non-bonded atomic radii for chlorine between 1·58 and 1·78 Å (Nyburg & Faerman, 1985).

The coordination about the Cu atom is intermediate between tetrahedral and square planar. The dihedral angle between the planes (Cl, Cu, Cl′) and (N, Cu, N′) is 95·7 (1)°. The Cu—N bond lengths are consistent with those of other Cu^{2+} complexes with imidazoles. The Cu—Cl bond lengths are typical for bonds of Cu^{2+} with non-bridging chloride anions. There are no close halide approaches from adjacent molecules to the Cu atom, the intermolecular Cu···Cl distances being larger than 4·0 Å. The intramolecular Cu···Cl(2) distance is 3·427 (2) Å.

The structure of the complex is different from that of the analogous complex with imidazole (II). The coordination in $[CuCl_2(C_3H_4N_2)_2]$ is a distorted square pyramid, with two N and two Cl atoms in an approximately square plane [Cu—N 1·973 (12) and 1·992 (12), Cu—Cl 2·321 (4) and 2·365 (4) Å, N—Cu—N 174·3 (10) and Cl—Cu—Cl 166·9 (3)°], and a Cl atom from an adjacent unit occupying the apical site [Cu—Cl 2·751 (6) Å; Lundberg, 1972].

The configuration of (I) is similar to that of the analogous complex with *N*-methylimidazole, $CuCl_2(C_4H_6N_2)_2$ (III): Cu—N 1·962 (4) and 1·975 (5), Cu—Cl 2·260 (3) and 2·256 (2) Å, N—Cu—N 149·7 (1) and Cl—Cu—Cl 143·6 (1)° (van Ooijen, Reedijk & Spek, 1979). However, the conformations about the Cu—N bonds are different in the two complexes. In (III) the Cl atoms lie very close to the planes through the imidazole rings. In (I) the Cu—Cl vectors are tipped from the planes through the 2-chloroimidazole ligands at angles higher than 40°. The dihedral angle between these planes is 52·7 (2)°. The resulting Cl(2)···Cl(2′) intramolecular contact is 3·795 (6) Å.

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Synthesis and Structure of Hexakis(thiourea)ruthenium(II) Trifluoromethanesulfonate

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Abstract. $[Ru(CH_4N_2S)_6][CF_3SO_3]_2$, $M_r = 855·9$, monoclinic, $P2_1/a$, $a = 11·173$ (1), $b = 11·064$ (1), $c = 13·722$ (1) Å, $\beta = 113·96$ (1)°, $V = 1550·1$ (3) Å^3, $Z = 2$, $D_x = 1·834$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 1·10 mm^{-1}, $T = 295$ K, $F(000) = 860$, $R = 0·026$ for 3609 observed reflections. The synthesis, solution properties and structure of the title complex are described. The $[Ru(tu)_6]^{2+}$ (tu = thiourea) cations lie on crystallographic inversion centres and the $[CF_3SO_3]^-$ anions in general positions. The tu ligands are S bonded [mean Ru—S 2·421 (3) Å] and the six S atoms define an elongated trigonal antiprism centred on the metal atom.

Introduction. The title complex was isolated during a study of the kinetics of the replacement of coordinated water in $[Ru(H_2O)_6]^{2+}$ (Patel, 1988). In aqueous solution the $[Ru(tu)_6]^{2+}$ ion can be oxidized to $[Ru^{III}\{SC(NH)NH_2\}_3]$ in which the deprotonated

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tu is thought to function as an N,S-chelating ligand. The solution and solid state studies on $[Ru(tu)_6]^{2+}$ reported below were therefore mainly directed at establishing the mode of attachment of tu to the metal. So far as we know, they represent the first complete characterization of any tu complex of Ru^{II}.

Experimental. Solutions containing $[Ru(H_2O)_6]^{2+}$ showed only slight changes in UV–visible absorbance after the addition of small amounts of tu (Ru:tu 10:1). With excess tu (Ru:tu 1:10) there was a discernible increase in absorbance below 600 nm over a period of 6 h. This increase was more rapid with an Ru:tu ratio of 1:50 (0·05 *M* tu, 0·001 *M* Ru^{2+}, $[H^+]$ = 0·1 *M*, ionic strength 1·0 *M*, $[CF_3SO_3]^-$ counter ion) but in neither case were the results consistent with a single first order process, possibly because the introduction of a single tu ligand into the coordination sphere of Ru facilitated further substitution. Consistent with this, the second solution deposited purple crystals of composition $[Ru(tu)_6][CF_3SO_3]_2$ (found: C 11·70, H 2·78, N 19·03%; calculated: C 11·23, H 2·81, N 19·65%). In the FAB mass spectrum the parent ion has a m/e ratio of 856, corresponding to $[^{102}Ru(tu)_6(CF_3SO_3)_2]^+$. The conductivity of a 10^{-3} *M* aqueous solution of the complex (202 Ω^{-1} cm^2 mol^{-1}) was that expected for a 2:1 electrolyte. Comparison of the infrared absorption bands [KBr disk; $\nu(NH_2)$ 3100–3300, ν(CN) 1575 and ν(CS) 655 cm^{-1}] with the corresponding bands for free tu (3050–3300, 1540 and 680 cm^{-1}) suggested S— rather than N—Ru bonding (Nakamoto, 1978).

