REACTION OF SUPERACID HGeCl₃ WITH AROMATIC COMPOUNDS. 8.* FORMATION OF LONG-LIVED CARBOCATIONS STABILIZED BY THE GeCl₃ ANION IN THE REACTION WITH 1,3-DIALKOXYBENZENES; CRYSTAL STRUCTURE OF 1,1,3,5-TETRAKIS(TRICHLOROGERMYL)-3-ETHOXYCYCLOHEXANE

S. P. Kolesnikov, S. L. Povarov, A. I. Lutsenko, D. S. Yufit, Yu. T. Struchkov, and O. M. Nefedov UDC 542.91:543.422.25:548.737:547.246+ 547.593.21

Cyclohexenyl cations that are long-lived at $-50\,^{\circ}$ C are stabilized by the GeCl₃⁻ anion and are detected and characterized spectrally in reactions of HGeCl₃ with 1,3-dialkoxybenzenes. Further reactions of the cyclohexenyl cations are studied. The x-ray crystal and molecular structure of the final reaction product of HGeCl₃ with 1,3-diethoxybenzene - 1,1,3,5-tetrakis(trichlorogermyl)-3-ethoxycyclohexane - is solved.

Earlier we demonstrated the ability of the superacid HGeCl_3 [2, 3] to add under mild conditions to alkoxybenzenes and other aromatic compounds to form tris- and bistrichloro-germyl derivatives of cyclohexane and cyclohexene [4-6]. This reaction was assumed to proceed with participation of cations. However, direct observation of carbocations in reactions of HGeCl₃ could not be found since the moderately nucleophilic anion GeCl₃⁻ readily recombines with them.

In the present work, we observed for the first time long-lived cyclohexenyl carbocations stabilized by the GeCl₃⁻ anion.* Reaction of 1,3-diethoxybenzene and 1,3-dimethoxybenzene with a 5-6-fold excess of HGeCl₃ at -50°C in CD_2Cl_2 produces 1,3-dialkoxy-5-trichlorogermyl-cyclohexenyl cations (I) that are stable under these conditions for many hours



R = Et (a) and Me (b).

The formation and further reactions of (I) were observed using NMR. Moreover, low-temperature UV and IR spectra were obtained for (Ia) to confirm its structure. As the temperature was raised to -10°C, signals of (I) disappeared in the ¹H and ¹³C NMR and those of the covalent 3,3,5-tris(trichlorogermyl)-1-alkoxycyclohexenes (II) appeared. Signals of (ROH)₂. HGeCl₃, which were then isolated from the reaction mixture as an immiscible layer, appeared simultaneously with those of (II).

Apparently, (II) forms by displacement of trichlorogermyl by the alkoxy groups to form intermediate 1-alkoxy-3,5-bis(trichlorogermyl)cyclohexenyl cations, which at -10° C recombine with the GeCl₃⁻ anion

*For previous communication, see [1].

N. D. Zelinskii Institute of Organic Chemistry, Academy of Sciences of the USSR, Moscow. Translated from Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No. 4, pp. 895-900, April, 1990. Original article submitted March 10, 1989.



Fig. 1. General view of 1,1,3,5-tetrakis-(trichlorogermy1)-3-ethoxycyclohexane.



R = Et (IIa) and Me (IIb).

Displacement of the GeCl₃ by an alkoxy group was observed for the first time in the present work. Occurrence of this reaction is apparently aided by formation of the stable $(ROH)_2$ ·HGeCl₃ complexes that were isolated pure and characterized by ¹H and ¹³C NMR and IR spectra.

The compounds (II) were not isolated pure. According to ¹H and ¹³C NMR spectra, they add another HGeCl₃ molecule on storage for 12 h at 20°C to form tetrakis(trichlorogermyl)-alkoxycyclohexanes (IV)



R = Et (a) and Me (b).

The ¹H and ¹³C NMR and spectral data are insufficient to prove that (IVa) and (IVb) contain geminal GeCl₃ groups. Therefore, an x-ray structural analysis was carried out for (IVa), which was isolated pure. This confirmed its structure as 1,1,3,5-tetrakis(trichlorogermy1)-3ethoxycyclohexane. A general view of (IVa) and the atomic numbering are shown in Fig. 1.

