The Crystal and Molecular Structure of Tetra- μ -chloro-tetra- μ -ethyloxycarbene-di- μ_3 -hydroxo dodecacarbonyl-di-chloro-hexaruthenium(II)

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The crystal and molecular structure of tetra-µchloro-tetra-µ-ethyloxycarbene-di-µ3-hydroxo-dodecacarbonyl-di-chloro-hexaruthenium(II)-benzene, [Ru₃- $Cl_3(COC_2H_5)_2(OH)(CO)_6]_2 \cdot C_6H_6$, have been determined. The complex crystallizes as monoclinic crystals in space group $P 2_1/c$ with unit cell dimensions a = 10.71(2) Å, b = 10.41(2) Å, c = 21.92(3) Å, β = 113° 15' (10') and Z = 2. A three-dimensional X-ray analysis, based on 3101 observed reflections, converged to a R of 0.076. The crystal structure of the complex consists of centrosymmetric hexanuclear molecules. The three ruthenium atoms of the asymmetric unit are octahedrally coordinated. The octahedra share a common vertex occupied by a hydroxo group which bridges three ruthenium atoms. Significant differences in the Ru–Cl bond lengths are accounted by the "trans influence" of the carbene nature of the C_2H_5CO groups.

Introduction

 η -Allyl ruthenium complexes of formula RuX(C₃-H₅)(CO)₃ (X = Cl, Br) have been used as catalysts in several reactions involving unsaturated substrates [1, 2]. The reactivity with different olefinic and acetylenic ligands was also widely investigated [3].

In the course of these studies some new ruthenium complexes, obtained in crystalline and amorphous forms, by reaction of $RuCl(C_3H_5)(CO)_3$ (I) with ethylene were isolated [2].

Since the analytical and spectroscopic data (IR and ¹H n.m.r.) did not allow to establish exactly the nature of the products and to understand some IR and analytical differences observed for the crystalline and amorphous product, recourse was had to X-ray crystallography.

Experimental

Preparation

By reaction of I (1 g, 3.8 mmol) with ethylene under pressure (50-60 atm) at 70-80 °C for 10 h in benzene solution and by standing the reaction mixture in the autoclave at room temperature for several days, a crystalline compound (II), initially formulated as $[RuCl(COC_2H_5)(CO)_2]_n$ with solvate benzene, was obtained [Found: C, 24.57; H, 2.17; Cl, 14.3; Ru, 40.2; M(benzene, Mechrolab Osmometer), 1600. Calc. for C₃₀H₂₈Cl₆O₁₈Ru₆ (on the basis of the formula determined by X-ray structural data): C, 24.09; H, 1.88; Cl, 14.22; Ru, 40.55%; M, 1495.7]. The complex could also be isolated as an amorphous product (III) either by adding n-pentane to the benzene solution or by evaporating to dryness [Found: C, 23.82; H, 2.18; Cl, 14.6; Ru, 39.2. C₅H₅-ClO₃Ru requires C, 24.05; H, 2.02; Cl, 14.21; Ru, 40.4%].

IR and ¹H n.m.r. Spectra

The IR spectra (Perkin-Elmer 225 spectrometer, in KBr) of the crystalline and amorphous products showed three bands in the carbonyl stretching region at 2130 vw, 2060vs, 2005vs cm⁻¹ and the bands assignable to a bridging acyl group (1543s cm⁻¹) [4] and to the terminal (329w cm⁻¹) and bridging chlorine atoms (290w and 260w cm⁻¹) [5]. The IR spectrum of the crystalline product differed from that of the amorphous one for a weak but sharp absorption at 3280 cm⁻¹ [ν (OH)], by a more complex pattern of bands in the Ru-C-O deformation and Ru-C stretching region (650-400 cm⁻¹) and by the bands due to solvate benzene.

