Table 2. *Molecular-packing analysis*

(a) With cell parameters and torques fixed at experimental values (*); (b) with optimized cell parameters and torques fixed; (c) with cell parameters fixed and torques relaxed.

	(a)	(b)	(c)
Cell parameters			
a (Å)	12·056 (7)*	12·398	12·056*
b (Å)	22·420 (8)*	22·255	22·420*
c (Å)	8·841 (2)*	8·820	8·841*
V (Å³)	2390 (2)*	2433	2390*
Molecular parameters			
Translation (Å)	0·31	0·19	0·12
Rotation (°)	0·51	0·57	0·99
Subrotations (°)			
C(2)–N(1)–C(9)–C(10)	111 (1)*	111*	116
C(1)–N(2)–C(15)–C(16)	92 (1)*	92*	74
C(3)–C(4)–O(2)–C(17)	85 (1)*	85*	76
C(5)–C(7)–O(4)–C(21)	−134 (1)*	−134*	−152
C(5)–C(7)–C(8)–O(5)	−52 (1)	−52*	−48

hydrogen bonds by van der Waals potential functions; rather, it should indicate an inherent limitation of the energy sets based on experimental data at room temperature, owing to the nonuniform effect of temperature on the unit-cell parameters: it is different in different crystals and it is different for different cell parameters of the same crystal (anisotropy in the thermal expansion coefficients).

Apart from this effect, the theoretical configuration agrees with the experimental structure. The optimized structure remains very similar with regard to the molecular parameters as shown in Table 2: shifts of positional and orientational molecular parameters are lower than 0·3 Å and 1° respectively. The torsional angles of selected molecular fragments show in two cases (the ethyl group and one of the acetyl chains) a large distortion and this could be attributed to the presence of intramolecular contacts with hydrogen-bond character linking the different rotational groups in the molecule.

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Structure of N-Aromatic Amides. II. XC₆H₄NHCOY*

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Abstract. $T = 296$ K, Cu$K\alpha$, $\lambda = 1·5418$ Å. (1) Salicylanilide ($X = $ H, $Y = o$-HOC₆H₄), C₁₃H₁₁NO₂, $M_r = 213·23$, m.p. $= 406·5$–$407·5$ K, orthorhombic, $Pbca$, $a = 11·018$ (2), $b = 24·781$ (5), $c = 7·760$ (1) Å, $V = 2118·7$ (5) Å³, $Z = 8$, $D_m = 1·33$, $D_x = 1·337$ Mg m⁻³, $\mu = 0·70$ mm⁻¹, $F(000) = 896$, $R = 0·050$ for 1577 unique reflections. (2) o-Nitroacetanilide ($X = o$-NO₂, $Y = $ CH₃), C₈H₈N₂O₃, $M_r = 180·16$, m.p. $= 364$–367 K, monoclinic, $P2_1/n$, $a = 15·507$ (3), $b = 4·9576$ (5), $c = 10·924$ (2) Å, $\beta = 97·68$ (1)°, V

* Part I: Haisa, Kashino, Ueno, Shinozaki & Matsuzaki (1980).

$= 832\cdot3$ (2) Å³, $Z = 4$, $D_m = 1\cdot45$, $D_x = 1\cdot438$ Mg m⁻³, $\mu = 0\cdot77$ mm⁻¹, $F(000) = 376$, $R = 0\cdot052$ for 1241 unique reflections. (3) p-Acetamido-benzoic acid $(X = p\text{-COOH}, Y = CH_3)$, $C_9H_9NO_3$, $M_r = 179\cdot09$, m.p. $= 541$–543 K, triclinic, $P\bar{1}$, $a = 6\cdot9858$ (5), $b = 12\cdot623$ (1), $c = 5\cdot0045$ (4) Å, $\alpha = 102\cdot575$ (6), $\beta = 101\cdot837$ (6), $\gamma = 83\cdot818$ (6)°, $V = 420\cdot64$ (6) Å³, $Z = 2$, $D_m = 1\cdot41$, $D_x = 1\cdot415$ Mg m⁻³, $\mu = 0\cdot86$ mm⁻¹, $F(000) = 188$, $R = 0\cdot042$ for 1250 unique reflections. The effect of the substituents X and Y on the crystal structure of acetanilides is discussed. The nitro and carboxyl groups dominate the structures, while the role of the amide group is only complementary. (1) The molecules form H-bonded ribbons held together with van der Waals forces to form sheets, which are stacked along **b**. (2) The molecules stack to form columns with dipole–dipole interactions between the nitro groups. The C=O group is not involved in any H bonding. (3) The acid dimers are H-bonded to form ribbons. The ribbons form sheets, which are stacked along **b**.

