Electronic Supplementary Material (ESI) for CrystEngComm.

Supporting information

Preparation of large-size single-crystal metal-organic frameworks via Marangoni effect

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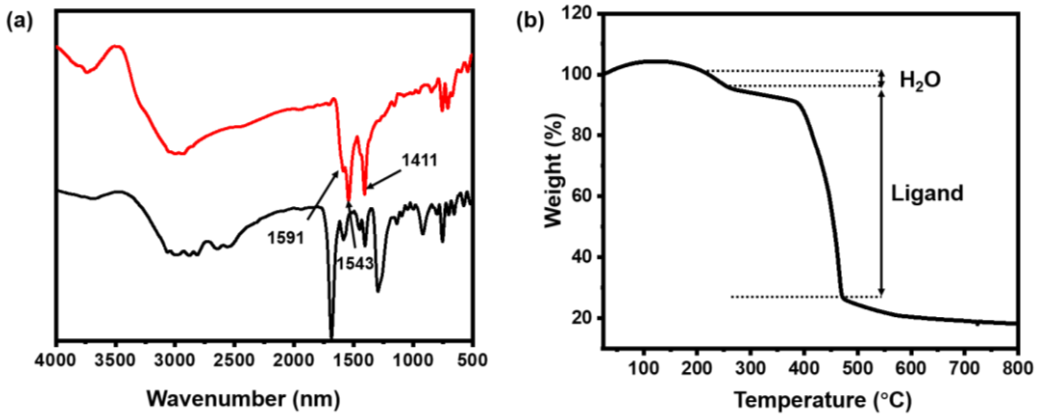

Figure S1. The characterization of Zn-MOF. (a) FT-IR spectra. Blank line: ligand; red line: Zn-MOF. (b) Thermogravimetric analysis.

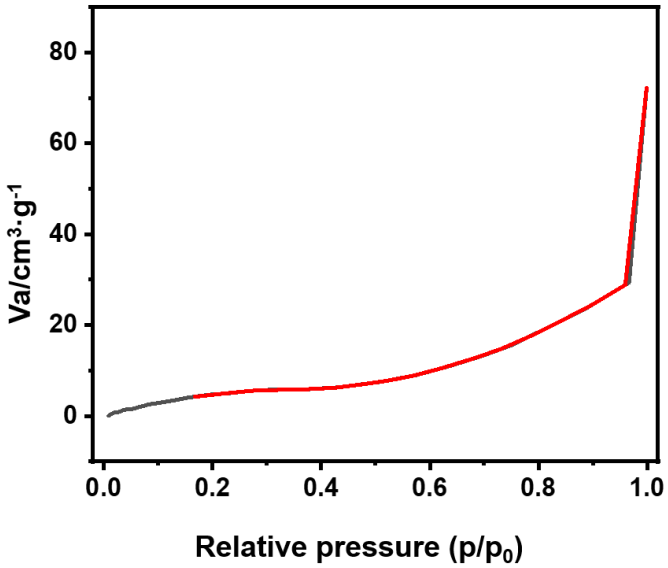

Figure S2. N_2 adsorption-desorption isotherm of Zn-MOF.

The surface area of the Zn-MOF is 16.446 m^2/g, and the N_2 adsorption isotherms show a type-II.

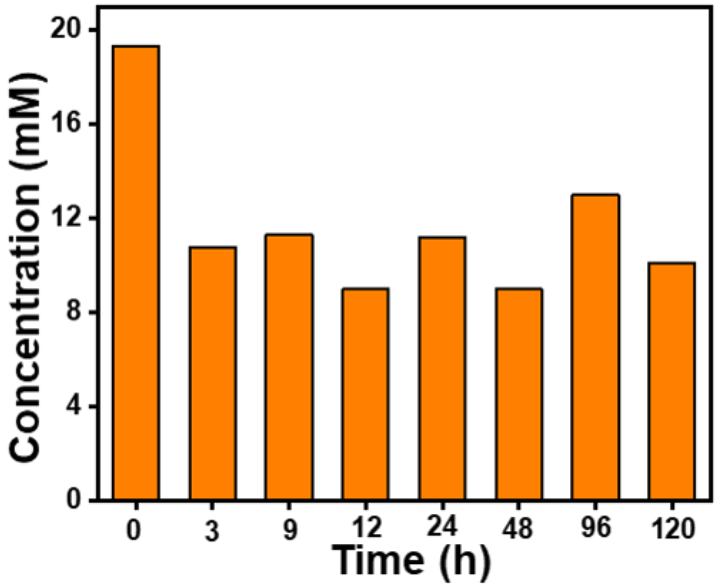

Figure S3. Concentration changing of Zn^{2+} ion in solution with reaction time calculated by ICP.

