$\left[\mathrm{Cu}(\mathrm{Hdmg})_{2}(\mathrm{im})\right]$ (Morehouse, Polychronopoulou \& Williams, 1980), $\left[\mathrm{Cu}(\mathrm{Hdpg})_{2}\left(\mathrm{H}_{2} \mathrm{O}\right)\right]$ (Falk, Ivanova, Roos \& Vänngard, 1970) (Hdmg - dimethylglyoximate, im - imidazole, Hdpg - dipropylglyoximate, im - imidazole, Hdpg - dipropylglyoximate) and $\left[\mathrm{Cu}_{2}(\mathrm{Hdmg})_{4}\right]$ (Vaciago \& Zambonelli, 1970) .

The $\mathrm{Cu}-\mathrm{S}$ distance of 2.941 (1) $\AA$ is similar to that found in the complex $\left[\mathrm{Cu}(\mathrm{pic})_{2}(\mathrm{tu})_{2}\right]$ [2.943 (1) $\AA$, pic - picolinate] (Ferrari, Capacchi, Fava, Montenero \& Nardelli, 1972) where the thiourea molecules are oriented almost parallel to the equatorial plane, in contrast with the present complex where the angle between the equatorial and thiourea planes is $81.3^{\circ}$.

The intramolecular $\mathrm{O}-\mathrm{H}-\mathrm{O}$ hydrogen bonds bridging the dioxime ligands are asymmetric $[\mathrm{O}(1) \cdots \mathrm{O}(2)=2.656(6), \quad \mathrm{O}(2)-\mathrm{H}(15)=0.79(8)$, $\mathrm{O}(1) \cdots \mathrm{H}(15)=1.87(8) \AA]$. The $\mathrm{O}(1)$ atom is also involved both in a second intramolecular hydrogen bond $[\mathrm{O}(1) \cdots \mathrm{N}(2)=3.089(5) \AA]$ and in two intermolecular hydrogen bonds with thiourea ligands.

Hexacoordination has not previously been structurally confirmed for copper dioximate complexes, although it has been assumed for a water adduct (Falk, Ivanova, Roos \& Vänngard, 1970); otherwise only pentacoordinate complexes have been reported. The complex $\left[\mathrm{Cu}(\mathrm{hd})_{2}(\mathrm{tu})_{2}\right]$ thus represents a new structural type among compounds of this class.

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# Structure of Two Crystalline Forms of $\mu$-(1,2-Phenylenediaminato- $\left.N, N, N^{\prime}, N^{\prime}\right)$ -bis[dicarbonyl(triphenylphosphine)ruthenium(I)](Ru-Ru) $\left[\left\{\mathrm{Ru}(\mathrm{CO})_{2}\left[\mathrm{P}\left(\mathrm{C}_{6} \mathrm{H}_{5}\right)_{3}\right]_{2^{-}}\right.\right.$ $\left.\left\{\boldsymbol{\mu}-\mathbf{1 , 2} \mathbf{- C}_{6} \mathrm{H}_{\mathbf{4}}(\mathrm{NH})_{2}\right\}\right]$ 

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#### Abstract

Ru}(\mathrm{CO})_{2}\left[\mathrm{P}_{2}\left(\mathrm{C}_{6} \mathrm{H}_{5}\right)_{3}\right]_{2}\left\{\mu-1,2-\mathrm{C}_{6} \mathrm{H}_{4}(\mathrm{NH})_{2}\right\}\right]\right.\).${ }_{3}^{2} \mathrm{C}_{7} \mathrm{H}_{8}(A), \quad \mathrm{C}_{46} \mathrm{H}_{36} \mathrm{~N}_{2} \mathrm{O}_{4} \mathrm{P}_{2} \mathrm{Ru}_{2}{ }_{2}^{2} \mathrm{C}_{7} \mathrm{H}_{8}, \quad M_{r}=1006 \cdot 3$, monoclinic, $P 2_{1} / c, a=10.040$ (4), $b=17.802$ (2), $c=$ 27.279 (9) $\AA, \beta=98.26$ (3) ${ }^{\circ}, V=4825$ (3) $\AA^{3}, Z=4$, $D_{x}=1.39 \mathrm{~g} \mathrm{~cm}^{-3}$, Mo $K \alpha$ raditation (graphite crystal monochromator, $\lambda=0.71073 \AA$ ), $\mu($ Mo K $\alpha)=$ $7.21 \mathrm{~cm}^{-1}, F(000)=2037, T=293 \mathrm{~K}$. Final conventional $R$ factor $=0.049$ for 3908 'observed' reflections and 512 variables. $\left[\left\{\mathrm{Ru}(\mathrm{CO})_{2}\left[\mathrm{P}\left(\mathrm{C}_{6} \mathrm{H}_{5}\right)_{3}\right]\right\}_{2}-\right.$ $\left.\left\{\mu-1,2-\mathrm{C}_{6} \mathrm{H}_{4}(\mathrm{NH})_{2}\right\}\right\} . \mathrm{C}_{9} \mathrm{H}_{12}(B), \mathrm{C}_{46} \mathrm{H}_{36} \mathrm{~N}_{2} \mathrm{O}_{4} \mathrm{P}_{2} \mathrm{Ru}_{2}$.$\mathrm{C}_{9} \mathrm{H}_{12}, \quad M_{r}=1065 \cdot 1, \quad$ monoclinic, $\quad P 2_{1} / m, \quad a=$ 9.9994 (1),$\quad b=25.477$ (2), $\quad c=10 \cdot 515$ (2) $\AA, \quad \beta=$


