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1,4-*trans*-Phenazinediol-1,2,3,4-tetrahydro-5,10-dioxide

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Abstract. Monoclinic, $P2_1/n$, $a=7{\cdot}229$ (1), $b=14{\cdot}787$ (2), $c=10{\cdot}432$ (1) Å, $\cos\beta=-0{\cdot}2417$ (2), $Z=4$, $D_m=1{\cdot}47$, $D_c=1{\cdot}46$ g cm^{-3}. The structure was determined by direct methods. Refinement of diffractometer data yielded a final R value of 0·031 on all observed data. The two independent N–O bond lengths are 1·302 (2) and 1·297 (2) Å.

Introduction. Preliminary X-ray photographs showed monoclinic symmetry and the following systematic absences were observed: $h0l$ when $h+l\neq 2n$, $0k0$ when $k\neq 2n$. These conditions establish the space group as $P2_1/n$. Cell constants were measured on a Picker 4-angle diffractometer by determining the centered values for 12 reflections and carrying out at least-squares analysis of these values. The cell constants are: $a=7{\cdot}229$ (1), $b=14{\cdot}787$ (2), $c=10{\cdot}432$ (1) Å, and $\beta=103{\cdot}99$ (2)°.

Data were collected by using Cu $K\alpha$ radiation with a monochromator to remove Cu $K\beta$ radiation. The θ–2θ scan used for data collection had a base width of 2·0° with a dispersion correction added to allow for α_1–α_2 splitting. The rate of scan was 1° min^{-1} and 10 s background counts were taken before and after each scan. One standard reflection was monitored every 50 measurements to check on crystal decomposition or movement. 1835 reflections were measured of which 1554 were considered above a threshold value, their intensity values being greater than three times the standard deviation.

The structure was solved by direct methods with the 1965 computer program of R. E. Long. Initially, a false solution was obtained which had no interpretable features on the E map. Reassignment of the starting set of reflections gave rise to the correct solution in a subsequent attempt. 14 of the 18 nonhydrogen atoms were found on the first correct E map and Fourier methods revealed the remaining four heavy atoms as well as all hydrogen atoms.

Refinement of the structure proceeded smoothly to a final R value of 0·031 based on all reflections above the threshold value and 0·037 on all measured reflections. A full-matrix least-squares computer program was used with the temperature factors of the nonhydrogen atoms being varied anisotropically while the hydrogen atoms had isotropic temperature factors. The scattering factors used for carbon, oxygen, and nitro-

Table 1. *Final positional and thermal parameters*

The parameters and standard deviations (in parentheses) for the least significant digit of the nonhydrogen atoms are × 10^4. The expression for the temperature factor is $\exp[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})]$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	8155 (3)	9703 (1)	1015 (2)	118 (4)	45 (1)	74 (2)	−13 (2)	15 (2)	16 (1)
C(2)	8094 (3)	10522 (2)	1605 (2)	169 (5)	41 (1)	90 (3)	−31 (2)	−13 (3)	19 (1)
C(3)	6589 (3)	10741 (1)	2178 (2)	207 (6)	27 (1)	82 (2)	−9 (2)	−17 (3)	3 (1)
C(4)	5160 (3)	10136 (1)	2181 (2)	161 (5)	27 (1)	65 (2)	5 (2)	8 (2)	1 (1)
C(5)	5207 (2)	9291 (1)	1582 (2)	105 (4)	24 (1)	47 (2)	−1 (1)	11 (2)	5 (1)
N(6)	3783 (2)	8655 (1)	1573 (1)	102 (3)	25 (1)	54 (1)	4 (1)	31 (2)	0 (1)
O(7)	2468 (2)	8854 (1)	2186 (1)	157 (3)	38 (1)	100 (2)	−2 (1)	86 (2)	−15 (1)
C(8)	3784 (2)	7864 (1)	932 (2)	101 (4)	24 (1)	43 (2)	7 (1)	17 (2)	0 (1)
C(9)	2219 (3)	7199 (1)	956 (2)	112 (4)	27 (1)	69 (2)	−2 (2)	28 (2)	1 (1)
O(10)	2751 (2)	6676 (1)	2131 (1)	230 (4)	32 (1)	75 (2)	3 (1)	67 (2)	7 (1)
C(11)	1950 (3)	6561 (1)	−205 (2)	162 (5)	34 (1)	78 (2)	−10 (2)	20 (3)	−9 (1)
C(12)	3838 (3)	6115 (1)	−226 (2)	215 (5)	27 (1)	70 (2)	10 (2)	27 (3)	−8 (1)
C(13)	5260 (3)	6806 (1)	−482 (2)	153 (4)	35 (1)	55 (2)	27 (2)	25 (2)	−5 (1)
O(14)	4789 (2)	7067 (1)	−1842 (1)	237 (4)	46 (1)	55 (1)	23 (1)	48 (2)	−6 (1)
C(15)	5242 (2)	7662 (1)	306 (2)	104 (4)	28 (1)	47 (2)	8 (1)	20 (2)	1 (1)
N(16)	6678 (2)	8250 (1)	348 (1)	95 (3)	40 (1)	49 (2)	9 (1)	26 (2)	3 (1)
O(17)	8050 (2)	8063 (1)	−212 (1)	107 (3)	67 (1)	79 (2)	12 (1)	53 (2)	−5 (1)
C(18)	6684 (2)	9079 (1)	981 (2)	104 (4)	31 (1)	48 (2)	0 (1)	12 (2)	8 (1)

