THE CRYSTAL STRUCTURE OF 1-(α-NAPHTHYL)GERMATRANE

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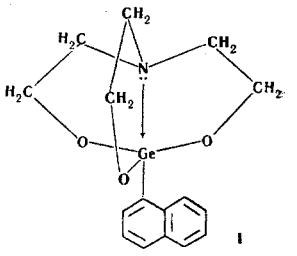
A calculation of the molecular and crystal structures of 1-ethylgermatrane [1] has shown the existence of a transannular donor-acceptor Ge← N bond in its molecule. In order to determine the change in the nature of this bond in 1-substituted germatranes as a function of the nature of the substituent, we have studied 1-(α-naphthyl)germatrane (I).



I

Compound I was synthesized by M. G. Voronkov and G. I. Zelchan [2,3] and was kindly given to us for x-ray investigations.

The experimental x-ray material was obtained with unfiltered Cu radiation and consists of a set of 0kl-3kl and hk0-hk4 "kforograms"* and also of hk5-hk9 Weissenberg rotating-crystal photographs. A total of 1023 nonzero independent reflections was recorded, their intensities being evaluated visually by means of density scale.

The following crystallographic characteristics were obtained for the crystals of I:

$a=9{,}57\pm0{,}02$ Å	$M=355{,}936$
$b=14{,}54\pm0{,}02$ Å	$d_n=1{,}61$ g/cm^3
$c=10{,}64\pm0{,}02$Å	$d_p=1{,}596$ g/cm^3
$\alpha=\beta=\gamma=90°$	$\mu(CuK_\alpha)=31{,}56$ cm^{-1}
$V=1480{,}53$ Å^3	$z=4$
$F_{(000)}=712$	

The space group of symmetry $Pna2_1$ was determined unambiguously from the statistics of the intensities of the reflections and the existence of a piezo effect. A model of the molecule was found by the heavy-atom method. The Ge atom was localized from the two-dimensional Patterson function, since the third coordinate of the Ge atom can be selected arbitrarily on the screw axis. The model of the structure of 1-(α-naphthyl)germatrane was obtained by successive calculations of the distribution of the electronic density. The unreliability factor (calculated from all the nonzero reflections) at this stage of the determination of the structure is 13%.

*Reciprocal lattice photographs (De Jong–Bouman method).

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The structural investigation showed the presence of a Ge ← N donor-acceptor bond the length of which is 2.26 Å. The Ge atom in the structure of I is located in a distorted trigonal bipyramid at the apices of which there are atoms of N and C (the α-atom of the naphthyl group). The N–Ge–C angle is close to 180°.

A further refinement of the structure of I is being performed.

LITERATURE CITED

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