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CYCLOPENTADIENYL-RUTHENIUM AND -OSMIUM COMPLEXES

V *. SYNTHESIS, REACTIVITY AND CRYSTAL STRUCTURE DETERMINATION OF CARBONYLCHLORO(η-CYCLOPENTADIENYL)-(TRIPHENYLPHOSPHINE)RUTHENIUM(II)

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Summary

$CpRuCl(CO)PPh_3$ is formed as the result of refluxing $CpRuCl(PPh_3)_2$ in ethylene glycol (yield up to 15%). A dissociation process is postulated with liberation of one PPh_3 molecule and simultaneous rearrangement of the cation formed earlier: $[CpRu(CO)(PPh_3)_2]^+Cl^- \rightarrow CpRuCl(CO)PPh_3 + PPh_3$. $CpRuCl(CO)PPh_3$ reacts reluctantly with the alkoxy anion to give $CpRuH(CO)PPh_3$, in contrast to $CpRuCl(PPh_3)_2$, which undergoes very facile transformation into $CpRuH(PPh_3)_2$.

The structure of $CpRuCl(CO)PPh_3$ has been determined by the single-crystal X-ray diffraction method. The compound is triclinic, space group $P\bar{1}$, a 9.378(2), b 10.584(2), c 16.590(4) Å, α 126.11(1), β 55.91(1), γ 101.49(1)°. The unit cell contains both R and S enantiomers. A shorter distance of the Ru–Cl bond has been noted in $CpRuCl(CO)PPh_3$ (2.396 Å) in comparison with the Ru–Cl distance in $CpRuCl(PPh_3)_2$ (2.453 Å). This causes a diminishing tendency to lose a chloride ion and as a result, nucleophilic attack of RO^- on $CpRuCl(CO)PPh_3$.

Introduction

The compound $CpRuCl(CO)PPh_3$ was obtained by Blackmore et al. in 1971 [1] by replacing one of the triphenylphosphine ligands in $CpRuCl(PPh_3)_2$ with carbon monoxide by carbonylating under pressure in benzene, or alternatively, in the reaction between tetrahydrofuran solutions of $CpRuCl(PPh_3)_2$ and enneacarbonyldi-iron $Fe_2(CO)_9$ at room temperature, yield 86%, m.p. 135–137°C.

* For part IV see ref. 18.

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$CpRuCl(CO)PPh_3$ can also be obtained in the reaction of $CpRuH(CO)PPh_3$ with chlorinated solvents, e.g. after stirring a sample in chloroform solution overnight [2].

As a result of refluxing $CpRuCl(PPh_3)_2$ in ethylene glycol, $CpRuCl(CO)PPh_3$ is also formed in a yield of up to about 15% (formed within the range 2–90 h of reflux), m.p. 220–222°C [3]. The melting temperature of $CpRuCl(CO)PPh_3$ obtained in this way rises even to 230–235°C after purification by chromatography.

Davies and Simpson [4] prepared $CpRuCl(CO)PPh_3$ in near-quantitative yields by heating a toluene or xylene solution of $CpRuCl(PPh_3)_2$ with sulphur in the presence of CO.

Considering the (η-C_5H_5) (Cp) group as occupying one coordination position, the compound $CpRuCl(CO)PPh_3$ has a pseudo-tetrahedral structure, in which the ruthenium atom is a chiral centre. Attempts to separate this type of compound into enantiomers, either by chromatography or during crystallization, were not carried out [5].

Such a large difference between the melting point of compound $CpRuCl(CO)PPh_3$ cited in the literature [1] and our value may be due to the possible formation of polymorphic compounds.

Experimental

The procedure adopted was similar to that given in [3]. 0.2757 g of $CpRuCl(PPh_3)_2$ and 25 cm^3 of ethylene glycol were refluxed for 22 h under nitrogen, by the use of a short reflux condenser. Next a colourless liquid (ca. 0.4 cm^3, water, phosphoroorganic compounds) was distilled off up to 150–160°C. Ruthenocene had not been found in the distillate. The obtained glycol phase was extracted with benzene (50 and 25 cm^3). The combined benzene extracts were evaporated and separated on a column (silica gel, benzene). The first colourless fraction (22 cm^3 of benzene) contained ruthenocene (yield 1.5%) and $CpRuH(CO)PPh_3$ (yield 2%).

