

ORGANOMETALLICS

Organometallics, 1998, 17(16), 3621-3623, DOI:[10.1021/om971139u](https://doi.org/10.1021/om971139u)

Terms & Conditions

Electronic Supporting Information files are available without a subscription to ACS Web Editions. The American Chemical Society holds a copyright ownership interest in any copyrightable Supporting Information. Files available from the ACS website may be downloaded for personal use only. Users are not otherwise permitted to reproduce, republish, redistribute, or sell any Supporting Information from the ACS website, either in whole or in part, in either machine-readable form or any other form without permission from the American Chemical Society. For permission to reproduce, republish and redistribute this material, requesters must process their own requests via the RightsLink permission system. Information about how to use the RightsLink permission system can be found at <http://pubs.acs.org/page/copyright/permissions.html>



ACS Publications

MOST TRUSTED. MOST CITED. MOST READ.

Copyright © 1998 American Chemical Society

Table 1. Crystal data and structure refinement for 5.

Crystal data

Identification code	3bo18c
Empirical formula	C ₂₇ H _{18.50} Br _{0.50} N ₂ O ₄ Pd ₂ S ₂
Formula weight	751.81
Crystal description	plate
Crystal colour	red
Crystal size	0.48 x 0.17 x 0.03 mm
Crystal system	Monoclinic
Space group	P2 ₁ /c
Unit cell dimensions	a = 9.8921(3) Å α = 90° b = 21.9373(7) Å β = 100.7190(10)° c = 11.7254(3) Å γ = 90°
Reflns to determine unit cell	8192
θ range to determine unit cell	1.86 to 25.36
F(000)	1472
Volume	2500.08(13) Å ³
Z	4
Density (calculated)	1.997 Mg/m ³
Radiation	MoKα λ = 0.71073Å fine-focus sealed tube, graphite monochromator
Linear absorption coefficient	2.444 mm ⁻¹
Temperature of measurement	141(2)
<i>Data collection</i>	
Diffractometer	Siemens SMART CCD
Temperature of data collection	141(2) K
Diffraction measurement method	Exposures over 0.3% φ or ω rotation
Absorption correction	Multi-scan (SADABS)
Max. and min. transmission	1.000000 and 0.260243

Reflections collected	11738
Independent reflections	4367 ($R_{int} = 0.0791$)
θ range for data collection	1.86 to 25.36 $^{\circ}$
Range of h , k and l	$-11 \leq h \leq 10$, $-25 \leq k \leq 26$, $-5 \leq l \leq 14$
Check reflections	no every ? reflections
Variation of check reflections	0

Refinement

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4367 / 23 / 355
Method of refining H atoms	calc
Goodness-of-fit on F^2	0.900
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0442$, $wR2 = 0.0871$
R indices (all data)	$R1 = 0.0809$, $wR2 = 0.0952$
Largest diff. peak and hole	0.876 and -0.807 $e\text{\AA}^{-3}$
Extinction method	none

Computer processing

Data collection	SMART (Siemens 1995)
Data reduction	SAINT (Siemens 1995)
Cell refinement	SAINT (Siemens 1995)
Structure solution	SHELXS-86 (Sheldrick, 1990)
Structure refinement	SHELXL-93 (Sheldrick, 1993)
Graphics by	SHELXL-93 (Sheldrick, 1993)
Computer publication	SHELXL-93 (Sheldrick, 1993)

