

- (a) Modified for the CDC 1604A by the authors.
 (b) Modified for the IBM 360/75 by G. Brunton of this Laboratory.
 (c) Modified for the IBM 360/75 by C. K. Johnson.

The multiple implication and superposition diagrams were prepared using programs written during a previous study; the functions evaluated are described in the report of that work (Ellison & Levy, 1965). Pseudo-normalized structure factor magnitudes for the 'squared scattering amplitude' structure were calculated using programs written by the authors for this investigation. Analysis of the thermal motion made extensive use of a program written by Johnson (1967).

References

- AMMON, H. L. (1964). *A System of Programs (PHASER and SIGNIE) for the IBM 709-7090-7094 for Application of the Symbolic Addition Procedure*. Unpublished work.
- AMMON, H. L. & JENSEN, L. H. (1967). *Acta Cryst.* **23**, 805.
- ATHERTON, N. M. & WHIFFEN, D. H. (1960). *Mol. Physics*, **3**, 1.
- BRENNER, H. (1967). *J. Colloid Interface Sci.* **23**, 407.
- BROWN, G. M. & LEVY, H. A. (1964). *J. Phys.* **25**, 497.
- BUSING, W. R. & LEVY, H. A. (1964). *Acta Cryst.* **17**, 142.
- BUSING, W. R., SMITH, H. G., PETERSON, S. W. & LEVY, H. A. (1964). *J. Phys.* **25**, 495.
- ELLISON, R. D. & LEVY, H. A. (1965). *Acta Cryst.* **19**, 260.
- ELLISON, R. D. & LEVY, H. A. (1967). Chemistry Division Annual Progress Report, Oak Ridge National Laboratory, Report No. ORNL-4164, p. 126.
- ELLISON, R. D. & LEVY, H. A. (1968). Chemistry Division Annual Progress Report, Oak Ridge National Laboratory, Report No. ORNL-4306, p. 151.
- GLUSKER, J. P., MINKIN, J. A. & PATTERSON, A. L. (1969). *Acta Cryst.* **B25**, 1066.
- GOLIČ, L. & SPEAKMAN, J. C. (1965). *J. Chem. Soc.* p. 2521.
- GUPTA, M. P. & GUPTA, N. P. (1968). *Acta Cryst.* **B24**, 631.
- JACOBSON, R. A., WUNDERLICH, J. A. & LIPSCOMB, W. N. (1961). *Acta Cryst.* **14**, 598.
- JOHNSON, C. K. (1966). Amer. Cryst. Assn. Program and Abstracts of Annual Meeting, Austin, Texas. Abstract K-9, p. 49; Chemistry Division Annual Progress Report, Oak Ridge National Laboratory, Report No. ORNL-3994, p. 125.
- JOHNSON, C. K. (1967). Chemistry Division Annual Progress Report, Oak Ridge National Laboratory, Report No. ORNL-4164, p. 127.
- KARLE, J. (1966). *Acta Cryst.* **20**, 881.
- KARLE, J. & KARLE, I. L. (1966). *Acta Cryst.* **21**, 849.
- LEVY, H. A. & ELLISON, R. D. (1967). Amer. Cryst. Assoc. Program and Abstracts of Winter Meeting, Atlanta, Georgia. Abstract A9, p. 21.
- OKAYA, Y., STEMPLE, N. R. & KAY, M. I. (1966). *Acta Cryst.* **21**, 237.
- PIJPER, W. P. (1971). *Acta Cryst.* **B20**, 344.
- SCHACHTSCHNEIDER, J. H. (1962). *Vibrational Analysis of Polyatomic Molecules*. III. *Vibrational Secular Equation Programs*. Shell Development Co. Tech. Report 263-62.
- SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* **B24**, 63.
- SHIMANOUCI, T. & SUZUKI, I. (1962). *J. Mol. Spectry.* **8**, 222.
- SHOEMAKER, D. P. (1966). *World List of Crystallographic Computer Programs*, 2nd Edition. Utrecht: Oosthoek.
- VAN DER HELM, D., GLUSKER, J. P., JOHNSON, C. K., MINKIN, J. A., BUROW, N. E. & PATTERSON, A. L. (1968). *Acta Cryst.* **B24**, 578.
- VAN EIJCK, B. P., KANTERS, J. A. & KROON, J. (1965). *Acta Cryst.* **19**, 435.
- WILSON, A. J. C. (1942). *Nature, Lond.* **150**, 152.
- WOOLFSON, M. M. (1956). *Acta Cryst.* **9**, 804.
- YAKEL, H. L. (1964). *ORSTAT, A Fortran Program for the Statistical Analysis of Diffraction Data*, Oak Ridge National Laboratory Report, ORNL-TM-750.
- ZACHARIASEN, W. H. (1965). *Trans. Amer. Cryst. Assoc.* **1**, 33.