The second lemon-yellow fraction (75 cm^3 of benzene) contained $CpRuCl(CO)PPh_3$. From this fraction, after evaporation $CpRuCl(CO)PPh_3$ crystallized as long lemon-yellow crystals (0.0680 g). They were washed with ethyl ether (3 cm^3) and dried. 0.0246 g of $CpRuCl(CO)PPh_3$, m.p. 218–220°C, yield 13%, was obtained.

From this mixture, crystals suitable for X-ray technique purposes were selected.

However, in the case of perturbation of the synthesis process of $CpRuCl(CO)PPh_3$ using a larger reflux condenser, absorbing a greater amount of condensates, the mixture in the reaction vessel darkened quickly. As a result, the yield of ruthenocene rose to 10% and the yield of $CpRuCl(CO)PPh_3$ decreased to 0%.

The identities of $CpRuCl(CO)PPh_3$ and the compound obtained by Blackmore et al. [1] were confirmed by comparison with authentic samples.

For the preparation of $CpRuCl(CO)PPh_3$, $Fe(CO)_5$ was used instead of $Fe_2(CO)_9$ and the mixture was exposed to a mercury-vapour lamp. Thus, 0.5106 g of $CpRuCl(PPh_3)_2$, 100 cm^3 of tetrahydrofuran and 1 cm^3 of $Fe(CO)_5$, with stirring, were exposed to UV light, with interruptions, for a total of 1 h, during 10 days' storage at room temperature.

Following the procedure of [1], 0.3590 g of the preparate was obtained. It was purified by chromatography (silica gel, chloroform) and the yellow chloroform effluent was evaporated. 25 cm^3 of hexane was then added, and the mixture was

pulverized and left to stand overnight. Next the n-hexane layer was decanted and the residue was collected and dried. 0.3170 g of $CpRuCl(CO)PPh_3$, m.p. 221–223°C, yield 91%, was obtained.

It was possible to obtain $CpRuH(CO)PPh_3$ from $CpRuCl(CO)PPh_3$ by prolonged action of MeONa. To a solution of 3 cm^3 of MeOH and 0.0375 g of metallic sodium (1.6 mmol) (and after obtaining MeONa), 0.0630 g of $CpRuCl(CO)PPh_3$ (0.13 mmol) was added. The mixture was heated at 60°C (stirring with interruptions) for 13 h. White-yellow needle-shaped crystals were obtained, which after filtration and washing were dried under vacuum. 0.0086 g of $CpRuH(CO)PPh_3$, m.p. 168–176°C (sealed capillaries), yield 14%, was obtained. IR (KBr) ν(Ru–H) 1972 m, ν(CO) 1934vs. TLC (Silufol) R_F 0.80 (benzene).

Determination of the crystal structure of $CpRuCl(CO)PPh_3$

Preliminary Weissenberg photographs showed the crystals to be triclinic. A crystal of dimensions $0.16 \times 0.25 \times 0.30$ mm was used for data collection on a Hilger and Watts Y290 computer-controlled four-circle diffractometer. Accurately measured θ-values for 16 reflexions gave the following cell dimensions from a least-squares fit: a 9.378(2), b 10.584(2), c 16.590(4) Å, α 126.11(1), β 55.91(1), γ 101.49(1)°, space group $P\bar{1}$, V 1080.4 $Å^3$, $Z = 2$ for $C_{24}H_{20}OPClRu$, MW 491.92, $F(000) = 496$, D_x 1.512 g cm^{-3}, λ(Cu-K_α) 1.5418 Å, μ 80.0 cm^{-1}.

Integrated intensities were collected up to θ_{max} 51° using the ω-2θ scan technique. Three standard intensities were monitored every 50 reflexions, but no significant variation was observed. 3445 unique reflexions were measured, of which 597 with $I < 2\sigma(I)$ were treated as unobserved in subsequent calculations. The intensities were corrected for Lorentz and polarization factors and an empirical absorption correction [6] was applied, based on the ψ-dependence of the intensity of the axial 002 reflexion ($I_{max}/I_{min} = 1/0.4$).

