Electronic Supplementary Information for

Synergic effect of unsaturated inner bridges and polymorphism for tuning the optoelectronic properties of 2,3-thieno(bis)imide based materials.

Massimo Zambianchi,[a] Laura Favaretto, [a] Margherita Durso, [a] Cristian Bettini, [b] Alberto Zanelli, [a] Ilse Manet, [a] Massimo Gazzano, [a] Lucia Maini,[c] Denis Gentili, [d] Stefano Toffanin, [d] Federico Gallino, [e] Michele Muccini, [d,f] Massimiliano Cavallini, [d,*] Manuela Melucci [a,*]

[a] *Consiglio Nazionale delle Ricerche, Istituto per la Sintesi Organica e la Fotoreattività, (CNR-ISOF), via P. Gobetti 101, 40129 Bologna, Italy*

[b] *MIST-ER Laboratory, via P. Gobetti 101, 40129 Bologna, Italy*

[c] *Dipartimento di Chimica "G. Ciamician", Università di Bologna, Via F. Selmi 2, 40126, Bologna, Italy*

[d] *Consiglio Nazionale delle Ricerche, Istituto per lo Studio dei Materiali Nanostrutturati (CNR-ISMN), via P. Gobetti 101, 40129 Bologna, Italy*

[e] *SAES-GETTERS S. p. A., Viale Italia 77, 20020 Lainate - MI – Italy*

[f] *E.T.C. s.r.l., via P. Gobetti 101, 40129 Bologna, Italy*

Table of Contents

1. SYNTHESIS

1.1 General Methods

Liquid chromatography purification were performed by flash chromatography using glass column packed with Merk silica gel 60 (230-400 mesh), or on a Teledyne-Isco CombiFlash Rf 200 system using RediSep Rf normal-phase silica columns. TLC was carried out on Merk silica gel 60 F254 plates; detection by UV (254 nm, and 365 nm). Melting points (uncorrected) were determined on a 'hot-stage' apparatus where the melting process was observed with the aid of a microscope. All NMR spectra were recorded at room temperature using a Varian Mercury 400 spectrometer (^{1}H: 400 MHz; ^{13}C: 100.6 MHz). Chemical shifts are referenced to the residual solvent signal ($CHCl_3$: δ ^{1}H = 7.26 ppm, δ^{13}C = 77.16 ppm), respectively. Mass spectra were obtained with a Trace 1300 GC coupled to an ISQ mass spectrometer (Thermo Scientific) controlled by a computer running XCalibur software. According to the mass to be detected two different analytical approaches were employed, gas chromatography mass spectrometry (GC-MS) and direct exposure electron ionization mass spectrometry (DE-MS). UV-Vis spectra were recorded using a Perkin Elmer Lambda 20 spectrometer. Photoluminescence spectra were obtained with a Perkin Elmer LS50B spectrofluorometer using an excitation wavelength corresponding to the maximum absorption lambda. Cyclic Voltammetric measurements were performed at room temperature with an AMEL System 5000.

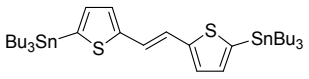
1.2 Chemicals and Solvents

Titanium (IV) chloride (*Fluka 99%*), zinc powder (*Aldrich 99.9%*), butyllithium (*Aldrich 2.5 M in hexanes*), *N,N,N',N'*-tetramethylethylenediamine (TMEDA, *Alfa Aesar 99.5%*), tributyltin chloride (TBTC, *Aldrich* 96%), tris(dibenzylideneacetone)dipalladium(0)-chloroform adduct $Pd_2(dba)_3 \cdot CHCl_3$, *Aldrich*), triphenylarsine ($AsPh_3$, *Aldrich 97%*), 2-thiophenecarboxaldheyde (*Aldrich 98%*), 2-thiophenecarboxylic acid (**7**) (*Acros Organics 99%*), thionyl chloride ($SOCl_2$, *Fluka 99%*), lithium diisopropylamide solution (LDA, *Acros Organics 2.0 M in THF/heptanes/ethylbenzene*), triethylamine (TEA, *Aldrich 99%*), diphenyl phosphoryl azide (DPPA, *Aldrich 97%*), trifluoroacetic acid (TFA, *Aldrich 99%*), ethylene glycol (*Aldrich 99.8%*), *p*-toluenesulfonic acid monohydrate (*p*-TsOH, *Aldrich 98.5%*), bis(triphenylphosphine)palladium(II) dichloride ($(PdCl_2(PPh_3)_2$, *Aldrich 99%*), copper(I) iodide (*Aldrich 99.5%*), 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU, *Aldrich 99%*), trimethylsilylacetilene (*Aldrich 98%,*), 2-iodothiophene (*Aldrich*, 98%) were used as received. *(E)-1,2-Di(thiophen-2-yl)ethane* (**4**), [1] 2-Bromo-5-hexyl-4H-thieno[2,3-c]pyrrole-4,6(5H)-dione (**6**),[2] *(5-(1,3-Dioxolan-2-yl)thiophen-2-yl)tributylstannane*, (**13**)[3] and *1,2-Bis(5-*

(tributylstannyl)thiophen-2-yl)ethyne (**15**), [4] were prepared according to previously reported methods.

1.3 Synthesis of NTE precursors 5

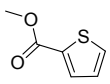
(E)-1,2-Bis(5-(tributylstannyl)thiophen-2-yl)ethane, (**5**)



The method reported in ref. 5 for the synthesis of this compound was modified as follow: To a solution of **4** (0.90 g, 4.69 mmol) in THF (25 ml), TMEDA (1.57 ml, 10.30 mmol) was added at -50 °C. After cooling to -78 °C a 2.5 M solution of *n*-butyllithium (4.31 ml, 10.80 mmol) was added dropwise. The mixture was then refluxed for 1 h. The reaction was cooled to -78 °C and TBTC (2.76 ml, 9.80 mmol) was added. The solution was warmed to room temperature and stirred overnight. The solvent was removed in vacuo, then the crude was dissolved in DCM (20 ml) and washed in water (2 x 20 ml). After drying (sodium sulfate) and removal of the solvent, the target material was obtained as a light brown oil in 98% yield. The material was virtually pure (^{1}H NMR) and was used without purification in the subsequent step. DE-MS (*m/z*): 770 ($M^{\cdot+}$); ^{1}H NMR ($CDCl_3$), δ [ppm]: 7.12 (d, ^3J= 3.2 Hz, 2H), 7.10 (s, 2H), 7.04 (d, ^3J= 3.2 Hz, 2H), 1.55 (m, 12H), 1.33 (m, 12H), 1.11 (m, 12H), 0.91 (m, 18H).