Cyclic voltammetry of 2 m*M* aqueous solutions under air-free conditions, using a PAR170 electrochemical system with mercury cup working, Pt wire secondary and saturated calomel reference electrodes, a scan rate of 200 mV s^{-1} and an ionic strength of 0·1 *M* ($NaCF_3SO_3/CF_3SO_3H$) gave the results in Table 1. The potentials are reported relative to a normal hydrogen electrode at 298 K. From Table 1 it can be concluded that tu is more effective in stabilizing Ru^{II} than H_2O or $C_2O_4^{2-}$. This result can be rationalized in terms of S-bonded tu having greater π-acidity than H_2O or $C_2O_4^{2-}$ since Ru^{II} is known to be an effective π donor.

Aqueous solutions of $[Ru(tu)_6]^{2+}$ slowly turn from yellow to blue-green (λ_{max} 670 nm). Yaffe & Voigt (1952) suggested that this change involves formation of a 1:3 Ru^{III}—SC(NH)NH_2 complex. Attempts to follow this change from the liberation of H^+ were inconclusive, probably because of the buffering action of the liberated tu. Attempts to isolate the blue-green Ru^{III} species by extraction with diethyl ether were unsuccessful.

Diffraction experiments were made with a crystal of dimensions 0·48 × 0·40 × 0·36 mm mounted on an Enraf–Nonius CAD-4 diffractometer. Cell dimensions are based on the setting angles of 22 reflections with $11 < \theta(\text{Mo } K\alpha) < 15°$.* The intensities of 6066 reflections with $2 < \theta(\text{Mo } K\alpha) < 30°$, h 0→15, k −15→4, l −19→19, were measured from $\omega/2\theta$ scans. After empirical correction for absorption [transmission factors on F 0·86–1·10 (Walker & Stuart, 1983)] duplicate estimations of the intensities of 1451 unique reflections were merged (R_{int} = 0·025) to give 4506 unique F values. Of these 3609 with $I > 3\sigma(I)$ were used subsequently. Intensity standards (020 and 004) showed random fluctuations of up to 8% of their mean values. The structure was solved by Patterson and difference Fourier methods and refined by full-matrix least squares on F with $w^{-1} = \sigma^2(F) + 0{\cdot}0023F^2$. Positional and anisotropic displacement parameters were adjusted for all non-H atoms. All H atoms were located in difference syntheses; their fractional coordinates and U_{iso} values were included in the least-squares refinement. Adjustment of 236 parameters, including an extinction parameter [g = 6·45 (13) × 10^{-3} (Larson, 1970)] converged (maximum shift/e.s.d. < 0·04) at R = 0·026, wR = 0·037, S = 1·98. Final $|\Delta\rho|$ values were < 0·43 e Å^{-3}. Neutral atom scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and all calculations were performed with the *GX* program package (Mallinson & Muir, 1985). Results are presented in Tables 2 and 3.†

Table 1. *Selected* Ru^{III}/Ru^{II} *reduction potentials* (V)

Couple	$E_{1/2}$	ΔE_p	Medium
$[Ru(tu)_6]^{3+}/[Ru(tu)_6]^{2+}$	+0·26	0·060	$NaCF_3SO_3$ (pH 7)
$[Ru(H_2O)_6]^{3+}/[Ru(H_2O)_6]^{2+}$	+0·20*	0·062	CF_3SO_3H
$[Ru(H_2O)_4(C_2O_4)]^+/[Ru(H_2O)_4(C_2O_4)]$	+0·04	0·064	CF_3SO_3H
$[Ru(C_2O_4)_3]^{3-}/[Ru(C_2O_4)_3]^{4-}$	−0·46†		H_2O

* *cf.* +0·21 V reported by Kallen & Earley (1971).
† From Kaziro, Hambley, Binstead & Beattie (1989).

