plane [6(h)], but the O atoms O(4) are disordered in position 12(i) with 50% occupancy. The intermolecular contacts O(4)—H$\cdots$O(1), 2·77 (1) Å, between solvent and cluster methoxide groups occur six times per unit cell. The contacts between the dinuclear cation unit $[Na_2(CH_3OH)_9]^{2+}$ and solvent molecules are through O(3)—H$\cdots$O(4), 2·727 (3) Å.

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Dichloro(p-cymene)(p-toluidine)ruthenium(II)

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Abstract. $[Ru(Cl)_2(C_{10}H_{14})(C_7H_9N)]$, $M_r = 413·33$, monoclinic, $P2_1/n$, $a = 8·774$ (2), $b = 12·399$ (3), $c = 15·628$ (3) Å, $\beta = 90·97$ (2)°, $V = 1699·91$ (2) Å^3, $Z = 4$, $D_x = 1·61$ g cm^{-3}, Mo $K\alpha$ (graphite monochromator), $\lambda = 0·71073$ Å, $\mu = 12·1$ cm^{-1}, $F(000) = 840$, $T = 295$ K, $R = 2·33\%$ for 282 variables and 2682 unique reflections having $I > 3\sigma(I)$. The structure confirms that the observed lability of the amine ligand is ascribable to the weak Ru—N bond, reflected in the relatively short Ru—arene bond, 1·637 (2) Å, rather than any steric constraint. Bond alternation, C—C (short) 1·395 (4) Å and C—C (long) 1·414 (4) Å, is present in the cymene ring.

Introduction. The expanding interest in the chemistry of Group 8 arene complexes (Le Bozec, Touchard & Dixneuf, 1989) has led to a need for the structural characterization of a number of simple prototype complexes. Our studies of systems containing the arene ruthenium unit with simple amine ligands (Bates, Begley & Wright, 1990) have provided (p-cymene)RuCl$_2$(p-toluidine) as an example. The complex is readily made by simple cleavage of the dimer

$[(p\text{-cymene})RuCl_2]_2$ in solutions containing an excess of the amine. However, the amine is only weakly bound to the metal and is readily substituted in solution (Bates, Begley & Wright, 1990).
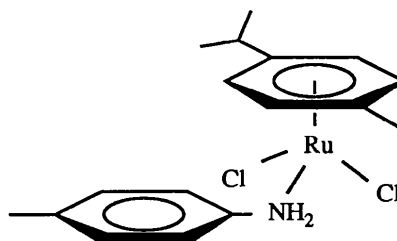
Experimental. The title compound was prepared by a similar method to that used by Bates, Begley & Wright (1990) in preparing the benzene analogue. A red crystal (0·9 × 0·2 × 0·05 mm) was grown from a dichloromethane/hexane solution. Space group $P2_1/n$ [non-standard setting of $P2_1/c$ (No. 14)]. Data collected on a Hilger & Watts Y290 diffractometer using $\omega/2\theta$ scans; 12 reflections having $10 < \theta < 12°$ used to determine lattice parameters; no absorption correction was considered necessary; data with $0 < h$

< 10, 0 < k < 14, $-18 < l < 18$; no intensity loss for three control reflections; maximum $(\sin\theta)/\lambda$ 0·5946 Å^{-1}; 3018 unique reflections measured; 2682 data having $I > 3\sigma(I)$ used for refinement; structure solved by Patterson and Fourier methods and refined by least squares with a weighting scheme based on a Chebyshev polynomial with coefficients 31·9713, 43·0118, 13·0014 and $-1\cdot3776$, using *CRYSTALS* (Watkin, Carruthers & Betheridge, 1985); H atoms located in difference Fourier maps and refined isotropically; 282 variables; refinement on F magnitudes; R, $wR = 2\cdot33$, 2·59%; max. Δ/σ in last cycle 0·3; largest peak in final difference Fourier synthesis has a height of 0·43 e Å^{-3} (all top peaks in the immediate vicinity of the heavy atoms); scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The molecular structure and the atomic numbering scheme are shown in Fig. 1. Atomic coordinates are listed in Table 1, and bond lengths and angles are given in Table 2.*