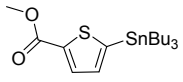
1.4 Synthesis of NTI precursors 7a, 8-12, 14

Methyl thiophene-2-carboxylate, (**7a**)



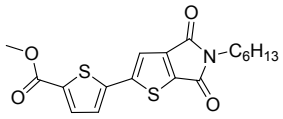
The protocol reported in ref. 6 was modified as follows: To a solution of commercial 2-thiophenecarboxylic acid **7** (1.0 g ; 7.8 mmol), in 50 ml of anhydrous MeOH under nitrogen, $SOCl_2$ (1.13 ml, 15.60 mmol) was added dropwise at 0 °C. Then the mixture was warmed to reflux for 5 h. The solvent was removed, and the crude product was purified by flash chromatography (silica gel, 7:3 pentane/EtOAc) to yield 0.89 g (80%) of a colorless oil. GC-MS (*m/z*): 142 ($M^{\cdot+}$); ^{1}H NMR ($CDCl_3$), δ [ppm]: 7.80 (dd, ^3J= 4.0 Hz, ^4J= 1.6 Hz 1H), 7.55 (dd, ^3J= 5.2Hz, ^4J=1.2 Hz 1H), 7.10 (dd, ^3J= 4.8 Hz, ^3J= 4.0 Hz 1H), 3.89 (s, 3H); ^{13}C NMR ($CDCl_3$), δ [ppm]: 162.7, 133.6, 133.4, 132.3, 127.7, 52.1.

Methyl 5-(tributylstannyl)thiophene-2-carboxylate, (**8**)



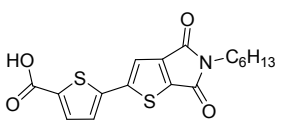
The protocol reported in ref. 7 was modified as follows: To a stirred 2M solution of LDA (2.5 ml, 5.04 mmol) at –78 °C under an N_2 atmosphere was added dropwise a solution of **7a** (0.6 g, 4.2 mmol) in dry THF (3 ml). After 1 hour, a solution of TBTC (1.5 ml, 4.62 mmol) in dry THF (4 ml) was added dropwise. After being stirred at -78 °C for further 2 h, the mixture was allowed to warm to rt, and the stirring was continued overnight. The resulting mixture was evaporated under reduced pressure, then diluted with DCM, and washed twice with water. After drying over sodium sulfate and evaporation of the solvent, the residue was purified by distillation (142-150 °C, 0.1 mmHg) to give the desired compound (725 mg, yield 40%) as a pale yellow oil. DE-MS (*m/z*): 400 ($M^{\cdot+}$ -OMe); ^{1}H NMR ($CDCl_3$), δ [ppm]: 7.88 (d, ^3J= 3.6 Hz, 1H), 7.15 (d, ^3J=3.6Hz, 1H), 3.88 (s, 3H), 1.55 (m, 6H), 1.33 (m, 6H), 1.12 (m, 6H), 0.89 (t, 9H); ^{13}C NMR ($CDCl_3$), δ [ppm]: 162.6, 147.3, 138.6, 135.7, 134.1, 52.0, 28.8, 27.2,13.6, 10.9.

Methyl 5-(5-hexyl-4,6-dioxo-5,6-dihydro-4H-thieno[2,3-c]pyrrol-2-yl)thiophene-2-carboxylate, (**9**)



To a refluxing toluene solution (13 ml) of bromo thienoimide **6** (200 mg, 0.63 mol) and in situ-prepared $Pd(AsPh_3)_4$ (8% mol, 26 mg of Pd_2dba_3 and 61 mg of $AsPh_3$) under N_2 atmosphere, **8** (300 mg, 0.70 mmol) in toluene (1.5 ml), was added dropwise. The solution was refluxed for 6 h then, after cooling and solvent evaporation, the crude product was purified by flash chromatography (silica gel, Pentane/EtOAc 80:20). The titled compound was obtained as a pale yellow powder (190 mg, yield 80%). DE-MS (*m/z*): 377 ($M^{\cdot+}$); ^{1}H NMR ($CDCl_3$), δ [ppm]: 7.74 (d, ^3J=4.0 Hz 1H), 7.41 (s, 1H), 7.29 (d, ^3J= 4.0 Hz 1H), 3.92 (s, 3H), 3.61 (t, 2H), 1.64 (m, 2H), 1.31 (m, 6H), 0.88 (t, 3H); ^{13}C NMR ($CDCl_3$), δ [ppm]: 163.7, 163.2, 161.9, 148.6, 145.1, 141.2, 134.2, 126.1, 117.9, 108.4, 52.5, 38.7, 31.3, 28.7, 26.4, 22.5, 14.0.

