The narrow N(1)–Al–N(2) angle, 91.3 (1)°, may have its origin in the 2p–3d $\pi$ interaction or it may be a consequence of a slight ring strain. However, the latter is not of sufficient magnitude to affect the Al–N bond lengths. All other distances and angles are within expected ranges.

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**Registry No.** $Cl_2AlN(C_2H_5)(C_2H_4N(CH_3)_2)$, 59738-07-7.

**Supplementary Material Available:** A listing of structure factor amplitudes and a stereoscopic view of the unit cell packing (11 pages). Ordering information is given on any current masthead page.

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# Synthesis and Characterization of Germanium(II) Dialkoxides

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An understanding of structure and bonding in compounds of the main group elements has evolved with advances in synthetic abilities. For example, exploration of the nonaqueous chemistry of tin(II) compounds in the past few years has been especially fruitful owing to the pioneering efforts and preparative techniques of Morrison and Haendler[1] and others.[2–4] The expansion of germanium(II) chemistry has been noticeably slower. The transient existence of a number of bivalent germanium(II) intermediates has been deduced from sealed tube experiments in which the intermediate is generated and trapped by oxidative addition reactions.[5,6] Relatively few compounds have been fully characterized either because of difficulties in isolating pure substances or because of their thermal, oxidative, or hydrolytic instability. The lag in the development of germanium(II) chemistry is due, in part, to a lack of stable, easily available intermediates and a paucity of information on the physical and spectroscopic properties of these compounds. Thus, the area of bivalent germanium chemistry represents a frontier in the development of an understanding of the behavior of the main group 4 elements. In the present study we wish to report the synthesis and properties of germanium(II) diethoxide which is stable in coordinating solvents. Additionally, we have isolated and characterized a diethanolate complex of germanium(II) diethoxide.

## Experimental Section

$GeI_2$ was synthesized from sublimed $GeI_4$ by a method described elsewhere.[7] Alcohol was dried over magnesium metal and distilled from magnesium alkoxide under dry nitrogen immediately before use. All other solvents were dried over either sodium metal or phosphorus pentoxide and freshly distilled prior to use. All manipulations were carried out in a glovebag flushed with nitrogen which was predried by passage through several columns of $P_2O_5$ and deoxygenated through several columns of reduced BTS catalyst (BASF Colors and Chemicals, Inc.).

**Table I.** Proton NMR Absorptions of Germanium(II) Dialkoxides

| | MHz spectrum | temp, °C | $\delta^a$ | |
|---|---|---|---|---|
| | | | O-$CH_2$ | -$CH_3$ |
| $Ge(OEt)_2\cdot xEtOH$ (A) | $60^b$ | 30 | 3.95 q | 1.30 t |
| | $60^b$ | −37 | 3.95 q | 1.15 t |
| $Ge(OEt)_2{}^d$ (B) | $220^c$ | 18 | 3.95 q, 4.0 br | 1.21 t, 1.29 br |
| | | 94 | 4.13 q | 1.39 t |

[a] Relative to $Me_4Si$: $J = 7$ Hz for all resolved multiplets; t, triplet; q, quartet; br, broad and unresolved. [b] In $C_6H_5Cl$. [c] Desolvated under vacuum at ambient temperature for 24 h. [d] In $C_6D_6$ run with FT mode.

**Table II.** $^{74}Ge$ Peaks for the Principal Fragment Ion Clusters of the Mass Spectrum of $Ge(OEt)_2\cdot 2EtOH$ at 80 eV and 90 °C

| *m/e* | % | ion |
|---|---|---|
| 255 | 59 | $[M - H]^+$ |
| 210 | 6 | $[M - EtOH]^{+\cdot}$ |
| $209^a$ | 14 | $[M - H, EtOH]^+$ |
| 165 | 7 | $[M - EtO, EtOH]^+$ |
| $164^a$ | 11 | $[M - 2EtOH]^{+\cdot}$ |
| 149 | 30 | $[M - Me, 2EtOH]^+$ |
| 135 | 15 | $[M - Et, 2EtOH]^+$ |
| 120 | 17 | $[M - 2EtO, EtOH]^+$ |
| $119^a$ | 96 | $[M - EtO, 2EtOH]^+$ |
| 91 | 81 | $[GeOH]^+$ |

[a] Corrected for overlapping germanium clusters.

