Crystal structure of $(Bu_4N)_2[Mo_6O_{18}N(o\text{-}CH_3C_6H_3OOCCH_2CH_3)]$, $C_{42}H_{83}Mo_6N_3O_{20}$

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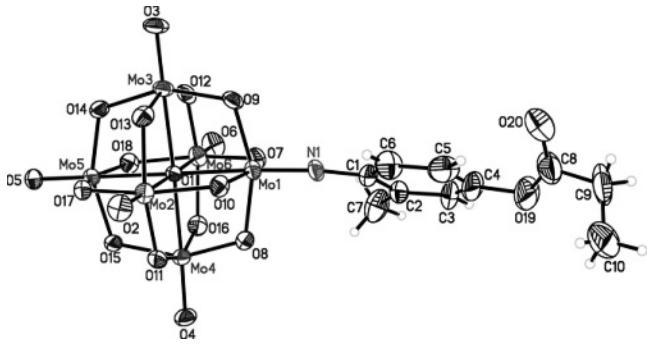

Abstract

$C_{42}H_{83}Mo_6N_3O_{20}$, triclinic, $P\overline{1}$ (no. 2), a = 12.683(3) Å, b = 12.878(4) Å, c = 19.433(6) Å, α = 74.27(2)°, β = 72.36(2)°, γ = 77.34(2)°, V = 2878.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.0664, $wR_{ref}(F^2)$ = 0.2268, T = 296 K.

Table 1. Data collection and handling.

Crystal:	yellow needles, size 0.03×0.40×0.53 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	13.37 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ and ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	18718, 9678
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5018
$N(param)_{refined}$:	614
Programs:	SHELX [4]

Source of material

Reference to literature [1], a mixture containing organoimido derivative (1.0mmol), a carboxylic acid (1.5 mmol) in anhydrous acetonitrile (15 mL) was heated ahead for 15 min, then 1,3-dicyclohexylcarbodiimide (DCC, 2.0 mmol in 5 mL anhydrous acetonitrile) was added. The reaction mixture was refluxed at a temperature of 85 °C for 7h. After being cooled to room temperature, the resulting red-brown solution was filtered to remove some yellow-green precipitates. The red-brown filtrate was poured into 40 mL ice-water mixture with stirring continuously. Then the resulting brown solid product was collected by filtration. After being dried, the crude product was dissolved in 15 mL acetonitrile for recrystallization, and the product was obtained by slow diffusion of the layered on Et_2O into an acetonitrile solution of the crude product.

Discussion

The title compound $(Bu_4N)_2[Mo_6O_{18}N(o\text{-}CH_3C_6H_3OOCCH_2CH_3)]$ is an esterified product of Lindqvist organimido derivative $(Bu_4N)_2[Mo_6O_{18}N(o\text{-}CH_3C_6H_3OH)]$. There are some typical features of the reported organoimido derivatives in the crystal structure of the title compound [2,3]. For example, the bond length of Mo–N is 1.734(8) Å showing special triple bond character; and the bond angle of Mo–N–C = 173.3(9)° is almost in a straight line. While the bond C(4)–O(19) = 1.41(2) Å is much longer than phemolic hydroxyl of raw material C(4)–O(19) = 1.353(5) Å [3], as the result of acylation.

