ordered atoms were refined with 50% occupancy factors and isotropic thermal parameters while the other non-H atoms were refined anisotropically for a total of 437 parameters, $R = 0.059$, $wR = 0.073$, GOF = 1.3, where non-Poisson $w^{-1} = [\sigma^2(I) + (0.08I)^2]/4F^2$. Final $(\Delta/\sigma)_{max} < 0.38$, $\Delta\rho_{max} = 1.19$ (11) (all six peaks above 0.64 were within 0.99 Å of the Rh position) and $\Delta\rho_{min} = -0.48$ (11) e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*.* Table 1 gives the atomic coordinates and Table 2 selected bond distances and angles. Figs. 1 and 2 show the molecule with the numbering scheme.

Related literature. The structure of this diene complex can be compared most directly with that of [RhI(PPh$_3$)$_2$(C$_3$H$_4$)] (Kashiwagi, Yasuoka, Kasai & Kukudo, 1969). Other Rh–diene complexes have been reported by Hewitt & DeBoer (1971) and by Racanelli, Pantini, Immirzi, Allegra & Porri (1969). Transition-metal–allene complexes are reviewed by Shaw & Stringer (1973).

We thank the National Science Foundation and the State of Arkansas for financial support.

* Lists of distances and angles in the phenyl groups and in the saturated portion of the cyclononadiene ring, anisotropic temperature factors, structure factors, and H-atom positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51893 (50 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Enraf–Nonius (1982). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

HEWITT, T. G. & DEBOER, J. J. (1971). *J.Chem. Soc A*, pp. 817–822.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

KASHIWAGI, T., YASUOKA, N., KASAI, N. & KUKUDO, M. (1969). *J. Chem. Soc. Chem. Commun.* pp. 317–318.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M.M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

RACANELLI, P., PANTINI, G., IMMIRZI, G., ALLEGRA, G. & PORRI, L. (1969). *J. Chem. Soc. Chem. Commun.* pp. 361–362.

SHAW, B. L. & STRINGER, A. J. (1973). *Inorg. Chim. Acta Rev.* **7**, 1–10.

Acta Cryst. (1989). C**45**, 1428–1429

Structure of Hexakis(acetonitrile)ruthenium(II) *p*-Toluenesulfonate Dihydrate at 100 K

BY WERNER LUGINBÜHL AND ANDREAS LUDI

Institut für Anorganische Chemie, Universität Bern, CH 3000 *Bern* 9, *Switzerland*

AND ANDREA RASELLI AND HANS-BEAT BÜRGI

Laboratorium für Kristallographie, Universität Bern, CH 3000 *Bern* 9, *Switzerland*

(*Received* 23 *January* 1989; *accepted* 6 *April* 1989)

Abstract. [Ru(C$_2$H$_3$N)$_6$][C$_7$H$_7$O$_3$S]$_2$.2H$_2$O, M_r = 725.8, triclinic, $P\bar{1}$, $a = 8.274$ (3), $b = 8.957$ (3), $c = 11.538$ (7) Å, $\alpha = 107.92$ (4), $\beta = 89.47$ (3), $\gamma = 92.44$ (3)°, $V = 812.9$ Å^3, $Z = 1$, D_m(298 K) = 1.430 (1), D_x(100 K) = 1.482 g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 6.47$ cm^{-1}, $F(000) = 374$, $R = 0.019$ for 3316 reflections with $I > 3\sigma(I)$ measured at 100 K. The Ru environment is close to octahedral with average Ru—N = 2.029 (2), C—N = 1.139 (1) and C—C = 1.456 (2) Å. The N—Ru—N angles are between 89.19 (3) and 90.87 (3)°.

