Electronic Supplementary Material (ESI) for ChemComm.

Supporting information

"DNA-Polymer Conjugates via the Graft-Through Polymerisation of Native DNA in Water"

Lucy A. Arkinstall,[a] Jonathan T. Husband,[a] Thomas. R. Wilks,[a] Jeffrey C. Foster[a] and Rachel K. O'Reilly*[a]*

[a]School of Chemistry, University of Birmingham, Edgbaston, Birmingham, B15 2TT, UK

*Corresponding Authors: j.c.foster@bham.ac.uk (J.C.F.) and r.oreilly@bham.ac.uk (R.K.O.R.)

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Materials and Methods

Materials

Cis-5-norbornene-exo-2,3-dicarboxylic anhydride (97%), dicyclopentadiene (90+%) and methyl acrylate (99%) were purchased from Alfa Aesar. 4-Dimethylamino pyridine (DMAP, ≥98 %), methoxy poly(ethylene glycol) (MW 2000 Da), *exo*-norbornene carboxylic acid (97%), 6-aminohexanoic acid (≥98.5 %), N-(3-Dimethylaminopropyl)-N'-ethylcarbodiimide hydrochloride (EDC.HCl, BioXtra grade), HOBt hydrate (97%), Grubbs 2nd generation catalyst and dimethylformamide (DMF, Biological grade ≥99 %) were purchased from Sigma-Aldrich. Tetrahydrofuran (THF, HPLC grade) was purchased from VWR Chemicals and was purified *via* passage through a column of basic alumina prior to use. The synthesis of **Nb-PEG[350]** was carried out according to a previously described protocol from our group.[1] The synthesis of **M2.1** was carried out according to a previously described protocol from our group.[2]

PBS buffer was prepared by disolving Dulbecco's phosphate buffered saline (9.6 g) from Sigma-Aldrich into 18 MΩ cm H_2O (1 L). Phosphate solution, pH = 2 (PB2) was prepared using 100 mM sodium phosphate monobasic dihydrate ($NaH_2PO_4 \cdot 2H_2O$) in 18 MΩ cm H_2O adjusted to pH 2 with HCl.

Oligonucleotides were purchased from Integrated DNA technologies, with and without the TAMRA modification at the 3' end. Oligonucleotides were resuspended in 18 MΩ cm H_2O to a concentration of 200 µM before use. Concentrations were calculated from the absorbance values at 260 nm using the reported extinction coefficients.

	Sequence (5'→3')	**Extinction coefficient/ L/(mole·cm)**
DNA-NH_2	5AmMC6/CGA GAC TCA ACG ACA TG	169 300
***DNA-NH_2**	5AmMC6/CGA GAC TCA ACG ACA TG/36-TAMSp	209 780

Micro Bio-spin™ 6 columns were purchased from Bio-Rad laboratories. Amicon® Ultra-0.5 mL centrifugal filters (3000 MWCO) were purchased from Millipore. Illustra™ NAP™-5 columns were purchased from GE healthcare.

Characterisation techniques

NMR Spectroscopy. ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker AVIII-300, Bruker AVIII-400 or Bruker Advance III AV600 spectrometer with $CDCl_3$ as the solvent. The chemical shifts of protons and carbons were reported in parts per million (ppm) relative to internal solvent resonances. NMR Spectra were processed using MestReNova software.

High-Resolution Mass Spectrometry. HRMS spectra were recorded by the MS Analytical Facility Service at the University of Birmingham on a Waters Xevo G2-XS QTof Quadrupole Time-of-Flight mass spectrometer.

Size Exclusion Chromatography. Size exclusion chromatography (SEC) in DMF was performed on an Agilent 1260 Infinity II LC system equipped with a Wyatt DAWN HELEOS II multi-angle laser light scattering (MALLS) detector, a Wyatt Optilab T-rEX differential refractive index detector, an Agilent 1260 Infinity II WR diode array detector, an Agilent guard column (PLGel 5 µM, 50 × 7.5 mm) and two Agilent Mixed-C columns (PLGel 5 µM, 300 × 7.5 mm). The mobile phase was DMF (HPLC grade) containing 5 mM NH_4BF_4 at 50 °C at flow rate of 1.0 mL min^{-1}. Number average molecular weights (M_n), weight average molecular weights (M_w) and dispersities ($Đ_M = M_w/M_n$) were determined using Wyatt ASTRA v7.1.3 software against poly(methyl methacrylate) (PMMA) standards.

Gel Electrophoresis. Native polyacrylamide gels were run at 2 °C in 1 x TAE buffer at 180V – 200V using a vertical nucleic acid electrophoresis cell connected to a PowerPack basic power supply (BioRad). Samples were combined with 20% loading buffer (0.05% bromophenol blue, 25% glycerol, 1x TAE) prior to running. Non-fluorescent DNA was stained using a 1:1000 aqueous SYBR® Gold nucleic acid gel stain (ThermoFisher) and visualized using a BioRad ChemiDoc™ MP Imaging system. The images were processed using ImageLab software. Denaturing polyacrylamide gels (15% acrylamide, 25% formamide) were run at room temperature in 1 x TBE buffer at 14 amps using a vertical nucleic acid electrophoresis cell connected to a PowerPack basic power supply (BioRad). The samples were diluted 1:1 v/v with formamide solution (90 % formamide) and heated at 70 °C for 10 min prior to running. Nonfluorescent DNA was stained using a 1:1000 aqueous SYBR® Gold nucleic acid gel stain (ThermoFisher) and visualized using a BioRad ChemiDoc™ MP Imaging system. The images were processed using ImageLab software.

Liquid Chromatography-Mass Spectrometry. Liquid chromatography-mass spectrometry (LCMS) analysis was performed on a Waters ACQUITY UPLC system coupled to a Xevo GS2-XS qToF mass spectrometer in negative ion mode. The oligonucleotides were eluted through an AQUITY UPLC oligonucleotide BEH C18 column (130Å, 1.7 µm, 2.1 × 50 mm) using a 75 mM triethylammonium acetate (TEAA, pH 7.0) solution in H_2O (buffer A) and a 75 mM TEAA solution in MeCN (buffer B) at 60 °C and a 0.2 mL min^{-1} flow rate. The data was processed using ProMass HR software.

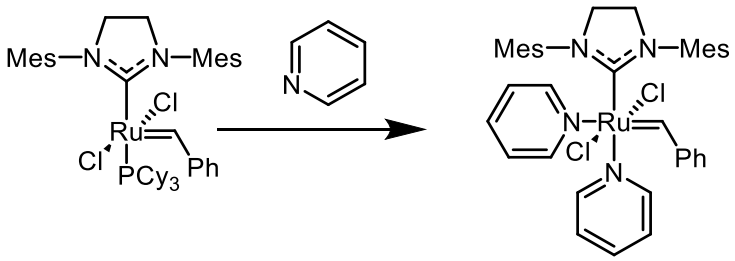
Fluorescence Spectroscopy. Fluorescence spectral data were determined using an Edinburgh Instruments FS5 spectrofluorometer. Quartz cells with four polished side were used for all measurements. The fluorescence of **•DNA-BBP1** and **•DNA-BBP2** (0.33 mg mL^{-1}) was recorded in differing volumes of H_2O/DMF recording the full emission spectra following excitation at 380 nm.

Synthetic procedures

General protocol for flash column chromatography

Flash chromatography was performed on a Teledyne ISCO CombiFlash Rf+ Lumen equipped with two UV detectors (254 nm and 280 nm) and an internal evaporative light scattering detector (ELSD). Samples were purified using RediSep RF normal phase columns.