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$114.51(1)^{\circ}, \quad V=2437.4(5) \AA^{3}, \quad Z=2, \quad D_{x}=$ $1.45 \mathrm{~g} \mathrm{~cm}^{-3}$, Mo $K \alpha$ radiation (graphite crystal monochromator, $\quad \lambda=0.71073 \AA), \quad \mu($ Mo $K \alpha)=$ $7 \cdot 17 \mathrm{~cm}^{-1}, F(000)=1084, T=293 \mathrm{~K}$. Final conventional $R$ factor $=0.036$ for 5414 'observed' reflections and 332 variables. The above structures correspond to two different crystalline forms (depending on the solvent) adopted by the compound previously formulated as $\left[\mathrm{Ru}_{2}\left\{\mu-1,2-\mathrm{C}_{6} \mathrm{H}_{4}\right.\right.$ $\left.\left.(\mathrm{NH})_{2}\right\}(\mathrm{CO})_{2}\left(\mathrm{PPh}_{3}\right)_{2}\right](C)$. The asymmetric unit contents are an entire formula unit in structure $(A)$ and a half formula unit in structure ( $B$ ). The $\mathrm{Ru}-\mathrm{Ru}$ lengths found in both cases are equal and significantly short ( $2 \cdot 56 \AA$ ).
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Introduction. When we studied the reactivity of compound (C) $\left[\mathrm{Ru}_{2}\left\{\mu-1,2-\mathrm{C}_{6} \mathrm{H}_{4}(\mathrm{NH})_{2}\right\}(\mathrm{CO})_{2}\left(\mathrm{PPh}_{3}\right)_{2}\right]$ (Anillo, Riera, Obeso-Rosete, Font-Altaba \& Solans, 1987), we found that it showed unexpected behaviour for a molecule with a triple $\mathrm{Ru}-\mathrm{Ru}$ bond as proposed on the basis of its previous structural analysis by X-ray diffraction. An assumed derivative of compound ( $C$ ), of which the crystal structure $(A)$ was determined by X-ray diffraction methods, gave the formula $\left[\mathrm{Ru}_{2}\left\{\mu-1,2-\mathrm{C}_{6} \mathrm{H}_{4}(\mathrm{NH})_{2}\right\}(\mathrm{CO})_{4}\left(\mathrm{PPh}_{3}\right)_{2}\right]$, with molecular parameters analogous to those previously found for compound ( $C$ ). This fact led us to the idea that both might be the same compound ( $C$ ) which was kept unreacted and used later as a sample for its structural characterization. The new formula is also supported by the mass spectrum, and its infrared spectrum is also better understood (four CO ligands) as for a unique isomer and not an isomeric syn-anti mixture in solution; the chemical reactivity also suggests this formula with a simple $\mathrm{Ru}-\mathrm{Ru}$ bond. As we had some crystals from the original crystallization of compound ( $C$ ) we decided to redetermine its structure by X-ray diffraction to compare it with that of compound $(A)$, which resulted in structure ( $B$ ) which crystallizes in a different space group but with the same molecular geometry as $(A)$. The different crystalline habits showed by $(A)$ and $(B)$ are related to the use of different solvents in each case, either toluene, $(A)$, or mesitylene, $(B)$, leading to different space groups.

