

The Crystal Structure of L-Cysteic Acid Monohydrate

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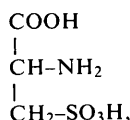
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(Received 6 February 1970)

The structure of L-cysteic acid monohydrate ($C_3H_7NO_5S \cdot H_2O$) has been determined by an X-ray diffraction analysis of a crystal in space group $P2_12_12_1$ having 4 molecules in a unit cell of dimensions $a = 6.927 \pm 1$, $b = 19.027 \pm 3$, $c = 5.305 \pm 1$ Å; density $D_x = 1.77$, $D_{meas} = 1.75 \text{ g.cm}^{-3}$. The intensity data were measured on an automatic diffractometer, phases were determined by the symbolic addition procedure and the structure was refined to an R of 6.7% by the method of least squares. The dimensions and conformation of the molecule are comparable to those of L-cysteine and taurine. The water of hydration is in the orientation determined by proton magnetic resonance. The molecules are packed in layers in the crystal and are held together by hydrogen bonding and ionic interactions.

Introduction

L-Cysteic acid, with structural formula



is a naturally occurring amino acid, although not one which is normally incorporated into proteins. It does occur in the part of a sheep's fleece exposed to light and weather and is an intermediate in the metabolic degradation of L-cysteine. It is also commonly used in the analysis of the amino-acid composition of proteins through the chromatographic identification of oxidized cysteine and cystine (Greenstein & Winitz, 1961).

Preliminary crystallographic work on cysteic acid, including proton magnetic resonance measurements on the proton-proton vectors of the water of hydration, has been reported (El Saffar, Hendrickson & Koski, 1969). The structures of the closely related compounds L-cysteine and taurine, decarboxylated cysteic acid, have also been reported (Ramachandra Ayyar, 1968; Harding & Long, 1968; Sutherland & Young, 1963; Okaya, 1966).

Experimental

A crystal from the same batch as those described by El Saffar *et al.* (1969) was mounted along the c axis for data collection on a Picker automated four-circle diffractometer. Measurements of 2θ values were fitted by a least-squares procedure to yield the lattice parameters and associated standard deviations which are listed in Table 1 together with other physical data. These cell constants differ from those based on film data (El Saffar *et al.*, 1969) by 3–5%. All the accessible intensity data ($\theta_{\max} = 66.25^\circ$) were measured by θ - 2θ scans of $2^\circ + 2\theta(\alpha_2) - 2\theta(\alpha_1)$ at 2 deg.min^{-1} with 10

sec background counts before and after each peak. Lorentz and polarization corrections were applied to the net counts and normalized structure factors $|E|$ and structure factors $|F|$ were derived on an approximately absolute scale from a K -curve (Karle & Hauptman, 1953) of the data. No correction for absorption is included in the results cited below. The effect of an absorption correction was not greater than $\frac{1}{3}\sigma$ in bond lengths and angles nor greater than $\frac{1}{2}\sigma$ in thermal parameters, excepting B_{33} values which were decreased by by 0.18 \AA^2 on the average.

Table 1. *Physical data*

Molecular formula	$C_3H_7O_5NS \cdot H_2O$
Molecular weight	187.17
Habit	Prismatic c
Crystal size	$0.08 \times 0.11 \times 0.21 \text{ mm}$
Space group	$P2_12_12_1$
Z	4 molecules/unit cell
a	$6.927 \pm 0.001 \text{ \AA}$
b	19.027 ± 0.003
c	5.305 ± 0.001
V	699.20 \AA^3
Density, flotation*	1.75 g.cm^{-3}
Density, calculated	1.77
Radiation	$\text{Cu } K\alpha$, 1.5418 \AA ; Ni-filtered
Linear absorption coefficient, μ	40.2 cm^{-1}
Temperature	22°C , ambient
Number of independent reflections	741

* El Saffar, Hendrickson & Koski (1969).

