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Crystal structure of bis[μ-1,1′-(1,4-butanediyl)bis(2-methylbenzimidazole)disilver(I)] 5,5′-(*p*-xylylenediamino)-1,1′,3,3′-(benzenetetracarboxylate) heptahydrate, $[Ag_2(C_{20}H_{22}N_4)_2]_2(C_{24}H_{14}N_2O_8) \cdot 7H_2O$

Li-Hui Zhang and Jin Yang*

Northeast Normal University, Department of Chemistry, Changchun 130024, P. R. China

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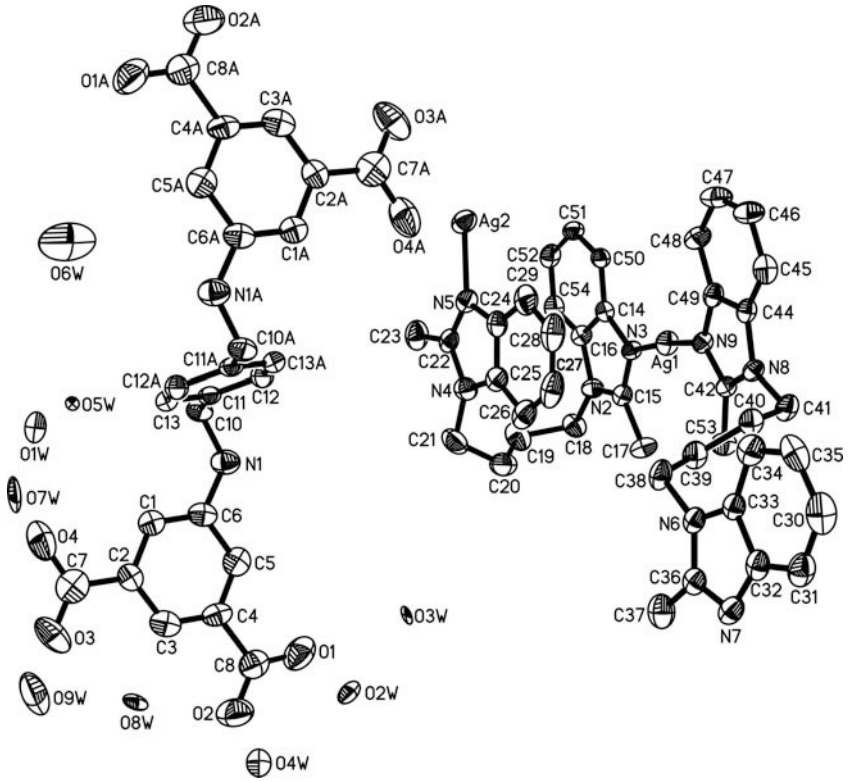

Abstract

$C_{104}H_{116}Ag_4N_{18}O_{15}$, monoclinic, $P2_1/n$ (no. 14), a = 15.234(1) Å, b = 25.536(1) Å, c = 15.9150(8) Å, β = 114.613(7)°, V = 5628.7 Å^3, Z = 2, $R(F)$ = 0.060, $wR_{ref}(F^2)$ = 0.173, T = 293 K.

Source of material

5,5′-(*p*-xylylenediamino)-1,1′,3,3′-(benzenetetracarboxylic acid) (L1) and 1,1′-(1,4-butanediyl)bis(2-methylbenzimidazole) ligand (L2) were sythesized [1]. A mixture of L1 (0.093 g, 0.2 mmol) in methanol (15 ml), L2 (0.064 g, 0.2 mmol) and Ag_2CO_3 (0.028 g, 0.1 mmol) were added to the solution. After the mixture was stirred for 30 min, white precipitate was collected, and dissolved in a minimum amount of ammonia (14 M). Colorless crystals were obtained from the solution by slow evaporation of the ammonia solution at ambient temperature (yield 49 %) after several days.

Experimental details

All hydrogen atoms on C atoms were generated geometrically and refined as riding atoms with d(C—H) = 0.93 - 0.97 Å, U_{iso}(H) = 1.2 or 1.5 U_{eq}(C). The hydrogen atoms bound to O1W, O2W and O4W are found in difference Fourier map and refined freely. The hydrogen atoms of the other water molecules could not be located. The huge residual values are caused by the vibration of the terminal O1 and O2 atoms at room temperature. The occupancies of the water molecules were established according to the thermal parameter.