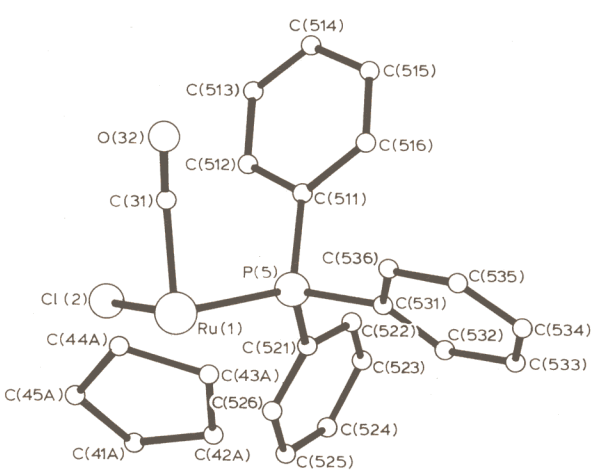


Fig. 1. The molecule of $CpRuCl(CO)PPh_3$ with the numbering scheme. Only one of the two positions of the Cp-ring is shown.

The structure was solved by direct methods, using the MULTAN-80 program [7]. On the E-map, the fragment consisting of ruthenium, chlorine, carbonyl and phosphorus atoms with parts of phenyl groups was visible. The first difference Fourier synthesis revealed all the missing atoms; however, some additional peaks were present in the vicinity of the five-membered ring. After few cycles of isotropic refinement [8], another difference Fourier map was calculated, from which it was evident that the cyclopentadienyl ring was disordered and had two possible orientations, differing by $\pi/5$. In subsequent anisotropic full-matrix refinement, both orientations of the cyclopentadienyl ring were included and their occupancy factors were refined.

TABLE 1

ATOMIC COORDINATES AND EQUIVALENT ISOTROPIC TEMPERATURE FACTORS ($\times 10^4$; e.s.d.s. in parentheses) FOR THE NON-HYDROGEN ATOMS OF $CpRuCl(CO)PPh_3$

Atom	x/a	y/b	z/c	U_{eq} [a]
Ru(1)	6071(1)	3102(1)	6597(1)	298
Cl(2)	3563(5)	3255(5)	6591(3)	457
C(31)	4743(17)	3005(15)	7943(13)	300
O(32)	4017(16)	3017(14)	8701(12)	506
C(41A)	7835(32)	1939(33)	4606(21)	257
C(42A)	8824(30)	2819(25)	5154(22)	240
C(43A)	8884(29)	2222(31)	5741(20)	245
C(44A)	7977(33)	864(29)	5526(24)	272
C(45A)	7478(34)	656(29)	4761(24)	296
C(41B)	8363(65)	2490(52)	4800(42)	140
C(42B)	9067(49)	2737(42)	5471(38)	97
C(43B)	8611(53)	1499(52)	5677(33)	95
C(44B)	7691(47)	520(39)	5091(37)	81
C(45B)	7527(58)	1088(58)	4520(39)	132
P(5)	5552(4)	5849(4)	7903(3)	246
C(511)	3649(14)	6697(12)	9404(9)	223
C(512)	2029(16)	6419(16)	9608(11)	379
C(513)	533(17)	7013(20)	10765(13)	437
C(514)	646(22)	7880(19)	11730(14)	471
C(515)	2223(20)	8247(21)	11580(10)	517
C(516)	3714(19)	7639(18)	10450(13)	395
C(521)	5234(16)	6968(14)	7486(9)	291
C(522)	4361(20)	8506(18)	8318(15)	400
C(523)	4062(24)	9407(21)	8049(19)	472
C(524)	4806(30)	8595(29)	6798(23)	613
C(525)	5706(31)	7093(27)	5976(20)	635
C(526)	5772(24)	6311(21)	6354(15)	494
C(531)	7312(15)	6578(16)	8118(11)	313
C(532)	8080(17)	7783(17)	8015(12)	368
C(533)	9488(18)	8233(23)	8159(14)	473
C(534)	10081(22)	7459(30)	8390(20)	623
C(535)	9354(26)	6244(26)	8454(21)	575
C(536)	7960(20)	5836(18)	8359(15)	389

[a] $U_{eq} = 1/3(U_{11} + U_{22} + U_{33} + 2U_{23}\cos\alpha + 2U_{13}\cos\beta + 2U_{12}\cos\gamma)$.