The ¹H n.m.r. spectrum of the crystalline product (Varian T 60 spectrometer, in benzene solution) showed a quartet and a triplet at 7.32 and 9.15 τ

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	x	•	7			SC 1	71 4	P13	57
Ru(1)	-0.08384(10)	-0.20176(11)	0.40900(5)	81.7(10)	23.3(15)	20.6(2)	5.1(8)	17.6(4)	-3.4(4)
Ru(2)	0.06520(10)	0.02238(12)	0.34289(5)	75.0(9)	40.2(15)	19.1(2)	0.8(8)	17.1(4)	0.0(4)
Ru(3)	-0.19243(10)	0.15187(12)	0.37316(5)	70.4(9)	36.0(15)	20.4(2)	1.9(8)	14.1(4)	-1.1(4)
CI(1)	0.13394(34)	-0.20980(38)	0.50463(16)	88.4(32)	44.7(51)	20.5(8)	9.8(28)	16.0(13)	-1.6(14)
CI(2)	0.03363(37)	-0.21990(39)	0.33623(18)	108.0(36)	33.5(50)	25.1(9)	3.4(30)	28.4(15)	-5.8(15)
C1(3)	-0.19267(36)	-0.15407(42)	0.48031(17)	99.0(35)	58.1(53)	23.3(8)	-4.0(32)	25.7(14)	-1.6(15)
C(1)	-0.1038(16)	-0.3769(18)	0.4170(8)	107(16)	75(26)	24(4)	-38(15)	16(6)	1(7)
C(2)	-0.2436(15)	-0.2023(16)	0.3344(8)	93(14)	47(21)	26(4)	-23(12)	27(6)	-10(6)
C(3)	0.1465(15)	0.0427(18)	0.2829(8)	90(15)	92(25)	23(4)	9(14)	14(6)	-3(7)
C(4)	0.2430(14)	0.0003(17)	0.4077(7)	84(14)	83(23)	24(4)	-7(13)	22(6)	-4(7)
C(5)	-0.3455(17)	0.0640(17)	0.3741(8)	122(19)	41(22)	30(4)	11(15)	19(7)	-6(7)
C(6)	-0.3053(15)	0.2876(17)	0.3438(7)	99(15)	93(25)	20(3)	1(14)	21(6)	6(6)
C(7)	-0.2311(14)	0.0923(16)	0.2809(6)	92(13)	56(20)	14(3)	-8(12)	9(5)	1(5)
C(8)	0.0700(11)	0.2065(15)	0.3584(6)	52(11)	67(22)	23(3)	19(10)	14(5)	6(6)
C(9)	-0.3624(15)	0.1058(19)	0.2217(8)	77(14)	108(27)	22(4)	0(14)	-1(6)	7(7)
C(10)	-0.3629(18)	0.2253(19)	0.1834(9)	136(20)	65(25)	28(5)	17(17)	20(8)	15(8)
C(11)	0.1766(15)	0.3009(18)	0.3580(8)	83(14)	101(26)	23(4)	-15(14)	17(6)	-8(7)
C(12)	0.1649(21)	0.4437(20)	0.3825(11)	166(26)	24(24)	51(8)	15(18)	31(12)	-4(9)
C(13)	0.4235(26)	0.0358(32)	0.0363(13)	171(32)	291(55)	36(7)	73(34)	14(12)	1(15)
C(14)	0.4899(34)	0.1302(29)	0.0120(13)	286(48)	165(45)	39(8)	-18(36)	14(16)	-19(14)
C(15)	0.4390(32)	-0.0909(34)	0.0246(14)	254(46)	243(53)	40(9)	-73(39)	4(16)	13(16)
0(1)	-0.1189(14)	-0.4803(14)	0.4243(7)	157(17)	86(19)	38(4)	-32(13)	29(7)	7(7)
0(2)	-0.3461(12)	-0.2025(13)	0.2887(6)	102(13)	127(20)	27(3)	17(12)	-1(5)	-8(6)
0(3)	0.1997(12)	0.0459(14)	0.2474(6)	135(15)	148(20)	30(3)	18(13)	46(6)	9(9)
0(4)	0.3509(11)	-0.0135(13)	0.4438(6)	94(12)	130(20)	30(3)	25(12)	13(5)	8(6)
0(5)	-0.4450(11)	0.0221(13)	0.3705(7)	87(12)	107(19)	48(5)	-8(11)	35(6)	10(7)
0(6)	-0.3744(13)	0.3776(12)	0.3265(6)	151(16)	57(17)	32(3)	48(12)	17(6)	7(5)
0(1)	-0.1351(9)	0.0436(10)	0.2705(4)	82(9)	54(14)	14(2)	4(8)	8(3)	4(4)
0(8)	-0.0279(9)	0.2556(10)	0.3698(5)	78(9)	12(12)	28(3)	-8(8)	22(4)	1(4)
0(9)	-0.0431(9)	0.0020(10)	0.4069(4)	84(9)	44(13)	17(2)	7(8)	21(4)	4(4)

from TMS, in the proper intensity ratio for an ethyl group, and a resonance at 10.9 τ assignable to a triple bridging OH group [6]. This last resonance was absent in the spectrum of the amorphous product.

