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The Structure of L-α-Aminoadipic Acid

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Abstract. $C_6H_{11}NO_4$, $M_r = 161{\cdot}16$, monoclinic, $P2_1$, $a = 5{\cdot}136\,(3)$, $b = 6{\cdot}992\,(2)$, $c = 10{\cdot}065\,(10)$ Å, $\beta = 93{\cdot}32\,(5)^\circ$, $U = 360{\cdot}8$ Å^3, $Z = 2$, $D_x = 1{\cdot}48$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}10$ mm^{-1}, $F(000) = 172$, $T = 293$ K. $R = 0{\cdot}058$ for 305 unique observed $[F > 5\sigma(F)]$ reflections. L-α-Aminoadipic acid exists as a zwitterion. The conformation around the amino terminus is similar to that of L-glutamic acid. There is a short intermolecular hydrogen bond between the carboxyl groups.

Introduction. Amino acids are precursors for proteins, hormones and neurotransmitters and can be neurotransmitters themselves. Membrane systems that make up different tissues in the body appear to be selective in the amino acids that they are able to recognize and take up. One example is the brain, where glutamate is an important excitatory amino acid and which has specific receptors for glutamate (Watkins & Olverton, 1987). Another example is the range of amino-acid transport mechanisms that exist for the import and export of amino acids in tissues such as liver, muscle *etc.* Such transport of amino acids is important for the amino-acid economy of the body and the description of the interaction between amino acids or analogues and receptors may prove important for altering the uptake or efflux of an amino acid from a particular organ (Rennie *et al.*, 1986). A knowledge of the molecular structure of naturally occurring amino acids or their analogues is necessary in order to understand and predict the necessary characteristics of an amino-acid agonist or antagonist, *i.e.* to be able to say whether a chemical substance will interact solely because of its shape or whether there are required chemical groups in the molecule before binding can take place. This may in turn give some insight into the nature of the receptive site for the molecule. We investigate here the structure of the amino acid L-2α-aminoadipic acid, an analogue of glutamic acid.

Experimental. Crystals were obtained from aqueous solution. Space group and initial cell dimensions were obtained from Weissenberg photographs. Data were collected on a Nicolet $P3$ (four-circle) diffractometer in Aberdeen by RAH. The crystal had dimensions $0{\cdot}175 \times 0{\cdot}1 \times 0{\cdot}04$ mm. Cell parameters were measured on the diffractometer using 14 reflections in the 2θ range 12–14°. Range of indices: $0 \le h \le 7$; $0 \le k \le 9$; $-13 \le l \le 13$. Data measured using $\omega/2\theta$ scans in the range $0 < 2\theta < 50^\circ$. Standard reflections 020 and 120 were measured every 50 reflections. No changes greater than 2σ from the mean of the intensities of these

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reflections were found throughout data collection. Lorentz and polarization factors were applied. No corrections were made for absorption or secondary extinction. 601 independent reflections measured, giving 305 observed [$F > 5\sigma(F)$] reflections used in the refinement. The structure was solved using the *SHELXS*86 program (Sheldrick, 1986).

Blocked full-matrix refinement (on F) was carried out using the program *SHELX*76 (Sheldrick, 1976). The H atoms attached to the C atoms were included at calculated positions. Those attached to N2 and O6*A* were located on a difference Fourier synthesis and set at standard bond lengths from the parent atoms. All H atoms were given isotropic temperature factors 1·5 times that of the parent atom and allowed to ride on that atom. Atoms C1 and C2 were refined isotropically since they were non-positive-definite when refined anisotropically. All other atoms were given anisotropic thermal parameters. The refinement converged at $R = 0{\cdot}058$, $wR = 0{\cdot}056$, $w = 1{\cdot}0359[\sigma^2(F) + 0{\cdot}000499F^2]^{-1}$. 89 refined parameters; max. shift/e.s.d. $<0{\cdot}01$; difference synthesis max. 0·29, min. $-0{\cdot}34$ e Å^{-3}. Anomalous-dispersion effects included in F.

Scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974). The program packages *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978) were also used. All calculations were carried out on the Dundee University Prime computer. Since the compound was supplied as the L form from the suppliers, this chirality was assumed throughout the calculations.

