

Fig. 2. Stereoscopic plot (ORTEP, Johnson, 1965) of the structure showing the thermal ellipsoids.

Discussion

As we have already observed, the introduction of anisotropic thermal parameters is imperative to obtain the best fit. The main feature of the thermal motion is the large amplitude of the K atom motion in the [001] direction. Equally, we note that the motion of the octahedra is characterized by a large value of u_{33} for F_{eq} which is correlated with the large values for u_{11} and u_{22} of F_{ax} . Fig. 2 shows a plot of the thermal ellipsoids.

All these observations can be connected with the vicinity of the structural phase transition observed at

250 K. In order to study the lattice dynamics of this crystal a low-temperature structure determination is now in progress.

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A Reinvestigation of the Structure of μ -Hyperoxo-bis(pentaamminecobalt) Tris(hydrogensulfate) Monosulfate at Room Temperature and at 17 K*

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Abstract

$[\text{Co}_2(\text{NH}_3)_{10}(\text{O}_2)](\text{HSO}_4)_3(\text{SO}_4)$, $M_r = 707.44$, is in space group $P2_12_12_1$ [$T = 293 \text{ K}$: $a = 16.360$ (1), $b = 13.946$ (3), $c = 9.978$ (1) Å; $T = 17 \text{ K}$: $a = 16.198$ (4), $b = 13.842$ (5), $c = 9.963$ (3) Å], with $Z = 4$, $\rho_c = 2.064$, $\rho_o = 2.060$ (5) Mg m^{-3} (293 K), $F(000) = 1460$, $\lambda(\text{Mo K}\alpha) = 0.71069 \text{ \AA}$. Final R values were 0.066 for both studies with 2377 and 2257 reflections respectively. The structure determined ear-

lier was confirmed in general. At room temperature the sulfate group has a twofold disorder. H atoms were located at both temperatures and a complete hydrogen-bonding pattern is presented.

Introduction

Schaefer & Marsh (1966) have previously reported the structure of the title compound, determined from film data. H atoms were not located, although a plausible hydrogen-bonding pattern was worked out, and bond distances had e.s.d.'s of 0.02 or 0.03 Å. To locate the

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H atoms and improve the precision of the bond distances, we have redetermined the structure using data collected with Mo $K\alpha$ radiation on an automatic diffractometer. For comparison purposes, we collected one data set at room temperature and another at 17 K. The two derived structures are very similar, the main differences being in the H atom positional parameters (see below).

Crystals of the compound used in the previous study were dissolved in hot, 2M H_2SO_4 . The solution was filtered into a Dewar flask. The flask was closed with a rubber stopper and placed in a refrigerator at 270 K. After 24 h the resulting needles were filtered and air-dried. Nearly cubic pieces were cut from the needles with a razor blade; they measured about 0.15 mm on a side. Some were mounted using an epoxy cement, but this apparently reacts with the complex; household cement worked better. Preliminary photographs showed the crystals to be the same as had been studied earlier.

Room-temperature data collection and refinement

Integrated intensity measurements were made on a locally modified Syntex $P2_1$ diffractometer using graphite-monochromated Mo $K\alpha$ radiation. A $\theta:2\theta$ scan was used, scanning from 0.9° below $2\theta(K\alpha_1)$ to 0.9° above $2\theta(K\alpha_2)$. The scan speed was 2° min^{-1} . The 2632 reflections scanned included all the unique data with $2\theta < 50^\circ$. Stationary background counts were measured at each end of the scan such that the total background time was half the scan time. The estimated variances in intensity were obtained from counting statistics plus an additional term ($0.02 \times \text{scan counts}$)², to account for intensity-dependent errors.

