Table 1. *Atomic positional and isotropic thermal parameters with e.s.d.'s in parentheses*

	x	*y*	*z*	*B*(Å²)
U	0·03719 (7)	0·27246 (4)	0·24365 (4)	2·45 (1)*
Br	0·0247 (2)	0·4603 (1)	0·2391 (1)	4·09 (4)*
C(1)	0·323 (2)	0·223 (1)	0·184 (1)	4·5 (4)
C(2)	0·293 (2)	0·163 (1)	0·247 (1)	3·9 (4)
C(3)	0·307 (2)	0·214 (1)	0·319 (1)	3·4 (4)
C(4)	0·389 (2)	0·384 (1)	0·343 (1)	5·1 (5)
C(5)	0·439 (2)	0·463 (1)	0·305 (1)	3·8 (4)
C(6)	0·445 (2)	0·465 (1)	0·222 (1)	4·9 (4)
C(7)	0·408 (2)	0·394 (1)	0·174 (1)	3·0 (4)
C(8)	0·366 (2)	0·312 (1)	0·212 (1)	3·9 (4)
C(9)	0·342 (2)	0·306 (1)	0·294 (1)	2·9 (4)
C(1′)	−0·232 (2)	0·278 (1)	0·331 (1)	3·6 (4)
C(2′)	−0·182 (2)	0·184 (1)	0·329 (1)	4·0 (4)
C(3′)	−0·052 (2)	0·175 (1)	0·374 (1)	4·4 (4)
C(4′)	0·090 (2)	0·289 (1)	0·473 (1)	4·7 (5)
C(5′)	0·079 (2)	0·376 (1)	0·503 (1)	5·4 (5)
C(6′)	−0·028 (2)	0·439 (2)	0·475 (1)	5·2 (5)
C(7′)	−0·138 (2)	0·414 (1)	0·419 (1)	5·1 (5)
C(8′)	−0·139 (2)	0·323 (1)	0·386 (1)	3·8 (4)
C(9′)	−0·020 (2)	0·261 (1)	0·415 (1)	3·5 (4)
C(1″)	0·012 (2)	0·180 (1)	0·104 (1)	3·2 (4)
C(2″)	−0·127 (2)	0·163 (1)	0·143 (1)	3·4 (4)
C(3″)	−0·217 (2)	0·243 (1)	0·146 (1)	3·0 (4)
C(4″)	−0·170 (2)	0·401 (1)	0·076 (1)	4·0 (4)
C(5″)	−0·073 (2)	0·444 (1)	0·028 (1)	4·6 (5)
C(6″)	0·081 (2)	0·411 (2)	0·008 (1)	5·7 (5)
C(7″)	0·121 (2)	0·327 (1)	0·028 (1)	4·1 (4)
C(8″)	0·015 (2)	0·269 (1)	0·077 (1)	4·5 (4)
C(9″)	−0·127 (2)	0·314 (1)	0·100 (1)	3·5 (4)

* $B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab\cos\gamma B(1,2) + ac\cos\beta B(1,3) + bc\cos\alpha B(2,3)]$.

Table 2. *Selected bond distances* (Å)

E.s.d.'s are given in parentheses.

U—Br	2·747 (2)	U—C(3′)	2·71 (2)
U—C(1)	2·73 (3)	U—C(8′)	2·91 (3)
U—C(2)	2·70 (2)	U—C(9′)	2·90 (2)
U—C(3)	2·76 (2)	U—C(1″)	2·69 (2)
U—C(8)	2·91 (3)	U—C(2″)	2·70 (2)
U—C(9)	2·78 (2)	U—C(3″)	2·74 (2)
U—C(1′)	2·72 (2)	U—C(8″)	2·78 (2)
U—C(2′)	2·68 (2)	U—C(9″)	2·84 (2)

The present structure analysis shows that the title compound is isostructural with the corresponding chloride derivative $[U(C_9H_7)_3]Cl$.