Structure analysis

The phases for the structure factor amplitudes were determined by an application of the symbolic addition procedure for non-centrosymmetric crystals (Karle & Karle, 1966). The reflections which were assigned phases to specify an origin and enantiomorph are listed in Table 2 along with those given symbolic assignments. Other phase indications were obtained by hand from these by using the relationship

$$\varphi_h = \langle \varphi_k + \varphi_{h-k} \rangle_k,$$

with interacting reflections restricted to those with

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$|E| > 1.75$. The recurring indications that $b = a$ and $c = a + \pi/2$ left two possible phase-sets depending on the value of a . Each of these sets of 48 phases was then gradually expanded with the tangent formula (Karle & Hauptman, 1956; program of Brenner & Gum, 1968) to generate phases for the 275 reflections with $|E| > 1.0$. The seven major peaks in an E -map synthesized from the set following from $a = -\pi/2$ were interpretable as atoms of the structure. Phases calculated from this partial structure were applied to reflections with $|E| > 1.4$ for which $|F|$ calculated at least 0.25 of the observed value. These reflections were then entered into the tangent formula for refinement of the partial structure phases and extension to all reflections with $|E| > 1.0$ (Karle, 1968). The four remaining non-hydrogen atoms were revealed in the succeeding E -map. The structure resulting from this phase determination proved to be opposite to the stereoconfiguration known for L-amino acids. The enantiomorphous structure was produced by inversion through the (001) plane and as a consequence is referred to an origin different from that specified by the phases in Table 2.

Table 2. Reflections used to initiate the phase determination

h	φ_h	$ E_h $
0 2 5	0	2.32
7 0 3	$+\pi/2$	2.08
2 3 0	0	2.07
0 11 1	$+\pi/2$	2.46
0 1 6	$a (\pm \pi/2)$	2.41
2 2 4	b	2.17
3 1 1	c	2.09

The structure was refined by full-matrix least-squares minimization of $\sum w(|F_o| - |F_c|)^2$ using the Busing, Martin & Levy (1962) program with scattering factors as listed in *International Tables for X-ray Crystallography* (1962). No allowance was made in the refinement for the small effect of anomalous dispersion. After three cycles of isotropic refinement using unit weights, the R value was reduced to 0.105. Inspection of the $(|F_o| - |F_c|)$ discrepancies at this point showed systematic deviations attributable to extinction for reflections with $|F_o| > 80$ and atypically large discrepancies for reflections with $h = 7, 8$ and $k \leq 4$. The validity of the measurements for this latter class of reflections was already suspect since reflections 740, 741, 820 and 821 had skewed peak profiles extending outside the scan range and had been eliminated from the data set. Tests indicated that neither stray radiation, additional crystal fragments nor instrumental failure was responsible for the discrepant observations. Although still unexplained, the 23 reflections with $h \geq 7$ and $k \leq 4$ were given zero weight in ensuing cycles of refinement as were the 7 reflections with $|F_o| > 80$. Four of the hydrogen atoms were located with confidence in a difference map following a cycle of anisotropic refinement and the other five were found in a second difference map computed after including these as constant contributors in another cycle of refinement. Hydrogen atoms were given the isotropic temperature factors of the atoms to which they were attached and positional parameters were varied in subsequent refinement. A plot of $(\Delta F)^2$ vs $|F_o|$ was used to establish the final weighting scheme: $w = (6 - |F_o|)^{-1}$, $|F_o| < 5$; $w = 1$, $5 \leq$

Table 3. Final atomic parameters†

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
S	0.8527	0.3906	-0.0298	1.27	1.91	1.40	-0.06	0.13	-0.01
O(1)	0.3893	0.3127	0.1788	2.85	2.46	1.78	-0.28	0.34	0.27
O(2)	0.3257	0.4194	0.3346	2.55	2.68	1.53	0.13	0.67	-0.24
O(3)	0.8844	0.4663	-0.0547	3.16	2.35	2.48	-0.50	-0.31	-0.09
O(4)	0.8199	0.3726	0.2328	3.17	4.01	1.51	-0.69	-0.24	0.90
O(5)	0.9982	0.3501	-0.1569	1.92	4.44	3.86	1.04	0.39	-1.13
N	0.4787	0.4868	-0.0567	2.07	2.55	2.93	0.78	0.48	1.06
C(1)	0.3838	0.3802	0.1690	0.93	2.53	1.95	0.14	-0.36	0.43
C(2)	0.4536	0.4089	-0.0802	1.44	2.74	1.87	0.05	-0.08	0.64
C(3)	0.6323	0.3736	-0.1935	2.20	3.03	1.13	-0.20	-0.06	-0.18
O(W)	0.2624	0.2653	0.5909	4.43	2.77	3.21	0.50	1.82	0.41
H(1, C2)	0.337	0.401	-0.161						
H(2, C3)	0.623	0.320	-0.218						
H(3, C3)	0.654	0.384	-0.402						
H(4, N)	0.364	0.507	-0.031						
H(5, N)	0.525	0.500	0.050						
H(6, N)	0.574	0.507	-0.183						
H(7, O1)	0.333	0.299	0.288						
H(8, W)	0.308	0.224	0.585						
H(9, W)	0.187	0.283	0.590						
Standard deviations									
S	0.0002	0.0001	0.0003	0.06	0.05	0.05	0.04	0.05	0.04
O(1)-O(5)	0.0007	0.0002	0.0009	0.22	0.19	0.20	0.17	0.18	0.16
N, C, O(W)	0.0009	0.0003	0.0011	0.25	0.23	0.24	0.20	0.21	0.20
H	0.014	0.004	0.018						

