

bond lengths and angles in Table 2. A stereo drawing of the unit cell is shown in Fig. 1.

Discussion. As expected, the crystallographically non-equivalent Pt(1)–C(1), Pt(1)–C(2), and C(1)–N(1), C(2)–N(2) bond lengths are equal to within one e.s.d., as shown in Table 2.

The Pt atom is octahedrally coordinated, with the cyanides forming a square-planar arrangement around it. The Br atoms occupy axial positions around the Pt atom. The tetracyanoplatinate interatomic distances are in close agreement with those previously reported (Washecheck, Peterson, Reis & Williams, 1976; Williams, Keefer, Washecheck & Enright, 1976; Maffly, Johnson, Koch & Williams, 1977).

The Rb interacts with only two species, the cyanide N atoms and the Br atoms. It is at the center of a somewhat distorted bi-capped trigonal prism, with N

atoms at the corners of the prism and Br atoms capping two of the side faces.

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4-Carboxy-1,2,4,8-tetramethyl-3,9-dioxatricyclo[5.2.1.0^{2,6}]decane-8-carboxamide

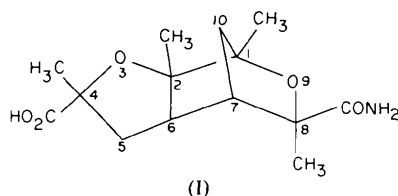
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(Received 21 September 1976; accepted 27 October 1976)

Abstract. C₁₄H₂₁NO₅, F.W. 283.3; monoclinic, *P*2₁/*c*, *a* = 11.770 (5), *b* = 10.811 (5), *c* = 11.346 (5) Å, β = 91.79 (5)° (20°C); *D_m* = 1.302, *Z* = 4, *D_x* = 1.304 g cm⁻³. The molecular configuration is found to be *rel*-(1*R*,2*R*,4*R*,6*R*,7*S*,8*R*).

Introduction. The title compound (I) was obtained by hydrolysis of a product of the AlCl₃-catalysed reaction of 2,5-dimethylfuran with ethyl cyanofornate (McCulloch & McInnes, 1976). The crystal-structure analysis was undertaken in order to establish the hitherto unknown molecular structure.



The crystals supplied were colourless, transparent, triangular plates <100>. One of these was cut to yield a specimen of dimensions 0.13 × 0.20 × 0.35 mm. This was mounted on a four-circle diffractometer with the longest direction (corresponding to *b*) parallel to the ϕ

axis. The intensities of the 2457 independent reflexions within the limiting sphere $2\theta = 130^\circ$ were measured with Cu *K*α radiation and a scintillation counter. The θ - 2θ scan mode was used, with scans of 2° for $2\theta < 100^\circ$, and 3° otherwise. Background intensities were estimated from an experimentally derived function of θ . 236 reflexions were considered to be unobserved, as their net intensities were less than either 30 counts or 10% of the corresponding background intensity. No correction was made for absorption ($\mu = 8.3$ cm⁻¹).

The structure was readily determined by symbolic addition procedures, with all non-hydrogen atoms appearing in the first *E* map. Refinement was by block-diagonal least squares, minimizing $\sum w\Delta F^2$. The identification of atoms, initially based on chemical plausibility, was subsequently verified by a study of ΔF syntheses. (At a later stage in refinement, a persistent peak and trough in the ΔF synthesis gave warning that a presumed OH group was in fact NH₂; the substitution caused the *R* index to drop from 0.047 to 0.039.) The seven most intense reflexions were corrected for extinction, the largest correction being 14% of *F_o*. The weighting scheme used in the final stages was $w = w_1 w_2$, where $w_1 = 5/F_o$ for $F_o > 5$, $w_1 = 1$ otherwise,

and $w_2 = 2.5 \sin^2 \theta$ for $\sin^2 \theta < 0.4$, $w_2 = 1$ otherwise. With this scheme the weighted residual showed no obvious dependence on F_o or θ . In the final cycle of refinement the largest coordinate shift of a non-hydrogen atom was about 30% of the corresponding e.s.d. of 0.0015 Å. The final R index (for observed reflexions only) was 0.038.* The final atomic positions are given in Table 1.

The computer programs used were those of Ahmed, Hall, Pippy & Huber (1966). Scattering factors were

* A list of structure factors and the isotropic (for H) and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32250 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Final atomic coordinates ($\times 10^3$ for hydrogen atoms, $\times 10^4$ otherwise)