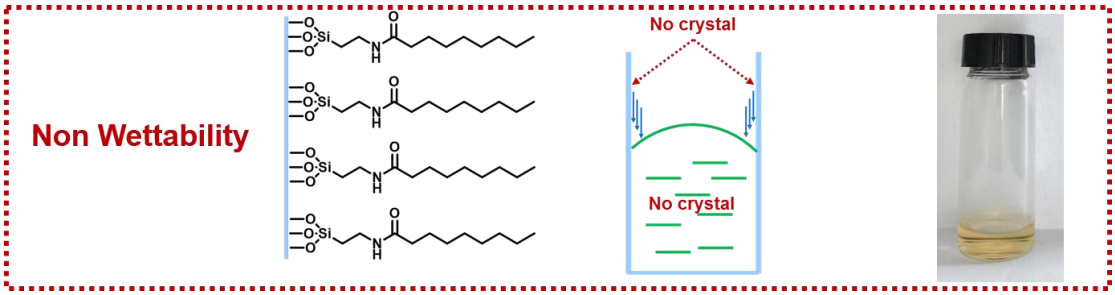

Figure S4. Modified the Pyrex vial with alkyl chain and used it to prepare the Zn-MOF.

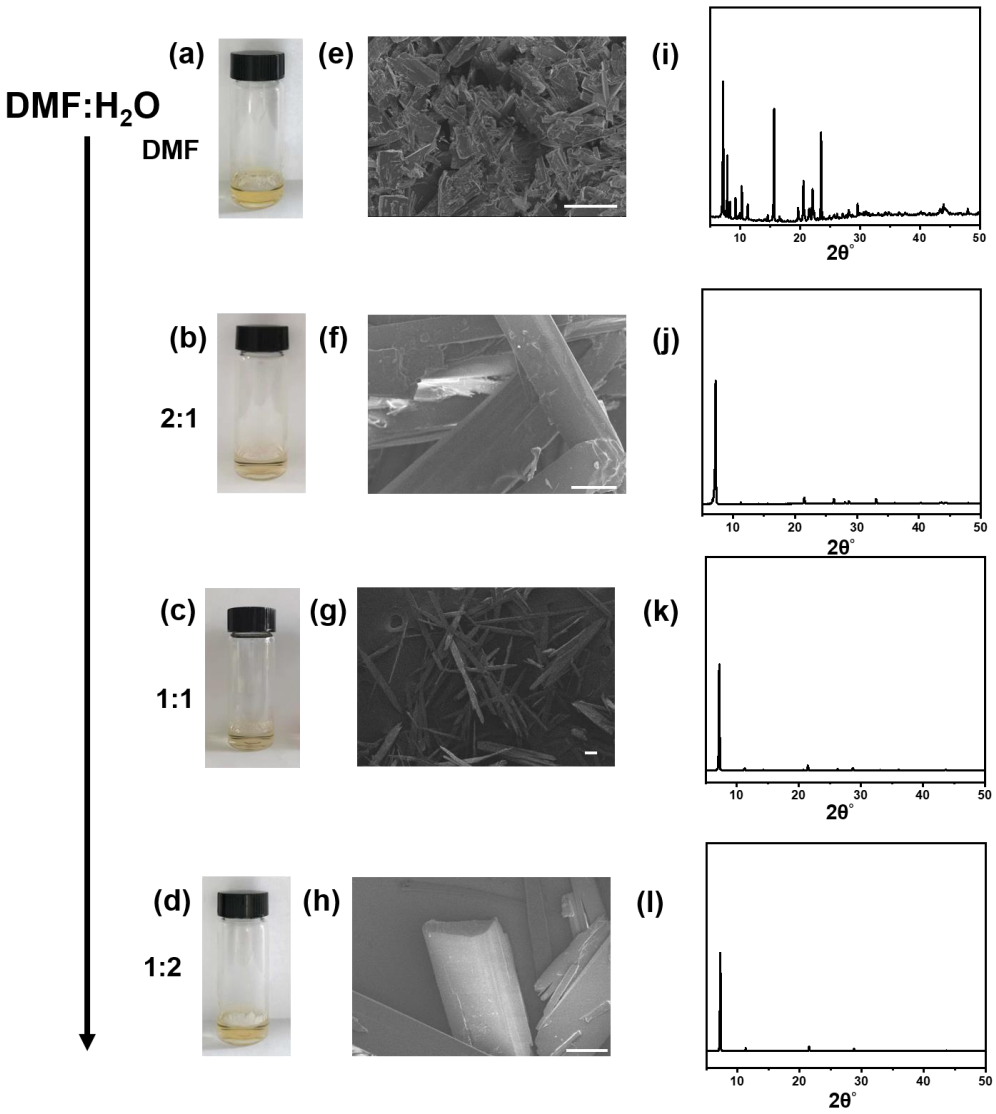

Figure S5. Synthesize Zn-MOF using different content of water in solution. (a-d) Photographs