X-ray Crystallography

Unit-cell dimensions and space group for the crystals of the complex II were obtained from precession photographs taken with Mo K α radiation. The crystals belonged to the monoclinic system with a = 10.71(2) Å, b = 10.41(2) Å, c = 21.92(3) Å, $\beta = 113^{\circ}$ 15' (10'). Systematic absences (hol absent for 1 = 2n + 1 and 0k0 absent for k = 2n + 1) indicated space group P 2₁/c. For Z = 2 the calculated density was 2.45 g/cm³.

The intensity data were recorded with Ni-filtered Cu K α radiation ($\lambda = 1.5418$ Å) by means of Weissenberg photographs with multiple film technique and integration process. A crystal elongated in the *b* direction was reduced to nearly cylindrical shape with a radius of 0.013 cm (μ R = 2.70 for Cu K α radiation). Nine layers with b as rotation axis (k = 0 through 8) were taken.

A total of 4138 independent reflections (3101 observed) were collected. The intensities measured with a Nonius microdensitometer have been corrected for Lorentz and polarization effects, for the incipient but incomplete $\alpha_1 - \alpha_2$ spot doubling and for the absorption factor using the three-constant formula proposed by Palm [7] for cylindrical crystals.

Solution and Refinement of the Structure

The ruthenium atoms were located from threedimensional Patterson function. All chlorine, carbon, and oxygen atoms were located by successive Fourier syntheses. Various cycles of block-matrix least squares refinement lowered the value of $R = \Sigma ||F_o|$ $- |F_c||/\Sigma|F_o|$ to 0.092: unit weights were assumed for all observed reflections, isotropic temperature factors were attributed to carbon and oxygen atoms, while anisotropic temperature factors were attributed to ruthenium and chlorine atoms.

The anisotropic refinement was extended to all the atoms and the weighting scheme $\sqrt{w} = 1/(a + |F_o| + b|F_o|^2)$ proposed by Cruickshank [8] was introduced with a = 40.0 and b = 0.005.

Seventeen strong reflections, probably affected by extinction, were excluded from the last refinement cycles. The final R value was 0.076.

Scattering factors used in the structure factor calculations were taken from International Tables for X-ray Crystallography [9]. All the programs used are incorporated in the X-ray 70 system [10]. Positional and thermal parameters for the atoms in the asymmetric unit are given in Table I. Observed and calculated structure factors are compared in Table II.

Results and Discussion

The resolution of the structure of the crystalline product II allows to formulate this as tetra- μ -chlorotetra- μ -ethyloxycarbene-di- μ_3 -hydroxo-dodecacarbonyl-dichloro-hexaruthenium(II)-benzene, [Ru₃Cl₃-(COC₂H₅)₂(OH)(CO)₆]₂·C₆H₆.

The crystal structure of II is composed of two discrete hexanuclear molecules located at the inversion centers at (0, 0, 1/2) and (0, 1/2, 0) in the monoclinic unit cell. At another pair of centers at (1/2, 0, 0) and (1/2, 1/2, 1/2) are two molecules of benzene of crystallization.

This structure is closely analogous to that of a toluene-containing form of di- μ -aquo-di- μ ₃-hydroxo-deca(1,1,1-trifluoropentane-2,4-dionato)hexanickel-(II) [11] which also crystallizes in the space group P 2₁/c with two hexanuclear molecules at two inversion centers and two disordered molecules of toluene at another pair of centers. The resemblance goes further as both the complex molecules are composed of two trinuclear units, related by a center of symmetry and joined by two bridging water molecules in the nickel complex and by two bridging chlorine atoms in II. In both cases the three metal atoms in the asymmetric unit are each octahedrally coordinated and the three octahedra share a common vertex at the position of the hydroxo group.

