

Supplementary Information for

Patterned Organic Layers on Gold Surfaces Prepared by Electro-grafting of Photolabile-Protected Aryl Diazonium Salts

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1. XPS Survey and high resolution spectra.

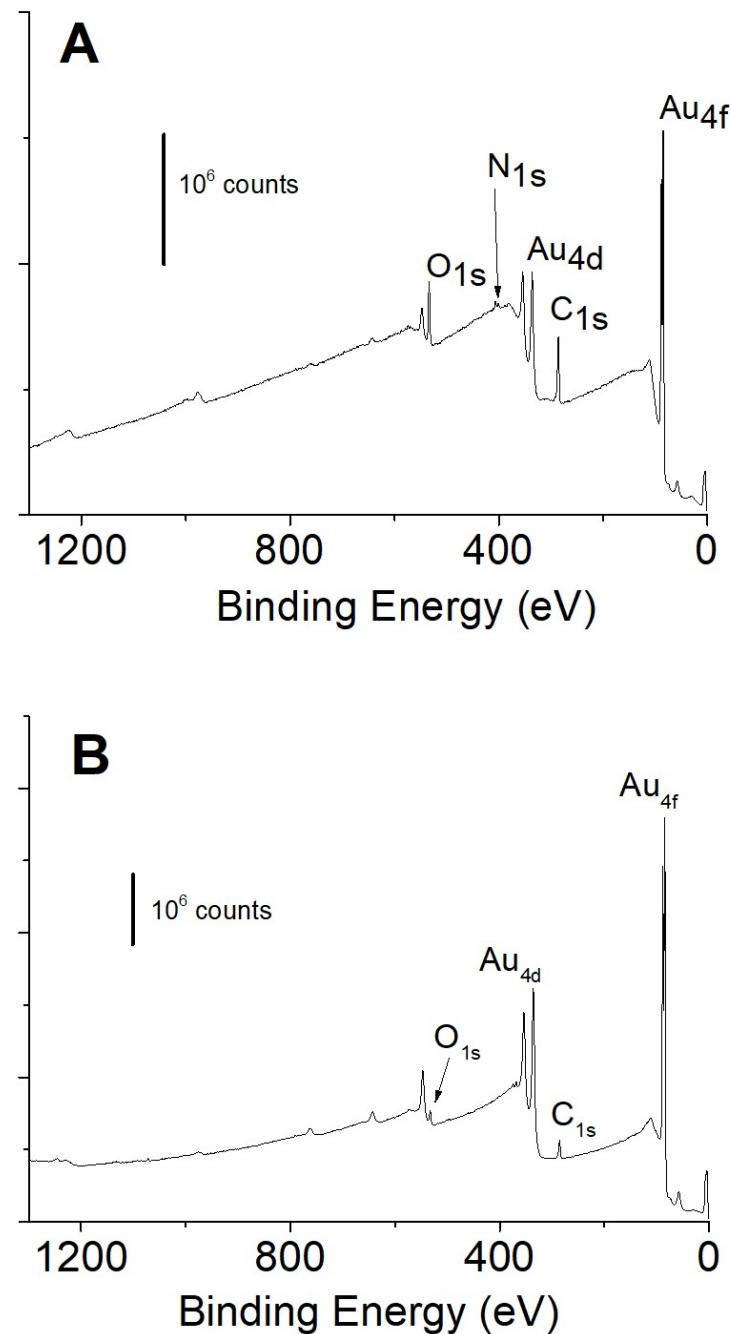


Figure S1. XPS survey spectra of the gold-modified samples with **1** (A) before and (B) after photo-deprotection at $\lambda = 365$ nm for 5 min (power: 40 mW/cm²).

