of additional transition-metal complexes of TEDTA is currently under study.

Acknowledgment. Discussion and exchange of data with Bruce Parkinson were helpful. The experimental assistance of Roger Baar and the expert maintenance of the computerized electrochemical instrumentation by John Turner are a pleasure to acknowledge. This work was supported by the National Science Foundation.

Registry No. I (violet form), 70983-09-4; I (blue-green form), 71031-50-0; II, 70983-10-7; CrPMDTA (violet form), 70983-11-8; CrPMDTA (peach form), 70983-12-9; CrEEDTA (violet form), 70983-13-0; CrEEDTA (blue-green form), 70983-14-1; Hg^{2+}, 14302-87-5; CH_3Hg^+, 22967-92-6; Ag^+, 14701-21-4; azide, 14343-69-2.

References and Notes

(1) Anson, F. C. *Acc. Chem. Res.* **1975**, *8*, 400.
(2) Reference 1 and references cited therein.
(3) Anson, F. C. *Anal. Chem.* **1964**, *36*, 932.
(4) Peerce, P. J.; Anson, F. C. *J. Electroanal. Chem.*, in press.
(5) Sawyer, D. T.; Roberts, J. L. "Experimental Electrochemistry for Chemists"; Wiley: New York, 1974; Chapter 5.
(6) Altman, C.; King, E. L. *J. Am. Chem. Soc.* **1961**, *83*, 2825.
(7) Haupt, G. W. *J. Res. Natl. Bur. Stand.* **1952**, *48*, 414.
(8) Smolin, D. D.; Kazbitnaya, L. M.; Viktovov, Yu. M. *J. Gen. Chem. USSR* **1964**, *34*, 3762.
(9) Peerce, P. J. Ph.D. Thesis, California Institute of Technology, 1978.
(10) Kaufman, S.; Keyes, L. S. *Anal. Chem.* **1964**, *36*, 1777.
(11) The spectrum of the oxo-bridged species in acid differs from that of isomer I by the presence of a shoulder at 309 nm and a much more intense absorption in the UV.
(12) Hamm, R. E. *J. Am. Chem. Soc.* **1953**, *75*, 5670.
(13) Schwarzenbach, G.; Biedermann, H. *Helv. Chim. Acta* **1948**, *31*, 459.
(14) Dwyer, F. P.; Garvan, F. L. *J. Am. Chem. Soc.* **1960**, *82*, 4823.
(15) Hoard, J. L.; Kennard, C. H. L.; Smith, G. S. *Inorg. Chem.* **1963**, *2*, 1316.
(16) Thorneley, R. N. F.; Sykes, A. G.; Gans, P. *J. Chem. Soc. A* **1971**, 1494.
(17) Ogino, H.; Tsukahara, K.; Tanaka, N. *Inorg. Chem.* **1977**, *16*, 1215.
(18) Ogino, H.; Watanabe, T.; Tanaka, N. *Chem. Lett.* **1974**, 91.
(19) Ogino, H.; Watanabe, T.; Tanaka, N. *Inorg. Chem.* **1975**, *14*, 2093.
(20) Sulfab, Y.; Taylor, R. S.; Sykes, A. G. *Inorg. Chem.* **1976**, *15*, 2388.
(21) Finn, M. G.; Anson, F. C., unpublished experiments.
(22) Schwarzenbach, G.; Senn, H.; Anderegg, G. *Helv. Chim. Acta* **1957**, *40*, 1886.
(23) Pavkovic, S. F.; Meek, D. W. *Inorg. Chem.* **1965**, *4*, 20. Legg, J. I.; Cooke, D. W. *Ibid.* **1965**, *4*, 1576; **1966**, *5*, 594. Hamilton, H. G., Jr.; Alexander, M. D. *J. Am. Chem. Soc.* **1967**, *89*, 5065.
(24) Wilkins, R G.; Williams, M. J. P. "Modern Coordination Chemistry. Principles and Methods", Lewis, J., Wilkins, R. G., Eds.; Interscience: New York, 1960; pp 187–91.
(25) Garner, C. S.; House, D. A. *Transition Met. Chem.* **1970**, *6*, 59.
(26) None of the complexes assigned cis configurations for the nitrogen atoms exhibited appreciable affinity for Selectacel anion-exchange resin, whereas the two isomers assigned trans configurations, isomer II and the peach isomer of CrPMDTA, were bound strongly by this resin.
(27) Weschler, C. J.; Deutsch, E. *Inorg. Chem.* **1973**, *12*, 2682.
(28) Lane, R. H.; Sedor, F. A.; Gilroy, M. J.; Bennett, L. E. *Inorg. Chem.* **1977**, *16*, 102.
(29) Kennard, G. J.; Deutsch, E. *Inorg. Chem.* **1978**, *17*, 2225.
(30) Schwarzenbach, G.; Schellenberg, M. *Helv. Chim. Acta* **1965**, *48*, 28.
(31) Complexes containing Ag–S bonds are often photosensitive: McAuliffe, C. A.; Quagliano, J. V.; Vallarino, L. M. *Inorg. Chem.* **1966**, *5*, 2000.
(32) (a) McAuliffe, C. A.; Quagliano, J. V.; Vallarino, L. M. *Inorg. Chem.* **1966**, *5*, 1996. (b) Natusch, D. F. S.; Porter, L. J. *J. Chem. Soc., Chem. Commun.* **1970**, 596. (c) Fairhurst, M. T.; Rabenstein, D. L. *Inorg. Chem.* **1975**, *14*, 1413.
(33) McAuliffe, C. A. *Inorg. Chem.* **1973**, *12*, 1699. Livingstone, S. E.; Nolan, J. D. *Ibid.* **1968**, *7*, 1447.
(34) (a) Williams, R. J. P. *Annu. Rep. Prog. Chem.* **1959**, *56*, 87. (b) Cotton, F. A.; Zingales, F. A. *Chem. Ind.* (*London*) **1960**, 1219. (c) Livingstone, S. E. *Q. Rev., Chem. Soc.* **1965**, *19*, 386.
(35) Barclay, D. J. *J. Electroanal. Chem.* **1968**, *19*, 318. Barclay, D. J.; Caja, J. *Croat. Chem. Acta* **1971**, *43*, 221.

