

is to be considered the upper limit of the acceptable coordination distances, Co(3) is surrounded by six Ho atoms. The coordination can be described by two equivalent figures: either a truncated Archimedian antiprism with two opposite corners through the center of the antiprism missing or a pentagon with one corner being replaced by a pair of atoms along a direction perpendicular to the pentagon plane. The occurrence of these different coordination polyhedra with Ho<sub>12</sub>Co<sub>7</sub> is rather unexpected, since the compounds on both sides, Ho<sub>3</sub>Co and Ho<sub>4</sub>Co<sub>3</sub>, have a much simpler construction. They are all composed of Co-centered trigonal rare-earth prisms. However, in Ho<sub>4</sub>Co<sub>3</sub> one of the nine Co atoms per unit cell is at the center of an octahedron. The structure of Y<sub>3</sub>Rh<sub>2</sub> (Moreau, Paccard & Parthé, 1976b) is characterized by the same four types of transition metal-centered rare-earth polyhedra found in Ho<sub>12</sub>Co<sub>7</sub>.

The R<sub>3</sub>Rh<sub>2</sub> alloys are brittle whilst those of the R<sub>12</sub>Co<sub>7</sub> phase show typical plastic behavior. Hence there seems to be no correlation between the mechanical properties and the type of coordination polyhedra of the transition metal atoms.

It is a pleasure to acknowledge the help of Dr Le Roy who constructed a model of Ho<sub>12</sub>Co<sub>7</sub> in Geneva. Two of us (W. A. and J. S.) are indebted to Professor A. Fratini (Dayton) for the refinement program and to Professor A. Ray (Dayton) for use of X-ray equipment.

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### N-2-(6'-Methyl)pyridyl-4-ethoxycarbonyl-2H,3H-pyrrole-2-one

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**Abstract.** C<sub>13</sub>H<sub>14</sub>N<sub>2</sub>O<sub>3</sub>, monoclinic, *P*2<sub>1</sub>/*c* C<sub>2h</sub><sup>5</sup>, *a* = 8·840 (2), *b* = 7·880 (2), *c* = 17·814 (4) Å, β = 97·21 (1)°, *D*<sub>x</sub> = 1·32 (1), *D*<sub>c</sub> = 1·328 g cm<sup>-3</sup>, *Z* = 4. The compound was prepared and kindly supplied by Vasvári-Debreczy and Mészáros at the Chinoin Research Laboratories, Budapest. Recrystallization from CHCl<sub>3</sub> yielded suitable crystals. The final *R* index is 0·048 for the full-matrix least-squares refinement of 164 variables based on 2074 observations. The dihedral angle between the best planes of the five and six-membered rings is 5·2°.

**Introduction.** As part of a systematic study of pharmaceutically interesting *N*-(pyridyl)pyrrole derivatives Dvortsák, Náray-Szabó & Vasvári-Debreczy (1975) have correlated PMR chemical shift data with quantum mechanical calculations of charge distributions. The present crystal structure study was undertaken to

provide details on the conformation in the solid state of one such derivative.

Details of data collection on a Picker FACS-I diffractometer are given in Table 1 and parallel those described earlier (Corfield, Doedens & Ibers, 1967). The unit-cell parameters were determined from a least-squares refinement of 12 reflections in the range 50 < 2θ (Cu Kα<sub>1</sub>) < 58°. No decomposition occurred during data collection. The data were processed in the usual way, using a value of *p* of 0·04 in the estimation of standard deviations. The structure was solved by direct methods and refined by full-matrix least-squares methods utilizing the 2074 reflections above background. In the refinement the quantity Σw(|*F*<sub>o</sub>| - |*F*<sub>c</sub>|)<sup>2</sup> was minimized, where *w* = 4*F*<sub>o</sub><sup>2</sup>/σ<sup>2</sup>(*F*<sub>o</sub><sup>2</sup>). Scattering factors for the nonhydrogen atoms were taken from Cromer & Waber (1974) while those for H were from Stewart, Davidson & Simpson (1965). From the initial *E* map the positions of all nonhydrogen atoms were clear. On a subsequent difference Fourier map the positions of all the H atoms were discernible. These

