C7 methyl group, on one side. While the acetoxy substitution at C4 presents the same conformation as in the bromoacetoxy derivative (C9 *trans* to C3 and C10 *trans* to C4), the main differences between them are due to the substitution at O3 and are reflected by the torsion angles C1—C2—C5—C6 and C1—C2—C5—O3, −21·9 (5) and 158·4 (4)° *vs* 56·1 (3) and 175·1 (3)° in the derivative. The above conformation gives rise to some steric interactions in the packing as well as within the molecule (Table 2) (Vainshtein, Fridkin & Indenbom, 1982).

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meso-α,α′-Diaminosuccinic Acid: An Example of Strong Intermolecular Hydrogen Bonding

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Abstract. $C_4H_8N_2O_4$, $M_r = 148\cdot12$, monoclinic, $P2_1/n$, $a = 7\cdot439$ (1), $b = 4\cdot758$ (1), $c = 8\cdot301$ (1) Å, $\beta = 102\cdot21$ (1)°, $V = 287\cdot2$ (1) Å^3, $Z = 2$, $D_x = 1\cdot713$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1\cdot54184$ Å, $\mu = 12\cdot9$ cm^{-1}, $F(000) = 156$, $T = 295$ K. The structure has been refined to $R = 0\cdot034$ for 516 unique reflections with $I \geq 2\cdot5\sigma(I)$. The asymmetric unit of the double zwitterionic title compound consists of one half molecule. The NH_3^+ protons are involved in N—H···O intermolecular hydrogen bonds. Each molecule is connected to eight neighbouring molecules by 12 hydrogen bonds. Two unique hydrogen bonds connect molecules which lie in sheets parallel to **b** and one hydrogen bond serves as a link between these sheets.

Introduction. The lack of structural information on the class of α,α′-diaminodicarboxylic acids and our general interest in hydrogen bonding of α-substituted carboxylic acids led us to the structure analysis of the first member of this class, *meso*-α,α′-diaminosuccinic acid. For a long time this compound attracted our attention because of its high density (1·713 g cm^{-3}) and its low solubility in water and in a wide variety of organic solvents, which are indicative of strong intermolecular hydrogen bonding of this supposedly double zwitterionic compound. However, numerous attempts to obtain single crystals of sufficient size and quality applying conventional methods were unsuccessful over a period of years. By using an alternative crystallization procedure crystals of excellent quality were obtained and we now report the crystal-structure analysis.

Experimental. Because of the insolubility of the title compound no standard crystallization technique was useful. Therefore lithium and hydrogen chloride salts were prepared and crystallization attempts were made in acidified gels (Li salt) and in basic gels (HCl salt). This procedure only yielded twinned crystals in different silica and agarose gels. Finally single crystals

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were obtained by the following procedure: A 0·1 *M* LiOH solution was saturated with *meso*-diaminosuccinic acid at the boiling point. After cooling 100 ml of this solution (pH = 10·3) was equilibrated against 1 ml 0·1 *M* potassium–sodium phosphate buffer acidified with 0·1 *M* HCl until pH = 3·0 by means of sitting-drop vapour diffusion (McPherson, 1967).

A plate-like crystal of diaminosuccinic acid (0·26 × 0·13 × 0·04 mm) was selected for data collection on an Enraf–Nonius CAD-4 diffractometer with Ni-filtered Cu *K*α radiation. Cell dimensions were obtained by least-squares fitting of 16 reflections in the range of $21·6 \leq 2\theta \leq 41·8°$. The intensity data of 2067 reflections were collected in the whole reflection sphere ($-9 \leq h \leq 9$, $-5 \leq k \leq 5$, $-10 \leq l \leq 10$; $2\theta_{max} = 140°$). Two reference reflections ($\bar{1}\bar{1}3$, 011) measured every hour showed no significant decay during 12 h of exposure time. After merging equivalent reflections, 516 unique reflections remained with $I \geq 2·5\sigma(I)$ ($R_{int} = 0·04$). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986). The solution with the highest figure of merit showed all non-hydrogen atoms. The H atoms were found in a difference map and included in the refinement with individual isotropic thermal parameters. Full-matrix least-squares refinement on *F* of positional and individual anisotropic thermal parameters for the non-H atoms resulted in a final *R* value of 0·034, $wR = 0·042$, $w = 1/[\sigma^2(F_o)]$, $S = 2·79$ for 62 refined parameters. The average and max. shift to e.s.d. ratio in the final stage were 0·002 and 0·019 respectively. Maximum and minimum densities in the final difference Fourier map were 0·25 and −0·27 e Å^{-3} respectively. Scattering factors for non-H atoms were taken from Cromer & Mann (1968), for H atoms from Stewart, Davidson & Simpson (1965) and anomalous-dispersion terms from Cromer & Liberman (1970). Programs used include *SHELX*76 (refinement) (Sheldrick, 1976) and the *EUCLID* package (geometry calculations and illustrations) (Spek, 1982).

