Table 4. *Comparison of the* O···O *intramolecular hydrogen bonds in hydrogen malonate ions*

Compound	O—H—O (°)	O···O (Å)
Lithium trihydrogen dimalonate	153 (5)	2·481 (3)
Potassium trihydrogen dimalonate	155	2·513 (9)
Guanidinium hydrogen malonate	153 (6)	2·407 (3)
Benzylammonium hydrogen malonate	158 (5)	2·430 (2)
4-Picolinium hydrogen malonate	159 (4)	2·493 (3)

in lithium and potassium trihydrogen dimalonate (Soriano-García & Parthasarathy, 1978; Currie, 1972), while the corresponding O···O hydrogen bond in (I) can be compared to the intramolecular hydrogen bond in guanidinium hydrogen malonate.

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Absolute Configuration of 1,1′-(1,2-Ethanediyl)bis(L-pyroglutamic acid), (*S*,*S*)-eddpg

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Abstract. $C_{12}H_{16}N_2O_6$, $M_r = 284{\cdot}3$, tetragonal, $P4_1$, $a = 7{\cdot}511\ (1)$, $c = 24{\cdot}278\ (3)$ Å, $V = 1369{\cdot}6\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}378$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 9{\cdot}6$ cm^{-1}, $F(000) = 600$, $T = 293$ K, final $R = 0{\cdot}022$ for 1371 observed reflections with $I > 2\sigma(I)$. The structure was solved by direct methods; the absolute configuration and space group were determined by synthesis and confirmed by use of the method of Rogers [*Acta Cryst.* (1981), A37, 734–741]. Both glutamate groups have the *S* configuration and are lactams. The dihedral angle and center-of-mass distance between the five-membered lactam rings are

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36° and 4·21 Å, respectively. Short intermolecular hydrogen-bonding contacts are found between a carboxylate —OH and a lactam carbonyl and between two neighboring carboxylic groups, O···O′ distances of 2·51 and 2·68 Å and O—H···O′ angles of 171 and 164°, respectively.

Introduction. Ethylenediaminetetraacetate (edta) and nitrilotriacetate (nta) are two organic multidentate ligands that have been used for many years to complex metal ions from a variety of solutions (Gardiner, 1976; Martell, 1975; Rompp, 1958; Schwarzenbach & Freitag, 1951). A secondary amine analog of edta, ethylenediamine disuccinic acid (Scarbrough & Voet, 1976), edds, also contains four carboxylate groups and chelates transition-metal ions (including Fe^{3+}, Co^{3+}, Ni^{2+} and Cu^{2+}) by binding to four, five or six of the octahedral metal sites (Pavelcik & Kettmann, 1983; Pavelcik, Garaj & Majer, 1980; Soldanova, Pavelcik, Majer & Garaj, 1984; Soldanova, Pavelcik & Majer, 1981). The practical applications of polycarboxylate analogs of edta are derived from their ability to selectively sequester transition-metal ions in solution. When the metal ions are chelated in this manner, their interactions with other solution constituents, such as oxidizers or anionic surfactants, are reduced or prevented. However, the industrial use of any compound can be restricted if it is not biodegradable, which is an important concern for polycarboxylate chelating agents. Fortunately, many tetracarboxylate compounds, like edds, are diastereomers, and the biodegradability of one enantiomer often differs substantially from that of other enantiomers (Hartman & Perkins, 1987). The determination of the biodegradability and the absolute configurations of the enantiomers of polycarboxylate compounds constitutes an essential step in determining their structure/biodegradation relationships and is the basis for this investigation.

Experimental. The title compound, namely 1,1′-(1,2-ethanediyl)bis(L-pyroglutamic acid), or eddpg (for ethylenediaminedipyroglutamic acid), was synthesized in a manner similar to the procedure for edds (Neal & Rose, 1968); however, L-glutamic acid was used in place of L-aspartic acid. Large, colorless crystals of eddpg were grown as follows: eddpg (2·00 g) was slurried in distilled water (2·00 mL). The solution was adjusted to pH 6 with aqueous NaOH. Absolute ethanol (4·00 mL) was slowly added to the solution with stirring. Scratching the glass walls of the vessel with a glass stirring rod initiated crystallization.