Discussion. The discrete centrosymmetric $[Ru(tu)_6]^{2+}$ cations (Fig. 1) contain nearly planar tu ligands which are coordinated to the metal through S as in the other homoleptic $M(tu)_6$ species which have been structurally characterized: $M = Pb^{2+}$ (Goldberg &

* The transformation (0$\bar{1}$0/100/102) applied to the cell whose dimensions are given in the *Abstract* yields an *A*-centred, metrically orthorhombic cell with a = 11·06, b = 11·17, c = 25·08 Å. Transformation of the intensities to correspond to this cell followed by merging in Laue group *mmm* gave R_{int} = 0·39, thereby confirming the monoclinic symmetry.

† Tables of H-atom parameters, anisotropic displacement parameters, a complete bond length and angle listing and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53919 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Fractional coordinates and equivalent isotropic displacement parameters* ($Å^2$) *for non-*H *atoms*

U is one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	*U*
Ru	0·00000	0·00000	0·00000	0·026
S(1)	−0·07517 (5)	−0·00101 (5)	0·14201 (4)	0·037
S(2)	−0·22020 (4)	0·07580 (5)	−0·09936 (4)	0·034
S(3)	−0·11327 (4)	−0·19199 (5)	−0·04361 (4)	0·035
S(4)	−0·17574 (6)	−0·53665 (6)	−0·40784 (4)	0·043
N(11)	0·15877 (18)	0·06882 (22)	0·28327 (15)	0·048
N(12)	0·00162 (25)	0·04317 (29)	0·34586 (17)	0·063
N(21)	−0·12864 (23)	0·24605 (24)	−0·18650 (19)	0·058
N(22)	−0·34587 (22)	0·20609 (24)	−0·27140 (17)	0·054
N(31)	−0·11106 (23)	−0·41116 (21)	−0·11399 (20)	0·058
N(32)	0·06893 (22)	−0·29938 (25)	−0·09250 (23)	0·064
O(1)	−0·30384 (17)	−0·57075 (20)	−0·41489 (15)	0·065
O(2)	−0·07525 (17)	−0·54965 (20)	−0·30203 (12)	0·057
O(3)	−0·14458 (19)	−0·58466 (18)	−0·49081 (14)	0·065
F(1)	−0·27341 (32)	−0·35235 (19)	−0·53121 (17)	0·136
F(2)	−0·23190 (32)	−0·32149 (20)	−0·36671 (19)	0·136
F(3)	−0·07996 (34)	−0·32725 (25)	−0·41768 (27)	0·163
C(1)	0·03825 (20)	0·04043 (21)	0·26598 (15)	0·041
C(2)	−0·23128 (19)	0·18336 (20)	−0·19289 (15)	0·040
C(3)	−0·04581 (18)	−0·30873 (20)	−0·08656 (15)	0·037
C(4)	−0·18913 (42)	−0·37585 (29)	−0·43146 (24)	0·085

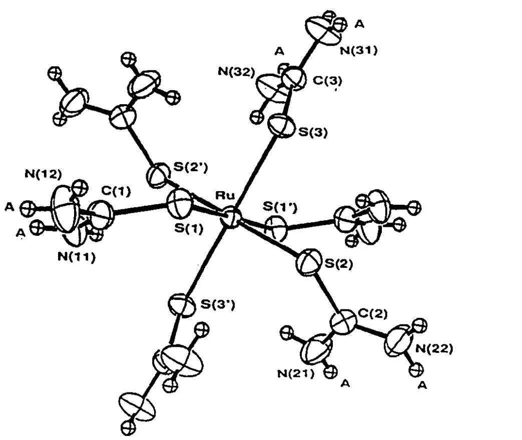

Fig. 1. A view of the $[Ru(tu)_6]^{2+}$ cation showing the atom numbering. Primed atoms are related to the corresponding unprimed atoms by the inversion centre at the Ru atom. H atoms are numbered H(11*A*), H(11*B*) *etc.* where the numeral is that of the attached N atom. For each NH_2 group only the *A*-numbered H atom is labelled. 50% probability ellipsoids are shown except for H atoms which are represented by spheres of arbitrary size.

