Amphiphilic anthanthrene trimers that exfoliate graphite and individualize single wall carbon nanotubes†

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Materials and methods

Chemicals

Natural Graphite was purchased from Kropfmühl AG whereas SWCNTs (704113-1G, statistical CoMoCAT®, > 75% carbon as SWCNT, 0.7 to 1.3 nm diameter) were purchased from SIGMA-ALDRICH and used as obtained. Dimethylformamide and Ethanol of HPLC quality were used as purchased from ROTH.

Spectroscopy

For UV/Vis/nIR absorption measurements a Lambda 2 spectrometer from Perkin Elmer or a Cary 5000 spectrometer from Varian, using 10x10mm quarz cuvettes, were utilized. A Fluoromax3 spectrometer from HORIBA Yobin Yvon was used for fluorescence measurements in the visible. A FluoroLog 3 spectrometer from HORIBA Yobin Yvon using a 450 W Xenon lamp and a Symphony InGaAs array in combination with an iHR320 imaging spectrometer was utilized for the measurements of nIR fluorescence measurements.
The transient absorption pump probe system HELIOS from Ultrafast Systems was the instrument for Femtosecond transient absorption measurements. All pump probe measurements were carried out in 2 mm OS quarz cuvettes. For the generation of laser pulses with a pulse width of 150 fs a CPA-2110 titanium:sapphire laser system from Clark-MXR Inc. was used (output 775 nm, 1 kHz). The 460 nm excitation pulses were generated by using a noncollinear optical parametric amplifier (NOPA, Clark MXR).

Raman spectroscopy / microscopy

For Raman measurement a WiTec alpha300r confocal Raman microscope was used. Samples were deposited onto silica wafers by drop casting. A frequency-doubled Nd:YAG Laser with an output of 532 nm or a HeNe Laser with an output of 633 nm were used for sample excitation.

Microscopy

Transmission electron microscopy was conducted on a Zeiss Leo EM912 Omega with an acceleration voltage of 80 kV. Specimen were prepared by drop casting the dispersion on Lacey Carbon/Copper grids.
Atomic force microscopy measurements were conducted with a JPK Nanowizard 4 Nanoscience microscope holding an ACTA cantilever from Applied Nanostructures APPNANO with a resonance frequency of 300 kHz and a tip radius below 10 nm. Samples were drop casted onto silica wafers.

Supplementary Section

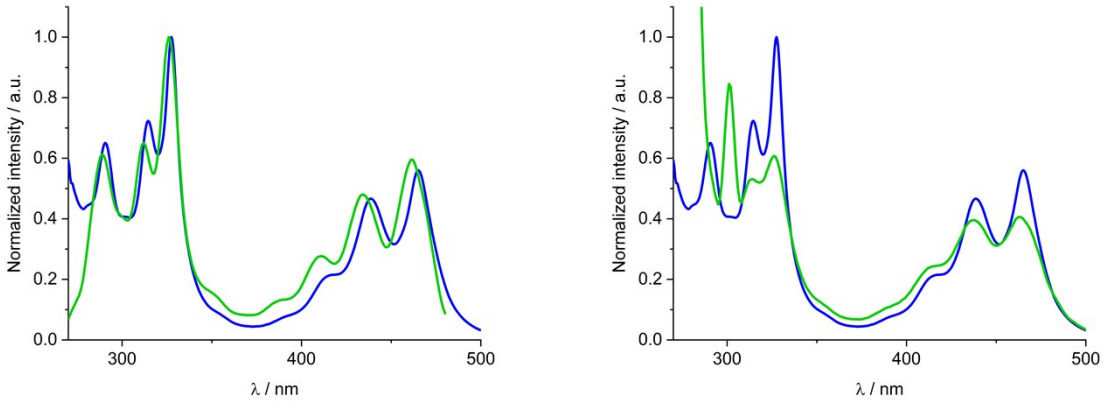


Figure S1: Absorption spectra (blue) and excitation spectra (green) of **1** recorded in DMF at 490 nm (left) and 600 nm (right).

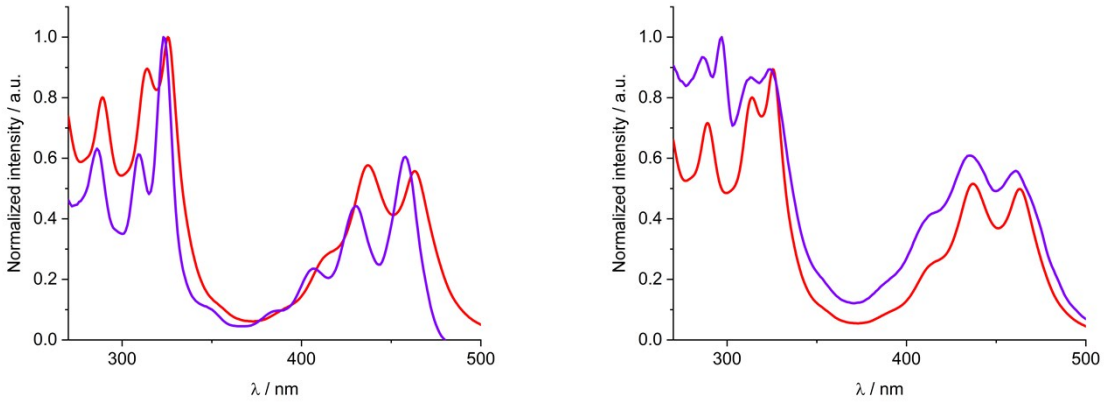


Figure S2: Absorption spectra (red) and excitation spectra (blue) of **1** recorded in ethanol at 490 nm (left) and 600 nm (right).

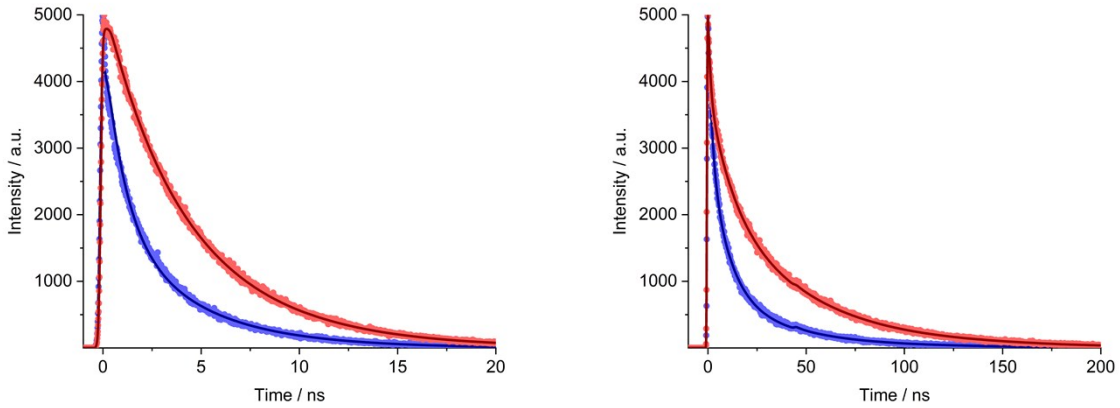


Figure S3: Fluorescence time profiles (dots) recorded at 480 nm (left) and 580 nm (right) and corresponding fits (line) of **1** in DMF (blue) and ethanol (red); 440 nm was used to photoexcite **1**.