IR spectra were obtained with a Perkin-Elmer 521 double beam spectrophotometer as Nujol and fluorolube mulls between KBr plates. The spectra were calibrated with polystyrene.

Proton NMR spectra were recorded on dilute solutions with a Varian A-60 or HR-220 (FT mode) spectrometer equipped with a variable-temperature probe. All chemical shifts were measured relative to $Me_4Si$ as an internal reference.

Mass spectral fragmentation data were recorded by using a Perkin-Elmer Hitachi RMU-6 mass spectrometer by direct injection of solids into the ionization beam. Thermogravimetric analysis (TGA) was carried out on a DuPont 900 thermal analyzer.

Elemental analyses were carried out by Schwarzkopf Microanalytical Laboratories, Woodside, N.Y.

**Preparation of $Ge(OEt)_2$ Ethanolate Complexes.** A 5-g sample of freshly prepared $GeI_2$ is added to 300 mL of EtOH containing a 3% excess of freshly prepared NaOEt. The yellow diiodide dissolves with loss of color and evolution of heat. The solution is filtered and the solvent is removed under vacuum at 40 °C. The white solid residue is kept under vacuum for 1 h (*extended periods of vacuum storage must be avoided in order to preserve the desired solubility properties*). The product (A) is a free flowing, white powder (2 g) which is soluble in benzene. The byproduct, NaI, is removed by filtration. Table I summarizes the 60-MHz proton NMR spectrum of A at 30 and −37 °C. The absorptions of A move upfield with addition of alcohol. If A is stored under vacuum, it gradually loses solubility in hydrocarbon solvents. The loss of solubility is initially reversible by addition of alcohol but with prolonged vacuum storage the reversibility is lost. The FT 220-MHz spectrum in benzene-$d_6$ at 18 and 94 °C of this material (B) after vacuum storage for 1 day is reported in Table I. Anal. H/C calcd for $Ge(OEt_2)$, 2.50; found, 2.61.

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[†] In partial fulfillment of the requirements for the Ph.D. Degree in Chemistry at the Polytechnic Institute of New York.

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Table III. Proton Chemical Shifts for $CH_3Ge(OEt)_nI_{3-n}$ at 220 MHz and 20 °C in $CDCl_3$

| | $\delta^a$ | | |
|---|---|---|---|
| compd | -O-$CH_2$ | -C-$CH_3$ | Ge-$CH_3$[b] |
| $CH_3Ge(OCH_2CH_3)_3$ | 3.81 q | 1.26 t[c] | 0.72 s |
| $CH_3Ge(OCH_2CH_3)_2I$ | 3.85 q | 1.26 t[c] | 1.26 s[c] |
| $CH_3Ge(OCH_2CH_3)I_2$ | 3.88 q | 1.30 t | 1.95 s |
| $CH_3GeI_3$ | | | 2.80 s |

[a] Relative to internal $Me_4Si$; s, singlet; t, triplet; q, quartet; all *J* values = 7 Hz. [b] For $CH_3Ge(OCH_3)_nI_{3-n}$ $\delta(CH_3\text{-}Ge)$ (in benzene): $n = 0$, 2.225; $n = 1$, 1.475; $n = 2$, 0.842; $n = 3$, 0.358 at 60 MHz.[16] [c] Resolvable at 220 MHz.

Addition of a dilute solution of water in alcohol to A causes precipitation of a cream-colored solid identified as $Ge(OH)_2$ by its aqueous solubility properties, reducing ability, and infrared spectrum.[8] The hydrolysis products of A in acid contain ethanol by the iodoform test and reduce mercuric chloride.