Synthesis of G3



G3

Grubbs' 2nd generation catalyst was converted to Grubbs' 3rd generation catalyst (**G3**) according to a previously reported procedure.[3] To a 20 mL scintillation vial equipped with a magnetic stir bar was added Grubbs' 2nd generation catalyst (100 mg, 0.12 mmol) and pyridine (1.0 mL). The mixture was stirred under a nitrogen atmosphere for 5 minutes at 25 °C until the red colour disappeared to yield a clear, dark green solution. Hexane (10 mL) was added and the solution was stirred for a further 5 minutes. The green precipitate that formed was isolated *via* filtration and washed with cold hexane (20 mL) to yield a green solid (0.0267 g, 31%). ^{1}H NMR (600 MHz; 298 K; $CDCl_3$) 19.18 (s, 1H, H-alkylidene), 8.63 (s, 2H, Arom-H), 7.83 (s, 2H, Arom-H), 7.67 (t, 1H, Arom-H), 7.63 (d, 2H, Arom-H), 7.48 (t, 2H, Arom-H), 7.27 (m, 2H, Arom-H), 7.06-7.09 (m, 4H, Arom-H), 6.97 (s, 2H, Arom-H), 6.75 (s, 2H, Arom-H), 4.12 (br d, 4H, NCH_2CH_2N), 2.65 (s, 6H, Ar-CH_3), 2.24-2.36 (m, 12H, Ar-CH_3). Characterisation data matched those reported in the literature.[3]

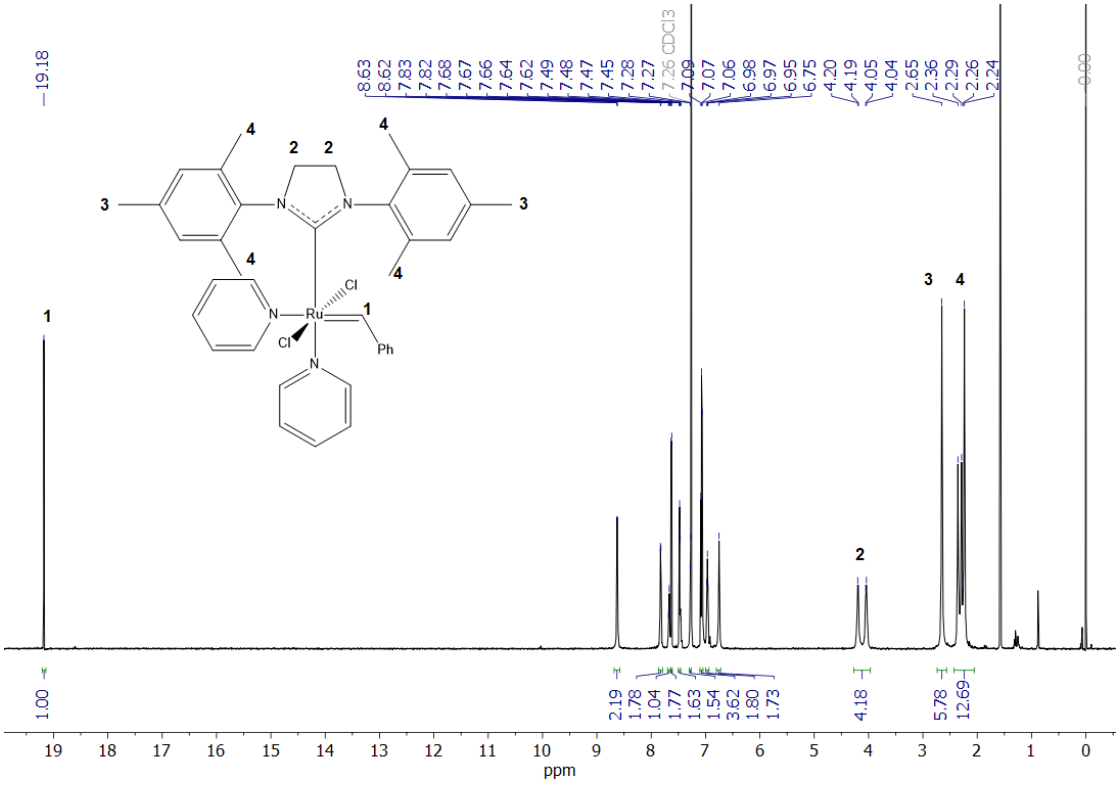
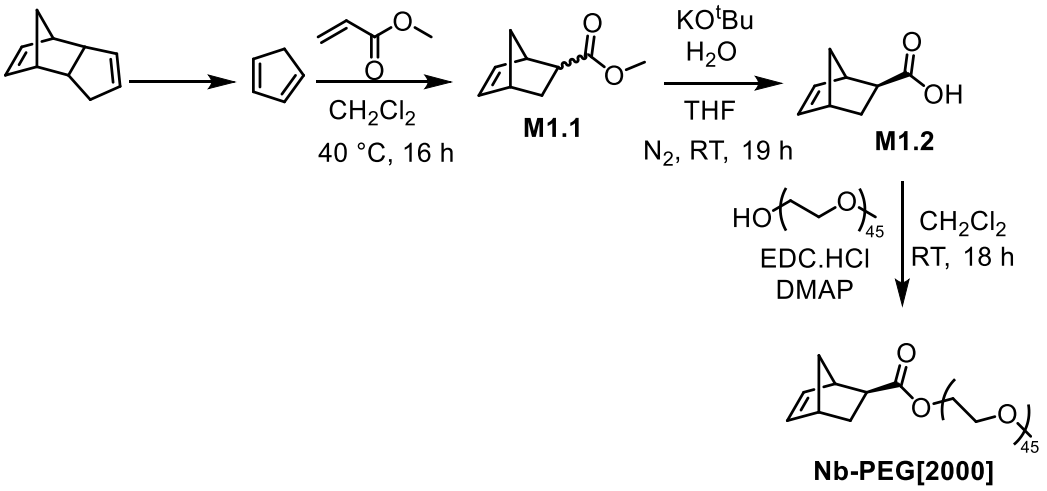


Fig. S1. ^{1}H-NMR spectrum of **G3** (600 MHz, $CDCl_3$).

Synthesis of Nb-PEG[2000]



Synthesis of M1.1

The synthesis of **M1.1** was adapted from a literature method.[4] Freshly prepared cyclopentadiene (34 mL, 0.40 mol) generated by the pyrolysis of dicyclopentadiene was added to methyl acrylate (40 mL, 0.44 mol) and dichloromethane (40 mL). The mixture was refluxed for 16 hours prior to concentration *in vacuo* to yield **M1.1** (ca. 20% *exo*) as a colourless oil (54.8 g, 90%).

Endo (major): ^{1}H NMR (300 MHz; 298K; $CDCl_3$) 6.19 (dd, 1H, J =5.7, 3.0 Hz, CHCH), 5.92 (dd, 1H, J = 5.7, 3.0 Hz, CHCH), 3.62 (s, 3H, CH3), 3.22-3.18 (m, 1H, CH), 2.95 (dt, 1H, J = 9.3, 3.9 Hz, CH), 2.90 (s, 1H, CH), 1.95-1.86 (m, 1H, CH_2), 1.55-1.24 (m, 3H, 2 x CH_2).

Exo (minor): ^{1}H NMR (300 MHz; 298K; $CDCl_3$) 6.16-6.08 (m, 2H, CHCH), 3.69 (s, 3H, CH_3), 3.04 (s, 1H, CH), 2.88 – 2.92 (m, 1H, CH), 2.26-2.20 (m, 1H, CH_3), 1.95-1.86 (m, 1H, CH_2), 1.55-1.24 (m, 3H, 2x CH_2).

Characterisation data matched those reported in the literature.[5]

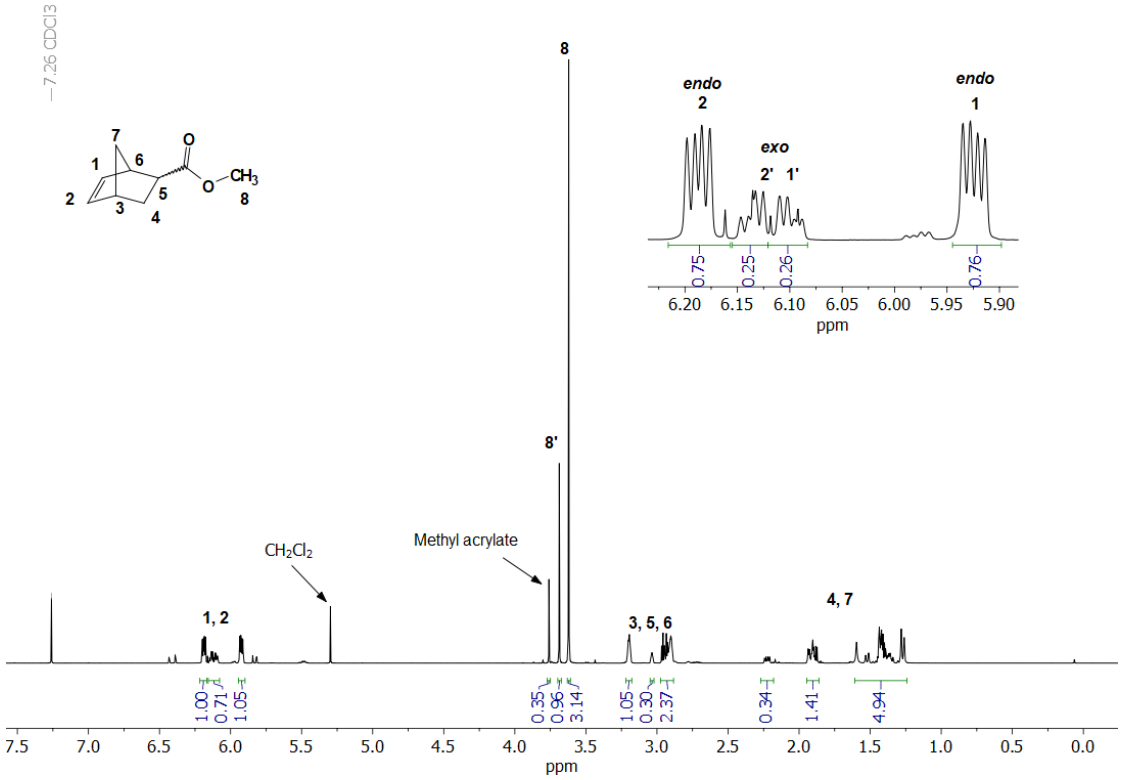


Fig. S2. ^{1}H-NMR spectrum of **M1.1** (300 MHz, $CDCl_3$).

