Supporting Information

The Crystalline Sponge Method: A Solvent-Based Strategy to Facilitate Noncovalent Ordered Trapping of Solid and Liquid Organic Compounds

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Table S1. Crystallographic details for **1·2**

	tim73-transstilbene-mtbe_a | CCDC 1545812
Crystal data	
Chemical formula	$C_{36}H_{24}I_6N_{12}Zn_3 \cdot 2.04(C_{14}H_{12})$
M_r	1949.83
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	100
a, *b*, *c* (Å)	34.9049 (14), 14.9409 (6), 34.5319 (14)
β (°)	107.5655 (8)
V (Å^3)	17169.0 (12)
Z	8
Radiation type	Synchrotron, λ = 0.41328 Å
μ (mm^{-1})	0.70
Crystal size (mm)	0.24 × 0.07 × 0.05
Data collection	
Diffractometer	Huber three-circle goniometer with free kappa
Absorption correction	Multi-scan *SADABS2014*/5 (Bruker, 2014/5) was used for absorption correction. wR_2(int) was 0.0716 before and 0.0555 after correction. The ratio of minimum to maximum transmission is 0.9295. The λ/2 correction factor is 0.00150.
T_{min}, T_{max}	0.884, 0.952
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	176286, 15424, 13344
R_{int}	0.047
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.601
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.052, 0.181, 1.03
No. of reflections	15424
No. of parameters	965
No. of restraints	573
H-atom treatment	H-atom parameters constrained
	$w = 1/[\sigma^2(F_o^2) + (0.1106P)^2 + 110.0348P]$ where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.97, -1.69

Table S2. Crystallographic details for **1·3**

	tim70c-vanillin_a	CCDC 1545813
Crystal data		
Chemical formula	$C_{72}H_{48}I_{12}N_{24}Zn_6 \cdot 2.07(C_8H_8O_3)$	
M_r	3479.51	
Crystal system, space group	Triclinic, $P\bar{1}$	
Temperature (K)	100	
a, *b*, *c* (Å)	14.9223 (8), 18.9078 (11), 32.5910 (18)	
α, β, γ (°)	102.7281 (10), 91.7744 (10), 110.7963 (9)	
V (Å^3)	8324.8 (8)	
Z	2	
Radiation type	Synchrotron, λ = 0.41328 Å	
μ (mm^{-1})	0.72	
Crystal size (mm)	0.3 × 0.07 × 0.04	
Data collection		
Diffractometer	Huber three-circle goniometer with free kappa	
Absorption correction	Multi-scan *SADABS2014*/5 (Bruker, 2014/5) was used for absorption correction. wR_2(int) was 0.0635 before and 0.0569 after correction. The ratio of minimum to maximum transmission is 0.8962. The λ/2 correction factor is 0.00150.	
T_{min}, T_{max}	0.853, 0.951	
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	175431, 29411, 23438	
R_{int}	0.051	
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.606	
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.072, 0.245, 1.06	
No. of reflections	29411	
No. of parameters	1471	
No. of restraints	392	
H-atom treatment	H-atom parameters constrained	
	$w = 1/[\sigma^2(F_o^2) + (0.1312P)^2 + 50.2672P]$ where $P = (F_o^2 + 2F_c^2)/3$	
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	2.18, -1.42	

