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Fig. 2. Empilement des entités bimoléculaires (NiHMK)$_2$.

Dans NiMMK (Abraham *et al.*, 1984) aucune interaction moléculaire notable n'a été relevée: aucune distance intermoléculaire faisant intervenir des atomes non-hydrogène n'est inférieure à 3,5 Å et il n'existe pas d'orientation privilégiée des plans des molécules les uns par rapport aux autres. La structure cristalline de NiHMK (Fig. 2) montre l'existence d'entités bimoléculaires constituées de molécules reliées par un centre de symétrie. Dans cette association cofaciale, les plans moyens des deux molécules sont parallèles et éloignés de 3,33 (1) Å, ce qui permet une approche des deux atomes de nickel à 3,303 (2) Å. Cette association

cofaciale de deux complexes plans du nickel a déjà été observée pour des ligands macrocycliques aza-annulène (Peng & Goedken, 1976). Toutefois les interactions métalliques ne peuvent se propager dans tout le cristal comme dans le cas de certains complexes plan carré du nickel (Gleizes, Marks & Ibers, 1975).

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Structure of 1,3-(1,1'-Ruthenocenediyl)-1-propanone, [Ru(C$_{13}$H$_{12}$O)], and 1,3-[1,1'-(3,4-Trimethyleneruthenocenediyl)]propane, [Ru(C$_{16}$H$_{18}$)]

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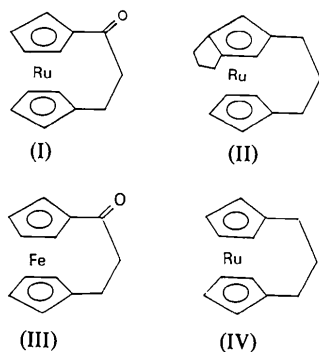
Abstract. C$_{13}$H$_{12}$ORu (I): $M_r = 285 \cdot 3$, triclinic, $P\bar{1}$, $a = 14 \cdot 041$ (3), $b = 14 \cdot 631$ (3), $c = 5 \cdot 767$ (1) Å, $\alpha = 101 \cdot 17$ (2), $\beta = 91 \cdot 31$ (2), $\gamma = 118 \cdot 97$ (1)°, $V = 1007 \cdot 4$ (4) Å^3, $Z = 4$, $D_m = 1 \cdot 87$ (2), $D_x = 1 \cdot 88$ Mg m^{-3}, Mo $K\alpha_1$, $\lambda = 0 \cdot 70926$ Å, $\mu = 1 \cdot 49$ mm^{-1}, $F(000) = 568$, $T = 294$ (2) K, $R = 0 \cdot 024$ for 3956 observed unique reflections. C$_{16}$H$_{18}$Ru (II): $M_r = 311 \cdot 4$, monoclinic, $P2_1/c$, $a = 5 \cdot 909$ (2), $b = 11 \cdot 299$ (1), $c = 19 \cdot 489$ (4) Å, $\beta = 106 \cdot 00$ (2)°, $V = 1250 \cdot 9$ (5) Å^3, $Z = 4$, $D_m = 1 \cdot 70$ (2), $D_x = 1 \cdot 65$ Mg m^{-3}, Mo $K\alpha_1$, $\mu = 1 \cdot 20$ mm^{-1}, $F(000) = 632$, $T = 297$ (2) K, $R = 0 \cdot 024$ for 2470 unique reflections.

The cyclopentadienyl (Cp) rings take the eclipsed conformation and the dihedral angles between them are 14·6 (1) and 15·0 (2)° for the two independent molecules of (I) and 15·6 (1)° for (II). The dihedral angles between the plane of the $\overset{C}{\underset{C}{\diagdown}}$C=O moiety and the adjacent Cp ring in (I) are 39·5 (2) and 39·7 (2)°. The five-membered ring fused to one Cp of (II) adopts an envelope conformation, the top of which bends towards the Ru atom.