for Zn-MOF crystal generated on the vessel wall of the Pyrex vessel. (e-h) SEM images. The scale bar is 50 μm. (i-l) PXRD patterns for the Zn-MOF crystal.

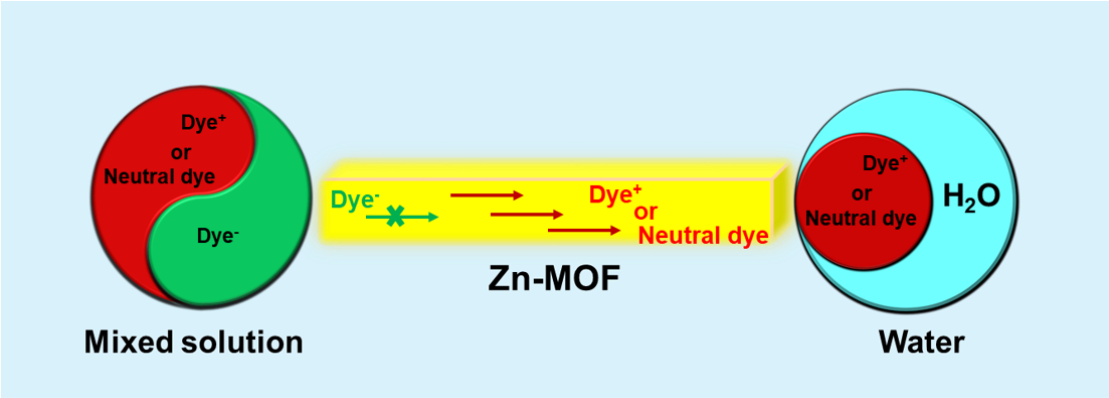

Figure S6. Schematic illustration for the mixed dye separation.