Discussion. The atomic numbering is shown in the perspective drawing (Fig. 1), and tables of atomic parameters, bond lengths and angles are given (Tables 1 and 2).*

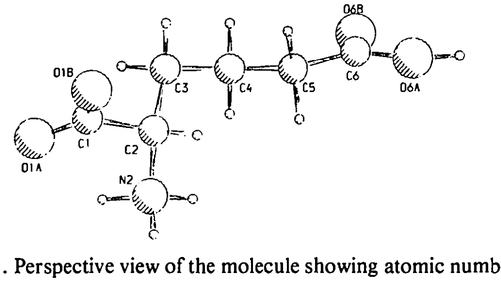

Fig. 1. Perspective view of the molecule showing atomic numbering.

Table 1. *Coordinates* (× 10^4) *for non-hydrogen atoms and equivalent isotropic thermal parameters* (Å^2 × 10^3) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}
C1	−659 (23)	−5141	136 (11)	18 (3)*
C1*A*	−2340 (14)	−5238 (29)	−821 (7)	30 (3)
C1*B*	1720 (15)	−5402 (26)	98 (7)	27 (2)
C2	−1750 (21)	−4719 (28)	1488 (11)	11 (3)*
N2	−4002 (17)	−3364 (27)	1338 (9)	25 (3)
C3	−2544 (27)	−6607 (30)	2097 (12)	30 (5)
C4	−3587 (24)	−6522 (30)	3462 (11)	23 (4)
C5	−1664 (25)	−5764 (29)	4504 (10)	27 (4)
C6	−2361 (30)	−6086 (30)	5899 (14)	33 (5)
C6*A*	−831 (17)	−5269 (28)	6818 (8)	46 (3)
C6*B*	−4252 (20)	−7021 (27)	6194 (8)	50 (4)

* Atoms C1 and C2 isotropic. $U_{eq} = \frac{1}{3}\sum_i\sum_j a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

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

C1*A*–C1	1·257 (11)	O1*B*–C1	1·238 (12)
C2–C1	1·531 (15)	N2–C2	1·496 (13)
C3–C2	1·521 (16)	C4–C3	1·505 (15)
C5–C4	1·496 (15)	C6–C5	1·487 (17)
C6*A*–C6	1·309 (15)	O6*B*–C6	1·221 (15)
C1*B*–C1–O1*A*	127·1 (10)	C2–C1–O1*A*	114·9 (10)
C2–C1–O1*B*	117·9 (10)	N2–C2–C1	110·7 (10)
C3–C2–C1	108·1 (10)	C3–C2–N2	111·5 (9)
C4–C3–C2	116·8 (11)	C5–C4–C3	113·5 (11)
C6–C5–C4	115·0 (11)	C6*A*–C6–C5	115·7 (13)
C6*B*–C6–C5	123·3 (14)	C6*B*–C6–O6*A*	121·0 (13)
O1*A*–C1–C2–C3	86 (2)	N2–C2–C3–C4	−60 (2)
O1*B*–C1–C2–C3	−91 (2)	C2–C3–C4–C5	−61 (2)
O1*A*–C1–C2–N2	−36 (2)	C3–C4–C5–C6	−165 (2)
O1*B*–C1–C2–N2	145 (2)	C4–C5–C6–O6*A*	−173 (2)
C1–C2–C3–C4	177 (2)	C4–C5–C6–O6*B*	5 (2)

The bonds and angles for the amino-acid terminus are in agreement with those found in the analogous L-glutamic acid structures (Taira & Watson, 1977; Lehmann & Nunes, 1980). The amino nitrogen N2 is not coplanar with the adjacent carboxyl group. The angle between the C1, C2, N2 plane and the O1*A*, O1*B*, C1 plane is −35 (1)°, similar to that in the L-glutamic acid molecule (−34·5°) (Taira & Watson, 1977). The torsion angles along the chain are listed in Table 2. There are four hydrogen bonds in the structure: N2···O1*B* ($-x$, y, z) 3·08 (2); N2···O1*B* ($-x$, $0{\cdot}5+y$, $-z$) 2·81 (2); N2···O6*B* ($-1-x$, $0{\cdot}5+y$, $1+z$) 2·84 (2); and O6*A*···O1*A* (x, y, $-1+z$) 2·54 (2) Å. Both the crystal forms of L-glutamic acid referred to above contain similar short O—H···O hydrogen bonds.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51108 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of the Cation Radical Salt 3,4;3′,4′-Bis(methylenedithio)tetrathiafulvalenium Triiodide