The crystal chosen for X-ray data collection was a parallelepiped measuring 0·10 × 0·10 × 0·24 mm along $a \times b \times c$, respectively, and was mounted on a Nicolet *R3m/E* diffractometer. Cell constants were determined by measuring 25 reflections with $25 < 2\theta < 35°$. Intensity data were collected by variable-speed ($2\text{–}29°\ \text{min}^{-1}$) ω-scans beginning 1° below $K\alpha_{12}$ and ending 1° beyond $K\alpha_{12}$ (Cu radiation), with stationary background counting time equal to scan time. A numerical absorption correction was applied ($\mu = 9{\cdot}6\ \text{cm}^{-1}$; T_{min}, $T_{\text{max}} = 0{\cdot}85$, $0{\cdot}91$; [001], [010] and [100] faces). The intensities of three standard reflections were measured after every 200 reflections and did not show significant variations throughout the data collection. The 1754 total data were measured to $2\theta_{\text{max}} = 100°$ ($0 \leq h$, $k \leq 7$ and $-24 \leq l \leq 24$). Out of 1414 unique non-zero intensities there were 1371 data with $F > 4\sigma(F)$, the latter being used in the structure determination and least-squares calculations. The only systematic absences in the data set were those consistent with the presence of 4_1 or 4_3 axes; the $(E^2 - 1)$ statistics indicated a noncentrosymmetric structure. The absolute configuration of this sample was *S,S* since it was synthesized by the addition of pure L-glutamic acid to 1,2-dibromoethane. The space group $P4_1$ was found to be consistent with this conformation, and was chosen. The structure was solved by direct methods. A refinement of $\Delta f''$ multipliers according to the method of Rogers (1981), using all 1371 $F(hkl)$ and $F(hk\bar{l})$ data with $F > 4\sigma(F)$, gave $\eta = 0{\cdot}8\ (4)$. An empirical isotropic extinction correction similar to Larson's (1967) was applied, where $F'_{\text{calc}} = F_{\text{calc}}/(1{\cdot}0 + 0{\cdot}002gF^2_{\text{calc}}/\sin 2\theta)^{0{\cdot}25}$, and the parameter g was refined to 0·044 (3). The reflections most affected by extinction were 01 ± 1, 01 ± 2, 01 ± 3, 110, 11 ± 1 and 11 ± 2. All calculations employed the *SHELXTL-Plus* crystallographic computer package (Sheldrick, 1988) on a MicroVAX II computer. The positions and anisotropic thermal parameters of the C, N and O atoms, and the scale factor were varied (182 variable parameters). All H atoms were located on an intermediate ΔF map and their positions were refined with isotropic thermal parameters to convergence. For the final cycles of refinement, the H-atom parameters were fixed to these values. Final H-atom positions did not differ significantly from their idealized positions. The weighting scheme for the least-squares refinement was $w = 1/[\sigma^2(F_{\text{obs}}) + 0{\cdot}0004(F_{\text{obs}})^2]$, with $\sigma(F_{\text{obs}})$ computed solely from counting statistics. Final $R(F)$ was 0·022 ($wR = 0{\cdot}029$, GOF = 1·193); the function $w(F_{\text{obs}} - F_{\text{calc}})^2$ was minimized. At convergence, final shift/e.s.d.'s were less than 0·01 for all atoms varied. The residual peaks on a final difference electron density map were less than $\pm 0{\cdot}2\ \text{e}\ \text{Å}^{-3}$. Atomic scattering factors were from *SHELXTL-Plus*.

Discussion. The fractional coordinates and the equivalent isotropic thermal parameters of all non-H

Table 1. *Fractional atomic coordinates* $(\times 10^4)$ *and equivalent isotropic thermal parameters* $(\text{Å}^2 \times 10^4)$ *for (S,S)-eddpg*

Equivalent isotropic U defined as 1/3 of the trace of the orthogonalized U_{ij} tensor. Estimated standard deviations are enclosed in parentheses. The z coordinate of N1 was fixed.

