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THE STRUCTURE OF HgACL₂ - A CHLORINE BRIDGED DIMER.(U)
SEP 79 O T BEACHLEY, R G SIMMONS
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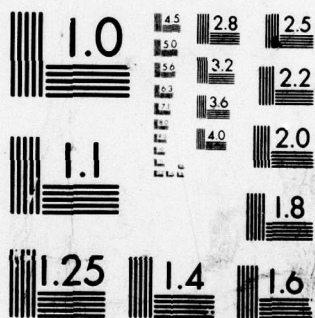


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by

O. T. Beachley, Jr. and Randall G. Simmons

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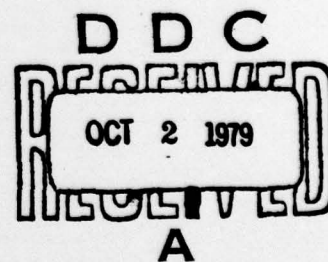
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The structure of HGaCl_2 has been determined using Raman and infrared spectroscopy. All data are consistent with the hypothesis that the compound exists as a dimeric molecule which has chlorine atoms in the bridging position. The hydride ligands occupy terminal positions. The reasons for the preference of gallium for chlorine bridges is discussed.		

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The Structure of HGaCl_2 - A Chlorine Bridged Dimer

by

O. T. Beachley, Jr.* and Randall G. Simmons

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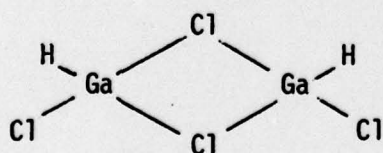
The compounds of Group IIA and IIIA (primarily Be, Mg, B and Al) which incorporate both hydrogen and halogen ligands have been the subject of numerous chemical investigations and spectroscopic studies. Interesting structural questions arise for this class of compounds because both hydrogen and halogen atoms can be bridging ligands between metal atoms. The spectroscopic properties of the dimeric Group IIA molecules, $[\text{HClBeO}(\text{C}_2\text{H}_5)_2]_2$ and $[\text{HClMgO}(\text{C}_2\text{H}_5)_2]_2$, are consistent with structures having bridging hydrogen atoms.^{1,2} The hydrogen bridge is also a dominant feature in boron chemistry³ whereas aluminum compounds³ are known with both bridging hydrogen and halogen atoms. Bond energy correlations for dimeric Al_2X_6 compounds⁴ suggest the following order of bridging potential, $\text{Cl} > \text{Br} > \text{I} > \text{H} > \text{CH}_3$. This sequence suggests that two center two electron bridge bonds are stronger than three center two electron bonds in aluminum chemistry.⁴ The compound HAlCl_2 would provide a test of this hypothesis but a simple dimeric species has not been observed.^{2,5,6,7} It is of interest that both AlH_3 and AlCl_3 have solid state structures with extended lattices involving six-coordinate aluminum with bridging hydrogen and chlorine atoms.⁸ Thus, the question of which

ligand, hydrogen or halogen, will be the preferential bridging ligand in Group IIIA compounds remains unanswered.

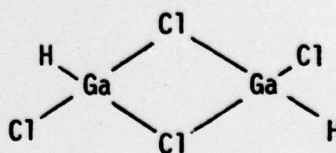
The gallium compound,⁹ HGaCl_2 , is known and exists as a simple dimeric species in benzene solution according to cryoscopic molecular weight measurements. However, no experimental data have been reported to ascertain whether $\text{H}_2\text{Ga}_2\text{Cl}_4$ exhibits hydrogen or chlorine bridging, or whether the compound is dimeric or more highly associated in the solid state. The task of answering these questions are hindered by the thermal instability of $\text{H}_2\text{Ga}_2\text{Cl}_4$ as it readily eliminates H_2 at temperatures above 20° to form GaGaCl_4 . Thus, the most applicable techniques available for studying the structure of HGaCl_2 are low temperature Raman and infrared spectroscopy. In this paper we report the results of our investigations of HGaCl_2 and DGaCl_2 . All spectroscopic data are consistent with the hypothesis that the dimeric molecule, which also exists in the solid state, has chlorine atoms in the bridging positions. The corresponding bromine compound, HGaBr_2 , was also prepared and was observed to decompose even more readily than HGaCl_2 .

