was done by extraction with THF (20 mL) followed by vacuum drying, dissolution in water (10 mL) and addition of NaCl (20% w/v, 7.5 mL), and, after overnight standing, filtration of a small precipitated impurity, followed by cautiously layering on the clear solution of CsCl (3% w/v, 20 mL). After complete diffusion (several weeks), the precipitated pure $Cs_3[Pt_2Rh_{11}(CO)_{24}]$ was filtered and washed with CsCl (3% w/v, 10 mL). The clear mother liquor and collected washings were treated with CH_3COOH (4 mL) to give after about 3 h a fine crystalline precipitate. After the mixture was allowed to stand overnight, $Cs_4[Pt_4Rh_{18}(CO)_{35}]$ was separated from the mother liquor by decantation and vacuum dried. Yields: 100–150 mg (3–4%). The product gave no hydrido signals in ^1H NMR.

(b) Synthesis as a Byproduct of the Synthesis of $[Pt_2Rh_9(CO)_{22}]^{3-}$. $[PPN][PtRh_5(CO)_{15}]$ (1.143 g, 0.685 mmol) and $[PPN]_2[PtRh_4(CO)_{14}]$ (1.405 g, 0.696 mmol) were dissolved under nitrogen in 25 mL of CH_3CN. The mixture was stirred at room temperature, until the IR spectrum did not change further (3–4 days). $NaBPh_4$ (0.733 g, 2.14 mmol) was added, and the solution was vacuum dried; the residue was redissolved in 5 mL of acetone and again vacuum dried. The residue was redissolved in water (20 mL) and filtered to remove the white precipitate of $[PPN]BPh_4$, which prior to being discarded was washed with a few milliliters of water. The solution with the collected washings (final volume 28 mL) was treated with 28 mL of saturated KBr solution and stirred for 3 h and then filtered to remove this first fraction of potassium salts. The filtered solution was concentrated in vacuum to a volume of 26 mL, reaching KBr saturation, and stirred overnight. This second fraction, containing $K_3[Pt_2Rh_9(CO)_{22}]$, was filtered, washed with saturated KBr, and vacuum dried. Further purification of this fraction was made by extraction with THF (20 mL) (to separate the excess KBr), followed by vacuum drying. The residue was redissolved in water (5 mL) and NaCl (20% w/v, 5 mL) and stirred overnight; this induced precipitation of a small amount of product, which was recovered by filtration and vacuum-dried. This precipitate was extracted with THF (5 mL) to give a solution which was evaporated to dryness in vacuum and the residue redissolved in MeOH (2 mL); cautious layering of a solution of NEt_4Br in 2-propanol (3 mg/mL) yielded, when the diffusion was completed, crystals which did not show a diffraction pattern. Further recrystallization from acetone/2-propanol yielded a few milligrams (10–20 mg, ca. 1%) of the acetone-solvated $[NEt_4]_4[Pt_4Rh_{18}(CO)_{35}]\cdot$acetone, in crystals suitable for X-ray diffraction.

X-ray Analysis. A summary of crystal data is reported in Table II. A crystal sample of 0.35 × 0.30 × 0.20 mm was mounted, on a glass fiber in air at room temperature, on an Enraf Nonius CAD4 diffractometer; graphite-monochromatized Mo Kα radiation was used with the generator operated at 55 kV and 25 mA. The cell parameters and the orientation matrix were obtained from a least-squares refinement of the setting angles of 25 randomly distributed intense reflections having $16 < 2\theta < 25°$. The intensity data were collected using the ω-scan technique within the limits $6 < 2\theta < 50°$, in the $+h,+k,+l$ octant. A variable scan speed (from 1 to 20°/min) and a variable scan range ($0.9 + 0.35 \tan \theta$) were used, with a 25% extension at each side of the peaks for background determination. Maximum time per scan was 50 s. The monitoring of the intensity of three intense reflections allowed correction for crystal decay, which was estimated about 13% at the end of the data collection. Lorentz, polarization, and absorption corrections were applied. The latter was applied to the reduced intensities based on ψ scans (ψ = 0–360°, every 10°)[16] of three reflections having χ values close to 90°; the minimum relative transmission factor was 0.41.

The structure was solved by direct methods and conventional difference Fourier techniques and refined with full-matrix least-squares methods on the basis of 4765 independent reflections having $I > 2\sigma(I)$; anisotropic temperature factors were assigned to all metal atoms; the hydrogen atom contributions were neglected. Scattering factors for neutral atoms were taken from the SDP package database;[17] anomalous scattering factors were taken from ref 18. The handness of the crystal was determined by refining the two enantiomeric models. The final conventional agreement indices R and R_w were 0.043 and 0.044, respectively, for the best enantiomer, while the other gave $R = 0.071$ and $R_w = 0.079$. The final difference Fourier map showed a maximum peak of 1.19 e/Å^3 in proximity of the Pt3 atom. All computations were performed on a PDP 11/73 microcomputer using the SDP package.[17] The final positional parameters are given in Table III.

Supplementary Material Available: Listings of crystal data (Table S1), atomic coordinates and isotropic thermal parameters (Table S2), anisotropic thermal parameters (Table S3), and bond distances and angles (Table S4) (24 pages); a listing of observed and calculated structure factor moduli (Table S5) (33 pages). Ordering information is given on any current masthead page.

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(16) North, A. C.; Phillips, D. C.; Scott Mathews, F. *Acta Crystallogr.* **1968**, *A24*, 351.

(17) Frenz, B. A. and Associates. SDP Plus Version 1.0, Enraf Nonius, 1980.

(18) Cromer, D. T. *International Tables for X-Ray Crystallography*; Kynoch Press: Birmingham, England 1974; Vol. 4, Tables 2.3.1.

Additions and Corrections

1991, Volume 30

Fausto Calderazzo,* Alberto Juris, Rinaldo Poli, and Fausto Ungari: Reactivity of Molecules Containing Element–Element Bonds. 2. Transition Elements.

Page 1276. In Table II, the correct carbonyl stretching vibrations of $CrCp(AsPh_2)(CO)_3$ are as follows (cm^{-1}; printed values in parentheses): 1998 s (1988 s), 1934 m (1934 m), 1929 s (1989 s).—Fausto Calderazzo

Antonio Belforte, Fausto Calderazzo,* Ulli Englert, and Joachim Strähle: Deoxygenation of Carbon Dioxide to Diethylformamide in the $Zn/HNEt_2/CO_2$ System. Crystal and Molecular Structure of $[Zn_4O(O_2CNEt_2)_6]$.

Pages 3780 and 3781. The last line of text on p 3780 and the first line on p 3781 should be modified as follows: "The X-ray crystal structure is available for $Zn_4O(O_2CMe)_6$,[24a,b] whose absorption and emission spectra were recently described;[24c]" Reference 24 should be modified as follows: (a) Koyama, H.; Saito, Y. *Bull. Chem. Soc. Jpn.* **1954**, *27*, 112. (b) Hiltunen, L.; Leskelä, M.; Mäkelä, M.; Niinistö, L. *Acta Chem. Scand.* **1987**, *A41*, 548. (c) Kunkely, H.; Vogler, A. *J. Chem. Soc., Chem. Commun.* **1990**, 1204.—Fausto Calderazzo