Electronic Supplementary Material (ESI) for ChemComm.

Development of 1,8-Naphthalimide dyes for a rapid imaging of subcellular compartments in plants

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Supporting Information
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I. General

Commercially available reagents were obtained from Tokyo Kasei, Wako Pure Chemical Industries Ltd., KANTO CHEMICAL CO., INC. and Nacalai tesque, and used without further purification.

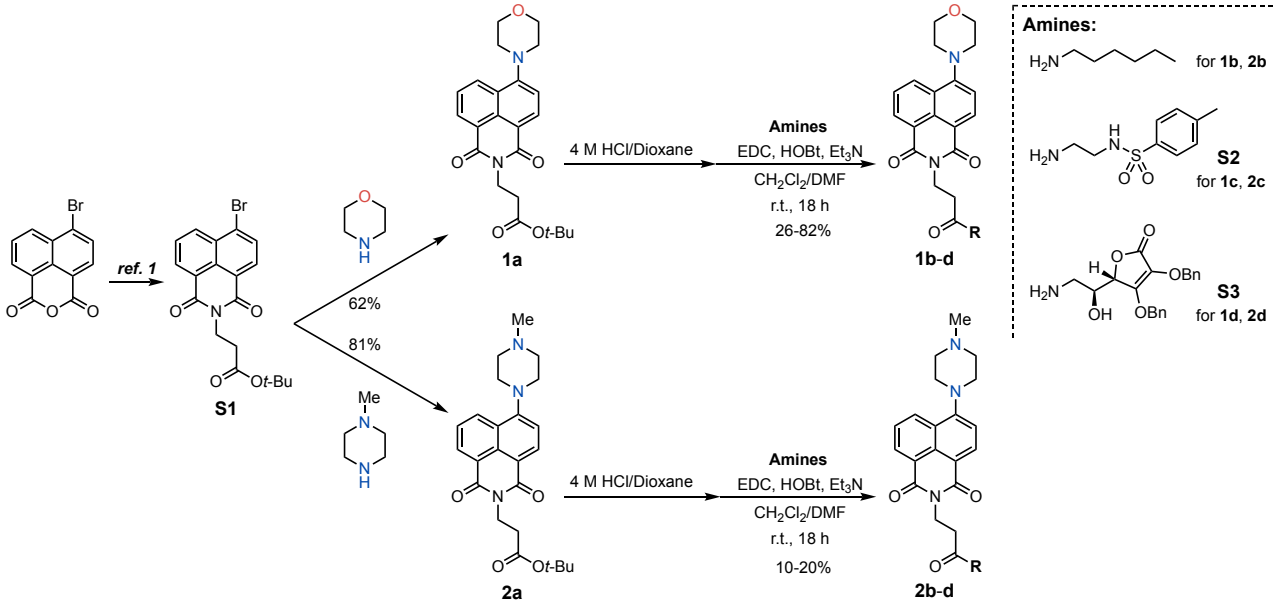
The ^{1}H and ^{13}C NMR were recorded on a Bruker 600 (600 MHz for ^{1}H, 150 MHz for ^{13}C) spectorometer. Chemical shifts were reported in ppm (δ), and coupling constants were reported in Hz. ^{1}H and ^{13}C-resonances were referenced to solvent residual peaks for $CDCl_3$ (^{1}H, 7.26 ppm), DMSO-*d*$_6$ (^{1}H, 2.50 ppm), $CDCl_3$ (^{13}C, 77.2 ppm) and DMSO-*d*$_6$ (^{13}C, 39.5 ppm). Multiplicity and qualifier abbreviations are as follows: s = singlet, d = doublet, t = triplet, q = quartet, quin = quintet, m = multiplet, br = broad, doublet of doublets (dd), doublet of doublets of triplets (dd). Spectra were processed by Bruker Top-spin.

High resolution mass analyses (HRSM) were submitted to the Mass Spectrometry Laboratory at RIKEN. For crude analysis, ultra high-performance liquid chromatography-mass spectrometry (UPLC/MS) was performed on a SHIMADZU LCMS-2020 equipped with a reverse phase C18 column (2.7 μm particle size, 2.1 x 100 mm) and a API/ESI mass spectrometry detector, and UV detector. UV-Vis spectra were recorded on a UV-1600. Fluorescent spectra were recorded on a RF-6000. Absolute quantum yields were determined using a SHIMADZU RF-6000 equipped with a calibrated integrating sphere system.

Thin-layer chromatography was performed on Merck 60 F254 precoated silica gel plates. Column chromatography was performed on silica gel (Silica Gel 60 N; 63−210 mesh, KANTO CHEMICAL CO., INC. or 40−50 mesh, KANTO CHEMICAL CO., INC.).

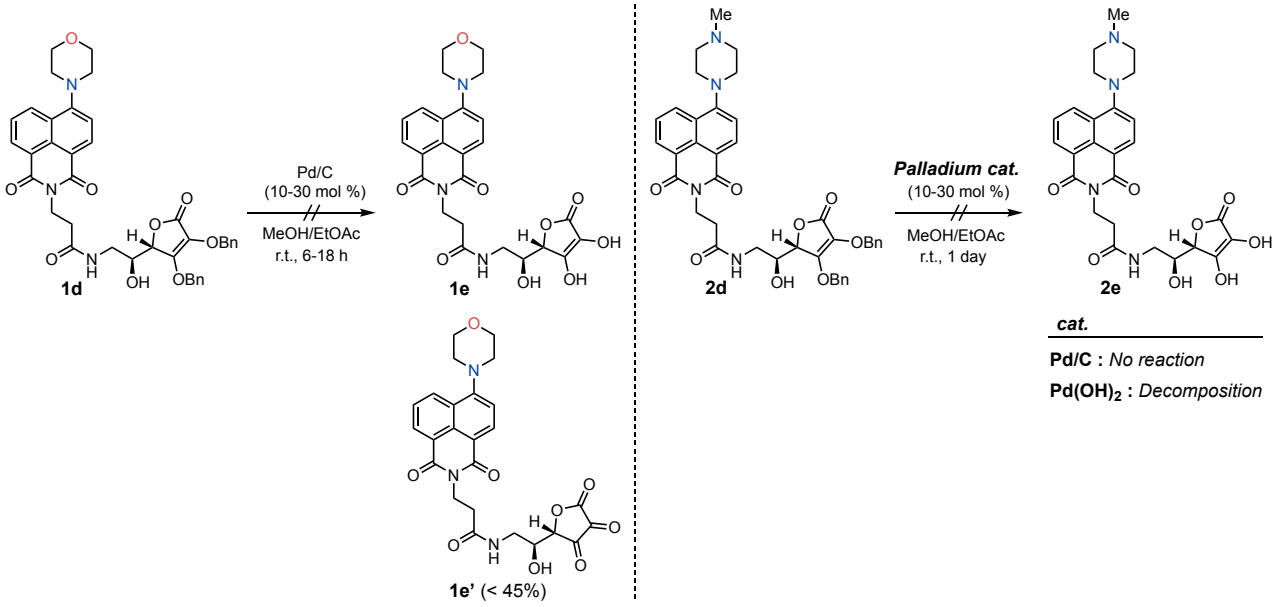
II. Synthesis of naphthalimide-based probes 1 and 2

Scheme S1. Synthesis of naphthalimide-based probes **1** and **2**



Comments: Probes **1** and **2** were synthesized from 4-bromo-1,8-naphthalic anhydride as a common starting material. After the acidic cleavage of the tertiary butyl group of **1a** and **2a**, the resulting product was used in subsequent steps without any purification.

Scheme S2. Synthesis of **1e** and **2e**



Comments: After the purification with reverse phase chromatography, the oxidized product **1e'** and unidentified product were provided. The chemical structure of **1e'** was determined from NMR and mass spectroscopy. Because the mass signal (m/z = 512, calculated for $[M + H]^+$) corresponding to **1e** was observed in LC/MS analysis for the reaction mixture, we assumed that the oxidization of **1e** occurred during the purification.

III. Photophysical properties of 1 and 2

For spectroscopic measurements, the stock solutions of **1** and **2** were prepared as 5 mM solution in DMSO. All spectra were obtained with 1.0 cm square quartz cuvette. Both excitation and emission slit widths were of 5 nm, respectively. To determine the pK_a values of the Me-piperazine moiety on **2**, the fluorescence spectra were measured under various pH conditions. The pKa values were calculated according to the Henderson-Hasselbach type equation; $\log[(F_{max} - F)/(F - F_{min})]) = pK_a - pH$, where F represents fluorescence intensity.

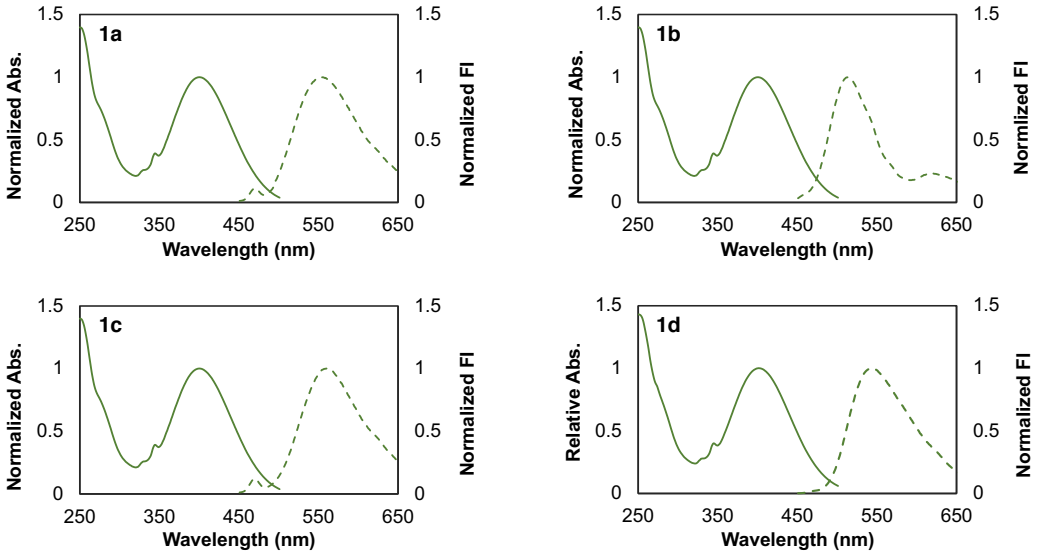


Fig. S1. Normalized absorption (solid line) and fluorescence spectra (dashed line) of **1** in phosphate buffer (20 mM, pH 7.2) containing 0.1% or 0.4% DMSO, λ_{ex} = 405 nm, at 23 ºC, [**1**] = 5 or 20 μM for measuring fluorescent and absorption spectra, respectively. A precipitation was slightly observed for **1b** in this measuring condition.

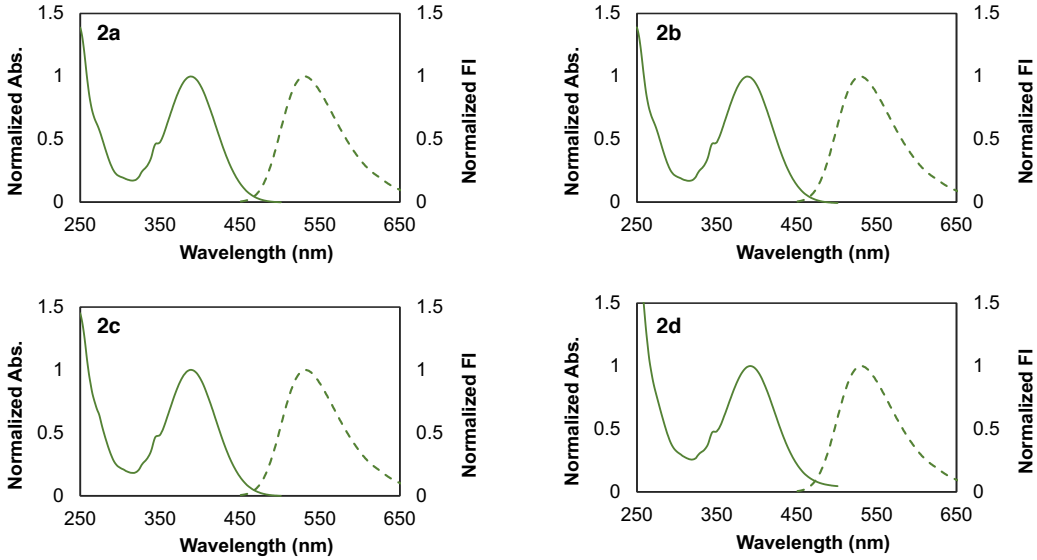


Fig. S2. Normalized absorption (solid line) and fluorescence spectra (dashed line) of **2** in phosphate buffer (20 mM, pH 5.7) containing 0.1% or 0.4% DMSO, λ_{ex} = 405 nm, at 23 ºC, [**2**] = 5 or 20 μM for measuring fluorescent and absorption spectra, respectively.

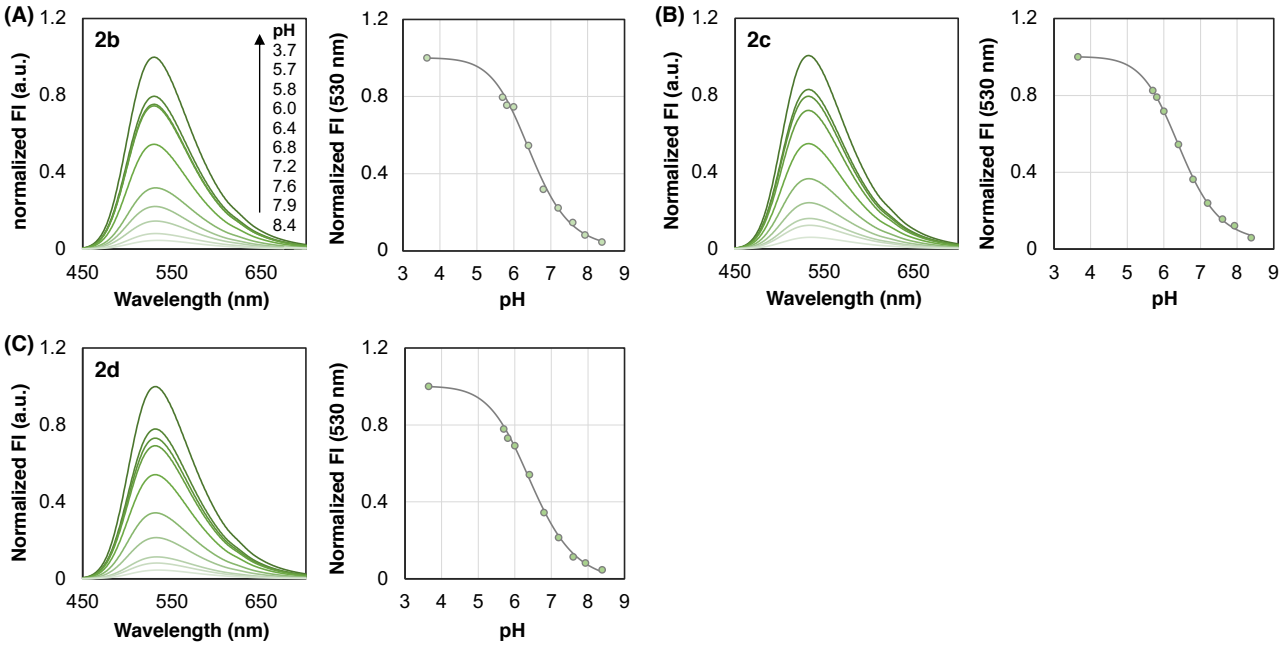


Fig. S3. Normalized fluorescence spectra of **2b**-**c** (5 μM) at different pH conditions and the plots of normalized fluorescence intensity against pH. All measurements were performed in pH-controlled 20 mM phosphate buffer containing 0.1% DMSO, λ_{Ex} = 405 nm, at 23 ºC.

Table S1. Quantum yields (Φ) of **2** in aqueous and organic solvents[a)]

	pH 7.2 Phosphate buffer	pH 5.7 Phosphate buffer	pH 3.7 Phosphate buffer	EtOH	CH_3CN	THF	CH_2Cl_2
2a	0.11	0.32	0.41	< 0.01	0.02	0.02	0.03
2b	0.1	0.37	0.44	< 0.01	0.02	0.02	0.04
2c	0.1[b)]	0.3	0.41	< 0.01	0.01	0.02	0.03
2d	0.09[b)]	0.27	0.35	< 0.01	0.01	0.02	0.04

a) Containing 0.1% DMSO in each solvent. b) In this measuring condition, small quantity of precipitation was observed.

IV. Imaging studies with confocal laser scanning microscopy (CLSM)

Arabidopsis (*Arabidopsis thaliana*) plants ecotype Columbia were used for live-cell imaging analysis of the naphthalimide probes. The plants were grown in soil in chamber at 23°C under a 14-h-light/10-h-dark photoperiod using LED lamps (140 μmol m^{-2} s^{-1}). Second or third rosette leaves of 14- to 21-d-old plants were used for the imaging via a CLSM system (LSM 900 system; Carl Zeiss) equipped with a 40x objective lens (C-Apochromat 40x/1.20 W Korr; Carl Zeiss). 10 mM MES-NaOH (pH 5.5) buffer containing 10 μM probes (**1** and **2**) were prepared and directly infiltrated into the excised leaves from its abaxial surface via a 1-ml syringe, followed by the observation by the confocal microscopy. The emissions between 410–546 nm for the naphthalimide probes and 650–700 nm for chlorophyll autofluorescence were detected simultaneously following the excitation by 405- and 640-nm diode lasers. For the fluorescence detection of red fluorescent protein (RFP) variants, the emission between 570–650 nm was detected following the excitation by 561-nm diode laser. Each detection was switched every line scan during the colocalization assay between the probes and RFP variants.

High light treatment was performed as previously described.[1] Plants were exposed to strong visible light (2,000 μmol m^{-2} s^{-1}) for 2 h at 23°C, and then cultivated in the indicated growth conditions for 2 d, followed by microscopic observations. 10 mM MES-NaOH (pH 5.5) buffer containing 20 μM **1a** or 10 μM **2a** was infiltrated into the leaves before the observations. The light was provided by a Xenon light source (MAX-303; Asahi Spectra) equipped with a mirror module (MAX-VIS; Asahi Spectra) and a rod lens (RLQL80-1; Asahi Spectra) to emit visible light (wavelength between 385 and 740 nm) with uniform intensity. The light intensity was measured with a data logger (LI-250A; LI-COR) equipped with a photosynthetic photon flux density sensor (LI-190R; LI-COR).

For colocalization assay between the probes and fluorescent protein markers visualizing intracellular components, we generated transgenic Arabidopsis plants expressing RFPs targeted to cytoplasm, vacuolar membrane, chloroplast outer envelope, endoplasmic reticulum (ER), respectively, as follows.

