Table VI. Substituent Contribution to the Stability of Monoligand Monocarboxylate Complexes Expressed in log *K* Units

metal ion	Δ log *K*(ML) CNAC	CLAC	PHAC	MAND	GLYCO	PYRU
Co^{2+}	0.04	0.01	0.01	0.76	0.86	0.34
Ni^{2+}	0.04	0.02	-0.04	0.80	1.06	0.32
Zn^{2+}	0.00	0.14		0.71	1.07	0.29
Cd^{2+}	0.03	0.12			0.44	0.09
Cu^{2+}	-0.04	0.18			0.89	0.50
Pb^{2+}	0.05	0.32			0.21	0.51

metal ion	Δ log *K*(ML) LACT	3OHP	GLYCE	2OHB	3OHB	4OHB
Co^{2+}	0.85	0.04	0.74	0.93	0.20	-0.21
Ni^{2+}	1.03	0.14	0.82	1.11	0.28	-0.20
Zn^{2+}	0.74	-0.05	0.75	0.96	0.08	-0.01
Cd^{2+}	0.36	-0.01	0.47	0.23	-0.02	0.08
Cu^{2+}	1.10	0.02	0.85	1.18	0.10	-0.13
Pb^{2+}	0.38	0.11	0.76	0.45	0.14	0.05

OH as a substituent on the acetate ligand has a pronounced stabilizing effect over the ligand basicity (from 0.70 to 1.07 log *K* units) due to the stable five-membered chelate ring coordination, which was confirmed by an IR study of glycolate complexes in aqueous solutions.[50] Cd^{2+} and Pb^{2+} ions have a smaller tendency toward chelate stabilization than other investigated ions (only 0.44 and 0.21 log *K* unit for Cd^{2+} and Pb^{2+} ions, respectively).

Substituents on the propanoate ligand in the 2-position contribute also to additional chelate stabilization. The oxygen atom in the PYRU ligand additionally stabilizes the complex by 0.3–0.5 log K unit, while the OH group in the LACT ligand stabilizes the complex by for 0.7–1.1 log K units. In both systems the Cd^{2+} ion again shows the least pronounced tendency to form a five-membered chelate ring (0.09 and 0.36 log *K* unit for PYRU and LACT complexes, respectively). The Pb^{2+} ion in LACT complexes has the same lower chelate stabilization as the Cd^{2+} ion, but in PYRU complexes its chelatation is more pronounced (0.51 log K unit) possibly due to its large ionic radius, which enables it to coordinate with both OH groups. An OH group in the 3-position on the PROP ligand does not have any additional stabilizing effect, and 3OHP behaves in accordance to its basicity. This is quite understandable because a six-membered chelate ring should be formed, which is obviously not favorable in these systems.

An OH group as substituent in the BUTY ligand has a pronounced chelating stabilization only in the 2-position (about 1 log *K* unit), while in the 3- and 4-positions no stabilization effect was registered. Again the Cd^{2+} and Pb^{2+} ions show the least pronounced chelatation (0.23 and 0.45 log *K* unit, respectively), but the Co^{2+} and Ni^{2+} ions show a slight tendency for additional stabilization over basicity with the 3OHB ligand of 0.2 log K unit, and a destabilization of the same amount with the 4OHB ligand below that expected from basicity relationship.

