

CONTENTS:

Table 1S. Full X-ray Data Collection and Structure Analysis Details for Empty β -Form of $[\text{CuL}_2]$

Table 2S. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β - $[\text{CuL}_2]$.

Table 3S. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β - $[\text{CuL}_2]$ at 173 K.

Table 4S. Bond lengths [$\text{\AA}$] for β - $[\text{CuL}_2]$.

Table 5S. Bond angles [deg] for β - $[\text{CuL}_2]$.

Table 6S. Parameters of intramolecular hydrogen bonding in β - $[\text{CuL}_2]$.

Table 7S. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β - $[\text{CuL}_2]$ at 293 K.

Table 8S. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β - $[\text{CuL}_2]$ at 173 K.

Table 9S. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β - $[\text{CuL}_2]$ at 293 K.

Table 10S. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β - $[\text{CuL}_2]$ at 173 K.

Table 11S. Channel profiles for β - $[\text{CuL}_2]$ matrix in a series of compounds.

Table 12S. Molecular volumes and packing parameters for a series of compounds based on β - $[\text{CuL}_2]$ matrix.

Table 13S. Unit cell parameters and selected torsion and dihedral angles for a series of compounds based on β - $[\text{CuL}_2]$ matrix.

Figure 1S. Channel profile for β - $[\text{CuL}_2]$ matrix at 293 K and at 173 K.

Figure 2S. Channel profile for β - $[\text{CuL}_2]$ matrix in inclusion compound with n-hexane and in pure ("empty") polymorph at 173 K.

Figure 3S. Channel profile for β - $[\text{CuL}_2]$ matrix in inclusion compound with tert-butylbenzene and in pure ("empty") polymorph at 173 K.

Table 1S. Full X-ray Data Collection and Structure Analysis Details for Empty β -Form of $[\text{CuL}_2]$

temperature (K)	293	173
empirical formula	$\text{C}_{16}\text{H}_{20}\text{CuF}_6\text{O}_6$	$\text{C}_{16}\text{H}_{20}\text{CuF}_6\text{O}_6$
formula weight	485.9	485.9
crystal system	trigonal	trigonal
space group	$R\text{-}\bar{3}$ (no 148)	$R\text{-}\bar{3}$ (no 148)
unit cell dimensions:		
<i>a</i> (Å)	24.358(5)	24.123(4)
<i>c</i> (Å)	10.530(2)	10.441(2)
<i>V</i> (Å ³)	5411(2)	5262(2)
<i>Z</i>	9	9
<i>D</i> _{calc} (g/cm ³)	1.342	1.380
<i>F</i> (000)	2223	2223
crystal color, habit	blue block	blue block
crystal size (mm)	0.3 0.4 0.4	0.3 0.4 0.4
μ (MoK α) (cm ⁻¹)	9.78	10.06
absorption correction	SADABS	SADABS
min/max transmission	0.642/0.746	0.651/0.746
data collection:		
θ range (deg)	2-29	2-29
<i>h,k,l</i> ranges min/max	-32/32, -32/32, -14/14	-32/32, -32/32, -14/14
unique sets coverage (%)	>99	>99
number of reflections:		
reflns for unit cell	all data	all data
all collected	20994	20130
unique, <i>R</i> _{int}	3056, 0.021	2933, 0.017
unique obsvd (<i>I</i> > 2 σ (<i>I</i>))	2430	2933
refined parameters	184	164
max shift/error	0.001	0.001
goodness of fit on <i>F</i> ²	1.040	1.057
final <i>R</i> 1, <i>wR</i> 2 (<i>I</i> > 2 σ (<i>I</i>))	0.035, 0.091	0.034, 0.095
final <i>R</i> 1, <i>wR</i> 2 (all data)	0.048, 0.098	0.039, 0.099
res. extrema (e/Å ³)	-0.28, +0.25	-0.32, +0.46