Four check reflections were measured every 100 reflections; they showed an initial rapid decrease in intensity (0.8% on F during 304 measurements) followed by a more gradual decay to the end of the data collection (2.0% on F for the remaining 2427 measurements). The decay correction was approximated as two linear functions with a maximum correction of 2.8%. The data were then merged into a set of 2377 independent intensities, which were corrected for Lorentz and polarization effects and placed on an approximately absolute scale using the results of a Wilson plot; no absorption correction was made ($\mu_{\text{max}} = 0.3$). The unit-cell parameters were taken as the previously reported values; the cell constants determined from the setting values of the diffractometer data agreed with the earlier ones within the statistical limits of accuracy.

Refinement began with the published coordinates and anisotropic temperature factors for all non-hydrogen atoms. After several least-squares cycles, the H atoms were located from difference maps and

included in subsequent calculations; neither their positions nor their thermal parameters were refined. The refinement with this model converged with $R = 0.069$ and the goodness of fit $\text{GOF} = 1.33$ ($R = (\sum |F_o| - |F_c|) / \sum |F_o|$; $\text{GOF} = [\sum w(F_o^2 - F_c^2)^2 / (N - P)]^{1/2}$, where w is the weight, N the number of reflections and P the number of parameters). A difference map showed several excursions greater than $1 \text{ e } \text{Å}^{-3}$, most in the vicinity of O atoms of the sulfate group. It appeared, in fact, that this group is disordered between two positions: O(6) and S(1) are ordered, but the other three O atoms have two alternate locations. A population parameter for these atoms was defined such that the three atoms in one orientation had the same population, p , and the three atoms in the other had population $(1 - p)$. The final refinement had the coordinates of all the non-hydrogen atoms in one matrix and a scale factor, a secondary-extinction factor (Larson, 1967), isotropic temperature factors for the disordered O atoms, the population parameter, and anisotropic thermal parameters for the rest of the non-hydrogen atoms in another matrix. The 33 H atoms were included with positions as improved by difference maps and with isotropic thermal parameters $B = 3.0 \text{ Å}^2$. This refinement converged with $R = 0.066$ and $\text{GOF} = 1.24$. The final difference map had maximum excursions of $+1.12$ and $-0.98 \text{ e } \text{Å}^{-3}$; none of the larger excursions was closely associated with any atom. All calculations were performed on an IBM 370/3032 computer using the programs of the CRYM crystallographic computing system (Duchamp, 1964). The function minimized was $\sum w(F_o^2 - F_c^2)^2$, including reflections for which F_o^2 was negative. The final parameters are given in Table 1.*

Low-temperature data collection and refinement

A full set of data was collected for each of two crystals. Each crystal was examined using a 30 min oscillation photograph prior to collecting diffractometer data. Integrated intensity data were collected at $17.0 \pm 0.5 \text{ K}$ using an extensively modified Syntex $P1$ diffractometer. The details of the instrument and the low-temperature device have been described elsewhere (Samson, Goldish & Dick, 1980). The data were collected, using graphite-monochromated Mo $K\alpha$ radiation and a $\theta:2\theta$ scan technique, for $2\theta < 50^\circ$. The scan range was from $[2\theta(K\alpha_1) - 1.0]^\circ$ to $[2\theta(K\alpha_2) + 1.0]^\circ$ and the scan speed was constant at $2.0^\circ \text{ min}^{-1}$. Backgrounds were measured at each end of the scan

* Lists of structure factors, anisotropic thermal parameters and H atom parameters for the room- and low-temperature studies have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35654 (41 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters of the heavy atoms*

All x, y, z values have been multiplied by 10⁴. $U_{eq} = (U_1 U_2 U_3)^{1/3}$ (F. R. Ahmed, 1979, private communication); $\sigma U_{eq} = (\langle \sigma U_{ii} \rangle \cdot U_{eq}) / (\langle U_{ii} \rangle \cdot \sqrt{6})$ (V. Schomaker, 1980, private communication).