To illustrate how this analysis can be useful in predicting the stability of some other substituted monoligand monocarboxylate complexes, let us try to predict, for example, the stability of methoxyacetate and ethoxyacetate complexes of Co^{2+} and Ni^{2+}. Methoxyacetates should behave like mandelate complexes having almost the same p*K*(HL) value (3.31) and having the possibility for additional chelate coordination through the oxygen atom in the methoxy group. Ethoxyacetate (p*K*(HL) = 3.51) will have a less pronounced chelating effect due to the induced attraction of electrons in the ethoxy group. Therefore, the approximate log *K*(ML) values could be obtained by taking the basicity contribution (using coefficients in Table IV for Co^{2+} and Ni^{2+}) and corresponding p*K* values for methoxy- and ethoxyacetic acids and adding about 0.76 and 0.80 log *K* unit (from Table VI) for cobalt and nickel methoxyacetates, respectively, and 0.56 and 0.60 log *K* unit for cobalt and nickel ethoxyacetates, respectively. The obtained log *K*(ML) values are 1.22 and 1.33 for cobalt and nickel methoxyacetates, which agree well with the values 1.16 and 1.29 obtained experimentally under identical experimental conditions. The calculated values for cobalt and nickel ethoxyacetates are 1.02 and 1.13, which also agree well with the experimental values of 1.05 and 1.08.[51] In conclusion, it can be said that the performed analyses show the behavior of the investigated class of weak monocarboxylate complexes, comparing the effect of metal ion and ligand on the solution stability. The factors influencing this stability are quantitatively determined. In principle, the same analysis can be performed with higher complex types, but the reliability of the data is poorer because the experimental and data evaluation errors accumulate in higher cumulative stability constants. Such an analysis can be used as a model system for any weak coordinative interaction, and the stability of many new systems can be easily predicted by knowing the general trends.

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Synthesis of New Carboxylate- and Phosphinate-Bridged Ruthenium(I) Dimers

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The carboxylate-bridged dimers of Ru(I) and Os(I) form an extensive class of derivatives with the general formula $[M(\mu\text{-}O_2CR)(CO)_2L]_2$ (R = alkyl, fluoroalkyl, or aromatic; L = tertiary phosphine,[1] acetonitrile,[1] pyrazole,[2] carbon monoxide,[3] carboxylic acid,[4,5] or pyridine[6]). Recent interest in these complexes stems in part from the discovery that they are catalyst precursors for the hydroformylation of olefins and the addition of carboxylic acids to alkynes.[7,8] Although a wealth of structural data is available for these complexes,[3,6,9,10] reports concerning their stoichiometric

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Table I. Infrared Data for New Compounds[a]

complex	ν(CO)	ν(PO) or ν(CO)		other
A	2035 s, 1972 w, 1944 s			
1a	2028 s, 1970 m, 1943 s, 1916 w	1594 m/1560 s	1420 s/1406 s	
1b	2032 s, 1969 m, 1945 s, 1911 w	1552 s	1417 s/1410 s	1596 m
1c	2029 s, 1973 m, 1944 s	1558 s	1407 s	1694 m (C═O)
1d	2024 m, 1971 m, 1945 s, 1914 w	1557 s	1423 s	1326 m
2a	2032 s, 1969 m, 1940 s, 1901 w	1151 s/1136 m	1045 m/1026 m	
2b	2028 s, 1967 m, 1935 s	1128 s	1034 s	1600 s
2c	2026 s, 1967 m, 1932 s, 1906 w	1147 m/1140 m	1045 m/1024 m	

[a] Measured in cm^{-1} as a Nujol mull between sodium chloride plates and calibrated with the 1601 cm^{-1} band of polystyrene. [b] Measured as a THF solution.

reactions are limited to the protonation of $[Os(O_2CCF_3)(CO)_2L]_2$ and multidentate phosphine substitution/oligomerization of $[Ru(O_2CCH_3)(CO)_2]_n$.[10,11] The reported syntheses of these materials invariably involve heating the respective metal carbonyls either in neat carboxylic acid or in high boiling solvents in the presence of phosphine. These conditions often lead to polymeric materials and preclude the possibility of incorporating many functional groups into these species. Herein we report (1) a simple low-temperature method for the preparation of these dimers, (2) the incorporation of functional groups into the molecule, and (3) the preparation of related phosphinate-bridged dimers by this technique.

