Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Di-μ-phenylmethanethiolato-μ-sulfido-bis[carbonyl(η^5-cyclopentadienyl)-molybdenum(III)]

Richard Chee Seng Wong, Mei Lee Ooi and Seik Weng Ng*

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.010 Å
R factor = 0.066
wR factor = 0.216
Data-to-parameter ratio = 22.2

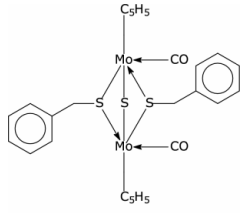
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 21 April 2004
Accepted 23 April 2004
Online 30 April 2004

The reaction of bis[cyclopentadienyltricarbonylmolybdenum(I)] with dibenzyl trisulfide affords the title compound, $[Mo_2S(C_5H_5)_2(C_7H_7S)_2(CO)_2]$, as one of the products. Three independent molecules exist in the asymmetric unit of the triclinic unit cell; one is related to another by a false inversion center and is related to the third by a false translation. In each dinuclear entity, each Mo atom is bonded to a cyclopentadienyl anion and a carbon monoxide molecule; the phenylmethanethiolate groups bridge a pair of Mo atoms, as does the sulfide atom.

Comment

The previous study reported the isolation of several compounds from the reaction of cyclopentadienyltricarbonylmolybdenum(I) with dibenzyl trisulfide (Wong *et al.*, 2004). Also present in the products of the reaction is $[(\eta^5\text{-}C_5H_5)(C_7H_7S)(CO)Mo^{III}]_2S$, (I) (Fig. 1), which differs from $[(\eta^5\text{-}C_5H_5)(C_7H_7S)(CO)_2Mo^{II}]_2$ in having a sulfide linkage in addition to the two phenylmethanethiolate linkages. In the related reaction of $[(\eta^5\text{-}C_5H_5)(CO)_3Cr]_2$ with diphenyl disulfide, one of the products is $[(\eta^5\text{-}C_5H_5)(C_6H_5S)Cr^{III}]_2S$, but the carbonyl ligands are cleaved in the reaction (Goh *et al.*, 1992). In the present structure, if the cyclopentadienyl ring is considered to occupy three sites, the geometry of the Mo atom is pseudo-pentagonal bipyramidal.



(I)

Experimental

The isolation of the title compound is described in the previous report (Wong *et al.*, 2004).

Crystal data

$[Mo_2S(C_5H_5)_2(C_7H_7S)_2(CO)_2]$
M_r = 656.51
Triclinic, $P\overline{1}$
a = 11.325 (1) Å
b = 16.104 (1) Å
c = 22.723 (1) Å
α = 74.15 (1)°
β = 86.10 (1)°
γ = 69.52 (1)°
V = 3732.9 (4) Å^3
Z = 6
D_x = 1.752 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6754 reflections
θ = 2.6–29.2°
μ = 1.28 mm^{-1}
T = 223 (2) K
Block, red
0.38 × 0.30 × 0.28 mm

© 2004 International Union of Crystallography
Printed in Great Britain – all rights reserved

Table 1
Selected geometric parameters (Å, °).