Contribution from the Department of Chemistry,
Texas A&M University, College Station, Texas 77843

Structural Studies of Some Multiply Bonded Diruthenium Tetracarboxylate Compounds

AVI BINO, F. ALBERT COTTON,* and TIMOTHY R. FELTHOUSE

Received March 9, 1979

Five compounds, three of types not previously described, containing the $Ru_2(O_2CR)_4^+$ unit have been structurally characterized by X-ray crystallography. These compounds and their crystallographic parameters are as follows: (**1**) $Ru_2(O_2CCH_3)_4Cl\cdot 2H_2O$, space group $C2/m$ with $a = 13.963$ (1) Å, $b = 7.399$ (1) Å, $c = 11.143$ (1) Å, $\beta = 138.88$ (2)°, and $Z = 2$; (**2**) $[Ru_2(O_2CCH_3)_4(H_2O)_2]BF_4$, space group Cc with $a = 14.246$ (2) Å, $b = 6.904$ (1) Å, $c = 17.687$ (2) Å, $\beta = 97.19$ (1)°, and $Z = 4$; (**3**) $Cs[Ru_2(O_2CCH_3)_4Cl_2]$, space group $P4_2/n$ with $a = 12.061$ (3) Å, $c = 11.335$ (2) Å, and $Z = 4$; (**4**) $Ru(O_2CCH_2CH_3)_4Cl$, space group $I\bar{4}$ with $a = 11.049$ (2) Å, $c = 7.423$ (1) Å, and $Z = 2$; (**5**) $K[Ru_2(O_2CH)_4Cl_2]$, space group $P4_2/n$ with $a = 11.483$ (2) Å, $c = 9.571$ (1) Å, and $Z = 4$. In **1** the $Ru_2(O_2CCH_3)_4^+$ ion has $2/m$ (C_{2h}) symmetry, with Ru–Ru = 2.267 (1) Å, and the Cl^- ions form perfectly linear symmetrical bridges between them. In **2** all atoms are on general positions. The $[Ru_2(O_2CCH_3)_4(OH_2)_2]^+$ ion has virtual D_{4h} symmetry with Ru–Ru = 2.248 (1) Å. In **3** the $[Ru_2(O_2CCH_3)_4Cl_2]^-$ ion has a crystallographic inversion center, and Ru–Ru = 2.286 (2) Å. In **4** the $[Ru_2(O_2CCH_2CH_3)_4]^+$ ion has $\bar{4}$ symmetry with Ru–Ru = 2.297 (7) Å. The Cl bridges are perfectly linear and symmetrical. In **5** the $[Ru_2(O_2CH)_4Cl_2]^-$ ion resides on a crystallographic inversion center with Ru–Ru = 2.290 (1) Å.

Introduction

The first report[1] of purportedly dinuclear complexes of ruthenium appeared in 1930. In 1966, Stephenson and Wilkinson[2a] published a study in which they found that this work could not be reproduced, but by different reactions they obtained other dinuclear diruthenium complexes, such as $Ru_2(O_2CR)_4Cl$. They suggested, however, that "the metal–metal distance in these systems is large enough to prevent direct orbital overlap." Independently, Japanese workers[2b,c] reported the preparation of the formato and acetato compounds $Ru_2(O_2CR)_4X$ (R = CH_3, H; X = Cl, Br, I). They described[2b] these substances as "the first spin-free complexes of ruthenium" and did not mention the possibility of Ru–Ru bonding. A few years[3] later it was suggested, and proven by an X-ray study of the compound with R = $CH_3CH_2CH_2$, that, on the contrary, a great deal of direct Ru–Ru overlap exists, since the Ru–Ru distance is 2.281 (4) Å. However, the reason for the ready formation of these particular systems with their odd-electron $Ru_2(O_2CR)_4^+$ unit was not at all obvious.

In an effort to account for the reported[2a] presence of three unpaired electrons, it was proposed[3] that in addition to eight electrons forming a $\sigma^2\pi^4\delta^2$ quadruple bond configuration, three more electrons with parallel spins occupy three orbitals of very similar but not equal energy, which were suggested to be the

0020-1669/79/1318-2599$01.00/0

Table I. Crystallographic Data

parameter	1	2	3	4	5
space group	$I2/m$[a]	Cc	$P4_2/n$	$I\bar{4}$	$P4_2/n$
a, Å	9.223 (1)	14.246 (2)	12.061 (3)	11.049 (2)	11.483 (2)
b, Å	7.399 (1)	6.904 (1)			
c, Å	11.143 (1)	17.687 (2)	11.335 (2)	7.423 (1)	9.571 (1)
β, deg	93.60 (1)	97.19 (1)			
V, Å^3	759 (1)	1726 (1)	1649 (1)	906.1 (3)	1262.0 (3)
d(calcd), g/cm^3	2.23	2.16	2.59	1.94	2.59
Z	2	4	4	2	4
fw	509.80	561.15	642.13	529.88	492.22
cryst size, mm	0.2 × 0.1 × 0.1	0.2 × 0.2 × 0.1	0.1 × 0.08 × 0.08	0.08 × 0.08 × 0.05	0.3 × 0.2 × 0.1
μ, cm^{-1} (for Mo Kα)	21.64	18.02	43.49	18.08	30.98
upper limit on 2θ, deg	45	50	45	45	45
no. of unique data	663	1253	834	258	811
data of $F_o^2 > 3\sigma(F_o^2)$	622	1171	686[b]	251[c]	765
R_1	0.045	0.038	0.041	0.053	0.035
R_2	0.056	0.053	0.051	0.071	0.060
goodness-of-fit	1.37	1.24	1.08	1.62	1.46

[a] The standard setting gives a cell belonging to the space group $C2/m$ with dimensions $a = 13.963$ Å, $b = 7.399$ Å, $c = 11.143$ Å, and $\beta = 138.88°$. The transformation matrix which yields the standard cell is

$$\begin{bmatrix} 1 & 0 & 1 \\ 0 & -1 & 0 \\ 0 & 0 & -1 \end{bmatrix}$$

[b] $F_o^2 > 2.5\sigma(F_o^2)$. [c] $F_o^2 > 2\sigma(F_o^2)$.

δ* orbital and two low-lying nonbonding orbitals directed outward along the Ru–Ru axis. This electron configuration implies a bond order of 3.5.

Some subsequent studies have modified this picture but left a great deal still to be clarified. It has been shown conclusively[4] that $Ru_2(O_2CC_3H_7)_4Cl$ does have three unpaired electrons per formula unit, as originally suggested by Stephenson and Wilkinson. There is a significant possibility that an inference drawn from the single datum of a room-temperature susceptibility could be erroneous, but the combined results of an extensive investigation of the temperature dependence of the magnetic susceptibility of the solid, the susceptibility of the compound in solution, and the EPR spectrum leave no doubt that one is dealing with a quartet ground state. An electrochemical study[4] produced rather complex results that did nothing to clarify the apparent preference of the $Ru_2(O_2CR)_4{}^n$ system for the value of $n = +1$.