The dimer, $\text{H}_2\text{Ga}_2\text{Cl}_4$, can have structures with bridging chlorine and

Bridging Chlorine Atoms

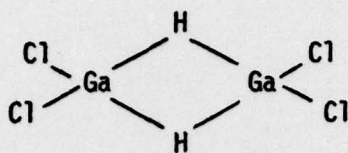


I
Cis Isomer



II
Trans Isomer

Bridging Hydrogen Atoms



III

hydrogen atoms. Structures which incorporate both a bridging chlorine and hydrogen are also possible but seem less likely. Three lines of experimental data suggest that HGaCl_2 exists at low temperature in the solid state as the geometrical isomers of the dimer with bridging chlorine atoms, Structures I and II.

- (1) The relative position of the GaH vibrational frequencies when compared with analogous Group IIIA compounds.
- (2) The comparison of the Raman spectra of $\text{H}_2\text{Ga}_2\text{Cl}_4$, $(\text{CH}_3)_2\text{Ga}_2\text{Cl}_4$ and $(\text{C}_6\text{H}_5)_2\text{Ga}_2\text{Cl}_4$.
- (3) The assignment of the vibrational modes, ν_2 and ν_3 , for a chlorine bridged dimeric skeleton.

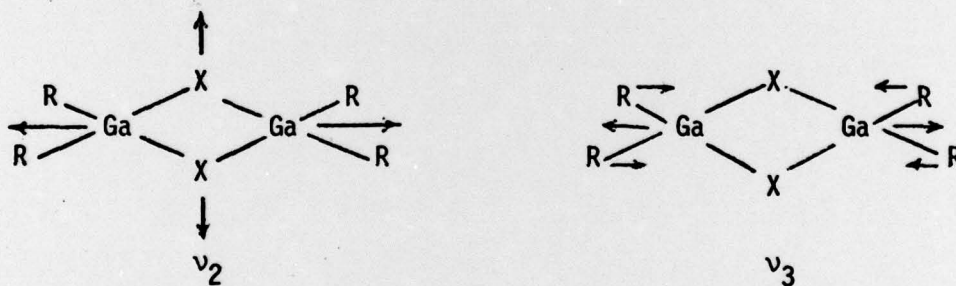
The gallium-hydrogen and gallium-chlorine vibrational motions are expected to give independent frequencies and not be coupled. Thus, pure Ga-H modes are readily assigned by comparing the infrared and Raman spectra of $\text{H}_2\text{Ga}_2\text{Cl}_4$ and $\text{D}_2\text{Ga}_2\text{Cl}_4$. In contrast, the motions of the terminal and bridging chlorine atoms will be coupled. The infrared Ga-H stretching frequencies have been observed as sharp bands at 2047 cm^{-1} for $\text{H}_2\text{Ga}_2\text{Cl}_4$ and 1465 cm^{-1} for $\text{D}_2\text{Ga}_2\text{Cl}_4$. The $\nu_{\text{GaH}}/\nu_{\text{D}}$ ratio for these frequencies is 1.40. The corresponding Raman bands occur at 2051 cm^{-1}

($\text{H}_2\text{Ga}_2\text{Cl}_4$) and 1468 cm^{-1} ($\text{D}_2\text{Ga}_2\text{Cl}_4$) with a $\nu\text{GaH/D}$ ratio of 1.40. The positions of these bands are consistent with terminal gallium-hydrogen bonds. If the hydrogen atoms had been in bridging positions, the vibrational modes of mainly stretching character should have absorptions at frequencies well below 2000 cm^{-1} . For comparison,³ terminal B-H bonds lead to absorptions in the $2500\text{--}2600\text{ cm}^{-1}$ region, whereas the vibrational modes of bridging hydrogens occur at $1500\text{--}1610\text{ cm}^{-1}$. In addition to the bands for the stretching motions, lower energy bond deformation modes were observed in the infrared spectrum of $\text{H}_2\text{Ga}_2\text{Cl}_4$ at 557 and 551 cm^{-1} . It is unfortunate that the KBr windows of the low temperature infrared cell prevented the observation of the bond deformation modes in $\text{D}_2\text{Ga}_2\text{Cl}_4$. The Ga-H bond deformation bands in the Raman spectra were observed at 518 cm^{-1} ($\text{H}_2\text{Ga}_2\text{Cl}_4$) and 419 cm^{-1} ($\text{D}_2\text{Ga}_2\text{Cl}_4$) with a $\nu\text{H/D}$ ratio of 1.39. These observations of the low energy bond deformation modes further support our hypothesis that the dimer has only terminal gallium-hydrogen bonds. The comparison of the number of observed Raman active bands (11) with those predicted by symmetry considerations further support our hypothesis. The cis and trans isomers of the chlorine bridged structure should have 11 and 9 Raman active bands, respectively. The dimer with the bridging hydrogen atoms should have only 9 bands, two fewer than that observed.