Experimental. A new method to obtain compound (C) with a better yield is as follows: A mixture of $\left[\mathrm{Ru}(\mathrm{CO})_{3}\left(\mathrm{PPh}_{3}\right)_{2}\right](1 \mathrm{~g})$ and $1,2-\mathrm{C}_{6} \mathrm{H}_{4}\left(\mathrm{NH}_{2}\right)_{2}(0.15 \mathrm{~g})$ is refluxed in mesitylene $\left(10 \mathrm{~cm}^{3}\right)$ for 1 h . The reaction solution is left at room temperature and after one day yellow crystals are collected, washed twice with hexane and dried in vacuo. Yield ca $80 \%$. A purer compound can be obtained by dissolving the former crystals in toluene and chromatographing the solution through $\mathrm{Al}_{2} \mathrm{O}_{3}$ (activity II), rejecting the first pale fraction $\{$ in which some unreacted $\left[\mathrm{Ru}(\mathrm{CO})_{3}\left(\mathrm{PPh}_{3}\right)_{2}\right]$ is usually present $\}$ and collecting the yellow fraction eluted with toluene. Further crystals can be formed by adding hexane at room temperature.
The procedure to obtain the crystals employed in the previous structural determination of $(C)$ is: A mixture of $\left[\mathrm{Ru}\left\{\mathrm{C}_{6} \mathrm{H}_{4}(\mathrm{NH})_{2}\right\}\left(\mathrm{PPh}_{3}\right)_{3}\right] \quad(0.1 \mathrm{~g})$ and $\mathrm{Cr}(\mathrm{CO})_{6}$ (excess) is refluxed in mesitylene ( $3 \mathrm{~cm}^{3}$ ) for 1 h . The reaction solution is chromotographed $\left(\mathrm{Al}_{2} \mathrm{O}_{3}\right.$, activity II) and the first yellow fraction eluted with toluene [in which the compound $(C)$ is detected]; by adding hexane to this solution yellow crystals are obtained. Analysis: Found C, 60.62; H, $4 \cdot 35$; N, $2 \cdot 60 \%$; calculated for $\mathrm{C}_{46} \mathrm{H}_{36} \mathrm{~N}_{2} \mathrm{O}_{4} \mathrm{P}_{2} \mathrm{Ru}_{2}$.$\mathrm{C}_{9} \mathrm{H}_{12}(B): \mathrm{C}, 62 \cdot 02 ; \mathrm{H}, 4 \cdot 54 ; \mathrm{N}, 2 \cdot 63 \% ;{ }^{1} \mathrm{H}$ NMR

Table 1. Experimental data

|  | (A) | (B) |
| :---: | :---: | :---: |
| Crystal colour | Brownish yellow | Yellow |
| Crystal shape | Nearly cubic | Prismatic |
| Crystal size (mm) | $0.2 \times 0.2 \times 0.2$ | $0.4 \times 0.2 \times 0.1$ |
| Drift correction | 0.996 to 1.025 | 0.98 to 1.01 |
| $\theta$ limits ( ${ }^{\circ}$ ) | 1-25 | 0-30 |
| $h, k, l$ range | $\begin{array}{r} -11,0,0 \text { to } \\ 11,21,32 \end{array}$ | $\begin{gathered} -14,0,0 \text { to } \\ 14,35,14 \end{gathered}$ |
| Number of measured reflections | 8974 | 7616 |
| Number of unique reflections | 8462 | 7251 |
| $R_{\text {int }}$ (for some doubly measured reflections) | 0.013 | 0.018 |
| Observed reflections $I>3 \sigma(I)$ | 3908 | 5414 |
| Number of refined parameters | 512 | 332 |
| $R, w R$ | $0.049,0.050$ | $0.032,0.036$ |
| $g$ value | 0.001 | 0.0002 |
| Shift/e.s.d. (max. ratio) | 1.782 | 0.040 |
| $\Delta \rho\left(\mathrm{e} \AA^{-3}\right)$ maximum | 1.14 | 0.45 |
| $\Delta \rho\left(\mathrm{e} \AA^{-3}\right)$ minimum | -0.63 | -0.45 |

$\Delta \rho\left(\mathrm{e} \AA^{-3}\right)$ minimum
( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}, 295 \mathrm{~K}$ ) $\delta=5.21(\mathrm{~m})$ and 4.83 (m) $\quad\left(4 \mathrm{H}, \quad \mathrm{C}_{6} \mathrm{H}_{4}\right.$ diimine), $\quad 2.27 \quad\left(s, \quad 9 \mathrm{H}, \quad \mathrm{CH}_{3}\right.$ mesitylene); ${ }^{31} \mathrm{P}\left\{{ }^{1} \mathrm{H}\right\}$ NMR ( $300 \mathrm{MHz}, \mathrm{CDCl}_{3}$, 295 K ) $\delta=28.03(s)$; IR (toluene, $\left.\mathrm{cm}^{-1}\right) \nu(\mathrm{CO}) 2002$, 1968, 1931. FAB mass spectrum: $m / e$ ratio: 946 $\left(M^{+}\right), 918\left(M^{+}-\mathrm{CO}\right), 889 \quad\left(M^{+}-2 \mathrm{CO}\right), 861$ ( $\left.M^{+}-3 \mathrm{CO}\right), 832\left(M^{+}-4 \mathrm{CO}\right)$.

