Electronic Supplementary Information

A Nonplanar 2D Covalent Organic Framework Derived from a Z-shaped Building Unit

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This work is dedicated to Prof. Susumu Kitagawa (Kyoto University) on the occasion of his 70th birthday

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Experimental Procedures

All the chemicals are commercially available, and used without further purification. All solvents were dried and distilled according to conventional methods, 4,4',4",4"'-(pyrene-1,3,6,8-tetrayl)tetraaniline (TAPPy)^[1] and 4',5'-bis(4-formylphenyl)-[1,1':2',1"-terphenyl]-4,4"-dicarbaldehyde (TPTF)^[2] were prepared in advance.

Powder X-ray diffraction (PXRD): PXRD patterns were collected on a Bruker D8 ADVANCE diffractometer using Cu Kα radiation.

Fourier transform infrared (FT-IR): IR spectrum was measured on a Thermo Fisher Scientific Optics NICOLETIS10 FT-IR spectrometer with Universal ATR accessory between the ranges of 4000 to 525 cm⁻¹.

Solution nuclear magnetic resonance (NMR): Liquid state 1H nuclear magnetic resonance spectroscopy were collected on a Bruker Avance III instrument with AS500 magnet equipped with a cryoprobe (500 MHz).

Solid-state nuclear magnetic resonance (ssNMR): All ssNMR spectra were recorded on a JEOL JNM-ECZ600R spectrometer at 14.1 T (¹H: 600 MHz) and 1.0 mm rotors. 1D ¹H and 2D ¹H-¹³C heteronuclear correlation ssNMR spectra were measured at MAS of 70 kHz and a recycle delay of 2.5 sec. $\pi/2$ pulse lengths of ¹H and ¹³C are 0.8 µs and 1.15 µs, respectively.

High resolution mass spectrometry (HRMS): HRMS mass spectra were collected on a Baird Acquity UPLC/XEVO G2-XS QTOF using CHCl₃ as a solvent.

Scanning electron microscope (SEM): SEM images were collected using a JSM-IT500HR system.

Transmission electron microscope (TEM): TEM images were obtained with a JEM-2100, JEOL.

Gas adsorption: Gas adsorption measurement N_2 adsorption and desorption measurements were performed at 77 K using ASAP 2020, Micromeritics Instrument Corp, USA. Pore size distributions and pore volumes were derived from the adsorption isotherms.

Thermogravimetric analysis (TGA): TGA was performed using a NETZSCH STA 449F5 under flowing N₂ with 10 K min⁻¹ ramp rate. Samples were heated in a Platinum pan (900 °C, 10 °C min⁻¹) under a N₂ flux (60 mL min⁻¹).

UV-vis spectroscopy (UV-vis): The UV-vis spectra of COF powders were collected using LAMBDA950, PerkinElmer, Germany. And the UV-vis spectra of monomers dispersing in CH₂Cl₂ were carried out by D-7PC, Nanjing Feile Instrument Co., Ltd.

Fluorescence measurement: The fluorescence spectra of samples were collected using RF-5301PC. As for fluorescence detection of PCP-COF with picric acid (PA), 5 mg COF powders were dispersed in 20 mL acetonitrile by ball-milling overnight to afford homogeneous acetonitrile suspensions. The fluorescence measurement of was carried out with the gradually adding 0 μ M, 10 μ M, 20 μ M, 30 μ M, 40 μ M, 50 μ M, 60 μ M, 70 μ M, 80 μ M, 90 μ M, and 100 μ M picric acid (100 μ g/mL in acetonitrile) in 2 mL acetonitrile COF suspensions after five minutes. The excitation wavelength used in the experiments was 410 nm, the width of excitation slit was 3.0 nm and launching slit was 3.0 nm, and the scan speed was 500 nm/min.