	x	y	z	U_{eq}
N1	9344 (2)	3592 (2)	1856 (0)	477 (6)
N2	11415 (2)	2008 (2)	2775 (1)	443 (6)
O1	11643 (3)	5538 (3)	1976 (1)	889 (8)
O2	8964 (2)	3199 (2)	3186 (1)	560 (6)
O3	7224 (2)	2767 (2)	941 (1)	684 (7)
O4	4986 (2)	2113 (2)	1494 (1)	600 (6)
O5	13084 (2)	−831 (3)	3269 (1)	787 (7)
O6	15742 (2)	108 (3)	2986 (1)	734 (7)
C1	10404 (3)	1985 (3)	1816 (1)	557 (8)
C2	10483 (4)	992 (3)	2358 (1)	513 (8)
C3	7426 (3)	3515 (3)	1903 (1)	472 (8)
C4	6896 (4)	5478 (4)	1977 (2)	835 (11)
C5	8551 (4)	6741 (3)	2084 (1)	722 (10)
C6	10042 (4)	5210 (4)	1963 (1)	598 (10)
C7	6560 (3)	2750 (3)	1396 (1)	456 (8)
C8	13341 (3)	2010 (3)	2822 (1)	484 (8)
C9	13720 (3)	3586 (3)	3213 (1)	656 (9)
C10	11980 (4)	3910 (3)	3499 (1)	648 (9)
C11	10618 (3)	3036 (3)	3149 (1)	459 (8)
C12	14002 (3)	262 (4)	3047 (1)	541 (9)

Table 2. *Bond lengths* (Å) *and angles* (°) *for (S,S)-eddpg*

Estimated standard deviations are enclosed in parentheses. See Fig. 1 for atom-labeling scheme.

N1—C1	1·449 (3)	N1—C3	1·446 (3)	N1—C6	1·349 (3)
N2—C2	1·448 (3)	N2—C8	1·451 (3)	N2—C11	1·334 (3)
O1—C6	1·228 (3)	O2—C11	1·252 (3)	O3—C7	1·211 (4)
O4—C7	1·297 (3)	O5—C12	1·200 (3)	O6—C12	1·321 (3)
C1—C2	1·515 (4)	C3—C4	1·538 (4)	C3—C7	1·508 (4)
C4—C5	1·473 (4)	C5—C6	1·496 (4)	C8—C9	1·543 (4)
C8—C12	1·506 (4)	C9—C10	1·500 (4)	C10—C11	1·482 (4)

C3—N1—C1	121·3 (2)	O4—C7—O3	123·2 (2)
C8—N2—C2	122·5 (2)	C9—C8—N2	103·5 (2)
C2—C1—N1	111·9 (2)	C10—C9—C8	104·4 (2)
C7—C3—N1	112·4 (2)	C10—C11—N2	109·7 (2)
C6—C5—C4	106·1 (2)	C8—C12—O5	124·7 (2)
C5—C6—O1	127·0 (3)	C6—N1—C3	114·1 (2)
C3—C7—O4	112·5 (2)	C11—N2—C8	113·2 (2)
C12—C8—C9	112·7 (2)	C4—C3—N1	103·2 (2)
O2—C11—N2	123·4 (2)	C5—C4—C3	106·7 (2)
O6—C12—O5	124·0 (2)	C5—C6—N1	108·5 (2)
C6—N1—C1	123·4 (2)	C3—C7—O3	124·3 (2)
C11—N2—C2	124·3 (2)	C12—C8—N2	110·9 (2)
C1—C2—N2	111·5 (2)	C11—C10—C9	105·3 (2)
C7—C3—C4	110·4 (2)	C10—C11—O2	126·9 (2)
O1—C6—N1	124·5 (3)	C8—C12—O6	111·3 (2)