Synthesis of M1.2

The synthesis of **M1.2** was adapted from a literature method.[6] Potassium t-Butoxide (80.8 g, 0.72 mol) and THF (720 mL) were added to a 2L 3-necked flask purged with N_2. **M1.1** (54.7 g, 0.36 mol) was added to the flask and stirred at room temperature for 1 hour. H_2O (6.5 mL, 0.36 mol) was diluted with THF (338 mL) and added dropwise to the reaction mixture over 18 hours. Excess H_2O (≈80 mL) was added to complete the reaction and the reaction mixture was stirred for a further 1 h. The reaction mixture was concentrated to remove THF, diluted with water and then adjusted to pH = 2 by the addition of concentrated HCl. The aqueous solution was then extracted with EtOAc (2 × 200 mL) and the organic layers were combined, dried over $MgSO_4$ and concentrated. Following rotary evaporation **M1.2** (ca. 85% *exo*) was obtained (40.3 g, 81%).

Removal of the *endo*-isomer was carried out using a previously described protocol.[7] **M1.2** (85% exo) (40 g, 0.29 mol) was dissolved in an aqueous solution of $NaHCO_3$ (26.76 g, 0.32 mol in 400 mL H_2O). $NaHCO_3$ was added until the reaction mixture was around pH = 10. In a separate flask, I_2 (49.2 g, 0.19 mol) and KI (52.8 g, 0.32 mol) were dissolved in H_2O (800 mL). The I_2/KI solution was added slowly to the flask containing the *endo*/*exo* norbornene acid until the solution retained a dark brown colour. The reaction mixture was then filtered and transferred to a separatory funnel. The aqueous solution was washed with Et_2O (5 × 500 mL) to remove the iodolactone formed by the endo isomer. The aqueous layer was decolorized using solid Na_2SO_3 and acidified to pH = 2 with conc. H_2SO_4. The product was extracted with Et_2O (4 × 500 mL) and the combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo* to yield **M1.2** (100% exo) as a pale yellow solid (20.2 g, 51%). ^{1}H NMR ($CDCl_3$; 298K; 300 MHz) 6.20-6.08 (m,

2H, CHCH), 3.11 (s, 1H, CH), 2.94 (s, 1H, CH), 2.29-2.24 (m, 1H, CH), 1.99-1.87 (m, 1H, CH_2), 1.58-1.34 (m, 3H, CH_2). Characterisation data matched those reported in the literature.[7]

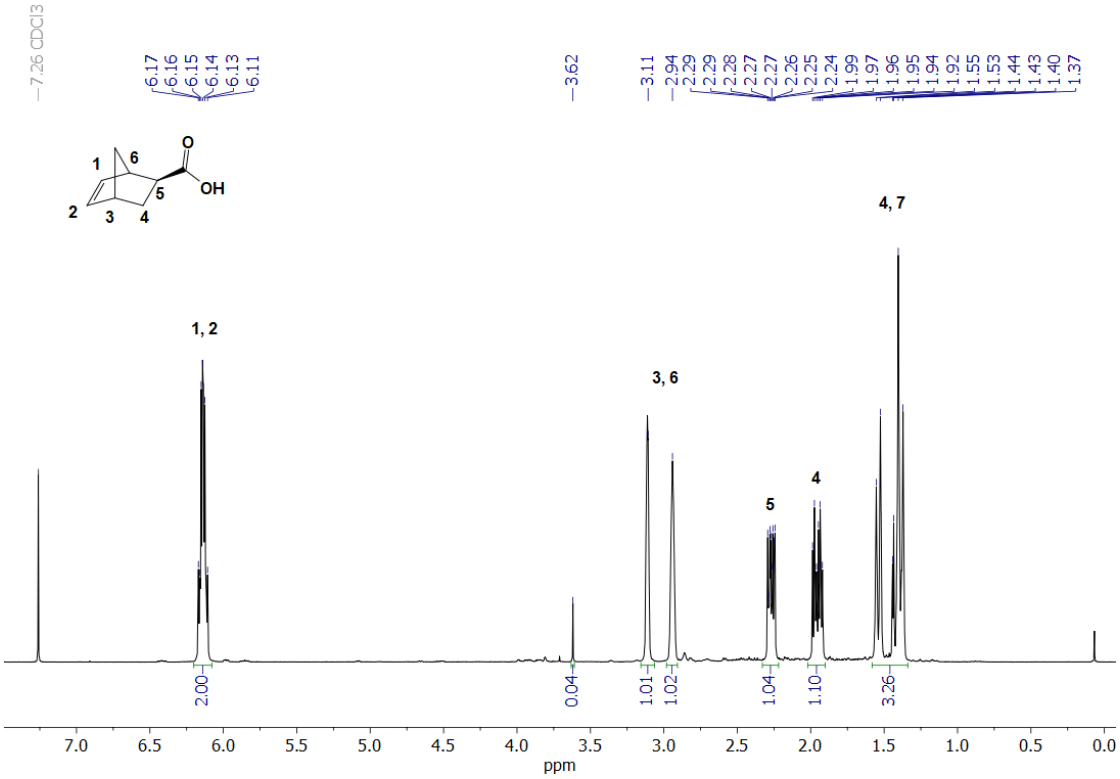


Fig. S3. ^{1}H-NMR spectrum of **M1.2** (300 MHz, $CDCl_3$).

Synthesis of Nb-PEG[2000]

M1.2 (2 g, 14.5 mmol), EDC.HCl (2.78 g, 14.5 mmol), 4-dimethylamino pyridine (0.17 g, 1.4 mmol) and methoxy polyethylene glycol (MW 2000 Da) were solubilised in dichloromethane (50 mL) and stirred at room temperature for 18 hours. The solution was washed with 1M HCl solution followed by H_2O and Sat. $NaHCO_3$ and finally brine prior to drying over $MgSO_4$ and concentrating *in vacuo*. The crude solid was precipitated from dichloromethane into diethyl ether, twice, to yield a white solid, **Nb-PEG[2000]** (3.18 g, 53%). ^{1}H NMR ($CDCl_3$; 298K; 300 MHz) 6.14-6.07 (m, 2H, CHCH), 4.28-4.15 (m, 2H, $COOCH_2$), 3.86-3.36 (m, 207H, OCH_2), 3.36 (s, 3H, OCH_3), 2.95 (d, 2H, CHCH), 2.31-2.19 (m, 1H, CH_2CH), 1.91 (dt, 1H, CH_2), 1.59-1.45 (m, 1H, CH_2), 1.34 (m, 2H, CH_2). ^{13}C NMR ($CDCl_3$; 298K; 100 MHz) 138.2 (*C*O), 135.89 (*C*H), 70.92 (CH_2), 46.82 (CH_3), 46.44 (*C*H), 43.18 (CH_2), 41.78 (*C*H), 30.49 (CH_2).

