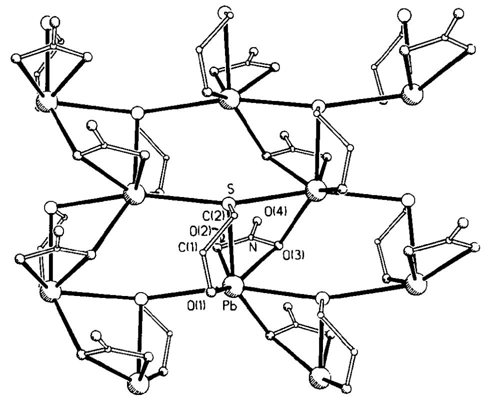

Fig. 2. Part of the polymeric sheet structure viewed along the *a* axis.

The Pb atom is seven-coordinate, but with a wide range of Pb—S and Pb—O bond lengths. The next-nearest S atom is 3·510 (4) Å distant, almost collinear with the 'primary' Pb—S bond (Fig. 2). All other Pb···O distances are >4·3 Å.

The polymeric sheets are based on a $(PbS)_\infty$ array perpendicular to the *a* axis. The nitrate ligands and the other atoms of the SCH_2CH_2OH ligand are arranged above and below the sheet. Each SCH_2CH_2OH ligand is chelating, and forms two additional S—Pb bonds within the sheet; the OH group is non-bridging. Nitrate acts as an essentially symmetrical bidentate ligand for the Pb atom (Addison, Logan, Wallwork & Garner, 1971); in addition, one of the bonded O atoms forms a bridge to another Pb atom.

The sevenfold coordination of Pb, chelating and bridging nitrate, and five-membered $PbSC_2X$ chelate ring are features also observed in 2-(morpholino)-ethanethiolatolead(II) nitrate (Dance & Guerney, 1981). Significant differences between the two structures are the number of bridging O atoms for the nitrate group (one or two), the degree of asymmetry of the Pb—NO_3 bonding (very slight or considerable), and the sheet *versus* chain polymerization. Pb—S bonds are longer, and Pb—O shorter, in the present structure than in 2-(morpholino)ethanethiolatolead(II) nitrate. As in this other Pb-thiolate structure, and in D-penicillaminatolead(II) (Freeman, Stevens & Taylor, 1974), the effect of a stereochemically active non-bonding electron pair on the Pb atom in $Pb(SCH_2CH_2OH)(NO_3)$ is marked: its angular location is approximately in the direction of the next-nearest S atom (Fig. 2).

The tendency of lead–thiolate complexes to polymerize appears to be aided by the propensity of Pb^{II} to have an irregular coordination sphere involving several secondary but significant bonding interactions.

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Structure of *N*-(3-Cyanophenyl)acetohydroxamic Acid Hydrate, $C_9H_8N_2O_2.\frac{1}{3}H_2O$

By Raman Mocharla, Douglas R. Powell and Dick van der Helm

Department of Chemistry, University of Oklahoma, Norman, OK 73019, *USA*

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Abstract. $M_r = 182{\cdot}18$, orthorhombic, $Pna2_1$, $a = 25{\cdot}123$ (7), $b = 3{\cdot}947$ (1), $c = 26{\cdot}107$ (7) Å, $V = 2588{\cdot}8$ (12) Å^3 at 138 (2) K, $a = 25{\cdot}202$ (6), $b = 4{\cdot}0433$ (12), $c = 26{\cdot}268$ (10) Å, $V = 2676{\cdot}5$ (14) Å^3 at 295 K, $Z = 12$, $D_m = 1{\cdot}354$, $D_x = 1{\cdot}356$ g cm^{-3} at 295 K, Ni-filtered Cu $K\bar{\alpha}$, $\lambda = 1{\cdot}5418$ Å, $\mu = 7{\cdot}71$ cm^{-1}, $F(000) = 1144$, $T = 138$ (2) K, $R = 0{\cdot}045$ for 2149 observed data. The three unique hydroxamic acid molecules all have a *trans* conformation, have the cyano groups *anti* to the hydroxyl O atom, have consistent bond distances and angles, and are connected through a network of hydrogen bonds.

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Introduction. Microorganisms elaborate a variety of small iron-chelating molecules, many of which use hydroxamate groups to bind the metal. Also, because of their ability to form stable metal complexes, hydroxamic acids have been used as analytical reagents (Agarwal & Roshania, 1980) and as therapeutic agents for treatment of iron overload (Anderson & Hiller, 1977). Crystal structures of synthetic hydroxamic acids include *N*,*N*′-dihydroxy-*N*,*N*′-diisopropylhexane diamide (HIPA) (Smith & Raymond, 1980), *N*-(4-cyanophenyl)acetohydroxamic acid (*p*-CNPA) (Mocherla, Powell, Barnes & van der Helm, 1983), and *N*-phenylbenzohydroxamic acid (PBHA) (Chou, Powell & van der Helm, 1984). This article describes the crystal and molecular structure of *m*-CNPA.

