Supporting Information

2D host-guest supramolecular chemistry for on-monolayer graphene emitting platform.

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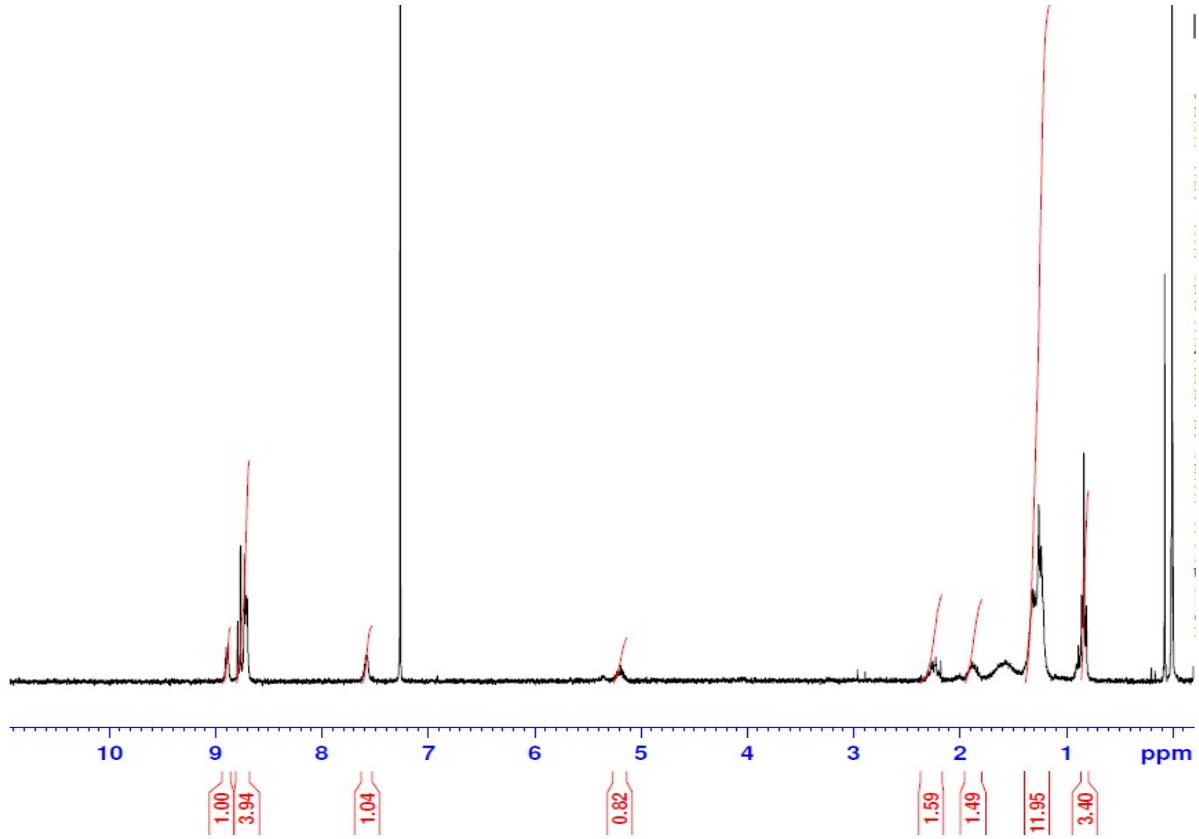
I. **Synthesis of *N*-(7-tridecan)-*N*'-(4-pyridine)-perylene-3,4,9,10-tetracarboxylic diimide (Py-PTCDI)**

Py-PTCDI was synthesized by the procedure described in the literature.[1] Briefly, a mixture of 7-amino tridecane (220 mg, 1.1 mmol) and 4-amino pyridine (103 mg, 1.1 mmol) was added into a 50 mL round-bottom flask equipped with a magnetic stir bar charged with perylene-3,4,9,10-tetracarboxylic dianhydride (PTCDA, 394 mg, 1.0 mmol) and imidazole (4.8 g, 70 mmol) and stirred at 130 °C for 2 h under an argon atmosphere. After cooling to room temperature, the mixture was extracted with dichloromethane (100 mL × 2) and washed with water (100 mL × 2). The combined organic layers were dried over anhydrous $MgSO_4$ and dried by rotary evaporator. The solid mixture containing dipyridyl PTCDI, dialkyl PTCDI, and the product isolated by silica gel column chromatography using dichloromethane/ethyl acetate (1:3) as an eluent to give a bright red solid (150 mg, 23%).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.85 (dd, 2H), 8.72−8.59 (m, 8H), 7.67 (dd, 2H), 5.17 (tt, J = 9.3, 5.7 Hz, 1H), 2.29−2.19 (m, 2H), 1.90−1.81 (m, 2H), 1.38−1.19 (m, 16H), 0.84 (t, J = 6.9 Hz, 6H); FTIR-ATR (solid, cm^{-1}): 2926, 2852, 1700, 1653, 1592, 1576, 1338; LC-MS (ESI, m/z) calcd for $[C_{42}H_{39}N_3O_4]^+$ (M^+), 649.2941; found, 649.2937.



