

Supporting Information

Precise Mono-Selective Aromatic C-H Bond Activation by Chemisorption of *Meta*-Aryne on a Metal Surface

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(1) Structure analysis and yield statistics

(a) STM image of 4',6'-dibromo-*meta*-terphenyl (DBMTP) on Cu(111) at 300 K

The deposition of DBMTP onto Cu(111) surface leads predominantly to the formation of organometallic dimers, as shown by Figure S1a. The magnified STM image of the dimer shows a protrusion between the two wing-shaped motifs. This protrusion is assigned to a Cu atom in a C-Cu-C bridge. For the STM image in Figure 1b, the absence of such bright protrusions at the debrominated carbon sites indicates their binding to substrate lattice Cu atoms rather than to Cu adatoms.

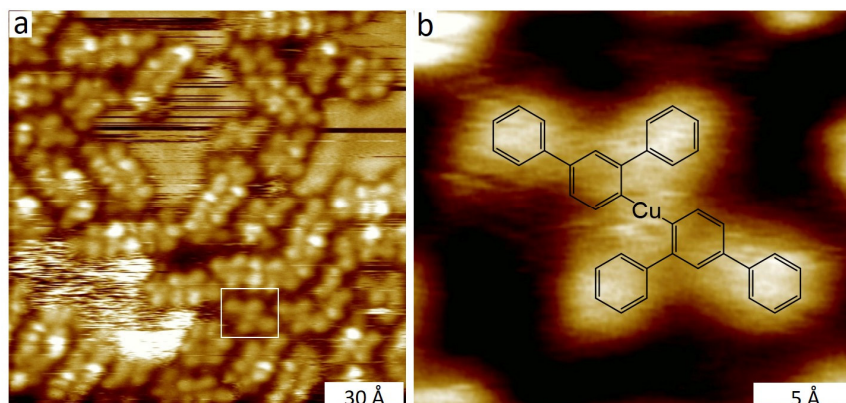


Figure S1. (a) Overview STM image taken after deposition of 4',6'-dibromo-*meta*-terphenyl (DBMTP) onto Cu(111) at 300 K. (b) Zoom-in of the white framed region in panel (a). Tunneling parameters: (a),(b) $I = 0.11$ nA, $U = 1.79$ V.

(b) STM image and molecular model of the *meta*-terphenyl biradical island on Cu(111) at 400 K

As shown by Figure S2b, the *meta*-terphenyl (MTP) biradicals are surrounded by bromine adatoms chemisorbed on the Cu(111) surface. The distance between the MTP biradical molecule and an adjacent Br adatom is measured to be 5.4 Å (shown in Figure S2c, the distance between the black dotted lines). Therefore, the distance between the carbon radical and the Br adatom is estimated to be $5.4 - 1.4$ (carbon atom diameter) = 4.0 Å. With the assumption that the radical carbon binds to a copper atom (red sphere in Figure S2d, C-Cu bond length of 1.97 Å),¹ the distance between the copper atom and its nearest Br adatom is estimated to be $4.00 - 1.97 = 2.03$ Å. However, this distance is much smaller than the Cu-Br bond length range (2.37 to 2.59 Å),² which means the space between the MTP biradicals and the Br adatoms is not sufficient for the insertion of an additional copper atom.

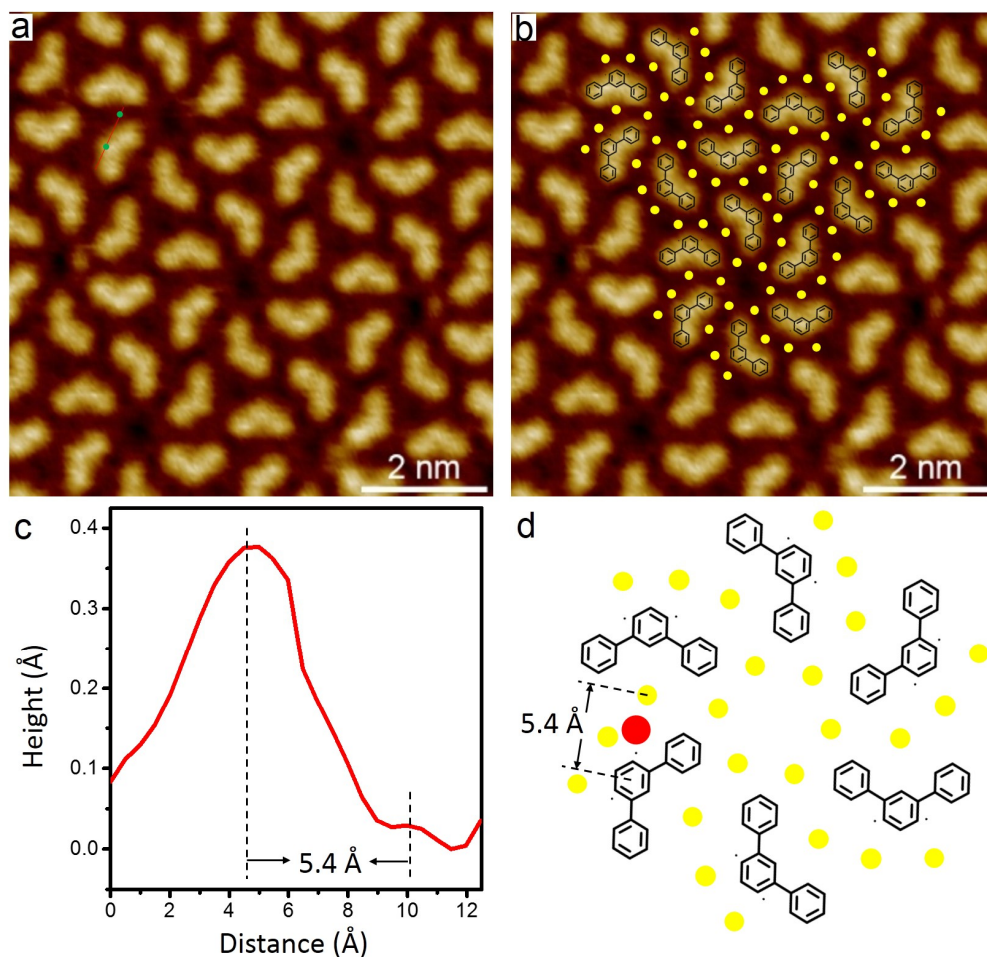


Figure S2. (a) STM image of the *meta*-terphenyl (MTP) biradical domain. (b) The STM image in panel (a) overlaid with molecular structures. (c) Apparent height profile along the red line in panel (a) from bottom to top. The green dots in panel (a) mark the centers of a MTP biradical and the Br adatom. (d) Molecular model for the MTP biradical island, showing the distance between the bromine adatom and the MTP biradical. The yellow and red spheres represent bromine and copper atoms, respectively.

(c) STM image of the organometallic dimer formed from 5'-bromo-*meta*-terphenyl (BMTP) on Cu(111) at 300 K

The deposition of BMTP onto the Cu(111) surface at 300 K is expected to result in the formation of organometallic dimers. As shown by Figure S3, the bright protrusion between the two wing-shaped motifs is assigned to the bridging Cu atom in C-Cu-C bond. The experimental distance between the terminals of the two wings is measured to be 1.08 nm, which is much larger than the corresponding distance of 0.85 nm for the butterfly-shaped species in Figure 1e in the manuscript. In addition, the butterfly-shaped species in Figure 1e are of uniform apparent height. Therefore, they are attributed to covalently linked dimers.

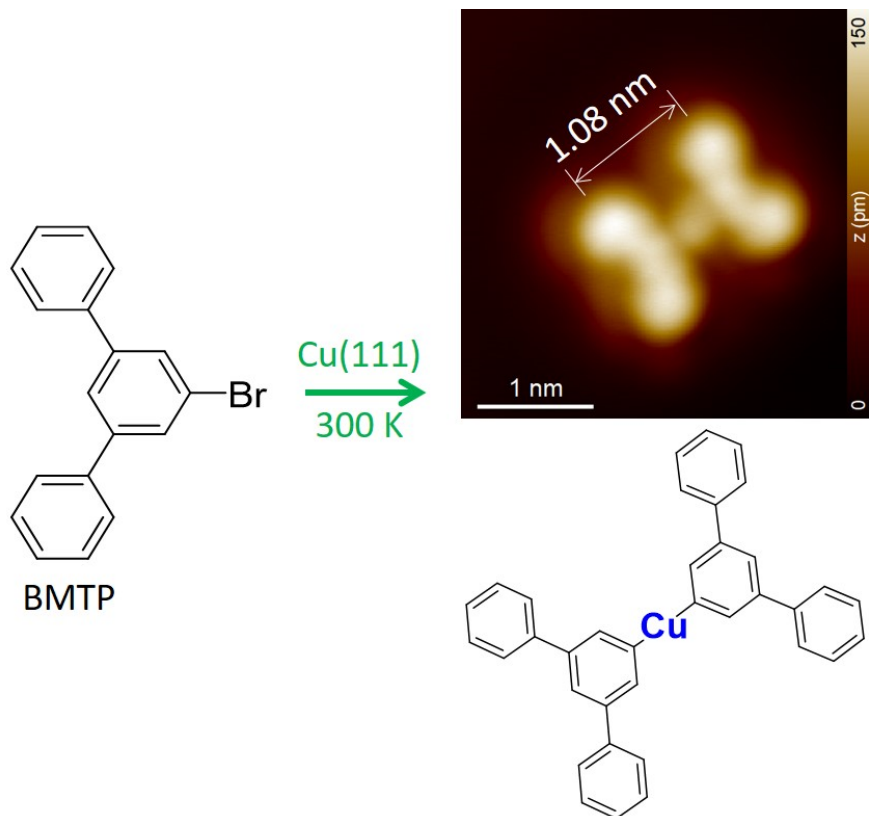


Figure S3. STM image and the corresponding chemical structure of the organometallic dimer species formed after deposition of 5'-bromo-*meta*-terphenyl (BMTP) onto Cu(111) at 300 K. Tunneling parameters: $I = 0.15$ nA, $U = -0.50$ V.