For the visualization of cytoplasm, we generated the plants expressing monomeric red fluorescent protein (mRFP) fused to nuclear export signal (NES; mRFP-NES). mRFP fragment was amplified by PCR using the primers mRFP_F and NES-mRFP_R (Table S2). The reverse primer contains NES from protein kinase inhibitor (PKI)[2] encoding the amino acids peptides ELALKLAGLDIN. The amplicon was cloned into the vector pENTR1A (Invitrogen) in a SLiCE (Seamless Ligation Cloning Extract) reaction[3], and then transferred to the vector pUB-Dest[4] in a LR clonase II (Invitrogen) reaction. For vacuolar membrane-marker expressing plants, the coding sequence of delta tonoplast intrinsic protein (δTIP; At3g16240) was amplified from Arabidopsis cDNA by PCR using the primers δTIP_F and δTIP_R (Table S2), cloned into pENTR/D-TOPO (Invitrogen), and transferred to the vector pUBC-mRFP-Dest[4] in a LR clonase II reaction.

For chloroplast outer envelope marker, the genomic fragment comprising 860-bp upstream from the start codon to the region just before the stop codon of Translocon at the outer membrane of chloroplasts 64-III (TOC64-III; AT3G17970) was amplified from Arabidopsis genomic DNA by PCR using the primers TOC64-III_F and TOC64-III_R (Table S2), cloned into the pDONR P4-P1r (Invitrogen) in a BP clonase II reaction. The coding sequence of an RFP derivative mRuby3[5] was amplified by PCR using the primers mRuby_F and mRuby_R (Table S2), and cloned into the pDONR221. The two fragments were then inserted into the vector

R4pGWB501[6] in a LR clonase II plus (Invitrogen) reaction. For the visualization of ER, the cDNA fragment comprising C-terminal transmembrane domain (TMD) of CYTOCHROME B5 ISOFORM B (CB5-B; At2g32720)[7] was amplified from the Arabidopsis cDNA by PCR using the primers CB5 TMD_F and CB5 TMD_R (Table S2), cloned into pENTR1A, and then transferred to the vector pUBN-mRFP-Dest[4].

The resulting vectors were introduced into Agrobacterium (*Agrobacterium tumefaciens*) strain GV3101 and then introduced into Arabidopsis ecotype Columbia by the floral dip method[8]. In the generated transgenic plants, each construct visualizes the target compartment (Fig. S4), respectively.

For the imaging of the model green algae *Chlamydomonas reinhardtii*, the wild-type strain CC-124 were used. The cells were grown in liquid Tris-acetate-phosphate (TAP) solution[9] in a rotation incubator at 23°C under continuous light (10 μmol m^{-2} s^{-1}). Cells in stationary phase were diluted to a density of 0.5 × 10^6 cells mL^{-1} in new TAP solution, then cultured for 4 d, and then harvested by centrifugation at 600 g for 3 min and resuspended in fresh TAP solution containing 50 μM **2a** probes. After the incubation for 5 minutes, the cells were observed via CLSM system (LSM 900 system; Carl Zeiss) equipped with a 63x objective lens (Plan-Apochromat 63x/1.40 Oil DIC M27; Carl Zeiss).

Table S2. The sequences of primers for gene cloning

Primer name	Primer sequence (5' to 3')	Amplicon size (bp)
mRFP_F	AGGAACCAATTCAGTCGACATGGCCTCCTCCGAGGACG	714
mRFP-NES_R	AAAGCTGGGTCTAGATATCTTAGTTAATATCAAGTCCAGCCAACTTAAGAGCAAGCTCGGCGCCGGTGGAGTGGCGG	
δTIP_F	CACCATGGCTGGAGTTGCCTTTG	753
δTIP_R	GAAATCAGCAGAAGCAAGAG	
TOC64-III_F	GGGGACAACTTTGTATAGAAAAGTTGATACGTCGGTTCATGTGTG	4622
TOC64-III_R	GGGGACTGCTTTTTTGTACAAACTTGCCTGGAATTTTCTCAGTCTC	
mRuby_F	GGGGACAAGTTTGTACAAAAAAGCAGGCTTCATGGTTTCAAAGGGCGAGG	681
mRuby_R	GGGGACCACTTTGTACAAGAAAGCTGGGTTCACTTATATAACTCATC	
CB5 TMD_F	AGGAACCAATTCAGTCGACTTCATAATCAAGCTCCTC	87
CB5 TMD_R	AAAGCTGGGTCTAGATATCCTACCCTGATTTGGTGTAG	

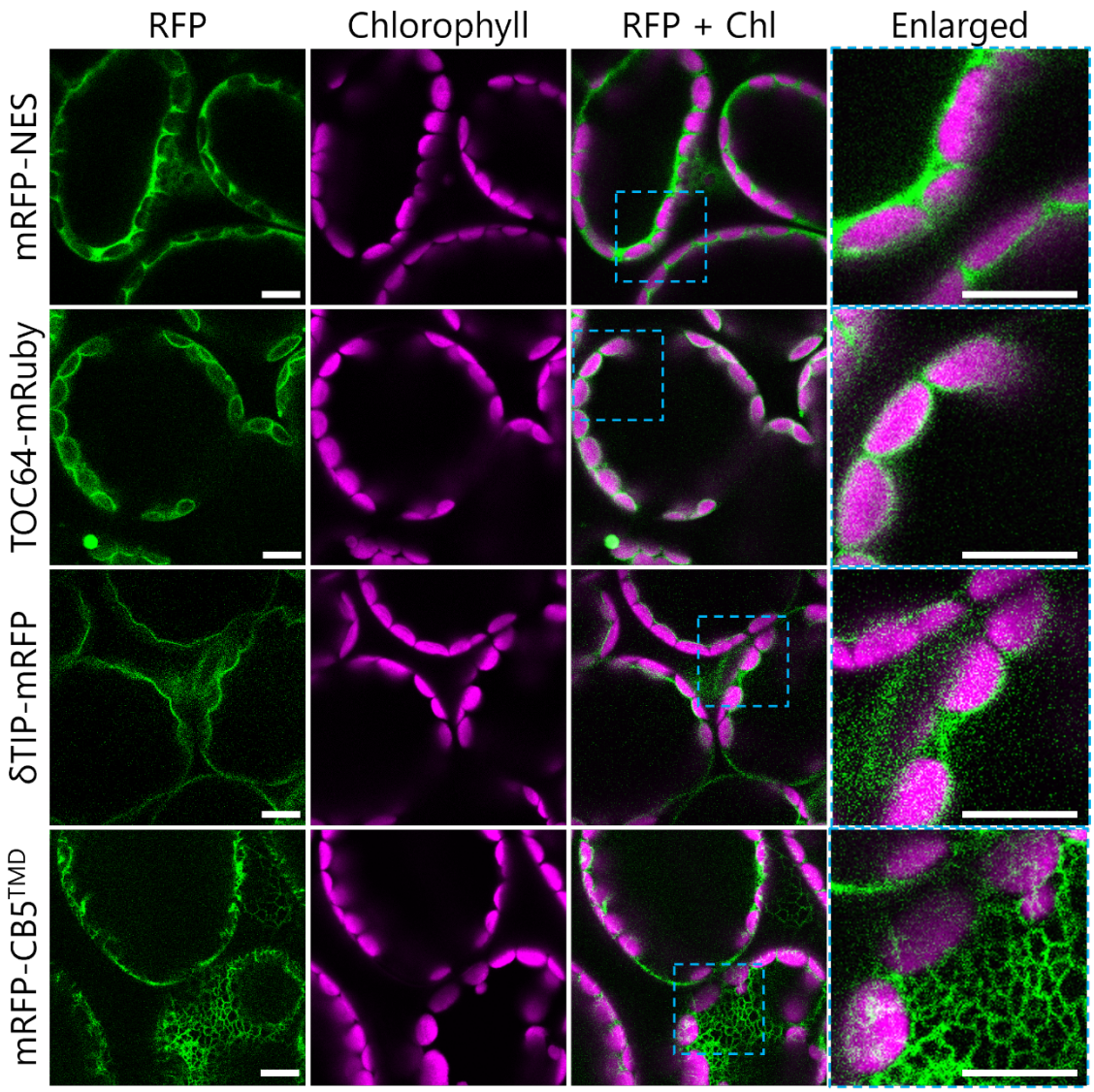


Fig. S4. Confocal images of mesophyll cells expressing red fluorescent proteins (RFPs) that are targeted to the cytoplasm (mRFP-NES), chloroplast outer envelope (TOC64-mRuby), the vacuolar membrane (δTIP-mRFP) or ER (mRFP-CB5^{TMD}) from leaves of transgenic plants expressing respective markers. Green, RFP fluorescence; magenta, chlorophyll autofluorescence (Chl). Scale Bars = 10 μm.

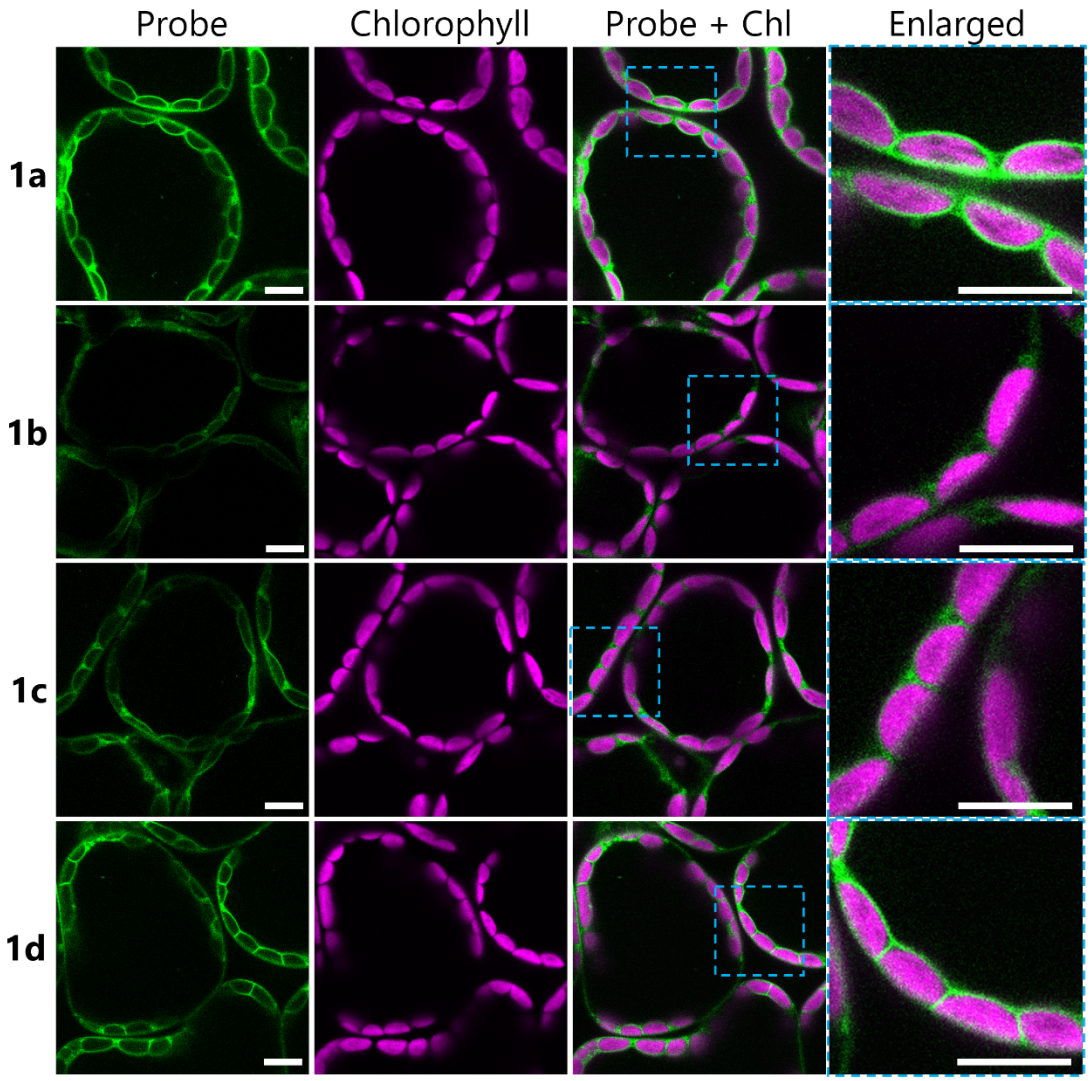


Fig. S5. Confocal images of mesophyll cells from wild-type leaves stained with naphthalimide-based probes **1a–d**. Green, probe fluorescence; magenta, chlorophyll autofluorescence (Chl). Scale Bars = 10 μm.

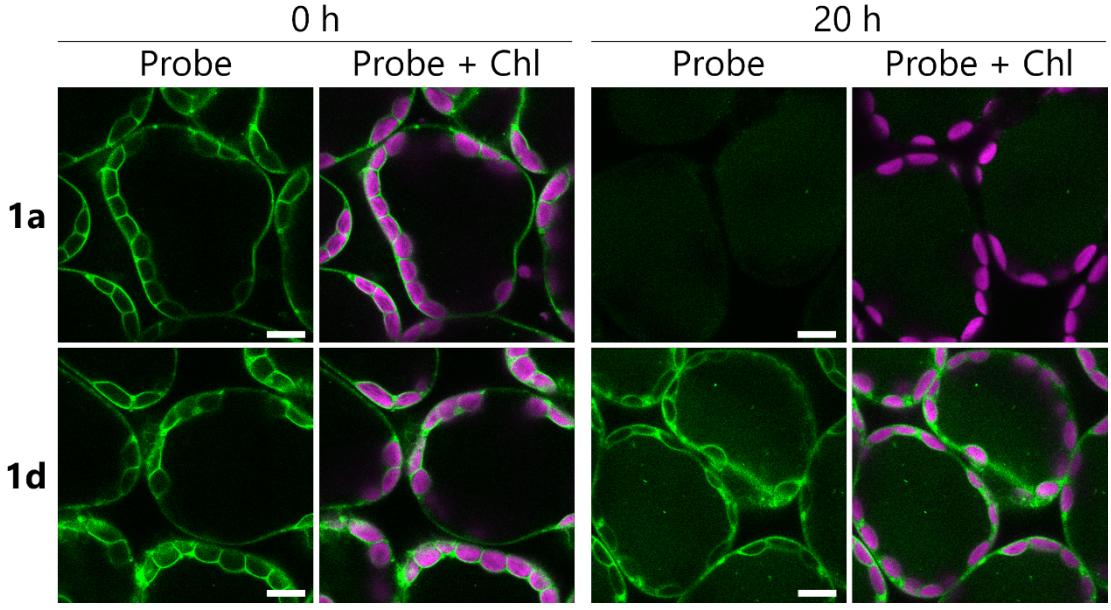


Fig. S6. Confocal images of mesophyll cells from wild-type leaves stained with naphthalimide-based probes **1a** and **1d**. Green, probe fluorescence; magenta, chlorophyll autofluorescence (Chl). Scale Bars = 10 μm.

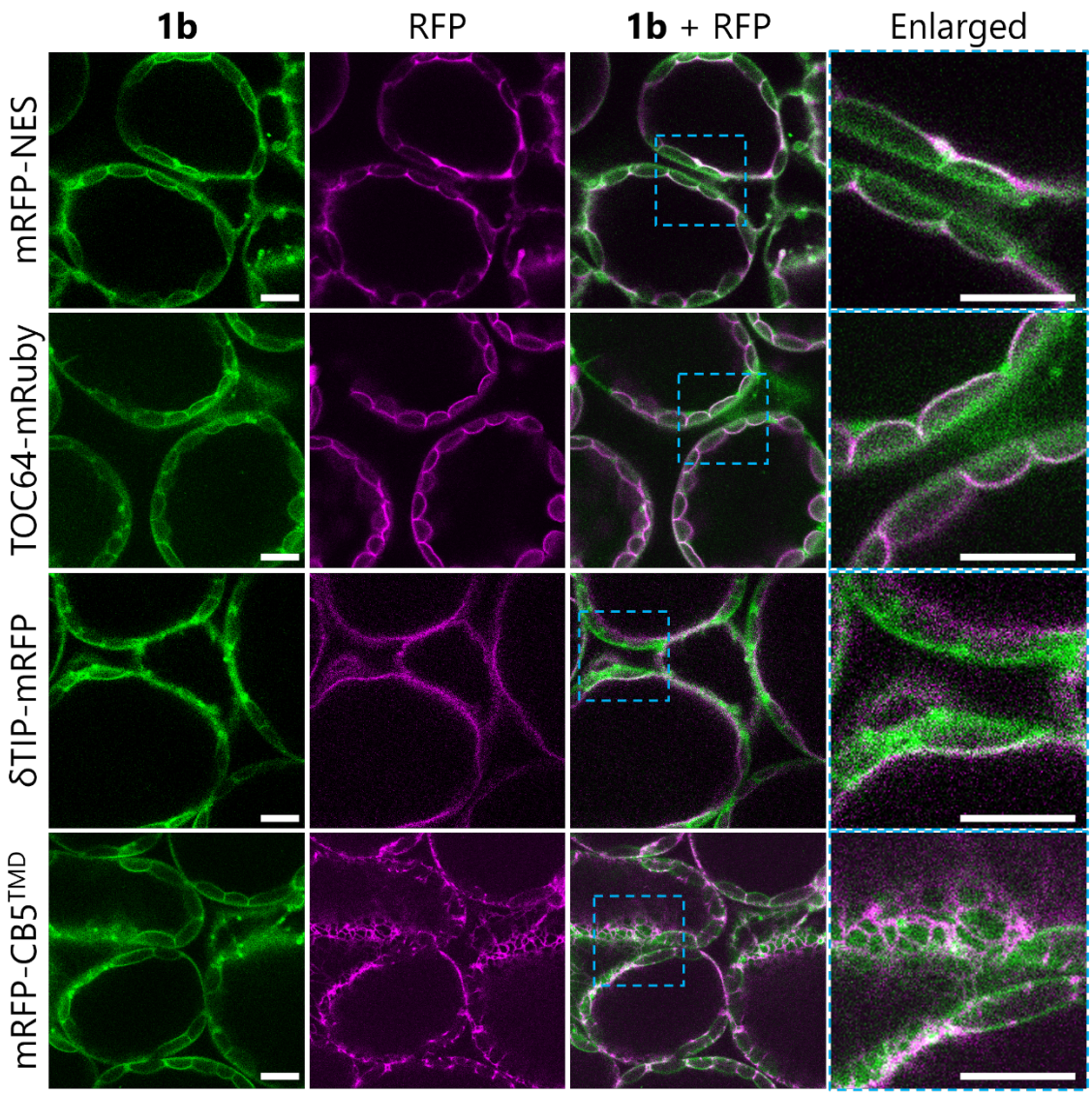


Fig. S7. Confocal images of mesophyll cells expressing red fluorescent proteins (RFPs) that are targeted to the cytoplasm (mRFP-NES), chloroplast outer envelope (TOC64-mRuby), the vacuolar membrane (δTIP-mRFP) or ER (mRFP-CB5^{TMD}) from leaves stained with the probe **1b**. Green, probe fluorescence; magenta, RFP fluorescence. Scale Bars = 10 μm.

