

corresponding edta complexes {2.378 Å in Na₂[Mg(H₂O)Y].5H₂O and 2.396 Å in [Mg(H₂O)₆][Mg(H₂O)Y].2H₂O}. This reflects the reduced basicity on the nitrogen atoms in phdta compared with those in edta. Configuration around the N atoms is well described as tetrahedral, since the sum of the three C—N—C bond angles is 333.9 (3)° for N(1) and 332.2 (3)° for N(2), respectively, although our preliminary work shows a quasiplanar configuration in neutral ligand H₄phdta and exclusive protonation on the carboxylate oxygens atoms alone (no zwitterion).

The pentahydrated Mg(2)O₆ moiety may be approximately described as octahedral, though the bond lengths Mg(2)—O are somewhat more divergent than those in the other edta complexes having a discrete hexahydrated moiety; *i.e.* the Mg(2)—O bond ranges from 2.033 to 2.118 Å (2.076 Å on the average) in [Mg(H₂O)₅][Mg(H₂O)phdta], while it ranges from 2.047 to 2.087 Å (ave. 2.062 Å) in [Mg(H₂O)₆][Mg(H₂O)Y] (Passer *et al.*, 1977) and from 2.053 to 2.087 Å (ave. 2.076 Å) in [Mg(H₂O)₆][H₂Y] (Julian *et al.*, 1973). These values may be compared with the mean Mg—O distance of 2.044 Å found for an aqueous solution (2 molar in MgCl₂ and CaCl₂) by an X-ray diffraction study (Caminiti, Licheri, Piccaluga & Pinna, 1977). All the water molecules are hydrated in this Mg—phdta complex, differing from those in the dimeric Mg—edta complex. These water molecules act as proton donor in a three-dimensional hydrogen-bonding network, as summarized in Table 2.

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Structure of *cis*-Bis(4-allyl-4*H*-1,2,4-triazole)bis(2,2'-bipyridine)ruthenium(II) Bis(hexafluorophosphate)

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Abstract. [Ru(C₅H₇N₃)₂(C₁₀H₈N₂)₂](PF₆)₂, *M_r* = 449.4, monoclinic, *P*2₁/*n*, *a* = 13.472 (4), *b* = 20.278 (6), *c* = 13.995 (4) Å, β = 101.34 (2)°, *V* = 3748.6 Å³, *Z* = 4, *D_x* = 1.633, *D_m* = 1.66 (1) Mg m⁻³, λ(Mo Kα) = 0.71069 Å, μ = 0.819 mm⁻¹, *F*(000) = 1848.80, *T* = 298 K, *R* = 0.0489 for 2635 unique reflections [*I* > 3σ(*I_o*)]. The planar 2,2'-bipyridine ligands are coordinated in a *cis* fashion to the central

metal ion. The Ru—N distances of 2.039 (6)–2.084 (6) Å are in the range expected for ruthenium(II) compounds.

Introduction. Ruthenium bis(bpy) (bpy = 2,2'-bipyridine) compounds have attracted much attention in the last few years because of their potential application as photochemical and electrochemical catalysts (Seddon & Seddon, 1984). We are interested in the photophysical and electrochemical properties of ruthenium bis(bpy) compounds containing ligands such as 1,2,4-triazoles and pyridyl-1,2,4-triazoles. In these investigations the effect of the presence of the five-membered ring on the physical properties and the structural parameters of the compounds is studied. The properties of these compounds are compared with those of the parent compound [Ru(bpy)₃]²⁺ (Vos, Haasnoot & Vos, 1983; Hage, Prins, Haasnoot, Reedijk & Vos, 1987). In this paper the first crystal structure of a ruthenium compound involving a 1,2,4-triazole ligand is reported.