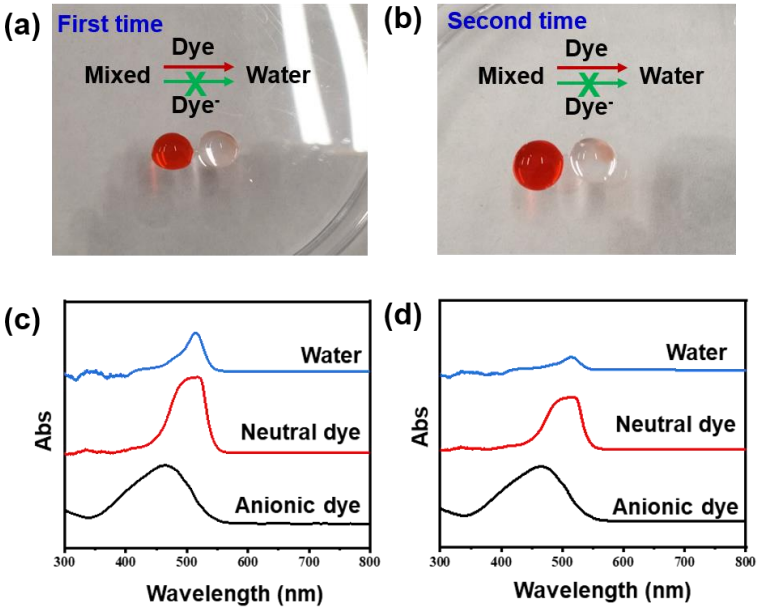

Figure S7. Reusability of Zn-MOF for the selective separation of dyes. Photographs of the selective separation of mixed dye solution: dye solution (left), Zn-MOF crystal (middle), and water (right): (a, b) mixed neutral and anionic dyes. Anionic dye: methyl orange; neutral dye: eosin Y. (c, d) UV absorption spectrum of different solution.

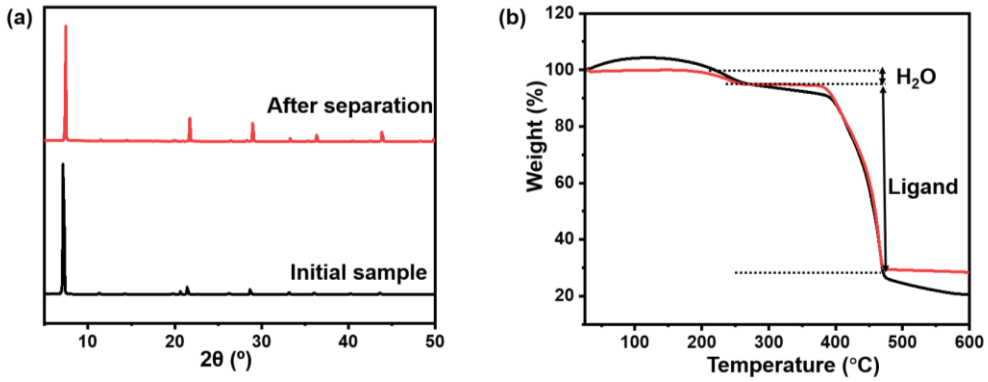

Figure S8. The characterization of the Zn-MOF before and after the separation of mixed dyes. (a) PXRD. (b) TGA. Black line: initial sample; red line: crystal after the dye separation.

Table 1. Crystal data and structure refinement for Zn-MOF.

Identification code	2107028977	
Empirical formula	C_{14} H_{10} O_5 Zn	
Formula weight	323.59	
Temperature	296.15 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 12.846(5) Å	= 90°.
	b = 5.943(2) Å	= 104.065(7)°.
	c = 17.311(7) Å	= 90°.
Volume	1281.9(8) $Å^3$	
Z	4	
Density (calculated)	1.677 Mg/m^3	
Absorption coefficient	1.931 mm^{-1}	
F(000)	656	
Crystal size	0.12 x 0.02 x 0.01 mm^3	
Theta range for data collection	1.634 to 26.577°.	
Index ranges	-16<=h<=14, -7<=k<=7, -21<=l<=21	
Reflections collected	8883	
Independent reflections	2637 [R(int) = 0.0535]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7456 and 0.4786	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2637 / 0 / 186	
Goodness-of-fit on F^2	0.984	
Final R indices [I>2sigma(I)]	R1 = 0.0355, wR2 = 0.0818	
R indices (all data)	R1 = 0.0538, wR2 = 0.0890	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.469 and -0.601 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for Zn-MOF. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Zn(1)	4409(1)	253(1)	3896(1)	30(1)
O(1)	5793(2)	1655(4)	4255(1)	45(1)
O(2)	6056(2)	2264(4)	5555(1)	40(1)
O(3)	6775(2)	7648(4)	6315(1)	39(1)
O(4)	6043(2)	5641(4)	7123(1)	42(1)
O(5)	4563(2)	-1367(4)	2941(1)	39(1)
C(1)	6246(2)	2644(5)	4890(2)	30(1)
C(2)	7044(2)	4415(5)	4825(2)	30(1)
C(3)	6900(3)	5512(6)	4093(2)	44(1)
C(4)	7574(3)	7225(6)	3992(2)	53(1)
C(5)	8421(3)	7831(6)	4612(2)	52(1)
C(6)	8586(2)	6727(6)	5327(2)	43(1)
C(7)	7899(2)	5051(5)	5460(2)	31(1)
C(8)	8168(2)	3882(5)	6244(2)	29(1)
C(9)	8975(2)	2262(5)	6374(2)	42(1)
C(10)	9257(3)	1086(6)	7075(2)	51(1)
C(11)	8729(3)	1465(6)	7669(2)	45(1)
C(12)	7932(2)	3041(5)	7554(2)	38(1)
C(13)	7645(2)	4295(5)	6853(2)	29(1)
C(14)	6757(2)	5981(5)	6756(2)	31(1)

Table 3. Bond lengths [Å] and angles [°] for Zn-MOF.