^{1}H NMR spectrum of Py-PTCDI.

II. **Confocal microscopy**

The depth of field and focused area were calculated as Equation S1-S2:[2]

$$DoF = \frac{\lambda n}{2(NA_{obj})^2} \qquad \text{(Equation S1)}$$

Focused area = ~DoF/sin θ. (Equation S2)

where λ is the wavelength of excitation light, n is the refractive index of medium (1), NA_{obj} is the numerical aperture of the objective (0.4), and θ is the angle between lateral plane (stage) and the sample. According to the calculation, 53.7 times smaller area was focused at 490 nm.

III. STM image processing

STM data analysis and image processing were performed using WSxM software (WSxM 5.0 Develop 9.1)[3] and Gwyddion software (Version 2.55).[4]

Regarding the statistical analysis of the apparent height distribution, all experiments were repeated several times with different STM tips, different spots on the sample, and the results presented in this work are representative and consistent with the more comprehensive data set. In order to determine the average apparent height of brighter spots, assuming that they correspond to phthalocyanine molecules or to ZnPc: Py-PTCDI molecules, Gwyddion software is used. Thanks to this software, this analysis consists in averaging over the entire STM image, the different apparent height of the bright spots while defining with as much precision as possible the zero of the heights of the STM image corresponding ideally to an empty cavity of the nanoporous network.

IV. Self assembly on HOPG: STM characterization

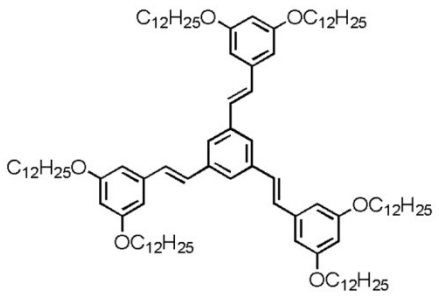
The first stage is the self-assembly of the TSB12 that forms a honeycomb network as evidenced in Figure S2. The extracted unit cell parameters of the honeycomb network ($a = b$ = 4.2 nm and α = 60°) are in agreement with our previous results on TSB12 networks.[23] Then, the solution of ZnPc:Py-PTCDI was drop-cast only once, expecting a low coverage for

the complex into the honeycomb network in order to observe the adlayer at an intermediate stage. Thanks to these host-guest system deposition conditions, STM reveals the host nanoporous molecular network with only some filled cavities (bright spots) as shown on a large-scale (200×200 nm^2) image (Figure S3a). The profile analysis (Figure S3b) extracted from this STM image along the white line AB (Figure S3a) gives an apparent height of 1.5 ± 0.1 nm and a periodicity of 4.2 nm. Further statistical analysis of the apparent height distribution of the spots reveals that the Gaussian fitting profile average distribution (Figure S3c) is 1.5 ± 0.2 nm. This value is much larger than the value for ZnPc adsorption into TSB12 network pores ($\sim 0.3 \pm 0.1$ nm) (Figure S4).

V. Self assembly on HOPG: Raman characterization

The HOPG spectrum (Figure S7a) exhibits two typical intense bands, the G-mode band (1579 cm^{-1}) and the 2D-mode asymmetric band (2717 cm^{-1}) with a shoulder at approximately 2675 cm^{-1}. Since most of the Raman bands of the conjugated molecular building blocks are in the 1000-1700 cm^{-1} region, we focused on this wavenumber range to compare the Raman spectra of HOPG surfaces at each stage of the host-guest system self-assembly (Figure S8). Starting from HOPG (Figure 8a), after the TSB12 deposition (Figure 8b) there is no change. No peak from the TSB12 monolayer is detected because we are far from its absorption wavelength (λ_{max} = 314 nm and $\lambda_{tail\text{-}end}$ = 354 nm). In contrast, subsequent deposition of the ZnPc:Py-PTCDI (1:100) system induces, before any rinsing, the appearance of the two bands at around 1300 and 1377 cm^{-1} (Figure 8c) that are matched to the vibronic coupling (~0.17 eV; ~1370 cm^{-1}) observed on UV-Vis spectra (Figure S5a). However, the characteristic PTCDI peak at 1580 cm^{-1} (C=C stretching) was not visible because of the large G-mode band of HOPG at 1579 cm^{-1}.[24, 25] After rinsing and elimination of Py-PTCDI excess, the two modes at 1300 and 1377 cm^{-1} are still visible with a lower intensity (Figure 8d). It is important to note that the PTCDI core emission is not quenched by the substrate (Figure 8d). Moreover, the

immobilization of the complex and the role of the ZnPc is clearly evidenced from Figure 8e and 8f recorded following the drop-casting, on the TSB12 modified substrate, of a Py-PTCDI solution, before and after rinsing, respectively. The Py-PTCDI Raman modes, visible just after deposition, completely disappeared after rinsing. Thus, all these results show that the ZnPc:Py-PTCDI could be trapped into the pores of the host TSB12 network on HOPG.