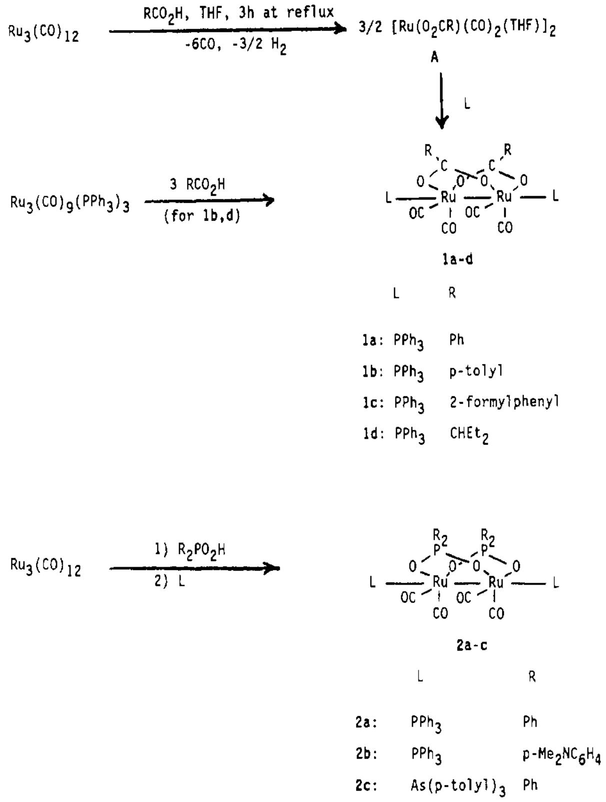
Results

Equations representing the reactions observed in this study are collected in Scheme I, and the spectroscopic data for new complexes are presented in Tables I and II. Treating $Ru_3(CO)_{12}$ with a slight excess of carboxylic acid in THF at reflux results in the formation of a red-orange solution and complete consumption of $Ru_3(CO)_{12}$ within 2 h. We formulate the product of this reaction as the dimeric species $[Ru(O_2CR)(CO)_2(THF)]_2$ (A, in Scheme I) on the basis of two experiments: (1) when the polymer $[Ru(O_2CCH_3)(CO)_2]_n$ is heated at reflux in THF for 2 h, a red-orange solution is also formed which has IR bands at 2035 s, 1972 w, and 1944 s cm^{-1}; (2) when $Ru_3(CO)_{12}$ is treated with acetic acid in THF with the conditions above, a solution with the same number, intensity, and pattern of IR bands is also obtained. With the slight excess of carboxylic acids that are present, there may also be some carboxylic acid coordination and oligomerization, as in the results of Shvo et al.[4,5] The THF ligands in A are labile, and attempts to isolate A from these solutions returns poorly soluble materials that have ν(CO) stretching bands in the IR spectra similar to those of the polymers $[Ru(O_2CR)(CO)_2]_n$ reported by Lewis and Johnson.[1]

The THF complexes can however be used directly by treating freshly prepared solutions of A with donor ligands. Thus, with phosphines or arsines, a rapid reaction ensues and within 1 h the complexes **1a–d** (Scheme I) can be isolated as bright yellow crystalline solids by recrystallization from dichloromethane/ethanol. Particularly significant is the derivative **1c** with an aldehyde ortho to the carboxylate bridging carbon. Incorporation of this substituent into the carboxylate bridge moiety allows for further condensation reactions, which in turn may allow for the construction of additional chelate rings.

It is also possible, although in slightly reduced yields, to prepare the compounds **1** in one step from the tris(triphenylphosphine)-substituted cluster $Ru_3(CO)_9(PPh_3)_3$ by using the above conditions.[12] This second approach is less general than the first in that the trisubstituted cluster must first be available and that the Lewis base is therefore fixed. The dimers **1b,d** are also prepared in very good yields when $Ru_3(CO)_{12}$ is heated to 100 °C in neat *p*-tolylaldehyde or in Et_2CHCHO. The nature of the aldehyde oxidation and the source of the oxygen are not clear because the reactions were performed under inert conditions by using standard Schlenk techniques and the aldehydes were dried, distilled, and stored under nitrogen. A possible explanation is the metal-catalyzed disproportionation[13] of the aldehydes into the corresponding alcohols and carboxylic acids. One interpretation of these results is that the formation of carboxylate-bridged dimers and polymers represents a pathway for catalyst poisoning of hydroformylation catalysts that are based on ruthenium clusters.

