Electronic Supplementary Material (ESI) for Chemical Communications.

Supporting Information

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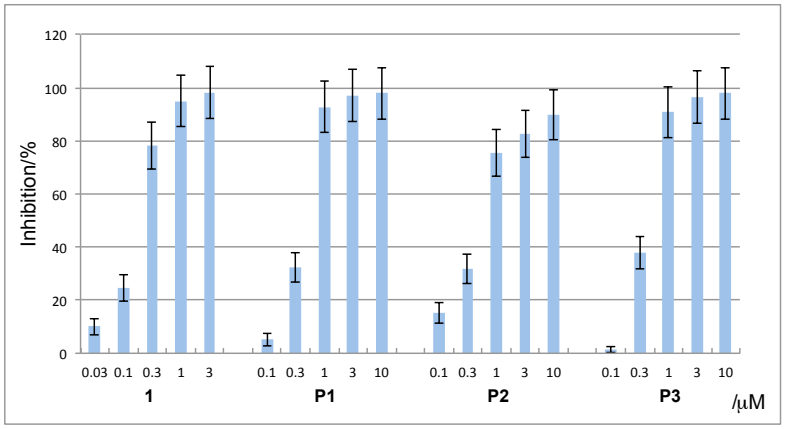
1. Supplementary Results



compound	IC_{50} (nM)
1	169 ± 22
P1	396 ± 67
P2	528 ± 76
P3	402 ± 33

Fig. S1. CA-II-inhibitory activity of the synthesized AzPI probes. *Measurement conditions*: 500 nM CA-II, 0.03–10 μM probe, 3 mM *p*-nitrophenyl acetate, 50 mM Tris-HCl buffer (pH 8.0).

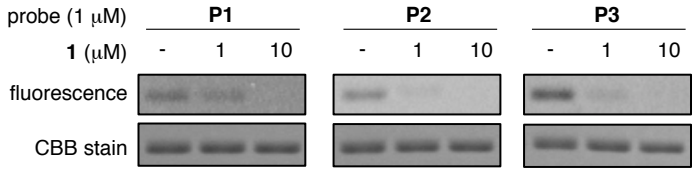


Fig. S2. Competition analysis. *Labeling conditions*: 1 μM CA-II, 1 μM **P1**–**P3**, 1–10 μM compound **1,** 20 mM phosphate buffer (pH 7.0), photo-irradiation (365 nm, 5 min, 0 ºC) after incubation at 0 ºC for 30 min. Comments: Labeling was inhibited by addition of **1**. This indicates that all the probes labeled the target CA-II in response to the ligand–target interactions.

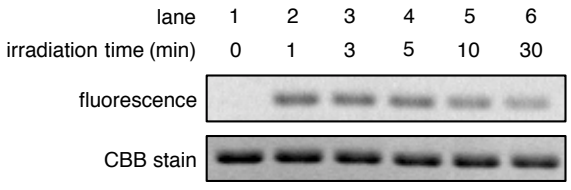


Fig. S3. Optimization of the photo irradiation time. *Labeling conditions*: 1 μM CA-II, 1 μM **P3**, 20 mM phosphate buffer (pH 7.0), photo-irradiation (365 nm, 0–30 min, 0 ºC) after incubation at 0 ºC for 30 min. Comments: Efficient fluorescence labeling proceeded with irradiation at 365 nm for ≤5 min.

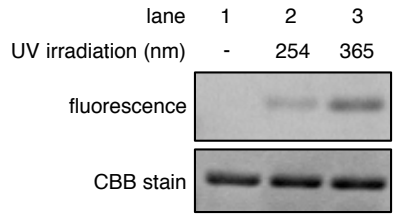
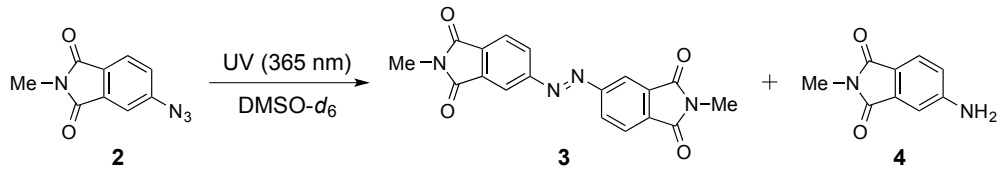


Fig. S4. Comparison of two irradiation wavelengths (254/365 nm). *Labeling conditions*: 1 μM CA-II, 1 μM **P3**, 20 mM phosphate buffer (pH 7.0), photo-irradiation (254 or 365 nm, 5 min, 0 ºC) after incubation at 0 ºC for 30 min. Comments: A handy mercury lamp (AS ONE, SLUV-4) was used in this experiment.

Table S1. Photoreaction of **2** in DMSO-d_6



		yields (%)		
entry	conditions	recovery of **2**	**3**	**4**
1	0 ºC, 1 h	81	10	–
2	diethylamine (100 equiv.), 0 ºC, 1 h	18	–	64

Comments: Photo-irradiation of **2** in DMSO-d_6 gave dimerized azo compound **3** in 10% yield. In the presence of diethylamine (100 equiv.), 4-amino-*N*-methylphthalimide (**4**) was obtained in 64% yield.

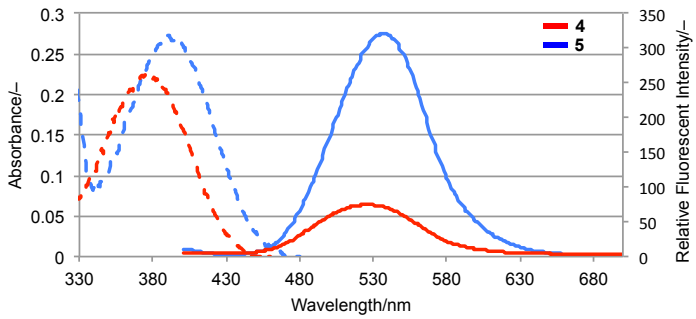
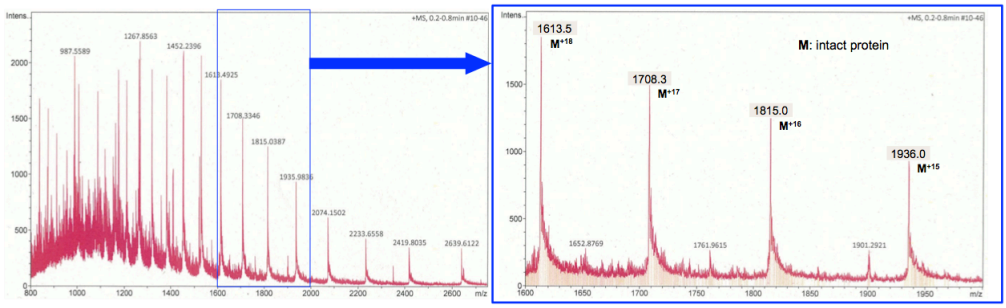


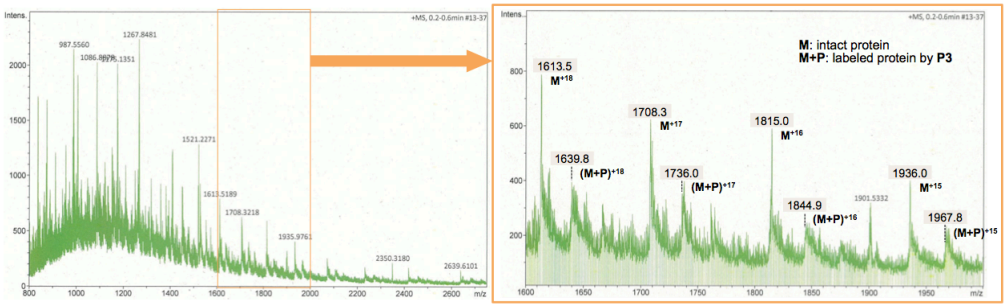
Fig. S5. UV-Vis absorbance (dotted lines) and fluorescence spectra (solid lines) of **4** and *N*-methyl-4-(propylamino)phthalimide (**5**). *Measurement conditions*: 100 μM compound in methanol.

The molar extinction coefficients (ε_{max}) were 2249 (373 nm) for **4** and 2729 (393 nm) for **5**. The fluorescence quantum yields (ϕ_f) were 0.053 (λ_{ex} = 373 nm) for **4** and 0.17 (λ_{ex} = 393 nm) for **5**, based on fluorescein disodium salt as a standard (λ_{ex} = 496.5 nm, ϕ_f = 0.93 in MeOH)[S1].

a) CA-II



b) Labeled CA-II by **P3**



Deconvolution [Calculated molecular weight: 29025.6 (intact CA-II), 29499.7 (labeled CA-II by **P3**)]

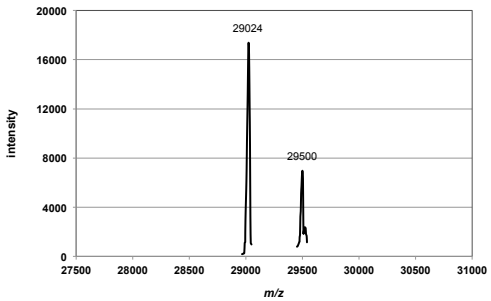


Fig. S6. MS analysis of the whole protein. a) MS analysis of CA-II. b) MS analysis of the labeled CA-II. Labeling conditions: **P3** (1 μM), CA-II (1 μM = 0.03 mg/mL), phosphate buffer (pH 7.0), and UV irradiation at 365 nm (5 min, 0 ºC). Deconvolution of the signals corresponding to the multiple charge states was performed using Compass 1.3 (Bruker).

