A ratiometric fluorescent probe with dual red/near-infrared emissions for monitoring lysosomal pH fluctuations

Huiying Mu, Ryo Tanaka, Ryo Kubota, Koji Miki, and Kouichi Ohe*

Department of Energy and Hydrocarbon Chemistry, Graduate School of Engineering, Kyoto University, Katsura, Nishikyo-ku, Kyoto 615-8510, Japan.

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1. Experimental Section

1.1 Materials and Methods

Tetrahydrofuran (THF), ethanol (EtOH), diethyl ether (Et_2O), methanol (MeOH), Triton X-100, potassium carbonate (K_2CO_3), and ammonium chloride (NH_4Cl) were purchased form nacalai tesque (Japan). Dichloromethane (CH_2Cl_2) and dimethyl sulfoxide (DMSO) were purchased from Tokyo Chemical Industry (TCI) Co., Ltd. (Japan). Anhydrous sodium sulfate, cinnamaldehyde, chloroquine, and Dulbecco's modified Eagle's medium (DMEM) with/without phenol red were purchased from FUJIFILM Wako Pure Chemicals Inc. (Japan). Solvents were distillated over CaH_2 under N_2 atmosphere. Silica gel (SiO_2, 230–400 mesh) for column chromatography was purchased from Silicycle (Canada). Buffered aqueous solutions (pH 2.0–2.2) were prepared by dissolving hydrochloric acid (HCl) and potassium chloride (KCl) in water (MilliQ). Buffered aqueous solutions (pH 3.1–6.9) were prepared by dissolving citric acid and sodium dihydrogenphosphate (NaH_2PO_4) in water (MilliQ). Buffered aqueous solutions (pH 7.2–8.7) were prepared by dissolving NaH_2PO_4 and disodium hydrogenphosphate (Na_2HPO_4) in water (MilliQ). Buffered aqueous solutions (pH 9.0–11.0) were prepared by dissolving sodium bicarbonate ($NaHCO_3$) and sodium carbonate (Na_2CO_3) in water (MilliQ). All buffered aqueous solutions were stored in refrigerator and used within one week. Saline solution for diluted dye in intraperitoneal (i.p.) injection was purchased from Otsuka Pharmaceutical Factory, Inc. Japan. Isoflurane inhalation solution used as anesthesia agent was purchased from Pfizer Inc. (Japan). LysoTracker™ Green DND-26, ER-Tracker™ Green (BODIPY™ FL glibenclamide), MitoTracker™ Green FM and CellLight™ Late Endosome-GFP were purchased from Thermo Fisher Scientific K. K. (Japan). were purchased from Thermo Fisher Scientific K. K. (Japan)

UV-vis absorption spectra of dyes were measured by UV-vis-NIR spectrophotometer (UH5300, Hitachi High-Technologies Co., Japan). The sample solutions (5.0×10^{-6} M) were prepared by mixing a solution of dyes in DMSO (10 μL, 1.5×10^{-3} M) with deoxygenated buffered solution (3 mL) with/without Triton X-100 (2.0×10^{-3} M). Emission spectra of dyes were measured by fluorescence spectrophotometer (RF-6000, Shimadzu Co., Japan). The sample solutions were prepared in the same manner shown in the method of UV-vis absorption measurement. The stability of fluorescence of probe was measured after 20 min incubation.

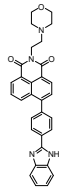
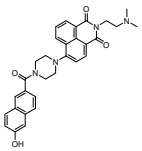
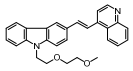
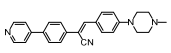
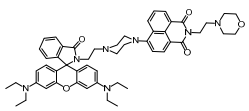
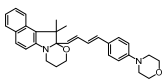
High resolution mass spectra were measured by EXACTICVE (ESI, Thermo Fisher Scientific Inc., USA). NMR spectra were recorded on JEOL EX-400 spectrometer (400 MHz for ^{1}H NMR

and 100 MHz for ^{13}C NMR).

Confocal microscopy was performed on LSM 710 (Carl Zeiss, Germany). The fluorescent intensities from confocal images were analyzed by ImageJ (Fiji) win64 software. Fluorescence images of dyes in mice model were measured by IVIS Imaging System 200 Series (PerkinElmer, Inc., USA.). Fluorescence intensities *in vivo* were quantified by Living Image 2.50-Igor Pro 4.09 software (PerkinElmer, Inc., USA.). Female BALB/cCrSlc mice, 6 weeks of age were purchased from SHIMIZU Laboratory Supplies Co., Ltd., Japan. All of our animal experiments were approved by the Animal Research Committee of Kyoto University and carried out with its guidelines.

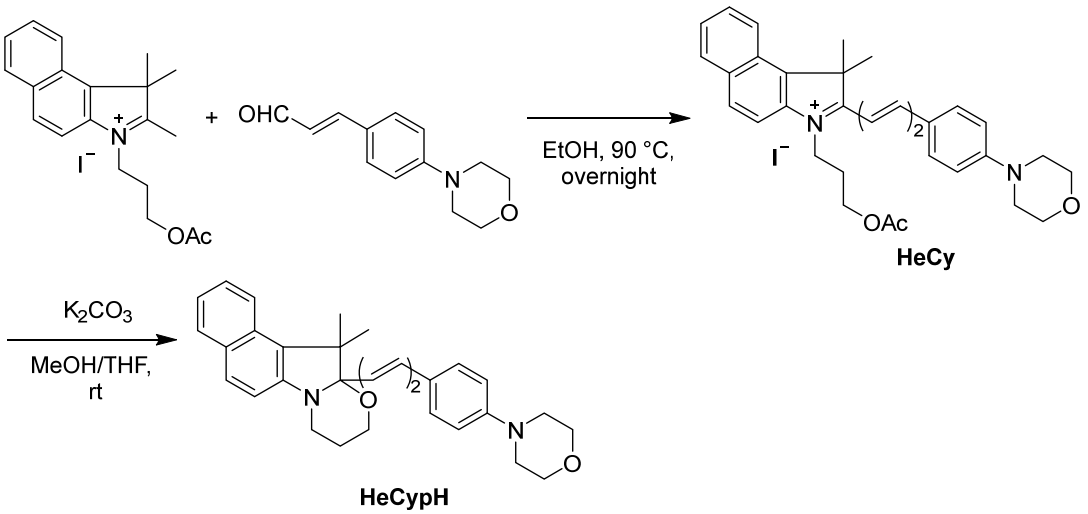
1.2 Comparison of the reported ratiometric probes and HeCypH

Table S1. Comparison of properties of recently reported ratiometric probes for lysosomal pH sensing in vivo.

probe	λ_{ex}/nm	λ_{em}/nm	pK_a	cellular pH measurements	in vivo applications	ref
	380	460/510	5.70	no	nematodes	1
	385	425/540	5.02	no	zebrafish	2
	415	530/637	4.60	no	mouse	3
	365	503/615	4.75	yes	medaka larva	4
	400	510/595	n.d.	no	vertebrate zebrafish	5
	550	615/722	6.55	yes	mouse	this work

1.3 Synthesis of hemicyanine dyes

HeCypH was prepared by following the procedure shown in Scheme S1. 3-Acetoxypropyl-substituted benz[e]indole was prepared according to our previous reported works.[6]



Scheme S1. Synthesis of probe **HeCypH**.

1.3.1 Preparation of protected hemicyanine derivative HeCy

3-Acetoxypropyl-substituted benz[e]indole (42 mg, 0.10 mmol), 3-(4-morpholinophenyl)acrylaldehyde (23 mg, 0.11 mmol), and EtOH (1.0 mL) were added in 25 mL Schlenk tube in N_2 atmosphere. After stirring at 90 °C for overnight, the solvent was removed under reduced pressure. The reaction mixture was dissolved in CH_2Cl_2 (1.5 mL), then cold Et_2O (20 mL) was poured into the mixture to precipitate solid product. The solid was collected by centrifuge, washed with cooled Et_2O and dried under vacuum to give **HeCy** as navy blue solid.

HeCy: a navy blue solid (92%); ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ 1.94 (s, 3H), 2.03 (s, 6H), 2.41–2.43 (m, 2H), 3.33 (t, J = 4.6 Hz, 4H), 3.84 (t, J = 4.6 Hz, 4H), 4.33 (t, J = 6.0 Hz, 2H), 4.95 (t, J = 6.9 Hz, 2H), 6.86 (d, J = 8.7 Hz, 2H), 7.50 (d, J = 14.6 Hz, 1H), 7.59–7.64 (m, 2H), 7.69–7.73 (m, 3H), 7.95 (d, J = 14.6 Hz, 1H), 8.02–8.08 (m, 3H), 8.16–8.24 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ 20.9, 27.2, 27.9, 44.5, 47.1, 52.8, 61.3, 66.4, 111.4, 112.8, 114.1, 122.4, 126.1, 126.3, 126.9, 127.5, 128.5, 130.3, 131.5, 132.1, 133.1, 136.6, 137.1, 138.4, 151.8, 153.4, 156.1, 170.6, 180.6. HRMS (ESI) calcd for $C_{33}H_{37}N_2O_3^+$ ([M]$^+$): 509.2799. Found: 509.2805.