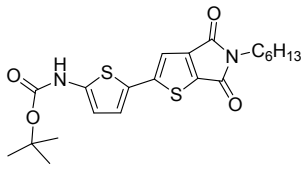
5-(5-Hexyl-4,6-dioxo-5,6-dihydro-4H-thieno[2,3-c]pyrrol-2-yl)thiophene-2-carboxylic acid, (**10**)



To a solution of **9** (180 mg, 0.48 mmol) in MeOH (4 ml), 1 N aq. NaOH (4 ml) was added dropwise at rt. The mixture was refluxed for 2 h. Methanol was removed on a rotary evaporator, then 4 ml of deionized water was added. The resulting mixture was brought to a pH below 2 with HCl, and the white solid formed was centrifuged, washed 2 times with water and dried at 50 °C. The solid was refluxed for 3 h in $SOCl_2$ (10 ml), then the solvent was removed by distillation. Distilled water (10 ml) was added dropwise, and the yellow solid formed was dissolved in DCM (10 ml). After separation of the two layers, the organic phase was washed with water (3 x 20 ml), dried and evaporated. The desiderated compound **10** was obtained as a yellow powder (170 mg, yield 98%). DE-MS (*m/z*): 363 ($M^{\cdot+}$); ^{1}H

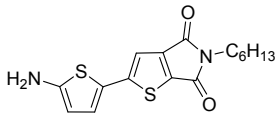
NMR ($CDCl_3$), δ [ppm]: 7.92 (d, ^3J=4.0 Hz 1H), 7.49 (s, 1H), 7.36 (d, ^3J=4.4 Hz 1H), 3.61 (t, 2H), 1.63 (m, 2H), 1.30 (m, 6H), 0.87 (t, 3H); ^{13}C NMR ($CDCl_3$), δ [ppm]: 163.3, 162.2, 159.2, 147.2, 146.1, 145.1, 140.3, 138.4, 137.3, 126.5, 119.0, 38.8, 31.3, 28.7, 26.4, 22.5, 14.0.

Tert-butyl (5-(5-hexyl-4,6-dioxo-5,6-dihydro-4H-thieno[2,3-c]pyrrol-2-yl)thiophen-2-yl)carbamate, (**11**)



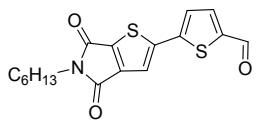
To a solution of compound **10** (176 mg, 0.48 mmol) and TEA (48 mg, 0.48 mmol) in t-BuOH (3 ml), DPPA (134 mg, 0.49 mmol) was added dropwise at room temperature. After refluxing for 6 h, the reaction was quenched with deionized water (10 ml). The mixture was extracted into DCM (3 x 20 ml), and the combined organic layers were washed three time with water, dried over Na_2SO_4, filtered and concentrated in vacuo. The crude product was purified by flash chromatography (silica gel, Pentane/EtOAc 80:20) to give the titled compound as an orange-red solid (88 mg, 42%). DE-MS (*m/z*): 434 ($M^{\cdot+}$); ^{1}H NMR ($CDCl_3$), δ [ppm]: 7.51 (bs, 1H), 7.16 (s, 1H), 7.04 (d, ^3J= 4.0 Hz, 1H), 6.43 (d, ^3J=4.0 Hz, 1H), 3.56 (t, 2H), 1.61 (m, 2H), 1.52 (s, 9H), 1.30 (m, 6H), 0.88 (t, 3H); ^{13}C NMR ($CDCl_3$), δ [ppm]: 164.1, 163.0, 152.0, 145.3, 142.5, 135.6, 129.8, 125.8, 123.8, 114.0, 38.4, 31.3, 29.6, 28.7, 28.2, 26.4, 22.5, 14.0.

2-(5-Aminothiophen-2-yl)-5-hexyl-4H-thieno[2,3-c]pyrrole-4,6(5H)-dione, (**12**)



To a 10 ml DCM solution of **11** (80 mg, 0.18 mmol), were added 10 ml of TFA while stirring. After 5 h at rt, the solution was brought to basic pH by introduction of 1N aq. NaOH. The organic phase was then washed with deionized water until neutral, dried over anhydrous sodium sulfate, and evaporated to give compound **12** in almost quantitative yield (60 mg) as a red solid. The product was stored at 4 °C under nitrogen, and used after short time for the subsequent reaction. DE-MS (*m/z*): 334 ($M^{\cdot+}$); ^{1}H NMR ($CDCl_3$), δ [ppm]: 7.05 (s, 1H), 6.95 (d, ^3J= 4.0 Hz, 1H), 6.11 (d, ^3J= 4.0 Hz, 1H), 4.08 (bs, 2H), 3.57 (t, 2H), 1.60 (m, 2H), 1.28 (m, 6H), 0.87 (t, 3H);

5-(5-Hexyl-4,6-dioxo-5,6-dihydro-4H-thieno[2,3-c]pyrrol-2-yl)thiophene-2-carbaldehyde, (**14**)



To a refluxing toluene solution (8 ml) of compound **6** (100 mg, 0.32 mmol) and in situ-prepared $Pd(AsPh_3)_4$ (8% mol, 13 mg of Pd_2dba_3 and 32 mg of $AsPh_3$) under N_2 atmosphere, **13** (148 mg, 0.33 mmol) in toluene (1 ml), was added dropwise. The solution was refluxed for 6 h then, after cooling and solvent evaporation, the

crude product was purified by flash chromatography on silica gel (eluent Pentane/DCM/EtOAc 85:15:15) to afford the titled product as a yellow powder (109 mg, yield 98%). DE-MS (*m/z*): 347 ($M^{\cdot+}$); ^{1}H NMR ($CDCl_3$), δ [ppm]: 9.90 (s, 1H), 7.72 (d, ^{3}J= 4.4 Hz 1H), 7.46 (s, 1H), 7.39 (d, ^{3}J=4.0 Hz 1H) 3.59 (m, 2H), 1.62 (m, 2H), 1.29 (m, 6H), 0.88 (t, 3H); ^{13}C NMR ($CDCl_3$), δ [ppm]: 182.3, 163.4, 162.2, 148.0, 145.0, 144.0, 143.8, 139.8, 136.7, 126.3, 118.6, 38.7, 31.3, 28.6, 26.4, 22.5, 13.9. (yield 98%). DE-MS (*m/z*): 347 ($M^{\cdot+}$); ^{1}H NMR ($CDCl_3$), δ [ppm]: 9.90 (s, 1H), 7.72 (d, ^{3}J= 4.4 Hz 1H), 7.46 (s, 1H), 7.39 (d, ^{3}J=4.0 Hz 1H) 3.59 (m, 2H), 1.62 (m, 2H), 1.29 (m, 6H), 0.88 (t, 3H); ^{13}C NMR ($CDCl_3$), δ [ppm]: 182.3, 163.4, 162.2, 148.0, 145.0, 144.0, 143.8, 139.8, 136.7, 126.3, 118.6, 38.7, 31.3, 28.6, 26.4, 22.5, 13.9.