The conformation of the cyclohexane ring in (IVa) is a slightly flattened and asymmetric chair. The angles $C^1C^6C^5$ and $C^2C^3C^4$ are displaced to different sides from the most planar (within 0.005(6) Å) four-atom fragment of the ring $(C^1C^2C^4C^5)$ and form dihedral angles of 44.1 and 52.0°, respectively, with it. The ethoxy group, with the smallest volume among the substituents in (IVa), and the Ge^2Cl_3 trichlorogermyl group are in axial positions. The remaining three GeCl₃ groups are in equatorial positions. Short intramolecular contacts between gem-trichlorogermyl substituents on C¹ and the ethoxy and trichlorogermyl groups on C³ lead to an increase in the lengths of the corresponding Ge-C bonds [1.978(6)-2.003(6) Å] in comparison to the Ge⁴-C⁵ bond, 1.948(6) Å. The small increase in Ge-Cl bond lengths for Cl³, Cl⁵, Cl⁶, and Cl⁸ are also caused by intramolecular interactions of these atoms with other substituents and with ring C atoms. A number of short intermolecular Cl-Cl contacts also occurs in (IVa). The shortest of these, Cl^{12} -Cl³ = 3.492(3) Å, is between atoms related by a translation along the x axis.

Reaction of (IVa) with MeMgBr gives the corresponding trimethylgermyl derivative in only 30% yield. This is apparently due to steric hindrance in geminal bulky trimethylgermyl groups



Compound (V) was isolated as crystals and was characterized by elemental analyses, ${}^{1}H$ and ${}^{13}C$ NMR spectra, and mass spectra. The structures of (VI) and (VII) are assumed on the basis of GCMS data.

EXPERIMENTAL

PMR spectra were recorded on Tesla BS-467 (60 MHz), Bruker WM-250 (250 MHz), and Bruker AM-300 (300 MHz) spectrometers in CDCl₃ and CD₂Cl₂ (TMS internal standard). Recording of ¹³C NMR spectra was done on the Bruker AM-300 (75.47 MHz) in CDCl₃ and CD₂Cl₂ (TMS internal standard). IR spectra were obtained on a Perkin-Elmer 580 B instrument. The UV spectra were obtained on a Specord M40 instrument. We carried out GCMS analysis on a Varian MAT-311 instrument. The m/z values were calculated for isotopes ⁷²Ge and ³⁵Cl. The GLC analysis was performed on a LKhM-8MD chroamtograph with a katharometer (He carrier gas) on 200 × 0.3 cm steel columns with 5% SE-30 on AW inerton (column 1) or 5% XE-60 on N-AW-DMCS chromaton (column 2). Purification of 1,3-diethoxybenzene and 1,3-dimethoxybenzene was done by refluxing over Na and distillation (GLC purity \geq 99.9%). Trichlorogermane was prepared according to [7].

Reaction of 1,3-Diethoxybenzene with HGeCl₃. HGeCl₃ (1.2 g, 6.7 mmoles) was added to a solution of 1,3-diethoxybenzene (0.20 g, 1.2 mmoles) in CD_2Cl_2 at -50°C. Spectra of the reaction mixture were recorded. Only signals of excess HGeCl₃ and the 1,3-diethoxy-5-trichlorogermylcyclohexenyl cation (Ia) were seen 10 min after mixing the reagents. PMR spectrum (-50°C, CD_2Cl_2 , δ , ppm): 1.61 t (6H, 2Me), 3.29 m (4H, 2CH₂), 3.39 m (1H, CH), 4.77 m (4H, 2CH₂O), 6.37 s (1H, CH). ¹³C NMR spectrum (-50°C, CD_2Cl_2 , δ , ppm): 14.0 (2Me), 31.8 (2CH₂), 34.8 (CH), 73.1 (2CH₂O), 99.4 (CH), 198.9 (2C). IR spectrum (-50°C, ν , cm⁻¹): 1530 (the only band in the range 1450-2100). UV spectrum (-50°C): λ_{max} 432 nm. At -50°C, (Ia) is stable for 5-6 h.

On warming the reaction mixture to -10° C, signals of (Ia) disappear and signals of 3,3,5tris(trichlorogermy1)-1-ethoxycyclohex-1-ene (IIa) appear. PMR spectrum (- -10° C, CD₂Cl₂, δ , ppm): 1.40 t (3H, Me), 2.57 m (2H), and 2.90 m (3H) (2CH₂, CH), 3.93 m (CH₂O), 4.84 s (CH). ¹³C NMR spectrum (- 10° C, CD₂Cl₂, δ , ppm): 14.5 (Me), 25.2 (CH₂), 27.2 (CH₂), 35.6 (CH), 56.4 (C), 64.1 (CH₂O), 84.8 (CH), 160.5 (C=). Signals of (EtOH)₂·HGeCl₃ (IIIa) appear simultaneously with (IIa). PMR spectrum (- 10° C, CD₂Cl₂, δ , ppm): 1.43 t (6H, 2Me), 4.12 q (4H, 2CH₂), 9.60 br.s (3H). ¹³C NMR spectrum (- 10° C, CD₂Cl₂, δ , ppm): 15.9 (2Me), 62.3 (2CH₂).