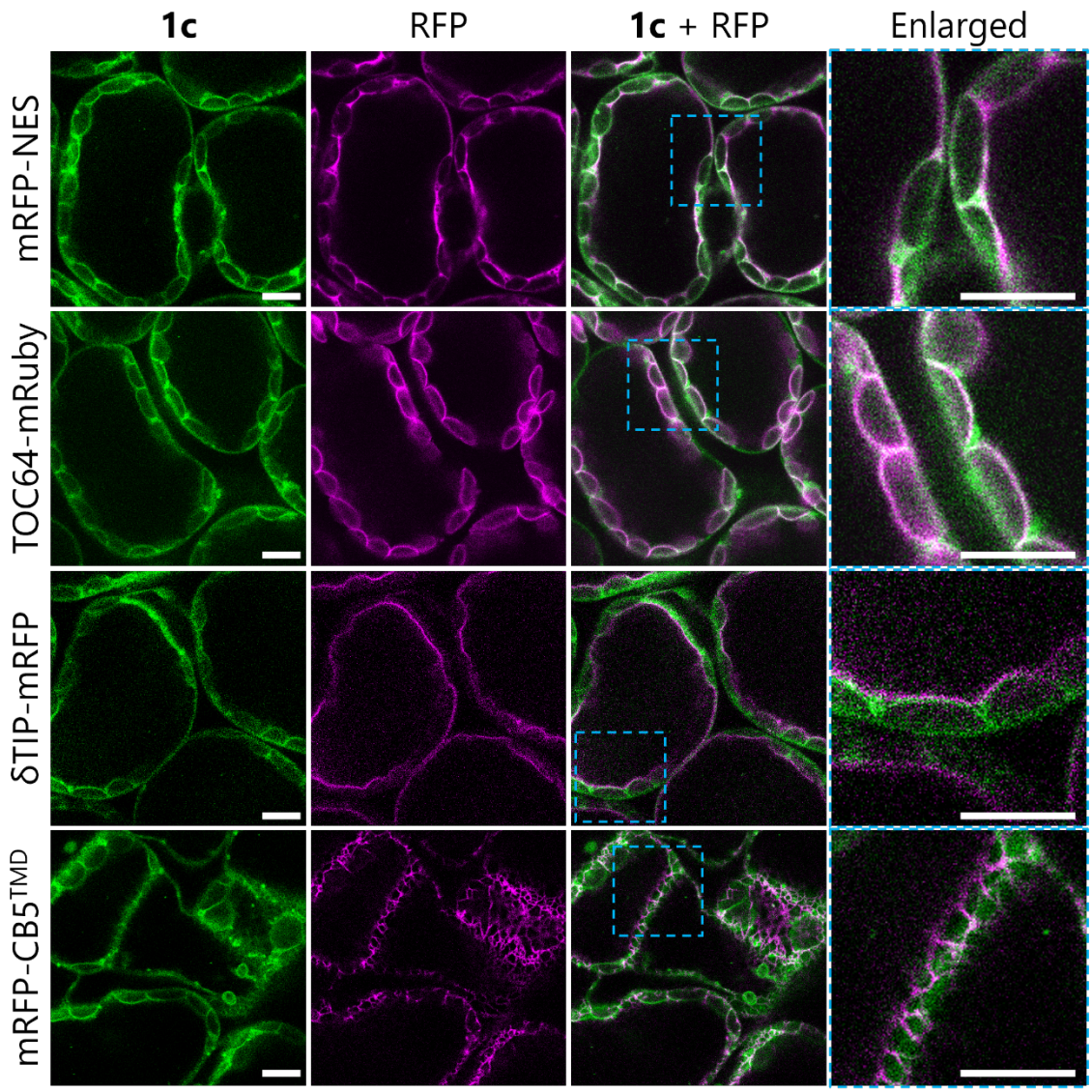


Fig. S8. Confocal images of mesophyll cells expressing red fluorescent proteins (RFPs) that are targeted to the cytoplasm (mRFP-NES), chloroplast outer envelope (TOC64-mRuby), the vacuolar membrane (δTIP-mRFP) or ER (mRFP-CB5^{TMD}) from leaves stained with the probe **1c**. Green, probe fluorescence; magenta, RFP fluorescence. Scale Bars = 10 μm.

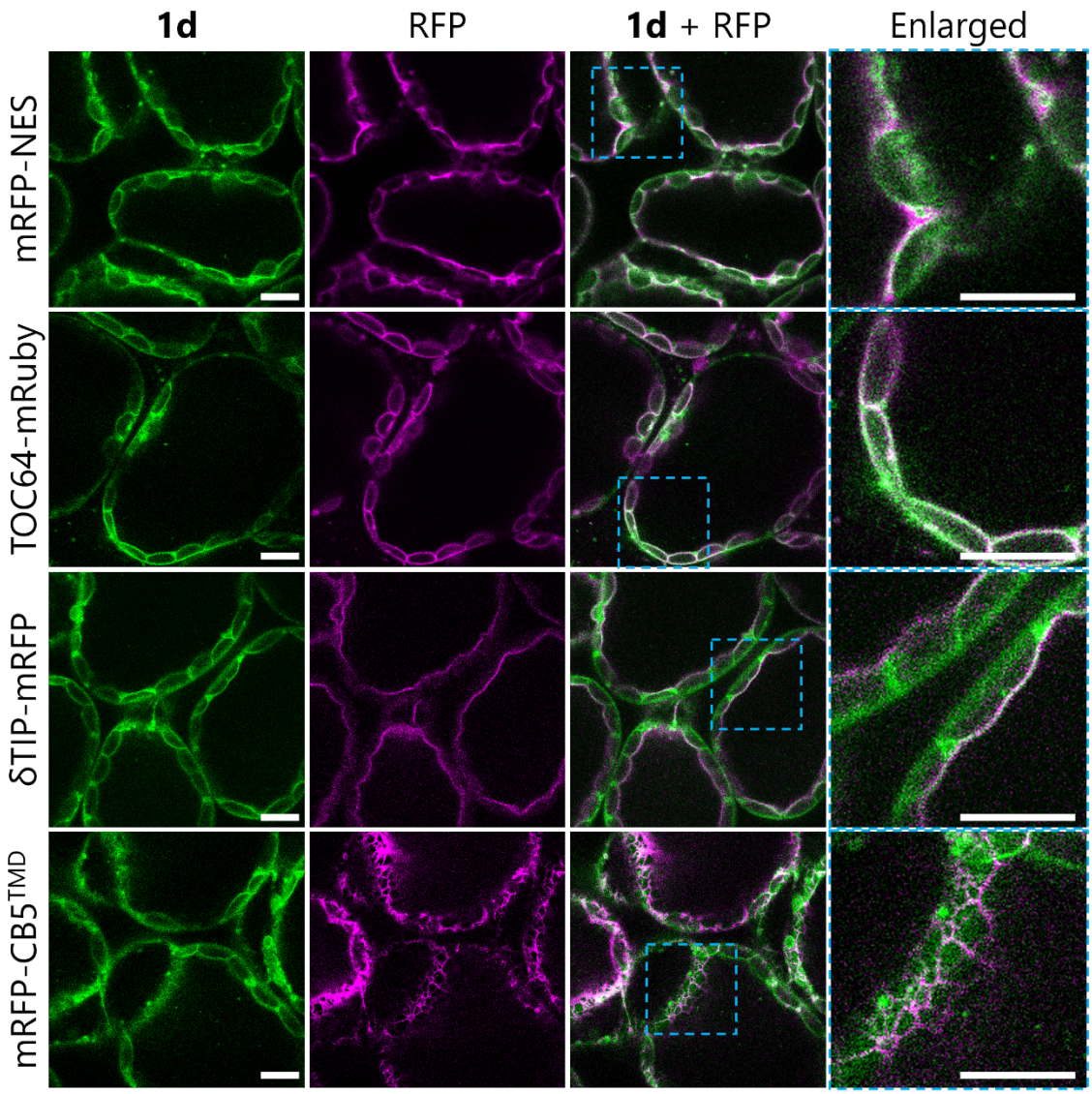


Fig. S9. Confocal images of mesophyll cells expressing red fluorescent proteins (RFPs) that are targeted to the cytoplasm (mRFP-NES), chloroplast outer envelope (TOC64-mRuby), the vacuolar membrane (δTIP-mRFP) or ER (mRFP-CB5^{TMD}) from leaves stained with the probe **1d**. Green, probe fluorescence; magenta, RFP fluorescence. Scale Bars = 10 μm.

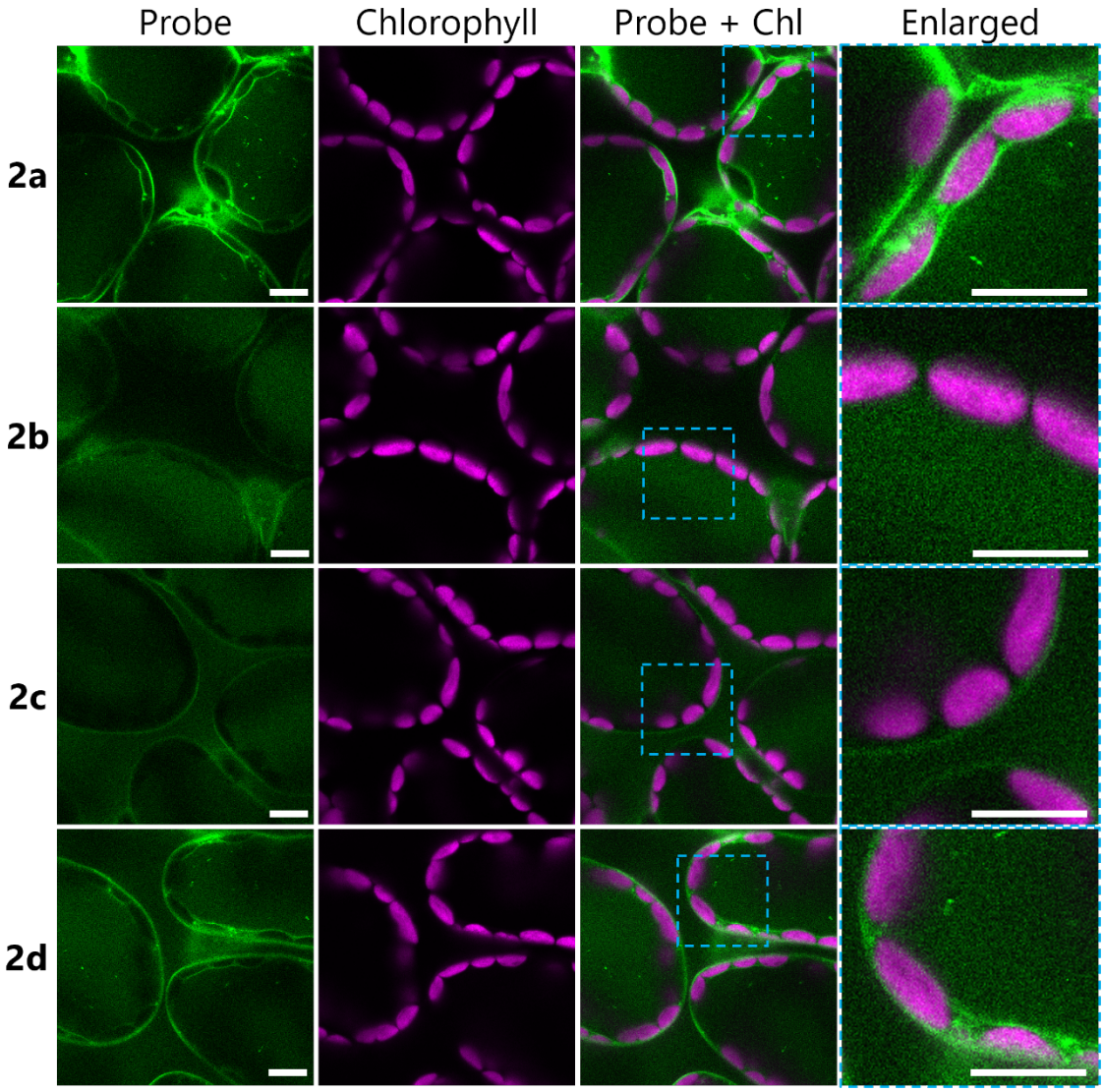


Fig. S10. Confocal images of mesophyll cells from wild-type leaves stained with naphthalimide-based probes **2a**–**d**. Green, probe fluorescence; magenta, chlorophyll autofluorescence (Chl). Scale Bars = 10 μm.

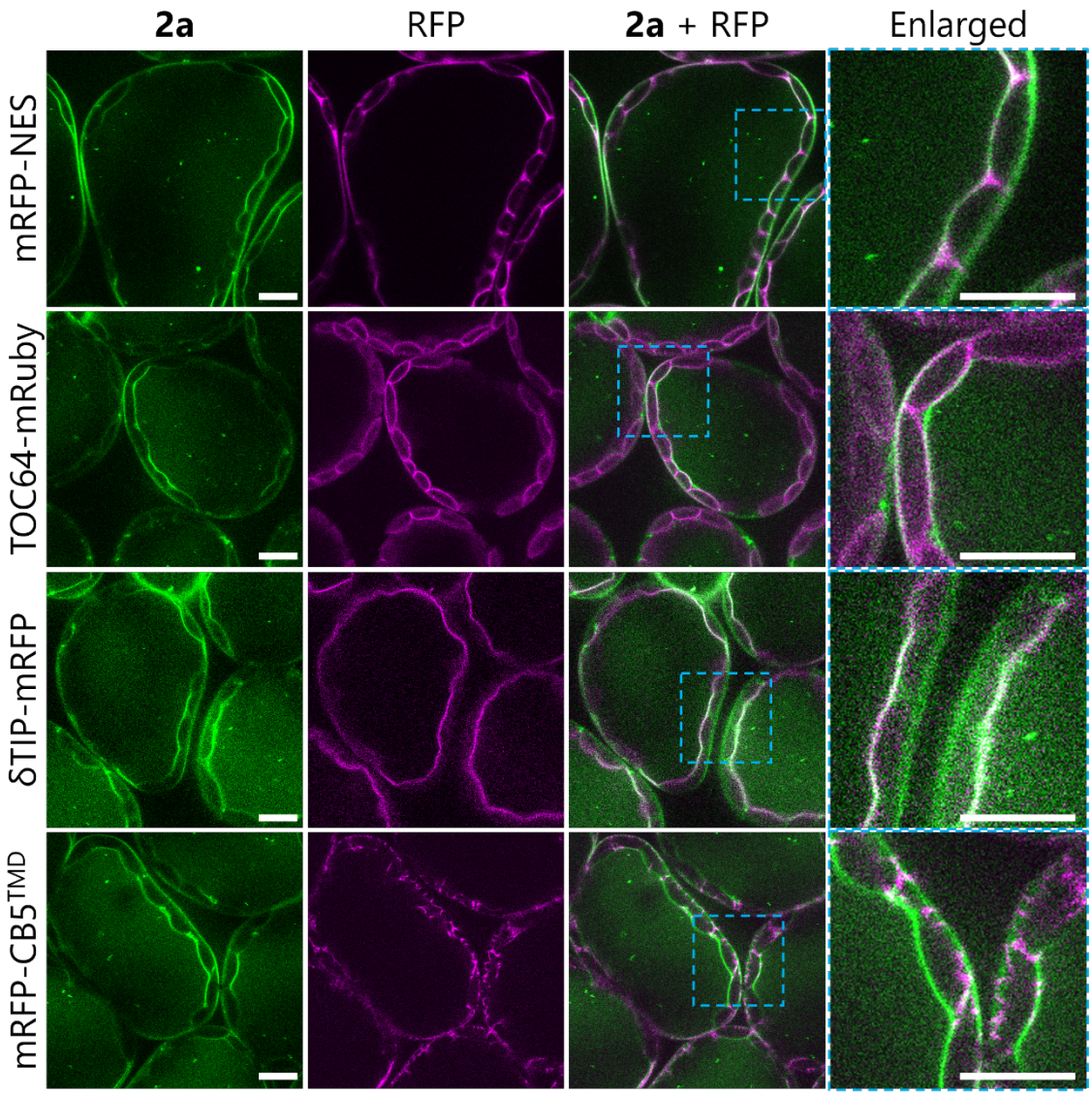


Fig. S11. Confocal images of mesophyll cells expressing red fluorescent proteins (RFPs) that are targeted to the cytoplasm (mRFP-NES), chloroplast outer envelope (TOC64-mRuby), the vacuolar membrane (δTIP-mRFP) or ER (mRFP-CB5^{TMD}) from leaves stained with the probe **2a**. Green, probe fluorescence; magenta, RFP fluorescence. Scale Bars = 10 μm.

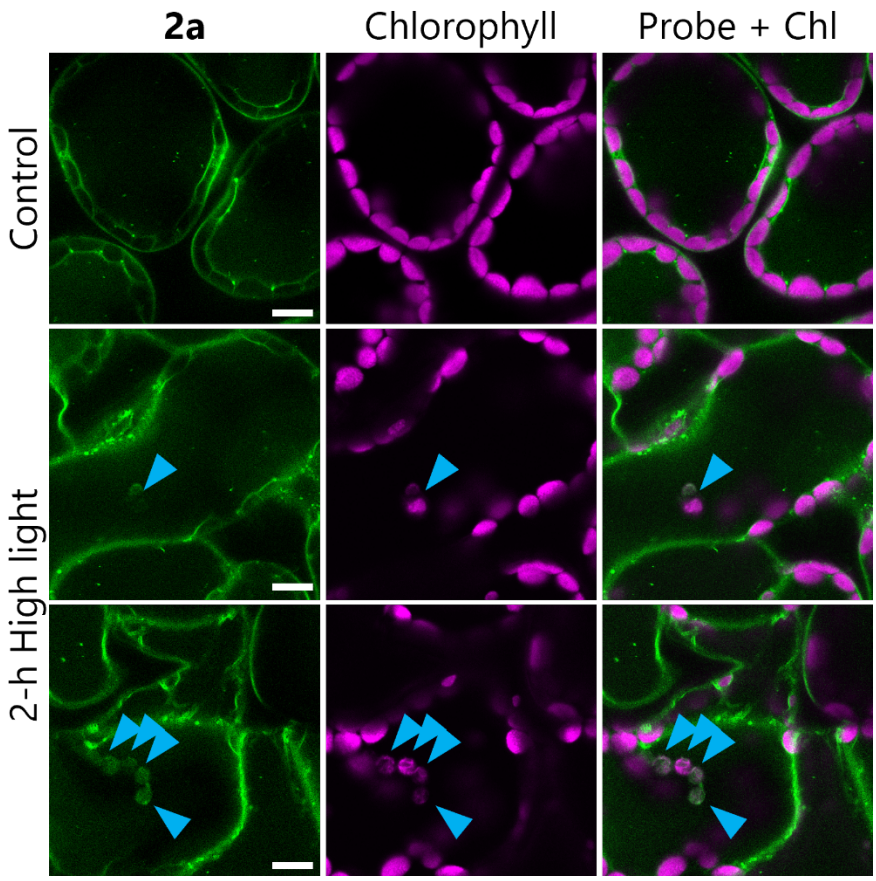


Fig. S12. Confocal images of mesophyll cells stained with the naphthalimide-based probe **2a** from non-treated control leaves or leaves 2 d after a high light treatment (2,000 μmol m^{-2} s^{-1}) for 2 h. Closed arrowheads indicate vacuole-enclosed chloroplasts. Green, probe fluorescence; magenta, chlorophyll autofluorescence (Chl). Scale Bars = 10 μm.