The positional parameters and standard deviations (in parentheses) for the least significant digit of the hydrogen atoms are × 10^3. Isotropic temperature factor and standard deviation in parentheses. The hydrogen atoms are numbered according to the atom to which they are bonded.

Table 1 (*cont.*)

	x	*y*	*z*	*B*
H(1)	917 (3)	950 (1)	60 (2)	4·3 (5)
H(2)	914 (3)	1096 (1)	170 (2)	5·3 (6)
H(3)	655 (3)	1131 (1)	258 (2)	4·1 (5)
H(4)	415 (3)	1027 (1)	257 (2)	3·6 (5)
H(9)	104 (3)	753 (1)	91 (2)	2·7 (4)
H(10)	256 (3)	695 (2)	283 (2)	5·4 (6)
H(11*a*)	141 (3)	692 (1)	−104 (2)	3·6 (5)
H(11*b*)	93 (3)	610 (1)	−13 (2)	4·2 (5)
H(12*a*)	434 (3)	581 (1)	64 (2)	3·3 (4)
H(12*b*)	366 (3)	564 (1)	−94 (2)	3·5 (4)
H(13)	659 (3)	653 (1)	−22 (2)	3·1 (4)
H(14)	551 (4)	678 (2)	−220 (2)	6·4 (7)

gen were those of Hanson, Herman, Lea & Skillman (1964) while the scattering factors for hydrogen were obtained from Stewart, Davidson & Simpson (1965).

Atomic coordinates and thermal parameters are given in Table 1. Bond lengths are on Fig. 1 and bond angles are in Table 2. A list of structure factors is obtainable.*

Discussion. It was not possible to tell with certainty if the OH groups of this compound were *cis* or *trans* from the chemical work. The results of this crystal structure unambiguously establish that the OH groups are *trans* to each other.

The bond lengths and angles are as expected for this type of compound. For the one structural feature that is observed least often, the N–O bond, the lengths are 1·302 (2) and 1·297 (2) Å. Bovio & Locchi (1971) have compiled the structural features of some *N*-oxide derivatives of phenazine and the N–O bond lengths of this study compare well with their information. The structure determinations of 8-hydroxyquinoline-*N*-oxide (Desiderato, Terry, Freeman & Levy, 1971), myxin (Hanson, 1968), and iodinin (Hanson & Huml, 1969) show an inverse relationship between the N–O bond length and the O···O contact and the data of this study support such a relationship.