Introduction. In the previous work on N-aromatic amides, the crystal structures of the amides have been classified by the scheme of the N—H···O=C hydrogen bonds into three types: 2_1, glide and translation (Haisa, Kashino, Ueno, Shinozaki & Matsuzaki, 1980). However, factors other than the hydrogen bonds might also dominate the structures. In the present work, the structures of salicylanilide (1), o-nitroacetanilide (2) and p-acetamidobenzoic acid (3) have been determined in order to examine the effects of polar groups on the structure of acetanilides as part of a study on the systematization of the crystal structures of aromatic compounds (Haisa, 1978).

Experimental. Experimental details for compounds (1) to (3) are listed in Table 1. Crystals grown by slow evaporation. D_m by flotation in aqueous KI; Laue group, systematic absences and preliminary lattice constants determined with Weissenberg goniometer; Rigaku AFC-5 four-circle diffractometer equipped with rotating anode; ω–2θ scan method [scan speed $4°$ min⁻¹ in ω, scan range (2θ): $1\cdot2°$ + $0\cdot15°\tan\theta$]; Ni-filtered Cu $K\alpha$, 40 kV, 200 mA; background measured for 4 s on either side of the peak; three standard reflections recorded every 57 reflections; Lorentz and polarization corrections; no absorption correction. All unique reflections within $\sin\theta/\lambda = 0\cdot5617$ Å⁻¹ used in structure analysis and refinement; structures solved by Patterson method, refined by block-diagonal least squares with anisotropic thermal parameters for non-H atoms and isotropic for H atoms; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = 1\cdot0/[\sigma(F_o)^2 + p|F_o| + q|F_o|^2]$ for $|F_o| > 0$, $w = r$ for $|F_o| = 0$. Correction for secondary extinction with $I_{corr} = I_o/(1 - gI_o)$ applied for strongest reflections of (2) and

Table 1. *Experimental details for* (1)–(3)

	(1)	(2)	(3)
Crystal habits	Prismatic c	Prismatic b with {100}, {101}, {10$\bar{1}$}	Plate developed {010}
Solvent for crystallization	Aqueous ethanol, 1:1 in volume	Ligroin	Ethanol
Size of specimen (mm)	$0\cdot15 \times 0\cdot08 \times 0\cdot40$	$0\cdot20 \times 0\cdot45 \times 0\cdot20$	$0\cdot20 \times 0\cdot05 \times 0\cdot43$
Reflections for lattice parameters			
Number	21	20	20
2θ range (°)	14–37	14–27	15–38
Systematic absences	$0kl$, k odd $h0l$, l odd $hk0$, k odd	$h0l$, $h+l$ odd $0k0$, k odd	No condition
$2\theta_{max}(°)$	120	120	120
Range of h, k, l	$0 \le h \le 12$ $0 \le k \le 27$ $0 \le l \le 8$	$-17 \le h \le 17$ $0 \le k \le 5$ $0 \le l \le 12$	$-7 \le h \le 7$ $-14 \le k \le 14$ $0 \le l \le 5$
Variation of standard reflections $\sum(|F_o|/|F_{oinitial}|)/3$	$0\cdot98$–$1\cdot00$	$0\cdot96$–$1\cdot00$	$0\cdot99$–$1\cdot00$
Number of unique reflections	1577	1241	1250
Number of reflections with $|F_o| > \sigma(|F_o|)$	1431	1193	1170
Number of parameters	190	151	155
Extinction correction g	No correction	$4\cdot7 \times 10^{-5}$	$1\cdot8 \times 10^{-5}$
Number of reflections		15	3
R	$0\cdot050$	$0\cdot052$	$0\cdot042$
wR	$0\cdot051$	$0\cdot055$	$0\cdot055$
Constants for w			
P	$-0\cdot0081$	$-0\cdot1116$	$0\cdot0093$
q	$0\cdot0007$	$0\cdot0100$	$0\cdot0003$
r	$1\cdot1586$	$0\cdot7885$	$10\cdot2158$
S	$1\cdot27$	$2\cdot35$	$1\cdot02$
$\Delta\rho$ max. (min.) (e Å⁻³) in final difference map	$0\cdot12$ ($-0\cdot13$)	$0\cdot22$ ($-0\cdot29$)	$0\cdot19$ ($-0\cdot18$)
$(\Delta/\sigma)_{max}$ for non-H (H) in final LS cycle	$0\cdot1$ ($0\cdot3$)	$0\cdot1$ ($0\cdot4$)	$0\cdot3$ ($1\cdot0$)

(3). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Programs *RSSFR*-5 (Sakurai, 1967), *SIGM, HBLS*-V and *DAPH* (Ashida, 1973), *MOLCON* (Fujii, 1979) and *ORTEP* (Johnson, 1965). Computations carried out at the Okayama University Computer Center.