Experimental. Orange crystals of the title complex were prepared as described before (Vos, Haasnoot & Vos, 1983). A needle-shaped crystal of dimensions 0.50 × 0.15 × 0.05 mm was selected for crystal structure determination. D_m was determined by flotation in dibromopropane/hexane. Intensity data were collected at room temperature using a Nicolet R3/M diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Cell parameters were obtained by a least-squares method using 25 reflections with $1.5 < \theta < 13.35^\circ$. Data were collected within $1.5 < \theta < 22.5^\circ$ using the ω - 2θ scan method and were corrected for Lorentz, polarization and absorption effects (transmission coefficients: min. 0.270, max. 0.294). The range for h was 0 to 15, for k 0 to 22, and for l -16 to 16. The intensity variation of a standard reflection (200) was $\pm 2\%$ about the mean value. The main computer program used was *SHELXTL* (Sheldrick, 1983). Of the 5471 reflections measured, 4885 were independent of which 2635 were observed [$I > 3\sigma(I)$] and were used in the refinement. The structure was solved by Patterson techniques. Full-matrix least-squares refinement on F of positional and anisotropic thermal parameters. H atoms were placed in calculated positions and were assigned isotropic thermal parameters ($U = 0.08 \text{ \AA}^2$). Final $R = 0.0489$, $wR = 0.0494$, max. $\Delta/\sigma = 0.042$. $w = [\sigma^2(F)]^{-1}$. Max., min. $\Delta\rho$ values in final difference synthesis, 0.44, -0.44 e \AA^{-3} . Scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Atomic fractional coordinates and equivalent isotropic thermal parameters for the non-H

Table 1. Fractional coordinates and equivalent isotropic thermal displacement parameters (\AA^2)

$U_{eq} = (8\pi^2/3) \text{ trace } U.$				
	x	y	z	U_{eq}
Ru	2865 (1)	6388 (1)	6264 (1)	39 (1)
P(1)	3267 (2)	2368 (1)	5036 (2)	63 (1)
F(11)	3864 (4)	1813 (3)	5694 (4)	91 (3)
F(12)	2561 (6)	2455 (4)	5784 (5)	160 (4)
F(13)	2640 (4)	2937 (3)	4373 (4)	84 (2)
F(14)	3961 (5)	2311 (4)	4286 (5)	133 (4)
F(15)	3984 (6)	2902 (3)	5599 (5)	155 (4)
F(16)	2526 (6)	1857 (3)	4446 (5)	141 (4)
P(2)	5873 (2)	435 (1)	7386 (2)	66 (1)
F(21)	5512 (7)	401 (6)	8403 (6)	210 (6)
F(22)	5248 (10)	-112 (5)	7702 (11)	269 (8)
F(23)	5270 (9)	426 (7)	6409 (7)	301 (9)
F(24)	6550 (7)	-97 (4)	7064 (7)	179 (5)
F(25)	5176 (6)	931 (4)	7744 (7)	182 (5)
F(26)	6564 (7)	955 (4)	7166 (8)	201 (6)
N(11)	1458 (5)	6810 (3)	6257 (4)	42 (3)
N(12)	672 (5)	6814 (4)	5439 (5)	57 (3)
N(13)	210 (5)	7354 (4)	6629 (5)	52 (3)
C(10)	1153 (6)	7134 (4)	6947 (6)	49 (3)
C(11)	-35 (7)	7139 (5)	5704 (5)	69 (4)
C(12)	-443 (7)	7718 (5)	7157 (7)	81 (5)
C(13)	-328 (10)	8407 (5)	7132 (9)	125 (7)
C(14)	310 (11)	8737 (5)	6827 (8)	139 (7)
N(21)	4195 (4)	5911 (3)	6252 (4)	44 (3)
N(22)	2365 (5)	5442 (3)	5975 (4)	44 (3)
C(20)	5113 (6)	6191 (4)	6371 (6)	54 (4)
C(21)	6001 (6)	5828 (5)	6413 (7)	66 (4)
C(22)	5931 (7)	5160 (5)	6339 (6)	66 (4)
C(23)	4993 (7)	4870 (5)	6179 (7)	60 (4)
C(24)	4129 (6)	5251 (4)	6146 (5)	44 (3)
C(25)	3115 (6)	4985 (4)	5999 (6)	46 (3)
C(26)	2882 (7)	4318 (5)	5884 (7)	66 (4)
C(27)	1887 (8)	4110 (5)	5767 (7)	71 (5)
C(28)	1136 (7)	4566 (4)	5771 (7)	63 (4)
C(29)	1407 (6)	5225 (4)	5868 (6)	55 (4)
N(31)	3036 (5)	6127 (3)	7730 (5)	46 (3)
N(32)	3942 (5)	6165 (4)	8383 (5)	74 (4)
N(33)	2824 (6)	5669 (4)	9090 (5)	66 (3)
C(30)	3798 (7)	5902 (6)	9186 (7)	86 (5)
C(31)	2389 (6)	5817 (4)	8161 (6)	54 (4)
C(32)	2384 (8)	5314 (5)	9808 (7)	94 (5)
C(33)	2917 (17)	4659 (11)	9922 (13)	188 (11)
C(34)	3319 (15)	4502 (11)	10621 (16)	237 (18)
N(41)	2705 (5)	6710 (3)	4852 (4)	44 (3)
N(42)	3482 (5)	7320 (3)	6478 (5)	49 (3)
C(40)	2263 (6)	6360 (5)	4065 (5)	54 (3)
C(41)	2078 (7)	6660 (5)	3138 (6)	69 (4)
C(42)	2312 (8)	7280 (5)	3026 (7)	82 (5)
C(43)	2780 (8)	7640 (5)	3806 (7)	75 (5)
C(44)	2976 (6)	7340 (4)	4728 (6)	48 (3)
C(45)	3461 (6)	7665 (4)	5638 (7)	50 (4)
C(46)	3871 (7)	8287 (5)	5647 (8)	74 (4)
C(47)	4342 (8)	8555 (5)	6562 (8)	93 (5)
C(48)	4340 (7)	8219 (5)	7379 (8)	78 (5)
C(49)	3906 (6)	7594 (5)	7331 (6)	62 (4)