Table 2S. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β -[CuL₂] at 293 K. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U (eq)
Cu	5000	0	5000	46 (1)
C (11)	5714 (1)	857 (1)	8520 (2)	87 (1)
F (11)	5227 (2)	887 (3)	8924 (4)	138 (2)
F (12)	5944 (3)	726 (3)	9506 (4)	162 (3)
F (13)	6117 (3)	1421 (2)	8140 (4)	175 (3)
F (14)	5383 (5)	624 (4)	9537 (6)	186 (9)
F (15)	6302 (3)	1077 (5)	8849 (11)	116 (4)
F (16)	5669 (7)	1344 (4)	8201 (8)	133 (6)
C (12)	5521 (1)	367 (1)	7452 (2)	55 (1)
O (12)	5288 (1)	499 (1)	6513 (1)	51 (1)
C (13)	5614 (1)	-138 (1)	7623 (2)	65 (1)
C (14)	5434 (1)	-615 (1)	6715 (2)	51 (1)
O (14)	5163 (1)	-638 (1)	5689 (1)	51 (1)
C (15)	5537 (1)	-1185 (1)	7013 (2)	56 (1)
C (16)	5419 (2)	-1584 (2)	5833 (3)	96 (2)
C (17)	5144 (1)	-1556 (2)	8153 (4)	84 (1)
O (15)	6189 (1)	-928 (1)	7392 (2)	56 (1)
C (18)	6646 (2)	-502 (2)	6506 (3)	77 (1)
C (25)	5757 (4)	-1029 (4)	6666 (8)	92 (3)
C (26)	6394 (6)	-677 (9)	6002 (14)	92 (3)
C (27)	5308 (9)	-1674 (5)	6100 (18)	92 (3)
O (25)	5877 (5)	-1131 (6)	7962 (10)	92 (3)
C (28)	5339 (8)	-1372 (12)	8779 (12)	92 (3)

Table 3S. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β -[CuL₂] at 173 K. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Cu	5000	0	5000	29 (1)
C(11)	5168 (1)	860 (1)	8571 (2)	57 (1)
F(11)	4814 (1)	713 (2)	9593 (3)	137 (2)
F(12)	5701 (1)	897 (2)	8947 (3)	98 (1)
F(13)	5315 (2)	1430 (1)	8201 (3)	137 (2)
F(14)	5335 (7)	636 (4)	9533 (8)	109 (7)
F(15)	5676 (5)	1380 (3)	8203 (8)	125 (9)
F(16)	4780 (4)	1042 (6)	9017 (12)	71 (4)
C(12)	4861 (1)	371 (1)	7485 (2)	38 (1)
O(12)	5224 (1)	508 (1)	6526 (1)	33 (1)
C(13)	4255 (1)	-144 (1)	7681 (2)	44 (1)
C(14)	3951 (1)	-627 (1)	6756 (2)	34 (1)
O(14)	4195 (1)	-649 (1)	5712 (1)	32 (1)
C(15)	3270 (1)	-1189 (1)	7048 (2)	41 (1)
C(16)	2998 (2)	-1621 (1)	5896 (3)	66 (1)
C(17)	3292 (1)	-1564 (1)	8206 (3)	62 (1)
O(15)	2874 (1)	-929 (1)	7441 (1)	40 (1)
C(18)	2843 (1)	-500 (1)	6547 (3)	57 (1)

Table 4S. Bond lengths [Å] for β -[CuL₂].

	at 293 K	at 173 K
Cu-O(12)	1.912(1)	1.916(1)
Cu-O(12)#1	1.912(1)	1.916(1)
Cu-O(14)	1.928(1)	1.932(1)
Cu-O(14)#1	1.928(1)	1.932(1)
Cu-O(15)#2	2.768(2)	2.708(1)
Cu-O(15)#3	2.768(2)	2.708(1)
Cu-O(25)#2	2.841(7)	
Cu-O(25)#3	2.841(7)	
C(11)-F(11)	1.298(3)	1.301(2)
C(11)-F(12)	1.292(2)	1.303(2)
C(11)-F(13)	1.290(3)	1.295(2)
C(11)-F(14)	1.289(3)	1.296(3)
C(11)-F(15)	1.299(3)	1.298(3)
C(11)-F(16)	1.289(3)	1.304(3)
C(11)-C(12)	1.532(3)	1.533(2)
C(12)-O(12)	1.260(2)	1.260(2)
C(12)-C(13)	1.369(3)	1.381(2)
C(13)-C(14)	1.395(3)	1.405(2)
C(14)-O(14)	1.253(2)	1.253(2)
C(14)-C(15)	1.561(3)	1.549(2)
C(15)-O(15)	1.443(3)	1.442(2)
C(15)-C(16)	1.514(3)	1.508(3)
C(15)-C(17)	1.519(3)	1.527(3)
O(15)-C(18)	1.425(3)	1.423(2)
C(14)-C(25)	1.560(3)	
C(25)-O(25)	1.443(3)	
C(25)-C(27)	1.516(3)	
C(25)-C(26)	1.516(3)	
O(25)-C(28)	1.426(4)	