† Positional parameters are in fractions of a cell edge and temperature factors are of the form $\exp[-\frac{1}{4}(h^2a^2B_{11} + k^2b^2B_{22} + l^2c^2B_{33} + 2hka^*b^*B_{12} + 2hla^*c^*B_{13} + 2klb^*c^*B_{23})]$.

Table 4. Observed and calculated structure factors

Successive columns contain k , $|F_{\text{obs}}| \times 10$, $|F_{\text{calc}}| \times 10$ and $\varphi_{\text{calc}}(\text{cycles}) \times 100$. The $|F_{\text{calc}}|$ contain a factor of 1.194 to bring them to the empirical absolute scale of the $|F_{\text{obs}}|$. Reflections given zero weight in the refinement are designated by asterisks.

0	k	0	87	96	0	13 494	486 75	100 104	82	12 126	123	0	10 183	175	88	20 188	195	25	18	147	142	30	2 234	235	75	12 214	211	0	2 128	131	42		
2	362	376	0	0	0	0	157 150	75	26	37 25	10	210	209	55	11	67	28	32	0	15	25	25	3 371	373	82	13 137	134	25	13 127	123	86		
4	922	1177	504	0	0	0	14 72	74	75	10	184	171	42	10	184	171	42	0	15	25	25	4	3 371	373	82	13 137	134	25	13 127	123	86		
6	244	245	50	0	0	0	11 139	133	25	16 215	215	25	11 230	217	73	15 210	205	50	10	184	171	42	5 107	103	78	0	42	48	50	6	307	294	25
8	812	651	64	0	0	0	10 184	171	42	17 86	48	0	12 238	353	41	16 76	86	56	14	137	137	0	4	4	4	4	4	4	4	4	4	4	
10	217	237	25	0	0	0	11 217	207	205	11 300	386	0	17 86	93	50	15 101	96	91	3	189	203	2	7 189	189	2	2 160	168	59	2 160	168	59		
12	170	169	50	0	0	0	14 169	159	50	19 287	288	25	16 143	149	30	16 07	67	50	16	135	145	39	8 101	102	78	3 161	156	80	3 161	156	80		
14	352	335	50	0	0	0	14 46	36	25	20 98	98	75	14 110	113	12	19 48	47	0	17	92	95	12	5 347	354	45	0	83	84	40	4 177	180	07	
16	76	73	0	0	0	0	17 120	119	0	21 111	111	0	18 213	227	90	20 43	78	0	18	110	113	83	6 223	230	83	6 130	136	16	6 130	136	16		
18	57	46	0	0	0	0	17 170	174	75	22 21	07	75	17 155	154	44	21 62	64	0	17	92	95	12	7 66	58	48	11 144	141	45	6 137	138	6		
20	64	67	50	0	0	0	18 34	32	50	23 72	74	85	18 72	74	85	2	0	0	18	110	113	83	8 325	326	67	12 102	107	88	7 157	151	19		
22	69	71	50	0	0	0	18 61	64	25	24 01	04	25	19 04	07	75	1	142	124	12	0	0	0	9 621	116	07	13 123	119	88	11 204	202	25		
0	k	0	0	0	0	0	2 025	205	00	1	k	0	1 558	594	28	2 441	441	26	10	293	289	29	14	36	44	80	123	127	80	14	106	106	25
2	123	131	0	0	0	0	0 184	171	42	3 476	474	84	3 003	307	08	4 213	216	1	12	162	175	35	1	k	0	11 175	124	38	14	0	17	25	
4	211	196	254	0	0	0	7 131	133	8	4 241	240	43	4 241	250	31	5 142	149	0	13	160	166	47	0	k	0	13 173	133	05	17	0	0	0	
6	432	430	75	0	0	0	1 18	14	75	5 343	337	3	5 289	291	85	6 168	176	71	14	142	145	86	1	k	0	11 144	141	45	5	177	177	28	
8	28	22	0	0	0	0	6 38	20	25	6 161	170	85	6 217	222	4	7 216	210	61	19	77	83	94	3	35	37	11	15 176	167	11	1	160	160	21
10	212	212	50	0	0	0	7 26	19	75	6 190	154	76	8 209	203	81	9 51	80	55	19	77	83	94	3	35	37	11	15 176	167	11	1	160	160	21
12	109	105	25	0	0	0	7 88	85	25	9 202	203	12	8 209	203	81	9 51	80	55	19	77	83	94	3	35	37	11	15 176	167	11	1	160	160	21
14	192	192	50	0	0	0	9 109	105	25	10 342	326	88	9 109	105	25	10 314	310	17	11	206	207	7	0	0	0	6 05	70	90	18	114	115	51	
16	172	172	50	0	0	0	11 152	151	0	11 244	244	21	12 233	235	23	13 94	86	21	3	1	2	0	8 147	151	18	0	266	266	25	7	4	25	0
