Table 4. In seven cases there is definite evidence for a C—H···O bond, as they fulfil the criteria proposed by Taylor & Kennard (1982) as well as by Murray-Rust & Glusker (1984).

Statistical analyses of the Cl···Cl (Gnanaguru, Murthy, Venkatesan & Ramamurthy, 1984) and OAc···OAc interactions show that, in general, there is a definite propensity for both chloro and acetoxy groups of symmetry-related molecules to come closer. While there is a significant directionality in the case of Cl···Cl interactions (Gnanaguru *et al.*, 1984), acetoxy interactions are not so specific for crystal engineering. Unlike chloro-substituted compounds, in the acetoxy compounds there is no preponderance for the β-type of packing. There exists a greater preference in acetoxy interactions to align with C=O groups in an anti-parallel orientation (Fig. 4) indicative of dipole–dipole interactions. It may be mentioned that only types III and IV would bring the parent molecules into a packing orientation similar to β type (Schmidt, 1964).

It is noteworthy that all the three acetoxy compounds which are photoreactive prefer the β type of packing. This indicates that not only the interaction between the functional groups (in this case acetoxy) but also the total interactions between the parent molecules are of crucial importance in reaching a packing suitable for reactivity.

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References

BORSCHE, W. (1907). *Chem. Ber.* **40**, 2731–2736.

BRASSY, P. C., BODO, B. & MOLHO, D. (1977). *Acta Cryst.* B**33**, 2559–2562.

BUSING, W. R. (1981). *WMIN. A Computer Program to Model Molecules and Crystals in Terms of Potential Energy Functions.* Oak Ridge National Laboratory, Tennessee, USA.

COHEN, M. D. & SCHMIDT, G. M. J. (1964). *J. Chem. Soc.* pp. 1996–2000.

COHEN, M. D., SCHMIDT, G. M. J. & SONNTAG, F. J. (1964). *J. Chem. Soc.* pp. 2000–2016.

GNANAGURU, K., MURTHY, G. S., VENKATESAN, K. & RAMAMURTHY, V. (1984). *Chem. Phys. Lett.* **109**, 255–258.

GNANAGURU, K., RAMASUBBU, N., VENKATESAN, K. & RAMAMURTHY, V. (1985). *J. Org. Chem.* **50**, 2337–2346.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

JURD, L. & WONG, R. Y. (1980). *Aust. J. Chem.* **33**, 137–154.

KURUTOVA, E. N., POTEKHIM, K. A., STRUCHKOV, YU. T. & ZEFIROV, N. S. (1979). *Sov. Phys. Crystallogr.* **24**, 215–217.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MORISKI, N., FURUKAMA, J., NOZOE, S., ITAI, A. & IITAKA, Y. (1980). *Chem. Pharm. Bull.* **28**, 500–507.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*78. Program for drawing crystal and molecular structures. Univ. of Cambridge, England.

MURRAY-RUST, P. & GLUSKER, J. P. (1984). *J. Am. Chem. Soc.* **106**, 1018–1025.

MURTHY, G. S., ARJUNAN, P., VENKATESAN, K. & RAMAMURTHY, V. (1986). *Tetrahedron*, **43**, 1225–1240.

PECHMANN, V. & WELSH, W. (1884). *Chem. Ber.* **7**, 1646.

PRECIGOUX, P. G., BUSETTA, B., COURSEILLE, C., HOSPITAL, M. & MIQUEL, J. F. (1978). *Acta Cryst.* B**34**, 3300–3305.

RAMASUBBU, N., GNANAGURU, K., VENKATESAN, K. & RAMAMURTHY, V. (1982). *Can. J. Chem.* **60**, 2159–2161.

RODRIGUEZ, J. G., CANO, F. H. & BLANCO, B. G. (1979). *Cryst. Struct. Commun.* **8**, 93–96.

RODRIQUEZ, J. G., VILCHER, J., VERDIR, P. S. & BLANCO, S. G. (1979). *Tetrahedron*, **39**, 199–201.

SCHMIDT, G. M. J. (1964). *J. Chem. Soc.* pp. 2014–2021.

