

and (2) because of the *Z* and *E* configuration: in particular, the strong interaction between the C(14)=O(15) group and the heterocycle in (1) enlarges the C(5)–C(12)–C(13) angle; in contrast, in (2), the carbonyl group interacts with C(11), increasing the C(11)–C(12)–C(13) angle. The π systems of the butenoic chain and the oxazine group interact poorly with each other in both structures; in fact the torsion angle C(4)–C(5)–C(12)–C(13) is -112.8 (3) and -68.9 (3) $^\circ$ in (1) and (2) respectively. The ring O(1), C(2), N(3), C(4), C(5), C(6) is not completely planar in both molecules: in (1) it has an approximate 1E conformation, with total puckering amplitude (Cremer & Pople, 1975) $Q_i = 0.036$ (2) \AA^2 , while its conformation in (2) is approximately 3S_2 , with $Q_i = 0.028$ (2) \AA^2 . In contrast, the phenyl ring of (2) is strictly planar; the dihedral angle between this and the heterocycle mean plane is 37.67 (6) $^\circ$. The cyclization of (I) to give (IV) (see scheme) requires a change of configuration around the C(4)=C(5) double bond in the case of *E* compounds and also, for *Z* isomers, around the C(12)=C(13) double bond. The similarity of the heterocycle in (1) and (2) indicates that the equilibrium (I) \leftrightarrow (II) [and probably (II) \leftrightarrow (III)] is not affected by the *E* or *Z* conformation of the C(12)=C(13) double bond; clearly this bond is thermally much more stable than C(4)=C(5), and does not permit the cyclization (I) \leftrightarrow (IV) of *Z* compounds.

The molecular packing is essentially due to van der Waals forces in both compounds, although some very weak hydrogen bonds are present.

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Acta Cryst. (1989). **C45**, 926–928

Structure of Methyl α -Benzamido- β -mercaptophenyl- β -phenylpropionate*

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Abstract. C₂₃H₂₁NO₃S, $M_r = 391.5$, triclinic, $P\bar{1}$, $a = 9.277$ (1), $b = 9.572$ (1), $c = 11.970$ (1) \AA , $\alpha = 103.09$ (1), $\beta = 92.42$ (1), $\gamma = 102.68$ (1) $^\circ$, $V = 1005.3$ (2) \AA^3 , $Z = 2$, D_m (floatation in KI solution) = 1.31, $D_x = 1.29$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ \AA , $\mu = 0.186$ mm⁻¹, $F(000) = 412$, $T = 293$ K, $R = 0.031$ for 1910 observed reflections. There is a short intramolecular N–H \cdots S contact [3.040 (2) \AA] and an intermolecular N–H \cdots O hydrogen bond [3.109 (3) \AA] which stabilize the crystal and molecular structure. The propionate group is *gauche* with respect to the β -phenyl ring.

Introduction. Phenylalanine is a molecule of importance as an essential amino acid and also as a requisite for the artificial sweetener 'aspartame'. The title compound resulted in an effort to prepare enantioselective α -phenylalanine as a precursor for such a synthesis (Keniya, Natu & Gogate, 1986). This article reports the stereochemical details as determined by X-ray crystallography.

Experimental. Crystal approx. $0.17 \times 0.35 \times 0.45$ mm, Nonius CAD-4F-11M diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode, scan speed 1° min⁻¹, $\theta < 23.5^\circ$, h 0 to 10, k -10 to 10, l -13 to 13, 3189 reflections collected, 1910 judged significant ($|F_o| > 3\sigma|F_o|$), lattice param-

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eters from 23 reflections ($20 < 2\theta < 38^\circ$). Three standard reflections (402, $14\bar{1}$, 142) every 2000s, 3% variation in intensity. No correction for absorption, structure solved by direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Least-squares refinement (*LALS*; Gantzel, Sparks & Trueblood, 1961) of scale factor, positional and anisotropic thermal parameters for non-hydrogen atoms (hydrogen coordinates from a difference Fourier map and isotropic thermal parameters for non-hydrogen atoms to which they are attached; all these parameters held fixed during refinement) converged to $R = 0.033$, $wR = 0.031$, $S = 0.81$, $w(|F_o| - |F_c|)^2$ minimized, $w = (5.0 + 1.0|F_o| + 0.02|F_o|^2)^{-1}$, $(\Delta/\sigma)_{\max} = 0.1$, final $\Delta\rho$ excursions $< 10.21 \text{ e } \text{\AA}^{-3}$. No corrections for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Fig. 1 gives a perspective view of the molecule with atomic numbering. The atomic parameters along with their e.s.d.'s and equivalent isotropic thermal parameters for the non-hydrogen atoms are given in Table 1* while Table 2 gives the bond lengths and angles.