atoms are given in Table 1.* Fig. 1 shows a projection of the molecule together with the atom numbering. Bond distances and angles are given in Table 2.

The two, flat, 2,2'-bipyridine ligands are bound to the central Ru atom in a *cis* coordination. The average Ru^{II}—N(bpy) bond length is 2.050 (6) Å, similar to that found in [Ru(bpy)₃](PF₆)₂ [2.056 (6) Å] (Rillema, Jones & Levy, 1979). In a number of compounds of the types [Ru(bpy)₂X₂]ⁿ⁺

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51967 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (Å) and bond angles (°)

Estimated standard deviations in the least significant digits are given in parentheses.

Ru—N(11)	2.078 (6)	Ru—N(21)	2.039 (6)
Ru—N(22)	2.045 (6)	Ru—N(31)	2.084 (6)
Ru—N(41)	2.050 (6)	Ru—N(42)	2.062 (7)
P(1)—F(11)	1.567 (6)	P(1)—F(12)	1.562 (8)
P(1)—F(13)	1.607 (6)	P(1)—F(14)	1.546 (8)
P(1)—F(15)	1.555 (7)	P(1)—F(16)	1.552 (7)
P(2)—F(21)	1.507 (8)	P(2)—F(22)	1.513 (13)
P(2)—F(23)	1.440 (10)	P(2)—F(24)	1.537 (10)
P(2)—F(25)	1.529 (9)	P(2)—F(26)	1.479 (10)
N(11)—N(12)	1.391 (8)	N(11)—C(10)	1.302 (11)
N(12)—C(11)	1.274 (12)	N(13)—C(10)	1.334 (10)
N(13)—C(11)	1.342 (11)	N(13)—C(12)	1.458 (13)
C(12)—C(13)	1.406 (16)	C(13)—C(14)	1.233 (19)
N(21)—C(20)	1.339 (10)	N(21)—C(24)	1.347 (10)
N(22)—C(25)	1.366 (10)	N(22)—C(29)	1.342 (11)
C(20)—C(21)	1.395 (12)	C(21)—C(22)	1.359 (14)
C(22)—C(23)	1.359 (14)	C(23)—C(24)	1.390 (12)
C(24)—C(25)	1.443 (11)	C(25)—C(26)	1.390 (13)
C(25)—C(27)	1.382 (14)	C(27)—C(28)	1.371 (13)
C(28)—C(29)	1.383 (12)	N(31)—N(32)	1.369 (9)
N(31)—C(31)	1.316 (11)	N(32)—C(30)	1.294 (13)
N(33)—C(30)	1.374 (13)	N(33)—C(31)	1.346 (10)
N(33)—C(32)	1.457 (14)	C(32)—C(33)	1.502 (24)
C(33)—C(34)	1.064 (27)	N(41)—C(40)	1.342 (10)
N(41)—C(44)	1.350 (11)	N(42)—C(45)	1.363 (11)
N(42)—C(49)	1.334 (10)	C(40)—C(41)	1.407 (12)
C(41)—C(42)	1.314 (14)	C(42)—C(43)	1.357 (13)
C(43)—C(44)	1.402 (13)	C(44)—C(45)	1.462 (11)
C(45)—C(46)	1.375 (13)	C(46)—C(47)	1.416 (14)
C(47)—C(48)	1.331 (15)	C(48)—C(49)	1.390 (13)
N(11)—Ru—N(21)	176.0 (2)	N(11)—Ru—N(22)	97.0 (3)
N(21)—Ru—N(22)	79.0 (3)	N(11)—Ru—N(31)	91.5 (2)
N(21)—Ru—N(31)	88.2 (3)	N(22)—Ru—N(31)	85.5 (3)
N(11)—Ru—N(41)	87.0 (3)	N(21)—Ru—N(41)	93.5 (3)
N(22)—Ru—N(41)	98.2 (2)	N(21)—Ru—N(42)	176.1 (3)
N(11)—Ru—N(42)	87.9 (2)	N(21)—Ru—N(42)	96.1 (3)
N(22)—Ru—N(42)	174.2 (3)	N(31)—Ru—N(42)	97.5 (3)
N(41)—Ru—N(42)	78.9 (3)	F(11)—P(1)—F(12)	89.8 (4)
F(11)—P(1)—F(13)	179.0 (4)	F(12)—P(1)—F(13)	89.2 (4)
