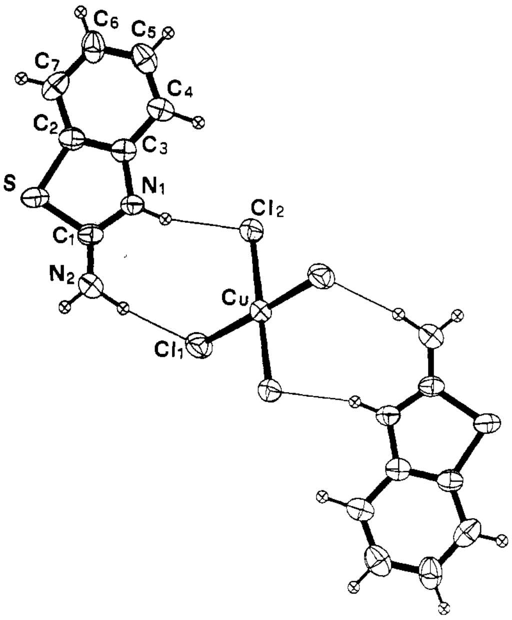

Figure 1. ORTEP view of the complex showing the atom numbering and thermal motion ellipsoids (50%) for the non-hydrogen atoms. The hydrogen atoms are represented as spheres of arbitrary radius.

This is a well-known result for square planar complexes when the ligand field models systematically underestimate the $x^2 - y^2 \rightarrow z^2$ separation. This has been attributed to d–s mixing, or to the residual electron density on the z axis, and it has been found to raise the energy of the $x^2 - y^2 \rightarrow z^2$ transition by 5000 cm^{-1} as in the present case.

The charge transfer bands are observed at 21 700 cm^{-1} as a shoulder, and at 26 100 cm^{-1}, respectively, comparing well with values previously reported, with essentially no change.[30]

EPR Spectrum. The EPR spectrum is axial; $g_{\parallel} = 2.13$, and $g_{\perp} = 2.05$. These values are crystal values, which correspond to averaged molecular values, due to the misalignment of the molecules in the unit cell and the presence of exchange narrowing effects, evidenced by the shape of the EPR line. If we assume axial symmetry for the chromophore, in agreement with the observed geometry of the $CuCl_4^{2-}$ anion, the molecular g values can be calculated and turn out to be $g_{\parallel} = 2.21$ and $g_{\perp} = 2.05$, in good accordance with the values previously reported for square-planar copper chloride complexes.[2] The fact that the two signals of the magnetically non-equivalent sites in the unit cell are averaged, indicates that the intermolecular exchange is larger than 0.02 cm^{-1}.[31]

Acknowledgment. This work has been supported by the financial aid of Consiglio Nazionale delle Ricerche of Italy. We are grateful to Prof. D. Gatteschi for very useful discussions and suggestions, to the "Centro Interdipartimentale di Calcolo Automatico ed Informatica Applicata" of Modena University for computer facilities, and to the "Centro Interdipartimentale Grandi Strumenti" of Modena University for spectra recording and intensity data collecting.

Registry No. $(HL)_2CuCl_4$, 114058-87-6.

Supplementary Material Available: Tables SI–SV, listing thermal parameters, positional and thermal parameters for hydrogen atoms, bond distances and angles involving hydrogen atoms, selected least-squares planes, and interatomic distances, respectively (5 pages); tables of calculated and observed structure factors (4 pages). Ordering information is given on any current masthead page.

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Contribution from the Department of Chemistry
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Tetranuclear Molybdenum Compounds Containing a μ_4-Disulfide Linkage

Joseph E. Pelati, J. C. Huffman,* and R. A. D. Wentworth*

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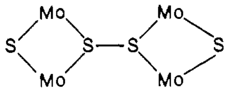
The addition of carboxylic acids[1] to an equilibrium mixture of cubane-like $[Mo(NC_6H_4CH_3)(\mu_3\text{-}S)(S_2P(OC_2H_5)_2)]_4$ and its dinuclear dissociation product[2] yields $[Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)]_2(\mu\text{-}S)(\mu\text{-}SH)(\mu\text{-}O_2CR)$ (R = alkyl). This type of compound is significant because of the bridging sulfhydryl ligand and because it resembles a thiol with moderate reactivity. Alkylation affords *S*-alkyl derivatives,[3] while oxidation with peroxides apparently leads[4] to products with the rare[5] μ_4-disulfide linkage