A sample of A (2 g), which was dried briefly, is taken up in 50 mL of pentane. Any undissolved material is removed by filtration. *No wash should be used* (see Results and Discussion). The solution is immersed in a dry ice–acetone bath (−76 °C). After several minutes a white solid (C) precipitates. Under optimum conditions, 50 mg of solid may be collected by low-temperature filtration. The TGA weight loss is 38%, calcd for 2EtOH, 36%. EtOH is the only volatile decomposition product as confirmed by the infrared spectrum of trapped gases. The mass spectrum of C is summarized in Table II. C can be sublimed at 90 °C and $10^{-6}$ torr with considerable reduction in yield. The mass spectrum of the sublimed white solid is identical with that of C. IR ($cm^{-1}$): 2985 (s), 2920 (w, br), 2885 (m, br), 2720 (w), 1490 (w), 1455 (s), 1386 (s), 1200 (w, br), 1155 (m), 1097 (s), 1050 (s), 910 (m), 887 (s), 635 (m, br), 585 (s, br). Anal. H/C calcd for $Ge(OEt)_2{\cdot}2EtOH$, 2.75; for $Ge(OEt)_2$, 2.50; found for C, 2.80; for C vacuum dried 18 h, 2.57.

**Reaction of A with MeI.** A fivefold excess of MeI is added to a toluene solution of 2 g of A. The mixture is refluxed under dry nitrogen overnight. The solution is filtered and solvents are removed under vacuum. The last 1–2 mL of liquid contains an equilibrium mixture of products which can be collected under vacuum as a pale yellow liquid by fractional condensation through a −23 °C trap. The FT 220-MHz NMR spectrum of these products in $CDCl_3$ is summarized in Table III. Hydrolysis of the products in dilute $HNO_3$ produces iodine, which can be extracted into $CCl_4$ forming a purple solution. Additionally the hydrolysate contains ethanol identified by the iodoform test and does not reduce mercuric chloride.

## Results and Discussions

The reaction of germanium(II) diiodide with sodium ethoxide was used to prepare the ethanol compexed germanium(II) diethoxide (A) (eq 1). A spontaneously degrades to an

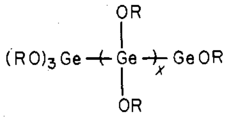
$$GeI_2 + 2NaOEt + xEtOH \rightarrow \underset{A}{Ge(OEt)_2{\cdot}xEtOH} + 2NaI \qquad (1)$$

insoluble, intractable material when stored under vacuum for several days. Product association and loss of solubility with loss of complexed ethanol appear to be the first steps in the degradation process. A salient example of the sensitivity of the solubility of A to alcohol concentrations is the conversion of a saturated pentane solution of A to a colloidal dispersion upon dilution with dry pentane. Solids redissolve with addition of a few drops of ethanol. These results have been interpreted in terms of a shift in equilibrium between A and less soluble, presumably associated, desolvated species.

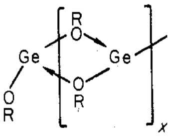
The proton NMR spectrum of A in chlorobenzene (Table I) exhibits a methylene quartet at 3.95 ppm and methyl triplet at 1.30 ppm which are shifted downfield from the corresponding absorptions of ethanol (3.66 (q), 1.15 (t)). As expected by inductive effects, the shift is greatest for the protons nearest the metal with the direction and magnitude of the shifts being similar to those observed for tin(II) diethoxide.[9]

The spectrum of A is temperature dependent, exhibiting clearly resolved multiplets at 30 °C and considerably broader lines at −37 °C. This effect is consistent with exchange of ethanol with the ethoxide groups on germanium. Addition of small increments of ethanol to the sample causes the absorptions to sharpen and move upfield indicating rapid exchange of all species.