F(11)—P(1)—F(14)	92.1 (4)	F(12)—P(1)—F(14)	177.7 (5)
F(13)—P(1)—F(14)	88.9 (4)	F(11)—P(1)—F(15)	90.6 (3)
F(12)—P(1)—F(15)	88.9 (5)	F(13)—P(1)—F(15)	89.4 (3)
F(14)—P(1)—F(15)	89.8 (4)	F(11)—P(1)—F(16)	91.8 (3)
F(12)—P(1)—F(16)	91.1 (4)	F(13)—P(1)—F(16)	88.2 (3)
F(14)—P(1)—F(16)	90.1 (4)	F(15)—P(1)—F(16)	177.6 (4)
F(21)—P(2)—F(22)	86.2 (7)	F(21)—P(2)—F(23)	176.6 (8)
F(22)—P(2)—F(23)	91.7 (8)	F(21)—P(2)—F(24)	89.2 (5)
F(22)—P(2)—F(24)	88.3 (6)	F(23)—P(2)—F(24)	88.1 (7)
F(21)—P(2)—F(25)	89.5 (6)	F(22)—P(2)—F(25)	88.2 (6)
F(23)—P(2)—F(25)	93.1 (7)	F(24)—P(2)—F(25)	176.4 (5)
F(21)—P(2)—F(26)	88.1 (6)	F(22)—P(2)—F(26)	174.2 (6)
F(23)—P(2)—F(26)	93.9 (7)	F(24)—P(2)—F(26)	90.2 (5)
F(25)—P(2)—F(26)	93.2 (5)	Ru—N(11)—N(12)	123.1 (5)
Ru—N(11)—C(10)	129.1 (5)	N(12)—N(11)—C(10)	107.8 (6)
N(11)—N(12)—C(11)	104.6 (7)	C(10)—N(13)—C(11)	103.9 (7)
C(10)—N(13)—C(12)	129.2 (7)	C(11)—N(13)—C(12)	126.8 (7)
N(11)—C(10)—N(13)	110.2 (7)	N(12)—C(11)—N(13)	113.5 (7)
N(13)—C(12)—C(13)	114.1 (10)	C(12)—C(13)—C(14)	129.4 (13)
Ru—N(21)—C(20)	126.1 (5)	Ru—N(21)—C(24)	115.6 (5)
C(20)—N(21)—C(24)	118.2 (7)	Ru—N(22)—C(25)	114.7 (5)
Ru—N(22)—C(29)	126.9 (6)	C(25)—N(22)—C(29)	118.0 (7)
N(21)—C(20)—C(21)	122.9 (8)	C(20)—C(21)—C(22)	118.5 (8)
C(21)—C(22)—C(23)	119.2 (9)	C(22)—C(23)—C(24)	120.3 (9)
N(21)—C(24)—C(25)	120.8 (7)	N(21)—C(24)—C(25)	115.2 (7)
C(23)—C(24)—C(25)	124.0 (8)	N(22)—C(25)—C(24)	115.1 (7)
N(22)—C(25)—C(26)	120.6 (8)	C(24)—C(25)—C(26)	124.4 (8)
C(25)—C(26)—C(27)	120.0 (9)	C(26)—C(27)—C(28)	119.5 (9)
C(27)—C(28)—C(29)	118.1 (8)	N(22)—C(29)—C(28)	123.8 (8)
Ru—N(31)—N(32)	123.0 (5)	Ru—N(31)—C(31)	128.5 (5)
N(32)—N(31)—C(31)	107.7 (6)	N(31)—N(32)—C(30)	106.5 (7)
C(30)—N(33)—C(31)	103.6 (8)	C(30)—N(33)—C(32)	128.1 (7)
C(31)—N(33)—C(32)	128.2 (8)	N(32)—C(30)—N(33)	111.6 (8)
N(31)—C(31)—N(33)	110.6 (7)	N(33)—C(32)—C(33)	104.8 (11)
C(32)—C(33)—C(34)	120.2 (21)	C(33)—H(33)—C(34)	53.1 (13)
Ru—N(41)—C(40)	124.4 (6)	Ru—N(41)—C(44)	116.6 (5)
C(40)—N(41)—C(44)	118.7 (7)	Ru—N(42)—C(45)	114.1 (5)
Ru—N(42)—C(49)	126.7 (6)	C(45)—N(42)—C(49)	119.2 (7)
N(41)—C(40)—C(41)	119.6 (8)	C(40)—C(41)—C(42)	121.4 (8)
C(41)—C(42)—C(43)	120.1 (9)	C(42)—C(43)—C(44)	118.4 (9)
N(41)—C(44)—C(43)	121.6 (7)	N(41)—C(44)—C(45)	113.5 (7)
C(43)—C(44)—C(45)	124.9 (8)	N(42)—C(45)—C(44)	116.5 (7)
N(42)—C(45)—C(46)	121.6 (8)	C(44)—C(45)—C(46)	121.8 (8)
C(45)—C(46)—C(47)	117.7 (9)	C(46)—C(47)—C(48)	120.1 (9)
C(47)—C(48)—C(49)	119.9 (9)	N(42)—C(49)—C(48)	121.4 (8)