An investigation[5] of the resonance Raman spectrum of the $Ru_2(O_2CR)_4Cl$ compounds with R = CH_3 and n-C_3H_7 provided evidence that the lowest energy electronic absorption band in these species, occurring at ca. 20 000 cm^{-1}, is due to an electric-dipole-allowed transition. A $\delta \rightarrow \delta^*$ assignment was suggested and the electron configuration was formulated as $\sigma^2\pi^4\delta^2\delta^*\pi^{*2}$; this means that the formal Ru–Ru bond order is 2.5. It remained, so far as we can see, as obscure as ever why the configuration is not $\sigma^2\pi^4\delta^2\delta^{*2}\pi^*$.

Most recently it has been stated[6] that an SCF-Xα-SW calculation on $Ru_2(O_2CH)_4Cl$ has shown that the ground-state configuration of electrons pertinent to Ru–Ru bonding should be $\sigma^2\pi^4\delta^2\pi^{*2}\delta^*$, with close spacing of the π^* and δ^* orbitals.

The chemical properties of $Ru_2{}^{n+}$ complexes have been but little studied. It is our own experience, which we understand has been the same elsewhere, that the isolation of oxidized or reduced products, i.e., compounds containing $Ru_2(O_2CR)_4$ or $Ru_2(O_2CR)_4{}^{2+}$, cannot be accomplished by any simple procedure. However, Warren and Goedken[7] have found that a macrocyclic tetradentate ligand, L ($C_{22}H_{22}N_4{}^{2-}$), will react with $Ru_2(O_2CCH_3)_4Cl$ to give $[Ru_2L_2]^+$ which can be reduced and oxidized. They found Ru–Ru bond lengths of ca. 2.28 Å in $[Ru_2L_2]^+$ and 2.625 Å in Ru_2L_2; they further found that Ru_2L_2, $[Ru_2L_2]^+$, and $[Ru_2L_2]^{2+}$ have 2, 1, and 0 unpaired electrons, respectively. These results are all consistent with the electron configurations (with implied bond orders in parentheses) for these three species as follows: Ru_2L_2, $\sigma^2\pi^4\delta^2\delta^{*2}\pi^{*2}$ (2.0); $[Ru_2L_2]^+$, $\sigma^2\pi^4\delta^2\delta^{*2}\pi^*$ (2.5); $[Ru_2L_2]^{2+}$, $\sigma^2\pi^4\delta^2\delta^{*2}$ (3.0). This situation is rather simple, intuitively satisfying, and at variance with what we know of the $Ru_2(O_2CR)_4{}^n$ systems.

It is clear that further studies of $Ru_2(O_2CR)_4{}^n$ species are needed. We have begun some new research on them and present here our first report. We have been looking for some crystalline compounds containing $Ru_2(O_2CR)_4{}^+$ ion with properties better suited to physical studies, especially crystal spectra, than is $Ru_2(O_2CC_3H_7)_4Cl$, and we have also been interested in varying the axially coordinated ligands from Cl^- to something quite different such as H_2O. We have succeeded in both of these efforts and report here our results on five compounds.

Experimental Section

Materials. $Ru_2(O_2CCH_3)_4Cl$ and $Ru_2(O_2CCH_2CH_3)_4Cl$ were prepared by using a procedure previously described.[8]

Preparation of $Ru_2(O_2CCH_3)_4Cl\cdot 2H_2O$, 1. An aqueous solution of $Ru_2(O_2CCH_3)_4Cl$ was absorbed on a cation-exchange column (Dowex 50W-X2) and eluted with 0.5 M LiCl. Slow evaporation of the reddish brown eluate gave beautiful dark red crystals.

Preparation of $[Ru_2(O_2CCH_3)_4(H_2O)_2]BF_4$, 2. This compound was prepared in an analogous fashion to **1**. Elution from the cation-exchange column was performed by using 0.5 M $NaBF_4$. Slow evaporation of the eluate produced reddish brown crystals.

Preparation of $Cs[Ru_2(O_2CCH_3)_4Cl_2]$, 3. An aqueous solution of $Ru_2(O_2CCH_3)_4Cl$ was mixed with an excess of CsCl. After a few days, reddish brown crystals were obtained.

Preparation of $Ru_2(O_2CCH_2CH_3)_4Cl$, 4. This compound was prepared by a procedure analogous to that used for the acetate, **1**. Elution from the cation-exchange column was accomplished with 0.25 M LiCl to give a yellowish brown eluate. Slow evaporation of the eluate gave small, dark purple crystals in the shape of flattened octahedra.

Preparation of $K[Ru_2(O_2CH)_4Cl_2]$, 5. The compound was prepared by using a carboxylate-exchange procedure. A sample of $Ru_2(O_2CCH_2CH_3)_4Cl$ was suspended in formic acid and heated to the boiling point. Addition of excess KCl followed by slow evaporation gave irregularly shaped brown-black crystals.

X-ray Crystallography. Collection of Data. Data were collected for all the compounds on a Syntex $P\bar{1}$ automated four-circle diffractometer using Mo (λ = 0.710 73 Å) radiation with a graphite crystal monochromator in the incident beam. Rotation photographs and ω scans of several strong reflections indicated in each case that the crystal was of satisfactory quality. The unit cell dimensions were obtained by a least-squares fit of 15 strong reflections in the range

Table II. Positional and Thermal Parameters and Their Estimated Standard Deviations for Compound 1[a]

atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ru	0.0000 (0)	0.15323 (9)	0.0000 (0)	3.20 (3)	0.69 (2)	1.97 (2)	0	−0.10 (2)	0
Cl	0.0000 (0)	0.5000 (0)	0.0000 (0)	4.1 (2)	0.69 (9)	3.5 (1)	0	0.3 (1)	0
O(1)	0.2049 (5)	0.1507 (6)	0.0745 (4)	3.4 (2)	1.1 (2)	3.5 (2)	0.0 (2)	−0.5 (2)	−0.1 (2)
O(2)	0.0810 (6)	0.1501 (6)	−0.1644 (4)	3.8 (2)	1.3 (2)	2.7 (2)	0.1 (2)	0.4 (2)	0.1 (2)
O(w)	0.1659 (14)	0.0000 (0)	0.3511 (13)	7.5 (7)	6.6 (7)	11.5 (7)	0	−2.1 (6)	0
C(2)	0.106 (1)	0.0000 (0)	−0.2137 (8)	3.3 (4)	2.2 (4)	2.4 (3)	0	−0.2 (3)	0
C(21)	0.170 (1)	0.0000 (0)	−0.3344 (9)	6.1 (6)	3.3 (5)	3.2 (4)	0	1.6 (4)	0
C(1)	0.266 (1)	0.0000 (0)	0.1005 (8)	3.4 (4)	1.9 (4)	2.8 (4)	0	−0.1 (3)	0
C(11)	0.419 (1)	0.0000 (0)	0.1592 (11)	3.8 (5)	3.4 (5)	5.0 (5)	0	−1.9 (4)	0