	<i>x</i>	<i>y</i>	<i>z</i>
C(1)	7129 (1)	-349 (1)	207 (1)
C(2)	7197 (1)	-1616 (1)	840 (1)
O(3)	7878 (1)	-2414 (1)	131 (1)
C(4)	8706 (1)	-3043 (1)	851 (1)
C(5)	8967 (1)	-2187 (1)	1900 (2)
C(6)	7944 (1)	-1310 (1)	1961 (1)
C(7)	8241 (1)	71 (1)	1781 (1)
C(8)	7161 (1)	844 (1)	1933 (1)
O(9)	6438 (1)	426 (1)	955 (1)
C(10)	8320 (1)	158 (1)	440 (1)
C(11)	6618 (1)	-346 (2)	-1024 (1)
C(12)	6047 (1)	-2198 (2)	1054 (2)
C(13)	9737 (2)	-3284 (2)	100 (2)
C(14)	8224 (1)	-4303 (1)	1207 (1)
O(15)	8874 (1)	-4914 (1)	1965 (1)
O(16)	7338 (1)	-4702 (1)	802 (2)
C(17)	6543 (2)	709 (2)	3077 (2)
C(18)	7400 (1)	2232 (1)	1770 (1)
N(19)	6710 (2)	2803 (1)	1017 (2)
O(20)	8155 (1)	2757 (1)	2357 (1)
H(5,1)	909 (2)	-265 (2)	263 (2)
H(5,2)	967 (2)	-169 (2)	174 (2)
H(6,1)	753 (1)	-142 (2)	270 (2)
H(7,1)	888 (2)	35 (2)	226 (2)
H(10,1)	841 (2)	100 (2)	14 (2)
H(10,2)	889 (2)	-38 (2)	9 (2)
H(11,1)	584 (2)	-58 (2)	-102 (2)
H(11,2)	703 (2)	-93 (2)	-149 (2)
H(11,3)	670 (2)	48 (2)	-138 (2)
H(12,1)	614 (2)	-290 (2)	155 (2)
H(12,2)	554 (2)	-158 (2)	141 (2)
H(12,3)	572 (2)	-251 (3)	31 (2)
H(13,1)	1031 (2)	-367 (2)	58 (2)
H(13,2)	999 (2)	-252 (2)	-21 (2)
H(13,3)	955 (2)	-390 (2)	-60 (2)
H(15,1)	854 (2)	-560 (2)	213 (2)
H(17,1)	710 (3)	97 (3)	370 (3)
H(17,2)	584 (2)	123 (3)	306 (2)
H(17,3)	622 (2)	-10 (3)	321 (2)
H(19,1)	675 (3)	355 (3)	90 (3)
H(19,2)	620 (2)	236 (2)	62 (2)

E.s.d.'s are given in parentheses.

taken from Stewart, Davidson & Simpson (1965) for H, and from Hanson, Herman, Lea & Skillman (1964).

Discussion. Bond lengths and angles are given in Table 2, and merit no detailed consideration. The fused-ring nucleus of the molecule may be described as consisting of the five-membered rings *A*, *B* and *C*, defined in Table

Table 2. Interatomic distances (Å) and bond angles (°)

E.s.d.'s of angles range from 0.11 to 0.17°. Atoms designated $X(n)^1$ are related to $X(n)$ by the *b* translation.

C(1)–C(2)	1.547 (2)	C(2)–C(1)–C(9)	105.1
C(2)–O(3)	1.440 (2)	C(2)–C(1)–C(10)	101.8
O(3)–C(4)	1.424 (2)	C(2)–C(1)–C(11)	116.5
C(4)–C(5)	1.531 (2)	C(9)–C(1)–C(10)	102.7
C(5)–C(6)	1.536 (2)	C(9)–C(1)–C(11)	109.0
C(2)–C(6)	1.559 (2)	C(10)–C(1)–C(11)	120.0
C(6)–C(7)	1.549 (2)	C(1)–C(2)–O(3)	107.1
C(7)–C(8)	1.535 (2)	C(1)–C(2)–C(6)	102.2
C(8)–O(9)	1.449 (2)	C(1)–C(2)–C(12)	114.0
C(1)–O(9)	1.459 (2)	O(3)–C(2)–C(6)	105.8
C(1)–C(10)	1.520 (2)	O(3)–C(2)–C(12)	110.8
C(7)–C(10)	1.530 (2)	C(6)–C(2)–C(12)	116.1
C(1)–C(11)	1.504 (2)	C(2)–O(3)–C(4)	110.4
C(2)–C(12)	1.519 (2)	O(3)–C(4)–C(5)	105.9
C(4)–C(13)	1.528 (3)	O(3)–C(4)–C(13)	107.6
C(4)–C(14)	1.534 (2)	O(3)–C(4)–C(14)	108.9
C(14)–O(15)	1.311 (2)	C(5)–C(4)–C(13)	113.1
C(14)–O(16)	1.207 (2)	C(5)–C(4)–C(14)	113.5
C(8)–C(17)	1.515 (3)	C(13)–C(4)–C(14)	107.6
C(8)–C(18)	1.539 (2)	C(4)–C(5)–C(6)	105.6
C(18)–N(19)	1.314 (3)	C(2)–C(6)–C(5)	104.8
C(18)–O(20)	1.232 (2)	C(2)–C(6)–C(7)	102.8
N(19)–H(19,1)	0.82 (3)	C(5)–C(6)–C(7)	114.1
N(19)–H(19,2)	0.88 (3)	C(6)–C(7)–C(8)	108.6
O(15)–H(15,1)	0.86 (2)	C(6)–C(7)–C(10)	102.2
O(15) ... O(20) ¹	2.698 (2)	C(8)–C(7)–C(10)	98.9
H(15,1) ... O(20) ¹	1.86 (2)	C(7)–C(8)–O(9)	102.3
N(19) ... O(16) ¹	2.809 (2)	C(7)–C(8)–C(17)	117.7
H(19,1) ... O(16) ¹	2.01 (3)	C(7)–C(8)–C(18)	111.3
N(19) ... O(9)	2.591 (2)	O(9)–C(8)–C(17)	109.8
H(19,2) ... O(9)	2.14 (2)	O(9)–C(8)–C(18)	108.5
C–H (mean)	0.98 (3)	C(17)–C(8)–C(18)	106.9
C–H (range)	0.95–1.05	C(8)–O(9)–C(1)	107.4
		C(1)–C(10)–C(7)	93.8
		C(4)–C(14)–O(15)	114.0
		C(4)–C(14)–O(16)	122.7
		O(15)–C(14)–O(16)	123.3
		C(8)–C(18)–N(19)	115.2
		C(8)–C(18)–O(20)	121.0
		N(19)–C(18)–O(20)	123.8