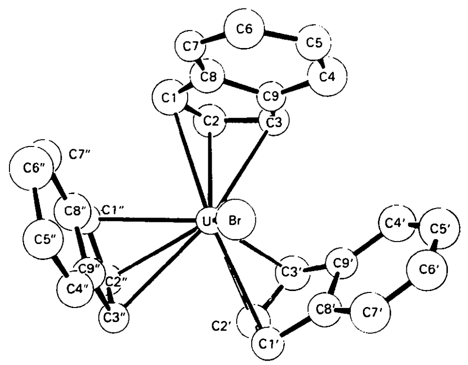

Fig. 1. The molecular structure of $[U(C_9H_7)_3]Br$.

The U—Br bond length of 2·747 (2) Å is comparable to those in $[U(C_9H_7)]Br_3.2C_4H_8O$ and to the U—Br (non-bridging apical Br atom) distance of 2·78 (3) Å in uranium tetrabromide (Taylor & Wilson, 1974).

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Bis(triphenylphosphoranediyl)ammonium Tetrachloronitridomolybdate(IV)

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(*Received* 14 *November* 1985; *accepted* 15 *August* 1986)

Abstract. $C_{36}H_{30}NP_2^+.MoNCl_4^-$, $M_r = 790·3$, orthorhombic, $Pna2_1$, $a = 21·635$ (10), $b = 16·922$ (6), $c = 9·709$ (3) Å, $U = 3555$ Å³, $Z = 4$, $D_x = 1·48$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 8·0$ cm⁻¹, $F(000) = 1600$, $T = 293$ K, $R = 0·050$ for 3643 reflections. The anion is approximately square pyramidal, with Mo 0·6 Å out

of the Cl_4 plane. Bond lengths: Mo—N 1·636 (5), Mo—Cl 2·325–2·347 Å. The cation is bent at N [P—N—P 146·7 (4)°].

Experimental. Stoe–Siemens four-circle diffractometer, monochromated Mo *Kα* radiation, profile-fitting mode (Clegg, 1981). 4323 reflections, $2\theta_{max}$ 50°, $+h+k+l$ and some $+h+k-l$. Three check reflections, no intensity change. Absorption correction based on ψ scans (crystal size 0·4 × 0·3 × 0·2 mm, transmissions 0·74–0·77). 3643 reflections with $F > 4\sigma(F)$ used for all calculations (program system *SHELXTL*; Sheldrick, 1983). Index ranges $h \leq 25$, $k \leq 20$, $|l| \leq 11$. Cell constants refined from $\pm 2\theta$ values of 40 reflections in the range 20–23°.

Structure solution by automatic Patterson interpretation (to locate Mo and Cl) and subsequent difference synthesis. Refinement on F to R 0·050, wR 0·054; all non-H atoms anisotropic; H atoms included using riding model with C—H 0·96 Å, $U(H) = 1·2 \times U_{eq}(C)$; 406 parameters; $S = 1·79$; weighting scheme $w^{-1} = \sigma^2(F) + 0·0003F^2$; max. Δ/σ 0·03, max. features in final $\Delta\rho$ map 0·6 e Å^{-3}. Weak indication of absolute structure (Jones, 1984) by η refinement (Rogers, 1981); $\eta = 0·9$ (2). Atomic scattering factors from *SHELXTL*. Final atomic coordinates are given in Table 1, and selected bond lengths and angles in Table 2.* Fig. 1 shows the atom-numbering scheme.

Related literature. For a review of compounds with transition-metal/nitrogen multiple bonds, see Dehnicke & Strähle (1981). Structure of $[Ph_4As][MoNCl_4]$: Knopp, Lörcher & Strähle (1977).

Table 1. *Atomic coordinates* (×10^4) *and isotropic thermal parameters* (Å^2 ×10^3)