Scheme I. Preparation of Carboxylate and Phosphinate Dimers



Phosphinate-bridged dimers are also readily prepared with the above conditions; cf. Scheme I. The complexes **2a–c** are isolated as air-stable yellow crystalline solids in very good yields. The structure of these dimers is probably directly comparable with that known for the carboxylates. There is therefore enough flexibility and separation between the bridges to accommodate the aromatic rings pointing inward from the phosphorus atoms.

The IR and NMR data for the new compounds are presented in Tables I and II. Particularly significant is the shift in the ν(CO) bands less than 2000 cm^{-1} to lower frequencies for the phosphinate species **2a–c** as compared to the carboxylate species **1a–d**. This can be interpreted as arising from better donation, and thus stronger bridges, by the phosphinate ligand or by reduced π-bonding between the bridges and the metal. The P–O stretching

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Table II. NMR Data for New Compounds[a]

1a	7.1–7.8 (m, C_6H_5)
1b	7.7 (m, C_6H_5), 7.01 (d, 2, $^3J_{HH} = 8.1$, C_6H_4), 6.82 (d, 2, $^3J_{HH} = 8.1$, C_6H_4), 2.26 (s, 3, CH_3)
1c	7.45 (m, C_6H_5), 9.89 (s, 1, CHO), 7.71 (d, 2, $^3J_{HH} = 7.5$, C_6H_4),[b] 6.98 (d, 2, $^3J_{HH} = 7.2$, C_6H_4)
1d	7.5 (m, C_6H_5), 1.84 (p, 1, $^3J_{HH} = 6.6$, $CHEt_2$), 1.14 (dq, 4, $^3J_{HH} = 6.6$ and 7.4, CH_2), 0.60 (t, 6, $^3J_{HH} = 7.4$, CH_3)
2a	7.5 (m, C_6H_5), 7.24 (d, $J = 7.1$)[b], 7.18, 7.15 (d, $J = 3.4$), 7.05 (t, $J = 5.8$), 7.00 (m), 6.97 (dd, $J = 5.1$ and 7.3)
2b	7.5 (m, C_6H_5), 7.14 (dd, 4, $^3J_{HP} = 9.8$, $^3J_{HH} = 6.0$, C_6H_4), 6.95 (dd, 4, $^3J_{HP} = 10.0$, $^3J_{HH} = 6.0$, C_6H_4), 6.44 (d, 4, $^3J_{HH} = 6.5$, C_6H_4), 6.29 (d, 4, $^3J_{HH} = 6.2$, C_6H_4), 2.93 (S, 12, CH_3), 2.92 (s, 12, CH_3)
2c	7.55 (d, 12, $^3J_{HH} = 8.1$, AsC_6H_4), 7.36, 7.21 (m, 4, $P(C_6H_5)_2$), 7.15 (d, 12, $^3J_{HH} = 7.8$, AsC_6H_4), 6.99, 6.94 (m, 4, $P(C_6H_5)_2$), 2.3 (s, 18, CH_3)

[a] Measured at 25 °C in $CDCl_3$; all chemical shifts (δ) are given in ppm with respect to internal TMS, and coupling constants are given in Hz. [b] Some peaks obscured by the triphenylphosphine multiplet.

modes for the phosphinate bridges are split, and two bands are seen for the asymmetric and symmetric modes, which probably originates from solid-state crystal splitting. The splitting in **2b** was obscured by the broadness of these bands.