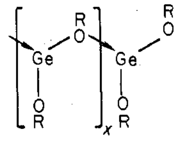
The 60-MHz proton NMR spectrum of B, which has been prepared by vacuum desolvation of A for 24 h, has two weak, broad signals in the ethoxy region. The FT NMR at 220 MHz of B at 19 °C exhibits a sharp quartet at 3.95 ppm and triplet at 1.21 ppm superimposed on the upfield sides of the broad signals (Table I). That these lines do not represent contamination by monomer is supported by the lack of exchange upon addition of ethanol. The spectrum can be interpreted in terms of an oligomeric structure (I, II, or III) with broad absorptions due to multiple environments of the ethoxide groups. Structure I was proposed by Satgé for aged solutions of $Ge(OMe)_2$ and $Ge(SMe)_2$ where evidence for catenation was obtained by isolation of Ge–Ge bonded derivatives with trapping reagents.[6] Bridging structures II and III were proposed for Sn(II) dialkoxides on the basis of NMR spectra;[9] however, Sn–Sn bonding is precluded by Mössbauer data.[10] Since the upfield resolved peaks in B represent a unique ethoxide environment (e.g., terminal ethoxides) by analogy to $Sn(OEt)_2$,[9] either II or III is preferred. Each broad absorption coalesces to a considerably sharper multiplet at 94 °C indicating exchange of bridge and terminal ethoxides on the 220-MHz time scale at the higher temperature.



I



II



III

Thermogravimetric analysis of C, which is precipitated from a pentane solution of A at −76 °C, indicates a 38% endothermic weight loss which corresponds to evolution of 2 mol of ethanol/mol of C. The only volatile product from thermal degradation is ethanol.

Oxidative and hydrolytic instability and ethanol evolution made reproducible elemental analysis difficult to obtain.[1,6] However, a H:C ratio of 2.5–2.6, which is in agreement with a diethoxide composition, was found for samples that have been dried under vacuum. A larger ratio (2.8) was determined for C and is consistent with the diethanolate formulation.

Attempts to purify C by vacuum sublimation have been frustrated by simultaneous dealcoholation. Thus, no sublimate is observed when C is heated slowly under vacuum to 90 °C; however, if a freshly prepared sample of C is immersed in a preheated bath at 90 °C, a small amount of white solid sublimes. The mass spectrum of C is identical with that of the sublimate.

Salient features of the mass spectrum of C (Table II) are the peaks at *m/e* 255, 210, and 164 which can be ascribed to the ions $[^{74}Ge(OEt)_2{\cdot}2EtOH) - H]^+$, $[^{74}Ge(OEt)_2{\cdot}EtOH]^{+\cdot}$, and $[^{74}Ge(OEt)_2]^{+\cdot}$, respectively.

Prominent features of the infrared spectrum of C are the characteristic CH, CO, and CCO modes found also in ethanol and the bands at 635 and 585 $cm^{-1}$ which have been tentatively assigned to GeO stretching modes since they appear in a similar spectral region to that of $Sn(OEt)_2$.[9] The lack of an observable OH stretching vibration is not unexpected insofar

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as a similar observation has been reported for other ethanolate complexes (e.g., mono- and dialcoholate of $BF_3$).[11]

A undergoes oxidative addition reactions with organohalides. Similar reactions have been reported for tin(II) dihalides,[12] for dialkyl and diamido compounds,[13,14] and for some Ge(II) compounds.[15] Generally organometallic(IV) derivatives are obtained. In the case of the reaction of A with MeI, the expected initial product is $MeGe(OEt)_2I$ (eq 2). Since alkoxy

$$Ge(OEt)_2{\cdot}xEtOH + MeI \rightarrow MeGe(OEt)_2I + xEtOH \quad (2)$$

and iodo substituents on germanium readily redistribute at ambient temperatures, at least four methyl germanium compounds are anticipated at equilibrium (eq 3 and 4). The

$$2MeGe(OEt)_2I \overset{K_1}{\rightleftharpoons} MeGe(OEt)_3 + MeGe(OEt)I_2 \quad (3)$$

$$2MeGe(OEt)I_2 \overset{K_2}{\rightleftharpoons} MeGeI_3 + MeGe(OEt)_2I \quad (4)$$

proton NMR data (Table III) of the product mixture indicate four distinct Me–Ge singlets corresponding to each of the species in the equilibrium distribution. Assignments were made on the basis of unique methyl on germanium to methyl on ethoxy integrated areas and on comparable shifts for $MeGe(OMe)_nI_{3-n}$, which have been reported by Moedritzer et al.[16] Furthermore, equilibrium constants ($K_1 = 0.1$; $K_2 = 0.2$) can be estimated from area ratio data and are in reasonable agreement to those values reported for the methoxy equilibrium system (vis., $K'_1 = 0.152$, $K'_2 = 0.106$).