C1—N1—C6—O1	8·6 (4)	C2—N2—C11—O2	1·6 (4)
C1—N1—C6—C5	−168·9 (2)	C2—N2—C11—C10	−178·5 (2)
C3—N1—C6—O1	175·8 (3)	C8—N2—C11—O2	−178·8 (2)
C3—N1—C6—C5	−1·7 (3)	C8—N2—C11—C10	1·1 (3)
C3—N1—C1—C2	−72·2 (2)	C8—N2—C2—C1	−83·4 (2)
C6—N1—C1—C2	94·1 (2)	C11—N2—C2—C1	96·1 (3)
C1—N1—C3—C4	176·0 (2)	C2—N2—C8—C9	166·6 (2)
C1—N1—C3—C7	−65·1 (2)	C2—N2—C8—C12	−72·4 (3)
C6—N1—C3—C4	8·4 (3)	C11—N2—C8—C9	−13·0 (3)
C6—N1—C3—C7	127·4 (2)	C11—N2—C8—C12	108·0 (2)
C4—C5—C6—N1	−6·2 (3)	C9—C10—C11—N2	11·7 (3)
C4—C5—C6—O1	176·4 (3)	C9—C10—C11—O2	−168·4 (2)
N1—C3—C4—C5	−11·7 (3)	N2—C8—C9—C10	19·1 (3)
N1—C3—C7—O3	−24·2 (3)	N2—C8—C12—O5	−15·2 (4)
N1—C3—C7—O4	157·8 (2)	N2—C8—C12—O6	167·2 (2)
C4—C3—C7—O3	90·5 (3)	C9—C8—C12—O5	100·2 (3)
C4—C3—C7—O4	−87·5 (2)	C9—C8—C12—O6	−77·4 (3)
C3—C4—C5—C6	11·0 (3)	C8—C9—C10—C11	−18·7 (3)
C7—C3—C4—C5	−132·0 (3)	C12—C8—C9—C10	−100·7 (2)
N1—C1—C2—N2	−65·5 (2)		

atoms in the title compound are listed in Table 1.* The atomic numbering scheme of the (*S,S*)-eddpg molecule is illustrated in Fig. 1. Interatomic distances and angles are listed in Table 2. The combination of chirality and lactam formation for the two glutamic acid moieties of eddpg results in several unusual structural features with respect to other chelants derived from ethylenediamine. The most outstanding feature is that eddpg is a *dilactam* (see Fig. 1). Similar five-membered lactam rings have been observed in many other glutamic acid derivatives, the most common of which are the pyrrolidin-2-ones (Admiraal, Eikelenboom & Vos, 1982; Fujiwara, Varley & van der Veen, 1977; Behmel & Weber, 1984; Langer, Huml & Zachova, 1984; Carrell, Glusker, Job & Bruice, 1977; Churchill & Rotella, 1979; Clarke, Pauling & Petcher, 1975; Bandoli, Nicolini, Pappalardo, Grassi & Perly, 1987; Flippen-Anderson, D'Antonio & Konnert, 1984; Troin, Gramain & Prange, 1985; Sheldrick, Borkenstein, Blacha-Puller & Gossauer, 1977; Bowie *et al.*, 1986). The conversion of pyrrolidonecarboxylic acid to glutamic acid is thermodynamically favored, but is exceedingly slow, except under extreme conditions (*e.g.*, at 373 K in 2M HCl or 0·5M NaOH) (Wilson & Cannan, 1937). The conversion of ethylenediaminedipyroglutamic acid (eddpg) is presumed to be equally difficult, such that the lactam form is the most likely species in solution. The presence of the stable lactam form for (*S,S*)-eddpg observed here is expected to affect profoundly its metal-binding ability. The approximate, but non-crystallographic, C_2 molecular symmetry observed in (*S,S*)-eddpg is similar to that found in (*S,S*)-edds (Scarbrough & Voet, 1976). Other ethylenediamine-based molecules containing two or more carboxylate groups, such as the succinate, tartrate or aspartate analogs, are generally flexible enough to bind N and O atoms to most (if not all) of the octahedral coordination sites of an appropriate metal atom [*e.g.*, the Fe^{3+}, Co^{3+}, Ni^{2+} and Cu^{2+} complexes of (*S,S*)-edds]. However, for the (*S,S*)-eddpg molecule, the covalent bonding about each N atom is *nearly trigonal* (*e.g.*, rather than tetrahedral) and the two lactam O atoms are

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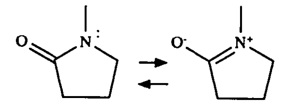
* Lists of anisotropic thermal parameters for the non-H atoms, positional and thermal parameters for the H atoms, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52185 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

directed toward the interior of the molecule. There are only two carboxylate groups in eddpg and they are directed outward from the center of the molecule, and are therefore not expected to interact with the same metal ion as the O atoms of the lactam carbonyl. Thus, for octahedrally coordinated metal atoms, eddpg is not expected to chelate as efficiently as other polycarboxylates that do not have the lactam form, such as edda (ethylenediamine diacetate), edds and eddg.