(d) Large-scale STM images of 5',5''-diphenyl-*meta*-quaterphenyl biradical (DMQBR) molecular islands

From an analysis of the four images below, we find a yield of $89 \pm 3 \%$ for DMQBR. This yield is defined as the number (8701) of *meta*-terphenyl (MTP) units in all the DMQBR molecules divided by the total number (9751) of MTP units in all the species. The errors stem from the number of the adsorbates with unclear shapes, which are difficult to assign to one of the two categories.

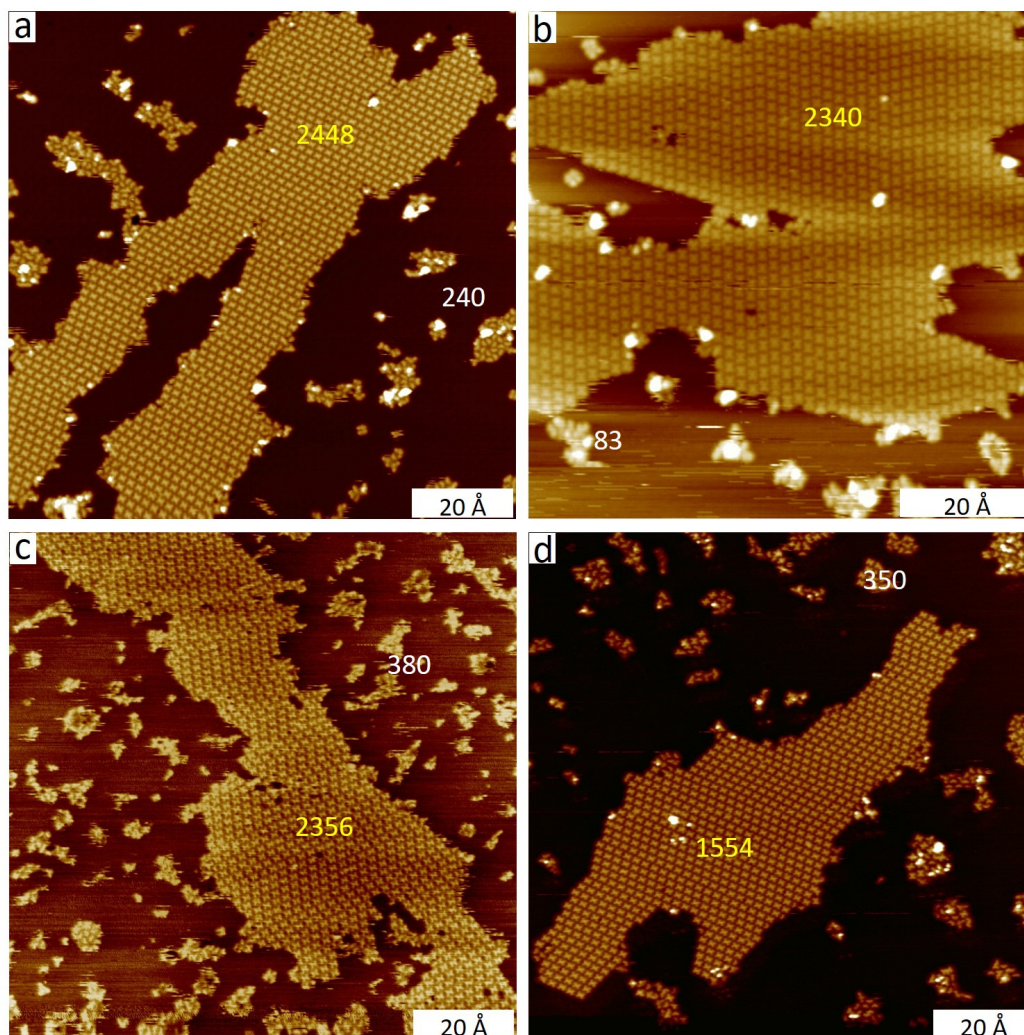


Figure S4. Large-scale STM images of the 5',5''-diphenyl-*meta*-quaterphenyl biradical (DMQBR) molecular island. The number of the *meta*-terphenyl (MTP) units inside and outside the DMQBR islands are given with yellow and white numbers, respectively. Tunneling parameters: (a) $I = 0.12$ nA, $U = -3.2$ V; (b) $I = 0.09$ nA, $U = -3.0$ V; (c) $I = 0.11$ nA, $U = -2.8$ V; (d) $I = 0.09$ nA, $U = -3.2$ V.

(e) STM images of the species formed after annealing of 5',5''-diphenyl-*meta*-quaterphenyl biradical (DMQBR) molecular islands to 480 K

Figure S5a shows the corresponding STM image after annealing the sample of DMQBR **2** to 480 K. Besides the biradical **2** that remains unchanged (at the bottom part), we observed the predominant formation of another species as marked by green arrows in Figure S5a. The distance between the two terminals of this species is measured to be 13.6 Å. Therefore, by size and shape consideration, we assign them as species **S1** with four- and eight-membered rings as shown by the molecular structure in Figure S5e. This means the higher temperature of 480 K triggers the intramolecular C-C coupling at the carbon radical sites and dehydrogenative coupling at the four C-H groups shown in green (Figure S5d). The relative high yield of species **S1** forming from DMQBR also indicates that scission of these four C-H bonds (green) is favored over the scission of the other bonds. Otherwise, intermolecular dehydrogenative coupling (such as the scarcely observed species marked by the red arrow in Figure S5a) should dominate, because of the larger number of other C-H bonds that can be activated. The reason for the preferential activation of the four green C-H groups is most likely related to the cooperative effects previously reported for the conversion of poly-anthracene into a graphene nanoribbon.³

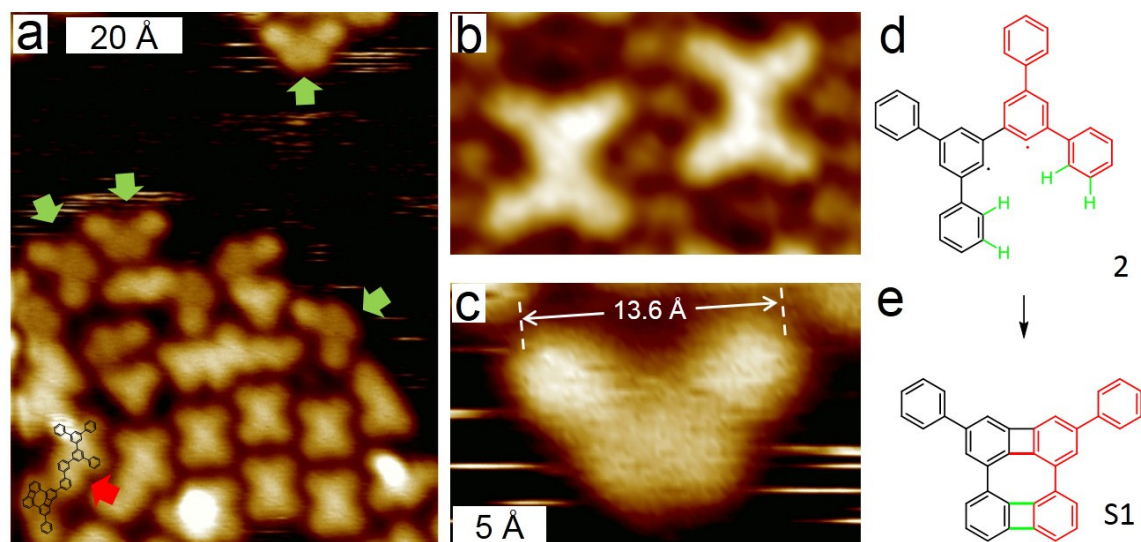


Figure S5. (a) STM image taken after annealing the sample in Figure 1d (biradical **2**) to 480 K. The green arrows mark the main product **S1** formed from biradical **2** by intramolecular C-C coupling. The red arrow marks the species formed by intermolecular dehydrogenative coupling between the biradical **2** and species **S1**. (b), (c) Zoom-in STM images of the biradical **2** and species **S1**. (d), (e) Chemical structures of the biradical **2** and species **S1**.

(f) Additional STM and non-contact (nc)-AFM images of an island of 3',5'-dibromo-*ortho*-terphenyl (DBOTP)

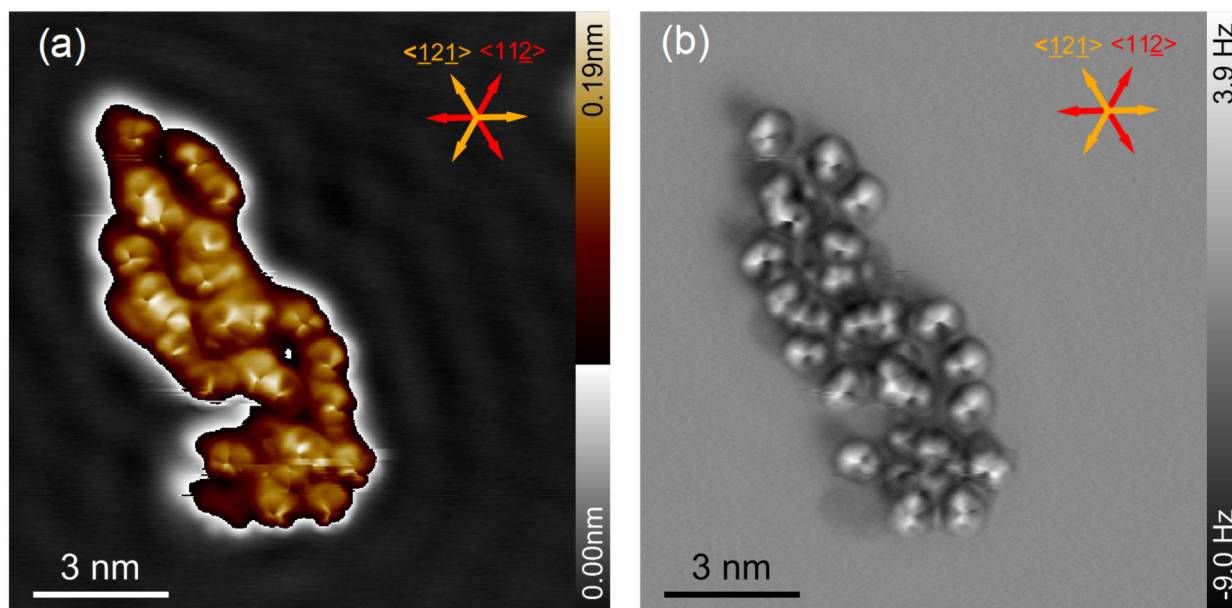


Figure S6. (a) STM and (b) nc-AFM images taken after deposition of a submonolayer of DBOTP 3 onto Cu(111) at <100 K. In the nc-AFM image, the individual molecules can more easily be discerned than in the STM image.