	x	y	z	U_{eq}*
P(1)	5447 (1)	3777 (1)	5368 (2)	32 (1)
P(2)	6008 (1)	2302 (1)	4163 (2)	36 (1)
C(11)	4625 (3)	3893 (3)	5129 (6)	34 (2)
C(12)	4343 (3)	4615 (4)	4969 (7)	44 (2)
C(13)	3722 (3)	4664 (4)	4676 (9)	62 (3)
C(14)	3372 (3)	4002 (4)	4530 (8)	55 (3)
C(15)	3634 (3)	3281 (4)	4715 (9)	56 (3)
C(16)	4260 (3)	3208 (4)	5012 (8)	48 (2)
C(21)	5800 (2)	4724 (3)	5017 (6)	34 (2)
C(22)	6034 (3)	4884 (4)	3699 (7)	47 (2)
C(23)	6299 (3)	5591 (4)	3404 (8)	58 (3)
C(24)	6333 (3)	6161 (4)	4423 (9)	62 (3)
C(25)	6106 (3)	6021 (4)	5706 (9)	58 (3)
C(26)	5832 (3)	5291 (3)	6014 (7)	46 (2)
C(31)	5605 (2)	3555 (3)	7143 (7)	34 (2)
C(32)	5154 (3)	3371 (4)	8063 (7)	42 (2)
C(33)	5314 (3)	3136 (4)	9373 (8)	59 (3)
C(34)	5923 (4)	3070 (4)	9744 (8)	58 (3)
C(35)	6382 (3)	3253 (4)	8833 (8)	54 (3)
C(36)	6236 (3)	3494 (4)	7514 (7)	46 (2)
C(41)	5495 (3)	1673 (3)	3188 (7)	39 (2)
C(42)	5013 (3)	1996 (4)	2464 (7)	48 (2)
C(43)	4630 (3)	1535 (5)	1693 (8)	67 (3)
C(44)	4739 (4)	713 (5)	1639 (10)	81 (4)
C(45)	5206 (3)	396 (4)	2368 (11)	72 (3)
C(46)	5587 (3)	857 (4)	3174 (9)	54 (2)
C(51)	6194 (3)	1783 (3)	5722 (7)	40 (2)
C(52)	6810 (3)	1625 (4)	6059 (10)	63 (3)
C(53)	6919 (4)	1228 (5)	7311 (11)	83 (4)
C(54)	6454 (4)	1010 (4)	8159 (9)	70 (3)
C(55)	5861 (4)	1185 (4)	7848 (9)	64 (3)
C(56)	5722 (3)	1568 (4)	6623 (7)	49 (2)
C(61)	6704 (2)	2424 (4)	3180 (7)	39 (2)
C(62)	6894 (3)	1886 (4)	2234 (9)	53 (2)
C(63)	7419 (3)	2032 (5)	1422 (10)	70 (3)
C(64)	7740 (3)	2719 (4)	1571 (9)	59 (3)
C(65)	7561 (3)	3246 (4)	2515 (10)	63 (3)
C(66)	7047 (3)	3116 (4)	3343 (9)	54 (2)
N(2)	5701 (2)	3141 (3)	4341 (6)	40 (2)
Mo	8214 (1)	3853 (1)	7036 (1)	48 (1)
Cl(1)	8848 (1)	3945 (1)	5109 (3)	74 (1)
Cl(2)	9079 (1)	3316 (1)	8152 (3)	81 (1)
Cl(3)	7995 (1)	4474 (1)	9120 (3)	93 (1)
Cl(4)	7605 (1)	4831 (1)	6002 (4)	95 (1)
N(1)	7773 (2)	3073 (3)	6894 (8)	57 (2)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Mo—Cl(1)	2·325 (3)	Mo—Cl(2)	2·347 (2)
Mo—Cl(3)	2·328 (3)	Mo—Cl(4)	2·341 (3)
Mo—N(1)	1·636 (5)		
P(1)—C(11)	1·806 (6)	P(1)—C(21)	1·808 (5)
P(1)—C(31)	1·796 (7)	P(1)—N(2)	1·566 (5)
P(2)—C(41)	1·805 (6)	P(2)—C(51)	1·796 (7)
P(2)—C(61)	1·793 (6)	P(2)—N(2)	1·579 (5)
P(1)—N(2)—P(2)	146·7 (4)	Cl(1)—Mo—Cl(2)	85·8 (1)
Cl(1)—Mo—Cl(3)	142·1 (1)	Cl(2)—Mo—Cl(3)	86·4 (1)
Cl(1)—Mo—Cl(4)	86·6 (1)	Cl(2)—Mo—Cl(4)	157·1 (1)
Cl(3)—Mo—Cl(4)	86·5 (1)	Cl(1)—Mo—N(1)	109·3 (3)
Cl(2)—Mo—N(1)	101·0 (2)	Cl(3)—Mo—N(1)	108·6 (3)
Cl(4)—Mo—N(1)	101·9 (2)		
C(11)—P(1)—C(21)	107·2 (2)	C(11)—P(1)—C(31)	109·5 (3)
C(21)—P(1)—C(31)	106·6 (3)	C(11)—P(1)—N(2)	109·8 (3)
C(21)—P(1)—N(2)	110·0 (3)	C(31)—P(1)—N(2)	113·6 (3)
C(41)—P(2)—C(51)	106·9 (3)	C(41)—P(2)—C(61)	107·8 (3)
C(51)—P(2)—C(61)	108·5 (3)	C(41)—P(2)—N(2)	109·2 (3)
C(51)—P(2)—N(2)	116·2 (3)	C(61)—P(2)—N(2)	107·9 (3)