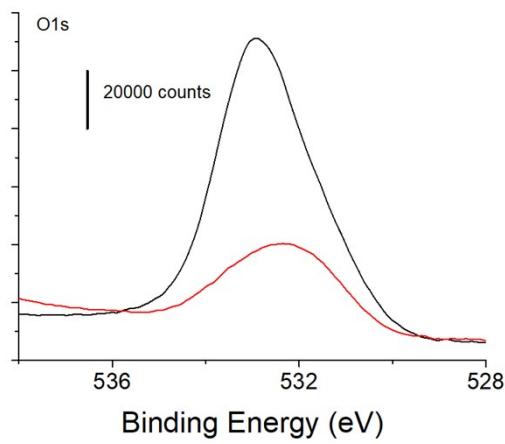
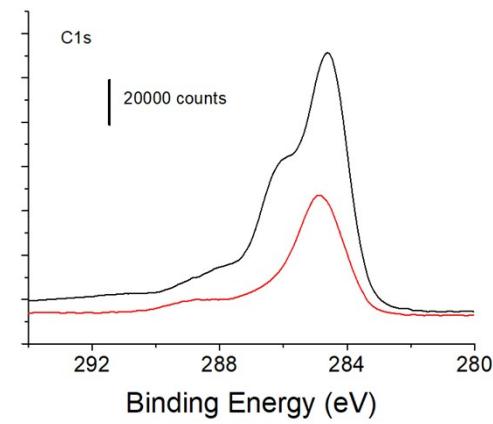
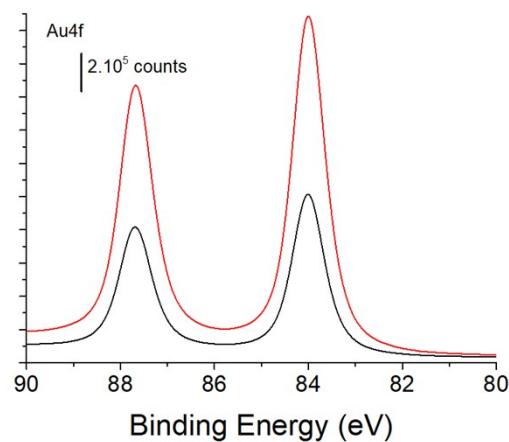


Figure S2. High resolution core level XPS spectra for the Au4f, C1s and O1s regions of gold substrate modified with **1**, before (black line) and after (red line) photo-deprotection at $\lambda = 365$ nm for 5 min (power: 40 mW/cm²).

Table S1. Ratio of atomic percentage determined through survey spectra quantification.

	C/Au	O/Au	C/O
Before irradiation	3.8 ± 0.2	1.2 ± 0.1	3.0 ± 0.1
After irradiation	0.83 ± 0.02	0.20 ± 0.02	4.3 ± 0.3

2. RMN spectra of molecules 2 and 3 and aryl diazonium 1.

^1H NMR (300 MHz, CDCl₃) δ 7.65 (s, 1H), 7.31 (d, $J = 9.2$ Hz, 2H), 7.26 (s, 1H), 6.93 (d, $J = 9.3$ Hz, 2H), 6.15 (s, 2H), 5.36 (d, $J = 0.8$ Hz, 2H), 1.47 (s, 9H).