Discussion

Metal-organic frameworks (MOFs) have received considerable interest in coordination chemistry and material science due to their structural diversity and potential applications in functional materials, nanotechnology, and biological recognition [2].

In the title crystal structure, the central Ag(I) atom is two-coordinated by two nitrogen atoms from two 1,1′-(1,4-butanediyl)bis(2-methylbenzimidazole) molecules (L2), showing a

* Correspondence author (e-mail: yang01chem@yahoo.com.cn)

chain structure. The bond lengths Ag1—N3, Ag1—N9, Ag2—N5 and Ag2—N7 of 2.071(4) Å, 2.081(4) Å, 2.080(5) Å and 2.118(5) Å, respectively, are within the normal range. The L2 ligands bridge adjacent Ag(I) cations to give a one-dimensional infinite chain. Notably, the carboxylate oxygen atoms of L1 anions are deprotonated, but do not coordinate the Ag(I) centers.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.24 × 0.32 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	7.51 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.38°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	28891, 1311
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4576
$N(param)_{refined}$:	698
Programs:	SHELXS-97, SHELXL-97 [3]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(41A)	4*e*		1.3138	1.1054	0.3679	0.062
H(41B)	4*e*		1.3107	1.0457	0.3893	0.062
H(38A)	4*e*		1.1738	1.0995	0.5819	0.071
H(38B)	4*e*		1.1593	1.0399	0.5954	0.071
H(13)	4*e*		0.3910	0.9530	0.5330	0.064
H(39A)	4*e*		1.2387	1.0190	0.4984	0.066
H(39B)	4*e*		1.1427	1.0500	0.4453	0.066
H(1)	4*e*		0.4080	0.8607	0.5371	0.068
H(48)	4*e*		0.9219	1.1130	0.0683	0.071
H(45)	4*e*		1.1945	1.1838	0.3085	0.068
H(40A)	4*e*		1.3240	1.0971	0.5168	0.061
H(40B)	4*e*		1.2268	1.1252	0.4559	0.061
H(10A)	4*e*		0.3577	0.8958	0.4024	0.067
H(10B)	4*e*		0.3906	0.9144	0.3262	0.067
H(37A)	4*e*		1.3466	0.9453	0.7409	0.110
H(37B)	4*e*		1.2933	0.9584	0.6353	0.110
H(37C)	4*e*		1.2410	0.9674	0.7006	0.110
H(29)	4*e*		0.7089	1.0305	0.0648	0.087
H(12)	4*e*		0.5375	0.9749	0.3802	0.058
H(18A)	4*e*		0.8628	0.7765	–0.0187	0.071
H(18B)	4*e*		0.7804	0.7825	–0.1185	0.071
H(17A)	4*e*		0.9738	0.8172	0.0850	0.099
H(17B)	4*e*		0.9847	0.8675	0.1451	0.099
H(17C)	4*e*		1.0361	0.8645	0.0780	0.099
H(5)	4*e*		0.6156	0.7953	0.4809	0.081
H(3)	4*e*		0.5862	0.7536	0.7096	0.077
H(46)	4*e*		1.0768	1.2362	0.2064	0.078
H(34)	4*e*		1.2897	1.1717	0.6287	0.076
H(31)	4*e*		1.5631	1.1086	0.8775	0.081
H(21A)	4*e*		0.6242	0.7927	0.0791	0.097
H(21B)	4*e*		0.7037	0.8099	0.1753	0.097
H(19A)	4*e*		0.7145	0.7405	–0.0321	0.093
H(19B)	4*e*		0.6691	0.7967	–0.0527	0.093
H(35)	4*e*		1.4137	1.2276	0.7182	0.086
H(47)	4*e*		0.9455	1.2028	0.0878	0.085
H(20A)	4*e*		0.7804	0.7537	0.1170	0.102
H(20B)	4*e*		0.8180	0.8094	0.1067	0.102
H(26)	4*e*		0.8521	0.8797	0.2299	0.095
H(23A)	4*e*		0.5153	0.8223	–0.0317	0.117
H(23B)	4*e*		0.4982	0.8613	–0.1131	0.117
H(23C)	4*e*		0.4490	0.8714	–0.0454	0.117
H(30)	4*e*		1.5512	1.1950	0.8363	0.096
H(28)	4*e*		0.8542	1.0342	0.1890	0.102
H(27)	4*e*		0.9246	0.9611	0.2702	0.098
H(50)	4*e*		0.7679	1.0128	–0.0999	0.052
H(53A)	4*e*		1.2310	0.9770	0.3374	0.085
H(53B)	4*e*		1.1800	0.9579	0.2346	0.085
H(53C)	4*e*		1.1240	0.9572	0.2979	0.085
H(52)	4*e*		0.5551	0.9207	–0.2741	0.065
H(51)	4*e*		0.6218	1.0025	–0.2279	0.067
H(54)	4*e*		0.6368	0.8459	–0.2015	0.059
O(3W)	4*e*	0.25	0.8583(9)	0.7822(5)	0.5092(8)	0.032(3)
H(4WB)	4*e*	0.50	0.711(8)	0.605(5)	0.569(2)	0.205
H(4WA)	4*e*	0.50	0.784(6)	0.585(5)	0.645(7)	0.205
H(2WA)	4*e*	0.50	0.723(3)	0.649(4)	0.462(7)	0.205
H(2WB)	4*e*	0.50	0.815(6)	0.668(3)	0.482(9)	0.205
H(1WB)	4*e*	0.50	0.264(7)	0.914(3)	0.508(8)	0.205
H(1WA)	4*e*	0.50	0.250(8)	0.863(3)	0.532(8)	0.205

