associated with a lengthening of the adjacent bonds. The rigorously planar carboxyl group (mean deviation 0·006 Å) makes a dihedral angle of 27° with the plane of the C(8)–C(13) ring. This tilt of the two planes is composed not only of a rotation about C(9)–C(14) but also of an inclination (~9°) of the two planes sharing a common point C(9).

The packing of the molecules in the crystals is stabilized by a system of hydrogen bonds. All H atoms which are covalently bonded to N and O atoms are involved in the formation of hydrogen bonds. One of the hydrogen atoms of the amino group, H(1), is involved in an intramolecular hydrogen bond, which is characterized by the distances N(18)–H(1)···O(17) = 2·690 (8), N(18)–H(1) = 1·11 (7), H(1)···O(17) = 2·05 (7) Å and the angle N(18)–H(1)···O(17) = 113 (5)°. The intramolecular hydrogen-bond angle is close, within experimental error, to the 119 (3)° found in anthranilic acid II (Boone, Derissen & Schoone, 1977) but significantly different from the 131° found in 2-amino-3-methylbenzoic acid (Brown & Marsh, 1963). The second hydrogen atom of the amino nitrogen, H(2), is directed towards the carbonyl O(16) atom of the carboxyl group of the molecule related by the symmetry operation $-x$, $2-y$, $\frac{1}{2}+z$, and is weakly bonded to this O atom: N(18)–H(2)···O(16) = 3·194 (13), N(18)–H(2) = 1·08 (7), H(2)···O(16) = 2·24 (7) Å, angle N(18)–H(2)···O(16) = 145 (5)°. A strong intermolecular hydrogen bond involving atom H(11) of the carboxyl group connects molecules related by the c glide plane. This intermolecular hydrogen bond, to the molecule at $\frac{1}{2}-x$, y, $z-\frac{1}{2}$, is characterized by the distances O(15)–H(11)···O(17) = 2·66 (2), O(15)–H(11) = 0·87 (7), H(11)···O(17) = 1·87 (7) Å and the angle O(15)–H(11)···O(17) = 152 (7)°. The molecules are linked through this bond in zigzag chains along c.

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2,2,6,6-Tetramethyl-5-phenyl-3-heptyl 3,5-Dinitrobenzoate

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Abstract. $C_{24}H_{30}N_2O_6$, M_r = 442·5, triclinic, $P\bar{1}$, a = 13·929 (2), b = 11·917 (1), c = 7·684 (1) Å, α = 99·02 (2), β = 104·90 (2), γ = 82·69 (1)°, U = 1212·3 (3) Å^3, Z = 2, D_x = 1·212 Mg m^{-3}, μ(Cu $K\alpha$) = 0·681 mm^{-1}. Final R = 0·056 for 2517 reflexions. The two chiral centres of the molecule are in the R,R configuration (S,S in the molecule related by a centre of symmetry).

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Introduction. All measurements for a crystal 0·5 × 0·5 × 0·6 mm, kindly supplied by Dr Arias Pérez, Departamento de Química Orgánica, Universidad de Alcala de Henares, Madrid, were made on a Philips PW 1100 diffractometer with graphite-monochromated Cu $K\alpha$ radiation. The cell parameters were determined by least squares from the 2θ angles for 18 reflexions measured for both positive and negative 2θ angles. Intensities of 4109 independent reflexions were measured up to $\theta = 65°$ with the $\omega/2\theta$ scan technique. No crystal decomposition was observed. After correction for Lorentz and polarization effects, 2517 reflexions were considered as observed with $I > 4\sigma(I)$ and were used in the calculations. The structure was solved with *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977). Anisotropic full-matrix least-squares refinement of the non-H atoms, with unit weights, led to $R = 0{\cdot}11$. A difference synthesis calculated with those reflexions with $\sin\theta/\lambda < 0{\cdot}5$ Å^{-1} showed all H atoms. To prevent bias on $\langle w\Delta^2F\rangle$ *vs* $\langle F_o\rangle$ and *vs* $\langle \sin\theta/\lambda\rangle$, the last steps of the refinement were carried out with weights given by $w = w_1w_2$, where $w_1 = 1/(a + b|F_o|)^2$ and $w_2 = 1/(c + d\sin\theta/\lambda)$, with the coefficients as shown in Table 1, calculated by *PESOS* (Martínez-Ripoll & Cano, 1975). Final refinement, including positional parameters of the H atoms, led to $R = 0{\cdot}056$ and $R_w = 0{\cdot}066$, where $R_w = (\sum w\Delta^2F/\sum w|F_o|^2)^{1/2}$. Table 2 lists the final atomic parameters.*