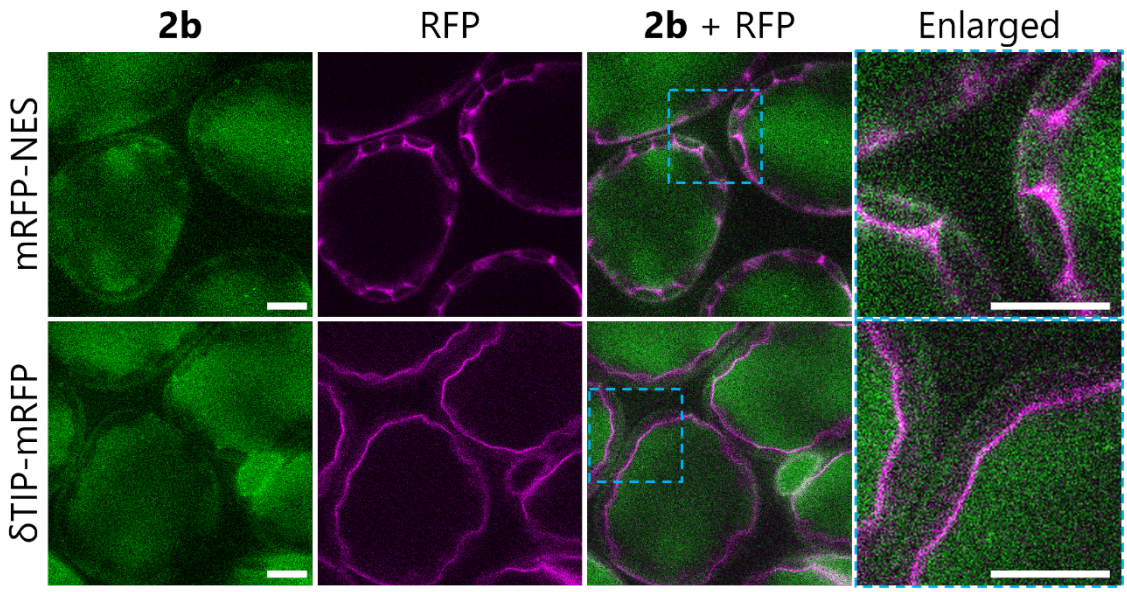


Fig. S13. Confocal images of mesophyll cells expressing red fluorescent proteins (RFPs) that are targeted to the cytoplasm (mRFP-NES) or the vacuolar membrane (δTIP-mRFP) from leaves stained with the probe **2b**. Green, probe fluorescence; magenta, RFP fluorescence. Scale Bars = 10 μm.

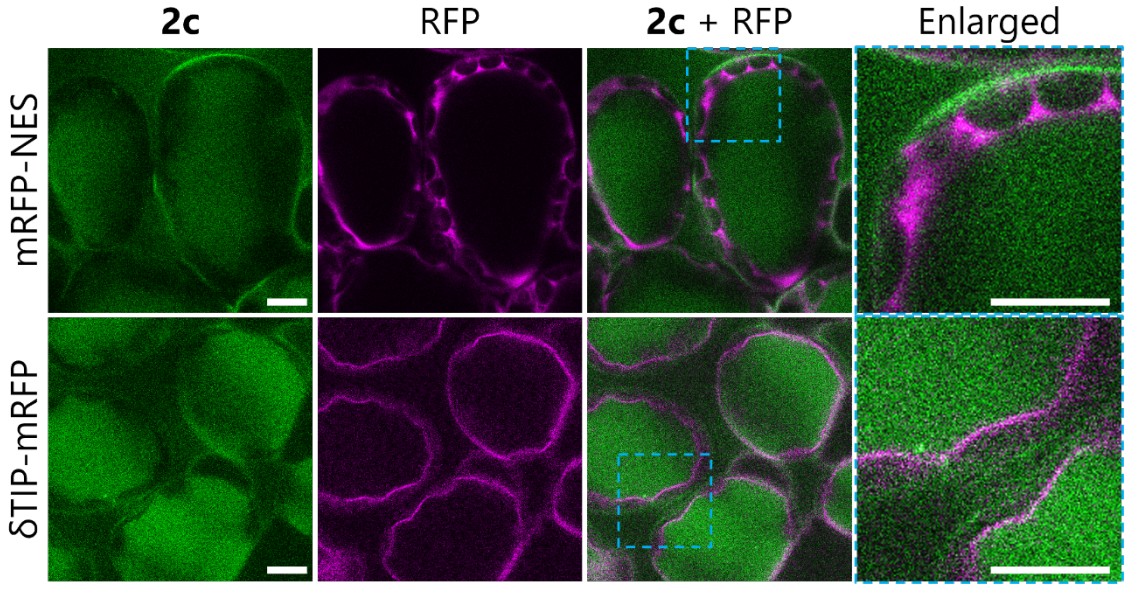


Fig. S14. Confocal images of mesophyll cells expressing red fluorescent proteins (RFPs) that are targeted to the cytoplasm (mRFP-NES) or the vacuolar membrane (δTIP-mRFP) from leaves stained with the probe **2c**. Green, probe fluorescence; magenta, RFP fluorescence. Scale Bars = 10 μm.

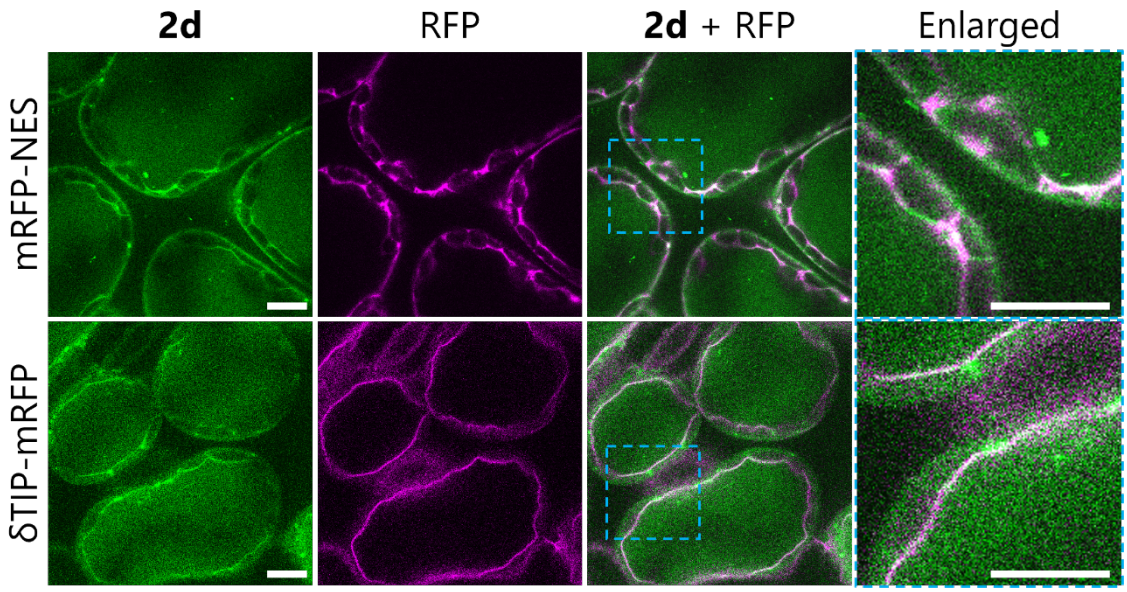


Fig. S15. Confocal images of mesophyll cells expressing red fluorescent proteins (RFPs) that are targeted to the cytoplasm (mRFP-NES) or the vacuolar membrane (δTIP-mRFP) from leaves stained with the probe **2d**. Green, probe fluorescence; magenta, RFP fluorescence. Scale Bars = 10 μm.

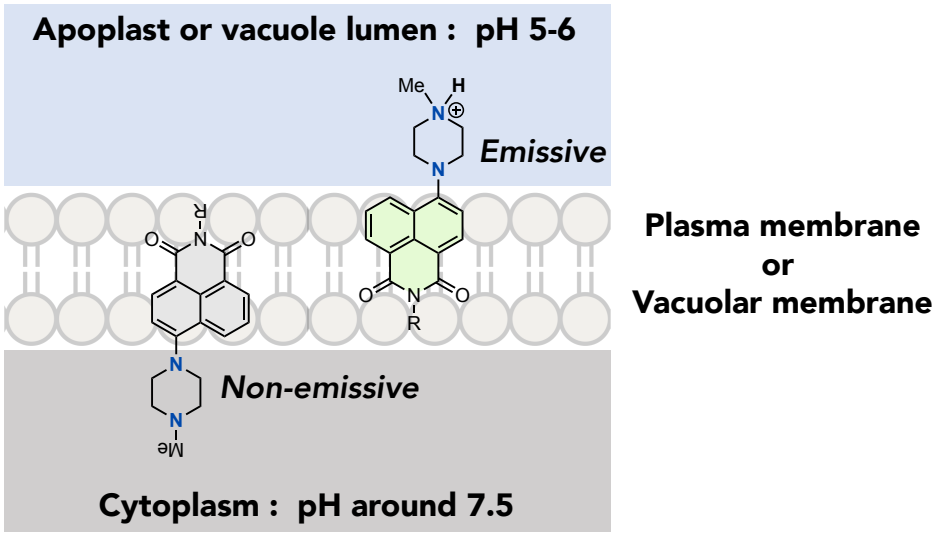


Fig. S16. Schematic illustration for the plasma- and vacuolar membrane-localized **2a**.

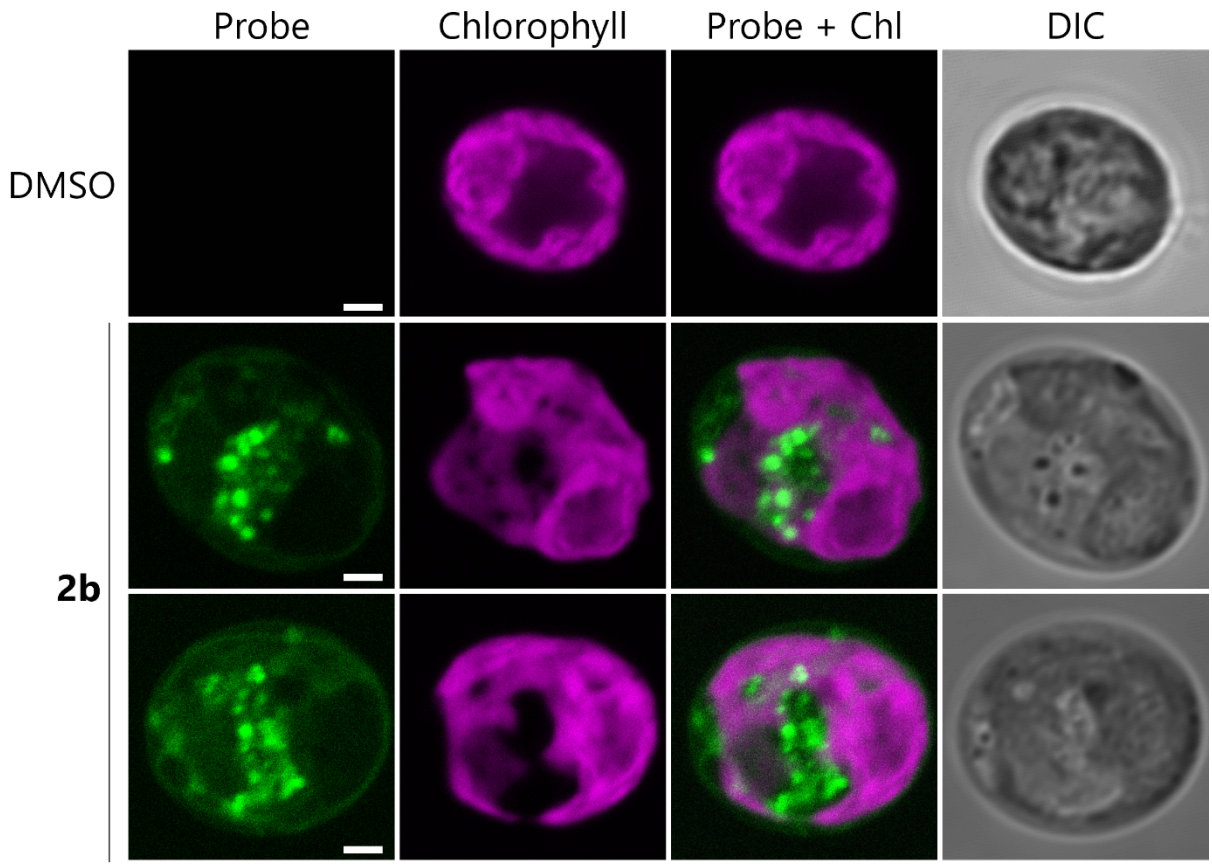


***Fig. S17*.** Confocal images of representative cells of *C. reinhardtii* strain CC-124 with or without the probe **2b**. Two independent representative cells are shown for the cells stained with the probe **2b**. Differential interference contrast (DIC) images are also shown. Green, probe fluorescence; magenta, chlorophyll autofluorescence (Chl). Scale Bars = 2 μm.

V. Assessment of cell toxicity of naphthalimide-based probes

We monitored cell death in the leaves treated with the probes to assess their cytotoxicity. 10 mM MES-NaOH (pH 5.5) buffer containing 10 μM probes (**1a**, **1d**, **2a**, or **2b**) were infiltrated into the second rosette leaves of Arabidopsis plants. After the incubation for 18 h, we confirmed that the presence of the probes does not lead to the appearance of dead cells. Dead cell stain was performed as previously described[10], with slight modification. The leaves were incubated for 5 min at 95°C in lactophenol trypan blue solution (10 mL of lactic acid, 10 mL of glycerol, 10 g of phenol, 10 mg of trypan blue, dissolved in 10 mL of distilled water), followed by destaining in chloral hydrate solution (25 g of chloral hydrate dissolved in 10 mL of distilled water) for 30 min. The images were obtained via a microscopy (Axio Observer; Carl Zeiss) equipped with a Fluar 5× objective (numerical aperture = 0.25; Carl Zeiss) and a CMOS camera (G3CMOS02300KPA; ToupTek).

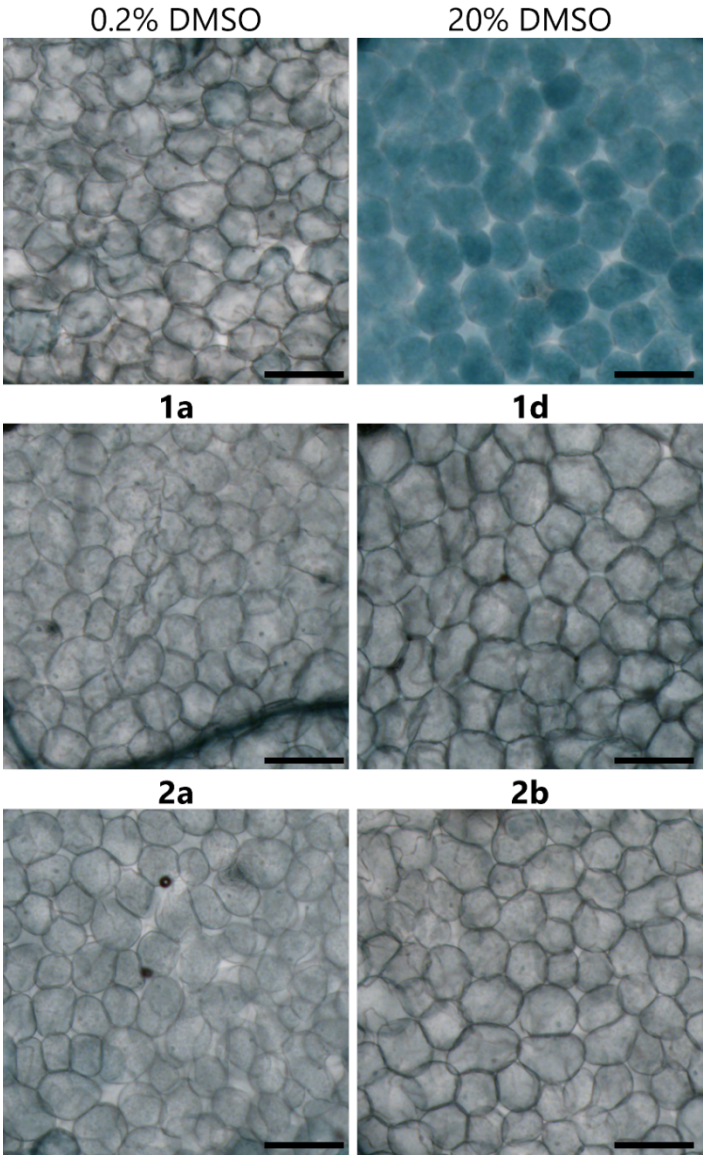


Fig. S18. Microscopy observations of dead cells in Arabidopsis leaf mesophyll cells. 0.2% DMSO (control)-infiltrated leaves and the probe **1a**, **1d**, **2a**, or **2b**-infiltrated leaves were incubated for 1 d and then stained with lactophenol trypan blue solution to detect dead cells. 20% DMSO treatment was performed as the condition that causes cell death largely. Scale Bars = 0.1 mm.

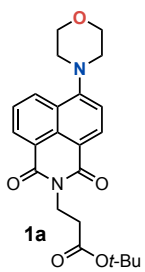
VI. Synthesis of naphtlaimide-based probes 1 and 2

Compound **S1**-**S4** were synthesized as described in a previous report.[11-13] Compounds **1a** and **2a** were synthesized as following the reported procedure.[14]

• General procedure for S_NAr reactions of S1

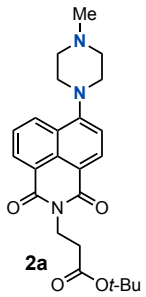
To a solution of **S1** in DMF was added a secondary amine (morpholine or *N*-methyl piperazine). The mixture was heated at 90 °C and allowed to stir for 18 hours. After cooling to room temperature, the solvent was removed. The residue was applied for was purified by flash chromatography on a Biotage One instrument (SNAP ultra-column or Sfär D column, 5–50% $CHCl_3$/AcOEt or 0–10% $CHCl_3$/MeOH over 12 column volumes) to provide a solid product. For imaging studies, this resulting solid was further purified by washing with 10% EtOAc/Hexane to afford **1a** and **2a**, respectively.