In this paper, we demonstrate alternate syntheses based on oxidation with Br_2 in CH_2Cl_2, we obtain the molecular structure of $[Mo_2(NC_6H_4CH_3)_2(S_2P(OC_2H_5)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2(\mu_4\text{-}S_2)\cdot 2CH_2Cl_2$ by X-ray methods, and we explore the reduction of the μ_4-disulfide linkage with dithiocarbamate ions.

Results and Discussion

Synthesis. The preparation of $[Mo_2(NC_6H_4CH_3)_2(S_2P(OC_2H_5)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2(\mu_4\text{-}S_2)$ (**1**) was accomplished easily by oxidizing the sulfhydryl-bridged compound with Br_2 in CH_2Cl_2:

$$2\ {>}S\text{—}H + Br_2 \longrightarrow {>}S\text{—}S{<} + 2HBr$$

The acid was scavenged with *N,N*-dimethylaniline. The selection of this base allowed a particularly easy separation of the desired compound from the ammonium salt. In contrast, this was not the case with other bases, such as triethylamine.

When the synthesis of $[Mo_2(NC_6H_4CH_3)_2(S_2CN(i\text{-}C_4H_9)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2(\mu_4\text{-}S_2)$ (**2**) was attempted by this method, an infrared spectrum suggested that considerable hydrolysis of the arylimide ligands had occurred. Presumably, the water necessary for hydrolysis was present as an impurity in the trifluoroacetic acid. The acid-assisted replacement of arylimide groups with oxo ligands has been studied previously.[6] The problem did not occur, however, when the sulfhydryl-bridged compound was replaced with its conjugate base.

Structure. An ORTEP drawing of the molecular structure of **1** is shown in Figure 1. A crystallographically imposed center of inversion is located at the midpoint of the μ_4-S_2 ligand. The drawing does not show the two molecules of CH_2Cl_2 that accompany each molybdenum-containing molecule even though they were located readily. This solvent of crystallization was not found during chemical analysis because it is easily lost and completely removed by vacuum. We are confident that the crystal selected for structural determination is representative of the bulk because it was taken from a sample that was very uniform in appearance.

Distances and angles within the dithiophosphate, trifluoroacetate, and arylimide ligands are unexceptional and do not merit

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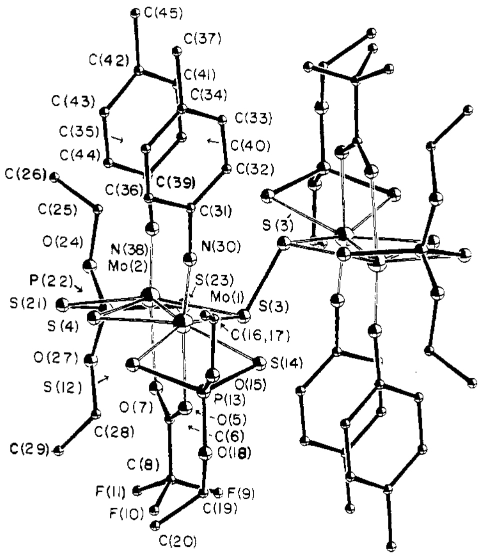

Figure 1. ORTEP drawing of $[Mo_2(NC_6H_4CH_3)_2(S_2P(OC_2H_5)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2(\mu_4\text{-}S_2)$.