[a] The form of the anisotropic thermal parameter is $\exp[-{}^1/_4(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

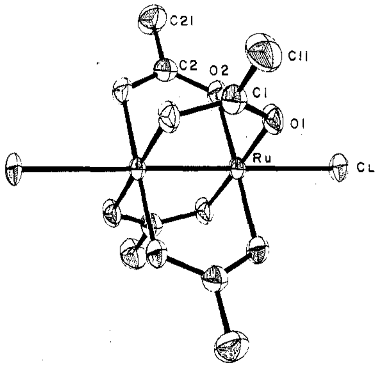

Figure 1. $Ru_2(O_2CCH_3)_4$ unit and its axial Cl atoms as they occur in $Ru_2(O_2CCH_3)_4Cl\cdot2H_2O$, **1**. Unlabeled atoms are related to labeled ones by the C_{2h} crystallographic symmetry.

$25° < 2\theta < 35°$. Data were collected at 22 ± 3° by using θ–2θ scan technique with a variable scan rate from 4.0 to 24.0°/min. General procedures for data collection have been reported elsewhere.[9] Crystallographic data and other pertinent information are given in Table I. For each crystal, Lorentz and polarization corrections were applied, but absorption corrections were omitted. In the case of compound **3**, ψ scans at $\chi = 90°$ for several reflections showed no variation greater than 5%.

Solution and Refinement of the Structures.[10] The heavy-atom positions in **1** and **4** were obtained through a three-dimensional Patterson function. In **2**, **3**, and **5** these positions were obtained by direct methods with the MULTAN program. All the structures were refined to convergence by full-matrix least-squares techniques. Compound **1** was refined in space group $I2/m$ (see footnote *a* in Table I) by using anisotropic thermal parameters for all atoms. Compound **2** was refined in space group Cc by using anisotropic thermal parameters for the metal atoms and all of the acetate oxygen atoms and isotropic thermal parameters for the rest of the nonhydrogen atoms. Compound **3** was refined in space group $P4_2/n$ by using anisotropic thermal parameters for all the atoms. Compound **4** was refined in space group $I\bar{4}$ by using anisotropic thermal parameters for Ru, Cl, and O and isotropic ones for the carbon atoms. Compound **5** was refined in space group $P4_2/n$ by using anisotropic thermal parameters for all atoms.

In both **2** and **4**, the refinement of the mirror image of the original structure suggested that the original choice of enantiomorph was right.

The final discrepancy indices defined as

$$R_1 = \frac{\sum||F_o| - |F_c||}{\sum|F_o|} \qquad R_2 = \left[\frac{\sum w(|F_o| - |F_c|)^2}{\sum w|F_o|^2}\right]^{1/2}$$

are listed in Table I, along with other crystal data. The final difference maps showed no peaks of structural significance.

Results and Discussion

Tables II–VI record the atomic positional and thermal parameters. Each of the structures refined very efficiently to relatively low discrepancy indices.

For each structure we present an ORTEP drawing of the unit containing the ruthenium atoms and all attached atoms, Figures 1–5, in each of which the atomic numbering scheme

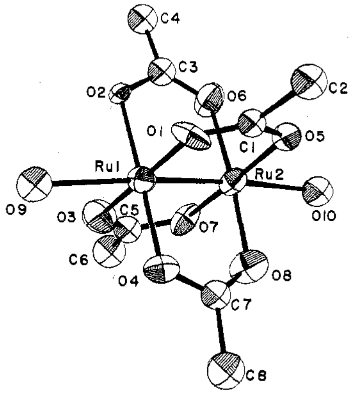

Figure 2. $[Ru_2(O_2CCH_3)_4(H_2O)_2]^+$ ion as it occurs in **2**, showing the atom numbering scheme.

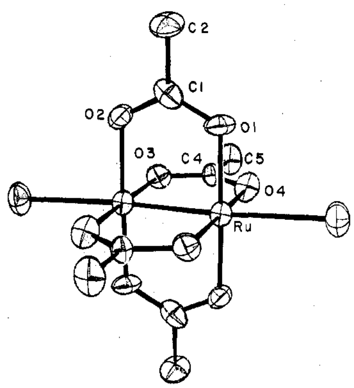

Figure 3. $[Ru_2(O_2CCH_3)_4Cl_2]^-$ ion as it occurs in **3**, showing the atomic numbering scheme. Unlabeled atoms are related to labeled ones by a center of inversion.

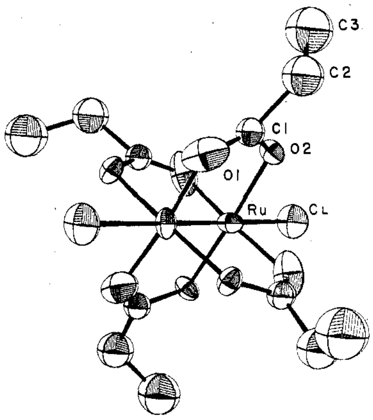

Figure 4. $Ru_2(O_2CC_2H_5)_4Cl_2$ unit in **4**, showing the atom numbering scheme. Unlabeled atoms are related by the $\bar{4}$ symmetry to the labeled ones.