TABLE 2

SELECTED BOND DISTANCES (Å) AND ANGLES (°)

	Ru(1)–Cl(2)	2.396(6)	Cl(2)–Ru(1)–C(31)	93.4(0.6)
	Ru(1)–C(31)	1.911(20)	Ru(1)–C(31)–O(32)	176.9(1.2)
	Ru(1)–P(5)	2.311(3)	C(511)–P(5)–Ru(1)	114.7(0.4)
	C(31)–O(32)	1.034(27)	C(521)–P(5)–Ru(1)	116.5(0.4)
	P(5)–C(511)	1.793(10)	C(531)–P(5)–Ru(1)	113.1(0.5)
	P(5)–C(521)	1.819(21)	C(521)–P(5)–C(531)	104.1(0.9)
	P(5)–C(531)	1.809(16)	C(531)–P(5)–C(511)	103.5(0.7)
Ph(1)	C(511)–C(512)	1.369(20)	C(511)–C(512)–C(513)	121.8(1.6)
	C(512)–C(513)	1.400(17)	C(512)–C(513)–C(514)	120.7(1.5)
	C(513)–C(514)	1.355(30)	C(513)–C(514)–C(515)	119.6(1.4)
	C(514)–C(515)	1.368(28)	C(514)–C(515)–C(516)	120.5(1.7)
	C(515)–C(516)	1.372(18)	C(515)–C(516)–C(511)	121.3(1.6)
	C(516)–C(511)	1.436(26)	C(516)–C(511)–C(512)	115.9(1.1)
Ph(2)	C(521)–C(522)	1.357(19)	C(521)–C(522)–C(523)	123.9(2.0)
	C(522)–C(523)	1.391(46)	C(522)–C(523)–C(524)	114.4(2.0)
	C(523)–C(524)	1.430(42)	C(523)–C(524)–C(525)	121.6(3.9)
	C(524)–C(525)	1.338(32)	C(524)–C(525)–C(526)	118.4(2.9)
	C(525)–C(526)	1.325(52)	C(525)–C(526)–C(521)	125.2(1.7)
	C(526)–C(521)	1.343(28)	C(526)–C(521)–C(522)	115.9(2.2)
Ph(3)	C(531)–C(532)	1.389(25)	C(531)–C(532)–C(533)	119.0(2.3)
	C(532)–C(533)	1.413(27)	C(532)–C(533)–C(534)	119.6(2.1)
	C(533)–C(534)	1.384(52)	C(533)–C(534)–C(535)	121.1(2.6)
	C(534)–C(535)	1.364(42)	C(534)–C(535)–C(536)	120.0(3.5)
	C(535)–C(536)	1.359(36)	C(535)–C(536)–C(531)	121.1(2.2)
	C(536)–C(531)	1.407(38)	C(536)–C(531)–C(532)	119.1(1.6)

TABLE 3

BOND DISTANCES (Å) AND ANGLES (°) FOR THE Ru-Cp PART

Cp(A)	C(41A)–C(42A)	1.469(47)	C(41A)–C(42A)–C(43A)	111.7(2.6)
	C(42A)–C(43A)	1.467(61)	C(42A)–C(43A)–C(44A)	107.2(3.1)
	C(43A)–C(44A)	1.472(39)	C(43A)–C(44A)–C(45A)	105.3(3.3)
	C(44A)–C(45A)	1.475(66)	C(44A)–C(45A)–C(41A)	112.4(2.6)
	C(45A)–C(41A)	1.469(55)	C(45A)–C(41A)–C(42A)	102.8(3.6)
Cp(B)	C(41B)–C(42B)	1.468(110)	C(41B)–C(42B)–C(43B)	106.6(4.2)
	C(42B)–C(43B)	1.469(85)	C(42B)–C(43B)–C(44B)	106.4(5.7)
	C(43B)–C(44B)	1.469(73)	C(43B)–C(44B)–C(45B)	112.0(4.4)
	C(44B)–C(45B)	1.468(110)	C(44B)–C(45B)–C(41B)	103.1(5.6)
	C(45B)–C(41B)	1.471(72)	C(45B)–C(41B)–C(42B)	111.8(6.3)
Cp(A)	Ru(1)–C(41A)	2.249(26)	C(41A)–Ru(1)–C(42A)	
	Ru(1)–C(42A)	2.241(22)	and so on,	
	Ru(1)–C(43A)	2.250(28)	37.7–38.1°	
	Ru(1)–C(44A)	2.265(31)		
	Ru(1)–C(45A)	2.298(20)		
Cp(B)	Ru(1)–C(41B)	2.241(50)	C(41B)–Ru(1)–C(42B)	
	Ru(1)–C(42B)	2.287(37)	and so on,	
	Ru(1)–C(43B)	2.316(54)	36.7–37.8°	
	Ru(1)–C(44B)	2.317(30)		
	Ru(1)–C(45B)	2.347(40)		