2. CYCLIC VOLTAMMETRY

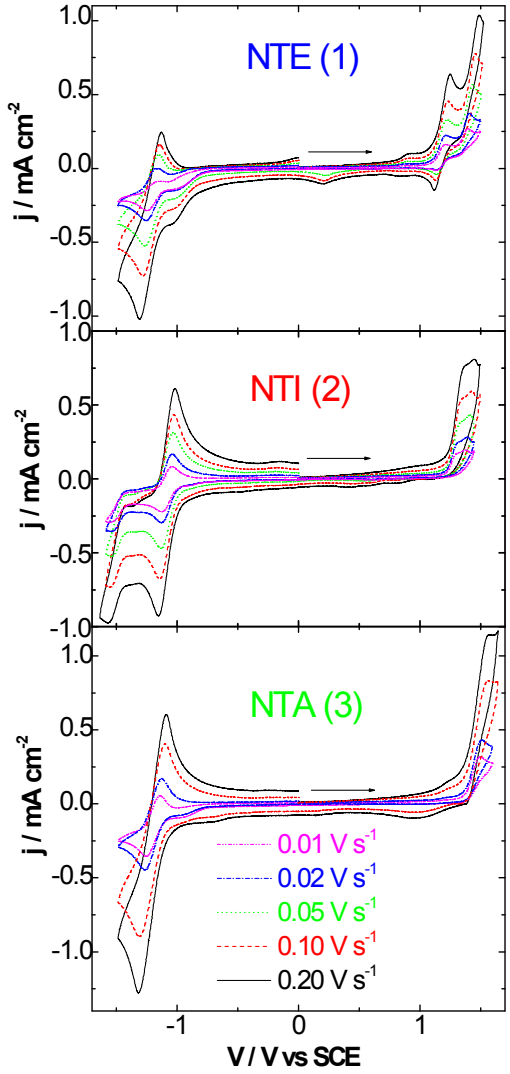


Figure S1. CVs of NTE, NTI and NTA at several scan rate in CH_2Cl_2 0.1 M $(C_4H_9)_4NClO_4$

CVs of NTE, NTI, and NTA have been carried out at scan rates ranging from 0.01 to 0.2 V s^{-1}. At 0.2 V s^{-1}, NTE has two quasi-reversible oxidation waves with the maxima at 1.25 and 1.50 V, respectively. In the voltammogram of NTI, these two waves became irreversible and the maxima shift to 1.39 and 1.45 V, respectively. At the slower scan rates the peaks are better resolved even if their distance remains about 60 nV. Finally, the voltammogram of NTA shows only an irreversible oxidation wave with the maximum at 1.57 V indicating that the acetylene-core decreases the conjugation of the molecule. The quasi-reversible reduction waves of NTE and NTA show the maxima at about the same potential (1.31 and 1.32 V) of other already described thienoimide based molecules. Whereas, the immune-core induces a significant shift of the LUMO energy and the voltammogram of NTI shows a quasi-reversible reduction wave with the maximum at -1.15 V, and

a further quasi-reversible wave at -1.56 V. Figure S1 depicts in detail the voltammograms of the three molecules at scan rates ranging from 0.2 to 0.01 V s^{-1}.

3. THERORETICAL CALCULATIONS

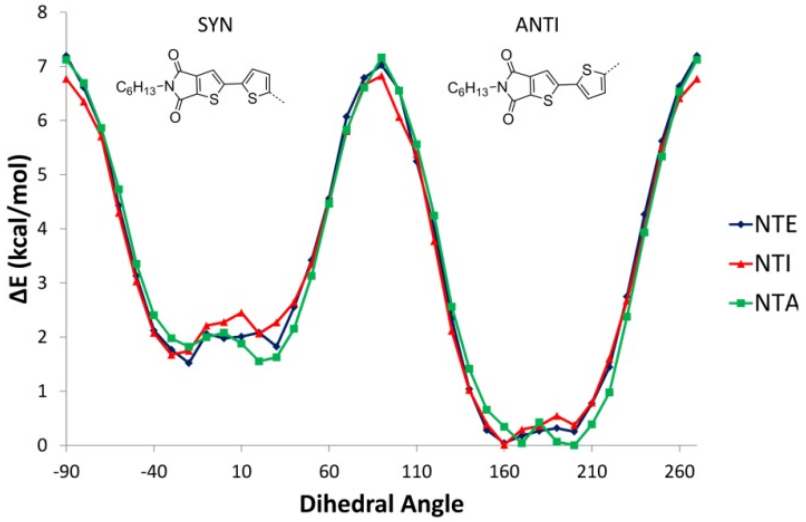


Figure S2. Conformation energy plot as a function of the dihedral angle between the thienoimide end moiety and the inner thiophene ring.

The conformation energy plot has been obtained from single point calculations starting from the optimized geometry at varying the dihedral angle between 2,3-thienoimide moiety and the inner thiophene ring and keeping the symmetry point group (resulting in concomitant rotation of two symmetric dihedral angles).