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Abstract. $C_8H_4S_8^+.I_3^-$, $M_r = 737\cdot31$, triclinic, $P\bar{1}$, $a = 13\cdot185\,(2)$, $b = 9\cdot433\,(1)$, $c = 7\cdot830\,(1)$ Å, $\alpha = 102\cdot99\,(1)$, $\beta = 104\cdot64\,(1)$, $\gamma = 67\cdot56\,(1)°$, $V = 861\cdot8\,(2)$ Å^3, $Z = 2$, $D_x = 2\cdot84$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu = 63\cdot0$ cm^{-1}, $F(000) = 678$, $T = 298$ K, $R = 0\cdot036$ for 4001 observed reflections. The cation radical and the anion form side-by-side molecular coordination pairs with short S···I contacts of 3·651–3·868 Å aligning their long molecular axes parallel. The cation radicals form face-to-face dimers with mean interplanar distance 3·41 Å. The adjacent dimers are connected to each other making one-dimensional arrays along the *c* axis by side-by-side S···S contacts of 3·395–3·772 Å between the cation radicals.

Introduction. Cation radical salts based on the π-electron-donor molecule 3,4;3′,4′-bis(ethylenedithio)-tetrathiafulvalene (BEDT-TTF) and quasi-linear anions exhibit superconductivity at ambient pressure and temperatures as high as 10 K (Urayama, Yamochi, Saito, Nozawa, Sugano, Kinoshita, Sato, Oshima, Kawamoto & Tanaka, 1988). Among the linear anions, triiodide gives four cation radical salts with BEDT-TTF exhibiting superconductivity at ambient pressure: β-(BEDT-TTF)$_2$I$_3$ (Yagubskii, Shchegolev, Laukhin, Kononovich, Kartsovnik, Zvarykina & Buravov, 1984), γ-(BEDT-TTF)$_6$(I$_3$)$_5$ (Yagubskii, Shchegolev, Pesotskii, Laukhin, Kononovich, Kartsovnik & Zvarykina, 1984), θ-(BEDT-TTF)$_2$I$_3$ (Kobayashi, Kato, Kobayashi, Nishio, Kajita & Sasaki, 1986), and κ-(BEDT-TTF)$_2$I$_3$ (Kato, Kobayashi, Kobayashi, Moriyama, Nishio, Kajita & Sasaki, 1987). Since the superconductivity of these salts is likely to be due to the quasi-two-dimensional character of the electrically conducting BEDT-TTF sheet, it is of great interest to investigate linear anion salts, in particular triiodide salts, of 3,4;3′,4′-bis(methylenedithio)tetrathiafulvalene (BMDT-TTF) in which the two-dimensional interaction is expected to be large compared with that of BEDT-TTF (Kato, Kobayashi, Kobayashi & Sasaki, 1985). In this paper, we report the crystal structure of the triiodide salt of BMDT-TTF.

Experimental. The crystals were prepared by electrochemical oxidation of BMDT-TTF in a benzonitrile solution of tetrabutylammonium triiodide at a platinum electrode (2 mm diameter). A constant current of 1 μA was passed through a fine fritted-glass separating anode and cathode of an electrochemical cell for a month to yield black rhombohedral crystals. A Rigaku automatic four-circle diffractometer with graphite-monochromatized Mo *Kα* radiation was used for intensity data collection from a crystal of dimensions 0·5 × 0·5 × 0·4 mm at room temperature [298 (2) K]. θ–2θ scan employed within $2\theta = 60°$ [$(\sin\theta)/\lambda = 0\cdot704$ Å^{-1}]. Four standard reflections (200, 00$\bar{5}$, $\bar{1}\bar{1}\bar{1}$ and $\bar{1}\bar{2}1$) were monitored every 50 reflections and showed no evidence of crystal deterioration. 4001 observed reflections with

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