Experimental. Title compound was a gift from Dr A. L. Crumbliss, P. M. Gross Chemical Laboratory, Department of Chemistry, Duke University, Durham, North Carolina. Crystals grown from dichloromethane–chloroform–acetonitrile solution equilibrated with petroleum ether at 277 K. Density measured by flotation using hexane and carbon tetrachloride. Colorless rods 0·10 × 0·25 × 0·50 mm. Low-temperature lattice constants from a least-squares fit to $\pm 2\theta$ values of 48 reflections; room-temperature lattice constants from 25 reflections. Systematic absences $0kl$, $k+l=2n+1$ and $h0l$, $h=2n+1$. $\langle |E^2-1| \rangle = 0{\cdot}740$. Enraf–Nonius CAD-4 diffractometer, ω–2θ scan mode, $2\theta < 150°$, *hkl* octant measured. Three intensity monitors (2,0,10, 414, and 10,0,4): max. variation 0·022 and overall e.s.d. 0·005. 2695 unique data measured, 2149 observed $[I > 2\sigma(I)]$. Structure by *MULTAN*80 (Main, Fiske, Hull Lessinger, Germain, Declercq & Woolfson, 1980). $\sum w(|F_o|-|F_c|)^2$ minimized; refinement was blocked with each molecule in a separate block. H atoms located by difference Fourier synthesis. Nonhydrogen atoms varied anisotropically, H atoms isotropically [U of H(2C) was kept fixed because this parameter consistently refined to a negative value], $wR = 0{\cdot}045$, $S = 1{\cdot}139$, $w = \sigma^{-2}(F)$. Max. and average Δ/σ 0·133 and 0·023. Max. and min. heights on a final difference map 0·22 and –0·23 e Å^{-3}. Scattering factors for O, N and C from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965). Structure factor, least-squares refinement and Fourier calculations with the *SHELX*76 package (Sheldrick, 1976).

Discussion. The final coordinates of the nonhydrogen atoms for *m*-CNPA are listed in Table 1. The atom-numbering scheme for all three hydroxamic acid molecules is shown in Fig. 1 (Johnson, 1965). The cyano groups are *anti* to oxygen O(9) in all three molecules. Bond distances, angles and selected torsion angles are presented in Table 2.* Bond distances and angles for the three unique hydroxamic acid molecules are consistent.

Table 1. *Fractional atomic coordinates* (x *and* $z \times 10^5$, $y \times 10^4$) *and equivalent isotropic thermal parameters* (Å$^2 \times 10^4$) *for the nonhydrogen atoms*

	x	y	z	U_{eq}†
C(1*A*)	74413 (15)	8941 (11)	53489 (14)	253 (20)
C(2*A*)	76198 (16)	7441 (12)	48910 (14)	256 (20)
C(3*A*)	81547 (16)	7551 (12)	47736 (14)	272 (21)
C(4*A*)	85305 (15)	9174 (12)	50839 (16)	304 (22)
C(5*A*)	83448 (17)	10639 (12)	55332 (16)	334 (24)
C(6*A*)	78153 (17)	10520 (12)	56644 (15)	309 (23)
C(7*A*)	83406 (16)	5889 (12)	43076 (15)	302 (23)
N(8*A*)	84977 (14)	4540 (11)	39524 (14)	419 (23)
N(9*A*)	69004 (13)	8884 (9)	55022 (11)	254 (17)
O(9*A*)	67752 (12)	11034 (8)	59120 (11)	342 (16)
C(10*A*)	64853 (15)	7264 (11)	52881 (14)	261 (20)
O(10*A*)	65420 (11)	5481 (8)	48983 (10)	299 (15)
C(11*A*)	59524 (19)	7668 (15)	55446 (20)	360 (26)
C(1*B*)	13575 (14)	11087 (10)	35899 (14)	235 (19)
C(2*B*)	14222 (15)	9552 (10)	31084 (14)	236 (20)
C(3*B*)	19189 (15)	9744 (10)	28694 (15)	263 (21)
C(4*B*)	23446 (17)	11433 (12)	30916 (17)	337 (24)
C(5*B*)	22718 (17)	12974 (12)	35630 (17)	344 (24)
C(6*B*)	17815 (17)	12826 (12)	38082 (15)	296 (22)
C(7*B*)	19966 (15)	8107 (11)	23786 (16)	274 (22)
N(8*B*)	20642 (13)	6775 (10)	19955 (13)	370 (22)
N(9*B*)	8692 (12)	10898 (9)	38630 (11)	240 (16)
O(9*B*)	8549 (12)	12770 (8)	43210 (10)	336 (16)
C(10*B*)	4079 (15)	9344 (11)	37364 (14)	253 (21)
O(10*B*)	3699 (10)	7799 (9)	33264 (9)	307 (15)
C(11*B*)	−513 (19)	9634 (15)	41003 (17)	348 (26)
C(1*C*)	−805 (13)	5162 (10)	20808 (13)	208 (18)
C(2*C*)	−1076 (14)	6084 (11)	15659 (14)	224 (19)
C(3*C*)	3161 (15)	5298 (11)	12482 (14)	249 (20)
C(4*C*)	7679 (15)	3629 (11)	14207 (15)	249 (20)
C(5*C*)	7843 (15)	2703 (12)	19331 (14)	283 (21)
C(6*C*)	3628 (15)	3394 (11)	22561 (14)	249 (20)
C(7*C*)	2882 (14)	6334 (12)	7147 (14)	282 (21)
N(8*C*)	2717 (14)	7107 (12)	2949 (13)	435 (23)
N(9*C*)	−4912 (13)	5945 (9)	24319 (11)	244 (16)
O(9*C*)	−4391 (11)	4472 (8)	29201 (9)	276 (15)
C(10*C*)	−9484 (15)	7718 (11)	23552 (14)	263 (20)
O(10*C*)	−10525 (10)	8948 (8)	19309 (10)	328 (16)
C(11*C*)	−13137 (18)	8109 (14)	28033 (17)	321 (25)
O*W*′	69569 (12)	7544 (9)	67481 (11)	327 (16)