Symmetry transformations used to generate equivalent atoms:

293 K experiment:

#1 $-x+1, -y, -z+1$; #2 $4/3-x+y, 2/3-x, -1/3+z$; #3 $-1/3+x-y, -2/3+x, 4/3-z$

173 K experiment:

#1 $-x+1, -y, -z+1$; #2 $1/3-y, -1/3+x-y, -1/3+z$; #3 $2/3+y, 1/3-x+y, 4/3-z$

Table 5S. Bond angles [deg] for β -[CuL₂].

	at 293 K	at 173 K
O(12)-Cu-O(14)	92.38(5)	92.60(5)
O(12)-Cu-O(12)#1	180.0	180.0
O(12)-Cu-O(14)#1	87.62(5)	87.40(5)
O(12)#1-Cu-O(14)#1	92.38(5)	92.60(5)
O(14)-Cu-O(14)#1	180.0	180.0
F(11)-C(11)-F(12)	105.8(3)	105.2(2)
F(11)-C(11)-F(13)	105.7(3)	109.1(2)
F(12)-C(11)-F(13)	108.6(3)	106.3(2)
F(14)-C(11)-F(15)	106.2(4)	107.1(3)
F(14)-C(11)-F(16)	107.3(4)	106.7(3)
F(15)-C(11)-F(16)	105.7(4)	105.6(3)
F(11)-C(11)-C(12)	110.6(2)	113.5(2)
F(12)-C(11)-C(12)	113.7(2)	110.9(2)
F(13)-C(11)-C(12)	112.0(2)	111.5(2)
F(14)-C(11)-C(12)	112.8(3)	112.6(3)
F(15)-C(11)-C(12)	111.3(3)	112.6(3)
F(16)-C(11)-C(12)	113.0(3)	111.7(3)
C(11)-C(12)-O(12)	112.3(2)	112.4(1)
C(13)-C(12)-O(12)	129.1(2)	129.6(2)
C(11)-C(12)-C(13)	118.6(2)	118.0(2)
Cu-O(12)-C(12)	124.0(1)	123.9(1)
C(12)-C(13)-C(14)	122.3(2)	121.6(2)
C(13)-C(14)-O(14)	124.6(2)	124.8(1)
C(13)-C(14)-C(15)	118.7(2)	118.5(1)
Cu-O(14)-C(14)	127.2(1)	127.2(1)
O(14)-C(14)-C(15)	116.7(2)	116.7(1)
C(14)-C(15)-O(15)	107.5(2)	108.5(1)
C(14)-C(15)-C(16)	110.0(2)	110.7(1)
C(14)-C(15)-C(17)	110.8(2)	109.9(2)
C(15)-O(15)-C(18)	115.1(2)	114.7(2)
O(15)-C(15)-C(16)	108.6(2)	111.8(2)
O(15)-C(15)-C(17)	105.6(2)	105.0(2)
C(16)-C(15)-C(17)	113.9(3)	110.8(2)
O(14)-C(14)-C(25)	111.6(3)	
C(14)-C(25)-O(25)	106.9(3)	
C(14)-C(25)-C(26)	110.7(3)	
C(14)-C(25)-C(27)	110.4(3)	
C(25)-O(25)-C(28)	114.7(4)	
O(25)-C(25)-C(26)	107.2(3)	
O(25)-C(25)-C(27)	107.3(3)	
C(26)-C(25)-C(27)	114.1(4)	

Symmetry transformation used to generate equivalent atoms:

#1 -x+1, -y, -z+1

Table 6S. Parameters of intramolecular hydrogen bonding in β -[CuL₂].

	atom D	atom A	atom H	D...A (Å)	D...H (Å)	D...H-A (deg)
at 293 K	O15	C13	H13	2.903(3)	2.68	94.6
	O25	C13	H13	2.817(8)	2.49	101.0
at 173 K	O15	C13	H13	2.905(2)	2.69	93.4

Table 7S. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β -[CuL₂] at 293 K.

