# The X-Ray Crystal Structure Determination of Tetraphenylarsonium Tris(n5-cyclopentadienyl)di-isothiocyanatouranate(IV)†

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The crystal structure of the title compound has been determined from three-dimensional X-ray diffraction data. The compound crystallises in space group  $P\overline{1}$ , with a = 15.570(7), b = 13.727(5), c = 9.082(4) Å,  $\alpha = 94.8(1)$ ,  $\beta = 104.9(1)$ ,  $\gamma = 99.6(1)$ °, and Z = 2. The structure was solved by Patterson and Fourier methods; full-matrix least-squares refinement based on 3 584 reflections gave a final R value of 0.053. The co-ordination about the uranium atom in the anion  $[U(cp)_3(NCS)_2]^{-1}$ cyclopentadienyl) is a trigonal bipyramid with three  $\pi$ -bonded cp rings in the equatorial plane (U-ring centre distances 2.48, 2.49, and 2.50 Å) and two nitrogen-bonded thiocyanate groups in the axial positions (U-N 2.46 and 2.50 Å). The As-C distances in the cation [AsPh<sub>4</sub>] + range from 1.873 to 1.886 Å and the C-As-C angles fall in the range 106.5—111.9°. The structure is discussed in conjunction with information available for [U(cp)<sub>3</sub>(NCS)(NCCH<sub>3</sub>)] and [U(cp)<sub>3</sub>(NCCH<sub>3</sub>)<sub>2</sub>]<sub>2</sub>[UO<sub>2</sub>Cl<sub>4</sub>]·2L (L = buta-1,3-diene).

The preparation of the first mononuclear, negatively charged, five-co-ordinate tris-cyclopentadienyl actinide(IV) complexes,  $[M(cp)_3X_2]^-$  (M = U, Np, or Pu; X = NCS) and  $[U(cp)_3XY]^-$ (X = NCS, Y = NCO), has recently been reported. Spectrophotometric evidence suggested trigonal bipyramidal coordination for the metal atom in the uranium complexes, with which the neptunium and plutonium analogues were shown to be isostructural by X-ray powder diffraction data, as in the known structure of [U(cp)<sub>3</sub>(NCS)(NCCH<sub>3</sub>)].<sup>2</sup> This has now been confirmed by the determination of the structure of [AsPh<sub>4</sub>][U(cp)<sub>3</sub>(NCS)<sub>2</sub>]. The detailed co-ordination within the anion [U(cp)<sub>3</sub>(NCS)<sub>2</sub>] is compared with similar information for the neutral complex [U(cp)<sub>3</sub>(NCS)(NCCH<sub>3</sub>)]<sup>2</sup> and the cation  $[U(cp)_3(NCCH_3)_2]^+$ .

## **Experimental**

The complex [AsPh<sub>4</sub>][U(cp)<sub>3</sub>(NCS)<sub>2</sub>] was prepared as described earlier 1 and recrystallised from acetonitrile. A single crystal of approximate dimensions  $0.03 \times 0.04 \times 0.1$  mm, mounted in a glass capillary, was used for data collection.

Crystal Data.— $C_{41}H_{35}AsN_2S_2U$ , M = 932.8, Triclinic, space group PI, a = 15.570(7), b = 13.727(5), c = 9.082(4) Å,  $\alpha = 94.8(1), \beta = 104.9(1), \gamma = 99.6(1)^{\circ}, U = 1 835.4 \text{ Å}^3$ Z = 2,  $D_c = 1.69 \,\mathrm{g \, cm^{-3}}$ , F(000) = 904,  $\mu(\text{Mo-}K_{\alpha}) = 52.3 \,\mathrm{cm^{-1}}$ ,  $\lambda(\text{Mo-}K_{\alpha}) = 0.710 \,69 \,\text{Å}$ .

Data Collection and Structure Refinement.—X-Ray intensity data were collected on a four-circle Philips PW1100 automated diffractometer with graphite monochromated Mo- $K_{\alpha}$ radiation. The unit cell was determined on the basis of 25 strong reflections found by mounting the crystal at random and varying the orientation angles  $\varphi$  and  $\chi$  in the range of 120° each, with the detector position varying between  $\theta = 6^{\circ}$ 

and  $\theta = 10^{\circ}$ . For the determination of precise lattice parameters 20 strong reflections with  $9 \le \theta \le 14^{\circ}$  were considered. Integrated intensities of hkl reflections with  $l \ge 0$  and  $3 \le 1$  $\theta \leq 25^{\circ}$  were measured using the  $\theta$ —2 $\theta$  scan method with a scan speed of 0.03° s<sup>-1</sup>, scan width 1.2°, and two background counts of 20 s at each end of the scans. Of the 6 949 reflections thus considered, 3 584 having a net intensity greater than 30  $(\sigma = \text{standard error based on counting statistics})$  were used in the structure determination and refinement. The intensities of three standard reflections, monitored at 100 reflection intervals, showed no greater fluctuations than those expected from Poisson statistics. The intensity data were corrected for Lorentz and polarization effects and for absorption following the method of North et al.4

The positions of the uranium and arsenic atoms were determined from a three-dimensional Patterson function. The Fourier-difference synthesis phased with the heavy-atom positions allowed the location of some of the lighter atoms including the two NCS groups. A further Fourier-difference synthesis showed all the missing non-hydrogen atoms. At this point the R factor was 0.152. Full-matrix least-squares refinement was carried out. The function minimized was  $\sum w(|F_0|$  $|F_c|$ ) where  $|F_c|$  and  $|F_c|$  are the observed and calculated structure factors; w = 1 showed reasonable consistency in a test of  $w\Delta^2$  for data sectioned with respect both to  $|F_0|$  and to  $(\sin\theta)/\lambda$ . Anisotropic thermal parameters were introduced for all the non-hydrogen atoms except those belonging to the phenyl rings, which were refined as rigid groups and restricted to their normal geometry ( $D_{6h}$  symmetry, C-C = 1.395 Å) using the group refinement procedure. Each ring was assigned six variable positional parameters and each ring carbon atom was assigned an individual isotropic thermal parameter. Hydrogen atom contributions were used as fixed atoms in calculated positions ( $d_{C-H} = 0.95 \text{ Å}$  and  $B_{iso} = 7 \text{ Å}^2$ ).