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

Atom	Site	x	y	z	U_{iso}
H(11A)	2*i*	0.9428	0.4347	–0.2285	0.093
H(11B)	2*i*	1.0645	0.4537	–0.2376	0.093
H(12A)	2*i*	1.0259	0.2751	–0.2801	0.141
H(12B)	2*i*	1.1415	0.3176	–0.3012	0.141
H(13A)	2*i*	1.0199	0.3924	–0.4012	0.298
H(13B)	2*i*	0.9616	0.4800	–0.3522	0.298
H(14A)	2*i*	1.1099	0.5281	–0.4343	0.417
H(14B)	2*i*	1.1793	0.4198	–0.4010	0.417
H(14C)	2*i*	1.1211	0.5073	–0.3534	0.417
H(15A)	2*i*	0.9932	0.3569	–0.0499	0.076
H(15B)	2*i*	1.0345	0.4533	–0.1148	0.076
H(16A)	2*i*	0.8157	0.4089	–0.0657	0.086
H(16B)	2*i*	0.8569	0.5051	–0.1316	0.086
H(17A)	2*i*	0.8594	0.4869	0.0171	0.090
H(17B)	2*i*	0.9061	0.5811	–0.0484	0.090
H(18A)	2*i*	0.7254	0.6434	0.0158	0.156
H(18B)	2*i*	0.6791	0.5514	–0.0001	0.156
H(18C)	2*i*	0.7258	0.6457	–0.0653	0.156
H(19A)	2*i*	0.8608	0.2902	–0.1404	0.083
H(19B)	2*i*	0.9569	0.2005	–0.1686	0.083
H(20A)	2*i*	0.8959	0.2200	–0.0213	0.117
H(20B)	2*i*	0.9747	0.1219	–0.0526	0.117
H(21A)	2*i*	0.8087	0.0987	–0.0856	0.140
H(21B)	2*i*	0.8155	0.0527	–0.0034	0.140
H(22A)	2*i*	0.6461	0.1463	–0.0079	0.310
H(22B)	2*i*	0.6999	0.2506	–0.0544	0.310
H(22C)	2*i*	0.7062	0.2039	0.0278	0.310
H(23A)	2*i*	1.1475	0.2064	–0.1792	0.084
H(23B)	2*i*	1.1242	0.2163	–0.0973	0.084
H(24A)	2*i*	1.2422	0.3574	–0.2227	0.110
H(24B)	2*i*	1.2096	0.3797	–0.1426	0.110
H(25A)	2*i*	1.3463	0.1882	–0.1814	0.136
H(25B)	2*i*	1.3157	0.2133	–0.1023	0.136
H(26A)	2*i*	1.4942	0.2556	–0.1675	0.234
H(26B)	2*i*	1.4143	0.3631	–0.1521	0.234
H(26C)	2*i*	1.4458	0.3368	–0.2307	0.234
H(27A)	2*i*	1.3147	0.0061	0.2701	0.134
H(27B)	2*i*	1.3059	0.1268	0.2245	0.134
H(28A)	2*i*	1.4994	–0.0265	0.2021	0.211
H(28B)	2*i*	1.4871	0.0936	0.1550	0.211
H(29A)	2*i*	1.4539	–0.0583	0.1098	0.249
H(29B)	2*i*	1.3317	–0.0321	0.1597	0.249
H(30A)	2*i*	1.3510	0.0738	0.0458	0.357
H(30B)	2*i*	1.4507	0.1180	0.0547	0.357
H(30C)	2*i*	1.3296	0.1443	0.1044	0.357

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Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(31A)	2*i*	1.2529	0.0640	0.3800	0.140
H(31B)	2*i*	1.2441	0.1873	0.3396	0.140
H(32A)	2*i*	1.3605	0.2139	0.4066	0.155
H(32B)	2*i*	1.3609	0.0917	0.4496	0.155
H(33A)	2*i*	1.1477	0.1407	0.4954	0.296
H(33B)	2*i*	1.2176	0.1837	0.5333	0.296
H(35A)	2*i*	1.5036	0.0414	0.3645	0.132
H(35B)	2*i*	1.5435	0.0003	0.2908	0.132
H(36A)	2*i*	1.4203	–0.1259	0.3392	0.169
H(36B)	2*i*	1.3739	–0.0822	0.4123	0.169
H(37A)	2*i*	1.5834	–0.1293	0.4068	0.280
H(37B)	2*i*	1.4950	–0.2008	0.4578	0.280
H(38A)	2*i*	1.6336	–0.2812	0.4310	0.709
H(38B)	2*i*	1.6454	–0.2335	0.3462	0.709
H(38C)	2*i*	1.5520	–0.3055	0.3927	0.709
H(39A)	2*i*	1.5202	0.1815	0.2267	0.150
H(39B)	2*i*	1.4817	0.2157	0.3027	0.150
H(40A)	2*i*	1.3583	0.2946	0.1934	0.172
H(40B)	2*i*	1.3228	0.3308	0.2691	0.172