Discussion. The complex is best thought of as containing an octahedrally coordinated ruthenium centre with the arene ligand occupying three sites. The small steric demand of the amine ligand is reflected in the 80·12 (6) and 83·16 (7)° N—

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53438 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
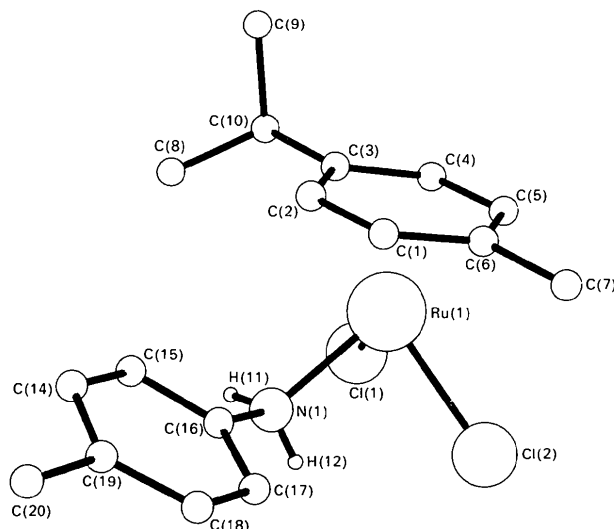
Table 1. *Fractional coordinates for* [Ru(Cl)$_2$(C$_{10}$H$_{14}$)(C$_7$H$_9$N)]

	x	y	z
Ru(1)	0·21325 (2)	0·09571 (1)	0·10557 (1)
Cl(1)	0·23078 (8)	−0·09596 (5)	0·13235 (5)
Cl(2)	0·29109 (7)	0·06379 (5)	−0·03950 (4)
C(1)	0·1834 (3)	0·2671 (2)	0·1238 (2)
C(2)	0·1838 (3)	0·2148 (2)	0·2040 (2)
C(3)	0·0859 (3)	0·1290 (2)	0·2211 (2)
C(4)	−0·0118 (3)	0·0954 (2)	0·1531 (2)
C(5)	−0·0140 (3)	0·1471 (2)	0·0739 (2)
C(6)	0·0845 (3)	0·2346 (2)	0·0580 (2)
C(7)	0·0853 (5)	0·2870 (3)	−0·0284 (2)
C(8)	0·2341 (4)	0·0637 (3)	0·3504 (2)
C(9)	−0·0270 (5)	0·1316 (5)	0·3632 (2)
C(10)	0·0821 (3)	0·0720 (2)	0·3066 (2)
N(1)	0·4499 (2)	0·0821 (2)	0·1324 (2)
C(14)	0·6687 (3)	0·2983 (2)	0·2400 (2)
C(15)	0·5747 (3)	0·2117 (2)	0·2272 (2)
C(16)	0·5433 (2)	0·1753 (2)	0·1457 (2)
C(17)	0·6027 (3)	0·2276 (2)	0·0769 (2)
C(18)	0·6968 (3)	0·3143 (3)	0·0905 (2)
C(19)	0·7336 (3)	0·3503 (2)	0·1717 (2)
C(20)	0·8436 (5)	0·4389 (4)	0·1868 (4)

Fig. 1. View of a single molecule of dichloro(p-cymene)(p-toluidine)ruthenium(II) showing the labelling scheme. All atoms are shown as spheres of arbitrary size. H atoms attached to carbons are omitted for clarity.