Scheme S1. Structure of TSB12

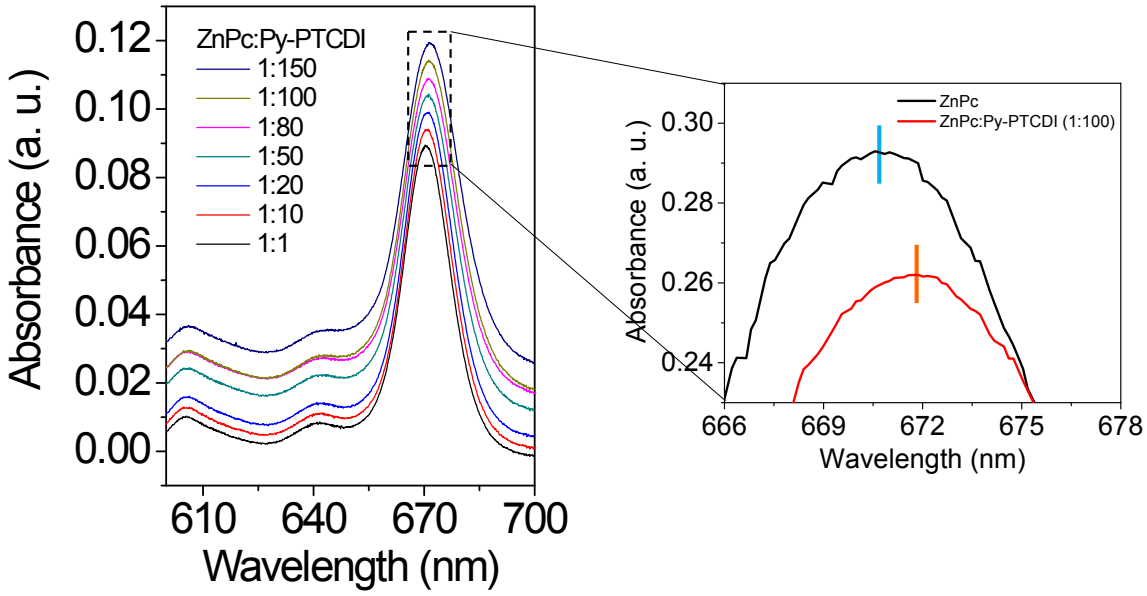


Figure S1. Evolution of absorption spectra of ZnPc in presence of different concentrations of Py-PTCDI ligand.

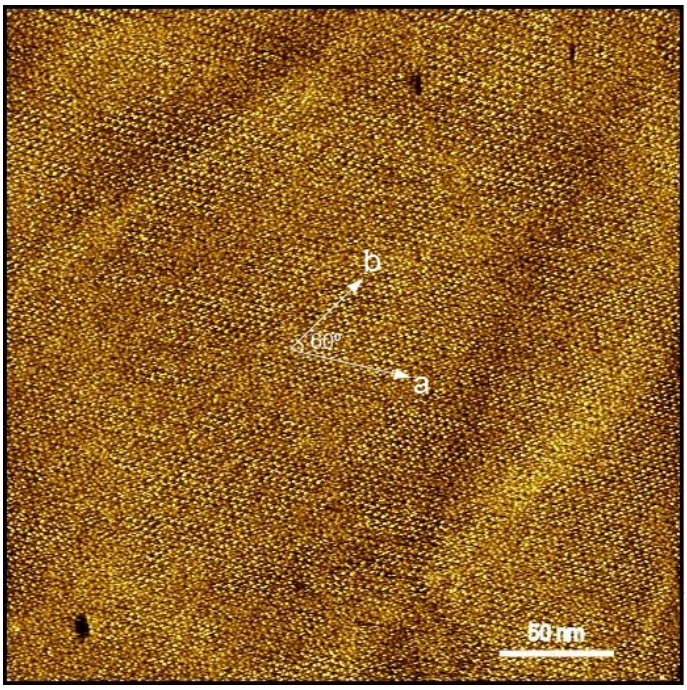


Figure S2. Large scale (300 × 300 nm^2) STM image of TSB12 self-assembled on HOPG; parameters a=b=4.2 nm, and α=60º; V_{bias}= -0.8 V and I_t= 16 pA.