Fig. 1 is an ORTEP [12] drawing of the molecule. Bond lengths and angles are given in Tables III and IV, together with their standard deviations.

As shown the molecular structure of II has a number of unusual features that seldom are present in a same molecule.

The first one is the presence of two bridging C₂H₅-CO groups between the Ru(2) and Ru(3) atoms; a similar situation was up to now found only in the crystal structure of bis-(µ-phenyloxycarbene-tricarbonyliron) [13]. In that case, however, the C atoms of the two bridging groups are both linked to the same iron atom, whereas the oxygen atoms are linked to the other iron atom. The C-O bond lengths of C₂H₅CO groups [C(7)-O(7): 1.246 Å; C(8)-O(8): 1.276 Å] longer than that of a keto group (1.23 Å) and analogous to those found in the "carbene compound" Fe₂(CO)₆[C(O)Ph]₂ [13] and the Ru-C distances [Ru(2)-C(8): 1.944 Å; Ru(3)-C(7): 1.996 A], longer than the ruthenium carbonyl lengths (1.89 Å) but shorter than those of a Ru–C (σ) bond (2.07 Å) [14], indicate for the bridging C_2H_5CO groups an arrangement of carbene type [13]. The carbene character of $C_2H_5C(O)$ groups, which display a high σ -donor and a low π -acceptor power, is also evidenced by the lengthening by trans effect [15, 16] of the Ru(2)-Cl(2) and Ru(3)-Cl(1)ⁱ bonds.

The C_2H_5 groups in II arise from the insertion of a C_2H_4 molecule into a ruthenium-hydrogen bond formed by β -elimination of pentadienes from the

TABLE II. Observed and Calculated Structure Factors ($\times 10^2$). The reflections marked with a star were excluded from the last refinement cycles.

