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Crystal Structure of 3-Phenylpyrrolidine-2,5-dione

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Abstract. The structure determination of the orthorhombic modification of the title compound [hereafter: PH–PD(*o*)] $C_{10}H_9NO_2$, $M=175{\cdot}2$, which crystallizes from ethanol in prismatic needles. Its space group is $Pna2_1$ (No. 33) $a=8{\cdot}660$ (10), $b=17{\cdot}313$ (10), $c=6{\cdot}054$ (10) Å (from oscillation and Weissenberg photographs and refined by diffractometer), $Z=4$, $D_c=1{\cdot}269$, $D_x=1{\cdot}287$ g cm^{-3} (by flotation). The other polymorph [PH–PD(*m*)] is monoclinic, space group $P2_1/c$.

Introduction. 870 independent reflexions were collected from layers *hk*0 to *hk*6 on a Stoe–Güttinger automatic off-line Weissenberg goniometer system using Cu $K\bar{\alpha}$ ($\lambda=1{\cdot}5418$ Å) radiation. 122 F_o values less than $2{\cdot}0\times\sigma(F_o)$ were considered as unobserved and were omitted later from the structure refinement. A crystal of approximate cross section $0{\cdot}15\times0{\cdot}24$ mm was used, which made an absorption correction unnecessary ($\mu R<0{\cdot}2$). The phase problem for 150 reflexions with $E>1{\cdot}2$ was solved by the program *MULTAN*, written by Germain, Main & Woolfson (1971). The starting reflexions for the weighted tangent formula given automatically by the *CONVERGE* section of the program were the three origin-determining reflexions: 5,15,0 (360°), 182 (45°), 121 (360°), and two additional reflexions: 864 and 652. The enantiomorphy was fixed by the reflexion 182. The third part of the program (*FASTAN*) gave two sets (Nos. 14 and 5) of phases with a nearly similar, good consistency (*ABS FOM* and *RESID* were 1·55, 1·54 and 27·4, 27·7 respectively). The *E* map computed from set No. 14 of best consistency gave a model which fitted the geometry of the PH–PD(*o*) molecule, and from which all 13 heavy atoms of the molecule could be obtained with a structure factor agreement of $R=0{\cdot}25$.

Full-matrix least-squares refinement of the fractional atomic coordinates with anisotropic vibrational

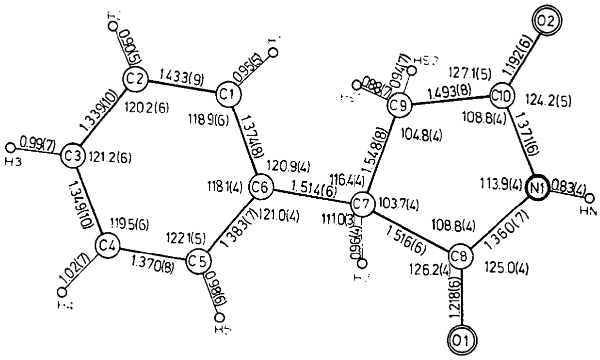

Fig. 1. Bond distances and angles with their e.s.d.'s.

Table 1. *Final fractional atomic positional* (×10⁴) *and vibrational parameters for* PH–PD(*o*)

The estimated standard deviations are in parentheses.
The b_{ij} are defined by $T=\exp[-10^{-4}(b_{11}h^2+b_{22}k^2+b_{33}l^2+2b_{12}hk+2b_{13}hl+2b_{23}kl)]$.
The *z* coordinate of atom O(2) was fixed throughout the structure refinement.