SCHMIDT, G. M. J. (1971). *Pure Appl. Chem.* **27**, 647–678.

SCHWEIZER, W. B. & DUNITZ, J. D. (1982). *Helv. Chim. Acta*, **65**, 1547–1554.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

TAYLOR, R. & KENNARD, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

VAN REMORTERE, F. P. & FLYNN, J. J. (1974). *J. Am. Chem. Soc.* **96**, 6593–6600.

WHITE, D. N. J., CARNDUFF, J., GUY, M. H. P. & BOVILL, M. J. (1977). *Acta Cryst.* B**33**, 2986–2988.

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2-Aminopyridinium Salicylate

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Abstract. $C_5H_7N_2^+.C_7H_5O_3^-$, $M_r = 232{\cdot}2$, orthorhombic, *Pbca*, $a = 15{\cdot}928\ (5)$, $b = 11{\cdot}830\ (5)$, $c = 11{\cdot}768\ (6)$ Å, $V = 2217\ (2)$ Å^3, $Z = 8$, $D_m = 1{\cdot}40$, $D_x = 1{\cdot}391$ g cm^{-3}, graphite-monochromated Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 8{\cdot}04$ cm^{-1}, $F(000) = 976$, $T = 295$ K, final $R = 0{\cdot}050$ for 1116 reflections. Both

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six-membered rings are planar. The cations and anions are linked together in chains along the [001] direction by N—H···O hydrogen bonds.

Introduction. As part of our research program involving the interaction of salicylate drugs with nucleic acid components, amides and amines, we have determined the crystal structure of a salt formed between 2-aminopyridine and salicyclic acid. Generally, the carboxyl —OH group of the acid is a strong hydrogen-bond donor whereas the C=O group is a weak hydrogen-bond acceptor. However, the negatively charged carboxylate group is a strong hydrogen-bond acceptor.

Table 1. *Atomic parameters*

E.s.d.'s given in parentheses refer to the least significant digit(s). B_{eq} is calculated according to Hamilton (1959).

	x	y	z	B_{eq}(Å^2)
O(1)	0·5685 (2)	0·3809 (2)	0·4411 (2)	3·71 (7)
O(2)	0·6254 (2)	0·4180 (2)	0·6093 (2)	3·65 (7)
O(3)	0·7240 (2)	0·5839 (3)	0·6371 (2)	4·87 (9)
C(11)	0·6596 (2)	0·5402 (3)	0·4571 (3)	2·81 (9)
C(12)	0·7113 (2)	0·6074 (3)	0·5250 (3)	3·22 (10)
C(13)	0·7523 (3)	0·7011 (3)	0·4794 (4)	4·14 (12)
C(14)	0·7431 (3)	0·7262 (4)	0·3662 (4)	4·32 (12)
C(15)	0·6931 (3)	0·6602 (4)	0·2976 (4)	4·32 (12)
C(16)	0·6527 (3)	0·5672 (4)	0·3420 (3)	3·81 (11)
C(17)	0·6138 (3)	0·4402 (3)	0·5049 (3)	2·97 (9)
N(2)	0·5532 (3)	0·2273 (4)	0·7196 (3)	4·30 (11)
C(2)	0·4998 (2)	0·1705 (3)	0·6526 (3)	3·20 (10)
N(1)	0·4894 (2)	0·2074 (3)	0·5446 (3)	3·18 (8)
C(3)	0·4532 (3)	0·0764 (4)	0·6887 (3)	3·82 (11)
C(4)	0·4008 (3)	0·0236 (4)	0·6142 (4)	4·22 (12)
C(5)	0·3918 (3)	0·0636 (4)	0·5019 (4)	4·16 (12)
C(6)	0·4367 (3)	0·1552 (4)	0·4703 (3)	3·70 (11)

Table 2. *Bond distances* (Å), *angles* (°) *and hydrogen bonding* (Å, °) *in 2-aminopyridinium salicylate*