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(41A)	2*i*	1.5194	0.3735	0.1588	0.299
H(41B)	2*i*	1.4848	0.4089	0.2348	0.299
H(42A)	2*i*	1.4348	0.5547	0.1441	0.354
H(42B)	2*i*	1.3265	0.5203	0.2040	0.354
H(42C)	2*i*	1.3615	0.4852	0.1280	0.354
H(34A)	2*i*	1.0872	0.3210	0.4989	0.680
H(34B)	2*i*	1.1288	0.3050	0.4173	0.680
H(34C)	2*i*	1.2052	0.3485	0.4499	0.680
H(7A)	2*i*	–0.8957	1.8487	0.5179	0.208
H(7B)	2*i*	–0.9830	1.8704	0.4713	0.208
H(7C)	2*i*	–0.8854	1.9403	0.4452	0.208
H(3)	2*i*	–0.7661	1.6902	0.5039	0.157
H(5)	2*i*	–0.6448	1.5957	0.3153	0.220
H(6)	2*i*	–0.7596	1.7432	0.2599	0.143
H(9A)	2*i*	–0.4701	1.3528	0.4586	0.281
H(9B)	2*i*	–0.4566	1.3488	0.3766	0.281
H(10A)	2*i*	–0.3077	1.4029	0.3955	0.351
H(10B)	2*i*	–0.3864	1.5102	0.4124	0.351
H(10C)	2*i*	–0.3583	1.4851	0.3337	0.351