	x/a	y/b	z/c	B or b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
O(1)	1249 (4)	296 (2)	3471 (8)	134 (5)	46 (1)	581 (12)	− 23 (3)	23 (8)	−88 (4)
O(2)	248 (5)	1798 (2)	9400	271 (8)	53 (2)	387 (10)	−7 (3)	100 (8)	−37 (4)
N	583 (5)	923 (2)	6658 (8)	137 (6)	31 (2)	370 (11)	− 18 (3)	53 (8)	2 (4)
C(1)	4794 (6)	879 (3)	5410 (11)	134 (9)	43 (2)	462 (16)	−6 (4)	−39 (10)	52 (5)
C(2)	6362 (8)	675 (3)	4933 (12)	177 (11)	38 (2)	663 (25)	4 (4)	−146 (16)	35 (7)
C(3)	7056 (7)	950 (4)	3119 (12)	116 (9)	45 (2)	649 (23)	−5 (4)	31 (13)	−47 (7)
C(4)	6299 (7)	1417 (4)	1700 (12)	145 (10)	60 (3)	430 (16)	−8 (5)	29 (12)	− 4 (7)
C(5)	4787 (6)	1604 (3)	2100 (8)	142 (9)	39 (2)	321 (12)	−7 (4)	− 5 (9)	17 (4)
C(6)	4007 (5)	1340 (2)	3943 (8)	100 (7)	23 (1)	302 (11)	− 11 (3)	−19 (8)	5 (3)
C(7)	2339 (5)	1559 (2)	4352 (8)	118 (7)	27 (2)	278 (9)	−9 (3)	−14 (7)	11 (4)
C(8)	1351 (5)	848 (2)	4710 (8)	94 (7)	33 (2)	319 (12)	−1 (3)	− 5 (8)	−10 (4)
C(9)	2018 (8)	2058 (3)	6424 (11)	148 (10)	31 (2)	379 (16)	−9 (4)	20 (11)	− 3 (5)
C(10)	851 (6)	1611 (3)	7712 (8)	151 (8)	31 (2)	287 (11)	2 (3)	4 (8)	− 1 (4)

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Table 1 (*cont.*)

Hydrogen atom parameters. Coordinates × 10^3.

H(1)	430 (5)	72 (3)	672 (8)	2·5 Å^2
H(2)	666 (6)	43 (3)	617 (8)	4·2
H(3)	816 (8)	83 (3)	287 (9)	5·1
H(4)	686 (8)	175 (4)	57 (11)	6·6
H(5)	423 (6)	191 (3)	98 (9)	3·9
H(7)	205 (5)	186 (3)	309 (7)	1·3
H(91)	294 (8)	207 (4)	705 (9)	4·1
H(92)	156 (8)	245 (4)	560 (9)	8·3
H(N)	−2 (5)	60 (2)	718 (6)	0·6

parameters for C, N and O atoms and isotropic ones for H atoms, using a modified version of the program written by Busing, Martin & Levy (1962), gave a final residual of 0·054 for the observed and 0·081 for the total 870 reflexions. All calculations were performed on a TR-440 computer in the German Computer Centre at Darmstadt, using atomic scattering factors taken from *International Tables for X-ray Crystallography* (1962). The final atomic parameters are given in Table 1. A table of calculated and observed structure factors has been deposited with the National Lending Library as Supplementary Publication No. SUP 30039.*

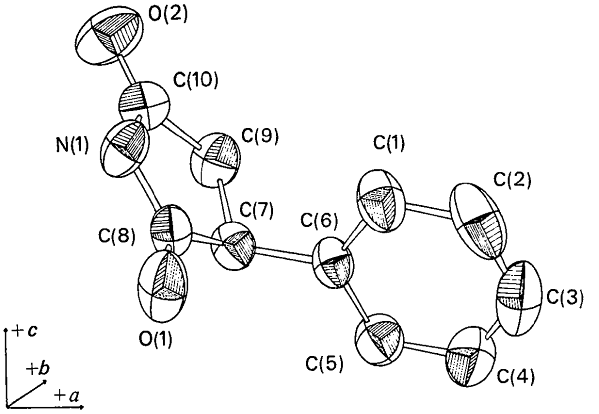

Fig. 2. A perspective view of the molecule.

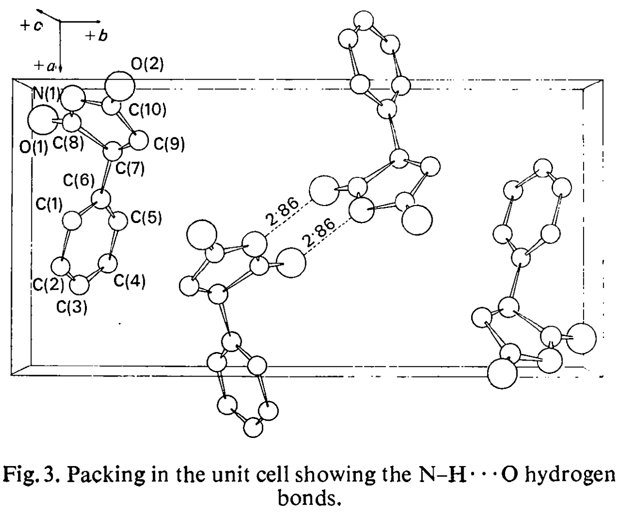

Fig. 3. Packing in the unit cell showing the N–H···O hydrogen bonds.