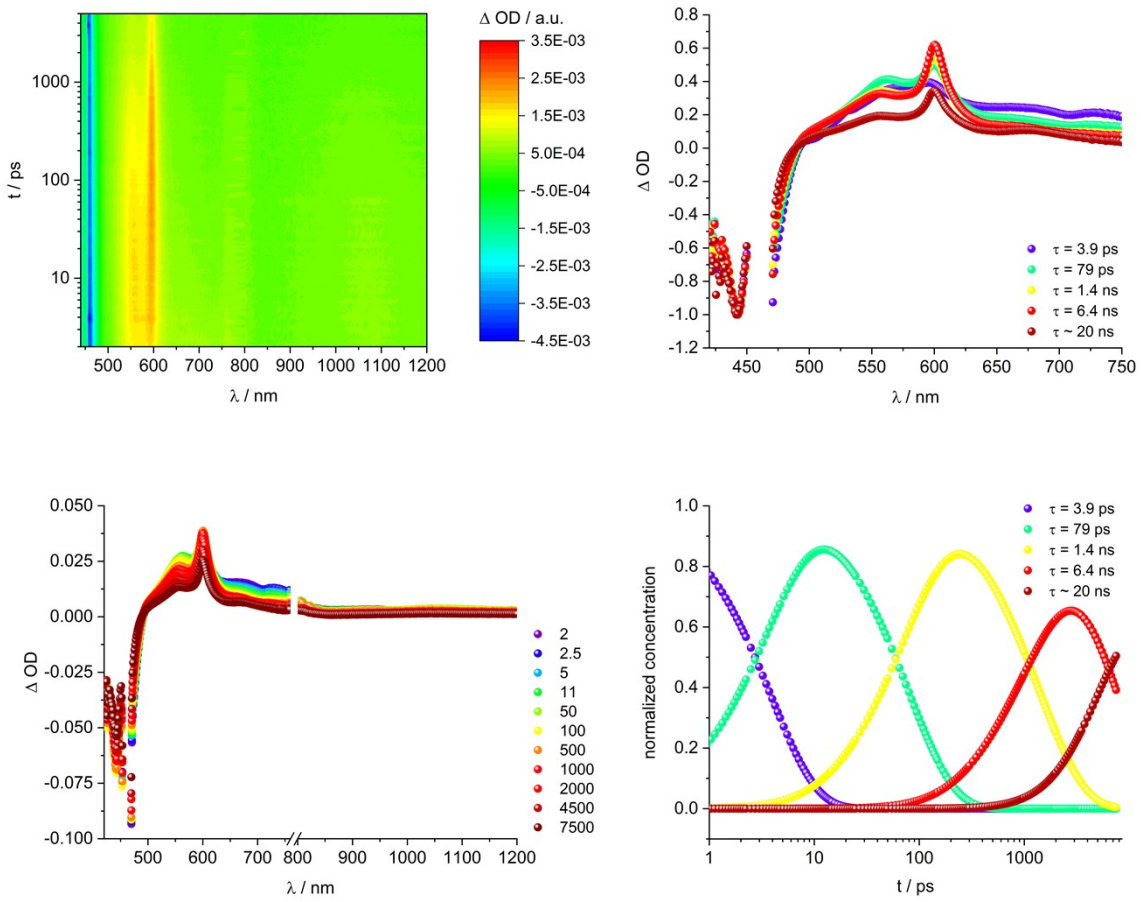


Figure S4: Differential absorption changes obtained upon femtosecond pump-probe experiments (460 nm) of **1** in DMF at room temperature with time delays between 2 and 5000 ps (upper left). Exemplary time evolution spectra of **1** in DMF at room temperature with time delays between 1.7 and 7500 ps (lower left) Corresponding evolution associated spectra of **1** in DMF obtained by global analysis (upper right). Corresponding concentration profiles (lower right).

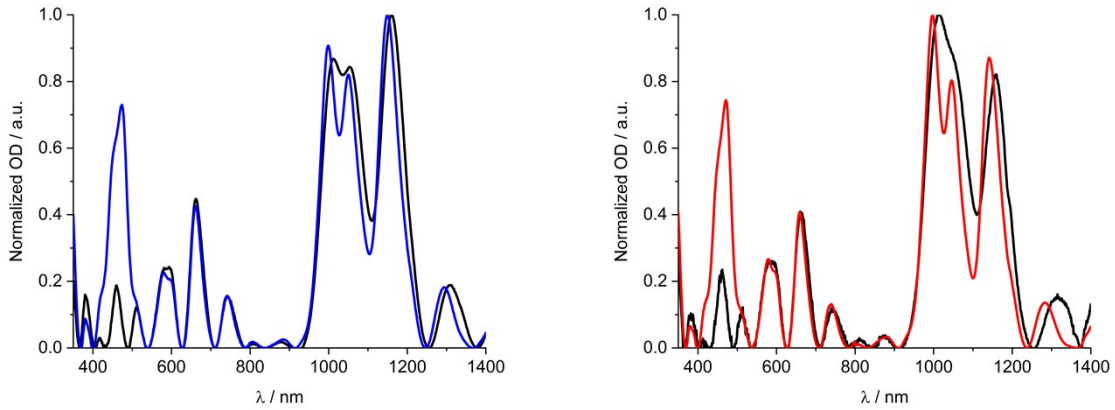


Figure S5: Normalized absorption spectra of the SWCNT reference (black) and **1**/SWCNT in DMF (left/blue) and ethanol (right/red) after subtraction of the scattering background.