4. FLUORESCENCE LIFETIME IMAGING

Fluorescence lifetime imaging (FLIM) was performed on the Nikon A1 laser scanning confocal microscope (Nikon Co., Shinjuku, Japan) equipped with a 485 nm pulsed/CW diode laser (PicoQuant GmbH, Berlin, Germany). Images were collected using a Nikon Plan Apo VC 60 oil immersion objective with NA 1.40. Fluorescence decays were collected with integrated PicoHarp300 electronics (PicoQuant GmbH, Berlin, Germany) for TCSPC measurements. Two single-photon avalanche diode detectors equipped with bandpass filters were set to register the fluorescence of different samples in the ranges 500-535 nm, 565-605 nm and 635-675 nm. The repetition rate of the pulsed excitation at 485 nm was 40 MHz. The instrument response function of the system is approximately 200 ps. A tail fit was performed on the histogram calculated for a

region of interest of the sample image. Fluorescence decay profiles were analyzed with a least-squares method, using multiexponential decay functions. The fitting function used is

$I(t) = b + \Sigma_j a_j e(-t/\tau_j)$ with j ranging from 1 to 2

The fractional intensity and the average fluorescence lifetime are calculated according to the following equations:

$f_i = a_i\tau_i / \Sigma_j a_j\tau_j \qquad \tau_{av} = \Sigma_j f_j\tau_j$

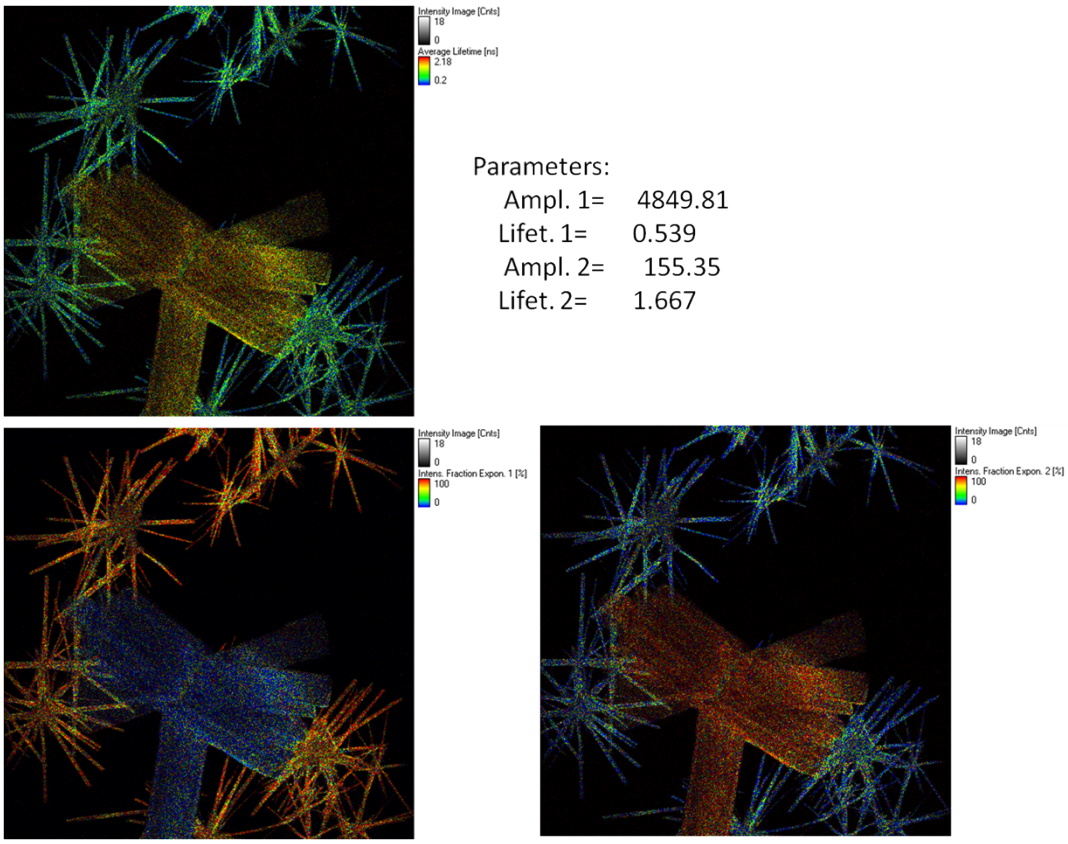


Figure S3. Average lifetime (top) as well as the fractional intensity of the two lifetimes, 0.54 ns (bottom left) and 1.67 ns (bottom right), for the different morphologies of NTE films. Parameters obtained from biexponential fit of the decay are indicated (tau in ns).