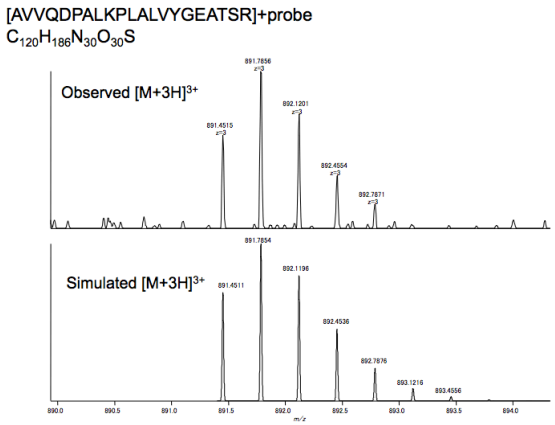
Comments: Labeling yield was estimated to be 29% based on the peak heights of the intact and the labeled CA-II after the deconvolution processing.

Table S2. SEQUEST search result (identified amino-**P3**-immobilized peptides)

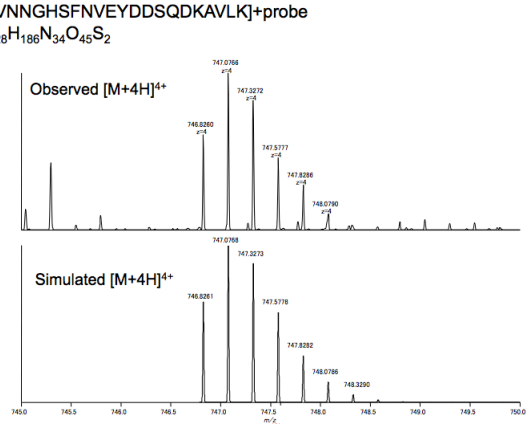
sequence	position	modifications	$[M+H]^+$	peak area
SHHWGYGK	2–9	N-term-Acetyl, amino-**P3**	1487.5796	$1.7x10^7$ ($[M+2H]^{2+}$) $2.9x10^7$ ($[M+3H]^{3+}$)
AVVQDPALKPLALVYGEATSR	37–57	amino-**P3**	2672.3399	$1.5x10^5$ ($[M+3H]^{3+}$)
MVNNGHSFNVEYDDSQDKAVLK	59–80	amino-**P3**	2984.2826	$2.8x10^5$ ($[M+4H]^{4+}$)
VLDALDSIKTK	159–169	amino-**P3**	1676.8200	$4.3x10^5$ ($[M+3H]^{3+}$)

Comments: Labeling occurred mainly on the sequence Ser2–Lys9.

a) Ala37–Arg57



b) Met59–Lys80



c) Val159–Lys169

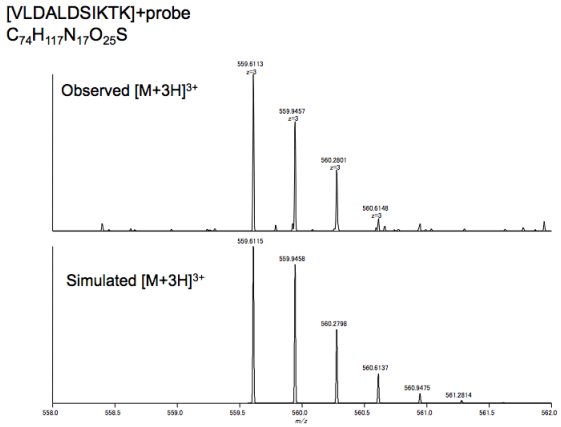
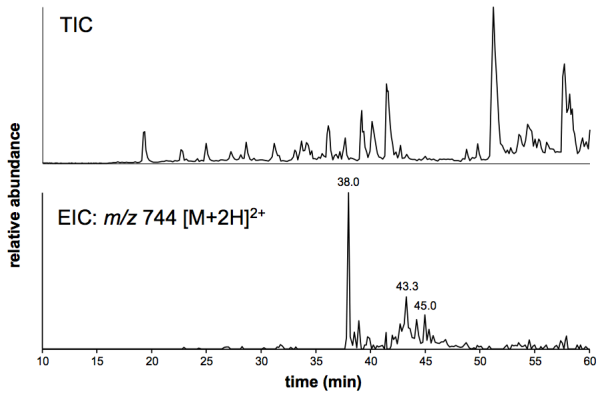
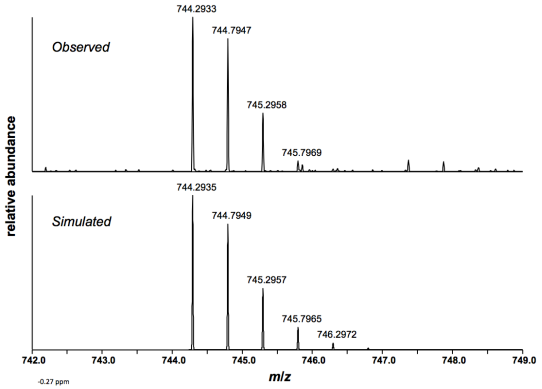


Fig. S7. Mass spectra of the identified amino-**P3**-immobilized peptide candidates (Ala37–Arg57, Met59–Lys80, and Val159–Lys169).

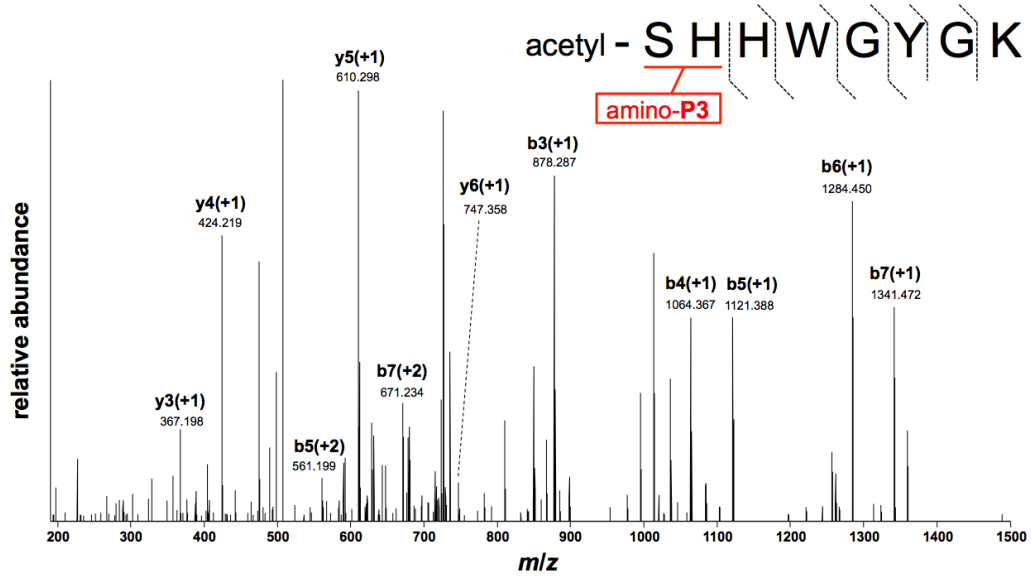
a)



b)



c)



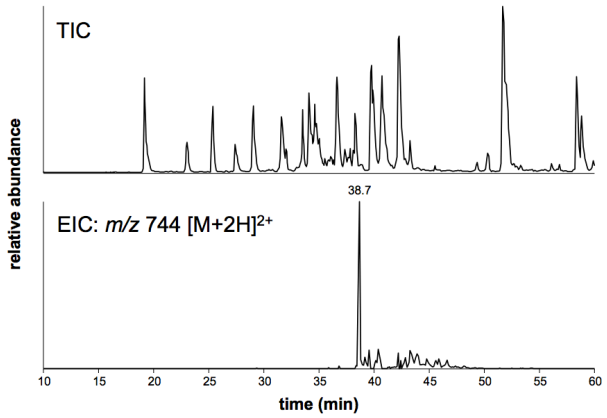
d)

CID mode in Orbitrap

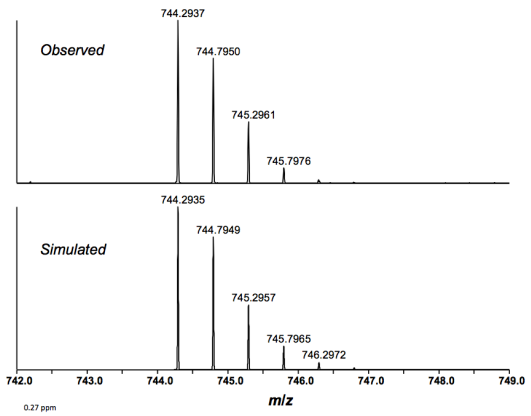
b-ion	b⁺	obs.	ppm	Seq.	y⁺	obs.	ppm	y-ion
2	741.230			acetyl-SH-amino-P3	1358.537			7
3	878.289	878.288	-1.14	H	747.357	747.358	1.34	6
4	1064.368	1064.367	-0.97	W	610.298	610.298	-0.66	5
5	1121.389	1121.388	-1.43	G	424.219	424.219	-0.71	4
6	1284.453	1284.450	-1.96	Y	367.198	367.198	-0.27	3
7	1341.474	1341.472	-1.78	G	204.134			2
8				K	147.113			1

Fig. S8. Detailed MS/MS results in CID ion-fragmentation mode. See also Figure 6. a) TIC and EIC chromatograms. b) MS1 of the amino-**P3**-immobilized Ser2–Lys9 (R_t 38.0 min). c,d) MS/MS analysis.