1.3.2 Preparation of probe HeCypH

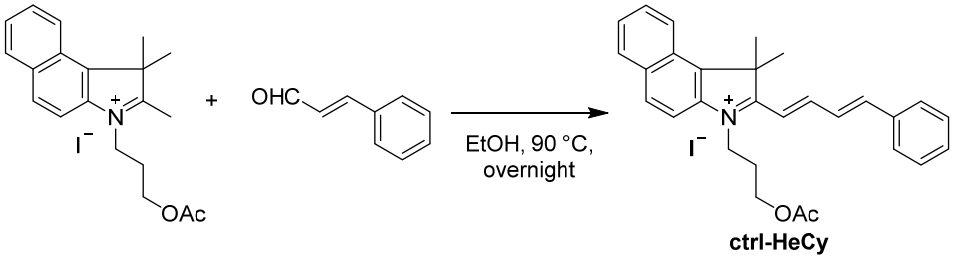
HeCy (10 mg, 0.016 mmol) and potassium carbonate (4.3 mg, 0.031 mmol) were added in the mixed solvents of MeOH/THF (1/1, v/v, 1.5 mL) and stirred at room temperature for 5 h.

After removal of the solvent, the residue was diluted with CH_2Cl_2 (10 mL) and washed with water (10 mL × 2). The organic layer was dried up with Na_2SO_4 and the organic solvent was removed under reduced pressure to obtain **HeCypH** as a yellow solid.

HeCypH: a yellow solid (quant.); ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ 1.14 (s, 3H), 1.22–1.27 (m, 1H), 1.65 (s, 3H), 1.94–2.02 (m, 1H), 3.20 (t, J = 5.0 Hz, 4H), 3.57 (t, J = 13.3 Hz, 1H), 3.72–3.81 (m, 2H), 3.87 (t, J = 5.0 Hz, 4H), 4.05–4.10 (m, 1H), 5.81 (d, J = 16.0 Hz, 1H), 6.59–6.72 (m, 2H), 6.83–6.94 (m, 3H), 7.06 (d, J = 8.6 Hz, 1H), 7.20 (t, J = 7.4 Hz, 1H), 7.35–7.39 (m, 3H), 7.71 (d, J = 8.6 Hz, 1H), 7.77 (d, J = 7.4 Hz, 1H), 7.95 (d, J = 8.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ 20.1, 22.8, 25.1, 40.3, 48.8, 51.0, 62.3, 66.8, 70.5, 101.8, 111.3, 115.3, 121.6, 121.8, 125.6, 125.9, 127.4, 128.5, 128.6, 128.7, 129.3, 129.4, 130.6, 133.1, 136.0, 145.4, 150.7. HRMS (ESI) calcd for $C_{31}H_{35}N_2O_2^+$ ([M+H]$^+$): 467.2693. Found: 467.2693.

1.3.3 Preparation of hemicyanine dye ctrl-HeCy

Hemicyanine dye **ctrl-HeCy** was prepared from a coupling reaction of cinnamaldehyde and 3-acetoxypropyl-substituted benz[e]indole in a similar manner of **HeCy** as shown above.

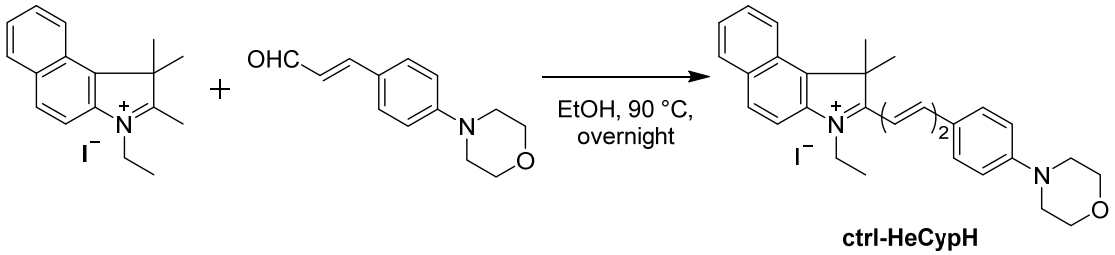


Scheme S2. Synthesis of **ctrl-HeCy**.

ctrl-HeCy: (37 mg, 95%); ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ 1.94 (s, 3H), 2.06 (s, 6H), 2.41 (quint, J = 6.4 Hz, 2H), 4.33 (t, J = 6.4 Hz, 2H), 5.08 (t, J = 6.4 Hz, 2H), 7.39–7.41 (m, 3H), 7.51 (d, J = 14.2 Hz, 1H), 7.65 (t, J = 8.0 Hz, 1H), 7.72–7.76 (m, 4H), 8.05 (d, J = 8.0 Hz, 1H), 8.10 (d, J = 9.0 Hz, 1H), 8.18–8.23 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ 20.9, 26.9, 28.2, 45.5, 53.5, 61.2, 111.9, 116.1, 122.6, 127.4, 127.5, 128.7, 129.1, 129.2, 129.3, 130.4, 131.4, 131.8, 133.6, 135.2, 138.1, 138.3, 150.0, 155.2, 170.5, 182.2. HRMS (ESI) calcd for $C_{29}H_{30}NO_2^+$ ([M]$^+$): 424.2271. Found: 424.2268.

1.3.4 Preparation of hemicyanine dye ctrl-HeCypH

Hemicyanine dye **ctrl-HeCypH** was prepared from a coupling reaction of 3-(4-morpholinophenyl)acrylaldehyde and 3-ethyl-1,1,2-trimethyl-1H-benzo[e]indol-3-ium in a similar manner of **HeCy** as shown above. 3-Ehyl-substituted benz[e]indole was prepared according to our previous reported works.[7]



Scheme S3. Synthesis of **Ctrl-HeCypH**.

ctrl-HeCypH: (68 mg, 93%); ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ 1.63 (t, J = 7.3 Hz, 3H), 2.03 (s, 6H), 3.35 (t, J = 5.0 Hz, 4H), 3.85 (t, J = 5.0 Hz, 4H), 4.83 (q, J = 7.3 Hz, 2H), 6.88 (d, J = 8.6 Hz, 2H), 7.45 (t, J = 14.8 Hz, 1H), 7.62–7.75 (m, 5H), 7.83 (d, J = 14.8 Hz, 1H), 7.95–8.20 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ 14.2, 27.1, 43.0, 47.2, 52.9, 66.5, 111.6, 111.7, 112.5, 114.1, 122.5, 126.2, 126.9, 127.6, 128.4, 130.2, 131.5, 131.9, 133.2, 137.5, 137.9, 151.4, 153.3, 155.8, 180.1. HRMS (ESI) calcd for $C_{30}H_{33}N_2O^+$ ($[M]^+$): 437.2587. Found: 437.2589.

1.4 Photophysical properties of hemicyanie dyes

Molar extinction coefficient value and quantum yields of **ctrl-HeCypH**, **HeCy**, and **HeCypH** were measured in EtOH and aqueous buffered solutions (Table S2). Relative quantum yields were determined by using Rhodamine B (Φ = 70%, in ethanol) as standard.[8] Due to low absorbance and fluorescence intensity in the red-emission range, molar extinction coefficient value and relative quantum yield of **HeCypH** could not be determined. The quantum yield of **HeCypH** might be underestimated, because the closed forms were major components in EtOH. The absolute quantum yields of **HeCypH** in buffered solutions at different pH values were determined.

Table S2. Photophysical properties of dyes.

dyes	relative fluorescence quantum yield Φ (%)[a]	absolute fluorescence quantum yield Φ (%)[b]	molar extinction coefficient ε ($L \times mol^{-1} \times cm^{-1}$)
ctrl-HeCypH	11.2 (EtOH)	-	5.10×10^4 (EtOH)
HeCy	10.7 (EtOH)	3.2 (pH 7.4 buffer)	4.80×10^4 (EtOH)
HeCypH	n.d. (EtOH)	2.9 (pH 3.8 buffer) 0.64 (pH 8.2 buffer)	3.80×10^4 (pH 3.8 buffer)

[a] λ_{ex} = 575 nm; [b] λ_{ex} = 550 nm.

1.5 pH-Dependent UV-vis and fluorescence spectra of HeCy

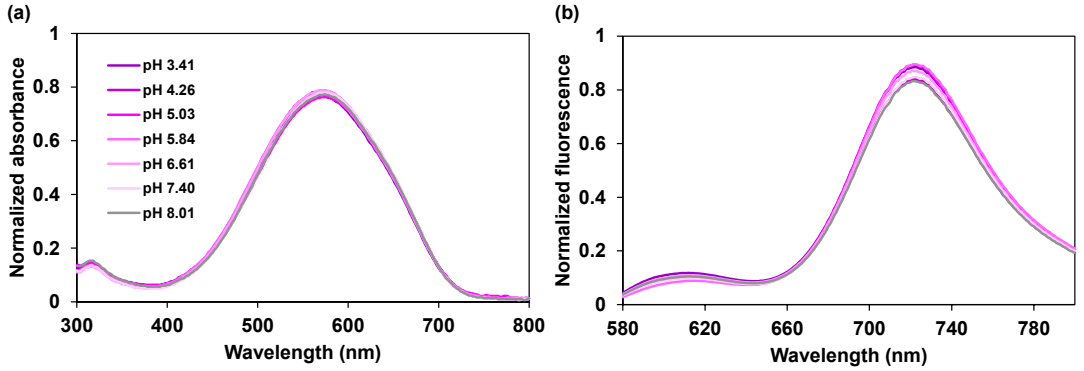


Figure S1. pH-Dependent (a) absorption and (b) fluorescence spectral changes of **HeCy** (5 μM) in various buffered aqueous solutions (pH 3–8, 0.1 M) with Triton X-100 (2.0×10^{-3} M). λ_{ex} = 550 nm.