Synthesis of 1a:



The reaction was performed with **S1** (2.82 g, 6.98 mmol) and morpholine (1.20 mL, 15.0 mmol) in DMF (10 mL). After the purification, compound **1a** (2.52 g, 62%) was afforded as a yellow-orange solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.59 (dd, *J* = 1.1, 7.2 Hz, 1H), 8.52 (d, *J* = 8.0 Hz, 1H), 8.42 (dd, *J* = 1.1, 8.4 Hz, 1H), 7.70 (dd, *J*= 7.2, 8.4 Hz, 1H), 7.23 (d, *J*= 8.0 Hz, 1H), 4.43 (t, *J*= 7.6 Hz, 2H), 4.02-4.01 (m, 4H), 3.27-3.26 (m, 4H), 2.68 (t, *J*= 7.6 Hz, 2H), 1.42 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.7, 164.4, 163.9, 155.9, 132.7, 161.4, 130.3, 130.1, 126.3, 126.0, 123.4, 117.2, 115.31, 80.9, 67.1, 53.6, 36.3, 34.1, 28.2; HRMS(ESI): calculated for $[M + Na]^+$ requires *m/z* = 433.1739, found 433.1739.

Synthesis of 2a



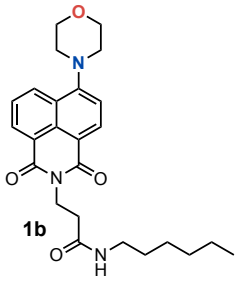
The reaction was performed with **S1** (350 mg, 0.866 mmol) and morpholine (1.43 mL, 13.0 mmol) in DMF (4.3 mL). After the purification, compound **2a** (297 mg, 81%) was afforded as a yellow-orange solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.58 (dd, *J* = 1.1, 7.2 Hz, 1H), 8.51 (d, *J* = 8.1 Hz, 1H), 8.41 (dd, *J* = 1.1, 8.4 Hz, 1H), 7.69 (dd, *J*= 7.2, 8.4 Hz, 1H), 7.22 (d, *J*= 8.1 Hz, 1H), 4.44 (t, *J*= 7.1 Hz, 2H), 3.31 (brs, 4H), 2.75 (brs, 4H), 2.68 (t, *J*= 7.1 Hz, 2H), 2.44 (2, 3H), 1.42 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.7, 164.5, 163.9, 156.2, 132.8, 131.3, 130.6, 130.1, 126.3, 125.8, 123.3, 116.7, 115.1, 80.8, 55.3, 53.2, 46.3, 36.2, 34.1, 28.2; HRMS(ESI): calculated for $[M + H]^+$ requires *m/z* = 424.2236, found 424.2231.

• General procedure for condensation reactions

To cleave the tertiary butyl group, **1a** and **2a** was treated with 4 M HCl/Dioxane (0.5-2 mL). After being stirred for 30-60 minutes at room temperature, HCl and dioxane were removed in vacuo. the resulting carboxylic acid was applied for the subsequent condensation reactions without any purification. To a solution of carboxylic acid in DMF/CH_2Cl_2 (0.01-0.05 M), EDC•HCl (1.10-1.25 eq.), HOBt (1.10-1.25 eq.), and triethyl amine (1.10-2.25 eq.) were added to form an activated ester. After being stirred for 15 minutes, the corresponding amine (1.0-1.2

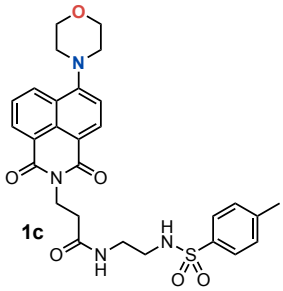
eq.) was added to the reaction solution. The mixture was allowed to stir at room temperature for 18 hours, after which the solution was diluted with CH_2Cl_2 and washed with H_2O and brine. The combined organic layer was washed with brine, dried over $Na2SO_4$, and concentrated in vacuo. The residue was purified by flash chromatography on a Biotage One instrument (SNAP ultra-column or Sfär D column, 5–50% $CHCl_3$/AcOEt or 0–10% $CHCl_3$/MeOH over 12 column volumes) to provide a solid product. For imaging studies, this resulting solid was further purified by washing with 10-30% EtOAc/Hexane to afford **1** or **2**.

Synthesis of 1b



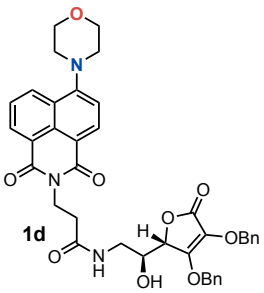
The reaction was performed with **1a** (41.0 mg, 0.100 mmol), hexylamine (12.1 mg, 0.120 mmol), EDC•HCl (22.2 mg, 0.120 mmol), HOBt (16.9 mg, 0.120 mmol), and triethyl amine (15.2 mg, 0.125 mmol) in DMF (1 mL) and CH_2Cl_2 (2 mL). After the purification, compound **1b** was afforded as a yellow solid (26.2 mg, 30%); ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.59 (dd, *J* = 1.1, 7.2 Hz, 1H), 8.53 (d, *J* = 8.0 Hz, 1H), 8.43 (dd, *J* = 1.1, 8.4 Hz, 1H), 7.71 (dd, *J*= 7.2, 8.4 Hz, 1H), 7.23 (d, *J*= 8.0 Hz, 1H), 5.07 (brs, 1H), 4.47 (t, *J*= 7.2 Hz, 2H), 4.03-4.01 (m, 4H), 3.28-3.26 (m, 4H), 3.23 (dt, *J*= 6.0, 7.6 Hz, 2H), 2.68 (t, *J*= 7.2 Hz, 2H), 1.46 (quint, *J*= 7.2 Hz, 2H), 1.28-1.22 (m, 6H), 0.866 (t, *J*= 7.0 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.3, 164.6, 164.2, 156.0, 132.9, 131.6, 130.5, 130.1, 126.3, 126.0, 123.2, 117.0, 115.1, 67.1, 53.6, 39.8, 36.9, 35.5, 31.6, 29.6, 26.8, 22.7, 14.2; HRMS(ESI): calculated for $[M + Na]^+$ requires *m/z* = 460.2212, found 460.2212.

Synthesis of 1c



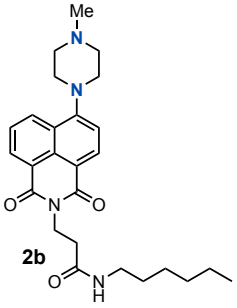
The reaction was performed with **1a** (17.4 mg, 42.3 µmol), **S2** (10.9 mg, 50.8 µmol), EDC•HCl (9.4 mg, 50.8 µmol), HOBt (7.2 mg, 50.8 µmol), and triethyl amine (6.4 mg, 0.635 mmol) in DMF (0.42 mL) and CH_2Cl_2 (84 mL). After the purification, compound **1c** was afforded as a yellow solid (19.0 mg, 82%); ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.48 (dd, *J* = 1.0, 8.4 Hz, 1H), 8.42 (dd, *J* = 1.0, 7.2 Hz, 1H), 8.36 (d, *J* = 8.0 Hz, 1H), 7.95 (t, *J* = 5.8 Hz, 1H), 7.79 (dd, *J*= 7.2, 8.4 Hz, 1H), 7.66 (d, *J* = 8.2 Hz, 2H), 7.54 (d, *J*= 6.0 Hz, 1H), 7.40 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 1H), 4.20 (t, *J*= 7.6 Hz, 2H), 3.92-3.90 (m, 4H), 3.22-3.21 (m, 4H), 3.03 (t, *J*= 5.8, 6.5 Hz, 2H), 2.72 (dt, *J*= 6.0, 6.5 Hz, 2H), 2.39 (s, 3H), 2.38 (t, *J*= 7.6 Hz, 2H, overlapped with neighboring signal); ^{13}C NMR (150 MHz, DMSO-d_6) δ 170.2, 163.4, 162.9, 155.4, 142.7, 137.5, 132.1, 130.6, 130.5, 129.7, 129.1, 126.5, 126.1, 125.3, 122.6, 115.9, 115.0, 79.2, 66.2, 53.0, 41.8, 38.5, 36.3, 33.8, 21.0; HRMS(ESI): calculated for $[M + Na]^+$ requires *m/z* = 573.1784, found 573.1783.

Synthesis of 1d



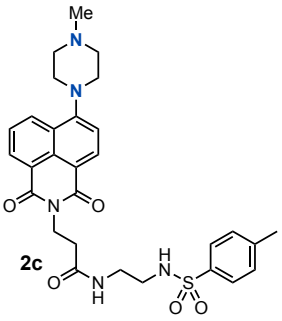
The reaction was performed with **1a** (143 mg, 0.348 mmol), **S3** (143 mg, 0.404 mmol), EDC•HCl (77.3 mg, 0.435 μmol), HOBt (58.8 mg, 0.435 mmol), and triethyl amine (52.8 mg, 0.522 mmol) in DMF (7 mL) and CH_2Cl_2 (14 mL). After the purification, compound **1d** was afforded as a yellow solid (61.8 mg, 26%); ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.51 (d, *J* = 7.8 Hz, 1H), 8.45 (d, *J* = 7.2 Hz, 1H), 8.38 (d, *J* = 7.8 Hz, 1H), 7.63 (dd, *J* = 7.2, 7.8 Hz, 1H), 7.39-7.32 (m, 8H), 7.23-7.22 (m, 2H), 7.18 (d, *J*= 7.8 Hz, 1H), 6.62 (brs, 1H), 5.22 (d, *J*= 11.8 Hz, 1H), 5.12 (d, *J*= 11.8 Hz, 1H), 5.07 (d, *J*= 12.0 Hz, 1H), 5.05 (d, *J*= 12.0 Hz, 1H), 4.58 (d, *J*= 2.0 Hz, 1H), 4.52 (ddd, *J*= 7.2, 7.2, 13.2 Hz, 1H), 4.43 (ddd, *J*= 6.3, 6.3, 13.2 Hz, 1H), 4.05-4.03 (m, 1H), 4.01-3.99 (m, 4H), 3.66-3.62 (m, 1H), 3.33-3.28 (m, 1H), 3.24-3.22 (m, 4H), 2.72 (ddd, *J*= 7.2, 7.2, 14.8 Hz, 1H), 2.66 (ddd, *J*= 6.3, 6.3, 14.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.1, 169.5, 164.7, 164.3, 157.2, 156.1, 136.2, 135.6, 133.0, 131.6, 130.6, 130.1, 129.3, 128.81, 128.79, 128.77, 127.9, 126.2, 126.0, 123.1, 121.4, 116.8, 115.2, 76.8, 74.0, 73.6, 68.3, 67.1, 53.6, 43.2, 37.0, 35.5; HRMS(ESI): calculated for $[M + Na]^+$ requires *m/z* = 714.2427, found 714.2430.

Synthesis of 2b



The reaction was performed with **2a** (99.4 mg, 0.235 mmol), hexylamine (28.6 mg, 0.282 mmol), EDC•HCl (52.2 mg, 0.294 μmol), HOBt (39.7 mg, 0.294 mmol), and triethyl amine (59.5 mg, 0.588 mmol) in DMF (2.5 mL) and CH_2Cl_2 (5 mL). After the purification, compound **2b** was afforded as a dark yellow solid (21.7 mg, 20%); ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.58 (dd, *J* = 1.0, 7.2 Hz, 1H), 8.51 (d, *J* = 8.1 Hz, 1H), 8.41 (dd, *J* = 1.0, 8.4 Hz, 1H), 7.69 (dd, *J*= 7.2, 8.4 Hz, 1H), 7.22 (d, *J*= 8.1 Hz, 1H), 6.09 (brs, 1H), 4.42 (t, *J*= 7.2 Hz, 2H), 3.31 (brs, 4H), 3.23 (dt, *J*= 5.8, 7.2 Hz, 2H), 2.75 (brs, 4H), 2.69 (t, *J*= 7.2 Hz, 2H), 2.44 (s, 3H), 1.46 (quint, *J*= 7.2 Hz, 2H), 1.28-1.22 (m, 6H), 0.849 (t, *J*= 6.8 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.4, 164.7, 164.2, 156.4, 133.0, 131.5, 130.7, 130.1, 126.3, 125.8, 123.1, 116.5, 115.1, 55.3, 53.2, 46.3, 39.8, 36.9, 35.5, 31.6, 29.6, 26.8, 22.7, 14.2; HRMS(ESI): calculated for $[M + H]^+$ requires *m/z* = 451.2709, found 451.2712.

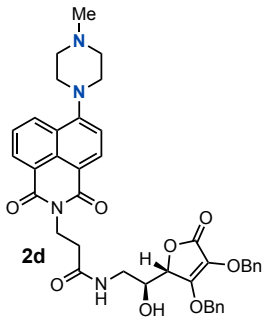
Synthesis of 2c



The reaction was performed with **2a** (99.4 mg, 0.235 mmol), **S2** (60.5 mg, 0.282 mmol), EDC•HCl (52.2 mg, 0.294 μmol), HOBt (39.7 mg, 0.294 mmol), and triethyl amine (59.5 mg, 0.588 mmol) in DMF (2.5 mL) and CH_2Cl_2 (5 mL). After the purification, compound **2c** was afforded as a yellow solid (18.3 mg, 14%); ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.48 (dd, *J* = 1.0, 8.4 Hz, 1H), 8.42 (dd, *J* = 1.0, 7.2 Hz, 1H), 8.36 (d, *J* = 8.0 Hz, 1H), 7.95 (t, *J* = 5.8 Hz, 1H), 7.79 (dd, *J*= 7.2, 8.4 Hz, 1H), 7.66 (d, *J* = 8.2 Hz, 2H), 7.54 (d, *J*= 6.0 Hz, 1H), 7.40 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 1H), 4.20 (t, *J*= 7.6 Hz, 2H), 3.92-3.90 (m, 4H), 3.22-3.21 (m, 4H), 3.03 (t, *J*= 5.8, 6.5 Hz, 2H), 2.72 (dt, *J*= 6.0, 6.5 Hz, 2H), 2.39 (s, 3H), 2.38 (t, *J*= 7.6 Hz, 2H, overlapped with neighboring signal); ^{13}C NMR (150 MHz, DMSO-d_6) δ 171.8, 164.8, 164.4, 156.5, 143.4, 137.4, 133.3, 131.7, 130.9, 130.0, 129.9,

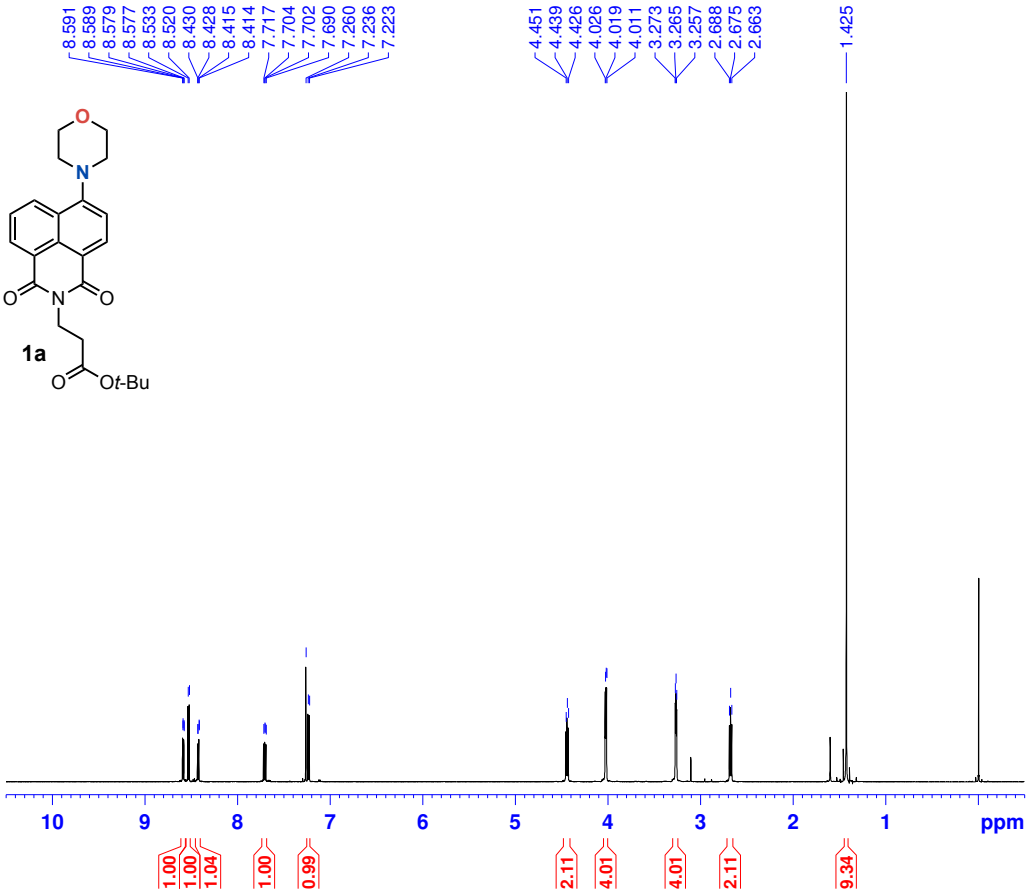
127.2, 126.1, 125.8, 122.8, 116.1, 115.2, 55.3, 53.1, 46.3, 42.7, 40.0, 37.0, 35.7, 21.7; $[M + H]^+$ requires *m/z* = 564.2281, found 564.2280.