Conclusion

The preparations described here represent a convenient, and probably general, entry into dimers of Ru(I) and possibly Os(I). The formation of **1c** has shown that reactive groups on the bridging ligand can easily be introduced. The hitherto unknown phosphinate dimers are a new class of reactive compounds. The dimers may be good starting materials for other M(I) complexes, the chemistry of which is at present very poorly understood in comparison to that of the M(II) and M(0) oxidation states.[2,14]

Experimental Section

The reactions described were performed under nitrogen with standard Schlenk techniques. However the air-stable products were recrystallized under open conditions by slow evaporation of the specified solvent pairs.

$[Ru_2(O_2CPh)_2(CO)_4(PPh_3)_2]$ (1a). $Ru_3(CO)_{12}$, (0.1 g, 0.16 mmol) and an excess of benzoic acid (0.096 g, 1.27 mmol) were heated under nitrogen in 20 mL of THF for 2 h. The mixture was briefly cooled, triphenylphosphine (0.12 g, 0.46 mmol) was added, and the resultant mixture was heated at reflux for a further 30 min. The resulting bright yellow solution was cooled, THF was removed in vacuo, and the yellow oil was recrystallized from dichloromethane/ethanol to give 0.165 g, 65%, of **1a** as yellow cubes, mp 267–275 °C. Anal. Calcd for $C_{54}H_{40}O_8P_2Ru_2\cdot{}^1/_2CH_2Cl_2$: C, 58.23; H, 3.68. Found: C, 57.76; H, 3.56. The presence of dichloromethane solvate was confirmed by ^1H NMR spectroscopy.

$[Ru_2(O_2C\text{-}p\text{-tolyl})_2(CO)_4(PPh_3)_2]$ (1b). From $Ru_3(CO)_{12}$. Compound **1b** was prepared in 63% yield as described for **1a**; mp 257–258 °C. Anal. Calcd for $C_{56}H_{44}O_8P_2Ru_2\cdot{}^1/_4CH_2Cl_2$: C, 59.77; H, 3.98. Found: C, 59.43; H, 3.34. The presence of dichloromethane solvate was confirmed by ^1H NMR spectroscopy.

From $Ru_3(CO)_9(PPh_3)_3$. $Ru_3(CO)_9(PPh_3)_3$ (0.05 g, 0.04 mmol) and 3 mL (ca. 25 mmol) of freshly distilled 4-methylbenzaldehyde were heated at 105 °C for 2 h, during which time the purple solution became a yellow oil. The mixture was then cooled, and the volatile materials were stripped off in vacuo. Recrystallization from dichloromethane/ethanol returned 0.04 g, 65%, of **1b**.

$[Ru_2\{O_2CC_6H_4CHO\}_2(CO)_4(PPh_3)_2]$ (1c). $Ru_3(CO)_{12}$ (0.078 g, 0.12 mmol) and an excess of 2-carboxybenzaldehyde (0.108 g, 0.72 mmol) were heated at reflux for 4 h. Triphenylphosphine (0.12 g, 0.46 mmol) was then added and the reaction mixture heated a further 20 min. Recrystallization from dichloromethane/ethanol returned 0.12 g, 59%, of yellow cubes, mp 216–220 °C. Anal. Calcd for $C_{56}H_{40}O_{10}P_2Ru_2\cdot{}^1/_2CH_2Cl_2$: C, 57.53; H, 3.51. Found: C, 57.96; H, 3.69. The presence of dichloromethane solvate was confirmed by ^1H NMR spectroscopy.

$[Ru_2(O_2CCHEt_2)_2(CO)_4(PPh_3)_2]$ (1d). From $Ru_3(CO)_{12}$ and 2-Ethylbutyric Acid. Compound **1d** was prepared as above from $Ru_3(CO)_{12}$ (0.30 g, 0.47 mmol), excess 2-ethylbutyric acid (1 mL, ca. 8 mmol), and triphenylphosphine (0.12 g, 0.46 mmol). Recrystallization yielded 0.65 g, 86%, of yellow cubes, mp 206–208 °C. Anal. Calcd for $C_{52}H_{52}O_8P_2Ru_2$: C, 58.41; H, 4.91. Found: C, 58.35; H, 4.74.