	Room temperature				Low temperature			
	x	y	z	U_{eq} or B (Å ²)	x	y	z	U_{eq} or B (Å ²)
Co(1)	-1243 (1)	-897 (1)	10910 (1)	0.0136 (3)	-1251 (1)	-914 (1)	10905 (1)	0.0102 (3)
Co(2)	1009 (1)	540 (1)	9109 (1)	0.0152 (3)	1023 (1)	531 (1)	9123 (1)	0.0096 (4)
O(1)	-403 (4)	-432 (4)	9743 (6)	0.0234 (14)	-403 (4)	-451 (4)	9732 (6)	1.0 (1)
O(2)	217 (4)	-15 (4)	10300 (6)	0.0216 (13)	248 (4)	-49 (4)	10323 (6)	1.1 (1)
N(1)	-2001 (5)	-257 (5)	9699 (7)	0.0256 (17)	-2017 (5)	-280 (5)	9695 (7)	1.0 (2)
N(2)	-1269 (5)	-2020 (5)	9752 (7)	0.0235 (16)	-1291 (5)	-2048 (5)	9726 (7)	1.0 (1)
N(3)	-1172 (5)	253 (4)	12041 (7)	0.0235 (16)	-1149 (5)	220 (5)	12073 (7)	1.0 (2)
N(4)	-2160 (4)	-1359 (5)	12013 (7)	0.0195 (16)	-2168 (5)	-1359 (5)	12020 (7)	1.1 (2)
N(5)	-481 (5)	-1541 (5)	12102 (8)	0.0260 (17)	-490 (5)	-1591 (5)	12073 (7)	0.8 (2)
N(6)	1012 (5)	1646 (5)	10350 (7)	0.0248 (16)	1057 (5)	1613 (5)	10366 (7)	1.0 (2)
N(7)	1845 (4)	-25 (5)	10247 (8)	0.0269 (17)	1885 (5)	-55 (5)	10227 (8)	1.1 (2)
N(8)	180 (5)	1172 (5)	8013 (7)	0.0214 (17)	168 (5)	1190 (5)	8071 (7)	1.3 (2)
N(9)	1841 (5)	1129 (5)	7983 (8)	0.0262 (17)	1848 (5)	1137 (5)	7967 (8)	1.1 (2)
N(10)	1007 (5)	-588 (5)	7929 (7)	0.0240 (16)	1009 (5)	-592 (5)	7921 (7)	1.3 (2)
S(1)	1020 (2)	497 (2)	3928 (2)	0.0202 (5)	1009 (2)	472 (2)	3902 (2)	0.0122 (5)
O(3A)	394 (5)	-310 (6)	3973 (9)	2.2 (2)	384 (4)	-351 (4)	3986 (6)	1.2 (1)
O(3B)	113 (11)	220 (14)	3895 (19)	1.8 (5)				
O(4A)	655 (6)	1274 (7)	3189 (10)	2.7 (2)	612 (4)	1267 (4)	3169 (6)	1.0 (1)
O(4B)	1087 (14)	1370 (16)	3117 (23)	2.1 (5)				
O(5A)	1744 (6)	106 (7)	3230 (10)	3.2 (2)	1736 (4)	115 (4)	3168 (6)	1.2 (1)
O(5B)	1477 (13)	-281 (15)	3402 (23)	2.1 (5)				
O(6)	1234 (4)	740 (4)	5310 (6)	0.0323 (14)	1216 (4)	749 (4)	5277 (6)	1.1 (1)
S(2)	-1215 (2)	-957 (2)	6115 (2)	0.0202 (5)	-1214 (2)	-957 (2)	6089 (2)	0.0118 (5)
O(7)	-445 (4)	-293 (5)	6124 (7)	0.0343 (14)	-440 (4)	-288 (4)	6114 (8)	0.9 (1)
O(8)	1850 (4)	434 (4)	6817 (6)	0.0289 (14)	-1851 (4)	-426 (4)	6790 (6)	0.9 (1)
O(9)	-986 (4)	-1827 (4)	6822 (6)	0.0273 (13)	-985 (4)	-1844 (4)	6793 (5)	1.0 (1)
O(10)	-1402 (4)	-1133 (4)	4714 (5)	0.0233 (12)	-1409 (4)	-1125 (4)	4673 (6)	1.0 (1)
S(3)	3399 (1)	2464 (2)	10021 (3)	0.0272 (5)	3406 (2)	2487 (2)	9966 (3)	0.0116 (5)
O(11)	4251 (4)	1989 (5)	9792 (8)	0.0508 (19)	4306 (4)	2079 (4)	9724 (6)	1.0 (1)
O(12)	3067 (4)	2698 (5)	8690 (7)	0.0435 (17)	3070 (4)	2748 (4)	8643 (6)	1.0 (1)
O(13)	2890 (4)	1790 (5)	10696 (7)	0.0467 (17)	2934 (4)	1726 (4)	10601 (6)	1.2 (1)
O(14)	3585 (4)	3321 (4)	10768 (7)	0.0410 (15)	3525 (4)	3339 (4)	10803 (6)	1.3 (1)
S(4)	3679 (2)	2622 (2)	5170 (3)	0.0284 (5)	3694 (2)	2591 (2)	5147 (2)	0.0136 (5)
O(15)	4438 (4)	2466 (5)	4517 (6)	0.0376 (17)	4462 (4)	2404 (4)	4456 (6)	1.3 (1)
O(16)	3479 (5)	3611 (4)	5464 (7)	0.0398 (15)	3532 (4)	3585 (4)	5450 (6)	1.2 (1)
O(17)	2997 (4)	2131 (4)	4533 (8)	0.0361 (15)	2994 (4)	2125 (4)	4464 (6)	1.3 (1)
O(18)	3790 (5)	2080 (5)	6556 (7)	0.0461 (15)	3777 (4)	2034 (4)	6524 (6)	1.3 (1)