Table 1. *Coefficients for the weighting scheme*

	a	b		c	d
$F_o \geq 0$	0·25	0·04	$\sin\theta/\lambda < 0{\cdot}43$ Å^{-1}	3·39	−6·13
			$\sin\theta/\lambda \geq 0{\cdot}43$	1·16	−0·72

Table 2. *Atomic parameters*

$U_{eq} = \frac{1}{3}\sum(U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j)$.

	x	y	z	U_{eq} ($\times10^4$ Å^2)
C(1)	0·3024 (2)	1·0524 (2)	1·0537 (4)	488 (9)
C(2)	0·3402 (2)	1·0316 (2)	1·2308 (4)	539 (9)
C(3)	0·3583 (2)	1·1238 (2)	1·3664 (4)	549 (9)
C(4)	0·3414 (2)	1·2349 (2)	1·3299 (4)	573 (10)
C(5)	0·3054 (2)	1·2519 (2)	1·1518 (4)	549 (9)
C(6)	0·2847 (2)	1·1636 (2)	1·0111 (4)	525 (9)
C(7)	0·2730 (2)	0·9578 (2)	0·9007 (4)	509 (9)
C(8)	0·2654 (2)	0·7523 (2)	0·8423 (4)	492 (8)
C(9)	0·2068 (2)	0·7018 (2)	0·9475 (4)	544 (9)
C(10)	0·1072 (2)	0·7732 (2)	0·9552 (4)	488 (9)
C(11)	0·0250 (2)	0·7438 (2)	0·7877 (4)	496 (9)
C(12)	−0·0028 (2)	0·6326 (2)	0·7291 (4)	633 (11)
C(13)	−0·0834 (3)	0·6110 (3)	0·5826 (5)	751 (13)
C(14)	−0·1373 (3)	0·6971 (3)	0·4914 (4)	757 (13)
C(15)	−0·1100 (2)	0·8067 (3)	0·5427 (4)	704 (12)
C(16)	−0·0291 (2)	0·8288 (2)	0·6890 (4)	587 (10)
C(17)	0·3573 (2)	0·6785 (2)	0·7990 (4)	619 (11)
C(18)	0·4298 (3)	0·6444 (3)	0·9708 (6)	898 (15)
C(19)	0·3228 (3)	0·5712 (3)	0·6733 (6)	911 (16)
C(20)	0·4080 (3)	0·7450 (3)	0·6994 (7)	905 (17)
C(21)	0·0756 (2)	0·7742 (2)	1·1366 (4)	621 (11)
C(22)	−0·0271 (3)	0·8403 (3)	1·1227 (5)	746 (13)
C(23)	0·0710 (3)	0·6548 (3)	1·1806 (6)	877 (16)
C(24)	0·1495 (3)	0·8404 (4)	1·2908 (5)	891 (15)
N(25)	0·2854 (2)	1·3697 (2)	1·1083 (4)	753 (12)
N(26)	0·3954 (2)	1·1023 (3)	1·5564 (4)	733 (11)
O(27)	0·4140 (2)	1·0039 (2)	1·5887 (3)	917 (11)
O(28)	0·4051 (3)	1·1838 (3)	1·6739 (4)	1214 (15)
O(29)	0·3163 (3)	1·4456 (2)	1·2270 (4)	1058 (13)
O(30)	0·2401 (3)	1·3860 (2)	0·9579 (4)	1270 (17)
O(31)	0·3002 (1)	0·8567 (1)	0·9608 (3)	540 (6)
O(32)	0·2292 (2)	0·9744 (1)	0·7514 (3)	701 (8)

Discussion. Recent interest in conformational studies made by proton NMR, applied to diastereoisomers whose chiral centres are separated by a methylene

Table 3. *Bond lengths* (Å)

E.s.d.'s are given in parentheses.