Acta Cryst. (1971). **B27**, 344

The Molecular and Crystal Structure of Glycollic Acid

By W. P. PIJPER

Laboratorium voor Kristalchemie, Rijksuniversiteit, Catharijnesingel 51, Utrecht, The Netherlands

(Received 3 March 1970)

The structure of glycollic acid has been determined by X-ray analysis, by a direct method. The structure crystallizes in the space group $P2_1/c$ with unit-cell dimensions: $a=9.061$, $b=10.562$, $c=7.828$ Å and $\beta=116.41^\circ$, with $Z=8$. The structure refined to $R=0.041$. The carboxylic-hydroxylic hydrogen bonding, in which the alcoholic hydroxylic oxygen atom serves as an acceptor, is unusual; the expected dimers were not found.

Introduction

Glycollic acid has been examined as part of a programme of investigation of carboxylic acids. The prob-

lem was whether glycollic acid in the crystal structure was likely to form dimers, probably with non-hydrogen-bonded hydroxyl groups, or a structure with hydroxyl-carbonyl coupling. An infrared analysis gave no reason to assume that there might be non-hydrogen-bonded hydroxyl groups in the structure (Kanters, Kroon, Peerdeman & Vliegthart, 1969), and the

Present address: Netherlands Foundation for Chemical Research, Laan van Meerdervoort 123 A, The Hague, Netherlands.

absence of such groups was later confirmed by the X-ray analysis.

Experimental

Crystals were obtained by slow evaporation of an aqueous solution at room temperature. The extinctions, $0k0$ for k odd and $h0l$ for l odd, uniquely determined the space group $P2_1/c$. From 45 reflexions, measured on a General Electric single-crystal orienter, the unit-cell dimensions were refined by least-squares;

they are: $a=9.061$, $b=10.562$, $c=7.828$ Å and $\beta=116.41^\circ$ (estimated standard deviations 0.002 Å and 0.02°). The calculated density with 8 molecules in the unit-cell was 1.51 g.cm $^{-3}$; the density found by flotation was 1.49 g.cm $^{-3}$. The integrated intensities of 1323 independent reflexions were measured with a General Electric single-crystal orienter equipped with a scintillation counter and discriminator. Ni filtered Cu-radiation (wave length 1.5418 Å) was used and the $\theta/2\theta$ scan was applied. The data were corrected for Lorentz