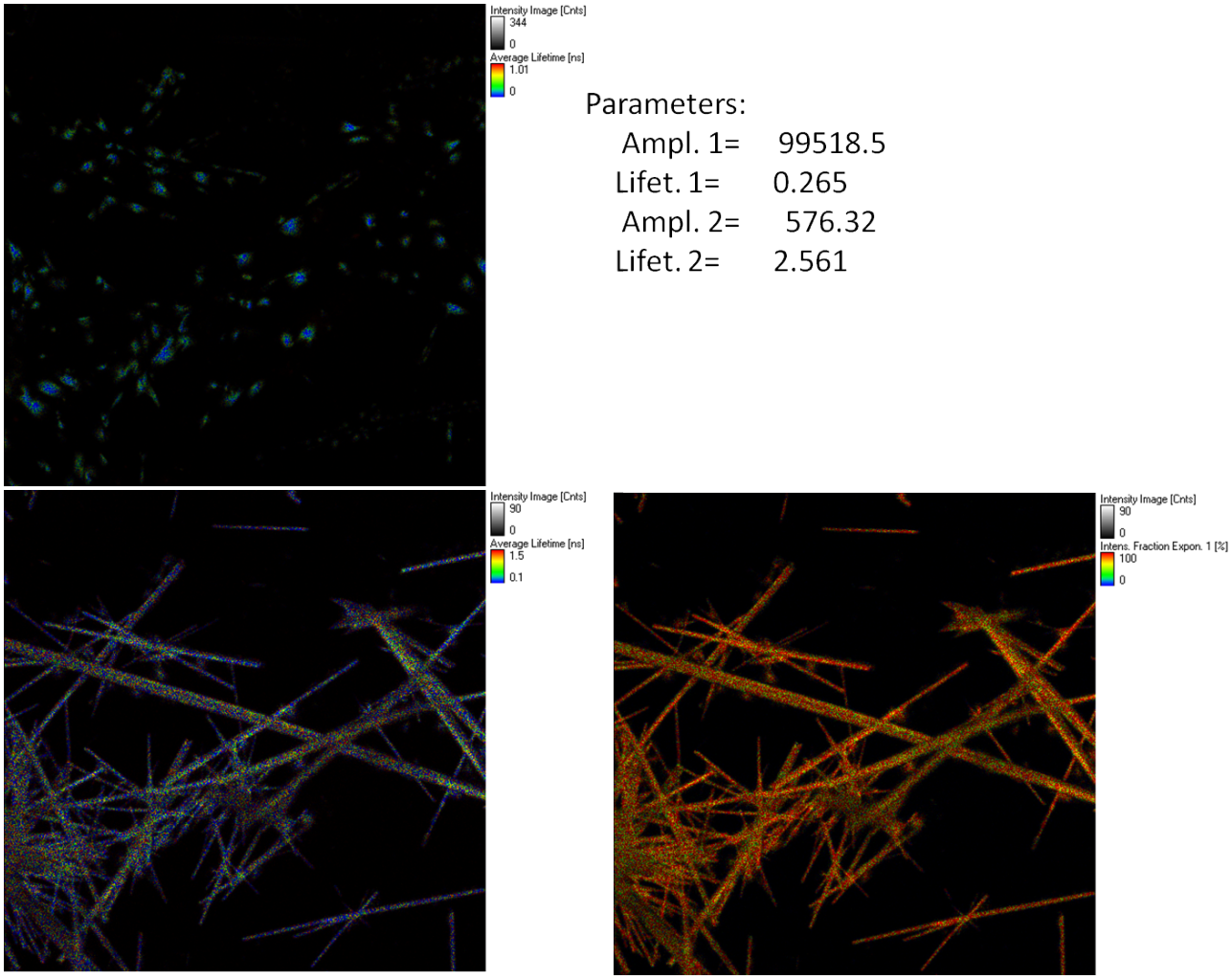


Figure S4. Average lifetime for the clusters (top) measuring emission at 520 nm, as well as the average lifetime (bottom left) and fractional intensity (bottom right) of the short lifetimes for the different morphologies of NTI films, obtained for fluorescence collected at 655 nm. Parameters obtained from biexponential fit of the decay are indicated (tau in ns).

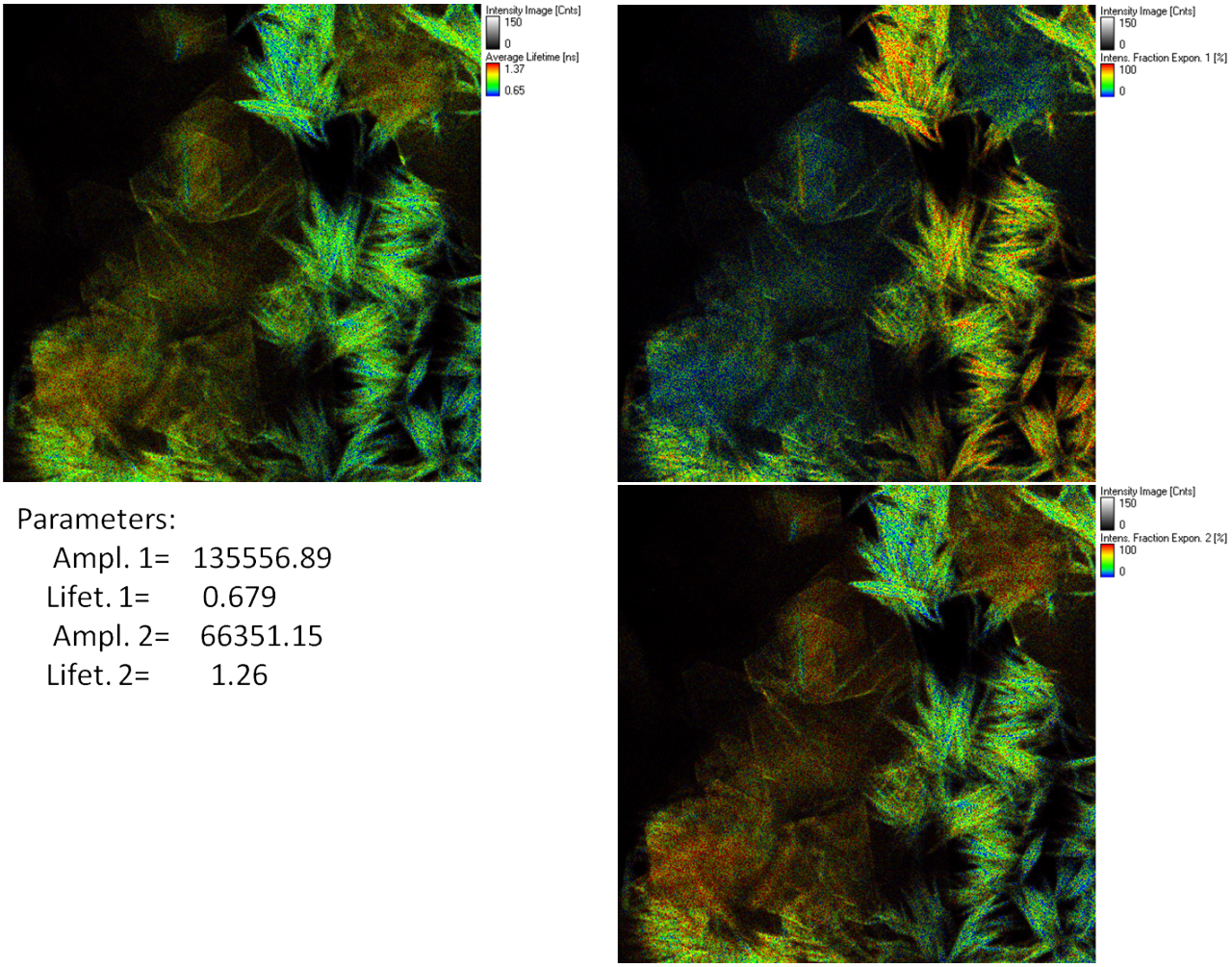


Figure S5. Sample NTA: the images below show the average lifetime (top left) as well as the fractional intensity of the two lifetimes, 0.68 ns (top right) and 1.26 ns (bottom) for the different morphologies. Parameters obtained from biexponential fit of the decay are indicated (tau in ns). Fluorescence was collected at 520 and 585 nm.