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positions were idealized ( $C-H=0.95 \text{ \AA}$ ) and used in the calculation of fixed structure factor contributions in ensuing cycles of refinement. The final anisotropic refinement of 164 variables (including an isotropic extinction parameter) converged to agreement indices of  $R=0.048$  and  $R_w=0.079$  and to an error in an observation of unit weight of  $2.83 e^* \text{ \AA}^{-3}$ . In a final difference Fourier synthesis the highest residual is  $0.18 e \text{ \AA}^{-3}$ , to be compared with the smallest hydrogen peak of  $0.28 e \text{ \AA}^{-3}$  found earlier. An analysis of

\* A list of structure amplitudes has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31773 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Summary of crystal data and intensity collection

Crystal dimensions	0.17 × 0.29 × 0.39 mm
Crystal shape	monoclinic prism (distorted) with {001}, {011}, {100} and {310} (cut face)
Temperature	22°C
Radiation	Cu $K\alpha_1$ ( $\lambda=1.54056 \text{ \AA}$ ) prefiltered with $1 \times 10^{-3}$ in of Ni foil
Transmission factors	0.786–0.905
$\mu$	$7.52 \text{ cm}^{-1}$
Receiving aperture	2.7 mm wide by 3.2 mm high, 32 cm from crystal
Take-off angle	$2.4^\circ$
Scan speed	$2^\circ \text{ min}^{-1}$ $4 < 2\theta < 100^\circ$ , $1^\circ \text{ min}^{-1}$ $100 < 2\theta < 160^\circ$
Scan range	$0.7^\circ$ below $K\alpha_1$ to $0.7^\circ$ above $K\alpha_2$ $4 < 2\theta < 125^\circ$ , $1.0^\circ$ below $K\alpha_1$ to $0.7^\circ$ above $K\alpha_2$ $125 < 2\theta < 160^\circ$
Background	10 s $4 < 2\theta < 80^\circ$ , 20 s $80 < 2\theta < 160^\circ$
Standard reflections	6 in the range of $4 < 2\theta < 125^\circ$ (bisecting mode), 3 in the range of $125 < 2\theta < 160^\circ$ (parallel mode)

Table 2. Positional and thermal parameters for the atoms of  $C_{13}H_{14}N_2O_3$

Estimated standard deviations in the least significant figure(s) are given in parentheses in this and all subsequent tables. The form of the anisotropic thermal ellipsoid is  $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$ . The quantities given are the thermal coefficients  $\times 10^4$ , and positional parameters  $\times 10^5$ .

	x	y	z	$\beta_{11}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
N(1)	29743 (19)	-2471 (21)	-8016 (9)	144.8 (25)	146.6 (26)	27.9 (5)	3.9 (20)	13.6 (8)	-1.8 (9)
C(2)	35530 (25)	-4485 (26)	-14559 (11)	154.7 (31)	167.7 (34)	30.0 (6)	-11.9 (25)	17.8 (11)	-7.0 (11)
C(3)	45733 (28)	-17369 (30)	-15579 (14)	169.7 (35)	196.5 (40)	41.6 (8)	-6.2 (30)	30.2 (13)	-17.2 (15)
C(4)	50371 (28)	-28100 (30)	-9598 (15)	165.2 (37)	173.9 (37)	51.3 (10)	25.2 (29)	26.5 (15)	-12.7 (15)
C(5)	44694 (26)	-25997 (27)	-2806 (13)	168.0 (35)	154.2 (34)	40.8 (8)	28.8 (27)	14.8 (13)	-0.5 (13)
C(6)	34298 (22)	-13008 (23)	-2342 (11)	134.4 (28)	128.8 (27)	30.8 (6)	2.5 (21)	10.8 (10)	-4.8 (10)
N(7)	27549 (20)	-9810 (20)	4324 (9)	156.4 (26)	132.1 (24)	27.7 (5)	21.9 (20)	14.4 (9)	4.1 (9)
C(8)	18120 (23)	4301 (23)	5103 (10)	141.8 (29)	135.2 (28)	28.3 (6)	8.5 (23)	7.4 (10)	3.9 (10)
C(9)	13477 (23)	4695 (23)	11990 (10)	151.3 (30)	136.0 (29)	26.8 (6)	4.2 (23)	11.6 (10)	1.7 (10)
C(10)	20155 (29)	-10145 (28)	16405 (12)	213.2 (40)	164.3 (35)	31.1 (7)	26.1 (30)	22.2 (12)	14.4 (12)
C(11)	29105 (28)	-19208 (26)	11030 (12)	200.5 (38)	149.0 (33)	33.0 (7)	24.0 (28)	15.4 (12)	13.7 (12)
C(12)	30257 (31)	7837 (35)	-20711 (13)	222.3 (45)	233.6 (47)	32.3 (7)	9.9 (36)	25.6 (14)	12.6 (15)
O(13)	36326 (26)	-32117 (22)	12176 (10)	338.3 (45)	197.3 (33)	46.8 (7)	112.3 (31)	40.7 (14)	35.3 (12)
C(14)	4281 (23)	17645 (23)	15055 (10)	143.0 (29)	142.8 (29)	25.8 (5)	-6.8 (22)	8.8 (9)	0.3 (10)
O(15)	787 (19)	17190 (19)	21411 (7)	220.6 (29)	188.9 (27)	25.89 (44)	21.3 (22)	24.2 (8)	4.2 (9)
O(16)	158 (17)	30183 (17)	10177 (7)	183.4 (25)	146.7 (23)	28.19 (43)	29.5 (18)	20.4 (8)	4.5 (8)
C(17)	-8921 (29)	43527 (27)	13051 (12)	206.1 (38)	163.9 (35)	32.3 (7)	32.6 (29)	21.1 (12)	-8.4 (12)
C(18)	-12021 (34)	56741 (32)	7027 (15)	267 (5)	180.1 (41)	44.4 (9)	65.3 (37)	31.5 (17)	10.0 (16)