Ru—Cl angles. These are significantly smaller than the comparable angles in the phosphine-substituted complexes [Ru(cymene)Cl$_2$(PPh$_2$CH$_2$-SiMe$_2$OH)] (Brost, Bruce & Stobart, 1986) and [Ru(cymene)Cl$_2$(PMePh$_2$)] (Bennett, Robertson & Smith, 1972), and also significantly smaller than the angles in [Ru(cymene)Cl(pyz)$_2$]PF$_6$ (pyz = pyrazine), but not [Ru(cymene)Cl$_2$(PMePh$_2$)]; in the latter case the creasing of the ring was ascribed to the strong *trans* influence of the phosphine. The planarity of the ring in this case probably reflects the lower *trans* influence of the amine relative to the phosphine.

A striking feature is the C—C bond alternation around the ring with an average C—C (short) of 1·395 (4) Å and C—C (long) of 1·414 (4) Å. This compares with the related complex [Ru(cymene)Cl(μ-N$_3$)]$_2$ (Bates, Begley & Wright, 1990), in which no signficant deviations from the average C—C distance of 1·411 (5) Å are observed. When the molecules are viewed down the arene–Ru axis the latter complex is almost perfectly eclipsed with the other ligands lying underneath C(2), C(4) and C(6) while the former is best regarded as staggered. (The description is only approximate because of the different requirements of the chloride and amine ligands.)

The pair of complexes therefore provides a clear example of bond alternation occurring with staggered rather than eclipsed conformations in three-legged piano-stool complexes. This is in accord with generalizations drawn from chromium–arene complex chemistry (Muetterties, Bleeke, Wucherer & Albright, 1982), as is the fact that the longer C—C bonds in (p-cymene)RuCl$_2$(p-toluidine) are eclipsed by the Ru—L bonds.

Table 2. *Bond lengths (Å) and angles (°) for* $[Ru(Cl)_2(C_{10}H_{14})(C_7H_9N)]$

Ru1—Cl1	2.418 (1)	C4—C5	1.393 (4)
Ru1—Cl2	2.412 (1)	C5—C6	1.412 (4)
Ru1—C1	2.161 (2)	C6—C7	1.498 (4)
Ru1—C2	2.151 (2)	C8—C10	1.493 (4)
Ru1—C3	2.180 (2)	C9—C10	1.508 (5)
Ru1—C4	2.122 (2)	N1—C16	1.430 (3)
Ru1—C5	2.143 (2)	C14—C15	1.366 (4)
Ru1—C6	2.184 (2)	C14—C19	1.379 (4)
Ru1—N1	2.118 (2)	C15—C16	1.374 (3)
C1—C2	1.412 (4)	C16—C17	1.366 (4)
C1—C6	1.394 (4)	C17—C18	1.369 (4)
C2—C3	1.397 (4)	C18—C19	1.378 (4)
C3—C4	1.417 (3)	C19—C20	1.479 (4)
C3—C10	1.511 (4)		