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	4*e*		0.96658(3)	0.98631(2)	0.08463(3)	0.0440(3)	0.0559(3)	0.0414(2)	–0.0097(2)	0.0095(2)	–0.0114(2)
Ag(2)	4*e*		0.51417(4)	0.98706(3)	–0.10696(3)	0.0606(3)	0.0867(5)	0.0524(3)	0.0197(3)	0.0216(2)	0.0082(3)
N(8)	4*e*		1.1834(3)	1.0730(2)	0.2969(3)	0.032(3)	0.045(3)	0.031(2)	0.003(2)	0.008(2)	–0.003(2)
N(6)	4*e*		1.2954(3)	1.0636(2)	0.6651(3)	0.038(3)	0.071(4)	0.031(2)	0.003(3)	0.017(2)	–0.003(3)
N(2)	4*e*		0.8167(4)	0.8492(2)	–0.0499(3)	0.051(3)	0.034(3)	0.045(3)	0.004(2)	0.018(2)	–0.003(2)
C(16)	4*e*		0.7515(4)	0.8856(2)	–0.1067(3)	0.045(4)	0.046(4)	0.033(3)	0.003(3)	0.015(3)	–0.007(3)
C(44)	4*e*		1.1293(4)	1.1155(3)	0.2492(3)	0.034(3)	0.052(4)	0.036(3)	–0.005(3)	0.013(2)	–0.008(3)
N(3)	4*e*		0.8810(3)	0.9288(2)	–0.0030(3)	0.035(3)	0.037(3)	0.037(2)	0.000(2)	0.009(2)	–0.004(2)
C(14)	4*e*		0.7905(4)	0.9350(2)	–0.0777(3)	0.040(3)	0.040(4)	0.033(3)	–0.008(3)	0.015(3)	–0.009(3)
N(9)	4*e*		1.0561(3)	1.0395(2)	0.1815(3)	0.035(3)	0.039(3)	0.040(2)	–0.002(2)	0.011(2)	–0.006(2)
C(25)	4*e*		0.7319(5)	0.9079(3)	0.1206(4)	0.056(4)	0.079(6)	0.035(3)	0.009(4)	0.024(3)	0.002(4)
N(4)	4*e*		0.6677(4)	0.8673(2)	0.0799(3)	0.069(4)	0.063(4)	0.046(3)	0.010(3)	0.031(3)	0.004(3)
C(41)	4*e*		1.2753(4)	1.0783(3)	0.3791(3)	0.037(3)	0.075(5)	0.036(3)	–0.003(3)	0.007(3)	–0.008(3)
N(5)	4*e*		0.6003(4)	0.9400(3)	0.0033(3)	0.047(3)	0.069(4)	0.043(3)	0.004(3)	0.017(2)	–0.002(3)
C(38)	4*e*		1.2019(4)	1.0650(3)	0.5858(4)	0.037(4)	0.084(5)	0.053(4)	–0.010(3)	0.015(3)	–0.010(3)
N(1)	4*e*		0.4792(4)	0.8629(2)	0.4127(4)	0.081(4)	0.065(4)	0.057(3)	0.009(3)	0.024(3)	0.001(3)
C(13)	4*e*		0.4356(4)	0.9719(3)	0.5193(4)	0.049(4)	0.051(5)	0.067(4)	0.013(3)	0.030(3)	0.017(3)
C(39)	4*e*		1.2077(4)	1.0527(3)	0.4935(3)	0.045(4)	0.079(5)	0.034(3)	–0.009(3)	0.009(3)	–0.007(3)
C(49)	4*e*		1.0483(4)	1.0934(3)	0.1772(4)	0.036(3)	0.057(5)	0.039(3)	–0.003(3)	0.015(3)	–0.007(3)
