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trans-Bis(carbazol-3-ylethynyl)bis(tri-*n*-butylphosphine)platinum(II)

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.005 Å
R factor = 0.024
wR factor = 0.062
Data-to-parameter ratio = 16.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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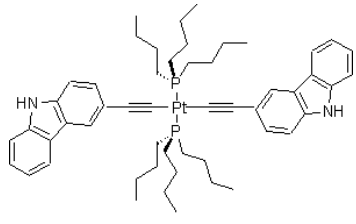
The title compound, $[Pt(C_{12}H_{27}P)_2(C_{14}H_8N)_2]$, is a mononuclear Pt^{II} di-yne exhibiting π-conjugation along the molecular backbone. It is used as a model species for rigid-rod platinum poly yne compounds of which it is a precursor. Such compounds are of interest due to the extended π-conjugation through the hetero-aromatic linker unit in the backbone.

Comment

Here we report the structural characterization of the title compound, (I), which is a mononuclear platinum(II) di-yne species, *trans*-$[Pt(P^nBu_3)_2(-C{\equiv}CR)_2]$ (R = carbazol-3-yl). Such platinum-containing species form the building blocks for rigid-rod organometallic poly-ynes of general formula *trans*-$[Pt(P^nBu_3)_2-C{\equiv}C-R-C{\equiv}C-]_\infty$ (R = aromatic or a heteroaromatic linker unit). Platinum(II) poly-ynes are of immense current interest due to π-electron conjugation along the backbone, donor–acceptor metal–ligand interactions and novel photophysical properties (Wittmann *et al.*, 1994; Beljonne *et al.*, 1996; Younus *et al.*, 1998; Chawdhury *et al.*, 1998, 1999; Khan, Al-Mandhary, Al-Suti, Hisahm *et al.*, 2002; Khan, Al-Mandhary, Al-Suti, Feeder *et al.*, 2002; Khan, Al-Mandhary, Al-Suti, Corcoran *et al.* 2003; Khan, Al-Suti *et al.*, 2003; Khan, Al-Mandhary, Al-Suti, Ahrens *et al.*, 2003). They possess interesting opto-electronic properties useful for application in light-emitting diodes and photocells (Wilson *et al.*, 2000; Wilson, Chawdhury *et al.*, 2001; Wilson, Dhoot *et al.*, 2001). Precursors to these species, such as the title compound, are studied as models of the molecular and electronic properties and structure–property relationships in the metal poly-ynes.



(I)

The structure of (I) exhibits a C—H···N close contact between the alkyl H atom H25*A* and nitrogen N1, with a C25···N1 distance of 3.674 (5) Å (Table 2).

Experimental

The title compound was synthesized by the following procedure. To a stirred solution of *trans*-$[(P^nBu_3)_2PtCl_2]$ (0.38 g, 0.5 mmol) and 3-ethynylcarbazole (0.19 g, 1.0 mmol) in $CH_2Cl_2/^iPr_2NH$ (50 ml, 1:1 *v*/*v*) under nitrogen was added a catalytic amount of CuI (5 mg). The