Introduction. The ^{13}C NMR chemical shift of the carbonyl carbon in (I) is higher by 3·4 p.p.m. than that in 1,3-(1,1'-ferrocenediyl)-1-propanone (III). Senda, Kamiyama, Kasahara, Izumi & Murakami (1980) predicted that the dihedral angle between the plane of the carbonyl group and the adjacent Cp ring in (I) would be smaller than that in (III). The crystal structure determination of (I) has been undertaken to compare the molecular structure with that of (III), which has already been published by Jones, Marsh & Richards (1965). The synthesis of [3][3]-ruthenocenophanes involving two heteroannular bridges has been previously reported (Kamiyama & Kasahara, 1984).* However, X-ray crystal-structure analysis revealed that (II) was [3](1,1')[3](3,4)-ruthenocenophane and not [3](1,1')[3](3,3')-ruthenocenophane. The analysis was carried out to obtain the conformational details of a homoannularly bridged carbon chain.


(I)   (II)

(III)   (IV)

Experimental. Experimental conditions shown in Table 1. D_m by flotation in aqueous solution of zinc iodide or potassium iodide. Rigaku AFC-5 four-circle diffractometer, Mo $K\alpha$ radiation monochromatized by graphite plate, θ–2θ scan, scan speed 6° min^{-1} in θ. During data collection, intensities of standard reflections of (I) decreased by 16% owing to sublimation; repeated measurements of five standard reflections used to correct. Corrections for Lorentz–polarization, not for absorption (μr for sphere < 0.25). Structure of (I) solved by direct methods with $MULTAN78$ (Main,

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* The nomenclature of metallocenophanes is according to that presented by Vögtle & Neumann (1970).

Table 1. *Experimental conditions and refinement details*

	(I)	(II)
Size of specimen (mm)	0·35 × 0·2 × 0·15	0·5 × 0·4 × 0·2
Number of reflections and 2θ range (°)	20	18
used for measuring lattice parameters	38 < 2θ < 40	40 < 2θ < 44
$2\theta_{max}(°)$	55	55
Range of h, k and l	$-18 \leq h \leq 18$	$-7 \leq h \leq 7$
	$-19 \leq k \leq 19$	$0 \leq k \leq 14$
	$-7 \leq l \leq 0$	$0 \leq l \leq 24$
Systematic absences	None	$h0l$, l odd;
		$0k0$, k odd
Variation of $|F_o|$ of 5 standard reflections,		
$\sum(|F_o|/|F_o|_{initial})/5$	0·92–1·00	0·99–1·01
Number of reflections measured	5115	3139
Number of reflections observed		
$||F_o| > 3\sigma(|F_o|)|$	4368	2545
R	0·024	0·024
wR	0·038	0·041
$w^{-1} = \sigma^2(|F_o|) + (c|F_o|)^2$	$c = 0.025$	$c = 0.025$
S	1·2	1·4
Number of observed unique reflections	3956	2470
Number of parameters refined	367	226
Final difference synthesis		
$(\Delta\rho)_{min}$ (e Å^{-3})	-0.85	-0.67
$(\Delta\rho)_{max}$ (e Å^{-3})	0·38	0·82
$(\Delta/\sigma)_{max}$	0·2	0·4 for non-H,
		1·3 for H atoms

Hull, Lessinger, Germain, Declercq & Woolfson, 1978), (II) by heavy-atom method. Non-H atoms refined with anisotropic thermal parameters, all H atoms found on difference synthesis, positional and isotropic thermal parameters of H atoms refined, $\sum w||F_o| - |F_c||^2$ minimized. $|F_o(100)|$ of (II) affected by secondary extinction was removed from refinement. Complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); *Universal Crystallographic Computation Program System UNICS*III (Sakurai & Kobayashi, 1979).*