(g) C 1s and Br 3p X-ray photoemission (XP) spectra of 3',5'-dibromo-*ortho*-terphenyl (DBOTP) on Cu(111) annealed at different temperatures

According to the Br 3p XP spectrum at 160 K, most of the C-Br groups remain intact, as indicated by the Br 3p_{3/2} binding energy (BE) of around 184 eV for the most intense signal. Annealing the sample to 270 K leads to the complete dissociation of all C-Br bonds, as indicated by the Br 3p_{3/2} BE of around 182 eV. Thus, it is reasonable to conclude that the DBOTP molecules deposited on the Cu(111) surface at the much lower temperature of 5 K stay intact.

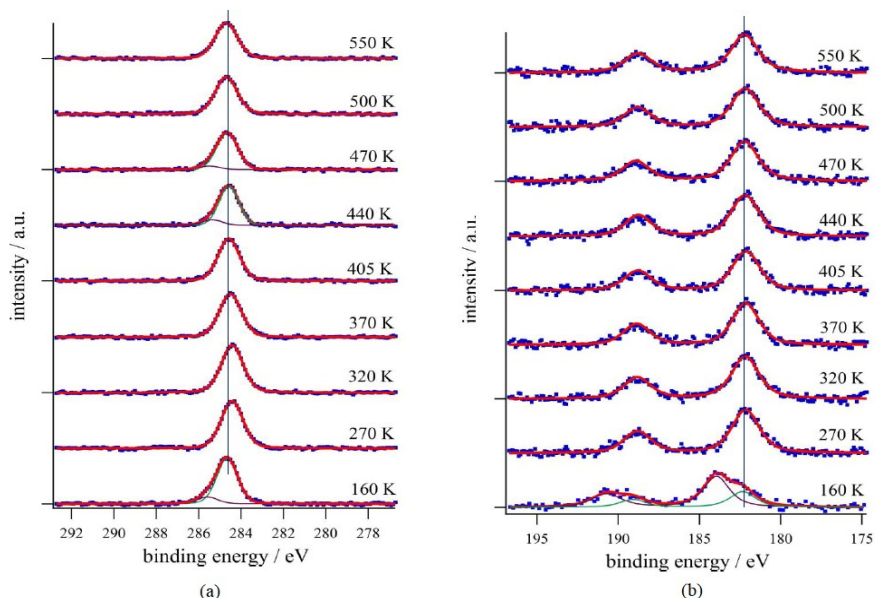


Figure S7. (a) C 1s and (b) Br 3p X-ray photoemission (XP) spectra of 3',5'-dibromo-*ortho*-terphenyl (DBOTP) on Cu(111) annealed at different temperatures as labeled. The vertical line marks the peak positions.

(h) Large-scale STM images taken after deposition of a submonolayer of DBOTP onto Cu(111) at 430 K

The yield of 2,3,6,7,10,11-hexaphenyl-triphenylene (HPTP) is defined as the number (729) of *ortho*-terphenyl (OTP) units in HPTP divided by the total number (855) of OTP units in all the adsorbates. The resulting yield is $86 \pm 5\%$. The errors again stems from the number of the adsorbates with unclear shapes, which are difficult to assign to one of the two categories.

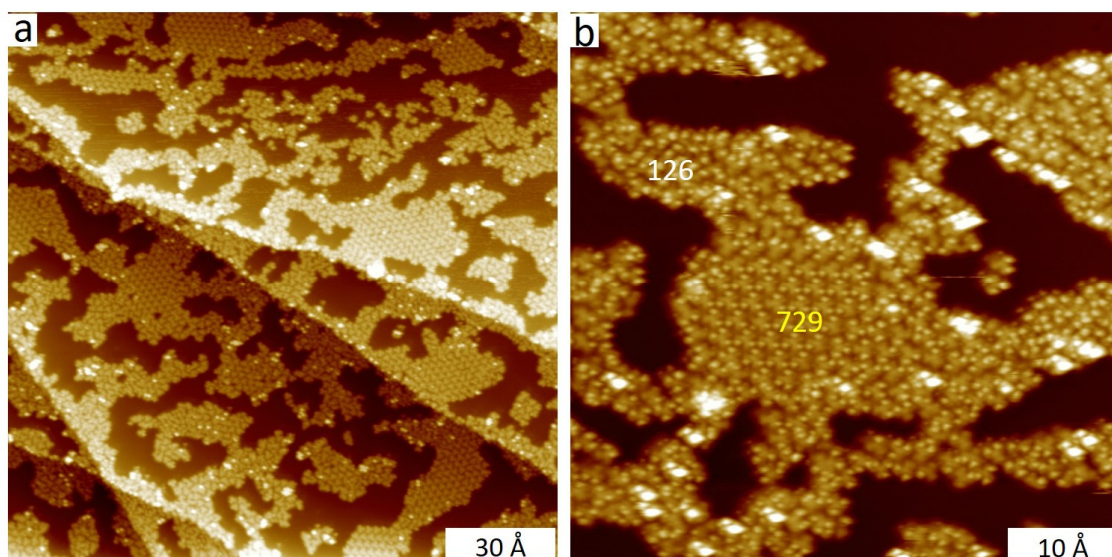


Figure S8. (a) Large-scale STM images taken after deposition of a submonolayer of DBOTP onto Cu(111) at 430 K. (b) Zoom-in STM image of the sample. The numbers of the OTP units in HPTP molecules and the by-products are labeled by yellow and white numbers, respectively. Tunneling parameters: (a) $I = 0.03$ nA, $U = 1.8$ V; (b) $I = 0.0$ nA, $U = 2.0$ V.

(i) Enlarged nc-AFM image of hexabenz[o,a,c,k,m,u,w]trinaphthylene (HBTN) 5

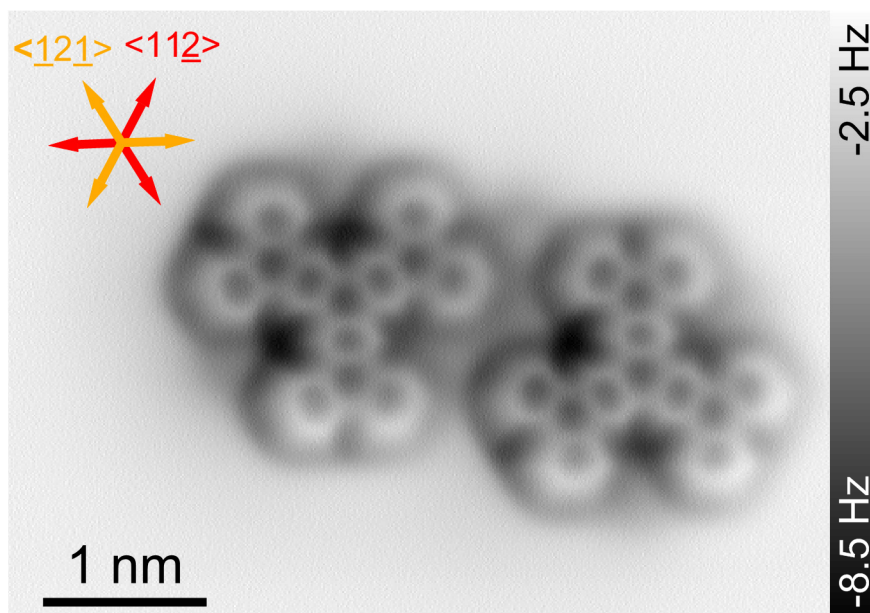


Figure S9. NC-AFM image of hexabenz[o,a,c,k,m,u,w]trinaphthylene (HBTN) **5**, taken at 5 K.

(j) STM images taken after annealing the sample in Figure S8 to 470 K

As can be seen, most of the trimer species are flat. They are assigned to hexabenzotriphenylene (HBTN) molecules formed by the complete cyclodehydrogenation of HPTP molecules in Figure S8. The proportion of the cyclodehydrogenated OTP units, *i.e.*, triphenylene, is $74 \pm 3\%$. This number is obtained via dividing the number (259) of the flat triphenylene units by the total number (351) of OTP and triphenylene units.