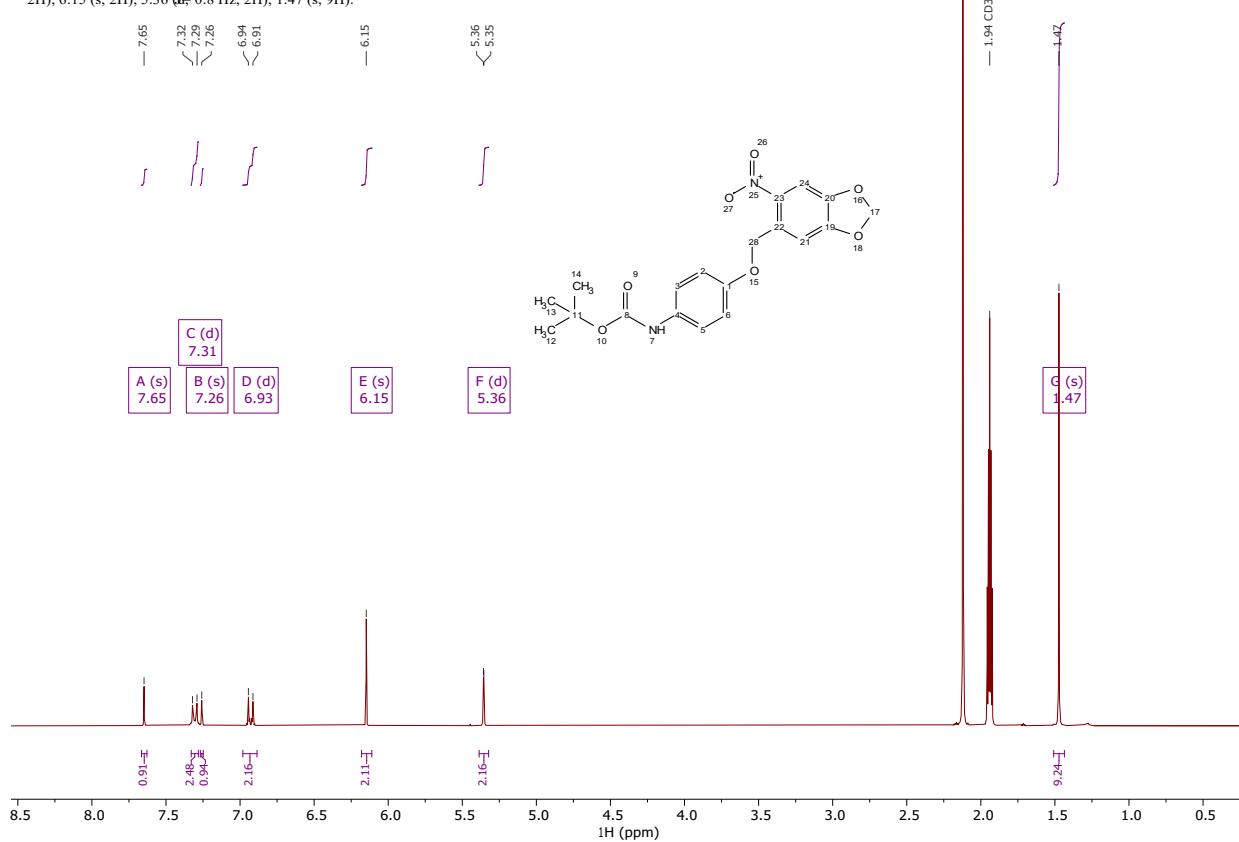


Figure S3. ^1H NMR spectra of O-[1-(6-Nitro-1,3-benzodioxol-5-yl)ethyl]-4-N-Boc-aminophenol (2).

^1H NMR (300 MHz, CD₃OD) δ 7.63 (s, 1H), 7.26 (*t*, 0.8 Hz, 1H), 6.80 – 6.74 (m, 2H), 6.62 – 6.55 (m, 2H), 6.14 (s, 2H), 5.28 (*d*, 0.8 Hz, 2H).