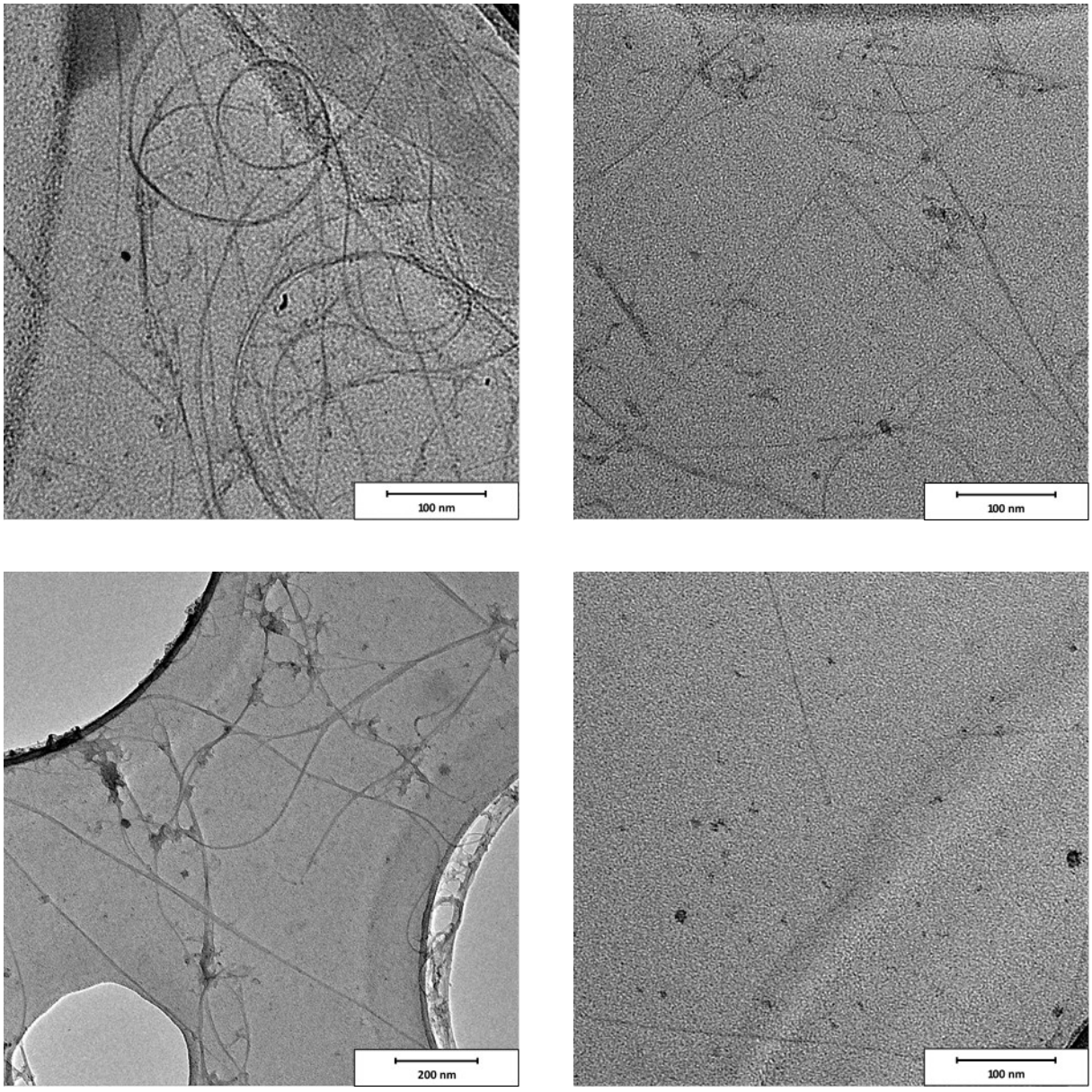
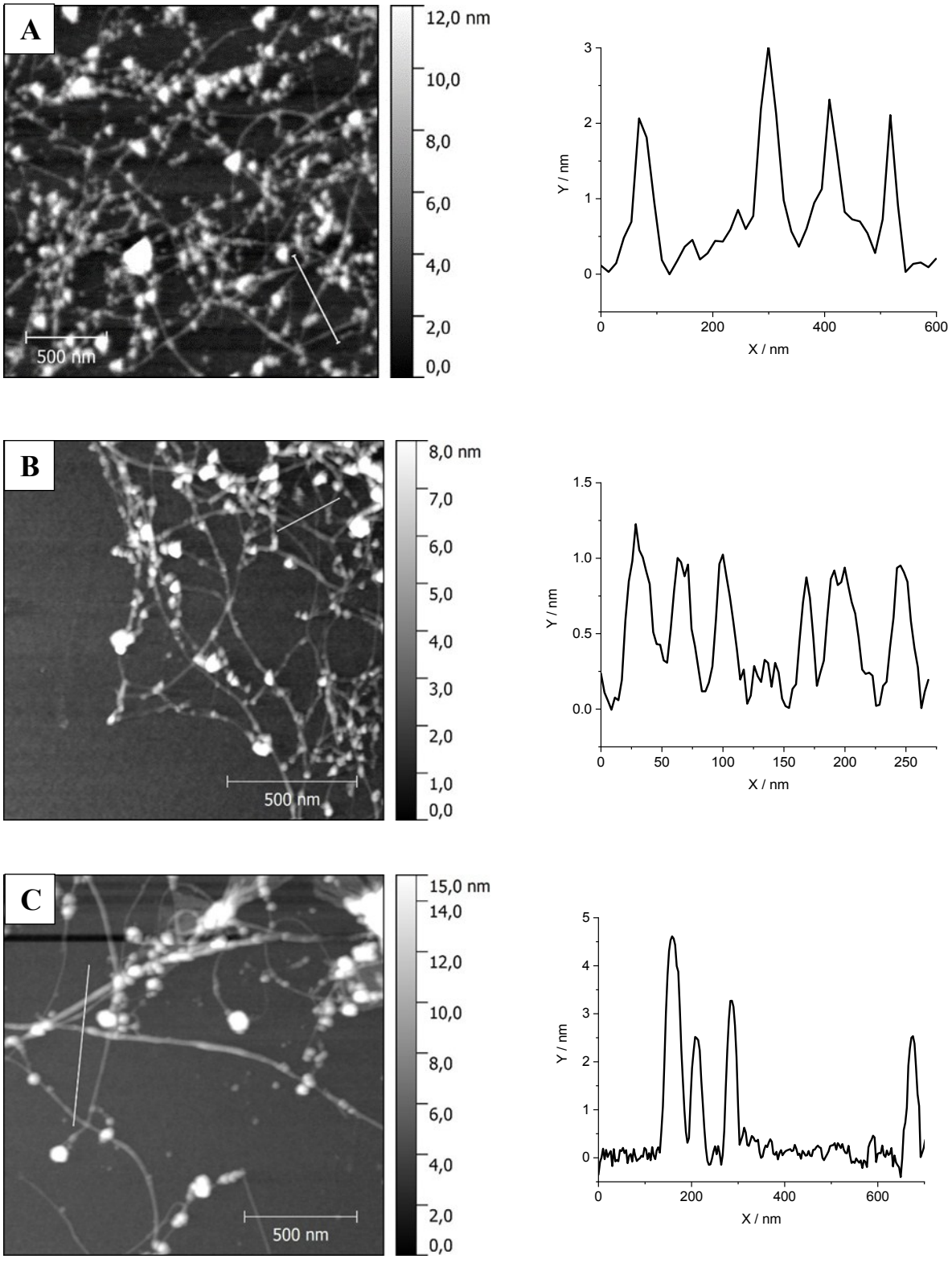


Figure S6: TEM images of a SWCNT reference (left) and **1**/SWCNT (right), drop-casted on a lacey carbon grid from DMF (top) and ethanol (bottom).