	U11	U22	U33	U23	U13	U12
Cu	56 (1)	50 (1)	41 (1)	-5 (1)	-10 (1)	34 (1)
C(11)	111 (2)	106 (2)	69 (2)	-35 (1)	-36 (2)	73 (2)
F(11)	181 (4)	209 (5)	95 (3)	-73 (3)	-29 (2)	150 (4)
F(12)	275 (7)	220 (5)	89 (3)	-88 (3)	-110 (4)	198 (6)
F(13)	188 (5)	109 (3)	152 (4)	-76 (3)	-23 (4)	18 (3)
F(14)	226 (14)	166 (9)	74 (7)	-43 (6)	54 (8)	30 (9)
F(15)	93 (5)	116 (7)	125 (8)	-51 (6)	-48 (5)	41 (5)
F(16)	239 (13)	114 (7)	104 (7)	-57 (6)	-81 (9)	132 (9)
C(12)	58 (1)	71 (1)	46 (1)	-12 (1)	-10 (1)	40 (1)
O(12)	58 (1)	58 (1)	47 (1)	-9 (1)	-10 (1)	36 (1)
C(13)	78 (1)	89 (2)	50 (1)	-11 (1)	-19 (1)	59 (1)
C(14)	53 (1)	62 (1)	52 (1)	2 (1)	-5 (1)	37 (1)
O(14)	59 (1)	55 (1)	50 (1)	-2 (1)	-10 (1)	36 (1)
C(15)	56 (1)	60 (1)	63 (1)	10 (1)	-5 (1)	37 (1)
C(16)	148 (4)	78 (2)	99 (3)	-16 (2)	-39 (3)	85 (3)
C(17)	58 (2)	91 (2)	108 (2)	50 (2)	19 (2)	41 (2)
O(15)	50 (1)	65 (1)	63 (1)	21 (1)	5 (1)	36 (1)
C(18)	66 (2)	82 (2)	96 (2)	44 (2)	29 (1)	47 (2)

Table 8S. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β -[CuL₂] at 173 K.

	U11	U22	U33	U23	U13	U12
Cu	25 (1)	31 (1)	25 (1)	-3 (1)	2 (1)	11 (1)
C (11)	43 (1)	72 (1)	44 (1)	-25 (1)	-2 (1)	20 (1)
F (11)	68 (2)	187 (4)	72 (2)	-82 (2)	19 (1)	1 (2)
F (12)	63 (1)	147 (3)	74 (2)	-57 (2)	-38 (1)	44 (2)
F (13)	227 (5)	86 (2)	114 (3)	-64 (2)	-74 (3)	91 (3)
F (14)	141 (15)	112 (9)	69 (8)	-32 (6)	-56 (9)	59 (9)
F (15)	93 (9)	81 (8)	70 (8)	-54 (6)	44 (7)	-53 (7)
F (16)	64 (6)	78 (7)	84 (8)	-42 (6)	-8 (5)	45 (5)
C (12)	31 (1)	51 (1)	31 (1)	-10 (1)	-3 (1)	21 (1)
O (12)	29 (1)	38 (1)	30 (1)	-4 (1)	0 (1)	15 (1)
C (13)	32 (1)	61 (1)	33 (1)	-8 (1)	5 (1)	18 (1)
C (14)	26 (1)	41 (1)	34 (1)	2 (1)	3 (1)	16 (1)
O (14)	28 (1)	34 (1)	31 (1)	0 (1)	4 (1)	13 (1)
C (15)	30 (1)	43 (1)	46 (1)	5 (1)	10 (1)	16 (1)
C (16)	39 (1)	56 (1)	71 (1)	-16 (1)	16 (1)	-2 (1)
C (17)	46 (1)	69 (1)	82 (2)	38 (1)	22 (1)	36 (1)
O (15)	31 (1)	47 (1)	42 (1)	12 (1)	9 (1)	20 (1)
C (18)	36 (1)	58 (1)	74 (1)	32 (1)	9 (1)	22 (1)