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(1)	2*i*	–0.98855(8)	2.00106(7)	0.28207(5)	0.0640(6)	0.0482(5)	0.0692(6)	–0.0024(4)	–0.0231(5)	–0.0040(4)
Mo(2)	2*i*	–0.96592(8)	2.16642(8)	0.12076(5)	0.0560(6)	0.0662(6)	0.0571(5)	–0.0056(5)	–0.0111(4)	–0.0107(4)
Mo(3)	2*i*	–1.19366(8)	2.05253(8)	0.20519(6)	0.0598(6)	0.0510(6)	0.1034(8)	–0.0115(5)	–0.0278(6)	–0.0240(5)
Mo(4)	2*i*	–0.99275(8)	2.25961(7)	0.26759(5)	0.0644(6)	0.0535(6)	0.0795(7)	–0.0102(5)	–0.0312(5)	–0.0160(5)
Mo(5)	2*i*	–1.20131(8)	2.31512(7)	0.18878(5)	0.0538(6)	0.0454(5)	0.0736(6)	–0.0052(4)	–0.0231(5)	–0.0078(4)
Mo(6)	2*i*	–1.22068(9)	2.14557(8)	0.35215(5)	0.0658(7)	0.0678(7)	0.0626(6)	–0.0084(5)	–0.0036(5)	–0.0086(5)
O(1)	2*i*	–1.0915(5)	2.1533(5)	0.2372(3)	0.045(3)	0.044(3)	0.054(4)	–0.009(3)	–0.013(3)	–0.009(3)
O(2)	2*i*	–0.8786(7)	2.1770(7)	0.0368(4)	0.073(5)	0.101(6)	0.067(5)	–0.004(5)	0.000(4)	–0.025(4)
O(3)	2*i*	–1.2715(7)	1.9801(6)	0.1850(5)	0.076(5)	0.060(5)	0.173(8)	–0.010(4)	–0.051(6)	–0.048(5)
O(4)	2*i*	–0.9188(7)	2.3390(6)	0.2861(5)	0.089(6)	0.069(5)	0.131(7)	–0.010(5)	–0.056(5)	–0.036(5)
O(5)	2*i*	–1.2795(6)	2.4305(6)	0.1533(4)	0.073(5)	0.053(4)	0.098(6)	0.004(4)	–0.038(4)	–0.009(4)
O(6)	2*i*	–1.3131(8)	2.1414(8)	0.4345(5)	0.098(7)	0.109(7)	0.081(6)	–0.008(6)	0.011(5)	–0.018(5)
O(7)	2*i*	–1.1108(6)	2.0249(6)	0.3699(4)	0.074(5)	0.068(5)	0.067(4)	–0.011(4)	–0.009(4)	–0.003(4)
O(8)	2*i*	–0.9241(6)	2.1152(6)	0.2989(4)	0.069(5)	0.062(5)	0.079(5)	–0.007(4)	–0.033(4)	–0.013(4)
O(9)	2*i*	–1.0903(6)	1.9422(5)	0.2511(4)	0.071(5)	0.042(4)	0.106(6)	–0.011(4)	–0.024(4)	–0.018(4)
O(10)	2*i*	–0.9033(6)	2.0355(6)	0.1807(4)	0.055(4)	0.060(4)	0.074(5)	0.009(3)	–0.017(4)	–0.021(3)
O(11)	2*i*	–0.9096(6)	2.2484(6)	0.1673(4)	0.059(4)	0.062(5)	0.080(5)	–0.022(4)	–0.019(4)	0.002(4)
O(12)	2*i*	–1.2773(6)	2.0648(6)	0.3056(4)	0.052(4)	0.057(5)	0.102(6)	–0.017(4)	–0.009(4)	0.003(4)
O(13)	2*i*	–1.0736(7)	2.0782(6)	0.1195(4)	0.079(5)	0.069(5)	0.082(5)	0.006(4)	–0.031(4)	–0.040(4)
O(14)	2*i*	–1.2615(6)	2.2018(6)	0.1738(4)	0.057(4)	0.061(4)	0.094(5)	–0.004(4)	–0.034(4)	–0.022(4)
O(15)	2*i*	–1.0979(6)	2.3691(5)	0.2196(4)	0.063(4)	0.047(4)	0.088(5)	–0.008(3)	–0.033(4)	–0.014(3)
O(16)	2*i*	–1.1147(7)	2.2373(6)	0.3523(4)	0.082(5)	0.069(5)	0.072(5)	–0.004(4)	–0.022(4)	–0.025(4)