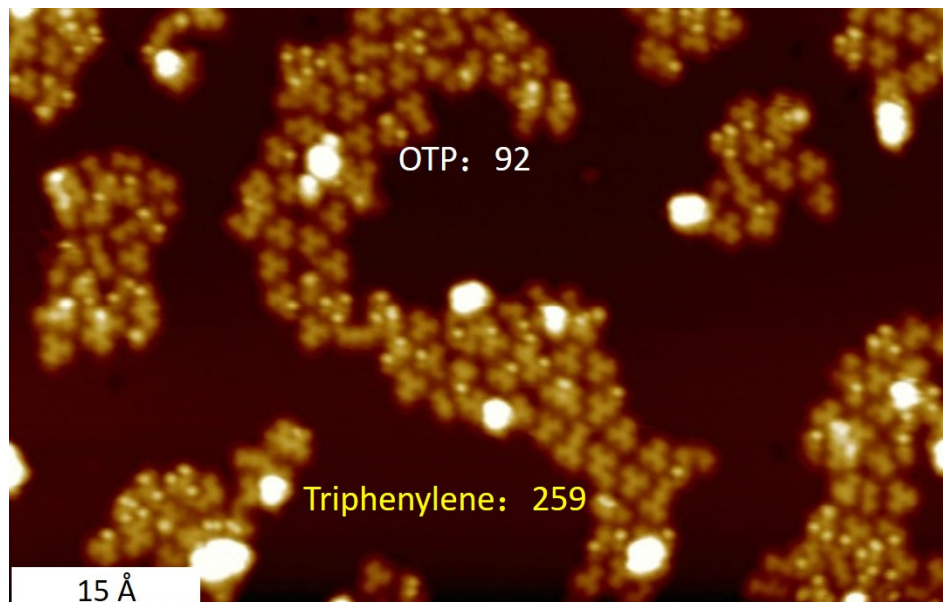


Figure S10. (a) STM images taken after annealing the sample in Figure S8 to 470 K. The number of the OTP and triphenylene units are labeled with white and yellow numbers, respectively. Tunneling parameters: (a) $I = 0.10$ nA, $U = -1.9$ V.

(2) Additional results from the DFT calculations.

(a) Optimized geometries and transition states for 1,1':3',1''-terphenyl (TP) **6** and its radical species **6-2r** on a Cu(111) slab in two different orientations

Two different orientations of the molecules with respect to the underlying Cu(111) surface lattice were considered as shown in Figure S11. In one orientation (denoted by X, Figure S11 a-f), the C-H bond to be activated (cf. Scheme 2 of the main paper) and the principal (C_2) symmetry axis of the molecule are parallel to a close-packed direction of the Cu(111) lattice (*i.e.*, parallel to a $\langle\bar{1}01\rangle$ direction in Figure S11 a-f). In the second orientation (denoted by Y, Figure S11 g-l), the target C-H bond and the molecular C_2 axis are perpendicular to the main crystallographic direction (*i.e.*, parallel to a $\langle\bar{1}\bar{2}1\rangle$ direction of the Cu(111) lattice). Hence, the two orientations considered can formally be transformed into each other via rotating the molecules by 90° around the surface normal. Figure 3 of the main text shows a subset of these structures.

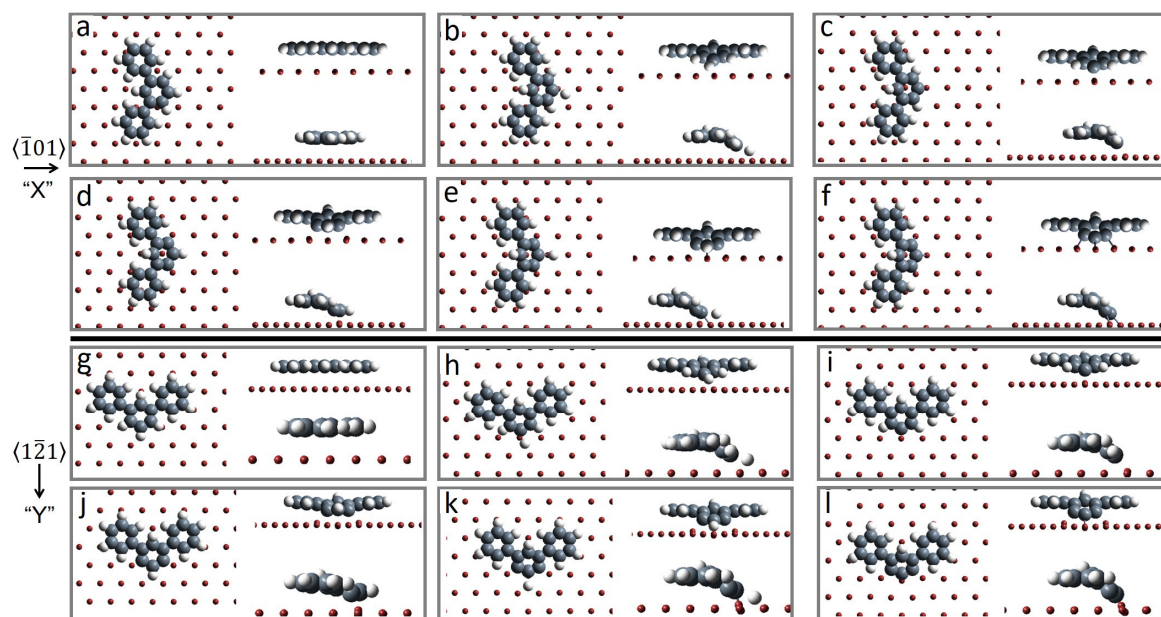


Figure S11. Optimized minimum and transition state geometries for 1,1':3',1''-terphenyl (TP) **6** (a-c, g-i) and its radical species **6-2r** (d-f, j-l) on a Cu(111) slab in two different orientations (top two rows / bottom two rows: target C-H bond parallel ("X") / perpendicular ("Y") to a high-symmetric direction of Cu(111)). Three different views are given for each: (a,g) intact 1,1':3',1''-terphenyl **6**, (b,h) transition structures for C-H activation from **6**, (c,i) monoradical product **6-1r**, (d,j) biradical **6-2r**, (e,k) transition structure for C-H activation from **6-2r**, (f,l) triradical product **6-3r**. Only the molecules and the atoms of the topmost Cu layer are shown (images generated with the p4vasp program).

(b) Calculated energies for the C-H activation of **6** and **6-2r**

Table S1 compares the relative energies of TP **6** and its radical derivatives **6-1r**, **6-2r**, **6-3r** (see Scheme 2) on the Cu(111) surface in the two orientations **X** and **Y** (see Figure S11). The species TP **6**, **6-1r** and **6-2r** slightly favor the **Y** orientation adsorption by 0.1 - 0.2 eV. In contrast, the triradical **6-3r** favors adsorption in **X** orientation by 0.43 eV. This can be rationalized considering the better alignment of the three radicalic carbon atoms in **6-3r** with adjacent atoms of the Cu(111) lattice (see Figure S11 f,l), which facilitates the formation of three C–Cu bonds in **X** orientation.

Table S1: Relative energies of intermediates in two different orientations on the surface [eV].

Intermediate	C-H along $\langle\bar{1}01\rangle$ ("X") - C-H along $\langle1\bar{2}1\rangle$ ("Y")
6	0.09 (Y lower)
6-1r	0.17 (Y lower)
6-2r	0.16 (Y lower)
6-3r	-0.43 (X lower)

Table S2: Reaction energies for C-H activation in [eV].

Orientation	6 → 6-1r	6-2r → 6-3r
C-H along $\langle\bar{1}01\rangle$, "X"	+1.14	+0.33
C-H along $\langle1\bar{2}1\rangle$, "Y"	+1.06	+0.92

Table S3: Activation barriers for C-H activation in [eV].

Orientation	6 → 6-1r	6-2r → 6-3r
C-H along $\langle\bar{1}01\rangle$, "X"	1.83 / 1.92 ^a	0.97 / 1.12 ^a
C-H along $\langle1\bar{2}1\rangle$, "Y"	1.80	1.39

a: effective barrier by adding the reorientation energy of **6** or **6-2r** in Table S1 to the direct barrier (Curtin-Hammett-principle), respectively.

(c) Structure of DMQBR **2** on Cu(111)

Figure S12 shows optimized geometries of biradical **2** (DMQBR, see Scheme 1 of the main text) in its *anti* and *syn* configuration. For these calculations, a larger unit cell than specified in the main text was used (9×10 Cu atoms per layer, three Cu layers, 270 Cu atoms in total, denoted "large" unit cell hereafter) with the same general computational setup. As discussed in the main text for the radicals of **6**, the interaction of the radical sites of DMQBR **2** with the Cu(111) surface leads to a distortion of the molecular geometry, see Figure S12. The DFT calculations indicate that the *anti* configuration is lower in energy by about 0.5 eV than the *syn* configuration.

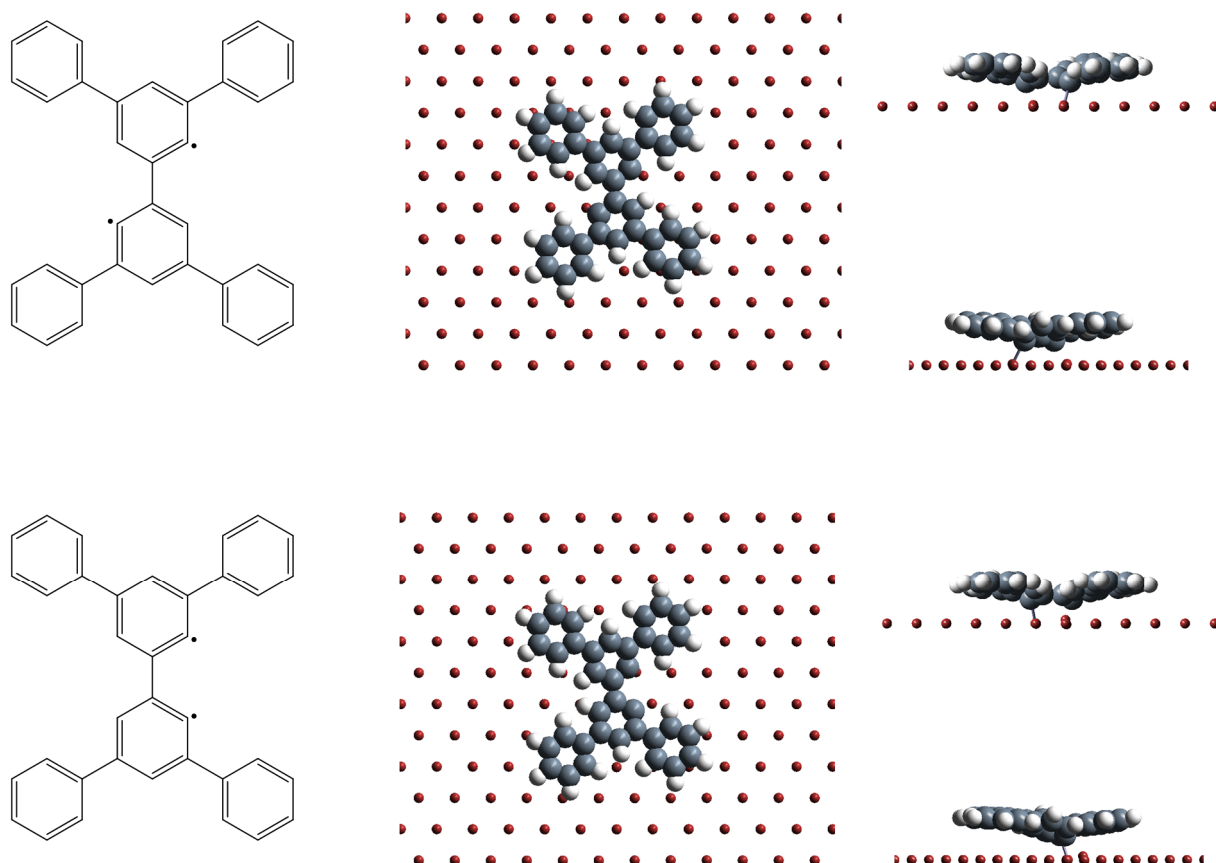
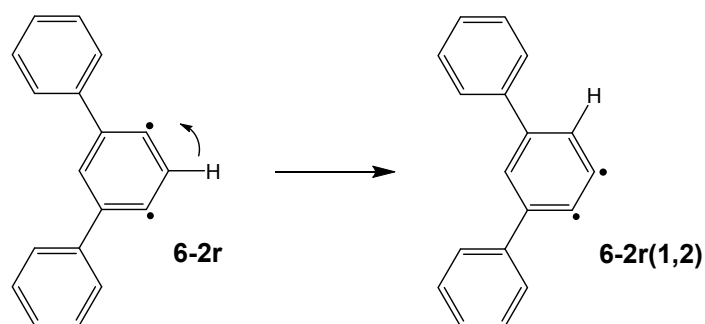