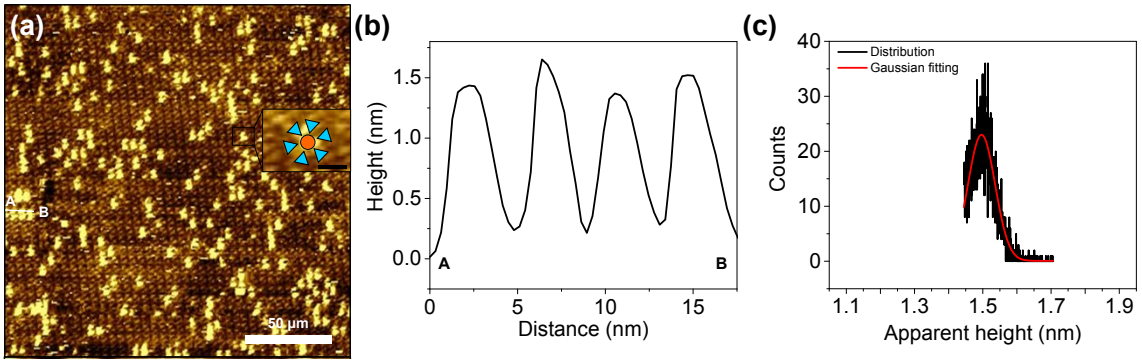


Figure S3. STM images and profiles on HOPG. (a) STM image at HOPG/phenyloctane interface of the ZnPc:Py-PTCDI (1:100) system drop-casted once and rinsed, onto the TSB12 honeycomb network. V_{bias}= -1.0 V and I_t = 13 pA. (b) Apparent-height cross section along the white line AB in (a). (c) The apparent height distribution and Gaussian fitting profile of the apparent height distribution for the spots of (a). The average distribution is 1.5±0.2 nm, $N(1.50, 0.08^2)$, p=0.95 where N and p are the normal distribution and the standard deviation, respectively. Inset on (a): the blue triangles indicate the TSB12 and orange dot indicates the ZnPc:Py-PTCDI. Scale bar is 3 nm.

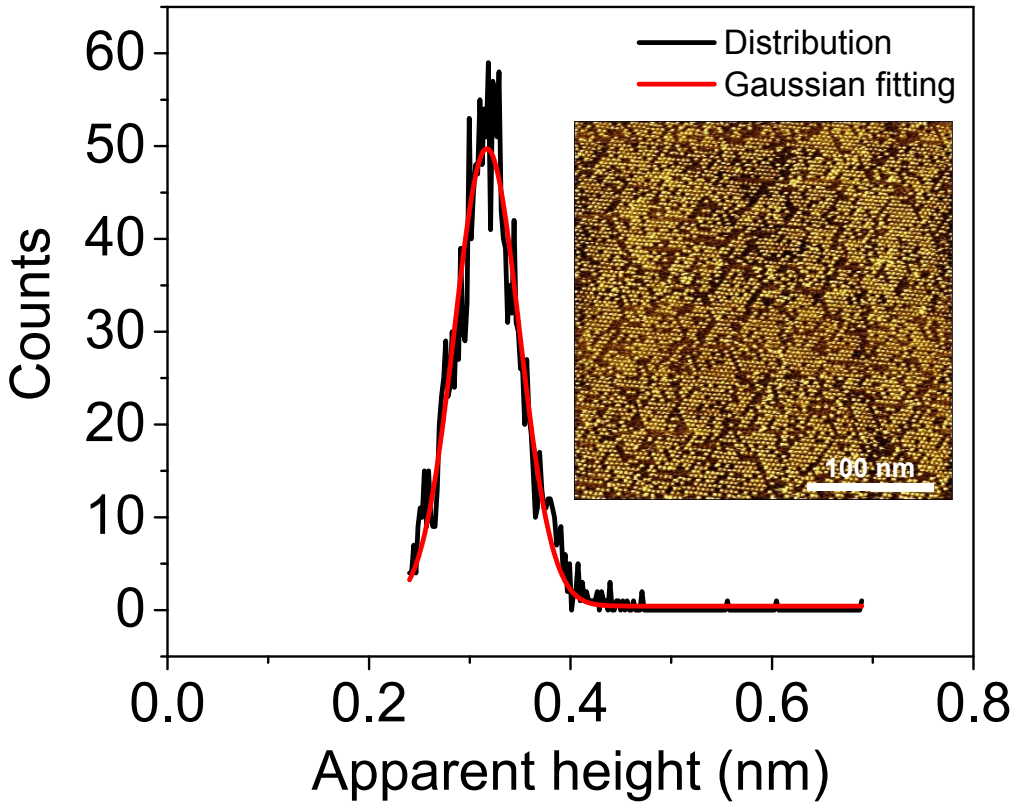


Figure S4. The apparent height distribution of ZnPc/TSB12/HOPG and Gaussian fitting profile of the apparent height distribution for the spots of the inset STM image. The average distributions is 1.5 ± 0.1 nm, $N(1.50,\ 0.06^2)$, p = 0.95, where N and p are the normal distribution and the standard deviation, respectively. Inset: STM image of ZnPc/TSB12/HOPG; parameters $a = b = 4.2$ nm, and $\alpha = 60°$; $V_{bias} = -1.1$ V and $I_t = 15$ pA.