0,0,1 12 1417 -1469 16 705 640	-12 1082 -1030	2 1516 -1721	-23 496 -421 -24 537 -520	8,1,L	-1 335 -2"A -4 215 -206	-11 368 37* -12 613 -658
4 2539#-3681 20 142 -148 6 1119 1095 -2 967 -975	-16 448 410	6 511 681 5 695 992	-27 268 223 -28 151 251	0 794 760 1 667 613	-5 537 539	-13 720 752 -14 521 -479
8 1737 1838 -4 1076 1453 12 1981 1066 -6 194 1180	-22 493 441	6 1662 1889 7 804 -775	5,1,1	3 869 -822 6 307 30 ⁶	-16 342 999	-15 277 -240
14 667 614 -8 395 -343 16 883 -840 -10 500 -432	-26 321 -448	6 1291 1365 9 811 815	0 823 -773	6 336 -305	-19 176 -147	-18 578 526
22 165 13 -12 145 +1172 24 506 440 -14 946 -953	11,0,L	11 608 533	2 968 975	12 519 553	-21 157 -195	-21 657 646
1,0,1	2 330 - 317	12 714 -638	# 857 851 5 1077 1112	18 286 299	13,1,L	-23 173 -153 -25 405 -390
0 3587# 4976 - 24 662 607 2 1704#+2208 - 26 466 - 465	-2 705 679 -4 851 -791	16 953 851 17 772 667	5 822 - P26 7 592 - 526	-2 748 710 -3 1003 -917	-2 115 162	-26 94 -90 -27 54 -90
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22 397 347 12 **12 +1137 -2 1734* 2302 14 715 659	· 12.0.L	-5 786 -773 -7 725 162	-2 1904 -2025	-14 936 -859 -16 792 -710	1 785=-1116	6 206 165 7 611 -589
-u 1677*-201" 16 683 556 -6 613 -662 19 217 -207	2 381 -449	-8 523	-4 595 -559 -5 775 -752	-20 284 236 -21 524 -472 -75 386 362	3 242 248	9 202 20E 9 529 543
-10 1277 1318 -2 749 -220 -12 2362 2396 -4 1447 1496 -14 355 315 -8 773 733	-4 363 -350 -6 861 -836	-11 1469 -1617	-7 433 370	-27 174 -193	5 818 901	13 416 -401
-16 671 -641 -10 *207 1203 -18 514 594 -12 1*07 -1028	-10 555 520	-13 91P -913	-9 503 398 -10 815 -769	9,1,L	7 933 9*1 8 523 -497	15 407 - 395 16 259 244
-22 342 -308 -14 1089 -1098 -24 691 517 -16 1917 1923	-14 100 -64 -16 319 -271	-15 1135 1131 -16 1111 1971	-12 697 -E75 -13 831 789	0 590 551	9 1225 -1326 10 1499 -156P	18 507 -435 21 145 -160
-26 307 330 -18 1065 987 -20 1250 -1159	-18 134 -131 -20 331 303	-10 757 -652	-14 356 357 -15 287 -253	a ⊴15 231 7 b€1 446 8 217 -194	15 558 562 16 816 .398	22 91 116 -1 320 -773
-24 278 322	13,0,L	-21 367 293	-19 70P 652	9 202 -207	17 812 - 77P 19 435 376	-2 425 529 -3 1609 -1653 -5 292 -281
2 500 -471 *,0,L 4 29023548	-2 146 208 -6 204 -202	-24 876 374	-21 681 656	11 191 -216 12 344 #8^	19 886 -870 20 159 153	-6 651 825 -7 659 665
6 480 436 ^ 1955 - 1932 8 1083 1079 2 173 174	-8 265 -259 -10 149 -139	-26 415 394	-24 504 -471 -26 321 -211	-1 308 240	21 212 30€ 22 562 - 510	-8 595 675 -10 E26 359
10 /67 -696 4 1720 1124 12 605 572 / 838 388 16 1362 -1252 10 592 -538	-14 324 319	1,1,L 0 1000 -1114	-28 125 179	-8 319 -250	24 223 214	-11 206 -210 -13 616 693
18 471 -445 12 148 -231 20 1053 973 14 472 439	-18 253 -276	1 1069 -1061	€,1,L	-10 653 534	1,2,1	-15 376 349 -17 574 -582
24 345 -435 16 211 233 -2 1069 1074 -2 275 258	0,1,L	3 163 -351 1 1929 2056	0 514 471 2 598 595	-12 1062 1019 -13 410 266	0 787 .494	-18 895 841 -19 331 -306
-6 622 595 -6 675 -556 -8 30704-5799 -10 1737 1835	2 153 137 3 1862+ 2670 8 982 - 1207	E 1683 1922	5 273 241	-16 789 -716	5 1567 16F5	-23 441 418
-10 267 250 -12 294 -406 -12 2487 2673 -14 1145 -1115	5 565 575 6 1565 - 1838	10 PR7 -922 11 31P 238	6 841 -928 9 512 -447	-20 385 334 -21 495 -463	6 324 7 ⁻⁵ 7 1017 -1049	-26 169 -174 -27 177 -240