C(42)	4*e*		1.1370(4)	1.0287(2)	0.2545(3)	0.035(3)	0.038(4)	0.032(3)	0.000(3)	0.011(2)	–0.003(2)
C(33)	4*e*		1.3547(4)	1.1073(3)	0.7024(4)	0.041(4)	0.061(5)	0.036(3)	–0.002(3)	0.017(3)	–0.004(3)
C(15)	4*e*		0.8918(4)	0.8779(3)	0.0105(4)	0.042(4)	0.052(5)	0.040(3)	0.000(3)	0.017(3)	–0.004(3)
C(1)	4*e*		0.4590(5)	0.8372(3)	0.5516(4)	0.060(4)	0.047(4)	0.058(4)	–0.005(3)	0.019(4)	–0.010(3)
C(48)	4*e*		0.9769(5)	1.1265(3)	0.1159(4)	0.049(4)	0.069(6)	0.050(4)	0.013(4)	0.010(3)	0.005(3)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(36)	4*e*		1.3395(4)	1.0214(3)	0.7142(4)	0.051(4)	0.060(5)	0.040(3)	0.004(4)	0.029(3)	–0.008(3)
C(45)	4*e*		1.1408(5)	1.1694(3)	0.2607(4)	0.059(4)	0.057(5)	0.058(4)	–0.013(4)	0.028(3)	–0.018(3)
C(11)	4*e*		0.4572(4)	0.9552(2)	0.4475(3)	0.045(4)	0.045(4)	0.036(3)	0.013(3)	0.014(3)	0.008(3)
C(32)	4*e*		1.4354(5)	1.0882(3)	0.7760(4)	0.047(4)	0.082(6)	0.037(3)	–0.005(4)	0.023(3)	–0.010(3)
C(6)	4*e*		0.5038(5)	0.8337(3)	0.4952(4)	0.070(5)	0.040(5)	0.055(4)	–0.008(3)	0.013(4)	–0.007(3)
C(40)	4*e*		1.2611(4)	1.0921(3)	0.4661(3)	0.043(4)	0.070(5)	0.029(3)	–0.009(3)	0.007(2)	–0.006(3)
C(2)	4*e*		0.4863(5)	0.8068(3)	0.6319(4)	0.062(5)	0.046(4)	0.052(4)	–0.017(4)	0.021(3)	–0.016(3)
N(7)	4*e*		1.4237(4)	1.0351(3)	0.7823(3)	0.045(3)	0.084(5)	0.039(3)	0.010(3)	0.019(2)	–0.002(3)
C(10)	4*e*		0.4133(5)	0.9062(3)	0.3913(4)	0.061(4)	0.057(5)	0.042(3)	0.003(4)	0.013(3)	0.007(3)
C(37)	4*e*		1.3019(6)	0.9687(3)	0.6963(5)	0.075(5)	0.089(7)	0.068(4)	–0.010(4)	0.042(4)	–0.017(4)
C(29)	4*e*		0.7365(6)	1.0003(3)	0.0978(5)	0.082(6)	0.080(7)	0.077(5)	0.000(4)	0.055(5)	–0.014(4)
O(4)	4*e*		0.3624(6)	0.8423(3)	0.6660(4)	0.150(7)	0.097(5)	0.122(5)	–0.021(5)	0.097(5)	–0.015(4)
C(12)	4*e*		0.5219(4)	0.9845(3)	0.4287(4)	0.054(4)	0.049(4)	0.045(3)	0.004(3)	0.024(3)	0.000(3)