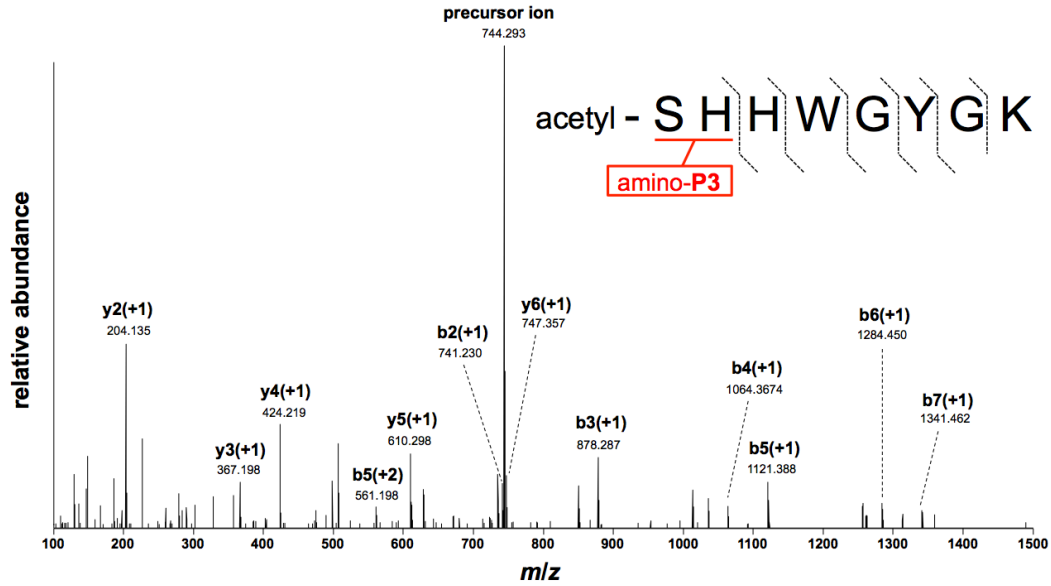
a)



b)



c)



d)

HCD mode in Orbitrap

b-ion	b^+	obs.	ppm	Seq.	y^+	obs.	ppm	y-ion
2	741.230	741.230	0.13	acetyl-SH-amino-P3	1358.537			7
3	878.289	878.287	-1.82	H	747.357	747.357	-0.40	6
4	1064.368	1064.367	-0.50	W	610.298	610.299	0.16	5
5	1121.389	1121.388	-1.43	G	424.219	424.219	-0.24	4
6	1284.453	1284.450	-2.04	Y	367.198	367.198	0.27	3
7	1341.474	1341.462	-8.94	G	204.134	204.135	0.98	2
8				K	147.113			1

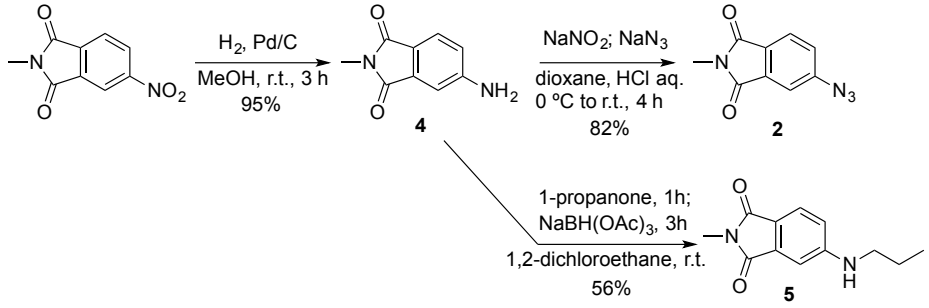
Fig. S9. Detailed MS/MS results in HCD ion-fragmentation mode. a) TIC and EIC chromatograms. b) MS1 of the amino-**P3**-immobilized Ser2–Lys9 (R_t 38.8 min). c,d) MS/MS analysis.

2. Chemistry

2-1. General information

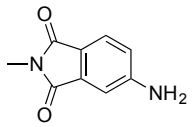
Chemicals were purchased from Sigma-Aldrich Co. LLC, Kanto Chemical Co. Inc., Tokyo Chemical Industry Co. Ltd., Acros Organics or Wako Pure Chemical Industries, Ltd., and used without further purification. Reactions were monitored by thin-layer chromatography (TLC, Merck silica gel 60 F_{254}) plate. Bands were visualized using UV light or appropriate reagents followed by heating. Flash chromatography was carried out with silica gel (Silica gel 60N, 40–50 mm particle size) purchased from Kanto Chemical Co. Inc. IR spectra were recorded on a JASCO FT/IR-470 spectrometer. NMR spectra were recorded on a JEOL JNM-ECA500 spectrometer, operating at 500 MHz for ^{1}H-NMR and at 125 MHz for ^{13}C-NMR. Proton and carbon chemical shifts are expressed in δ values (ppm) relative to internal $CHCl_3$ (7.24 ppm), CHD_2OD (3.30 ppm), C_2HD_5SO (2.49 ppm) for ^{1}H-NMR, and internal $CDCl_3$ (77.00 ppm), methanol-d_4 (49.00 ppm), dimethylsulfoxide-d_6 (39.50 ppm) for ^{13}C-NMR. Data are reported as follows: chemical shift, multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad), coupling constants (Hz), integration. High-resolution mass spectra were obtained using a BRUKER micrOTOF II mass spectrometer.

2-2. Synthetic Procedures



Scheme S1. Synthesis of AzPI **2** and 4-aminophthalimide derivatives **4** and **5**.

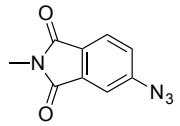
*Synthesis of 4-amino-N-methylphthalimide (**4**) (Compound **4** was prepared by a reported procedure.*[S2]*)*



10% Pd/C (50 mg) was added to a solution of *N*-methyl-4-nitrophthalimide (500 mg, 2.4 mmol) in MeOH (16 mL), and the mixture was stirred for 3 h at room temperature under a hydrogen atmosphere.

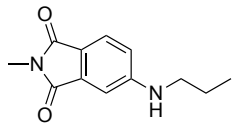
The mixture was then filtrated through Celite pad and washed with MeOH. The solvent was removed under reduced pressure to afford the product **4** (405 mg, 2.3 mmol, 95%) as a yellow solid. **4**: ^{1}H-NMR (DMSO-d_6) δ 2.93 (s, 3H), 6.42 (br s, 2H), 6.76 (dd, J = 2.0, 8.3 Hz, 1H), 6.89 (d, J = 1.7 Hz, 1H), 7.46 (d, J = 8.1 Hz, 1H); ^{13}C-NMR (DMSO-d_6) δ 23.4, 106.9, 116.4, 116.8, 124.7, 134.6, 154.8, 168.1, 168.4; HRMS (ESI) Calcd. for $C_9H_8N_2O_2Na$: 199.0478, Found 199.0470.

*Synthesis of 4-azido-1-methylphthalimide (**2**)*



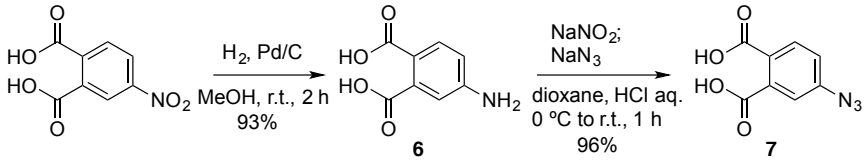
Sodium nitrate (59 mg, 0.85 mmol) in water (0.5 mL) was added to a solution of **4** (100 mg, 0.57 mmol) in 1,4-dioxane (5.0 mL) and 6 *N* HCl aq (4.0 mL) at 0 ºC. Sodium azide (55 mg, 0.85 mmol) in water (0.5 mL) was then added to the reaction mixture at 0 ºC, and the resulting mixture was stirred for 1 h at room temperature. The reaction was quenched by an addition of saturated $NaHCO_3$ aq. and extracted with AcOEt. The combined organic layer was dried over Na_2SO_4, and the solvent was removed under reduced pressure. The residue was purified by silica-gel column chromatography (AcOEt/*n*-hexane = 1/6) to afford **2** (95 mg, 0.47 mmol, 82%) as a white solid. **2**: ^{1}H-NMR ($CDCl_3$) δ: 3.15 (s, 3H), 7.26 (dd, J = 2.3, 8.0 Hz, 1H), 7.46 (d, J = 1.7 Hz, 1H), 7.79 (d, J = 8.1 Hz, 1H); ^{13}C-NMR ($CDCl_3$) δ: 24.1, 113.7, 123.8, 124.9, 128.1, 134.5, 146.5, 167.5, 167.6; HR-MS (ESI) Calcd. for $C_9H_6N_4O_2Na$: 225.0383, Found 225.0372.

*Synthesis of N-methyl-4-(propylamino)phthalimide (**5**)*

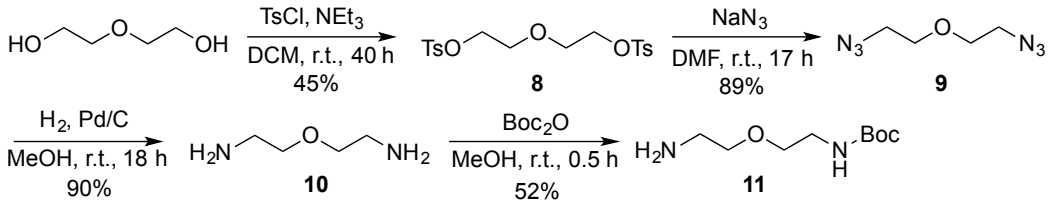


Propionaldehyde (41 μL, 0.57 mmol) was added to a solution of **4** (100 mg, 0.57 mmol) in 1,2-dichloroethane (4.0 mL) at 0 ºC, and the mixture was stirred for 1.5 h at room temperature. Sodium triacetoxyborohydride (181 mg, 0.86 mmol) was added to the mixture, and the resulting mixture was further stirred for 3 h at room temperature. Saturated NaHCO3 aq. was added, and the mixture was extracted with AcOEt. The combined organic layer was dried over Na_2SO_4, and the solvent was removed under reduced pressure. The residue was purified by Chromatorex NH column chromatography

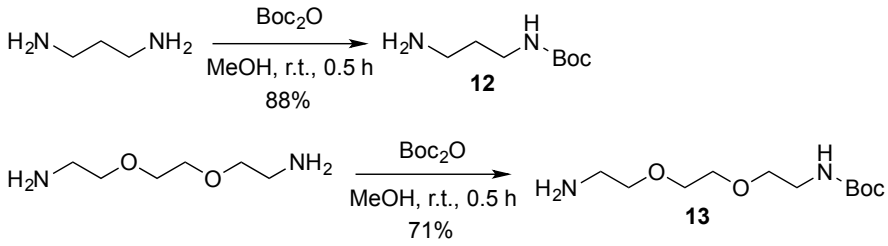
(AcOEt/*n*-hexane = 1/3) to afford **5** (70 mg, 0.32 mmol, 56%) as a yellow solid. **5**: ^{1}H-NMR ($CDCl_3$) δ: 0.97 (t, *J* = 7.5 Hz, 3H), 1.64 (tq, *J* = 7.2, 7.2 Hz, 2H), 3.06 (s, 3H), 3.18 (td, *J* = 5.0, 7.5 Hz, 2H), 4.61 (br s, 1H), 6.65 (dd, *J* = 2.0, 8.5 Hz, 1H), 6.92 (d, *J* = 3.0 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 1H); ^{13}C-NMR ($CDCl_3$) δ: 11.4, 22.2, 23.6, 45.2, 105.9, 115.4, 118.7, 124.8, 135.0, 153.3, 168.8, 169.1; HR-MS (ESI) Calcd. for $C_{12}H_{14}N_2O_2Na$: 241.0947, Found 241.0944.