Discussion. The final atomic parameters of (1)–(3) are listed in Table 2.* The thermal ellipsoids of the molecules are shown in Fig. 1, with atomic numbering. Bond lengths and interbond angles are listed in Table 3.

The molecule of (1) consists of three groups: (I) benzene ring (A), (II) amide group, and (III) benzene ring (B), which are planar within $0\cdot009$ (3), $0\cdot006$ (3) and $0\cdot010$ (2) Å, respectively. The dihedral angles between these planes are $10\cdot03$ (7)° for (I) and (II), $21\cdot08$ (6)° for (II) and (III) and $12\cdot06$ (8)° for (I) and (III). The hydroxyl group accepts an intramolecular hydrogen bond from the amide NH $[O(1)\cdots N$ $2\cdot662$ (1), $O(1)\cdots H(N)$ $1\cdot85$ (2), $H(N)$–N $0\cdot98$ (2) Å, $O(1)\cdots H(N)$–N 139 (2)°] and donates a fairly strong hydrogen bond to the amide C=O of the molecule

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42648 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Final atomic coordinates* $(\times 10^4)$ *and equivalent isotropic thermal parameters*

$$B_{eq} = \tfrac{4}{3}\Sigma\beta_{ij}/a_{ij}^{*2}.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
(1) Salicylanilide				
O(1)	2566 (1)	2412·3 (4)	−238 (2)	4·50 (5)
O(2)	−576·8 (9)	3084·6 (5)	1684 (2)	4·66 (6)
N	1418 (1)	3243·8 (5)	1198 (2)	4·16 (6)
C(1)	715 (1)	2321·8 (6)	1309 (2)	3·64 (7)
C(2)	1731 (1)	2084·9 (7)	541 (2)	3·72 (7)
C(3)	1886 (2)	1531·2 (7)	553 (2)	4·45 (8)
C(4)	1039 (2)	1205·8 (7)	1360 (3)	5·14 (9)
C(5)	31 (2)	1428·7 (7)	2140 (2)	4·94 (9)
C(6)	−127 (1)	1980·7 (7)	2098 (2)	4·20 (8)
C(7)	462 (1)	2912·7 (7)	1395 (2)	3·77 (8)
C(8)	1458 (1)	3813·3 (7)	1215 (2)	4·05 (8)
C(9)	520 (2)	4141·8 (8)	1757 (3)	5·40 (9)
C(10)	675 (2)	4694·1 (8)	1719 (3)	6·8 (1)
C(11)	1740 (2)	4923·3 (8)	1197 (3)	7·4 (1)
C(12)	2679 (2)	4597·3 (9)	682 (3)	6·8 (1)
C(13)	2542 (2)	4042·2 (8)	693 (3)	5·36 (9)
(2) *o*-Nitroacetanilide				
O(1)	5167·9 (7)	2134 (3)	7056 (1)	6·14 (6)
O(2)	2581·3 (6)	−3590 (2)	6666 (1)	4·62 (5)
O(3)	1379·5 (7)	−1623 (3)	6002 (1)	6·28 (7)
N(1)	4027·0 (7)	−741 (2)	6612 (1)	4·12 (5)
N(2)	2167·6 (7)	−1810 (2)	6064 (1)	3·95 (5)
C(1)	3515·3 (8)	625 (3)	5662 (1)	3·76 (6)
C(2)	2621·0 (8)	147 (3)	5383 (1)	3·59 (6)
C(3)	2107 (1)	1544 (3)	4445 (1)	4·27 (7)
C(4)	2477 (1)	3421 (3)	3756 (1)	4·81 (7)
C(5)	3357 (1)	3924 (3)	4014 (2)	5·07 (7)
C(6)	3869 (1)	2569 (3)	4936 (2)	4·68 (7)
C(7)	4787·7 (8)	122 (3)	7282 (1)	4·35 (7)
C(8)	5102 (1)	−1683 (4)	8348 (2)	5·60 (8)
(3) *p*-Acetamidobenzoic acid				
O(1)	8956 (2)	931 (1)	2377 (3)	4·41 (6)
O(2)	7732 (2)	245 (1)	−2063 (3)	4·70 (6)
O(3)	−513 (2)	3676 (1)	−3015 (3)	4·61 (6)
N(1)	358 (2)	3284 (1)	1287 (3)	3·62 (6)
C(1)	5694 (2)	1484 (1)	536 (3)	3·35 (7)
C(2)	5482 (3)	2218 (1)	2964 (4)	3·82 (8)
C(3)	3724 (3)	2811 (1)	3175 (4)	3·84 (8)
C(4)	2156 (2)	2675 (1)	966 (3)	3·32 (7)
C(5)	2339 (2)	1928 (1)	−1455 (4)	3·66 (8)
C(6)	4109 (3)	1339 (1)	−1676 (4)	3·69 (7)
C(7)	7588 (2)	854 (1)	299 (4)	3·59 (7)
C(8)	−853 (2)	3737 (1)	−691 (4)	3·45 (7)
C(9)	−2682 (3)	4329 (2)	173 (4)	4·52 (9)