$\sum w(|F_o| - |F_c|)^2$  as a function of  $|F_o|$ , Miller indices, and setting angles revealed no unusual trends. All but one of the 644 reflections omitted from the refinement satisfied the condition  $|F_o^2 - F_c^2| < 4\sigma(F_o^2)$ . Final positional and thermal parameters are given in Tables 2 and 3. The atomic labelling scheme, together with important metrical information, is given in Fig. 1. A drawing of the molecule is given in Fig. 2.

**Discussion.** The striking feature of the molecular structure is its near planarity. The least-squares planes for the *N*-picoline and pyrrole groups are given in Table 4. The angle between these planes is only  $5.2^\circ$ . Thus the suggestion of Dvortsák *et al.* (1975) from spectroscopic data and quantum mechanical calculations that the molecule is planar is essentially correct. Perhaps this planarity may be explained by the following considerations. The lone pair electrons of atom N(7) can be delocalized in three directions toward atoms C(6), C(8), and C(11). This delocalization is manifested

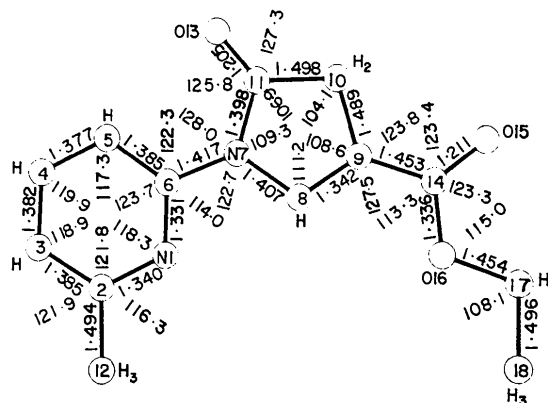


Fig. 1. Bond distances and angles and the numbering scheme for the molecule. Standard deviations of the bond lengths are  $0.002\text{--}0.004 \text{ \AA}$ , of the angles  $0.2^\circ$ .