Scheme S2. Synthesis of the AzPI unit of probe molecules.

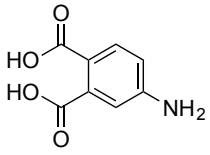


Scheme S3. Synthesis of the linker unit of **P2**.



Scheme S4. Synthesis of the linker units of **P1** and **P3**.

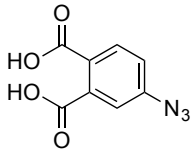
*Synthesis of 4-aminophthalic acid (**6**)*



10% Pd/C (50 mg) was added to a solution of 4-nitrophthalic acid (500 mg, 2.4 mmol) in MeOH (15 mL), and the mixture was stirred for 2 h at room temperature under a hydrogen atmosphere. The mixture was then filtrated through Celite pad and washed with MeOH. The solvent was removed under reduced

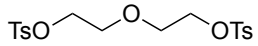
pressure to afford the product **6** (401 mg, 2.2 mmol, 93%) as an orange solid. **6**: ^{1}H-NMR (DMSO-d_6) δ: 6.00 (br s, 2H), 6.54–6.56 (m, 2H), 7.52 (d, J = 8.6 Hz, 1H); ^{13}C-NMR (DMSO-d_6) δ: 111.6, 113.0, 114.9, 131.6, 138.2, 152.1, 167.1, 170.6; HRMS (ESI) Calcd. for $C_8H_7NO_4Na$: 204.0267, Found 204.0260.

Synthesis of 4-azidophthalic acid (7)



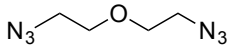
Sodium nitrate (57 mg, 0.83 mmol) in water (1.0 mL) was added to a solution of **6** (100 mg, 0.55 mmol) in 1,4-dioxane (8.0 mL) and 6*N* HCl aq. (6.0 mL) at 0 ºC. Sodium azide (54 mg, 0.83 mmol) in water (1.0 mL) was then added to the mixture at 0 ºC, and the resulting mixture was stirred at room temperature for 1 h and extracted with AcOEt. The combined organic layer was dried over Na_2SO_4, and the solvent was removed under reduced pressure to afford **7** (110 mg, 0.53 mmol, 96%) as a white solid. **7**: ^{1}H-NMR (CD_3OD) δ: 7.24 (d, J = 2.3 Hz, 1H), 7.27 (dd, J = 2.0, 7.7 Hz, 1H), 7.83 (d, J = 8.6 Hz, 1H); ^{13}C-NMR (CD_3OD) δ: 119.8, 121.6, 128.9, 132.3, 137.1, 144.8, 169.7, 170.8; HRMS (ESI) Calcd. for $C_8H_4N_3O_4Na$: 206.0196, Found 206.0184.

*Synthesis of 1,5-bis(p-tosyloxy)-3-oxapentane (**8**) (Compound **8** was prepared by a small modification of a known procedure.*[S3]*)*



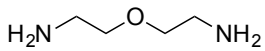
Tosyl chloride (2.9 g, 15 mmol) and triethylamine (1.4 mL, 10 mmol) were added to a solution of diethyleneglycol (531 mg, 5.0 mmol) in CH_2Cl_2 (20 mL), and the mixture was stirred at room temperature for 40 h. The resulting mixture was then added H_2O, and the whole was extracted with AcOEt. The combined organic layer was dried over Na_2SO_4, and the solvent was removed under reduced pressure. The residue was purified by silica-gel chromatography (AcOEt/*n*-hexane = 1/3) to afford **8** (942 mg, 2.3 mmol, 45%) as a white solid. **8**: ^{1}H-NMR ($CDCl_3$) δ: 2.43 (s, 6H), 3.59 (t, J = 4.6 Hz, 4H), 4.07 (t, J = 4.6 Hz, 4H), 7.33 (d, J = 8.0 Hz, 4H), 7.76 (d, J = 8.6 Hz, 4H); HRMS (ESI) Calcd. for $C_{18}H_2O_7S_2Na$: 437.0699, Found 403.0712.

*Synthesis of 1,5-diazido-3-oxapentane (**9**) (Compound **9** was prepared by a small modification of a known procedure.*[S4]*)*



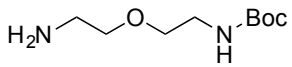
Sodium azide (488 mg, 7.5 mmol) was added to a solution of **8** in *N,N*-dimethylformamide (DMF) (10 mL), and the mixture was stirred at room temperature for 17 h. The resulting mixture was then added H_2O and extracted with AcOEt. The combined organic layer was washed with brine and dried over Na_2SO_4, and the solvent was removed under reduced pressure. The residue was purified by silica-gel chromatography (AcOEt/*n*-hexane = 1/6) to afford **9** (416 mg, 2.7 mmol, 89%) as a colorless oil. **9**: ^{1}H-NMR ($CDCl_3$) δ: 3.35 (t, *J* = 5.2 Hz, 4H), 3.62 (t, *J* = 5.2 Hz, 4H); ^{13}C-NMR ($CDCl_3$) δ: 50.5, 69.8; HRMS (ESI) Calcd. for $C_4H_9N_6O$: 157.0832, Found 157.0820.

*Synthesis of 1,5-diamino-3-oxapentane (**10**) (Compound **10** was prepared by a different method from the reported procedure.*[S5]*)*



10% Pd/C (57 mg) was added to a solution of **9** (381 mg, 2.4 mmol) in MeOH (10 mL), and the mixture was stirred at room temperature for 18 h under a hydrogen atmosphere. The mixture was then filtrated through Celite pad and washed with MeOH. The solvent was removed under reduced pressure to afford the product **10** (229 mg, 2.2 mmol, 90%) as a pale yellow oil. **10**: ^{1}H-NMR (CD_3OD) δ: 3.01 (t, *J* = 5.2 Hz, 4H), 3.64 (t, *J* = 4.9 Hz, 4H), 4.92 (br s, 4H); HRMS (ESI) Calcd. for $C_4H_{13}N_2O$: 105.1022, Found 105.1019.

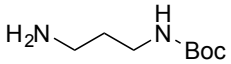
*Synthesis of N-tert-butoxycarbonyl-1,5-diamino-3-oxapentane (**11**) (Compound **11** was prepared by a small modification of a known procedure.*[S6]*)*



Di-*tert*-butyl dicarbonate (202 μL, 0.88 mmol) in MeOH (5.0 mL) was slowly added to a solution of **10** (229 mg, 2.2 mmol) in MeOH (5.0 mL) at 0 ºC. The mixture was stirred at 0 ºC for 30 min, and the solvent was removed under reduced pressure. The residue was added $NaHCO_3$ aq. and extracted with AcOEt. The combined organic layer was dried over Na_2SO_4, and the solvent was removed under reduced pressure. The residue was purified by silica-gel chromatography (AcOEt/*n*-hexane = 1/6) to afford **11** (94 mg, 0.46 mmol, 52%) as a pale yellow oil. **11**: ^{1}H-NMR ($CDCl_3$) δ: 1.42 (s, 9H), 2.84 (t, *J* = 5.5 Hz,

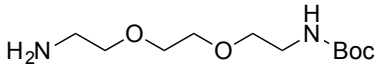
2H), 3.27–3.32 (m, 2H), 3.45 (t, *J* = 5.5 Hz, 2H), 3.49 (t, *J* = 5.2 Hz, 2H), 4.92 (br s, 1H); HRMS (ESI) Calcd. for $C_9H_{21}N_2O_3$: 205.1547, Found 205.1545.

*Synthesis of N-tert-butoxycarbonyl-1,3-diaminopropane (**12**) (Compound **12** was prepared by a small modification of a known procedure.*[S7]*)*

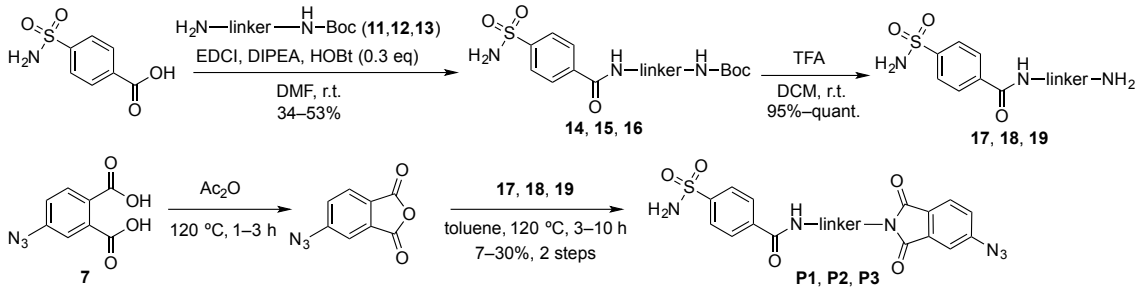


Di-*tert*-butyl dicarbonate (1.8 mL, 8.0 mmol) in MeOH (30 mL) was slowly added to a solution of 1,3-diaminopropane (2.0 mL, 24 mmol) in MeOH (50 mL) at 0 ºC. The mixture was stirred at 0 ºC for 1 h, and the solvent was removed under reduced pressure. The residue was added $NaHCO_3$ aq. and extracted with AcOEt. The combined organic layer was dried over Na_2SO_4, and the solvent was removed under reduced pressure to afford **12** (1.2 g, 7.0 mmol, 88%) as a pale yellow oil. **12**: ^{1}H-NMR ($CDCl_3$) δ: 1.41 (s, 9H), 1.58 (tt, *J* = 6.6, 6.6 Hz, 2H), 2.73 (t, *J* = 6.6 Hz, 2H), 3.18 (dt, *J* = 6.0 Hz, 2H), 4.89 (br s, 1H); HRMS (ESI) Calcd. for $C_8H_{18}N_2O_2$: 175.1441, Found 175.1449.