Table 1. Final fractional atomic coordinates and thermal parameters

	x	y	z	β_{11}	β_{22}	β_{33}	$2\beta_{12}$	$2\beta_{23}$	$2\beta_{31}$
O(1)	-0.1315	+0.1888	+0.2785	+0.0160	+0.0053	+0.0422	-0.0033	-0.0060	+0.0345
O(2)	+0.0979	+0.1880	+0.2437	+0.0111	+0.0054	+0.0352	-0.0003	-0.0031	+0.0211
O(3)	+0.1324	+0.4443	+0.2959	+0.0109	+0.0047	+0.0247	-0.0014	+0.0013	+0.0100
O(4)	+0.6296	+0.0081	+0.3970	+0.0118	+0.0059	+0.0234	+0.0026	-0.0010	+0.0056
O(5)	+0.3910	+0.0141	+0.1374	+0.0152	+0.0076	+0.0287	+0.0032	-0.0078	-0.0026
O(6)	+0.3572	+0.2678	+0.1863	+0.0109	+0.0060	+0.0300	+0.0026	+0.0052	+0.0174
C(1)	-0.0063	+0.2444	+0.2684	+0.0100	+0.0053	+0.0187	-0.0015	-0.0019	+0.0108
C(2)	-0.0105	+0.3862	+0.2891	+0.0127	+0.0052	+0.0240	-0.0006	-0.0008	+0.0176
C(3)	+0.4972	+0.0667	+0.2723	+0.0103	+0.0060	+0.0192	+0.0008	-0.0000	+0.0125
C(4)	+0.4961	+0.2034	+0.3226	+0.0121	+0.0055	+0.0218	+0.0012	+0.0007	+0.0141
Isotropic B									
H(1)	-0.1259	+0.1029	+0.2541	+3.6 Å 2					
H(2)	+0.2038	+0.4459	+0.4094	+5.1					
H(3)	-0.1157	+0.4176	+0.1632	+2.5					
H(4)	-0.0363	+0.4081	+0.3890	+2.4					
H(5)	+0.6270	-0.0728	+0.3606	+4.0					
H(6)	+0.2826	+0.2569	+0.2168	+3.9					
H(7)	+0.5015	+0.2067	+0.4483	+2.9					
H(8)	+0.5936	+0.2448	+0.3220	+1.5					

The e.s.d.'s are

	Coordinates
Oxygen	0.0015 Å
Carbon	0.0020
Hydrogen	0.026

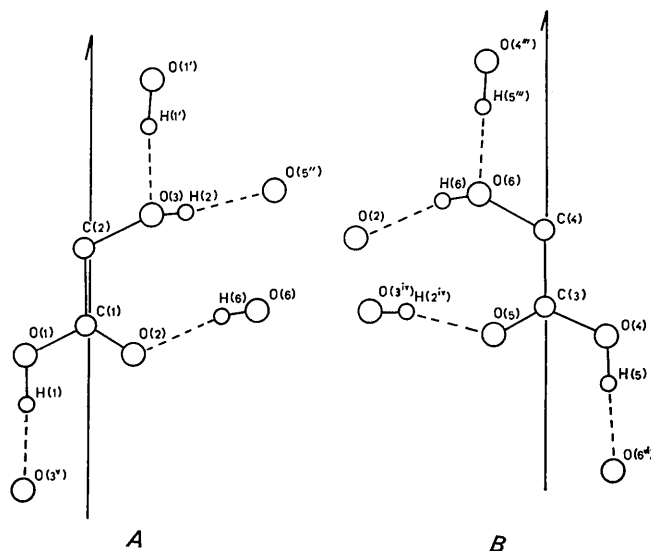


Fig. 1. The independent molecules A and B of glycolic acid with their environment. The aliphatic hydrogen atoms are omitted.

and polarization factors. Correction for absorption was neglected; the value of the linear absorption factor was 13.11 cm^{-1} , and the size of the crystal was $0.3 \times 0.2 \times 0.2 \text{ mm}$.

Structure determination

The presence of two independent molecules, *A* and *B*, in the asymmetric unit gave rise to some difficulty in the structure determination. A sharpened three-dimen-

sional Patterson synthesis yielded the positions of the oxygen atoms of the carboxylic group of molecule *A* while only the orientations of the oxygen atoms of the carboxylic group of molecule *B* could be established. Further interpretation of the Patterson synthesis was not successful. Fig. 1 shows that this was due to a combination of coincidences in the structure, giving rise to too large a number of possible interpretations.

The symbolic sign procedure was therefore applied (Beurskens, 1963): it led to several Fourier syntheses,

Table 2. Observed and calculated structure factors on absolute scale

Reflexions marked with an asterisk are apparently suffering from extinction and were omitted from the refinement. The columns contain *h, k, l, F_{obs}* and *F_{calc}*.

