Supplementary Material (ESI) for Chemical Communications

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X-ray crystal structure determination of 3

Crystal suitable for X-ray analysis were obtained by slow concentration of a acetonitril solution of 3. [C₆₄H₄₃N₁₀O₄Ru₂](PF₆) · 3 CH₃CN, Fw = 1486.36, dark red needle, 0.30 x 0.12 x 0.06 mm³, triclinic, P T (no.2 ), a = 9.5350(1), b = 15.4639(2), c = 21.5139(3) Å, \( \alpha = 97.2304(4) \), \( \beta = 96.0597(5) \), \( \gamma = 92.7702(6)^\circ \), \( V = 3123.27(7) \) Å³, \( Z = 2 \), \( D_\chi = 1.580 \) g/cm³, \( \mu = 0.59 \) mm⁻¹. 57455 Reflections were measured on a Nonius Kappa CCD diffractometer with rotating anode (graphite monochromator, \( \lambda = 0.71073 \) Å) up to a resolution of (\( \sin \theta/\lambda \)max = 0.65 Å⁻¹) at a temperature of 110 K. An absorption correction based on multiple measured reflections was applied (0.94-0.97 correction range). 14126 Reflections were unique (Rint = 0.048). The structure was solved with automated Patterson methods (program DIRDIF-99[1]) and refined with SHELXL-97[2] against F² of all reflections. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were introduced in calculated positions and refined with a riding model. The assignment of C11/N12 and C21/N22 was based on the refinements of the occupancies on the corresponding sites. The PF₆ anion and one acetonitrile molecule were disordered. 941 Parameters were refined with 447 restraints. R1/wR2 [I > 2\( \sigma \)(I)]: 0.0362/0.0878. R1/wR2 [all refl.]: 0.0500/0.0955. S = 1.035. Residual electron density between -0.83 and 1.77 e/Å³. Geometry calculations and checking for higher symmetry was performed with the PLATON program[3].

References