Crystal structure of methylpyridylbenzophenoneazine tetracarbonylmolybdenum(0), $C_{24}H_{17}MoN_3O_4$

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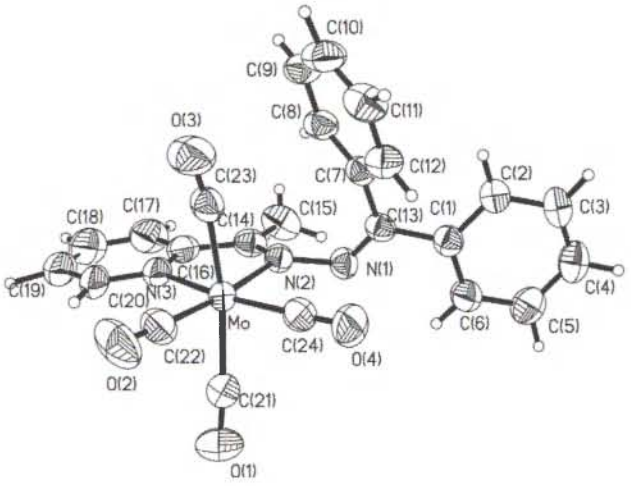
Received March 14, 1999, CCDC-No. 1267/144



Abstract

$C_{24}H_{17}MoN_3O_4$, orthorhombic, $P2_12_12_1$ (No. 19), $a = 8.7185(1)$ Å, $b = 11.4066(1)$ Å, $c = 22.9699(3)$ Å, $V = 2284.3$ Å^3, $Z = 4$, $R_{gt}(F) = 0.027$, $wR(F^2) = 0.051$, $T = 293$ K.

Source of material

2-Acetylpyridine benzophenone azine (apba) was prepared by refluxing a mixture of 2-acetylpyridine (1.21 g, 0.01 mol) and benzophenone hydrazone (1.96 g, 0.01 mol) in dry ethanol for 6 hours. The yellow reaction mixture was then concentrated and cooled to 278 K overnight. The yellow solid formed was filtered off, washed by petroleum ether, recrystallized from hot ethanol and dried in vacuo. $Mo(CO)_6$ (0.264 g, 0.001 mol) and apba (0.2994 g, 0.001 mol) were mixed together in dry toluene (30 ml) under argon in a Schlenk flask fitted with a condenser. The reaction mixture was slowly heated until all the reactants had dissolved to give a yellow solution. This was then refluxed for 6 hours after which the resultant red-brown solution was allowed to cool to room temperature and evaporated to low volume. The dark brown microcrystalline solid formed after evaporation was washed with n-pentane and recrystallized from n-hexane and dichloromethane mixture [1, 2].

Table 1. Data collection and handling.

Crystal:	dark brown slab, size 0.12 × 0.26 × 0.52 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.09 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15172, 5238
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4661
$N(param)_{refined}$:	357
Programs:	SHELXTL/PC [3], SHELXL-93 [4], PARST [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	4a	0.072(3)	1.167(2)	0.9794(9)	0.038(6)
H(3)	4a	0.093(4)	1.349(3)	1.022(1)	0.08(1)
H(4)	4a	0.264(4)	1.498(3)	0.986(1)	0.071(9)
H(5)	4a	0.426(5)	1.441(3)	0.907(1)	0.09(1)
H(6)	4a	0.388(4)	1.261(3)	0.862(1)	0.08(1)
H(8)	4a	-0.034(3)	0.962(2)	0.846(1)	0.055(8)
H(9)	4a	-0.174(4)	0.821(3)	0.895(1)	0.09(1)
H(10)	4a	-0.110(4)	0.744(3)	0.985(1)	0.070(9)
H(11)	4a	0.105(4)	0.840(3)	1.033(1)	0.064(8)
H(12)	4a	0.246(3)	0.984(2)	0.984(1)	0.036(7)
H(15A)	4a	-0.008(7)	1.057(5)	0.741(2)	0.15(2)
H(15B)	4a	0.077(5)	1.089(3)	0.695(2)	0.11(1)
H(15C)	4a	0.113(4)	1.139(3)	0.758(2)	0.08(1)
H(17)	4a	0.071(4)	0.918(3)	0.651(1)	0.057(9)
H(18)	4a	0.116(4)	0.746(3)	0.591(2)	0.08(1)
H(19)	4a	0.314(5)	0.599(4)	0.624(2)	0.12(1)
H(20)	4a	0.418(4)	0.639(3)	0.717(1)	0.08(1)