1.6 UV-vis absorption and fluorescence spectra of HeCy and ctrl-HeCy

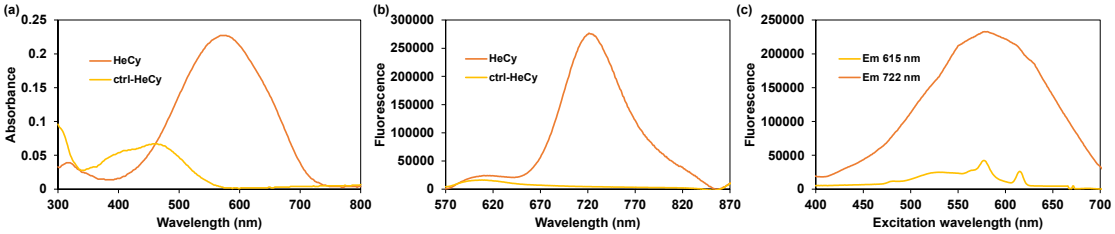


Figure S2. (a) UV-vis absorption, (b) fluorescence spectra of **HeCy** and **ctrl-HeCy** (5 μM), and (c) excitation spectra of **HeCy** (5 μM) in buffered aqueous solution (pH 7.2, 0.1M) with Triton X-100 (2.0×10^{-3} M). λ_{ex} = 550 nm.

1.7 Solvent-dependency of HeCy

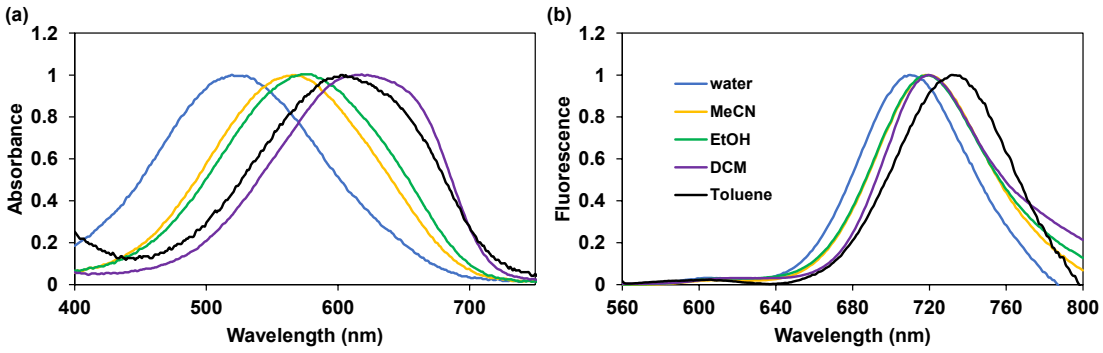


Figure S3. (a) UV-vis absorption and (b) fluorescence spectra of **HeCy** (5 μM) in different solvents including toluene, dichloromethane (DCM), ethanol (EtOH), acetonitrile (MeCN), and water. λ_{ex} = 550 nm.

1.8 Concentration-dependency in absorbance and fluorescence of HeCy

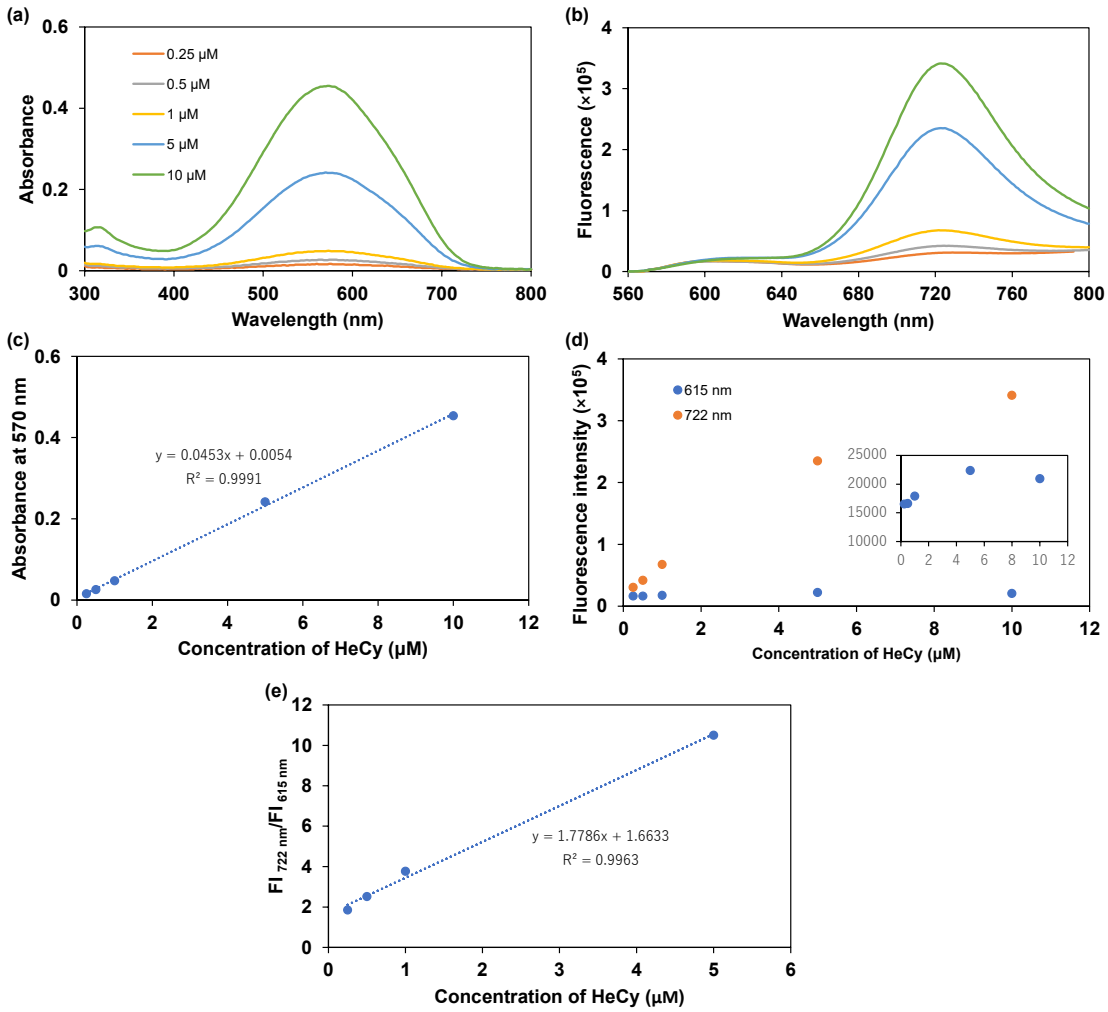


Figure S4. Concentration-dependent (a) UV-vis absorption and (b) fluorescence spectra of **HeCy** in buffered aqueous solution (pH 7.2, 0.1 M) with Triton X-100 (2.0×10^{-3} M). (c) Absorbance at 570 nm and (d) fluorescence intensities at 615 nm and 722 nm of **HeCy** towards concentrations (0.25–20 μM). (e) Fluorescence intensity ratio ($FI_{722\ nm}/FI_{615\ nm}$) of **HeCy** towards concentration in the range of 0–5 μM, respectively. λ_{ex} = 550 nm.

1.9 Selectivity of probe HeCypH towards various analytes

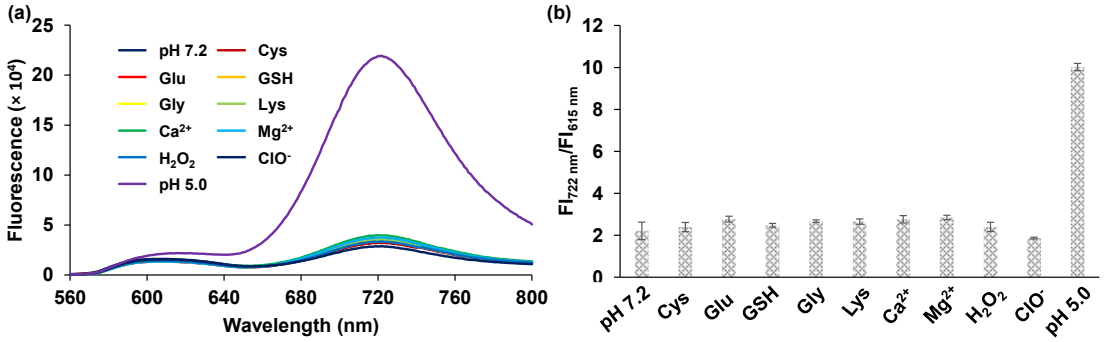


Figure S5. Fluorescent spectra (a) and quantification (b) of **HeCypH** (5 μM) without or with other biological analytes (bovine serum albumin (BSA, 10 μM); typical reactive oxygen species (H_2O_2, ClO^-, 100 μM); metal ions (Ca^{2+}, Mg^{2+}, 100 μM) and amino acids (cysteine, glutamine, glutathione, lysine, 100 μM)) in PBS (pH 7.2) and probe in PBS (pH 5.0). λ_{ex} = 550 nm.