for a combined time equal to half the scan time. Altogether, 3282 reflections for crystal (1) (including some duplicates) and 2612 reflections for crystal (2) were measured. Variances for the intensities were estimated from counting statistics plus an additional term $(0.02 \times \text{scan counts})^2$ to correct for intensity-dependent errors.

For each crystal four check reflections (400, 460, 12 $\bar{2}$, 024) were measured every 100 reflections. The check reflections showed a linear decrease in intensity for both data sets which was later found to be caused by a real variation in the space current of the X-ray tube. The data were corrected for this change in X-ray source intensity using a linear function. The maximum correction was 11.3% on F for crystal (1) and 6.8% for crystal (2).

The cell parameters given in the *Abstract* were determined by averaging the values found for each crystal; these were determined by a least-squares fit to the setting angles of fifteen reflections. The two sets of values agreed within their calculated standard deviations.

The data were corrected for Lorentz and polarization effects and structure factor magnitudes were derived. The data were not corrected for absorption ($\mu_{r \max} = 0.3$). The two data sets were merged to give 2257 unique reflections. Least-squares refinement was begun with the parameters from the ordered room-temperature refinement. The refinement quickly converged, but the anisotropic thermal parameters of several atoms were unsatisfactory; those for O(1) were non-positive definite and the thermal ellipsoids for five

of the N atoms had major:minor axis ratios of 5:1 or larger. The H atoms were located in difference maps (these maps were significantly easier to interpret than those of the room-temperature structure) and the H atoms were included as fixed contributions, with $B = 2.0 \text{ \AA}^2$, in subsequent calculations. The thermal parameters improved somewhat, but O(1) remained non-positive definite. Close inspection of the thermal parameters of the O and N atoms revealed that, although some were severely anisotropic, the actual values were so small that they were all within 3σ of being isotropic. The structure was therefore refined as follows: One matrix contained the positional coordinates of all the non-hydrogen atoms and a second matrix contained anisotropic thermal parameters for the Co and S atoms, isotropic thermal parameters for the O and N atoms, a scale factor and a secondary-extinction parameter. All 33 H atoms were included in the structure factor calculations but not refined.