Table 2. Atomic coordinates [$\times 10^4$] and equivalent isotropic displacement parameters [$\text{\AA}^2 \times 10^3$] for $\mathcal{S} \cdot U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Pd1	7479(1)	4315(1)	3085(1)	27(1)
Pd2	3927(1)	6429(1)	5480(1)	30(1)
C1	5285(6)	5160(3)	3124(6)	26(2)
C2	6640(7)	5025(3)	3678(5)	26(2)
C3	7161(6)	5360(3)	4663(6)	23(2)
C4	6327(6)	5797(3)	5087(5)	24(2)
C5	4945(6)	5878(3)	4587(6)	28(2)
C6	4426(7)	5572(3)	3563(6)	29(2)
S1'	3371(2)	4822(1)	1092(2)	34(1)
C2'	4899(6)	4799(3)	2078(5)	24(2)
N3'	5829(5)	4411(2)	1785(4)	27(1)
C3A'	5338(7)	4113(3)	740(6)	30(2)
C4'	6042(7)	3687(3)	211(6)	31(2)
C5'	5393(7)	3441(3)	-844(6)	38(2)
C6'	4065(7)	3621(3)	-1338(6)	38(2)
C7'	3367(7)	4033(3)	-814(6)	40(2)
C7A'	4003(7)	4280(3)	233(6)	34(2)
S1''	8429(2)	6219(1)	6887(2)	32(1)
C2''	6788(6)	6167(3)	6108(6)	27(2)
N3''	5858(5)	6517(2)	6461(5)	27(1)
C3A''	6444(7)	6836(3)	7469(6)	28(2)
C4''	5774(7)	7235(3)	8104(6)	37(2)
C5''	6517(8)	7500(3)	9107(6)	43(2)
C6''	7911(8)	7385(3)	9444(6)	40(2)
C7''	8599(7)	7002(3)	8818(6)	39(2)
C7A''	7851(7)	6732(3)	7837(6)	30(2)
O1A	8277(4)	3493(2)	2497(4)	32(1)
C1A	7927(7)	2999(3)	2888(7)	43(2)
O2A	7142(5)	2930(2)	3624(5)	46(1)
C2AA	8691(19)	2389(10)	2721(19)	36(6)
C2AB	8219(21)	2485(11)	2170(21)	47(6)
O1B	9045(4)	4254(2)	4493(4)	29(1)
C1B	8971(6)	3953(3)	5365(6)	28(2)
O2B	7919(4)	3707(2)	5637(4)	36(1)
C2B	10286(7)	3872(4)	6227(7)	47(2)
Br1	9119(2)	3818(1)	8892(2)	71(1)
C1C	9602(10)	4456(5)	9474(11)	87(3)
C2C	9566(9)	4555(5)	10655(10)	80(3)
C3C	9958(10)	5087(5)	11226(11)	98(3)

Table 3. Selected bond lengths [Å] and angles [°] for **5**.

Pd1-C2	1.953 (6)	Pd1-N3'	2.026 (5)
Pd1-O1B	2.047 (4)	Pd1-O1A	2.135 (4)
Pd1-Pd2#1	2.8810 (7)	Pd2-C5	1.991 (6)
Pd2-N3"	2.048 (5)	Pd2-O2B#1	2.062 (4)
Pd2-O2A#1	2.148 (4)	Pd2-Pd1#1	2.8810 (7)
C1-C6	1.402 (8)	C1-C2	1.408 (8)
C1-C2'	1.450 (8)	C2-C3	1.383 (8)
C3-C4	1.415 (8)	C4-C5	1.395 (8)
C4-C2"	1.447 (9)	C5-C6	1.388 (9)
S1'-C2'	1.725 (6)	S1'-C7A'	1.748 (7)
C2'-N3'	1.344 (7)	N3'-C3A'	1.394 (8)
C3A'-C4'	1.379 (8)	C3A'-C7A'	1.394 (9)
C4'-C5'	1.392 (9)	C5'-C6'	1.391 (9)
C6'-C7'	1.353 (9)	C7'-C7A'	1.381 (9)
S1"-C2"	1.712 (7)	S1"-C7A"	1.753 (7)
C2"-N3"	1.323 (7)	N3"-C3A"	1.403 (8)
C3A"-C4"	1.393 (8)	C3A"-C7A"	1.397 (9)
C4"-C5"	1.393 (9)	C5"-C6"	1.386 (10)
C6"-C7"	1.376 (9)	C7"-C7A"	1.379 (9)
O1A-C1A	1.249 (8)	C1A-O2A	1.273 (8)
C1A-C2AB	1.47 (3)	C1A-C2AA	1.57 (2)
O2A-Pd2#1	2.148 (4)	O1B-C1B	1.231 (7)
C1B-O2B	1.265 (7)	C1B-C2B	1.502 (9)
O2B-Pd2#1	2.062 (4)	Br1-C1C	1.592 (12)
C1C-C2C	1.409 (14)	C1C-C3C#2	1.414 (12)
C2C-C3C	1.366 (14)	C3C-C1C#2	1.414 (12)
C2-Pd1-N3'	81.3 (2)	C2-Pd1-O1B	94.2 (2)
N3'-Pd1-O1B	175.0 (2)	C2-Pd1-O1A	175.2 (2)
N3'-Pd1-O1A	98.0 (2)	O1B-Pd1-O1A	86.3 (2)
C2-Pd1-Pd2#1	87.8 (2)	N3'-Pd1-Pd2#1	95.13 (14)
O1B-Pd1-Pd2#1	82.37 (12)	O1A-Pd1-Pd2#1	87.56 (12)
C5-Pd2-N3"	80.9 (2)	C5-Pd2-O2B#1	93.0 (2)
N3"-Pd2-O2B#1	173.8 (2)	C5-Pd2-O2A#1	176.4 (2)
N3"-Pd2-O2A#1	99.0 (2)	O2B#1-Pd2-O2A#1	87.0 (2)
C5-Pd2-Pd1#1	108.0 (2)	N3"-Pd2-Pd1#1	103.08 (14)
O2B#1-Pd2-Pd1#1	79.75 (13)	O2A#1-Pd2-Pd1#1	75.55 (12)
C6-C1-C2	123.8 (6)	C6-C1-C2'	125.5 (6)
C2-C1-C2'	110.7 (5)	C3-C2-C1	116.7 (6)
C3-C2-Pd1	127.1 (5)	C1-C2-Pd1	115.7 (5)
C2-C3-C4	120.1 (6)	C5-C4-C3	121.9 (6)
C5-C4-C2"	114.2 (6)	C3-C4-C2"	123.8 (6)
C6-C5-C4	118.5 (6)	C6-C5-Pd2	127.4 (5)
C4-C5-Pd2	114.1 (5)	C5-C6-C1	118.5 (6)
C2'-S1'-C7A'	90.2 (3)	N3'-C2'-C1	118.5 (6)
N3'-C2'-S1'	113.8 (5)	C1-C2'-S1'	127.7 (5)
C2'-N3'-C3A'	112.3 (5)	C2'-N3'-Pd1	111.5 (4)
C3A'-N3'-Pd1	135.5 (5)	C4'-C3A'-C7A'	120.3 (6)
C4'-C3A'-N3'	126.2 (6)	C7A'-C3A'-N3'	113.5 (6)
C3A'-C4'-C5'	118.4 (7)	C6'-C5'-C4'	120.2 (7)
C7'-C6'-C5'	121.5 (7)	C6'-C7'-C7A'	118.7 (7)
C7'-C7A'-C3A'	121.0 (6)	C7'-C7A'-S1'	128.8 (6)
C3A'-C7A'-S1'	110.2 (5)	C2"-S1"-C7A"	89.6 (3)
N3"-C2"-C4	117.0 (6)	N3"-C2"-S1"	116.0 (5)