1.10 *In vitro* cell experiments

Human lung carcinoma cell line, A549, was purchased from American Type Culture Collection (Manassas, VA). A549 cells were cultured in 10% FBS-Dulbecco's modified Eagle's medium (DMEM). Cells were cultured in well-humidified incubator with 5% CO_2 and 95% air at 37 °C.

1.10.1 Cytotoxicity experiments (MTT assay)

Measurement of cell viability was evaluated by reducing of MTT (3-(4,5-dimethyl-2-thiazolyl)-2,5-diphenyltetrazolium bromide) to formazan crystals using mitochondrial dehydrogenases. The cytotoxicity assays were performed by A549 cells in 96-well plates with a seeding density of 2×10^4 cells per well. After 24 h of cell attachment at 37 °C in 5% CO_2 humidified incubator before adding test substances, the plate was washed with 100 μL/well PBS. Then the cells were cultured in medium with 0.05, 0.5, 5, 50 μM of probe **HeCypH** under 37 °C for 6 h. Cells in culture medium without probe were used as the control. Six replicate wells were used for each control and test concentration. 100 μL of MTT (0.5 mg/mL) prepared in medium was added to each well and the plates were incubated at 37 °C. Wells without cell were used as the blank. The medium was carefully removed and washed with PBS, and the purple crystals were lysed in 200 μL DMSO. Absorbance at 550 nm was measured with micro-plate spectrophotometer (800TS, Biotek Instruments, Inc. USA). Cell viability was calculated using the following equation:

$$\text{Cell viability (\%)} = (A_{test}-A_{blank})/(A_{control}-A_{blank}) \times 100$$

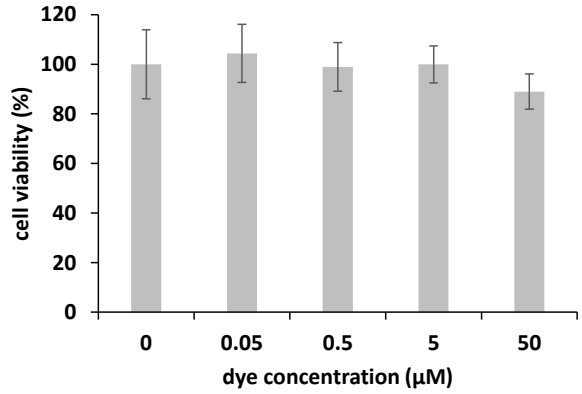


Figure S6. Cytotoxicity of **HeCypH** in varied concentration (0, 0.05, 0.5, 5 and 50 μM) in living A549 cells for 6 h. Error bar indicates s.d., n = 6.

1.10.2 Confocal imaging

A549 cells (2×10^5 cells/dish) were passaged to μ-35 mm dish (ibidi) for confocal imaging after 24 h attachment, following a washing thrice with DMEM without phenol red. The fluorescence imaging was performed with LSM 710 inverted fluorescence microscope with 63× objective lens. For cell internalization assay: confocal images were taken before and after **HeCypH** (5 μM) staining at 0, 5, 10, 15, 30, 60, and 80 min without further washing procedures (Figure S5). To evaluate the pH-dependent fluorescence response, high K^+ buffered solution (0.5 mM $MgSO_4$, 30 mM NaCl, 120 mM KCl, 5 mM glucose, 1 mM $CaCl_2$, 1 mM NaH_2PO_4, 20 mM NaOAc and 20 mM HEPES) containing H^+/K^+ ionophore at different pH values were firstly prepared. To investigate the reversibility of **HeCypH** in living cells, A549 cells treated with **HeCypH** (5 μM) for 0–30 min, following a washing twice with high K^+ buffered solution (pH 8.0). Then the fresh buffered solution (pH 8.0) was added and incubate for 0–20 min. Finally, the culture medium was replaced again by DMEM without phenol red. The red emission was detected at 580 nm–758 nm when excited at 561 nm. For the colocalization experiments, A549 cells were treated with **HeCypH** (5 μM) for 30 min at 37 °C and wash with DMEM, then treated with LysoTracker green DND-26 (2 μM), ER-Tracker^{TM} Green (5 μM), or MitoTracker^{TM} Green FM (2 μM) for 15 min, respectively. Conditions: For **HeCypH**, λ_{ex} = 561 nm, λ_{em} = 580–758 nm; For LysoTracker Green, and Mito-Tracker Green, ER-Tracker Green, λ_{ex} = 488 nm, λ_{em} = 500–560 nm. A549 cells were treated with **HeCypH** (5 μM) for 30 min at 37 °C, following a washing procedure by DMEM without phenol red (1 mL × 2). Then the new buffered solutions with varied pH values (4.5, 5.0, 5.5, 6.0, 6.5, 7.0, 7.5) was added to the dishes and cultured for a further 30 min. For visualization of pH fluctuation under stimulation conditions, A549 cells were pretreated with NH_4Cl (30 mM), or chloroquine (75 μM) for 30 min at 37 °C under 5% CO_2, followed by staining with **HeCypH** (5 μM) for 30 min. In these

experiments, the fluorescence signals were detected at two channels, 596–636 nm and 696–736 nm when excited at 561 nm. The fluorescence intensities were extracted from seven ROIs in each image by ImageJ win64 software.

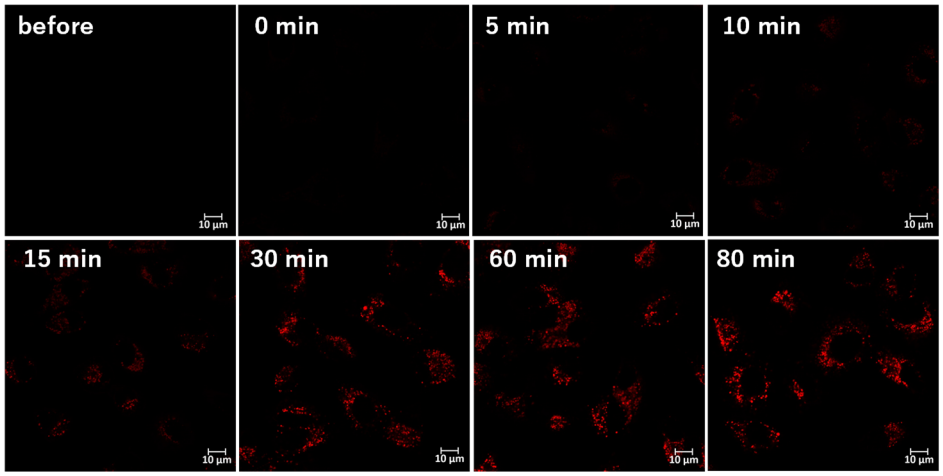


Figure S7. Confocal images of living A549 cells after stained with **HeCypH** (5 μM) for 0–80 min without washing procedure.

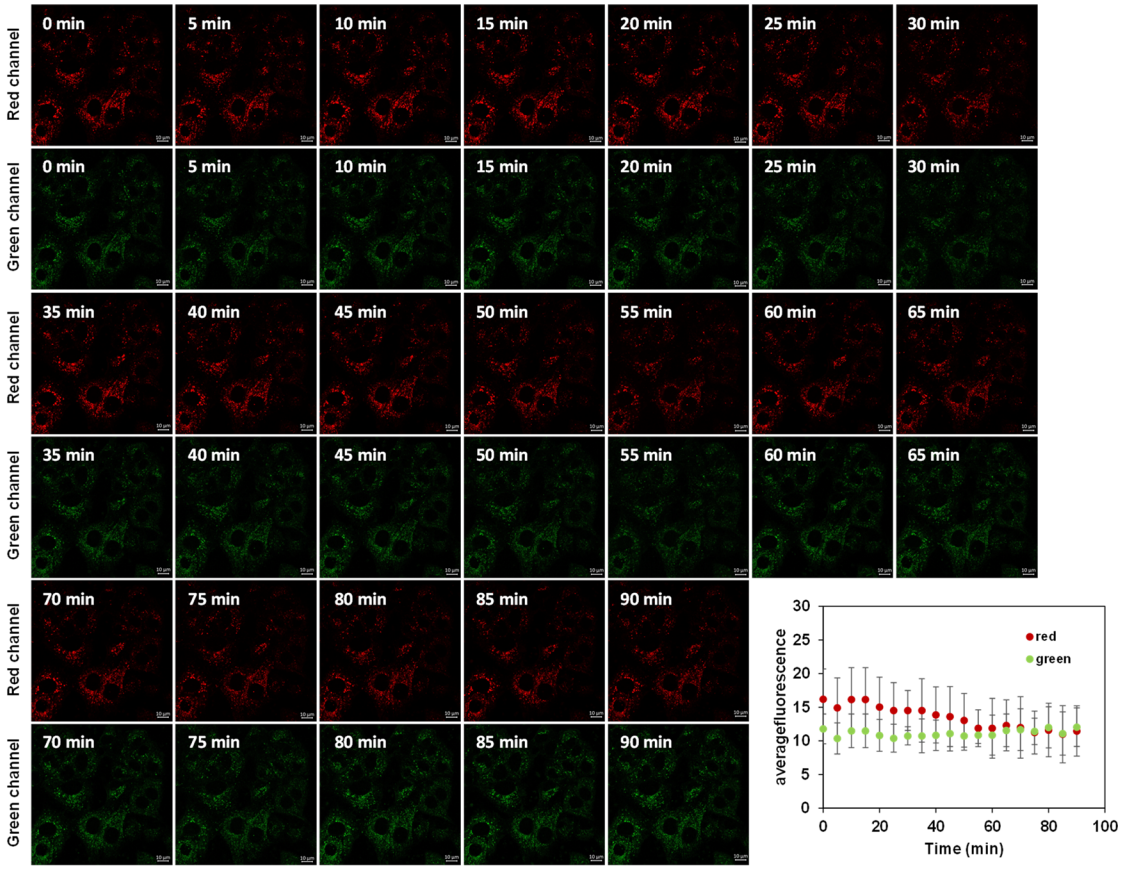


Figure S8. Confocal images of living A549 cells acquired from 0 to 90 min (at 5 min intervals) after