Figure S12. Optimized geometries of the biradical **2** in *anti* (top row) and *syn* (bottom row) configuration ("large" unit cell, see text). Only the molecules and the topmost Cu layer of the three-layer Cu(111) slab are shown.

(d) Formation of DMQBR **2** on Cu(111) from **6-2r** by 1,2-hydrogen shift

As discussed in the main text, formation of **2** can be envisioned via C-H activation of **6-2r**, subsequent C-C coupling of two molecules of **6-3r**, and reattachment of the two hydrogen atoms released in the C-H activation step. C-H activation from **6-2r** to yield **6-3r** is thermodynamically uphill by +0.5 eV (cf. Figure 3 bottom) according to the present DFT calculations, while the subsequent C-C coupling of two **6-3r** molecules and reattachment of two hydrogen atoms is exothermic by -3.8 eV ("large" unit cell, see above). Hence, the total reaction of two molecules of **6-2r** to yield one molecule of **2** is exothermic by -2.8 eV.

As an alternative, **2** could also be formed from two molecules of **6-2r** by 1,2-hydrogen shift, see Scheme S1, and subsequent C-C coupling of the 1,2 biradicals **6-2r(1,2)**.



Scheme S1. 1,2 hydrogen shift in **6-2r**; Cu(111) surface not shown.

The 1,2-hydrogen shift reaction (Scheme S1) on Cu(111) is computed to be exothermic by 0.6 eV per molecule, and the subsequent C-C coupling step of two **6-2r(1,2)** molecules is exothermic by 1.6 eV. However, preliminary DFT calculations⁴ on possible transition states for the reaction shown in Scheme S1 on Cu(111), see Figure S13, indicate that the energy barrier for this process is significantly higher than that of C-H activation shown in Figure 3 of the main text. Specifically, the transition structures found⁴ correspond to energy barriers of 1.7 eV (**X** orientation, cf. Figure S13 (top row) and Figure S11d) or 2.4 eV (**Y** orientation, cf. Figure S13 (bottom row) and Figure S11j). These DFT data thus suggest that C-H activation according to Figure 3 of the main text is to be favored due to its lower barrier of 1.1 eV.

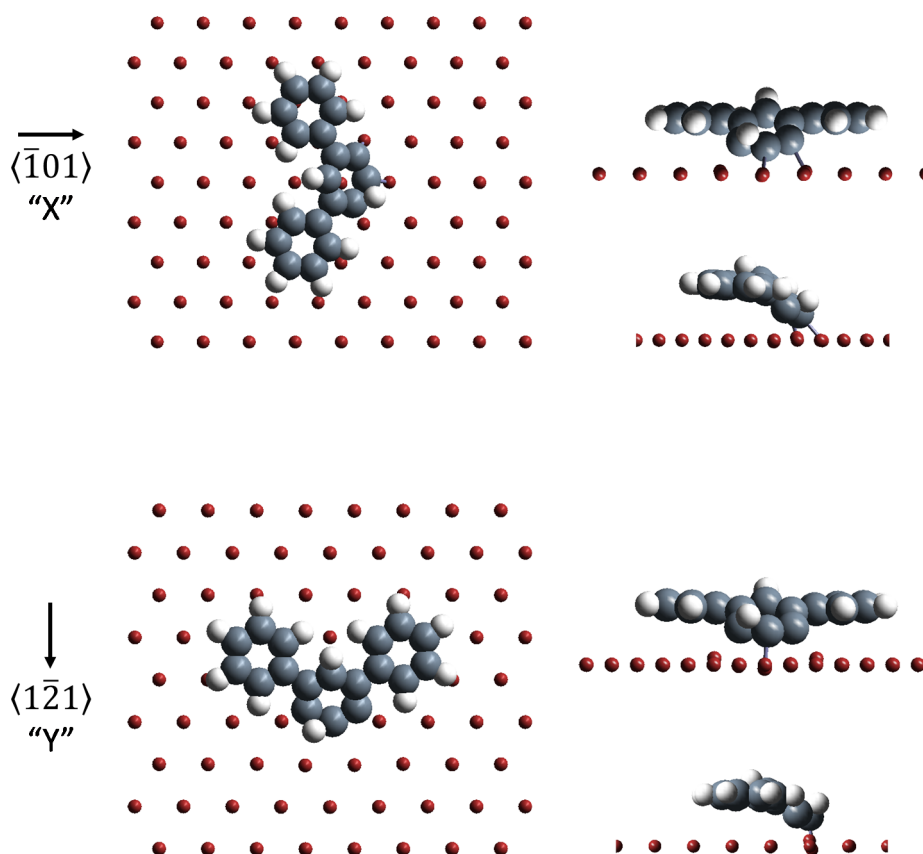


Figure S13. Representations of optimized⁴ transition structures for 1,2-H shift in **6-2r** (Scheme S1) in two different orientations on the Cu(111) surface, cf. Figure S11. Only molecules and topmost Cu layer of the three-layer Cu(111) slab are shown.

(e) Structure of biradical **7-2r**, derived from precursor DBOTP **3**

Figure S14 compares the optimized molecular structure of the biradical species **6-2r** and **7-2r**, which are assumed to form as intermediates from DBMTP **1** and DBOTP **3**, respectively; see Scheme 1 of the main text. The images show that the peculiar distortion of the central ring, which is due to the interaction of the radical sites with the Cu(111) surface, is similar for both **6-2r** and **7-2r**. It may therefore be anticipated that C-H bond activation is facilitated also for **7-2r** in analogy to the C-H activation of **6-2r**, which is discussed in the main text (Scheme 2 and Figure 3).

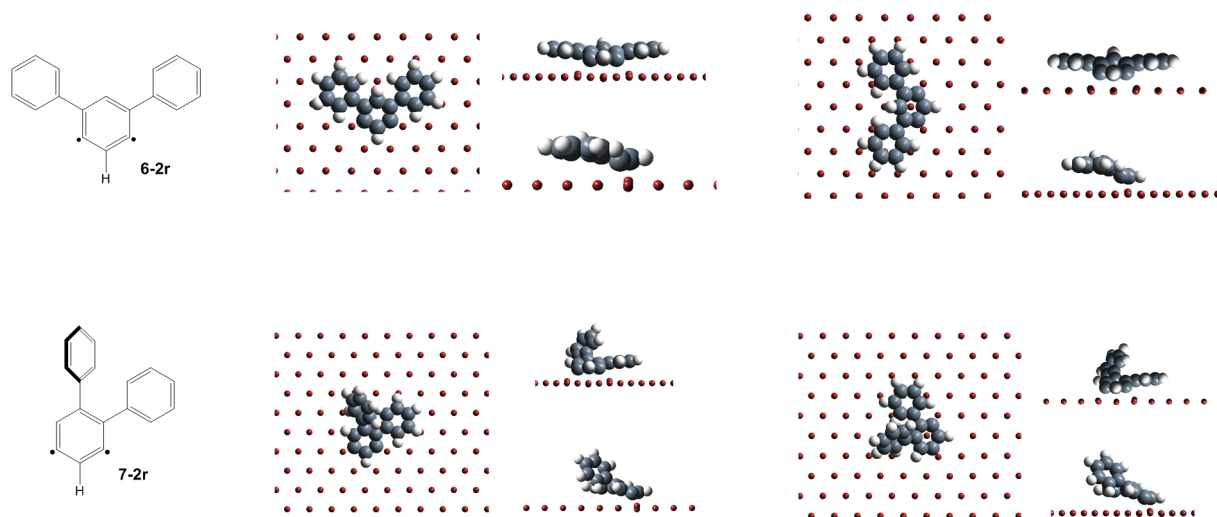


Figure S14. Comparison of optimized geometries of **6-2r** (top row, see also the main text) and **7-2r** (bottom row), which are assumed to form as intermediates from DBMTP **1** and DBOTP **3**, respectively. Cf. Scheme 1 of the main text. The energy difference of the two orientations amounts to 0.16 eV in favor of Y orientation in both cases. Only molecules and topmost Cu layer of the three-layer Cu(111) slab are shown.