From $Ru_3(CO)_{12}$ and 2-Ethylbutyraldehyde. $Ru_3(CO)_{12}$ (0.075 g, 0.12 mmol) and 3 mL (ca. 25 mmol) of freshly distilled 2-ethylbutyraldehyde were heated at reflux, 117 °C, for 4 h. The resulting red solution was cooled and stripped to dryness in vacuo. Triphenylphosphine (0.092 g, 0.35 mmol) and 20 mL of ethanol were then added. The resulting yellow suspension was heated at reflux for an additional hour, cooled, and filtered. The solid was then washed with ethanol and *n*-hexane, to give 0.153 g, 85%, of **1d**.

From $Ru_3(CO)_9(PPh_3)_3$. $Ru_3(CO)_9(PPh_3)_3$ (0.056 g, 0.04 mmol) and 2 mL (ca. 15 mmol) of freshly distilled 2-ethylbutyraldehyde were heated at reflux for 1 h, during which time the purple suspension became a yellow oil. The mixture was then cooled, and the volatile materials were stripped off in vacuo. Recrystallization from dichloromethane/ethanol returned 0.039 g, 60%, of **1d**.

$[Ru_2(O_2PPh_2)_2(CO)_4(PPh_3)_2]$ (2a). $Ru_3(CO)_{12}$ (0.06 g, 0.09 mmol) and diphenylphosphinic acid (0.123 g, 0.56 mmol) were heated at reflux for 14 h. The solution was then briefly cooled, triphenylphosphine (0.065 g, 0.24 mmol) was added, and this mixture was heated at reflux for an additional hour. The resulting yellow solution was cooled, and THF was removed in vacuo. Recrystallization of the oily residue from dichloromethane/ethanol returned 0.122 g, 68%, of **2a**, mp 244–246 °C. Anal. Calcd for $C_{64}H_{50}O_8P_4Ru_2$: C, 60.38; H, 3.96. Found: C, 59.80; H, 3.92.

$[Ru_2\{O_2P(p\text{-}Me_2NC_6H_4)_2\}_2(CO)_4(PPh_3)_2]$ (2b). $Ru_3(CO)_{12}$ (0.043 g, 0.07 mmol), bis(*p*-(dimethylamino)phenyl)phosphinic acid (0.123 g, 0.40 mmol), and triphenylphosphine (0.293 g, 1.12 mmol) were utilized as described above for **2a**. Yellow crystals (0.11 g, 75%; mp 236–242 °C) were obtained after recrystallization from dichloromethane/ethanol. Anal. Calcd for $C_{72}H_{70}N_4O_8P_4Ru_2$: C, 59.63; H, 4.83; N, 3.88. Found: C, 59.83; H, 4.88; N, 3.68.

$[Ru_2(O_2PPh_2)_2(CO)_4\{As(p\text{-tolyl})_3\}_2]$ (2c). This preparation is identical with that of **2a** except that 0.131 g (0.38 mmol) of tri-*p*-tolylarsine was added instead of triphenylphosphine. Workup as for **2a** returned 0.13 g, 64%, of yellow crystals, mp 240–248 °C. Anal. Calcd for $C_{70}H_{62}As_2O_8P_2Ru_2$: C, 58.18; H, 4.32. Found: C, 57.78; H, 4.27.

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Synthesis and Structure of the Trinuclear Palladium Cluster $[Pd_3(PEt_3)_3(\mu_2\text{-}NPh)_2(\mu_2\text{-}NHPh)]Cl$, Containing Bridging Imido and Amido Ligands

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The chemistry of nitrene or imido ligands[1] coordinated to electron-rich metals has been little developed, whereas much is known about the chemistry of such ligands coordinated to electron-deficient early transition metals.[2] Such species are of possible importance as intermediates in metal-catalyzed reductions of nitroaromatics.[3] We recently used the tetrazenido dianion, I, $[RNN{=}NNR]^{2-}$, to synthesize main-group and transition-metal metallacyclotetrazene complexes.[4] Although palladium is the

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