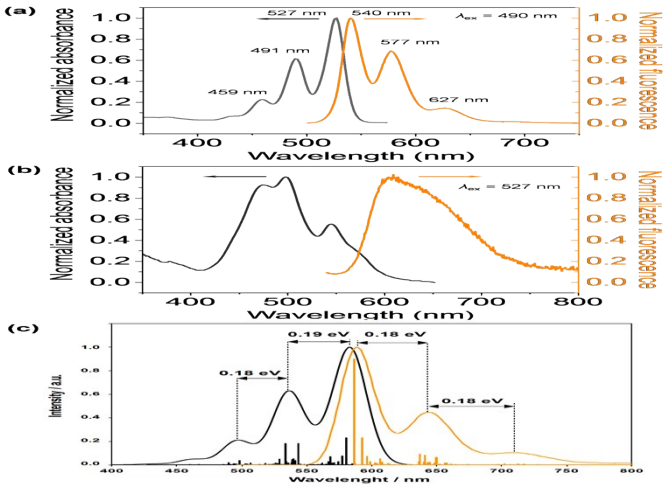


Figure S5. UV-Vis and fluorescence spectra of Py-PTCDI (a) solution in toluene (14 μM, ε_{527nm} = 50,000 M^{-1} cm^{-1}), (b) film and (c) computed. Black and orange lines correspond to absorption and emission spectra, respectively. Vertical sticks on (c) are the decomposition into vibronic transitions.

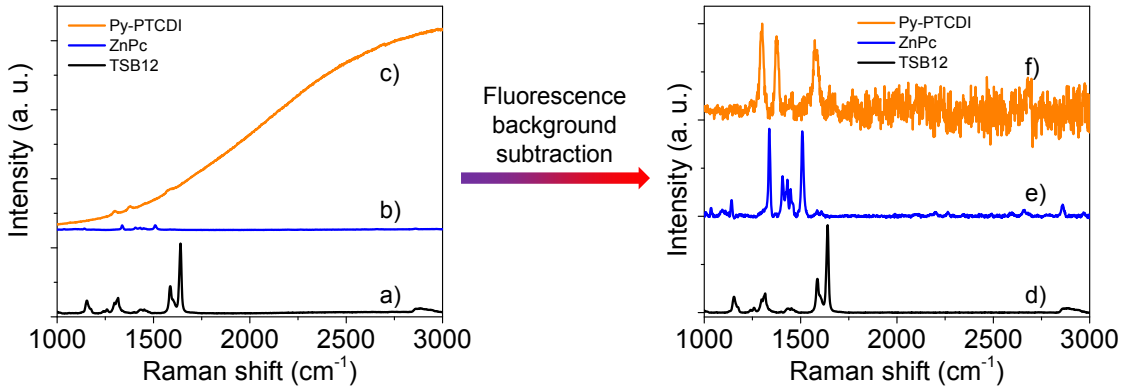


Figure S6. Raman spectra of films deposited onto a quartz substrate: (a,d) TSB12, (b,e) ZnPc, and (c,f) Py-PTCDI before and after subtraction of fluorescence background, respectively. Laser line at 532 nm.