Experimental. The Ru(acetonitrile)$_6^{2+}$ complex was prepared as previously described (Rapaport, Helm, Merbach, Bernhard & Ludi, 1988). Density at 298 K by flotation. Crystals grown by cooling an acetone–water solution from 313 to 277 K. A section (0.29 × 0.26 × 0.22 mm) cut from a prismatic crystal was sealed in a Lindemann capillary and measured on an Enraf–Nonius CAD-4 diffractometer equipped with a liquid-nitrogen attachment. Unit-cell data from 22 reflections with $13 < \theta < 19°$. Data collection [ω scan, $(1.6 + 0.35\tan\theta)°$ (20–27°), $(1.7 + 0.35\tan\theta)°$

0108-2701/89/091428-02$03.00

Table 1. *Final atomic positional parameters and B_{eq} values, with e.s.d.'s in parentheses*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq} (Å^2)
Ru	0·00000	0·00000	0·00000	0·930 (2)
N1	0·2135 (2)	−0·0924 (1)	−0·0667 (1)	1·30 (2)
N2	0·0762 (1)	0·0330 (1)	0·1733 (1)	1·22 (2)
N3	0·0945 (1)	0·2164 (1)	0·0105 (1)	1·23 (2)
C1	0·3365 (2)	−0·1385 (2)	−0·1012 (1)	1·38 (2)
C2	0·4954 (2)	−0·1952 (2)	−0·1433 (1)	1·80 (2)
C3	0·1098 (2)	0·0591 (2)	0·2733 (1)	1·25 (2)
C4	0·1435 (2)	0·0959 (2)	0·4026 (1)	1·66 (2)
C5	0·1552 (2)	0·3339 (2)	0·0135 (1)	1·31 (2)
C6	0·2359 (2)	0·4830 (2)	0·0165 (1)	1·82 (2)
C7	0·3557 (2)	0·2779 (2)	0·7092 (1)	1·19 (2)
C8	0·1994 (2)	0·2250 (2)	0·7268 (1)	1·23 (2)
C9	0·0681 (2)	0·3071 (2)	0·7101 (1)	1·34 (2)
C10	0·0897 (2)	0·4420 (2)	0·6740 (1)	1·41 (2)
C11	0·2465 (2)	0·4905 (2)	0·6525 (1)	1·75 (2)
C12	0·3791 (2)	0·4090 (2)	0·6699 (1)	1·62 (2)
C13	−0·0534 (2)	0·5326 (2)	0·6582 (2)	2·04 (2)
S	0·52171 (4)	0·18332 (4)	0·74810 (3)	1·284 (5)
O1	0·6664 (1)	0·2548 (1)	0·7100 (1)	1·81 (2)
O2	0·5127 (1)	0·2168 (1)	0·87929 (9)	1·95 (2)
O3	0·4988 (1)	0·0170 (1)	0·6819 (1)	2·06 (2)
O4	0·7470 (2)	0·1885 (1)	0·4569 (1)	2·18 (2)

Table 2. *Bond lengths* (Å) *and angles* (°)

	Uncorrected	Corrected for thermal motion
Ru—N1	2·026 (1)	2·027 (1)
Ru—N2	2·031 (1)	2·032 (1)
Ru—N3	2·027 (1)	2·028 (1)
N1—C1	1·137 (1)	1·138 (1)
N2—C3	1·140 (1)	1·140 (1)
N3—C5	1·138 (1)	1·139 (1)
C1—C2	1·454 (1)	1·455 (1)
C3—C4	1·453 (1)	1·453 (1)
C5—C6	1·458 (1)	1·459 (1)

N1—Ru—N2	90·87 (3)	Ru—N3—C5	176·20 (8)
N1—Ru—N3	89·29 (3)	N1—C1—C2	178·87 (10)
N2—Ru—N3	89·19 (3)	N2—C3—C4	176·70 (10)
Ru—N1—C1	177·07 (8)	N3—C5—C6	178·84 (11)
Ru—N2—C3	174·89 (8)		