staining with **HeCypH** (5 μM) for 1 h. Red channel and green channel were collected at 596–636 nm and 696–736 nm when excited at 561 nm. Scale bar: 10 μm.

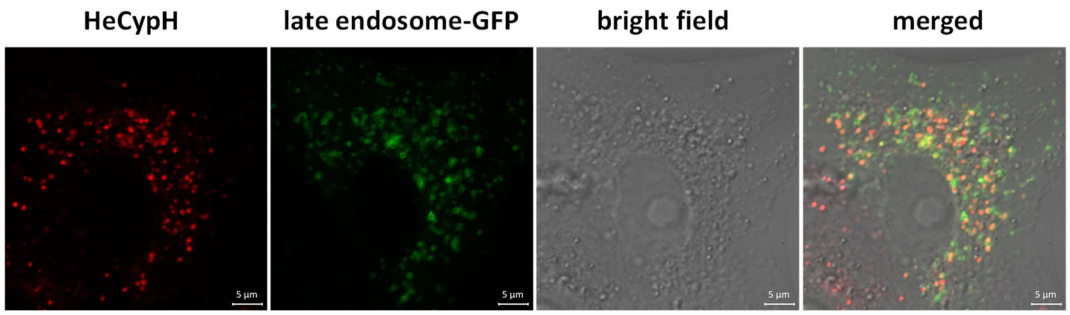


Figure S9. Representative confocal images of living A549 cells pretreated with CellLight™ Late Endosomes-GFP (Rab7a sequence, 50 particles/cell, 16 h), followed by **HeCypH** (5 μM) incubation for 30 min. Scale bar: 5 μm. Colocalization areas are in yellow. Conditions: for **HeCypH**, λ_{ex} = 561 nm, λ_{em} = 580–758 nm. For commercial tracker, λ_{ex} = 488 nm, λ_{em} = 500–560 nm.

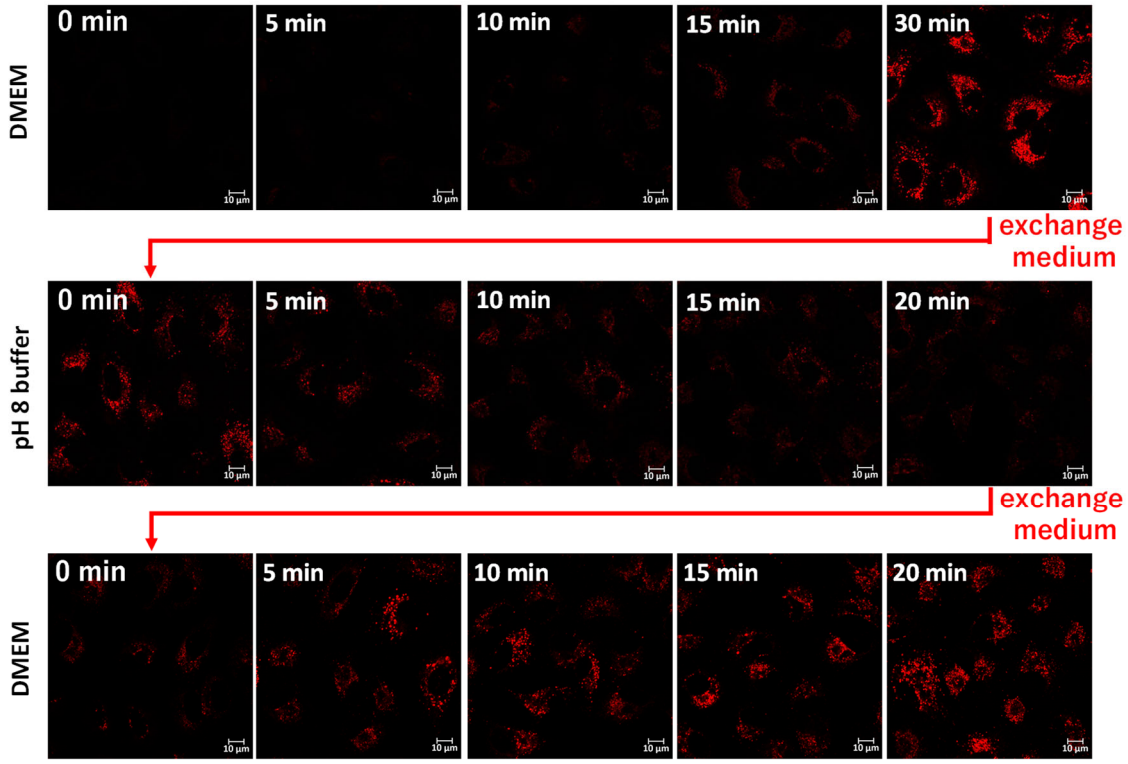


Figure S10. pH-Dependent reversible response of **HeCypH** (5 μM) in living A549 cells.

1.11 IVIS imaging of HeCypH or ctrl-HeCypH in solutions and *in vivo*

For a demonstration using IVIS imaging, the solutions of **HeCypH** (5 μM) in pH 5.4, 5.8, 6.2, 6.6, 7.0, 7.4, 7.8 PBS buffer with Triton X-100 (2 mM) were prepared. Fluorescence images of **HeCypH** were measured by IVIS Imaging System (λ_{ex} = 558 nm, DsRed filter; λ_{em1} = 583 nm, DsRed filter; and λ_{em2} = 695 nm, Cy5.5 filter). Fluorescent intensities of dyes in solutions from two channels were collected based on the shape of centrifuge tube.

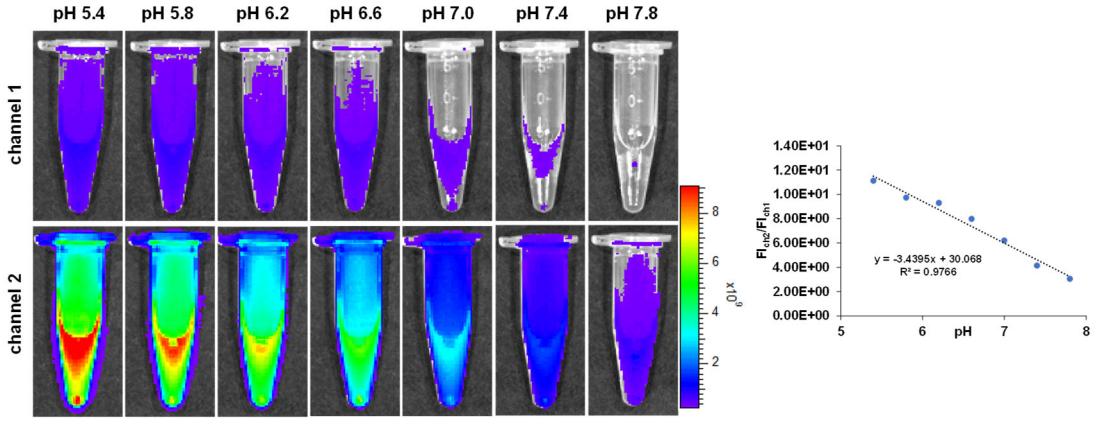


Figure S11. Representative IVIS images of **HeCypH** (5 μM) in PBS buffered solutions at various pH values.

For in vivo experiments, six BALB/cCrSlc mice (6 weeks old) were randomly divided in two groups and used as controlled and chloroquine (CQ)-pretreated mice, respectively. Mice of group 1 were administrated with saline (100 μL) and mice of group 2 were treated with chloroquine (10 mg/kg in 100 μL saline) via i.p. injection and cultured for 4 h. Then a second i.p. injection of **HeCypH** (100 μM in 200 μL saline) was operated to the same place in each mouse. Then fluorescence images of mice were measured by IVIS imaging system equipped with DsRed and Cy5.5 filters. **Ctrl-HeCypH** were examined through same methods as mentioned above (Figure S8). Fluorescence intensities of ROIs were indicated and detected in the same size (diameter = 1.60 cm) using Living Image 2.50-Igor Pro 4.09 software.

