

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U <sub>eq</sub>
S2	0.8883 (1)	0.25851 (9)	0.54815 (2)	0.0511 (2)
O1	1.0470 (3)	0.1106 (2)	0.56745 (7)	0.0528 (4)
O6	0.7320 (3)	0.2339 (3)	0.58579 (8)	0.073 (1)
N3	1.0247 (3)	0.4371 (2)	0.56643 (7)	0.040 (1)
C4	1.1289 (3)	0.3847 (3)	0.61623 (8)	0.040 (1)
C5	1.1920 (4)	0.1889 (3)	0.60281 (9)	0.043 (1)
C7	0.9432 (3)	0.6263 (3)	0.5636 (1)	0.047 (1)
C8	0.8028 (5)	0.6700 (4)	0.6084 (1)	0.069 (1)
C9	0.8626 (4)	0.6594 (4)	0.5066 (1)	0.071 (1)
C10	1.2914 (4)	0.5122 (4)	0.6281 (1)	0.063 (1)
C11	1.2221 (4)	0.0686 (3)	0.6523 (1)	0.043 (1)
C12	1.4011 (4)	0.0235 (4)	0.6689 (1)	0.057 (1)
C13	1.4263 (5)	-0.0773 (4)	0.7172 (1)	0.075 (1)
C14	1.2765 (6)	-0.1313 (4)	0.7479 (1)	0.075 (1)
C15	1.0977 (5)	-0.0897 (4)	0.7307 (1)	0.068 (1)
C16	1.0709 (3)	0.0104 (4)	0.6832 (1)	0.052 (1)

Table 2. Selected geometric parameters (Å, °)

S2—O1	1.626 (2)	C7—C8	1.515 (4)
S2—O6	1.456 (2)	C7—C9	1.525 (4)
S2—N3	1.676 (2)	C11—C12	1.379 (4)
O1—C5	1.461 (3)	C11—C16	1.382 (3)
N3—C4	1.474 (3)	C12—C13	1.397 (4)
N3—C7	1.487 (3)	C13—C14	1.362 (4)
C4—C5	1.520 (3)	C14—C15	1.376 (4)
C4—C10	1.508 (3)	C15—C16	1.381 (4)
C5—C11	1.503 (3)		
O1—S2—O6	105.7 (1)	C4—C5—C11	114.0 (2)
O1—S2—N3	91.37 (9)	N3—C7—C8	114.6 (2)
O6—S2—N3	111.8 (1)	N3—C7—C9	109.5 (2)
S2—O1—C5	114.2 (1)	C8—C7—C9	112.1 (2)
S2—N3—C4	108.4 (1)	C5—C11—C12	120.3 (2)
S2—N3—C7	117.9 (2)	C5—C11—C16	120.2 (2)
C4—N3—C7	118.1 (2)	C12—C11—C16	119.4 (2)
N3—C4—C5	102.2 (2)	C11—C12—C13	119.4 (3)
N3—C4—C10	112.9 (2)	C12—C13—C14	120.8 (3)
C5—C4—C10	112.4 (2)	C13—C14—C15	119.9 (2)
O1—C5—C4	106.1 (2)	C14—C15—C16	119.9 (3)
O1—C5—C11	110.6 (2)	C11—C16—C15	120.7 (3)

The absolute configuration was deduced from the known configuration of the starting material, (3), and confirmed by refinement of the alternative enantiomer ( $R = 0.032$ ,  $wR = 0.038$ ). Weak reflections [ $I < 10\sigma(I)$ ] were rescanned (maximum of three rescans) and the counts accumulated to improve accuracy. The H atoms were placed at assumed positions (C—H = 0.95 Å,  $U = 1.2U_{eq}$  of the associated atom) and fixed.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *TEXSAN ORTEP* (Johnson, 1965). Software used to prepare material for publication: *TEXSAN FINISH*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data, and a stereoscopic packing diagram have been deposited with the IUCr (Reference: FG1054). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 2,3-Diphenylmaleic Anhydride, an Analogue of *cis*-Stilbene