O(17)	2*i*	–1.0794(6)	2.2893(6)	0.1049(4)	0.066(5)	0.059(4)	0.071(4)	–0.010(4)	–0.026(4)	–0.002(3)
O(18)	2*i*	–1.2822(6)	2.2785(6)	0.2903(4)	0.057(4)	0.061(4)	0.083(5)	0.004(4)	–0.013(4)	–0.022(4)
N(1)	2*i*	–0.9043(8)	1.8865(7)	0.3178(5)	0.067(6)	0.043(5)	0.086(6)	0.003(4)	–0.025(5)	–0.001(4)
N(2)	2*i*	1.0166(8)	0.3252(6)	–0.1516(4)	0.085(7)	0.046(5)	0.065(5)	–0.017(5)	–0.024(5)	–0.013(4)
N(3)	2*i*	1.396(1)	0.100(1)	0.3004(7)	0.070(7)	0.14(1)	0.120(9)	–0.039(8)	–0.034(7)	–0.018(8)
C(11)	2*i*	1.018(1)	0.3980(9)	–0.2285(6)	0.11(1)	0.065(7)	0.062(7)	–0.015(7)	–0.027(7)	–0.012(6)
C(12)	2*i*	1.061(2)	0.340(1)	–0.2918(7)	0.19(2)	0.08(1)	0.075(9)	–0.02(1)	–0.02(1)	–0.028(8)
C(13)	2*i*	1.029(3)	0.429(2)	–0.366(1)	0.49(6)	0.17(2)	0.08(1)	–0.18(3)	0.05(2)	–0.05(1)
C(14)	2*i*	1.115(4)	0.474(3)	–0.390(2)	0.41(6)	0.24(4)	0.15(3)	–0.03(4)	–0.01(3)	–0.08(3)
C(15)	2*i*	0.9829(9)	0.4004(8)	–0.0974(6)	0.075(8)	0.050(6)	0.075(7)	–0.013(6)	–0.026(6)	–0.020(5)
C(16)	2*i*	0.869(1)	0.4602(9)	–0.0850(6)	0.070(8)	0.068(7)	0.082(8)	–0.001(6)	–0.025(6)	–0.027(6)
C(17)	2*i*	0.850(1)	0.5325(9)	–0.0301(6)	0.087(9)	0.066(7)	0.071(7)	–0.011(7)	–0.010(6)	–0.023(6)
C(18)	2*i*	0.735(1)	0.599(1)	–0.0189(7)	0.10(1)	0.11(1)	0.10(1)	0.007(9)	–0.024(9)	–0.049(9)
C(19)	2*i*	0.931(1)	0.2470(8)	–0.1332(6)	0.081(8)	0.049(6)	0.085(8)	–0.012(6)	–0.021(6)	–0.027(6)
C(20)	2*i*	0.909(1)	0.176(1)	–0.0573(7)	0.10(1)	0.085(9)	0.11(1)	–0.053(9)	–0.016(8)	–0.005(8)
C(21)	2*i*	0.809(2)	0.119(1)	–0.0412(9)	0.13(2)	0.11(1)	0.12(1)	–0.05(1)	–0.02(1)	–0.03(1)
C(22)	2*i*	0.707(2)	0.185(2)	–0.017(1)	0.13(2)	0.27(3)	0.24(3)	–0.07(2)	0.04(2)	–0.16(2)
C(23)	2*i*	1.130(1)	0.2581(8)	–0.1477(6)	0.075(8)	0.047(6)	0.094(8)	–0.013(6)	–0.017(6)	–0.025(6)
C(24)	2*i*	1.228(1)	0.323(1)	–0.1704(8)	0.09(1)	0.076(9)	0.10(1)	–0.017(8)	0.002(8)	–0.032(7)
C(25)	2*i*	1.330(1)	0.246(1)	–0.155(1)	0.068(9)	0.09(1)	0.18(2)	–0.008(8)	–0.02(1)	–0.06(1)
C(26)	2*i*	1.430(1)	0.306(2)	–0.179(1)	0.09(1)	0.15(2)	0.25(2)	–0.03(1)	–0.03(1)	–0.10(2)
C(27)	2*i*	1.357(1)	0.066(1)	0.2446(9)	0.08(1)	0.13(1)	0.13(1)	–0.02(1)	–0.02(1)	–0.03(1)
C(28)	2*i*	1.447(2)	0.033(2)	0.183(1)	0.11(2)	0.30(3)	0.16(2)	–0.02(2)	–0.04(1)	–0.12(2)