This model converged at a final R 0.053. A difference-Fourier map calculated at this point was practically featureless except for a few peaks of ca. 0.8 e Å<sup>-3</sup> in the proximities of the uranium and arsenic positions.

The anomalous dispersion terms 5 for U and As were taken

<sup>†</sup> Supplementary data available (No. SUP 23439, 25 pp.): observed and calculated structure factors, thermal parameters. See Notices to Authors No. 7, J. Chem. Soc., Dalton Trans., 1981, Index issue.

**Table 1.** Atomic co-ordinates ( $\times$  10<sup>4</sup>) with e.s.d.s in parentheses

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C(8) 3 136(17) -256(22) 2 089(28) H(81) 314 25 143 C(9) 2 477(18) -1 084(25) 1 868(30) H(91) 196 -126 101 C(10) 2 711(15) -1 585(14) 3 103(38) H(101) 236 -219 323 C(11) 3 539(14) -1 059(17) 4 046(29) H(111) 386 -124 499 C(12) 3 774(12) -243(15) 3 399(27) H(121) 431 25 379 C(13) 1 979(16) 742(17) 7 066(22) H(131) 153 114 704 C(14) 1 833(14) -277(16) 6 958(21) H(141) 127 -71 687 C(15) 2 651(13) -595(15) 6 993(20) H(151) 275 -126 689 C(16) 3 308(13) 237(15) 7 182(23) H(161) 394 28 731 C(17) 2 917(19) 1 077(18) 7 229(22) H(171) 320 176 732 C(18) 6 458(6) 3 301(7) 1 360(9) C(19) 5 988(6) 2 790(7) 2 445(9) H(19) 615 320 341 C(20) 5 275(6) 1 969(7) 2 114(9) H(20) 496 182 285 C(21) 5 032(6) 1 369(7) 697(9) H(21) 455 81 47 C(22) 5 503(6) 1 589(7) -388(9) H(22) 534 118 -135 C(23) 6 215(6) 2 410(7) -57(9) H(23) 654 256 -80 C(24) 8 370(7) 3 665(7) 1 166(10) C(25) 8 744(7) 2 926(7) 1 920(10) H(25) 853 268 273 C(26) 9 437(7) 2 556(7) 1 488(10) H(26) 969 205 200 C(27) 9 757(7) 2 925(7) 303(10) H(27) 23 267 1 C(28) 9 383(7) 3 664(7) -451(10) H(26) 969 C(29) 8 689(7) 4 034(7) -201(10) H(29) 843 454 -53 C(30) 7 011(6) 5 094(6) 488(12) C(31) 6 211(6) 4 878(6) -708(12) H(31) 586 422 -96 C(32) 5 925(6) 5 629(6) -1 \$42(17) H(31) 586 422 -96 C(33) 6 438(6) 6 596(6) -1 180(12) H(34) 759 747 26 C(35) 7 525(6) 6 606(6) 849(12) H(31) 586 600 779 C(39) 8 889(6) 5 577(7) 6 788(12) H(31) 589 60 397 515 698 C(30) 7 746(6) 4 725(7) 3 757(12) H(34) 759 747 26 C(36) 7 746(6) 4 725(7) 3 757(12) H(34) 759 747 26 C(37) 8 578(6) 5 629(6) -1 180(12) H(31) 586 600 779 C(40) 7 368(6) 5 707(7) 5 748(12) H(39) 836 600 779 C(40) 7 368(6) 5 707(7) 5 748(12) H(39) 836 600 779 C(40) 7 368(6) 5 707(7) 5 748(12) H(40) 696 605 608	C(7)							
$ \begin{array}{c} C(9) \\ C(9) \\ C(10) \\ C(10) \\ C(10) \\ C(11) \\ C(11) \\ C(11) \\ C(11) \\ C(11) \\ C(11) \\ C(12) \\ C(13) \\ C(14) \\ C(14) \\ C(15) \\ C(15) \\ C(15) \\ C(16) \\ C(16) \\ C(16) \\ C(16) \\ C(17) \\ C(17) \\ C(19) \\ C(11) \\ $	C(8)		-256(22)		H(81)			
$ \begin{array}{c} C(10) & 2711(15) & -1585(14) & 3103(38) & H(101) & 236 & -219 & 323 \\ C(11) & 3539(14) & -1059(17) & 4046(29) & H(111) & 386 & -124 & 499 \\ C(12) & 3774(12) & -2243(15) & 3399(27) & H(121) & 431 & 25 & 379 \\ C(13) & 1979(16) & 742(17) & 7066(22) & H(131) & 153 & 114 & 704 \\ C(14) & 1833(14) & -277(16) & 6958(21) & H(141) & 127 & -71 & 687 \\ C(15) & 2651(13) & -595(15) & 6993(20) & H(151) & 275 & -126 & 689 \\ C(16) & 3308(13) & 237(15) & 7182(23) & H(161) & 394 & 28 & 731 \\ C(17) & 2917(19) & 1077(18) & 7229(22) & H(171) & 320 & 176 & 732 \\ C(18) & 6458(6) & 3010(7) & 1360(9) & & & & & & & & & & & \\ C(19) & 5988(6) & 2790(7) & 2445(9) & H(19) & 615 & 320 & 341 \\ C(20) & 5275(6) & 1969(7) & 2114(9) & H(20) & 496 & 182 & 285 \\ C(21) & 5032(6) & 1369(7) & 697(9) & H(21) & 455 & 81 & 47 \\ C(22) & 5275(6) & 1369(7) & -388(9) & H(22) & 534 & 118 & -135 \\ C(23) & 6215(6) & 2410(7) & -57(9) & H(23) & 654 & 256 & -80 \\ C(24) & 8370(7) & 3665(7) & 1166(10) & & & & & \\ C(25) & 8744(7) & 2926(7) & 1920(10) & H(25) & 853 & 268 & 273 \\ C(26) & 9437(7) & 2556(7) & 1488(10) & H(26) & 969 & 205 & 200 \\ C(27) & 9757(7) & 2925(7) & 303(10) & H(27) & 23 & 267 & 1 \\ C(28) & 9383(7) & 3664(7) & -451(10) & H(28) & 960 & 392 & -126 \\ C(29) & 8689(7) & 4034(7) & -20(10) & H(28) & 960 & 392 & -126 \\ C(29) & 8689(7) & 4034(7) & -20(10) & H(29) & 843 & 454 & -53 \\ C(30) & 7011(6) & 5094(6) & 488(12) & & & & \\ C(31) & 6211(6) & 4878(6) & -708(12) & H(31) & 586 & 422 & -96 \\ C(32) & 5925(6) & 5629(6) & -1542(12) & H(31) & 586 & 422 & -96 \\ C(32) & 5925(6) & 5629(6) & -1542(12) & H(34) & 759 & 747 & 26 \\ C(35) & 7525(6) & 6060(6) & 849(12) & H(35) & 807 & 621 & 166 \\ C(36) & 7746(6) & 4725(7) & 3757(12) & H(34) & 599 & 435 & 445 \\ C(38) & 8805(6) & 5171(7) & 6282(12) & H(38) & 937 & 515 & 698 \\ C(39) & 8200(6) & 5677(7) & 6768(12) & H(39) & 336 & 600 & 779 \\ C(40) & 7368(6) & 5707(7) & 5748(12) & H(39) & 336 & 600 & 779 \\ C(40) & 7368(6) & 5707(7) & 5748($	C(9)		-1 084(25)		H(01)			101