Table 9S. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β -[CuL₂] at 293 K.

	x	y	z	U (eq)
H(13)	5803	-164	8370	78
H(16A)	5644	-1314	5129	143
H(16B)	4973	-1812	5649	143
H(16C)	5564	-1880	5973	143
H(17A)	5272	-1852	8419	126
H(17B)	4703	-1782	7924	126
H(17C)	5208	-1269	8836	126
H(18A)	6615	-724	5731	115
H(18B)	7064	-336	6855	115
H(18C)	6568	-160	6335	115
H(26A)	6439	-300	5614	138
H(26B)	6418	-945	5362	138
H(26C)	6727	-564	6611	138
H(27A)	5494	-1940	6165	138
H(27B)	5234	-1626	5223	138
H(27C)	4914	-1864	6555	138
H(28A)	4959	-1569	8278	138
H(28B)	5354	-1030	9261	138
H(28C)	5342	-1679	9346	138

Table 10S. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β -[CuL₂] at 173 K.

	x	y	z	U (eq)
H(13)	4039	-172	8460	53
H(16A)	2553	-1948	6069	99
H(16B)	3250	-1828	5721	99
H(16C)	3013	-1366	5152	99
H(17A)	3457	-1279	8950	93
H(17B)	3573	-1738	8021	93
H(17C)	2860	-1916	8393	93
H(18A)	3271	-129	6425	85
H(18B)	2555	-356	6875	85
H(18C)	2680	-719	5726	85

Table 11S. Channel profiles for β -[CuL₂] matrix in a series of compounds. Diameter (Å) of a sphere inscribed in the channel and centered at (0,0,Z) as a function of Z.

Z/c	(1) 293 K	(1) 173 K	(2)	(3)	(4)	(5)	(6)
0.00	5.310	5.400	5.444	5.256	5.490	5.346	5.420
0.02	5.174	5.258	5.316	5.130	5.400	5.206	5.288
0.04	5.060	5.134	5.212	5.026	5.332	5.086	5.178
0.06	4.968	5.034	5.132	4.946	5.268	4.988	5.092
0.08	4.900	4.956	5.074	4.888	5.200	4.912	5.028
0.10	4.856	4.902	5.040	4.854	5.156	4.860	4.986
0.12	4.838	4.872	5.030	4.846	5.136	4.832	4.970
0.14	4.842	4.864	5.046	4.862	5.138	4.828	4.980
0.16	4.872	4.882	5.086	4.902	5.164	4.850	5.012
0.18	4.926	4.924	5.148	4.966	5.214	4.894	5.068
0.20	5.006	4.990	5.240	5.054	5.286	4.962	5.150
0.22	5.106	5.078	5.346	5.166	5.354	5.054	5.252
0.24	5.230	5.188	5.476	5.298	5.432	5.168	5.378
0.26	5.374	5.320	5.628	5.450	5.528	5.302	5.522
0.28	5.540	5.472	5.800	5.622	5.642	5.458	5.688
0.30	5.722	5.644	5.990	5.812	5.776	5.632	5.872
0.32	5.924	5.834	6.196	6.020	5.926	5.824	6.072
0.34	6.118	6.038	6.338	6.242	5.982	6.032	6.144
0.36	5.986	5.900	6.206	6.356	5.846	5.994	6.008
0.38	5.872	5.782	6.092	6.242	5.728	5.874	5.890
0.40	5.778	5.682	5.996	6.146	5.628	5.772	5.790
0.42	5.704	5.602	5.920	6.068	5.550	5.690	5.712
0.44	5.650	5.542	5.866	6.012	5.492	5.628	5.654
0.46	5.618	5.502	5.832	5.974	5.456	5.586	5.616
0.48	5.606	5.484	5.818	5.958	5.440	5.566	5.602
0.50	5.616	5.488	5.826	5.962	5.448	5.566	5.608
0.52	5.606	5.484	5.818	5.958	5.440	5.566	5.602
0.54	5.618	5.502	5.832	5.974	5.456	5.586	5.616
0.56	5.650	5.542	5.866	6.012	5.492	5.628	5.654
0.58	5.704	5.602	5.920	6.068	5.550	5.690	5.712
0.60	5.778	5.682	5.996	6.146	5.628	5.772	5.790
0.62	5.872	5.782	6.092	6.242	5.728	5.874	5.890
0.64	5.986	5.900	6.206	6.356	5.846	5.994	6.008
0.66	6.118	6.038	6.338	6.242	5.982	6.032	6.144
0.68	5.924	5.834	6.196	6.020	5.926	5.824	6.072
0.70	5.722	5.644	5.990	5.812	5.776	5.632	5.872
0.72	5.540	5.472	5.800	5.622	5.642	5.458	5.688
0.74	5.374	5.320	5.628	5.450	5.528	5.302	5.522
0.76	5.230	5.188	5.476	5.298	5.432	5.168	5.378
0.78	5.106	5.078	5.346	5.166	5.354	5.054	5.252
0.80	5.006	4.990	5.240	5.054	5.286	4.962	5.150
0.82	4.926	4.924	5.148	4.966	5.214	4.894	5.068

