

UP

Empirical formula	$C_{24}H_{28}Mo_3N_4O_{11} \cdot C_{24}H_{27}Mo_3N_4O_{11} \cdot 2(C_2H_6OS)$ (1)	$C_{52}H_{52}Cu_4Mo_8N_8O_{34}S_2 \cdot 4(C_2H_6OS)$ (2)
Formula weight	1827.89	2743.42
$T$ (K)	100(2) K	100(2) K
Radiation, $\lambda$	0.71073 Å	0.71073 Å
Crystal system, space group	Monoclinic	Monoclinic
Space Group	P2(1)/n	C2/c
Unit cell dimensions	$a = 17.4435$ (6) Å $b = 15.6194$ (5) Å $c = 24.9582$ (9) Å	$a = 28.767$ (11) Å $b = 14.296$ (5) Å $c = 22.177$ (8) Å
	$\alpha = 90^\circ$ $\beta = 100.844$ (3) $^\circ$ $\gamma = 90^\circ$	$\alpha = 90^\circ$ $\beta = 109.920$ (14) $^\circ$ $\gamma = 90^\circ$
Volume	6678.6 (4) Å <sup>3</sup>	8575 (5) Å <sup>3</sup>
Z, Calculated Density	4, 1.818 Mg m <sup>-3</sup>	2.125 Mg m <sup>-3</sup>
Absorption coefficient	1.24 mm <sup>-1</sup>	2.34 mm <sup>-1</sup>
$F(000)$	3644	5424
Crystal size (mm)	0.23 × 0.15 × 0.1	0.17 × 0.15 × 0.13 mm <sup>3</sup>
Theta range / $^\circ$	2.9 to 26.9 $^\circ$	2.8 to 26.4
Index ranges	-21 ≤ $h$ ≤ 21 -18 ≤ $k$ ≤ 18 -30 ≤ $l$ ≤ 30	-35 ≤ $h$ ≤ 35 -17 ≤ $k$ ≤ 17 -27 ≤ $l$ ≤ 27
Reflections collected	58140	165405
Independent reflections	12210 [ $R_{int} = 0.138$ ]	8783 [ $R_{int} = 0.042$ ]
Completeness to theta max /	99.5 %	99.5
Refinement method	Full-matrix least-squares on $F^2$	Full-matrix least-squares on $F^2$
Data / restraints / parameters	12210 / 49 / 837	8783 / 650 / 564
Goodness-of-fit on $F^2$	1.03	1.06
Final $R$ indices [ $F^2 > 2\sigma(F^2)$ ]	$R_1 = 0.0897$ $wR_2 = 0.2309$	$R_1 = 0.049$ $wR_2 = 0.137$
$R$ indices (all data)	$R_1 = 0.1554$ $wR_2 = 0.2863$	$R_1 = 0.0544$ $wR_2 = 0.1427$
Largest diff. peak and hole	6.73 and -1.02 e.Å <sup>-3</sup>	2.93, -3.22

## Supplementary data

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C CDC 1050597 and 1051415 contain the supplementary crystallographic data for this

paper. These data can be obtained free of charge at

[www.ccdc.cam.ac.uk/conts/retrieving.html](http://www.ccdc.cam.ac.uk/conts/retrieving.html) [or from the Cambridge Crystallographic

Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223- 336033; email: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk)].