On warming the reaction mixture to 20°C, signals of (IIa) gradually disapper and those of 1,1,3,5-tetrakis(trichlorogermyl)-3-ethoxycyclohexane (IVa) appear. PMR spectrum (CD_2Cl_2 , δ , ppm): 1.41 t (3H, Me), 1.94 t (1H), 2.29 d.d (1H), 2.77 m (2H), 2.90 m (1H), 3.17 t.t (1H, 3CH₂, CH), 3.82 m (2H, CH₂O). ¹³C NMR spectrum (CD_2Cl_2 , δ , ppm): 26.3 (CH₂), 27.8 (CH₂), 34.8 (CH₂), 34.8 (CH), 53.7 (C), 64.5 (CH₂O), 88.0 (C). The reaction is complete after 12 h at 20°C. Signals of only (IIIa) and (IVa) are seen.

Reaction of 1,3-Dimethoxybenzene with HGeCl₃. Spectra of the 1,3-dimethoxy-5-trichlorogermylcyclohexenyl cation (Ib) were obtained analogously at -50°C. PMR spectrum (-50°C, CD₂Cl₂, δ , ppm): 3.33 m (4H, 2CH₂), 3.44 m (1H, CH), 4.52 s (6H, 2MeO), 6.51 s (1H, CH). ¹³C NMR spectrum (-50°C, CD₂Cl₂, δ , ppm): 31.6 (2CH₂), 34.9 (CH), 62.9 (2MeO), 99.1 (CH), 200.2 (2C). Spectra of 3,3,5-tris(trichlorogermyl)-1-methoxycyclohex-1-ene (IIb) were obtained at -10°C. PMR spectrum (-10°C, CD₂Cl₂, δ , ppm): 2.61 m (3H), 2.87 m (1H), 3.01 m (1H and 2CH₂, CH), 3.78 s (3H, MeO), 4.90 s (1H, CH). ¹³C NMR spectrum (-10°C, CD₂Cl₂, δ , ppm): 25.2 (CH₂), 26.9 (CH₂), 35.5 (CH), 55.7 (MeO), 56.2 (C), 84.7 (CH), 161.2 (C). Spectra of (MeOH)₂·HGeCl₃ (IIIb) were also obtained. PMR spectrum (CD₂Cl₂, δ , ppm): 3.76 s (6H, 2MeO), 9.38 br.s (3H). ¹³C NMR spectrum (CD₂Cl₂, δ , ppm): 51.9 (MeO). At 20°C for 12 h, (IIb) reacts with HGeCl₃ to form 1,1,3,5-tetrakis(trichlorogermyl)-3-methoxycyclohexane (IVb). PMR spectrum (CD₂Cl₂, δ , ppm): 2.04 t (1H), 2.39 d.d (1H), 2.88 m (3H), 3.03 m (1H), 3.19 t.t (1H and 3CH₂, CH), 3.64 s (3H, MeO). ¹³C NMR spectrum (CD₂Cl₂, δ , ppm): 26.2 (CH₂), 26.7 (CH₂), 35.2 (CH), 35.4(CH₂), 54.5 (C), 54.9 (MeO), 87.7 (C).

1,1,3,5-Tetrakis(trichlorogermyl)-3-ethoxycyclohexane (IVa). HGeCl₃ (9.0 g, 50 mmoles) was added to 1,3-diethoxybenzene (1.66 g, 10 mmoles) at -10°C. The mixture was gradually