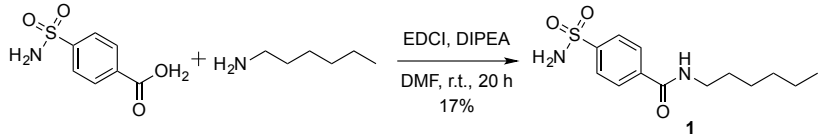
*Synthesis of N-tert-butoxycarbonyl-1,8-diamino-3,6-dioxaoctane (**13**) (Compound **13** was prepared by a different method from the reported procedure.*[S8]*)*



Di-*tert*-butyl dicarbonate (230 μL, 1.0 mmol) in MeOH (6.0 mL) was slowly added to a solution of 1,8-diamino-3,6-dioxaoctane (436 μL, 3.0 mmol) in MeOH (6.0 mL) at 0 ºC. The mixture was stirred at 0 ºC for 1 h, and the solvent was removed under reduced pressure. The residue was added H_2O and extracted with AcOEt. The combined organic layer was dried over Na_2SO_4, and the solvent was removed under reduced pressure to afford **13** (166 mg, 0.71 mmol, 71%) as a pale yellow oil. **13**: ^{1}H-NMR ($CDCl_3$) δ: 1.42 (s, 9H), 2.86 (t, *J* = 5.2 Hz, 2H), 3.28–3.33 (m, 2H), 3.49 (t, *J* = 5.2 Hz, 2H), 3.52 (t, *J* = 4.9 Hz, 2H), 3.58–3.61 (m, 2H); HRMS (ESI) Calcd. for $C_{11}H_{24}N_2O_4Na$: 271.1628, Found 271.1637.

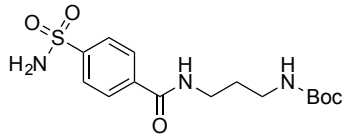


Scheme S5. Synthesis of the designed probes **P1**–**P3**.



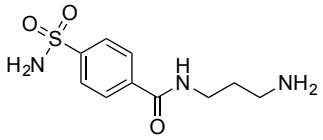
Scheme S6. Synthesis of the reported ligand **1**.

*Synthesis of N-(3-(tert-butoxycarbonylamino)propyl)-4-sulfamoylbenzamide (**14**)*



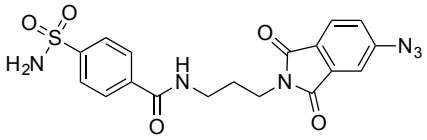
1-(3-Dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDCI) (288 mg, 1.5 mmol) and *N,N*-diisopropylethylamine (517 μL, 3.0 mmol) were added to a solution of 4-sulfamoylbenzoic acid (201 mg, 1.0 mmol) in anhydrous DMF (4.0 mL), and the mixture was stirred at room temperature for 15 min under an Ar atmosphere. Compound **12** (192 mg, 1.1 mmol) in DMF (1.0 mL) was then added to the mixture, and the mixture was stirred for 4 h at room temperature. The solvent was then removed under reduced pressure, and the residue was purified by silica-gel chromatography ($CHCl_3$/MeOH = 20/1 to 1/1) to afford **14** (57 mg, 0.16 mmol, 16%) as a white solid. **14**: ^{1}H-NMR (CD_3OD) δ: 1.42 (s, 9H), 1.76 (tt, *J* = 6.7, 6.7 Hz, 2H), 3.13 (dt, *J* = 6.3, 6.3 Hz, 2H), 3.42 (t, *J* = 6.9 Hz, 2H), 7.94–7.98 (m, 4H); ^{13}C-NMR (CD_3OD) δ: 28.8, 30.7, 38.5, 38.8, 80.1, 127.3, 128.9, 139.0, 147.6, 158.6, 168.8; HRMS (ESI) Calcd. for $C_{15}H_{22}N_3O_5S$: 356.1275, Found 356.1281.

*Synthesis of N-(3-aminopropyl)-4-sulfamoylbenzamide (**17**)*



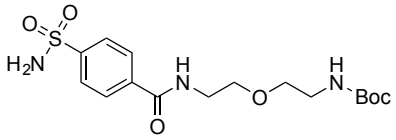
Trifluoroacetic acid (TFA) (122 μL, 1.6 mmol) was added dropwise to a solution of **14** (57 mg, 0.16 mmol) in CH_2Cl_2 (1.5 mL) at 0 ºC, and the mixture was stirred at room temperature for 6 h. The solvent and reagent were then removed under reduced pressure to afford the product **17** (62 mg, 0.17 mmol, quant.) as a yellow amorphous solid. **17**: ^{1}H-NMR (CD_3OD) δ: 1.94 (tt, *J* = 6.9, 6.9 Hz, 2H), 2.98 (t, *J* = 7.5 Hz, 2H), 3.47 (t, *J* = 6.3 Hz, 2H), 7.89–7.96 (m, 4H); ^{13}C-NMR (CD_3OD) δ: 28.7, 37.6, 38.3, 127.3, 129.0, 138.5, 147.7, 169.3; HRMS (ESI) Calcd. for $C_{10}H_{16}N_3O_3S$: 258.0907, Found 258.0906.

*Synthesis of N-(3-(4-sulfamoylbenzamido)propyl)-4-azidophthalimide (**P1**)*



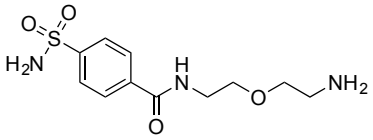
Compound **7** (34 mg, 0.17 mmol) in acetic anhydride (0.50 mL) was stirred at 120 ºC for 2.5 h. The solvents were then removed under reduced pressure, and the residue was dissolved in anhydrous toluene (0.50 mL). Compound **17** (58 mg, 0.15 mmol, TFA adduct) was added to the mixture, and the resulting mixture was refluxed for 10 h. The solvent was removed under reduced pressure, and the residue was purified by Chromatorex NH column chromatography ($CHCl_3$/MeOH = 50/1) to afford **P1** (7.6 mg, 0.018 mmol, 12%) as a white solid. **P1**: IR (KBr) 3327, 2926, 2124, 1770, 1715, 1630, 1550, 1469, 1443, 1339, 1306, 1166, 857, 802, 747 cm^{-1}; ^{1}H-NMR (DMSO-d_6) δ: 1.87 (tt, *J* = 7.0, 7.0 Hz, 2H), 3.29 (dt, *J* = 5.5, 6.9 Hz, 2H), 3.62 (t, *J* = 7.2 Hz, 2H), 7.47 (br s, 2H), 7.48 (dd, *J* = 1.7, 8.0 Hz, 1H), 7.53 (d, *J* = 1.8 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.87 (d, *J* = 8.1 Hz, 2H), 7.94 (d, *J* = 8.6 Hz, 2H), 8.66 (t, *J* = 5.7 Hz, 1H); ^{13}C-NMR (DMSO-d_6) δ: 28.4, 36.2, 37.6, 114.3, 124.9, 125.3, 126.1, 128.1, 128.3, 134.4, 137.9, 146.5, 146.7, 165.7, 167.6, 167.7; HRMS (ESI) Calcd. for $C_{18}H_{16}N_6O_5SNa$: 451.0795, Found 451.0796.

*Synthesis of N-tert-butoxycarbonyl-2-(2-(4-sulfamoylbenzamido)ethoxy)ethylamine (**15**)*



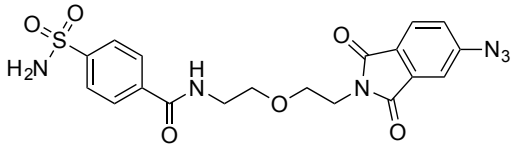
EDCI (234 mg, 1.2 mmol), *N,N*-diisopropylethylamine (419 μL, 2.4 mmol) and 1-hydroxybenzotriazole (HOBt) (33 mg, 0.24 mmol) were added to a solution of 4-sulfamoylbenzoic acid (163 mg, 0.81 mmol) in anhydrous DMF (4.0 mL), and the mixture was stirred at room temperature for 15 min under an Ar atmosphere. Compound **11** (165 mg, 0.81 mmol) in DMF (1.0 mL) was then added to the mixture, and the resulting mixture was further stirred for 24 h at room temperature. The solvent was then removed under reduced pressure, and the residue was purified by silica-gel chromatography ($CHCl_3$/MeOH = 20/1) to afford **15** (113 mg, 0.31 mmol, 38%) as a white solid. **15**: ^{1}H-NMR (CD_3OD) δ: 1.35 (s, 9H), 3.18 (t, *J* = 5.2 Hz, 2H), 3.47 (t, *J* = 5.7 Hz, 2H), 3.53 (t, *J* = 5.4 Hz, 2H), 3.59 (t, *J* = 5.2 Hz, 2H), 7.88–7.94 (m, 4H); ^{13}C-NMR (CD_3OD) δ: 28.8, 41.0, 41.3, 70.3, 70.9, 80.1, 139.0, 147.6, 158.5, 168.9; HRMS (ESI) Calcd. for $C_{16}H_{25}N_3O_6SNa$: 410.1356, Found 410.1352.