Discussion. Table 2 lists atomic coordinates and Table 3 bond lengths and angles. There are two crystallographically independent molecules in the unit cell of (I). They are chemically equivalent and the geometries are quite similar. Perspective views of (I) and (II) are shown in Fig. 1. The dihedral angles* (Ito, 1982) between the Cp rings agree with that of 14·8 (2)° in 1,3-(1,1'-ruthenocenediyl)propane (IV) (Ohba, Saito, Kamiyama & Kasahara, 1984), ca 6° greater than the dihedral angle in (III). The dihedral angle between the plane of the $\overset{C}{\underset{C}{\diagdown}}$C=O moiety and the adjacent Cp ring in (I) is ca 2° less than 41·6 (3)° found in (III) (Jones, Marsh & Richards, 1965).
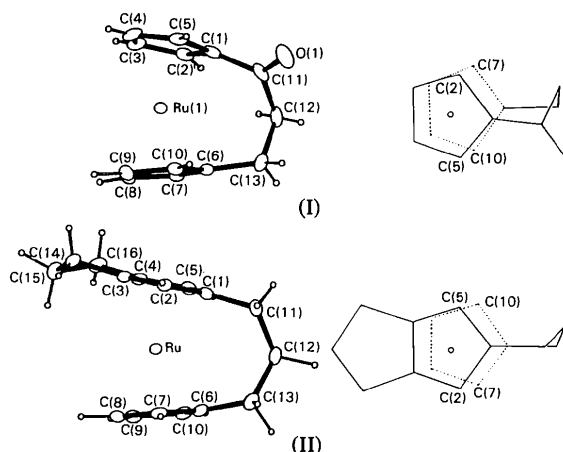
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* Lists of structure factors, anisotropic thermal parameters, atomic parameters for H atoms, bond lengths and bond angles involving H atoms, and least-squares planes through the Cp rings and the $\overset{C}{\underset{C}{\diagdown}}$C=O moiety and deviations of atoms from them have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42003 (62 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Positional parameters* ($\times 10^4$; *for* Ru $\times 10^5$) *and equivalent isotropic thermal parameters* (Hamilton, 1959)

	x	y	z	$B_{eq}(\text{Å}^2)$
(I)				
Ru(1)	18436 (2)	1247 (2)	22921 (4)	2·1
Ru(2)	80334 (2)	46743 (2)	33089 (4)	1·9
O(1)	4320 (2)	1597 (3)	7346 (5)	5·9
O(2)	6975 (2)	6043 (2)	−403 (4)	4·5
C(1)	3052 (3)	1586 (2)	4603 (5)	3·3
C(2)	2770 (3)	1795 (2)	2409 (6)	3·3
C(3)	1665 (3)	1530 (3)	2229 (6)	3·7
C(4)	1241 (3)	1153 (3)	4286 (7)	4·1
C(5)	2088 (3)	1196 (3)	5744 (6)	3·6
C(6)	2719 (2)	−731 (2)	1622 (5)	2·6
C(7)	2350 (3)	−636 (2)	−611 (5)	2·9
C(8)	1187 (3)	−1186 (3)	−944 (6)	3·4
C(9)	812 (3)	−1637 (2)	1053 (7)	3·5
C(10)	1760 (3)	−1366 (2)	2612 (6)	3·0
C(11)	4003 (3)	1458 (3)	5259 (6)	3·9
C(12)	4476 (3)	1031 (3)	3292 (7)	4·0
C(13)	3898 (3)	−197 (3)	2764 (7)	4·0
C(01)	8233 (2)	6095 (2)	2443 (5)	2·5
C(02)	8702 (2)	6340 (2)	4868 (5)	2·7
C(03)	9602 (2)	6148 (2)	4827 (5)	2·8
C(04)	9676 (2)	5761 (2)	2441 (6)	3·0
C(05)	8860 (2)	5747 (2)	953 (5)	2·7
C(06)	6280 (2)	3729 (2)	2960 (5)	2·6
C(07)	6735 (2)	3850 (2)	5327 (5)	2·8
C(08)	7406 (3)	3364 (2)	5157 (6)	3·1
C(09)	7360 (3)	2928 (2)	2691 (6)	3·3
C(010)	6655 (2)	3144 (2)	1355 (5)	2·8
C(011)	7146 (3)	5916 (2)	1532 (5)	2·9
C(012)	6208 (3)	5451 (3)	3008 (6)	3·3
C(013)	5607 (3)	4220 (3)	2299 (6)	3·5
(II)				
Ru	93681 (3)	80065 (1)	59414 (1)	2·1
C(1)	11281 (5)	6468 (2)	6374 (1)	2·9
C(2)	12184 (5)	6885 (2)	5800 (2)	2·7
C(3)	10345 (4)	6842 (2)	5159 (1)	2·5
C(4)	8303 (4)	6386 (2)	5315 (1)	2·7
C(5)	8837 (5)	6137 (2)	6041 (2)	3·0
C(6)	10010 (5)	9049 (2)	6888 (1)	3·0
C(7)	10769 (5)	9704 (2)	6369 (2)	3·2
C(8)	8786 (5)	9927 (2)	5767 (2)	3·5
C(9)	6792 (5)	9417 (3)	5922 (2)	3·6
C(10)	7533 (5)	8889 (2)	6604 (2)	3·3
C(11)	12557 (5)	6459 (3)	7153 (2)	3·8
C(12)	11469 (7)	7183 (3)	7639 (2)	4·2
C(13)	11576 (6)	8531 (3)	7566 (2)	4·3
C(14)	9942 (6)	6988 (2)	4370 (2)	3·3
C(15)	7239 (7)	6984 (3)	4105 (2)	4·5
C(16)	6370 (5)	6221 (3)	4628 (2)	3·8