TABLE 1. Bond Lengths d, Å

Bond	d	Bond	d	Bond	d
$\begin{array}{c} Ge^{1}-Cl^{1}\\ Ge^{1}-Cl^{2}\\ Ge^{1}-Cl^{2}\\ Ge^{1}-Cl^{3}\\ Ge^{2}-Cl^{4}\\ Ge^{2}-Cl^{5}\\ Ge^{2}-Cl^{5}\\ Ge^{2}-Cl^{6}\\ Ge^{2}-Cl^{6}\\ Ge^{2}-Cl^{7}\\ \end{array}$	2,110 (2) 2,110 (2) 2,126 (2) 1,980 (6) 2,111 (2) 2,128 (2) 2,128 (2) 1,978 (6) 2,110 (2)	$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	$\begin{array}{c} 2.134 (2) \\ 2.115 (2) \\ 2.003 (6) \\ 2.115 (3) \\ 2.118 (2) \\ 2.108 (3) \\ 1.948 (6) \\ 1.456 (7) \end{array}$	$\begin{array}{c} O-C^{7} \\ C^{1}-C^{2} \\ C^{1}-C^{6} \\ C^{2}-C^{3} \\ C^{3}-C^{4} \\ C^{5}-C^{5} \\ C^{5}-C^{6} \\ C^{7}-C^{8} \end{array}$	1,448 (8) 1,549 (8) 1,562 (8) 1,514 (8) 1,523 (8) 1,533 (8) 1,529 (8) 1,47 (1)

TABLE 2. Bond Angles ω , deg

Angle	ω	Angle	ω	Angle	ω
Cl ¹ Ge ¹ Cl ² Cl ¹ Ge ¹ Cl ² Cl ² Ge ¹ Cl ³ Cl ² Ge ¹ Cl ³ Cl ² Ge ¹ Cl ³ Cl ³ Ge ¹ C ¹ Cl ⁴ Ge ² Cl ⁵ Cl ⁴ Ge ² Cl ⁶ Cl ⁴ Ge ² Cl ⁶ Cl ⁵ Ge ² C ³ Cl ⁷ Ge ³ Cl ⁸ Cl ⁷ Ge ³ Cl ⁸	$\begin{array}{c} 110,83(9)\\ 107,17(8)\\ 112,1(2)\\ 104,49(9)\\ 114,7(2)\\ 106,8(2)\\ 110,05(8)\\ 104,75(8)\\ 104,75(8)\\ 104,75(8)\\ 114,4(2)\\ 103,20(9)\\ 114,2(2)\\ 109,24(2)\\ 109,24(2)\\ 109,24(8)\\ 109,44(8)\\ 110,8(2)\\ \end{array}$	Cl ⁸ Ge ³ Cl ⁹ Cl ⁶ Ge ³ C ³ Cl ⁹ Ge ³ C ³ Cl ¹⁰ Ge ⁴ Cl ¹¹ Cl ¹⁰ Ge ⁴ Cl ¹² Cl ¹⁰ Ge ⁴ C ⁵ Cl ¹¹ Ge ⁴ C ⁵ Cl ¹¹ Ge ⁴ C ⁵ Cl ¹² Ge ⁴ C ⁵ Cl ¹² Ge ⁴ C ⁵ C ³ OC ⁷ Ge ¹ Cl ² Ge ² Ge ⁴ Cl ² C ² Ge ⁴ Cl ² C ² Ge ² Cl ² C ⁸	$\begin{array}{c} 103,94(8)\\ 108,7(2)\\ 115,0(2)\\ 108,07(9)\\ 108,04(9)\\ 108,6(2)\\ 107,48(9)\\ 111,1(2)\\ 113,4(2)\\ 113,4(2)\\ 114,9(4)\\ 109,8(3)\\ 105,2(4)\\ 106,4(4)\\ 111,5(4)\\ 111,5(4)\\ 112,5(4)\end{array}$	C ² C ¹ C ⁶ C ¹ C ² C ³ Ge ³ C ³ C ² Ge ³ C ³ C ⁴ OC ³ C ² OC ³ C ⁴ C ² C ³ C ⁴ C ² C ³ C ⁴ C ³ C ⁴ C ⁵ C ⁵ Ge ⁴ C ⁵ C ⁵ Ge ⁴ C ⁵ C ⁶ C ⁴ C ⁵ C ⁷ C ³ C ⁷ C ³	$\begin{array}{c} 111,0 (5)\\ 112,3 (5)\\ 111,0 (4)\\ 106,7 (4)\\ 108,1 (4)\\ 104,9 (4)\\ 114,4 (4)\\ 111,5 (5)\\ 110,5 (5)\\ 100,9 (4)\\ 109,1 (4)\\ 113,2 (5)\\ 113,2 (5)\\ 105,7 (6)\\ \end{array}$