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo	4*a*	0.42009(2)	0.82330(2)	0.820501(8)	0.03966(9)	0.03594(9)	0.04139(9)	0.00006(9)	0.0018(1)	–0.00527(9)
O(1)	4*a*	0.7252(3)	0.9119(2)	0.7604(1)	0.071(2)	0.109(2)	0.120(2)	–0.031(2)	0.036(2)	–0.009(2)
O(2)	4*a*	0.6114(3)	0.5950(2)	0.8376(1)	0.092(2)	0.069(2)	0.115(2)	0.040(1)	0.029(2)	0.012(1)
O(3)	4*a*	0.1836(3)	0.6576(2)	0.8868(1)	0.082(2)	0.060(2)	0.119(2)	0.000(1)	0.033(2)	0.021(1)
O(4)	4*a*	0.5412(2)	0.9266(2)	0.93753(9)	0.065(2)	0.081(2)	0.060(1)	–0.002(1)	–0.014(1)	–0.019(1)
N(1)	4*a*	0.2640(3)	1.0783(2)	0.83248(8)	0.051(1)	0.037(1)	0.045(1)	0.0012(9)	0.004(1)	–0.0017(8)
N(2)	4*a*	0.2604(2)	0.9728(2)	0.80084(8)	0.044(1)	0.039(1)	0.038(1)	–0.0028(9)	0.0053(9)	–0.0003(8)
N(3)	4*a*	0.3058(3)	0.7897(2)	0.73517(9)	0.049(1)	0.050(1)	0.042(1)	–0.010(1)	0.005(1)	–0.0090(9)
C(1)	4*a*	0.2308(3)	1.1952(2)	0.9157(1)	0.041(1)	0.040(1)	0.048(1)	0.007(1)	–0.002(1)	–0.001(1)
C(2)	4*a*	0.1442(3)	1.2232(2)	0.9648(1)	0.053(2)	0.049(2)	0.046(1)	0.001(1)	0.000(1)	–0.004(1)
C(3)	4*a*	0.1562(4)	1.3337(3)	0.9903(1)	0.075(2)	0.059(2)	0.051(2)	0.005(2)	0.001(1)	–0.016(2)
C(4)	4*a*	0.2552(4)	1.4151(3)	0.9690(2)	0.085(2)	0.045(2)	0.071(2)	0.002(2)	–0.005(2)	–0.016(2)
C(5)	4*a*	0.3441(5)	1.3887(3)	0.9210(2)	0.077(2)	0.048(2)	0.089(2)	–0.012(2)	0.008(2)	–0.005(2)
C(6)	4*a*	0.3316(4)	1.2792(3)	0.8941(2)	0.059(2)	0.046(2)	0.070(2)	–0.005(1)	0.016(2)	–0.007(1)
C(7)	4*a*	0.1217(3)	0.9854(2)	0.9135(1)	0.038(1)	0.038(1)	0.045(1)	0.004(1)	0.006(1)	–0.001(1)
C(8)	4*a*	–0.0050(3)	0.9349(2)	0.8866(1)	0.039(2)	0.049(2)	0.058(2)	0.005(1)	0.005(1)	0.003(1)
C(9)	4*a*	–0.0890(4)	0.8483(3)	0.9143(1)	0.041(2)	0.059(2)	0.081(2)	–0.003(2)	0.009(2)	–0.003(1)
C(10)	4*a*	–0.0462(3)	0.8107(3)	0.9695(2)	0.050(2)	0.055(2)	0.090(2)	–0.002(1)	0.018(2)	0.015(2)
C(11)	4*a*	0.0778(5)	0.8597(3)	0.9964(1)	0.067(2)	0.061(2)	0.061(2)	0.010(2)	0.013(2)	0.021(1)
C(12)	4*a*	0.1631(3)	0.9474(3)	0.9693(1)	0.046(2)	0.053(2)	0.054(2)	0.002(1)	0.000(1)	0.007(1)
C(13)	4*a*	0.2091(3)	1.0817(2)	0.8843(1)	0.036(1)	0.038(1)	0.044(1)	0.006(1)	0.002(1)	0.003(1)
C(14)	4*a*	0.1816(3)	0.9739(2)	0.7530(1)	0.042(1)	0.056(2)	0.041(1)	–0.002(1)	0.001(1)	0.005(1)
C(15)	4*a*	0.0776(6)	1.0719(4)	0.7358(2)	0.061(2)	0.082(2)	0.059(2)	0.016(2)	–0.001(2)	0.013(2)
C(16)	4*a*	0.2039(3)	0.8692(3)	0.7157(1)	0.043(2)	0.067(2)	0.042(1)	–0.014(1)	0.003(1)	–0.006(1)
C(17)	4*a*	0.1303(4)	0.8553(4)	0.6625(1)	0.055(2)	0.100(3)	0.058(2)	–0.004(2)	–0.011(1)	–0.008(2)
C(18)	4*a*	0.1663(5)	0.7565(4)	0.6289(2)	0.073(2)	0.124(3)	0.056(2)	–0.024(2)	–0.006(2)	–0.035(2)
C(19)	4*a*	0.2690(4)	0.6769(4)	0.6487(2)	0.071(2)	0.087(2)	0.069(2)	–0.023(2)	0.011(2)	–0.031(2)
C(20)	4*a*	0.3369(4)	0.6946(3)	0.7020(1)	0.066(2)	0.058(2)	0.055(2)	–0.016(2)	0.011(1)	–0.018(1)
C(21)	4*a*	0.6092(4)	0.8875(3)	0.7806(1)	0.056(2)	0.057(2)	0.062(2)	–0.007(1)	0.005(1)	–0.010(1)
C(22)	4*a*	0.5420(3)	0.6815(3)	0.8305(1)	0.055(2)	0.053(2)	0.063(2)	0.006(1)	0.012(1)	–0.001(2)
C(23)	4*a*	0.2583(4)	0.7251(2)	0.8637(1)	0.054(2)	0.043(2)	0.059(2)	0.007(1)	0.008(1)	0.000(1)
C(24)	4*a*	0.4983(3)	0.8850(2)	0.8944(1)	0.044(2)	0.046(2)	0.053(2)	0.005(1)	0.000(1)	–0.001(1)

Acknowledgments. The authors would like to thank the Malaysian Government, Universiti Teknologi Malaysia for grants UPP/UTM vot No:26-67100, Universiti Kebangsaan Malaysia and Universiti Sains Malaysia for providing the facilities for the collaborative research project.

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