related by a 2_1 axis along **a** [O(1)⋯O(2) 2·639 (1), O(1)—H(O) 0·96 (2), H(O)⋯O(2) 1·68 (2) Å, O(1)—H(O)⋯O(2) 175 (2)°]. The C=O bond length is comparable to that in some salicylanilides (Sindt & Mackay, 1978, 1979*a*), but larger than 1·22–1·23 Å in other anilides in which the C=O participates in a rather weak C=O⋯H—N hydrogen bond (Haisa, Kashino, Ueno, Shinozaki & Matsuzaki, 1980; Kashino & Haisa, 1977). The dihedral angle between (I) and (II) is smaller than the 17–48° found in the anilides whose intermolecular hydrogen bonds are the 2_1 or translation type. The angles between (II) and (III) and (I) and (III) are also small. Such small angles are observed in salicylanilides where the molecular planes overlap each other by a large amount in the crystals (Sindt & Mackay, 1979*b*, 1980), although the corresponding angle between (II) and (III) in 3,5-diiodo-3′,4′-dichloro-salicylanilide is somewhat large, 30·5° (Sindt & Mackay, 1978).

In the molecule of (2), benzene ring (I), amide group (II) and nitro group (III) are planar within 0·003 (2), 0·059 (2) and 0·003 (1) Å, respectively. The dihedral angles between these planes are 21·07 (6)° for (I) and (II), 18·97 (6)° for (I) and (III) and 36·10 (6)° for (II) and (III). The N(1)—C(7) bond is lengthened and C(1)—N(1) is shortened by the electron-withdrawing effect of the nitro group, as evidenced by an increase in C(1)—C(2)—C(3) and a decrease in C(2)—C(1)—C(6). The decrease in double-bond character of the N(1)—C(7) bond thus induced results in a deviation of τ [C(1)—N(1)—C(7)—C(8)] from 180·0° and relatively poor planarity of the amide group: $\tau = 172·0(2)°$ for (2), 179·9 (2)° for (1) and 179·2 (2)° for (3). The amide NH donates the intramolecular hydrogen bond to the
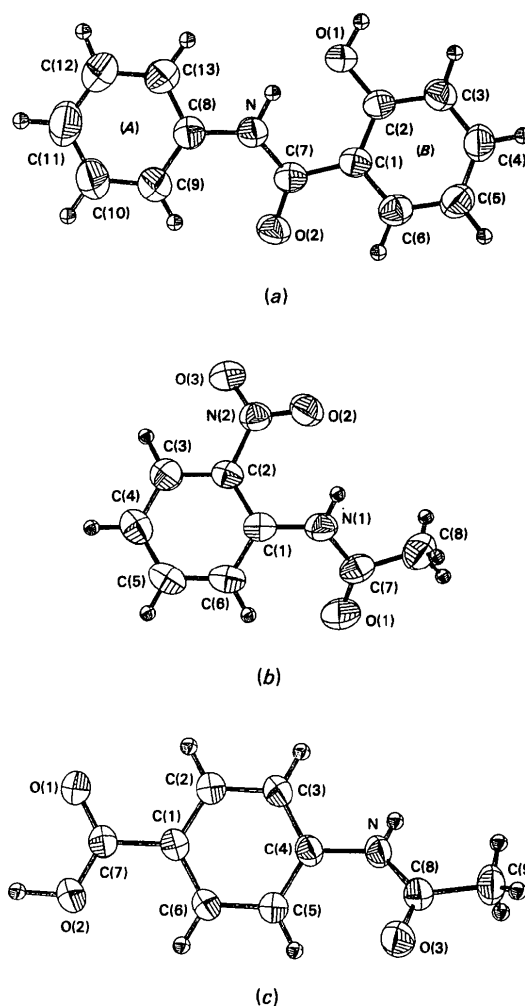