Table I. Selected Bond Distances (Å) and Angles (deg)

Mo(1)–Mo(2)	2.867 (1)	S(3)–S(3′)	2.080 (4)
Mo(1)–S(3)	2.457 (2)	Mo(2)–S(3)	2.468 (2)
Mo(1)–S(4)	2.342 (2)	Mo(2)–S(4)	2.354 (2)
Mo(1)–S(12)	2.508 (2)	Mo(2)–S(21)	2.498 (2)
Mo(1)–S(14)	2.550 (3)	Mo(2)–S(23)	2.554 (2)
Mo(1)–N(30)	1.717 (2)	Mo(2)–N(38)	1.717 (7)
Mo(1)–O(5)	2.281 (6)	Mo(2)–O(7)	2.278 (6)
S(3)–Mo(1)–S(4)	106.48 (8)	S(21)–Mo(2)–S(23)	77.70 (8)
S(3)–Mo(2)–S(4)	105.75 (8)	Mo(1)–N(30)–C(31)	168.0 (6)
Mo(1)–S(3)–Mo(2)	71.20 (6)	Mo(2)–N(38)–C(39)	173.2 (6)
Mo(1)–S(4)–Mo(2)	75.26 (7)	Mo(1)–S(3)–S(3′)	106.7 (1)
S(12)–Mo(1)–S(14)	78.23 (8)	Mo(2)–S(3)–S(3′)	110.4 (2)

further comment. Many of the bond distances and angles within and around the $[Mo_2(\mu\text{-}S)]_2(\mu_4\text{-}S_2)$ core can be found in Table I. The S(3)–S(3′) bond length of 2.080 (4) Å is within the range of distances observed for many bridging disulfide ligands.[5] Table II compares some of the similarities and differences in the average distances and angles in this compound and in $[Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)]_2(\mu\text{-}S)(\mu\text{-}SH)(\mu\text{-}O_2CCF_3)$.

As shown in Figure 1, **1** has adopted a configuration that can be described as proximal. This designation refers to the juxtaposition of one sulfur atom from the disulfide bridge with the arylimide ligands on the other end of the molecule. Only one conformer[7] of **1** and **2** can be detected in solution according to our NMR spectra. Both proximal and distal conformers have been observed in solution with other carboxylate ligands, but the abundance of the proximal conformer is always preponderant.[4]

An Aspect of Reactivity. It would seem possible for **2** to be obtained from **1** by a reaction involving an exchange of ligands. However, when **1** was allowed to react with various quantities of $NaS_2CN(i\text{-}C_4H_9)_2$ in tetrahydrofuran, reduction of the disulfide ligand accompanied ligand exchange. The products of each reaction were the thiuram disulfide $[(i\text{-}C_4H_9)_2NCS_2]_2$ and $[Mo(NC_6H_4CH_3)(\mu_3\text{-}S)(S_2CN(i\text{-}C_4H_9)_2)]_4$, a known substance with a cubane-like structure.[8] This reaction resembles the reduction of certain disulfides by thiolates.[9]

Conclusion. The formation of **1** represents a novel ligand oxidation.[10] This is a rare example of a sulfide oxidation in which the metal center is not involved in the oxidation–reduction process and oligomerization occurs through the formation of a $\mu_4\text{-}S_2$ ligand. The structure of **1** is important because it represents yet another structural variation known[11] for a compound containing a Mo_4S_4 core.

Table II. Comparison of Molecular Parameters[a,b] from $[Mo_2(NC_6H_4CH_3)_2(S_2P(OC_2H_5)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2(\mu_4\text{-}S)\cdot 2CH_2Cl_2$ ($\mathbf{1}\cdot 2CH_2Cl_2$) and $[Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)]_2(\mu\text{-}S)(\mu\text{-}SH)(\mu\text{-}O_2CCF_3)$ (**3**)

	$\mathbf{1}\cdot 2CH_2Cl_2$	**3**
Mo–Mo, Å	2.867 (1)	2.839 (2)
Mo–S_b, Å	2.348 (8)	2.348 (9)
Mo–SR (R = S or H), Å	2.462 (8)	2.44 (1)
Mo–S_l(trans to S_b), Å	2.552 (3)	2.540 (4)
Mo–S_l(trans to SR), Å	2.503 (7)	2.509 (4)
Mo–N, Å	1.717 (0)	1.74 (1)
Mo–O, Å	2.279 (2)	2.26 (1)
Mo–S_b–Mo, deg	75.26 (7)	74.4 (1)
Mo–S(R)–Mo, deg	71.20 (6)	71.1 (1)
S_b–Mo–SR, deg	106.1 (5)	107.3 (6)
S_l–Mo–S_l, deg	78.0 (4)	78.4 (3)
Mo–N–C, deg	171 (4)	174 (2)
sum of angles around C(6), deg[c]	360 (3)	361 (3)

[a] Averages are given where required. Standard deviations have been calculated from the differences between individual values and the average value. [b] Abbreviations: S_b = bridging sulfur atom; SR = bridging disulfide or sulfhydryl ligand; S_l = sulfur from a dithiophosphate ligand. [c] This parameter indicates the planarity of the carboxylate ligand. The estimated standard deviation for the sum of the angles has been obtained from standard procedures for the propagation of errors.