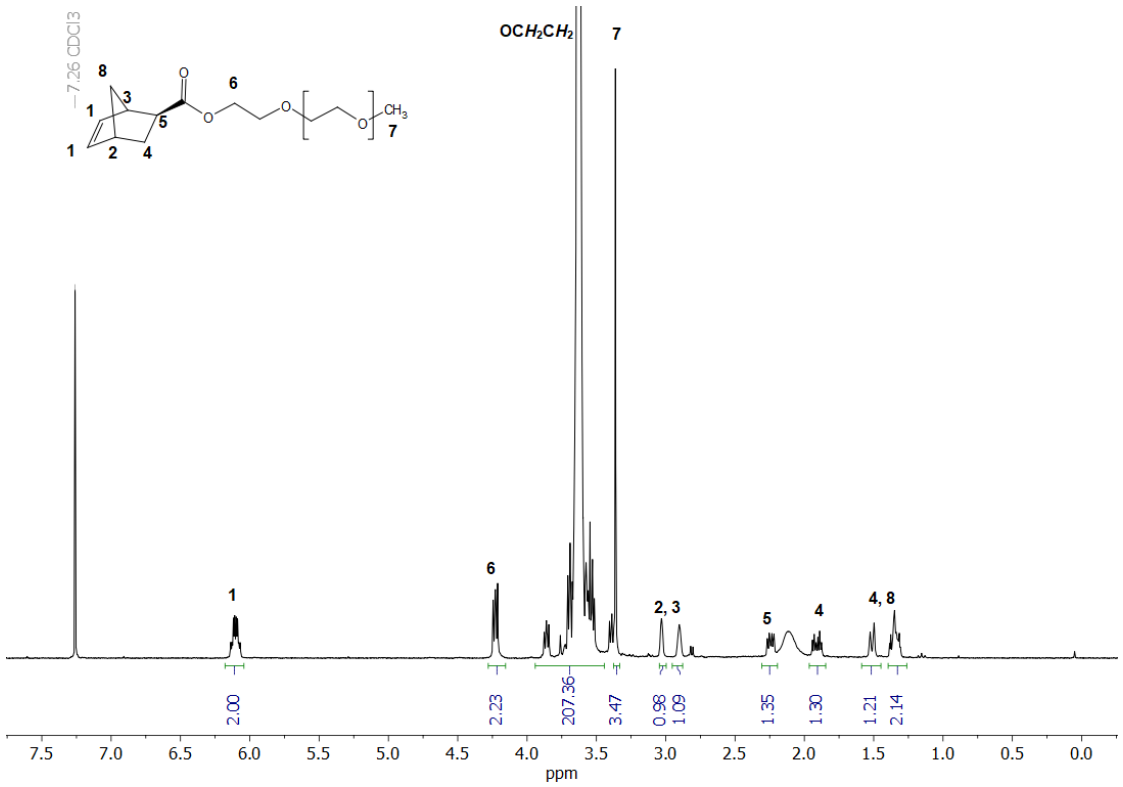


Fig. S4. ^{1}H-NMR spectrum of **Nb-PEG[2000]** in $CDCl_3$ (300 MHz, $CDCl_3$).

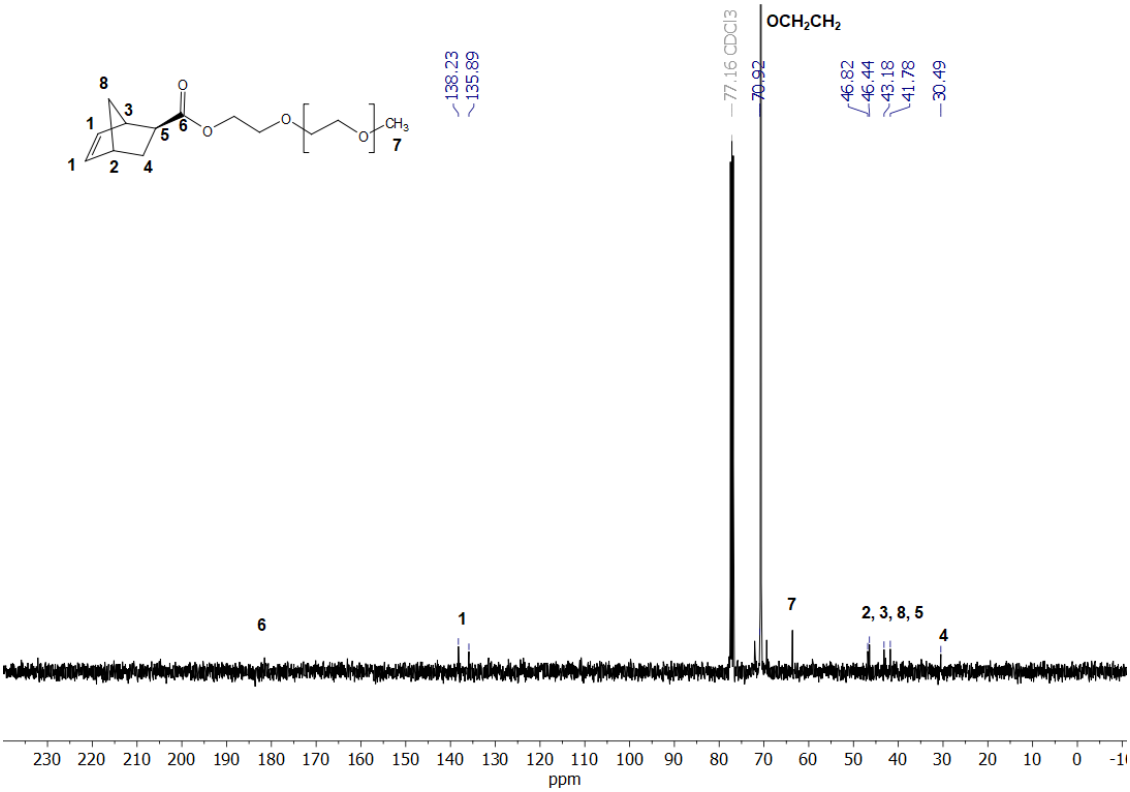
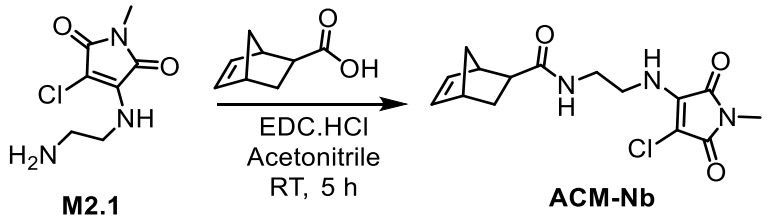


Fig. S5. ^{13}C-NMR spectrum of **Nb-PEG[2000]** (100 MHz, $CDCl_3$).

Synthesis of ACM-Nb



A round-bottom flask was charged with *exo*-norbornene carboxylic acid (85 mg, 0.41 mmol), acetonitrile (20 mL) and a magnetic stirrer bar. EDC (130 mg, 0.62 mmol) was added, followed by ethylamine-ACM **M2.3** (100 mg, 0.41 mmol). The reaction mixture was stirred for 5 hours at which point the solution was dried and purified *via* silica flash column chromatography and eluted with a methanol (0-5%) in CH_2Cl_2 gradient, to yield solid **ACM-Nb** (73 mg, 49%). ^{1}H NMR ($CDCl_3$; 298K; 300 MHz) 6.08 (dd, J = 5.7, 2.9 Hz, 1H, CH), 6.02 (dd, J = 5.7, 3.0 Hz, 1H, CH), 5.90 (s, 1H, NH), 5.82 (s, 1H, NH), 3.71 (q, ^{3}JHH = 5.9 Hz, 2H, CH_2), 3.54 (q, ^{3}JHH = 5.9 Hz, 2H, CH_2), 2.99 (s, 3H, CH_3), 2.93 (m, 2H, CH), 1.99 – 1.89 (m, 1H, CH), 1.89 – 1.79 (m, 1H, CH), 1.65 – 1.55 (m, 1H, CH_2), 1.36 – 1.16 (m, 3H, CH_2). ^{13}C NMR ($CDCl_3$; 298K; 100 MHz) 177.1 (CO), 167.8 (CO), 165.5 (CO), 140.8 (CN), 138.4 (CH), 135.8 (CH), 89.8, 47.3 (CH), 46.3 (CH2), 44.7 (CH), 43.8 (CH2), 41.6 (CH_2), 40.0 (CH), 30.6 (CH_2), 24.2 (CH_3). HR-MS [M+H+] m/z calculated 346.0934, found 346.0932.