The isotropic refinement converged with $R = 0.066$ and $\text{GOF} = 1.37$. In the final difference map there are several peaks of 1 e \AA^{-3} , some closely associated with the O atoms. While the anisotropic refinement might have reduced these, the final agreement between observed and calculated structure factors indicates that the isotropic model is satisfactory. The final positional parameters for the heavy atoms are given in Table 1.*

Discussion

The cation

Fig. 1 shows a drawing of a cation prepared by ORTEP (Johnson, 1965) using the room-temperature parameters. The atom-labelling scheme for both structures is shown in Fig. 2. All the heavy-atom distances are given in Table 2. There are no significant changes from the structure originally reported. The O—O distances confirm that the O_2 group is certainly best called a hyperoxide group. Individual e.s.d.'s in Co—O

* See deposition footnote.

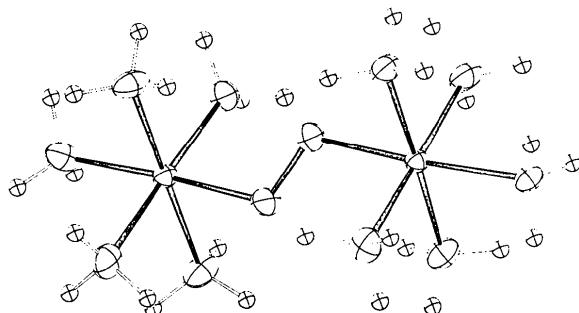


Fig. 1. A view of the $[\text{Co}_2(\text{NH}_3)_{10}(\text{O}_2)]^{2+}$ cation.

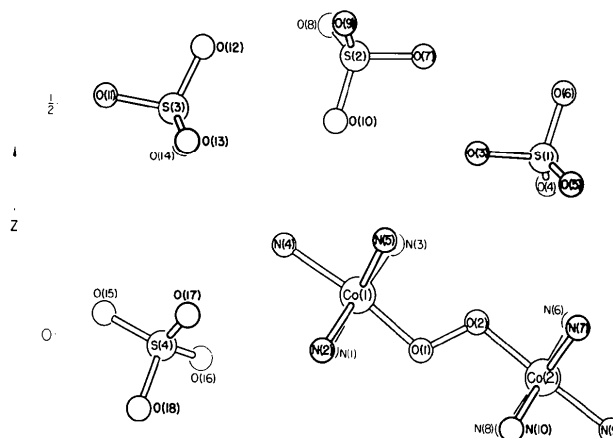


Fig. 2. A view perpendicular to the (230) plane showing the atom labelling for both structures.

Table 2. Distances within the ions (\AA)

	Room temperature	Low temperature	Room temperature (original film data)
Co(1)—O(1)	1.914 (6)	1.916 (6)	1.894 (20)
N(1)	1.949 (7)	1.940 (7)	1.903 (20)
N(2)	1.946 (7)	1.962 (7)	1.958 (20)
N(3)	1.965 (7)	1.962 (7)	1.933 (20)
N(4)	1.969 (7)	1.953 (7)	1.969 (20)
N(5)	1.942 (8)	1.937 (7)	1.968 (20)
Co(2)—O(2)	1.921 (6)	1.909 (6)	1.894 (20)
N(6)	1.978 (7)	1.945 (7)	1.931 (20)
N(7)	1.945 (7)	1.954 (7)	1.955 (20)
N(8)	1.953 (7)	1.961 (8)	1.947 (20)
N(9)	1.946 (8)	1.955 (8)	1.946 (20)
N(10)	1.966 (7)	1.962 (7)	1.951 (20)
O(1)—O(2)	1.295 (8)	1.329 (8)	1.312 (30)
S(1)—O(3)	1.524 (9), 1.535 (19)	1.527 (6)	1.483 (20)
O(4)	1.440 (10), 1.465 (23)	1.469 (6)	1.421 (20)
O(5)	1.479 (10), 1.419 (22)	1.472 (6)	1.434 (20)
O(6)	1.462 (7)	1.461 (6)	1.431 (20)
S(2)—O(7)	1.563 (7)	1.559 (6)	1.563 (20)
O(8)	1.449 (7)	1.445 (6)	1.428 (20)
O(9)	1.452 (6)	1.462 (6)	1.448 (20)
O(10)	1.453 (6)	1.464 (6)	1.479 (20)
S(3)—O(11)	1.560 (8)	1.582 (6)	1.535 (20)
O(12)	1.472 (7)	1.470 (6)	1.478 (20)
O(13)	1.425 (7)	1.448 (6)	1.442 (20)
O(14)	1.441 (7)	1.457 (6)	1.456 (20)
S(4)—O(15)	1.419 (7)	1.446 (6)	1.461 (20)
O(16)	1.448 (7)	1.433 (6)	1.438 (20)
O(17)	1.455 (7)	1.472 (6)	1.460 (20)
O(18)	1.586 (8)	1.580 (6)	1.566 (20)