Zn(1)-O(1)	1.924(2)
Zn(1)-O(2)#1	1.943(2)
Zn(1)-O(3)#2	1.933(2)
Zn(1)-O(5)	1.964(2)
O(1)-C(1)	1.257(3)
O(2)-C(1)	1.253(3)
O(3)-C(14)	1.256(3)
O(4)-C(14)	1.252(3)
O(5)-H(5A)	0.8526
O(5)-H(5B)	0.79(4)
C(1)-C(2)	1.493(4)
C(2)-C(3)	1.397(4)
C(2)-C(7)	1.404(4)
C(3)-H(3)	0.9300
C(3)-C(4)	1.375(5)
C(4)-H(4)	0.9300
C(4)-C(5)	1.376(5)
C(5)-H(5)	0.9300
C(5)-C(6)	1.372(5)
C(6)-H(6)	0.9300
C(6)-C(7)	1.386(4)
C(7)-C(8)	1.489(4)
C(8)-C(9)	1.392(4)
C(8)-C(13)	1.403(4)
C(9)-H(9)	0.9300
C(9)-C(10)	1.370(5)
C(10)-H(10)	0.9300
C(10)-C(11)	1.383(5)
C(11)-H(11)	0.9300
C(11)-C(12)	1.365(4)
C(12)-H(12)	0.9300
C(12)-C(13)	1.394(4)
C(13)-C(14)	1.497(4)
O(1)-Zn(1)-O(2)#1	123.00(9)
O(1)-Zn(1)-O(3)#2	113.85(10)
O(1)-Zn(1)-O(5)	102.17(9)

O(2)#1-Zn(1)-O(5)	98.09(10)
O(3)#2-Zn(1)-O(2)#1	105.49(9)
O(3)#2-Zn(1)-O(5)	113.18(10)
C(1)-O(1)-Zn(1)	132.84(19)
C(1)-O(2)-Zn(1)#1	137.7(2)
C(14)-O(3)-Zn(1)#2	119.56(18)
Zn(1)-O(5)-H(5A)	109.5
Zn(1)-O(5)-H(5B)	117(3)
H(5A)-O(5)-H(5B)	122.5
O(1)-C(1)-C(2)	116.3(3)
O(2)-C(1)-O(1)	124.5(3)
O(2)-C(1)-C(2)	119.1(3)
C(3)-C(2)-C(1)	117.1(3)
C(3)-C(2)-C(7)	119.3(3)
C(7)-C(2)-C(1)	123.5(3)
C(2)-C(3)-H(3)	119.6
C(4)-C(3)-C(2)	120.8(3)
C(4)-C(3)-H(3)	119.6
C(3)-C(4)-H(4)	120.1
C(3)-C(4)-C(5)	119.8(3)
C(5)-C(4)-H(4)	120.1
C(4)-C(5)-H(5)	120.1
C(6)-C(5)-C(4)	119.8(3)
C(6)-C(5)-H(5)	120.1
C(5)-C(6)-H(6)	119.0
C(5)-C(6)-C(7)	121.9(3)
C(7)-C(6)-H(6)	119.0
C(2)-C(7)-C(8)	123.5(2)
C(6)-C(7)-C(2)	118.2(3)
C(6)-C(7)-C(8)	118.1(3)
C(9)-C(8)-C(7)	118.0(3)
C(9)-C(8)-C(13)	118.2(3)
C(13)-C(8)-C(7)	123.8(3)
C(8)-C(9)-H(9)	119.2
C(10)-C(9)-C(8)	121.5(3)
C(10)-C(9)-H(9)	119.2
C(9)-C(10)-H(10)	119.9
C(9)-C(10)-C(11)	120.2(3)