Results and Discussion

Synthesis of 4,5,12,13-tetrabromo[2.2]paracyclophane



4,5,12,13-tetrabromo[2.2]paracyclophane was synthesized according to the reported literature with a modified procedure.^[3] Bromine (87.3 g, 546.46 mmol) was added slowly to the 100 ml dry Schlenk flask containing a catalytic amount of iodine (150 mg, 1.18 mmol) at ice-water bath. Under agitation, [2.2]paracyclophane (10.0 g, 48.01 mmol) was added to the solution in batches. Then the reaction mixture was warmed up slowly and stirred at room temperature for a week. Add 50 mL dichloromethane to dilute the reaction solution. The reaction was quenched by cautiously adding saturated aqueous NaHSO₃ (150 mL) and extracted with CH₂Cl₂ (3×50 mL). The organic layer was washed with aqueous NaHSO₃ (3×35 mL) and water (3×35 mL) successively and then dried over MgSO₄. After removing the solvent under reduced pressure and the residue was obtained as a crude product. After extracted by column chromatography using petroleum ether as eluent, the pure product was obtained compound **1** as a white solid (11.3 g, 45%). ¹H NMR (500 MHz, CDCl₃): δ 7.00 (s, 4H), 3.34-3.38 (m, 4H), 3.08-3.12 (m, 4H).







PCP-CHO was synthesized according to Suzuki coupling. Compound **1** (523 mg, 1.0 mmol), *p*-formylphenylboric acid (898 mg, 6.0 mmol), Pd(PPh₃)₄ (138 mg, 0.12 mmol) and anhydrous K₂CO₃ (1.65 g, 12 mmol) were added in 100 mL dry Schlenk, and then the reaction system was vacuumed for 30 minutes, during which argon gas was pumped three times. Under the protection of argon, 30 mL anoxic tetrahydrofuran and 10 mL anoxic water were added. The mixture reaction was heated to 100 °C, and stirred for 48 hours. The mixture was extracted into DCM (3×50 mL) and the solvent was removed under reduced pressure. The crude product was washed with MeOH and extracted by column chromatography using DCM as eluent to afford off-white solid, PCP-CHO (505 mg, 81%). ¹H NMR (500 MHz, CDCl₃): δ 9.96 (s, 4H), 7.73 (m, 16 H), 6.81 (s, 4H), 3.05-3.09 (m, 4H), 2.96-3.00 (m, 4H). ¹³C NMR (125 MHz, CDCl₃): δ 191.83, 146.03, 138.93, 138.48, 134.43, 131.78, 129.11, 33.58.



Fig. S2 ¹H NMR spectra of PCP-CHO











Fig. S5 The crystal structures of PCP-CHO (CCDC 2088970).

Note: The two benzene rings are fixed by two ethylene chains, and the distance between the two benzene rings is approximate 3.01 Å, indicating the intramolecular π - π interaction. The proximity of the two benzene rings leads to structural distortions, which may provide unusual reaction behavior. In the solid state, PCP-CHO molecules exhibit fishbone-like accumulation with distinct intermolecular π - π stacking. Based on the unique Z-shaped structure of PCP-CHO, we assumed that a novel nonplanar stair-stepped COF structure could be constructed through the aldehyde group condensation with amines.

Synthesis of PCP-COF



PCP-CHO (31.2 mg, 0.05 mmol) and TAPPy (28.3 mg, 0.05 mmol) were weighed into a glass ampoule with odichlorobenzene (2.0 mL) and n-butyl alcohol (2.0 mL). The solution was ultrasound for 5 minutes to obtain yellow turbid solution. 6 M acetic acid (0.3 mL) was pure into the glass ampoule as catalyst. The glass ampoule was flash frozen at 77 K using the liquid nitrogen bath and degassed by freeze-pump-thaw three times, and then was sealed. The glass ampoule was placed at 120 oC for 3 days. The yellow solid was isolated by centrifugation and washed with MeOH (3×10 mL) and then filtered. The sample was then transferred to vacuum chamber and evacuated to 20 mTorr at 50 °C for 24 h, yielding yellow powder PCP-COF (Yield : 47.5 mg, 85%).



Fig. S6 Comparison of ¹³C CPMAS spectra for PCP-COF.



Fig. S7 ¹H detected ¹H/¹³C HETCOR spectrum for PCP-COF.

