

There are two (9-methyladenine)zinc complexes reported in the Cambridge Structural Database by McCall & Taylor (1975, 1976) obtained from the same slightly acid solution. The (9-methyladenine)-zinc complex $[\text{ZnCl}_3(\text{C}_6\text{H}_7\text{N}_5)]^- \cdot [\text{C}_6\text{H}_8\text{N}_5]^+ \cdot \text{H}_2\text{O}$ shows that the N(1) site of one adenine molecule is protonated, while the N(1) site of the second molecule is strongly bound by Zn (McCall & Taylor, 1975) and the *catena*-dichloro- μ -(9-methyladenine)-zinc(II) complex exhibits two coordination sites, N(1) and N(7), bridging neighbouring adenine molecules (McCall & Taylor, 1976).

In addition, Taylor (1973) has published the crystal structure of (trichloroadeninium)zinc(II) crystallized from hydrochloric acid solution. The Zn ion is coordinated to N(7) and the adenine ring is protonated at N(1) and is significantly non-planar with a dihedral angle of $4.3(1)^\circ$ between the adenine moieties. The complex presented in this work is very similar to the latter compound as shown in Table 3.

The structure may mimic the way in which the zinc cation can interact with an adenine molecule in nucleic acids.

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Structure of Tris(η^5 -cyclopentadienyl)uranium Thiocyanate

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Abstract. Tris(η^5 -cyclopentadienyl)(thiocyanato-*N*)-uranium, $[\text{U}(\text{NCS})(\text{C}_5\text{H}_5)_3]$, $M_r = 491.40$, orthorhombic, $Pca2_1$, $a = 15.427(2)$, $b = 8.323(3)$, $c = 11.772(5)$ Å, $V = 1511(1)$ Å³, $Z = 4$, $D_x = 2.159$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 103.0$ cm⁻¹, $F(000) = 904$, $T = 295(1)$ K, $R = 0.043$ for 902 reflections [$I > 3\sigma(I)$]. The U atom is surrounded by three η^5 -covalently bonded cyclopentadienyl rings and by one thiocyanate N atom in a

Table 3. Comparison of selected bond distances (Å) and angles ($^\circ$) in (adenine)zinc(II) complexes

	Protonation			
	site	Zn—N	C(5)—N(7)—C(8)	C(6)—N(1)—C(2)
$\text{C}_6\text{H}_{10}\text{Cl}_3\text{N}_5\text{OZn}^a$	N(1)(H ⁺)	2.077 (3)	104.1 (2)	124.4 (2)
$\text{C}_3\text{H}_6\text{Cl}_3\text{N}_5\text{Zn}^b$	N(1)(H ⁺)	2.094 (5)	104.2 (3)	124.6 (3)
$\text{C}_{17}\text{H}_{17}\text{Cl}_3\text{N}_{10}\text{OZn}^c$	N(1)	2.05 (2)		120 (1)
	N(1)(H ⁺)			124 (1)
$\text{C}_6\text{H}_7\text{Cl}_3\text{N}_5\text{OZn}^d$	N(1)	2.068 (2)		119.6 (2)
	N(7)	2.040 (2)	104.7 (2)	

References: (a) Present work; (b) Taylor (1973); (c) McCall & Taylor (1975); (d) McCall & Taylor (1976).

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distorted tetrahedral configuration. The U—C bond distances average 2.73 (4) Å, in agreement with the values previously reported for tris(cyclopentadienyl)uranium compounds. These distances do not appear to be influenced by the nature of the basic ligand. The U—N bond length, 2.34 (4) Å, is, as expected, shorter than the corresponding distance observed in the adduct $[\text{U}(\text{C}_5\text{H}_5)_3(\text{NCS})(\text{NCCH}_3)]$.

Experimental. The title compound was prepared by reaction of tris(cyclopentadienyl)uranium chloride