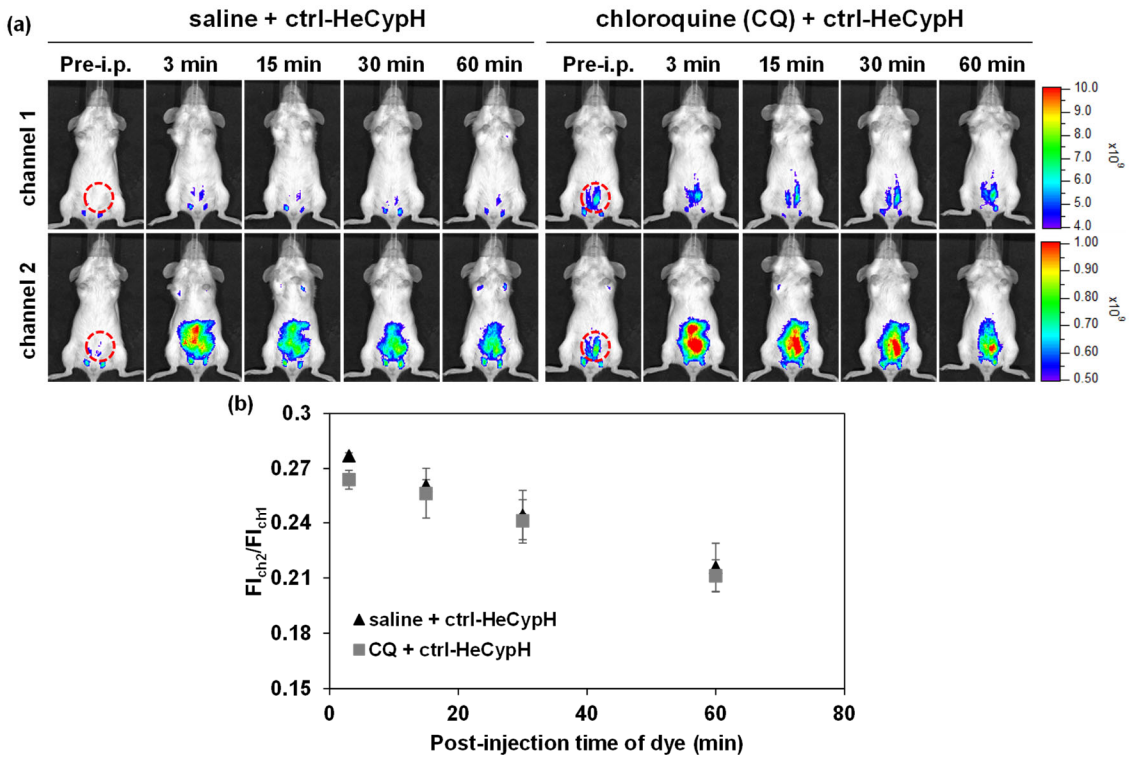
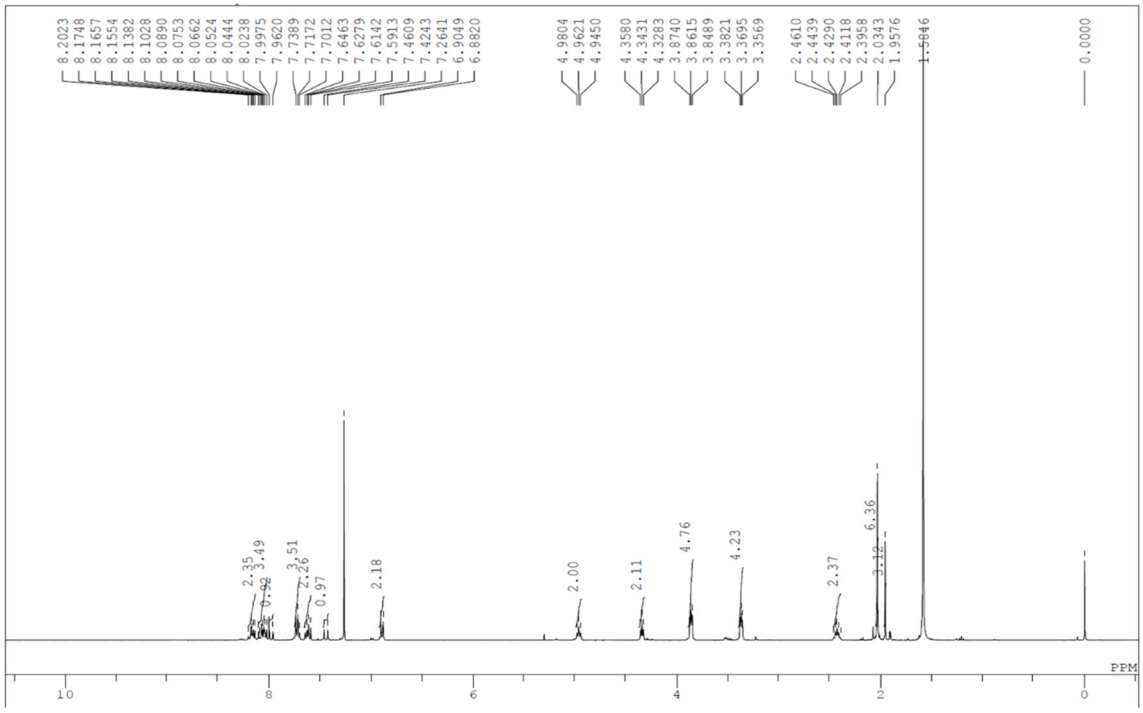


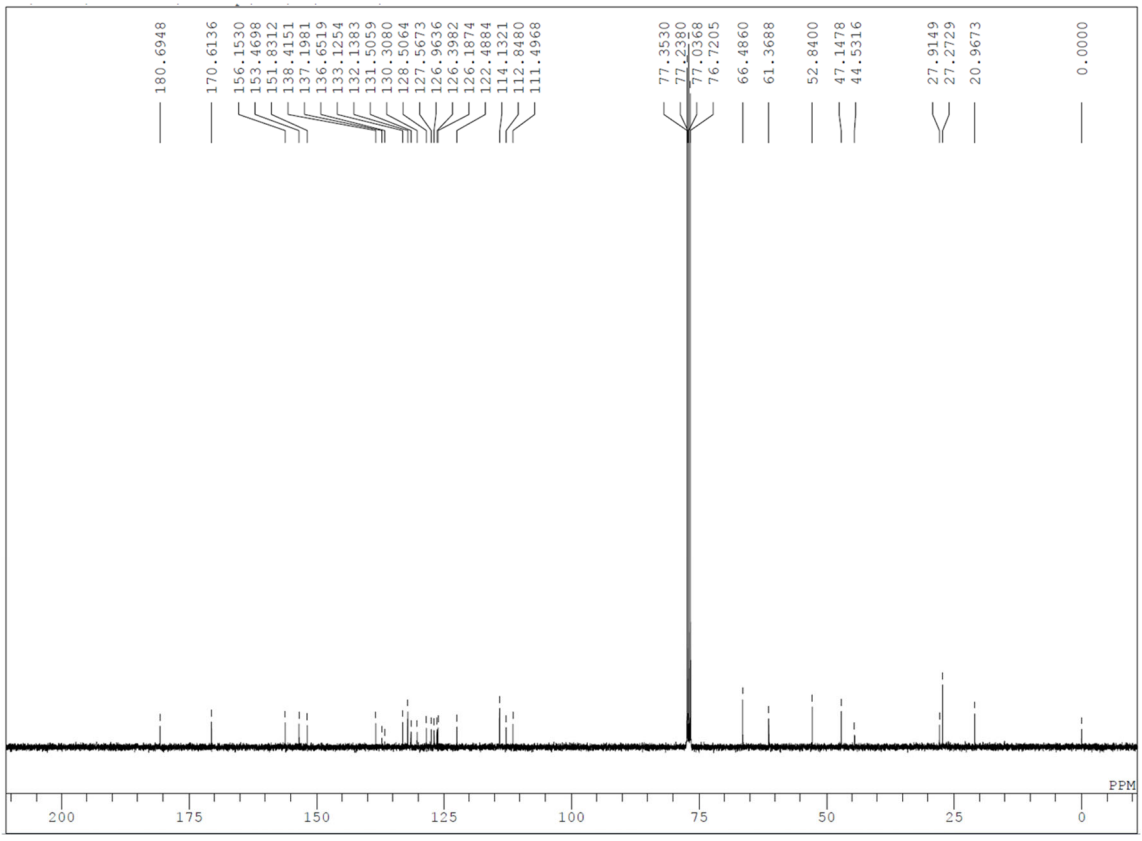
Figure S12. (a) Representative IVIS imaging of living mice exposed to saline (100 μL, left) or chloroquine (CQ, 10 mg/kg, 100 μL, right) for 4 h, followed by an injection of **ctrl-HeCypH** (100 μM in 200 μL saline). (b) Normalized fluorescent ratio of channel 2 and channel 1 of ROIs (indicated by red circle) at different time points from (a) after **ctrl-HeCypH** injection. All procedures were performed via intraperitoneal (i.p.) injection, and the centers of the red circles on the mice indicate the injection sites.