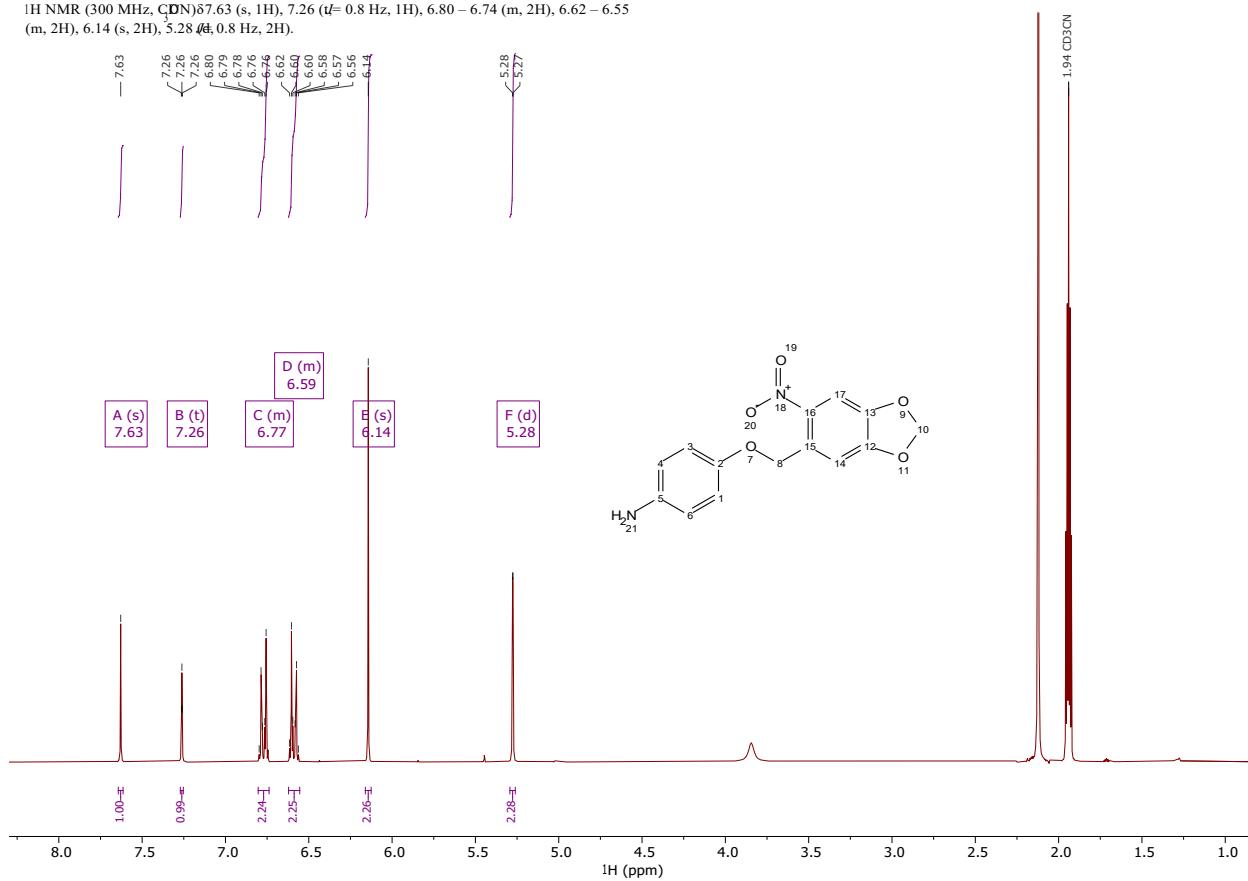


Figure S4. ^1H NMR spectra of O-[1-(6-Nitro-1,3-benzodioxol-5-yl)ethyl]-aminophenol (3).

¹H NMR (300 MHz, CDCl₃) δ 8.41 (d, *J*= 9.4 Hz, 2H), 7.72 (s, 1H), 7.46 (d, *J*= 9.5 Hz, 2H), 7.25 (s, 1H), 6.19 (s, 2H), 5.66 (s, 2H).



Figure S5. ¹H NMR spectra of O-[1-(6-Nitro-1,3-benzodioxol-5-yl)ethyl]-hydroxybenzene diazonium tetrafluoroborate (1).