TABLE 3. Coordinates (×10⁴, ×10⁵ for Ge) of Nonhydrogen Atoms

Atom	x	Y	z	Atom	X	Y	z
Ge ¹ Ge ² Ge ³ Cl ¹ Cl ² Cl ³ Cl ⁴ Cl ⁵ Cl ⁶ Cl ⁷ Cl ⁸ Cl ⁹	$\begin{array}{c} 7869(10)\\ 32381(9)\\ -21436(8)\\ 30268(9)\\ 48(3)\\ 2541(3)\\ -1421(3)\\ 2160(2)\\ 5234(2)\\ 4576(3)\\ -2857(3)\\ -4015(2)\\ -2340(2) \end{array}$	$\begin{array}{c} -13075 (6) \\ -2274 (7) \\ 41945 (6) \\ 27689 (7) \\ -2252 (2) \\ -2796 (2) \\ -533 (2) \\ 47 (2) \\ 701 (2) \\ -2385 (2) \\ 3902 (2) \\ 3861 (2) \\ 6304 (2) \end{array}$	26271 (4) 36330 (4) 28335 (4) 5173 (4) 3794 (1) 2074 (1) 1810 (1) 3391 (1) 3391 (1) 3391 (1) 3391 (1) 2104 (1) 2104 (1)	Cl ¹⁰ Cl ¹¹ Cl ¹² O C ¹ C ² C ³ C ⁴ C ⁵ C ⁶ C ⁷ C ⁸	$\begin{array}{c} 1318 (3) \\ 2801 (3) \\ 5545 (3) \\ 1347 (5) \\ 1540 (8) \\ -113 (7) \\ 146 (7) \\ 746 (7) \\ 2417 (7) \\ 2297 (8) \\ 1823 (9) \\ 2461 (12) \end{array}$	2405 (2) 4906 (2) 1663 (3) 2710 (4) 301 (5) 1435 (5) 2846 (5) 3294 (5) 2225 (6) 753 (6) 3942 (6) 3781 (8)	-274(1) 192(1) 269(1) 3379(3) 2696(3) 2840(3) 2710(3) 1834(3) 1672(3) 1832(4) 3370(4) 4222(5)

TABLE 4. Coordinates $(\times 10^3)$ of Hydrogen Atoms

Atom	X	Y	z	Atom		Y	Z
H ^{2.1}	-107 (7)	149(5)	246 (3)	H ^{6.2}	346 (8)	3(6)	172 (3)
H ^{2.2}	-47 (6)	123(5)	336 (3)	H ^{7.1}	317 (10)	349(7)	296 (5)
H ^{4.1}	-6 (7)	336(5)	150 (3)	H ^{7.2}	71 (12)	481(9)	327 (5)
H ^{4.2}	81 (7)	422(5)	170 (3)	H ^{8.1}	314 (11)	288(8)	448 (5)
H ⁵	329 (7)	231(5)	206 (3)	H ^{8.2}	128 (7)	404(5)	440 (3)
H ^{6.1}	136 (8)	75(5)	136 (3)	H ^{8.3}	282 (8)	424(6)	430 (4)

heated to 20°C. The reaction mixture layered after 2 h. The upper layer $[(EtOH)_2 \cdot HGeCl_3]$ was separated. For PMR and ¹³C NMR spectra, see above. IR spectrum (thin layer, v, cm⁻¹): 850, 1015, 1390, 1445, 2905, 2960, and 2985.

Crystals (IVa) formed in the lower layer after 12 h. These were separated, washed with pentane cooled to -10°C, and dried in vacuum. Pure (IVa) (6.55 g) was obtained; yield 78% based on 1,3-diethoxybenzene, mp 132-134°C (from $CHCl_3$). According to PMR data, the total yield of (IVa) was 93% based on 1,3-diethoxybenzene. For the PMR and ¹³C NMR spectra, see above. Found: C 11.30; H 1.46; C1 50.73; Ge 34.43%. $C_8H_{12}Cl_{12}Ge_4O$. Calculated : C 11.42; H 1.43; Cl 50.69; Ge 34.56%.

