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N-(4-Acetylphenyl)acetohydroxamic Acid

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Abstract. $C_{10}H_{11}NO_3$, $M_r = 193{\cdot}20$, monoclinic, $P2_1/n$, $a = 23{\cdot}539\,(5)$, $b = 8{\cdot}473\,(2)$, $c = 9{\cdot}540$ Å, $\beta = 99{\cdot}44\,(2)°$, $V = 1876{\cdot}9\,(7)$ Å^3, $Z = 8$, $D_x = 1{\cdot}367$ g cm^{-3}, Ni-filtered Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 7{\cdot}55$ cm^{-1}, $F(000) = 816$, $T = 138\,(2)$ K, $R = 0{\cdot}057$ for 3018 data. The hydroxamate groups of the two crystallographically unique molecules have a *trans* conformation. Both molecules are roughly planar and have similar molecular conformations and identical dimensions, which are compared with those of other *N*-substituted acetohydroxamic acids. The molecules are linked by two intermolecular hydrogen bonds with lengths 2·631 (2) and 2·636 (2) Å.

Introduction. Under iron-limiting conditions, microorganisms produce a variety of small chelating agents which solubilize ferric iron in the environment and transport the iron to the cell. Many of these metabolites chelate the iron through hydroxamic acid groups. Both naturally occurring and synthetic hydroxamic acids have been used as therapeutic agents for treatment of iron overload (Anderson & Hiller, 1977). Crystal structures of synthetic hydroxamic acids with a *trans* conformation include *N,N'*-dihydroxy-*N,N'*-diisopropylhexanediamide (hipa) (Smith & Raymond, 1980), *N*-(4-cyanophenyl)acetohydroxamic acid (*p*-cnpa) (Mocherla, Powell, Barnes & van der Helm, 1983) and *N*-(3-cyanophenyl)acetohydroxamic acid (*m*-cnpa) (Mocharla, Powell & van der Helm, 1984). This article describes the crystal and molecular structure of *N*-(4-acetylphenyl)acetohydroxamic acid (apa).

Experimental. A sample of the title compound was kindly supplied by Dr A. L. Crumbliss, P. M. Gross Chemical Laboratory, Department of Chemistry, Duke University, Durham, North Carolina. Colorless plate-shaped crystals were grown by diffusing benzene into a solution of the compound in ethyl acetate. Crystal: 0·08 × 0·25 × 0·50 mm, Enraf–Nonius CAD-4, 48 reflections used for lattice constants, systematic absences: $h0l$, $h + l = 2n + 1$, $0k0$, $k = 2n + 1$, no absorption correction applied, all data $2\theta < 150°$ in $-29 \le h \le 29$, $0 \le k \le 10$, $0 \le l \le 11$ collected, three intensity monitors had a maximum difference of 0·074 and an e.s.d. of 0·019, 3826 unique data measured, 808 unobserved data [$F < 3{\cdot}8\sigma(F)$], solved by *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), refined by block full-matrix least squares minimizing $\sum w(|F_o| - |F_c|)^2$, hydrogens located on a difference electron density map, all heavy atoms refined anisotropically, hydrogens refined isotropically, $R = 0{\cdot}057$, $wR = 0{\cdot}078$, $S = 2{\cdot}79$, $w = 1/\sigma^2(F)$, maximum and average shift-to-e.s.d. ratios = 0·48, 0·10, maximum and minimum on final difference map = 0·35 and 0·34 e Å^{-3}, C, N and O scattering factors from Cromer & Mann (1968), H scattering factors from Stewart, Davidson & Simpson (1965), programs used include *SHELX* (Sheldrick, 1976) and *ORTEP* (Johnson, 1965).

Discussion. The final coordinates of the nonhydrogen atoms are given in Table 1. The atom-numbering scheme is shown in Fig. 1 (Johnson, 1965). Bond

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distances, angles and selected torsion angles are presented in Table 2.* The two independent molecules have identical bond distances and angles. They are related by a noncrystallographic c glide plane at $y = 0{\cdot}34$.

Crumbliss and co-workers have investigated the pK_a values and the ΔH_a and ΔS_a of acid dissociation for a series of synthetic hydroxamic acids (Monzyk & Crumbliss, 1980; Brink & Crumbliss, 1984; Brink, Fish & Crumbliss, 1985). For substituted *N*-phenyl acetohydroxamic acids the pK_a values vary very little and the variations are only weakly correlated with the Hammett parameters. In the case of the substituted *N*-methylbenzohydroxamic acids the variation in pK_a is larger and the correlation with the Hammett parameter is also stronger. For both types of compounds there exists a positive correlation between the values of ΔH_a and ΔS_a of acid dissociation. The ΔH_a values vary between 4·2 and 46 kJ mol^{-1}. One does not expect a variation of bond distances for the hydroxamic acids as a function of pK_a. Instead, a correlation with the ΔH_a values could exist. However, thermodynamic measurements indicate that the solvation effects largely determine the values of ΔH_a and a correlation between ΔH_a values and geometry, therefore, is not expected. It should therefore be possible to obtain an average geometry for the hydroxamic acid group from the results of a number of structure determinations of these compounds (Table 2, column *C*).