<i>h</i>	<i>k</i>	<i>l</i>	<i>F_{obs}</i>	<i>F_{calc}</i>	<i>h</i>	<i>k</i>	<i>l</i>	<i>F_{obs}</i>	<i>F_{calc}</i>	<i>h</i>	<i>k</i>	<i>l</i>	<i>F_{obs}</i>	<i>F_{calc}</i>	<i>h</i>	<i>k</i>	<i>l</i>	<i>F_{obs}</i>	<i>F_{calc}</i>
1	0	0	2.0	2.0	1	0	0	49.8	49.8	5	1	2	7.3	7.0	11	2	2	2.0	2.0
2	0	0	27.4	27.4	2	0	0	10.0	10.0	6	1	2	3.4	3.7	12	2	2	2.0	2.0
3	0	0	97.8	98.1	3	0	0	18.3	18.3	7	0	0	3.9	4.0	13	2	2	2.0	2.0
4	0	0	151.9	151.9	4	0	0	11.9	11.9	8	0	0	4.1	4.1	14	2	2	2.0	2.0
5	0	0	216.2	216.2	5	0	0	3.2	3.2	9	0	0	0.9	0.9	15	2	2	2.0	2.0
6	0	0	280.5	280.5	6	0	0	1.0	1.0	10	0	0	0.2	0.2	16	2	2	2.0	2.0
7	0	0	344.8	344.8	7	0	0	0.3	0.3	11	0	0	0.1	0.1	17	2	2	2.0	2.0
8	0	0	409.1	409.1	8	0	0	0.1	0.1	12	0	0	0.0	0.0	18	2	2	2.0	2.0
9	0	0	473.4	473.4	9	0	0	0.0	0.0	13	0	0	0.0	0.0	19	2	2	2.0	2.0
10	0	0	537.7	537.7	10	0	0	0.0	0.0	14	0	0	0.0	0.0	20	2	2	2.0	2.0
11	0	0	602.0	602.0	11	0	0	0.0	0.0	15	0	0	0.0	0.0	21	2	2	2.0	2.0
12	0	0	666.3	666.3	12	0	0	0.0	0.0	16	0	0	0.0	0.0	22	2	2	2.0	2.0
13	0	0	730.6	730.6	13	0	0	0.0	0.0	17	0	0	0.0	0.0	23	2	2	2.0	2.0
14	0	0	794.9	794.9	14	0	0	0.0	0.0	18	0	0	0.0	0.0	24	2	2	2.0	2.0
15	0	0	859.2	859.2	15	0	0	0.0	0.0	19	0	0	0.0	0.0	25	2	2	2.0	2.0
16	0	0	923.5	923.5	16	0	0	0.0	0.0	20	0	0	0.0	0.0	26	2	2	2.0	2.0
17	0	0	987.8	987.8	17	0	0	0.0	0.0	21	0	0	0.0	0.0	27	2	2	2.0	2.0
18	0	0	1052.1	1052.1	18	0	0	0.0	0.0	22	0	0	0.0	0.0	28	2	2	2.0	2.0
19	0	0	1116.4	1116.4	19	0	0	0.0	0.0	23	0	0	0.0	0.0	29	2	2	2.0	2.0
20	0	0	1180.7	1180.7	20	0	0	0.0	0.0	24	0	0	0.0	0.0	30	2	2	2.0	2.0
21	0	0	1245.0	1245.0	21	0	0	0.0	0.0	25	0	0	0.0	0.0	31	2	2	2.0	2.0
22	0	0	1309.3	1309.3	22	0	0	0.0	0.0	26	0	0	0.0	0.0	32	2	2	2.0	2.0
23	0	0	1373.6	1373.6	23	0	0	0.0	0.0	27	0	0	0.0	0.0	33	2	2	2.0	2.0
24	0	0	1437.9	1437.9	24	0	0	0.0	0.0	28	0	0	0.0	0.0	34	2	2	2.0	2.0
25	0	0	1502.2	1502.2	25	0	0	0.0	0.0	29	0	0	0.0	0.0	35	2	2	2.0	2.0
26	0	0	1566.5	1566.5	26	0	0	0.0	0.0	30	0	0	0.0	0.0	36	2	2	2.0	2.0
27	0	0	1630.8	1630.8	27	0	0	0.0	0.0	31	0	0	0.0	0.0	37	2	2	2.0	2.0
28	0	0	1695.1	1695.1	28	0	0	0.0	0.0	32	0	0	0.0	0.0	38	2	2	2.0	2.0
29	0	0	1759.4	1759.4	29	0	0	0.0	0.0	33	0	0	0.0	0.0	39	2	2	2.0	2.0
30	0	0	1823.7	1823.7	30	0	0	0.0	0.0	34	0	0	0.0	0.0	40	2	2	2.0	2.0
31	0	0	1888.0	1888.0	31	0	0	0.0	0.0	35	0	0	0.0	0.0	41	2	2	2.0	2.0
32	0	0	1952.3	1952.3	32	0	0	0.0	0.0	36	0	0	0.0	0.0	42	2	2	2.0	2.0
33	0	0	2016.6	2016.6	33	0	0	0.0	0.0	37	0	0	0.0	0.0	43	2	2	2.0	2.0
34	0	0	2080.9	2080.9	34	0	0	0.0	0.0	38	0	0	0.0	0.0	44	2	2	2.0	2.0
35	0	0	2145.2	2145.2	35	0	0	0.0	0.0	39	0	0	0.0	0.0	45	2	2	2.0	2.0
36	0	0	2209.5	2209.5	36	0	0	0.0	0.0	40	0	0	0.0	0.0	46	2	2	2.0	2.0
37	0	0	2273.8	2273.8	37	0	0	0.0	0.0	41	0	0	0.0	0.0	47	2	2	2.0	2.0