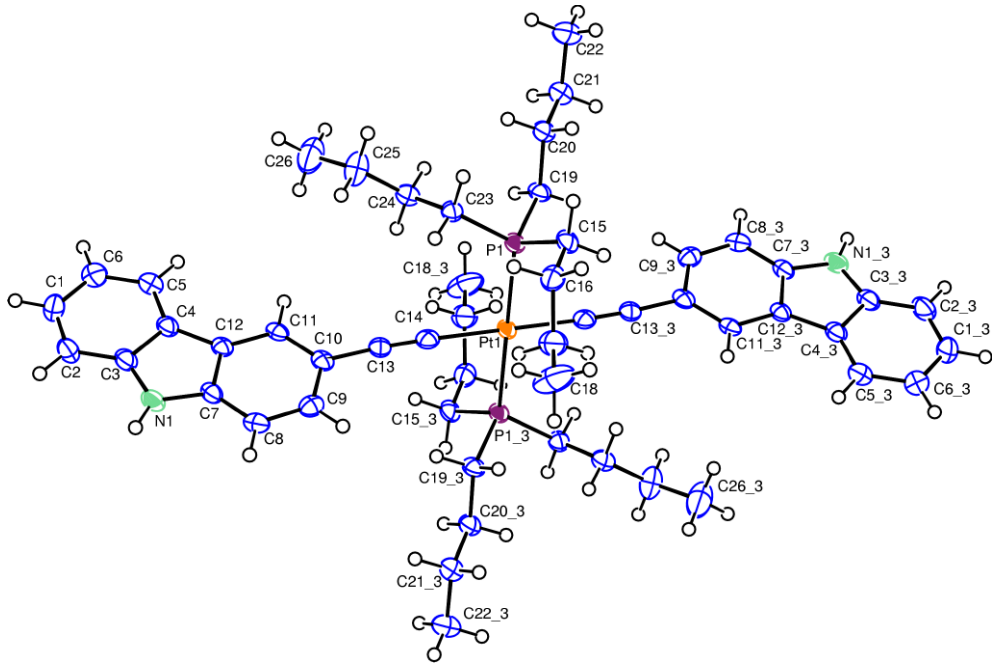


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

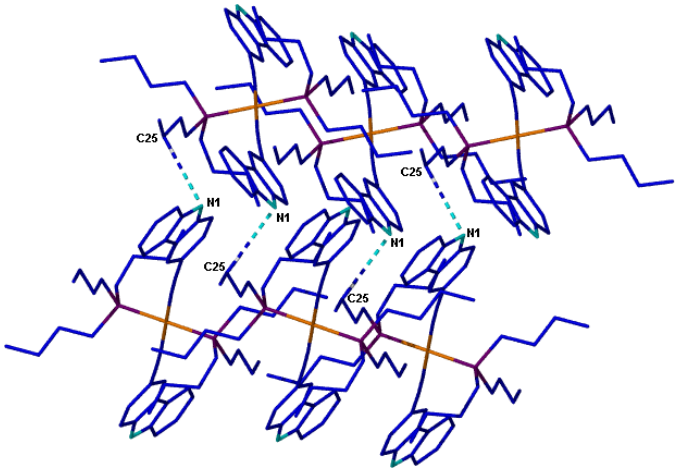


Figure 2
Packing diagram of compound (I), showing the close intermolecular contact between atoms N1 and H25*A*.

yellow solution was stirred at room temperature for 15 h, after which all volatile components were removed under reduced pressure. The residue was dissolved in CH_2Cl_2 and passed through a silica column eluting with hexane/CH_2Cl_2 (1:1, *v*/*v*). Removal of the solvents under vacuo gave the title complex as a pale-yellow solid in 72% yield.

Crystal data

$[Pt(C_{12}H_{27}P)_2(C_{14}H_8N)_2]$	D_x = 1.359 Mg m^{-3}
M_r = 980.13	Mo $K\alpha$ radiation
Monoclinic, $P2_1/a$	Cell parameters from 25483 reflections
a = 9.2860 (1) Å	θ = 2.9–27.5°
b = 19.4330 (3) Å	μ = 3.03 mm^{-1}
c = 13.6980 (2) Å	T = 150 (2) K
β = 104.238 (1)°	Block, white
V = 2395.94 (6) Å^3	0.33 × 0.25 × 0.13 mm
Z = 2	

Data collection

Bruker–Nonius KappaCCD diffractometer	4221 independent reflections
φ and ω scans	3270 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)	R_{int} = 0.070
T_{min} = 0.538, T_{max} = 0.683	θ_{max} = 25.0°
35095 measured reflections	$h = -11 \rightarrow 11$
	$k = -23 \rightarrow 23$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0312P)^2 + 0.9277P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.024	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.062	$\Delta\rho_{max}$ = 0.70 e Å^{-3}
S = 1.03	$\Delta\rho_{min}$ = −1.32 e Å^{-3}
4221 reflections	Extinction correction: *SHELXL*97
260 parameters	Extinction coefficient: 0.0016 (3)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C3—N1	1.383 (4)	C13—C14	1.199 (4)
C3—C4	1.408 (4)	C14—Pt1	2.013 (3)
C4—C12	1.453 (4)	C15—P1	1.825 (3)
C7—N1	1.381 (4)	C19—P1	1.830 (3)
C7—C12	1.414 (4)	C23—P1	1.819 (3)
C10—C13	1.447 (4)	P1—Pt1	2.2935 (8)
N1—C3—C4	108.5 (3)	C7—N1—C3	109.5 (3)
C3—C4—C12	106.9 (3)	C23—P1—Pt1	117.91 (11)
N1—C7—C12	108.7 (3)	C15—P1—Pt1	112.13 (11)
C7—C12—C4	106.3 (3)	C19—P1—Pt1	111.94 (10)
C14—C13—C10	176.7 (3)	C14—Pt1—P1	93.30 (8)
C13—C14—Pt1	177.5 (3)		
N1—C3—C4—C12	−0.6 (3)	C4—C3—N1—C7	1.0 (3)
C8—C9—C10—C13	−178.2 (3)	C13—C14—Pt1—P1	−170 (7)
N1—C7—C12—C4	0.5 (3)	C23—P1—Pt1—C14	−5.91 (15)
C3—C4—C12—C7	0.1 (3)	C15—P1—Pt1—C14	115.13 (14)
C12—C7—N1—C3	−0.9 (3)	C19—P1—Pt1—C14	−127.65 (15)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C25—H25A···N1^{i}	0.99	2.69	3.674 (5)	171

Symmetry code: (i) $\frac{1}{2}+x, \frac{1}{2}-y, z$.