Table III. Positional and Thermal Parameters and Their Estimated Standard Deviations for Compound **2**[a]

atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ru(1)	0.22852 (0)	0.1521 (1)	0.36523 (0)	2.68 (4)	2.53 (4)	2.64 (3)	−0.14 (4)	0.66 (3)	−0.22 (4)
Ru(2)	0.12035 (7)	0.3463 (2)	0.40847 (6)	2.16 (3)	3.44 (5)	3.22 (4)	−0.02 (4)	0.44 (3)	−0.12 (4)
O(1)	0.2286 (7)	0.322 (1)	0.2721 (5)	5.2 (4)	1.6 (3)	3.5 (4)	−0.9 (3)	1.2 (3)	−0.6 (3)
O(2)	0.3373 (5)	0.308 (1)	0.4238 (5)	1.3 (3)	2.1 (3)	3.5 (3)	−0.2 (3)	−0.4 (3)	0.7 (3)
O(3)	0.2280 (8)	−0.014 (2)	0.4539 (6)	5.2 (5)	4.1 (5)	4.6 (5)	−0.2 (4)	0.5 (4)	1.8 (4)
O(4)	0.1266 (6)	−0.004 (1)	0.3075 (4)	3.3 (3)	3.5 (4)	2.1 (3)	−0.9 (3)	0.9 (3)	0.5 (3)
O(5)	0.1244 (6)	0.523 (1)	0.3153 (5)	2.5 (3)	2.6 (3)	2.4 (3)	0.1 (3)	0.6 (2)	−0.7 (3)
O(6)	0.2286 (7)	0.499 (1)	0.4641 (6)	3.2 (4)	4.3 (5)	5.8 (5)	0.7 (4)	−0.4 (4)	−2.6 (4)
O(7)	0.1190 (6)	0.170 (2)	0.4989 (5)	2.2 (3)	6.3 (6)	2.2 (3)	0.1 (4)	0.6 (3)	1.0 (4)
O(8)	0.0240 (7)	0.176 (2)	0.3538 (6)	3.9 (4)	5.4 (6)	3.8 (4)	−0.4 (4)	0.9 (3)	−1.2 (4)

atom	x	y	z	B, Å^2	atom	x	y	z	B, Å^2
F(1)	−0.1481 (9)	0.692 (2)	0.5929 (7)	7.2 (3)	C(1)	0.1813 (8)	0.460 (2)	0.2642 (6)	2.4 (2)
F(2)	−0.0046 (10)	0.726 (2)	0.6609 (9)	9.3 (4)	C(2)	0.1737 (10)	0.616 (2)	0.2020 (8)	3.4 (3)
F(3)	−0.1229 (7)	0.459 (2)	0.6791 (6)	6.3 (2)	C(3)	0.3136 (9)	0.459 (2)	0.4646 (7)	3.1 (2)
F(4)	−0.0533 (9)	0.500 (2)	0.5761 (7)	7.9 (3)	C(4)	0.3921 (10)	0.556 (2)	0.5052 (8)	3.6 (3)
O(9)	0.3358 (7)	−0.068 (1)	0.3227 (6)	3.8 (2)	C(5)	0.1769 (10)	0.014 (2)	0.5028 (8)	3.3 (2)
O(10)	0.0021 (7)	0.523 (1)	0.4499 (5)	3.8 (2)	C(6)	0.1648 (12)	−0.085 (3)	0.5774 (10)	5.1 (4)
					C(7)	0.0418 (9)	0.046 (2)	0.3150 (7)	2.9 (2)
					C(8)	−0.0307 (11)	−0.095 (2)	0.2685 (9)	4.3 (3)
					B	−0.072 (3)	0.603 (2)	0.640 (2)	3.6 (2)

[a] The form of the anisotropic thermal parameter is $\exp[-\frac{1}{4}(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

Table IV. Positional and Thermal Parameters and Their Estimated Standard Deviations for Compound **3**[a]

atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cs	0.7500 (0)	0.2500 (0)	0.3061 (1)	5.39 (8)	3.06 (7)	3.07 (6)	−1.28 (8)	0	0
Ru	0.5304 (1)	0.0875 (1)	0.5212 (1)	2.20 (5)	1.75 (4)	1.73 (4)	−0.23 (5)	0.01 (5)	−0.01 (5)
Cl	0.5843 (4)	0.2849 (3)	0.5669 (4)	4.2 (2)	1.9 (2)	3.7 (2)	−0.4 (2)	−0.8 (2)	−0.0 (1)
O(1)	0.3774 (8)	0.1261 (8)	0.5806 (9)	1.4 (4)	3.1 (5)	2.8 (5)	0.1 (4)	0.3 (4)	−0.6 (4)
O(2)	0.6813 (8)	0.0452 (8)	0.4583 (9)	1.8 (4)	2.4 (4)	2.7 (4)	−0.6 (4)	−0.0 (4)	0.3 (4)
O(3)	0.5738 (8)	0.0335 (8)	0.6835 (8)	2.5 (5)	2.4 (4)	2.2 (4)	0.0 (4)	−0.6 (4)	−0.1 (4)
O(4)	0.4881 (9)	0.1337 (8)	0.3567 (9)	3.4 (5)	2.7 (5)	2.0 (4)	−0.5 (4)	0.3 (4)	−0.2 (4)
C(1)	0.303 (1)	0.052 (1)	0.580 (1)	3.6 (8)	2.6 (7)	1.6 (6)	0.9 (6)	0.6 (6)	0.6 (6)
C(2)	0.192 (1)	0.081 (1)	0.630 (1)	2.5 (7)	3.8 (8)	3.2 (8)	0.0 (7)	1.4 (7)	−0.8 (7)
C(4)	0.555 (1)	−0.067 (1)	0.716 (1)	2.1 (7)	1.4 (6)	1.3 (6)	−0.2 (5)	−0.2 (5)	−0.2 (5)
C(5)	0.585 (1)	−0.102 (1)	0.836 (1)	4.4 (8)	2.9 (7)	1.7 (6)	−0.5 (7)	−1.8 (6)	0.7 (6)

[a] The form of the anisotropic thermal parameter is $\exp[-\frac{1}{4}(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

Table V. Positional and Thermal Parameters and Their Estimated Standard Deviations for Compound **4**[a]

atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ru	0.0000 (0)	0.0000 (0)	−0.1544 (3)	4.0 (1)	1.97 (8)	1.21 (6)	−0.2 (4)	0	0
Cl	0.0000 (0)	0.0000 (0)	−0.5000 (0)	6.2 (8)	6	1.9 (4)	0	0	0
O(1)	−0.146 (2)	−0.093 (2)	−0.175 (2)	5 (1)	9 (1)	2.4 (9)	2 (1)	−0.2 (8)	−1 (1)
O(2)	−0.159 (1)	−0.093 (1)	0.144 (2)	2.6 (5)	3.4 (6)	1.6 (4)	−0.8 (4)	−0.2 (5)	0.1 (6)

atom	x	y	z	B, Å^2	atom	x	y	z	B, Å^2
C(1)	−0.196 (2)	−0.122 (2)	−0.036 (3)	3.1 (5)	C(3)	−0.412 (4)	−0.147 (4)	0.153 (6)	8.5 (11)
C(2)	−0.313 (3)	−0.203 (3)	0.018 (8)	6.1 (7)					

[a] The form of the anisotropic thermal parameter is $\exp[-\frac{1}{4}(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