$ \begin{array}{c} C(11) & 3  539(14) & -1  059(17) & 4  046(29) & H(111) & 386 & -124 & 499 \\ C(12) & 3  774(12) & -243(15) & 3  399(27) & H(121) & 431 & 25 & 379 \\ C(13) & 1  979(16) & 742(17) & 7  066(22) & H(131) & 153 & 114 & 704 \\ C(14) & 1  833(14) & -277(16) & 6  958(21) & H(141) & 127 & -71 & 687 \\ C(15) & 2  651(13) & -595(15) & 6  993(20) & H(151) & 275 & -126 & 689 \\ C(16) & 3  308(13) & 237(15) & 7  182(23) & H(161) & 394 & 28 & 731 \\ C(17) & 2  917(19) & 1  077(18) & 7  229(22) & H(171) & 320 & 176 & 732 \\ C(18) & 6  458(6) & 3  010(7) & 1  360(9) & & & & & & & & & & & & & & & & & & &$	C(10)							
$ \begin{array}{c} C(12) & 3774(12) & -243(15) & 3399(27) & H(121) & 431 & 25 & 379 \\ C(13) & 1979(16) & 742(17) & 7066(22) & H(131) & 153 & 114 & 704 \\ C(14) & 1833(14) & -277(16) & 6958(21) & H(141) & 127 & -71 & 687 \\ C(15) & 2 651(13) & -595(15) & 6993(20) & H(151) & 275 & -126 & 689 \\ C(16) & 3 308(13) & 237(15) & 7182(23) & H(161) & 394 & 28 & 731 \\ C(17) & 2 917(19) & 1 077(18) & 7 229(22) & H(171) & 320 & 176 & 732 \\ C(18) & 6 458(6) & 3 010(7) & 1 360(9) & & & & & \\ C(19) & 5 988(6) & 2 790(7) & 2 445(9) & H(19) & 615 & 320 & 341 \\ C(20) & 5 275(6) & 1 969(7) & 2 114(9) & H(20) & 496 & 182 & 285 \\ C(21) & 5 032(6) & 1 369(7) & 697(9) & H(21) & 455 & 81 & 47 \\ C(22) & 5 503(6) & 1 589(7) & -388(9) & H(21) & 455 & 81 & 47 \\ C(22) & 5 503(6) & 2 410(7) & -57(9) & H(23) & 654 & 256 & -80 \\ C(24) & 8 370(7) & 3 665(7) & 1 166(10) & & & & \\ C(24) & 8 370(7) & 3 665(7) & 1 166(10) & & & & \\ C(25) & 8 744(7) & 2 926(7) & 1 920(10) & H(25) & 853 & 268 & 273 \\ C(26) & 9 437(7) & 2 556(7) & 1 488(10) & H(26) & 969 & 205 & 200 \\ C(27) & 9 757(7) & 2 925(7) & 303(10) & H(27) & 23 & 267 & 1 \\ C(28) & 9 88(9(7) & 4 034(7) & -20(10) & H(28) & 960 & 392 & -126 \\ C(29) & 8 689(7) & 4 034(7) & -20(10) & H(29) & 843 & 454 & -53 \\ C(30) & 7 011(6) & 5 094(6) & 488(12) & & & & \\ C(31) & 6 211(6) & 4 878(6) & -708(12) & H(31) & 586 & 422 & -96 \\ C(32) & 5 925(6) & 5 629(6) & -1 542(12) & H(32) & 538 & 548 & -236 \\ C(33) & 6 438(6) & 6 596(6) & -1 180(12) & H(33) & 624 & 711 & -175 \\ C(34) & 7 238(6) & 6 811(6) & 16(12) & H(34) & 759 & 747 & 26 \\ C(35) & 7 725(6) & 6 060(6) & 849(12) & H(35) & 807 & 621 & 166 \\ C(36) & 7 746(6) & 4 725(7) & 3 757(12) & & & & \\ C(39) & 8 200(6) & 5 677(7) & 6 282(12) & H(38) & 937 & 515 & 698 \\ C(39) & 8 200(6) & 5 677(7) & 6 788(12) & H(39) & 836 & 600 & 799 \\ C(40) & 7 368(6) & 5 707(7) & 5 748(12) & H(40) & 696 & 605 & 608 \\ \end{array}$	C(11)							
$\begin{array}{c} C(13) & 1 \ 979(16) & 742(17) & 7066(22) & H(131) & 153 & 114 & 704 \\ C(14) & 1 \ 833(14) & -277(16) & 6958(21) & H(141) & 127 & -71 & 687 \\ C(15) & 2 \ 651(13) & -595(15) & 6993(20) & H(151) & 275 & -126 & 689 \\ C(16) & 3 \ 308(13) & 237(15) & 7182(23) & H(161) & 394 & 28 & 731 \\ C(17) & 2 \ 917(19) & 1 \ 077(18) & 7229(22) & H(171) & 320 & 176 & 732 \\ C(18) & 6458(6) & 3010(7) & 1 \ 360(9) & & & & & & & & & & & & & & & & & & &$								
$\begin{array}{c} C(14) & 1 \ 833(14) & -277(16) & 6 \ 958(21) & H(141) & 127 & -71 & 687 \\ C(15) & 2 \ 651(13) & -595(15) & 6 \ 993(20) & H(161) & 394 & 28 & 731 \\ C(16) & 3 \ 308(13) & 237(15) & 7 \ 182(23) & H(161) & 394 & 28 & 731 \\ C(17) & 2 \ 917(19) & 1 \ 077(18) & 7 \ 229(22) & H(171) & 320 & 176 & 732 \\ C(18) & 6 \ 458(6) & 3 \ 010(7) & 1 \ 360(9) & & & & & & & & & & & & & & & & & & &$		` '						313 704
C(15) 2 651(13) -595(15) 6 993(20) H(151) 275 -126 689 C(16) 3 308(13) 237(15) 7 182(23) H(161) 394 28 731 C(17) 2 917(19) 1 077(18) 7 229(22) H(171) 320 176 732 C(18) 6 458(6) 3 010(7) 1 360(9) C(19) 5 988(6) 2 790(7) 2 445(9) H(19) 615 320 341 C(20) 5 275(6) 1 969(7) 2 114(9) H(20) 496 182 285 C(21) 5 032(6) 1 369(7) 697(9) H(21) 455 81 47 C(22) 5 503(6) 1 589(7) -388(9) H(22) 534 118 -135 C(23) 6 215(6) 2 410(7) -57(9) H(23) 654 256 -80 C(24) 8 370(7) 3 665(7) 1 166(10) C(25) 8 744(7) 2 926(7) 1 920(10) H(25) 853 268 273 C(26) 9 437(7) 2 556(7) 1 488(10) H(26) 969 205 200 C(27) 9 757(7) 2 925(7) 303(10) H(27) 23 267 1 C(28) 9 383(7) 3 664(7) -451(10) H(28) 960 392 -126 C(29) 8 689(7) 4 034(7) -20(10) H(28) 960 392 -126 C(29) 8 689(7) 4 034(7) -20(10) H(28) 960 392 -126 C(29) 8 689(7) 4 034(7) -20(10) H(28) 960 392 -126 C(31) 6 211(6) 4 878(6) -708(12) H(31) 586 422 -96 C(32) 5 925(6) 5 629(6) -1 542(12) H(32) 538 548 -236 C(33) 6 438(6) 6 596(6) -1 180(12) H(33) 6624 711 -175 C(34) 7 238(6) 6 811(6) 16(12) H(34) 759 747 26 C(35) 8 578(6) 4 695(7) 4 777(12) H(37) 899 435 445 C(38) 8 805(6) 5 171(7) 6 282(12) H(38) 937 515 698 C(39) 8 200(6) 5 677(7) 6 768(12) H(39) 836 600 779 C(40) 7 368(6) 5 707(7) 5 748(12) H(40) 696 605 608			277(16)					