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

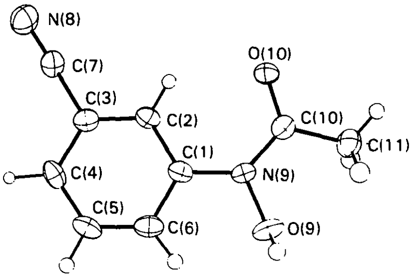

Fig. 1. Schematic drawing of *m*-CNPA.

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* Lists of structure factors, anisotropic thermal parameters and positional parameters for the hydrogen atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39282 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

	A	*B*	*C*	Average
C(1)–C(2)	1·408 (5)	1·405 (5)	1·394 (5)	1·402 (5)
C(2)–C(3)	1·379 (6)	1·397 (5)	1·385 (5)	1·387 (5)
C(3)–C(4)	1·399 (6)	1·387 (6)	1·388 (6)	1·391 (6)
C(4)–C(5)	1·389 (6)	1·385 (6)	1·387 (5)	1·387 (6)
C(5)–C(6)	1·374 (6)	1·389 (6)	1·381 (5)	1·381 (6)
C(6)–C(1)	1·396 (6)	1·389 (6)	1·392 (5)	1·392 (6)
C(3)–C(7)	1·459 (6)	1·448 (6)	1·453 (5)	1·453 (6)
C(7)–N(8)	1·140 (6)	1·143 (6)	1·138 (5)	1·140 (6)
C(1)–N(9)	1·417 (5)	1·421 (5)	1·414 (5)	1·417 (5)
N(9)–O(9)	1·401 (4)	1·406 (4)	1·407 (4)	1·405 (4)
N(9)–C(10)	1·345 (5)	1·352 (5)	1·360 (5)	1·352 (5)
C(10)–O(10)	1·245 (5)	1·236 (5)	1·238 (5)	1·240 (5)
C(10)–C(11)	1·505 (6)	1·499 (6)	1·495 (6)	1·500 (6)
O(9)–N(9)–C(1)	114·9 (3)	114·9 (3)	115·4 (3)	115·1 (3)
C(10)–N(9)–C(1)	129·3 (3)	129·9 (3)	129·3 (3)	129·5 (3)
O(9)–N(9)–C(10)	115·6 (3)	115·1 (3)	115·1 (3)	115·3 (3)
N(9)–C(10)–O(10)	121·3 (3)	120·1 (3)	120·8 (3)	120·7 (3)
N(9)–C(10)–C(11)	117·0 (4)	118·0 (4)	117·2 (3)	117·4 (4)
O(10)–C(10)–C(11)	121·7 (4)	121·8 (4)	122·0 (4)	121·8 (4)
N(9)–C(1)–C(2)	122·6 (3)	121·8 (3)	122·2 (3)	122·2 (3)
N(9)–C(1)–C(6)	119·1 (3)	118·8 (3)	118·7 (3)	118·9 (3)
C(6)–C(1)–C(2)	118·3 (4)	119·4 (3)	119·1 (3)	118·9 (3)
C(1)–C(2)–C(3)	119·1 (4)	118·7 (3)	118·8 (3)	118·9 (3)
C(2)–C(3)–C(4)	122·9 (4)	121·9 (4)	122·7 (4)	122·5 (4)
C(3)–C(4)–C(5)	116·9 (4)	118·7 (4)	117·5 (4)	117·7 (4)
C(4)–C(5)–C(6)	121·4 (4)	120·5 (4)	120·9 (4)	120·9 (4)
C(5)–C(6)–C(1)	121·3 (4)	120·8 (4)	120·8 (3)	121·0 (4)
C(4)–C(3)–C(7)	118·2 (4)	118·7 (4)	119·0 (3)	118·6 (4)
C(2)–C(3)–C(7)	118·9 (4)	119·4 (4)	118·3 (3)	118·9 (4)
C(3)–C(7)–N(8)	177·8 (4)	178·8 (4)	178·9 (5)	178·5 (4)