and [Ru(bpy)₂XY]ⁿ⁺, where X and Y are monodentate ligands, a strong *trans* effect has been observed. In the compound reported here this effect is not very strong. From the σ -donor properties of the triazole ligand one would expect a shortening of the *trans* Ru—N(bpy) bond. The observed *trans* effect, estimated from the difference of Ru—N distances in the same bpy ligand, is insignificant (0.006 and 0.012 Å), much smaller than observed in compounds such as [Ru(bpy)₂CO(H)](PF₆) (Haasnoot, Hinrichs, Weir & Vos, 1986) and [Ru(bpy)₂Cl₂] (Egglestone, Goldsby, Hodgson & Meyer, 1985). The Ru—N distances for one of the bpy ligands are, however, significantly shorter [2.039 (6) and 2.045 (6) Å] than those observed for the other ligand [2.050 (6) and 2.062 (7) Å]. The average Ru^{II}—N(triazole) distance is 2.080 (6) Å. This is longer than the Ru—N bond observed for the bpy ligands but still shorter than the values found for ammonia and pyridine in [Ru(NH₃)₆]²⁺ (Stynes & Ibers, 1971) and [Ru(py)₆]²⁺ (Templeton, 1979) of 2.144 (4) and 2.12 (1) Å, respectively. The Ru—N distances found in the molecule are consistent with the expected σ -donor properties of the triazole ligand. The distortions observed in the Ru—N(bpy) distances as a result of the presence of the triazole ligands are small.