Mo1*a*—C1*a*	2.360 (5)	Mo2*b*—C6*b*	2.360 (5)
Mo1*a*—C2*a*	2.339 (5)	Mo2*b*—C7*b*	2.350 (6)
Mo1*a*—C3*a*	2.297 (5)	Mo2*b*—C8*b*	2.303 (5)
Mo1*a*—C4*a*	2.290 (5)	Mo2*b*—C9*b*	2.284 (5)
Mo1*a*—C5*a*	2.330 (5)	Mo2*b*—C10*b*	2.320 (6)
Mo1*a*—C11*a*	1.952 (8)	Mo2*b*—C12*b*	1.967 (8)
Mo1*a*—S1*a*	2.460 (2)	Mo2*b*—S1*b*	2.453 (2)
Mo1*a*—S2*a*	2.452 (2)	Mo2*b*—S2*b*	2.449 (2)
Mo1*a*—S3*a*	2.437 (2)	Mo2*b*—S3*b*	2.440 (2)
Mo2*a*—C12*a*	1.965 (9)	Mo1*c*—C1*c*	2.362 (5)
Mo2*a*—C6*a*	2.361 (5)	Mo1*c*—C2*c*	2.322 (5)
Mo2*a*—C7*a*	2.340 (5)	Mo1*c*—C3*c*	2.278 (6)
Mo2*a*—C8*a*	2.294 (5)	Mo1*c*—C4*c*	2.292 (5)
Mo2*a*—C9*a*	2.286 (5)	Mo1*c*—C5*c*	2.344 (5)
Mo2*a*—C10*a*	2.329 (5)	Mo1*c*—C11*c*	2.007 (8)
Mo2*a*—S1*a*	2.454 (2)	Mo1*c*—S1*c*	2.463 (2)
Mo2*a*—S2*a*	2.462 (2)	Mo1*c*—S2*c*	2.455 (2)
Mo2*a*—S3*a*	2.443 (2)	Mo1*c*—S3*c*	2.438 (2)
Mo1*b*—C1*b*	2.357 (6)	Mo2*c*—C6*c*	2.356 (6)
Mo1*b*—C2*b*	2.318 (6)	Mo2*c*—C7*c*	2.322 (6)
Mo1*b*—C3*b*	2.291 (6)	Mo2*c*—C8*c*	2.287 (5)
Mo1*b*—C4*b*	2.313 (6)	Mo2*c*—C9*c*	2.300 (6)
Mo1*b*—C5*b*	2.354 (6)	Mo2*c*—C10*c*	2.343 (5)
Mo1*b*—C11*b*	1.975 (8)	Mo2*c*—C12*c*	2.001 (7)
Mo1*b*—S1*b*	2.461 (2)	Mo2*c*—S1*c*	2.457 (2)
Mo1*b*—S2*b*	2.471 (2)	Mo2*c*—S2*c*	2.461 (2)
Mo1*b*—S3*b*	2.439 (2)	Mo2*c*—S3*c*	2.438 (2)

