Fig. 2b.

The above conclusions are partially at variance with the assumptions by Matsuda et al. [4] on $N_3Bu_2Sn(OSnBu_2)_2N_3$ based on an X-ray study, according to which the dimeric tristannoxane chain would contain four-coordinate as well as five-coordinate tin(IV) atoms (the latter arising from bonds with bridging oxygens). The presence of two coordinatively different types of tin atoms in $N_3(CH_3)_2Sn[OSn(CH_3)_2]_2N_3$ is ruled out by the magnitude of the linewidth parameter extracted from the Mössbauer data as already noted above. Five-coordination for all of the metal atoms, including the terminal tin atom of Matsuda's dimer, can be attained by α-bridging through two of the four azide groups. The near equivalence of the six tin atoms in the dimeric structure is then readily understood in terms of the similarity in the bonding environment around each of the trigonal bipyramidally coordinated metal atoms as indicated in Fig. 2b, in which the major contribution to the field gradient is provided by the difference in the bonding properties of the two alkyl groups and the O (or N_3) ligand in the trigonal plane and the ligands along the orthogonal chain axis. The fact that the metal atom which sees an N_3—Sn—O environment along this bond axis gives essentially the same Mössbauer parameters as one which sees an O—Sn—O array can be rationalized in terms of small departures from 180° bond angles along the axis perpendicular to the alkyl-group-containing plane. These conclusions are further supported — at least qualitatively — by the following considerations. Numerical values of the Mössbauer parameters of

$Cl(C_4H_9)_2Sn[OSn(C_4H_9)_2]_2Cl$, $Cl(C_4H_9)_2Sn[(OSn(C_4H_9)_2)_4]_5Cl$ and a number of related oligomeric organotin oxides [20] have been summarized in a recent review by Smith [16] although the source of some of these data is ascribed to unpublished results by Chapman, Davies and Harrison (ref. 52, cited in ref. 16) and Davies and Smith (ibid, ref. 26), and no full description of the experimental results is referenced. Nevertheless the implication is clear that the two different tin sites in these molecules (e.g. a dibutyltin(IV) fragment bonded to a chlorine and a bridging oxygen atom and the same fragment bonded to two bridging oxygen atoms) are indistinguishable by their Mössbauer hyperfine interactions and hence give rise to what is detected experimentally as a single resonance doublet.

Two X-ray crystal structures of related $R_2Sn(IV)$ distannoxane have recently been reported [21,23]. The dimer $[\{(CH_3)_2SnNCS\}_2O]_2$ has a complex structure in which two tin atoms are pentacoordinated by the two doubly bridging oxygens and by NCS nitrogen, the resulting configuration being of the trigonal bipyramidal type discussed above, although highly distorted from the ideal geometry [21]. The other two tin atoms of these molecules are six-coordinated by one oxygen, two NCS nitrogens (one doubly bridging) and one S from NCS of an adjacent oligomer, forming a very distorted *trans*-$(CH_3)_2$ octahedral configuration [21]. Structures of this type, or perhaps non-polymeric ones containing five-coordinated Sn atoms [22], may be associated with distannoxanes of the type $[(XAlk_2Sn)_2O]_2$, where the X ligands all have well known bridging properties. The Mössbauer parameters for these compounds (X = OH, Cl, Br, NCS, RCOO) are I.S. = 1.25 - 1.54 mm·sec^{-1}, Q.S. = 3.20 - 3.57 mm·sec^{-1} [16,20]. The crystal structure of $[(CH_3)_3SiO(CH_3)_2Sn]_2O_2$ consists of distorted trigonal bipyramidal $(CH_3)_2SnO_3$ units with equatorial CH_3 groups, where one of the Sn—O bonds (that corresponding to inter-chain bridging) is considerably longer (2.8 Å), and thus weaker, than the other two (2.2 Å) [23]. This type of structure may be associated with distannoxanes $[(XR_2Sn)_2O]_2$ [with X = F, $OSi(CH_3)_3$, OC_6H_4Y (Y = H, *p*-Cl, *p*-CH_3, *p*-OCH_3)] which show an I.S. = 1.08 - 1.24 mm·sec^{-1} and a Q.S. = 2.45 - 2.99 mm·sec^{-1} [16], as well as to the polystannoxanes including $[N_3(CH_3)_2Sn\{OSn(CH_3)_2\}_2N_3]_2$, in view of the similarity of their Mössbauer parameters.

Using this structural model it is possible to account for the vibrational spectroscopic data as follows. The (Sn—O—Sn) vibrational mode is associated with the strong Raman active bands at 530 - 540 cm^{-1} and contributes to the infrared band observed at 565 cm^{-1} (see Fig. 3) and perhaps to the IR band at 720 cm^{-1} (Table 2).

In fact dimeric diethyltin(IV) distannoxanes show IR bands assigned to the (Sn—O—Sn) stretching mode in the ranges 532 - 571 and 595 - 638 cm^{-1} [24,13], and triorganotin(IV) distannoxanes exhibit ν(Sn—O—Sn) around 750 cm^{-1} [13]. The bent structure of the SnC_2 moiety is indicated by the presence of the Raman active ν_s- and $\nu_{as}(SnC_2)$ modes at 512 and 572 cm^{-1}. These modes are probably associated with the IR absorptions at 525 and 565 cm^{-1}, respectively (Fig. 3 and Table 2). The N_3 vibrations are as expected [5,13,14]. Moreover it has been observed that terminal and bridging azides are structurally equivalent [25] and the respective $\nu_{as}(N_3)$ modes occur in about the same

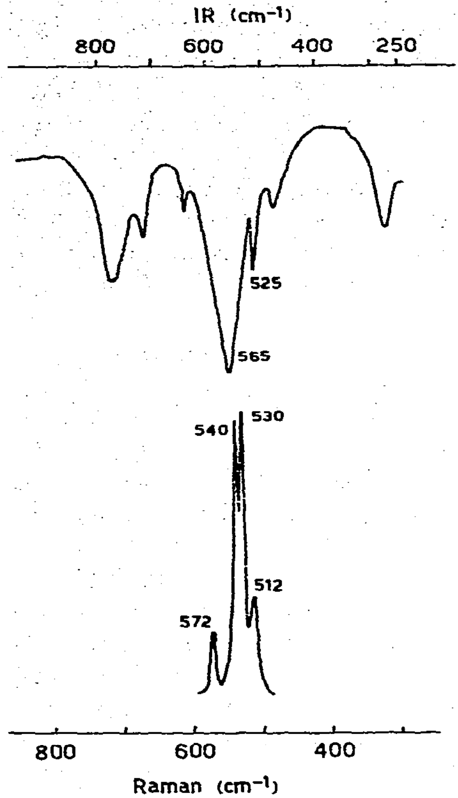

Fig. 3. Portion of the low frequency infrared and Raman spectra of $[N_3(CH_3)_2Sn\{OSn(CH_3)_2\}_2$.

region [26], so that the presence of a single $\nu_{as}(N_3)$ band, as well as of other single bands associated with N_3 vibrations, in $[N_3(CH_3)_2$-$Sn\{OSn(CH_3)_2\}N_3]_2$, is not at variance with the structure proposed in Fig. 2b, where both terminal and bridging azides are present.

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