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## Key indicators

Single-crystal X-ray study  
 $T = 103$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$   
 $R$  factor = 0.034  
 $wR$  factor = 0.080  
Data-to-parameter ratio = 16.4For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.*trans*-Dichlorobis(10-dodecyl-9-phospha-  
10-silatriptycene- $\kappa P$ )palladium(II)The title compound,  $[\text{PdCl}_2(\text{C}_{30}\text{H}_{37}\text{PSi})_2]$ , has small C—P—C and C—Si—C angles and a short intramolecular contact between the P and Si atoms due to the structural constraint imposed by the rigid triptycene framework. The molecule lies on a center of inversion.

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## Comment

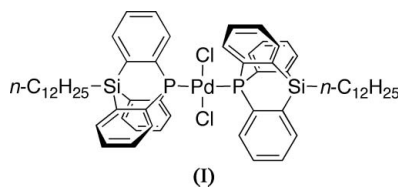
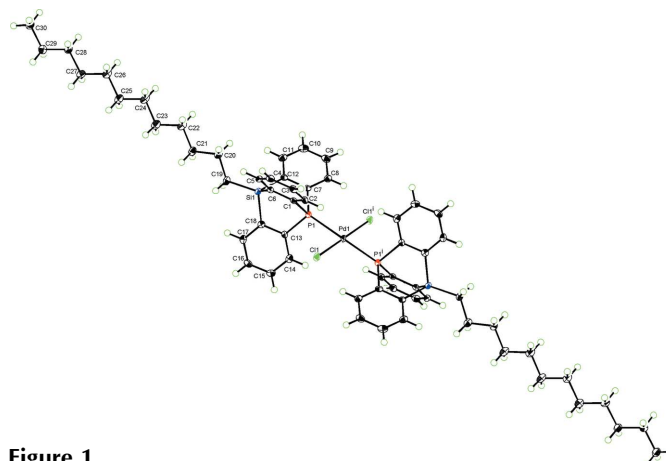
As part of our studies on the synthesis and properties of 9-phospha-10-silatriptycenes (Tsuji *et al.*, 2006), we have prepared the title compound, (I). Although there are some reports on the preparation of 9-phosphatriptycene–transition metal complexes (Agou *et al.*, 2004), no X-ray structure determinations of such compounds have been reported.As shown in Fig. 1 and summarized in Table 1, the centrosymmetric complex (I) has a *trans* configuration about the square-planar palladium center [ $\text{P}1-\text{Pd}1-\text{P}1^i = 180^\circ$  and  $\text{Cl}1-\text{Pd}1-\text{P}1 = 82.77(5)^\circ$ ; symmetry code: (i)  $2 - x, -y, 2 - z$ ], as is usually seen for this type of complex. The  $\text{Pd}1-\text{Cl}1$  and  $\text{Pd}1-\text{P}1$  bond lengths are similar to those of *trans*- $\text{PdCl}_2(\text{PPh}_3)_2$  [ $\text{Pd}1-\text{Cl}1 = 2.2997(7) \text{ \AA}$  and  $\text{Pd}1-\text{P}1 = 2.3247(6) \text{ \AA}$ ; Oilunkaniemi *et al.*, 2003]The characteristic features of this complex are as follows: (i) the dodecyl group has the all-*anti* conformation, with C—C—

Figure 1

A view of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size. Symmetry code: (i)  $2 - x, -y, 2 - z$ .

C—C torsion angles of 175.6 (2)–180.0 (2)°; (ii) the intramolecular P—Si distance [P1—Si1 = 3.0774 (15) Å] is significantly smaller than the sum of the van der Waals radii [1.90 Å for P and 2.00 Å for Si; Emsley (1999)], suggesting a possible intramolecular interaction between these two atoms; (iii) the C—P—C angles [99.25 (11)–103.27 (11)°; average = 101.2°] are narrower than those of *trans*-PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> [102.99 (11)–108.02 (11)°; average = 105.0°; Oilunkaniemi *et al.*, 2003]. The C—Si—C angles [100.03 (11)–103.75 (11)°; average = 101.7°] within the triptycene framework are also significantly narrowed for the *Sisp*<sup>3</sup> atoms. These structural features are due to the constraint imposed by the triptycene framework composed of three bridging benzene rings.

### Experimental

A mixture of PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (140 mg, 0.540 mmol) and 10-dodecyl-9-phospha-10-silatrypticene (500 mg, 1.09 mmol) in benzene (7.0 ml) was stirred for 2 h at room temperature. The solvent was evaporated and the resulting yellow solid was recrystallized from benzene to give the title compound, (I) [266 mg, 45% yield; m.p. 505–507 K (decomposition)]. Single crystals suitable for X-ray crystallography were obtained by slow evaporation of an excess of diethyl ether into a concentrated solution of (I) in dichloromethane. <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 9.26 (*dd*, 6H, *J* = 6.9, 14.1 Hz), 7.75 (*dd*, 6H, *J* = 0.6, 7.5 Hz), 7.40 (*ddd*, 6H, *J* = 1.5, 7.5, 7.5 Hz), 7.31 (*dd*, 6H, *J* = 6.9, 7.5 Hz), 2.10–2.18 (*m*, 4H), 1.85–1.91 (*m*, 4H), 1.69–1.78 (*m*, 4H), 1.48–1.58 (*m*, 4H), 1.26–1.48 (*m*, 28H), 0.87–0.91 (*m*, 6H); <sup>13</sup>C NMR (CDCl<sub>3</sub>): δ 142.2 (virtual triplet, *J*<sub>C—P</sub> = 3.3 Hz), 140.7 (*vt*, *J*<sub>C—P</sub> = 23 Hz), 136.7 (*vt*, *J*<sub>C—P</sub> = 11 Hz), 131.8 (*vt*, *J*<sub>C—P</sub> = 3.3 Hz), 128.4, 127.8 (*vt*, *J*<sub>C—P</sub> = 6.6 Hz), 34.3, 32.0, 29.81, 29.77, 29.7, 29.5, 29.3, 23.7, 22.8, 14.3, 5.54 (one aliphatic signal is overlapped); <sup>29</sup>Si NMR (CDCl<sub>3</sub>): δ -30.7 (*vt*, *J*<sub>Si—P</sub> = 10.7 Hz); <sup>31</sup>P NMR (CDCl<sub>3</sub>): δ -10.4; elemental analysis calculated for C<sub>60</sub>H<sub>74</sub>Cl<sub>2</sub>P<sub>2</sub>PdSi<sub>2</sub>: C 66.07, H 6.84%; found: C 65.77, H 6.65%.