C4-C2"-S1"	126.9 (5)	C2"-N3"-C3A"	110.6 (6)
C2"-N3"-Pd2	113.6 (5)	C3A"-N3"-Pd2	135.7 (4)
C4"-C3A"-C7A"	118.9 (6)	C4"-C3A"-N3"	126.9 (6)
C7A"-C3A"-N3"	114.2 (6)	C3A"-C4"-C5"	119.0 (7)
C6"-C5"-C4"	120.3 (7)	C7"-C6"-C5"	121.6 (7)
C6"-C7"-C7A"	117.7 (7)	C7"-C7A"-C3A"	122.5 (6)
C7"-C7A"-S1"	128.1 (5)	C3A"-C7A"-S1"	109.4 (5)
C1A-O1A-Pd1	118.0 (4)	O1A-C1A-O2A	126.8 (6)
O1A-C1A-C2AB	110.9 (11)	O2A-C1A-C2AB	120.4 (11)
O1A-C1A-C2AA	121.4 (10)	O2A-C1A-C2AA	110.6 (10)
C1A-O2A-Pd2#1	132.1 (5)	C1B-O1B-Pd1	123.5 (4)
O1B-C1B-O2B	128.1 (6)	O1B-C1B-C2B	116.5 (6)
O2B-C1B-C2B	115.5 (6)	C1B-O2B-Pd2#1	125.1 (5)
C2C-C1C-C3C#2	121.9 (11)	C2C-C1C-Br1	119.7 (9)
C3C#2-C1C-Br1	118.4 (11)	C3C-C2C-C1C	123.8 (11)
C2C-C3C-C1C#2	114.4 (12)		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z+1 #2 -x+2,-y+1,-z+2

Table 4. Anisotropic displacement parameters [$\text{\AA}^2 \times 10^3$] for **5**.

The anisotropic displacement factor exponent takes the form:

$$-2\pi^2 [(ha^*)^2 U_{11} + \dots + 2hka^* b^* U_{12}]$$

	U11	U22	U33	U23	U13	U12
Pd1	27 (1)	28 (1)	26 (1)	-2 (1)	6 (1)	-1 (1)
Pd2	27 (1)	24 (1)	39 (1)	1 (1)	10 (1)	1 (1)
C1	27 (4)	25 (4)	28 (4)	-2 (3)	11 (3)	-3 (3)
C2	37 (4)	24 (4)	18 (4)	0 (3)	9 (3)	-6 (3)
C3	22 (3)	20 (4)	28 (4)	4 (3)	5 (3)	1 (3)
C4	34 (4)	21 (4)	18 (4)	-2 (3)	6 (3)	-7 (3)
C5	24 (4)	14 (4)	47 (5)	20 (3)	11 (3)	3 (3)
C6	29 (4)	34 (4)	21 (4)	6 (3)	0 (3)	-4 (3)
S1'	33 (1)	37 (1)	30 (1)	0 (1)	-1 (1)	-1 (1)
C2'	26 (4)	28 (4)	18 (4)	3 (3)	1 (3)	-3 (3)
N3'	33 (3)	29 (3)	20 (3)	0 (3)	3 (3)	-1 (3)
C3A'	37 (4)	30 (4)	22 (4)	1 (3)	6 (3)	-10 (3)
C4'	35 (4)	42 (5)	17 (4)	2 (3)	6 (3)	-2 (3)
C5'	50 (5)	41 (5)	26 (4)	1 (4)	16 (4)	-7 (4)
C6'	49 (5)	40 (5)	20 (4)	-8 (4)	-5 (4)	-6 (4)
C7'	41 (5)	36 (5)	37 (5)	0 (4)	-4 (4)	-2 (4)
C7A'	42 (4)	33 (4)	29 (4)	-5 (4)	9 (4)	-9 (4)
S1''	32 (1)	34 (1)	30 (1)	-5 (1)	4 (1)	0 (1)
C2''	34 (4)	19 (4)	32 (4)	5 (3)	13 (3)	2 (3)
N3''	29 (3)	22 (3)	31 (3)	2 (3)	7 (3)	-2 (2)
C3A''	41 (4)	19 (4)	24 (4)	1 (3)	9 (3)	0 (3)
C4''	50 (5)	32 (4)	32 (5)	7 (4)	17 (4)	2 (4)
C5''	71 (6)	35 (5)	28 (5)	-9 (4)	17 (4)	-1 (4)
C6''	64 (6)	38 (5)	17 (4)	-9 (3)	0 (4)	-7 (4)
C7''	34 (4)	42 (5)	38 (5)	-7 (4)	2 (4)	-7 (3)
C7A''	37 (4)	27 (4)	25 (4)	1 (3)	4 (3)	0 (3)
O1A	31 (3)	36 (3)	30 (3)	-8 (2)	6 (2)	-6 (2)
C1A	29 (4)	33 (5)	71 (6)	-26 (4)	20 (4)	-2 (4)
O2A	40 (3)	21 (3)	85 (4)	-2 (3)	28 (3)	4 (2)
C2AA	21 (11)	17 (10)	73 (19)	6 (11)	14 (11)	17 (8)
C2AB	39 (15)	28 (12)	72 (19)	12 (12)	7 (12)	-2 (10)
O1B	30 (3)	33 (3)	25 (3)	-1 (2)	3 (2)	0 (2)
C1B	22 (4)	24 (4)	38 (5)	-3 (4)	3 (3)	2 (3)
O2B	29 (3)	35 (3)	40 (3)	1 (2)	-4 (2)	-1 (2)
C2B	34 (4)	56 (5)	50 (6)	6 (4)	6 (4)	-4 (4)
Br1	54 (1)	104 (2)	54 (1)	-9 (1)	5 (1)	6 (1)
C1C	68 (7)	67 (6)	132 (8)	43 (5)	38 (7)	30 (5)
C2C	50 (6)	97 (8)	97 (7)	36 (6)	28 (6)	33 (6)
C3C	86 (8)	80 (7)	143 (10)	48 (6)	65 (7)	42 (6)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **5**.

	x	y	z	U(eq)
H3	8080 (6)	5295 (3)	5055 (6)	28
H6	3510 (7)	5640 (3)	3168 (6)	34
H4'	6946 (7)	3565 (3)	557 (6)	37
H5'	5859 (7)	3148 (3)	-1228 (6)	45
H6'	3638 (7)	3451 (3)	-2061 (6)	45
H7'	2459 (7)	4151 (3)	-1159 (6)	48
H4''	4825 (7)	7324 (3)	7857 (6)	44
H5''	6065 (8)	7762 (3)	9563 (6)	52
H6''	8404 (8)	7575 (3)	10124 (6)	48
H7''	9556 (7)	6925 (3)	9053 (6)	46
H2A1	9555 (48)	2481 (10)	2461 (75)	55
H2A2	8107 (42)	2137 (20)	2138 (58)	55
H2A3	8890 (81)	2169 (21)	3460 (26)	55
H2A4	9210 (24)	2401 (31)	2324 (68)	70
H2A5	7920 (97)	2586 (21)	1347 (21)	70
H2A6	7723 (84)	2123 (16)	2357 (65)	70
H2B1	10089 (9)	3671 (19)	6925 (17)	70
H2B2	10704 (25)	4271 (4)	6435 (30)	70
H2B3	10923 (19)	3620 (17)	5883 (16)	70
H1C	9324 (10)	4071 (5)	9138 (11)	104
H2C	9250 (9)	4232 (5)	11080 (10)	96
H3C	9938 (10)	5146 (5)	12025 (11)	117