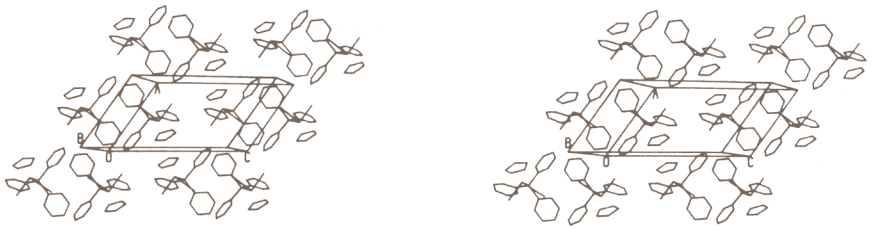


Fig. 2. Packing of the molecules of $CpRuCl(CO)PPh_3$ in the triclinic unit cell.

In general, the atoms within the five-membered ring behaved poorly in the course of refinement and their bond distances were constrained and refined as one of the parameters. Attempts to refine the structure in the non-centrosymmetric space group $P1$ gave no better results: neither the R-factor decreased nor the electron density around the five-membered ring was clearer. The positions of the hydrogen atoms within the phenyl groups were calculated at distances 1.08 Å from their parent carbon atoms: cyclopentadienyl hydrogens were not included at all.

The final R value was 0.1081. The final positional parameters and selected bond lengths and angles are given in Tables 1–3. The numbering scheme and structure are shown in Figs. 1 and 2. The final list of the calculated structure factors can be obtained from the author (Z.D.).

Results and discussion

The compound $CpRuCl(CO)PPh_3$ is formed as a result of dissociation of the complex cation, which is formed at shorter reflux times; one molecule of PPh_3 leaves

$$[CpRu^+(CO)(PPh_3)_2]Cl^- \rightarrow CpRuCl(CO)PPh_3 + PPh_3$$

with simultaneously rearrangement of the ionic structure into a covalent one. In polar solvents (e.g. ethylene glycol) this process is hindered, thus the yield of $CpRuCl(CO)PPh_3$ is insignificant (10–15%) in comparison with the yield of $[CpRu^+(CO)(PPh_3)_2]$, isolated from the solution as a sparingly soluble tetraphenylborate (up to 60% yield [3]).

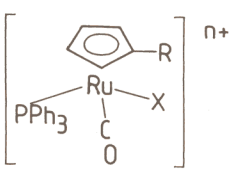
An essential difference between $CpRuCl(CO)PPh_3$ and $CpRuCl(PPh_3)_2$ occurs in the reactivity with the alkoxy anion in an adequate alcohol medium. In the case of $CpRuCl(PPh_3)_2$, the reaction takes place with formation of the alkoxy complex (with liberation of Cl^-) in which an intramolecular hydride shifts occurs, giving the hydride $CpRuH(PPh_3)_2$ (yield 90%) [9,10]. However, nucleophilic attack of the alkoxy anion on the compound $CpRuCl(CO)PPh_3$ is less effective and thus the yield of $CpRuH(CO)PPh_3$ is insigificant (14%). This results from the shorter Ru–Cl bond (2.396 Å; present work) in $CpRuCl(CO)PPh_3$ in comparison with the Ru–Cl distance (2.453 Å) in $CpRuCl(PPh_3)_2$ [11–13].