C(16)         3 308(13)         237(15)         7 182(23)         H(161)         394         28         731           C(17)         2 917(19)         1 077(18)         7 229(22)         H(171)         320         176         732           C(18)         6 458(6)         3 010(7)         1 360(9)         1         176         732           C(19)         5 988(6)         2 790(7)         2 445(9)         H(19)         615         320         341           C(20)         5 275(6)         1 969(7)         2 114(9)         H(20)         496         182         285           C(21)         5 032(6)         1 369(7)         697(9)         H(21)         455         81         47           C(22)         5 503(6)         1 589(7)         -388(9)         H(22)         534         118         -135           C(23)         6 215(6)         2 410(7)         -57(9)         H(23)         654         256         -80           C(24)         8 370(7)         3 665(7)         1 166(10)         202         206         205         200           C(24)         8 744(7)         2 925(7)         1 920(10)         H(25)         853         268         273								
C(17)         2 917(19)         1 077(18)         7 229(22)         H(171)         320         176         732           C(18)         6 458(6)         3 010(7)         1 360(9)								
C(18)         6 458(6)         3 010(7)         1 360(9)           C(19)         5 988(6)         2 790(7)         2 445(9)         H(19)         615         320         341           C(20)         5 275(6)         1 969(7)         2 114(9)         H(20)         496         182         285           C(21)         5 032(6)         1 369(7)         697(9)         H(21)         455         81         47           C(22)         5 503(6)         1 589(7)         -388(9)         H(22)         534         118         -135           C(23)         6 215(6)         2 410(7)         -57(9)         H(23)         654         256         -80           C(24)         8 370(7)         3 665(7)         1 166(10)         200         400	C(10)				` ,			731
C(19)         5 988(6)         2 790(7)         2 445(9)         H(19)         615         320         341           C(20)         5 275(6)         1 969(7)         2 114(9)         H(20)         496         182         285           C(21)         5 032(6)         1 369(7)         697(9)         H(21)         455         81         47           C(22)         5 503(6)         1 589(7)         -388(9)         H(22)         534         118         -135           C(23)         6 215(6)         2 410(7)         -57(9)         H(23)         654         256         -80           C(24)         8 370(7)         3 665(7)         1 166(10)         365(2) <td></td> <td></td> <td></td> <td></td> <td><b>II</b>(1/1)</td> <td>320</td> <td>176</td> <td>132</td>					<b>II</b> (1/1)	320	176	132
$\begin{array}{c} C(20) & 5\ 275(6) & 1\ 969(7) & 2\ 114(9) & H(20) & 496 & 182 & 285 \\ C(21) & 5\ 032(6) & 1\ 369(7) & 697(9) & H(21) & 455 & 81 & 47 \\ C(22) & 5\ 503(6) & 1\ 589(7) & -388(9) & H(22) & 534 & 118 & -135 \\ C(23) & 6\ 215(6) & 2\ 410(7) & -57(9) & H(23) & 654 & 256 & -80 \\ C(24) & 8\ 370(7) & 3\ 665(7) & 1\ 166(10) & & & & & & & & & \\ C(25) & 8\ 744(7) & 2\ 926(7) & 1\ 920(10) & H(25) & 853 & 268 & 273 \\ C(26) & 9\ 437(7) & 2\ 556(7) & 1\ 488(10) & H(26) & 969 & 205 & 200 \\ C(27) & 9\ 757(7) & 2\ 925(7) & 303(10) & H(27) & 23 & 267 & 1 \\ C(28) & 9\ 383(7) & 3\ 664(7) & -451(10) & H(28) & 960 & 392 & -126 \\ C(29) & 8\ 689(7) & 4\ 034(7) & -20(10) & H(29) & 843 & 454 & -53 \\ C(30) & 7\ 011(6) & 5\ 094(6) & 488(12) & & & & & \\ C(31) & 6\ 211(6) & 4\ 878(6) & -708(12) & H(31) & 586 & 422 & -96 \\ C(32) & 5\ 925(6) & 5\ 629(6) & -1\ 542(12) & H(32) & 538 & 548 & -236 \\ C(33) & 6\ 438(6) & 6\ 596(6) & -1\ 180(12) & H(34) & 759 & 747 & 26 \\ C(34) & 7\ 238(6) & 6\ 811(6) & 16(12) & H(34) & 759 & 747 & 26 \\ C(35) & 7\ 525(6) & 6\ 060(6) & 849(12) & H(35) & 807 & 621 & 166 \\ C(36) & 7\ 746(6) & 4\ 725(7) & 3\ 757(12) & & & & & \\ C(37) & 8\ 578(6) & 4\ 695(7) & 4\ 777(12) & H(37) & 899 & 435 & 445 \\ C(38) & 8\ 805(6) & 5\ 171(7) & 6\ 282(12) & H(38) & 937 & 515 & 698 \\ C(39) & 8\ 200(6) & 5\ 677(7) & 6\ 788(12) & H(39) & 836 & 600 & 779 \\ C(40) & 7\ 368(6) & 5\ 707(7) & 5\ 748(12) & H(40) & 696 & 605 & 608 \\ \end{array}$	C(10)				11(10)	615	220	241