The conformation of the hydroxamate group is *trans* and approximately planar with some out-of-plane deformation at *N* (χ_N) and twisting around the C–N bond (τ) (Winkler & Dunitz, 1971).

* Lists of anisotropic thermal parameters, hydrogen parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43414 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters* ($x \times 10^5$, y *and* $z \times 10^4$) *and isotropic equivalent thermal parameters* ($\times 10^4$ Å^2)

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

	x	y	z	U_{eq}
C(1*A*)	28846 (8)	4294 (2)	4125 (2)	189 (8)
C(2*A*)	25334 (9)	3200 (2)	4677 (2)	216 (9)
C(3*A*)	19419 (9)	3317 (2)	4342 (2)	223 (9)
C(4*A*)	16804 (9)	4494 (2)	3427 (2)	204 (8)
C(5*A*)	20350 (9)	5568 (3)	2879 (2)	232 (9)
C(6*A*)	26262 (9)	5485 (2)	3212 (2)	236 (9)
C(7*A*)	10417 (9)	4661 (3)	3045 (2)	268 (10)
O(7*A*)	8365 (7)	5685 (2)	2221 (2)	416 (9)
C(8*A*)	6616 (10)	3566 (3)	3712 (3)	359 (12)
N(9*A*)	34965 (7)	4225 (2)	4412 (2)	206 (7)
O(9*A*)	37858 (6)	5402 (2)	3774 (2)	249 (7)
C(10*A*)	38359 (9)	3282 (2)	5352 (2)	225 (9)
O(10*A*)	36231 (6)	2312 (2)	6080 (2)	288 (7)
C(11*A*)	44759 (10)	3449 (3)	5430 (3)	310 (11)
C(1*B*)	28932 (8)	2588 (2)	9126 (2)	185 (8)
C(2*B*)	25513 (8)	3698 (2)	9698 (2)	208 (9)
C(3*B*)	19552 (9)	3605 (2)	9358 (2)	209 (9)
C(4*B*)	16860 (8)	2436 (2)	8452 (2)	213 (8)
C(5*B*)	20345 (9)	1343 (3)	7898 (2)	228 (9)
C(6*B*)	26275 (9)	1406 (2)	8226 (2)	203 (8)
C(7*B*)	10482 (9)	2299 (3)	8075 (2)	257 (9)
O(7*B*)	8330 (7)	1229 (2)	7311 (2)	421 (9)
C(8*B*)	6792 (10)	3485 (3)	8685 (3)	333 (11)
N(9*B*)	35031 (7)	2638 (2)	9419 (2)	197 (7)
O(9*B*)	37854 (6)	1453 (2)	8766 (2)	241 (7)
C(10*B*)	38540 (9)	3568 (2)	10353 (2)	217 (9)
O(10*B*)	36477 (6)	4557 (2)	11081 (2)	275 (7)
C(11*B*)	44919 (9)	3374 (3)	10419 (3)	296 (11)

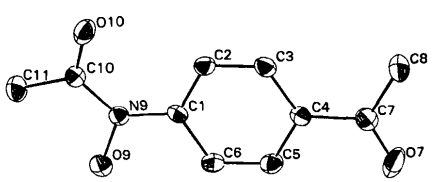

Fig. 1. Perspective view with atom numbers.

Table 2. *Bond distances* (Å), *angles* (°) *and selected conformational angles* (°) *with e.s.d.'s in parentheses*

Columns *A* and *B* refer to the two independent molecules in the asymmetric unit, while column *C* lists weighted average bond distances for the *N*-phenylacetyl hydroxamic acid from the results on *p*-cnpa (Mocherla *et al.*, 1983), *m*-cmpa (Mocharla *et al.*, 1984) and the present structure.