(a)

(b)

(c)

Fig. 1. The thermal ellipsoids of the molecules with atomic numbering for non-H atoms. Ellipsoids of 50% probability are used for the non-H atoms; the H atoms are represented as spheres equivalent to $B = 1·0 \, \text{Å}^2$: (a) salicylanilide (1), (b) *o*-nitroacetanilide (2), (c) *p*-acetamidobenzoic acid (3).

nitro group [N(1)···O(2) 2·657 (2), N(1)—H(5) 0·84 (2), H(5)···O(2) 2·01 (2) Å, N(1)—H···O(2) 133 (2)°]. Thus, the amide C=O is free from any hydrogen bond in contrast with those of other anilides whose crystal structures have so far been reported. The C=O bond is shorter than those in these anilides (Haisa, Kashino, Ueno, Shinozaki & Matsuzaki, 1980; Kashino & Haisa, 1977).

In the molecule of (3), the benzene ring (I), amide group (II) and carboxyl group (III) are planar within 0·008 (2), 0·006 (2) and 0·002 (2) Å, respectively. The dihedral angles between these planes are 40·40 (7)° for

(I) and (II), 5·20 (6)° for (I) and (III) and 35·21 (6)° for (II) and (III), the first being typical for the anilides whose intermolecular hydrogen bonds are of the translation type (Haisa, Kashino, Ueno, Shinozaki & Matsuzaki, 1980). In the crystals of (3) the hydrogen bond is formed between the molecules related by a c translation [N(1)···O(3) 2·963 (2), N(1)—H(5) 0·86 (2), H(5)···O(3) 2·10 (2) Å, N(1)—H(5)···O(3) 179 (2), H(5)···O(3)=C(8) 174·6 (6)°]. The other hydrogen bond is formed between the carboxyl groups related by $\bar{1}$ as found in many carboxylic acids (Kashino & Haisa, 1980) [O(2)···O(1) 2·625 (1), O(2)—H(9) 1·14, H(9)···O(1) 1·49 (3) Å, O(2)—H(9)···O(1) 172 (3), H(9)···O(1)—C(7) 119 (1)°]. A partly disordered model of the carboxyl group is suggested by somewhat short C(7)—O(2) and long C(7)=O(1) and O(2)—H(9), and by the large $B_{iso} = 9$ (1) Å^2 for H(9).

Table 3. Bond lengths (Å) and interbond angles (°) with e.s.d.'s in parentheses

(1) Salicylanilide

O(1)—C(2)	1·368 (2)	C(4)—C(5)	1·380 (3)
O(2)—C(7)	1·242 (2)	C(5)—C(6)	1·379 (3)
N—C(7)	1·344 (2)	C(8)—C(9)	1·381 (3)
N—C(8)	1·412 (2)	C(8)—C(13)	1·383 (3)
C(1)—C(2)	1·397 (3)	C(9)—C(10)	1·380 (3)
C(1)—C(6)	1·396 (3)	C(10)—C(11)	1·365 (4)
C(1)—C(7)	1·492 (3)	C(11)—C(12)	1·372 (4)
C(2)—C(3)	1·383 (3)	C(12)—C(13)	1·384 (4)
C(3)—C(4)	1·383 (3)		

C(7)—N—C(8)	129·3 (1)	O(2)—C(7)—N	122·2 (2)
C(2)—C(1)—C(6)	117·7 (2)	O(2)—C(7)—C(1)	121·1 (2)
C(2)—C(1)—C(7)	125·5 (2)	N—C(7)—C(1)	116·6 (2)
C(6)—C(1)—C(7)	116·8 (2)	N—C(8)—C(9)	124·7 (2)
O(1)—C(2)—C(1)	118·6 (2)	N—C(8)—C(13)	115·7 (2)
O(1)—C(2)—C(3)	120·6 (2)	C(9)—C(8)—C(13)	119·6 (2)
C(1)—C(2)—C(3)	120·9 (2)	C(8)—C(9)—C(10)	119·1 (2)
C(2)—C(3)—C(4)	119·9 (2)	C(9)—C(10)—C(11)	121·7 (2)
C(3)—C(4)—C(5)	120·5 (2)	C(10)—C(11)—C(12)	119·3 (3)
C(4)—C(5)—C(6)	119·2 (2)	C(11)—C(12)—C(13)	120·1 (3)
C(1)—C(6)—C(5)	121·8 (2)	C(8)—C(13)—C(12)	120·3 (2)