-16 571 -071 -16 1245 1179 -18 894 840 -18 196 1146	7 1212 -1291 8 1180 1304	14 47P 330 16 4PO 375	9 431 -426 11 793 780	-22 237 210 -25 459 491	9 1248 -1728	4,2,L
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-28 C97 385	12 962 -961 13 822 -752	-1 075 983 -7 PS& 920	-1 516 5C2 -2 1655 -1722	0 525 501	13 989 95P 18 545 513	3 1699 -1792 5 1073 -1053
P,0,L	14 1574 + 1549 15 365 292	-3 244 207	-3 1306 -1310 -4 1960 -210P	1 271 199 2 372 326	15 623 -577 17 679 -63P	6 433 -405 7 667 661
2 925 - 892 ^ 1462 - 1437 4 2706 - 2936 2 140 674 8 571 571 4 1325 1365	17 312 234 18 861 742 19 439 -315	-6 1320 1314	-6 878 807	3 313 -285 4 299 -299 6 250 -272	20 269 245 22 894 -451 23 171 -105	9 264 262 9 616 637
12 376 - 353 - 512 - 499 14 457 - 430 - 8 742 - 758	20 244 168	-8 755 -311	-9 1034 1037 -9 121 -352	7 201 176	24 89 -95	14 734 753
16 903 -837 10 746 307 18 351 278 13 713 190	24 162 -115 25 191 -185	-12 433 -412	-11 761 -666 -12 991 -953	-5 5#2 536 -6 327 275	-3 329 300	18 647 -651 20 182 180
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-# 2255 2458 -6 242 -208 -6 858 696 -3 250 -309	0 405 411 1 1274 -1452	-16 587 497 -18 968 862	-17 1099 -1104	-12 1017 961	-9 190 -147	-3 1448 -1427
-8 4066+-5499 -10 551 926 -10 1198 -1309 -12 285 -243	2 997 -1100 3 1553 1730	-21 266 241 -22 834 -686	-21 711 628 -22 203 267	-14 753 70P -15 600 -560	-11 834 855 -14 263 -299	-5 1512 -1536 -6 273 -219
+12 2112 2324 -14 554 -514 -16 753 -706 -16 762 204 -18 201 266 -13 607 560	4 1329 -1574 5 1058 -1023	-23 247 -177 -25 2PC -256	-23 340 -296 -26 404 -411	-16 362 -328	-15 1170 -1107 -16 617 -589	-7 298 259
-20 599 -434 -20 545 219 -22 184 -159 -22 713 636	7 1615 -1893 8 1504 1646	-27 170 149	-28 67 -76	-19 879 812	-18 657 59A	-10 1699 1051 -11 356 -372 -12 569 1352
+24 561 500 -24 525 -423 +26 108 117 -26 004 -176	9 780 744 10 940 878	a,,,I	7,1,L	-21 324 -273 -22 485 457	-21 202 165	-14 1056 -1160 -15 283 270
4,0,L ^c ,C,L	11 1005 1010 12 1271 -1336	n 1346 -1424 1 1414 -1434 2 255 - 225	0 898 873 1 992 961 3 353 - 366	-24 171 171 -25 270 326	-23 488 -456	-18 720 689 -19 409 -364
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10 299 236 P 779 -368 12 950 -930 10 411 422 10 871 -872 10 69 62	18 843 115 21 335 - 291	8 1207 -1264 9 903 -901	11 260 208	6 126 -160	1 557 E17 2 1606 1719 3 1301 1368	-27 181 -213
19 361 221 -2 636 -538 20 302 300 -6 253 -227	-3 940 1071	10 526 -502 11 385 325	16 108 108 -1 904 834	-8 294 -221	4 329 - 289 5 702 - 666	2 1143 1216
-2 1737 -1936 -3 676 633 -4 1287 1302 -10 261 241	-5 407 -390 -6 1062 1186	12 211 193	-2 369 -278 -3 1360 -1410	-6 341 340 -7 431 -358	6 388 293 7 1452 - 1519	3 801 -998 6 1243 -1318
-6 1309 1362 -12 111 -733 -8 2590 -2960 -15 739 321	-8 238 283 -10 1594 -1751	10 176 -143	-5 293 -265	-9 559 -514 -10 327 -309 -11 334 306	9 505 571 9 818 - 370 10 662 - 708	7 1269 1320
-12 524 540 -20 779 205 -16 259 245 -22 796 689	-12 24P -216 -13 535 -400	-2 979 -791	-7 1171 1131 +0 1060 1042	-12 376 349 -13 328 315	11 909 954 12 271 227	13 428 422
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alkyl intermediate derived by a primary insertion of ethylene into a ruthenium-allyl bond of I [2].