C1*a*—Mo1*a*—C2*a*	35.2 (1)	C7*a*—Mo2*a*—S1*a*	147.5 (1)
C1*a*—Mo1*a*—C3*a*	59.1 (1)	C7*a*—Mo2*a*—S2*a*	93.6 (1)
C1*a*—Mo1*a*—C4*a*	59.2 (1)	C7*a*—Mo2*a*—S3*a*	99.4 (2)
C1*a*—Mo1*a*—C5*a*	35.3 (1)	C8*a*—Mo2*a*—C9*a*	36.1 (1)
C1*a*—Mo1*a*—C11*a*	134.3 (2)	C8*a*—Mo2*a*—C10*a*	59.6 (1)
C1*a*—Mo1*a*—S1*a*	115.9 (2)	C8*a*—Mo2*a*—C12*a*	78.2 (3)
C1*a*—Mo1*a*—S2*a*	112.4 (2)	C8*a*—Mo2*a*—S1*a*	141.8 (2)
C1*a*—Mo1*a*—S3*a*	82.2 (1)	C8*a*—Mo2*a*—S2*a*	105.9 (1)
C2*a*—Mo1*a*—C3*a*	35.7 (1)	C8*a*—Mo2*a*—S3*a*	135.0 (2)
C2*a*—Mo1*a*—C4*a*	59.5 (1)	C9*a*—Mo2*a*—C10*a*	35.8 (2)
C2*a*—Mo1*a*—C5*a*	59.0 (1)	C9*a*—Mo2*a*—C12*a*	76.8 (3)
C2*a*—Mo1*a*—C11*a*	112.4 (3)	C9*a*—Mo2*a*—S1*a*	105.9 (2)
C2*a*—Mo1*a*—S1*a*	94.1 (1)	C9*a*—Mo2*a*—S2*a*	141.3 (2)
C2*a*—Mo1*a*—S2*a*	147.4 (2)	C9*a*—Mo2*a*—S3*a*	136.5 (1)
C2*a*—Mo1*a*—S3*a*	100.3 (1)	C10*a*—Mo2*a*—C12*a*	109.7 (3)
C3*a*—Mo1*a*—C4*a*	36.1 (1)	C10*a*—Mo2*a*—S1*a*	91.8 (1)
C3*a*—Mo1*a*—C5*a*	59.6 (1)	C10*a*—Mo2*a*—S2*a*	150.1 (2)
C3*a*—Mo1*a*—C11*a*	78.6 (2)	C10*a*—Mo2*a*—S3*a*	101.0 (2)
C3*a*—Mo1*a*—S1*a*	106.1 (2)	C12*a*—Mo2*a*—S1*a*	90.1 (2)
C3*a*—Mo1*a*—S2*a*	141.2 (2)	C12*a*—Mo2*a*—S2*a*	90.5 (2)
C3*a*—Mo1*a*—S3*a*	135.9 (1)	C12*a*—Mo2*a*—S3*a*	144.2 (2)
C4*a*—Mo1*a*—C5*a*	35.8 (1)	S1*a*—Mo2*a*—S3*a*	70.59 (7)
C4*a*—Mo1*a*—C11*a*	76.6 (3)	S1*a*—Mo2*a*—S2*a*	110.55 (7)
C4*a*—Mo1*a*—S1*a*	141.3 (2)	S2*a*—Mo2*a*—S3*a*	69.99 (7)
C4*a*—Mo1*a*—S2*a*	105.4 (1)	C1*b*—Mo1*b*—C2*b*	35.4 (1)
C4*a*—Mo1*a*—S3*a*	136.9 (2)	C1*b*—Mo1*b*—C3*b*	59.2 (1)
C5*a*—Mo1*a*—C11*a*	109.3 (3)	C1*b*—Mo1*b*—C4*b*	58.9 (1)
C5*a*—Mo1*a*—S1*a*	150.8 (2)	C1*b*—Mo1*b*—C5*b*	35.1 (1)
C5*a*—Mo1*a*—S2*a*	91.5 (1)	C1*b*—Mo1*b*—C11*b*	135.3 (2)
C5*a*—Mo1*a*—S3*a*	101.3 (2)	C1*b*—Mo1*b*—S1*b*	117.3 (2)
C11*a*—Mo1*a*—S1*a*	90.5 (2)	C1*b*—Mo1*b*—S2*b*	110.8 (2)
C11*a*—Mo1*a*—S2*a*	89.0 (2)	C1*b*—Mo1*b*—S3*b*	80.7 (1)
C11*a*—Mo1*a*—S3*a*	143.5 (2)	C2*b*—Mo1*b*—C3*b*	35.9 (1)
S1*a*—Mo1*a*—S2*a*	110.7 (1)	C2*b*—Mo1*b*—C4*b*	59.5 (1)
S1*a*—Mo1*a*—S3*a*	70.6 (1)	C2*b*—Mo1*b*—C5*b*	58.9 (1)
S2*a*—Mo1*a*—S3*a*	70.2 (1)	C2*b*—Mo1*b*—C11*b*	116.8 (3)