Table VI. Positional and Thermal Parameters and Their Estimated Standard Deviations for Compound **5**[a]

atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ru	0.47761 (6)	0.59624 (6)	0.98407 (7)	4.13 (3)	3.88 (3)	1.93 (3)	1.60 (2)	0.04 (2)	0.10 (2)
K	0.2500 (0)	0.7500 (0)	0.2133 (3)	5.2 (1)	3.9 (1)	2.79 (9)	0.6 (1)	0	0
Cl	0.4312 (2)	0.8079 (2)	0.9440 (2)	5.6 (1)	3.97 (9)	2.83 (8)	1.55 (8)	−0.90 (8)	−0.22 (8)
O(1)	0.6386 (5)	0.6256 (5)	0.9080 (6)	4.2 (2)	3.8 (2)	4.8 (3)	1.2 (2)	1.4 (2)	0.6 (2)
O(2)	0.6835 (4)	0.4379 (4)	0.9377 (7)	3.5 (2)	4.1 (3)	4.2 (3)	1.6 (2)	0.5 (2)	0.4 (2)
O(3)	0.4222 (5)	0.5580 (5)	0.7903 (6)	5.6 (3)	4.0 (2)	3.0 (2)	1.9 (2)	−0.6 (2)	0.1 (2)
O(4)	0.5333 (5)	0.6303 (5)	1.1805 (6)	6.3 (3)	3.6 (2)	2.5 (2)	1.9 (2)	0.2 (2)	−0.5 (2)
C(1)	0.4292 (8)	0.4550 (8)	0.7450 (9)	5.8 (5)	5.8 (4)	2.5 (3)	2.2 (4)	−0.2 (3)	0.7 (3)
C(2)	0.7061 (9)	0.5389 (9)	0.9023 (10)	5.8 (5)	6.6 (5)	3.6 (4)	1.0 (4)	1.1 (4)	−0.1 (4)

[a] The form of the anisotropic thermal parameter is $\exp[-\frac{1}{4}(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

Table VII. Bond Distances (Å) and Bond Angles (deg) for $Ru_2(O_2CCH_3)_4Cl \cdot 2H_2O$, **1**

Distances

Ru-Ru′	2.267 (1)	O(1)-C(1)	1.276 (6)
Cl	2.566 (1)	O(2)-C(2)	1.266 (5)
O(1)	2.016 (4)	C(1)-C(11)	1.52 (1)
O(2)	2.022 (4)	C(2)-C(21)	1.50 (1)

Angles

Ru′-Ru-O(1)	89.5 (1)	Ru-O(1)-C(1)	119.6 (5)
O(2)	89.4 (1)	Ru-O(2)-C(2)	119.3 (4)
O(1)-Ru-O(1)′	178.9 (2)	O(1)-C(1)-O(1)″	121.8 (8)
O(2)	89.0 (2)	C(11)	119.1 (4)
O(1)-Ru-O(2)′	91.0 (2)	O(2)-C(2)-O(2)″	122.6 (8)
		C(21)	118.7 (4)

Table VIII. Bond Distances (Å) and Bond Angles (deg) for $[Ru_2(O_2CCH_3)_4(H_2O)_2]BF_4$, **2**

Distances

Ru(1)-Ru(2)	2.248 (1)	Ru(2)-O(5)	2.05 (2)
O(1)	2.02 (2)	O(6)	2.02 (2)
O(2)	2.06 (2)	O(7)	2.01 (2)
O(3)	1.90 (2)	O(8)	1.97 (2)
O(4)	1.99 (2)	O(10)	2.27 (1)
O(9)	2.34 (1)	O(3)-C(5)	1.21 (3)
O(1)-C(1)	1.16 (3)	O(7)-C(5)	1.35 (3)
O(5)-C(1)	1.36 (2)	C(5)-C(6)	1.52 (3)
C(1)-C(2)	1.54 (3)	O(4)-C(7)	1.28 (3)
O(2)-C(3)	1.34 (3)	O(8)-C(7)	1.17 (3)
O(6)-C(3)	1.24 (3)	C(7)-C(8)	1.57 (3)
C(3)-C(4)	1.42 (3)		
B-F(1)	1.42 (4)		
F(2)	1.30 (4)		
F(3)	1.46 (4)		
F(4)	1.38 (4)		

Angles

Ru(2)-Ru(1)-O(1)	90.1 (3)	Ru(1)-Ru(2)-O(5)	89.5 (3)
O(2)	91.3 (3)	O(6)	87.9 (3)
O(3)	90.3 (3)	O(7)	89.1 (3)
O(4)	90.6 (3)	O(8)	86.7 (4)
O(9)	176.3 (3)	O(10)	175.3 (3)
O(1)-Ru(1)-O(2)	91.7 (8)	O(5)-Ru(2)-O(6)	89.1 (8)
O(3)	179.3 (8)	O(7)	178.6 (7)
O(4)	88.4 (7)	O(8)	92.7 (8)
O(2)-Ru(1)-O(3)	88.9 (9)	O(6)-Ru(2)-O(7)	90.7 (8)
O(4)	178.1 (8)	O(8)	174.2 (9)
O(3)-Ru(1)-O(4)	91.0 (8)	O(7)-Ru(2)-O(8)	87.3 (9)
Ru(1)-O(1)-C(1)	121 (2)	Ru(1)-O(3)-C(5)	123 (2)
Ru(2)-O(5)-C(1)	115 (1)	Ru(2)-O(7)-C(5)	117 (1)
O(1)-C(1)-O(5)	124 (2)	O(3)-C(5)-O(7)	121 (2)
C(2)	130 (2)	C(6)	134 (2)
O(5)-C(1)-C(2)	105 (2)	O(7)-C(5)-C(6)	106 (2)
Ru(1)-O(2)-C(3)	117 (1)	Ru(1)-O(4)-C(7)	116 (2)
Ru(2)-O(6)-C(3)	125 (2)	Ru(2)-O(8)-C(7)	124 (2)
O(2)-C(3)-O(6)	119 (2)	O(4)-C(7)-O(8)	123 (3)
C(4)	114 (2)	C(8)	110 (2)
O(6)-C(3)-C(4)	127 (2)	O(8)-C(7)-C(8)	127 (2)
F(1)-B-F(2)	112 (1)	F(2)-B-F(3)	134 (4)
F(3)	100 (3)	F(4)	111 (3)
F(4)	89 (3)	F(3)-B-F(4)	101 (1)

for that species is defined. For each structure the significant interatomic distances and angles are presented in Tables VII–XI.