Synthesis of TPTF-COF



4',5'-bis(4-formylphenyl)-[1,1':2',1"-terphenyl]-4,4"-dicarbaldehyde (TPTF) (24.7 mg, 0.05 mmol) and TAPPy (28.3 mg, 0.05 mmol) were weighed into a glass ampoule with mesitylene (3.5 mL) and dioxane (0.5 mL). The solution was ultrasound for 5 minutes to obtain yellow turbid solution. 6 M acetic acid (0.3 mL) was pure into the glass ampoule as catalyst. The glass ampoule was flash frozen at 77 K using the liquid nitrogen bath and degassed by freeze-pump-thaw three times, and then was sealed. The glass ampoule was placed at 120 oC for 3 days. The yellow solid was isolated by centrifugation and washed with MeOH (3×10 mL) and then filtered. The sample was then transferred to vacuum chamber and evacuated to 20 mTorr at 50 °C for 24 h, yielding yellow powder TPTF-COF (Yield : 39.5 mg, 80%).



Fig. S8 FTIR spectra of (a) PCP-COF, (b) TAPPy, (c) PCP-CHO.



Fig. S9 FTIR spectra of (a) TPTF-COF, (b) TAPPy, (c) TPTF.



Fig. S10 PXRD patterns of PCP-COF, TAPPy and PCP-CHO.



Fig. S11 PXRD patterns of TPTF-COF, TAPPy and TPTF.



Fig. S12 TGA curve of PCP-COF.

Table S1. The comparison of specific surface area and pore size of nonplanar 2D COFs

Name	Structure type	Bond type	BET (m²/g)	Pore size (nm)	Reference
CTC-COF-2	undulated	B-0	541	2.52	Chin. J. Chem. 2016, 34,
CTC-COF-3	undunated	D U	1013	2.34	783-787
Marta-COF-1	Wavy	B-O	1300	3.5	J. Am. Chem. Soc. 2019, 141, 14403-14410
COF-NUST- 1	Wavy	B-O	410	2.4	J. Am. Chem. Soc. 2020 , 142, 21279-21284
CX4-BD-1	Bowl-shaped	C=N	~24	-	J. Am. Chem. Soc. 2021, 143, 3407-3415
QTD-COF-1	Colyliform	C=N	~3	1.36/1.72	Angew. Chem. Int. Ed. 2020 , 59, 22697 -22705
PCP-COF	Z-shaped	C=N	1123	1.2/1.5	This work

Experimental and Simulation X-ray Diffraction Patterns for COFs Structures



Fig. S13 (a) PXRD pattern of the as-synthesized PCP-COF (red), compared with the simulated PXRD pattern of the AB stacking model (blue), (b) The simulated structure of AB stacking.



Fig. S14 (a) PXRD pattern of the as-synthesized PCP-COF (red), compared with the simulated PXRD pattern of the concave-convex stacking model (blue), (b) The simulated structure of concave-convex stacking and (c) Lateral sketch of concave-convex stacking.



Fig. S15 PXRD pattern of the as-synthesized PCP-COF (red), compared with the simulated PXRD pattern of sql-AA stacking model (blue); (a, b) Stair-stepped structure, (c, d) Concaved-convex structure.

Topology	Framework energy (Kcal/mol)
Bex-AA stair-stepped	233.364
Bex-AB	532.098
Bex-AA concave-convex	478.918
Sql-AA stair-stepped	300.501
Sql-AA concave-convex	1155.337

Table S2. The energies for the 2D frameworks with steric hindrance

Table S3. Crystal structure data of PCP-CHO (CCDC 2088970)

Space group: P21/N				
Monoclinic;	Monoclinic; $a = 8.33$ Å, $b = 14.28$ Å, $c = 15.65$ Å; $\alpha = 90.0^{\circ}$, $\beta = 100.3^{\circ}$, $\gamma = 90.0^{\circ}$			
Atom	Х	у	Z	
Cl1	0.41362(16)	0.29127(10)	0.73275(9)	
Cl2	0.74904(17)	0.22539(9)	0.76690(9)	
O3	0.4630(4)	0.6256(2)	1.0045(2)	
O4	0.5501(4)	0.9730(2)	0.6170(2)	
C5	0.1301(4)	0.4174(3)	0.5853(2)	
C6	0.4722(5)	0.5346(3)	0.7109(3)	
C7	0.5453(5)	0.5570(3)	0.7947(3)	