(2) o-Nitroacetanilide

O(1)—C(7)	1·201 (2)	C(1)—C(6)	1·405 (2)
O(2)—N(2)	1·229 (2)	C(2)—C(3)	1·395 (2)
O(3)—N(2)	1·218 (2)	C(3)—C(4)	1·370 (2)
N(1)—C(1)	1·395 (2)	C(4)—C(5)	1·379 (2)
N(1)—C(7)	1·371 (2)	C(5)—C(6)	1·372 (2)
N(2)—C(2)	1·459 (2)	C(7)—C(8)	1·498 (3)
C(1)—C(2)	1·400 (2)		

C(1)—N(1)—C(7)	127·5 (1)	C(1)—C(2)—C(3)	121·9 (1)
O(2)—N(2)—O(3)	122·2 (1)	C(2)—C(3)—C(4)	120·0 (1)
O(2)—N(2)—C(2)	119·9 (1)	C(3)—C(4)—C(5)	119·1 (2)
O(3)—N(2)—C(2)	118·0 (1)	C(4)—C(5)—C(6)	121·4 (2)
N(1)—C(1)—C(2)	121·9 (1)	C(5)—C(6)—C(5)	121·3 (2)
N(1)—C(1)—C(6)	121·8 (1)	O(1)—C(7)—N(1)	124·1 (2)
C(2)—C(1)—C(6)	116·3 (1)	O(1)—C(7)—C(8)	122·6 (2)
N(2)—C(2)—C(1)	122·2 (1)	N(1)—C(7)—C(8)	113·3 (1)
N(2)—C(2)—C(3)	116·0 (1)		

(3) p-Acetamidobenzoic acid

O(1)—C(7)	1·254 (2)	C(1)—C(7)	1·481 (3)
O(2)—C(7)	1·279 (2)	C(2)—C(3)	1·379 (3)
O(3)—C(8)	1·219 (2)	C(3)—C(4)	1·383 (3)
N—C(4)	1·420 (3)	C(4)—C(5)	1·383 (3)
N—C(8)	1·350 (3)	C(5)—C(6)	1·384 (3)
C(1)—C(2)	1·382 (3)	C(8)—C(9)	1·503 (3)
C(1)—C(6)	1·392 (3)		

C(4)—N—C(8)	125·6 (2)	C(4)—C(5)—C(6)	119·7 (2)
C(2)—C(1)—C(6)	119·4 (2)	C(1)—C(6)—C(5)	120·4 (2)
C(2)—C(1)—C(7)	120·2 (2)	O(1)—C(7)—O(2)	123·1 (2)
C(6)—C(1)—C(7)	120·4 (2)	O(1)—C(7)—C(1)	119·7 (2)
C(1)—C(2)—C(3)	120·2 (2)	O(2)—C(7)—C(1)	117·2 (2)
C(2)—C(3)—C(4)	120·4 (2)	O(3)—C(8)—N	123·3 (2)
N—C(4)—C(3)	118·6 (2)	O(3)—C(8)—C(9)	121·7 (2)
N—C(4)—C(5)	121·4 (2)	N—C(8)—C(9)	115·0 (2)
C(3)—C(4)—C(5)	119·9 (2)		

(a)

(b)

(c)
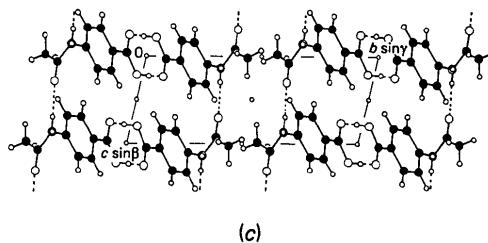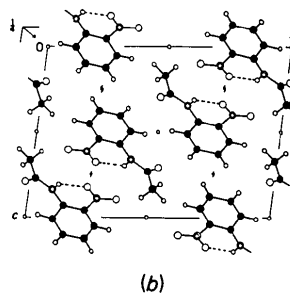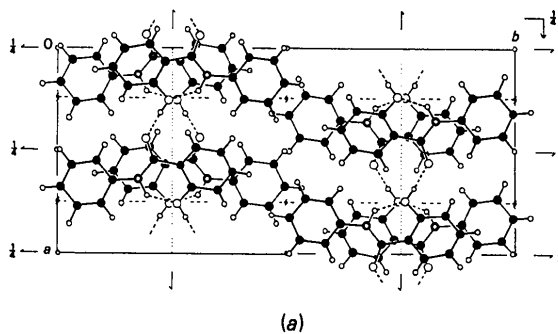
Fig. 2. Projections of the crystal structures: (a) salicylanilide (1) viewed along c, (b) o-nitroacetanilide (2) viewed along b, (c) p-acetamidobenzoic acid (3) viewed along a.