A
500 nm
12,0 nm
10,0
8,0
6,0
4,0
2,0
0,0
Y / nm
X / nm
B
500 nm
8,0 nm
7,0
6,0
5,0
4,0
3,0
2,0
1,0
0,0
Y / nm
X / nm
C
500 nm
15,0 nm
14,0
12,0
10,0
8,0
6,0
4,0
2,0
0,0
Y / nm
X / nm

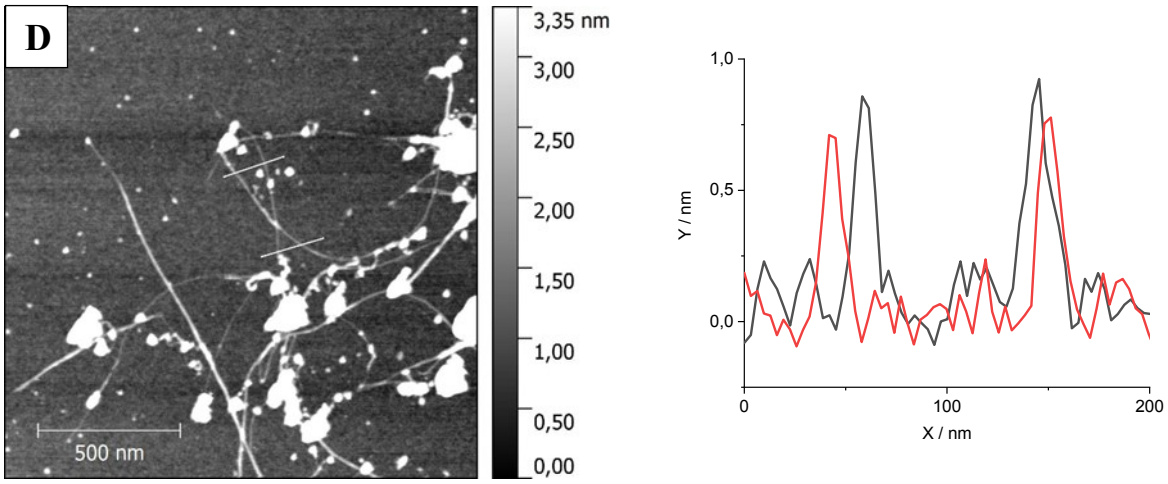


Figure S7: Tapping mode AFM images (left) and corresponding height profiles (right) of SWCNT (A) and **1**/SWCNT (B) drop-casted from DMF and SWCNT (C) and **1**/SWCNT (D) drop-casted from ethanol on a Si/SiO_2 surface.

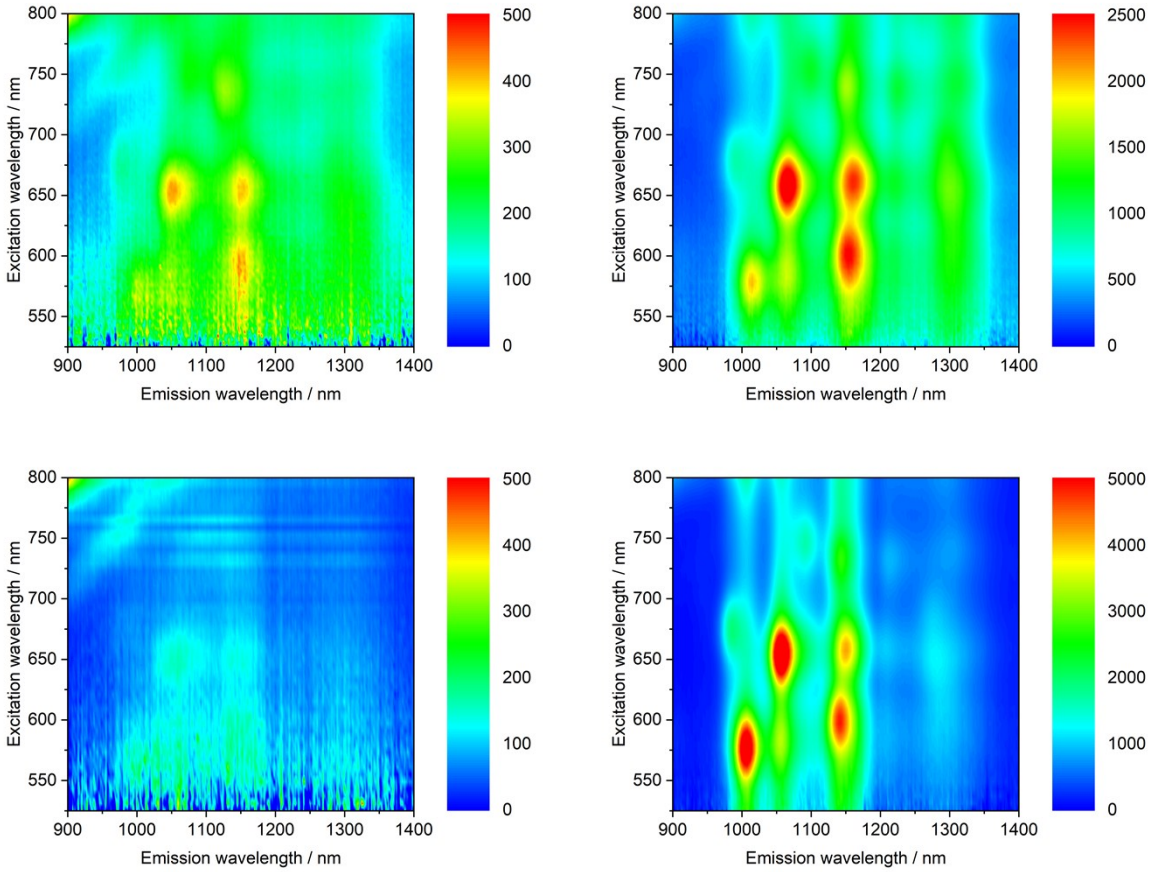


Figure S8: 3D nIR fluorescence spectra of a SWCNT reference (left) and **1**/SWCNT (right) in DMF (top) and ethanol (bottom).