MINJOONG YOON,\* YONG HEE KIM AND DAE WON CHO

*Department of Chemistry, Chungnam National University, Taejeon 305-764, Korea*

IL-HWAN SUH,\* JIN-HO LEE, BO-YOUNG RYU  
AND JEONG-RAN PARK

*Department of Physics, Chungnam National University, Taejeon 305-764, Korea*

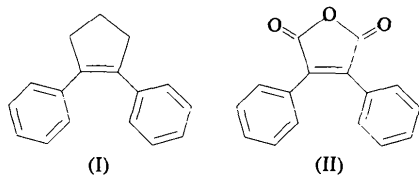
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## Abstract

The asymmetric unit of the title structure, C<sub>16</sub>H<sub>10</sub>O<sub>3</sub>, consists of two molecules which have similar conformations. Each of the two pentagonal rings is planar, while the average dihedral angle between the two phenyl rings within each molecule is 57.4°. The molecules are held together by van der Waals forces, with the shortest intermolecular distance being 2.914(11) Å for O(3')···C(4)( $\frac{1}{2}-x, -y, \frac{1}{2}+z$ ).

### Comment

In order to investigate the structural effects on the *cis* → *trans* photoisomerization of stilbene, a number of 1,2-diphenylcycloalkenes have been used as analogues of *cis*-stilbene (Penn, Gan, Eaton, Chan & Lin, 1988; Hohlneicher *et al.*, 1988). The intramolecular photoreactions of these analogues are strongly hindered, with variations in the reactivity depending on the size and structure of the cycloalkene ring. For example, in a five-membered-ring compound such as 1,2-diphenylcyclopentene, (I) (Bernstein, 1975), the relative twist of the phenyl groups is pronounced and leads to an increased through-space  $\pi$  interaction between the two rings. It has been proposed that this effect results in a higher intramolecular photoreactivity of the five-membered-ring compound compared to that of other 1,2-diphenylcycloalkenes (Hohlneicher *et al.*, 1988). Not all of the diphenylcycloalkenes used in the photoisomerization studies have non-bonding character and it would be interesting to see the effect of the non-bonding electronic character on the relationship between the structure and the intramolecular photoreactivity of a 1,2-diphenylcycloalkene. Thus, we chose 2,3-diphenylmaleic anhydride, (II), an analogue of *cis*-stilbene, and determined its structure by X-ray crystal analysis.



The average O(1)—C(1) and O(3)—C(4) bond length of 1.18 Å, including the exocyclic O atoms, and the average O(2)—C(1) and O(2)—C(4) bond length of 1.39 Å in the pentagonal rings, reflect the double- and single-bond characters, respectively, of compound (II). These values can be compared with those described previously for *N*-carboxy-*L*-valine anhydride (Kanazawa, Ohashi & Sasada, 1984). The average C(2)—C(3) bond length of 1.34 Å in the two pentagonal rings (see Table 2) indicates double-bond character. This value is in good agreement with the C=C distance of 1.337 (6) Å given by Lonsdale (1983), the distance of 1.350 (6) Å in methyl  $\alpha$ -(3-hydroxy-5-oxo-4-phenyl-2*H*-2-furylidene)phenylacetate (C<sub>19</sub>H<sub>14</sub>O<sub>5</sub>; Brassy, Bachet, Molho & Molho, 1985) and 1.328 (8) Å in the ethanol-water solvates of anacrotine and madurensine (C<sub>18</sub>H<sub>25</sub>NO<sub>6</sub> · ½C<sub>2</sub>H<sub>6</sub>O · ½H<sub>2</sub>O; Mackay, Sadek & Culvenor, 1984).

The atoms of the two pentagonal rings lie within 0.032 (9) and 0.009 (8) Å of their respective least-squares planes for molecules *A* and *B*. The exocyclic atoms O(1) and O(3), and atoms C(11) and C(21) of the phenyl rings are displaced alternately on opposite

sides of the best plane. The aromatic rings have normal *sp*<sup>2</sup>-hybrid bond lengths and angles. The aromatic ring atoms C(11)—C(16) and C(21)—C(26) are coplanar.