C8	0.2846(5)	0.7153(3)	0.5890(2)
C9	0.5417(5)	0.7524(3)	0.5044(3)
C10	0.3011(5)	0.5289(3)	0.6888(2)
C11	0.2266(4)	0.4996(3)	0.5988(2)
C12	0.1040(4)	0.3785(3)	0.5024(3)
C13	0.4529(5)	0.5761(3)	0.8579(2)
C14	0.3373(4)	0.6450(3)	0.5381(2)
C15	0.2089(5)	0.5472(3)	0.7530(2)
C16	0.1344(4)	0.4289(3)	0.4319(2)
C17	0.1908(4)	0.5206(3)	0.4412(2)
C18	0.2556(4)	0.5525(3)	0.5260(2)
C19	0.1478(5)	0.5839(3)	0.3631(2)
C20	0.3584(5)	0.8023(3)	0.5985(2)
C21	0.0332(5)	0.3752(3)	0.6491(3)
C22	0.2830(5)	0.5713(3)	0.8366(3)
C23	0.4678(5)	0.6656(3)	0.4957(3)
C24	0.4893(5)	0.8212(3)	0.5562(3)
C25	0.5763(5)	0.9119(3)	0.5685(3)
C26	0.5330(6)	0.6026(3)	0.9463(3)
C27	0.6066(7)	0.3053(4)	0.7967(4)
H22	0.2184(5)	0.5845(3)	0.8794(3)
H15	0.0933(5)	0.5431(3)	0.7392(2)
H6	0.5374(5)	0.5231(3)	0.6681(3)
H7	0.6609(5)	0.5594(3)	0.8093(3)
H23	0.5058(5)	0.6193(3)	0.4606(3)
Н9	0.6292(5)	0.7654(3)	0.4748(3)
H20	0.3207(5)	0.8488(3)	0.6335(2)
H8	0.1957(5)	0.7029(3)	0.6178(2)
H12	0.0645(4)	0.3161(3)	0.4943(3)
H16	0.1166(4)	0.4005(3)	0.3761(2)
H19a	0.2260(5)	0.6368(3)	0.3690(2)
H19b	0.1600(5)	0.5485(3)	0.3103(2)
H21a	0.0279(5)	0.3064(3)	0.6410(3)
H21b	0.0903(5)	0.3880(3)	0.7090(3)
H25	0.6595(5)	0.9227(3)	0.5354(3)
H26	0.6488(6)	0.6015(3)	0.9587(3)
H27a	0.6452(7)	0.3701(4)	0.7902(4)
H27b	0.5992(7)	0.2957(4)	0.8585(4)

Table S4. Fractional atomic coordinates for simulated PCP-COF powders

Space group: P	Space group: <i>P1</i>			
3D triclinic; a =	= 25.10 Å, $b = 20.69$ A	Å, c = 7.52 Å; α = 121.4	$4^{\circ}, \beta = 130.9^{\circ}, \gamma = 89^{\circ}$	
Atom	X	у	Z	
C1	0.16457	0.11136	0.33677	
C2	0.17346	0.04965	0.16495	
C3	0.21288	0.07376	0.12357	
C4	0.24229	0.15962	0.24652	
C5	0.23223	0.22128	0.41152	

C6	0.19435	0.19744	0.45826
C7	0.28202	0.18327	0.19578
N8	0.30072	0.25828	0.26936
С9	0.33931	0.28412	0.22295
C10	0.34881	0.36145	0.27482
C11	0.38532	0.38785	0.22758
C12	0.41316	0.33783	0.12737
C13	0.40376	0.26027	0.07832
C14	0.36721	0.23353	0.12509
C15	0.45205	0.36462	0.07279
C16	0.46089	0.43898	0.09901
C17	0.43088	0.49271	0.1706
C18	0.84693	0.88357	-0.20499
C19	0.81715	0.79749	-0.32648
C20	0.77927	0.77365	-0.27975
C21	0.76921	0.83531	-0.11474
C22	0.79862	0.92117	0.00821
C23	0.83804	0.94528	-0.03317
C24	0.72948	0.81166	-0.064
N25	0.71078	0.73664	-0.13758
C26	0.67219	0.7108	-0.09117
C27	0.66269	0.63348	-0.14304
C28	0.62618	0.60708	-0.09581
C29	0.59834	0.6571	0.00441
C30	0.60774	0.73466	0.05346
C31	0.64429	0.7614	0.00669
C32	0.55945	0.63031	0.05899
C33	0.55061	0.55595	0.03277
C34	0.58062	0.50222	-0.03883
C35	0.8474	0.02368	-0.24525
C36	0.88886	0.1124	0.02284
C37	0.84868	0.15582	0.05202
C38	0.76633	0.11089	-0.18547
C39	0.72371	0.02213	-0.45725
C40	0.76436	-0.02129	-0.48687
C41	0.72822	0.15948	-0.13385
N42	0.65317	0.12328	-0.31816
C43	0.62016	0.17543	-0.25016
C44	0.66779	0.26692	0.00818
C45	0.63559	0.31746	0.07256
C46	0.55517	0.27772	-0.11969
C47	0.50731	0.18617	-0.37546
C48	0.53978	0.13521	-0.44082
C49	0.52189	0.33312	-0.05268
C50	0.53203	0.4076	-0.02082
C51	0.57134	0.42941	-0.06649
C52	0.1641	0.97125	0.37703
C53	0.24714	1.01622	0.61866
C54	0.28778	0.9728	0.58903