38	0	0	2338.1	2338.1	38	0	0	0.0	0.0	42	0	0	0.0	0.0	48	2	2	2.0	2.0
39	0	0	2402.4	2402.4	39	0	0	0.0	0.0	43	0	0	0.0	0.0	49	2	2	2.0	2.0
40	0	0	2466.7	2466.7	40	0	0	0.0	0.0	44	0	0	0.0	0.0	50	2	2	2.0	2.0
41	0	0	2531.0	2531.0	41	0	0	0.0	0.0	45	0	0	0.0	0.0	51	2	2	2.0	2.0
42	0	0	2595.3	2595.3	42	0	0	0.0	0.0	46	0	0	0.0	0.0	52	2	2	2.0	2.0
43	0	0	2659.6	2659.6	43	0	0	0.0	0.0	47	0	0	0.0	0.0	53	2	2	2.0	2.0
44	0	0	2723.9	2723.9	44	0	0	0.0	0.0	48	0	0	0.0	0.0	54	2	2	2.0	2.0
45	0	0	2788.2	2788.2	45	0	0	0.0	0.0	49	0	0	0.0	0.0	55	2	2	2.0	2.0
46	0	0	2852.5	2852.5	46	0	0	0.0	0.0	50	0	0	0.0	0.0	56	2	2	2.0	2.0
47	0	0	2916.8	2916.8	47	0	0	0.0	0.0	51	0	0	0.0	0.0	57	2	2	2.0	2.0
48	0	0	2981.1	2981.1	48	0	0	0.0	0.0	52	0	0	0.0	0.0	58	2	2	2.0	2.0
49	0	0	3045.4	3045.4	49	0	0	0.0	0.0	53	0	0	0.0	0.0	59	2	2	2.0	2.0
50	0	0	3109.7	3109.7	50	0	0	0.0	0.0	54	0	0	0.0	0.0	60	2	2	2.0	2.0
51	0	0	3174.0	3174.0	51	0	0	0.0	0.0	55	0	0	0.0	0.0	61	2	2	2.0	2.0
52	0	0	3238.3	3238.3	52	0	0	0.0	0.0	56	0	0	0.0	0.0	62	2	2	2.0	2.0
53	0	0	3302.6	3302.6	53	0	0	0.0	0.0	57	0	0	0.0	0.0	63	2	2	2.0	2.0
54	0	0	3366.9	3366.9	54	0	0	0.0	0.0	58	0	0	0.0	0.0	64	2	2	2.0	2.0
55	0	0	3431.2	3431.2	55	0	0	0.0	0.0	59	0	0	0.0	0.0	65	2	2	2.0	2.0
56	0	0	3495.5	3495.5	56	0	0	0.0	0.0	60	0	0	0.0	0.0	66	2	2	2.0	2.0
57	0	0	3559.8	3559.8	57	0	0	0.0	0.0	61	0	0	0.0	0.0	67	2	2	2.0	2.0
58	0	0	3624.1	3624.1	58	0	0	0.0	0.0	62	0	0	0.0	0.0	68	2	2	2.0	2.0
59	0	0	3688.4	3688.4	59	0	0	0.0	0.0	63	0	0	0.0	0.0	69	2	2	2.0	2.0
60	0	0	3752.7	3752.7	60	0	0	0.0	0.0	64	0	0	0.0	0.0	70	2	2	2.0	2.0
61	0	0	3817.0	3817.0	61	0	0	0.0	0.0	65	0	0	0.0	0.0	71	2	2	2.0	2.0
62	0	0	3881.3	3881.3	62	0	0	0.0	0.0	66	0	0	0.0	0.0	72	2	2	2.0	2.0
63	0	0	3945.6	3945.6	63	0	0	0.0	0.0	67	0	0	0.0	0.0	73	2	2	2.0	2.0
64	0	0	4010.0	4010.0	64	0	0	0.0	0.0	68	0	0	0.0	0.0	74	2	2	2.0	2.0
65	0	0	4074.3	4074.3	65	0	0	0.0	0.0	69	0	0	0.0	0.0	75	2	2	2.0	2.0
66	0	0	4138.6	4138.6	66	0	0	0.0	0.0	70	0	0	0.0	0.0	76	2	2	2.0	2.0
67	0	0	4203.0	4203.0	67	0	0	0.0	0.0	71	0	0	0.0	0.0	77	2	2	2.0	2.0
68	0	0	4267.3	4267.3	68	0	0	0.0	0.0	72	0	0	0.0	0.0	78	2	2	2.0	2.0
69	0	0	4331.6	4331.6	69	0	0	0.0	0.0	73	0	0	0.0	0.0	79	2	2	2.0	2.0
70	0	0	4396.0	4396.0	70	0	0	0.0	0.0	74	0	0	0.0	0.0	80	2	2	2.0	2.0
71	0	0	4460.3	4460.3	71	0	0	0.0	0.0	75	0	0	0.0	0.0	81	2	2	2.0	2.0
72	0	0	4524.6	4524.6	72	0	0	0.0	0.0	76	0	0	0.0	0.0	82	2	2	2.0	2.0
73	0	0	4589.0	4589.0	73	0	0	0.0	0.0	77	0	0	0.0	0.0	83	2	2	2.0	2.0
74	0	0	4653.3	4653.3	74	0	0	0.0	0.0	78	0	0	0.0	0.0	84	2	2	2.0	2.0
75	0	0	4717.6	4717.6	75	0	0	0.0	0.0	79	0	0	0.0	0.0	85	2	2	2.0	2.0
76	0	0	4782.0	4782.0	76	0	0	0.0	0.0	80	0	0	0.0	0.0	86	2	2	2.0	2.0
77	0																		