The angles C(3)=C(2)—C(11) [129.0 (8) in molecule *A* and 132.5 (8)° in molecule *B*] and C(2)=C(3)—C(21) [132.1 (8) and 130.8 (8)°] are larger than angles C(1)—C(2)—C(11) [122.0 (9) and 119.9 (8)°] and C(4)—C(3)—C(21) [121.1 (8) and 121.0 (8)°], and the dihedral angles between the phenyl ring planes for the separate molecules *A* and *B* are 62.1 (4) and 52.7 (4)°, respectively. This implies that the repulsion between phenyl rings is diminished (see Fig. 1). It is also noteworthy that the average angle between the double and single bonds [C(2)=C(3)—C(21) and C(3)=C(2)—C(11)] is 131.1°, which is larger than that for compound (I) (128.6°; Bernstein, 1975).

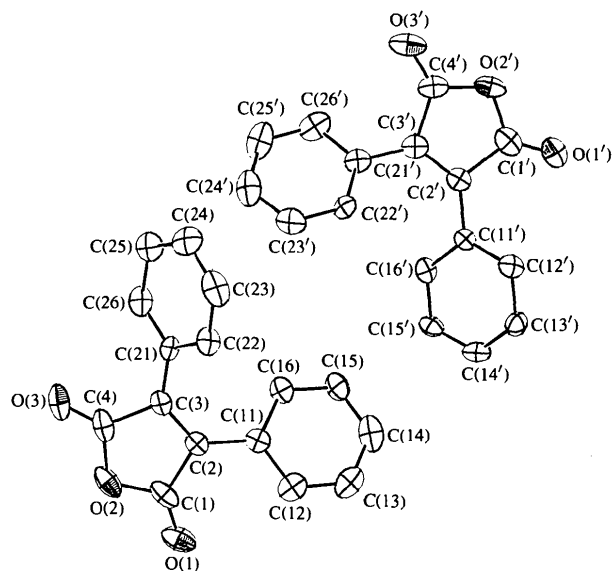


Fig. 1. ORTEP (Johnson, 1971) drawing of the title compound showing the atom-labelling scheme. H atoms are omitted for clarity.

The minimum dihedral angle between the pentagonal and phenyl rings in a molecule is 34.7 (4)°, and that between phenyl rings is 52.7 (4)°. These dihedral angles are smaller than those of compound (I) (Bernstein, 1975). The torsion angle C(11)—C(2)=C(3)—C(21) of  $-14.8$  (8)° for molecule *A* is larger than the value of 6.5 (9)° for molecule *B*, which is rather close to the value of  $-7.5$ ° for 1,2-diphenylcyclopentene (Bernstein, 1975). These geometric differences between compounds (I) and (II) suggest that the through-space  $\pi$  interactions should be lower in compound (II) than in compound (I). Thus, the reactivity of *cis* → *trans* photoisomerization of compound (II) is likely to be reduced when compared to that of compound (I).

## Experimental

The title compound may be purchased from the Aldrich Chemical Co. or synthesized by refluxing  $\alpha,\beta$ -diphenylsuccinonitrile in a mixed solution of sulfuric acid and glacial acetic acid, according to the method of Wawzonek (1940). The compound was further purified by repeated recrystallization from dry diphenyl ether and acetone, alternately. Single crystals (m.p. 432–433 K) were obtained by slow evaporation of an acetone solution of the compound in the final stage of the recrystallization process.

## Crystal data

C <sub>16</sub> H <sub>10</sub> O <sub>3</sub>	Mo K $\alpha$ radiation
$M_r = 250.26$	$\lambda = 0.71069 \text{ \AA}$
Orthorhombic	Cell parameters from 25
<i>Pbca</i>	reflections
$a = 18.959 (3) \text{ \AA}$	$\theta = 9.8\text{--}13.2^\circ$
$b = 13.339 (3) \text{ \AA}$	$\mu = 0.086 \text{ mm}^{-1}$
$c = 19.684 (2) \text{ \AA}$	$T = 297 \text{ K}$
$V = 4977.8 (5) \text{ \AA}^3$	Platelet
$Z = 16$	$0.12 \times 0.1 \times 0.06 \text{ mm}$
$D_x = 1.336 \text{ Mg m}^{-3}$	Green

## Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{\max} = 23^\circ$
$\omega$ - $2\theta$ scans	$h = 0 \rightarrow 20$
Absorption correction: none	$k = 0 \rightarrow 14$
3877 measured reflections	$l = 0 \rightarrow 21$
3861 independent reflections	3 standard reflections
2574 observed reflections	frequency: 100 min
$[ F_o  > 3\sigma( F_o )]$	intensity decay: 2%

## Refinement

Refinement on $F^2$	Unit weights applied
$R = 0.0657$	$(\Delta/\sigma)_{\max} = 0.049$
$wR = 0.0657$	$\Delta\rho_{\max} = 0.23 \text{ e \AA}^{-3}$
$S = 3.0897$	$\Delta\rho_{\min} = -0.23 \text{ e \AA}^{-3}$
2574 reflections	Atomic scattering fac-
423 parameters	tors from <i>SHELX76</i>
All H-atom parameters refined	(Sheldrick, 1976)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

Molecule A	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}$
O(1)	0.1742 (4)	0.1146 (5)	0.1942 (4)	0.086 (6)
O(2)	0.0825 (4)	0.0853 (5)	0.2622 (4)	0.075 (5)
O(3)	0.0197 (3)	0.0498 (6)	0.3569 (4)	0.084 (6)
C(1)	0.1545 (6)	0.0881 (7)	0.2477 (5)	0.063 (7)
C(2)	0.1940 (4)	0.0552 (6)	0.3084 (4)	0.045 (5)
C(3)	0.1476 (4)	0.0282 (6)	0.3567 (4)	0.043 (5)
C(4)	0.0754 (5)	0.0517 (7)	0.3293 (6)	0.060 (7)
C(11)	0.2715 (4)	0.0646 (6)	0.3131 (4)	0.046 (6)
C(12)	0.3145 (6)	0.0453 (7)	0.2572 (6)	0.060 (7)
C(13)	0.3874 (6)	0.0566 (8)	0.2640 (6)	0.070 (8)
C(14)	0.4164 (6)	0.0872 (8)	0.3233 (7)	0.075 (8)
C(15)	0.3740 (5)	0.1074 (9)	0.3794 (6)	0.073 (8)
C(16)	0.3025 (5)	0.0962 (8)	0.3743 (5)	0.062 (7)

C(21)	0.1565 (4)	-0.0194 (7)	0.4223 (4)	0.049 (6)
C(22)	0.2019 (5)	-0.0997 (8)	0.4296 (6)	0.055 (6)
C(23)	0.2087 (6)	-0.1483 (9)	0.4920 (7)	0.074 (8)
C(24)	0.1744 (7)	-0.1103 (10)	0.5489 (6)	0.079 (9)
C(25)	0.1298 (6)	-0.0312 (10)	0.5441 (6)	0.074 (8)
C(26)	0.1205 (5)	0.0141 (9)	0.4797 (6)	0.064 (7)
Molecule B				
O(1')	0.6549 (4)	0.2052 (5)	0.6719 (3)	0.074 (5)
O(2')	0.5705 (4)	0.1781 (5)	0.7498 (3)	0.067 (5)
O(3')	0.4643 (4)	0.1544 (5)	0.7987 (3)	0.083 (5)
C(1')	0.5949 (6)	0.1896 (7)	0.6842 (5)	0.063 (7)
C(2')	0.5321 (5)	0.1808 (6)	0.6372 (4)	0.043 (5)
C(3')	0.4749 (5)	0.1669 (6)	0.6761 (4)	0.050 (6)
C(4')	0.4979 (6)	0.1649 (7)	0.7488 (5)	0.060 (6)
C(11')	0.5411 (4)	0.1961 (7)	0.5643 (4)	0.044 (5)
C(12')	0.5968 (5)	0.1502 (7)	0.5304 (4)	0.054 (6)
C(13')	0.6050 (5)	0.1655 (8)	0.4602 (5)	0.066 (7)
C(14')	0.5591 (6)	0.2287 (9)	0.4263 (5)	0.065 (7)
C(15')	0.5040 (5)	0.2745 (8)	0.4590 (4)	0.056 (6)
C(16')	0.4955 (5)	0.2571 (7)	0.5277 (4)	0.050 (6)
C(21')	0.4006 (5)	0.1528 (6)	0.6576 (4)	0.047 (6)
C(22')	0.3817 (5)	0.1020 (8)	0.5988 (5)	0.053 (6)
C(23')	0.3127 (6)	0.0921 (8)	0.5804 (5)	0.067 (8)
C(24')	0.2605 (6)	0.1307 (9)	0.6207 (7)	0.080 (9)
C(25')	0.2763 (6)	0.1790 (9)	0.6806 (7)	0.082 (9)
C(26')	0.3467 (6)	0.1898 (7)	0.6996 (5)	0.068 (7)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