Fig. 1. *ORTEP* drawings (Johnson, 1965) of (I) and (II) with 50% probability ellipsoids. H atoms are represented by circles of radius 0·08 Å. Projections of the molecules on the Cp ring containing the C(1) atom are also given, where open circles denote Ru atoms.

Table 3. *Bond lengths* (Å) *and bond angles* (°) *and non-bonded distances between the Cp rings* (Å)

(I)

Ru(1)–C(1)	2·122 (2)	Ru(2)–C(01)	2·120 (3)
Ru(1)–C(2)	2·126 (3)	Ru(2)–C(02)	2·127 (3)
Ru(1)–C(3)	2·195 (5)	Ru(2)–C(03)	2·202 (2)
Ru(1)–C(4)	2·216 (5)	Ru(2)–C(04)	2·215 (3)
Ru(1)–C(5)	2·185 (4)	Ru(2)–C(05)	2·189 (3)
Ru(1)–C(6)	2·134 (3)	Ru(2)–C(06)	2·141 (2)
Ru(1)–C(7)	2·156 (4)	Ru(2)–C(07)	2·155 (3)
Ru(1)–C(8)	2·198 (3)	Ru(2)–C(08)	2·188 (3)
Ru(1)–C(9)	2·208 (2)	Ru(2)–C(09)	2·200 (3)
Ru(1)–C(10)	2·171 (4)	Ru(2)–C(010)	2·180 (2)
O(1)–C(11)	1·217 (5)	O(2)–C(011)	1·204 (3)
C(1)–C(2)	1·448 (5)	C(01)–C(02)	1·433 (4)
C(1)–C(5)	1·424 (5)	C(01)–C(05)	1·439 (4)
C(1)–C(11)	1·486 (7)	C(01)–C(011)	1·482 (5)
C(2)–C(3)	1·401 (6)	C(02)–C(03)	1·421 (5)
C(3)–C(4)	1·426 (6)	C(03)–C(04)	1·409 (4)
C(4)–C(5)	1·409 (6)	C(04)–C(05)	1·405 (5)
C(6)–C(7)	1·433 (5)	C(06)–C(07)	1·435 (4)
C(6)–C(10)	1·425 (4)	C(06)–C(010)	1·421 (4)
C(6)–C(13)	1·511 (5)	C(06)–C(013)	1·517 (6)
C(7)–C(8)	1·416 (5)	C(07)–C(08)	1·424 (6)
C(8)–C(9)	1·423 (5)	C(08)–C(09)	1·428 (5)
C(9)–C(10)	1·426 (6)	C(09)–C(010)	1·427 (5)
C(11)–C(12)	1·510 (6)	C(011)–C(012)	1·526 (5)
C(12)–C(13)	1·531 (5)	C(012)–C(013)	1·533 (5)
C(2)–C(1)–C(5)	106·7 (3)	C(02)–C(01)–C(05)	107·3 (3)
C(2)–C(1)–C(11)	128·0 (4)	C(02)–C(01)–C(011)	128·5 (3)
C(5)–C(1)–C(11)	122·9 (3)	C(05)–C(01)–C(011)	122·2 (3)
C(1)–C(2)–C(3)	108·6 (3)	C(01)–C(02)–C(03)	107·3 (3)
C(2)–C(3)–C(4)	107·7 (4)	C(02)–C(03)–C(04)	108·8 (3)
C(3)–C(4)–C(5)	108·7 (4)	C(03)–C(04)–C(05)	108·4 (3)
C(1)–C(5)–C(4)	108·3 (3)	C(01)–C(05)–C(04)	108·1 (3)
C(7)–C(6)–C(10)	106·8 (3)	C(07)–C(06)–C(010)	107·2 (3)
C(7)–C(6)–C(13)	126·2 (3)	C(07)–C(06)–C(013)	126·0 (3)
C(10)–C(6)–C(13)	126·9 (3)	C(010)–C(06)–C(013)	126·6 (3)
C(6)–C(7)–C(8)	108·5 (3)	C(06)–C(07)–C(08)	108·4 (3)
C(7)–C(8)–C(9)	108·5 (3)	C(07)–C(08)–C(09)	107·9 (3)
C(8)–C(9)–C(10)	107·3 (3)	C(08)–C(09)–C(010)	107·7 (3)
C(6)–C(10)–C(9)	109·0 (3)	C(06)–C(010)–C(09)	108·8 (3)
O(1)–C(11)–C(1)	120·2 (4)	O(2)–C(011)–C(01)	121·5 (3)
O(1)–C(11)–C(12)	120·9 (4)	O(2)–C(011)–C(012)	120·3 (4)
C(1)–C(11)–C(12)	118·6 (3)	C(01)–C(011)–C(012)	117·9 (3)
C(11)–C(12)–C(13)	110·6 (3)	C(011)–C(012)–C(013)	110·2 (3)
C(6)–C(13)–C(12)	112·9 (4)	C(06)–C(013)–C(012)	114·5 (3)
C(1)···C(6)	3·292 (4)	C(01)···C(06)	3·291 (3)
C(2)···C(7)	3·399 (4)	C(02)···C(07)	3·410 (3)
C(3)···C(8)	3·759 (6)	C(03)···C(08)	3·781 (4)
C(4)···C(9)	3·888 (5)	C(04)···C(09)	3·869 (4)
C(5)···C(10)	3·632 (5)	C(05)···C(010)	3·619 (3)