Table 3. Distances of some atoms from certain mean planes

Atoms marked by an asterisk define the plane. Units of distance are 0.001 Å.

Ring	
<i>A</i>	C(2)* 24; O(3) 364; C(4)* -24; C(5)* 37; C(6)* -37
<i>B</i>	C(1)* -10; C(2)* 15; C(6)* -15; C(7)* 10; C(10) 871
<i>C</i>	C(1)* 35; O(9)* -53; C(8)* 50; C(7)* -32; C(10) 855

Dihedral angles: *AB* 56.7°; *BC* 68.2°.

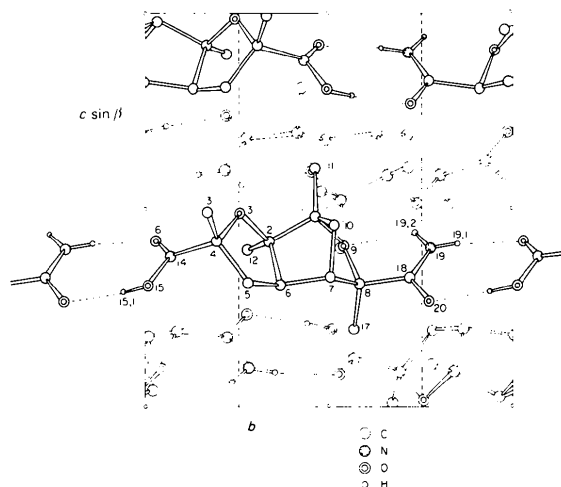


Fig. 1. The structure viewed along *a*. Molecules drawn in heavy outline lie at about $x = \frac{3}{4}$; those in lighter outline at about $x = \frac{1}{4}$. H atoms not involved in hydrogen bonding have been omitted.

3. *A* and *B* share one side, and *B* and *C* two sides. Each of these rings is in the envelope conformation, with one atom lying significantly out of the plane of the remaining four. C(10), the apex of the shared flap of envelopes

B and *C*, is almost equidistant from the four-atom planes.

The orientation of the carboxamide group is stabilized by the intramolecular hydrogen bridge N(19)–H(19,2)···O(9). Molecules related by the *b* translation are joined by N–H···O and O–H···O bonds, as shown in Fig. 1. Contacts between the chains so formed appear to be consistent with van der Waals interactions.

The author is grateful to Dr A. W. McCulloch for suggesting the problem and for supplying specimen material.

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Dimethyl 3-Phenyl-1*H*-indene-1,2-dicarboxylate

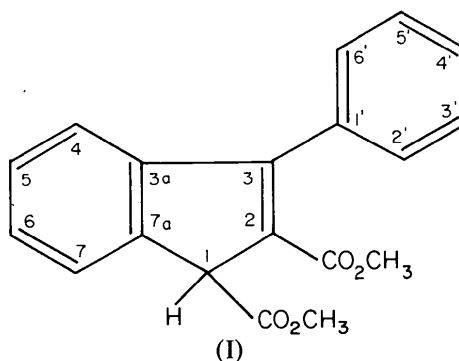
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(Received 13 September 1976; accepted 22 October 1976)

Abstract. C₁₉H₁₆O₄, F.W. 308.3; triclinic, $P\bar{1}$, $a = 10.470$ (5), $b = 12.141$ (6), $c = 8.098$ (4) Å, $\alpha = 122.7$ (1), $\beta = 113.7$ (1), $\gamma = 68.1$ (1)° (20°C). $D_m = 1.322$, $Z = 2$, $D_x = 1.317$ g cm⁻³. The final *R* (observed reflexions only) is 0.040. Van der Waals forces govern the molecular packing, which is dominated by face-to-face stacking of the indene nuclei.

Introduction. The title compound (I) has been characterized by chemical and spectroscopic techniques (Matheson, McCulloch, McInnes & Smith, 1976). However, it was considered essential to have further proof of the proposed structure, and to establish the molecular conformation. The crystal-structure analysis was undertaken for this purpose.



The crystals supplied were clear and colourless, with well defined faces. The crystal class was established