2. NMR spectra

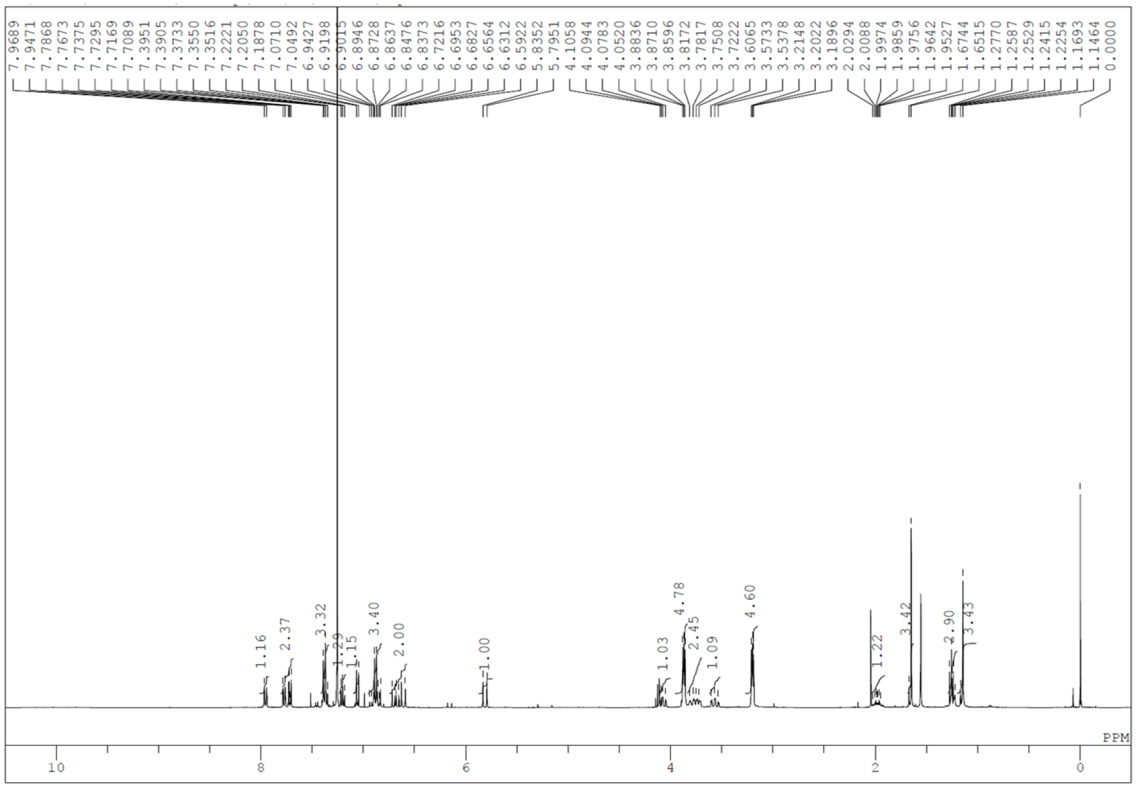
^{1}H NMR of **HeCy** ($CDCl_3$)



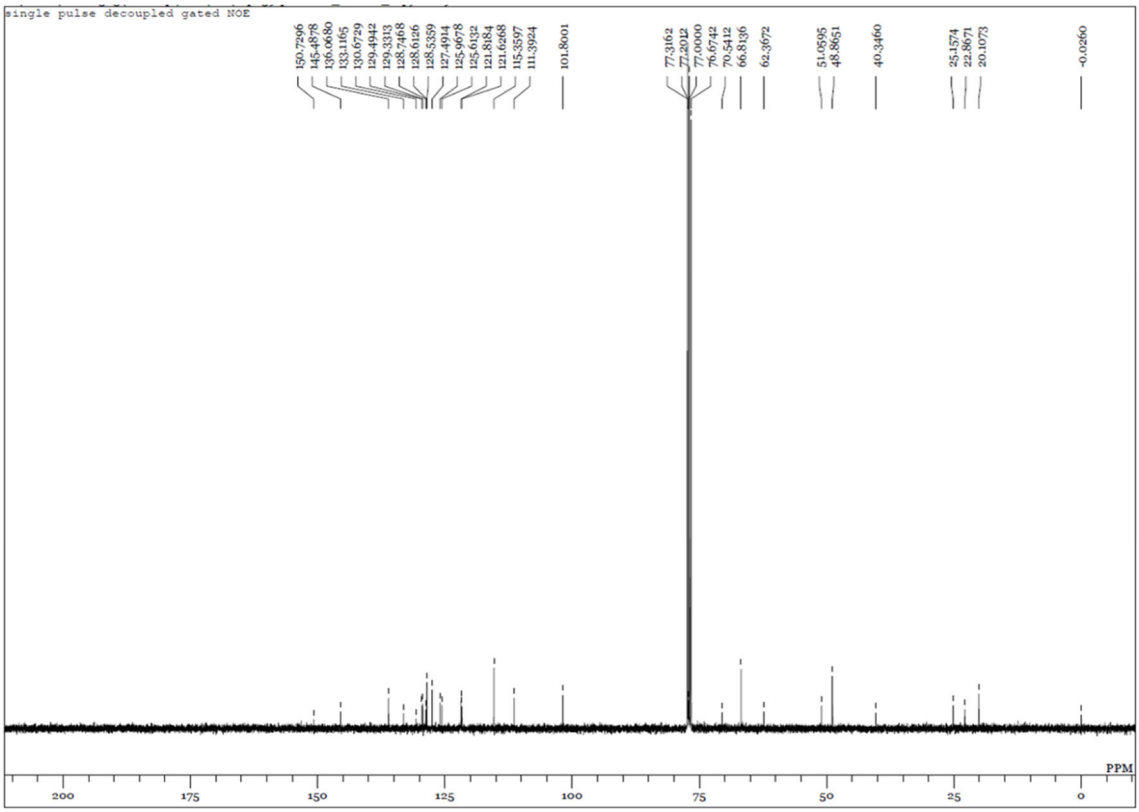
^{13}C NMR of **HeCy** ($CDCl_3$)



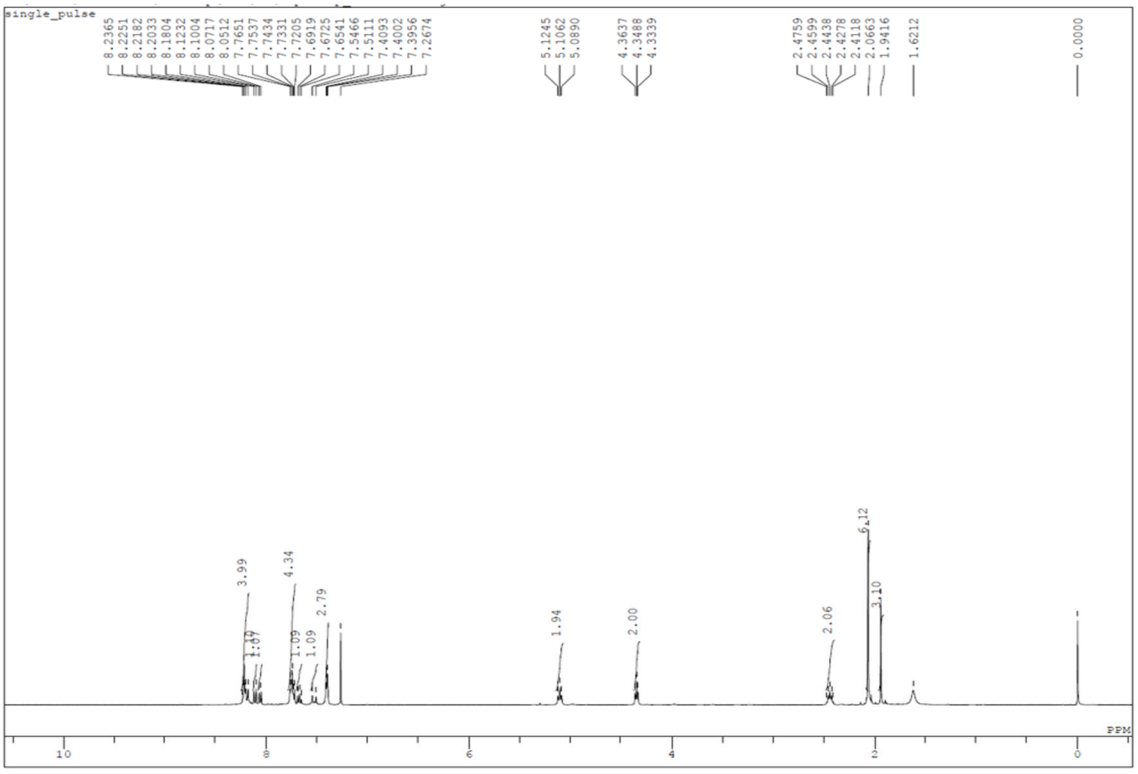
^{1}H NMR of **HeCypH** ($CDCl_3$)