	A	*B*	*C*
N(9)–O(9)	1·401 (2)	1·404 (2)	1·402 (2)
N(9)–C(10)	1·359 (3)	1·363 (3)	1·358 (3)
C(10)–O(10)	1·234 (3)	1·237 (3)	1·236 (2)
C(10)–C(11)	1·503 (3)	1·502 (3)	1·502 (1)
N(9)–C(1)	1·423 (2)	1·418 (2)	1·421 (2)
C(1)–C(2)	1·401 (3)	1·405 (3)	
C(2)–C(3)	1·380 (3)	1·390 (3)	
C(3)–C(4)	1·400 (3)	1·396 (3)	
C(4)–C(5)	1·394 (3)	1·397 (3)	
C(5)–C(6)	1·378 (3)	1·381 (3)	
C(6)–C(1)	1·404 (3)	1·398 (3)	
C(4)–C(7)	1·494 (3)	1·490 (3)	
C(7)–O(7)	1·217 (3)	1·220 (3)	
C(7)–C(8)	1·501 (4)	1·506 (3)	
	A	*B*	
O(9)–N(9)–C(10)	115·5 (2)	115·0 (2)	
O(9)–N(9)–C(1)	115·8 (2)	115·4 (2)	
C(10)–N(9)–C(1)	128·2 (2)	129·2 (2)	
O(10)–C(10)–N(9)	120·9 (2)	120·5 (2)	
O(10)–C(10)–C(11)	122·1 (2)	122·2 (2)	
N(9)–C(10)–C(11)	116·9 (2)	117·3 (2)	
N(9)–C(1)–C(2)	122·8 (2)	122·0 (2)	
N(9)–C(1)–C(6)	118·1 (2)	118·6 (2)	
C(6)–C(1)–C(2)	119·1 (2)	119·4 (2)	
C(1)–C(2)–C(3)	120·0 (2)	119·5 (2)	
C(2)–C(3)–C(4)	121·3 (2)	121·5 (2)	
C(3)–C(4)–C(5)	118·0 (2)	118·0 (2)	
C(4)–C(5)–C(6)	121·6 (2)	121·5 (2)	
C(5)–C(6)–C(1)	119·9 (2)	120·0 (2)	
C(3)–C(4)–C(7)	122·8 (2)	122·8 (2)	
C(5)–C(4)–C(7)	119·2 (2)	119·2 (2)	
C(4)–C(7)–O(7)	120·1 (2)	120·3 (2)	
C(4)–C(7)–C(8)	118·9 (2)	118·5 (2)	
O(7)–C(7)–C(8)	120·9 (2)	121·1 (2)	
ω_1 = C(1)–N(9)–C(10)–C(11)	179·6 (2)	−179·1 (2)	
ω_2 = O(9)–N(9)–C(10)–O(10)	172·7 (2)	−173·6 (2)	
ω_3 = C(1)–N(9)–C(10)–O(10)	1·1 (3)	−1·1 (3)	
ω_4 = O(9)–N(9)–C(10)–C(11)	−8·9 (3)	8·4 (3)	
$\chi_c = \pi + \omega_1 - \omega_3$	−1·5 (3)	2·0 (3)	
$\chi_N = \pi + \omega_2 - \omega_3$	−8·4 (3)	7·5 (3)	
$\tau = (\omega_1 + \omega_2)/2$	176·2 (2)	−176·4 (2)	

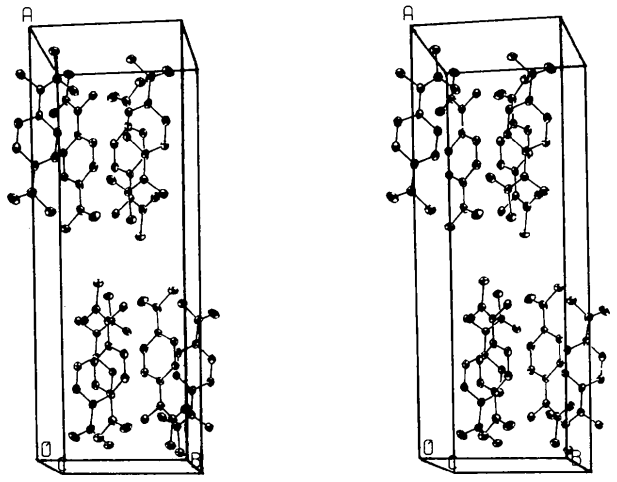

Fig. 2. Packing diagram.

A packing diagram (Johnson, 1965) is shown in Fig. 2. The two molecules are linked by two intermolecular hydrogen bonds [O(10*A*)···O(9*B*) 2·631 (2) Å and O(9*A*)···O(10*B*) ($x, y, z-1$) 2·636 (2) Å].

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2-(*p*-Chlorophenyl)-3-nitro-2*H*-chromene

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Abstract. $C_{15}H_{10}ClNO_3$, $M_r = 287{\cdot}70$, triclinic, $P\bar{1}$, $Z = 2$, $F(000) = 296$, $a = 5{\cdot}422$ (1), $b = 9{\cdot}624$ (1), $c = 12{\cdot}636$ (2) Å, $\alpha = 76{\cdot}66$ (2), $\beta = 78{\cdot}67$ (2), $\gamma = 87{\cdot}97$ (2)°, $V = 629{\cdot}03$ Å^3, $D_m = 1{\cdot}507$ (3), $D_x = 1{\cdot}519$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 26{\cdot}25$ mm^{-1}, $T = 413$ K, final $R = 0{\cdot}0577$ for 1859 observed reflections [$I > 2{\cdot}5\sigma(I)$]. Bond lengths [1·512 (5) Å] and angles [109·2 (3)°] at the phenyl substitution site are comparable with those in other molecules. The bond angle at the nitro substitution site C(7)–C(8)–C(9) is 122·9 (3)° owing to the electron-withdrawing character of the nitro group. The pyran ring adapts a half-chair conformation.

Introduction. The title compound is of therapeutic value in the treatment of infectious diseases.

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