O(1)–C(17)	1·256 (4)	C(15)–C(16)	1·378 (6)
O(2)–C(17)	1·270 (4)	N(1)–C(6)	1·361 (5)
C(11)–C(17)	1·499 (5)	N(1)–C(2)	1·354 (5)
C(11)–C(12)	1·396 (5)	C(2)–C(3)	1·403 (6)
C(11)–C(16)	1·396 (5)	C(3)–C(4)	1·363 (6)
C(12)–O(3)	1·363 (4)	C(4)–C(5)	1·410 (6)
C(12)–C(13)	1·394 (6)	C(5)–C(6)	1·350 (6)
C(13)–C(14)	1·372 (6)	C(2)–N(2)	1·340 (5)
C(14)–C(15)	1·377 (6)		

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

D–H···A	D···A	H···A	∠D–H···A
N(1)–H(N1)···O(1)	2·699 (4)	1·73 (4)	170 (3)
N(2)–H2(N2)···O(2)	2·845 (4)	1·95 (5)	158 (4)
O(3)–H(O3)···O(2)	2·535 (4)	1·54 (6)	163 (6)
N(2)–H1(N2)···O(1^i)	2·914 (4)	2·06 (4)	169 (4)

Symmetry code: (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

Experimental. Prismatic crystal (from alcohol/hexane, *v:v*), 0·4 × 0·3 × 0·2 mm; D_m by flotation, CCl_4/hexane; Syntex $P2_1$ diffractometer; lattice parameters from 25 reflections ($40 < 2\theta < 60°$); θ–2θ scan; $2\theta < 130°$; $h = 0$–18, $k = 0$–13, $l = 0$–13; 2248 reflections measured, 1743 unique, 1116 with $I > 2\sigma(I)$. Intensities of three standard reflections (032, 220, 223) varied within 3%. Lp and semi-empirical absorption corrections (North, Phillips & Mathews, 1968), min., max. transmission coefficients 0·92, 1·04; direct methods (*MULTAN*78; Main, 1980); anisotropic full-matrix refinement on F [*CRYGLS;* modified version of *ORFLS* (Busing, Martin & Levy, 1962)]; H (from ΔF synthesis) isotropic; final $R = 0·050$,* $wR = 0·048$, $w = 1/\sigma^2$, $(\Delta/\sigma)_{max} = 0·05$, $S = 1·220$; max., min. heights in final ΔF synthesis 0·20, −0·25 e Å^{-3}; atomic scattering factors are those of *International Tables for X-ray Crystallography* (1974). Table 1 lists atomic coordinates and Table 2 bond lengths and angles.

Discussion. 2-Aminopyridine and salicylic acid formed a salt, with the proton from the carboxyl group covalently bonded to the ring N(1) atom of

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44427 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

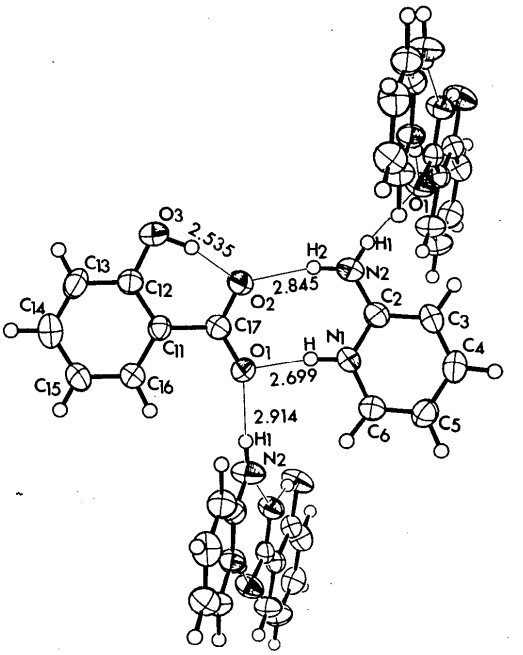

Fig. 1. Molecular structure and atomic numbering scheme. Hydrogen bonds are represented by thin lines.