Discussion. Several succinimide derivatives have recently been prepared (Seres, 1970) in order to study their pharmaceutical effects. One of them, Perlepsyn, is now registered by the Chinoin Factory as an efficacious anti-epileptic drug. A series of the most promising models (including Perlepsyn itself) have been subjected to X-ray structure analysis (Argay, 1972). Because the PH–PD(*o*) molecule is a significant part of Perlepsyn (Argay & Seres, 1973), its structure is the first to be described.

The bond lengths and angles shown in Fig. 1 are comparable with the corresponding data found in the literature. The lone pair of the nitrogen atom is delocalized on the OC(8)NC(10)O moiety, thus forming two equally strong C–N multiple bonds. The phenyl ring is planar [the mean atomic deviation of 0·007 Å from the best plane $2{\cdot}627x+13{\cdot}992y+3{\cdot}095z-4{\cdot}145=0$ is less than the mean e.s.d. (0·009 Å) for the C–C distances]. It is, however, somewhat distorted [*cf.* the short C(2)–C(3) and C(3)–C(4) distances], which is presumably due to the limited number of reflexions compared with the number of parameters refined. The C–C bond lengths in the benzene rings are, as we have frequently observed, particularly influenced by the errors, the limited number and the non-spherical distribution of the reflexions. In particular, the distances that show considerable deviations from the mean value of 1·392 Å given for benzene by Cox, Cruickshank & Smith (1958) are those formed by carbon atoms furthest from the centre of gravity of the molecular electron density. This observation is supported in this case by the large thermal motions of C(2) and C(3), as shown in Fig. 2. The dihedral angle between the best planes of the phenyl and succinimide rings is 82·2°.

In most succinimide derivatives there are N–H···O intermolecular hydrogen bonds which maintain molecular dimers. In PH–PD(*o*) these are $N(x,y,z)\cdots O[1]\ (\bar{x},\bar{y},z+\frac{1}{2})$ and its symmetry equivalent $O[1]\ (x,y,z)\cdots N(\bar{x},\bar{y},z+\frac{1}{2})$ with length of 2·86 Å (Fig. 3).

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* Copies may be obtained from the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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Manganous Citrate Decahydrate

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Abstract. Crystals of manganese citrate, $[Mn(H_2O)_6]$ $[MnC_6H_5O_7(H_2O)_2]$ $2H_2O$, were prepared by evaporation of an aqueous mixture of freshly precipitated manganous hydroxide and citric acid. They are monoclinic, space group $P2_1/n$, $Z=2$, F.W. 723·19, $a=$ 20·575 (5), $b=6·755$ (2), $c=9·230$ (2) Å, $\beta=96·74°$ (1), $V=1273·97$ Å^3, $D_x=1·89$, $D_{\text{meas}}=1·88$ g cm^{-3}, λ(Mo $K\alpha$)$=0·7107$ Å. The final residual $R=0·045$. All hydrogen atoms were located and were refined isotropically. The structure is isomorphous with that of the magnesium salt [Johnson *Acta Cryst.* (1965). **18**, 1004]. Each citrate ion forms a tridentate chelate to one manganous ion.

Experimental. A roughly spherical crystal, 0·26 mm diameter, was used in the collection of three-dimensional data on a Syntex automated diffractometer with monochromatic Mo $K\alpha$ radiation using the θ–2θ scan technique. Intensities were measured for 3731 unique reflections. Values for $\sigma(F)$ were derived from counting statistics and measured instrumental uncertainties. The formula used was: $\sigma(F)=F/2\{\sigma^2(I)/I^2+\delta^2\}^{1/2}$ [where $\sigma(I)$ is derived from counting statistics alone and δ is the measured instrumental uncertainty]. Reflections for which the measured intensity, I_{obs}, was less than $2·33\sigma(I)$ were considered to be unobserved. For these 'unobserved reflections', when $I\geq\sigma(I)$, the

Table 1. *Atomic parameters for manganese citrate*

Positional parameters are listed as fractions of cell edges.
Anisotropic temperature factors are expressed as

$$\exp\left[-(\tfrac{1}{4})\,(B_{11}h^2a^{*2}+\ldots+2B_{12}hka^*b^*+\ldots)\right]$$

and isotropic temperature factors as

$$\exp\,(-B\sin^2\theta/\lambda^2)$$

with values of B given in Å^2. Estimated standard deviations, determined from the inverted full matrices, are listed beside each parameter, with respect to the last decimal place given.