which could not be interpreted. Finally a direct method was employed, developed and programmed by Spek (1968, 1970). Eight sign-sets with 497 independent reflexions were obtained, one of which gave the correct solution by inspection of the Fourier syntheses. However, the correct solution was afterwards automatically obtained from these 8 sets by subjecting them to a sign reversing procedure using Sayre's equation (Krabben-dam & Kroon, 1971).

Refinement of the structure

Only the 1229 non-zero observed reflexions were used in the refinement and 6 reflexions apparently suffering from extinction were omitted. The model was refined by least squares, by means of the block-diagonal program of Palm & Peterse (1964). The atomic scattering factors were taken from the tables of Moore (1963).

The heavy-atom model was refined with anisotropic thermal parameters to $R=0.078$, in which R was defined as:

$$R = \frac{\sum ||F_{\text{obs}}| - |F_{\text{calc}}||}{\sum |F_{\text{obs}}|}$$

A difference Fourier synthesis clearly revealed the positions of the hydrogen atoms. The refinement was continued for all the atomic positions and anisotropic thermal parameters for the C and O atoms, as well as isotropic thermal parameters for the H atoms. The initial values of the isotropic thermal parameters of the H atoms were obtained from the peak densities in the difference Fourier synthesis, according to McDonald (1956). The final R value was 0.041. No weighting scheme was applied. The final fractional atomic coordinates, their e.s.d.'s, as calculated by least-squares, and thermal parameters are given in Table 1. In the

Table 3. Bond lengths and bond angles

E.s.d.'s are given in parentheses. Numbering of the atoms as in Fig. 1.