#### Crystal data

[PdCl <sub>2</sub> (C <sub>30</sub> H <sub>37</sub> PSi) <sub>2</sub> ]	Z = 1
<i>M<sub>r</sub></i> = 1090.61	<i>D<sub>x</sub></i> = 1.345 Mg m <sup>-3</sup>
Triclinic, <i>P</i> $\bar{1}$	Mo <i>K</i> α radiation
<i>a</i> = 9.253 (6) Å	Cell parameters from 3738 reflections
<i>b</i> = 10.897 (7) Å	<i>θ</i> = 3.1–25.5°
<i>c</i> = 14.311 (9) Å	<i>μ</i> = 0.59 mm <sup>-1</sup>
<i>α</i> = 106.403 (10)°	<i>T</i> = 103 (2) K
<i>β</i> = 92.296 (10)°	Prism, yellow
<i>γ</i> = 102.087 (9)°	0.20 × 0.15 × 0.05 mm
<i>V</i> = 1346.0 (15) Å <sup>3</sup>	

#### Data collection

Rigaku Saturn70 CCD diffractometer	4993 independent reflections
<i>ω</i> scans	4405 reflections with <i>I</i> > 2σ( <i>I</i> )
Absorption correction: multi-scan (Jacobson, 1998)	<i>R</i> <sub>int</sub> = 0.041
<i>T</i> <sub>min</sub> = 0.892, <i>T</i> <sub>max</sub> = 0.971	<i>θ</i> <sub>max</sub> = 25.5°
14206 measured reflections	<i>h</i> = -11 → 11
	<i>k</i> = -13 → 13
	<i>l</i> = -17 → 17

#### Refinement

Refinement on <i>F</i> <sup>2</sup>	<i>w</i> = 1/[σ <sup>2</sup> ( <i>F</i> <sub>o</sub> <sup>2</sup> ) + (0.0324 <i>P</i> ) <sup>2</sup> + 0.108 <i>P</i> ]
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )] = 0.034	where <i>P</i> = ( <i>F</i> <sub>o</sub> <sup>2</sup> + 2 <i>F</i> <sub>c</sub> <sup>2</sup> )/3
<i>wR</i> ( <i>F</i> <sup>2</sup> ) = 0.080	(Δ/σ) <sub>max</sub> = 0.001
<i>S</i> = 1.09	Δρ <sub>max</sub> = 0.50 e Å <sup>-3</sup>
4993 reflections	Δρ <sub>min</sub> = -0.28 e Å <sup>-3</sup>
304 parameters	
H-atom parameters constrained	

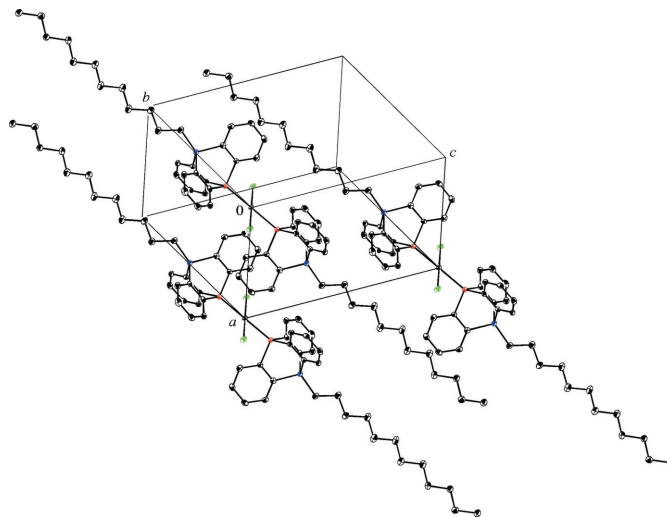


Figure 2

The molecular packing of (I). H atoms bonded to C atoms have been omitted.

Table 1

Selected geometric parameters (Å, °).

Pd1—Cl1	2.3014 (13)	P1—Si1	3.0774 (15)
Pd1—P1	2.3596 (11)		
Cl1—Pd1—Cl1 <sup>i</sup>	180	C1—P1—C7	99.25 (11)
Cl1—Pd1—P1	82.77 (5)	C13—P1—C7	101.15 (11)
Cl1 <sup>i</sup> —Pd1—P1	97.23 (5)	C18—Si1—C6	103.75 (11)
P1—Pd1—P1 <sup>i</sup>	180	C18—Si1—C12	101.45 (11)
C1—P1—C13	103.27 (11)	C6—Si1—C12	100.03 (11)

Symmetry code: (i) -*x* + 2, -*y*, -*z* + 2.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.95–1.00 Å and with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C).

Data collection: *CRYSTALCLEAR* (Rigaku, 2004); cell refinement: *CRYSTALCLEAR*; data reduction: *CRYSTALCLEAR*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* and *Yadokari-XG* (Wakita, 2005).

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