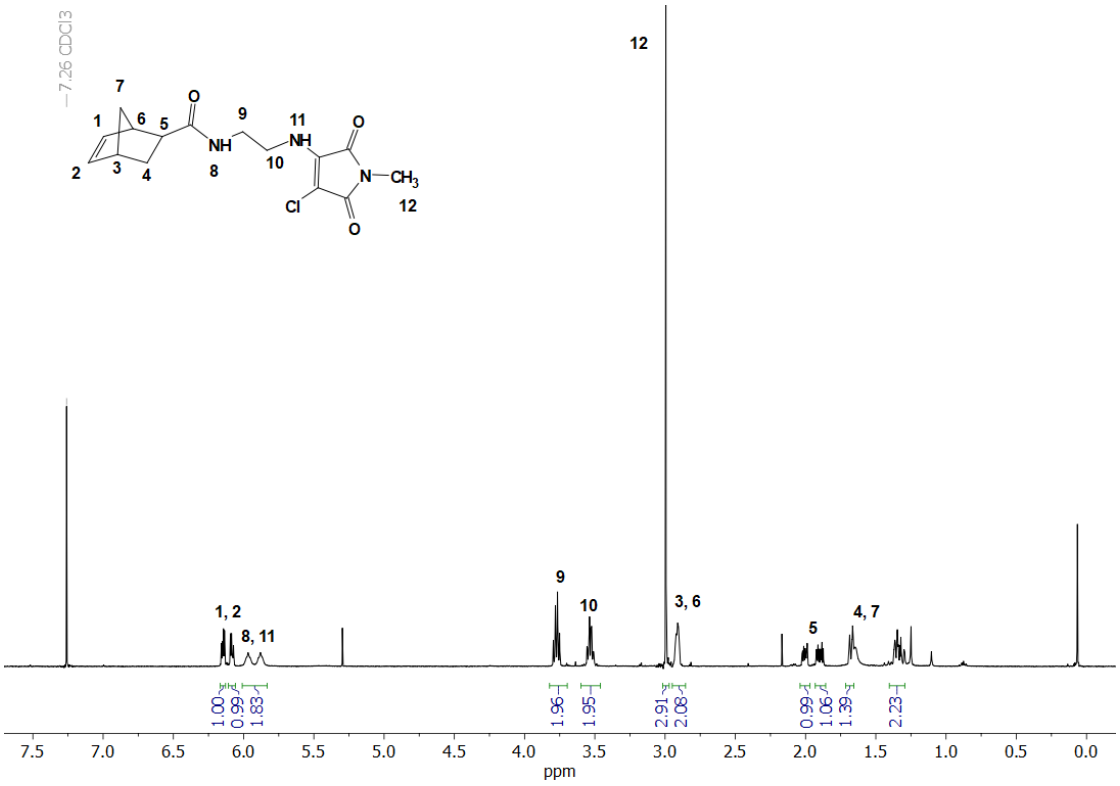


Fig. S6. ^{1}H-NMR spectrum of **ACM-Nb** (300 MHz, $CDCl_3$).

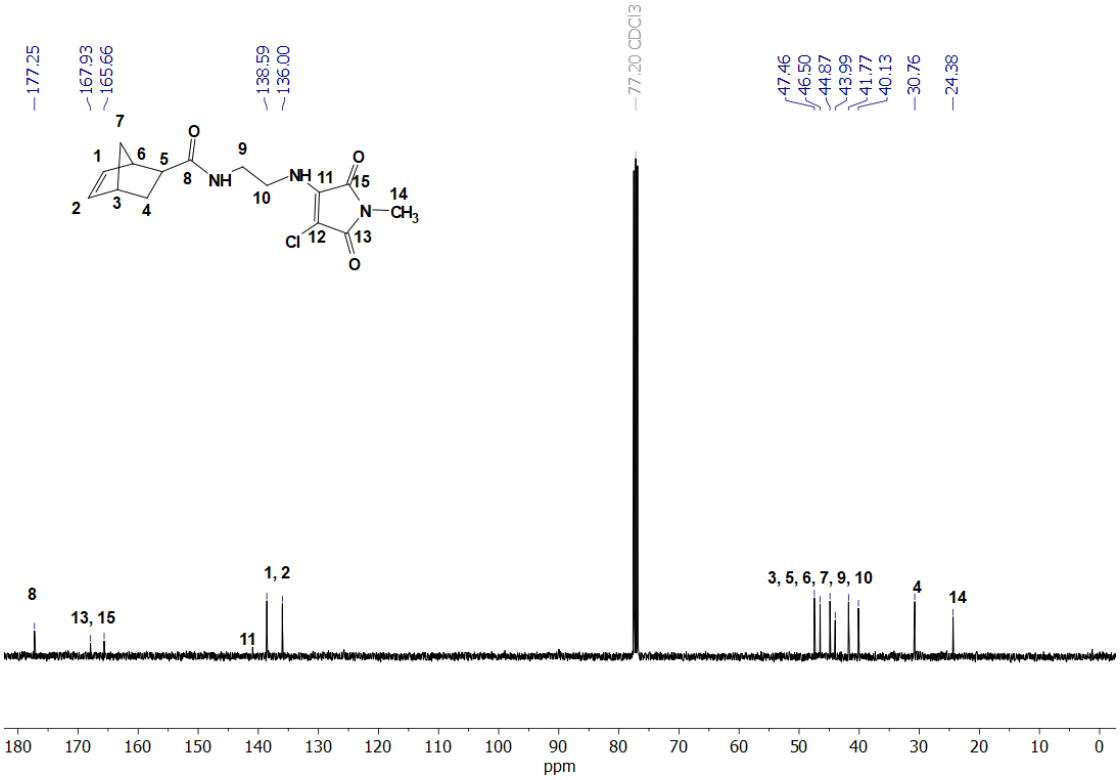
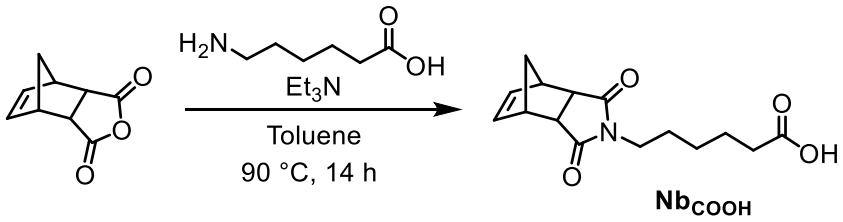


Fig. S7. ^{13}C-NMR spectrum of **ACM-Nb** (100 MHz, $CDCl_3$).

Synthesis of Nb_{COOH}



A round bottom flask was charged with cis-5-Norbornene-exo-2,3-dicarboxylic anhydride, aminohexanoic acid, triethylamine, and toluene. The flask was fitted with a condenser, and the reaction mixture was heated at reflux for 14 hours. The reaction mixture was then cooled to room temperature and concentrated under reduced pressure. The resulting residue was re-dissolved in EtOAc, and the organic solution was washed with 1M HCl (2 ×) and brine, dried over $MgSO_4$, and concentrated under reduced pressure to yield the pure product as an off-white solid. ^{1}H NMR ($CDCl_3$; 298K; 400 MHz) 6.28 (s, 2H, CH), 3.51 – 3.43 (m, 2H, CH), 3.27 (s, 2H, CH), 2.67 (m, 2H, CH_2), 2.34 (t, J = 7.4 Hz, 2H, CH_2), 1.71 – 1.47 (m, 5H, CH_2), 1.35 (m, 2H, CH_2), 1.21 (d, J = 9.8 Hz, 1H, CH_2). ^{13}C NMR ($CDCl_3$; 298K; 100 MHz) 137.86, 47.82, 47.82, 45.18, 42.74, 38.43, 33.58, 27.43, 26.35, 24.15. HR-MS [M+H$^+$] m/z calculated 276.1236, found 276.1239.

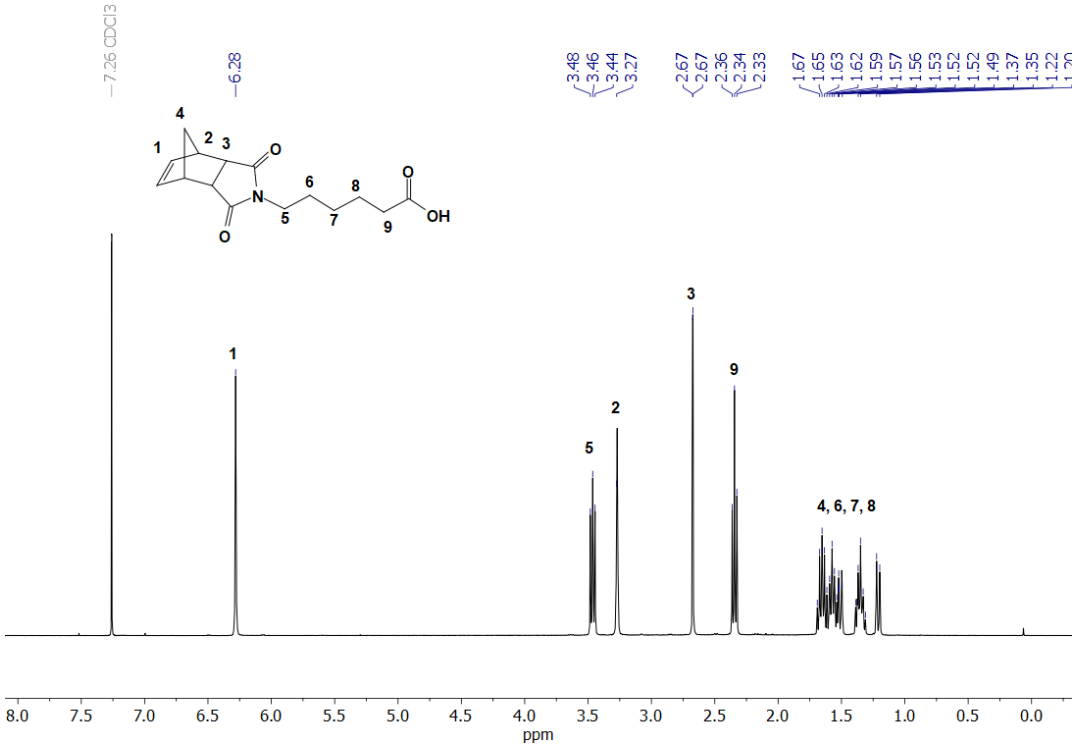


Fig. S8. ^{1}H-NMR spectrum of **Nb$_{COOH}$** (400 MHz, $CDCl_3$).