<i>d</i>		<i>d</i>	
O(1)–C(1)	1.311 (2) Å	H(7)–C(4)	0.964 (28) Å
O(2)–C(1)	1.202 (3)	H(8)–C(4)	0.987 (25)
O(3)–C(2)	1.412 (2)	O(1)–H(1)---O(3 ^v)	2.646 (2)
C(1)–C(2)	1.508 (3)	O(1)–H(1)	0.934 (25)
O(4)–C(3)	1.317 (2)	O(4)–H(5)---O(6 ^{vi})	2.637 (2)
O(5)–C(3)	1.202 (3)	O(4)–H(5)	0.898 (25)
O(6)–C(4)	1.411 (2)	O(6)–H(6)---O(2)	2.715 (2)
C(3)–C(4)	1.498 (3)	O(6)–H(6)	0.819 (26)
H(3)–C(2)	1.075 (25)	O(3)–H(2)---O(5 ⁱⁱ)	2.694 (2)
H(4)–C(2)	0.940 (28)	O(3)–H(2)	0.834 (26)
Angle		Angle	
O(1)–C(1)–O(2)	123.4 (2)°	O(4)–C(3)–O(5)	123.0 (2)°
O(1)–C(1)–C(2)	112.0 (2)	O(4)–C(3)–C(4)	111.6 (2)
O(2)–C(1)–C(2)	124.6 (2)	O(5)–C(3)–C(4)	125.4 (2)
C(1)–C(2)–O(3)	111.7 (2)	C(3)–C(4)–O(6)	112.9 (2)
C(1)–C(2)–H(3)	105.0 (1.3)	C(3)–C(4)–H(7)	107.0 (1.4)
C(1)–C(2)–H(4)	111.0 (1.4)	C(3)–C(4)–H(8)	108.0 (1.4)
O(3)–C(2)–H(3)	109.0 (1.3)	O(6)–C(4)–H(7)	112.0 (1.3)
O(3)–C(2)–H(4)	115.0 (1.3)	O(6)–C(4)–H(8)	106.0 (1.4)
H(3)–C(2)–H(4)	104.0 (2.0)	H(7)–C(4)–H(8)	111.0 (2.0)
C(1)–O(1)–H(1)	111.0 (1.4)	C(3)–O(4)–H(5)	109.0 (1.5)
C(2)–O(3)–H(2)	108.0 (1.7)	C(4)–O(6)–H(6)	107.0 (1.7)
C(2)–O(3)---H(1')	118.6 (7)	C(4)–O(6)---H(5''')	119.7 (7)
H(2)–O(3)---H(1')	99.0 (1.9)	C(3)–O(5)---H(2 ^{iv})	139.5 (5)
C(1)–O(2)---H(6)	127.9 (6)	H(6)–O(6)---H(5''')	111.0 (1.8)

Donor	Acceptor	Angle	Distance of the H atoms from the straight O–O line
O(1)–H(1)---O(3 ^v)		175 (2.3)°	0.05 Å
O(3)–H(2)---O(5 ⁱⁱ)		163 (2.5)	0.17
O(4)–H(5)---O(6 ^{vi})		173 (2.4)	0.07
O(6)–H(6)---O(2)		163 (2.5)	0.17

Atoms related by symmetry are marked as follows:

'	–x	$\frac{1}{2}+y$	$\frac{1}{2}-z$
''	x	$\frac{1}{2}-y$	$\frac{1}{2}+z$
'''	1–x	$\frac{1}{2}+y$	$\frac{1}{2}-z$
iv	x	$\frac{1}{2}-y$	$z-\frac{1}{2}$
v	–x	$y-\frac{1}{2}$	$\frac{1}{2}-z$
vi	1–x	$y-\frac{1}{2}$	$\frac{1}{2}-z$