Synthesis of 2d



The reaction was performed with **2a** (119 mg, 0.282 mmol), **S2** (120 mg, 0.339 mmol), EDC•HCl (62.7 mg, 0.352 µmol), HOBt (47.7 mg, 0.352 mmol), and triethyl amine (64.3 mg, 0.635 mmol) in DMF (2.8 mL) and CH_2Cl_2 (5.6 mL). After the purification, compound **2d** was afforded as a yellow solid (20.5 mg, 10%); ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.52 (dd, *J* = 1.1, 7.3 Hz, 1H), 8.44 (d, *J* = 8.1 Hz, 1H), 8.38 (d, *J* = 1.1, 8.4 Hz, 1H), 7.62 (dd, *J* = 7.3, 8.4 Hz, 1H), 7.39-7.33 (m, 8H), 7.23-7.22 (m, 2H), 7.13 (d, *J*= 8.1 Hz, 1H), 6.54 (t, *J*= 3.6 Hz, 1H), 5.22 (d, *J*= 11.8 Hz, 1H), 5.12 (d, *J*= 11.8 Hz, 1H), 5.09 (d, *J*= 11.3 Hz, 1H), 5.06 (d, *J*= 11.3 Hz, 1H), 4.59 (d, *J*=4.4 Hz, 1H), 4.53 (ddd, *J*= 6.4, 7.9, 13.2 Hz, 1H), 4.44 (ddd, *J*= 6.3, 6.3, 13.2 Hz, 1H), 4.05-4.03 (m, 1H), 3.66-3.62 (m, 1H), 3.34-3.29 (m, 4H), 2.76-2.71 (m, 5H), 2.67 (ddd, *J*= 6.3, 6.3, 15.0 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.1, 169.5, 164., 164.4, 157.2, 156.4, 136.2, 135.6, 133.1, 131.5, 130.8, 130.1, 129.3, 128.8, 128.8, 127.9, 126.2, 125.8, 123.0, 121.3, 116.3, 115.2, 76.8, 74.0, 73.6, 68.3, 55.2, 53.0, 46.2, 43.2, 37.0, 35.5; HRMS(ESI): calculated for $[M + H]^+$ requires *m/z* = 705.2924, found 705.2923.

1a: ^{1}H NMR (600 MHz, $CDCl_3$)

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PROCNO                1
Date_          20210609
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TD                65536
SOLVENT           CDC13
NS                    4
DS                    2
SWH           12376.237 Hz
FIDRES         0.188846 Hz
AQ             2.6477449 sec
RG                228.1
DW               40.400 usec
DE                6.50 usec
TE                298.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                8.00 usec
PL1              -1.00 dB
PL1W        31.62277603 W
SFO1        600.0337054 MHz
SI                32768
SF          600.0300139 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
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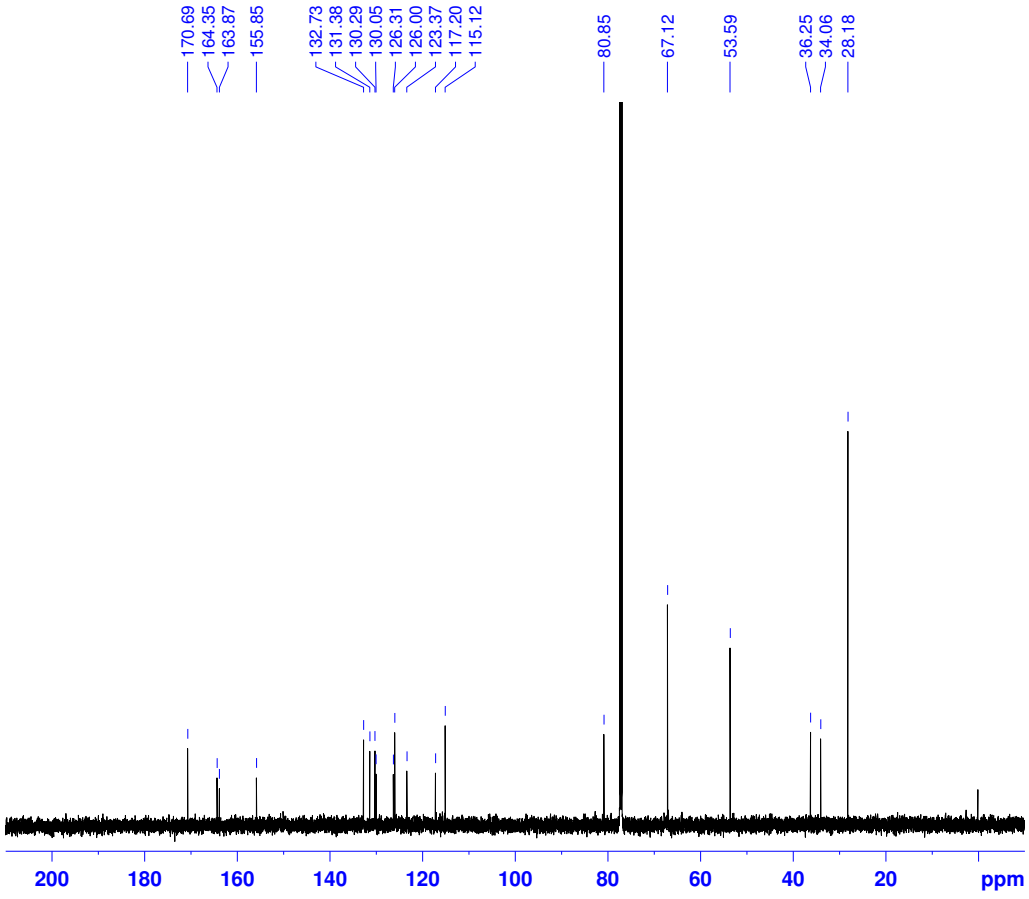
1a: ^{13}C NMR (150 MHz, $CDCl_3$)

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PROCNO                1
Date_          20210609
Time              11.17
INSTRUM           spect
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PULPROG         zgpg30
TD                65536
SOLVENT           CDC13
NS                  662
DS                    4
SWH           35971.223 Hz
FIDRES         0.548877 Hz
AQ             0.9110143 sec
RG              20642.5
DW               13.900 usec
DE                6.50 usec
TE                298.0 K
D1            4.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1               15.00 usec
PL1              -3.20 dB
PL1W       262.40374756 W
SFO1        150.8927508 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2               1.00 dB
PL12             19.00 dB
PL13             19.00 dB
PL2W       19.95262337 W
PL12W       0.31622776 W
PL13W       0.31622776 W
SFO2        600.0324001 MHz
SI                32768
SF          150.8776431 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
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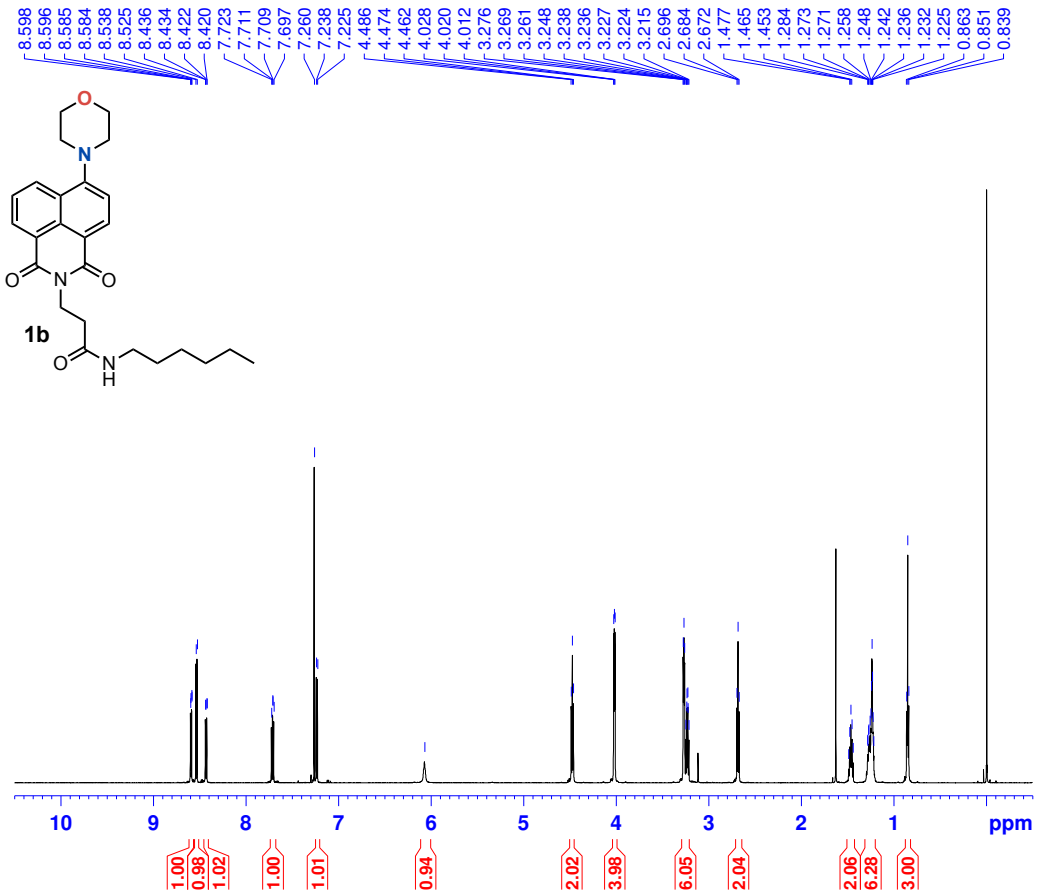
1b: ^{1}H NMR (600 MHz, $CDCl_3$)

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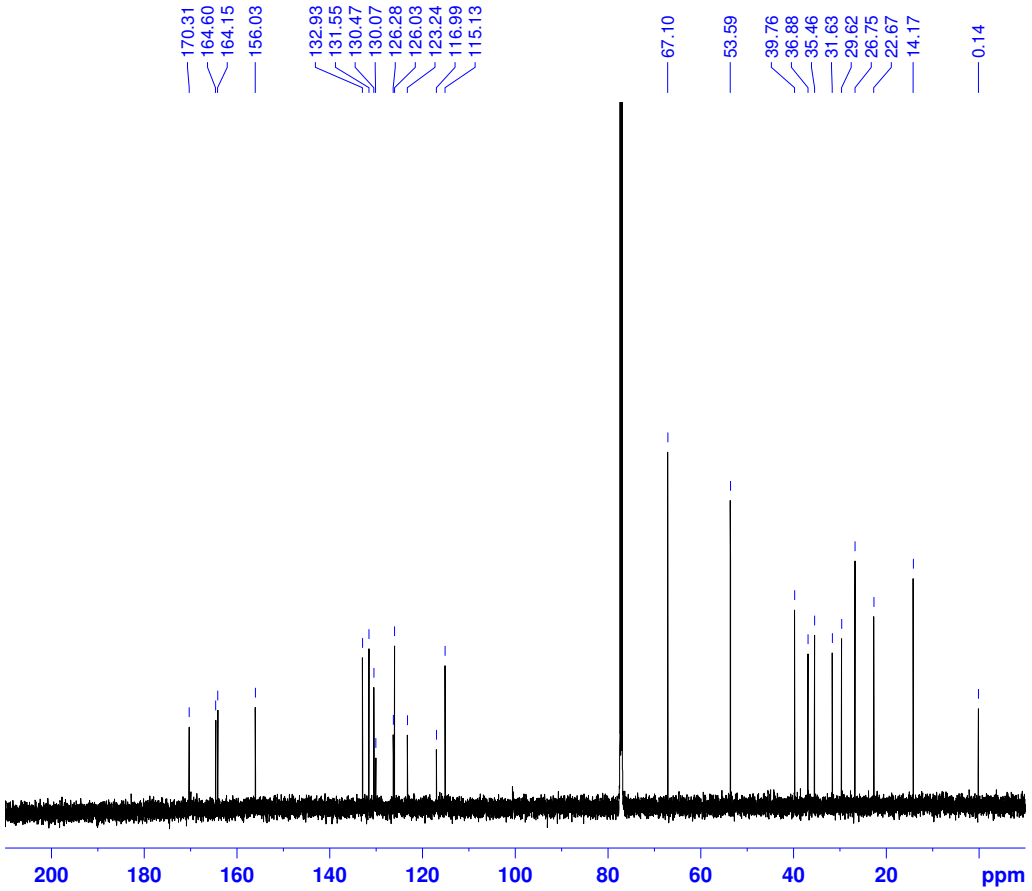
EXPNO                   1
PROCNO                  1
Date_            20210610
Time                11.10
INSTRUM             spect
PROBHD    5 mm TXI 1H/2H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      4
DS                      2
SWH             12376.237 Hz
FIDRES              0.188846 Hz
AQ               2.6477449 sec
RG                    256
DW                 40.400 usec
DE                  6.50 usec
TE                  298.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  8.00 usec
PL1                -1.00 dB
PL1W          31.62277603 W
SFO1          600.0337054 MHz
SI                  32768
SF            600.0300138 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                  1.00
```

1b: ^{13}C NMR (150 MHz, $CDCl_3$)

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PROCNO                  1
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INSTRUM             spect
PROBHD    5 mm TXI 1H/2H
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   3224
DS                      4
SWH            35971.223 Hz
FIDRES             0.548877 Hz
AQ              0.9110143 sec
RG                  20642.5
DW                 13.900 usec
DE                  6.50 usec
TE                  298.0 K
D1              4.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                 15.00 usec
PL1                -3.20 dB
PL1W          262.40374756 W
SFO1           150.8927508 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               19.00 dB
PL13               19.00 dB
PL2W          19.95262337 W
PL12W          0.31622776 W
PL13W          0.31622776 W
SFO2          600.0324001 MHz
SI                  32768
SF            150.8776430 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                  1.40
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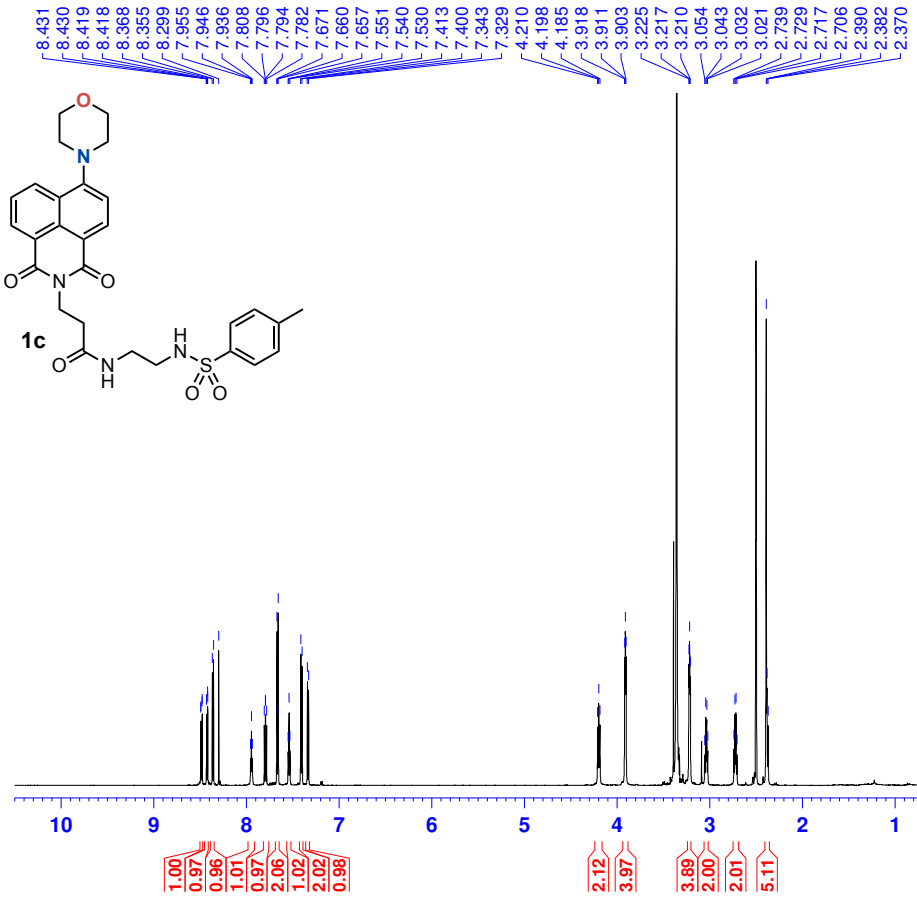
1c: ^{1}H NMR (600 MHz, DMSO-d_6)

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PROCNO                   1
Date_             20210217
Time                 10.32
INSTRUM              spect
PROBHD    5 mm TXI 1H/2H
PULPROG               zg30
TD                   65536
SOLVENT               DMSO
NS                       8
DS                       2
SWH              12376.237 Hz
FIDRES           0.188846 Hz
AQ               2.6477449 sec
RG                    90.5
DW                  40.400 usec
DE                   6.50 usec
TE                  300.5 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   8.00 usec
PL1                 -1.00 dB
PL1W          31.62277603 W
SFO1          600.0337054 MHz
SI                   32768
SF            600.0300043 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00
```
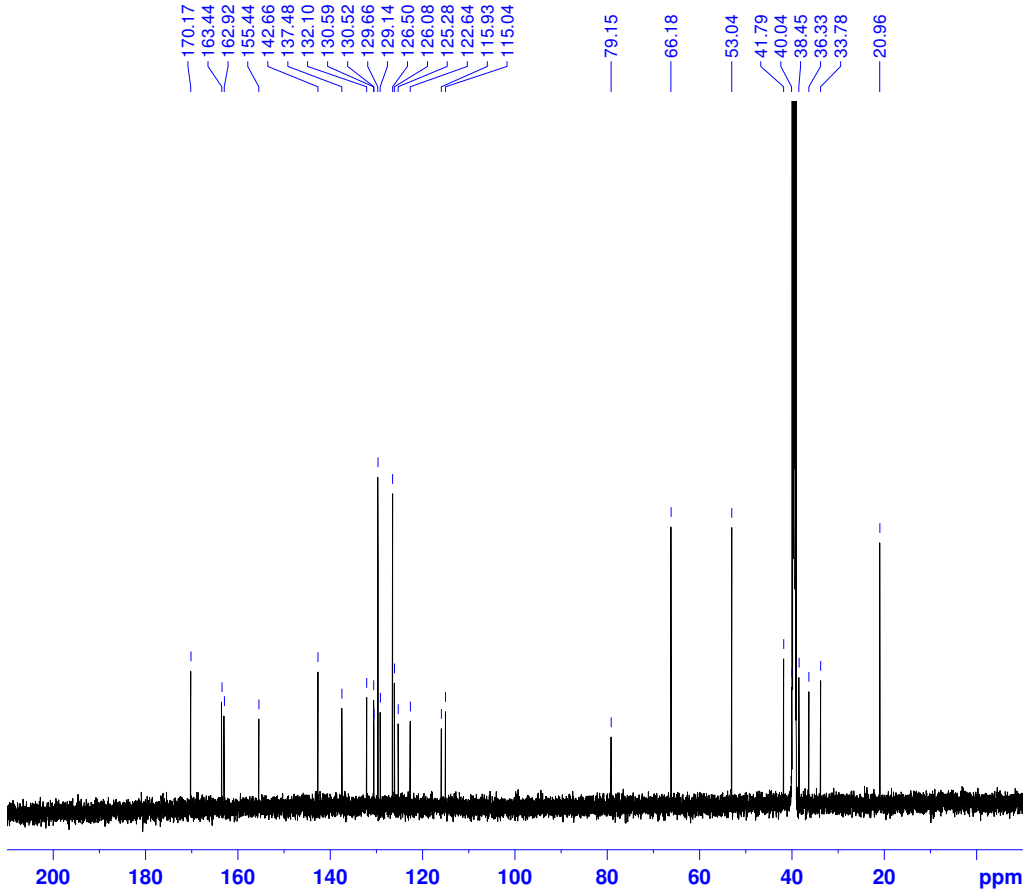