18	120	120	0	0	0	0	12 150	150	0	12 134	134	45	13 144	144	13	14 246	246	80	10	125	125	93	3	1	2	0	8 147	151	18	0	266	266	25
20	202	202	50	0	0	0	13 150	150	0	13 134	134	45	14 144	144	13	15 150	150	0	10	125	125	93	3	1	2	0	8 147	151	18	0	266	266	25
22	167	167	50	0	0	0	14 152	152	0	14 134	134	45	15 144	144	13	16 150	150	0	10	125	125	93	3	1	2	0	8 147	151	18	0	266	266	25
24	148	148	75	0	0	0	15 112	112	0	15 104	104	0	16 112	112	0	17 112	112	0	10	125	125	93	3	1	2	0	8 147	151	18	0	266	266	25
26	121	121	0	0	0	0	16 102	97	0	17 40	41	74	18 97	108	00	19 157	154	97	10	125	125	93	3	1	2	0	8 147	151	18	0	266	266	25
28	60	54	25	0	0	0	18 60	54	25	20 72	68	50	21 97	58	77	22 95	62	84	0	12	124	124	74	0	0	0	10 100	100	00	11	74	75	75
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0	k	0	0	0	0	0	0 213	210	50	0 283	284	50	0 120	101	0	0 283	284	50	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0
2	171	171	0	0	0	0	1 016	143	254	2 005	470	87	3 253	366	96	4 140	450	24	5 190	203	161	6 148	450	24	7 189	203	2	8 140	450	24	9 202	203	12
4	486	484	0	0	0	0	3 283	242	25	4 163	177	0	5 130	165	0	6 422	483	60	7 251	203	161	8 148	450	24	9 202	203	12	10 148	450	24	11 167	167	16
6	430	437	75	0	0	0	4 266	295	50	5 144	166	25	6 292	242	88	7 189	203	12	8 148	450	24	9 202	203	12	10 148	450	24	11 167	167	16	12 167	167	16
8	247	235	0	0	0	0	5 152	152	25	6 247	235	0	7 189	203	12	8 148	450	24	9 202	203	12	10 148	450	24	11 167	167	16	12 167	167	16	13 167	167	16
10	9	9	75	0	0	0	10 231	234	0	11 184	184	25	12 184	184	25	13 184	184	25	14 184	184	25	15 184	184	25	16 184	184	25	17 184	184	25	18 184	184	25
12	148	148	50	0	0	0	13 148	148	50	14 148	148	50	15 148	148	50	16 148	148	50	17 148	148	50	18 148	148	50	19 148	148	50	20 148	148	50	21 148	148	50
14	322	310	50	0	0	0	14 322	310	50	15 322	310	50	16 322	310	50	17 322	310	50	18 322	310	50	19 322	310	50	20 322	310	50	21 322	310	50	22 322	310	50
16	181	180	50	0	0	0	15 181	180	50	16 181	180	50	17 181	180	50	18 181	180	50	19 181	180	50	20 181	180	50	21 181	180	50	22 181	180	50	23 181	180	50
18	107	106	25	0	0	0	16 107	106	25	17 107	106	25	18 107	106	25	19 107	106	25	20 107	106	25	21 107	106	25	22 107	106	25	23 107	106	25	24 107	106	25
20	177	181	25	0	0	0	17 177	181	25	18 177	181	25	19 177	181	25	20 177	181	25	21 177	181	25	22 177	181	25	23 177	181	25	24 177	181	25	25 177	181	25
22	123	123	0	0	0	0	18 123	123	0	19 123	123	0	20 123	123	0	21 123	123	0	22 123	123	0	23 123	123	0	24 123	123	0	25 123	123	0	26 123	123	0
24	186	186	75	0	0	0	19 186	186	75	20 186	186	75	21 186	186	75	22 186	186	75	23 186	186	75	24 186	186	75	25 186	186	75	26 186	186	75	27 186	186	75
26	547	550	50	0	0	0	20 547	550	50	21 547	550	50	22 547	550	50	23 547	550	50	24 547	550	50	25 547	550	50	26 547	550	50	27 547	550	50	28 547	550	50
28	103	103	25	0	0	0	21 103	103	25	22 103	103	25	23 103	103	25	24 103	103	25	25 103	103	25	26 103	103	25	27 103	103	25	28 103	103	25	29 103	103	25
30	485	486	25	0	0	0	22 485	486	25	23 485	486	25	24 485	486	25	25 485	486	25	26 485	486	25	27 485	486	25	28 485	486	25	29 485	486	25	30 485	486	25
32	28	28	0	0	0	0	23																										