Herbstein, 1972), $M = Tc^{3+}$ (Abrams, Davison, Faggiani, Jones & Lock, 1984) and $M = Ni^{2+}$ (Weininger, O'Connor & Amma, 1969). The metal coordination approximates to D_{3d} trigonal antiprismatic rather than regular octahedral, with the RuS_6 unit elongated along an axis defined by Ru and the centroid of S(1), S(2) and S(3) so that these atoms subtend S—Ru—S angles of 81·4 (1)–82·9 (1)°. Similar distortions occur in $[Ni(tu)_6]^{2+}$ and $[Tc(tu)_6]^{3+}$ The variety of metal ions which show the distortion [Ni^{2+} d^8, Ru^{2+} d^6, Tc^{3+} d^4] makes it unlikely that electronic factors are responsible. The mean Ru—S

Table 3. *Selected bond lengths, bond angles, torsion angles and hydrogen-bond interactions* (Å, °)

Ru—S(1)	2·415 (1)	Ru—S(2)	2·427 (1)
Ru—S(3)	2·420 (1)	S(1)—C(1)	1·720 (3)
S(2)—C(2)	1·717 (3)	S(3)—C(3)	1·716 (3)
S(4)—O(1)	1·445 (2)	S(4)—O(2)	1·437 (2)
S(4)—O(3)	1·422 (2)	S(4)—C(4)	1·804 (4)
N(11)—C(1)	1·307 (3)	N(12)—C(1)	1·317 (3)
N(21)—C(2)	1·312 (4)	N(22)—C(2)	1·320 (3)
N(31)—C(3)	1·317 (4)	N(32)—C(3)	1·321 (3)
F(1)—C(4)	1·335 (5)	F(2)—C(4)	1·313 (5)
F(3)—C(4)	1·275 (6)	N—H	0·70 (3)–0·87 (4)
S(1)—Ru—S(2)	81·3 (1)	S(1)—Ru—S(3)	82·9 (1)
S(2)—Ru—S(3)	81·7 (1)	Ru—S(1)—C(1)	116·0 (1)
Ru—S(2)—C(2)	114·1 (1)	Ru—S(3)—C(3)	119·1 (1)
O(1)—S(4)—O(2)	112·8 (2)	O(1)—S(4)—O(3)	114·0 (2)
O(1)—S(4)—C(4)	103·8 (2)	O(2)—S(4)—O(3)	115·3 (2)
O(2)—S(4)—C(4)	104·7 (2)	O(3)—S(4)—C(4)	104·7 (2)
S(1)—C(1)—N(11)	122·3 (2)	S(1)—C(1)—N(12)	118·4 (2)
N(11)—C(1)—N(12)	119·3 (2)	S(2)—C(2)—N(21)	121·4 (2)
S(2)—C(2)—N(22)	119·6 (2)	N(21)—C(2)—N(22)	119·0 (3)
S(3)—C(3)—N(31)	118·5 (2)	S(3)—C(3)—N(32)	122·3 (2)
N(31)—C(3)—N(32)	119·2 (3)	S(4)—C(4)—F(1)	110·4 (3)
S(4)—C(4)—F(2)	110·9 (3)	S(4)—C(4)—F(3)	112·6 (3)
F(1)—C(4)—F(2)	107·8 (4)	F(1)—C(4)—F(3)	107·7 (4)
F(2)—C(4)—F(3)	107·2 (3)		
S(3)—Ru—S(1)—C(1)	135·3 (1)	Ru—S(1)—C(1)—N(11)	−1·2 (2)
S(1)—Ru—S(2)—C(2)	143·4 (1)	Ru—S(2)—C(2)—N(21)	−21·3 (2)
S(2)—Ru—S(3)—C(3)	136·2 (1)	Ru—S(3)—C(3)—N(32)	2·9 (2)

A—H···*B*	*A*—H	H···*B*	*A*···*B*	*A*—H···*B*
N(11)—H(11*A*)···O(3^{i})	0·83 (3)	2·16 (3)	2·989 (3)	174 (2)
N(12)—H(12*A*)···O(1^{i})	0·77 (3)	2·36 (3)	3·131 (3)	172 (3)
N(12)—H(12*B*)···O(1^{ii})	0·80 (3)	2·29 (3)	3·012 (3)	150 (3)
N(21)—H(21*A*)···O(2^{iii})	0·70 (3)	2·30 (3)	2·958 (3)	158 (3)
N(22)—H(22*A*)···O(1^{iii})	0·87 (3)	2·47 (3)	3·308 (3)	161 (3)
N(22)—H(22*B*)···O(2^{iv})	0·74 (3)	2·24 (3)	2·978 (3)	172 (3)
N(31)—H(31*A*)···S(1^{v})	0·85 (3)	2·70 (3)	3·512 (2)	161 (3)

Symmetry code: (i) $\frac{1}{2}+x, -\frac{1}{2}-y, 1+z$; (ii) $-\frac{1}{2}-x, \frac{1}{2}+y, -z$; (iii) $x, 1+y, z$, (iv) $-\frac{1}{2}+x, -\frac{1}{2}-y, z$; (v) $-\frac{1}{2}-x, -\frac{1}{2}+y, -z$.