2-aminopyridine (Fig. 1). Crystal integrity is maintained through a series of intermolecular (N—H···O) hydrogen bonds with N···O distances in the range 2·699 (4) to 2·914 (5) Å. There is a strong intramolecular hydrogen bond in the salicylate anion with O(hydroxyl)···O(carboxyl) = 2·535 (4) Å. The six-membered rings of pyridine and salicylate are planar within 0·007 and 0·011 Å, respectively. The distance and angles in the complex are normal with N(1)—C(2) and N(1)—C(6) longer and C(2)—N(2) shorter than those observed in the unprotonated parent compound (Hsu & Craven, 1974). The nature of the molecular interaction and the mode of hydrogen bonding in this structure are quite similar to those found in 9-methyladeninium salicylate (Gellert & Hsu, 1983) except that the N(1)···O(carbonyl) separation is longer in the present compound. This suggests that 9-methyladenine is a stronger electron donor than 2-aminopyridine.

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References

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee, USA.

GELLERT, R. W. & HSU, I.-N. (1983). *J. Cryst. Spectrosc. Res.* **13**, 99–105.

HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.

HSU, I.-N. & CRAVEN, B. M. (1974). *Acta Cryst.* B**30**, 994–997.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

MAIN, P. (1980). *National Resources Computer Chemistry Software Catalog*, Vol. 1, Program No. XS01 (*MULTAN*78).

NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

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Structure of 7-Chloro-5-(2,6-dichlorophenyl)-1,3-dihydro-2*H*-1,4-benzodiazepin-2-one*

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Abstract. $C_{15}H_9Cl_3N_2O$, $M_r = 339{\cdot}6$, monoclinic, $P2_1/n$ (non-standard setting of $P2_1/c$), $a = 12{\cdot}311$ (1), $b = 10{\cdot}082$ (2), $c = 12{\cdot}360$ (1) Å, $\beta = 104{\cdot}76$ (7)°, $V = 1483{\cdot}5$ Å^3, $Z = 4$, $D_x = 1{\cdot}52$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}55$ mm^{-1}, $F(000) = 688$, $T = 293$ K, $R = 0{\cdot}036$ for 1868 observed reflections. The seven-membered ring adopts a slightly distorted cycloheptatriene-like boat conformation with bow and stern angles of 59·4 (5) and 31·7 (5)°, respectively. The angle between the 5-phenyl ring and the fused benzo moiety is 78·7 (3)°. The C(5)—C(phenyl) bond length is 1·498 (3) Å.

Introduction. The 5-phenyl-1,4-benzodiazepines have been extensively used in clinical practice as anxiolytics, hypnotics, anticonvulsants and muscle relaxants (*e.g.* Hamor & Martin, 1983). The title compound (Fryer, Leimgruber & Trybulski, 1982) differs from the clinically used benzodiazepines in that the 5-phenyl ring is substituted with chlorine at both *ortho* positions. Its affinity for the benzodiazepine receptor *in vitro* is the same, within the limits of experimental error, as that of the mono-substituted analogue (Squires & Braestrup, 1977; Braestrup & Squires, 1978). As part of a continuing study of structure–activity relationships for this class of compounds, we now report the crystal structure of the title compound.

Experimental. Crystals were grown from ethanol. A crystal of size 0·5 × 0·4 × 0·1 mm was mounted on an Enraf–Nonius CAD-4 diffractometer. Lattice parameters from 25 reflections having $10 < \theta < 19°$. Data collected using ω–2θ scans, $2 < \theta < 25°$; two standard reflections measured every 2 h showed no significant variation over period of data collection; 3231 reflections scanned, 2906 unique, $R_{int} = 0{\cdot}019$; of these 1868 having $I > 2{\cdot}5\sigma(I)$ were used in the analysis, index range h −15 to 14, k 0 to 12, l 0 to 15; no absorption corrections applied; structure solved by direct methods; all H atoms located in difference Fourier maps; heavier atoms refined with anisotropic temperature factors, H atoms refined isotropically; full-matrix least-squares refinement on F magnitudes. $R = 0{\cdot}036$, $wR = 0{\cdot}049$, $w = 1/[\sigma^2(F) + 0{\cdot}0008F^2]$, max. Δ/σ 0·06; residual electron density in final Fourier difference map within +0·25 and −0·38 e Å^{-3}; atomic scattering factors were taken from *International Tables*

* Contribution from the Crystallography Unit, Universities of Aston and Birmingham.

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