Through the compounds (1) to (3) the C—N—C angles tend to increase with decrease in the dihedral angle between the planes of the benzene ring and the amide group (Haisa, Kashino, Ueno, Shinozaki & Matsuzaki, 1980).

Projections of the crystal structures of (1) to (3) are shown in Fig. 2. In the crystal of (1), nearly flat molecules lie on (001) extending the long axes along **b**. The dihedral angles of the benzene ring (A), amide group and benzene ring (B) with (001) are 20·07 (6), 10·05 (6) and 30·62 (6)°, respectively. A ribbon along **a** is formed by the O—H···O=C hydrogen bond in place of the N—H···O=C bond in acetanilide. The O—H···O angle is close to the ideal value for hydrogen bonding, and the O···H distance is short. The ribbons are held together by van der Waals interactions between the molecules related by a c-glide plane to form a sheet parallel to (010). The sheets related by a b-glide plane are stacked along **b** to form the $Pbca$ structure. Thus, no change occurs in the space group on replacement of the CH_3 group of acetanilide by the o-HOC_6H_4 group (for acetanilide, $Pcab$, $Z = 8$, $a = 9·483$, $b = 19·640$, $c = 7·779$ Å; Brown & Corbridge, 1954). By this choice of $Pcab$ for acetanilide the dimensions of a, b and c are roughly determined by the molecular width, molecular length and molecular overlapping, respectively, for both (1) and acetanilide. Similar interchange between $Pbca$ and $Pcab$ caused by packing requirements has been found in the methyl derivatives of 15,16-dihydrocyclopenta[a]phenanthren-17-one, which have similar molecular shape to (1): for the 1-methyl derivative, $Pbca$, $Z = 8$, $a = 14·344$ (2), $b = 23·121$ (3), $c = 7·571$ (1) Å; for the 11-methyl derivative, $Pcab$, $Z = 8$, $a = 14·355$ (2), $b = 23·153$ (4), $c = 7·526$ (3) Å (Kashino, Zacharias, Peck, Glusker, Bhatt & Coombs, 1986).

In the crystal of (2), the molecules related by a b translation are stacked to form a column with a spacing of 3·519 (2) Å between the benzene ring planes. The columns related by the 2_1 axis at $x = \frac{1}{4}$ and $z = \frac{3}{4}$ are joined together to form a double column. The dihedral angle between the benzene ring planes related by the 2_1 axis is 89·57 (5)°. The columns related by a $\bar{1}$ and an n-glide plane are packed hexagonally as suggested by the morphology of the crystals. Calculations of energies for the van der Waals interactions (E_v) and dipole–dipole interactions (E_d) [by the equations given by Kitaigorodsky (1973), and by assuming the bond moments for N—O, C—O and N—H bonds to be 12·94, 7·67 and 4·37 × 10^{-30} C m, respectively] show that the dipole–dipole interactions between the nitro groups, $(E_d)_{nitro}$, play an appreciable role between the molecules related by the 2_1 axis: $E_v = -10·5$, $(E_d)_{nitro} = -10·9$, $(E_d)_{amide} = 1·7$ kJ mol^{-1}. The dipole–dipole interactions including those between the amide groups exist also between the molecules related by the b translation [$E_v = -13·4$, $(E_d)_{nitro} = -3·8$, $(E_d)_{amide} =$

$-6·3$ kJ mol^{-1}] and the $\bar{1}$ [$E_v = -13·0$, $(E_d)_{nitro} = 0·0$, $(E_d)_{amide} = -3·3$ kJ mol^{-1}]. The 2_1 symmetry operation and the b translation in the $P2_1/n$ structure fit the parallel arrangements of the molecules due to the dipole–dipole interactions in addition to the oblique arrangement of the benzene rings due to the quadrupole–quadrupole interactions. Many derivatives of mononitrobenzene adopt this space group: nitrobenzene (Trotter, 1959), mononitrophenols (Haisa, Kashino & Kawashima, 1980). Whether the hydrogen bond in o-nitroacetanilides is inter- or intramolecular depends on the molecular packing in the crystals: it is intermolecular in form I of 4-methyl-2-nitroacetanilide, $P2_1/c$, and intramolecular in forms II and III (structures undetermined) (Moore, Palmer & Yeadon, 1978).