C6*a*—Mo2*a*—C7*a*	35.2 (1)	C2*b*—Mo1*b*—S1*b*	92.8 (1)
C6*a*—Mo2*a*—C8*a*	59.1 (1)	C2*b*—Mo1*b*—S2*b*	146.1 (2)
C6*a*—Mo2*a*—C9*a*	59.2 (1)	C2*b*—Mo1*b*—S3*b*	95.6 (2)
C6*a*—Mo2*a*—C10*a*	35.2 (1)	C3*b*—Mo1*b*—C4*b*	35.9 (1)
C6*a*—Mo2*a*—C12*a*	134.2 (3)	C3*b*—Mo1*b*—C5*b*	59.3 (1)
C6*a*—Mo2*a*—S1*a*	112.5 (2)	C3*b*—Mo1*b*—C11*b*	82.0 (3)
C6*a*—Mo2*a*—S2*a*	115.2 (2)	C3*b*—Mo1*b*—S1*b*	102.5 (2)
C6*a*—Mo2*a*—S3*a*	81.5 (1)	C3*b*—Mo1*b*—S2*b*	146.2 (2)
C7*a*—Mo2*a*—C8*a*	35.7 (1)	C3*b*—Mo1*b*—S3*b*	131.5 (2)
C7*a*—Mo2*a*—C9*a*	59.5 (1)	C4*b*—Mo1*b*—C5*b*	35.4 (1)
C7*a*—Mo2*a*—C10*a*	59.0 (1)	C4*b*—Mo1*b*—C11*b*	76.7 (2)
C7*a*—Mo2*a*—C12*a*	111.9 (3)	C4*b*—Mo1*b*—S1*b*	137.2 (2)
C4*b*—Mo1*b*—S2*b*	110.3 (2)	C3*c*—Mo1*c*—S2*c*	142.3 (2)
C4*b*—Mo1*b*—S3*b*	137.5 (2)	C2*c*—Mo1*c*—S3*c*	98.3 (2)
C5*b*—Mo1*b*—C11*b*	107.4 (3)	C3*c*—Mo1*c*—C4*c*	36.2 (1)
C5*b*—Mo1*b*—S1*b*	151.0 (2)	C3*c*—Mo1*c*—C5*c*	59.6 (1)
C5*b*—Mo1*b*—S2*b*	93.5 (1)	C3*c*—Mo1*c*—C11*c*	78.9 (3)
C5*b*—Mo1*b*—S3*b*	103.0 (2)	C3*c*—Mo1*c*—S1*c*	105.2 (2)
C11*b*—Mo1*b*—S1*b*	90.5 (2)	C3*c*—Mo1*c*—S2*c*	142.3 (2)
C11*b*—Mo1*b*—S2*b*	88.5 (2)	C3*c*—Mo1*c*—S3*c*	134.2 (2)
C11*b*—Mo1*b*—S3*b*	143.7 (2)	C4*c*—Mo1*c*—C5*c*	35.65 (7)
S1*b*—Mo1*b*—S2*b*	110.0 (1)	C4*c*—Mo1*c*—C11*c*	77.0 (2)
S1*b*—Mo1*b*—S3*b*	71.1 (1)	C4*c*—Mo1*c*—S1*c*	140.7 (2)
S2*b*—Mo1*b*—S3*b*	70.1 (1)	C4*c*—Mo1*c*—S2*c*	106.3 (2)
C6*b*—Mo2*b*—C7*b*	35.1 (1)	C4*c*—Mo1*c*—S3*c*	136.4 (2)
C6*b*—Mo2*b*—C8*b*	59.0 (1)	C5*c*—Mo1*c*—C11*c*	109.4 (3)
C6*b*—Mo2*b*—C9*b*	59.3 (1)	C5*c*—Mo1*c*—S1*c*	150.4 (2)
C6*b*—Mo2*b*—C10*b*	35.3 (1)	C5*c*—Mo1*c*—S2*c*	91.7 (1)
C6*b*—Mo2*b*—C12*b*	134.0 (2)	C5*c*—Mo1*c*—S3*c*	101.1 (2)
C6*b*—Mo2*b*—S1*b*	112.2 (2)	C11*c*—Mo1*c*—S1*c*	90.2 (2)
C6*b*—Mo2*b*—S2*b*	115.5 (2)	C11*c*—Mo1*c*—S2*c*	89.9 (2)
C6*b*—Mo2*b*—S3*b*	81.0 (1)	C11*c*—Mo1*c*—S3*c*	144.3 (2)
C7*b*—Mo2*b*—C8*b*	35.5 (1)	S1*c*—Mo1*c*—S3*c*	70.4 (1)
C7*b*—Mo2*b*—C9*b*	59.4 (1)	S1*c*—Mo1*c*—S2*c*	110.7 (1)
C7*b*—Mo2*b*—C10*b*	58.9 (1)	S2*c*—Mo1*c*—S3*c*	70.8 (1)
C7*b*—Mo2*b*—C12*b*	108.8 (3)	C6*c*—Mo2*c*—C7*c*	35.3 (1)
C7*b*—Mo2*b*—S1*b*	92.4 (1)	C6*c*—Mo2*c*—C8*c*	59.3 (1)