*Synthesis of 2-(2-(4-sulfamoylbenzamido)ethoxy)ethylamine (**18**)*



TFA (237 μL, 3.1 mmol) was added dropwise to a solution of **15** (113 mg, 0.31 mmol) in CH_2Cl_2 (3.0 mL) at 0 ºC, and the mixture was stirred at room temperature for 24 h. The solvent and reagent were then removed under reduced pressure to afford the product **18** (118 mg, 0.29 mmol, 95%) as a yellow oil. **18**: ^{1}H-NMR (CD_3OD) δ: 3.04–3.15 (m, 2H), 3.58 (t, *J* = 4.6 Hz, 2H), 3.58–3.74 (m, 4H), 7.85–8.03 (m, 4H); ^{13}C-NMR (CD_3OD) δ: 40.6, 40.7, 67.5, 70.7, 127.3, 129.0, 138.8, 147.5, 169.0; HRMS (ESI) Calcd. for $C_{11}H_{18}N_3O_4S$: 288.1013, Found 288.1011.

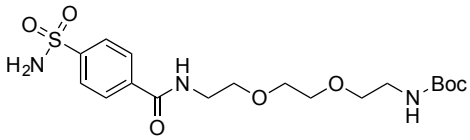
*Synthesis of N-(2-(2-(4-sulfamoylbenzamido)ethoxy)ethyl)-4-azidophthalimide (**P2**)*



Compound **7** (46 mg, 0.22 mmol) in acetic anhydride (0.60 mL) was stirred at 120 ºC for 1 h. The solvents were then removed under reduced pressure, and the residue was dissolved in anhydrous toluene

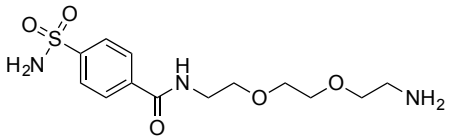
(0.50 mL). Compound **18** (60 mg, 0.15 mmol, TFA adduct) was added to the mixture, and the resulting mixture was refluxed for 3.5 h. The solvent was removed under reduced pressure, and the residue was purified by Chromatorex NH column chromatography ($CHCl_3$/MeOH = 100/1) to afford **P2** (7.2 mg, 0.016 mmol, 7.0%) as a white solid. **P2**: IR (KBr) 3358, 3087, 2923, 2122, 1767, 1703, 1660, 1611, 1556, 1397, 1343, 1302, 1157, 1122, 913, 849, 803, 746 cm^{-1}; ^{1}H-NMR (DMSO-d_6) δ: 3.32–3.38 (m, 2H), 3.54 (t, J = 5.5 Hz, 2H), 3.63 (t, J = 5.2 Hz, 2H), 3.73 (t, J =5.2 Hz, 2H), 7.41–7.49 (m, 2H), 7.75 (d, J = 7.5 Hz, 1H), 7.82 (d, J = 8.1 Hz, 2H), 7.86 (d, J = 8.6 Hz, 2H), 8.56 (br s, 1H); ^{13}C-NMR (DMSO-d_6) δ: 37.2, 37.2, 66.7, 68.2, 113.7, 124.4, 124.7, 125.5, 127.4, 127.7, 133.7, 137.1, 146.0, 146.1, 165.1, 166.9, 167.0; HRMS (ESI) Calcd. for $C_{19}H_{18}N_6O_6SNa$: 481.0901, Found 481.0901.

*Synthesis of N-tert-butoxycarbonyl-2-(2-(2-(4-sulfamoylbenzamido)ethoxy)ethoxy)ethylamine (**16**) (Compound **15** was prepared by a different method from the reported procedure.*[S9]*)*



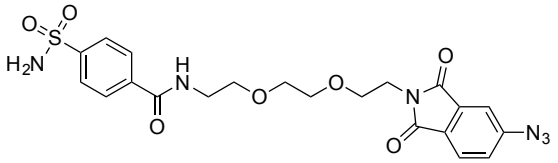
EDCI (288 mg, 1.5 mmol), *N,N*-diisopropylethylamine (517 μL, 3.0 mmol) and HOBt (41 mg, 0.30 mmol) were added to a solution of 4-sulfamoylbenzoic acid (201 mg, 1.0 mmol) in anhydrous DMF (4.0 mL), and the mixture was stirred at room temperature for 15 min under an Ar atmosphere. Compound **13** (258 mg, 1.1 mmol) in DMF (1.0 mL) was then added to the mixture, and the resulting mixture was further stirred for 24 h at room temperature. The solvent was then removed under reduced pressure, and the residue was purified by silica-gel chromatography ($CHCl_3$/MeOH = 20/1) to afford **16** (148 mg, 0.34 mmol, 34%) as a colorless oil. **16**: ^{1}H-NMR ($CDCl_3$) δ: 1.31 (s, 9H), 3.07–3.18 (m, 2H), 3.33–3.43 (m, 2H), 3.41–3.60 (m, 6H), 3.59 (t, J = 4.6 Hz, 2H), 5.15 (br s, 1H), 6.28 (br s, 2H), 7.43 (br s, 1H), 7.62–7.75 (m, 4H); ^{13}C-NMR ($CDCl_3$) δ: 28.2, 39.8, 40.0, 69.2, 69.9, 69.9, 69.9, 79.3, 126.1, 127.7, 137.5, 144.9, 156.1, 166.5; HRMS (ESI) Calcd. for $C_{18}H_{29}N_3O_7SNa$: 454.1618, Found 454.1596.

*Synthesis of 2-(2-(2-(4-sulfamoylbenzamido)ethoxy)ethoxy)ethylamine (**19**)* (*Compound **19** was prepared by a small modification of the reported procedure*.[S9])



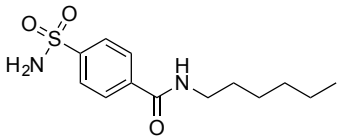
TFA (262 μL, 3.4 mmol) was added dropwise to a solution of **16** (148 mg, 0.34 mmol) in CH_2Cl_2 (3.0 mL) at 0 ºC, and the mixture was stirred at room temperature for 10 h. The solvent and reagent were then removed under reduced pressure to afford the product **19** (170 mg, 0.38 mmol, quant.) as a yellow oil. **19**: ^{1}H-NMR (CD_3OD) δ: 3.02–3.09 (m, 2H), 3.52–3.59 (m, 2H), 3.60–3.72 (m, 8H), 7.88–7.96 (m, 4H); ^{13}C-NMR (CD_3OD) δ: 40.6, 40.8, 67.8, 70.5, 71.3, 71.3, 127.3, 129.0, 138.9, 147.6, 169.0; HRMS (ESI) Calcd. for $C_{13}H_{22}N_3O_5S$: 332.1275, Found 332.1248.

*Synthesis of N-(2-(2-(2-(4-sulfamoylbenzamido)ethoxy)ethoxy)ethyl)-4-azidophthalimide (**P3**)*

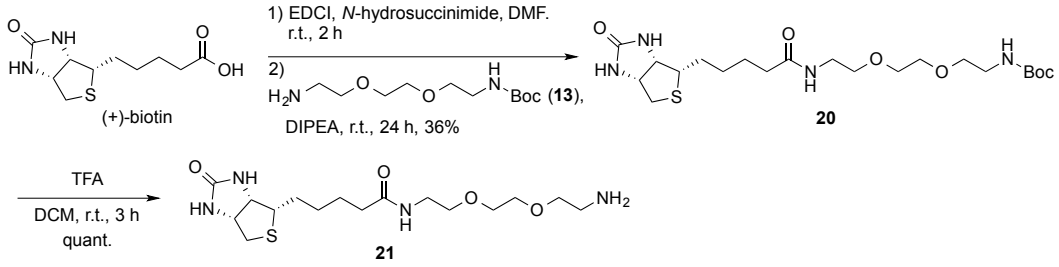


Compound **7** (34 mg, 0.17 mmol) in acetic anhydride (0.50 mL) was stirred at 120 ºC for 3 h. The solvents were then removed under reduced pressure, and the residue was dissolved in anhydrous toluene (0.50 mL). Compound **19** (67 mg, 0.15 mmol, TFA adduct) was added to the mixture, and the resulting mixture was refluxed for 3 h. The solvent was removed under reduced pressure, and the residue was purified by Chromatorex NH column chromatography ($CHCl_3$/MeOH = 50/1) to afford **P3** (23 mg, 0.046 mmol, 30%) as a white solid. **P3**: IR (KBr) 3364, 3334, 3169, 3087, 2873, 2129, 1769, 1703, 1638, 1621, 1554, 1479, 1445, 1397, 1337, 1164, 1118, 908, 856, 745 cm^{-1}; ^{1}H-NMR (DMSO-d_6) δ: 3.32–3.39 (m, 2H), 3.44–3.48 (m, 4H), 3.50–3.52 (m, 2H), 3.60 (t, J = 6.0 Hz, 2H), 3.72 (t, J = 6.0 Hz, 2H), 7.46 (br s, 1H), 7.48 (dd, J = 2.3, 8.1 Hz, 1H), 7.54 (d, J = 1.7 Hz, 1H), 7.85 (d, J = 8.6 Hz, 1H), 7.87 (d, J = 8.6 Hz, 2H), 7.96 (d, J = 8.6 Hz, 2H), 8.64 (t, J = 5.7 Hz, 1H), ^{13}C-NMR (DMSO-d_6) δ: 37.2, 37.2, 66.9, 68.7, 69.4, 69.5, 113.9, 124.5, 124.8, 125.6, 127.4, 127.8, 133.7, 137.2, 146.1, 146.2, 165.2, 166.9, 167.0; HRMS (ESI) Calcd. for $C_{21}H_{22}N_6O_7SNa$: 525.1163, Found 525.1167.

