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## Bis(triphenylphosphine-*P*)iminium Trichloro( $\eta^5$ -cyclopentadienyl)chromide(III)

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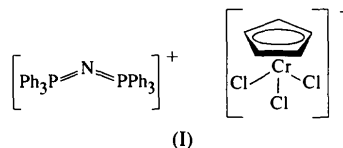
### Abstract

The two independent formula units of the title compound,  $[(C_{18}H_{15}P)_2N][CrCl_3(C_5H_5)]$ , display similar pseudo-tetrahedral anions. The Cr—Cl bond lengths of 2.291(2)–2.318(2) Å are consistent with values from previous studies.

### Comment

We are interested in the reactions of  $PX_5$  ( $X = Cl, Br$ ) with hexacarbonyls of chromium (Scheer *et al.*, 1989; Scheer, Nam, Herrmann, Fedin & Fedorov, 1990), molybdenum and tungsten (Scheer *et al.*, 1988; Scheer, Nam, Herrmann, Fedin, Ikorski & Fedorov, 1990), and with cyclopentadienyl complexes of the type  $[CpM(CO)_3]_2$  (Scheer, Nam, Schenzel *et al.*, 1990). Our studies led to a simple synthesis of new halogeno complexes of group 6 transition metals. The confirmation

of the Cp-containing products as ionic species was of particular importance. We report here the synthesis and structure of  $[PPN][CpCrCl_3]$ , (I), where PPN is bis(triphenylphosphine)iminium.



The asymmetric unit contains two formula units of the title compound. The Cr atoms are tetrahedrally coordinated by the cyclopentadienyl (interpreted as one position) and three chloro ligands (Table 1; 'Cent' refers to the centre of gravity of the Cp rings). The Cl—Cr—Cl angles are narrower than Cp—Cr—Cl. The chloro ligands are so oriented that Cl3 eclipses C1 and Cl5 eclipses C6.

The distances from Cr to the least-squares plane of the Cp ligand are 1.907(2) Å for Cr1 and 1.900(3) Å for Cr2. The Cr—Cl bond lengths lie in the range 2.291(2)–2.318(2) Å. Similar Cr—Cl distances were observed in two other structure determinations of the same anion: 2.316(3) and 2.322(2) Å (mirror symmetry) in  $[W(H)_2(Cl)(H_2O)(Pr_3P)_2][CpCrCl_3].CH_3CN$  (Eremenko, Rosenberger, Nefedov, Berke & Novotortsev, 1995), and 2.290(5) and 2.314(5) Å in  $[Li_2(thf)_4(dioxane)]_2[CpCrCl_3]_2$  (Müller & Krause, 1972); in the latter compound, the third Cr—Cl bond is lengthened to 2.346(4) Å by bridging to lithium. The PPN cations display the usual bent geometry at nitrogen, with P1—N1—P2 140.8(2), P3—N2—P4 141.7(2)° and P—N 1.578(3)–1.581(3) Å.

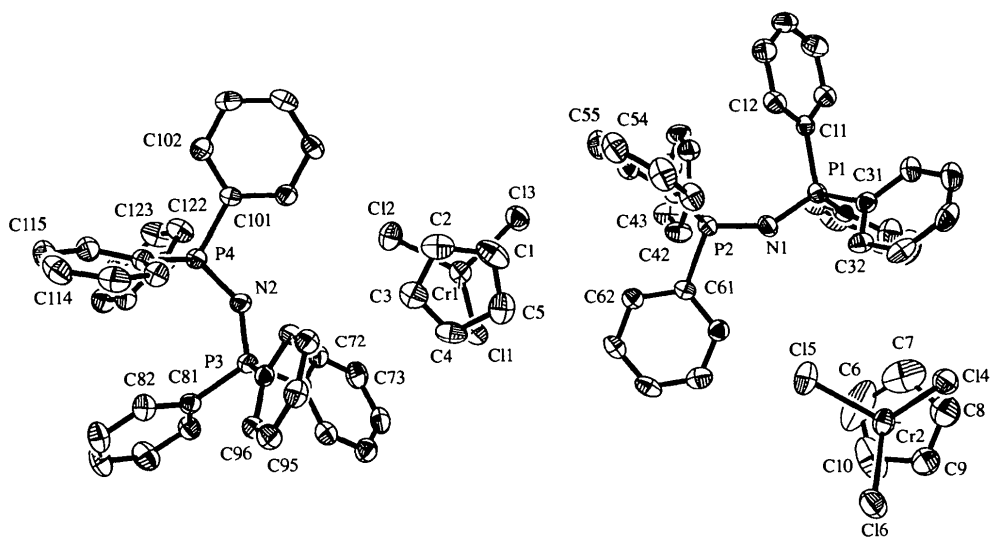


Fig. 1. A view of the two independent formula units of the title compound in the crystal. H atoms have been omitted for clarity. Ellipsoids represent 50% probability levels.

