Acta Cryst. (1978). B**34**, 2379–2381

L-Serylglycine

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(*Received* 22 *March* 1978; *accepted* 29 *March* 1978)

Abstract. $C_5H_{10}N_2O_4$, $M_r = 162{\cdot}15$, orthorhombic, $P2_12_12_1$, $a = 4{\cdot}550\ (2)$, $b = 8{\cdot}506\ (3)$, $c = 17{\cdot}380\ (4)$ Å, $U = 672{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}601$ g cm^{-3}, μ(Cu $K\alpha$) $= 10{\cdot}8$ cm^{-1}. The structure was refined to $R = 0{\cdot}043$ for 713 unique reflexions. The molecule is a zwitterion; the peptide backbone is in an extended conformation.

Introduction. The crystal structure of L-serylglycine was studied as part of a series of peptide structures determined in this laboratory. Small colourless crystals were grown from acetone/water. Intensities were measured on a Syntex $P2_1$ diffractometer with graphite-monochromated Cu $K\alpha$ radiation and a crystal $0{\cdot}1 \times 0{\cdot}04 \times 0{\cdot}025$ mm. Systematic absences $h00$ (h odd), $0k0$ (k odd) and $00l$ (l odd) indicated space group $P2_12_12_1$. Cell dimensions were obtained by a least-squares refinement of 15 strong reflexions. 1186 reflexions were measured in the range $0 < 2\theta < 116°$; after application of Lp corrections, averaging equivalent reflexions gave 713 data with $F > 4\sigma(F)$.

The structure was solved by multisolution tangent refinement using the program *SHELX*. A starting set of nine reflexions (chosen from a convergence map) provided 2^5 phase permutations. The best E map showed all non-hydrogen atoms. Isotropic refinement proceeded to $R = 0{\cdot}09$, and anisotropic to $R = 0{\cdot}07$; difference maps then revealed all H atoms. In the final stages of refinement H(3) and H(10) refined freely; C–H distances were fixed at 1·08 Å and H–C–H angles at 109·5°; N–H distances and H–N–H angles of the $-NH_3^+$ group were fixed at 0·95 Å and 109·5° respectively; all H atoms were assigned an overall isotropic temperature factor. The final $R' = \sum w^{1/2}\Delta / \sum w^{1/2}|F_o|$ was 0·039, with a corresponding R of 0·043. The weighting scheme was $w = 1/[\sigma^2(F) + 0{\cdot}0005F^2]$, which gave mean values of $w\Delta^2$ varying only slightly with $\sin\theta$ or $|F_o|$. A final difference map showed no peaks >0·37 e Å^{-3}. Final atomic coordinates are given in Table 1,† with derived bond lengths, angles and torsion angles in Tables 2–4. Diagrams of the structure are given in Figs. 1 and 2.

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† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33498 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atom coordinates* ($\times 10^4$)

Overall isotropic temperature factor for H atoms = 0·038 (4) Å^2.

	x	y	z
C(1)	5940 (8)	5051 (4)	6110 (2)
C(2)	4909 (10)	6682 (4)	5881 (2)
C(3)	6598 (9)	9373 (4)	6009 (2)
C(4)	8810 (9)	10480 (4)	6386 (2)
C(5)	7541 (10)	11212 (4)	7123 (2)
N(1)	6687 (8)	7882 (3)	6242 (2)
N(2)	9431 (8)	11754 (3)	5816 (2)
O(1)	4793 (7)	3884 (3)	5767 (1)
O(2)	7855 (7)	4979 (3)	6624 (2)
O(3)	4864 (7)	9877 (3)	5516 (1)
O(4)	9380 (7)	12460 (3)	7392 (2)
H(1)	2648	6833	6055
H(2)	5073	6801	5264
H(4)	10770	9841	6544
H(5)	7382	10319	7563
H(6)	5380	11676	7003
H(3)	8012 (79)	7455 (41)	6592 (21)
H(7)	11099	12367	5950
H(8)	7743	12409	5794
H(9)	9741	11280	5328
H(10)	8913 (76)	13265 (39)	7187 (23)

Table 2. *Bond lengths* (Å)

C(1)–C(2)	1·517 (7)	C(1)–O(1)	1·270 (5)
C(1)–O(2)	1·250 (6)	C(2)–N(1)	1·446 (6)
C(3)–C(4)	1·526 (7)	C(3)–N(1)	1·332 (5)
C(3)–O(3)	1·241 (6)	C(4)–C(5)	1·537 (7)
C(4)–N(2)	1·495 (5)	C(5)–O(4)	1·430 (6)
N(1)–H(3)	0·930 (37)	O(4)–H(10)	0·800 (36)

Table 3. *Bond angles* (°)

