Journal of Structural Chemistry, Vol. 35, No. 3, 1994

CRYSTAL AND MOLECULAR STRUCTURE OF BIURET PERCHLORATE

Yu. P. Gladii, A. Tashenov, M. A. Tanival', and N. N. Nurakhmetov

UDC 548.737

To study complex formation of amides with mineral acids, we investigated biuret perchlorate by X-ray analysis. Experimental data were obtained on a DRON-2.0 X-ray diffractometer (λCuK_α, graphite monochromator, ω-scan mode, 662 unique reflections for $\theta \leq 65°$). The crystals are monoclinic, $a = 4.859(1)$, $b = 10.588(2)$, $c = 7.120(1)$, $\beta = 100.90(2)°$, $V = 359.7$ Å^3, $Z = 2$, space group $P2_1$, $d_{calc} = 1.878$ g·cm^{-3}. The structure was solved by direct methods. Nonhydrogen atoms were located on electron density E-maps and refined by the least-squares method at first isotropically and then anisotropically. Hydrogen atoms were located on difference Fourier maps and included in the refinement in an anisotropic approximation. The final R factor is 0.054. All calculations were performed with a "ROENTGEN-75" program package.* Atomic coordinates, bond lengths, and bond angles in the cation and anion and parameters of hydrogen bonds are given in Tables 1-3; projection of the structure on a (100) plane is shown in Fig. 1.

As a result of our study, we found that the biuret molecule is protonated at the oxygen atom to form a salt of the formula $\left[\begin{matrix}H_2NCONH\\ H_2N\end{matrix}\!\!>\!COH\right]^+ ClO_4^-$. The nonhydrogen atoms of the cation lie in the plane with an accuracy of 0.05 Å; the oxygen atoms of the carbonyl groups are in the *cis*-position. The hydrogen atoms of the cation involved in hydrogen bonds deviate from the plane; for example, the torsion angles at the C(1)–N(1) and C(2)–N(3) bonds are –13.9 and 10.1°, respectively. Differences in the C=O and C–N bond lengths and bond angles for C(1) and C(2) atoms are probably due to the influence of the steric environment of the cation and H-bonding.

The anions in the structure are slightly distorted tetrahedra with Cl–O bond lengths of 1.379-1.415(2) Å and OClO bond angles of 106.3-111.7(2)°.

TABLE 1. Atomic Coordinates ($\times 10^4$, $\times 10^3$ for H atoms) in the Structure

Atom	*x/a*	*y/b*	*z/c*	Atom	*x/a*	*y/b*	*z/c*
Cl	4912(1)	1940(1)	1626(1)	N(3)	8774(10)	–362(4)	7547(6)
O(1)	2099(4)	1916(4)	1890(3)	C(1)	9720(10)	3044(4)	7007(6)
O(2)	5201(7)	2882(3)	280(5)	C(2)	9847(9)	791(4)	7080(6)
O(3)	6663(6)	2236(4)	3328(4)	H(1)	1001(11)	491(5)	747(7)
O(4)	5679(7)	803(3)	873(4)	H(2)	813(11)	396(5)	890(7)
O(5)	11611(7)	3089(3)	6037(4)	H(3)	744(10)	195(9)	824(7)
O(6)	11732(6)	787(3)	6066(4)	H(4)	726(12)	–38(6)	860(8)
N(1)	8720(9)	4072(4)	7749(6)	H(5)	954(10)	–110(5)	716(7)
N(2)	8781(5)	1891(4)	7567(3)	H(6)	1351(12)	303(6)	563(7)

* V. I. Andrianov, Z. Sh. Safina, and B. L. Tarnapolskii, *"ROENTGEN-75." Program Package for Solving Crystal Structures* [in Russian], Chernogolovka (1975).

Al-Farabi Kazakh State National University. Translated from *Zhurnal Strukturnoi Khimii*, Vol. 35, No. 3, pp. 159-161, May-June, 1994. Original article submitted July 6, 1993.