Average distances

Co—O	1.918 (6)	1.913 (6)	1.894 (20)
Co—N	1.956 (13)	1.953 (9)	1.946 (20)
S—O	1.449 (15)	1.459 (14)	1.448 (19)
S—OH	1.570 (14)	1.574 (13)	1.555 (17)

and Co—N bond distances are about the same in the two new structures and half to a third of those estimated for the film-data determination. The average distances are also nearly the same, although their

scatter does not decrease much, perhaps reflecting the fact that each N atom is hydrogen bonded differently and so the ten Co—N distances and the 12 S—O distances may not be chemically equivalent in the crystal. The axial Co—N distances are not significantly different from the equatorial distances.

The sulfate and hydrogensulfate groups

In the original determination, we represented O(3), O(4), and O(5) as single atoms with extremely large anisotropic motions. The better-quality diffractometer data allowed us to resolve these atoms at room temperature into two sets of atoms, one set of positions (*A*) occupied 72.4% of the time and the other (*B*) 27.6% of the time. At 17 K the atoms occupy only the *A* sites. The geometries of the sulfate and hydrogensulfate ions are similar, with three normal S—O bonds and one longer one; the longer one bears the H atom of the hydrogensulfate groups and, in the sulfate ion, it accepts two strong hydrogen bonds. The angles are all normal, with those involving the long S—O bond systematically smaller than the rest [av. 106.3 (16) vs 112.4 (20)°]; this presumably reflects the equalization of the non-bonded O—O distances in the ions (Donohue, 1965).

The hydrogen bonding

The locations for the H atoms are based on difference maps calculated in the planes in which the H atoms are expected to lie. These maps (especially for the room-temperature structure) were not always unequivocal, and the H positions in the two structures show some differences. We are pleased to note that in all cases but one [H(2) of N(6)], the H atom positions we chose from the difference maps lead to the same hydrogen-bonding pattern in both structures, albeit the distances and angles involving the H atoms are not identical. The hydrogen-bonding pattern is summarized in Table 3. It is quite possible that with so many acceptor atoms in the structure and with three protons on each N donor atom, individual hydrogen bonds in the classic sense are not energetically favorable enough and the ammine groups must simply orient themselves in some overall electrostatic field. In this connection, one should note that there is also a 'gearing' effect in the cation which does not allow each ammine group complete freedom of orientation. The problem is complicated and Table 3 may oversimplify the situation.

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Table 3. *The hydrogen bonding*