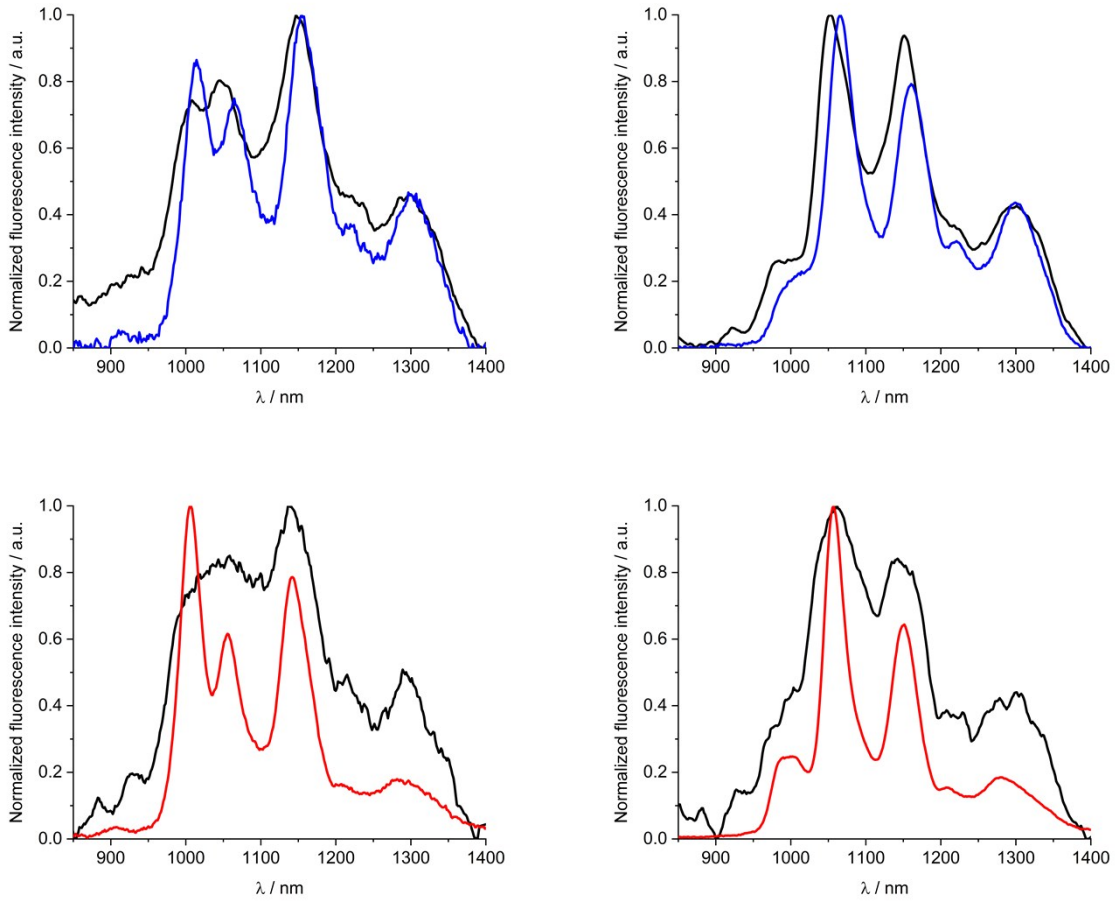


Figure S9: Normalized nIR fluorescence spectra of SWCNT (black) and **1**/SWCNT in DMF (top/blue) and ethanol (bottom/red) at 585 (left) and 657nm (right) excitation.

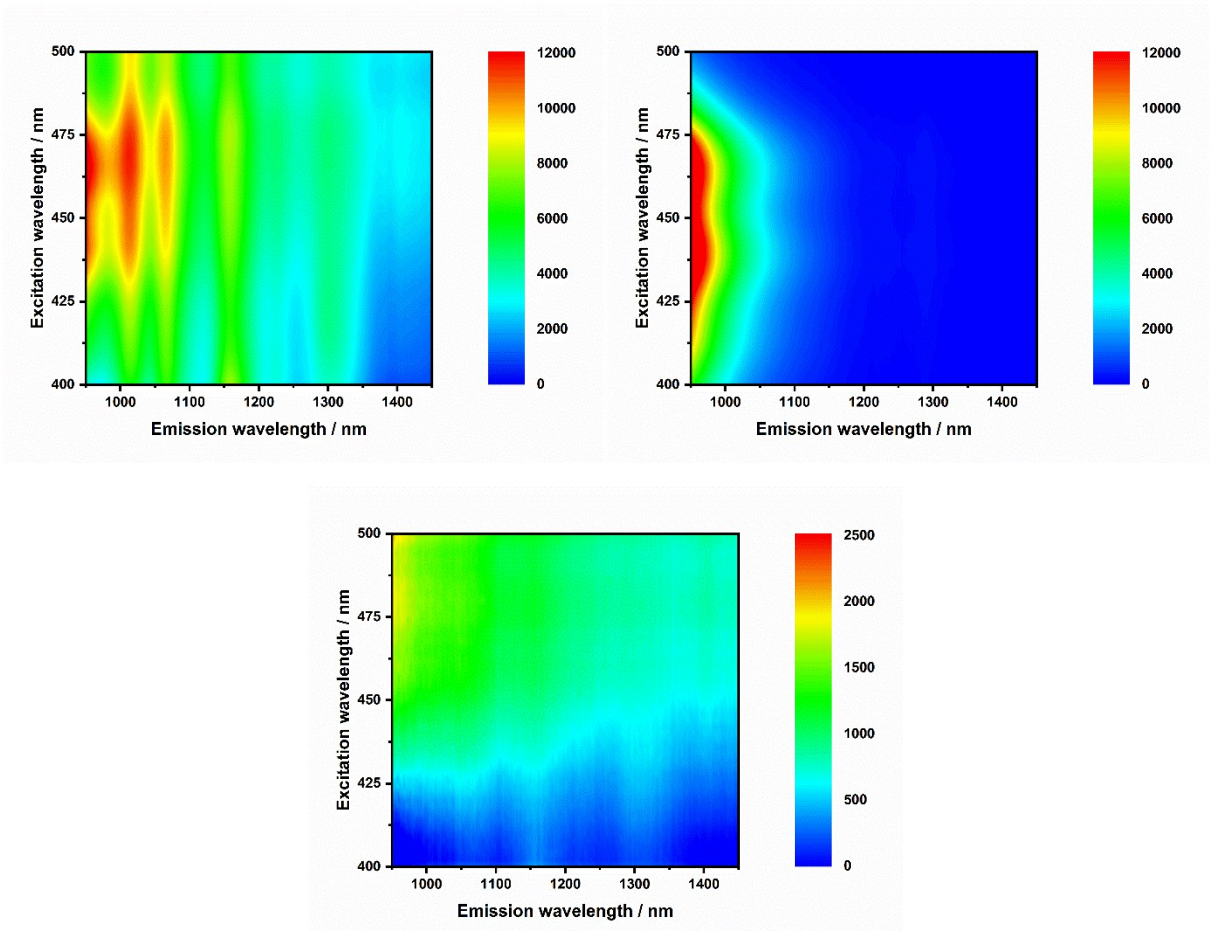


Figure S10: 3D nIR fluorescence spectra of **1**/SWCNT (top left), **1** (top right), and a SWCNT reference (bottom) in DMF.