Cl1—Ru1—Cl2	89.04 (2)	Ru1—C1—C6	72.2 (2)
Cl1—Ru1—C1	162.10 (8)	C2—C1—C6	121.0 (3)
Cl1—Ru1—C2	123.99 (7)	Ru1—C2—C1	71.3 (1)
Cl1—Ru—C3	94.28 (7)	Ru1—C2—C3	72.3 (1)
Cl1—Ru1—C4	89.69 (8)	C1—C2—C3	121.9 (2)
Cl1—Ru1—C5	112.87 (8)	Ru1—C3—C2	70.1 (1)
Cl1—Ru1—C6	149.93 (7)	Ru1—C3—C4	68.6 (1)
Cl1—Ru1—N1	80.12 (6)	Ru1—C3—C10	131.5 (2)
Cl2—Ru1—C1	108.79 (7)	C2—C3—C4	116.5 (2)
Cl2—Ru1—C2	145.52 (7)	C2—C3—C10	123.2 (2)
Cl2—Ru1—C3	165.36 (7)	C4—C3—C10	120.3 (2)
Cl2—Ru1—C4	127.50 (7)	Ru1—C4—C3	73.0 (1)
Cl2—Ru1—C5	96.27 (7)	Ru1—C4—C5	71.8 (1)
Cl2—Ru1—C6	87.84 (7)	C3—C4—C5	122.0 (3)
Cl2—Ru1—N1	83.15 (7)	Ru1—C5—C4	70.1 (1)
C1—Ru1—C2	38.2 (1)	Ru1—C5—C6	72.5 (1)
C1—Ru1—C3	68.9 (1)	C4—C5—C6	120.8 (2)
C1—Ru1—C4	80.8 (1)	Ru1—C6—C1	70.4 (1)
C1—Ru1—C5	67.9 (1)	Ru1—C6—C5	69.4 (1)
C1—Ru1—C6	37.4 (1)	Ru1—C6—C7	129.7 (2)
C1—Ru1—N1	99.9 (1)	C1—C6—C5	117.8 (2)
C2—Ru1—C3	37.6 (1)	C1—C6—C7	121.8 (3)
C2—Ru1—C4	68.1 (1)	C5—C6—C7	120.3 (3)
C2—Ru1—C5	80.7 (1)	C3—C10—C8	113.9 (3)
C2—Ru1—C6	68.6 (1)	C3—C10—C9	108.3 (3)
C2—Ru1—N1	92.4 (1)	C8—C10—C9	109.6 (3)
C3—Ru1—C4	38.5 (1)	Ru1—N1—C16	121.5 (2)
C3—Ru1—C5	69.31 (9)	C15—C14—C19	120.7 (3)
C3—Ru1—C6	82.2 (1)	C14—C15—C16	120.4 (3)
C3—Ru1—N1	111.46 (9)	N1—C16—C15	120.4 (2)
C4—Ru1—C5	38.1 (1)	N1—C16—C17	119.6 (2)
C4—Ru1—C6	69.0 (1)	C15—C16—C17	120.0 (2)
C4—Ru1—N1	147.8 (1)	C16—C17—C18	119.1 (3)
C4—Ru1—C6	38.1 (1)	C17—C18—C19	121.9 (3)
C5—Ru1—N1	167.0 (1)	C14—C19—C18	117.9 (3)
C6—Ru1—N1	129.04 (9)	C14—C19—C20	120.0 (3)
Ru1—C1—C2	70.5 (1)	C18—C19—C20	122.0 (3)

The Ru–ring distance of 1.637 (2) Å is slightly shorter than that observed for [Ru(cymene)Cl(μ-N$_3$)]$_2$ (Bates, Begley & Wright, 1990) (1.66 Å) and [Ru(cymene)Cl(pyz)$_2$]PF$_6$ (1.68 Å). Since the major metal–ligand interactions between the arene and the metal involve both σ and π donation from the arene, this shorter distance must reflect the stronger metal–arene interaction which in turn is a consequence of the relatively poor donating capacity of the amine ligand. The Ru—Cl bond lengths, however, do not differ significantly from those in the other determinations already mentioned and there is no detectable lengthening of the Ru—N bond.

The relatively small coordination site occupied by the amine ligand makes if seem likely that there are no great steric demands on the ligand that might lead to the observed lability. Rather, the lability must be attributed to the weak Ru—N bond. However, the detection of this weakness is not possible in terms of the bond length itself, and can only be inferred from the relatively short Ru—arene distance.

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Structure of *N,N*-Dimethyl-1-[2-(phenylthio)phenyl]-2-propylammonium Hydrogen Maleate

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Abstract. $C_{21}H_{25}NO_4S$, $M_r = 387.49$, monoclinic, $P2_1/c$, $a = 10.092$ (2), $b = 32.095$ (7), $c = 6.498$ (1) Å, $\beta = 100.97$ (2)°, $V = 2066.4$ (8) Å^3, $Z = 4$, $D_x =$ 1.246 g cm^{-3}, Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å, $\mu = 15.58$ cm^{-1}, $F(000) = 824$, $T = 295$ K, $R = 0.045$, $wR = 0.066$ for 2411 observed reflections. The crystal