1971). Nevertheless, the intra- and intermolecular H···N distances noted here, 2·21 (4) and 2·24 (4) Å, are equal within the range of the experimental errors and are considerably shorter than the sum of the van der Waals radii. So, we can state that the interaction O—H$\begin{smallmatrix}\cdots N'\cdots\\ \cdots N\cdots\end{smallmatrix}$H—O′ observed in the crystal structure of 1-isoquinolyl(phenyl)methanol is an interesting example of a bifurcated hydrogen bond.

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The Structure of 2-(2-Aminobenzoyl)benzoic Acid

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Abstract. $C_{14}H_{11}NO_3$, orthorhombic, $Pca2_1$, a = 13·860 (1), b = 10·135 (1), c = 8·286 (1) Å, Z = 4, V = 1163·9 Å^3, D_x = 1·38, D_m = 1·37 Mg m^{-3}, $F(000)$ = 504. The crystal structure was solved from diffractometer data by direct methods and refined by full-matrix least-squares procedures to an R of 0·040 for 844 observed reflexions. The packing of the molecules in the crystals is stabilized by hydrogen bonds: O(15)—H(11)···O(17) = 2·663 (21) and N(18)—H(2)···O(16) = 3·194 (13) Å. An intramolecular hydrogen bond, N(18)—H(1)···O(17) = 2·690 (8) Å, is present.

Introduction. This determination of the crystal and molecular structure of 2-(2-aminobenzoyl)benzoic acid is a continuation of our studies on substituted 2-aroylbenzoic acids. Suitable crystals for X-ray analysis were kindly provided by Professor J. Gronowska of the Organic Chemistry Department of this University.

Crystal symmetry and approximate cell parameters were determined from rotation and Weissenberg photographs. Space group $Pca2_1$ or $Pcam$ was deduced from systematic absences. On the basis of the distribution of normalized structure factors the noncentrosymmetric space group $Pca2_1$ was selected.

Accurate cell dimensions and intensities were measured on an automated Syntex $P2_1$ four-circle diffractometer, with Cu $K\alpha$ radiation and a graphite monochromator. Reflexions were collected up to $2\theta_{\max}$ = 116°. 844 reflexions from the data set had I >

Table 1. *Fractional atomic coordinates* (×10^4, *for* H ×10^3), *with estimated standard deviations, and* B_{eq} (Å^2)

$$B_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\,\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
C(1)	−503 (3)	8104 (4)	10607 (34)	2·1
C(2)	−788 (3)	9337 (5)	11288 (35)	2·5
C(3)	−1600 (3)	9986 (5)	10630 (35)	2·8
C(4)	−2086 (3)	9482 (5)	9348 (35)	3·0
C(5)	−1808 (4)	8275 (5)	8652 (34)	2·9
C(6)	−1037 (3)	7609 (5)	9275 (35)	2·4
C(7)	331 (3)	7384 (5)	11182 (34)	2·3
C(8)	517 (3)	5990 (4)	10602 (35)	2·3
C(9)	1376 (3)	5645 (4)	9822 (35)	2·2
C(10)	1592 (3)	4328 (5)	9543 (35)	3·0
C(11)	950 (4)	3355 (5)	9972 (35)	3·3
C(12)	76 (4)	3684 (5)	10691 (35)	3·3
C(13)	−134 (4)	5001 (6)	11036 (35)	2·8
C(14)	2000 (3)	6694 (5)	9100 (35)	2·5
O(15)	2897 (2)	6323 (4)	8885 (34)	4·1
O(16)	1696 (2)	7765 (3)	8718 (34)	3·6
O(17)	894 (2)	7838 (3)	12210 (34)	3·3
N(18)	−323 (4)	9885 (5)	12569 (35)	3·8
H(1)	38 (5)	959 (7)	1311 (9)	
H(2)	−50 (5)	1088 (6)	1293 (9)	
H(11)	313 (5)	699 (7)	836 (10)	

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1·96$\sigma(I)$ and were used for structure refinement. No absorption correction was applied. The structure was solved with *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined by a full-matrix least-squares procedure using the program *CRYLSQ* of the XRAY 70 system (Stewart, Kundell & Baldwin, 1970). All heavy atoms were located from an E map calculated with the set of phases with the highest combined figure of merit. After full-matrix least-squares refinement with anisotropic thermal parameters (R = 0·073), a difference map showed all H atoms with electron densities ranging from 0·25 to 0·39 e Å^{-3}. The H atoms, with isotropic thermal parameters equal to those of the carrier atoms, were included in the model during the further refinement but their parameters were not refined. After three subsequent cycles of refinement (R = 0·041) the positional parameters of the H atoms involved in hydrogen bonds were refined. The final R value was 0·040 for 844 reflexions. Unit weights were used throughout. The average and maximum values of the shift/error in the last cycle were 0·01 and 0·25 (for H atoms) respectively. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). Atomic coordinates are listed in Table 1.*