C7*b*—Mo2*b*—S2*b*	149.9 (2)	C6*c*—Mo2*c*—C9*c*	59.1 (1)
C7*b*—Mo2*b*—S3*b*	101.5 (2)	C6*c*—Mo2*c*—C10*c*	35.2 (1)
C8*b*—Mo2*b*—C9*b*	36.1 (1)	C6*c*—Mo2*c*—C12*c*	135.1 (2)
C8*b*—Mo2*b*—C10*b*	59.6 (1)	C6*c*—Mo2*c*—S1*c*	111.7 (2)
C8*b*—Mo2*b*—C12*b*	76.4 (2)	C6*c*—Mo2*c*—S2*c*	115.7 (2)
C8*b*—Mo2*b*—S1*b*	107.0 (1)	C6*c*—Mo2*c*—S3*c*	80.9 (1)
C8*b*—Mo2*b*—S2*b*	139.6 (2)	C7*c*—Mo2*c*—C8*c*	35.9 (1)
C8*b*—Mo2*b*—S3*b*	136.6 (2)	C7*c*—Mo2*c*—C9*c*	59.6 (1)
C9*b*—Mo2*b*—C10*b*	35.9 (1)	C7*c*—Mo2*c*—C10*c*	59.0 (1)
C9*b*—Mo2*b*—C12*b*	78.3 (3)	C7*c*—Mo2*c*—C12*c*	113.8 (3)
C9*b*—Mo2*b*—S1*b*	142.9 (2)	C7*c*—Mo2*c*—S1*c*	147.0 (2)
C9*b*—Mo2*b*—S2*b*	104.3 (1)	C7*c*—Mo2*c*—S2*c*	93.0 (1)
C9*b*—Mo2*b*—S3*b*	133.7 (2)	C7*c*—Mo2*c*—S3*c*	98.0 (2)
C10*b*—Mo2*b*—C12*b*	112.4 (3)	C8*c*—Mo2*c*—C9*c*	36.1 (1)
C10*b*—Mo2*b*—S1*b*	147.4 (2)	C8*c*—Mo2*c*—C10*c*	59.5 (1)
C10*b*—Mo2*b*—S2*b*	92.7 (1)	C8*c*—Mo2*c*—C12*c*	79.7 (2)
C10*b*—Mo2*b*—S3*b*	97.8 (2)	C8*c*—Mo2*c*—S1*c*	143.0 (2)
C12*b*—Mo2*b*—S1*b*	90.3 (2)	C8*c*—Mo2*c*—S2*c*	104.7 (2)
C12*b*—Mo2*b*—S2*b*	90.2 (2)	C8*c*—Mo2*c*—S3*c*	133.8 (2)
C12*b*—Mo2*b*—S3*b*	145.1 (2)	C9*c*—Mo2*c*—C10*c*	35.6 (1)
S1*b*—Mo2*b*—S2*b*	111.02 (7)	C9*c*—Mo2*c*—C12*c*	77.3 (2)
S1*b*—Mo2*b*—S3*b*	71.23 (7)	C9*c*—Mo2*c*—S1*c*	107.1 (2)
S2*b*—Mo2*b*—S3*b*	70.49 (6)	C9*c*—Mo2*c*—S2*c*	140.0 (2)
C1*c*—Mo1*c*—C2*c*	35.3 (1)	C9*c*—Mo2*c*—S3*c*	136.5 (2)
C1*c*—Mo1*c*—C3*c*	59.3 (1)	C10*c*—Mo2*c*—C12*c*	109.4 (3)
C1*c*—Mo1*c*—C4*c*	59.1 (1)	C10*c*—Mo2*c*—S1*c*	92.1 (1)
C1*c*—Mo1*c*—C5*c*	35.1 (1)	C10*c*—Mo2*c*—S2*c*	150.3 (2)
C1*c*—Mo1*c*—C11*c*	134.6 (2)	C10*c*—Mo2*c*—S3*c*	101.3 (2)
C1*c*—Mo1*c*—S1*c*	115.8 (2)	C12*c*—Mo2*c*—S1*c*	89.5 (2)
C1*c*—Mo1*c*—S2*c*	111.8 (2)	C12*c*—Mo2*c*—S2*c*	90.2 (2)
C1*c*—Mo1*c*—S3*c*	81.0 ;(1)	C12*c*—Mo2*c*—S3*c*	143.9 (2)
C2*c*—Mo1*c*—C3*c*	36.0 (1)	S1*c*—Mo2*c*—S2*c*	110.7 (1)
C2*c*—Mo1*c*—C4*c*	59.7 (1)	S1*c*—Mo2*c*—S3*c*	70.5 (1)
C2*c*—Mo1*c*—C5*c*	59.0 (1)	S2*c*—Mo2*c*—S3*c*	70.7 (1)
C2*c*—Mo1*c*—C11*c*	113.0 (3)		
C2*c*—Mo1*c*—S1*c*	93.4 (1)		
C2*c*—Mo1*c*—S2*c*	147.0 (2)		