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**Registry No.** $Ge(OEt)_2$, 71872-05-4; $Ge(OEt)_2{\cdot}2EtOH$, 71872-04-3; $GeI_2$, 13573-08-5; NaOEt, 141-52-6; $CH_3Ge(OCH_2CH_3)_3$, 5865-91-8; $CH_3Ge(OCH_2CH_3)_2I$, 71872-06-5; $CH_3Ge(OCH_2CH_3)I_2$, 71872-07-6; $CH_3GeI_3$, 1111-91-7; MeI, 74-88-4.

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## Synthesis and Characterization of a New Class of Bivalent Germanium Compounds

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There has been considerable interest in the development of the chemistry of group 4 elements in the second oxidation state. Compounds of Sn(II) and Ge(II) have been viewed as electronic and structural analogues to singlet carbenes and silenes. These electron-deficient compounds have an extensive and varied chemistry owing to their capacity to act as acceptor species toward Lewis bases,[1–4] as donor ligands in transition metal complexes,[5,6] as precursors to new heterocyclic compounds,[7,8] and as intermediates in a variety of oxidative addition reactions.[9,10]

Two approaches have been used to elucidate Sn(II) and Ge(II) chemistry. One is the generation of bifunctional intermediates, generally by α-elimination in the presence of a trapping reagent.[11] The existence of the M(II) intermediate is deduced from the isolation and characterization of the M(IV) species. The second approach is to isolate and characterize an M(II) species which has been stabilized though steric or electronic effects. Although the latter approach has been successful for Sn(II) compounds,[12,13] it has been less fruitfully applied to Ge(II) compounds which appear to be more labile.[14]

We now wish to report the synthesis of a new class of Ge(II) compounds which are heterocyclic derivatives of the dialkoxides and are stabilized through an intramolecular, transannular dative bond between nitrogen and germanium. The compound, 5-methyl-5-aza-2,8-dioxa-1-germana(II)bicyclo[3.3.0]octane (A), is prepared by the transesterification of germanium(II) diethoxide–ethanol complex[14] with *N*-methyldiethanolamine (eq 1 and 2).

$$GeI_2 + 2NaOC_2H_5 + xC_2H_5OH \rightarrow Ge(OC_2H_5)_2{\cdot}xC_2H_5OH + 2NaI \quad (1)$$

$$Ge(OC_2H_5)_2{\cdot}xC_2H_5OH + MeN(CH_2CH_2OH)_2 \rightarrow \underset{\textstyle A}{\overline{Ge}(OCH_2CH_2)_2\overline{N}Me} + (x + 2)C_2H_5OH \quad (2)$$

## Experimental Section

$GeI_2$ was prepared by methods described elsewhere.[15] Ethanol was dried by refluxing over magnesium metal and distilled under dry nitrogen immediately before use. *N*-Methyldiethanolamine was dried over sodium and distilled under vacuum. All solvents were dried and distilled by standard procedures. The infrared spectrum from 4000 to 400 cm$^{-1}$ was recorded on a Perkin-Elmer 457 double beam spectrometer as Nujol and Fluorolube mulls on CsBr plates. Mass spectral data were obtained by using a Finnigan 3300 spectrometer. The proton NMR spectrum was recorded on dilute solutions with either a Varian A60 or HR220 spectrometer using $Me_4Si$ as an internal reference.

In a typical reaction in the synthesis of $Ge(OCH_2CH_2)_2NMe$ (A), 1.5 g of freshly cut sodium was reacted with 50 mL of ethanol. A 31-mL sample of the resulting alcoholic sodium ethoxide solution was then added to 200 mL of toluene in a 500 mL round-bottom flask. The solution was degassed under vacuum and 2.5 g of $GeI_2$ was added

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† Submitted in partial fulfillment of the requirements for the Ph.D. degree in Chemistry at the Polytechnic Institute of New York.

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