and potassium thiocyanate, in tetrahydrofuran. Extraction followed by recrystallization with *n*-pentane yielded dark-brown prismatic single crystals. A selected specimen (0.25 × 0.25 × 0.30 mm) was sealed in a thin-walled glass capillary under an inert atmosphere. Intensity data were recorded on an Enraf-Nonius CAD-4 X-ray diffractometer, with graphite-monochromated Mo K α radiation and using ω -2 θ scans. The unit-cell dimensions were determined from 25 reflections with $4 < \theta < 23^\circ$. The space group was established from systematic absences. 1855 unique reflections were collected in the range $4 < \theta < 26^\circ$ (h 0 \rightarrow -11, k 0 \rightarrow -20, l 0 \rightarrow 15). Of these, 1301 were considered observed [$I > \sigma(I)$] and 902 with $I > 3\sigma(I)$ were used in refinement. The intensities of three standard reflections were measured at 30 min intervals and an anisotropic decay correction was applied to the data (total decay 1.68%). The intensities were corrected for Lorentz-polarization effects. Empirical absorption corrections, based on a method which used a set of scans of reflections with χ values close to 90° (North, Phillips & Mathews, 1968), gave transmission factors ranging from 48.6 to 99.5%. The structure was solved by direct methods and refined by full-matrix least-squares minimization of $\sum w(|F_o| - |F_c|)^2$ $\{w = 1/[\sigma(F_o)]^2$ with $\sigma(F_o) = \sigma(F_o^2)/2F_o$ and $\sigma(F_o^2) = [\sigma(I) + (0.081)^2]^{1/2}/Lp\}$. Anisotropic thermal parameters were refined for the U and for the thiocyanate atoms while the cyclopentadienyl C atoms were treated isotropically. H atoms, in calculated positions (C—H = 0.95 Å), were included in the final structure-factor calculation with individual isotropic temperature factors $B = 5 \text{ \AA}^2$. A secondary-extinction coefficient was refined to a value of $g = 2.1 (2) \times 10^{-7}$, $\{F_c = F_c/[1 + g(F_c)^2 Lp]\}$. The absolute structure which gave the better refinement was chosen. The final agreement factors were $R = 0.042$, $wR = 0.057$, $S = 1.24$. The final $(\Delta/\sigma)_{\max}$ was less than 0.01. The maximum and minimum heights in the final difference Fourier map were 2.07 and $-1.36 e \text{ \AA}^{-3}$ (around the U atom). The atomic scattering factors and anomalous-dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations were performed with the Enraf-Nonius (1986) SDP programs. Atomic coordinates and temperature factors are given in Table 1.* Selected bond distances and angles are presented in Table 2. An ORTEPII illustration (Johnson, 1976) of the molecular structure is given in Fig. 1.

Table 1. Atomic positional parameters and isotropic or equivalent isotropic thermal parameters (\AA^2)

The U atom and thiocyanate atoms N, C(16) and S were refined anisotropically; $B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab\cos\gamma B(1,2) + accos\beta B(1,3) + bccos\alpha B(2,3)]$.

	x	y	z	B_{iso}/B_{eq}
U	0.09239 (4)	-0.23850 (7)	0.125	2.95 (1)
N	0.160 (1)	-0.424 (2)	0.246 (2)	4.9 (5)
C(16)	0.197 (1)	-0.532 (2)	0.280 (2)	3.0 (4)
S	0.2498 (5)	-0.6818 (8)	0.3311 (7)	6.0 (2)
C(1)	0.240 (2)	-0.052 (3)	0.105 (3)	7.2 (7)
C(2)	0.267 (2)	-0.196 (3)	0.091 (3)	6.0 (7)
C(3)	0.238 (2)	-0.267 (3)	-0.001 (4)	7.4 (8)
C(4)	0.180 (2)	-0.126 (4)	-0.053 (3)	9 (1)
C(5)	0.185 (2)	-0.004 (3)	0.024 (3)	5.7 (6)
C(6)	0.092 (2)	-0.033 (3)	0.308 (2)	5.4 (6)
C(7)	0.056 (1)	0.048 (3)	0.224 (3)	6.8 (7)
C(8)	-0.031 (2)	-0.038 (3)	0.220 (3)	7.2 (8)
C(9)	-0.025 (2)	-0.155 (3)	0.286 (3)	6.4 (7)
C(10)	0.043 (2)	-0.164 (3)	0.355 (3)	6.7 (7)
C(11)	0.016 (2)	-0.522 (4)	0.079 (2)	7.7 (8)
C(12)	-0.054 (2)	-0.432 (3)	0.109 (4)	10 (1)
C(13)	-0.052 (2)	-0.338 (4)	0.011 (3)	8.8 (9)
C(14)	0.015 (2)	-0.373 (4)	-0.056 (3)	8.9 (9)
C(15)	0.059 (2)	-0.490 (4)	-0.011 (3)	6.3 (7)

Table 2. Selected distances (\AA) and angles ($^\circ$)

Cp(I) denotes centroid of ring (I) [C(1), C(2), C(3), C(4), C(5)], Cp(II), centroid of ring (II) [C(6), C(7), C(8), C(9), C(10)], and Cp(III), centroid of ring (III) [C(11), C(12), C(13), C(14), C(15)].