5. PHOTOLUMINESCENCE IN THIN DEPOSITS

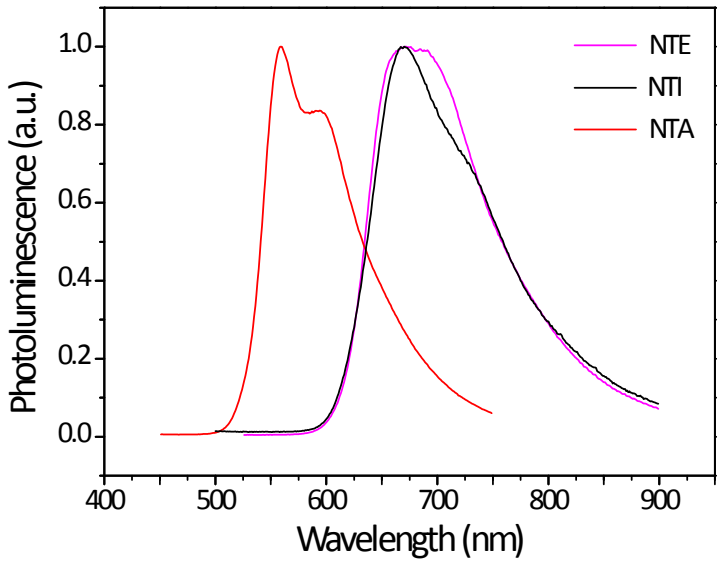


Figure S6. Fluorescence spectra of cast films obtained by exciting the samples at 375 nm collected with a calibrated optical multichannel analyzer (PMA-11, Hamamatsu).

6. DIFFERENTIAL SCANNING CALORIMETRY

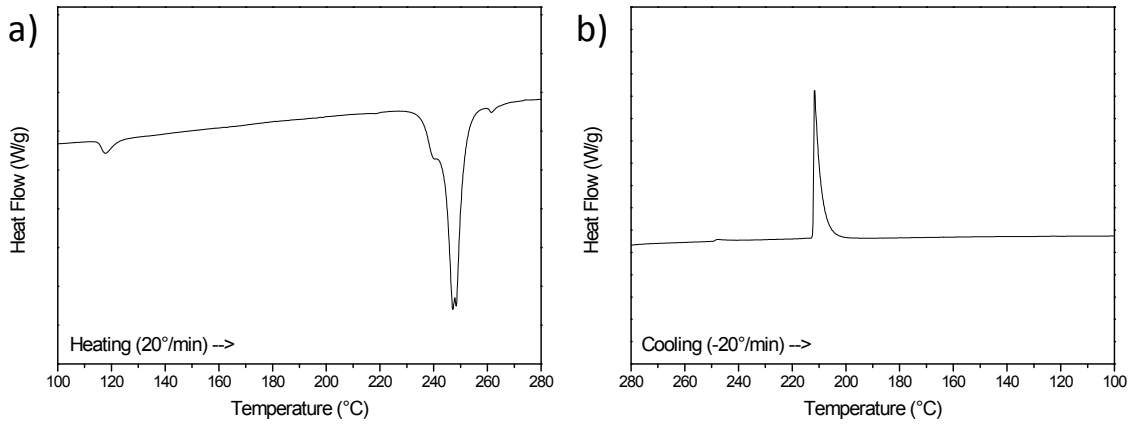


Figure S7. Thermograms of a): heating and b) cooling, of NTE under nitrogen atmosphere. This sample is mainly formed by rod- like crystal phase.

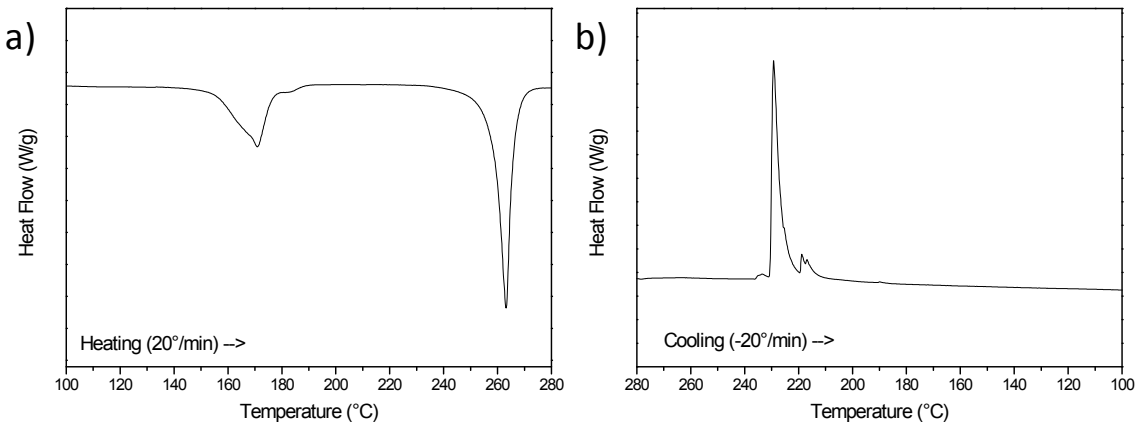


Figure S8. Thermograms of a): heating and b) cooling, of NTI under nitrogen atmosphere.