The phenyl rings *A*, *B* and *C* are planar within experimental errors with their mean C—C distances being 1.383 (4), 1.378 (4) and 1.384 (4) Å respectively. The dihedral angle between the rings *A* and *B* is $68.3 (2)^\circ$, *B* and *C* is $86.8 (2)^\circ$ and *A* and *C* is $75.8 (2)^\circ$ respectively. The phenylalanine moiety adopts a conformation similar to that in the structure of *tert*-butoxycarbonyl-*l*- α -phenylalanine (Bats, Fuess, Kessler &

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and some important torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51670 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

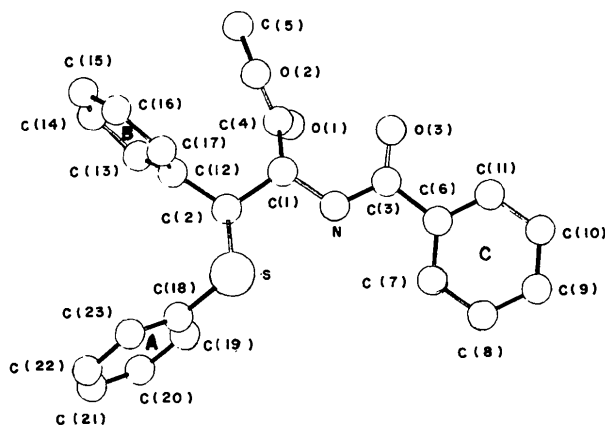


Fig. 1. *PLUTO* diagram (Motherwell & Clegg, 1978) of the molecule showing the crystallographic numbering scheme.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters for non-hydrogen atoms with e.s.d.'s in parentheses

$$B_{eq} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
S	5678 (1)	1157 (1)	8884	3.68 (2)
N	4933 (2)	3388 (2)	10884 (1)	3.62 (5)
O(1)	2601 (2)	4525 (2)	10043 (1)	4.31 (5)
O(2)	1108 (2)	2336 (2)	10026 (1)	4.31 (5)
O(3)	3640 (2)	3359 (2)	12424 (1)	4.61 (5)
C(1)	3661 (2)	2446 (2)	10124 (2)	3.02 (6)
C(2)	4070 (2)	1989 (2)	8881 (2)	2.80 (6)
C(3)	4822 (3)	3722 (2)	12032 (2)	3.15 (6)
C(4)	2419 (2)	3258 (3)	10081 (2)	3.22 (6)
C(5)	-178 (3)	2865 (4)	9763 (3)	6.87 (10)
C(6)	6220 (3)	4509 (2)	12792 (2)	3.07 (6)
C(7)	7611 (3)	4425 (3)	12453 (2)	4.21 (7)
C(8)	8867 (3)	5123 (3)	13221 (3)	4.98 (8)
C(9)	8726 (3)	5900 (3)	14315 (2)	4.45 (7)
C(10)	7352 (3)	5992 (3)	14652 (2)	4.31 (7)
C(11)	6085 (3)	5301 (3)	13889 (2)	3.81 (6)
C(12)	2729 (2)	986 (2)	8118 (2)	2.88 (6)
C(13)	2116 (3)	-408 (3)	8260 (2)	4.57 (7)
C(14)	774 (3)	-1213 (3)	7659 (3)	5.41 (8)
C(15)	51 (3)	-662 (3)	6900 (2)	4.89 (8)
C(16)	680 (3)	697 (3)	6723 (2)	4.66 (7)
C(17)	2020 (3)	1517 (3)	7327 (2)	3.60 (6)
C(18)	6239 (2)	1296 (3)	7505 (2)	3.32 (6)
C(19)	6235 (3)	25 (3)	6672 (2)	4.42 (7)
C(20)	6695 (3)	127 (4)	5604 (2)	5.82 (9)
C(21)	7131 (3)	1459 (5)	5350 (3)	5.93 (8)
C(22)	7149 (3)	2742 (4)	6178 (3)	5.62 (8)
C(23)	6719 (3)	2666 (3)	7270 (2)	4.52 (7)

Table 2. Bond distances (Å) and bond angles ($^\circ$) with e.s.d.'s in parentheses (except phenyl rings)