Table 3. Idealized positional and isotropic thermal parameters for the hydrogen atoms

	x	y	z	B (Å <sup>2</sup> )
H[C(3)]	0.498	-0.186	-0.203	5.81
H[C(4)]	0.577	-0.369	-0.102	6.07
H[C(5)]	0.477	-0.332	0.014	5.50
H[C(8)]	0.153	0.124	0.012	4.61
Ha[C(10)]	0.266	-0.064	0.208	5.65
Hb[C(10)]	0.124	-0.172	0.180	5.65
Ha[C(17)]	-0.182	0.390	0.143	5.73
Hb[C(17)]	-0.034	0.485	0.175	5.73
Ha[C(12)]	0.352	0.184	-0.198	6.32
Hb[C(12)]	0.325	0.034	-0.255	6.32
Hc[C(12)]	0.195	0.093	-0.210	6.32
Ha[C(18)]	-0.029	0.613	0.058	6.82
Hb[C(18)]	-0.176	0.517	0.026	6.82
Hc[C(18)]	-0.182	0.655	0.088	6.82

Table 4. Best weighted least-squares planes

Plane I  $6.362x + 4.858y + 4.036z = 1.453$

Plane II  $6.784x + 4.280y + 4.302z = 1.633$

Distances (Å) from the planes. For the atoms defining the plane the errors are given.

	Plane I	Plane II
N(1)	-0.004 (2)	-0.066
C(2)	0.002 (2)	
C(3)	-0.016 (2)	
C(4)	-0.001 (3)	
C(5)	0.014 (2)	0.166
C(6)	0.003 (3)	0.036
N(7)	-0.003 (2)	0.002 (2)
C(8)	0.115	0.000 (2)
C(9)	0.116	-0.002 (2)
C(10)	-0.002	0.006 (3)
C(11)	-0.089	-0.006 (2)
C(12)	0.017 (3)	
O(13)	-0.211	-0.020
C(14)	0.284	0.060
O(15)	0.296	0.077
O(16)	0.434	0.107
C(17)	0.621	0.186
C(18)	0.822	0.282

Dihedral angle between planes I and II is 5.2°.

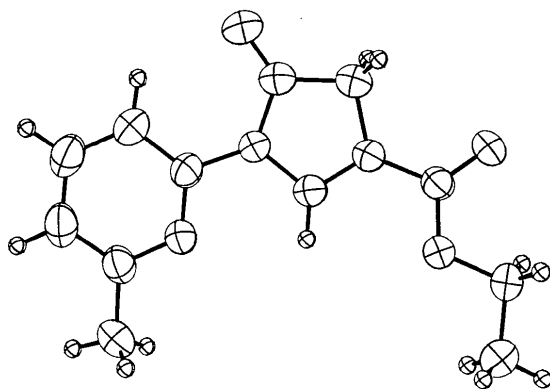


Fig. 2. A diagram of the molecule showing vibrational ellipsoids at the 50% probability level except for hydrogen atoms, which are artificially small.

in the N(1)-C(6)-N(7)-C(11) torsion angle of 175.2 (2)° and the fact that the N(7)-C(6), N(7)-C(8), and N(7)-C(11) bond lengths of 1.417 (2), 1.407 (2), and 1.398 (2) Å are all significantly shorter than a normal N-C(sp<sup>2</sup>) single bond. The 5.2° tilt between the two rings along C(6)-N(7) and the increased angles of C(5)-C(6)-N(7) [122.3 (2)°] and C(6)-N(7)-C(11) [128.0 (2)°] compared with the angles of N(1)-C(6)-N(7) [114.0 (2)°] and C(6)-N(7)-C(8) [122.7 (2)°] probably arise from O(13)-H[C(5)] repulsion. This distance is calculated to be 2.27 Å, about 0.2 Å shorter than any intermolecular distance.

Table 5. Comparison of some structural data of 5-methoxycarbonylmethylene-2-piperidino-Δ<sup>2</sup>-thiazolin-4-one (I), N-(α-glutarimido)phthalimide (II), and the present structure (III)

	I	II	III
Bonds (Å)			
a	1.314 (9)	1.455 (3)	1.417 (2)
b	1.467 (10)	1.391 (3)	1.407 (2)
c	1.459 (10)	1.391 (3)	1.398 (2)
d	1.324 (9)	1.522 (4)	1.331 (2)
e	1.782 (7)	1.506 (4)	1.385 (3)
Torsion angles (°)			
c-a-d	-173.2	103.6	175.2
References			
	Cameron & Hair (1971)	Allen & Trotter (1971)	Present structure

Table 5 compares the present results with those on two related structures. In compound I planarity presumably results from the multiple nature of the N-C bond connecting the two rings. In compound II there is no opportunity for multiple bonding and the rings are nearly perpendicular to one another.