Conformational angles of the hydroxamate group

	A	*B*	*C*
ω_1 = C(1)–N(9)–C(10)–C(11)	179·4 (5)	−178·8 (4)	179·4 (5)
ω_2 = O(9)–N(9)–C(10)–O(10)	174·7 (4)	175·8 (4)	−175·6 (4)
ω_3 = C(1)–N(9)–C(10)–O(10)	0·2 (10)	−0·2 (10)	−0·8 (7)
ω_4 = O(9)–N(9)–C(10)–C(11)	−6·0 (5)	−2·8 (5)	4·6 (5)
$\chi_C = \pi + \omega_1 - \omega_3$	−0·8 (10)	1·4 (10)	0·2 (7)
$\chi_N = \pi + \omega_2 - \omega_3$	−5·5 (10)	−4·0 (10)	5·2 (7)
$\tau = (\omega_1 + \omega_2)/2$	177·1 (5)	178·5 (4)	−178·1 (5)

The electron-withdrawing effect of the *m*-cyanophenyl group is somewhat less than was observed for the *p*-cyanophenyl group in *p*-CNPA. The average C(1)—N(9) and N(9)—C(10) bond lengths in *m*-CNPA are 1·417 (5) and 1·352 (5) Å, respectively. These values compare with 1·427 (3) and 1·365 (4) Å in *p*-CNPA (Mocherla, Powell, Barnes & van der Helm, 1983), 1·436 (6) and 1·353 (6) Å in PBHA (Chou, Powell & van der Helm, 1984) and 1·461 (2) and 1·328 (2) Å in HIPA (Smith & Raymond, 1980). The N—O and C=O bond lengths of *m*-CNPA [1·405 (5) and 1·240 (5) Å] are close to the literature values of 1·393 (5) and 1·228 (5) Å in *p*-CNPA, 1·398 (6) and 1·240 (7) Å in PBHA and 1·396 (2) and 1·241 (2) Å in HIPA.

The hydroxamate groups are in the *trans* conformation. The torsion angles of the three hydroxamate groups indicate that these groups have small but significant deformations from planarity. These deformations involve some N out-of-plane bending and a small C—N rotation according to calculations described by Winkler & Dunitz (1971) (see Table 2). The phenyl rings are nearly coplanar with their respective hydroxamate group, having interplanar angles of 8·3 (7), 2·4 (6) and 5·0 (7)°.

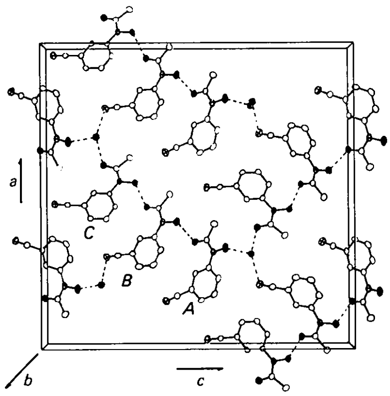

Fig. 2. Packing diagram for *m*-CNPA. The unit-cell enclosure is drawn for *a* and *b* at 0 and 1, and for *c* at $-\frac{1}{2}$ and $+\frac{1}{2}$.

Each of the three hydroxamic acid molecules in *m*-CNPA has a somewhat different hydrogen-bonding scheme (see Fig. 2). The water molecule is bonded to the hydroxyl oxygen O(9*A*), 2·621 (5) Å, and to the carbonyl oxygen O(10*C*)($\frac{1}{2}-x$, $y-\frac{1}{2}$, $\frac{1}{2}+z$), 2·721 (5) Å. Molecule *B* is bonded to both molecules *A* and *C* through O(9*B*)···O(10*A*)($x-\frac{1}{2}$, $\frac{3}{2}-y$, z), 2·627 (5) Å, and O(10*B*)···O(9*C*), 2·642 (5) Å. Finally there is a weak hydrogen bond between the water molecule and the cyano group of molecule *B* (3·061 Å), and thus all active H atoms are used for hydrogen bonding.

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