(II)

Ru–C(1)	2·118 (2)	C(3)–C(4)	1·420 (3)
Ru–C(2)	2·169 (3)	C(3)–C(14)	1·499 (4)
Ru–C(3)	2·208 (2)	C(4)–C(5)	1·391 (4)
Ru–C(4)	2·194 (2)	C(4)–C(16)	1·513 (4)
Ru–C(5)	2·152 (2)	C(6)–C(7)	1·422 (4)
Ru–C(6)	2·133 (2)	C(6)–C(10)	1·426 (4)
Ru–C(7)	2·163 (2)	C(6)–C(13)	1·507 (4)
Ru–C(8)	2·209 (2)	C(7)–C(8)	1·434 (4)
Ru–C(9)	2·197 (3)	C(8)–C(9)	1·417 (5)
Ru–C(10)	2·147 (4)	C(9)–C(10)	1·412 (5)
C(1)–C(2)	1·445 (5)	C(11)–C(12)	1·522 (6)
C(1)–C(5)	1·459 (4)	C(12)–C(13)	1·533 (5)
C(1)–C(11)	1·497 (4)	C(14)–C(15)	1·537 (5)
C(2)–C(3)	1·413 (4)	C(15)–C(16)	1·528 (6)
C(2)–C(1)–C(5)	105·7 (2)	C(7)–C(6)–C(13)	126·0 (3)
C(2)–C(1)–C(11)	126·7 (3)	C(10)–C(6)–C(13)	127·4 (2)
C(5)–C(1)–C(11)	127·5 (3)	C(6)–C(7)–C(8)	109·1 (2)
C(1)–C(2)–C(3)	108·4 (2)	C(7)–C(8)–C(9)	107·1 (3)
C(2)–C(3)–C(4)	108·4 (2)	C(8)–C(9)–C(10)	108·2 (3)
C(2)–C(3)–C(14)	140·3 (3)	C(6)–C(10)–C(9)	109·3 (3)
C(4)–C(3)–C(14)	110·8 (2)	C(1)–C(11)–C(12)	116·5 (3)
C(3)–C(4)–C(5)	108·9 (2)	C(11)–C(12)–C(13)	116·2 (3)
C(3)–C(4)–C(16)	109·5 (2)	C(6)–C(13)–C(12)	115·9 (3)
C(5)–C(4)–C(16)	141·2 (3)	C(3)–C(14)–C(15)	101·6 (3)
C(1)–C(5)–C(4)	108·6 (3)	C(14)–C(15)–C(16)	106·7 (3)
C(7)–C(6)–C(10)	106·3 (2)	C(4)–C(16)–C(15)	102·1 (2)
C(1)···C(6)	3·238 (3)	C(3)···C(8)	3·874 (4)
C(2)···C(7)	3·548 (4)	C(4)···C(9)	3·809 (4)
		C(5)···C(10)	3·454 (4)