(3) Precursor synthesis and characterization

(a) 1,5-Dibromo-2,3-diiodobenzene

According to a synthesis protocol of Müllen *et al.*⁵ 1.35 g (5.34 mmol, 1.07 eq) iodine and 0.85 mL (6.32 mmol, 1.27 eq) isopentyl nitrite were dissolved in 100 mL 1,2-dichloroethane and heated to reflux. Then 1.46 g (5.00 mmol, 1.00 eq) 3,5-dibromo anthranilic acid (dissolved in 30 mL 1,4-dioxane) were added dropwise to the boiling solution during a time period of 1 h. A gas evolution could be observed. The dark red solution was refluxed for 2 h and cooled down afterwards. After cooling to room temperature, 50 mL of a 5% aqueous sodium thiosulfate solution were added, which caused a decoloration of the reaction solution. The aqueous layer was extracted with chloroform (2 x 25 mL) and the combined organic layers were dried over MgSO₄. The crude product was purified by column chromatography over silica gel (eluent: *n*-pentane) to afford 1.10 g (2.26 mmol, 45 %) of the desired product as colorless needles.

TLC: R_f = 0.75 (*n*-pentane).

¹H NMR (300 MHz, CDCl₃): δ = 7.94 (d, 1H, ⁴*J* = 2.1 Hz), 7.72 (d, 1H, ⁴*J* = 2.1 Hz) ppm.

¹³C NMR (75 MHz, CDCl₃): δ = 140.2, 134.3, 130.0, 123.3, 114.2, 109.8 ppm.

HRMS: *m/z* for [C₁₈H₁₃⁷⁹Br]⁺ calc.: 487.6592, found 487.6589 (EI⁺).

IR (ATR): 3072 (w), 3035 (w), 2952 (w), 2922 (w), 2866 (m), 1720 (w), 1582 (m), 1555 (s), 1517 (m), 1435 (m), 1340 (s), 1252 (m), 1178 (m), 1099 (s), 1049 (s), 989 (s), 851 (vs), 742 (s), 704 (m), 665 (m), 521 (w), 436 (w) cm⁻¹.

Mp: 105-107°C

(b) 1,5-Dibromo-2,4-diiodobenzene

According to a synthesis protocol of Hart *et al.*⁶ 1.04 g (4.41 mmol, 1.00 eq) 1,3-dibromobenzene were dissolved in 30 mL concentrated sulfuric acid and heated to 90 °C. In a time period of 10 min 4.43 g (17.5 mmol, 3.96 eq) iodine were added. The solution was heated to 115 °C and stirred for 1 day. After cooling down to room temperature, the reaction solution was dissolved in 200 mL water and ice and extracted with 50 mL dichloromethane (3x). The organic layer was washed with saturated sodium bicarbonate solution (2x 100 mL), saturated sodium sulfite solution (50 mL) and dried over MgSO₄. The crude product was purified by column chromatography over silica gel (eluent: *n*-pentane), the mono-iodinated side product could be separated by short path distillation (1×10⁻¹ mbar, 115 °C) to afford 603 mg (1.48 mmol, 34%) of the desired product as colorless needles.

¹H NMR (300 MHz, CDCl₃): δ = 8.28 (s, 1H, *H* 3), 7.83 (s, 1H, *H* 6) ppm.

¹³C NMR (75 MHz, CDCl₃): δ = 149.6 (2C), 135.5 (2C), 130.6, 100.5 ppm.

(c) 3',5'-Dibromo-1,1':2',1''-terphenyl

According to a synthesis protocol of Müllen *et al.*⁵ 533 mg (1.09 mmol, 1.00 eq) 1,5-dibromo-2,3-diiodobenzene and 265 mg (2.17 mmol, 1.99 eq) benzene boronic acid were dissolved in 20 mL 1,4-dioxane and 4 mL of a 10 % aqueous sodium carbonate solution and 5 mL toluene were added. The reaction solution was degassed with argon for 30 min and 130 mg (0.11 mmol, 10 mol%) tetrakis(triphenylphosphine)palladium(0) were added in one portion. The reaction was stirred at 90 °C for 3 d until complete conversion could be detected by GC-MS analysis. After cooling to room temperature, 25 mL water were added and the aqueous layer was extracted with dichloromethane (3 x 15 mL) and the combined organic layers were dried over MgSO₄. The crude product was purified by column chromatography on silica (eluent: *n*-pentane/dichloromethane 25:1) to afford 174 mg (0.45 mmol, 41 %) of the desired compound **3** as colorless solid.

TLC: R_f = 0.40 (*n*-pentane/dichloromethane 25:1).

¹H NMR (300 MHz, CDCl₃): δ = 7.84 (d, 1H, ⁴*J* = 2.0 Hz), 7.52 (d, 1H, ⁴*J* = 2.0 Hz), 7.21-7.24 (m, 3H) 7.13-7.15 (m, 3H), 6.99-7.06 (m, 4H) ppm.

¹³C NMR (75 MHz, CDCl₃): δ = 145.0, 140.4, 139.9, 139.2, 134.2, 132.3, 130.5 (2C), 129.6 (2C), 127.9 (2C), 127.8 (2C), 127.5, 127.3, 125.3, 121.5 ppm.

HRMS: m/z for $[C_{18}H_{12}^{79}Br_2]^+$ calc.: 385.93058, found: 385.93057 (EI⁺).

IR (ATR): 3061 (w), 2922 (w), 2854 (w), 1566 (w), 1533 (m), 1491 (w), 1424 (m), 1379 (m), 1273 (w), 1235 (w), 1174 (w), 1101 (w), 998 (m), 889 (m), 761 (s), 694 (w), 624 (m), 516 (w) cm⁻¹.

Mp: 83-85 °C.

(d) 4',6'-Dibromo-1,1':3',1''-terphenyl

For the synthesis of the product, 438 mg (0.90 mmol, 1.00 eq) 1,5-dibromo-2,4-diiodobenzene and 223 mg (1.83 mmol, 2.03 eq) benzene boronic acid were dissolved in 6 mL 1,4-dioxane, 3 mL water and 6 mL toluene. Then 370 mg (2.68 mmol, 2.97 eq) potassium carbonate were added and the reaction mixture was degassed with argon for 30 min. Then tetrakis(triphenylphosphine)palladium(0) 103 mg (0.09 mmol, 10 mol%) were added in one portion. The reaction mixture was stirred at 110 °C for 3 d until complete conversion could be detected by GC-MS analysis. After cooling down to room temperature, 10 mL water were added and the aqueous layer was extracted with dichloromethane (3 x 10 mL) and the combined organic layers were dried over MgSO₄. The crude product was purified by column chromatography on silica (eluent: *n*-pentane/dichloromethane 20:1) to afford 252 mg (0.65 mmol, 73 %) of the desired compound as a colorless solid.

TLC: R_f = 0.45 (*n*-pentane/dichloromethane 20:1).

¹H NMR (300 MHz, CDCl₃): δ = 8.00 (s, 1H), 7.40-7.45 (m, 10H), 7.31 (s, 1H) ppm.

¹³C NMR (75 MHz, CDCl₃): δ = 142.0, 140.0, 136.8, 133.4, 129.4, 128.3, 128.1, 121.9 ppm.

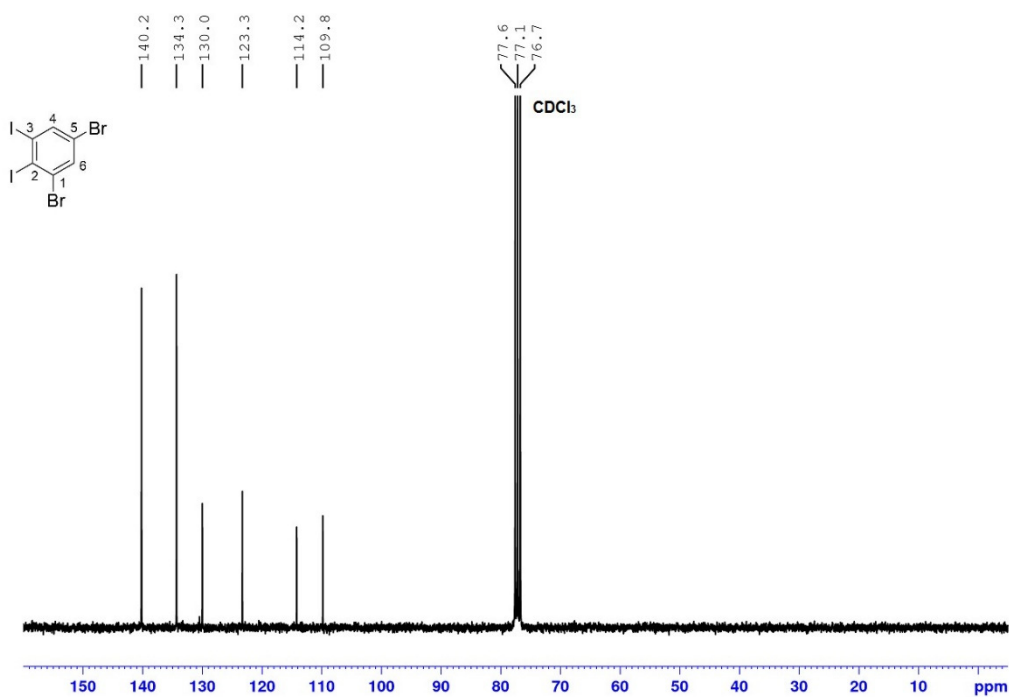
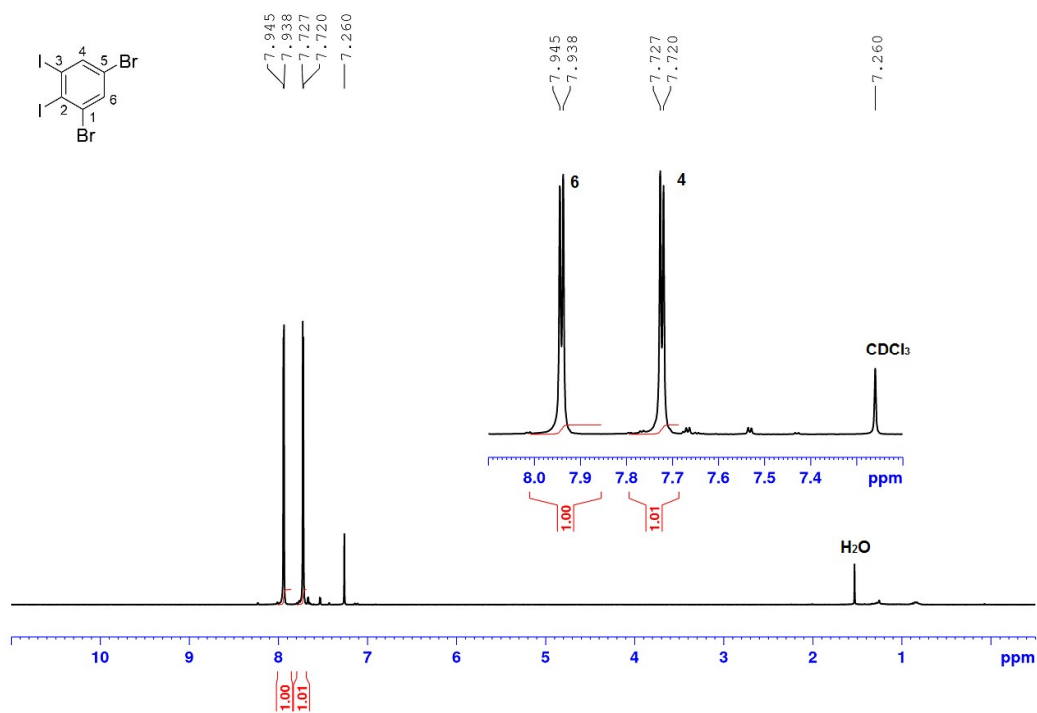
HRMS: m/z for $[C_{18}H_{12}^{79}Br_2]^+$ calc.: 385.93058, found: 385.93057 (EI⁺).