	Molecule A	Molecule B
O(1)—C(1)	1.17 (1)	1.18 (1)
O(2)—C(1)	1.39 (1)	1.38 (1)
O(2)—C(4)	1.40 (1)	1.39 (1)
O(3)—C(4)	1.19 (1)	1.18 (1)
C(1)—C(2)	1.48 (1)	1.51 (1)
C(2)—C(3)	1.34 (1)	1.34 (1)
C(2)—C(11)	1.48 (1)	1.46 (1)
C(3)—C(4)	1.50 (1)	1.50 (1)
C(3)—C(21)	1.45 (1)	1.47 (1)
C(11)—C(12)	1.39 (1)	1.39 (1)
C(11)—C(16)	1.41 (1)	1.39 (1)
C(12)—C(13)	1.40 (1)	1.41 (1)
C(13)—C(14)	1.35 (1)	1.38 (1)
C(14)—C(15)	1.39 (2)	1.37 (1)
C(15)—C(16)	1.37 (1)	1.38 (1)
C(21)—C(22)	1.38 (1)	1.39 (1)
C(21)—C(26)	1.39 (1)	1.41 (1)
C(22)—C(23)	1.40 (1)	1.36 (1)
C(23)—C(24)	1.39 (2)	1.37 (2)
C(24)—C(25)	1.36 (2)	1.38 (2)
C(25)—C(26)	1.42 (1)	1.39 (1)
O(1)—C(1)—O(2)	120.3 (9)	122.3 (9)
O(1)—C(1)—C(2)	130.9 (10)	130.2 (10)
O(2)—C(4)—O(3)	121.6 (9)	122.7 (9)
O(2)—C(1)—C(2)	108.8 (9)	107.5 (9)
O(2)—C(4)—C(3)	108.5 (8)	107.4 (8)
O(3)—C(4)—C(3)	129.8 (10)	129.9 (10)
C(1)—O(2)—C(4)	107.2 (7)	109.5 (7)
C(1)—C(2)—C(3)	108.6 (8)	107.4 (8)
C(1)—C(2)—C(11)	122.0 (9)	119.9 (8)
C(2)—C(3)—C(4)	106.6 (8)	108.2 (8)
C(2)—C(3)—C(21)	132.1 (8)	130.8 (8)
C(2)—C(11)—C(12)	121.2 (9)	119.9 (8)
C(2)—C(11)—C(16)	119.6 (8)	121.3 (8)
C(3)—C(2)—C(11)	129.0 (8)	132.5 (8)
C(3)—C(21)—C(22)	120.4 (8)	121.2 (8)
C(3)—C(21)—C(26)	121.6 (8)	120.5 (9)
C(4)—C(3)—C(21)	121.1 (8)	121.0 (8)
C(11)—C(12)—C(13)	118.9 (11)	119.4 (9)
C(11)—C(16)—C(15)	120.7 (10)	122.3 (9)
C(12)—C(11)—C(16)	119.2 (9)	118.8 (8)
C(12)—C(13)—C(14)	121.2 (11)	119.6 (10)
C(13)—C(14)—C(15)	120.5 (10)	121.5 (9)
C(14)—C(15)—C(16)	119.6 (10)	118.2 (10)
C(21)—C(22)—C(23)	120.7 (11)	121.2 (10)
C(21)—C(26)—C(25)	121.9 (11)	120.2 (11)
C(22)—C(21)—C(26)	118.0 (10)	118.3 (9)