The second feature is the presence of a triple bridging hydroxo group: such a group has been previously characterized in the crystal structure of $[Pt(OH)(CH_3)_3]_4$ [17,18], Ni₆(CF₃COCHCOCH₃)₁₀-(OH)₂(H₂O)₂ [11] and $[W(OH)(CO)_3H]_4$ ·4 OPPh₂-Et [19]. As the hydroxo group is a five-electron donor, in one molecule of II there are a total of sixty ligand-donated electrons, so each ruthenium atom can conform to the closed shell configuration without any metal-metal bond.

The close approach $O(9)...Cl(3)^i$ [3.174(9) Å] between the oxygen of the hydroxo group and the terminal chlorine atom is indicative of hydrogen bonding: as appears from Fig. 1, the relative orientation of the two atoms is favourable for hydrogen bonding which assures a further linking between the

two centrosymmetrically related trinuclear units in the molecule. This is also confirmed by the sharp IR absorption at 3280 cm⁻¹, consistent with the O-H stretching frequency for an O...Cl bond distance of 3.19 Å [20]. The origin of the hydroxo group of II, indicated also by a resonance at 10.9 τ in the ¹H n.m.r. spectrum [6], is not clear: it might arise from the moisture of the commercial ethylene used as well as from decomposition of carbon monoxide [21].

The last feature of the molecular structure of II is the different coordination of the three ruthenium atoms in the asymmetric unit: in fact, Ru(2) and Ru-(3) are identically coordinated, each being linked to two carbonyl groups, to the "carbene atom" of one ethyloxycarbene group and to the oxygen atom of the other ethyloxycarbene group, to a bridging chlorine atom and to the triple bridging hydroxo group. Ru(1) on the contrary is linked to three chlorine



Figure 1. The molecular structure of [Ru₃Cl₃(COC₂H₅)₂(OH)(CO)₆]₂·C₆H₆.

Ru(1)-Cl(1)	2.446(3)	Ru(3)O(8)	2.094(10)
Ru(1)-Cl(2)	2.398(5)	Ru(3)-O(9)	2.146(9)
Ru(1)–Cl(3)	2.343(5)		
Ru(1)-C(1)	1.851(19)	C(1)-O(1)	1.109(24)
Ru(1)-C(2)	1.842(12)	C(2) - O(2)	1.158(16)
Ru(1)-O(9)	2.169(10)	C(3)-O(3)	1.134(25)
		C(4)O(4)	1.123(16)
Ru(2)–Cl(2)	2.541(4)	C(5)-O(5)	1.125(23)
Ru(2)-C(3)	1.854(20)	C(6)-O(6)	1.162(21)
Ru(2)-C(4)	1.887(13)	C(7) - O(7)	1.246(19)
Ru(2)–C(8)	1.944(16)	C(8)–O(8)	1.276(19)
Ru(2)–O(7)	2.118(8)	C(7)-C(9)	1.497(17)
Ru(2)–O(9)	2.155(11)	C(9) - C(10)	1.499(28)
		C(8) - C(11)	1.509(23)
Ru(3)–Cl(1) ⁱ	2.571(4)	C(11)-C(12)	1.602(29)
Ru(3)C(5)	1.885(19)	C(13)-C(14)	1.43(5)
Ru(3)–C(6)	1.805(17)	C(13)-C(15)	1.37(5)
Ru(3)-C(7)	1.996(14)	C(14)-C(15) ⁱⁱ	1.37(5)

TABLE III. Bond Distances (Å)^{a,b}.

^aThe figures in parentheses are the estimated standard deviations. ^bThe symmetry code used in this and in the subsequent table is the following:

i	atom	at	- x	— y	1 - z
ii	atom	at	1 – x	— y	— z

atoms (one terminal and two bridging), to two carbonyl groups and to the triple bridging hydroxo group.

Concerning the structure of the amorphous product, the IR spectrum does not show the presence of the triple bridging OH group and indicates a simpler arrangement of the CO groups as results from the lower Ru–C stretchings between 650 and 400 cm⁻¹. The high value of the molecular weight (~2000) furthermore suggests for this product a " μ -catena" arrangement of RuCl(COC₂H₅)(CO)₂ units of type:



Both the crystalline and amorphous products are stable in the solid state and give by treatment with

TABLE IV. Bond Angles (°)^a.