Experimental data for both structures $(A)$ and $(B)$ are collected in Table 1.
Enraf-Nonius CAD-4 single-crystal diffractometer, Mo $K \alpha$ radiation, graphite crystal monochromator. Unit-cell dimensions were determined from the angular settings of 25 reflections with $20<$ $\theta<30^{\circ}$. Space group from systematic absences ( $A$, $B)$ and structure determination (B). $\omega-2 \theta$ scan technique with a variable scan rate and a maximum scan time of 60 s per reflection. Intensity checked by monitoring three standard reflections every 60 min . Drift corrections. Profile analysis (Lehmann \& Larsen, 1974; Grant \& Gabe, 1978) and empirical absorption correction using $\psi$ scans (North, Phillips \& Mathews, 1968) (correction factors 0.90 to 1.00 ) were applied to all the reflections of $(B)$.
Lorentz and polarization corrections were applied and data reduced to $\left|F_{o}\right|$ values. The structures were solved by Patterson method using the program SHELXS86 (Sheldrick, 1985) and Fourier synthesis. Isotropic and anisotropic least-squares refinements using SHELX76 (Sheldrick, 1976), followed by a difference Fourier synthesis allowed the location of some hydrogen atoms; the rest of the hydrogen atoms were geometrically placed. Positional and anisotropic thermal parameters of the non-hydrogen atoms were refined. Hydrogen atoms were geometrically placed and the overall isotropic temperature factors refined, except some of those belonging to molecule ( $B$ ), for which positional parameters were refined. Function minimized $\sum w\left(F_{o}-F_{c}\right)^{2}, w=1 /$ $\left[\sigma^{2}\left(F_{o}\right)+g F_{o}^{2}\right]$ with $\sigma\left(F_{o}\right)$ from counting statistics ( $g$ in Table 1). Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV). The plot was drawn with the PLUTO program

Table 2. Fractional positional and thermal parameters (with e.s.d.'s)