C(11)-C(10)-H(10)	119.9
C(10)-C(11)-H(11)	120.4
C(12)-C(11)-C(10)	119.3(3)
C(12)-C(11)-H(11)	120.4
C(11)-C(12)-H(12)	119.2
C(11)-C(12)-C(13)	121.6(3)
C(13)-C(12)-H(12)	119.2
C(8)-C(13)-C(14)	121.6(3)
C(12)-C(13)-C(8)	119.1(3)
C(12)-C(13)-C(14)	119.2(3)
O(3)-C(14)-C(13)	118.0(2)
O(4)-C(14)-O(3)	124.1(3)
O(4)-C(14)-C(13)	117.9(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+1 #2 -x+1,-y+1,-z+1

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for Zn-MOF. The anisotropic displacement factor exponent takes the form: $-2\ ^2[\ h^2\ a^{*2}U^{11} + ... \quad + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Zn(1)	32(1)	27(1)	30(1)	-1(1)	7(1)	-4(1)
O(1)	41(1)	54(2)	38(1)	-9(1)	6(1)	-18(1)
O(2)	48(1)	38(1)	35(1)	0(1)	9(1)	-14(1)
O(3)	39(1)	31(1)	49(1)	5(1)	16(1)	3(1)
O(4)	45(1)	37(1)	52(1)	-4(1)	27(1)	0(1)
O(5)	41(1)	34(1)	44(1)	-8(1)	19(1)	-11(1)
C(1)	27(1)	25(2)	35(2)	1(1)	3(1)	2(1)
C(2)	32(2)	26(2)	32(2)	1(1)	8(1)	-1(1)
C(3)	50(2)	44(2)	34(2)	6(2)	4(2)	-4(2)
C(4)	72(2)	48(2)	41(2)	15(2)	18(2)	-9(2)
C(5)	58(2)	45(2)	56(2)	4(2)	22(2)	-19(2)
C(6)	41(2)	44(2)	44(2)	-3(2)	9(1)	-12(2)
C(7)	29(1)	32(2)	33(1)	-4(1)	10(1)	-1(1)
C(8)	24(1)	28(2)	35(2)	-4(1)	3(1)	-3(1)
C(9)	33(2)	43(2)	47(2)	-4(2)	6(1)	9(1)
C(10)	44(2)	40(2)	63(2)	6(2)	2(2)	12(2)
C(11)	46(2)	36(2)	46(2)	10(2)	-4(2)	-1(2)
C(12)	41(2)	37(2)	33(2)	-1(1)	3(1)	-8(1)
C(13)	27(1)	26(2)	33(2)	-3(1)	3(1)	-3(1)
C(14)	31(2)	29(2)	32(2)	-9(1)	6(1)	-6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for Zn-MOF.

	x	y	z	U(eq)
H(5A)	5154	-997	2834	58
H(3)	6342	5078	3668	53
H(4)	7458	7974	3507	64
H(5)	8880	8986	4545	62
H(6)	9176	7113	5735	52
H(9)	9330	1973	5976	50
H(10)	9807	29	7150	61
H(11)	8913	656	8143	54
H(12)	7572	3284	7952	46
H(5B)	4340(30)	-2610(70)	2890(20)	50(11)

Table S6. Elemental analysis of Zn-MOF.

Sample	C		O		H	
	The.	Exp.	The.	Exp.	The.	Exp.
Zn-MOF	51.8%	51.7%	24.6	24.2	3.0%	3.0%

Element analysis:

The chemical formula of Zn-MOF crystal is $\{Zn(C_{14}H_8O_4)\cdot H_2O\}$.

The theoretical value of element was calculated based on the following equation.

$$\text{Theoretical value}_{(\text{element})}=\frac{M(element)\times N_{number}}{M_{total}}$$

$$\text{Theoretical value}_{(C)}=\frac{M(element)\times N_{number}}{M_{total}}=\frac{12\times 14}{324}=51.8\%$$

$$\text{Theoretical value}_{(H)}=\frac{M(element)\times N_{number}}{M_{total}}=\frac{1\times 10}{324}=3.0\%$$

$$\text{Theoretical value}_{(O)}=\frac{M(element)\times N_{number}}{M_{total}}=\frac{16\times 5}{324}=24.6\%$$