Table III. Summary of Crystallographic Data

formula	$Mo_4C_{48}H_{68}N_4O_{12}F_6P_4S_{12}\cdot 2CH_2Cl_2$
mol wt	2069.32
space group	*Pcab*
a, Å	15.343 (4)
b, Å	20.787 (6)
c, Å	25.422 (8)
Z	4
V, Å^3	8108.03
density (calcd), g/cm^3	1.715
cryst size, mm	0.23 × 0.32 × 0.14
cryst color	red
radiation	Mo Kα (λ = 0.71069 Å), graphite monochromator
linear abs coeff, cm^{-1}	11.642
temp, °C	−158
instrument	Picker 4-circle diffractometer locally modified and interfaced
detector aperture	3.0 mm wide × 4.0 mm high, 22.5 cm from cryst
sample to source dist, cm	23.5
takeoff angle, deg	2.0
scan speed, deg/min	4.0
scan width, deg	2.0 + dispersion
bkgd counts, s	6 at each end of scan
2θ range, deg	6–45
total no. of data collected	5938
no. of unique data	5281
no. of unique data with $F > 2.33\sigma(F)$	4147
R(*F*)	0.0560
$R_w(F)$	0.0576
goodness of fit	1.085
largest Δ/σ	0.05

Experimental Section

Reagents and Procedures. All manipulations were performed under a nitrogen atmosphere by using standard vacuum line techniques. Solvents were dried with CaH_2 or P_4O_{10} and distilled under vacuum. The sulfhydryl-bridged compound was prepared as previously described.[2] Other reagents were used as received.

Physical and Analytical Methods. The ^{1}H NMR spectra were recorded with a Nicolet NT 360 spectrometer. The ^{31}P NMR spectra were

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(7) We assume that inversion at the sulfur atoms in the disulfide ligand is not rapid on the NMR time scale in keeping with previous results.[1,3,4]

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Table IV. Fractional Coordinates for $[Mo_2(NC_6H_4CH_3)_2(S_2P(OC_2H_5)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2\cdot 2CH_2Cl_2$[a]