C55	0.24517	0.88404	0.31724
C56	0.16282	0.83911	0.07973
C57	0.12263	0.88253	0.10892
C58	0.28328	0.83545	0.26561
N59	0.35832	0.87164	0.44992
C60	0.39134	0.81949	0.38192
C61	0.47172	0.85972	0.57262
C62	0.50419	0.80875	0.50726
C63	0.45633	0.71721	0.25147
C64	0.37591	0.67747	0.05918
C65	0.3437	0.72801	0.12356
C66	0.48961	0.6618	0.18446
C67	0.47947	0.58733	0.15259
C68	0.44016	0.56552	0.19826
C69	0.52908	0.68192	0.13782
C70	0.51041	0.53462	0.07925
C71	0.48242	0.31301	-0.00604
C72	0.50109	0.46031	0.05253
C73	0.89113	0.97646	-0.26182
C74	0.94028	1.00284	-0.26827
C75	0.96885	0.94872	-0.34702
C76	0.96491	0.88067	-0.34781
C77	0.93333	0.86696	-0.26603
C78	0.88855	0.90783	-0.25422
C79	0.96739	0.82906	-0.11067
C80	0.97853	1.09575	-0.12494
C81	1.03297	0.89918	0.25672
C82	1.04411	1.16586	0.24245
C83	1.07817	1.12797	0.39781
C84	1.12295	1.0871	0.386
C85	1.12037	1.01846	0.39359
C86	1.07122	0.99209	0.40005
C87	1.04265	1.0462	0.47879
C88	1.04659	1.11425	0.47958

Table S5. Fractional atomic coordinates for simulated TPTF-COF powders

Space group: <i>P2/M</i>				
3D monoclinic;	3D monoclinic; $a = 19.49 \text{ Å}, b = 24.43 \text{ Å}, c = 4.28 \text{ Å}; \alpha = \gamma = 90^{\circ}, \beta = 79.6^{\circ}, \beta =$			
Atom	X	у	Z	
C1	-0.08045	-0.09908	0.00328	
C2	-0.13581	-0.10028	-0.17166	
C3	-0.17633	-0.14711	-0.17299	
C4	-0.15979	-0.19381	-0.01214	
C5	-0.10373	-0.19292	0.15392	
C6	-0.06825	-0.14502	0.17736	
C7	-0.20962	-0.23813	0.04438	
N8	-0.20252	-0.27398	0.25999	

C9	-0.26012	-0.30166	0.43938
C10	-0.24949	-0.35063	0.59126
C11	-0.30583	-0.37644	0.77924
C12	-0.37329	-0.35421	0.80263
C13	-0.37984	-0.30035	0.69993
C14	-0.32457	-0.27508	0.51226
C15	-0.43717	-0.38684	0.91039
C16	-0.44116	-0.44304	0.84936
C17	-0.39042	-0.47174	0.63833
C18	0.03694	-0.0496	-0.00802
H19	-0.14752	-0.06535	-0.30898
H20	-0.22064	-0.14675	-0.29716
H21	-0.09084	-0.22772	0.28953
H22	-0.03012	-0.1443	0.32815
H23	-0.25926	-0.23254	-0.03388
H24	-0.19811	-0.36895	0.56035
H25	-0.29734	-0.41501	0.89003
H26	-0.42803	-0.27767	0.75315
H27	-0.33196	-0.23397	0.43224
H28	-0.35122	-0.45122	0.46929
C29	-0.07212	0	0.02719
C30	-0.5	-0.64347	1
C31	-0.5	-0.52853	1
H32	0.5	1.31929	1.5
H33	1.12868	1	-1.06256



Fig. S16 SEM images of PCP-COF.