C(2)–C(1)–O(1)	117·7 (4)	C(2)–C(1)–O(2)	116·6 (4)
O(1)–C(1)–O(2)	125·7 (4)	C(1)–C(2)–N(1)	111·0 (4)
C(4)–C(3)–N(1)	115·9 (4)	C(4)–C(3)–O(3)	120·2 (4)
N(1)–C(3)–O(3)	123·9 (4)	C(3)–C(4)–N(2)	106·7 (4)
C(5)–C(4)–N(2)	109·3 (4)	C(4)–C(5)–O(4)	110·7 (4)
C(2)–N(1)–C(3)	121·6 (4)	C(2)–N(1)–H(3)	111·8 (23)
C(3)–N(1)–H(3)	126·2 (23)	C(5)–O(4)–H(10)	109·6 (27)

Discussion. The peptide group displays the usual *trans* geometry. Details of the planes containing the peptide and carboxyl groups are given in Table 5. The carboxyl group is ionized, and the molecule is thus a zwitterion.

The peptide backbone is extended, with torsion angles $\psi_T^2 = -172{\cdot}1°$, $\varphi_2 = 166{\cdot}0°$, $\omega = -174{\cdot}1°$, $\psi_1 = 151{\cdot}9°$ [*cf.* fully extended values, all 180°; see Table 4 and the IUPAC–IUB Commission on Biochemical Nomenclature (1971)].

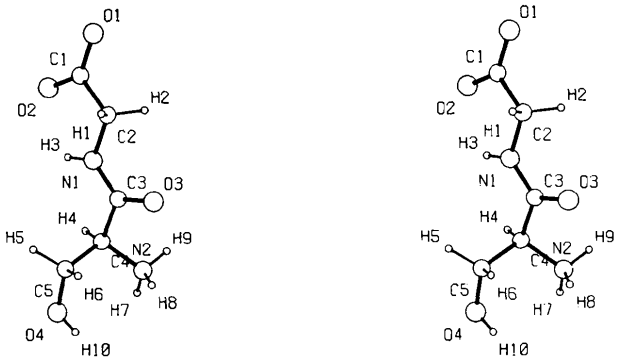

Fig. 1. Stereo pair of the L-serylglycine molecule showing the atomic numbering scheme.

Table 4. *Torsion angles* (°)

The sign convention is as defined by Klyne & Prelog (1960).

O(1)–C(1)–C(2)–N(1)	−172·1 (4)
O(2)–C(1)–C(2)–N(1)	8·0 (6)
C(1)–C(2)–N(1)–C(3)	166·0 (4)
N(1)–C(3)–C(4)–C(5)	−89·1 (4)
N(1)–C(3)–C(4)–N(2)	151·9 (4)
O(3)–C(3)–C(4)–C(5)	91·1 (5)
O(3)–C(3)–C(4)–N(2)	−27·8 (6)
C(4)–C(3)–N(1)–C(2)	−174·1 (4)
O(3)–C(3)–N(1)–C(2)	5·7 (7)
C(3)–C(4)–C(5)–O(4)	−169·0 (4)
N(2)–C(4)–C(5)–O(4)	−51·6 (5)
O(3)–C(3)–N(1)–H(3)	176·8 (28)
C(4)–C(3)–N(1)–H(3)	−2·9 (29)
C(4)–C(5)–O(4)–H(10)	87·2 (29)

Hydrogen-bonding interactions are given in Table 6. The hydrogen-bonding pattern is unusual in that the carboxyl oxygen atom O(1) takes part in three H bonds and O(2) in only one, rather than two each (*e.g.* L-alanyl-L-serine, Jones, Falvello & Kennard, 1978). The peptide oxygen O(3) is not involved in H bonding.

Table 5. *Least-squares planes*

The equations of the planes are of the form $ax + by + cz + d = 0$.

(i) Peptide group		Deviations from plane (Å)	
a =	−0·6743	C(2)	−0·038
b =	0·1784	N(1)	0·048
c =	0·7166	C(3)	0·012
d =	6·8706	O(3)	0·006
		C(4)	−0·029
(ii) Carboxyl group			
a =	0·7164	C(2)	0·000
b =	0·0422	C(1)	0·001
c =	−0·6964	O(1)	0·000
d =	−5·2772	O(2)	0·000

The dihedral angle between planes (i) and (ii) is 13·0°.