atom	*x*	*y*	*z*
Mo(1)	9685.4 (5)	1258.5 (3)	4516.8 (3)
Mo(2)	9911.0 (5)	1210.4 (3)	5635.6 (3)
S(3)	9472 (1)	302 (1)	5070 (1)
S(4)	9880 (1)	2127 (1)	5092 (1)
O(5)	8251 (4)	1340 (3)	4744 (2)
C(6)	7996 (6)	1335 (4)	5211 (4)
O(7)	8428 (4)	1255 (3)	5622 (2)
C(8)	7007 (6)	1392 (5)	5291 (4)
F(9)	6659 (8)	834 (6)	5318 (9)
F(10)	6780 (8)	1740 (8)	5682 (6)
F(11)	6617 (9)	1685 (9)	4872 (5)
S(12)	9363 (2)	2141 (1)	3873 (1)
P(13)	8840 (2)	1480 (1)	3396 (1)
S(14)	8949 (1)	641 (1)	3773 (1)
O(15)	9246 (5)	1473 (3)	2834 (3)
C(16)	10168 (8)	1303 (6)	2775 (5)
C(17)	10545 (8)	1667 (6)	2334 (5)
O(18)	7880 (4)	1656 (3)	3229 (3)
C(19)	7223 (7)	1764 (6)	3642 (5)
C(20)	6541 (8)	2185 (7)	3426 (5)
S(21)	9796 (2)	2060 (1)	6327 (1)
P(22)	9495 (2)	1380 (1)	6854 (1)
S(23)	9554 (1)	555 (1)	6457 (1)
O(24)	10086 (5)	1403 (3)	7354 (2)
C(25)	11031 (7)	1325 (6)	7310 (4)
C(26)	11446 (9)	1568 (7)	7796 (5)
O(27)	8620 (4)	1498 (3)	7153 (3)
C(28)	7771 (8)	1475 (7)	6887 (6)
C(29)	7355 (9)	2100 (7)	6917 (6)
N(30)	10760 (4)	1124 (3)	4365 (3)
C(31)	11666 (5)	1117 (4)	4318 (3)
C(32)	12105 (6)	598 (5)	4093 (4)
C(33)	13006 (6)	609 (5)	4073 (4)
C(34)	13477 (6)	1118 (5)	4286 (4)
C(35)	13036 (6)	1639 (5)	4510 (4)
C(36)	12139 (6)	1640 (4)	4521 (4)
C(37)	14463 (6)	1111 (6)	4274 (5)
N(38)	11014 (5)	1073 (3)	5661 (3)
C(39)	11927 (5)	1035 (4)	5706 (3)
C(40)	12401 (6)	509 (4)	5554 (3)
C(41)	13298 (6)	530 (4)	5588 (4)
C(42)	13742 (6)	1064 (5)	5774 (4)
C(43)	13237 (6)	1587 (5)	5929 (4)
C(44)	12342 (6)	1588 (4)	5898 (4)
C(45)	14724 (6)	1082 (5)	5794 (5)
Cl(46)	5774 (4)	5169 (2)	1839 (2)
C(47)	6887 (11)	5029 (11)	1947 (7)
Cl(48)	7126 (3)	4768 (2)	2551 (2)
F(9)	6734 (9)	1121 (12)	5745 (6)
F(10)	6555 (9)	1132 (15)	4935 (8)
F(11)	6821 (11)	1978 (7)	5342 (17)

[a] Fractional coordinates are ×10^4.

recorded with a Varian XL 100 spectrometer at 40.5 MHz using ^1H decoupling and an external standard of 85% H_3PO_4. The infrared spectra were recorded with a Perkin-Elmer Model 283 spectrophotometer using KBr pellets of the samples.

$[Mo_2(NC_6H_4CH_3)_2(S_2P(OC_2H_5)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2(\mu_4\text{-}S_2)$. A sample of 1.0 g of the sulfhydryl-bridged compound was dissolved in CH_2Cl_2. To prevent decomposition, 0.10 mL of CF_3CO_2H was added. Next, 0.5 mL of a freshly prepared 1.04 M Br_2 in CH_2Cl_2 solution was added. The solution color changed to a lighter orange. A large excess of *N*,*N*-dimethylaniline was added, causing the formation of a red color. The volume of the solution was reduced, and then a layer of ether was added carefully. Red crystals were produced in excess of a 50% yield and dried under a vacuum. Recrystallization was accomplished by using an ether/CH_2Cl_2 layering procedure. Anal. Calcd for $[Mo_2(NC_6H_4CH_3)_2(S_2P(OC_2H_5)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2(\mu_4\text{-}S_2)$: C, 30.4; H, 3.6; N, 3.0. Found: C, 30.6; H, 3.9; N, 2.8. Selected IR bands (cm^{-1}): 1639 (s), 1467 (m), 1388 (w), 1202 (s), 1153 (s), 1047 (m), 1003 (s), 959 (s), 854 (m), 820 (s), 790 (s). ^1H NMR ($CDCl_3$): δ 6.60 (4 H, q, aryl H), 4.0–4.4 (4 H, m, OCH_2), 2.08 (3 H, s, aryl CH_3), 1.31 and 1.23 (6 H, 2 t, $OCCH_3$). ^{31}P NMR ($CDCl_3$): δ 114.3 (s).