S—C(2)	1.839 (2)	S—C(18)	1.776 (2)
N—C(1)	1.450 (3)	N—C(3)	1.353 (3)
O(1)—C(4)	1.199 (3)	O(2)—C(4)	1.326 (3)
O(2)—C(5)	1.444 (4)	O(3)—C(3)	1.222 (3)
C(1)—C(2)	1.541 (3)	C(1)—C(4)	1.531 (3)
C(2)—C(12)	1.512 (3)	C(3)—C(6)	1.496 (3)
C(2)—S—C(18)	99.1 (1)	C(1)—N—C(3)	118.7 (2)
C(4)—O(2)—C(5)	116.5 (2)	N—C(1)—C(2)	111.2 (2)
N—C(1)—C(4)	110.6 (2)	C(2)—C(1)—C(4)	107.0 (2)
S—C(2)—C(1)	109.5 (2)	S—C(2)—C(12)	113.4 (2)
C(1)—C(2)—C(12)	109.8 (2)	N—C(3)—O(3)	121.2 (2)
N—C(3)—C(6)	116.8 (2)	O(3)—C(3)—C(6)	122.0 (2)
O(1)—C(4)—O(2)	124.9 (2)	O(1)—C(4)—C(1)	124.8 (2)
O(2)—C(4)—C(1)	110.1 (2)	C(3)—C(6)—C(7)	122.5 (2)
C(3)—C(6)—C(11)	117.7 (2)	C(2)—C(12)—C(13)	121.7 (2)
C(2)—C(12)—C(17)	119.3 (2)	S—C(18)—C(19)	119.6 (2)
S—C(18)—C(23)	120.4 (2)		

Schuck, 1980). The $C\beta$ phenyl group (*B*) is *gauche* to the $C\alpha$ carbon chain [$C(4)—C(1)—C(2)—C(12) = -60.1 (2)^\circ$]. The structure is stabilized by bifurcated short $N—H \cdots S$ and $N—H \cdots O$ interactions. The amide group in fact has short bifurcated $N—H \cdots S$ [$3.040 (2)$; $H \cdots S = 2.82 \text{ \AA}$] and $N—H \cdots O(1)$ [$3.109 (3)$; $H \cdots O(1) = 2.16 \text{ \AA}$] interactions. Such short $N—H \cdots S$ contacts are observed in several compounds involving cysteine and have been discussed in detail earlier (Lee & Suh, 1980; Takasugawa, Koetzle, Kou & Parthasarathy, 1981; Rosenfield & Parthasarathy, 1975). Occurrences of bifurcated short interactions of

this type are also reported in similar compounds (Koetzle, Hamilton & Parthasarathy, 1972; Johnson & Kvik, 1972).

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Structure of 1-Formyl-8-methoxy-3-methyl-5,6-dihydrobenz[*f*]isoquinolin-2(3*H*)-one*

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Abstract. 8-Methoxy-3-methyl-2-oxo-2,3,5,6-tetrahydrobenz[*f*]isoquinoline-1-carbaldehyde, C₁₆H₁₅NO₃, *M_r* = 269.14, monoclinic, *P*2₁/*a*, *a* = 7.509 (1), *b* = 10.123 (1), *c* = 17.391 (2) Å, β = 100.06 (1)°, *V* = 1301.63 (3) Å³, *Z* = 4, *D_m*(floatation in KI solution) = 1.38, *D_x* = 1.37 Mg m⁻³, λ(Mo *Kα*) = 0.7107 Å, μ = 0.1028 mm⁻¹, *F*(000) = 568.0, *T* = 293 K, *R* = 0.037 for 1085 observed reflections. There is a short intramolecular C–H...O interaction owing to the preferred conformational orientation of the aldehyde group. This 'locked-in' conformation results in the formation of a seven-membered ring including the aromatic H atom [C...O = 2.898 (5), H...O = 2.58 Å; angle C–H...O = 99.7°]. The tricyclic moiety has two of its flanking rings planar while the central ring has a 'twist-boat' conformation.

Introduction. The title compound is an unusual derivative which resulted from a Reformatsky–Vilsmeier reaction on tetralone during an attempted preparation of a dihydronaphthaldehyde (Rao & Krishna Rao, 1986). The aim of the present investigation is to determine the stereochemical features of this novel intermediate.

Experimental. Crystal approx. 0.4 × 0.18 × 0.3 mm, Nonius CAD-4F-11M diffractometer, graphite-

monochromated Mo *Kα*, ω/2θ scan mode, scan speed 1° min⁻¹, θ < 23.5°, *h* 0 to 8, *k* 0 to 11, *l* –19 to 19, 2041 reflections collected, 1085 judged significant (*|F_o|* > 3σ*|F_o|*), lattice parameters from 20 reflections

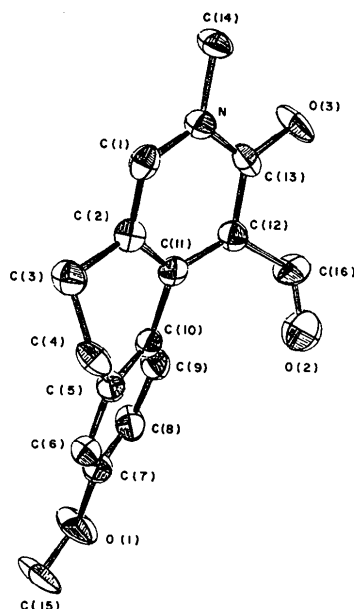


Fig. 1. ORTEP (Johnson, 1965) drawing of the molecule with crystallographic numbering scheme. Thermal ellipsoids are drawn at the 50% probability level.

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