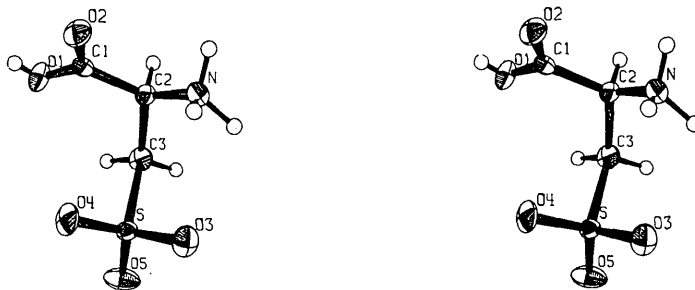


Fig. 1. Stereodrawing of the L-cysteic acid molecule. The Figure was drawn by computer with a program due to Johnson (1965).

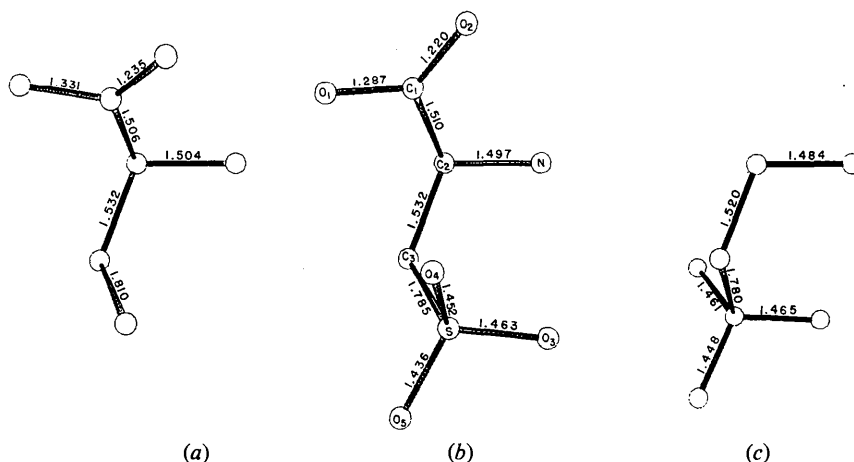


Fig. 2. Comparison of (a) L-cysteine, (b) L-cysteic acid and (c) taurine. The configuration and C-O bond lengths of L-cysteine are taken from Ramachandra Ayyar (1968); other L-cysteine bond lengths are averaged from three molecules (Ramachandra Ayyar, 1968; Harding & Long, 1968). The configurations and bond lengths of taurine and L-cysteic acid are taken respectively from Okaya (1966) and from this work. The molecules were drawn by computer (Johnson, 1965) with the N-C(2)-C(3) plane of each in the same orientation.