In the crystal of (3), the planes of the acid dimers lie approximately on ($13\bar{1}$). The dimers related by a c translation are held together by N—H···O=C hydrogen bonds at both ends to form a ribbon. The ribbons are held together along [101] to form a sheet. The sheets are stacked along **b** by van der Waals interactions. The structure is similar to that of some p-substituted benzoic acids in $P\bar{1}$. In particular, a side-by-side arrangement of the molecules in the sheet as shown in Fig. 3 is commonly observed in p-chlorobenzoic acid (Colapietro & Domenicano, 1982), p-dimethylaminobenzoic acid (Vyas, Sakore & Biswas, 1978) and p-methoxycinnamic acid (Bryan & Freyberg, 1975). This arrangement is characterized by the C=O···H interactions and antiparallel orientation of the carboxyl groups.

Judging from the present results, the nitro and carboxyl groups dominate the crystal structures of the acetanilides, while the role of the amide group is only complementary.
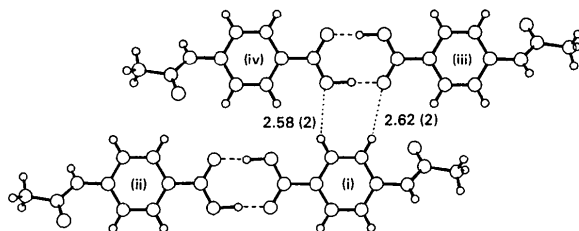
Fig. 3. A side-by-side arrangement of the dimers viewed perpendicular to the benzene ring plane of the molecule (i) of (3). Symmetry code: (i) x, y, z; (ii) $2-x, -y, -z$; (iii) $-1+x, y, -1+z$; (iv) $1-x, -y, -1-z$. Spacing between the benzene ring planes: 0·299 (3) Å for (i) and (ii); 0·237 (3) Å for (i) and (iii); 0·062 (3) Å for (i) and (iv).

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Structure of p-Aminobenzoic Acid–1,3-Dimethyl-2-imidazolidinone (1/1)

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Abstract. $C_7H_7NO_2.C_5H_{10}N_2O$, $M_r = 251\cdot3$, monoclinic, $P2_1/c$, $a = 7\cdot965$ (2), $b = 12\cdot559$ (3), $c = 14\cdot250$ (2) Å, $\beta = 115\cdot01$ (2)°, $V = 1291\cdot8$ (5) Å³, $Z = 4$, $D_x = 1\cdot29$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0\cdot71070$ Å, $\mu = 0\cdot09$ mm⁻¹, $F(000) = 536$, $T = 293$ K, final $R = 0\cdot077$ for 1665 unique reflections. The complex is formed by hydrogen bonding between the carbonyl group of 1,3-dimethyl-2-imidazolidinone and the hydroxy group of p-aminobenzoic acid. The units are packed together with hydrogen bonding between amino groups and carbonyl groups of the p-aminobenzoic acid moieties. Both constituents of the complex are planar with normal bond lengths and angles.

Introduction. 1,3-Dimethyl-2-imidazolidinone (DMI) is an interesting compound in the pharmaceutical field. It is known to have low toxicity (Lien & Kumler, 1968) and also does not bring about any primary irritation with redness, itching and scaling in contact with rabbit skin (Ueda, 1976). Recently, DMI has been studied as an absorption promoter on percutaneous absorption (Sato, Kobayashi, Nishiu & Tamada, 1983). Therefore, it is important to elucidate the interaction of DMI with

a drug. The X-ray crystal structure analysis of the complex of DMI and p-aminobenzoic acid (PABA) has been undertaken to reveal the interaction between the two components.

Experimental. To a solution of 1 g p-aminobenzoic acid in 200 ml ethyl ether, 1 ml of 1,3-dimethyl-2-imidazolidinone was added slowly with stirring at 303 K. Solution was kept standing for at least 48 h at room temperature in the dark. Crystals were filtered out, washed with cold ethyl ether and dried under vacuum. Crystal is transparent and colorless column, m.p. 380 K. Analysis: calc. for $C_{12}H_{17}N_3O_3$: C, $57\cdot11$; H, $6\cdot94$; N, $16\cdot75$%; found: C, $57\cdot37$; H, $6\cdot82$; N, $16\cdot73$%. Electron-impact mass spectrometry: m/z 114 (M^+), 137 (M^+). Single crystal $0\cdot6 \times 0\cdot3 \times 0\cdot3$ mm. Rigaku AFC-5FOS four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Cell parameters refined by least squares for 20 reflections with $7\cdot25 < \theta < 11\cdot23°$. No corrections for absorption. 3184 independent reflections measured, 2θ 0–54° without $F(000)$; range of hkl: h 0→10, k 0→16, l −18→16; $\omega/2\theta$ scan mode; 1666 reflections with $F \geq 3\sigma(F)$. Linear