U—N	2.34 (2)	U—Cp(I)	2.45 (4)
U—C(1)	2.77 (3)	U—Cp(II)	2.51 (4)
U—C(2)	2.74 (3)	U—Cp(III)	2.47 (4)
U—C(3)	2.70 (4)		
U—C(4)	2.67 (4)	N—U—Cp(I)	99 (1)
U—C(5)	2.69 (3)	N—U—Cp(II)	96 (1)
U—C(6)	2.75 (3)	N—U—Cp(III)	97 (1)
U—C(7)	2.72 (3)	Cp(I)—U—Cp(II)	116 (1)
U—C(8)	2.77 (3)	Cp(I)—U—Cp(III)	122 (1)
U—C(9)	2.71 (3)	Cp(II)—U—Cp(III)	118 (1)
U—C(10)	2.88 (4)		
U—C(11)	2.69 (3)	N—C(16)	1.14 (3)
U—C(12)	2.78 (3)	C(16)—S	1.61 (3)
U—C(13)	2.74 (4)		
U—C(14)	2.69 (4)	U—N—C(16)	163 (1)
U—C(15)	2.69 (3)	N—C(16)—S	178 (2)

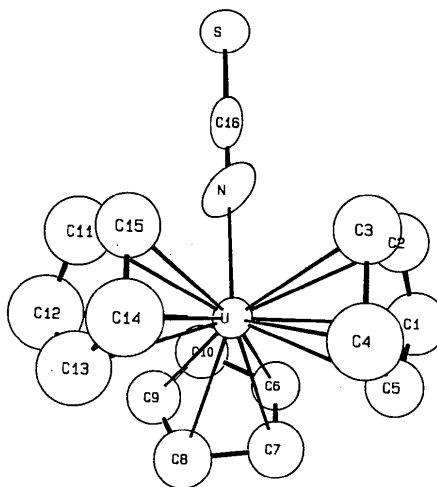


Fig. 1. The molecular structure. Thermal ellipsoids are at the 50% probability level.

* Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates, C—C bond lengths and C—C—C bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55740 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1000]

Related literature. The structures of a large number of tris(cyclopentadienyl)uranium complexes of the series $[U(C_5H_5)_3]X$, where X is an anion or a monodentate Lewis base, have been previously reported (Burns, 1986, and references therein; Rogers & Rogers, 1991, and references therein; Spirlet, Rebizant, Apostolidis, Andreotti & Kanellakopoulos, 1989; Spirlet, Rebizant, Apostolidis, Van den Bossche & Kanellakopoulos, 1990; Rebizant, Spirlet, Apostolidis & Kanellakopoulos, 1991). The structure analysis of the title complex was carried out in order to complete the series.

Tris(cyclopentadienyl)uranium thiocyanate is known to form adducts with Lewis bases such as CH_3CN and water but only the structure of the complex $[(C_5H_5)_3U(NCS)(NCCH_3)]$ has been previously reported (Aslan, Yunlu, Fischer, Bombieri & Benetollo, 1988).

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Structure of 3-Ferrocenyl-2-butenic Acid

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Abstract. [1-(1-Carboxyisopropenyl)- η^5 -cyclopentadienyl](η^5 -cyclopentadienyl)iron, $[Fe(C_9H_9O_2)(C_5H_5)]$, $M_r = 270.11$, monoclinic, $P2_1/c$, $a = 11.813$ (4), $b = 8.353$ (5), $c = 12.297$ (4) Å, $\beta = 102.31$ (3)°, $V = 1185.4$ Å³, $Z = 4$, $D_x = 1.513$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 12.55$ cm⁻¹, $F(000) = 560$, $T = 293$ K, $R = 0.061$ for 1655 observed reflections. The molecular geometry shows the (*E*) stereochemical conformation. The Fe atom and the centroid of the Cp rings form an angle of 179.7 (3)°. The two cyclopentadienide rings are almost eclipsed, forming a 1.1° twist angle.

Experimental. An orange crystal synthesized by Professor Yin You-jin (Zhang, Yin, Zhou & Wang, 1990), of size $0.4 \times 0.3 \times 0.29$ mm, was used for data collection on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated $Mo K\alpha$ radiation. Cell dimension were refined from 25 accurately centred reflections in the range $18 < 2\theta < 30$ °. Intensities were measured using ω - 2θ scans of width $(0.64 + 0.35\tan\theta)$ °, for θ range 1 to 26° ($h = -14$ to 14, $k = 0$ to 10, $l = 0$ to 5). 2621 reflections were measured, of which 2387 were unique. 1655 observed

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reflections [$I \geq 3\sigma(I)$] were retained for structure determination. Three standard reflections, monitored every 3600 s, showed 0.5% variation in intensity; correction applied. $R_{int} = 0.052$. Lp corrections, as well as ψ semi-empirical absorption corrections (maximum and minimum transmission 1.000 and 0.874, respectively), were applied.

The structure was solved by Patterson methods and difference Fourier syntheses, with anisotropic full-matrix least-squares refinement (on F) for non-H atoms. All H atoms were found on a difference

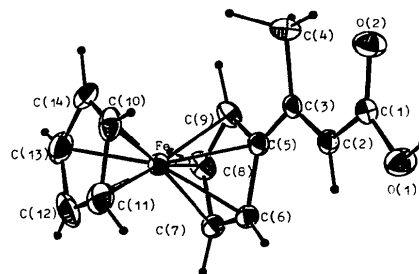


Fig. 1. Structure of title compound showing the atomic numbering.