Table 6. *Non-bonded distances* (Å)

(i) Hydrogen-bonding interactions

D–H···A	D···A	H···A	Symmetry transformation of A
O(4)–H(10)···O(2)	2·62	1·82	$x, 1+y, z$
N(2)–H(8)···O(1)	2·78	1·84	$x, -1+y, z$
N(2)–H(7)···O(1)	3·04	2·14	$-1+x, -1+y, z$
N(2)–H(9)···O(1)	2·81	1·91	$-\frac{1}{2}+x, 1\frac{1}{2}-y, 1-z$
N(1)–H(3)···O(4)	3·00	2·13	$2-x, \frac{1}{2}+y, 1\frac{1}{2}-z$

(ii) Other contacts <3·2 Å

		Symmetry transformation of second atom
O(3)···N(2)	2·99	$-1+x, y, z$
O(3)···C(4)	3·18	$-1+x, y, z$
O(2)···N(2)	3·16	$x, -1+y, z$
O(2)···O(4)	2·99	$2-x, \frac{1}{2}+y, 1\frac{1}{2}-z$

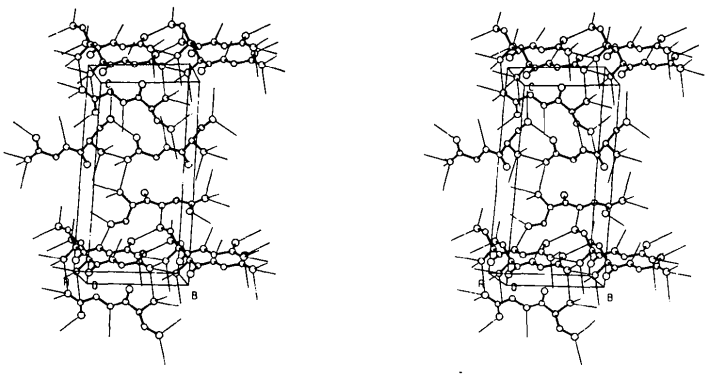

Fig. 2. Stereo packing diagram. Hydrogen bonds are indicated by narrow lines (H atoms omitted).

We thank the MRC and the Churchill Foundation for financial support. The figures were drawn with the program *PLUTO* written by Dr W. D. S. Motherwell. All other crystallographic programs were written by Dr G. M. Sheldrick.

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Acta Cryst. (1978). B**34**, 2381–2384

2,3,7,8-Tetrachlorodibenzofuran*

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(*Received* 30 *November* 1977; *accepted* 7 *March* 1978)

Abstract. $C_{12}H_4Cl_4O$, $M_r = 305{\cdot}97$, monoclinic, $C2/c$, $a = 14{\cdot}702$ (4), $b = 12{\cdot}886$ (4), $c = 6{\cdot}256$ (1) Å, $\beta = 99{\cdot}90$ (2)°, $V = 1168$ Å, $\rho_{obs} = 1{\cdot}72$ (flotation), $\rho_{calc} = 1{\cdot}74$ g cm^{-3}, $Z = 4$. The structure has been determined by direct methods and refined to $R = 0{\cdot}042$ for 1863 independent reflections. The molecule is essentially planar. A crystallographic twofold axis bisects a C—C bond and passes through the O atom of the five-membered furan ring. The two unique C—Cl distances are 1·725 (2) and 1·732 (2) Å, the C—O distance is 1·385 (2) Å, and the benzenoid ring C—C distances range between 1·366 (2) and 1·404 (2) Å. The longest C—C bond distance within the benzenoid rings joins the C atoms to which the Cl atoms are attached. The title compound is closely related in structure to the highly toxic 2,3,7,8-tetrachlorodibenzo-*p*-dioxin.

Introduction. Chlorinated dibenzofurans have recently been recognized as significant contaminants in some industrial chemicals; 2,3,7,8-tetrachlorodibenzofuran (TCDBF; Fig. 1*a*) has been reported to be extremely active biologically (see *Discussion* for references). This specific tetrachloro isomer was synthesized under an FDA-supported contract by Gray, Dipinto & Solomon (1976). Crystals of the compound suitable for structural analysis were crystallized from 2,2,4-trimethylpentane ('iso-octane'; distilled in glass) by one of us (IHP) and dried with paraffin under vacuum. The space group $C2/c$ was confirmed by a successful refinement; no calculations were made

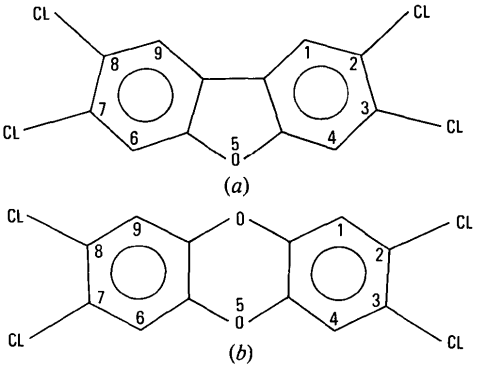

Fig. 1. Molecular structures of (*a*) 2,3,7,8-tetrachlorodibenzofuran and (*b*) 2,3,7,8-tetrachlorodibenzo-*p*-dioxin.

* Named in accordance with the accepted system of organic chemical nomenclature. Because of the crystallographic twofold axis in the molecule, the labeling of the atoms in the figures (except Fig. 1) and tables of this paper is shown as 3,4,3′,4′-tetrachlorodibenzofuran.