Table S3. Crystallographic details for **1·4**

	zni2mof-cf3phenylazide_a	CCDC 1545814
Crystal data		
Chemical formula	$C_{36}H_{24}I_6N_{12}Zn_3 \cdot 0.68(C_5H_{12}O) \cdot 0.66(C_7H_4F_3N_3)$	
M_r	1765.98	
Crystal system, space group	Monoclinic, *C*2/*c*	
Temperature (K)	100	
a, *b*, *c* (Å)	35.2912 (17), 14.8832 (7), 30.9415 (15)	
β (°)	101.827 (1)	
V (Å^3)	15906.9 (13)	
Z	8	
Radiation type	Synchrotron, λ = 0.41328 Å	
μ (mm^{-1})	0.75	
Crystal size (mm)	0.28 × 0.1 × 0.06	
Data collection		
Diffractometer	Bruker D8 goniometer	
Absorption correction	Multi-scan *SADABS2014*/5 (Bruker, 2014/5) was used for absorption correction. wR_2(int) was 0.1317 before and 0.0730 after correction. The ratio of minimum to maximum transmission is 0.8854. The λ/2 correction factor is 0.00150.	
T_{min}, T_{max}	0.828, 0.936	
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	113242, 14007, 11742	
R_{int}	0.057	
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.596	
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.058, 0.227, 1.16	
No. of reflections	14007	
No. of parameters	711	
No. of restraints	73	
H-atom treatment	H-atom parameters constrained	
	$w = 1/[\sigma^2(F_o^2) + (0.1337P)^2 + 128.1977P]$ where $P = (F_o^2 + 2F_c^2)/3$	
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	2.61, -2.10	

Table S4. Crystallographic details for **1·5**

	tim83-artemisinin-c2_c	CCDC 1545815
Crystal data		
Chemical formula	$C_{72}H_{48}I_{12}N_{24}Zn_6 \cdot 0.93(C_{15}H_{22}O_5) \cdot 0.62(C_5H_{12}O)$	
M_r	3482.07	
Crystal system, space group	Monoclinic, *C*2	
Temperature (K)	100	
a, *b*, *c* (Å)	35.252 (2), 14.8612 (8), 35.233 (2)	
β (°)	108.1167 (11)	
V (Å^3)	17543.1 (17)	
Z	4	
Radiation type	Synchrotron, λ = 0.41328 Å	
μ (mm^{-1})	0.68	
Crystal size (mm)	0.16 × 0.07 × 0.05	
Data collection		
Diffractometer	Huber three-circle goniometer with free kappa	
Absorption correction	Multi-scan *SADABS2014*/5 (Bruker, 2014/5) was used for absorption correction. wR_2(int) was 0.1028 before and 0.0728 after correction. The ratio of minimum to maximum transmission is 0.8555. The λ/2 correction factor is 0.00150.	
T_{min}, T_{max}	0.822, 0.961	
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	181019, 30760, 25384	
R_{int}	0.071	
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.597	
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.070, 0.234, 1.07	
No. of reflections	30760	
No. of parameters	1310	
No. of restraints	156	
H-atom treatment	H-atom parameters constrained	
	$w = 1/[\sigma^2(F_o^2) + (0.1379P)^2 + 110.1P]$ where $P = (F_o^2 + 2F_c^2)/3$	
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	2.62, -1.22	
Absolute structure	Flack *x* determined using 10229 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, *Acta Cryst. B*69 (2013) 249-259). Hooft *y* (Hooft, Straver and Spek, *J. Appl. Cryst.* (2008) 96-103.). A three-hypothesis model based on Bayesian statistical analysis on Bijvoet pairs using a Student's *t*-distribution was employed to determine absolute configuration. *p*3(true) = 1.000, *p*3(false) = 0.0E+00, *p*3(rac-twin) = 0.3E-37	
Absolute structure parameter	Flack *x*: 0.18(3); Hooft *y*: 0.15(2); *p*3(true) = 1.000, *p*3(false) = 0.0E+00, *p*3(rac-twin) = 0.3E-37	

Void Analysis

Void volume and total void percentage (relative to unit cell volume) for the soaked crystalline sponges were determined using PLATON/SQUEEZE (A. L. Spek, *Acta Crystallogr., Sect. C.: Struct. Chem.*, 2015, **71**, 9) (note: all structures reported in this article are UNSQUEEZED and have not been treated with other residual electron density attenuation programs).

1·**2**: Void Volume: 2987 Å^3, Void Percentage: 17%

1·**3**: Void Volume: 2590 Å^3, Void Percentage: 31%

1·**4**: Void Volume: 4420 Å^3, Void Percentage: 28%

1·**5**: Void Volume: 7300 Å^3, Void Percentage: 42%