The two types of N—C bonds about each N atom in (*S*,*S*)-eddpg (*e.g.*, two with distances of ~1·45 and one of ~1·34 Å) are typical for N-substituted, five-membered lactams, as shown in Table 3. This adjacent bond-length difference reflects the two resonance forms of the C(sp^2)—N(sp^2) bond in the ring, as described by Birnbaum (1970):

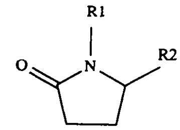


In agreement with the expected sp^2 hybridization of these N and C atoms, the atoms N1, C1, C3, C5 and C6 (or N2, C2, C8, C10 and C11), as labeled in Fig. 1, form approximate planes. The planarity about the N atom in other five-membered lactam rings is a common feature, as shown in Table 3. Comparatively, the C2—N2—C11—C10 dihedral angle is somewhat closer to 180° than the C1—N1—C6—C5 dihedral angle (see Table 2*c*), indicative of the more planar region about N2 than about N1. The lack of hydrogen-bonding interactions for O1 (described below) may have some effect on this difference.

Table 3 also lists selected geometries of lactams with different ring sizes. The pairs of values for the C—N—C—C dihedral angles and the central C—N bond length of the O—C—N—C group for (*S*,*S*)-eddpg (which is a C_4N lactam) given in Table 3 are in-between those of the C_3N propiolactam (Yang, Seiler & Dunitz, 1987) and the C_5N valerolactam (Romers, Rutten, van Driel & Sanders, 1967). Even though the skewed or *cis* conformation about O—C—N—C is observed for the larger, more-flexible caprylolactam (C_8N), perlargolactam (C_9N) and caprinolactam ($C_{10}N$), the central C—N bond length of the O—C—N—C group is still ~1·33 Å (Winkler & Dunitz, 1975). Similar central C—N bond lengths in (*S*,*S*)-eddpg, at 1·349 (3) and 1·334 (3) Å for N1 and N2, respectively, are also consistent with that observed in the neutron diffraction study of L-glutamine, 1·331 Å (Koetzle, Frey, Lehmann & Hamilton, 1973).

The projection of the *ac* plane of (*S*,*S*)-eddpg in Fig. 2 shows the O—H···O′ hydrogen-bonding interactions that are important for its crystal packing, namely, O4—H4O···O2 and O6—H6O···O3. The C6—N1 and C6—C5 bond distances are noticeably longer than the C11—N2 and C11—C10 distances, and the O1—C6 bond distance is significantly shorter than the O2—C11 distance (atomic labels from Fig. 1). These observations are consistent with

Table 3. *Bond distances* (Å) *and dihedral angles* (°) *about the* N *atoms of selected lactams and* (*S*,*S*)-*eddpg*



(*a*) Five-membered-ring lactams*

	Ring N=C bond	Ring N—C bond	C—N=C—O dihedral	C—N=C—C dihedral
Mean (e.s.d.)	1·32 (3)	1·45 (2)	180 (3)	−1 (9)
Min./max.	1·24/1.36	1·41/1·48	176/188	−22/13
(*S*,*S*)-eddpg	1·349/1·334	1·446/1·451	176·1/181·1	−2.0/1·0

(*b*) C_5-substituted five-membered-ring lactams†

	N=C bond	N—C bond	C—N=C—O dihedral	C—N=C—C dihedral
Mean (e.s.d.)	1·35 (2)	1·46 (2)	179 (7)	0 (15)
Min./max.	1·32/1.38	1·42/1·49	163/188	−21/21

(*c*) Lactams with four- to eleven-atom rings‡

	N=C bond	N—C bond	C—N=C—O dihedral	C—N=C—C dihedral
Propiolactam, C_3N	1·333	1·466	180·6	−0·1
(*S*,*S*)-eddpg, C_4N	1·34	1·45	179	−1§
Valerolactam, C_5N	1·365	1·497	174·3	16·2
Caprolactam, C_6N	1·328	1·469	176·6	67·8
7-Heptanelactam, C_7N	1·334	1·462	182·2	81·7
Caprylolactam, C_8N	1·334	1·448	−26·0	−90·4¶
Perlargolactam, C_9N	1·305	1·456	+10·8	75·4
Caprinolactam, $C_{10}N$	1·305	1·460	+4·1	75·2

* Averages of 22 pyrrolidones (as illustrated above, with R_2 = H) from the 1988 Cambridge Structural Database (CSD). The two values for (*S*,*S*)-eddpg correspond to the two lactam rings per molecule.