The π orbital of the carbonyl group in (I) seems to be able to conjugate with the π system of the Cp ring a little more effectively than in (III) and the electrons of the carbonyl carbon in (I) are more delocalized, providing a higher ^{13}C NMR chemical shift than in (III) (Stothers, 1972). Bond distances of the carbonyl carbon atom to the adjacent Cp ring in (I) are 1·486 (7) and 1·482 (5) Å in agreement with that in (III), 1·493 (10) Å, within the experimental error. The C(1)—C(11) bond length of (I) is significantly shorter than C(11)—C(12) and C(6)—C(13), suggesting the smaller atomic radius of C sp^2 rather than C sp^3. The molecular structure of (I) is distorted by the rigidity of the carbon chain owing to the attached carbonyl group. The distance of C(11) from the plane through the adjacent Cp ring is 0·368 (7) Å, much larger than that of C(13), 0·108 (7) Å. The C(5)—C(1)—C(11) angle is ca 5° smaller than C(2)—C(1)—C(11). The Cp rings are skewed to one side. The C(2)···C(7) interatomic distance is shorter than C(5)···C(10) by 0·209, 0·233 (7) Å. The corresponding value for (IV) is 0·066 (6) Å. These distortions were also observed for (III).

The five-membered ring C(3), C(4), C(16), C(15) and C(14) in (II) has an envelope conformation, the C(14), C(15) and C(16) atoms being shifted from the Cp plane by −0·145 (5), 0·259 (6) and −0·115 (5) Å, respectively. The C—C bond lengths next to the Cp ring in this homoannularly bridged carbon chain are significantly shorter than the other C—C bonds in the bridge as was observed in the heteroannular bridge. The geometry of the 1,3-(1,1'-ruthenocenediyl)propane moiety of (II) is essentially the same as that of (IV).

The crystal structures consist of discrete molecules separated by normal van der Waals distances. The shortest intermolecular distances are 3·451 (4) Å for C(9)···C(05) ($-1+x$, y, z) and 3·591 (4) Å for C(8)···C(14) ($-x$, $-y$, $1-z$) for (I) and (II), respectively.

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Structure d'un Complexe du Nickel(II) avec l'Aminoacétohydroxamate Monosodique, Na[Ni(C₂H₄N₂O₂)(C₂H₅N₂O₂)].3H₂O

$Na[Ni(C_2H_4N_2O_2)(C_2H_5N_2O_2)].3H_2O$

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Abstract. $M_r = 312.88$, monoclinic, $P2_1/b$, $a = 7.172$ (4), $b = 13.166$ (5), $c = 11.865$ (3) Å, $\gamma = 92.30$ (4)°, $V = 1119$ (1) Å^3, $Z = 4$, $D_m(293 \text{ K}) = 1.87$ (1), $D_x = 1.856$ Mg m^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0.71069$ Å, $\mu(\text{Mo } K\alpha) = 1.766$ mm^{-1}, $F(000) = 744$, $T = 293$ K, $R = 0.056$ for 2570 unique reflexions. The