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## Chloro(*p*-chloroanilido-*N*)bis( $\eta^5$ -pentamethylcyclopentadienyl)uranium(IV)

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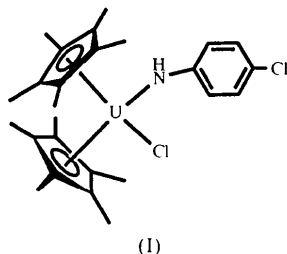
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### Abstract

The title complex,  $[UCl(C_{10}H_{15})_2(C_6H_4ClN)]$ , adopts the pseudo-tetrahedral geometry typical for bis(pentamethylcyclopentadienyl)uranium complexes, with a chloride and a *para*-substituted amide ligand occupying the plane bisecting the metallocene unit. Some important bond lengths include U—N 2.237 (3) and U—Cl 2.613 (1) Å.

### Comment

We have been examining the chemistry of uranium mono- and dialkyls with substituted anilines as a synthetic tool to yield mono and bis amide and imide complexes of actinides. Treatment of  $(C_5Me_5)_2U(CH_3)Cl$  with an excess of *p*-chloroaniline in hexane solution gave the title compound, (I), in modest yield.



In the solid state, the title molecule adopts a typical metallocene geometry, with an average  $M-C_{ring}$  bond length of 2.736 Å. This value is well within

the range typically found in bis(pentamethylcyclopentadienyl)uranium(IV) complexes (Arney *et al.*, 1992; Hall *et al.*, 1993; Arney & Burns, 1995). The  $C_p-M-C_p$  angle of  $135.1^\circ$  ( $C_p$  is the ring centroid) falls within the range of  $132-141^\circ$  reported for similar uranium(IV) structures (Arney & Burns, 1995; Arney *et al.*, 1992). The U—Cl bond length of 2.613 (1) Å is about 0.04 Å shorter than the U—Cl distance of 2.658 (2) Å reported for  $(C_5Me_5)_2U[(CH_2)(CH_2)P(Me)(Ph)]Cl$  (Cramer *et al.*, 1989), possibly reflecting the increased electron density in the title compound. However, the U—Cl bond length of the title compound is longer than the U—Cl distance of 2.583 (6) Å reported for the dichloride  $(C_5Me_5)_2UCl_2$  (Spirlet *et al.*, 1992). The U—N distance of 2.237 (3) Å is shorter than the U—N bond distance of 2.267 (6) Å reported for  $(C_5Me_5)_2U[NH(2,6-Me_2C_6H_3)]_2$  (Straub *et al.*, 1996). These differences in bond-length data qualitatively indicate that uranium(IV) complexes are structurally sensitive to the electronic and steric influences of the ancillary ligands.

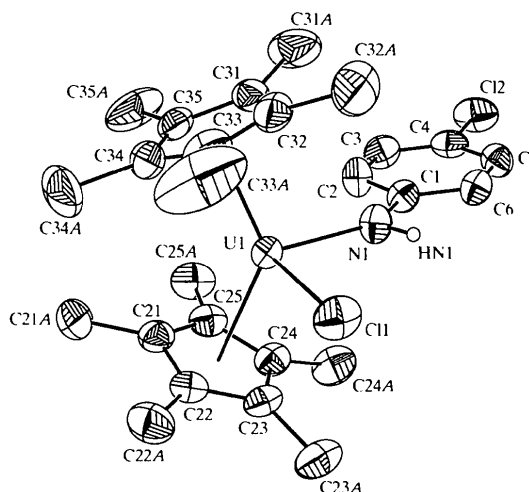


Fig. 1. View of the title compound showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level. H atoms, except for the amide H atom, have been omitted for clarity. The  $C_5Me_5$  ring (C31–C35A) is disordered over two positions and only the major component is shown.

### Experimental

The title compound was prepared by treatment of  $(C_5Me_5)_2U(CH_3)Cl$  with *p*-chloroaniline in hexane. The crude product was isolated by filtration of the reaction mixture, and recrystallization from a saturated hexane solution at 238 K yielded the pure product as dark-red blocks.