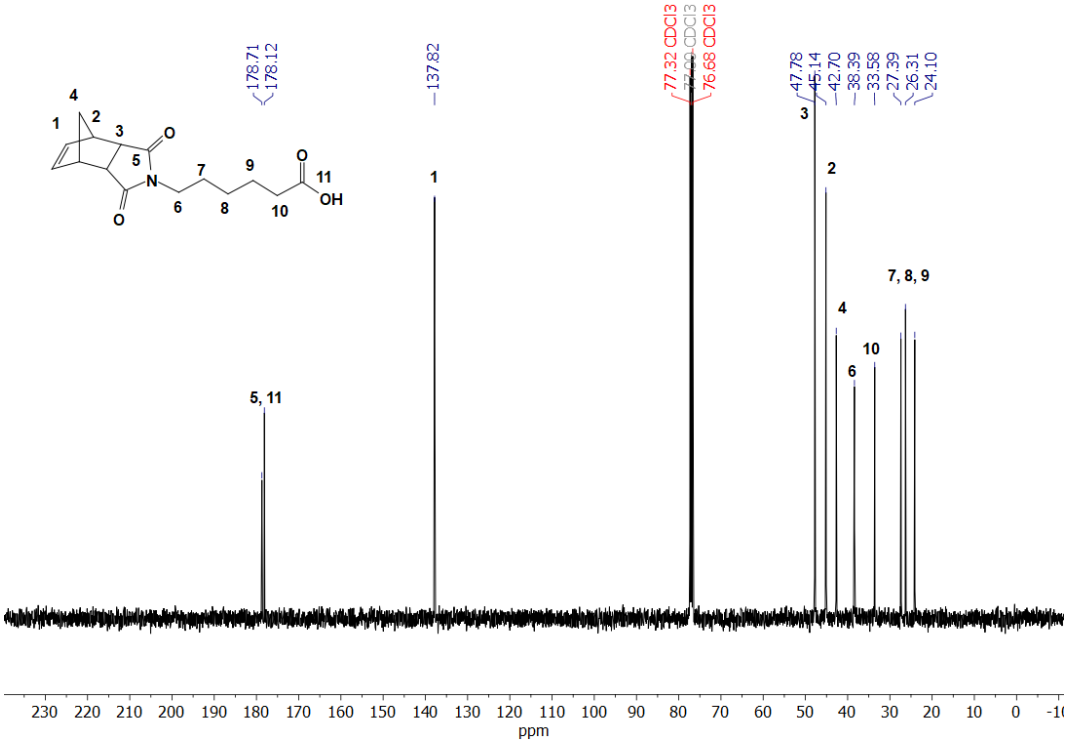
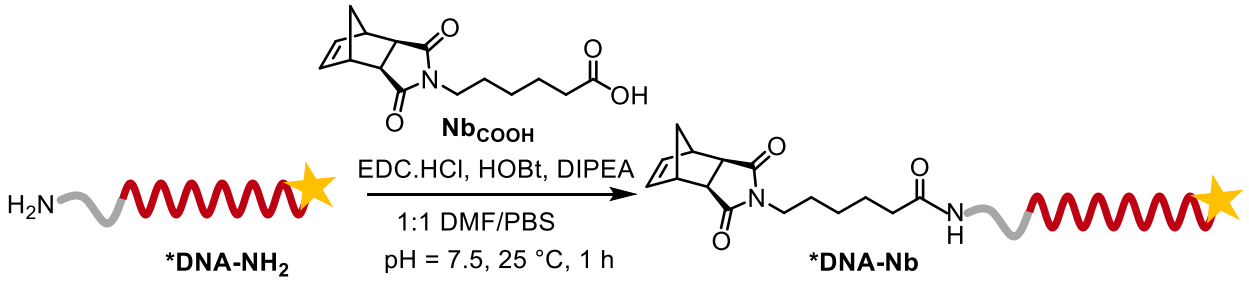


Fig. S9. ^{13}C-NMR spectrum of **Nb$_{COOH}$** in $CDCl_3$.

Synthesis of *DNA-Nb



EDC.HCl (100 µL, 300 mM in DMF) was mixed with HOBt (100 µL, 300 mM in DMF), **Nb$_{COOH}$** (100 µL, 300 mM in DMF) and PBS (150 µL) and thoroughly mixed. 300 µL of this solution was mixed with ***DNA-NH$_2$** (100 µL, 200 µM in 18 mΩ cm H_2O) and DIPEA (3.6 µL). After 2 hours shaking the flask at room temperature the mixture was filtered through a NAP™-5 column equilibrated with 18 mΩ cm H_2O and then concentrated using an Amicon® Ultra centrifugal filter (3000 Da MWCO). LC-MS (ESI-) m/z calculated 6633.2, found 6633.2.

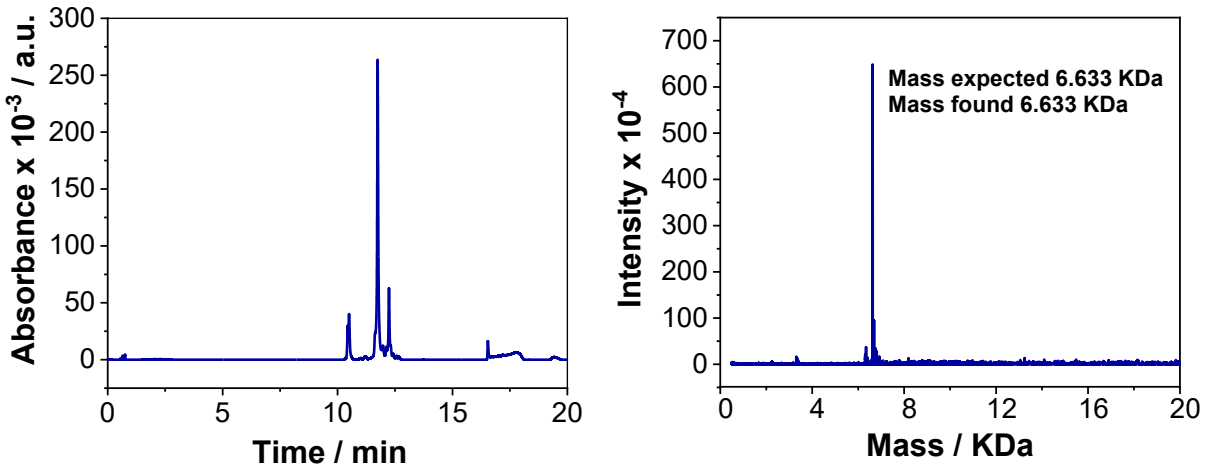


Fig. S10. (Left) HPLC-UV chromatogram of ***DNA-Nb**. (Right) Deconvoluted mass spectrum of ***DNA-Nb**.

Example polymerisation procedure

A vial was charged with **Nb-PEG** and THF (0.9 mL, freshly filtered through basic alumina). To the vial was added 100 µL of a 15 mg mL^{-1} solution of **G3** in THF. The reaction mixture was stirred for 30 s at room temperature and then 100 µL of this solution was transferred to a vial containing **Nb-PEG**, ***DNA-Nb** and succinimide (10 µL, 0.05 mg mL^{-1}) in PB2 (0.9 mL). After 1 h stirring, the polymerisation was quenched by the addition of 1 drop of ethyl vinyl ether and neutralised with Sat. $NaHCO_3$ prior to analysis.