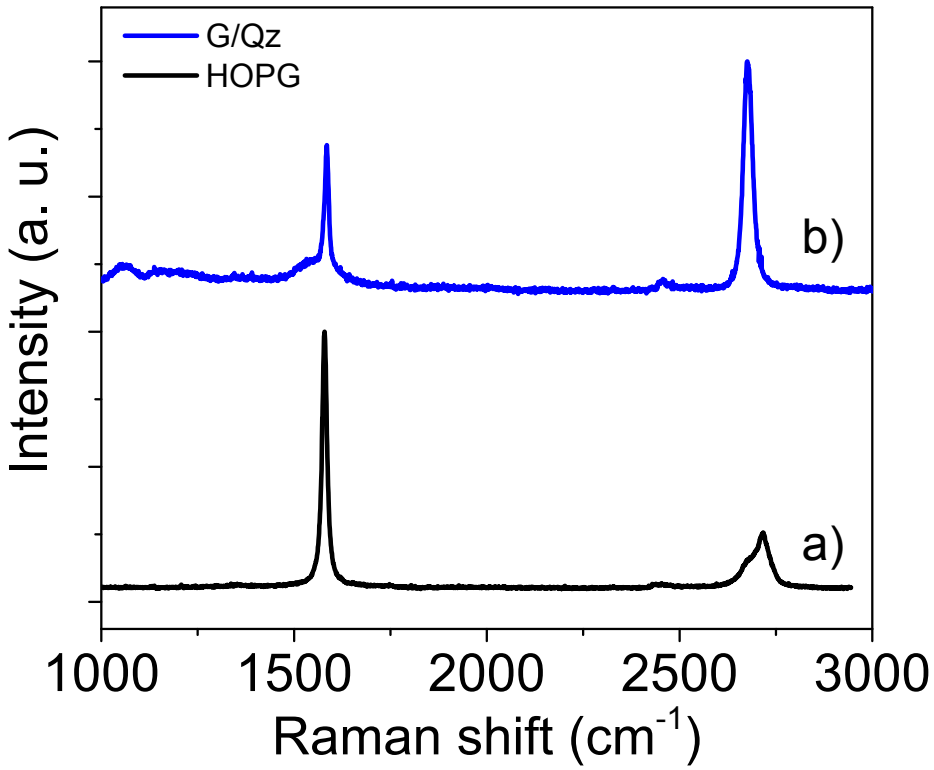


Figure S7. Raman spectra of (a) freshly cleaved HOPG and (b) monolayer graphene transferred on quartz. Laser line at 532 nm.

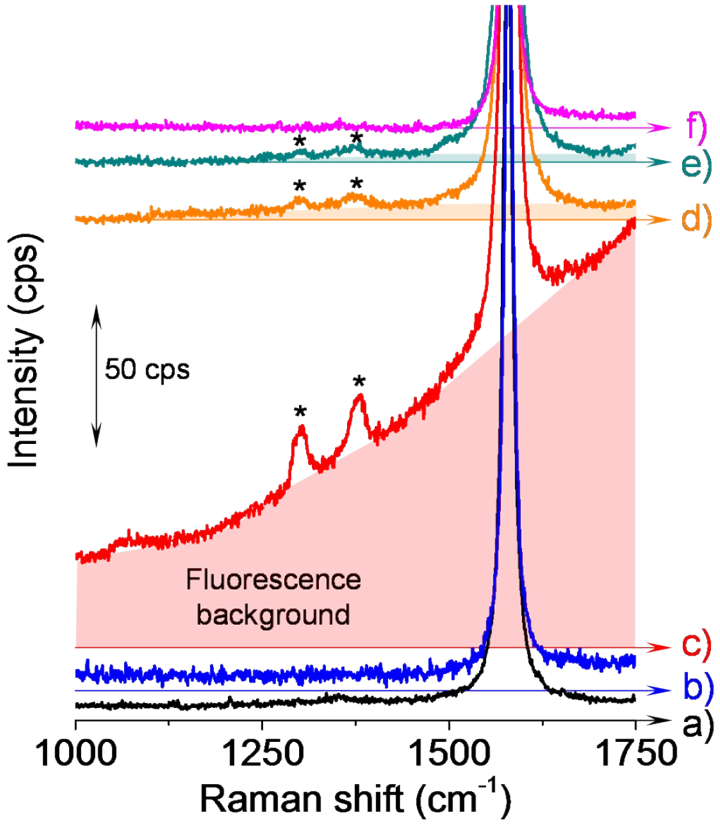


Figure S8. Raman spectra of freshly cleaved and functionalized HOPG surfaces. (a) freshly cleaved HOPG. HOPG with deposits of (b) TSB12, (c) subsequent addition of ZnPc:Py-PTCDI (1:100) before and (d) after rinsing. HOPG modified with TSB12-based network and subsequent addition of (e) Py-PTCDI (1:100) before and (f) after rinsing. Laser line at 532 nm. Asterisks show the characteristic Raman modes of Py-PTCDI, and one way arrows indicate the line with zero intensity values for each spectrum.