 0022-4766/94/3503-0416$12.50

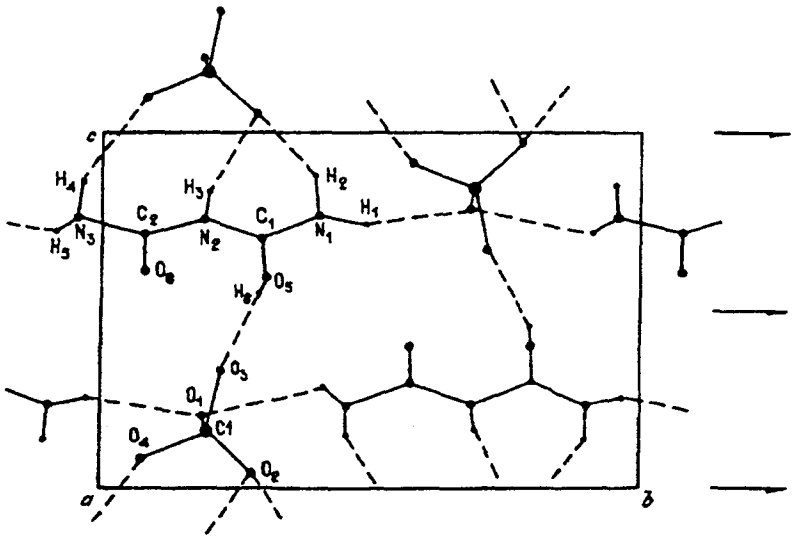

Fig. 1. Projection of the structure of biuret perchlorate on a (100) plane.

TABLE 2. Bond Lengths d (Å) and Bond Angles ω (deg) in the Structure

Bond	d	Angle	ω
C(1)–O(5)	1.251(6)	N(1)C(1)O(5)	123.0(4)
C(2)–O(6)	1.269(5)	N(2)C(1)O(5)	120.6(4)
C(1)–N(1)	1.341(6)	N(1)C(1)N(2)	116.0(4)
C(1)–N(2)	1.388(5)	C(1)N(2)C(2)	121.6(3)
C(2)–N(2)	1.347(5)	N(2)C(2)O(6)	120.2(4)
C(2)–N(3)	1.391(6)	N(3)C(2)O(6)	118.4(4)
Cl–O(1)	1.415(2)	N(2)C(2)N(3)	121.2(4)
Cl–O(2)	1.408(3)	O(1)ClO(2)	109.1(2)
Cl–O(3)	1.379(2)	O(1)ClO(3)	109.8(2)
Cl–O(4)	1.397(3)	O(1)ClO(4)	111.7(2)
		O(2)ClO(3)	108.2(2)
		O(2)ClO(4)	106.3(2)
		O(3)ClO(4)	111.6(2)

TABLE 3. Parameters of Hydrogen Bonds in the Structure

Bond	D...A, Å	D–H, Å	A...H, Å	∠DHA, deg
N(1)–H(1)...O(1) $(1-x, y+1/2, 1-z)$	3.054(6)	1.13(7)	2.43(8)	113(3)
N(1)–H(2)...O(2) $(x, y, 1+z)$	2.988(6)	0.92(8)	2.20(6)	143(3)
N(2)–H(3)...O(2) $(x, y, 1+z)$	3.022(5)	0.88(8)	2.21(7)	153(3)
N(3)–H(4)...O(4) $(x, y, 1+z)$	3.277(6)	1.15(9)	2.29(7)	142(3)
N(3)–H(5)...O(1) $(1-x, y-1/2, 1-z)$	2.952(5)	0.93(8)	2.39(8)	119(3)

In the structure, cations and anions are packed by means of a three-dimensional system of H-bonds involving all hydrogen atoms of the structure (see Fig. 1 and Table 3). The O(5)...O(3) distance is 2.926(7) Å, which is typical for hydrogen bonds (O(5)–H(6) = 1.02(9) Å, O(3)–H(6) = 2.59(8) Å) but the O(5)H(6)O(3) angle is only 120(3)°.

Translated by L. Smolina