CRYSTAL STRUCTURE OF ORGANO SILICON COMPOUNDS. 

XL.* BIS(3-DICHLOROALUMINODISILOXANE-1,3-DIOLATO)ALUMINUM CHLORIDE

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An x-ray diffraction structural analysis was carried out for a tetracyclic alumosiloxane using 4281 reflections. The final R = 0.024. The molecule studied has two tetrahedral silicon atoms and two tetrahedral and one tetragonal pyramidal aluminum atoms within two planar four-membered \( \text{Al}_2\text{O}_2 \) rings and two six-members \( \text{Al}(\text{OSi})_2\text{O} \) rings with boat conformation.

INTRODUCTION

Alumosiloxanes (compounds with an Si-O-Al bond) hold special interest among the heterosiloxanes (compounds with an Si-O-M bond, in which M is a metal or nonmetal atom) since a) their "inorganic" frame is an analog for alumosilicates which are common in the earth's crust, and b) the Si-O-Al fragment is superior in its strength and chemical stability to almost all Si-O-M groups [2].

The crystalline alumodimethylsiloxane, \( \text{Me}_3\text{OClSiAl}_3 \) (I) was first obtained by one of the authors of the present work [3]. A structure consisting of six-membered alumosiloxane rings was assigned on the basis of elemental analysis and the chemical properties of this compound. In addition, inequivalence of the aluminum atoms was noted in the structure proposed. A structural analysis of the bromine analog (II) of this compound indicated the presence of six-membered alumosiloxane rings and also inequivalence of the aluminum atoms, but the structure of II was considerably more complicated [4-6]. This molecule consists of four condensed rings, namely two four-membered and two six-membered rings. An analogous structure was proposed for I on the basis of its isomorphism with II [4-6]. However, a detailed structural analysis of I has not been carried out.

For this study, we took chloroalumodimethylsiloxane I obtained according to our previous procedure [3]. The crystals for the x-ray diffraction measurements were obtained by recrystallization from toluene.

EXPERIMENTAL AND STRUCTURE DETERMINATION

The unit cell parameters for monoclinic crystals of I at -120°C are as follows: \( a = 10.841(2) \text{ Å}, b = 12.951(3) \text{ Å}, c = 18.459(4) \text{ Å}, \beta = 93.59(2)^\circ, V = 2586.7(9) \text{ Å}^3 \), \( d_{alc} = 1.507 \text{ g/cm}^3 \), \( Z = 4 \), \( \text{C}_{6}\text{H}_{12}\text{Al}_{2}\text{Cl}_{2}\text{O}_{4}\text{Si}_{4} \), space group \( \text{P}2_1/n \). The unit cell parameters and intensities of 4939 independent reflections were measured on a Syntex P2_1 four-circle diffractometer.
using λCuKα radiation and θ/2θ scanning to θmax = 25°. In the structure determination, we assumed the isostructural nature of the crystals of I and II. The coordinates of the Si, Al, and Cl atoms were taken to be as in the structure of II. The position of all the non-hydrogen atoms were found from the first F map (R = 0.45). The structure was refined by the method of least squares to R = 0.024 (Rw = 0.024) relative to 4281 reflections with I > 2σ in the block diagonal anisotropic approximation for the nonhydrogen atoms and the iso-