Supplementary Characterisation data

Optimisation of ROMP in the presence of DNA

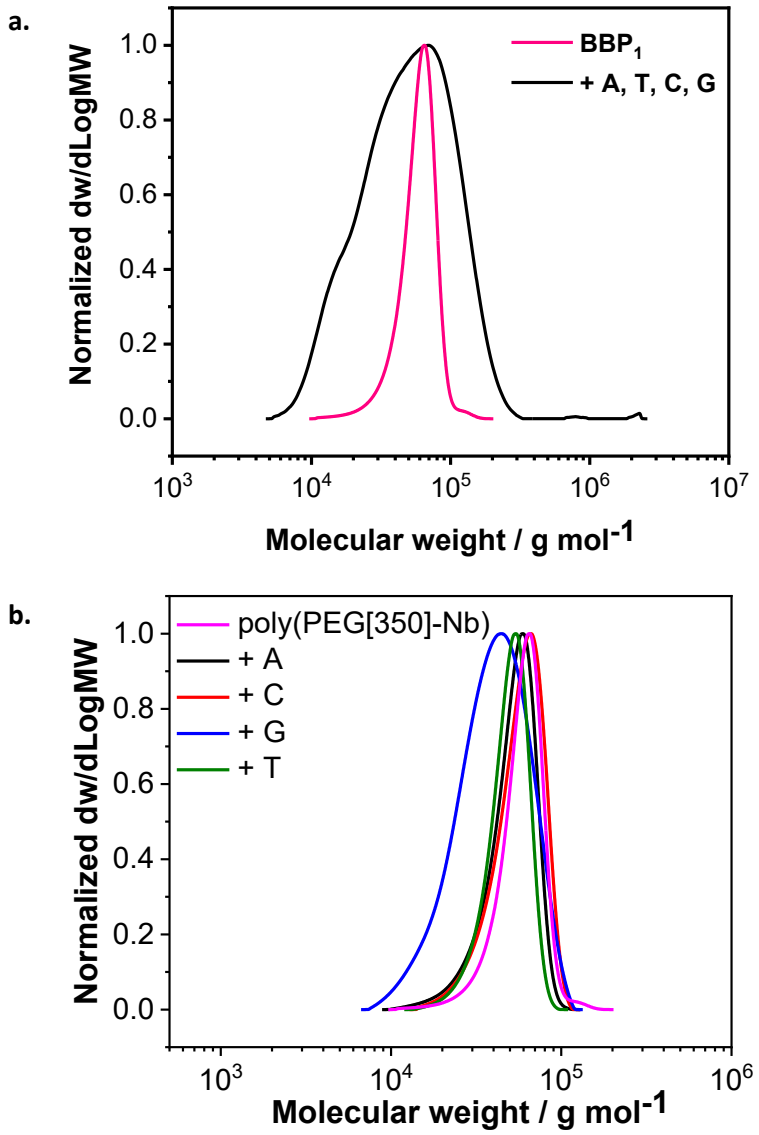


Fig. S11. (a) Normalized SEC RI molecular weight distributions of poly(PEG[350]-Nb) synthesized in the absence (pink trace) and presence of 100 nmol of each of the four nucleobases (black trace). (b) Normalized SEC RI molecular weight distributions of poly(PEG[350]-Nb) synthesized synthesized in the absence (pink trace) and presence of 100 nmol of either adenine (black trace), cytosine (red trace), guanine (blue trace) or thymine (green trace). Eluent: DMF + 5 mM NH_4BF_4, PMMA standards.

Stability of DNA under the polymerisation conditions

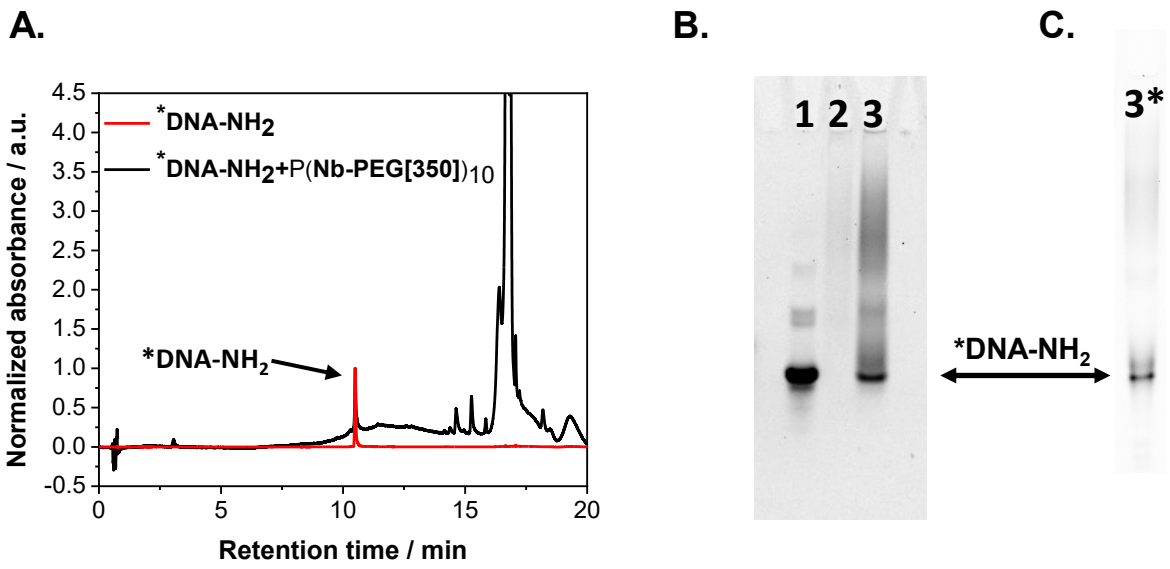


Fig. S12. Stability study: ***DNA-NH₂** was subjected to 10 equivalents P(**Nb-PEG[350]**)$_{10}$ and 2.5 equivalents of succinimide in 9:1 v/v PB2:THF and incubated for 1 h at room temperature. (A) LC-MS-UV chromatogram at 260 nm of ***DNA-NH₂** (red trace) and ***DNA-NH₂** after incubation with P(**Nb-PEG[350]**)$_{10}$ (black trace). (B) 15% native PAGE of ***DNA-NH₂** (lane 1) and ***DNA-NH₂** incubated with P(**Nb-PEG[350]**)$_{10}$ lane 3). (C) 15% denaturing PAGE (lane 3*) ***DNA-NH₂** incubated with P(**Nb-PEG[350]**)$_{10}$ incubated with the macroinitator.

Table S1. Summary of optimisation of PEG ROMP in the presence of nucleobases. [a]Monomer conversion calculated from ^{1}H NMR spectra in $CDCl_3$. [b]Calculated from conversion using the initial [Nb]/[**G3**] ratio. [c]Measured by SEC using poly (methyl methacrylate) standards with DMF + 5 mM NH_4BF_4 eluent.

Run	Additive/Conditions	Conversion[a]	$M_{n,theo}$ (kDa)[b]	$M_{n,SEC}$ (kDa)[c]	$Đ_M$[c]
1	None	*Quant*	54.5	55.4	1.10
2	Nucleobase mixture	0.30		36.3	1.79
3	Cytosine	*Quant*		53.1	1.12
4	Guanine	0.67		34.7	1.26
5	Thymine	*Quant*		46.9	1.08
6	Guanine + Succinimide	*Quant*		77.6	1.28

Polymerisation characterisation

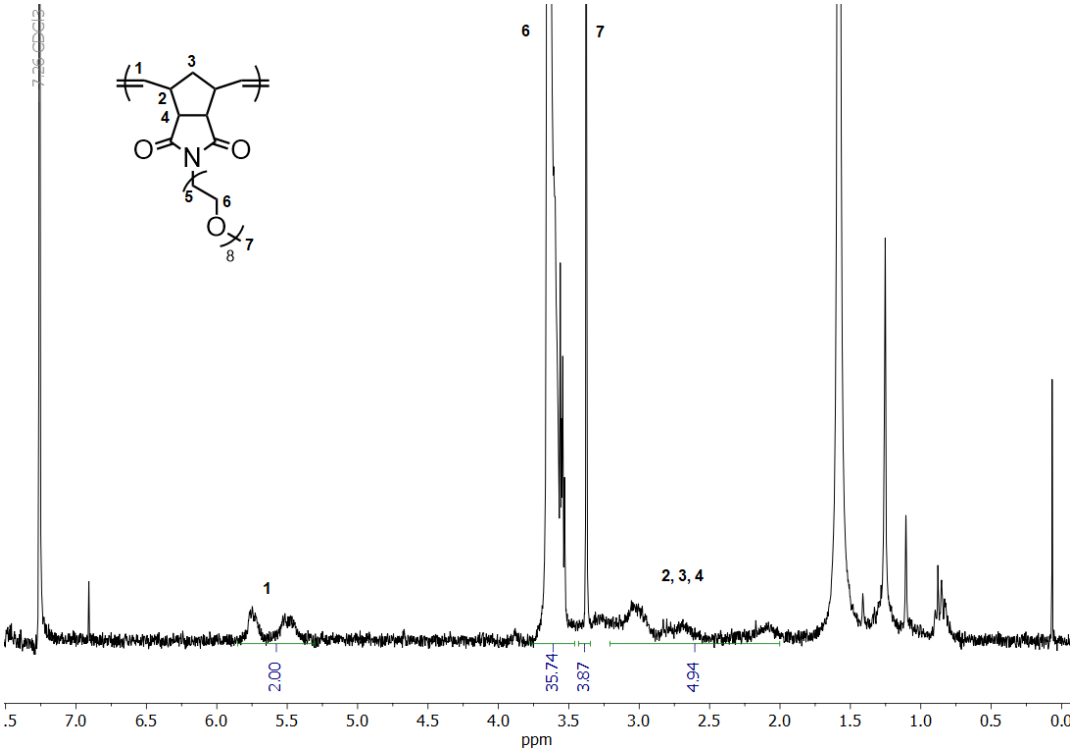


Fig. S13. *^{1}H NMR spectrum of **BBP1** in $CDCl_3$. Complete conversion was determined by the disappearance of the norbornene protons expected at 6.2 ppm.*