C(21)         5 032(6)         1 369(7)         697(9)         H(21)         455         81         47           C(22)         5 503(6)         1 589(7)         -388(9)         H(22)         534         118         -135           C(23)         6 215(6)         2 410(7)         -57(9)         H(23)         654         256         -80           C(24)         8 370(7)         3 665(7)         1 166(10)         11 166(10)	C(19)		1 060(7)					
C(22)         5 503(6)         1 589(7)         -388(9)         H(22)         534         118         -135           C(23)         6 215(6)         2 410(7)         -57(9)         H(23)         654         256         -80           C(24)         8 370(7)         3 665(7)         1 166(10)	C(21)				H(20)		102	
C(23)         6 215(6)         2 410(7)         -57(9)         H(23)         654         256         -80           C(24)         8 370(7)         3 665(7)         1 166(10)				_ 288(Q)				
C(24)       8 370(7)       3 665(7)       1 166(10)         C(25)       8 744(7)       2 926(7)       1 920(10)       H(25)       853       268       273         C(26)       9 437(7)       2 556(7)       1 488(10)       H(26)       969       205       200         C(27)       9 757(7)       2 925(7)       303(10)       H(27)       23       267       1         C(28)       9 383(7)       3 664(7)       -451(10)       H(28)       960       392       -126         C(29)       8 689(7)       4 034(7)       -20(10)       H(29)       843       454       -53         C(30)       7 011(6)       5 094(6)       488(12)       488(12)       488(12)       488(12)       586       422       -96         C(31)       6 211(6)       4 878(6)       -708(12)       H(31)       586       422       -96         C(32)       5 925(6)       5 629(6)       -1 542(12)       H(32)       538       548       -236         C(33)       6 438(6)       6 596(6)       -1 180(12)       H(33)       624       711       -175         C(34)       7 238(6)       6 811(6)       16(12)       H(34)       759       747	C(22)							
$\begin{array}{cccccccccccccccccccccccccccccccccccc$					П(23)	034	230	80
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(24)			` '	11/25)	052	260	272
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(25)			` ,				2/3
C(28)       9 383(7)       3 664(7)       -451(10)       H(28)       960       392       -126         C(29)       8 689(7)       4 034(7)       -20(10)       H(29)       843       454       -53         C(30)       7 011(6)       5 094(6)       488(12)			2 330(7)	• •				
C(29)       8 689(7)       4 034(7)       -20(10)       H(29)       843       454       -53         C(30)       7 011(6)       5 094(6)       488(12)       -708(12)       H(31)       586       422       -96         C(31)       6 211(6)       4 878(6)       -708(12)       H(31)       586       422       -96         C(32)       5 925(6)       5 629(6)       -1 542(12)       H(32)       538       548       -236         C(33)       6 438(6)       6 596(6)       -1 180(12)       H(33)       624       711       -175         C(34)       7 238(6)       6 811(6)       16(12)       H(34)       759       747       26         C(35)       7 525(6)       6 060(6)       849(12)       H(35)       807       621       166         C(36)       7 746(6)       4 725(7)       3 757(12)       747       747       748       <	C(27)							
C(30)       7 011(6)       5 094(6)       488(12)         C(31)       6 211(6)       4 878(6)       -708(12)       H(31)       586       422       -96         C(32)       5 925(6)       5 629(6)       -1 542(12)       H(32)       538       548       -236         C(33)       6 438(6)       6 596(6)       -1 180(12)       H(33)       624       711       -175         C(34)       7 238(6)       6 811(6)       16(12)       H(34)       759       747       26         C(35)       7 525(6)       6 060(6)       849(12)       H(35)       807       621       166         C(36)       7 746(6)       4 725(7)       3 757(12)       3 757(12)       757       747       26         C(37)       8 578(6)       4 695(7)       4 777(12)       H(37)       899       435       445         C(38)       8 805(6)       5 171(7)       6 282(12)       H(38)       937       515       698         C(39)       8 200(6)       5 677(7)       6 768(12)       H(39)       836       600       779         C(40)       7 368(6)       5 707(7)       5 748(12)       H(40)       696       605       608 </td <td>C(20)</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	C(20)							