Computer programs used in this study were local modifications of Zalkin's *FORDAP* Fourier program, Dewar's *FAME* scaling program, the Main, Woolfson & Germain *MULTAN* direct-methods program, Johnson's *ORTEP* thermal ellipsoid plotting program, and the Busing & Levy *ORFFE* error-function program. Our least-squares program *NUCLS*, in its non-group form, closely resembles the Busing-Levy *ORFLS* program. The absorption-correction program, *AGNOST*, included in our case the Tompa & de Meulenaer analytical method. All computations were carried out on a CDC 6400 computer.

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## 1,2,3,5-Tetra-O-acetyl- $\beta$ -D-ribofuranose

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**Abstract.** C<sub>13</sub>H<sub>18</sub>O<sub>9</sub>, orthorhombic,  $P2_12_12_1$ ,  $a=7.501$  (1),  $b=13.665$  (1),  $c=15.337$  (1) Å,  $Z=4$ ,  $D_m=1.345$  g cm<sup>-3</sup>. The acetyl groups each adopt the expected planar conformation with the carbonyl and acetyl CO bonds in an eclipsed disposition; in addition, for three of the four methyl groups, one CH bond is *cis* to the carbonyl bond. The furanose ring has the <sup>3</sup>T<sub>2</sub> twist conformation, with normal bond lengths and angles except for the C(5)C(4) bond which is 1.508 (3) Å. Final *R* value was 0.052 with 1891 observed reflections. The structure agrees well with that determined by James & Stevens [*Cryst. Struct. Commun.* (1973), **2**, 609–612].

**Introduction.** The title compound is reported to crystallize in two forms: *A*, m.p. 58°C, and *B*, m.p. 85°C (Brown, Davoll & Lowy, 1955). Form *B* will catalyze the transformation of *A* to *B* in both the liquid and solid state (Davoll, Brown & Visser, 1952). A preliminary X-ray investigation of the solid-state transformation gave the cell parameters of Table 1 for the two forms (Patterson & Groshens, 1954), which imply

an increase of the molecular volume by about 2% in passing from *A* to the more stable *B* form. The mechanism of the transformation is unknown and if the reported loss of acetic acid (Davoll, Brown & Visser, 1952) is indeed associated with the change then the system is most unusual.

An attempt to produce the rare *A* form by application of high pressure, based on the assumption that the transformation was conformational rather than chemical, was not successful. The infrared spectrum and polarization in visible light were followed up to a pressure of 4.2 GPa, but no variations attributable to the desired change were noted (Linton, 1973).

In an initial step to resolve this unusual behavior the structure determination of the title compound was undertaken. Subsequently a report of an independent determination appeared (James & Stevens, 1973). The present paper reports the results of our analysis and briefly compares the two structure analyses.

### Structure analysis

A crystal was ground approximately spherical to a radius of 0.21 (1) mm from a sample supplied by Dr C. M. Stewart of the CSIRO Division of Chemical Technology, Melbourne. The unit-cell parameters were derived by least-squares refinement against  $2\theta$  values for 15 reflections ( $45^\circ \leq 2\theta \leq 90^\circ$ ) determined on a Nonius CAD-4 diffractometer with graphite-monochromated Cu radiation ( $\lambda=1.5418$  Å). A total of 1871 integrated intensities were measured with an  $\omega$ - $2\theta$  scan; of these reflections 140 were less than  $3\sigma_I$ . The usual Lorentz and polarization corrections were applied in addition to an absorption correction based on a spherical crystal.

The structure was solved by *MULTAN* (Germain, Main & Woolfson, 1971) and completed with, initially, the system of Shiono (1971) and later the X-RAY

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Table 1. Cell parameters

	Form <i>A</i>	Form <i>B</i>
<i>a</i>	12.49 Å	7.50 Å
<i>b</i>	5.58	13.66
<i>c</i>	11.12	15.33
$\beta$	97.75°	
<i>Z</i>	2	4
<i>V</i>	767.9 Å <sup>3</sup>	1570.6 Å <sup>3</sup>
Space group	$P2_1$ Monoclinic	$P2_12_1$ Orthorhombic