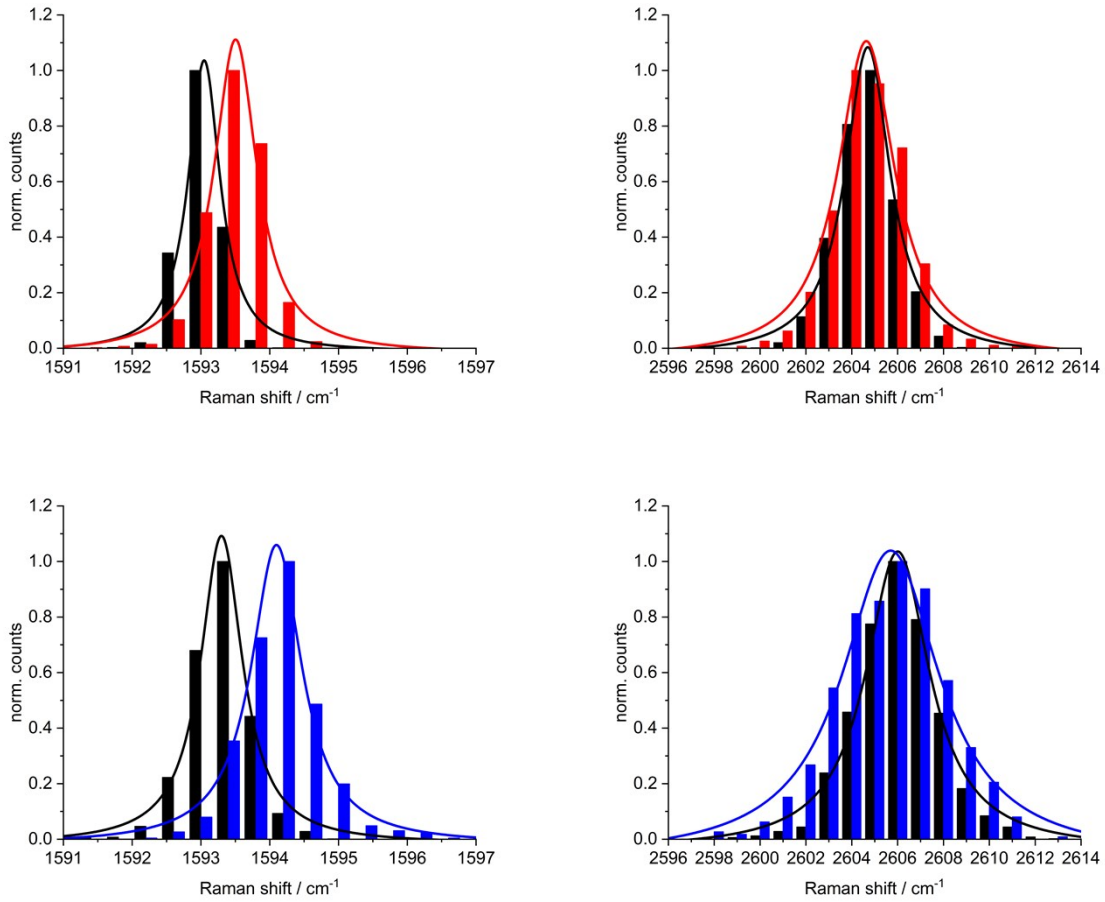


Figure S11: Histograms of Raman spectra for the G-band (left) and 2D-band (right) of the SWCNT reference (black) and **1**/SWCNT (red/blue) drop casted from DMF (top) or ethanol (bottom) on a Si/SiO_2 surface.

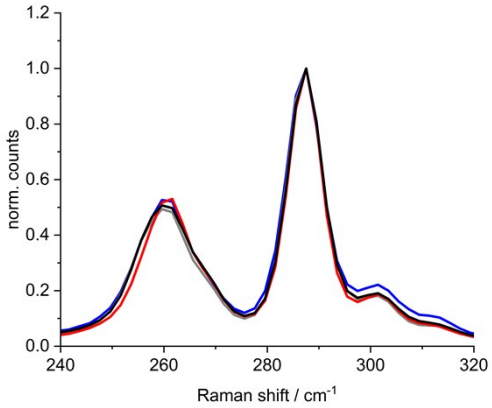


Figure S12: Radial breathing modes of SWCNT drop casted from DMF (black) or ethanol (grey) and **1**/SWCNT drop casted from DMF (red) or ethanol (blue) on a Si/SiO_2 surface.

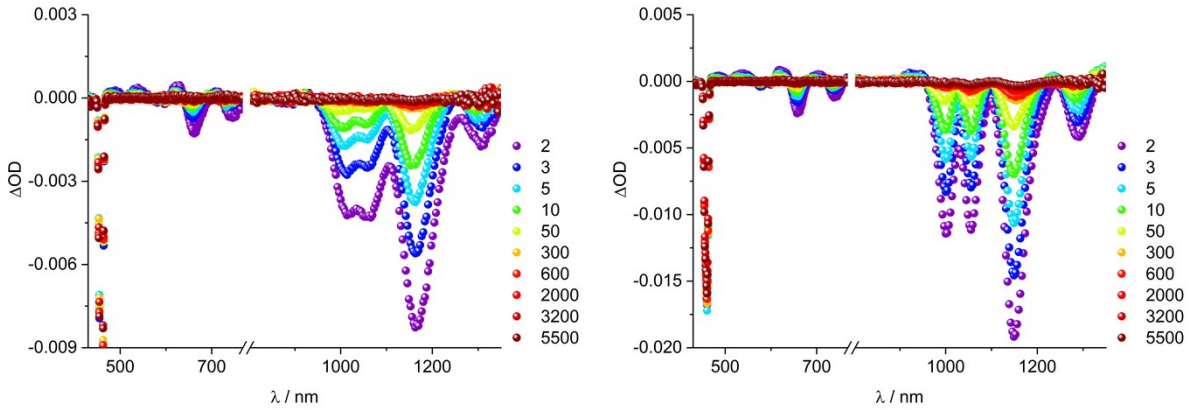


Figure S13: Differential absorption spectra obtained upon femtosecond pump-probe experiments (460 nm) of the SWCNT reference (left) and **1**/SWCNT (right) in DMF at room temperature with time delays between 2 and 5500 ps.

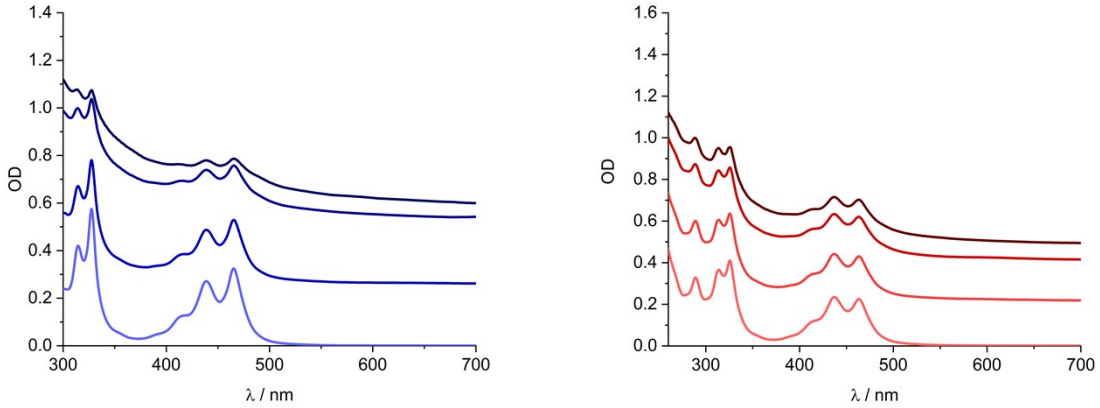


Figure S14: Absorption spectra of **1** in DMF (left) and ethanol (right) with increasing number of graphene enrichment cycles from blue/red to dark blue/dark red.