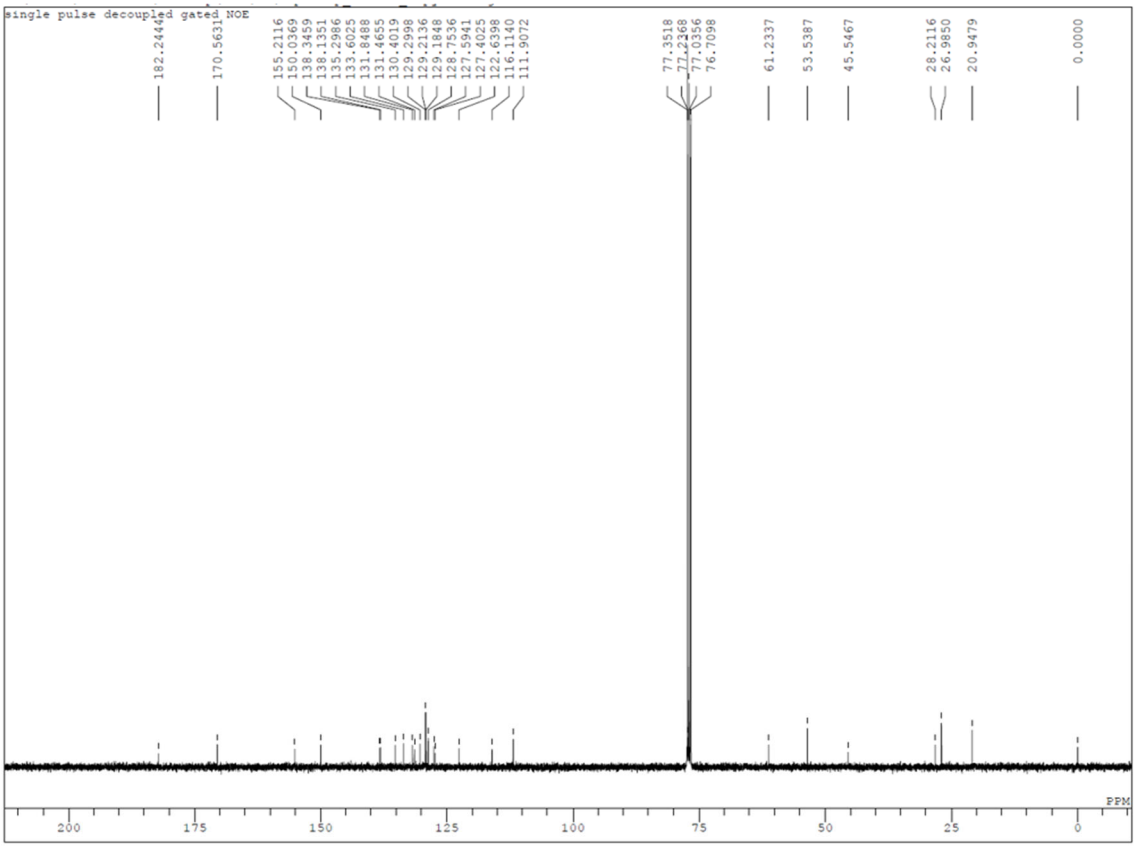
^{13}C NMR of **HeCypH** ($CDCl_3$)



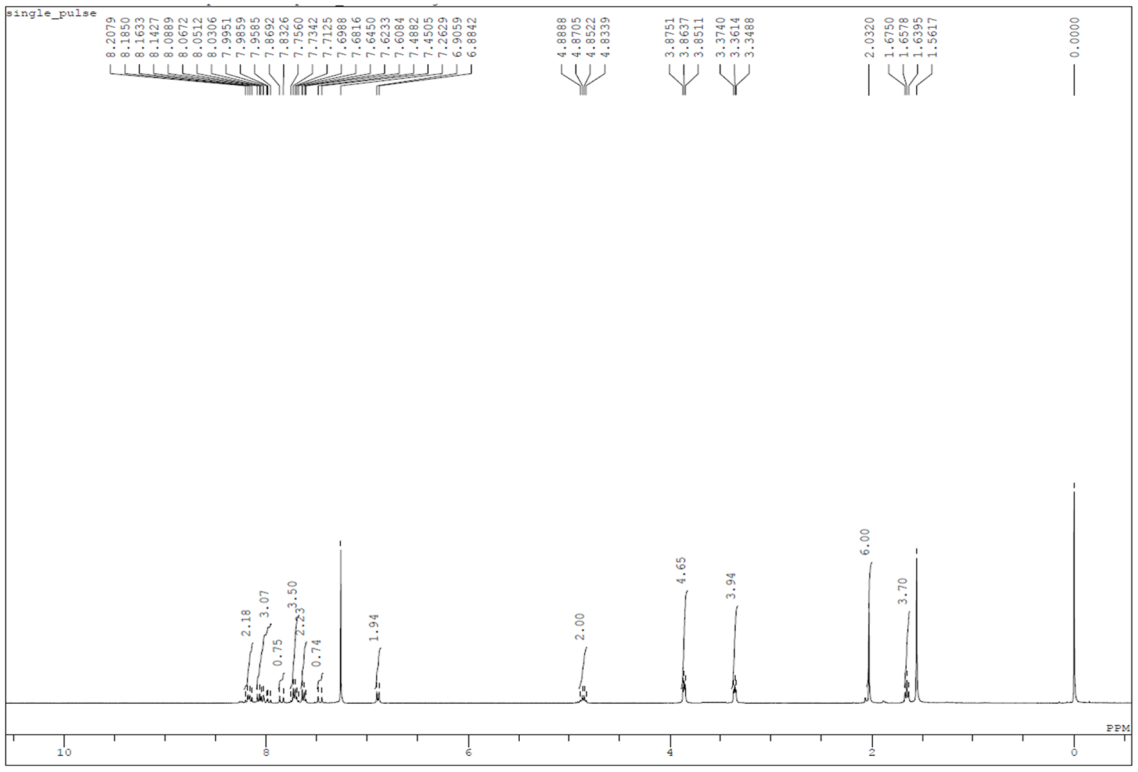
^{1}H NMR of **ctrl-HeCy** ($CDCl_3$)



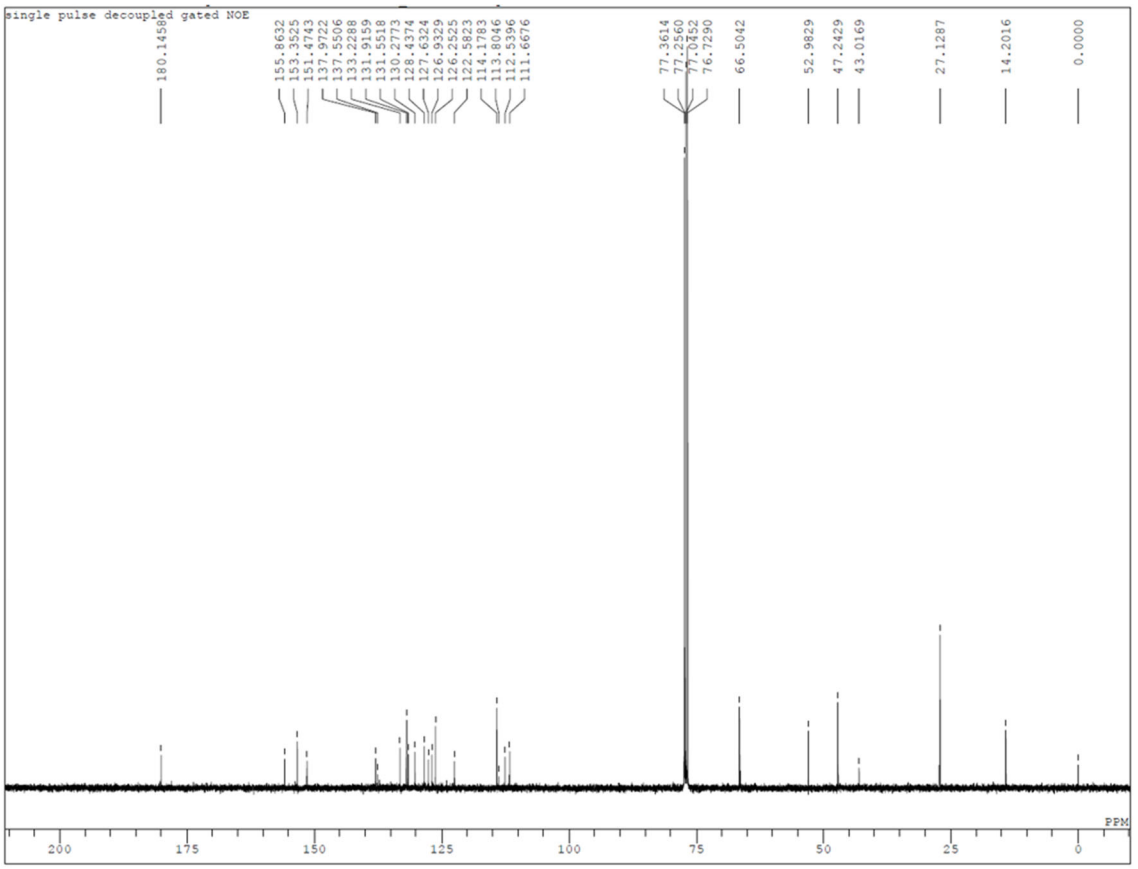
^{13}C NMR of **ctrl-HeCy** ($CDCl_3$)



^{1}H NMR of **ctrl-HeCypH** ($CDCl_3$)



^{13}C NMR of **ctrl-HeCypH** ($CDCl_3$)



3. References

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