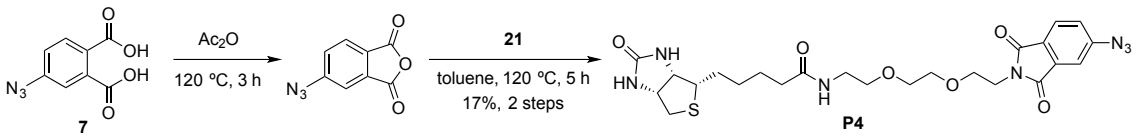
*Synthesis of N-hexyl-4-sulfamoylbenzamide (**1**)*



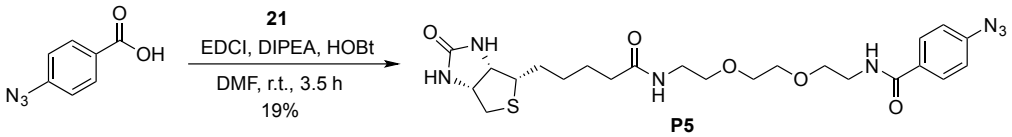
EDCI (144 mg, 0.75 mmol), *N,N*-diisopropylethylamine (259 μL, 1.5 mmol) and *n*-hexylamine (73 μL, 0.55 mmol) were added to a solution of 4-sulfamoylbenzoic acid (101 mg, 0.50 mmol) in anhydrous DMF (5.0 mL), and the mixture was stirred at room temperature for 20 h under an Ar atmosphere. The solvent was then removed under reduced pressure, and the residue was purified by silica-gel chromatography ($CHCl_3$/MeOH = 30/1 to 1/1) to afford **1** (24 mg, 0.084 mmol, 17%) as a white solid. **1**: ^{1}H-NMR (CD_3OD) δ: 0.91 (t, *J* = 6.9 Hz, 3H), 1.32–1.41 (m, 6H), 1.62 (tt, *J* = 7.2, 7.2 Hz, 2H), 3.37 (t, *J* = 7.2 Hz, 2H), 7.91–7.97 (m, 4H); ^{13}C-NMR (CD_3OD) δ: 14.4, 23.7, 27.8, 30.4, 32.7, 41.2, 127.3, 128.9, 139.2, 147.6, 168.7; HRMS (ESI) Calcd. for $C_{13}H_{20}N_2O_3SNa$: 307.1087, Found 307.1092.



Scheme S7. Synthesis of the biotin-linker conjugated unit.

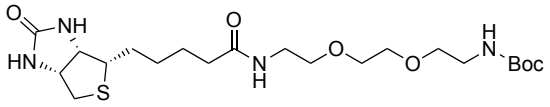


Scheme S8. Synthesis of biotin–AzPI conjugate **P4**.



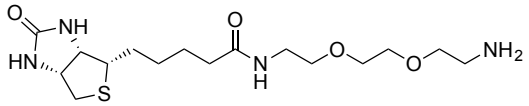
Scheme S9. Synthesis of non-AzPI probe **P5**.

*Synthesis of N-tert-butoxycarbonyl-2-(2-(2-(biotinylamino)ethoxy)ethoxy)ethylamine (**20**) (Compound **20** was prepared by a different method from the reported procedure.* [S8]*)*



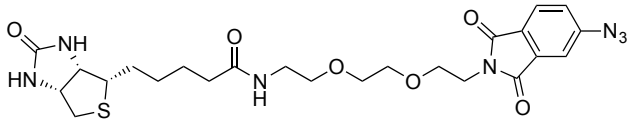
N-Hydroxysuccinimide (90 mg, 0.78 mmol) and EDCI (205 mg, 1.1 mmol) were added to a solution of (+)-biotin (173 mg, 0.71 mmol) in anhydrous DMF (3.0 mL), and the mixture was stirred at room temperature for 2 h under an Ar atmosphere. Compound **13** (166 mg, 0.71 mmol) in DMF (2.0 mL) and *N*,*N*-diisopropylethylamine (367 μL, 2.1 mmol) were then added, and the resulting mixture was stirred at room temperature for 24 h. After removal of the solvent, the residue was purified by silica-gel chromatography ($CHCl_3$/MeOH = 10/1) to afford **20** (121 mg, 0.26 mmol, 36%) as a white solid. **20**: ^{1}H-NMR ($CDCl_3$) δ: 1,28–1.37 (m, 11H), 151–1.62 (m, 4H), 2.11 (t, *J* = 7.5 Hz, 2H), 2.62 (d, *J* = 12.6 Hz, 1H), 2.78 (dd, *J* = 4.9, 12.9 Hz, 1H), 3.02 (t, *J* = 5.7 Hz, 2H), 3.18 (t, *J* = 4.6 Hz, 2H), 3.31 (td, *J* = 5.0, 5.0 Hz, 2H), 3.42–3.46 (m, 4H), 3.47–3.53 (m, 4H), 4.18 (dd, *J* = 6.0, 6.0 Hz, 1H), 4.38 (dd, *J* = 6.3 Hz, 1H), 5.25 (br s, 1H), 6.07 (br s, 1H), 6.67 (br s, 1H), 6.94 (br s, 1H); ^{13}C-NMR ($CDCl_3$) δ: 25.3, 27.8, 28.1, 35.6, 38.8, 39.9, 40.2, 55.5, 59.8, 60.0, 61.4, 61.6, 69.6, 69.7, 69.8, 78.9, 155.9, 164.0, 173.4; HRMS (ESI) Calcd. for $C_{21}H_{38}N_4O_6SNa$: 497.2404, Found 497.2401.

*Synthesis of 2-(2-(2-(biotinylamino)ethoxy)ethoxy)ethylamine (**21**) (Compound **21** was prepared by a different method from the reported procedure.*[S8]*)*



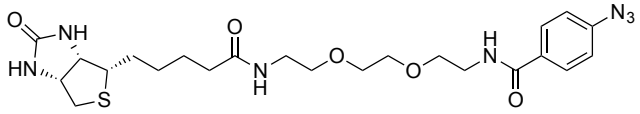
TFA (200 μL, 2.6 mmol) was added dropwise to a solution of the compound **20** (121 mg, 0.26 mmol) in CH_2Cl_2 (2.0 mL) at 0 ºC, and the mixture was stirred at room temperature for 6 h. The solvent and reagent were then removed under reduced pressure to afford the product **21** (129 mg, 0.26 mmol, quant.) as a yellow oil. **21**: ^{1}H-NMR (CD_3OD) δ: 1.31–1.48 (m, 4H), 1.50–1.72 (m, 4H), 2.17 (t, *J* = 7.2 Hz, 2H), 2.70 (d, *J* = 12.6 Hz, 1H), 3.01 (dd, *J* = 4.6, 12.6 Hz, 1H), 3.02–3.11 (m, 2H), 3.11–3.22 (m, 1H), 3.29–3.38 (m, 2H), 3.46–3.55 (m, 2H), 3.57–3.64 (m, 4H), 3.62–3.69 (m, 2H), 4.27 (dd, *J* = 4.0, 4.0 Hz, 1H), 4.46 (dd, *J* = 5.2, 5.2 Hz, 1H); ^{13}C-NMR (CD_3OD) δ: 26.6, 29.2, 29.5, 36.5, 39.9, 40.4, 40.9, 56.8, 61.4, 63.2, 67.6, 70.5, 71.0, 71.1, 165.8, 176.0; HRMS (ESI) Calcd. for $C_{16}H_{30}N_4O_4SNa$: 397.1880, Found 397.1872.

Synthesis of N-(2-(2-(2-(biotinylamino)ethoxy)ethoxy)ethyl)-4-azidophthalimide (**P4**)



Compound **7** (21 mg, 0.10 mmol) in acetic anhydride (0.50 mL) was stirred at 120 ºC for 3 h. The solvents were then removed under reduced pressure, and the residue was dissolved in anhydrous toluene (0.50 mL). Compound **21** (49 mg, 0.10 mmol, TFA adduct) was added to the mixture, and the resulting mixture was refluxed for 5 h. The solvent was removed under reduced pressure, and the residue was purified by Chromatorex NH column chromatography ($CHCl_3$/MeOH = 70/1) to afford **P4** (9.3 mg, 0.017 mmol, 30%) as a white solid. **P4**: IR (KBr) 3291, 2929, 2123, 1771, 1705, 1646, 1395, 1305, 1120, 854, 745 cm^{-1}; ^{1}H-NMR ($CDCl_3$) δ: 1.42 (tt, *J* = 7.6, 7.6 Hz, 2H), 1.60–1.76 (m, 4H), 2.22 (t, *J* = 8.3 Hz, 2H), 2.69 (d, *J* = 13.2 Hz, 1H), 2.87 (dd, *J* = 4.9, 4.9 Hz, 1H), 3.12 (td, *J* = 6.5 Hz, 1H), 3.30–3.40 (m, 2H), 3.47 (t, *J* = 5.2 Hz, 2H), 3.51–3.55 (m, 2H), 3.58–3.61 (m, 2H), 3.71 (t, *J* = 5.7 Hz, 2H), 3.87 (t, *J* = 5.7 Hz, 2H), 4.28 (dd, *J* = 5.1, 6.9 Hz, 1H), 4.46 (dd, *J* = 4.9, 7.7 Hz, 1H), 5.41 (br s, 1H), 6.34 (br s, 1H), 6.72 (t, *J* = 5.2 Hz, 1H), 7.29 (dd, *J* = 1.7, 8.0 Hz, 1H), 7.45 (d, *J* = 2.3 Hz, 1H), 7.79 (d, *J* = 8.1 Hz, 1H); ^{13}C-NMR ($CDCl_3$) δ: 25.6, 28.1, 35.9, 37.4, 39.0, 40.5, 55.5, 60.1, 61.7, 67.8, 69.8, 69.9, 70.0, 113.8, 124.0, 125.0, 127.8, 134.2, 146.7, 163.8, 167.4, 167.5, 173.3; HRMS (ESI) Calcd. for $C_{24}H_{31}N_7O_6SNa$: 568.1949, Found 568.1971.