Data collection

Bruker SMART CCD area-detector diffractometer	17028 independent reflections
φ and ω scans	11206 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	$R_{int} = 0.041$
$T_{min} = 0.680$, $T_{max} = 0.715$	$\theta_{max} = 27.5^\circ$
36947 measured reflections	$h = -14 \rightarrow 14$
	$k = -20 \rightarrow 19$
	$l = -29 \rightarrow 29$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0898P)^2 + 20.1592P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.066$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.216$	$\Delta\rho_{max} = 2.42$ e Å^{-3}
$S = 1.07$	$\Delta\rho_{min} = -1.69$ e Å^{-3}
17028 reflections	
766 parameters	
H-atom parameters constrained	

Molecule *a* is related to molecule *b* by a false inversion at approximately (2/3, 2/3, 2/3); it is also related to molecule *c* by an approximate translation of (1/3, 1/3, 1/3). The false symmetry/translational symmetry necessitated constraining the five-membered rings to be refined as regular pentagons (C—C = 1.42 Å) and the six-membered rings as regular hexagons (C—C = 1.39 Å). H atoms were placed at calculated positions in the riding-model approximation [aromatic C—H = 0.94 Å and aliphatic C—H = 0.98 Å; U_{iso}(H) = 1.2U_{eq}(C)]. The final difference Fourier had a large peak at about 1.5 Å from atom Mo1*a* and the deepest hole at about 1.5 Å from atom C11*a*.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

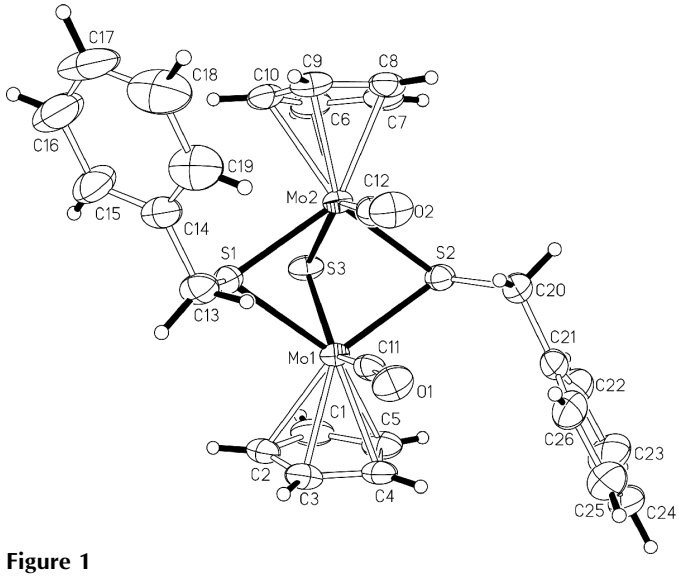


Figure 1
*ORTEP*II (Johnson, 1976) plot of one of the three independent molecules of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

We thank the National University of Singapore for the diffraction measurements, and the University of Malaya (PJP FP005/2003A) for generously supporting this work.

References

Bruker (2001). *SADABS, SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Goh, L. Y., Tay, M. S., Mak, T. C. W. & Wang, R.-J. (1992). *Organometallics*, **11**, 1711–1717.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Wong, R. C. S., Ooi, M. L., Lo, K. M. & Ng, S. W. (2004). *Acta Cryst.* E**60**, m687–m689.