0.84	4.872	4.882	5.086	4.902	5.164	4.850	5.012
0.86	4.842	4.864	5.046	4.862	5.138	4.828	4.980
0.88	4.838	4.872	5.030	4.846	5.136	4.832	4.970
0.90	4.856	4.902	5.040	4.854	5.156	4.860	4.986
0.92	4.900	4.956	5.074	4.888	5.200	4.912	5.028
0.94	4.968	5.034	5.132	4.946	5.268	4.988	5.092
0.96	5.060	5.134	5.212	5.026	5.332	5.086	5.178
0.98	5.174	5.258	5.316	5.130	5.400	5.206	5.288
1.00	5.310	5.400	5.444	5.256	5.490	5.346	5.420

Comments:

- (1) Empty polymorph. $c=10.530(2)$ Å at 293 K and $c=10.441(2)$ Å at 173 K. This work.
- (2) In benzene inclusion compound. $c=10.623(3)$ Å. Data refer to room T. Soldatov, D. V.; Ripmeester, J. A.; Shergina, S. I.; Sokolov, I. E.; Zanina, A. S.; Gromilov, S. A.; Dyadin, Yu. A. *J. Am. Chem. Soc.* **1999**, *121*, 4179-4188.
- (3) In tert-butylbenzene inclusion compound. $c=10.510(2)$ Å. Data refer to room T. Soldatov, D. V.; Ripmeester, J. A. *J. Inclusion Phenom.* **2001**, *39*, 81-84.
- (4) In chloroform inclusion compound. $c=10.491(2)$ Å. Data refer to $T=173$ K. Manakov, A. Yu.; Soldatov, D. V.; Ripmeester, J. A.; Lipkowski, J. *J. Phys. Chem. B* **2000**, *104*, 12111-12118.
- (5) In n-hexane inclusion compound. $c=10.434(2)$ Å. Data refer to $T=173$ K. Soldatov, D. V.; Ripmeester, J. A. *Chem. Mater.* **2000**, *12*, 1827-1839.
- (6) In tert-butanol inclusion compound. $c=10.558(2)$ Å. Data refer to $T=173$ K. Soldatov, D. V.; Ripmeester, J. A. *Chem. Mater.* **2000**, *12*, 1827-1839.

Table 12S. Molecular volumes and packing parameters for a series of compounds based on β -[CuL₂] matrix. Volumes are given in Å³. For references see previous Table.

Compound	(1) 293 K	(1) 173 K	(2)	(3)	(4)	(5)	(6)
Guest	no guest	no guest	benzene	tert-butyl-benzene	chloroform	n-hexane	tert-butanol
Guest:Host			2/3	1/3	2/3	0.373(4)	2/3
Unit cell volume	5411(2)	5262(2)	5522(2)	5461(1)	5282(2)	5267(1)	5364(2)
Volume occupied by host matrix	2738(1)	2755(1)	2733(1)	2693(1)	2709(1)	2716(1)	2682(1)
Packing coefficient for host matrix	0.506	0.524	0.495	0.493	0.513	0.516	0.500
Volume per guest molecule			78.4(1)	137.8(1)	82.2(1)	102.4(1) 100.9(1)	75.8(1)
Packing coefficient	0.506	0.524	0.580	0.569	0.606	0.582	0.585
Temperature of study	room	173 K	room	room	173 K	173 K	173 K

Comments:

For n-hexane molecule volumes for two found conformations are given separately.