Further support for our hypothesis that the hydrogen atoms occupy terminal positions comes from comparing the Ga-H infrared stretching frequencies in $\text{H}_2\text{Ga}_2\text{Cl}_4$ with a variety of simple donor-acceptor adducts. All of these compounds involve four coordinate gallium. The GaH stretching bands for $\text{H}_2\text{Ga}_2\text{Cl}_4$, $\text{HCl}_2\text{Ga}\cdot\text{N}(\text{CH}_3)_3$, HCl_2Ga dioxane, and $\text{HCl}_2\text{Ga}\cdot\cdot$

bipyridyl occur as sharp bands at 2047, 1986, 1975, and 1855 cm^{-1} , respectively. If there had been a major change in structure from bridging hydrogen in $\text{H}_2\text{Ga}_2\text{Cl}_4$ to terminal hydrogens in the adducts, a large change in frequency would have been expected.

The organogallium dichloride compounds, $(\text{CH}_3)_2\text{Ga}_2\text{Cl}_4$ and $(\text{C}_6\text{H}_5)_2\text{Ga}_2\text{Cl}_4$, have chlorine bridged dimeric structures analogous to that proposed for $\text{H}_2\text{Ga}_2\text{Cl}_4$. The structure of the methyl compound was determined by an X-ray study¹⁰ whereas Raman and NQR data¹¹ were used to assign a structure for the phenyl derivative. The comparison of the Raman spectra of these compounds support our hypothesis. The frequencies of the Raman active vibrational modes, ν_2 and ν_3 , are related to the bridged dimeric skeleton and are dependent on the nature of the bridging atom.¹²⁻¹⁶ In the



series of compounds $(\text{C}_6\text{H}_5)_2\text{Ga}_2\text{X}_4$ ($\text{X} = \text{Cl}, \text{Br}, \text{I}$), the frequency¹¹ assigned to ν_2 for different halogens is 279 cm^{-1} (Cl), 185 cm^{-1} (Br) and 153 cm^{-1} (I). Similarly, bands for $(\text{C}_6\text{H}_5)_4\text{Ga}_2\text{X}_2$ ($\text{X} = \text{Cl}, \text{Br}, \text{I}$) occur at 263 cm^{-1} (Cl), 181 cm^{-1} (Br) and 150 cm^{-1} (I). Consequently, ν_2 is assigned to the band at 320 cm^{-1} for $\text{H}_2\text{Ga}_2\text{Cl}_4$; 311 cm^{-1} for $(\text{CH}_3)_2\text{Ga}_2\text{Cl}_4$ and 330 cm^{-1} for Ga_2Cl_6 ¹²⁻¹⁶. The vibrational mode, ν_3 , is also dependent on the nature of the bridging and terminal groups in dimeric struc-

tures. Assignments for ν_3 are 167 cm^{-1} (Ga_2Cl_6);¹²⁻¹⁶ $140, 133\text{ cm}^{-1}$ $(\text{C}_6\text{H}_5)_2\text{Ga}_2\text{Cl}_4$;¹¹ $165, 132\text{ cm}^{-1}$ $(\text{CH}_3)_2\text{Ga}_2\text{Cl}_4$ and $160, 148\text{ cm}^{-1}$ $\text{H}_2\text{Ga}_2\text{Cl}_4$. In conclusion, all spectroscopic data are fully consistent with a dimeric structure involving bridging chlorine atoms for $\text{H}_2\text{Ga}_2\text{Cl}_4$.

An attempt was made to differentiate the cis and trans isomers of $\text{H}_2\text{Ga}_2\text{Cl}_4$ by variable temperature ^1H nmr spectroscopy. However, the broadness of the single observed line prevented isomer identification. The increased broadness of the line with increased temperature is consistent with the high spin and quadrupole moments of the two gallium isotopes.