### Crystal data

$[UCl(C_{10}H_{15})_2(C_6H_4ClN)]$   
 $M_r = 670.48$

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073$  Å

Monoclinic  
 $P2_1/c$   
 $a = 9.6467(4) \text{ \AA}$   
 $b = 12.7276(6) \text{ \AA}$   
 $c = 20.804(1) \text{ \AA}$   
 $\beta = 90.129(1)^\circ$   
 $V = 2554.3(2) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.743 \text{ Mg m}^{-3}$   
 $D_m$  not measured

#### Data collection

Bruker P4/CCD/PC diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: empirical (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.19$ ,  $T_{\max} = 0.35$   
 13 422 measured reflections  
 5158 independent reflections

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.024$   
 $wR(F^2) = 0.064$   
 $S = 1.272$   
 5158 reflections  
 316 parameters  
 H atoms treated by a mixture of independent and constrained refinement

Cell parameters from 2734 reflections  
 $\theta = 2.0\text{--}26.5^\circ$   
 $\mu = 6.58 \text{ mm}^{-1}$   
 $T = 203(2) \text{ K}$   
 Block  
 $0.25 \times 0.16 \times 0.16 \text{ mm}$   
 Red

4564 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.021$   
 $\theta_{\text{max}} = 26.5^\circ$   
 $h = -12 \rightarrow 12$   
 $k = 0 \rightarrow 15$   
 $l = 0 \rightarrow 25$   
 Intensity decay: none

$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.750 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.661 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

U1—N1	2.237 (3)	C1—C2	1.396 (5)
U1—C11	2.613 (1)	C2—C3	1.381 (6)
C12—C4	1.748 (4)	C3—C4	1.374 (6)
N1—HN1	0.93 (5)	C4—C5	1.371 (6)
N1—C1	1.396 (5)	C5—C6	1.386 (5)
C1—C6	1.395 (5)		
N1—U1—C11	93.11 (9)	C3—C2—C1	121.4 (4)
HN1—N1—C1	113 (3)	C4—C3—C2	119.7 (4)
HN1—N1—U1	103 (3)	C5—C4—C3	120.3 (4)
C1—N1—U1	143.7 (3)	C5—C4—C12	120.0 (3)
C6—C1—N1	122.4 (3)	C3—C4—C12	119.7 (3)
C6—C1—C2	117.6 (4)	C4—C5—C6	120.2 (4)
N1—C1—C2	120.0 (4)	C5—C6—C1	120.8 (4)

The pentamethylcyclopentadienyl ring defined by atoms C31—C35 was refined as two disordered pentamethylcyclopentadienyl rings. The second ring consists of atoms C31'—C35' and C31B—C35B. The site-occupancy factors of the two rings refined to 0.832(6) for the former and 0.168(6) for the latter. The second ring was refined with isotropic displacement parameters and no H atoms. H atoms were fixed in positions of ideal geometry, with C—H distances of 0.96 (methyl) and 0.93  $\text{\AA}$  (aromatic). The H-atom positions were refined using a riding model and the  $U_{\text{iso}}$  values were fixed to 1.2 (aromatic) or 1.5 (methyl) times the  $U_{\text{eq}}$  of the parent C atom. The amido H atom was found in the difference map and was refined with  $U_{\text{iso}}$  set to 0.08  $\text{\AA}^2$ . H atoms were not placed on the methyl groups of the disordered  $\text{C}_5\text{Me}_5$  ligands.

Data collection: SMART (Bruker, 1996a). Cell refinement: SAINT (Bruker, 1996b). Data reduction: SAINT. Program(s)

used to solve structure: SHELXTL (Bruker, 1997). Program(s) used to refine structure: SHELXTL. Molecular graphics: SHELXTL. Software used to prepare material for publication: SHELXTL.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1192). Services for accessing these data are described at the back of the journal.

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### Tetrakis( $\mu$ -6-chloropyridin-2-olato)-methoxydiruthenium(II,III)

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#### Abstract

The title complex, tetrakis( $\mu$ -6-chloropyridin-2-olato)- $1\kappa^4\text{O}$ ,  $2\kappa^4\text{N}$ -methoxy- $1\kappa\text{O}$ -diruthenium(II,III)(Ru—Ru),  $[\text{Ru}_2(\text{C}_5\text{H}_3\text{ClNO})_4(\text{CH}_3\text{O})]$ , has a totally polar arrangement of the chp ligands (chp is 6-chloro-2-hydroxypyridine), and one methoxy group occupies the sterically available axial position. The bond distance between the