For α -[CuL₂] unit cell volume is 4290(1) Å³ (Z=8), volume occupied by host structure is 2424(1) Å³, and packing coefficient equals **0.565**. These data refer to room T. Soldatov, D. V.; Ripmeester, J. A.; Shergina, S. I.; Sokolov, I. E.; Zanina, A. S.; Gromilov, S. A.; Dyadin, Yu. A. *J. Am. Chem. Soc.* **1999**, *121*, 4179-4188.

Table 13S. Unit cell parameters ($\text{\AA}$, $\text{\AA}^3$) and selected torsion and dihedral angles (deg.) for a series of compounds based on β -[CuL₂] matrix. Volumes are given in $\text{\AA}^3$. For references see Table 11S.

Compound	(1) 293 K	(1) 173 K	(2)	(3)	(4)	(5)	(6)
Guest	no guest	no guest	benzene	tert-butyl-benzene	chloro-form	n-hexane	tert-butanol
Parameter a	24.358(5)	24.123(4)	24.492(1)	24.495(3)	24.111(4)	24.144(3)	24.221(4)
Parameter c	10.530(2)	10.441(2)	10.624(1)	10.510(2)	10.491(2)	10.434(2)	10.558(2)
Unit cell volume	5411(2)	5262(2)	5522(2)	5461(1)	5282(2)	5267(1)	5364(2)
Angle 1	-52.2	+51.2	+50.1	+51.3	-50.1	-51.0	-50.0
Angle 2	9.7	9.6	11.2	9.2	10.0	9.3	11.8
Angle 3	66.5	67.2	65.9	66.1	67.0	67.2	65.9
Angle 4	74.8	74.0	75.5	75.3	74.3	74.1	75.5
Temperature of study	room	173 K	room	room	173 K	173 K	173 K

Comments:

Angle 1: Torsion angle C13-C14-C14-O14

Angle 2: Angle between normal to bischelate fragment and (Cu-O15B) bond

Angle 3: Dihedral angle between bischelate fragment and (ab) crystallographic plane

Angle 4: Dihedral angle between bischelate fragments of two adjacent [CuL₂] units

Figure 1S. Channel profile for β -[CuL₂] matrix at 293 K (solid line) and at 173 K (dashes). (Diameter (Å) of a sphere inscribed in the channel and centered at (0,0,Z) as a function of Z.)