Aromatic, methylene and methyl H atoms were constrained as riding atoms, fixed to the parent atoms with distances of 0.95, 0.99 and 0.98 Å, respectively. The isotropic displacement parameters were fixed to 120% of that of the parent atom for aromatic and methylene H atoms and 150% for methyl H atoms.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997), *X-SEED* (Barbour, 2001) and *POV-Ray for Windows* (Cason, 1999); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.

Beljonne, D., Wittmann, H. F., Köhler, A., Graham, S., Younus, M., Lewis, J., Raithby, P. R., Khan, M. S., Friend, R. H. & Bredas, J. L. (1996). *J. Chem. Phys.* **105**, 3868–3877.

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38

Cason, C. (1999). *POV-Ray for Windows*. POV-Team, Williamstown, Australia.

Chawdhury, N., Köhler, A., Friend, R. H., Wong, W.-Y., Lewis, J., Younus, M., Raithby, P. R., Corcoran, T. C., Al-Mandhary, M. R. A. & Khan, M. S. (1999). *J. Chem. Phys.* **110**, 4963–4970.

Chawdhury, N., Köhler, A., Friend, R. H., Younus, M., Long, N. J., Raithby, P. R. & Lewis, J. (1998). *Macromolecules*, **31**, 722–727.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Khan, M. S., Al-Mandhary, M. R. A., Al-Suti, M. K., Ahrens, B., Mahon, M. F., Male, L., Raithby, P. R., Boothby, C. E. & Köhler, A. (2003). *J. Chem. Soc. Dalton Trans.* pp. 74–84.

Khan, M. S., Al-Mandhary, M. R. A., Al-Suti, M. K., Corcoran, T. C., Al-Mahrooqi, Y., Attfield, J. P., Feeder, N., David, W. I. F., Shankland, K., Friend, R. H., Köhler, A., Marseglia, E. A., Tedesco, E., Tang, C. C., Raithby, P. R., Collings, J. C., Roscoe, K. P., Batsanov, A. S., Stimson, L. M. & Marder, T. B. (2003). *New J. Chem.* **27**, 140–149.

Khan, M. S., Al-Mandhary, M. R. A., Al-Suti, M. K., Feeder, N., Nahar, S., Köhler, A., Friend, R. H., Wilson, P. J. & Raithby, P. R. (2002). *J. Chem. Soc. Dalton Trans.* pp. 2441–2448.

Khan, M. S., Al-Mandhary, M. R. A., Al-Suti, M. K., Hisahm, A. K., Raithby, P. R., Ahrens, B., Mahon, M. F., Male, L., Marseglia, E. A., Tedesco, E., Friend, R. H., Köhler, A., Feeder, N. & Teat, S. J. (2002). *J. Chem. Soc. Dalton Trans.* pp. 1358–1368.

Khan, M. S., Al-Suti, M. K., Al-Mandhary, M. R. A., Ahrens, B., Bjernemose, J. K., Mahon, M. F., Male, L., Raithby, P. R., Friend, R. H., Köhler, A. & Wilson, J. S. (2003). *J. Chem. Soc. Dalton Trans.* pp. 65–73.

Nonius (1997). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Wilson, J. S., Chawdhury, N., Al-Mandhary, M. R. A., Younus, M., Khan, M. S., Raithby, P. R., Köhler, A. & Friend, R. H. (2001). *J. Am. Chem. Soc.* **123**, 9412–9417.

Wilson, J. S., Dhoot, A. S., Seeley, A. J. A. B., Khan, M. S., Köhler, A. & Friend, R. H. (2001). *Nature (London)*, **413**, 828–831.

Wilson, J. S., Köhler, A., Friend, R. H., Al-Suti, M. K., Al-Mandhary, M. R. A., Khan, M. S. & Raithby, P. R. (2000). *J. Chem. Phys.* **113**, 7627–7634.

Wittmann, H. F., Friend, R. H., Kahn, M. S. & Lewis, J. (1994). *J. Chem. Phys.* **101**, 2693–2698.

Younus, M., Köhler, A., Cron, S., Chawdhury, N., Al-Mandhary, M. R. A., Khan, M. S., Lewis, J., Long, N. J., Friend, R. H. & Raithby, P. R. (1998). *Angew. Chem. Int. Ed.* **37**, 3036–3039.