IR (ATR): = 3055 (w), 3022 (w), 1575 (w), 1490 (m), 1454 (m), 1429 (w), 1349 (w), 1250 (w), 1058 (m), 1028 (w), 999 (m), 915 (w), 833 (w), 754 (s), 689 (vs), 580 (m), 503 (w), 471 (w) cm⁻¹.

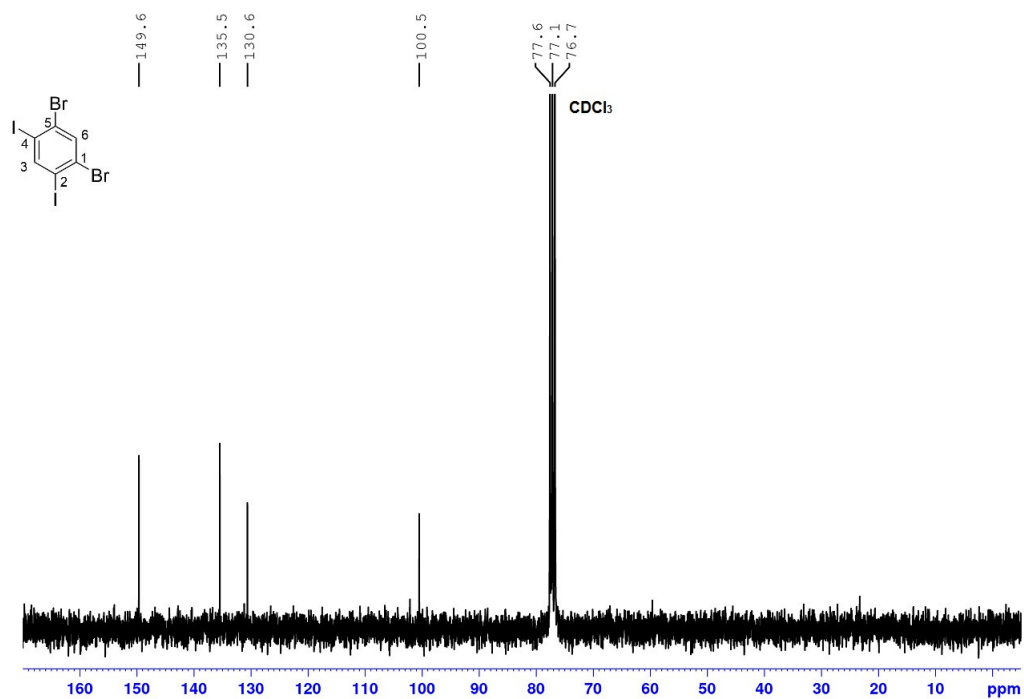
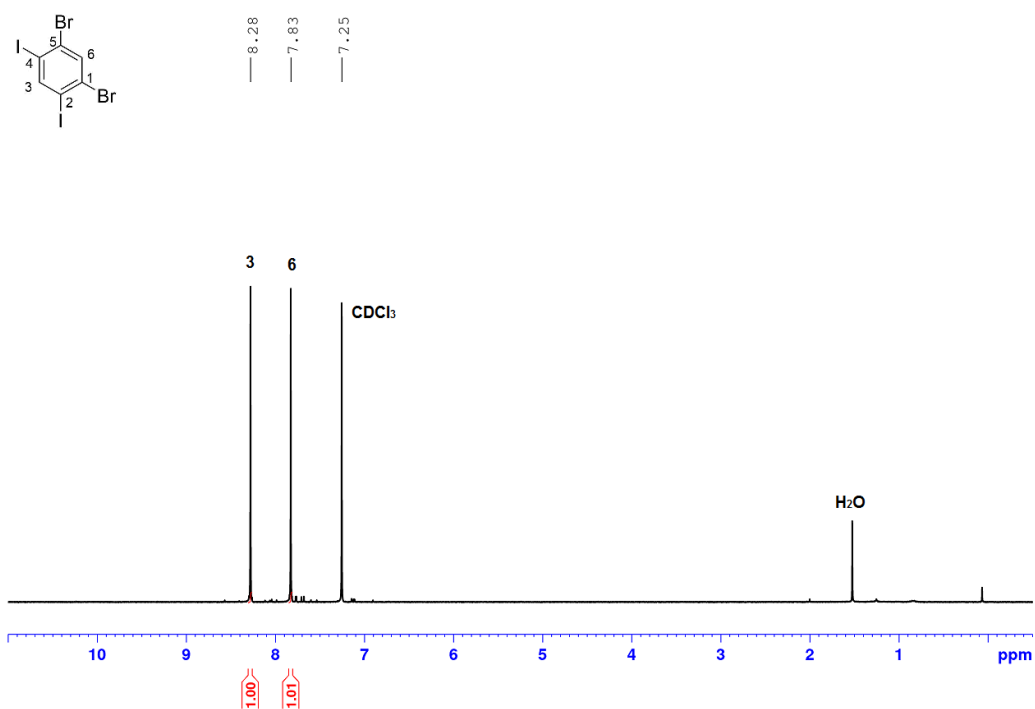
Mp: 63-65 °C.

(e) NMR-spectra

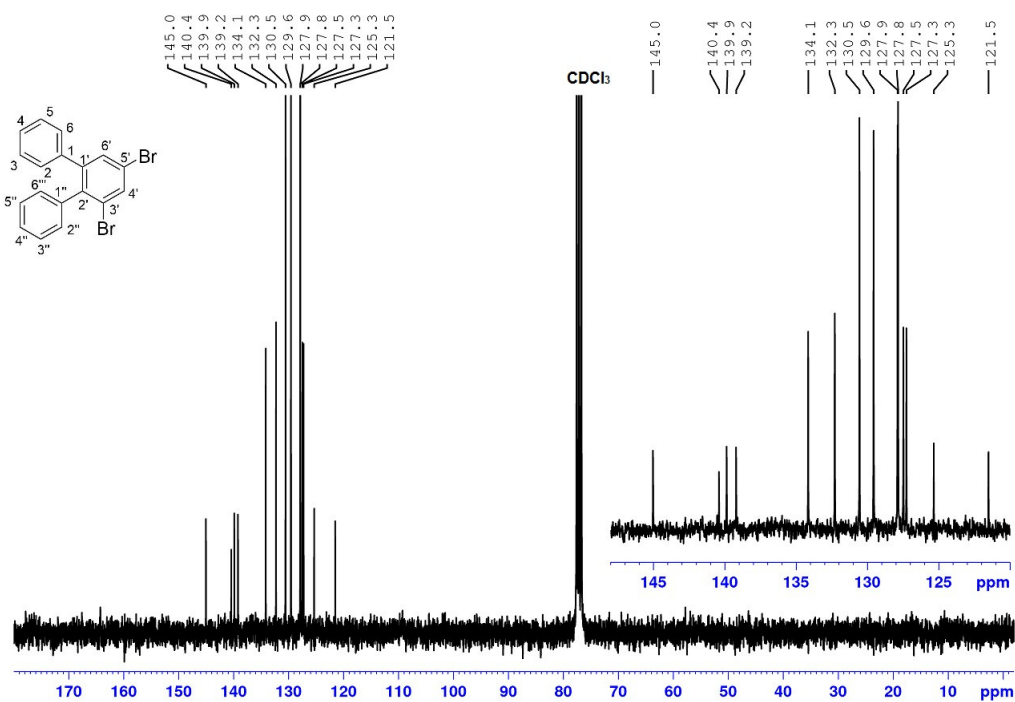
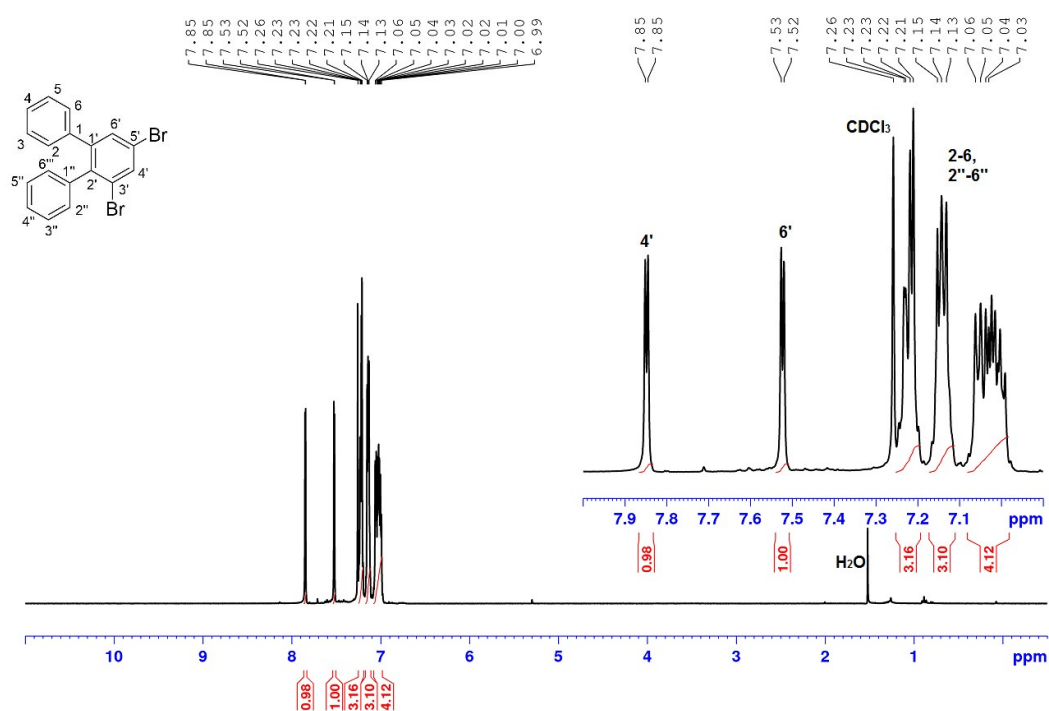
1,5-Dibromo-2,3-diiodobenzene