| $U_{\mathrm{eq}}=\frac{1}{3} \sum_{i} \sum_{j} U_{i j} a_{i}^{*} a_{j}^{*} \mathbf{a}_{i} \cdot \mathbf{a}_{j}$. |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: |
|  | $x$ | $y$ | $z$ | $U_{\text {eq }}\left(\AA^{2} \times 10^{2}\right)$ |
| (A) |  |  |  |  |
| Rul | 0.50550 (7) | 0.17836 (4) | 0.55076 (3) | 3.68 (3) |
| Ru2 | 0.46214 (8) | 0.23936 (4) | 0.46504 (3) | 3.76 (3) |
| P1 | 0.6564 (3) | 0.1388 (1) | 0.6208 (1) | 3.77 (9) |
| P2 | $0 \cdot 5407$ (2) | 0.2945 (2) | 0.3953 (1) | 3.96 (9) |
| N1 | 0.5880 (7) | 0.2843 (5) | 0.5292 (3) | 4.0 (3) |
| N2 | 0.6219 (7) | 0.1620 (4) | 0.4919 (3) | 3.5 (3) |
| Cl | 0.727 (1) | 0.2713 (6) | 0.5247 (3) | 4.0 (3) |
| C2 | 0.746 (1) | $0 \cdot 2014$ (5) | 0.5024 (3) | 4.0 (3) |
| C3 | 0.873 (1) | $0 \cdot 1780$ (6) | 0.4962 (4) | 4.9 (4) |
| C4 | 0.982 (1) | 0.2257 (8) | 0.5110 (4) | 6.6 (5) |
| C5 | 0.964 (1) | $0 \cdot 2926$ (8) | 0.5313 (4) | 6.7 (5) |
| C6 | 0.835 (1) | 0.3174 (6) | 0.5390 (4) | 5.9 (4) |
| C11 | 0.414 (1) | 0.0861 (7) | 0.5448 (4) | 4.8 (4) |
| 011 | 0.363 (1) | 0.0298 (5) | 0.5400 (3) | 7.7 (4) |
| C12 | 0.378 (1) | 0.2192 (6) | 0.5859 (4) | 5.3 (4) |
| O 2 | 0.301 (1) | 0.2470 (5) | 0.6079 (3) | 7.6 (4) |
| C21 | 0.348 (1) | 0.1710 (6) | 0.4282 (4) | 5.0 (4) |
| 021 | 0.275 (1) | 0.1295 (4) | 0.4068 (3) | 8.1 (4) |
| C22 | $0 \cdot 323$ (1) | $0 \cdot 3056$ (6) | 0.4674 (4) | 4.9 (4) |
| 022 | $0 \cdot 238$ (1) | 0.3475 (5) | 0.4706 (4) | 8.5 (4) |
| C31 | 0.588 (1) | $0 \cdot 0658$ (6) | 0.6575 (4) | 4.9 (4) |
| C32 | 0.651 (1) | -0.0040 (7) | 0.6694 (5) | 7.2 (5) |
| C33 | 0.593 (1) | -0.0575 (8) | 0.6962 (5) | 7.9 (6) |
| C34 | 0.475 (2) | -0.0412 (9) | 0.7125 (5) | 8.6 (6) |
| C35 | 0.409 (1) | 0.0271 (9) | 0.7030 (5) | 8.1 (6) |
| C36 | $0 \cdot 463$ (1) | 0.0809 (8) | 0.6738 (5) | 7.6 (5) |
| C41 | 0.812 (1) | 0.0982 (6) | $0 \cdot 6062$ (4) | 4.5 (4) |
| C42 | 0.940 (1) | 0.1285 (7) | 0.6246 (4) | 6.3 (5) |
| C43 | 1.056 (1) | 0.0955 (9) | 0.6114 (5) | 7.6 (6) |
| C44 | 1.045 (1) | 0.0333 (8) | 0.5786 (6) | $8 \cdot 2$ (6) |
| C45 | 0.918 (1) | 0.0052 (7) | 0.5602 (5) | 6.8 (5) |
| C46 | 0.806 (1) | 0.0375 (6) | 0.5738 (4) | 5.0 (4) |
| C 51 | 0.714 (1) | $0 \cdot 2085$ (6) | 0.6681 (4) | 4.4 (4) |
| C52 | 0.769 (1) | 0.1864 (7) | 0.7161 (4) | 5.5 (4) |
| C53 | 0.820 (1) | 0.2397 (8) | 0.7507 (4) | 6.5 (5) |
| C54 | 0.812 (1) | 0.3148 (9) | 0.7381 (4) | 8.3 (6) |
| C55 | 0.755 (2) | 0.3373 (8) | 0.6917 (5) | 9.9 (7) |