	To	Room temperature			Low temperature		
		N...O (Å)	H...O (Å)	N—H ...O (°)	N...O (Å)	H...O (Å)	N—H ...O (°)
N(1)							
H(11)	O(8)	2.90 (1)	1.99	159	2.91 (1)	2.01	161
H(21)	O(16) ⁵	2.89 (1)	2.05	146	2.92 (1)	2.01	158
H(31)	O(14) ⁶	2.90 (1)	2.04	150	2.87 (1)	2.08	138
N(2)							
H(12)	O(15) ³	3.07 (1)	2.13	168	3.01 (1)	2.33	128
H(22)	O(17) ⁵	3.15 (1)	2.37	138	3.09 (1)	2.17	166
H(32)	O(9)	2.97 (1)	2.30	128	2.98 (1)	2.05	162
N(3)							
H(13)	O(2)	2.88 (1)	2.42	109	2.88 (1)	2.25	122
H(23)*	O(3B) ¹	2.80 (2)	1.95	148	3.21 (1)	1.81	107
H(33)	O(12) ⁶	2.95 (1)	2.76	92	3.17 (1)	2.32	148
N(4)							
H(14)	O(10) ¹	2.98 (1)	2.05	166	2.93 (1)	2.00	165
H(24)	O(17) ⁵	2.95 (1)	2.10	146	2.90 (1)	2.03	149
H(34)	O(8) ²	2.99 (1)	2.11	153	2.95 (1)	2.07	152
N(5)							
H(15)	O(18) ³	2.92 (1)	2.03	151	2.89 (1)	2.09	141
H(25)	O(3A) ¹	2.91 (1)	2.07	146	2.93 (1)	2.39	116
H(35)*	O(4B) ¹	3.09 (2)	2.22	152	2.98 (1)	2.02	173
N(6)							
H(16)	O(13)	3.10 (1)	2.17	164	3.05 (1)	2.53	115
H(26)	O(9) ⁴	3.04 (1)	2.25	140	3.03 (1)	2.92	87
H(36)	O(11) ⁶	3.46 (1)	2.76	132	3.37 (1)	2.43	165
H(36)	O(4A) ¹	2.94 (1)	2.10	150	2.92 (1)	2.17	134
N(7)							
H(17)	O(13)	3.09 (1)	2.17	158	3.02 (1)	2.20	144
H(27)	O(5A) ¹	2.99 (1)	2.18	145	2.95 (1)	2.05	157
H(37)	O(17) ³	3.03 (1)	2.17	152	2.97 (1)	2.02	170
N(8)							
H(18)	O(7)	2.96 (1)	2.17	140	2.99 (1)	2.17	142
H(28)	O(1)	2.98 (1)	2.48	112	2.96 (1)	2.47	111
H(38)	O(9) ⁴	3.09 (1)	2.26	124	3.03 (1)	2.12	160
N(9)							
H(19)	O(5A) ³	2.90 (1)	2.04	150	2.88 (1)	1.93	173
H(29)	O(6)	2.90 (1)	1.96	165	2.92 (1)	2.01	159
H(39)	O(9) ⁴	3.18 (1)	2.31	151	3.13 (1)	2.52	123
N(10)							
H(110)	O(17) ³	3.14 (1)	2.64	114	3.08 (1)	2.13	170
H(210)	O(15) ³	3.15 (1)	2.26	154	3.04 (1)	2.72	100
H(310)	O(7)	3.01 (1)	2.19	144	2.99 (1)	2.37	122
O(7)							
H(O7)	O(3A)	2.55 (1)	1.87	123	2.51 (1)	1.46	169
	O(3B)	2.51 (2)	1.61	149			
O(11)							
H(O11)	O(3A) ³	2.55 (1)	1.70	143	2.55 (1)	1.72	144
O(18)							
H(O18)	O(12)	2.58 (1)	1.67	154	2.60 (1)	1.59	153

Symmetry relations: the hydrogen bond is between the O atom at x, y, z and the N atom at: (1) $x, y, -1 + z$; (2) $-\frac{1}{2} - x, -y, -\frac{1}{2} + z$; (3) $\frac{1}{2} - x, -y, -\frac{1}{2} + z$; (4) $-x, -\frac{1}{2} + y, \frac{1}{2} - z$; (5) $-x, \frac{1}{2} + y, \frac{1}{2} - z$; (6) $\frac{1}{2} + x, \frac{1}{2} - y, 2 - z$.

* In the low-temperature structure, the hydrogen bond is to the O atom in the *A* position.

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