C(1)–C(2)	1·376 (4)	C(11)–C(12)	1·401 (4)
C(1)–C(6)	1·392 (4)	C(11)–C(16)	1·386 (4)
C(1)–C(7)	1·509 (3)	C(12)–C(13)	1·387 (4)
C(2)–C(3)	1·390 (4)	C(13)–C(14)	1·366 (5)
C(3)–C(4)	1·375 (4)	C(14)–C(15)	1·376 (5)
C(3)–N(26)	1·470 (4)	C(15)–C(16)	1·392 (4)
C(4)–C(5)	1·368 (4)	C(17)–C(18)	1·519 (5)
C(5)–C(6)	1·385 (3)	C(17)–C(19)	1·527 (4)
C(5)–N(25)	1·471 (4)	C(17)–C(20)	1·524 (6)
C(7)–O(31)	1·333 (3)	C(21)–C(22)	1·529 (5)
C(7)–O(32)	1·187 (3)	C(21)–C(23)	1·526 (5)
C(8)–C(9)	1·516 (4)	C(21)–C(24)	1·540 (5)
C(8)–C(17)	1·538 (4)	N(25)–O(29)	1·214 (4)
C(8)–O(31)	1·478 (3)	N(25)–O(30)	1·198 (4)
C(9)–C(10)	1·542 (4)	N(26)–O(27)	1·218 (4)
C(10)–C(11)	1·515 (3)	N(26)–O(28)	1·213 (4)
C(10)–C(21)	1·564 (5)		

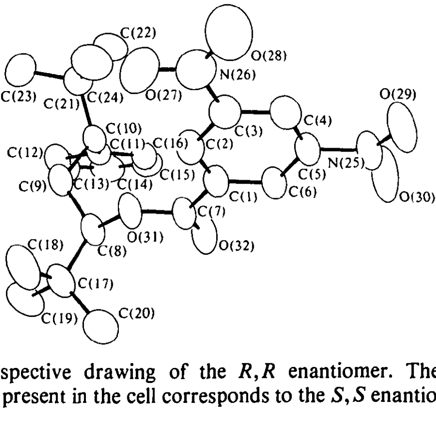

Fig. 1. Perspective drawing of the R,R enantiomer. The other molecule present in the cell corresponds to the S,S enantiomer.

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* Lists of structure factors, anisotropic thermal parameters and H atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35815 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *Bond angles* (°)

E.s.d.'s are given in parentheses.

C(6)–C(1)–C(7)	117·4 (2)	C(11)–C(12)–C(13)	120·7 (3)
C(2)–C(1)–C(7)	122·0 (3)	C(12)–C(13)–C(14)	121·1 (4)
C(2)–C(1)–C(6)	120·5 (2)	C(13)–C(14)–C(15)	119·6 (4)
C(1)–C(2)–C(3)	118·8 (3)	C(14)–C(15)–C(16)	119·6 (3)
C(2)–C(3)–N(26)	119·0 (3)	C(11)–C(16)–C(15)	122·1 (3)
C(2)–C(3)–C(4)	122·4 (3)	C(8)–C(17)–C(20)	109·0 (3)
C(4)–C(3)–N(26)	118·5 (3)	C(8)–C(17)–C(19)	108·7 (3)
C(3)–C(4)–C(5)	116·9 (3)	C(8)–C(17)–C(18)	111·2 (3)
C(4)–C(5)–N(25)	118·3 (3)	C(19)–C(17)–C(20)	108·3 (3)
C(4)–C(5)–C(6)	123·3 (3)	C(18)–C(17)–C(20)	110·4 (3)
C(6)–C(5)–N(25)	118·4 (3)	C(18)–C(17)–C(19)	109·2 (3)
C(1)–C(6)–C(5)	118·0 (3)	C(10)–C(21)–C(24)	108·3 (3)
C(1)–C(7)–O(32)	123·2 (3)	C(10)–C(21)–C(23)	113·0 (3)
C(1)–C(7)–O(31)	110·1 (3)	C(10)–C(21)–C(22)	108·9 (3)
O(31)–C(7)–O(32)	126·6 (4)	C(23)–C(21)–C(24)	110·6 (3)
C(17)–C(8)–O(31)	107·8 (3)	C(22)–C(21)–C(24)	107·0 (3)
C(9)–C(8)–O(31)	104·4 (3)	C(22)–C(21)–C(23)	108·9 (3)
C(9)–C(8)–C(17)	116·7 (3)	C(5)–N(25)–O(30)	118·6 (3)
C(8)–C(9)–C(10)	112·3 (3)	C(5)–N(25)–O(29)	118·0 (3)
C(9)–C(10)–C(21)	113·8 (3)	O(29)–N(25)–O(30)	123·4 (4)
C(9)–C(10)–C(11)	112·3 (3)	C(3)–N(26)–O(28)	118·1 (3)
C(11)–C(10)–C(21)	113·2 (3)	C(3)–N(26)–O(27)	118·7 (3)
C(10)–C(11)–C(16)	120·4 (3)	O(27)–N(26)–O(28)	123·2 (4)
C(10)–C(11)–C(12)	122·6 (3)	C(7)–O(31)–C(8)	119·6 (3)
C(12)–C(11)–C(16)	117·0 (3)		