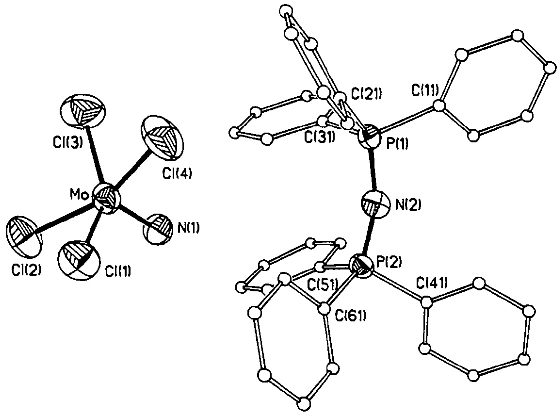

Fig. 1. Asymmetric unit of the title compound, showing the atom-numbering scheme.

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* Lists of structure factors, H-atom coordinates, bond lengths and angles, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43071 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

We thank the Fonds der Chemischen Industrie for financial support.

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Acta Cryst. (1987). C**43**, 357–358

Bis(triphenylphosphoranediyl)ammonium Tetrachloroferrate(III)

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(*Received* 14 *November* 1985; *accepted* 15 *August* 1986)

Abstract. $C_{36}H_{30}NP_2^+.FeCl_4^-$, $M_r = 736{\cdot}2$, monoclinic, $P2_1/c$, $a = 17{\cdot}629\,(4)$, $b = 9{\cdot}173\,(2)$, $c = 22{\cdot}225\,(6)$ Å, $\beta = 95{\cdot}73\,(2)°$, $U = 3576$ Å^3, $Z = 4$, $D_x = 1{\cdot}37$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 8{\cdot}0$ cm^{-1}, $F(000) = 1508$, $T = 293$ K, $R = 0{\cdot}056$ for 3090 reflections. The anion is tetrahedral, with Fe—Cl 2·170–2·191 Å. The cation is bent at N; P—N—P 141·8 (3)°.

Experimental. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode (Clegg, 1981). 4785 reflections, $2\theta_{\max}$ 45°, $\pm h+k+l$. Three check reflections, no intensity change. Crystal size 0·4 × 0·3 × 0·1 mm; no absorption correction. 4642 unique reflections (R_{int} 0·020), 3090 with $F > 4\sigma(F)$ used for all calculations (program system *SHELXTL*; Sheldrick, 1983). Index range $|h| \leq 18$, $k \leq 9$, $l \leq 23$. Cell constants refined from $\pm 2\theta$ values of 32 reflections in the range 20–23°.

Structure solution by routine direct methods (to locate P, Fe, Cl) and subsequent difference synthesis. Refinement on F to R 0·056, wR 0·050; all non-H atoms anisotropic; H atoms included using riding model with C—H 0·96 Å, $U(\text{H}) = 1{\cdot}2U_{\text{eq}}(\text{C})$; weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}0002F^2$; 397 parameters; $S = 1{\cdot}52$; max. Δ/σ 0·08; max. features in final $\Delta\rho$ map $\pm 0{\cdot}3$ e Å^{-3}. Atomic scattering factors from *SHELXTL*. Final atomic coordinates are given in

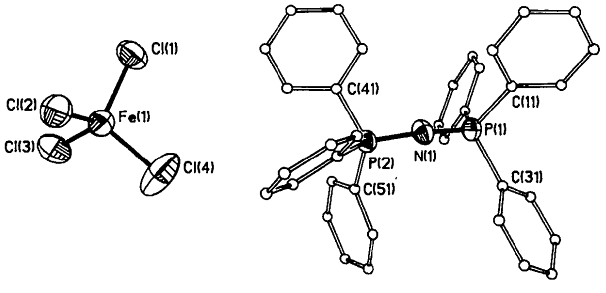

Fig. 1. Asymmetric unit of the title compound, showing the atom-numbering scheme.