Table 3. continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(29)	2*i*	1.401(2)	–0.003(3)	0.133(2)	0.15(2)	0.28(4)	0.17(2)	–0.02(2)	–0.05(2)	–0.01(2)
C(30)	2*i*	1.382(2)	0.090(3)	0.081(2)	0.16(2)	0.31(4)	0.29(4)	–0.05(3)	–0.11(3)	–0.07(3)
C(31)	2*i*	1.293(1)	0.126(2)	0.3611(8)	0.070(9)	0.18(2)	0.11(1)	–0.03(1)	–0.015(8)	–0.04(1)
C(32)	2*i*	1.317(2)	0.154(2)	0.425(1)	0.11(1)	0.14(2)	0.13(1)	–0.01(1)	–0.03(1)	–0.04(1)
C(33)	2*i*	1.201(3)	0.190(2)	0.487(2)	0.21(3)	0.16(2)	0.30(4)	–0.06(2)	–0.02(3)	0.05(3)
C(35)	2*i*	1.479(1)	0.014(1)	0.3313(9)	0.068(9)	0.13(1)	0.14(1)	–0.014(9)	–0.035(9)	–0.03(1)
C(36)	2*i*	1.440(2)	–0.094(2)	0.373(1)	0.10(1)	0.16(2)	0.16(2)	–0.04(1)	–0.03(1)	–0.01(1)
C(37)	2*i*	1.534(3)	–0.177(2)	0.406(2)	0.22(3)	0.10(2)	0.34(4)	–0.00(2)	–0.11(3)	0.05(2)
C(38)	2*i*	1.593(5)	–0.252(4)	0.394(3)	0.49(9)	0.39(7)	0.6(1)	0.22(7)	–0.26(8)	–0.35(8)
C(39)	2*i*	1.455(1)	0.200(2)	0.265(1)	0.08(1)	0.16(2)	0.15(1)	–0.03(1)	–0.03(1)	–0.03(1)
C(40)	2*i*	1.387(2)	0.308(2)	0.231(1)	0.09(1)	0.14(2)	0.17(2)	–0.01(1)	–0.03(1)	0.00(1)
C(41)	2*i*	1.456(2)	0.396(2)	0.198(2)	0.12(2)	0.15(2)	0.43(4)	–0.06(2)	–0.13(2)	0.11(3)
C(42)	2*i*	1.389(2)	0.498(2)	0.166(2)	0.20(3)	0.17(2)	0.40(4)	–0.05(2)	–0.16(3)	–0.07(3)
C(34)	2*i*	1.151(5)	0.300(5)	0.461(2)	0.8(1)	0.47(7)	0.25(4)	–0.41(9)	–0.23(6)	0.08(5)
C(1)	2*i*	–0.835(1)	1.802(1)	0.3517(8)	0.074(8)	0.066(8)	0.10(1)	0.003(7)	–0.041(7)	–0.013(7)
C(2)	2*i*	–0.834(1)	1.789(1)	0.4251(8)	0.078(9)	0.066(8)	0.11(1)	0.002(7)	–0.038(8)	–0.010(8)
C(7)	2*i*	–0.906(2)	1.869(2)	0.4688(8)	0.12(1)	0.19(2)	0.09(1)	0.02(1)	–0.03(1)	–0.03(1)
C(3)	2*i*	–0.764(2)	1.701(2)	0.454(1)	0.14(2)	0.10(1)	0.16(2)	0.01(1)	–0.10(1)	0.00(1)
C(4)	2*i*	–0.697(2)	1.634(2)	0.418(2)	0.16(2)	0.09(1)	0.36(4)	0.06(1)	–0.17(3)	–0.07(2)
C(5)	2*i*	–0.693(2)	1.645(2)	0.342(2)	0.10(1)	0.10(2)	0.39(4)	0.04(1)	–0.10(2)	–0.11(2)
C(6)	2*i*	–0.762(1)	1.732(1)	0.310(1)	0.09(1)	0.09(1)	0.17(2)	0.028(9)	–0.02(1)	–0.07(1)
O(19)	2*i*	–0.625(2)	1.551(1)	0.453(1)	0.24(2)	0.102(9)	0.46(3)	0.08(1)	–0.24(2)	–0.12(1)
O(20)	2*i*	–0.6256	1.431	0.3831	0.31	0.095	0.452	–0.017	–0.229	–0.019
C(8)	2*i*	–0.5792	1.4699	0.4107	0.364	0.087	0.266	–0.009	–0.247	–0.016
C(9)	2*i*	–0.4673	1.3975	0.4093	0.191	0.108	0.345	0.025	–0.119	0.065
C(10)	2*i*	–0.372(2)	1.454(2)	0.386(2)	0.30(4)	0.10(2)	0.37(4)	0.01(2)	–0.17(3)	–0.08(2)

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