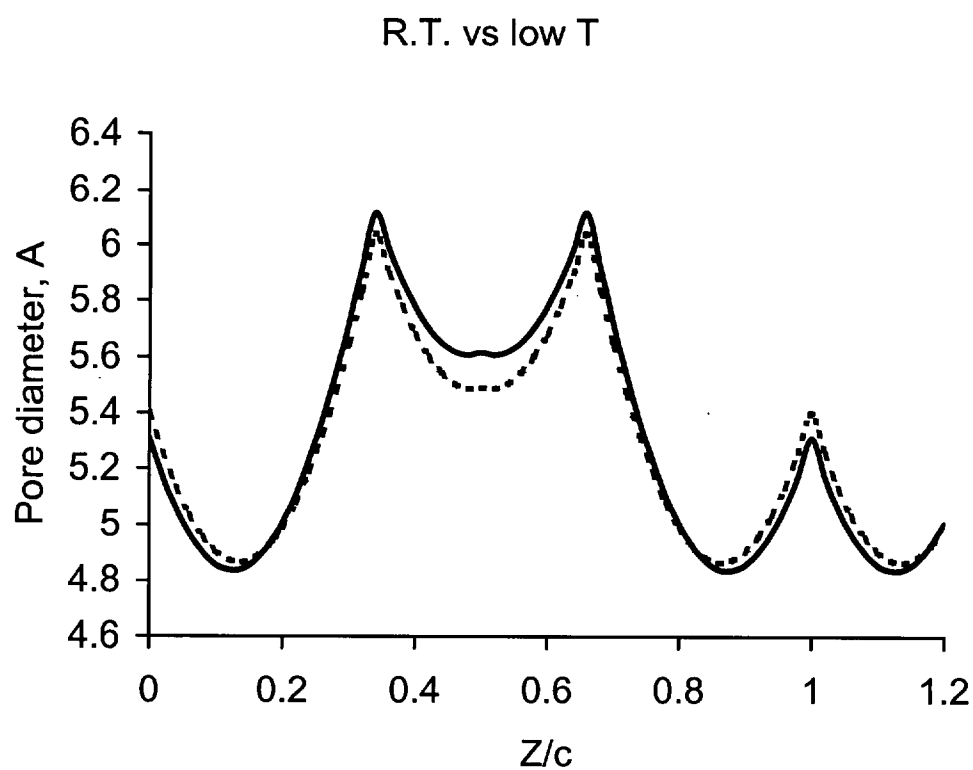


Figure 2S. Channel profile for β -[CuL₂] matrix in inclusion compound with n-hexane (solid line) and in pure ("empty") polymorph (dashes). Data refer to 173 K.
(Diameter (Å) of a sphere inscribed in the channel and centered at (0,0,Z) as a function of Z.)

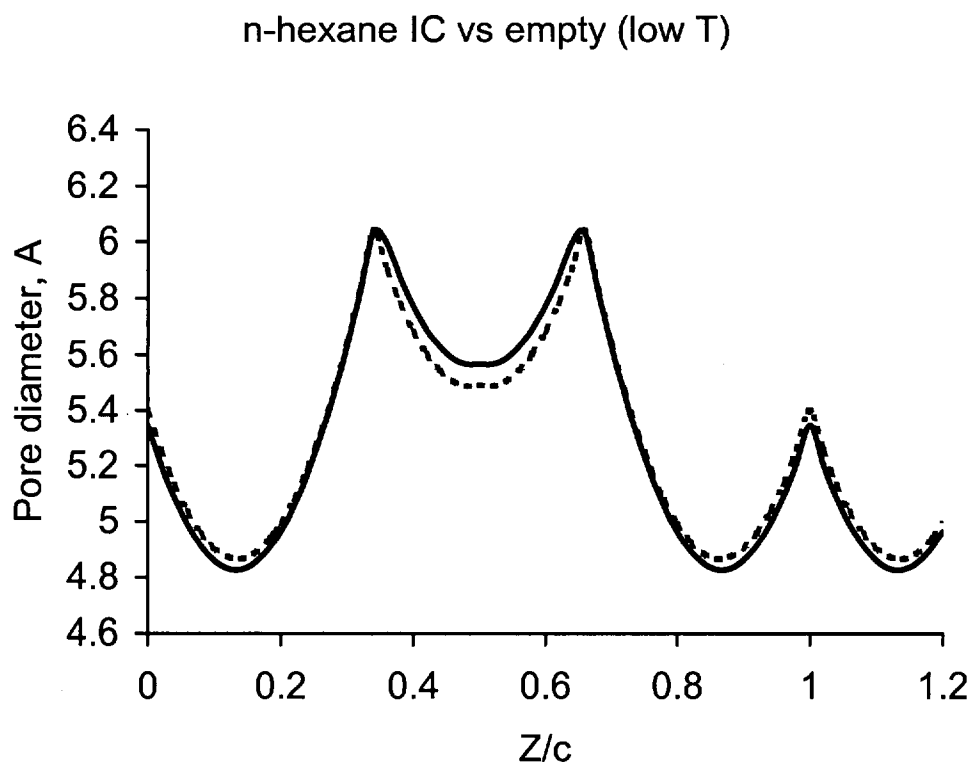


Figure 3S. Channel profile for β -[CuL₂] matrix in inclusion compound with tert-butylbenzene (solid line) and in pure ("empty") polymorph (dashes). Data refer to 173 K. (Diameter (Å) of a sphere inscribed in the channel and centered at (0,0,Z) as a function of Z.)