C(18)	4*e*		0.8020(5)	0.7938(3)	–0.0548(5)	0.080(5)	0.039(4)	0.069(4)	–0.003(4)	0.041(4)	–0.006(3)
C(17)	4*e*		0.9795(5)	0.8547(3)	0.0865(4)	0.058(5)	0.066(5)	0.061(4)	0.018(4)	0.012(3)	0.011(3)
C(22)	4*e*		0.5896(5)	0.8889(3)	0.0098(4)	0.047(4)	0.080(6)	0.039(3)	0.009(4)	0.025(3)	0.000(3)
C(5)	4*e*		0.5830(5)	0.7985(3)	0.5187(5)	0.070(5)	0.039(4)	0.091(5)	–0.015(4)	0.032(4)	–0.020(4)
O(1)	4*e*		0.7330(5)	0.7284(3)	0.5654(5)	0.092(5)	0.087(5)	0.162(6)	0.019(4)	0.057(5)	–0.006(4)
C(3)	4*e*		0.5651(6)	0.7732(3)	0.6554(5)	0.078(5)	0.042(5)	0.056(4)	–0.019(4)	0.013(4)	–0.014(3)
C(46)	4*e*		1.0706(6)	1.2001(3)	0.1997(5)	0.077(5)	0.044(4)	0.069(4)	0.018(4)	0.027(4)	0.005(4)
C(34)	4*e*		1.3442(5)	1.1592(3)	0.6782(4)	0.060(5)	0.079(6)	0.053(4)	0.000(4)	0.026(3)	–0.007(4)
C(4)	4*e*		0.6105(5)	0.7692(3)	0.5984(5)	0.055(4)	0.029(4)	0.076(5)	–0.008(3)	0.003(4)	–0.006(4)
C(31)	4*e*		1.5091(5)	1.1211(4)	0.8276(4)	0.050(4)	0.097(7)	0.050(4)	–0.011(4)	0.016(3)	–0.010(4)
C(24)	4*e*		0.6892(5)	0.9522(3)	0.0722(4)	0.058(4)	0.067(5)	0.049(4)	–0.001(4)	0.035(3)	–0.010(4)
C(21)	4*e*		0.6841(6)	0.8120(3)	0.1091(5)	0.106(7)	0.081(6)	0.073(5)	0.002(5)	0.053(5)	0.020(4)
C(19)	4*e*		0.7285(6)	0.7775(3)	–0.0193(5)	0.110(7)	0.048(5)	0.094(6)	–0.012(4)	0.063(5)	–0.013(4)
C(35)	4*e*		1.4183(6)	1.1919(3)	0.7310(5)	0.098(6)	0.060(5)	0.080(5)	–0.023(5)	0.060(5)	–0.015(4)
O(3)	4*e*		0.4591(5)	0.7841(3)	0.7629(4)	0.150(6)	0.126(6)	0.056(3)	–0.039(4)	0.043(4)	0.002(3)
C(7)	4*e*		0.4357(7)	0.8109(4)	0.6924(7)	0.091(7)	0.070(7)	0.089(7)	–0.016(5)	0.028(6)	–0.034(5)
C(47)	4*e*		0.9910(6)	1.1800(3)	0.1285(5)	0.087(6)	0.052(5)	0.069(5)	0.025(4)	0.029(4)	0.015(4)
C(20)	4*e*		0.7615(6)	0.7869(4)	0.0848(5)	0.094(6)	0.085(7)	0.087(6)	0.028(5)	0.048(5)	0.026(5)
O(2)	4*e*		0.7159(5)	0.7024(3)	0.6902(5)	0.118(6)	0.092(5)	0.139(5)	0.029(4)	0.043(4)	0.031(4)
C(26)	4*e*		0.8227(6)	0.9097(4)	0.1969(5)	0.065(5)	0.132(9)	0.049(4)	0.021(5)	0.034(4)	0.004(5)