3. AFM images and line profiles.

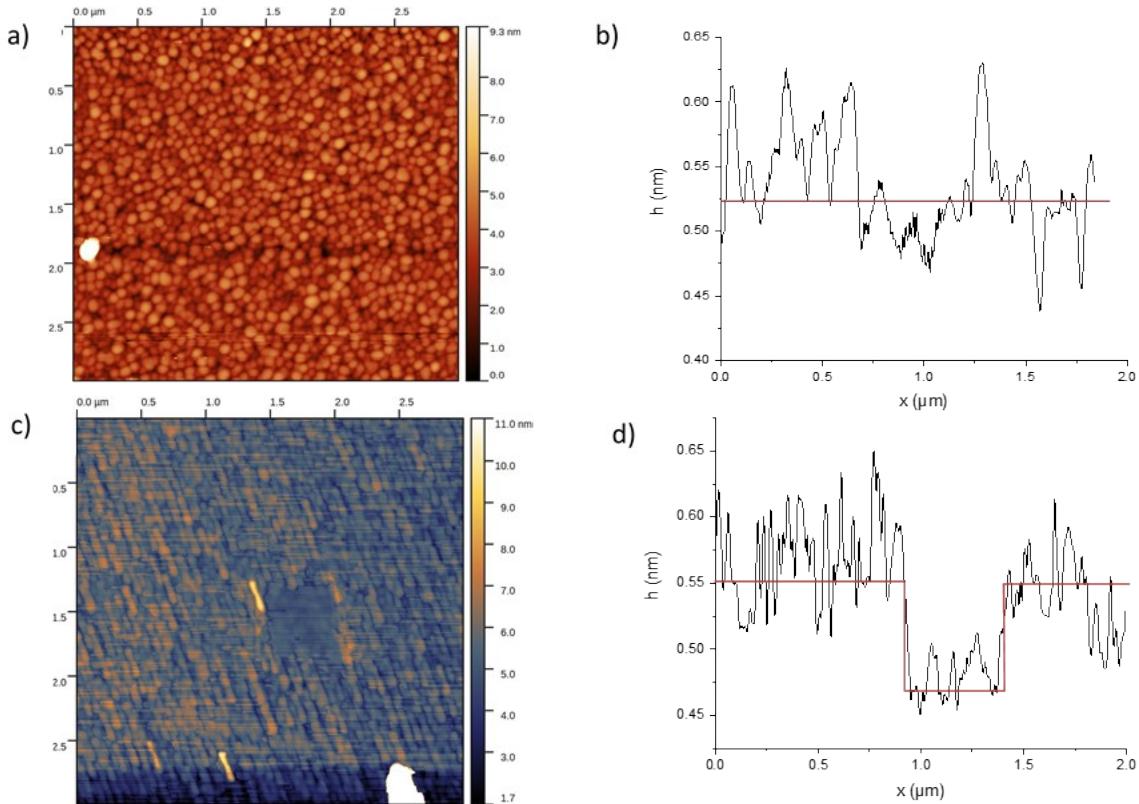


Figure S6. a) $3 \times 3 \mu\text{m}^2$ AFM image of a bare gold substrate. b) Line profile (black) and average height profile curve (red) for image presented in Figure S6a. c) $3 \times 3 \mu\text{m}^2$ AFM image of a bare gold substrate, showing a $0.5 \times 0.5 \mu\text{m}^2$ scratch. d) Line profile (black) and average height profile curve (red) for image presented in Figure S6c.