† Averages of 15 substituted pyrrolidones (as above, with $R_2 \neq$ H) from CSD.

‡ References for structures are: the C_3N lactam, Yang, Seiler & Dunitz (1987); C_5N, Romers, Rutten, van Driel & Sanders (1967); C_6N, C_8N, C_9N and $C_{10}N$, Winkler & Dunitz (1975); C_7N, Winkler & Seiler (1979).

§ Average of two for (*S*,*S*)-eddpg, which has an approximate C_2 molecular symmetry.

¶ The *trans* conformation is observed for lactams C_3N, C_5N, C_6N and C_7N; skewed for C_8N; *cis* for C_9N and $C_{10}N$.

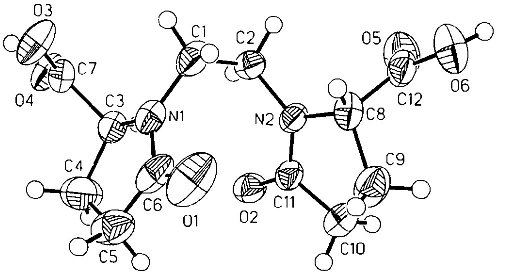


Fig. 1. Perspective view of the (*S*,*S*)-eddpg molecule perpendicular to the mean molecular plane. H atoms are represented by open circles. Thermal ellipsoids for non-H atoms are drawn at the 30% probability level.

the presence of a hydrogen-bonding interaction for O2, but not for O1. The H40···O2 and H60···O3 distances, at 1·52 and 1·62 Å, respectively, are relatively short (Taylor, 1981). These hydrogen bonds involve the carboxylate groups on the 'outside' of the molecule and the lactam carbonyls on the 'interior' (*e.g.*, with respect to the molecular center of mass). Thus, each (*S*,*S*)-eddpg molecule contains four hydrogen-bond acceptors and two donors. A three-dimensional network of intermolecular interactions arises from the arrangement of two hydrogen bonds per molecule. These hydrogen bonds serve to link two molecules related by a unit translation along the crystallographic *a* axis and two molecules related by the 4_1 screw axis along the crystallographic *c* axis. Of the four O atoms involved in these two hydrogen-bond interactions, one is from the *lactam carbonyl*, O2. The distance between this lactam O2 atom and the hydrogen-bonding carboxylate O4 atom is 2·511 (3) Å, which is 0·165 Å shorter than the distance between the two carboxylate O atoms comprising the other hydrogen bond [*e.g.*, O3···O6 is 2·676 (3) Å]. The shorter O···O distance may be the result of a more negative charge on the lactam O2 compared with the carboxylate O3. These hydrogen bonds extend from the center of the (*S*,*S*)-eddpg molecule in *approximately opposite directions*. It is expected that any other interaction involving these O atoms, including metal binding, would have a similar orientation. In such a scheme, multidentate binding to metal ions is not favored.

In summary, because (*S*,*S*)-eddpg exists in the lactam form, the N atoms are sp^2 and the C—O bond vectors are approximately antipodal. As a result, only mono- or bidentate complexing to metal ions is expected. Since other polycarboxylate chelants, such as edta, edda and edds, often form tetra-, penta- or hexadentate complexes, (*S*,*S*)-eddpg is not expected to be an efficient complexing agent if the solid-state structure observed here is maintained in solution.

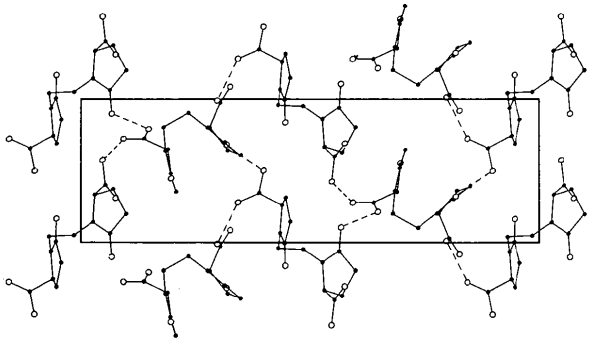


Fig. 2. Projection of the unit cell of (*S*,*S*)-eddpg along the *a* axis (*c* axis horizontal). Four of the six O atoms (open circles) contribute to the three-dimensional network of hydrogen-bonding interactions (broken lines). H atoms are omitted for clarity.

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