C(23)	4*e*		0.5059(5)	0.8583(4)	–0.0502(5)	0.059(5)	0.107(7)	0.065(4)	–0.017(4)	0.023(4)	–0.009(4)
C(30)	4*e*		1.5008(6)	1.1722(4)	0.8036(5)	0.078(6)	0.107(8)	0.060(5)	–0.031(5)	0.034(4)	–0.034(5)
C(28)	4*e*		0.8228(6)	1.0022(4)	0.1712(6)	0.071(6)	0.120(9)	0.077(5)	–0.028(6)	0.043(5)	–0.042(6)
C(27)	4*e*		0.8651(6)	0.9582(5)	0.2198(5)	0.055(5)	0.137(9)	0.058(5)	–0.013(6)	0.028(4)	–0.032(6)
C(8)	4*e*		0.6932(6)	0.7293(4)	0.6183(7)	0.067(6)	0.065(6)	0.095(6)	–0.010(5)	0.019(5)	–0.007(5)
C(50)	4*e*		0.7426(4)	0.9797(3)	–0.1205(4)	0.047(3)	0.044(4)	0.044(3)	0.004(3)	0.024(3)	0.004(3)
C(53)	4*e*		1.1709(5)	0.9757(3)	0.2836(4)	0.058(4)	0.052(5)	0.053(3)	0.001(3)	0.016(3)	0.003(3)
C(52)	4*e*		0.6152(4)	0.9235(3)	–0.2246(4)	0.038(4)	0.082(6)	0.035(3)	–0.003(4)	0.007(3)	–0.002(3)
C(51)	4*e*		0.6551(5)	0.9731(3)	–0.1957(4)	0.056(4)	0.065(5)	0.052(4)	0.021(4)	0.029(3)	0.022(3)
C(54)	4*e*		0.6626(4)	0.8790(3)	–0.1817(4)	0.047(4)	0.057(5)	0.043(3)	–0.015(3)	0.018(3)	–0.017(3)
O(1W)	4*e*	0.50	0.2260(5)	0.8898(4)	0.5002(5)	0.046(5)	0.069(7)	0.052(5)	–0.018(4)	0.024(4)	–0.032(4)
O(2W)	4*e*	0.50	0.7803(6)	0.6412(3)	0.4755(6)	0.036(5)	0.044(6)	0.080(6)	0.009(4)	0.018(4)	–0.005(4)
O(4W)	4*e*	0.50	0.7355(8)	0.6036(3)	0.6270(8)	0.095(8)	0.006(5)	0.146(9)	0.008(4)	0.078(7)	0.004(5)
O(5W)	4*e*	0.25	0.1690(8)	0.8423(5)	0.3271(7)	0.020(3)	0.018(3)	0.018(3)	–0.001(1)	0.008(1)	–0.001(1)
O(7W)	4*e*	0.25	0.252(1)	0.8454(7)	0.771(1)	0.06(1)	0.13(2)	0.07(1)	–0.01(1)	0.06(1)	–0.01(1)
O(6W)	4*e*	0.50	0.048(1)	0.9486(9)	0.519(2)	0.045(8)	0.22(2)	0.44(3)	–0.06(1)	–0.02(1)	0.16(2)
O(8W)	4*e*	0.25	0.496(1)	0.6860(5)	0.8186(9)	0.044(9)	0.025(9)	0.037(8)	–0.016(7)	0.004(7)	0.011(6)
O(9W)	4*e*	0.50	0.328(1)	0.7565(6)	0.8342(9)	0.126(8)	0.19(1)	0.113(7)	–0.048(7)	0.072(6)	0.015(7)

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