Table 5. *Torsion angles* (°)

E.s.d.'s are given in parentheses.

C(2)–C(3)–N(26)–O(27)	3·7 (5)
C(4)–C(5)–N(25)–O(29)	11·5 (4)
C(2)–C(1)–C(7)–O(32)	170·4 (3)
C(2)–C(1)–C(7)–O(31)	−7·4 (4)
C(1)–C(7)–O(31)–C(8)	170·5 (3)
O(32)–C(7)–O(31)–C(8)	−7·2 (5)
C(17)–C(8)–O(31)–C(7)	115·2 (3)
C(9)–C(8)–O(31)–C(7)	−120·1 (3)
C(9)–C(8)–C(17)–C(18)	−56·1 (4)
O(31)–C(8)–C(17)–C(18)	60·9 (4)
O(31)–C(8)–C(9)–C(10)	70·8 (3)
C(17)–C(8)–C(9)–C(10)	−170·3 (3)
C(8)–C(9)–C(10)–C(11)	83·9 (4)
C(8)–C(9)–C(10)–C(21)	−145·9 (3)
C(9)–C(10)–C(21)–C(22)	−176·1 (3)
H(81)–C(8)–C(9)–H(91)	−166 (3)
H(81)–C(8)–C(9)–H(92)	80 (4)
H(91)–C(9)–C(10)–H(101)	90 (3)
H(92)–C(9)–C(10)–H(101)	−160 (3)

bridge (Alvarez Ibarra, Arias Perez, Fernandez Gonzalez & Rico Sarompas, 1977), justifies the present X-ray analysis.

Table 6. *Atomic deviations* (Å) *from the least-squares planes* (*Nardelli, Musatti, Domiano & Andreetti,* 1965)

Atoms marked with asterisks were not included in the calculation of the plane. E.s.d.'s are in parentheses.

Plane 1		Plane 2		Plane 4	
C(1)	−0·005 (3)	N(25)	0·000 (4)	C(11)	−0·014 (3)
C(2)	0·007 (3)	O(29)	0·000 (4)	C(12)	0·009 (4)
C(3)	−0·003 (3)	O(30)	0·000 (5)	C(13)	0·006 (4)
C(4)	−0·004 (3)			C(14)	−0·012 (4)
C(5)	0·006 (3)	Plane 3		C(15)	0·003 (4)
C(6)	0·000 (3)			C(16)	0·011 (4)
*N(25)	−0·010 (4)	N(26)	0·000 (4)	*C(10)	−0·119 (4)
*O(29)	0·182 (4)	O(27)	0·000 (4)		
*O(30)	−0·205 (6)	O(28)	0·000 (5)		
*N(26)	−0·036 (4)				
*O(27)	0·015 (4)				
*O(28)	−0·127 (5)				
*C(7)	−0·106 (4)				
*O(32)	−0·283 (4)				
*O(31)	−0·015 (4)				

Angles between planes (°)

1 and 2 = 10·5 (8)
1 and 3 = 4·3 (7)
2 and 3 = 14·3 (8)
1 and 4 = 32·1 (6)

Fig. 1 shows the final X-ray model of the *R*,*R* configuration of the molecule in the cell. Tables 3, 4 and 5 list the bond lengths, bond angles and torsion angles, respectively. Table 6 shows some molecular planes. All intermolecular distances are within the van der Waals limits.

Most of the calculations were performed with XRAY 70 (Stewart, Kundell & Baldwin, 1970). We thank the Centro de Proceso de Datos del Ministerio de Educación for computing facilities on the Univac 1108 computer.

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