C(31)       6 211(6)       4 878(6)       -708(12)       H(31)       586       422       -96         C(32)       5 925(6)       5 629(6)       -1 542(12)       H(32)       538       548       -236         C(33)       6 438(6)       6 596(6)       -1 180(12)       H(33)       624       711       -175         C(34)       7 238(6)       6 811(6)       16(12)       H(34)       759       747       26         C(35)       7 525(6)       6 060(6)       849(12)       H(35)       807       621       166         C(36)       7 746(6)       4 725(7)       3 757(12)       72       73       738(6)       4 695(7)       4 777(12)       H(37)       899       435       445         C(38)       8 805(6)       5 171(7)       6 282(12)       H(38)       937       515       698         C(39)       8 200(6)       5 677(7)       6 768(12)       H(39)       836       600       779         C(40)       7 368(6)       5 707(7)       5 748(12)       H(40)       696       605       608					H(29)	843	434	-53
C(32)       5 925(6)       5 629(6)       -1 542(12)       H(32)       538       548       -236         C(33)       6 438(6)       6 596(6)       -1 180(12)       H(33)       624       711       -175         C(34)       7 238(6)       6 811(6)       16(12)       H(34)       759       747       26         C(35)       7 525(6)       6 060(6)       849(12)       H(35)       807       621       166         C(36)       7 746(6)       4 725(7)       3 757(12)       72       73       738(6)       4 695(7)       4 777(12)       H(37)       899       435       445         C(38)       8 805(6)       5 171(7)       6 282(12)       H(38)       937       515       698         C(39)       8 200(6)       5 677(7)       6 768(12)       H(39)       836       600       779         C(40)       7 368(6)       5 707(7)       5 748(12)       H(40)       696       605       608	C(30)				11/21)	£0.6	422	06
C(33)       6 438(6)       6 596(6)       -1 180(12)       H(33)       624       711       -175         C(34)       7 238(6)       6 811(6)       16(12)       H(34)       759       747       26         C(35)       7 525(6)       6 060(6)       8 49(12)       H(35)       807       621       166         C(36)       7 746(6)       4 725(7)       3 757(12)       3 757(12)       73       73       73       74	C(31)							
C(34)       7 238(6)       6 811(6)       16(12)       H(34)       759       747       26         C(35)       7 525(6)       6 060(6)       849(12)       H(35)       807       621       166         C(36)       7 746(6)       4 725(7)       3 757(12)       3 757(12)       3 757(12)       3 757(12)       3 757(12)       3 757(12)       3 757(12)       3 757(12)       3 757(12)       3 757(12)       3 757(12)       3 757(12)       3 757(12)       3 899       435       445       445       <	C(32)							-236
C(35)       7 525(6)       6 060(6)       849(12)       H(35)       807       621       166         C(36)       7 746(6)       4 725(7)       3 757(12)              C(37)       8 578(6)       4 695(7)       4 777(12)       H(37)       899       435       445       C(38)       8 805(6)       5 171(7)       6 282(12)       H(38)       937       515       698       C(39)       8 200(6)       5 677(7)       6 768(12)       H(39)       836       600       779       C(40)       7 368(6)       5 707(7)       5 748(12)       H(40)       696       605       608	C(33)							
C(36)       7 746(6)       4 725(7)       3 757(12)         C(37)       8 578(6)       4 695(7)       4 777(12)       H(37)       899       435       445         C(38)       8 805(6)       5 171(7)       6 282(12)       H(38)       937       515       698         C(39)       8 200(6)       5 677(7)       6 768(12)       H(39)       836       600       779         C(40)       7 368(6)       5 707(7)       5 748(12)       H(40)       696       605       608	C(34)						/4/	
C(37)       8 578(6)       4 695(7)       4 777(12)       H(37)       899       435       445         C(38)       8 805(6)       5 171(7)       6 282(12)       H(38)       937       515       698         C(39)       8 200(6)       5 677(7)       6 768(12)       H(39)       836       600       779         C(40)       7 368(6)       5 707(7)       5 748(12)       H(40)       696       605       608	C(35)				H(33)	807	621	166
C(38) 8 805(6) 5 171(7) 6 282(12) H(38) 937 515 698 C(39) 8 200(6) 5 677(7) 6 768(12) H(39) 836 600 779 C(40) 7 368(6) 5 707(7) 5 748(12) H(40) 696 605 608	C(30)				11(27)	900	435	445
C(39) 8 200(6) 5 677(7) 6 768(12) H(39) 836 600 779 C(40) 7 368(6) 5 707(7) 5 748(12) H(40) 696 605 608								
C(40) 7 368(6) 5 707(7) 5 748(12) H(40) 696 605 608								
C(41) / 141(0) 3 231(/) 4 243(12) $H(41)$ 63/ 323 333								
	C(41)	/ 141(0)	3 231(7)	4 243(12)	H(41)	637	323	333