Comparison of the positions of the signals of Cp in the ^1H NMR spectra (4.84 s and 4.03 s for $CpRuCl(CO)PPh_3$ and $CpRuCl(PPh_3)_2$, respectively) and the analogous positions of the signals of the phosphorus atom in the ^1H-decoupled ^{31}P NMR spectra (48.4 s and 38.4 s, respectively, as above) indicates downfield shifts for $CpRuCl(CO)PPh_3$ as a result of a decrease of the electron density on the phos-

phorus atom. This leads to an increase in the covalent character of the Ru–Cl bond. This shows that the steric arguments are relatively unimportant in this case, because nucleophilic attack of the alkoxylate anion in the case of a less crowded molecule of $CpRuCl(CO)PPh_3$ might be more effective than for $CpRuCl(PPh_3)_2$.

The increase in the back-bonding proportion in the Ru–CO bonding manifests itself by a decrease of the order of the bonding $\bar{C}\equiv\overset{+}{O}$. This is usually connected with a decrease in the IR frequency ν(CO).

Comparison of the data (Table 4) of the ν(CO) frequency for compounds I and II with the related C–O bond lengths shows that ν(CO) decreases only slightly with a significant increase of the C–O bond distance from 1.034 to 1.149 Å for I and II, respectively). The increased proportion of back-bonding Ru–$\bar{C}\equiv\overset{+}{O}$ for II is also confirmed by the significant shortening of the Ru–C distances (from 1.911 to 1.838 Å for I and II, respectively). This is caused by the introduction of a menthyl group (σ-donor ligand) into the cyclopentadienyl ring (compound II). Its influence by Cp–Ru π-bonding increases the electron density on the ruthenium atom. This should lead to growth in the ionic character of Ru–Cl bonding for compound II, manifesting itself by an increase in the bond lengths. In fact, an increase of the Ru–Cl distances was observed, from 2.396 to 2.425 Å for I and II, respectively.



	R	X	n
I	H	Cl	0
II	menthyl	Cl	0
III	neomenthyl	NC-Me	1

Haines and DuPreez [16] stated that the share increase of π-back-donation from the ruthenium atom to the anti-bonding orbitals of the cyanide group for $CpRu(CN)(PPh_3)_2$ in comparison with $CpRu(CN)(CO)_2$ manifests itself by a marked diminution of the frequency of the C≡N stretching mode (2083 and 2125 cm^{-1} for $CpRu(CN)(PPh_3)_2$ and $CpRu(CN)(CO)_2$, respectively). This is due to the PPh_3 ligand being a more marked σ-donor than CO.

One can expect that for compound II the ionization connected with removal of the chloro group will be facilitated. It is known [17] that complexes of the type $(\eta\text{-}C_5Me_5)RuCl(CO)PPh_3$ ionize very readily in methanol.

A change in the electron density of the ruthenium atom (directly determining the back-bonding participation in Ru–CO) may also be caused by an electronic configuration change of molecule II into ionic III · PF_6^-. As a result of displacement of the chloro group in II (formally as Cl^-) by the NC-Me ligand, the ruthenium atom gains a formal positive charge. A decrease of the electron density on ruthenium also decreases the back-bonding proportion, and ν(CO) should increase; this in fact can be observed. Also, the Ru–C bond lengths increase markedly (from 1.838 to

TABLE 4

COMPARISON OF THE SELECTED VALUES OF BOND LENGTHS, ANGLES AND ν(CO) FREQUENCIES IN $CpRuCl(CO)PPh_3$ AND ITS DERIVATIVES

	Compound		
	I (this work)	II [14]	III [15]
Bond lengths (Å)			
Ru–Cl	2.396	2.425	–
Ru–P	2.311	2.310	2.324
Ru–C	1.911	1.838	1.870
C–O	1.034	1.149	1.139
Ru–C(1) (Cp)	2.249	2.245	2.262
Ru–C(2)	2.241	2.250	2.187
Ru–C(3)	2.250	2.207	2.180
Ru–C(4)	2.265	2.236	2.241
Ru–C(5)	2.298	2.249	2.251
Angles (°)			
Ru–C–O	176.9	177.0	175.9
Ru–P–C (Ph(1))	114.7	110.8	117.5
Ru–P–C (Ph(2))	116.5	119.8	114.8
Ru–P–C (Ph(3))	113.1	115.6	112.8
IR (ν(CO) (cm^{-1})	1958s (CS_2) [1]	1950br (pentane)	1990br ($CHCl_3$)
	1958s (Nujol) [4]		

1.870 Å for II and III, respectively). This is followed by a slight shortening of the C–O distances.

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