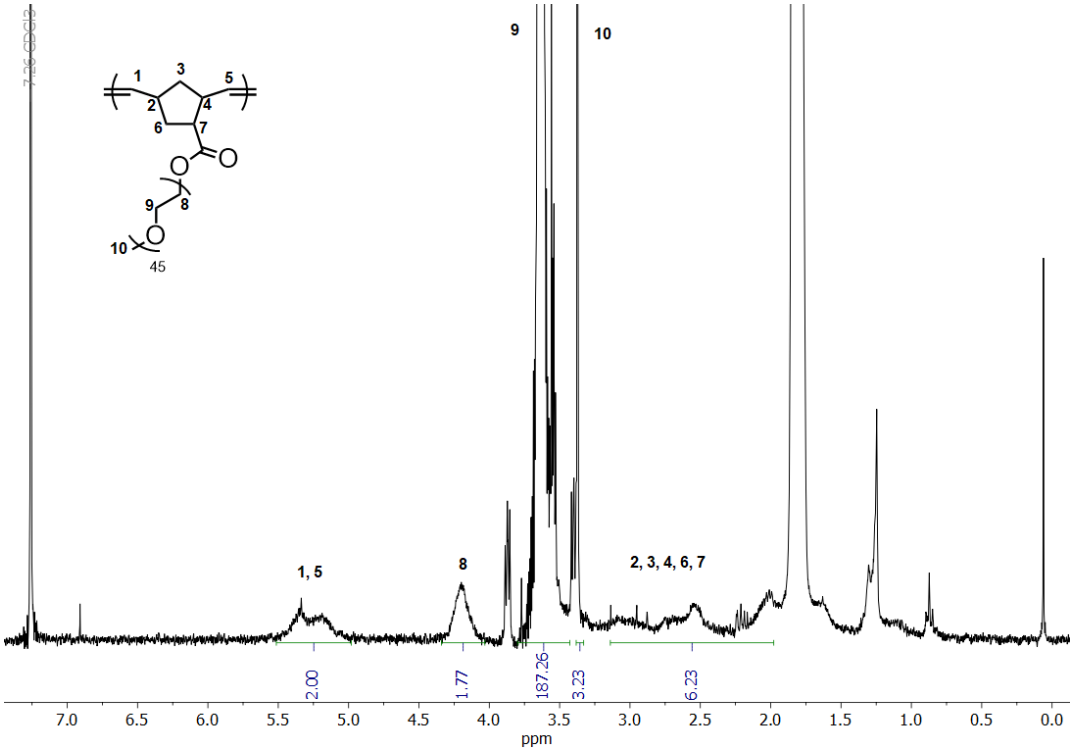


Fig. S14. *^{1}H NMR spectrum of **BBP2** in $CDCl_3$. Complete conversion was determined by the disappearance of the norbornene protons expected at 6.2 ppm.*

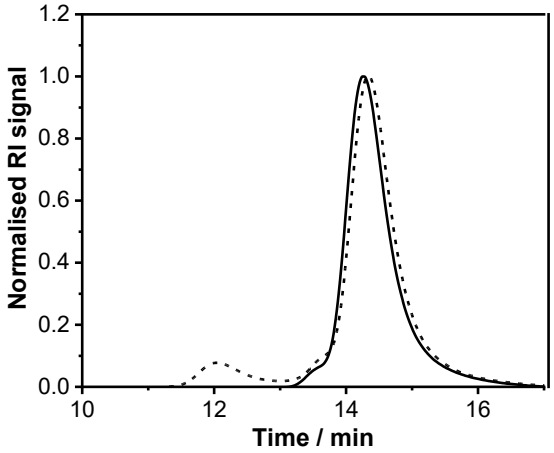


***Fig. S15**. DMF-SEC analysis of **DNA-BBP1** (dash line) and **BBP1** (solid line).*

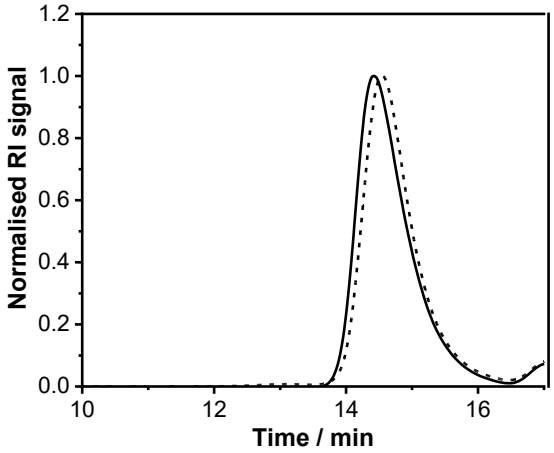


***Fig. S16**. DMF-SEC analysis of **DNA-BBP2** (dash line) and **BBP2** (solid line).*

Fluorescence quenching of *DNA-NH_2

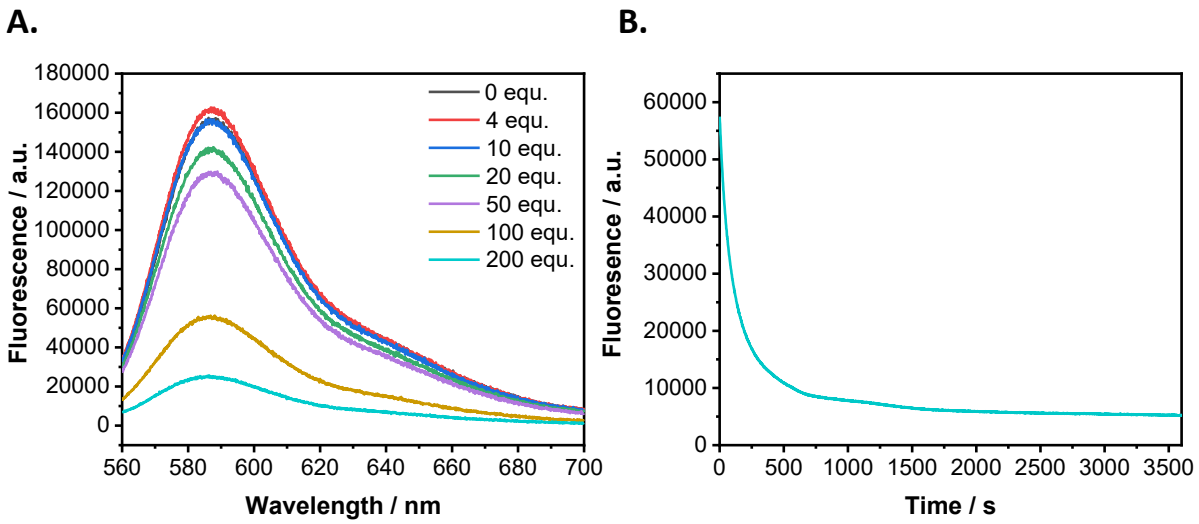


***Fig. S17**. (A) Fluorescence emission spectrum of *DNA-NH_2 (167 nM) in the presence of the macroinitiator. (B) Fluorescence emission at 580 nm in the presence of 200 equivalents of macroinitiator. Excitation λ = 545 nm.*

Purified DNA-BBP2

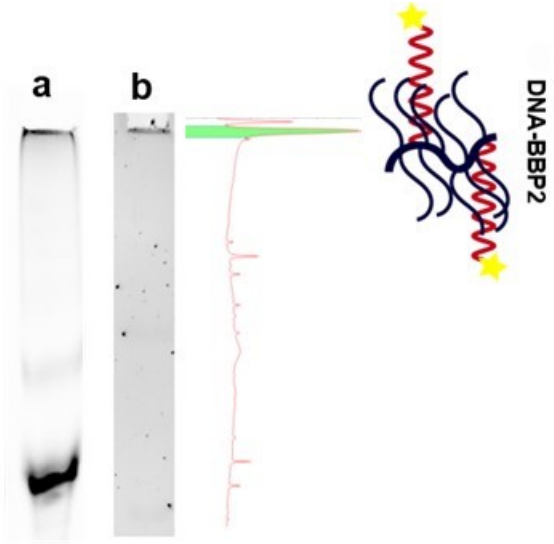


***Fig. S18**. Denaturing PAGE analysis of DNA-BBP2 without staining. (a) Before purification by prep-SEC; and (b) after purification prep-SEC. The corresponding densitometric measurement of lane b is also displayed.*

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