into account in the refinement. Atomic scattering factors for U and As were taken from ref. 6, for the other non-hydrogen atoms from ref. 7, and for the hydrogen atoms from ref. 8. Data processing and computation were carried out using the SHELX 76 programs package. The final positional parameters are listed in Table 1.

# **Results and Discussion**

Figure 1 shows the stereochemistry of the  $[U(cp)_3(NCS)_2]^-$  anion and  $[AsPh_4]^+$  cation and the atom numbering scheme. The relevant interatomic distances and bond angles are given in Tables 2 and 3, respectively, and the significant best mean planes in the structure are shown in Table 4. The packing of the ionic units viewed down c is shown in Figure 2.

The co-ordination around the uranium atom is a trigonal bipyramid, comprising three  $\pi$ -bonded  $\eta^5$ -cyclopentadienyl rings, the centres of which form the equatorial plane of the bipyramid, and two axially co-ordinated thiocyanate groups

which are bonded via the nitrogen atoms. This arrangement is similar to those in the neutral complex [U(cp)<sub>3</sub>(NCS)-(NCCH<sub>3</sub>)]<sup>2</sup> and in the cationic group [U(cp)<sub>3</sub>(NCCH<sub>3</sub>)<sub>2</sub>]<sup>+</sup>.3 Selected bond distances and angles for the three species are compared in Table 5, from which it is seen that the average U-C and U-M (M = ring centre) distances and the M-U-M' angles are very similar. These values are also comparable to those reported for tetrahedral compounds of the type  $[U(cp)_3X]$  (X = halogen or aryl group); for example, in  $[U(cp)_3F]$ , U-C = 2.74 Å and the average M-U-M' angle is 117°.10 Thus the conversion from four-co-ordinate tetrahedral geometry to five-co-ordinate trigonal bipyramidal geometry, at least for the presently known trans thiocyanates and methyl cyanide adducts of the latter class, involves little change in the steric arrangement of the U(cp)<sub>3</sub>+ nucleus. Indeed, the main change on the evidence available appears to be a small increase in the average M-U-M' angle from ca. 117° to ca. 120°. This undoubtedly explains the ease with which five-coordinate actinide(iv) compounds of the types [M(cp)<sub>3</sub>X<sub>2</sub>] and

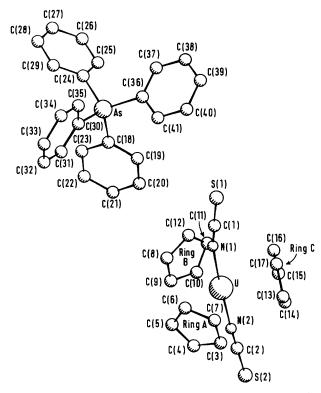


Figure 1. The [U(cp)<sub>3</sub>(NCS)<sub>2</sub>]<sup>-</sup> anion and [AsPh<sub>4</sub>]<sup>+</sup> cation showing the atomic numbering system

Table 2. Interatomic distances (Å) with e.s.d.s in parentheses

( )	50(1) 46(1)	N(1)-C(1) N(2)-C(2)	1.15(2) 1.16(2)	C(1)-S C(2)-S	` ' ' '	
As-C(18 As-C(24	,	882(9) 873(12)	As-C	` '	.886(10) .874(10)	
Rin	g A	Ring	В	Ring	C	
- ' '	2.76(2) 2.71(2) 2.75	U-C(8) U-C(9) U-C(10) U-C(11) U-C(12) U-C (average) U-M <sub>B</sub>	2.72(3) 2.73(3) 2.74(2) 2.79(2) 2.77(2) 2.75	U-C(13) U-C(14) U-C(15) U-C(16) U-C(17) U-C (average) U-M <sub>C</sub>	2.79(2) 2.77(2) 2.71(2) 2.71(2) 2.76(2) 2.75	
Averages: U-C 2.75, C-C 1.38, U-M 2.49						
M = Centre	M = Centre of a C5H5 ring.					

 $[M(cp)_3XY]$  (where X and Y are anionic or neutral ligands) are formed, subject, of course, to the proviso that  $M^{4+}$  should be sufficiently large and that the donor atom from X and Y should be suitably small to allow bonding without steric hindrance.