1c: ^{13}C NMR (150 MHz, DMSO-d_6)




```
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PROCNO                   1
Date_             20210217
Time                 15.06
INSTRUM              spect
PROBHD    5 mm TXI 1H/2H
PULPROG           zgpg30
TD                   65536
SOLVENT               DMSO
NS                   4198
DS                       4
SWH              35971.223 Hz
FIDRES           0.548877 Hz
AQ               0.9110143 sec
RG                 23170.5
DW                  13.900 usec
DE                   6.50 usec
TE                  300.7 K
D1             6.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                  15.00 usec
PL1                 -3.20 dB
PL1W         262.40374756 W
SFO1          150.8927508 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                  1.00 dB
PL12                19.00 dB
PL13                19.00 dB
PL2W          19.95262337 W
PL12W          0.31622776 W
PL13W          0.31622776 W
SFO2          600.0324001 MHz
SI                   32768
SF            150.8777352 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                    1.40
```
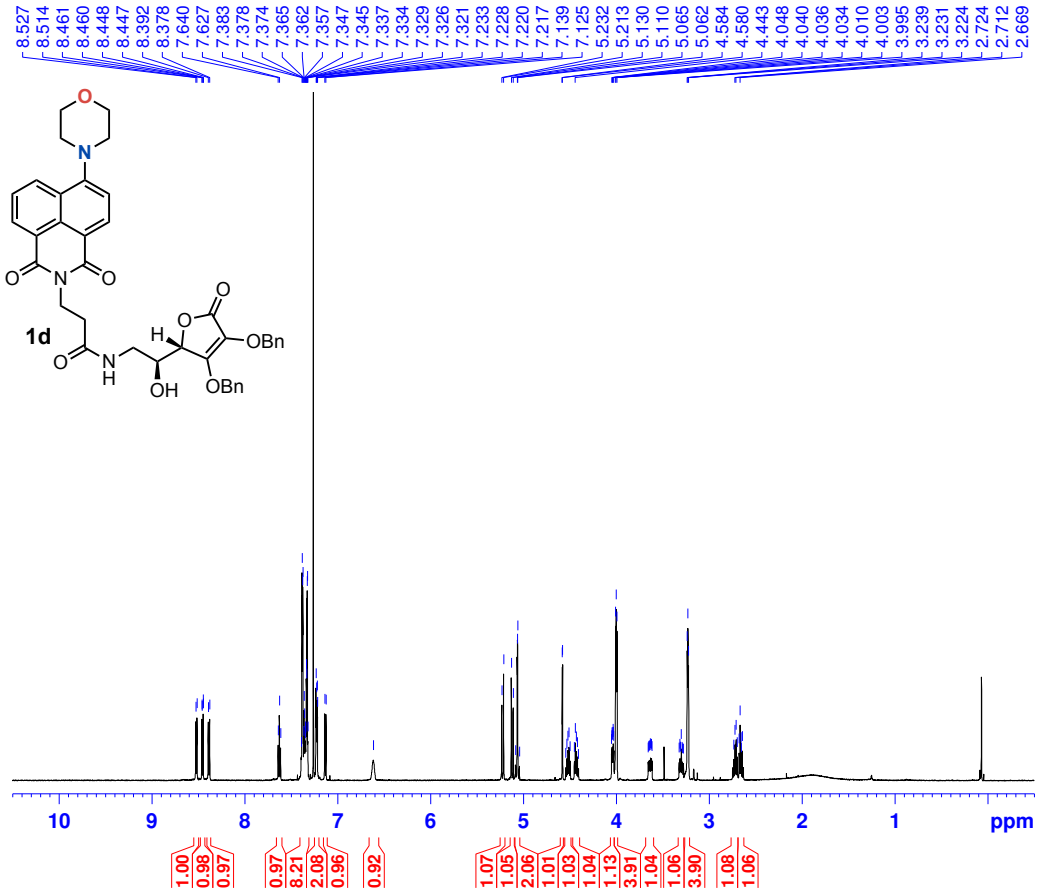
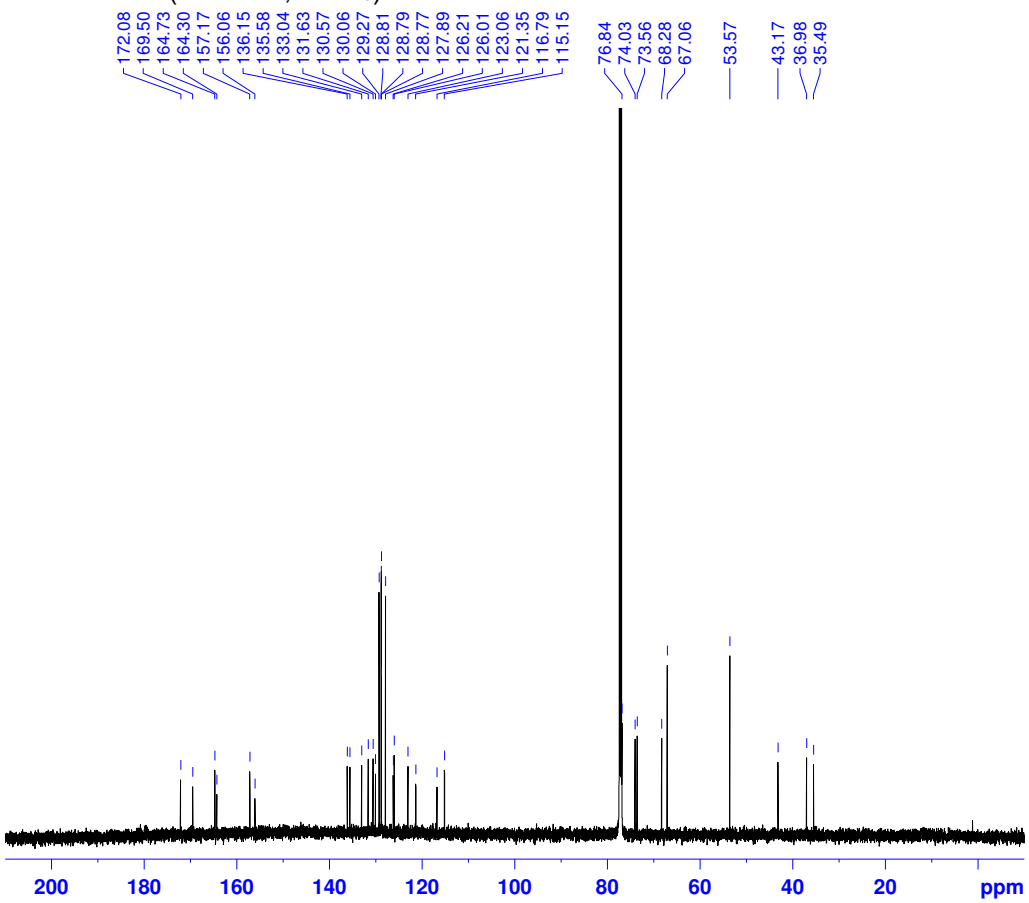

1d: ^{1}H NMR (600 MHz, $CDCl_3$)



BRUKER

```
NAME              sk-1-363
EXPNO                    2
PROCNO                   1
Date_             20190715
Time                 10.54
INSTRUM              spect
PROBHD   5 mm TXI 1H/2H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                       4
DS                       2
SWH              12376.237 Hz
FIDRES           0.188846 Hz
AQ               2.6477449 sec
RG                     362
DW                  40.400 usec
DE                   6.50 usec
TE                  298.0 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   8.00 usec
PL1                 -1.00 dB
PL1W           31.62277603 W
SFO1           600.0337054 MHz
SI                   32768
SF             600.0300142 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00
```

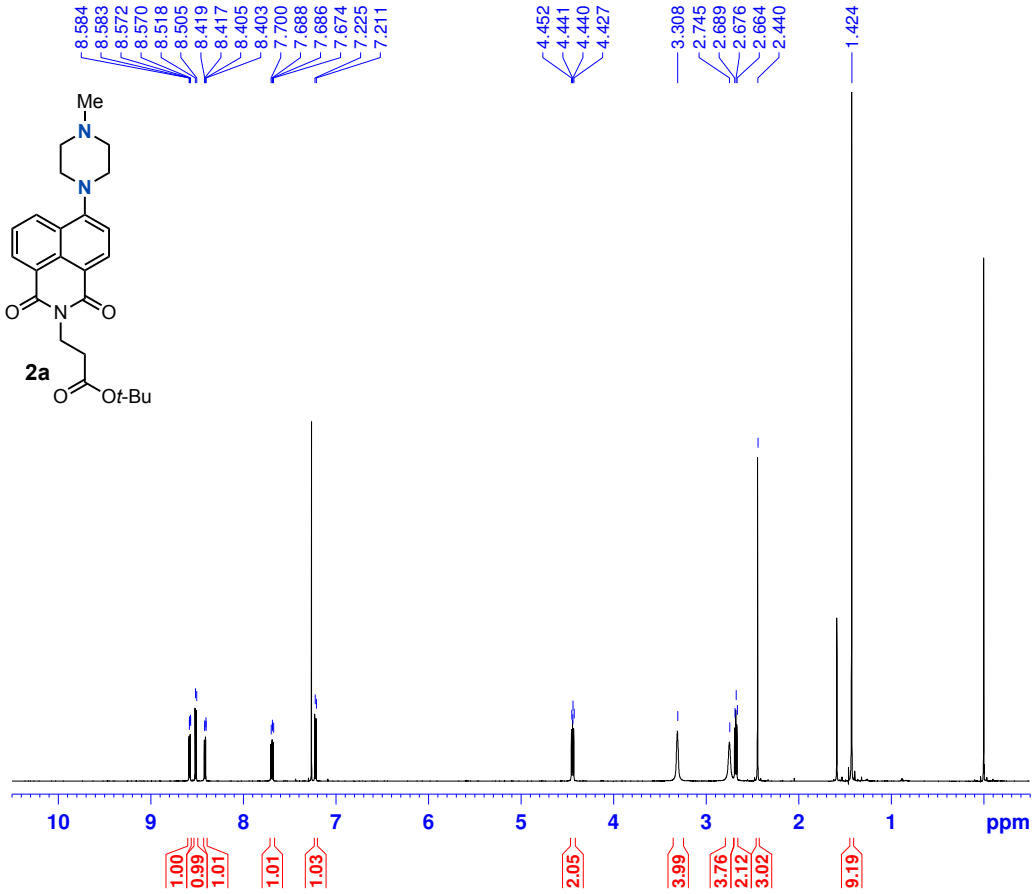
1d: ^{13}C NMR (150 MHz, $CDCl_3$)



BRUKER

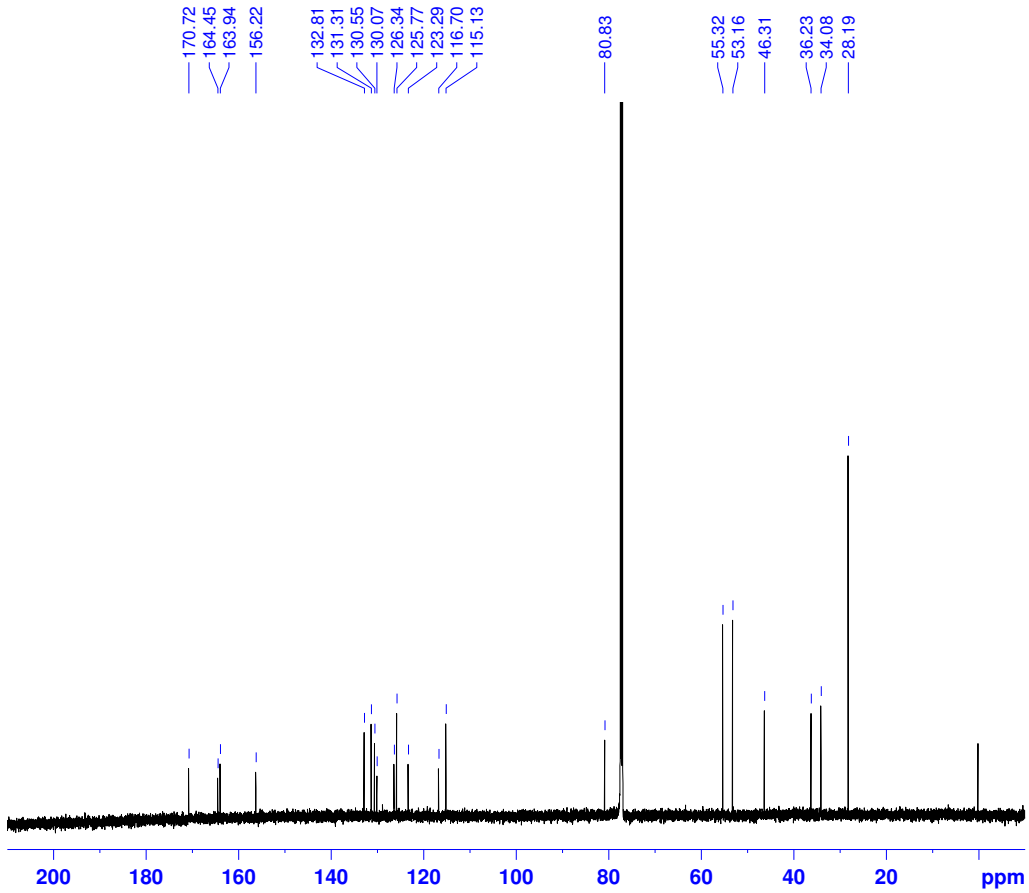
```
NAME          sk-1-363 13C
EXPNO                    2
PROCNO                   1
Date_             20190716
Time                 11.42
INSTRUM              spect
PROBHD   5 mm TXI 1H/2H
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   12000
DS                       4
SWH              35971.223 Hz
FIDRES           0.548877 Hz
AQ               0.9110143 sec
RG                 18390.4
DW                  13.900 usec
DE                   6.50 usec
TE                  298.0 K
D1               4.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                  15.00 usec
PL1                 -3.20 dB
PL1W          262.40374756 W
SFO1           150.8927508 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 19.00 dB
PL13                 19.00 dB
PL2W          19.95262337 W
PL12W          0.31622776 W
PL13W          0.31622776 W
SFO2           600.0324001 MHz
SI                   32768
SF             150.8776429 MHz
WDW                     EM
SSB                      0
LB                   1.00 Hz
GB                       0
PC                    1.40
```

2a: ^{1}H NMR (600 MHz, $CDCl_3$)



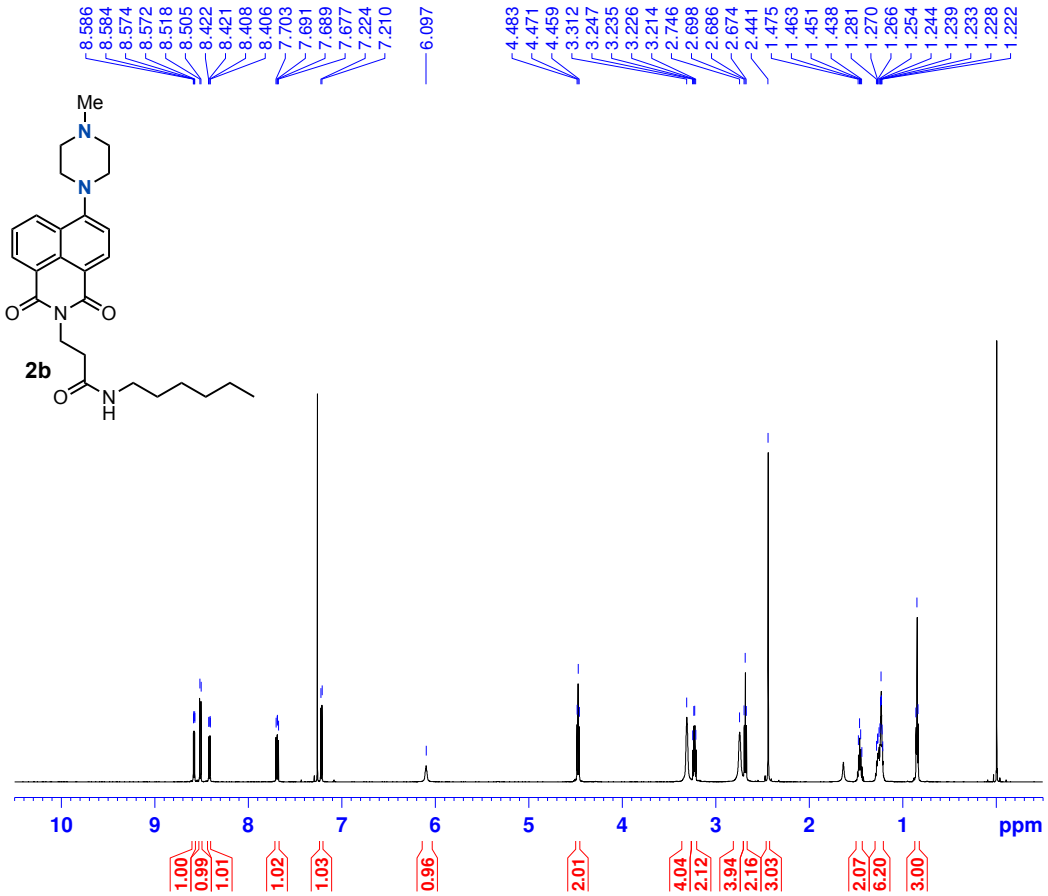

```
NAME            sk-1-1066
EXPNO                   2
PROCNO                  1
Date_            20210108
Time                20.37
INSTRUM             spect
PROBHD    5 mm TXI 1H/2H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      4
DS                      2
SWH             12376.237 Hz
FIDRES          0.188846 Hz
AQ              2.6477449 sec
RG                    362
DW                40.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.00 usec
PL1                 -1.00 dB
PL1W          31.62277603 W
SFO1          600.0337054 MHz
SI                  32768
SF            600.0300138 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

2a: ^{13}C NMR (150 MHz, $CDCl_3$)



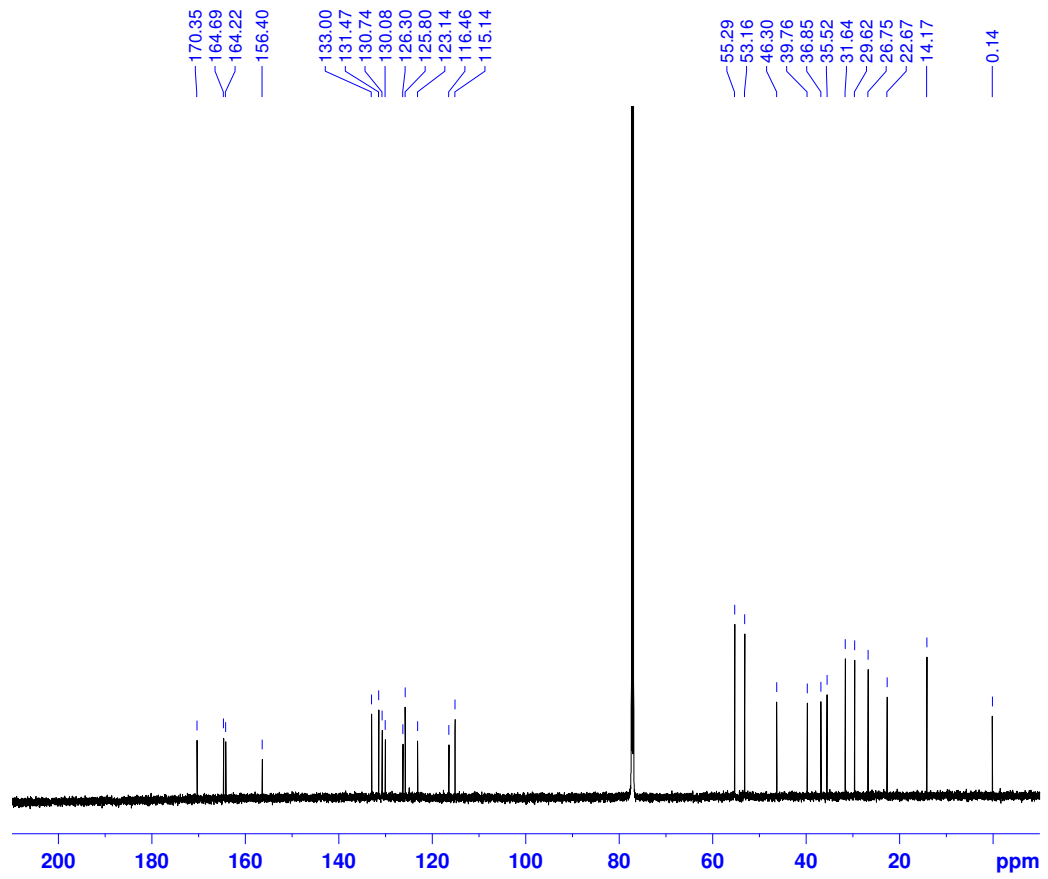