* A table of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30241 (9 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

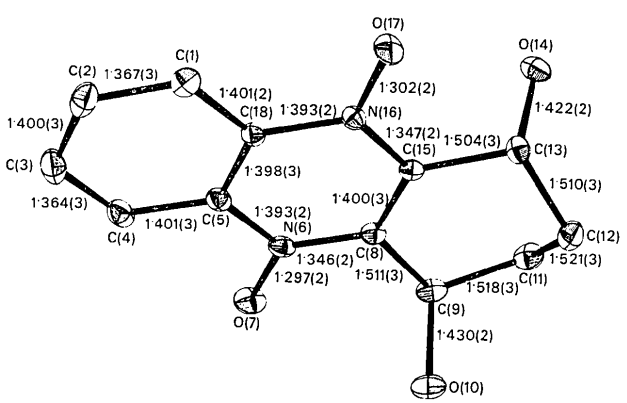

Fig. 1. Bond lengths of all nonhydrogen atoms with their standard deviations (in parentheses) for the least significant figure.

Table 2. *Bond angles of all nonhydrogen atoms and their standard deviations (in parentheses) for the least significant figure*

C(1)—C(2)—C(3)	121·1 (2)°
C(2)—C(3)—C(4)	120·7 (2)
C(3)—C(4)—C(5)	119·3 (2)
C(4)—C(5)—C(18)	119·9 (2)
C(5)—C(18)–C(1)	120·2 (2)
C(18)–C(1)—C(2)	118·8 (2)
C(4)—C(5)—N(6)	120·5 (2)
C(5)—N(6)—O(7)	119·1 (1)
C(5)—N(6)—C(8)	119·9 (1)
O(7)—N(6)—C(8)	121·0 (1)
N(6)—C(8)—C(15)	120·6 (1)
C(8)—C(15)–C(16)	120·5 (1)
C(15)–N(16)–O(17)	121·9 (1)
C(15)–N(16)–C(18)	120·1 (1)
O(17)–N(16)–C(18)	118·0 (1)
N(16)–C(18)–C(1)	120·5 (2)
N(16)–C(18)–C(5)	119·2 (1)
C(18)–C(5)—N(6)	119·6 (1)
N(6)—C(8)—C(9)	116·8 (1)
C(8)—C(9)—O(10)	106·3 (1)
C(8)—C(9)—C(11)	111·9 (2)
O(10)–C(9)—C(11)	110·3 (2)
C(9)—C(11)–C(12)	111·1 (2)
C(11)–C(12)–C(13)	109·9 (2)
C(12)–C(13)–O(14)	107·9 (1)
C(12)–C(13)–C(15)	110·3 (1)
O(14)–C(13)–C(15)	108·8 (1)
C(13)–C(15)–N(16)	118·0 (1)
C(13)–C(15)–C(8)	121·5 (1)
C(15)–C(8)—C(9)	122·6 (1)

The intermolecular hydrogen bonds are both of length 2·754 (2) Å. Infinite chains are formed involving all four oxygen atoms of the molecule. O(7), O(10), O(14), and O(17) in position (x,y,z) form hydrogen bonds respectively with O(14) and O(17) of a symmetry-related molecule $(\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z)$ and O(7) and O(10) of a symmetry-related molecule $(-\frac{1}{2}+x, 1\frac{1}{2}-y, -\frac{1}{2}+z)$. The intermolecular hydrogen-bond angles are 159·4° (15) for O(14)–H(14)–O(7) and 146·6° (15) for O(10)–H(10)–O(17). The intramolecular distances O(14)–O(17) and O(7)–O(10) are 2·946 (2) and 3·229 (2) Å respectively. There does not appear to be any intramolecular hydrogen bonding however. The O(14)–H(14)–O(17) angle is 73·2° (16) and the O(7)–H(10)–O(10) angle is 104·5° (15).

Computer programs used in this study consisted of Raymond's *UCFACS*, Dewar's *FAME*, Long's *REL*, Zalkin's *FORDAP*, Ibers's *NUCLS4*, Rao's *MASTER*, and Johnson's *ORTEP*.