C(22)—C(23)—C(24)	119.7 (11)	120.2 (11)
C(23)—C(24)—C(25)	121.3 (12)	121.0 (11)
C(24)—C(25)—C(26)	118.1 (12)	119.1 (11)
C(1)—C(2)—C(3)—C(4)	-4.6 (7)	-0.8 (8)
C(1)—C(2)—C(11)—C(12)	-39.4 (9)	46.3 (9)
C(1)—C(2)—C(11)—C(16)	138.8 (12)	-132.8 (12)
C(1)—C(2)—C(3)—C(21)	172.2 (13)	-179.3 (13)
C(2)—C(3)—C(21)—C(22)	-43.3 (10)	34.0 (10)
C(2)—C(3)—C(21)—C(26)	136.2 (14)	-146.7 (14)
C(3)—C(2)—C(11)—C(12)	148.4 (13)	-140.1 (14)
C(3)—C(2)—C(11)—C(16)	-33.4 (9)	40.8 (10)
C(4)—C(3)—C(2)—C(11)	168.4 (12)	-174.8 (13)
C(4)—C(3)—C(21)—C(22)	133.1 (12)	-144.5 (13)
C(11)—C(2)—C(3)—O(2)	171.1 (10)	-174.8 (11)
C(11)—C(2)—C(3)—C(1)	173.0 (12)	-174.2 (14)
C(11)—C(2)—C(3)—C(21)	-14.8 (8)	6.5 (9)

Intensity data were corrected for Lorentz and polarization effects. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985) and refined by full-matrix least squares using *SHELX76* (Sheldrick, 1976), with anisotropic displacement factors for all the non-H atoms. All the H atoms were located from difference Fourier maps and were refined isotropically in the final cycles.

All computations were performed using a MicroVAX 3400 computer at the Chungnam National University. Data collection: *CAD-4 Software* (Enraf-Nonius, 1985). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Molecular graphics: *ORTEPII* (Johnson, 1971).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data, and a stereo packing diagram have been deposited with the IUCr (Reference: TA1003). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Anhydrous DL-Glutamic Acid

JACK D. DUNITZ AND W. BERND SCHWEIZER

*Laboratorium für Organische Chemie, ETH-Zürich, Universitätsstrasse 16, CH-8092 Zürich, Switzerland*

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### Abstract

The conformation of the glutamic acid molecule,  $C_5H_9NO_4$ , in the crystalline racemate differs from that observed previously in the two known chiral modifications. All three forms have a short  $O \cdots O$  hydrogen bond between the side-chain carboxylic acid and the ionized carboxylate of another molecule.

### Comment

There seems to be a persistent legend that DL-glutamic acid crystallizes only as a conglomerate of D- and L-crystals (for example, see Prelog, 1991). This may appear to be supported by the fact that the anhydrous crystalline racemate has not so far been described whereas crystal structures are known for two chiral forms:  $\alpha$ -form,  $P2_12_12_1$ ,  $a = 10.282$ ,  $b = 8.779$ ,  $c = 7.068$  Å (Lehmann & Nunes, 1980);  $\beta$ -form,  $P2_12_12_1$ ,  $a = 5.159$ ,  $b = 17.30$ ,  $c = 6.948$  Å (Lehmann, Koetzle & Hamilton, 1972). According to Dunn & Stoddart (1937), crystallization of aqueous solutions of DL-glutamic acid can yield several anhydrous forms as well as a monohydrate, often crystallizing together out of the same solution. Some of these forms may not have been definitely identified but they include the known chiral forms (Sakata, Susuki & Takenouchi, 1962). In the course of our study of the stability of racemic crystals compared with their chiral counterparts (Brock, Schweizer & Dunitz, 1991) we became interested in the question of whether DL-glutamic acid was indeed obtainable or not. A commercial sample of DL-glutamic acid was examined and found to contain a mixture of small needles and plates corresponding probably to the monohydrate and the anhydrous form described by Dunn & Stoddart (1937). Recrystallization from ethanol gave plate-formed crystals that were found to correspond to the long-sought racemate, the structure of which is described here.