The two axial NCS groups in  $[U(cp)_3(NCS)_2]^-$  are collinear  $[N(1)-U-N(2)=178.4(5)^\circ]$  and the average M-U-N angle is  $90^\circ$ , close to the values obtained for the other two species (Table 5). However, there are significant differences between the U-N bond lengths in the three trigonal bipyramidal units. Thus (Table 5) the U-N (thiocyanate) distance is 2.407(15) Å when it is *trans* to CH<sub>3</sub>CN in  $[U(cp)_3(NCS)(NCCH_3)]$  compared to the average U-NCS distance of 2.48(1) Å in  $[U(cp)_3(NCS)_2]^-$ , whilst the U-NCCH<sub>3</sub> bond length in the former complex is

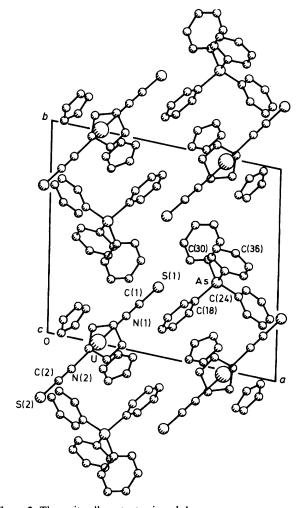


Figure 2. The unit-cell contents viewed down c

Table 3. Bond angles (°) with e.s.d.s in parentheses

U-N(1)-C(1)	168(1)	U-N(2)-C(2)	177(2)
N(1)-C(1)-S(1)	179(2)	N(2)-C(2)-S(2)	179(2)
N(1)-U-N(2)	178.4(5)		
C(18)-As- $C(24)$	106.7(4)	C(24)-As- $C(36)$	111.9(4)
C(18)-As- $C(30)$	109.4(4)	C(24)-As- $C(30)$	110.9(5)
C(18)-As- $C(36)$	111.4(4)	C(30)-As- $C(36)$	106.5(4)
$M_A - U - M_B$	118.8(1)	$M_A$ -U- $M_C$	120.2(1)
$M_B-U-M_C$	121.0(1)		
$N(1)-U-M_A$	90.6(4)	$N(2)-U-M_A$	90.8(4)
$N(1)-U-M_B$	89.4(4)	$N(2)-U-M_B$	90.7(4)
$N(1)-U-M_c$	89.4(4)	$N(2)-U-M_c$	89.2(4)

Averages: C-C-C 108, M-U-M' 120, N(1)-U-M' 89.8, N(2)-U-M 90.2

2.678(16) Å compared with an average value of 2.60 Å in the cation [U(cp)<sub>3</sub>(NCCH<sub>3</sub>)<sub>2</sub>]<sup>+</sup>. The anionic ligand NCS<sup>-</sup> is clearly co-ordinated more strongly to the Lewis acid U<sup>IV</sup> than is the neutral ligand NCCH<sub>3</sub> and it also exerts a *trans* influence on the U<sup>-</sup>NCCH<sub>3</sub> bond in [U(cp)<sub>3</sub>(NCS)(NCCH<sub>3</sub>)] as indicated by significant lengthening of the U<sup>-</sup>NCCH<sub>3</sub> distance in this complex relative to the average value in [U(cp)<sub>3</sub>(NCCH<sub>3</sub>)<sub>2</sub>]<sup>+</sup>.

In the [AsPh<sub>4</sub>]<sup>+</sup> cation the As-C distances range from 1.873(12) to 1.886(10) Å whilst the C-As-C angles deviate

**Table 4.** Least-squares planes with deviations (Å) of the relevant atoms in square brackets. The equation of a plane in the direct space is given by pX + qY + rZ = s

Plane 1.	C(3)—C(7) [C(3) 0.0, C(4) 0.002, C(5) -0.003, C(6) 0.003, C(7) -0.002]	<i>p</i> 10.4162	<i>q</i> -8.9361	r 3.4518	s 1.2345
Plane 2.	C(8)—C(12) [C(8) -0.003, C(9) 0.009, C(10) -0.011, C(11) 0.009, C(12) -0.003]	11.5078	-7.8400	-5.5229	2.6596
Plane 3.	C(13)—C(17) [C(13) -0.01, C(14) 0.012, C(15) -0.01, C(16) 0.005, C(17) 0.003]	-1.0539	-1.0087	8.9049	6.0184
Plane 4.	M <sub>A</sub> , M <sub>B</sub> , M <sub>C</sub> [U -0.01, S(1) 5.181, S(2) -5.248, N(1) 2.486, N(2) -2.467, C(1) 3.608, C(2) -3.620]	8.8547	9.7202	1.2669	1.5800
Plane 5.	C(18)—C(23)	10.4823	-10.1097	2.4886	4.0647
Plane 6.	C(24)—C(29)	6.5058	7.4462	4.0711	8.6489
Plane 7.	C(30)—C(35)	11.7239	-3.5304	<b>-7.2079</b>	6.0700
Plane 8.	C(36)—C(41)	5.7733	11.1106	-4.3839	8.0746
Angles (°) between the planes					
	1—2 60.5 1—3 5—6 81.9 6—7 8—5 65.2 8—6	78.7	2—3 59.6 7—5 69.8 8—7 69.8		

Table 5. Molecular parameters in trigonal bipyramidal U<sup>IV</sup> complexes containing cyclopentadienyl ligands

		$[U(cp)_3(NCS)(NCCH_3)]$ (Ref. 2)	$[U(cp)_3(NCCH_3)_2]^+$ (Ref. 3)	$[U(cp)_3(NCS)_2]^-$ (This work)
(a) Distances (Å)				
	U <b>-C</b>	2.763	2.74	2.75
	U-NCCH <sub>3</sub>	2.678(16)	<b>§2.61(2) §2.58(2)</b>	
	U-NCS	2.407(15)		∫2.50(1)  2.46(1)
	U-M	2.484	2.47	2.49
	C-C(ring)	1.42	1.39	1.38
(b) Angles (°)				
	M-U-M'	119.9	119.9	120
	M-U-NCCH <sub>3</sub>	87.8	∫89.2 ∫90.9	
	M-U-NCS	92.2	•	<b>{89.8</b> <b>{90.2</b>
M = Centre of a C <sub>5</sub> F	I <sub>s</sub> ring.			

somewhat from the ideal tetrahedral value, two being less than 109° [106.7(4) and 106.5(4)°], three larger [110.9(5), 111.4(4), and 111.9(4)°], and one close to the expected value [109.4(4)°]. Thus the interatomic interactions only influence the valence angles, as noted in other ionic compounds.<sup>11</sup>

Intermolecular contacts in the compound are not of particular significance.

#### Acknowledgements

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