All data are consistent with the hypothesis that $\text{H}_2\text{Ga}_2\text{Cl}_4$ has a chlorine bridged dimeric skeleton. Thus, when there is a choice between hydrogen and chlorine as bridging atoms, beryllium, magnesium and boron have bridging hydrogen; gallium has bridging chlorine. The preference of gallium for chlorine bridges may be attributed to several factors. The greater bond energy for the gallium-chlorine bond would favor chlorine bridges. The longer gallium-chlorine bonds will also minimize repulsions between gallium atoms. Furthermore, internuclear and interelectronic repulsion terms will be greater for bridge bonds than terminal bonds, thus making the weak gallium-hydrogen bond even weaker, if it is in a bridging position. Consequently, chlorine bridges are favored in gallium chemistry. Since the other elements have more stable bonds to hydrogen, hydrogen bridges with their greater angular variation can be accommodated.

Experimental Section

Materials. All compounds described in this investigation were manipulated in a standard vacuum system. The stopcocks were lubricated with Halocarbon 25-5S grease which is not reactive to gallium(III) chlorine compounds. The solvents were dried by conventional means. The reagents were purified by sublimation and/or fractionation. The gallanes,⁹ $\text{H}_2\text{Ga}_2\text{Cl}_4$ and $\text{D}_2\text{Ga}_2\text{Cl}_4$, were prepared by reacting one mole of GaCl_3 with one mole of $\text{HSi}(\text{CH}_3)_3$ or $\text{DSi}(\text{CH}_3)_3$ at -23° for 8 hrs. The $\text{DSi}(\text{CH}_3)_3$ was prepared by reacting $\text{ClSi}(\text{CH}_3)_3$ with LiAlD_4 in dry dibutylether at 0° and purified by trap to trap fractionation. Its purity was confirmed by spectroscopy and vapor pressure measurements. The compound, HGABr_2 , was prepared from GaBr_3 and $\text{HSi}(\text{CH}_3)_3$ by the analogous procedure but it was observed to be significantly more unstable at room temperature than $\text{H}_2\text{Ga}_2\text{Cl}_4$.

Methylgallium dichloride,¹⁷ $(\text{CH}_3)_2\text{Ga}_2\text{Cl}_4$, was synthesized by reacting equimolar quantities of GaCl_3 and $\text{Si}(\text{CH}_3)_4$ in benzene at 35° for 4 hrs.

Analyses. Gallium was determined by EDTA titration.¹⁸ Chlorine was determined by standard gravimetric procedures. Hydrogen was measured after acid hydrolysis with a Toepler pump-gas burette assembly. $\text{H}_2\text{Ga}_2\text{Cl}_4$, Calc: Ga, 49.2; Cl, 50.1; H, 0.71. Found: Ga, 49.3; Cl, 50.0; H, 0.68. $\text{D}_2\text{Ga}_2\text{Cl}_4$, Calc: Ga, 48.9; Cl, 49.7. Found: Ga, 48.9; Cl, 49.8.

Infrared Spectra. The infrared spectra were recorded in the range $4000\text{--}250\text{ cm}^{-1}$ using a Perkin-Elmer Model 457 spectrometer. Condensed phase spectra were observed using a low temperature cell¹⁹ equipped with

KBr optics. The sample was cooled by a cold finger which contained liquid nitrogen.

Raman Spectra. The Raman spectra were recorded using a Spex Model 14018 double monochromator instrument and the 5145 or 4880⁰Å line of a coherent radiation argon ion laser. Spectra were observed when the sample was cooled to 120°K. Direct current detection was employed. Band positions were measured relative to the laser line and should be accurate to ± 1 cm⁻¹.

The following give the spectra [frequency, cm⁻¹ (intensity; vs, very strong; s, strong; m, medium, w, weak; sh, shoulder)]. H₂Ga₂Cl₄: 2051 (vs), 581 (vs), 338 (s), 320 (m), 202 (m), 160 (m), 148 (m), 136 (m), 114 (m), 105 (m), 52 (vs). D₂Ga₂Cl₄: 1468 (vs), 419 (vs), 383 (s), 321 (m), 314 (m), 202 (m), 160 (m), 148 (m), 135 (m), 112 (m), 103 (m), 52 (vs).

Nuclear Magnetic Resonance Spectra. The ¹H nmr spectra were recorded at 100 MHz using the Varian XL-100 spectrometer equipped with variable temperature capability. The solvent for the 25% solution of H₂Ga₂Cl₄ was d⁸-toluene. Tetramethylsilane was used as internal reference. The following give the temperature, chemical shift (δ ppm) and width of line at half height (Hz): -10°, 4.06, 64; -60°, 4.58, 22; -80°, 4.86, 21.

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