Table XII compares the Ru–Ru, Ru–O(carboxylate), and Ru–Cl distances for all six compounds now structurally characterized. The Ru–O distances are invariant within the uncertainties. There are, however, significant variations in the Ru–Ru and Ru–Cl distances, and these will be discussed after each structure has been described in detail. Before this is done, it should be recalled that in the previously reported structure of $Ru_2(O_2CC_3H_7)_4Cl$ the $Ru_2(O_2CR)_4^+$ units are connected into infinite chains by bridging Cl^- ions which occupy axial positions (Ru′–Ru–Cl, 175.1 (1)°). However,

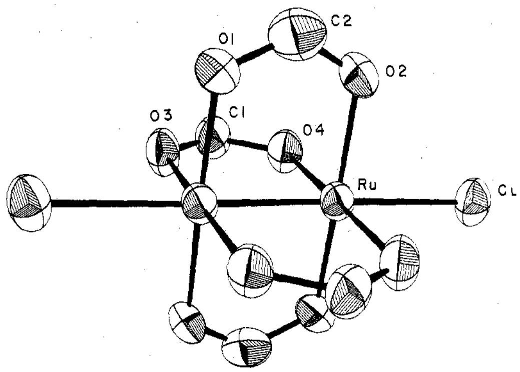

Figure 5. $[Ru_2(O_2CH)_4Cl_2]^-$ ion that occurs in **5**. Unlabeled atoms are related to labeled ones by the center of inversion.

Table IX. Bond Distances (Å) and Bond Angles (deg) for $Cs[Ru_2(O_2CCH_3)_4Cl_2]$, **3**

Distances

Ru-Ru′	2.286 (2)	O(1)-C(1)	1.27 (2)
Cl	2.521 (4)	O(2)-C(1)	1.26 (2)
O(1)	2.02 (1)	C(1)-C(2)	1.49 (2)
O(2)′	2.02 (1)	O(3)-C(4)	1.28 (2)
O(3)′	2.02 (1)	O(4)-C(4)	1.26 (2)
O(4)	2.01 (1)	C(4)-C(5)	1.47 (2)

Angles

Ru′-Ru-Cl	176.2 (1)	Ru-O(1)-C(1)	118 (1)
O(1)	89.4 (3)	Ru-O(2)′-C(1)	119 (1)
O(2)′	88.9 (3)	O(1)-C(1)-O(2)	123 (1)
O(3)′	88.7 (3)	C(2)	117 (1)
O(4)	88.8 (3)	O(2)-C(1)-C(2)	119 (1)
O(1)-Ru-O(2)′	178.1 (4)	Ru-O(3)′-C(4)′	119 (1)
O(3)′	90.5 (4)	Ru-O(4)-C(4)	122 (1)
O(4)	90.7 (4)	O(3)-C(4)-O(4)	119 (1)
O(2)′-Ru-O(3)′	90.4 (4)	C(5)	119 (1)
O(4)	88.3 (4)	O(4)-C(4)-C(5)	121 (1)
O(3)′-Ru-O(4)	177.2 (4)		

Table X. Bond Distances (Å) and Bond Angles (deg) for $Ru_2(O_2CCH_2CH_3)_4Cl$, 4

Distances

Ru-Ru′	2.292 (7)	O(1)-C(1)	1.22 (3)
Cl	2.566 (4)	O(2)-C(1)	1.43 (3)
O(1)′	1.92 (3)	C(1)-C(2)	1.61 (3)
O(2)	2.04 (2)	C(2)-C(3)	1.60 (5)

Angles

Ru′-Ru-O(1)′	95 (1)	Ru-O(1)-C(1)	117 (2)
O(2)	88.0 (7)	Ru-O(2)-C(1)	113 (2)
O(1)′-Ru-O(1)″	171 (2)	O(1)-C(1)-O(2)	127 (2)
O(2)	88 (1)	C(2)	134 (3)
O(2)′	92 (2)	O(2)-C(1)-C(2)	100 (2)
O(2)-Ru-O(2)′	176 (1)	C(1)-C(2)-C(3)	119 (2)

Table XI. Bond Distances (Å) and Bond Angles (deg) for $K[Ru_2(O_2CH)_4Cl_2]$, **5**

Distances

Ru-Ru′	2.290 (1)	O(1)-C(2)	1.26 (1)
Cl	2.517 (2)	O(2)-C(2)	1.24 (1)
O(1)′	2.016 (5)	O(3)-C(1)	1.262 (9)
O(2)	2.034 (5)	O(4)-C(1)	1.286 (9)
O(3)′	2.010 (5)		
O(4)	2.024 (5)		

Angles

Ru′-Ru-Cl	178.68 (6)	O(2)-Ru-O(3)′	90.5 (2)
O(1)′	90.2 (1)	O(4)	89.0 (2)
O(2)	88.3 (1)	O(3)′-Ru-O(4)	178.5 (2)
O(3)′	89.0 (1)	Ru-O(1)′-C(2)′	116.6 (5)
O(4)	89.5 (1)	Ru-O(2)-C(2)	118.2 (5)
O(1)′-Ru-O(2)	178.4 (2)	O(1)-C(2)-O(2)	126.8 (8)
O(3)′	89.6 (2)	Ru-O(3)′-C(1)′	120.1 (5)
O(4)	90.8 (2)	Ru-O(4)-C(1)	118.2 (5)
		O(3)-C(1)-O(4)	123.1 (7)

Table XII. Principal Bond Lengths (Å) in Six $Ru_2(O_2CR)_4^+$ Compounds

compd	Ru-Ru	Ru-O(carboxylate)	Ru-Cl
$K[Ru_2(O_2CH)_4Cl_2]$	2.290 (1)	2.021 (5)	2.517 (2)
$Ru_2(O_2CCH_3)_4Cl \cdot 2H_2O$	2.267 (1)	2.019 (3)	2.566 (1)
$[Ru_2(O_2CCH_3)_4(H_2O)_2]BF_4$	2.248 (1)	2.00 (4)	
$Cs[Ru_2(O_2CCH_3)_4Cl_2]$	2.286 (2)	2.02 (1)	2.521 (4)
$Ru_2(O_2CC_2H_5)_4Cl$	2.292 (7)	1.99 (2)	2.566 (4)
$Ru_2(O_2CC_3H_7)_4Cl$	2.281 (4)	2.00 (3)	2.587 (5)

these chains are zigzag, with an angle of 125.4 (1)° at the Cl atom. This angle, not stated in ref 3, has been calculated by us.