distance of 2·421 (3) Å appears long since a recent survey of Ru^{II}—S bond lengths gave a range of 2·262 (1)–2·393 (2) Å for such distances (Rawle & Cooper, 1987). It is, however, comparable with the mean Tc—S distance of 2·428 (8) Å in $[Tc(tu)_6]^{3+}$. Bond lengths and angles in the tu ligands of $[Ru(tu)_6]^{2+}$ agree well with those from room-temperature studies of the free ligand (Truter, 1967; Elcombe & Taylor, 1968) apart from a marginal shortening of the mean N—C bond length [1·316 (2) compared with 1·340 (6) and 1·333 (3) Å]. The Ru—S—C—N torsion angles (Table 3) indicate that, as expected (see Abrams *et al.*, 1984), the Ru atom lies close to each tu plane and the S—Ru—S—C tilt angles (see Berta, Spofford, Boldrini & Amma, 1970) indicate that each Ru(tu) plane approximately bisects an S—Ru—S angle [*e.g.* Ru(tu1) bisects S(2′)—Ru—S(3′)]. This is usual in $M(tu)_n$ ($n = 4$, 6) species and helps to lessen interligand repulsions (Berta *et al.*, 1970).

The $[CF_3SO_3]^-$ anions adopt near staggered C_{3v} conformations (Fig. 2) and their dimensions are unexceptional (see Lundgren, 1978) apart from variations in the C—F bond lengths which, together

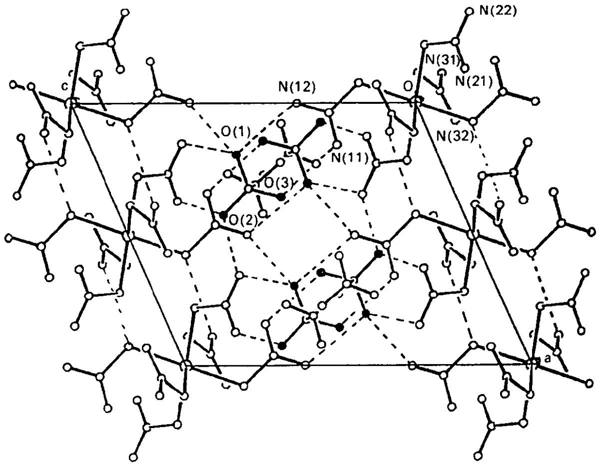

Fig. 2. The unit-cell contents projected down the *b* axis. O atoms are represented by filled circles and hydrogen bonds by dashed lines. The labelling is shown only for N atoms in the cation and for O atoms in the anion related to that defined by the coordinates in Table 2 by the transformation $\frac{1}{2}+x$, $-\frac{1}{2}-y$, $1+z$. H atoms are omitted.

with the large and very anisotropic displacement parameters of the F atoms, may indicate some disorder of the trifluoromethyl group.

The crystal structure consists of layers of $[Ru(tu)_6]^{2+}$ cations with the metal atoms lying exactly on the 001 planes. Anions lie between these layers; each is hydrogen bonded to four cations, three in the same layer and one in an adjacent layer. Adjacent cations are also directly linked by N—H···S hydrogen bonds (see Fig. 2 and Table 3).

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Structures of *c*-Acetonitrile-*ab*-dibromo-*d*-carbonyl-*ef*-bis(triphenylphosphine)osmium(2+) and *b*-Acetonitrile-*af*-dibromo-*d*-carbonyl-*ce*-bis(triphenylphosphine)osmium(2+)

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Abstract. (II). $[cis\text{-}OsBr_2(C_2H_3N)(CO)\{P(C_6H_5)_3\}_2]$, $M_r = 943{\cdot}65$, orthorhombic, $P2_12_12_1$, $a = 13{\cdot}278\,(5)$, $b = 24{\cdot}462\,(8)$, $c = 10{\cdot}823\,(3)$ Å, $V = 3515\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}783$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 60{\cdot}03$ cm^{-1}, $F(000) = 1832$, $T = 296$ K, $R = 0{\cdot}031$, 2802 unique observed reflections. (III) $[trans\text{-}OsBr_2(C_2H_3N)(CO)\{P(C_6H_5)_3\}_2]$, $M_r = 943{\cdot}65$, monoclinic, $P2_1/n$, $a = 10{\cdot}326\,(5)$, $b = 15{\cdot}461\,(10)$, $c = 22{\cdot}961\,(8)$ Å, $\beta = 91{\cdot}15\,(5)°$, $V = 3665\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}710$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 57{\cdot}58$ cm^{-1}, $F(000) = 1832$, $T = 296$ K, $R = 0{\cdot}060$, 3089 unique observed reflections. Compounds (II)

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