```
NAME            sk-1-1066 13C
EXPNO                   1
PROCNO                  1
Date_            20210109
Time                10.28
INSTRUM             spect
PROBHD    5 mm TXI 1H/2H
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  10000
DS                      4
SWH             35971.223 Hz
FIDRES          0.548877 Hz
AQ              0.9110143 sec
RG                18390.4
DW                13.900 usec
DE                 6.50 usec
TE                298.0 K
D1             4.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  15.00 usec
PL1                 -3.20 dB
PL1W         262.40374756 W
SFO1          150.8927508 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  1.00 dB
PL12                19.00 dB
PL13                19.00 dB
PL2W         19.95262337 W
PL12W         0.31622776 W
PL13W         0.31622776 W
SFO2          600.0324001 MHz
SI                  32768
SF            150.8776420 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

2b: ^{1}H NMR (600 MHz, $CDCl_3$)



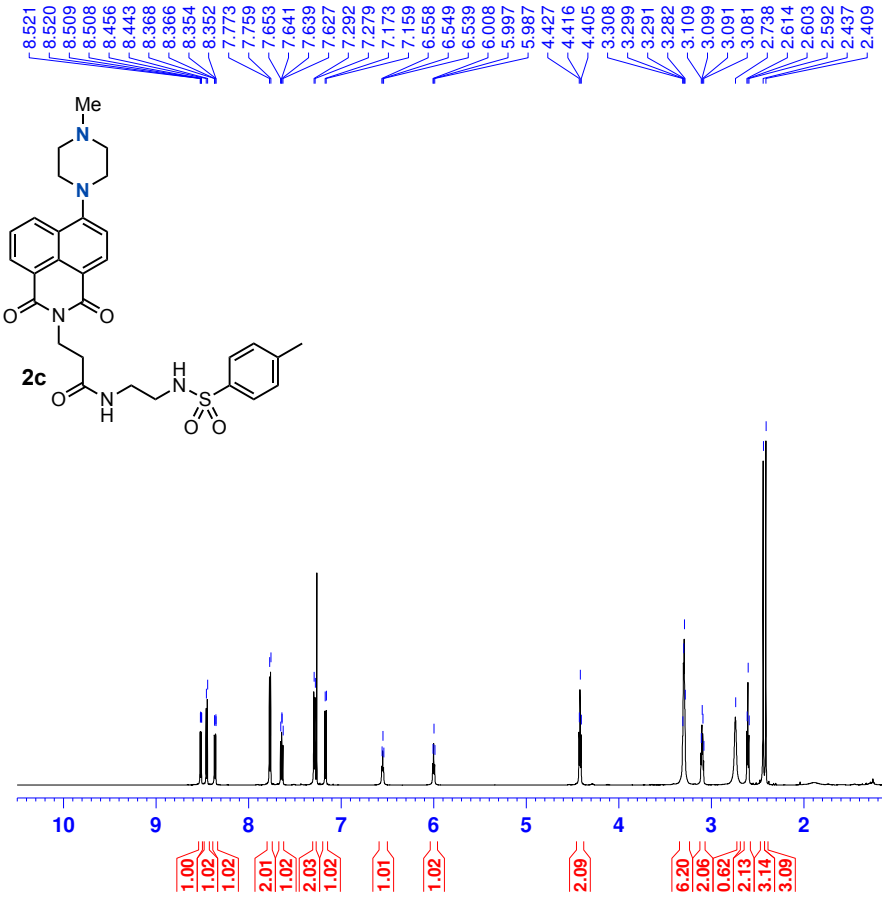

```
NAME      sk-1-1103 20210610
EXPNO                  1
PROCNO                 1
Date_           20210610
Time               15.51
INSTRUM            spect
PROBHD   5 mm TXI 1H/2H
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           12376.237 Hz
FIDRES         0.188846 Hz
AQ             2.6477449 sec
RG                  362
DW                40.400 usec
DE                 6.50 usec
TE                298.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.00 usec
PL1               -1.00 dB
PL1W        31.62277603 W
SFO1        600.0337054 MHz
SI                32768
SF          600.0300141 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

2b: ^{13}C NMR (150 MHz, $CDCl_3$)



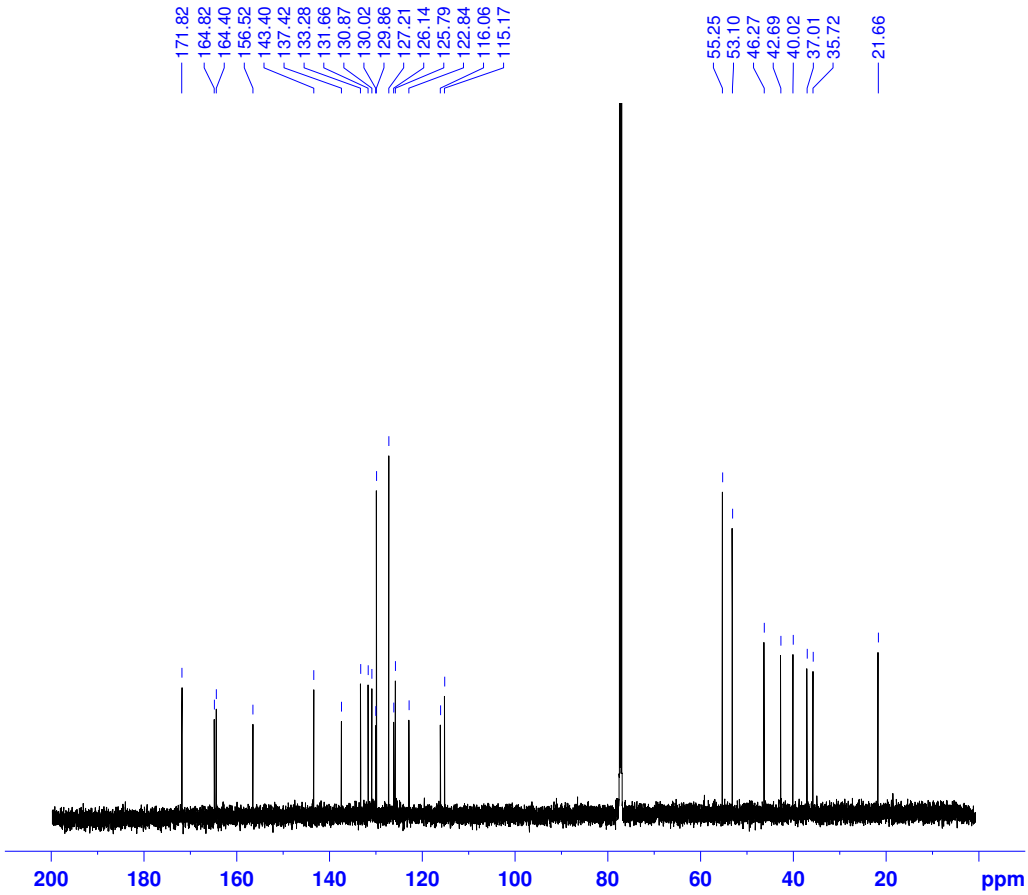

```
NAME      sk-1-1103 20210610 13C
EXPNO                  1
PROCNO                 1
Date_           20210611
Time               10.26
INSTRUM            spect
PROBHD   5 mm TXI 1H/2H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                13384
DS                    4
SWH           35971.223 Hz
FIDRES         0.548877 Hz
AQ             0.9110143 sec
RG                23170.5
DW                13.900 usec
DE                 6.50 usec
TE                298.0 K
D1            4.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PL1               -3.20 dB
PL1W       262.40374756 W
SFO1        150.8927508 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                1.00 dB
PL12              19.00 dB
PL13              19.00 dB
PL2W       19.95262337 W
PL12W       0.31622776 W
PL13W       0.31622776 W
SFO2        600.0324001 MHz
SI                32768
SF          150.8776428 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

2c: ^{1}H NMR (600 MHz, $CDCl_3$)



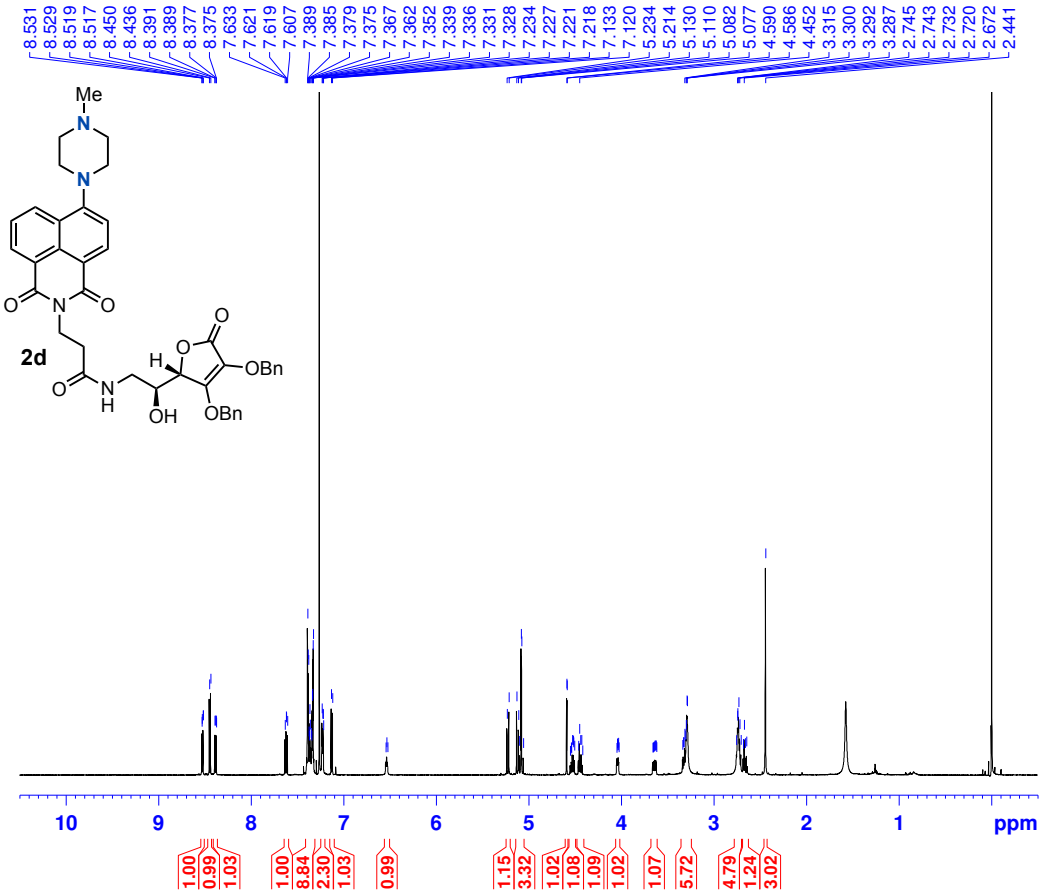

```
NAME            sk-1-1104
EXPNO                   1
PROCNO                  1
Date_            20210203
Time                18.01
INSTRUM             spect
PROBHD   5 mm TXI 1H/2H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      4
DS                      2
SWH            12376.237 Hz
FIDRES          0.188846 Hz
AQ              2.6477449 sec
RG                    128
DW                 40.400 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.00 usec
PL1                 -1.00 dB
PL1W         31.62277603 W
SFO1         600.0337054 MHz
SI                  32768
SF           600.0300138 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

2c: ^{13}C NMR (150 MHz, $CDCl_3$)



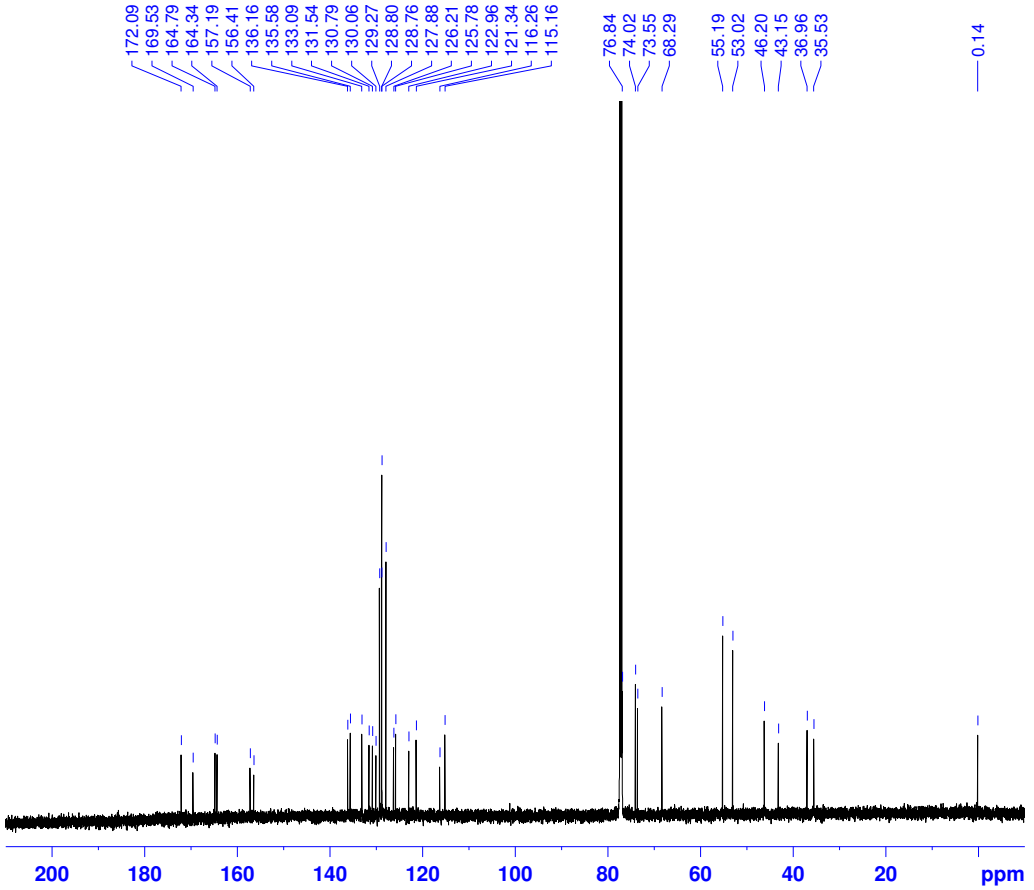

```
NAME       sk-1-1104 13C
EXPNO                 1
PROCNO                1
Date_          20210203
Time              18.12
INSTRUM           spect
PROBHD   5 mm TXI 1H/2H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1410
DS                    4
SWH           30030.029 Hz
FIDRES            0.458222 Hz
AQ                1.0912410 sec
RG                23170.5
DW                  16.650 usec
DE                   6.50 usec
TE                 298.0 K
D1            4.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 15.00 usec
PL1                -3.20 dB
PL1W       262.40374756 W
SFO1        150.8927508 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               19.00 dB
PL13               19.00 dB
PL2W        19.95262337 W
PL12W        0.31622776 W
PL13W        0.31622776 W
SFO2        600.0324001 MHz
SI                32768
SF          150.8776443 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

2d: ^{1}H NMR (600 MHz, $CDCl_3$)




```
NAME      sk-1-1069 bottom
EXPNO                1
PROCNO               1
Date_         20210111
Time             19.25
INSTRUM          spect
PROBHD   5 mm TXI 1H/2H
PULPROG          zg30
TD               65536
SOLVENT          CDC13
NS                  16
DS                   2
SWH          12376.237 Hz
FIDRES       0.188846 Hz
AQ           2.6477449 sec
RG               574.7
DW              40.400 usec
DE               6.50 usec
TE              298.0 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                8.00 usec
PL1              -1.00 dB
PL1W       31.62277603 W
SFO1       600.0337054 MHz
SI               32768
SF         600.0300138 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

2d: ^{13}C NMR (150 MHz, $CDCl_3$)




```
NAME      sk-1-1214 PTLC 13C
EXPNO                1
PROCNO               1
Date_         20210623
Time             18.09
INSTRUM          spect
PROBHD   5 mm TXI 1H/2H
PULPROG        zgpg30
TD               65536
SOLVENT          CDC13
NS               11506
DS                   4
SWH          35971.223 Hz
FIDRES       0.548877 Hz
AQ           0.9110143 sec
RG             20642.5
DW              13.900 usec
DE               6.50 usec
TE              298.0 K
D1          4.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                15.00 usec
PL1              -3.20 dB
PL1W       262.40374756 W
SFO1        150.8927508 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            80.00 usec
PL2               1.00 dB
PL12             19.00 dB
PL13             19.00 dB
PL2W       19.95262337 W
PL12W       0.31622776 W
PL13W       0.31622776 W
SFO2       600.0324001 MHz
SI               32768
SF         150.8776435 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

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