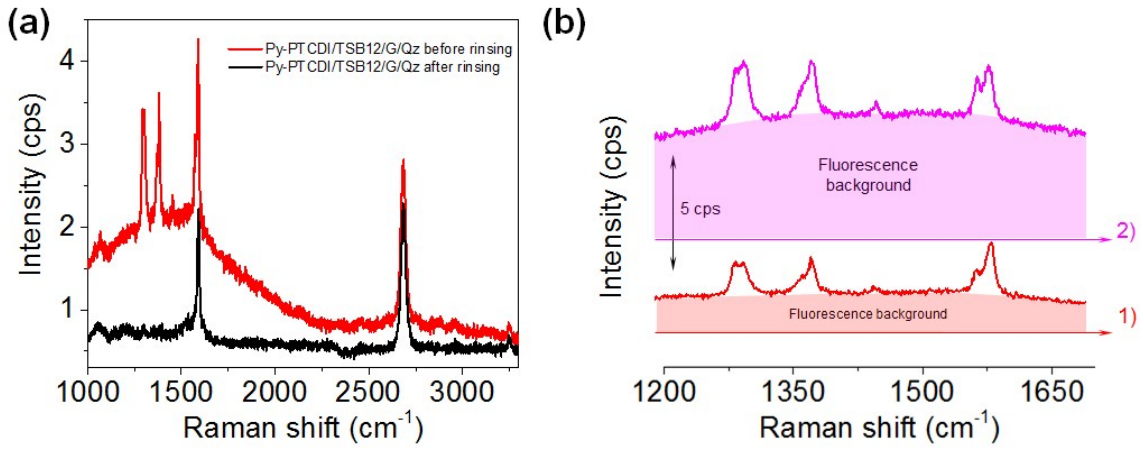


Figure S9. (a) Raman spectra of graphene with self-assembled TSB12 based network and subsequent addition of Py-PTCDI drop-casted before (red curve) and after rinsing (black curve). (b) Fluorescence background intensity comparison with and without ZnPc. Raman spectra of graphene with deposits of TSB12, subsequent addition of (1) Py-PTCDI drop-casted and before rinsing, (2) ZnPc:Py-PTCDI (1:100): drop-casted and rinsed.

Figure S9 b1 and b2 show the comparison of the Raman spectra, before subtracting fluorescence background, of PTCDI layer/aggregates on the surface (because no ZnPc) before rinsing and of our platform after rinsing, respectively. The fluorescence background on Figure S9 b2 is less intense than the one on Figure S9 b3.

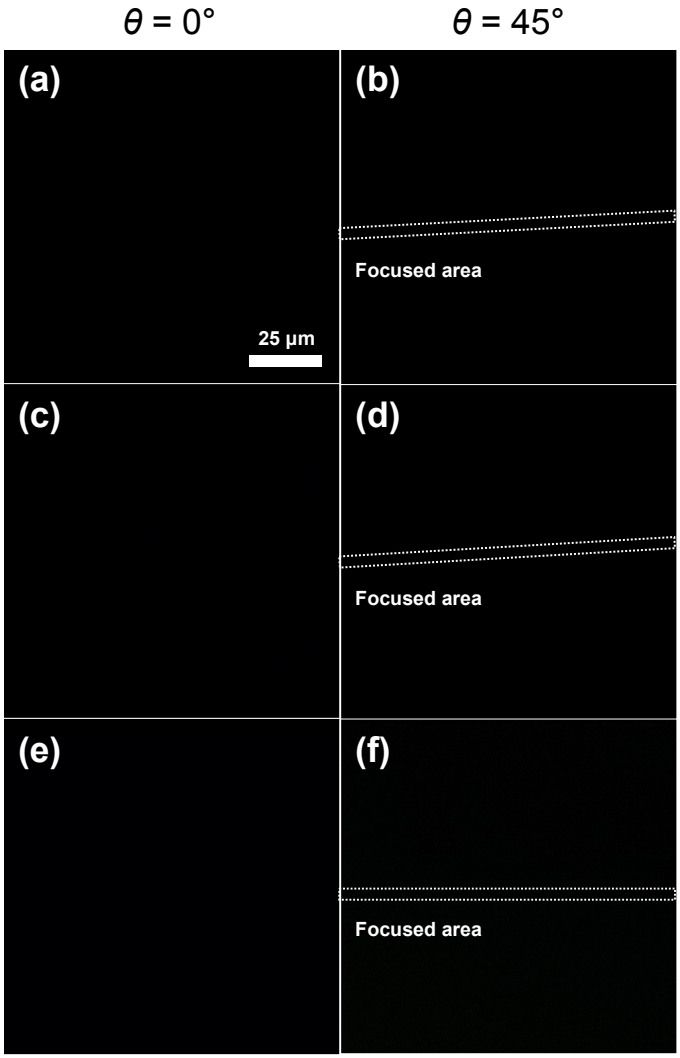


Figure S10. Fluorescence confocal microscopy images of graphene on quartz, (a) $\theta = 0°$ and (b) $\theta = 45°$, covered with the TSB12-based self-assembled network, (c) $\theta = 0°$ and (d) $\theta = 45°$, and subsequent addition of ZnPc, (e) $\theta = 0°$ and (f) $\theta = 45°$. Excitation wavelength 490 nm.

STM simulated images were obtained by plotting the charge density inside a plane perpendicular to the z-direction of the slab, placed 2 Å above the uppermost atom of the system from a charge density file (cube format) generated by CP2K integrating all monoelectronic states included in 1 eV window energy starting from the last occupied level (thus corresponding to a −1 V bias in STM).