Discussion. The molecular structure and atomic numbering are shown in Fig. 1. The projection of the structure along the *b* axis is illustrated in Fig. 2. Bond lengths and angles are listed in Table 2. Bond lengths in the benzene rings C(1)–C(6) and C(8)–C(13) range from 1·357 (32) to 1·427 (17) Å and from 1·375 (13) to 1·400 (20) Å, respectively. The mean bond lengths, 1·398 (22) and 1·391 (14) Å, are close to the accepted value. The benzene rings are planar (mean deviations 0·006 and 0·014 Å respectively) and are mutually inclined at 71°. The N(18)—C(2) bond length, 1·361 (32) Å, reflects some double-bond character and is consistent with the nearly zero deviation of N(18) from the aromatic plane. According to Chao & Schempp (1977) and Ashfaquzzaman & Pant (1979), there is a correlation between C—NH$_2$ length and N-atom deviation from the ring plane. The amino group is only approximately planar (mean deviation 0·056 Å) and is twisted by ~4° with respect to the adjacent benzene ring.

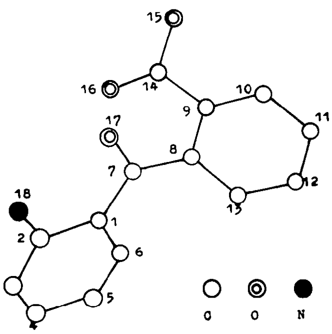

Fig. 1. The molecule viewed along the *c* axis with the numbering system.

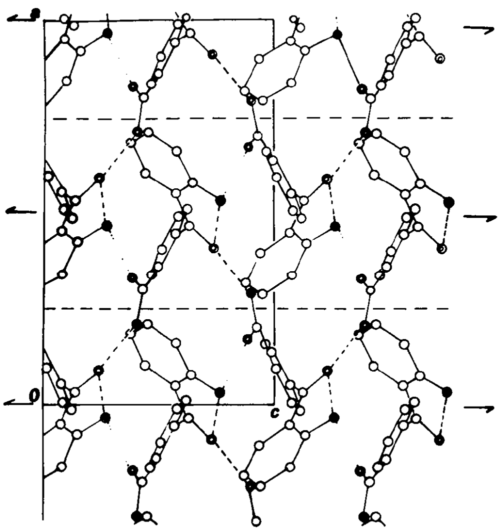

Fig. 2. The crystal structure projected along the *b* axis. The hydrogen bonds are indicated by broken and dotted lines.

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)–C(2)	1·427 (17)	C(8)–C(9)	1·400 (20)
C(2)–C(3)	1·413 (17)	C(9)–C(10)	1·387 (10)
C(3)–C(4)	1·357 (32)	C(10)–C(11)	1·375 (13)
C(4)–C(5)	1·406 (18)	C(11)–C(12)	1·390 (19)
C(5)–C(6)	1·365 (16)	C(12)–C(13)	1·396 (11)
C(6)–C(1)	1·421 (32)	C(13)–C(8)	1·396 (13)
C(2)–N(18)	1·361 (32)	C(9)–C(14)	1·495 (17)
C(1)–C(7)	1·449 (14)	C(14)–O(15)	1·311 (8)
C(7)–O(17)	1·243 (28)	C(14)–O(16)	1·207 (12)
C(7)–C(8)	1·514 (14)		

C(1)–C(2)–C(3)	118·4 (1·9)	C(7)–C(8)–C(9)	121·6 (0·6)
C(2)–C(3)–C(4)	121·5 (1·2)	C(7)–C(8)–C(13)	118·6 (1·4)
C(3)–C(4)–C(5)	120·8 (1·2)	C(13)–C(8)–C(9)	119·3 (0·7)
C(4)–C(5)–C(6)	119·3 (1·9)	C(8)–C(9)–C(10)	120·0 (0·9)
C(5)–C(6)–C(1)	121·8 (1·2)	C(9)–C(10)–C(11)	120·5 (1·2)
C(6)–C(1)–C(2)	118·2 (1·1)	C(10)–C(11)–C(12)	120·2 (0·7)
C(1)–C(2)–N(18)	122·3 (1·2)	C(12)–C(13)–C(8)	120·0 (1·2)
C(3)–C(2)–N(18)	119·3 (1·2)	C(11)–C(12)–C(13)	119·8 (0·8)
C(6)–C(1)–C(7)	119·6 (1·2)	C(8)–C(9)–C(14)	120·0 (0·6)
C(2)–C(1)–C(7)	122·1 (1·8)	C(10)–C(9)–C(14)	119·6 (0·6)
C(1)–C(7)–O(17)	122·7 (1·0)	C(9)–C(14)–O(15)	113·5 (0·8)
O(17)–C(7)–C(8)	117·2 (1·0)	C(9)–C(14)–O(16)	122·8 (0·7)
C(1)–C(7)–C(8)	120·1 (1·5)	O(15)–C(14)–O(16)	123·6 (1·3)

The distortion of the C(1)–C(6) ring geometry is in agreement with the observation that electron-releasing groups (NH$_2$) decrease the endocyclic angles (Domenicano, Vaciago & Coulson, 1975). The decrease in ring angles at the point of substitution is

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35813 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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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