## Experimental

The title compound was prepared by refluxing 0.12 g (0.5 mmol)  $\text{CpCrCl}_2(\text{CH}_3\text{CN})$  (Scheer, Nam, Schenzel *et al.*, 1990) and 0.3 g (0.52 mmol)  $[\text{PPN}]\text{Cl}$  for 2 h in 30 ml  $\text{CH}_3\text{CN}$ . The solvent volume was reduced to 10 ml and 5 ml diethyl ether added. Storage at 278 K gave 0.25 g (63%) blue crystals. Analysis:  $\text{C}_{41}\text{H}_{35}\text{Cl}_3\text{CrNP}_2$ , calculated C 64.62, H 4.63, N 1.84%; found C 64.24, H 4.52, N 1.51%.

### Crystal data

$(\text{C}_{36}\text{H}_{30}\text{NP}_2)[\text{CrCl}_3(\text{C}_5\text{H}_5)]$

$M_r = 761.99$

Triclinic

$P\bar{1}$

$a = 10.307(5) \text{ \AA}$

$b = 16.161(10) \text{ \AA}$

$c = 22.496(10) \text{ \AA}$

$\alpha = 91.57(4)^\circ$

$\beta = 89.81(4)^\circ$

$\gamma = 101.10(4)^\circ$

$V = 3676(3) \text{ \AA}^3$

$Z = 4$

$D_x = 1.377 \text{ Mg m}^{-3}$

$D_m$  not measured

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 50 reflections

$\theta = 10.0\text{--}11.5^\circ$

$\mu = 0.647 \text{ mm}^{-1}$

$T = 178(2) \text{ K}$

Irregular fragment

$0.50 \times 0.45 \times 0.40 \text{ mm}$

Blue

### Data collection

Siemens R3 diffractometer

$\omega$  scans

Absorption correction:

$\psi$  scans (XEMP; Nicolet, 1987)

$T_{\min} = 0.689$ ,  $T_{\max} = 0.772$

24 201 measured reflections

12 995 independent reflections

8181 reflections with

$I > 2\sigma(I)$

$R_{\text{int}} = 0.052$

$\theta_{\max} = 25.05^\circ$

$h = -12 \rightarrow 8$

$k = -19 \rightarrow 19$

$l = -26 \rightarrow 26$

3 standard reflections every 147 reflections intensity decay: 2%

### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.164$

$S = 0.950$

12 995 reflections

865 parameters

H atoms riding

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$

$\Delta\rho_{\max} = 0.497 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.623 \text{ e \AA}^{-3}$

Extinction correction: none

Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Cr1—Cent1	1.907	Cr2—Cent2	1.901
Cr1—Cl3	2.291 (2)	Cr2—Cl5	2.294 (2)
Cr1—Cl2	2.300 (2)	Cr2—Cl6	2.306 (2)
Cr1—Cl1	2.312 (2)	Cr2—Cl4	2.318 (2)
Cl3—Cr1—Cl2	96.27 (6)	Cl5—Cr2—Cl6	98.12 (7)
Cl3—Cr1—Cl1	93.18 (6)	Cl5—Cr2—Cl4	96.95 (6)
Cl2—Cr1—Cl1	100.05 (6)	Cl6—Cr2—Cl4	96.15 (6)
Cl1—Cr1—Cent1	121.0	Cl4—Cr2—Cent2	119.4
Cl2—Cr1—Cent1	118.5	Cl5—Cr2—Cent2	120.1
Cl3—Cr1—Cent1	122.1	Cl6—Cr2—Cent2	120.8

Data collection: *P3 Program System* (Nicolet, 1987). Cell refinement: *P3 Program System*. Data reduction: *XDISK* in *P3 Program System*. Program(s) used to solve structure:

*SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1314). Services for accessing these data are described at the back of the journal.

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## Triphenyl[2-(triphenylsilyl)phenylthio]phosphonium Tetraoxorhenate(1–)–Dichloromethane (1/1)

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## Abstract

The cation of the title compound,  $\text{C}_{42}\text{H}_{34}\text{PSSi}^+ \cdot \text{ReO}_4^- \cdot \text{CH}_2\text{Cl}_2$ , is formed by a bond between the S atom of the 2-(triphenylsilyl)benzenethiol and the P atom of the