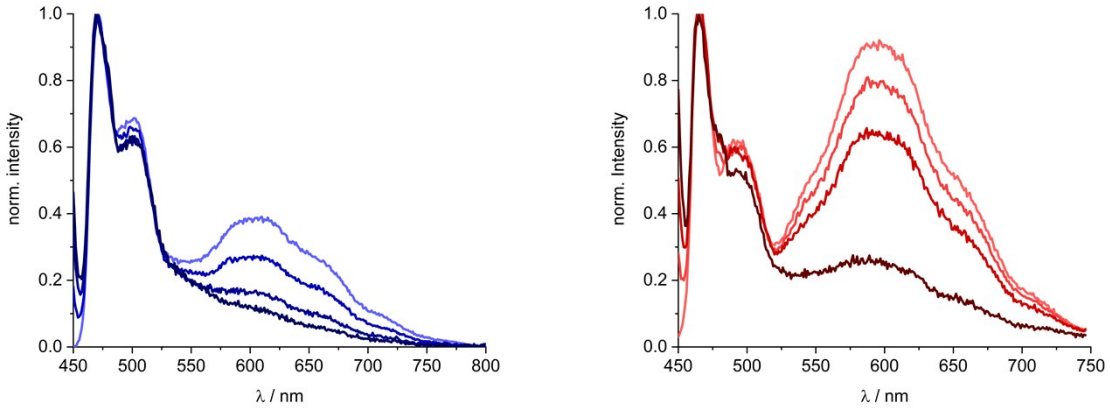


Figure S15: Normalized fluorescence spectra of **1** in DMF (left) and ethanol (right) with increasing number of graphene enrichment cycles from blue/red to dark blue/dark red.

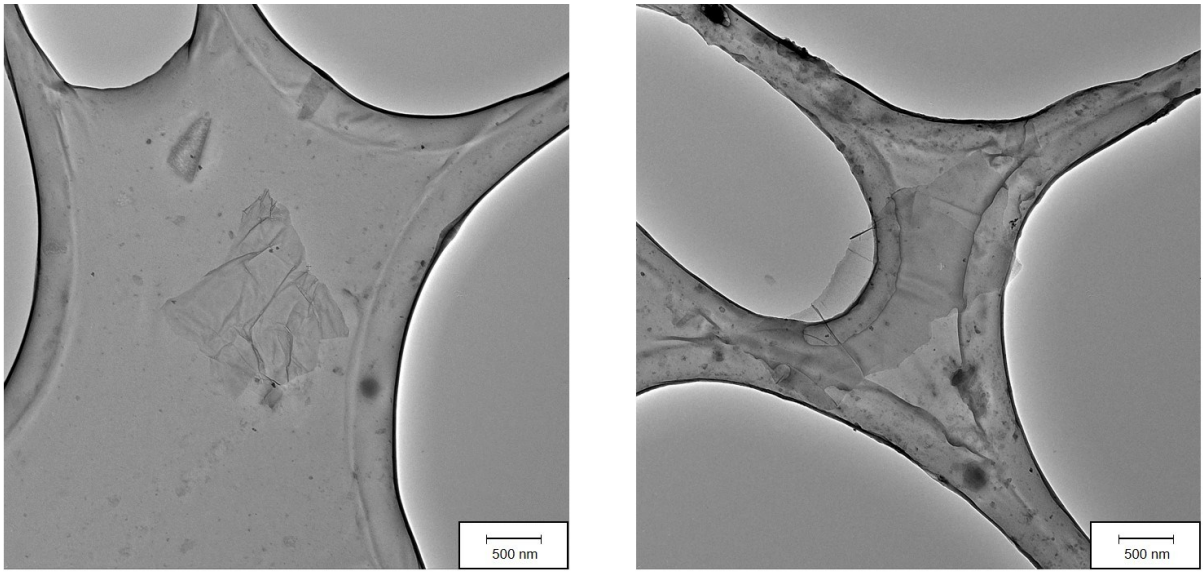


Figure S16: TEM image of **1**/NG in DMF (Left) and **1**/NG in ethanol (right) drop casted on a lacey carbon grid.