Compound 1. The ruthenium atom resides on a twofold axis at 0, y, 0. A mirror plane bisects the twofold axis within the dinuclear units. Thus, the entire group of atoms shown in Figure 1 is arranged in accord with crystallographically imposed C_{2h} symmetry. However, as Table VII shows, the Ru–O distances and O–Ru–O angles conform, within experimental error, with D_{4h} symmetry. The chlorine atoms bridge the dinuclear units symmetrically, creating linear one-dimensional chains. All atoms not yet mentioned, except O(1) and O(2), reside on the mirror plane at x, 0, z.

Compound 2. All atoms are located on general positions, and therefore no symmetry is imposed on the molecule by the crystal structure. However, variations in Ru–O and O–Ru–O angles (Table VIII), though real in a statistical sense, show no pattern implying any systematic descent from D_{4h} symmetry for the unit shown in Figure 2.

Compound 3. The $[Ru_2(O_2CCH_3)_4Cl_2]^-$ ion, Figure 3, resides on a crystallographic center of inversion. Again, however, the actual values of bond lengths and angles are consistent with virtual D_{4h} symmetry.

Compound 4. The midpoint of the Ru–Ru bond is a position of crystallographic $\bar{4}$ (S_4) symmetry. The –Cl–Ru–Ru–Cl– chains are thus precisely linear with all Ru–Cl distances equal, and they run exactly along the crystallographic z axis. The $Ru_2(O_2CC_2H_5)_4Cl_2$ unit is shown in Figure 4. The errors in light-atom positions, and hence in all bond distances (except Ru–Ru) and angles, are rather large. In view of this, the apparent deviations from D_{4h} symmetry are not statistically real, with but one possible exception: the difference between the two crystallographically independent Ru–Ru–O angles (7°), each of which has an esd of ca. 1°. The crystals of this compound were all rather smaller than optimum and the data were therefore limited and were of relatively low quality. The structure determination was carried out despite this because the very favorable orientation of the molecular units will make this substance useful in future studies of spectra.

Compound 5. The $[Ru_2(O_2CH)_4Cl_2]^-$ ion, Figure 5, resides on a crystallographic inversion center, and the entire ion has effective D_{4h} symmetry to within the experimental uncertainities, as the distances and angles in Table XI show.

We turn now to Table XII which collects all available results on the key bond lengths in compounds containing the $Ru_2(O_2CR)_4^+$ unit. The six compounds listed comprise the five new ones reported in this paper plus the previously described $Ru_2(O_2CC_3H_7)_4Cl$.[3] There is clearly no significant variation in the Ru–O distances, and a mean value of 2.01 ± 0.01 Å adequately represents all of them.

There are significant variations in the axial Ru···Cl distances which appear to follow an understandable pattern. Of the five available ones, two are 2.519 ± 0.003 Å, two are 2.566 ± 0.003 Å, and one is 2.587 (5) Å. The first two occur in the $[Ru(O_2CR)_4Cl_2]^-$ ions, the next two occur in the chain structures where the angles at the bridging Cl atoms are 180°, and the last one occurs in the chain structure where the angle at the bridging Cl atom is 125.4°.

There is also a spread in the Ru–Ru distances, but actually few of the differences are large enough to be significant in both the statistical and chemical senses. For the five compounds in which the axial ligands are Cl^- ions, the Ru–Ru distances range from 2.267 (1) to 2.292 (7) Å. However, when the esd's are considered, the 3σ criterion of significance indicates that there are only two significant differences, namely, those between 2.267 (1) Å in compound **1** where there are infinite chains and the two values 2.290 (1) and 2.286 (2) Å in the compounds containing discrete $[Ru_2(O_2CR)_4Cl_2]^-$ ions. This, in turn, correlates with the fact that the Ru–Cl distance in the former compound is significantly longer than those in each of the other two compounds. It seems that as the nonbridging Cl^- axial ligands move closer to the Ru atoms, by ca. 0.045 Å, than do the bridging Cl^- ions, the Ru–Ru bond is lengthened by ca. 0.022 Å. This is, admittedly, a small effect, but it is consistent in both direction and magnitude with the effects seen previously in dimolybdenum[11] and dirhenium[12] compounds.

By far the most dramatic difference to be seen among the six compounds in Table XII is in the Ru–Ru distance, 2.248 (1) Å, for the diaquo species, $[Ru_2(O_2CR)_4(H_2O)_2]^+$, as compared to all the others which average 2.283 ± 0.007 Å.

Acknowledgment. We thank the National Science Foundation for generous financial support.

Supplementary Material Available: Tables of observed and calculated structure factors (17 pages). Ordering information is given on any current masthead page.

References and Notes

(1) Mond, A. W. *J. Chem. Soc.* **1930**, 1247.
(2) (a) Stephenson, T. A.; Wilkinson, G. *J. Inorg. Nucl. Chem.* **1966**, *28*, 2285. (b) Mukaida, M.; Nomura, T.; Ishimori, T. *Bull. Soc. Chem. Jpn.* **1967**, *40*, 2462. (c) *Ibid.* **1972**, *45*, 2143.
(3) Bennett, M. J.; Caulton, K. G.; Cotton, F. A. *Inorg. Chem.* **1969**, *8*, 1.
(4) Cotton, F. A.; Pedersen, E. *Inorg. Chem.* **1975**, *14*, 388.
(5) Clark, R. J. H.; Franks, M. L. *J. Chem. Soc., Dalton Trans.* **1976**, 1825.
(6) Norman, J. G., Jr.; Kolari, H. J. *J. Am. Chem. Soc.* **1978**, *100*, 791.
(7) Warren, L. F.; Goedken, V. L. *J. Chem. Soc., Chem. Commun.* **1978**, 909. Also private communications from V.L.G. to F.A.C.
(8) Mitchell, R. W.; Spencer, A.; Wilkinson, G. *J. Chem. Soc., Dalton Trans.* **1973**, 846.
(9) Cotton, F. A.; Frenz, B. A.; Deganello, G.; Shaver, A. *J. Organomet. Chem.* **1973**, *50*, 227; Adams, R. D.; Collins, D. M.; Cotton, F. A. *J. Am. Chem. Soc.* **1974**, *96*, 749.
(10) All crystallographic computing was done on a PDP 11/45 computer at the Molecular Structure Corp. College Station, TX, using the Enraf-Nonius structure determination package.
(11) Cotton, F. A.; Norman, J. G., Jr.; *J. Am. Chem. Soc.* **1972**, *94*, 5697. Collins, D. M.; Cotton, F. A.; Murillo, C. A. *Inorg. Chem.* **1976**, *15*, 1861; Cotton, F. A.; Shive, L. W. *Ibid.* **1975**, *14*, 2027.
(12) Cotton, F. A.; Hall, W. T. *Inorg. Chem.* **1977**, *16*, 1867.