bonds in the nitrate group are somewhat longer than the sum of the covalent radii. However, the nitrate group structure in (I) differs from that in 1,4-difluoro-1,4-tetranitro-2,3-butenediol dinitrate [6], in which the O-NO₂ bonds are significantly extended. Otherwise, the specific bond angles of the nitrate group and its difference relative to the C-NO₂ group are retained in all the compounds studied.

**CONCLUSIONS**

1. Cis-3,4-oxolanediol dinitrate was obtained by the O-nitration of the corresponding glycol using the HNO₃·H₂SO₄·H₂O system.

2. The cis-3,4-oxolanediol dinitrate molecule is asymmetric and the crystal structure consists of two enantiomers, i.e., is a racemate.

**LITERATURE CITED**


**COMPARATIVE STUDY OF B₁₀H₁₂(NH₃)₂ AND B₁₀H₁₂(NEt₃)₂**

BY THE METHODS OF X-RAY SPECTROSCOPY

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The nonionic compounds 6,9-bis(ammonia)-nido-decaborane(12)B₁₀H₁₂(NH₃)₂ and 6,9-bis(triethylamine)-nido-decaborane(12)B₁₀H₁₂(NEt₃)₂ belong to the group of derivatives of decaborane (14)B₁₀H₁₄ with the general formula B₁₀H₁₂L₂, where L is a Lewis base. Structural investigations of the compounds B₁₀H₁₂(NMe₃)₂, B₁₀H₁₂(NEt₃)₂, B₁₀H₁₂(NH₂)₂, and B₁₀H₁₂(SMe₂)₂ showed that the B₁₀H₁₂ fragment basically maintains the configuration of the framework of B atoms characteristic of decaborane(14) [1-4] and has some similar to the B₁₀H₁₂⁻ anion, i.e., it is an isostructural and isoelectronic analog of the compounds of the type B₁₀H₁₂L [5]. The complexes B₁₀H₁₂L₂ are formed by attaching bases of the Lewis type by means of donor N or S atoms to the B atoms in positions 6 and 9 of the decaborane(14) molecule with the accompanying elimination of two "bridging" hydrogens from the latter. The typical structure of the complexes B₁₀H₁₂L₂, which is presented in Fig. 1, consists of two pentagonal pyramids joined along a common edge of the bases.

The complexes of the B₁₀H₁₂L₂ type are of scientific and technological interest, since reactions resulting in the formation of close structures such as B₁₀H₁₀²⁻ and o-carborane(12)B₁₀C₂H₁₂, which have high chemical and thermal stabilities, are realized with their participation [6-8]. In this case, the processes resulting in the formation of these structures include a step involving the elimination of L from B₁₀H₁₂L₂. The latter is achieved when some L groups are replaced by Lewis bases of other types. The following empirical series of L has been derived on the basis of the capacity for displacement in the complex B₁₀H₁₂L₂ [6]: Et₃N, C₅H₅N, PPh₃, P(OR)₃ > MeCONMe₂, HCONMe₂, AsEt₃, P(SEt)₃ > Et₂NCN > MeCN > SMe₂.

The high reactivity of B₁₀H₁₂(NEt₃)₂ in processes resulting in the formation of the
Structure of Complexes of the type $B_{10}H_{12}(NR_2)_2$, $R = H, Et$.

$B_{10}H_{10}^{2-}$ anion and o-carborane(12) is known [7], while $B_{10}H_{12}(NH_3)_2$ is totally inert in processes of this kind. The place of NH$_3$ in the displacement series just given has not been determined, but it has been established that it is not displaced from $B_{10}H_{12}(NH_3)_2$ by tertiary amines, pyridine, and phosphorus bases [9]. In order to account for this difference it would be useful to investigate the complexes $B_{10}H_{12}(NH_3)_2$ and $B_{10}H_{12}(NEt_3)_2$ by x-ray photoelectron spectroscopy and x-ray spectroscopy, which make it possible to obtain information on the electronic structure.

**EXPERIMENTAL**

The complexes $B_{10}H_{12}(NH_3)_2$ and $B_{10}H_{12}(NEt_3)_2$ were synthesized according to [9] by treating a solution of $B_{10}H_{12}$ in toluene with NH$_3$ (gas) or NEt$_3$, respectively, with heating followed by recrystallization of the products from water. The latter were identified according to their IR and $^1B$ NMR spectra and by chemical analysis [9].

The B-K$_3$ emission spectra of the compounds were obtained on a Stearat ultralong-wavelength x-ray spectrometer with a Miristat crystal analyzer ($2d = 81 \text{ Å}$). The K$_3$ line of elemental boron ($\lambda = 67.6 \text{ Å}$) and the M$_5$ line of molybdenum ($\lambda = 64.38 \text{ Å}$) were used for the calibration of the spectrometer. The mean-square error of the measurements of the energies of the lines was ±0.2 eV. The resolution was ≈2 eV. The operating conditions of the x-ray tube were 4 kV × 800 mA.

The $B$ $1s$ and $N$ $1s$ x-ray photoelectron spectra were obtained on the spectrometer described in [10]. The spectrometer was calibrated with the use of the C $1s$ line of graphite (284.3 eV). The mean-square error in the measurements of the energies was ±0.15 eV. The resolution was 1.5 eV. The operating conditions of the x-ray tube were 8 kV × 100 mA. The radiation corresponded to Al Ka (1487 eV).

During the recording of the x-ray photoelectron spectra it was found that in the case of $B_{10}H_{12}(NH_3)_2$, the effects of the charging of the sample cause significant distortions in the form of the spectrum being analyzed when the usual method for preparing the samples is used. The testing of a number of different methods for applying the samples to the substrate showed that charging can be successfully avoided when a graphite matrix is used. The finely ground samples were rubbed into a graphite plate with a thickness of 1 mm, which was glued onto the metallic substrate of the sample holder by a current-conducting glue for the purpose of forming an electrical contact between the graphite matrix and the substrate.

Ten $B$ $1s$ and $N$ $1s$ spectra were obtained for each compound with the use of this method. Five $B$ $1s$ spectra were selected among them for $B_{10}H_{12}(NH_3)_2$ and for $B_{10}H_{12}(NEt_3)_2$, and they were then used to construct the averaged spectra of the complex. The criterion of smallest deviation of the halfwidths and the asymmetry indices of the spectral lines from their mean values served as a basis for the selection. The superposition of the spectra for the averaging was carried out according to their centers of gravity. The standard deviations in the location of the special features of the spectra with respect to the intensity were ±7% in the region of the right-hand branch and ±8% in the region of the left-hand branch of the spectra; the deviations with respect to the $x$ coordinate were ±0.1 and ±0.15 eV, respectively (Fig. 2). In order to reveal the fine structure, the averaged $B$ $1s$ spectra were subjected to an operation which is the inverse of the convolution operation [11] [this made it possible...