ence in composition between Cu$_2$In$_2$S$_{12}$ (cation ordering of 2:1 type, space group I$_4_1$/amd) and CuIn$_8$S$_8$ (cation ordering of 1:1 type, space group P4$_3$m), as those phases have been observed in studies on ordered CuIn$_3$S$_8$ phases [11]. That stoichiometry can be obtained if one assumes that two ordered 2:1 phases can coexist in a 1:1 relation: Cu$_2$In$_2$S$_{12}$ and CuIn$_8$S$_8$. Those phases are isostructural (Table I) and should have similar unit-cell parameters. Formally speaking, the CuIn$_8$S$_8$ stoichiometry can be obtained in a polytype with packing code $cA\sim BpBr\sim rA\sim BpA$ that simultaneously contains ordered 2:1 fragments in the [Cu$_2$In] and $2\sim v2\sim v2\sim v2'$ [CuIn$_2$] forms, but the symmetry of that spineloid differs from I$_4_1$/amd as determined by experiment in [11].

LITERATURE CITED


CRYSTAL STRUCTURE OF DIAQUABIS(NITROAMINO-
GUANIDINE)NICKEL(II)PERCHLORATE \( [\text{Ni(CH}_3\text{N}_5\text{O}_2]_2^- \)
\((\text{H}_2\text{O})_2](\text{ClO}_4)_2\)

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The synthesis of diaquabis(nitroaminoguanidine)nickel(II)nitrate \([\text{Ni(CH}_3\text{N}_5\text{O}_2]_2^- \)
\((\text{ClO}_4)_2\) and the results from its investigation by IR spectroscopy and x-ray crystallographic analysis were reported in [1]. It was concluded on the basis of these data that the nitro-
imine structure is formed in the nitroaminoguanidine ligand of this compound:

\[
\begin{align*}
\text{NH}_3 \quad \text{C} \quad \text{NH} \quad \text{NH}_2
\end{align*}
\]

In the present work we give the results from determination of the crystal structure of another complex of Ni(II) with nitroaminoguanidine, i.e., diaquabis(nitroaminoguanidine)-nickel(II) perchlorate \([\text{Ni(CH}_3\text{N}_5\text{O}_2](\text{H}_2\text{O})_2](\text{ClO}_4)_2\).
EXPERIMENTAL

The violet single crystals of \([\text{Ni(CH}_3\text{NSO}_2\text{)}_2\text{H}_2\text{O}_2\text{]}(\text{CIO}_4)_2\) with irregular form have a monoclinic unit cell with the following parameters: \(a = 16.113(4)\) Å, \(b = 12.243(3)\) Å, \(c = 8.336(2)\) Å, \(\alpha = 90.01(2)°\), \(V = 1644.5(7)\) Å\(^3\), space group Pb\(\text{bI}_1\), \(Z = 4\), \(\text{d}_{\text{calc}} = 2.16\) g/cm\(^3\). A set of experimental data was obtained from a single crystal measuring 0.25 × 0.50 × 0.35 mm on an automatic Syntex P2\(_1\) diffractometer by the standard procedure (MoK\(_\lambda\) radiation, graphite monochromator, \(\theta/2\theta\) scan with variable rate \(V_{\text{min}} = 5\) deg/min, \(2\theta_{\text{max}} = 48°\), 4569 \(I_{hkj} > 2\sigma_I\) from 5549 measured intensities). The structure was solved by the heavy atom method and refined by least-squares treatment in anisotropic approximation for all the nonhydrogen atoms. All the hydrogen atoms were localized at the last stage of refinement in the \(\Delta p(xyz)\) difference synthesis, and their joint refinement (in isotropic approximation) with the nonhydrogen atoms led to a final \(R\) value of 0.049. All the calculations on the solution and refinement of the structure were conducted by means of the YANX software [2]. The atomic coordinates and equivalent temperature parameters \(U_{\text{eq}} = 1/3(U_{11} + U_{22} + U_{33})\) for the H atoms (\(U_{\text{iso}}\)) are given in Table 1.

DESCRIPTION OF STRUCTURE

A projection of the structure of \([\text{Ni(CH}_3\text{NSO}_2\text{)}_2\text{H}_2\text{O}_2\text{]}(\text{CIO}_4)_2\) on to the (001) plane is given in Fig. 1. The ionic-type structure consists of \([\text{Ni(CH}_3\text{NSO}_2\text{)}_2\text{H}_2\text{O}_2\text{]}^{2+}\) cations and \(\text{CIO}_4^-\) anions. Both crystallographically independent nickel atoms have a pseudocentrosymmetric environment (CN of Ni = 6, octahedron), formed in the equatorial plane of the N atoms of the two bidentate \(\text{CH}_3\text{NSO}_2\) ligands, while the axial positions are occupied by the oxygen atoms of the water molecules. The average Ni-N and Ni-O bond lengths and ONiN chelate angles are 2.076(8), 2.099(8) Å, and 77.0(3)°. The \(\text{CH}_3\text{NSO}_2\) ligands are coordinated with the central nickel atom by the N atom of the terminal NH\(_2\) group and by the imine N atom attached to the nitro group and form with it almost planar five-membered chelate rings. (The deviation of the atoms is not greater than 0.22 Å.) The \(\text{CIO}_4^-\) anions have the usual tetrahedral structure.

The complex cations and \(\text{CIO}_4^-\) anions are arranged in the structure in layers parallel to the (110) plane. The neighboring cations are linked to each other by O\(_\text{H}_2\text{O}\)−H...O\(_\text{NO}_2\) hydrogen bonds of 2.78(1) Å. All the bond lengths and bond angles in the complex are given in Fig. 2. The coordinated water molecules also form O−H...O hydrogen bonds with the oxygen atoms of the \(\text{CIO}_4^-\) anions [2.73(1)-2.74(1) Å]. In addition, as in the nitrate complex [1], very strong