Table 5. *Molecular dimensions*

Bond lengths ($\sigma=0.005-8$)		Bond angles ($\sigma=0.3-5$)	
C(1)-O(1)	1.287 Å	C(2)-C(1)-O(1)	112.7°
C(1)-O(2)	1.220	C(2)-C(1)-O(2)	121.0
C(2)-N	1.497	O(1)-C(1)-O(2)	126.2
C(2)-C(1)	1.510	C(1)-C(2)-C(3)	116.3
C(2)-C(3)	1.532	C(1)-C(2)-N	108.8
S-C(3)	1.785	C(3)-C(2)-N	111.9
S-O(3)	1.463	C(2)-C(3)-S	114.9
S-O(4)	1.452	C(3)-S-O(3)	105.2
S-O(5)	1.436	C(3)-S-O(4)	106.9
		C(3)-S-O(5)	105.9
		O(3)-S-O(5)	110.0
		O(4)-S-O(5)	115.7
		O(5)-S-O(3)	112.3
Average lengths to hydrogen atoms		Torsion angles*	
C-H	1.03	ψ_1	190.0
N-H	0.87	ψ_2	12.6
O-H	0.73	χ_1	55.4
		χ_2	292.2

* The torsion angles follow the conventions defined by Lakshminarayanan, Sasisekharan & Ramachandran (1967) with χ_2 referring to O(3).

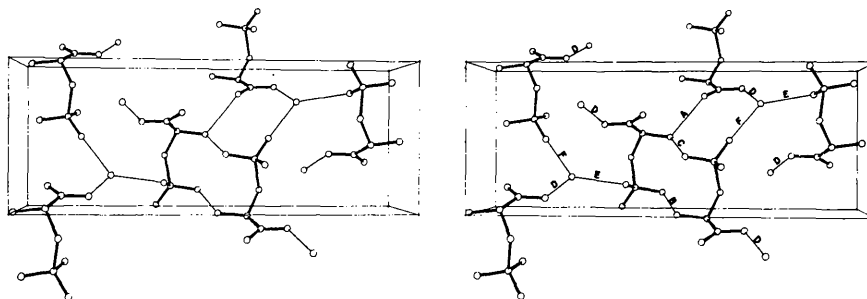


Fig. 3. Stereographic packing diagram. The unit cell is oriented with *b* across, *a* down and *c* out of the page. Hydrogen bonds are designated by the letters assigned in Table 6. The Figure was drawn by the computer program of Johnson (1965).

Table 6. Hydrogen bond lengths

Bond	Symmetry operation on acceptor atom	Length	X-H...O angle
A N-H...O(2)	$\frac{1}{2}-x$ $1-y$ $-\frac{1}{2}+z$	2.823 Å	144.1°
B N-H...O(3)	$1\frac{1}{2}-x$ $1-y$ $-\frac{1}{2}+z$	2.946	167.0
C N-H...O(3')	$1\frac{1}{2}-x$ $1-y$ $\frac{1}{2}+z$	2.965	144.6
D O(1)-H...O(W)	x y z	2.523	164.3
E O(W)-H...O(4)	$-\frac{1}{2}+x$ $\frac{1}{2}-y$ $1-z$	2.813	144.8
F O(W)-H...O(5)	$-1+x$ y $1+z$	2.783	142.2
Configuration about hydrogen donors			
(C-N).A	C(2)—N...O(2)	121.5°	
(C-N).B	C(2)—N...O(3)	105.3	
(C-N).C	C(2)—N...O(3')	114.2	
A.B	O(2)...N...O(3)	97.7	
A.C	O(2)...N...O(3')	103.4	
B.C	O(3)...N...O(3')	127.7	
(C-O).D	C(1)—O(1)...O(W)	111.2	
D.E	O(1)...W...O(4)	124.9	
D.F	O(1)...W...O(5)	116.0	
E.F	O(4)...W...O(5)	118.3	

molecule. All oxygen atoms and the nitrogen atom participate in hydrogen bonding (Table 6). This layer structure is no doubt responsible for the cleavage plane parallel to (010) noted by El Saffar *et al.* (1969).

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