The octahedral coordination about the central metal atom is not regular with an average N—Ru—N angle of 79.0 (3)° for the bpy ligands and 91.5 (2)° for the N—Ru—N angle involving the triazole ligands. The bite angle observed for the bpy ligand is similar to the value obtained in other ruthenium(II) compounds (Egglestone, Goldsby, Hodgson & Meyer 1985; Rillema, Jones & Levy, 1979).

In one of the triazole ligands the bond length observed for the C=C bond [C(33)—C(34)] of 1.06 (3) Å is too short for an ordered double bond. This suggests the presence of disorder in this allyl group.

The two PF₆⁻ groups are octahedrally coordinated with an average P—F distances of 1.56 Å for the first PF₆⁻ group, but a much shorter average P—F dis-

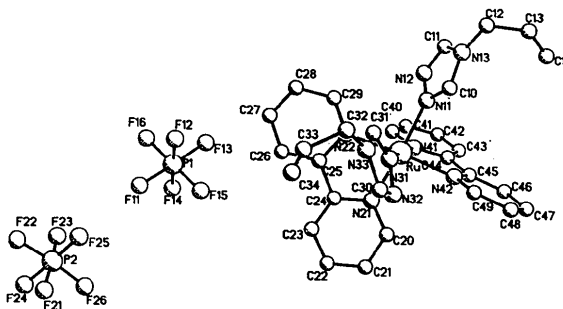


Fig. 1. Molecular structure and atom numbering of the title compound. H atoms are omitted for clarity.

tance of 1.50 Å for the second anion. This suggests the presence of some disorder in the PF_6^- groups.

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cis-Bis(cyclopropylamine)diiodoplatinum(II)

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Abstract. $[\text{PtI}_2(\text{C}_3\text{H}_7\text{N})_2]$, $M_r = 563.1$, monoclinic, $C2/c$, $a = 23.252$ (3), $b = 8.577$ (1), $c = 15.456$ (2) Å, $\beta = 128.38$ (1)°, $V = 2416$ (1) Å³, $Z = 8$, $D_x = 3.09$ g cm⁻³, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 173$ cm⁻¹, $F(000) = 1984$, $T = 298$ K, $R = 0.027$ for 1044 unique observed reflections. The structure consists of monomeric molecules with the four ligand atoms and the central metal ion in an essentially *cis*-planar arrangement. One of the two cyclopropane rings is partly disordered with one C atom occupying two positions.

Introduction. Studies on oligomeric platinum complexes raised a question about the effect of amine-ligand size on the tendency towards oligomerization. Traditionally research has been carried out with ammonia ligands, and various coloured species have been reported (Rosenberg, van Camp, Trosko & Mansour, 1969; Shimura, Tomohiro, Laitalainen, Moriyama, Uemura & Okuno, 1988; Laitalainen, Okuno & Tomohiro, 1987). The cyclopropylamine ligand forms blue oligomeric complexes (Laitalainen & Oksanen, 1989). The title compound was prepared, isolated and characterized for our studies on oligomerization reactions of platinum.

Experimental. The crystals were prepared by dissolving K_2PtCl_4 (0.1245 g, 0.300 mmol) and KI (0.1992 g, 1.200 mmol) in water (1.26 ml) with ultrasonication at room temperature. The reaction mixture was heated to 313 K and aqueous cyclopropylamine solution (14%, 250 μl , 0.613 mmol) was added dropwise within 10 minutes, the mixture was kept at 313 K under magnetic stirring for 20 additional minutes, then cooled on ice. A yellow powdery precipitate was filtered off. Very small crystals slowly formed in the filtrate. The whole synthesis was carried out in the dark.

A small crystal (0.02 × 0.06 × 0.10 mm) was measured with a CAD-4 diffractometer. The unit-cell parameters were determined on the basis of 25 well centred reflections in the angular range $24 < 2\theta < 34^\circ$. The intensity data were collected in the $\omega/2\theta$ scan mode, with the scan speed 0.9–16.5° min⁻¹ in the range $4.0 < 2\theta < 60^\circ$. The absorption correction was calculated with the DIFABS program (Walker & Stuart, 1983); the minimum/maximum corrections were 0.78/1.22. Intensities were collected up to $(\sin\theta)/\lambda = 0.7035$ Å⁻¹. Five standard reflections were measured once every hour. The intensities decreased 15% during the measurements and the data were