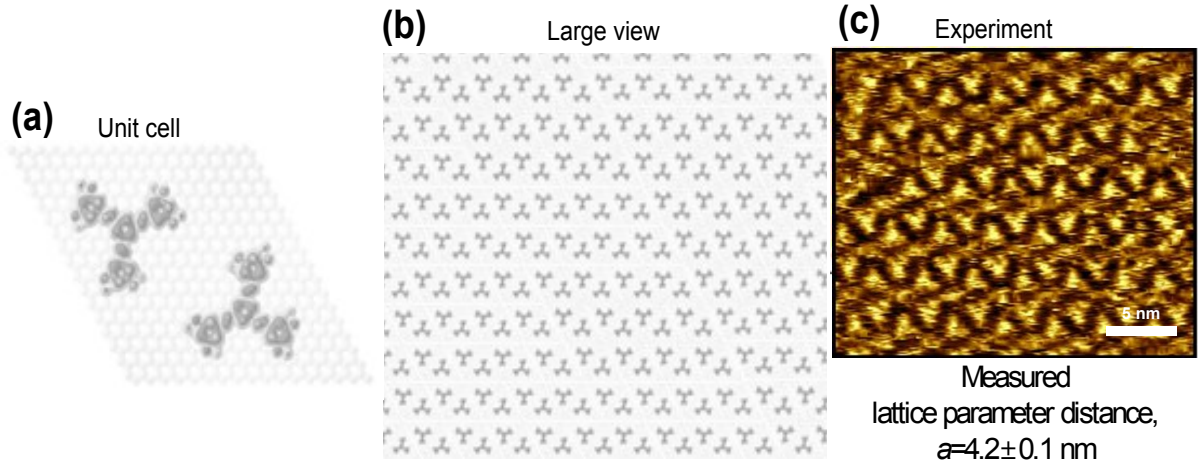


Figure S11. (a,b) Two different view of the simulated STM image. (c) Experimental STM image.[5]

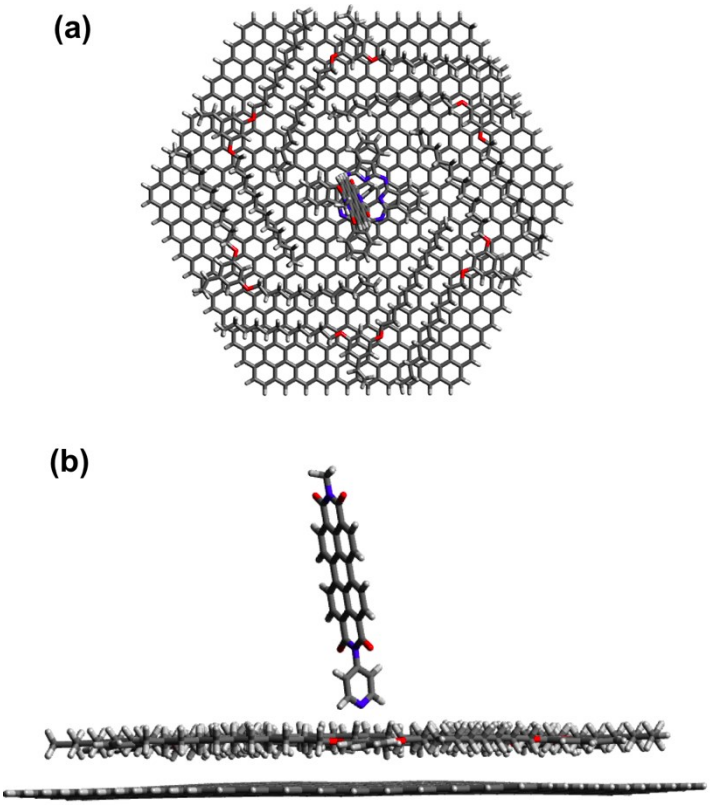


Figure S12. (a) Top and (b) side view of the cluster used to simulate the Mulliken point charges.

VII. Geometry obtained from the DFT optimization (in POSCAR format).

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ZnPc:Py-PTCDI-Graphene-TSB
   1.00000000000000
    36.2833683313999984  -20.9482124732999999    0.0000000000000000
     0.0000000000000000   41.8964249464999980    0.0000000000000000
     0.0000000000000000    0.0000000000000000   35.0000000000000000
   C  C   H   N  O  Zn
   578 268 371 11 16 1
Direct
 0.041237  0.017744  0.032004
 0.041227  0.076556  0.030902
 0.041208  0.135363  0.030039
 0.041185  0.194174  0.029542
 0.041176  0.253013  0.027942
 0.041163  0.311808  0.025056
 0.041150  0.370597  0.022259
 0.041131  0.429414  0.020954
 0.041138  0.488239  0.021763
 0.041161  0.547073  0.023774
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 0.041224  0.841250  0.033715
 0.041240  0.900108  0.033303
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1.049786  1.038748  0.138943
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