| C56 | 0.704 (1) | 0.2837 (7) | 0.6561 (4) | 7.5 (5) |
| C61 | 0.703 (1) | 0.2578 (6) | 0.3854 (3) | 4.3 (4) |
| C62 | 0.711 (1) | 0.1827 (6) | 0.3745 (4) | 5.0 (4) |
| C63 | 0.834 (1) | 0.1504 (7) | 0.3688 (4) | 6.4 (5) |
| C64 | 0.949 (1) | 0.195 (1) | 0.3746 (4) | 7.7 (6) |
| C65 | 0.944 (1) | 0.2689 (9) | 0.3852 (4) | 7.2 (6) |
| C66 | 0.820 (1) | $0 \cdot 3009$ (7) | 0.3904 (4) | 5.4 (4) |
| C71 | 0.439 (1) | 0.2791 (6) | 0.3355 (4) | 4.5 (4) |
| C72 | $0 \cdot 299$ (1) | $0 \cdot 2822$ (7) | 0.3324 (4) | 6.3 (5) |
| C73 | $0 \cdot 214$ (1) | 0.2723 (8) | 0.2872 (5) | 8.5 (6) |
| C74 | 0.272 (1) | 0.2550 (7) | 0.2447 (4) | 7.8 (5) |
| C75 | 0.410 (1) | 0.2518 (7) | 0.2479 (4) | 6.5 (5) |
| C76 | 0.490 (1) | 0.2629 (6) | 0.2923 (4) | 5.7 (4) |
| C 81 | 0.560 (1) | $0 \cdot 3968$ (6) | 0.3971 (4) | 4.7 (4) |
| C82 | 0.547 (1) | 0.4398 (8) | 0.3547 (5) | 8.4 (6) |
| C83 | 0.569 (2) | 0.5176 (8) | 0.3584 (7) | 11.0 (8) |
| C84 | 0.593 (2) | 0.5513 (8) | $0 \cdot 4020$ (7) | 10.4 (8) |
| C85 | 0.604 (2) | 0.5093 (9) | 0.4427 (6) | 10.2 (8) |
| C86 | 0.590 (1) | 0.4312 (7) | 0.4404 (5) | 7.6 (6) |
| C91 | 1.077 (1) | 0.4793 (8) | 0.6526 (5) | 12.00 |
| C 92 | 1.075 (1) | 0.4828 (8) | 0.7036 (5) | 12.00 |
| C93 | 0.964 (1) | 0.5136 (8) | 0.7218 (5) | 12.00 |
| C94 | 0.855 (1) | 0.5409 (8) | $0 \cdot 6891$ (5) | 12.00 |
| C95 | 0.857 (1) | 0.5373 (8) | 0.6382 (5) | 12.00 |
| C96 | 0.968 (1) | 0.5065 (8) | 0.6199 (5) | 12.00 |
| C97 | 0.970 (1) | 0.5027 (8) | $0 \cdot 5652$ (5) | 12.00 |
| (B) |  |  |  |  |
| Rul | 0.24623 (2) | 0.19976 (1) | 0.06059 (2) | 3.00 (1) |
| Pl | 0.34682 (7) | $0 \cdot 11770$ (2) | 0.03520 (6) | $3 \cdot 20$ (2) |
| N1 | 0.4348 (3) | 0.25000 | 0.1646 (3) | $3 \cdot 3$ (1) |
| N2 | 0.2846 (3) | 0.25000 | -0.0865 (3) | $3 \cdot 3$ (1) |
| Oll | -0.0613 (2) | 0.1664 (1) | -0.1272 (3) | 7.3 (1) |
| 012 | 0.1843 (3) | 0.1763 (1) | 0.3105 (2) | 7.9 (1) |
| C1 | 0.5236 (4) | 0.25000 | 0.0887 (4) | 3.4 (1) |
| C2 | 0.4379 (4) | 0.25000 | -0.0561 (4) | 3.4 (1) |
| C3 | 0.5013 (5) | $0 \cdot 25000$ | -0.1487 (4) | 4.5 (1) |
| C4 | 0.6556 (5) | 0.25000 | -0.0959 (5) | 5.5 (2) |
| C5 | 0.7392 (4) | 0.25000 | 0.0439 (5) | 5.4 (2) |
| C6 | 0.6745 (4) | 0.25000 | 0.1395 (4) | 4.3 (1) |
| Cll | 0.0551 (3) | 0.1793 (1) | -0.0560 (3) | $4 \cdot 2$ (1) |
| C12 | 0.2098 (3) | 0.1835 (1) | 0.2156 (3) | 4.6 (1) |
| C31 | 0.2717 (3) | 0.0922 (1) | -0.1440 (3) | 3.6 (1) |
| C32 | 0.2449 (3) | 0.0395 (1) | -0.1753 (3) | 4.9 (1) |