	$x\times10^5$	$y\times10^4$	$z\times10^4$	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Mn(1)	0	0	0	1·62 (2)	1·61 (2)	1·53 (2)	0·07 (2)	−0·04 (2)	0·03 (2)
Mn(2)	21778 (2)	5140·6 (6)	5400·8 (4)	1·50 (2)	1·47 (2)	1·46 (1)	−0·17 (2)	−0·13 (1)	−0·12 (1)
O(1)	18672 (9)	−56 (3)	8339 (2)	1·70 (7)	1·83 (7)	1·53 (7)	0·26 (8)	−0·16 (6)	−0·29 (7)
O(2)	20744 (11)	−2426 (3)	6811 (2)	2·96 (8)	2·34 (8)	2·33 (9)	1·05 (8)	−0·46 (8)	−0·88 (8)
O(3)	2549 (10)	4940 (3)	2914 (2)	2·07 (7)	2·05 (8)	2·96 (9)	0·04 (8)	−1·20 (7)	0·54 (8)
O(4)	12844 (10)	5271 (3)	3877 (2)	1·90 (7)	2·42 (9)	2·44 (8)	−0·57 (8)	−0·70 (7)	0·98 (7)
O(5)	23341 (9)	2181 (3)	4525 (2)	1·52 (7)	2·08 (8)	2·48 (9)	−0·22 (7)	0·40 (6)	−0·67 (7)
O(6)	16588 (10)	26 (3)	3321 (2)	2·14 (7)	2·35 (8)	1·62 (7)	−0·30 (8)	0·18 (6)	−0·67 (8)
O(7)	15438 (9)	3074 (3)	6479 (2)	2·17 (8)	1·38 (7)	1·10 (6)	−0·03 (7)	−0·17 (6)	- 0·03 (6)
O(8)	26057 (11)	6813 (3)	3786 (2)	2·94 (8)	2·68 (9)	2·00 (8)	0·12 (8)	0·67 (8)	0·45 (8)
O(9)	4049 (10)	−1191 (3)	2110 (2)	2·25 (8)	1·88 (8)	2·20 (9)	−0·28 (7)	−0·60 (7)	0·41 (7)
O(10)	8938 (11)	1685 (3)	− 150 (3)	2·27 (8)	2·47 (9)	3·73 (11)	−0·50 (8)	0·83 (8)	−1·06 (9)
O(11)	− 4541 (11)	2427 (3)	1047 (2)	2·62 (8)	2·29 (8)	2·44 (9)	0·49 (8)	−0·69 (7)	−0·54 (8)
O(12)	13989 (11)	5558 (4)	748 (3)	2·27 (8)	3·00 (10)	2·78 (10)	0·11 (8)	0·21 (8)	0·09 (8)
C(1)	17273 (13)	−1050 (4)	7166 (3)	1·69 (10)	1·38 (10)	1·24 (9)	−0·32 (9)	−0·02 (8)	0·19 (8)
C(2)	11368 (13)	− 340 (4)	6171 (3)	1·50 (8)	1·44 (10)	1·42 (9)	−0·35 (8)	−0·11 (8)	0·12 (8)
C(3)	12912 (13)	1627 (4)	5423 (3)	1·47 (8)	1·34 (9)	1·10 (9)	−0·09 (8)	−0·05 (8)	−0·13 (8)
C(4)	6589 (13)	2496 (4)	4632 (3)	1·39 (8)	1·60 (10)	1·68 (10)	−0·04 (9)	0·10 (8)	0·17 (9)
C(5)	7415 (14)	4366 (4)	3750 (3)	1·75 (10)	1·63 (10)	1·48 (10)	0·08 (9)	−0·20 (9)	0·06 (8)
C(6)	18023 (13)	1246 (4)	4335 (3)	1·45 (10)	1·32 (9)	1·26 (10)	0·09 (8)	−0·05 (8)	0·20 (8)