<u>Reaction of (IVa) with MeMgBr</u>. A solution of (IVa) (4.2 g, 5 mmoles) in 100 ml absolute ether was treated with MeMgBr (0.1 mole) in ether. A yellow oil (2.77 g) was isolated after the usual workup. Vacuum distillation gave a number of fractions. Pure 1,1,3,5-tetrakis-(trimethylgermyl)-3-ethoxycyclohexane (V) (0.29 g) was obtained form the fraction with bp 120-140°C (0.1 mm) by chromatography on a silica gel column as white crystals with mp 78°C (from ethanol). According to GLC, the total yield of (V) was 30% based on (IVa). PMR spectrum (CDCl₃, δ , ppm): 0.09 s (9H, GeMe₃), 0.11 s (9H. GeMe₃), 0.18 s (9H, GeMe₃), 0.20 s (9H, GeMe₃), 1.19 t (3H, Me), 1.33 m (2H), 1.48 t (1H), 1.77 (2H), 2.12 m (2H, 3CH₂, CH), 3.30 m (2H, CH₂O). ¹³C NMR spectrum (CDCl₃, δ , ppm): -4.55 (GeMe₃), -2.44 (2GeMe₃), -1.43 (GeMe₃), 15.8 (Me), 18.8 (CH), 19.1 (C), 31.0 (CH₂), 31.5 (CH₂), 37.4 (CH₂), 59.9 (CH₂O), 75.7 (C). Found: C 40.25; H 7.99; Ge 48.98%. C₂₀H₄₈Ge₄O. Calculated: C 40.38; H 8.07; Ge 48.86%. Mass spectrum: [M]⁺ is absent; 475 [Me-GeMe₃]⁺, 415 [M-GeMe₃-OEt-Me]⁺, 358 [M-2GeMe₃]⁺.

According to GCMS, 3,3,5-tris(trimethylgermyl)cyclohexanone (VI), yield 19% based on (IVa), mass spectrum: 466 [M]⁺, 431 [M-Me]⁺, 331 [M-GeMe₃]⁺; and 1,3,5-tris(trimethylgermyl)-1-dimethylchlorogermyl-3-ethoxycyclohexane (VII), yield 9% based on (IVa), mass spectrum: [M]⁺ is absent; 495 [M-GeMe₃]⁺, 298 [M-2GeMe₃-EtO-C1]⁺.

<u>X-Ray Structural Study</u>. Crystals of $C_8H_{12}Cl_{12}Ge_40$ are triclinic, at -120°C a = 8.203(2), b = 10.402(2), c = 16.419(3) Å, α = 77.16(1), β = 87.96(2), γ = 72.03(2)°, Z = 2, space group P1. Unit-cell constants and intensities of 3979 independent reflections with I $\geq 2\sigma$ were measured on a Syntex P2₁ automatic 4-circle diffractometer at -120°C (λ MoK_{α}, graphite monochromator, $\theta/2\theta$ scanning, $\theta \leq 30^\circ$).

The structure was solved by direct methods and refined using anisotropic full matrix least squares for all nonhydrogen atoms. Absorption ($\mu = 45.6 \text{ cm}^{-1}$) was calculated using the program DIFABS [8]. All hydrogen atoms were found in a difference synthesis and refined isotropically. The final agreement factors were R = 0.042 and R_w = 0.044. All calculations were performed using INEXTL programs [9] on an Eclipse S/200 computer.

Bond lengths and angles are given in Tables 1 and 2. Coordinates of nonhydrogen atoms are in Table 3, those of H atoms are in Table 4. Equivalent isotropic thermal factors can be obtained from the authors.

LITERATURE CITED

- S. P. Kolesnikov, S. L. Povarov, A. I. Lutsenko, and O. M. Nefedov, Izv. Akad. Nauk SSSR, Ser. Khim., No. 12, 2874 (1987).
- S. P. Kolesnikov, I. V. Lyudovskaya, and O. M. Nefedov, Izv. Akad. Nauk SSSR, Ser. Khim., 1127 (1984).
- 3. V. B. Kazanskii, O. M. Nefedov, A. A. Pankov, et al., Izv. Akad. Nauk SSSR, 698 (1983).
- 4. S. P. Kolesnikov, O. M. Nefedov, and V. I. Sheichenko, Izv. Akad. Nauk SSSR, Ser. Khim., 443 (1966).
- 5. S. P. Kolesnikov, S. L. Povarov, and O. M. Nefedov, Izv. Akad. Nauk SSSR, Ser. Khim., 666 (1988).
- 6. S. P. Kolesnikov, S. L. Povarov, V. V. Samoshin, and A. I. Lutsenko, Izv. Akad. Nauk SSSR, Ser. Khim., 2826 (1987).
- 7. A. D. Petrov, V. K. Mironov, and N. G. Dzhurinskaya, Dokl. Akad. Nauk SSSR, <u>128</u>, 302 (1959).
- 8. N. Walker and D. Stuart, Acta Crystallogr., Sec. A: Found. Crystallogr., <u>39</u>, 158 (1983).
- 9. R. G. Gerr, A. I. Yanovskii, and Yu. T. Struchkov, Kristallografiya, <u>28</u>, No. 5, 1029 (1983).