Table 2 (cont.)

|  |  |  |  |  |
| :--- | ---: | ---: | ---: | ---: |
| C33 | $0.1909(4)$ | $0.0224(1)$ | $-0.3129(4)$ | $6.3(1)$ |
| C34 | $0.1628(4)$ | $0.0573(2)$ | $-0.4193(4)$ | $6.4(1)$ |
| C35 | $0.1906(4)$ | $0.1099(1)$ | $-0.3895(3)$ | $6.6(1)$ |
| C36 | $0.2429(4)$ | $0.1276(1)$ | $-0.2524(3)$ | $5.4(1)$ |
| C41 | $0.129(3)$ | $0.0635(1)$ | $0.1329(3)$ | $3.8(1)$ |
| C42 | $0.1698(3)$ | $0.0547(1)$ | $0.1170(4)$ | $5.2(1)$ |
| C43 | $0.1366(4)$ | $0.0145(1)$ | $0.1877(4)$ | $6.5(2)$ |
| C44 | $0.2465(5)$ | $-0.0168(1)$ | $0.2777(4)$ | $7.1(2)$ |
| C45 | $0.3896(4)$ | $-0.0089(1)$ | $0.2941(4)$ | $7.0(2)$ |
| C46 | $0.425(4)$ | $0.0305(1)$ | $0.2216(3)$ | $5.2(1)$ |
| C51 | $0.5449(3)$ | $0.1145(1)$ | $0.0869(3)$ | $3.9(1)$ |
| C52 | $0.6372(3)$ | $0.1297(1)$ | $0.2217(3)$ | $5.3(1)$ |
| C53 | $0.7884(3)$ | $0.1274(1)$ | $0.2669(4)$ | $6.5(1)$ |
| C54 | $0.8478(3)$ | $0.1117(1)$ | $0.1765(5)$ | $6.8(2)$ |
| C55 | $0.7582(4)$ | $0.0979(2)$ | $0.0416(4)$ | $6.5(2)$ |
| C56 | $0.6070(3)$ | $0.0989(1)$ | $-0.0031(3)$ | $5.1(1)$ |
| C61 | $-0.0246(9)$ | 0.25000 | $-0.4456(10)$ | $11.2(2)$ |
| C62 | $-0.1759(6)$ | 0.25000 | $-0.4495(6)$ | $7.3(1)$ |
| C63 | $-0.2499(4)$ | $0.2967(2)$ | $-0.4510(4)$ | $7.4(1)$ |
| C64 | $-0.3889(5)$ | $0.2973(2)$ | $-0.4580(5)$ | $7.4(1)$ |
| C65 | $-0.4583(7)$ | 0.25000 | $-0.4655(7)$ | $7.5(2)$ |
| C66 | $-0.4577(6)$ | $0.3482(2)$ | $-0.4492(6)$ | $10.9(2)$ |

(Motherwell, 1976). Geometrical calculations were performed with PARST (Nardelli, 1983).

Discussion. Positional parameters and equivalent isotropic thermal parameters for $(A)$ and $(B)$ are shown in Table 2.* A selection of distances and angles for both structures is collected in Table 3. For molecule $(A)$, Fig. 1 shows a plot with the atomic numbering scheme. Molecule ( $B$ ) follows a similar labelling.

The space group for compound $(A)$ was determined unequivocally from systematic absences. During the structure determination and refinement no problems were found and the four carbonyl groups came out very clearly from the beginning and refined well (see Table 2 for thermal factors).

For structure ( $B$ ) the space group assignment was not evident from systematic absences. We initially solved the structure in the noncentrosymmetric space group $P 2_{1}$, finding the mirror plane and strong correlations. Therefore we decided to solve the structure in the centrosymmetric space group. The agreement factors were $R=0.024$ and $R=0.032$ for the noncentrosymmetric and centrosymmetric space groups, respectively. In both space groups four carbonyl ligands were unequivocally determined (see Table 2 for thermal factors). In addition, the FAB mass spectrum of the compound shows (see Experimental) the maximum m/e ratio at 946 corresponding to the cation $\left[\mathrm{Ru}_{2}\left(\mathrm{C}_{6} \mathrm{H}_{6} \mathrm{~N}_{2}\right)(\mathrm{CO})_{4}\left\{\mathrm{P}\left(\mathrm{C}_{6} \mathrm{H}_{5}\right)_{3}\right\}_{2}\right]^{+}$ ( $M^{+}$) and other peaks for the cations resulting from a stepwise release of ligands which indicate the above stoichiometry for compound ( $A$ ).

[^1]Table 3. Selected bond lengths ( $\AA$ ) and bond angles ( ${ }^{\circ}$ ) (with e.s.d.'s)