Fig. S17 SEM images of TPTF-COF.



Fig. S18 TEM images of PCP-COF.



Fig. S19 UV-vis DRS spectrum of PCP-COF and PCP-CHO in solid and PCP-CHO in CH₂Cl₂ solution.



Fig. S20 Fluorescence intensity of PCP-CHO in acetonitrile (a) and in solid (b) ($\lambda_{ex} = 350$ nm).



Fig. S21 Tindall effect for different standing time of PCP-COF and TPTF-COF.



Fig. S22 Tindall effect for different standing time of PCP-COF dispersion in acetone.



Fig. S23 Fluorescence intensity of PCP-COF in acetone, acetonitrile and NMP.



Fig. S24 Fluorescence intensity contrast of PCP-COF and TPTF-COF suspension in acetonitrile.

Considering the solubility of nitro compounds, we chose acetone, acetonitrile and NMP as dispersant. After standing for 12 hours, the fluorescence of the dispersion was measured. The fluorescence of the material in acetone and acetonitrile does not change, but in the polarity of NMP there is a red shift (Fig. S23). It may be that the enhanced polarity of the solvent reduces the energy of $\pi \rightarrow \pi$ * transition, resulting in the red shift of the fluorescence peak. However, after standing for 24 hours, the Dundall effect of PCP-COF dispersed in acetone disappeared and obvious aggregates appeared at the bottom (Fig. S22). Considering the dispersibility of the material and the purchased nitro compound acetonitrile standard solution, combined with literature research, we chose acetonitrile as the dispersant.



Fig. S25 Fluorescence quenching upon addition of 2,4-dinitrophenol (DNP) (0-100 µM) in acetonitrile



Fig. S26 Fluorescence quenching upon addition of *p*-nitrotoluene (NT) (0-100 μ M) in acetonitrile.



Fig. S27 Fluorescence quenching upon addition of *m*-dinitrobenzene (DNB) (0-100 μ M) in acetonitrile.



Fig. S28 Fluorescence quenching upon addition of nitrobenzene (NB) (0-100 μ M) in acetonitrile.



Fig. S29 Fluorescence quenching upon addition of 2, 4-dinitrotoluene (DNT) (0-100 µM) in acetonitrile.



Fig. S30 Fluorescence quenching upon addition of *p*-nitrophenol (NP) (0-100 µM) in acetonitrile.

Chemosenors	Fluorescence quenching (%)	$K_{\rm SV}$ (M ⁻¹)	References	
PCP-COF	80	4.1×10 ⁴	This work	
3D-Py-COF	75	3.1×10 ⁴	J. Am. Chem. Soc. 2016, 138, 3302-3305	
Py-Azine-COF	69	7.8×10 ⁴	J. Am. Chem. Soc. 2013, 135, 17310-17313	
COP-63	-	8.0×10^{4}	J. Mater. Chem. C, 2015, 3, 8490-8494	
TAPB-TFPB	80	5.9×10 ⁴	J. Mater. Chem. C. 2015, 3.	
TAPB-TFP	67	3.2×10 ⁴	7159-7171	

Table S6. Stern-Volmer quenching constants (Ksv) towards PA of different POP-based chemosenors

TfpBDH	63	2.6×10 ⁴	Chem. Sci., 2015, 6, 3931- 3939
PCTF-8	71	1.3×10 ⁵	J. Mater. Chem. A, 2016, 4, 13450-13457
TRIPTA	61.7	2.7×10^{6}	<i>RSC Adv.</i> , 2016, 6, 28047–28054
TPECz	76	6.4×10^{4}	Angew. Chem. Int. Ed. 2015, 54, 11540-11544
COP-301	99	2.6×10 ⁵	J. Mater. Chem. A, 2015, 3, 92-96
DTF	-	2.1×10 ³	<i>Talanta</i> , 2017, 165, 282-288.
COF-BABD-DB	79	5.7×10 ⁵	<i>Chem. Commun.</i> , 2018, 54,
COF-BABD-BZ	92.3	4.5×10 ⁵	2308-2311

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