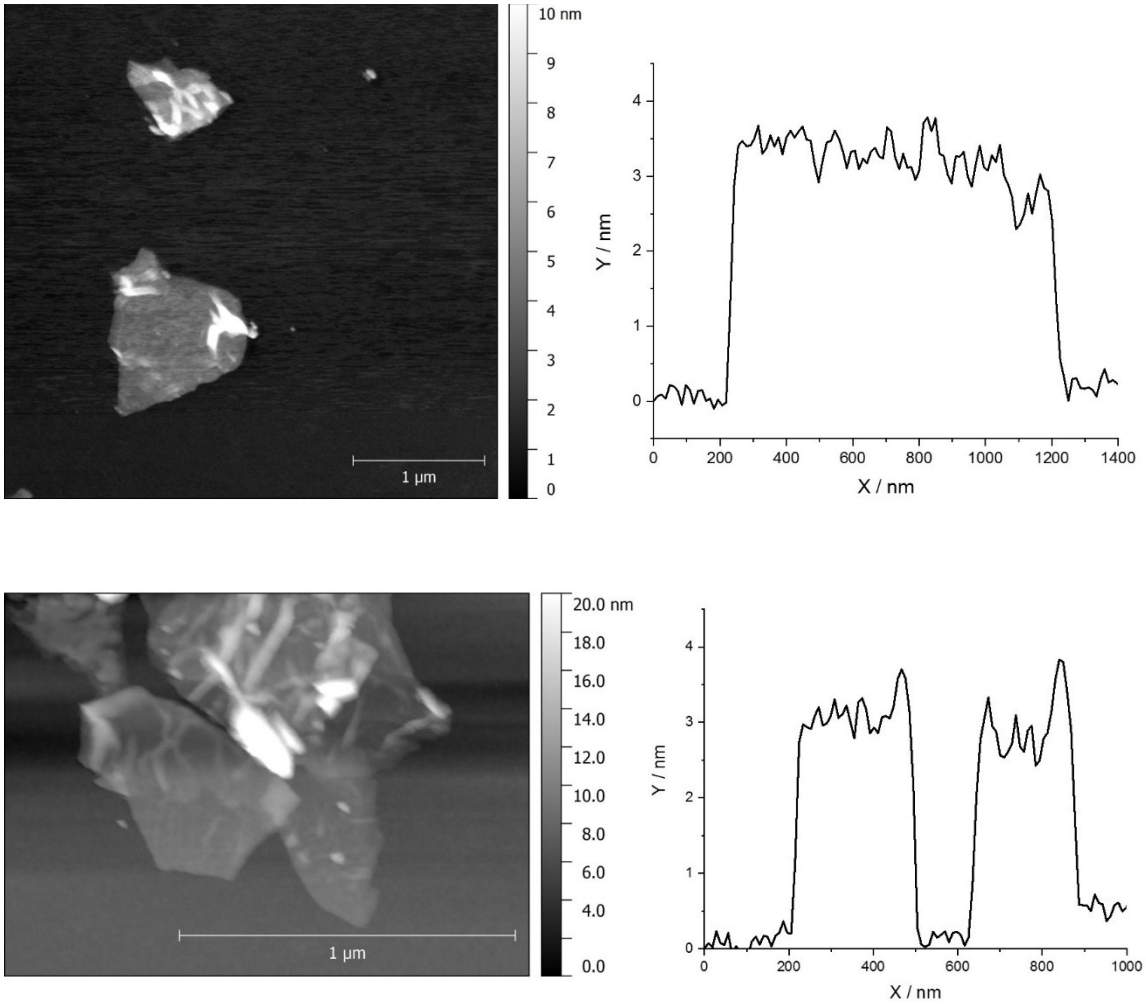


Figure S17: Tapping mode AFM images (left) and the corresponding height profiles (right) of **1**/NG (A) drop casted from DMF and **1**/NG (B) drop casted from ethanol on a Si/SiO_2 surface.

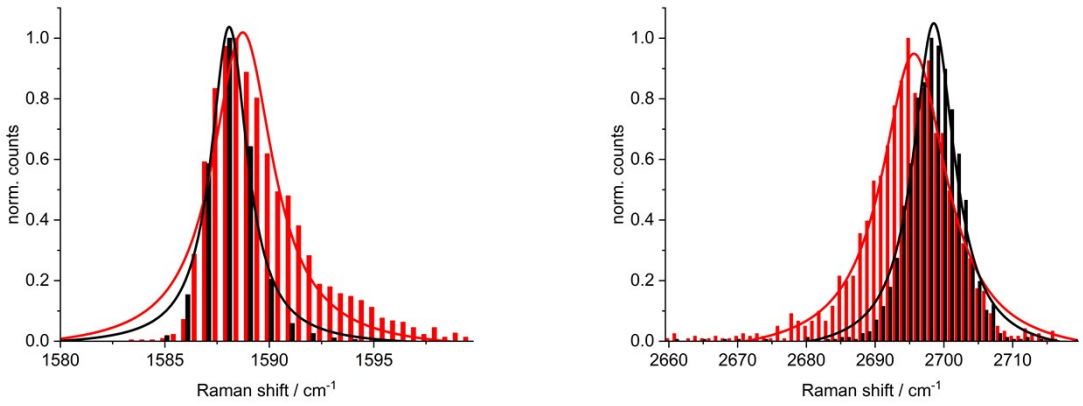


Figure S18: Histograms of Raman spectra for the G- band (left) and 2 D band (right) of the graphene reference (black) and **1**/NG (red) drop casted from DMF on a Si/SiO_2 surface.

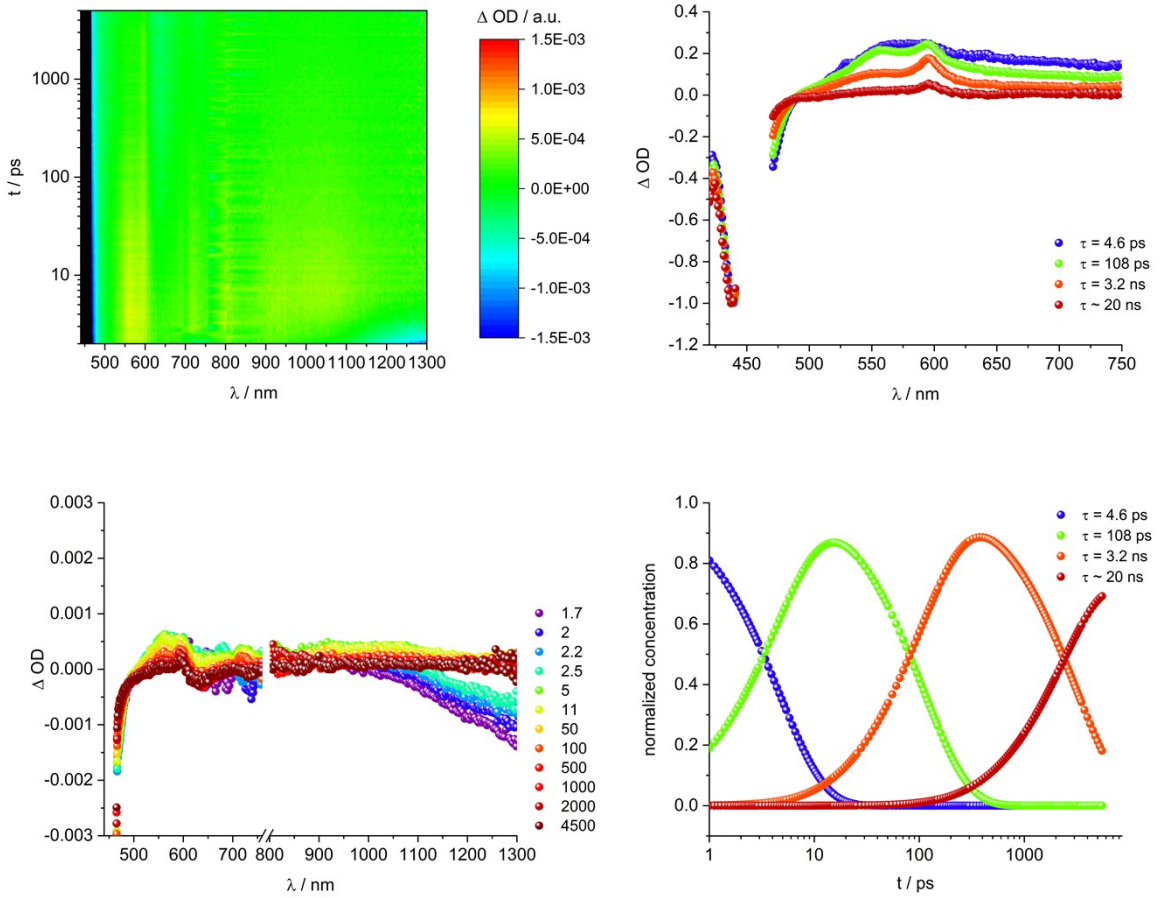


Figure S19: Differential absorption changes obtained upon femtosecond pump-probe experiments (460 nm) of **1**/NG in DMF at room temperature with time delays between 2 and 5000 ps (upper left). Exemplary time evolution spectra of **1**/NG in DMF at room temperature with time delays between 1.7 and 4500 ps (lower left) Corresponding evolution associated spectra of **1**/NG in DMF obtained by global analysis (upper right). Corresponding concentration profiles (lower right).