$[Mo_2(NC_6H_4CH_3)_2(S_2CN(i\text{-}C_4H_9)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2(\mu_4\text{-}S)$. After a 10-fold excess of CF_3CO_2H was added to 0.60 mL of 0.19 M NaOH, the solution was evaporated to dryness under a vacuum. A sample of 0.10 g of $[Mo(NC_6H_4CH_3)(\mu_3\text{-}S)(S_2CN(i\text{-}C_4H_9)_2)]_4$ was added to the residue along with 15 mL of tetrahydrofuran. While the mixture was stirred, 0.14 mL of 0.40 M Br_2 was added. The color changed immediately to red-orange. After the mixture was stirred for 2 h, the solvent was evaporated under a vacuum, 20 mL of CH_2Cl_2 was added, and the mixture was filtered. Hexanes (15 mL) were added carefully to produce red-orange crystals in approximately 50% yield. Anal. Calcd for $[Mo_2(NC_6H_4CH_3)_2(S_2CN(i\text{-}C_4H_9)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2(\mu_4\text{-}S_2)$: C, 41.3; H, 5.1; N, 5.7. found: C, 41.2; H, 5.2; N, 5.4. Selected IR bands (cm^{-1}): 1642 (s), 1495 (s), 1254 (s), 1205 (s), 1154 (s), 819 (m), 732 (m), 443 (w), 430 (w). ^1H NMR ($CDCl_3$): δ 6.59 (4 H, q, aryl H), 3.74 (4 H, m, CH_2), 2.2–2.4 (2 H, m, CCH), 2.01 (3 H, s, aryl CH_3), 0.94 (12 H, m, CCH_3).

X-ray Crystallography. Cell dimensions, some of the details for the collection of the data, and final residuals are given in Table III. The methods used for the solution and refinement of the structure are available as supplementary material. Positional parameters for all non-hydrogen atoms can be found in Table IV.

Supplementary Material Available: Details of the solution and refinement of the structure, Tables SI and SII (fractional coordinates of the hydrogen atoms and anisotropic thermal parameters of all heavy atoms), and Tables SIII and SIV (all bond distances and angles) (7 pages); Table SV (F_o and F_c) (11 pages). Ordering information is given on any current masthead page.

Contribution from the Chemistry Department,
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Stereochemistry of a 2,5-Disubstituted 1,4-Diazonia-2,5-diboratacyclohexane

Norman E. Miller

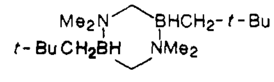
Received August 7, 1987

A small amount of co-subliming impurity was reported[1] to be formed in the synthesis of 1,1-dimethyl-1-azonia-3-boratacyclobutane from reaction of *tert*-butyllithium in pentane/hexane with $Me_3NCH_2BH_2NMe_3^+Cl^-$. This material was at that time recognized as a boron hydride derivative but was unidentified. Characterization of this substance, now, as 2,5-dineopentyl-1,1,4,4-tetramethyl-1,4-diazonia-2,5-diboratacyclohexane (**1**) allows stereochemical features of the parent six-membered ring to be established.

Synthesis, Separation, and Characterization

With several scaled-up syntheses (for a total 2-g yield) of 1,1-dimethyl-1-azonia-3-boratacyclobutane, sufficient material was available for separation and identification of the impurity. Fractional recrystallization from methylene chloride or fractional vaporization gave two isomeric fractions with related mass spectral fragmentation and the same high mass peaks. The less soluble, lower vapor pressure fraction had a higher melting point of 156–158 °C; the more soluble, higher vapor pressure fraction had a much lower melting point of 59–60 °C. High-resolution mass spectra (Table I) show $P - 1$ peaks at m/e 281 and 280 expected for a $C_{16}H_{40}N_2B_2$ parent composition. A nearby intense 211 peak, however, might mean that the higher masses are impurities. Under chemical ionization (with NH_3) conditions (Table I) the 100% peak is m/e 281, and associated high fragments accord with the $C_{16}H_{40}N_2B_2$ parent of electron-ionization spectra. Elemental analyses also support such a composition.

Infrared data and low-field proton NMR data, along with synthesis conditions, would be compatible with six- or eight-membered heterocycles with neopentyl or *tert*-butyl substitution, respectively.



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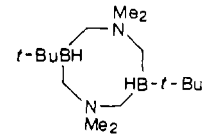

Detailed analysis of mass data and study of temperature dependence of the proton NMR spectrum accord with a six-membered

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