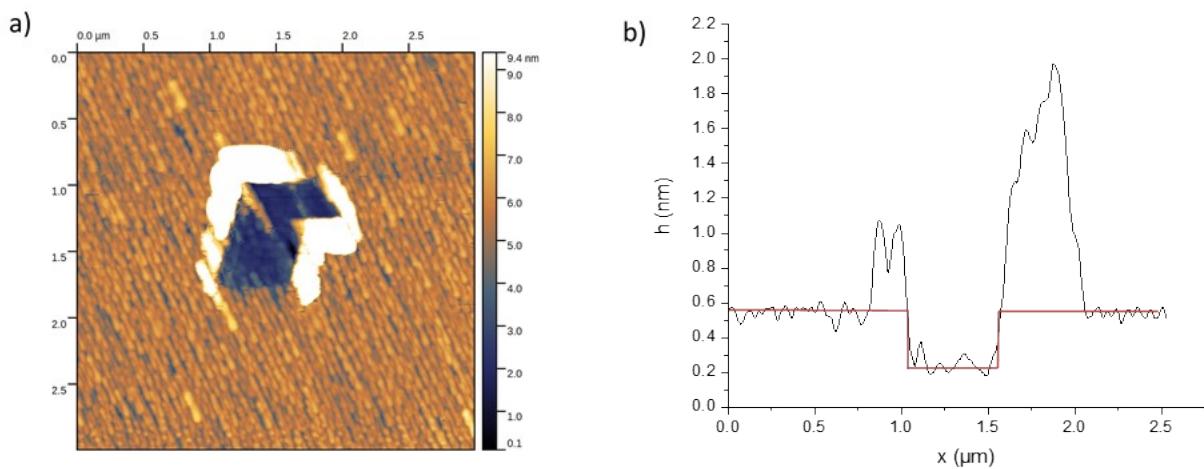


Figure S7. a) $3 \times 3 \mu\text{m}^2$ AFM image of a gold substrate modified with o-[1-(6-nitro-1,3-benzodioxol-5-yl)ethyl]-hydroxybenzene diazonium tetrafluoroborate salt showing a $1 \times 1 \mu\text{m}^2$ scratch. b) Line profile (black) of the scratch and average height profile curve (red) for image presented in Figure S7a.

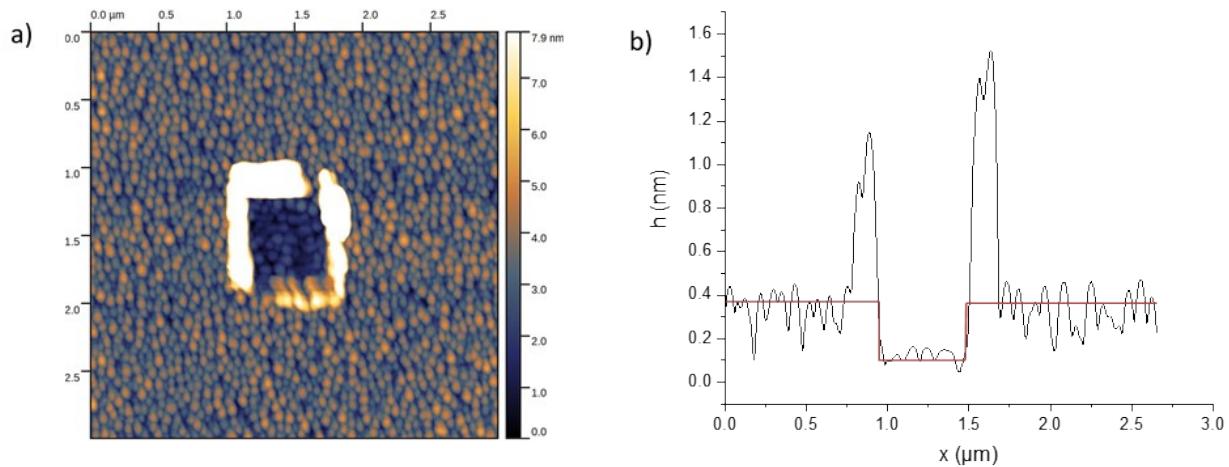


Figure S8. a) $3 \times 3 \mu\text{m}^2$ AFM image of a gold substrate modified with o-[1-(6-nitro-1,3-benzodioxol-5-yl)ethyl]-hydroxybenzene diazonium tetrafluoroborate salt after irradiation at 365 nm and elimination of the photolabile protective group, showing a $1 \times 1 \mu\text{m}^2$ scratch. b) Line profile (black) of the scratch and average height profile curve (red) for image presented in Figure S8a.