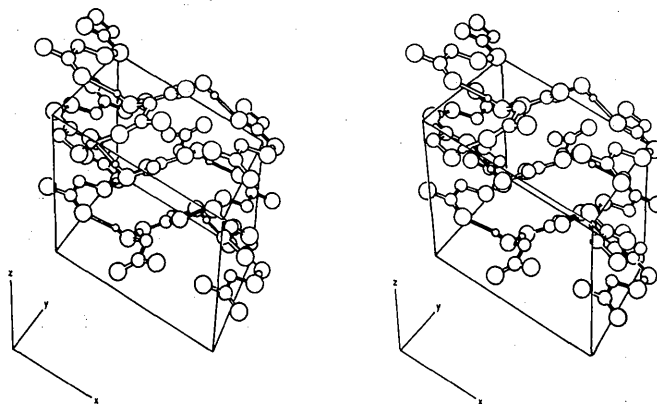


Fig. 2. Stereographic drawing of glycollic acid. For clarity the aliphatic hydrogen atoms are omitted.

last cycle of the refinement the maximum and average shifts, as fractions of the e.s.d.'s, in the atomic coordinates of the heavy atoms and of the hydrogen atoms were 0.03, 0.01 and 0.3, 0.1 respectively. The expression for the anisotropic thermal parameters was:

$$\exp \left[- (h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2kl\beta_{23} + 2lh\beta_{31}) \right].$$

The observed and calculated structure factors are listed in Table 2. In the final difference Fourier synthesis the electron density was not above a level of $0.21 \text{ e.}\text{\AA}^{-3}$.

Molecular structure

Bond lengths and bond angles together with their e.s.d.'s are shown in Table 3. The molecules are almost planar. In the molecules *A* and *B* the hydroxylic oxygen atom is turned away from the plane through the carboxylic group by 5.8 and 2.4° respectively. The C-C bond lengths are short in the two independent molecules (1.508 and 1.498 \AA).

The crystal structure

Let us consider the crystal structure shown as a stereographic drawing in Fig. 2. The structure is made up of chains which lie along the screw axis. The hydrogen bonding in the chains is effected by an unusual carboxylic-hydroxylic coupling, in which the alcoholic hydroxylic oxygen atom serves as an acceptor (see also Fig. 1). The chains are linked through the hydrogen atoms of the hydroxylic groups. Inspection of the

hydrogen bonds shows that the hydrogen atoms involved in the hydrogen bonding are a significant distance from the straight line between the oxygen atoms concerned (Table 3).

Note: After this manuscript had been completed we happened to learn from an internal report that the structure of this compound had already been determined by means of neutron diffraction (Ellison & Levy, 1967, Ellison, Johnson & Levy, 1971).

References

- BEURSKENS, P. T. (1963). Technical Report on Sign Correlation by the Sayre Equation. The Crystallographic Laboratory, Univ. of Pittsburgh, Pittsburgh, Pa. 15213.
- ELLISON, R. D. & LEVY, H. A. (1967). Chemistry Division Annual Progress Reports ORNL-4164, p. 126. Oak Ridge National Laboratory, Oak Ridge, Tennessee.
- ELLISON, R. D., JOHNSON, C. K. & LEVY, H. A. (1971). *Acta Cryst.* B26, 333.
- KANTERS, J. A., KROON, J., PEERDEMAN, A. F. & VLIENGENTHART, J. A. (1969). *Nature, Lond.* 222, 370.
- KRABBENDAM, H. & KROON, J. (1971). *Acta Cryst.* A 27, 48.
- MCDONALD, T. R. R. (1956). *Acta Cryst.* 9, 162.
- MOORE, F. H. (1963). *Acta Cryst.* 16, 1169.
- PALM, J. H. & PETERSE, W. J. A. M. (1964). General Structure Factors and Least-Squares Program in Algol for the TR-4 Computer (Version for EL X-8 Computer).
- SPEK, A. L. (1968). Algol program for EL X-8 computer. Laboratorium voor Kristalchemie, Rijksuniversiteit, Utrecht, The Netherlands.
- SPEK, A. L. (1970). To be published.