|  | (A)* | (B) $\dagger$ |
| :---: | :---: | :---: |
| Rul-Ru2 | $2 \cdot 558$ (1) | $2 \cdot 560$ (1) |
| Rul-P1 | $2 \cdot 372$ (3) | $2 \cdot 382$ (1) |
| Rul-N1 | $2 \cdot 167$ (8) | 2.163 (2) |
| Rul-N2 | 2.149 (8) | 2.161 (2) |
| Rul-Cl1 | 1.87 (1) | 1.866 (3) |
| Rul-C12 | 1.85 (1) | 1.858 (3) |
| P1-C31 | 1.83 (1) | 1.834 (2) |
| Pl-C41 | 1.81 (1) | 1.835 (3) |
| Pl-C51 | 1.83 (1) | 1.825 (3) |
| $\mathrm{C} 11-\mathrm{O} 11$ | 1.13 (1) | $1 \cdot 140$ (3) |
| $\mathrm{Cl} 2-\mathrm{Ol2}$ | $1 \cdot 15$ (1) | 1-143 (3) |
| $\mathrm{Nl}-\mathrm{Cl}$ | 1.44 (1) | 1.419 (4) |
| $\mathrm{N} 2-\mathrm{C} 2$ | 1.42 (1) | 1.431 (4) |
| $\mathrm{Cl}-\mathrm{C} 2$ | 1.41 (1) | 1.403 (5) |
| C2-C3 | 1.37 (1) | 1.364 (5) |
| C3-C4 | 1.40 (2) | 1.407 (6) |
| C4-C5 | 1.33 (2) | 1.357 (6) |
| C5-C6 | 1.41 (2) | 1.404 (6) |
| C6- Cl | 1.37 (1) | $1 \cdot 376$ (5) |
| Ru2-Rul-P1 | 150.7 (1) | $151 \cdot 3$ (1) |
| Ru2-Rul-N1 | 53.8 (2) | 53.7 (1) |
| Ru2-Rul-N2 | $53 \cdot 5$ (2) | 53.7 (1) |
| Ru2-Rul-C11 | $104 \cdot 5$ (3) | $106 \cdot 2$ (1) |
| Ru2-Rul-C12 | $105 \cdot 6$ (4) | $102 \cdot 9$ (1) |
| Pl-Rul-N1 | $105 \cdot 2$ (2) | 104.8 (1) |
| P1—Rul-N2 | 102.4 (2) | $103 \cdot 4$ (1) |
| Pl-Rul-C11 | 94.2 (3) | 92.7 (1) |
| Pl-Rul-C12 | 96.2 (4) | 98.5 (1) |
| N1-Rul-N2 | 69.1 (3) | 68.6 (1) |
| N1-Rul-Cll | 158.3 (4) | 159.7 (1) |
| N1-Rul-C12 | 96.1 (4) | 97.8 (1) |
| N2-Rul-Cll | 97.6 (4) | 97.6 (1) |
| N2-Rul-Cl2 | 158.7 (4) | 156.6 (1) |
| C11-Rul-Cl2 | 91.4 (5) | 89.7 (1) |
| *Averaged values. <br> $\dagger$ Mirror plane related. |  |  |

A comparison of bond distances and angles for molecules ( $A$ ) and ( $B$ ) shows that both are virtually identical, with normal differences derived from distinct packings due to different solvents. All this information allows us to conclude that molecules of structures $(A)$ and $(B)$ are of the same compound, mentioned in the title of this paper, and therefore structure ( $C$ ) should be substituted by structure ( $B$ ) of the present communication.


Fig. 1. PLUTO (Motherwell, 1976) diagram showing the atomic arrangement and numbering for molecule ( $A$ ); molecule ( $B$ ) follows a similar labelling.

The molecule shows a mirror plane containing the diimine ring, which is a crystallographic mirror plane for structure ( $B$ ).

The distance between the two ruthenium atoms, $2 \cdot 559$ (1) $\AA$, is significantly short for a single $\mathrm{Ru}^{\mathrm{I}}-\mathrm{Ru}^{\mathrm{I}}$ bond.

Both ruthenium atoms exhibit a significantly distorted octahedral environment due to the very diverse nature of the ligands.

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# Structure of $\boldsymbol{c i s}$-Tetrachlorobis(1-ethylimidazole- $\kappa \boldsymbol{N}^{\boldsymbol{\beta}}$ )platinum(IV) 

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[^2]$\mu=9.61 \mathrm{~mm}^{-1}, F(000)=1000, T=294(2) \mathrm{K}, R=$ 0.036 for 2110 unique observed reflections. The complex consists of mononuclear $\mathrm{PtCl}_{4}(1$-ethylimidazole) $)_{2}$ units. The coordination around $\mathrm{Pt}^{\text {IV }}$ is © 1990 International Union of Crystallography


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[^1]:    * Lists of structure amplitudes, anisotropic thermal parameters, H -atom parameters, distances, angles and principal torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53041 ( 71 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

[^2]:    Abstract. $\left[\mathrm{PtCl}_{4}\left(\mathrm{C}_{5} \mathrm{H}_{8} \mathrm{~N}_{2}\right)_{2}\right], M_{r}=529 \cdot 2$, monoclinic, $P 2_{1} / n, a=12.749$ (4), $b=9.217$ (3), $c=14.898$ (5) $\AA$, $\beta=110.06$ (5) ${ }^{\circ}, \quad V=1644.4$ (9) $\AA^{3}, \quad Z=4, \quad D_{m}=$ 2.12 (3), $D_{x}=2.14 \mathrm{Mg} \mathrm{m}^{-3}$, Мо $K \alpha, \lambda=0.71069 \AA$,