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *for all atoms, with e.s.d.'s in parentheses*

	x	y	z	U_{iso} or U_{eq}
C(1)	0·4974 (2)	0·1271 (3)	0·7220 (2)	0·0183 (4)†
C(2)	0·4510 (2)	−0·0568 (3)	0·5664 (2)	0·0168 (4)†
O(1)	0·6362 (2)	0·0569 (2)	0·8283 (1)	0·0265 (4)†
O(2)	0·3982 (1)	0·3362 (2)	0·7265 (1)	0·0246 (4)†
N	0·2490 (2)	−0·0533 (3)	0·5009 (2)	0·0195 (4)†
H(1)	0·192 (3)	−0·080 (5)	0·587 (3)	0·044 (6)
H(2)	0·212 (3)	0·113 (5)	0·452 (3)	0·033 (5)
H(3)	0·211 (3)	−0·194 (5)	0·424 (3)	0·041 (6)
H(4)	0·485 (2)	−0·250 (4)	0·591 (2)	0·018 (4)

† $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

O(1)–C(1)	1·253 (2)	C(1)–C(2)	1·538 (2)
O(2)–C(1)	1·244 (2)	C(2)–H(4)	0·96 (2)
N–C(2)	1·486 (2)	C(2)–C(2*a*)*	1·542 (2)
N–H(1)	0·91 (2)	N–H(2)	0·91 (2)
N–H(3)	0·93 (2)		

O(1)–C(1)–O(2)	126·7 (1)	O(1)–C(1)–C(2)	116·4 (1)
O(2)–C(1)–C(2)	116·8 (1)	C(1)–C(2)–N	109·3 (1)
C(1)–C(2)–C(2*a*)*	110·2 (1)	N–C(2)–C(2*a*)*	110·2 (1)
N–C(2)–H(4)	106·7 (9)	C(1)–C(2)–H(4)	111 (1)
C(2)–N–H(1)	108 (2)	H(4)–C(2)–C(2*a*)*	110·1 (9)
C(2)–N–H(2)	111 (2)	H(1)–N–H(2)	109 (2)
C(2)–N–H(3)	112 (1)	H(1)–N–H(3)	108 (2)
		H(2)–N–H(3)	108 (2)

* Related to C(2) by the operation 1−*x*, −*y*, 1−*z*.

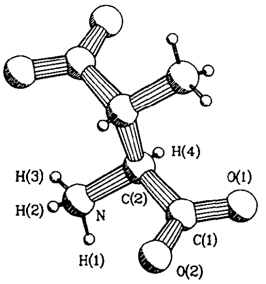

Fig. 1. Perspective view of the molecule with numbering scheme.

Discussion. The final fractional coordinates and equivalent isotropic thermal parameters are listed in Table 1.* Bond distances and angles are listed in Table 2. A perspective view of the molecule with adopted numbering scheme is shown in Fig. 1. As the middle of the C—C bond coincides with a centre of inversion, the carbon chain has an extended form which minimizes the electrostatic interaction, whereas in the related *meso*-tartaric acid the conformation is synclinal (Kroon, 1982). The title compound is a double zwitterion with C—O distances of 1·244 (2) and 1·253 (2) Å. The acetic-acid group is planar ($\sigma_{plane} = 0·001$ Å) and the N atom deviates from this plane [distance: 0·72 (6) Å, torsion angle O(2)—C(1)—C(2)—N: −32·8 (2)°]. The plane of the acetic-acid group is almost perpendicular [88·6 (1)°] to the plane of the carbon-atom skeleton.