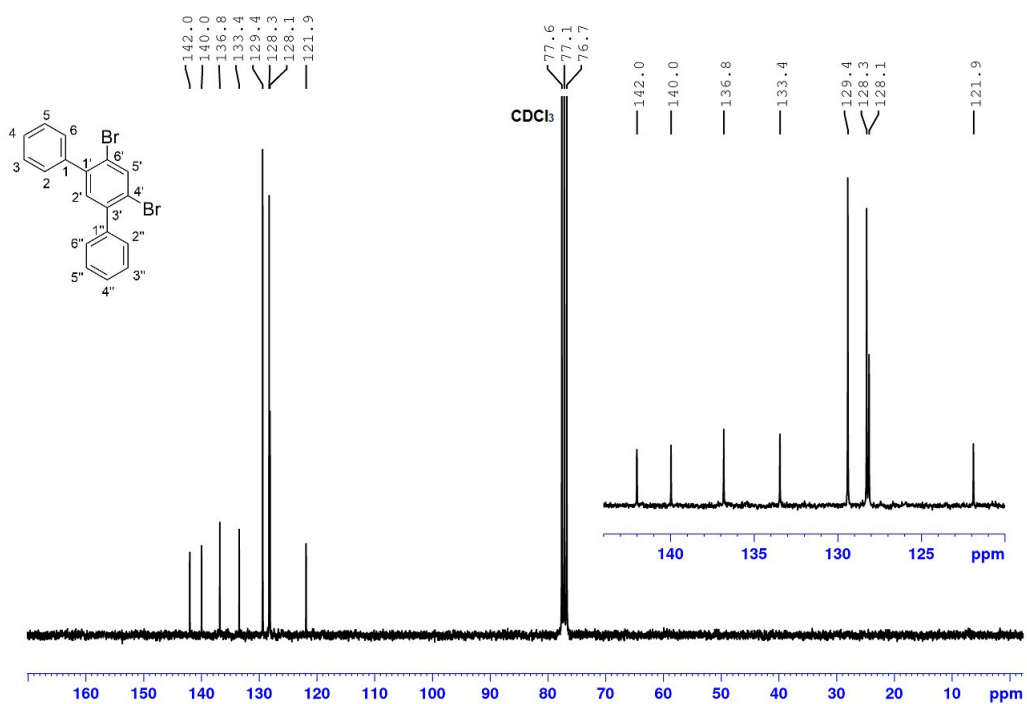
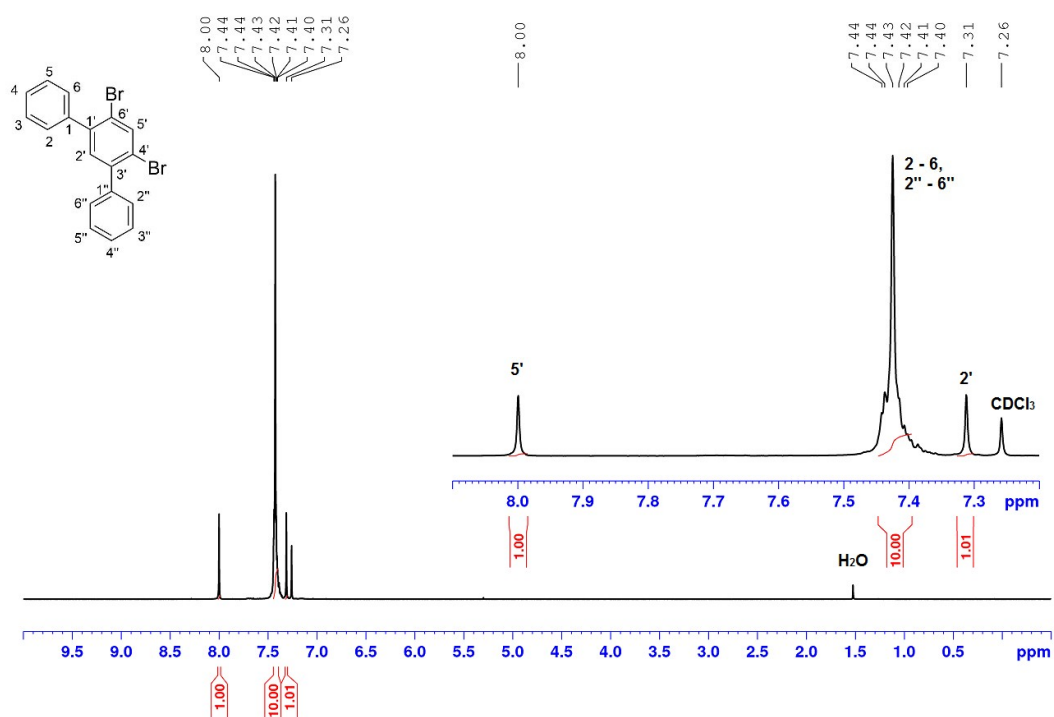
1,5-Dibromo-2,4-diiodobenzene



3',5'-Dibromo-1,1':2',1''-terphenyl



4',6'-Dibromo-1,1':3,1''-terphenyl



(4) References

1. Fan, Q.; Wang, C.; Han, Y.; Zhu, J.; Kuttner, J.; Hilt, G.; Gottfried, J. M., *ACS Nano* **2014**, *8*, 709-718.
2. Ma, L.; Sun, J.; Lu, X.; Zhang, S.; Qi, H.; Liu, L.; Shao, Y.; Shao, X., *Beilstein J. Org. Chem.* **2015**, *11*, 850-859.
3. Björk, J.; Stafström, S.; Hanke, F., *J. Am. Chem. Soc.* **2011**, *133*, 14884-14887.
4. Transition states for the 1,2-H-shift reaction (Scheme S1, Figure S13) were investigated using the general setup and procedures described in the Methods section of the main text. Transition states were optimized for the molecules in two different orientations on the surface as shown in Figure S11. However, for the molecule in **Y** orientation, see Figures S11j and S13, a transition state was found in optimizations using the gamma point only. The energy of this transition structure was subsequently calculated without further optimization using the 2x2x1 k-point mesh using the gamma-point geometry. For the molecule in **X** orientation, cf. Figures S11d and S13, a transition structure was found for the full 2x2x1 k-point mesh and a slightly reduced convergence criterion for the gradient (0.01 eV/Å). .
5. Talirz, L.; Söde, H.; Dumsclaff, T.; Wang, S.; Sanchez-Valencia, J. R.; Liu, J.; Shinde, P.; Pignedoli, C. A.; Liang, L.; Meunier, V.; Plumb, N. C.; Shi, M.; Feng, X.; Narita, A.; Müllen, K.; Fasel, R.; Ruffieux, P., *ACS Nano* **2017**, *11*, 1380-1388.
6. Hart, H.; Harada, K.; Du, C. J. F., *J. Org. Chem.* **1985**, *50*, 3104-3110.

(5) Geometry data of stationary points from the DFT calculations

Geometries of stationary points found with the procedures described in the main paper; VASP POSCAR format; VASP version 5.3.5. Adsorbate systems (molecule + surface) resulted in (essentially) zero total magnetization (below 0.0001 μ_B) in the DFT calculations. All plots of optimized geometries (Figures 3, S11-S14) were generated with the p4vasp program.

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Selective

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TS 6 -> 6-1r X

1.0

+17.6699932067 +0.0000000000 +0.0000000000
 +0.0000000000 +17.4887577163 +0.0000000000
 +0.0000000000 +0.0000000000 +24.6900000000

C H Cu

18 14 168

Selective

Cartesian

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TS 6-2r -> 6-3r Y

1.0

+17.6699932067 +0.0000000000 +0.0000000000
+0.0000000000 +17.4887577163 +0.0000000000
+0.0000000000 +0.0000000000 +24.6900000000

C H Cu

18 12 168

Selective

Cartesian

+8.8371236863 +6.9794333657 +9.8941833657 T T T
+7.5691002673 +7.5025748358 +10.0726038746 T T T
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TS 6-2r -> 6-3r X

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TS 1,2-H-shift 6-2r X

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