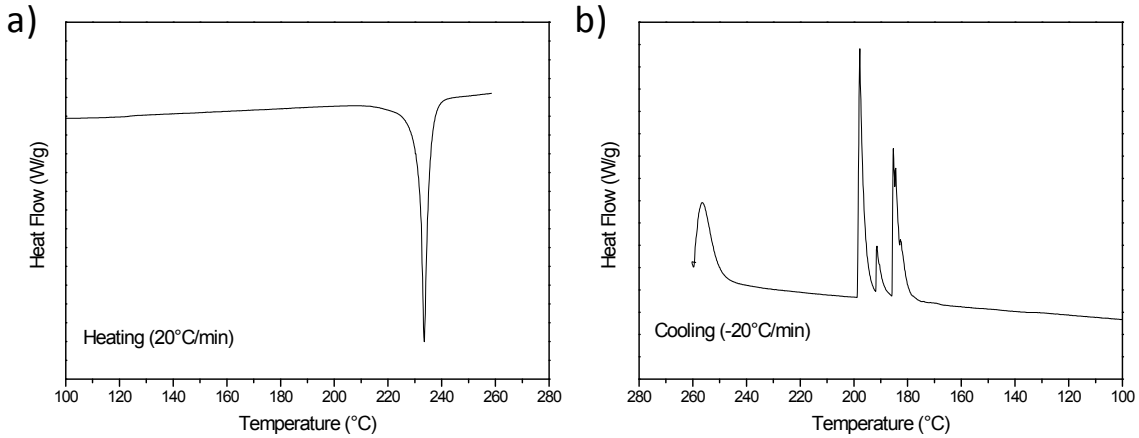


Figure S9. Thermograms of a): heating and b) cooling, of NTA under nitrogen atmosphere.

Table. S1. Summary of the thermal transitions of NTE, NTA and NTI samples.

	Transition temperature (°C) and melting enthalpy (J/g, in brackets)	
Item	Heating (20°C/min)	Cooling (-20°C/min)
NTE	118 (3.5) 240, 246, 248 (80.2) 261 (0.7)	248 (- 0.9) 212 (-78.4)
NTI	171 (28.5) 263 (54.7)	233 (-0.4) 230 (-35.0) 219 (-2.6) 198 (-24.2) 192 (-3.9)
NTA	233 (53.7)	186,184, 183 (-20.5)

7. X-RAY DIFFRACTION DATA

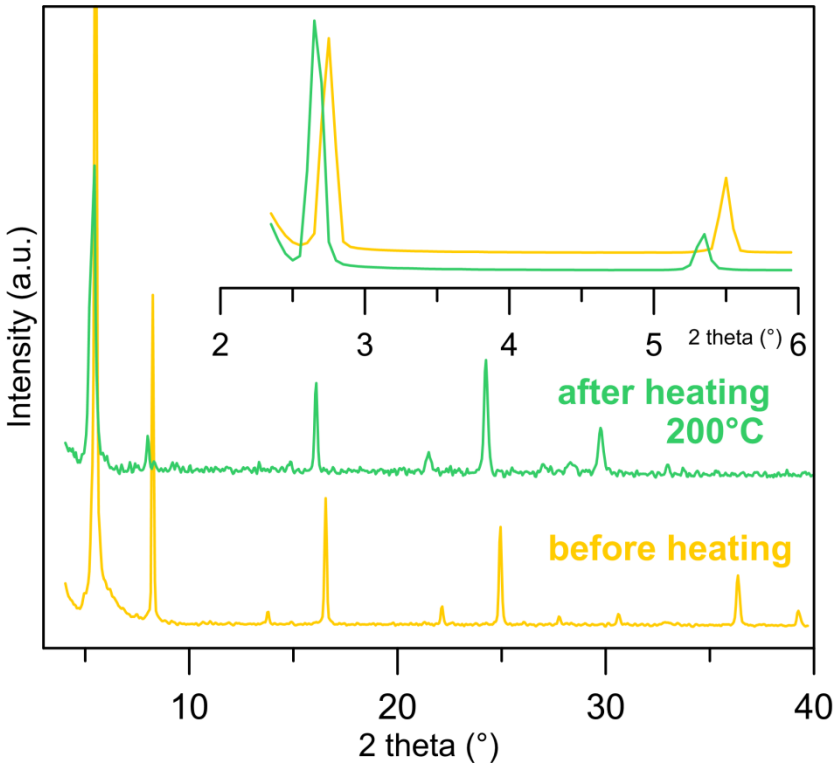


Figure S10. XRD patterns of the NTA sample before and after the thermal cycle.

Main peaks in the XRD patterns for NTE

Pos. [°2Th.]	d-spacing [Å]	Rel. Int. [%]
2.6562	32.26239	1.0**
5.3624	16.46682	1.5**
6.0952	14.50059	100
7.6821	11.50847	2.84
9.8297	8.99837	0.52
12.1651	7.27566	1.04
14.4471	6.13115	2.02
16.5805	5.34676	0.3
18.2853	4.85193	20.04
19.7919	4.48586	0.69
24.4514	3.64057	8.87

37.0109	2.42895	1.84

** peaks belonging to the orange emitting phase

Main peaks for NTE after heat treatment at 200 °C

Pos. [°2Th.]	d-spacing [Å]	Rel. Int. [%]
2.7102	32.59945	400
5.3826	16.41877	100
8.0657	10.96198	8.24
16.152	5.48763	2.69
21.5859	4.11692	0.95
24.2955	3.66356	1.81

Main peaks in the XRD patterns for NTA

Pos. [°2Th.]	d-spacing [Å]	Rel. Int. [%]
2.7447	32.18943	400
5.4973	16.07639	100
8.2519	10.71502	9.42
13.7837	6.4247	0.28
16.548	5.35717	3.71
22.1368	4.01571	0.61
24.9414	3.57014	3.00
27.7733	3.21222	0.24
30.621	2.91967	0.32
36.3558	2.4712	1.31
39.2547	2.29513	0.38

Main peaks for NTA after heat treatment at 200 °C

Pos. [°2Th.]	d-spacing [Å]	Rel. Int. [%]
2.6645	33.15900	500
5.3376	16.55717	100
8.0117	11.0357	0.94
16.0853	5.51022	2.37
21.4819	4.13663	0.42
24.2431	3.67136	2.86
26.9643	3.30672	0.51
28.3494	3.14824	0.64
29.7465	3.00347	1.46

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