The conformation of the NH_3^+ group with respect to the C(2)—N bond deviates 14 (1)° from the ideal staggered conformation which is probably caused by intermolecular hydrogen bonding. There are three unique intermolecular hydrogen bonds, all involving the N—H bonds of the NH_3^+ group as donors (Table 3). The N—H(2) and N—H(3) bonds donate hydrogen bonds to O(1) atoms of the carboxyl groups of the two

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* Lists of structure factors, anisotropic thermal parameters and the complete internal geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44733 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Intermolecular hydrogen bonds in the structure of meso-diaminosuccinic acid, with e.s.d.'s in parentheses*

X–H···Y	X···Y (Å)	H···Y (Å)	X–H···Y (°)
N–H(1)···O(2^{i})	2·765 (2)	1·86 (2)	173 (2)
N–H(2)···O(1^{ii})	2·798 (2)	1·90 (2)	174 (2)
N–H(3)···O(1^{iii})	2·827 (2)	1·93 (2)	161 (2)

Symmetry code: (i) $\frac{1}{2}-x, -\frac{1}{2}+y, 1\frac{1}{2}-z$; (ii) $-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$; (iii) $-\frac{1}{2}+x, -\frac{1}{2}-y, -\frac{1}{2}+z$.

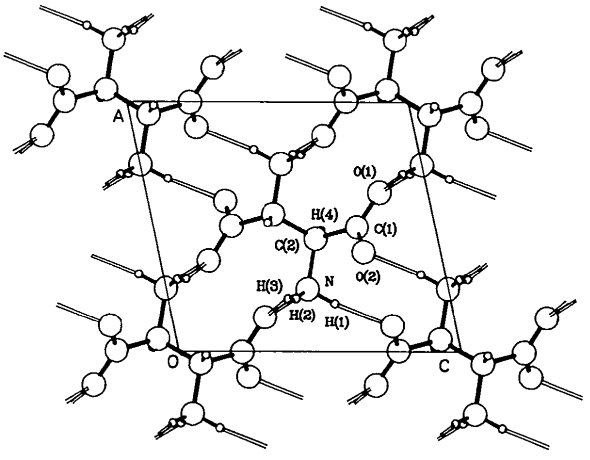

Fig. 2. Projection of the structure along **b**, showing the sheets of hydrogen-bonded molecules parallel to (10$\bar{1}$) and the hydrogen bond between the sheets.

symmetry-related (*n*-glide) molecules, differing by one period along **b** (Figs. 2 and 3). This pair of hydrogen bonds is generated by a 2_1 axis into an infinite double-ribbon pattern propagating in the **b** direction. These chains form a sheet in the (10$\bar{1}$) plane. The third hydrogen bond, between N–H(1) and O(2), connects molecules related by a 2_1 axis and serves as a link between the sheets. The relatively high number of effective donors and acceptors in the molecule provides an ideal basis for a tight efficient three-dimensional hydrogen-bond network, in which each molecule is connected to eight surrounding molecules by 12 hydrogen bonds. The dense packing of the structure is also reflected by the low thermal parameters (Table 1). Besides the three N–H···O hydrogen bonds, there is a short intermolecular C(2)–H(4)···O(2)′ (x, $-1+y$, z) contact with C···O, H···O and C–H···O 3·238 (2), 2·42 (2) Å and 142(1)° respectively.

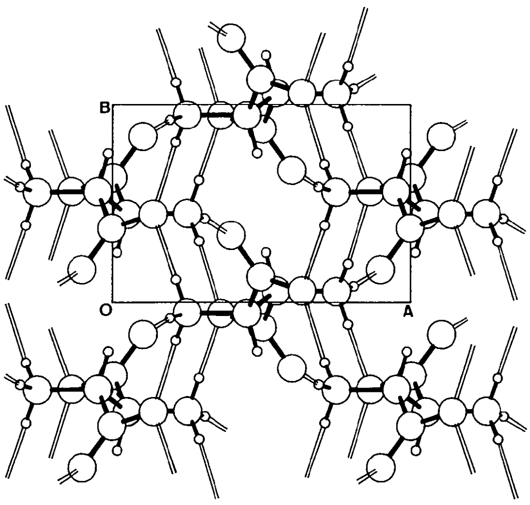

Fig. 3. Projection of the structure along **c**, showing the ribbons of hydrogen bonds in the **b** direction.

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