Table 1. *Atomic coordinates* (×10^4) *and isotropic thermal parameters* (Å^2 ×10^3)

	x	y	z	U_{eq}*
P(1)	1696 (1)	7391 (1)	1055 (1)	38 (1)
P(2)	3359 (1)	7922 (2)	1015 (1)	37 (1)
N(1)	2497 (2)	8169 (4)	1126 (2)	42 (1)
C(11)	1172 (3)	8065 (5)	1651 (2)	41 (2)
C(12)	1552 (3)	8670 (7)	2155 (3)	66 (3)
C(13)	1158 (3)	9149 (7)	2629 (3)	89 (3)
C(14)	385 (3)	8978 (8)	2589 (3)	94 (3)
C(15)	−6 (3)	8379 (8)	2091 (3)	95 (3)
C(16)	391 (3)	7912 (7)	1624 (3)	69 (2)
C(21)	1714 (2)	5434 (5)	1107 (2)	37 (2)
C(22)	1490 (3)	4721 (5)	1612 (2)	45 (2)
C(23)	1530 (3)	3213 (6)	1650 (2)	57 (2)
C(24)	1789 (3)	2419 (6)	1191 (2)	62 (2)
C(25)	2021 (3)	3111 (6)	693 (3)	62 (2)
C(26)	1981 (3)	4615 (5)	645 (2)	50 (2)
C(31)	1160 (3)	7831 (5)	347 (2)	41 (2)
C(32)	621 (3)	6890 (7)	73 (3)	82 (3)
C(33)	194 (4)	7271 (7)	−462 (3)	99 (3)
C(34)	320 (3)	8583 (7)	−724 (3)	77 (3)
C(35)	839 (3)	9512 (6)	−463 (3)	65 (2)
C(36)	1265 (3)	9160 (6)	76 (2)	50 (2)
C(41)	3868 (3)	6855 (5)	1606 (2)	39 (2)
C(42)	3491 (3)	6230 (6)	2055 (2)	55 (2)
C(43)	3899 (4)	5458 (7)	2518 (3)	70 (3)
C(44)	4667 (4)	5295 (7)	2525 (3)	71 (3)
C(45)	5053 (3)	5927 (7)	2089 (3)	66 (3)
C(46)	4654 (3)	6707 (6)	1631 (2)	53 (2)
C(51)	3483 (2)	7086 (5)	299 (2)	39 (2)
C(52)	3844 (3)	5760 (6)	257 (3)	63 (2)
C(53)	3886 (4)	5138 (7)	−304 (3)	84 (3)
C(54)	3556 (4)	5797 (9)	−812 (3)	82 (3)
C(55)	3185 (4)	7100 (8)	−773 (3)	75 (3)
C(56)	3162 (3)	7757 (6)	−224 (2)	57 (2)
C(61)	3817 (2)	9671 (5)	1028 (2)	36 (2)
C(62)	4463 (3)	9916 (6)	733 (2)	56 (2)
C(63)	4835 (3)	11256 (7)	794 (3)	69 (3)
C(64)	4569 (4)	12327 (7)	1146 (3)	62 (3)
C(65)	3933 (3)	12081 (6)	1433 (3)	65 (2)
C(66)	3562 (3)	10760 (6)	1385 (2)	49 (2)
Fe(1)	7523 (1)	7994 (1)	1403 (1)	57 (1)
Cl(1)	7154 (1)	6676 (2)	2142 (1)	80 (1)
Cl(2)	8025 (1)	10032 (2)	1771 (1)	75 (1)
Cl(3)	8376 (1)	6747 (2)	971 (1)	82 (1)
Cl(4)	6546 (1)	8534 (2)	770 (1)	118 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

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