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Bis-(2-hydroxy-5-methylacetophenato)nickel (II)

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Abstract. Monoclinic, $C2/c$, $a=11{\cdot}432\,(2)$, $b=8{\cdot}579\,(2)$, $c=16{\cdot}693\,(4)$ Å, $\beta=101{\cdot}3\,(1)°$, 22°C, $(C_9H_9O_2)_2Ni$, $M=357{\cdot}05$, $Z=4$, $D_m=1{\cdot}45$, $D_x=1{\cdot}48$ g cm^{-3}, crystallized from aqueous ethanol. The compound is diamagnetic and monomeric. The Ni atom is at a center of symmetry with square planar coordination to four oxygen atoms; the entire complex is planar.

Introduction. Cell dimensions were obtained from 10 high-angle reflections measured with Mo $K\alpha_1$ radiation ($\lambda=0{\cdot}70926$) on a Picker FACS-1 four-circle diffractometer. The red parallelepiped crystal measured 0·31 × 0·16 × 0·36 mm. Systematic absences: hkl for $h+k$ odd, $h0l$ for l odd. One quadrant of data was collected in the range $3°<2\theta<52°$. Of the 1678 unique data in this range, 1134 with an intensity greater than $3\sigma(F_0^2)$ based on counting statistics were used for the structure determination. Lorentz, polarization and absorption (Ni only) corrections were applied. The structure was solved by analysis of a series of Fourier syntheses. Refinement was by full-matrix least-squares techniques with weights derived from counting statistics. Three positional parameters for all atoms except Ni, anisotropic temperature factors for non-hydrogen atoms, isotropic temperature factors for hydrogen atoms and one scale factor (total of 142) were refined. The final conventional residual index was 0·049. Inclusion of 544 unobserved data without further refinement raised the residual to 0·080. Atomic coordinates and thermal parameters are listed in Table 1. Bond lengths and angles are included in Fig. 1.*

Discussion. Graddon & Mockler (1968) report that this compound forms as a deep green monomeric high-

* The table of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30259 (8 pp.). Copies may be obtained from the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Final structure parameters with standard deviations in parentheses*

(*a*) Heavy atoms (anisotropic thermal parameters)

The anisotropic thermal parameters are in the form $\times 10^{-4}$ exp $[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})]$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Ni	0·2500	0·2500	0·5000	80 (1)	65 (1)	25 (1)	−2 (1)	14 (1)	−2 (1)
O(1)	0·1831 (3)	0·4097 (4)	0·4316 (2)	121 (4)	121 (6)	43 (2)	−8 (4)	20 (2)	−2 (2)
O(2)	0·2692 (3)	0·1476 (3)	0·4069 (2)	105 (3)	74 (4)	26 (1)	10 (3)	16 (2)	1 (2)
C(1)	0·1431 (4)	0·4080 (5)	0·0315 (2)	53 (4)	77 (5)	22 (2)	6 (4)	11 (2)	4 (2)
C(2)	0·1880 (4)	0·5060 (5)	0·0986 (3)	64 (4)	70 (6)	27 (2)	7 (4)	14 (2)	−1 (2)
C(3)	0·1855 (5)	0·4487 (5)	0·1773 (3)	93 (5)	90 (6)	24 (2)	−5 (5)	11 (3)	−4 (3)
C(4)	0·1410 (4)	0·3041 (6)	0·1894 (3)	85 (5)	102 (6)	23 (2)	−3 (5)	14 (3)	8 (3)
C(5)	0·0957 (4)	0·2054 (5)	0·1244 (3)	62 (4)	92 (6)	31 (2)	−2 (4)	13 (2)	9 (3)
C(6)	0·0987 (4)	0·2599 (6)	0·0469 (3)	58 (4)	86 (6)	25 (2)	5 (5)	7 (2)	−3 (3)
C(7)	0·3591 (4)	0·0452 (5)	0·0529 (3)	52 (4)	75 (6)	29 (2)	15 (4)	8 (2)	4 (3)
C(8)	0·4095 (5)	0·1506 (6)	0·1222 (3)	73 (5)	100 (7)	24 (2)	0 (5)	7 (3)	−6 (3)
C(9)	0·0458 (7)	0·0468 (8)	0·1369 (4)	112 (7)	121 (9)	36 (2)	−41 (6)	15 (4)	9 (4)