Bond Angles at the Ruther	nium Atoms		
Cl(1)-Ru(1)-Cl(2)	89.7(1)	O(9)-Ru(2)-C(8)	88.1(6)
Cl(1)-Ru(1)-Cl(3)	89.6(1)	C(4)-Ru(2)-O(7)	178.9(11)
Cl(1)-Ru(1)-C(1)	89.5(4)	C(4) - Ru(2) - C(3)	86.0(7)
Cl(1)-Ru(1)-C(2)	176.6(5)	C(4)–Ru(2)–C(8)	92.2(6)
Cl(1)-Ru(1)-O(9)	85.4(2)	O(7)–Ru(2)–C(3)	94.4(5)
Cl(2)-Ru(1)-Cl(3)	172.3(2)	O(7)–Ru(2)–C(8)	88.7(4)
Cl(2)-Ru(1)-C(1)	95.5(6)	C(3)–Ru(2)–C(8)	91.4(8)
Cl(2)-Ru(1)-C(2)	87.6(6)		
Cl(2)-Ru(1)-O(9)	84.2(3)	O(9)–Ru(3)–Cl(1) ⁱ	88.3(3)
Cl(3)-Ru(1)-C(1)	92.2(6)	O(9)-Ru(3)-C(5)	100.2(6)
Cl(3)–Ru(1)–C(2)	93.4(6)	O(9)-Ru(3)-C(7)	87.2(5)
Cl(3)–Ru(1)–O(9)	88.0(3)	O(9)-Ru(3)-C(6)	174.4(7)
C(1)-Ru(1)-C(2)	88.8(6)	O(9)-Ru(3)-O(8)	82.2(4)
C(1)-Ru(1)-O(9)	174.9(4)	Cl(1) ⁱ -Ru(3)-C(5)	87.6(6)
C(2)-Ru(1)-O(9)	96.2(6)	$Cl(1)^{i}-Ru(3)-C(7)$	175.2(5)
		$Cl(1)^{i}-Ru(3)-C(6)$	92.4(5)
Cl(2)-Ru(2)-O(9)	81.2(3)	Cl(1) ⁱ –Ru(3)–O(8)	93.1(3)
Cl(2)–Ru(2)–C(4)	89.7(5)	C(5)–Ru(3)–C(7)	91.6(7)
Cl(2)-Ru(2)-O(7)	89.3(3)	C(5)–Ru(3)–C(6)	85.4(8)
Cl(2)-Ru(2)-C(3)	99.3(6)	C(5)-Ru(3)-O(8)	177.6(5)
Cl(2)-Ru(2)-C(8)	169.2(6)	C(7)–Ru(3)–C(6)	92.3(7)
O(9)-Ru(2)-C(4)	98.1(6)	C(7)–Ru(3)–O(8)	88.0(5)
O(9)-Ru(2)-O(7)	81.5(4)	C(6)–Ru(3)–O(8)	92.2(6)
O(9)-Ru(2)-C(3)	175.9(6)		
Bond Angles in the Ligand	s		
Ru(1)-Cl(1)-Ru(3) ⁱ	130.0(2)	Ru(2)–O(7)–C(7)	124.7(8)
Ru(1)-Cl(2)-Ru(2)	88.7(1)	Ru(3)-C(7)-O(7)	117.2(8)
Ru(1)-C(1)-O(1)	176.1(19)	Ru(3)-C(7)-C(9)	126.7(12)
Ru(1)-C(2)-O(2)	178.1(17)	O(7)-C(7)-C(9)	116.1(13)
Ru(2)-C(3)-O(3)	174.8(16)	C(7)-C(9)-C(10)	110.9(12)
Ru(2)-C(4)-O(4)	176.6(17)	Ru(3)–O(8)–C(8)	124.7(10)
Ru(3)-C(5)-O(5)	172.5(15)	Ru(2)C(8)O(8)	117.5(11)
Ru(3)-C(6)-O(6)	177.6(13)	Ru(2)C(8)C(11)	127.5(12)
Ru(1)-O(9)-Ru(2)	106.0(4)	O(8)-C(8)-C(11)	115.0(14)
Ru(1)-O(9)-Ru(3)	126.1(4)	C(8)-C(11)-C(12)	116.3(16)
Ru(2)–O(9)–Ru(3)	103.0(4)		
Bond angles in the Benzen	e Molecule		
C(14)-C(13)-C(15)	118.6(31)		
C(13)-C(14)-C(15) ⁱⁱ	119.1(29)		
C(13)-C(15)-C(14) ⁱⁱ	122.3(33)		

^aThe numbers in parentheses are the corresponding standard deviations.

triphenylphosphine or pyridine, with releasing of carbon monoxide and breaking of the bridging structure, the same mononuclear compounds $Ru(COC_2-H_5)(CO)L_2Cl\cdot Sv$ (L = PPh₃, Py; Sv = benzene, methylene chloride, tetrahydrofuran) [2].

Acknowledgements

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