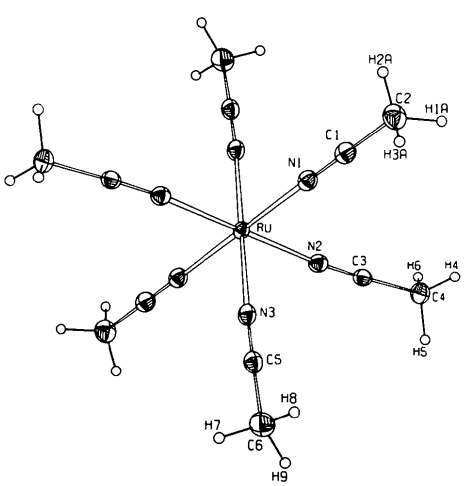

Fig. 1. The molecular structure and labelling scheme for $[Ru(NCCH_3)_6]^{2+}$.

(15–20°), (1·8 + 0·35tanθ)° (0–15°)] to $\theta = 27°$ with $0 \le h \le 10$, $-11 \le k \le 11$, $-14 \le l \le 14$. Three check reflections (600, 1$\bar{2}$1, 03$\bar{5}$) showed total intensity loss of 0·9% in 98 h. No absorption correction, T_{min} = 97% from ψ scans. 3795 reflections measured, 3540 unique reflections, 3316 with $I > 3\sigma(I)$ classified as observed. Structure solved by Patterson and Fourier methods. Full-matrix refinement minimized $\sum w(|F_o| - |F_c|)^2$, $w = 4F_o/[\sigma^2(I) + (0{\cdot}01I)^2]$. H atoms from difference maps included in refinement with individual isotropic temperature factors, all other atoms refined anisotropically. H atoms in two methyl groups (C2 and C13) showed disorder, which was accounted for by a split-atom model; torsional angles between the two orientations of the methyl groups are close to 60°. Final $R = 0{\cdot}019$, $wR = 0{\cdot}024$, S = 2·31, 292 parameters. Final $(\Delta/\sigma)_{max} = 0{\cdot}04$ (H22), $\Delta\rho_{max} = 0{\cdot}31$ e Å^{-3} on the C—S bond of the anion. Atomic scattering factors and anomalous-dispersion corrections included in the Enraf–Nonius (1982) programs used for all calculations. Final atomic parameters are given in Table 1.* Important interatomic distances, corrected for thermal motion (Maverick & Trueblood, 1987) and angles are collected in Table 2; the molecular structure with numbering scheme is shown in Fig. 1 (*ORTEP*, 1988).

Related literature. The room-temperature structure of $[Ru(NCCH_3)_6][7\text{-}(\eta^6\text{-}C_6Me_6)\text{-}nido\text{-}7\text{-}RuB_{10}H_{13}]_2$ without thermal-motion correction shows a shorter Ru—N distance of 2·013 Å (Bown, Fontaine, Greenwood, Kennedy & Thornton-Pett, 1987). Ligand exchange for the complex ion in solution is extremely slow (Rapaport, Helm, Merbach, Bernhard & Ludi, 1988).

This work was supported by the Swiss National Science Foundation (Grant No. 2.622-087).

* Lists of structure factors, anisotropic thermal parameters, H-atom positions, and a complete table of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51875 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

BOWN, M., FONTAINE, X. L. R., GREENWOOD, N. N., KENNEDY, J. D. & THORNTON-PETT, M. (1987). *J. Chem. Soc. Dalton Trans.* pp. 1169–1174.

Enraf–Nonius (1982). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

MAVERICK, E. F. & TRUEBLOOD, K. N. (1987). *THMA*11. Univ. of California, Los Angeles, USA.

ORTEP (1988). Version of DAVENPORT, G. & HALL, S. R. in *XTAL*2.4, *Users Manual*, edited by S. R. HALL & J. M. STEWART. Univs. of Western Australia, Australia, and Maryland, USA.

RAPAPORT, I., HELM, L., MERBACH, A. E., BERNHARD, P. & LUDI, A. (1988). *Inorg. Chem.* **27**, 873–879.