Synthesis of 4-azido-N-(2-(2-(2-(biotinylamino)ethoxy)ethoxy)ethyl)benzamide (**P5**)



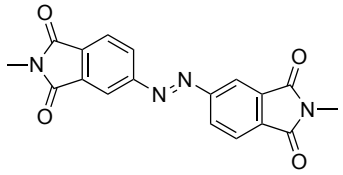
EDCI (39 mg, 0.20 mmol), *N,N*-diisopropylethylamine (70 μL, 0.41 mmol) and HOBt (5.5 mg, 0.040 mmol) were added to a solution of 4-azidobenzoic acid (22 mg, 0.14 mmol) in anhydrous DMF (1.0 mL), and the mixture was stirred at room temperature for 5 min under an Ar atmosphere. Compound **21** (73 mg, 0.15 mmol) in DMF (0.5 mL) was then added to the mixture, and the resulting mixture was stirred for 3.5 h at room temperature. The solvent was then removed under reduced pressure, and the residue was purified by silica-gel chromatography ($CHCl_3$/MeOH = 15/1 to 10/1) to afford **P5** (13 mg, 0.025 mmol, 19%) as a white solid. **P5**: IR (KBr) 3296, 2930, 2865, 2126, 1704, 1646, 1542, 1499, 1283, 1127,

847, 763 cm^{-1}; ^{1}H-NMR ($CDCl_3$) δ: 1.37 (tt, *J* = 7.6, 7.6 Hz, 2H), 1.53–1.69 (m, 4H), 2.16 (t, *J* = 7.9 Hz, 2H), 2.69 (d, *J* = 13.2 Hz, 1H), 2.85 (dd, *J* = 5.2, 13.2 Hz, 1H), 3.07 (td, *J* = 6.5, 6.5 Hz, 1H), 3.38 (t, *J* = 4.6 Hz, 2H), 3.53 (t, *J* = 5.2 Hz, 2H), 3.56–3.65 (m, 6H), 3.65 (t, *J* = 6.3 Hz, 2H), 4.23 (dd, *J* = 5.2, 7.5 Hz, 1H), 4.44 (dd, *J* = 5.2, 7.5 Hz, 1H), 5.72 (br s, 1H), 6.72 (br s, 1H), 6.76 (br s, 1H), 7.03 (d, *J* = 8.6 Hz, 2H), 7.26 (br s, 1H), 7.83 (d, *J* = 8.6 Hz, 1H); ^{13}C-NMR ($CDCl_3$) δ: 25.5, 28.0, 28.1, 35.9, 39.1, 39.9, 40.4, 55.6, 60.2, 61.7, 69.8, 69.9, 70.1, 118.9, 128.9, 130.8, 143.2, 164.2, 166.6, 173.4; HRMS (ESI) Calcd. for $C_{23}H_{33}N_7O_5SNa$: 542.2156, Found 542.2146.

2-3. Photoreaction conditions

A solution of **1** (10 mg, 0.050 mmol) in DMSO-d_6 (0.75 mL) was irradiated using 365 nm LED light (HLV-24UV365-4WNRBT, CCS Inc., 525 mW/cm^2) for 1 h at 0 ºC in the presence/absence of diethylamine (507 μL, 5.0 mmol). The resulting mixture was then added H_2O and extracted with AcOEt. After removal of the solvent, the residue was purified by silica-gel chromatography (AcOEt/*n*-hexane). The yields of the isolated products are shown in Table S1.

*1,2-Bis(N-methylphthalimid-4-yl)diazene (**3**)*



Compound **3** was obtained as an orange solid. **3**: ^{1}H-NMR ($CDCl_3$) δ: 3.23 (s, 6H), 8.03 (d, *J* = 8.0 Hz, 2H), 8.31 (dd, *J* = 1.4, 7.7 Hz, 2H), 8.36 (d, *J* = 1.2 Hz, 2H); ^{13}C-NMR ($CDCl_3$) δ: 24.3, 116.2, 124.4, 130.7, 133.6, 134.2, 155.8, 167.4, 167.5; HRMS (ESI) Calcd. for $C_{18}H_{12}N_4O_4Na$: 371.0751, Found 371.0732.

2-4. Spectrometric analyses

Methanol (for spectrochemical analysis) was purchased from Wako Pure Chemical Industries, Ltd.. The absorption spectra were recorded on a Shimadzu UV-2400PC spectrometer equipped with a 50 W halogen lamp and a deuterium lamp (slit width: 0.2 nm). The fluorescence measurements were performed on a JASCO FP-6500 equipped with a 150 W xenon lamp (slit width: 3 nm). A 1.00 cm

quartz cell was used for the measurements.

3. CA-II-Inhibitory Activity Assay

Evaluation of enzymatic activity of CA-II[S10]

The hydrolytic (esterase) activity of CA-II was evaluated using *p*-nitrophenyl acetate (*p*-NPA). The initial rates of *p*-NPA hydrolysis were determined in Tris-HCl buffer (50 mM, pH 8.0) at 25 ºC by measuring the increase of absorbance at 355 nm (EnVision, Perkin Elmer). The Michaelis-Menten constant (K_m) was obtained by fitting plots of initial rate versus *p*-NPA concentration. Measurement conditions were as follows: 500 nM CA-II, 0.03–3 mM *p*-nitrophenyl acetate, 50 mM Tris-HCl buffer (pH 8.0). The K_m value was 1.45 mM.

Evaluation of CA-II-inhibitory activity of the chemical probes

CA-II (1 μM) was incubated with test compound (**1**, **P1**, **P2** or **P3**; concentration: 0.06–20 μM) for 30 min at room temperature in Tris-HCl buffer (50 mM, pH 8.0). To this mixture (50 μL/well in 96-well plate) was added *p*-NPA (6 mM in Tris-HCl buffer) (50 μL/well in 96 well plate), and the absorbance at 355 nm was measured for the initial 5 min (final concentration: 500 nM CA-II, 0.03–10 μM test compound, and 3 mM *p*-NPA). The inhibition constant (K_i) was determined as the concentration that inhibited 50% of the hydrolysis of *p*-NPA. The IC_{50} values were calculated from the equation below[S11].

$$K_i = IC_{50}/(1+[S]/K_m) \qquad ([S]: \text{substrate concentration})$$

4. Protein Labeling

4-1. Material preparations

Streptavidin (from *Streptomyces avidinii*, >90%, 10–20 units/mg) and CAII (carbonic anhydrase isozyme II from bovine erythrocytes, ≥ 3,000 W-A units/mg protein) were purchased from Sigma-Aldrich Co., LLC. These proteins were used without further purification. Streptavidin and CAII were dissolved in MilliQ water, and the concentrations were determined by measuring the absorbance at 280 nm. The HEK-293 cell lysate was prepared as follows. HEK293 cells were cultured in Dulbecco's modified Eagle's medium (DMEM) containing 10% fetal bovine serum (FBS) and penicillin/streptomycin at 37 ºC in a humidified atmosphere of 5% CO_2. The cell pellet was lysed with solubilization buffer (20 mM Hepes-Na (pH 7.0), 1.0 mM ethylenediaminetetraacetic acid (EDTA), 0.5% Triton X-100, and complete mini (Roche)) (15 min on ice), and the crude lysate was centrifuged at 15,000 g for 10 min at 4 ºC. The supernatant was collected and used as HEK293 cell lysate.

4-2. Labeling procedures

General method for labeling of CA-II

CA-II was incubated with ligand–AzPI conjugate in 20 mM phosphate buffer (100 μL, pH 7.0) for 30 min at 0 ºC. For the competition analysis, CA-II was pre-incubated with compound **1** at 0 ºC for 30 min before addition of ligand–AzPI conjugate. The samples were irradiated at 365 nm with an LED lamp (HLV-24UV365-4WNRBT, CCS Inc., 612 mW/cm^2) for 5 min at 0 ºC, and then mixed with 5x SDS-PAGE loading buffer (250 mM Tris, pH 6.8, 10% SDS, 50% glycerol, 8% 1,4-dithiothreitol, and 1 μg/mL bromophenol blue), heated at 98 ºC for 20 min, and subjected to SDS-polyacrylamide gel electrophoresis (PAGE) (10–20% acrylamide, Wako Pure Chemical Industries, Ltd.). The gel was visualized with an in-gel fluorescence imager (ATTO Ez-Capture II) (λ_{em}> 540 nm, diaphragm: 3.0, exposure time: 5 s) equipped with WSE-5500 VariRays (λ_{ex}: 440–500 nm, peak: 470 nm), and stained with Commassie Brilliant Blue (See Pico^{TM} CBB Stain Kit).

Labeling of CA-II in the presence of HEK293 cell lysate

To a mixture of CA-II (0.015 mg/mL) and HEK293 cell lysate (final concentration: 0.72 mg/mL) in phosphate buffer (20 mM, pH 7.0) was added ligand–AzPI conjugate **P3**, and the sample was incubated at 0 ºC for 30 min. For competition analysis, the sample was pre-incubated with compound **1** (30 min, 0

ºC) before addition of **P3**. The labeling was performed under UV irradiation at 365 nm for 5 min (0 ºC).

General method for the labeling of streptavidin (Chemiluminescence detection of labeled streptavidin)

Streptavidin was incubated with probe for 30 min at 0 ºC in 20 mM phosphate buffer (100 μL, pH 7.0). For the competition analysis, streptavidin was pre-incubated with biotin (30 min, 0 ºC) before addition of the probe molecule. The sample was then irradiated at 365 nm (5 min, 0 ºC), mixed with 5x SDS-PAGE loading buffer and heated at 120 ºC for 20 min before SDS-PAGE. For chemiluminescence detection, the proteins on the gel were transferred to PVDF film (Immobilon®-P). After blocking with 1% BSA in 1x Tris-buffered saline with Tween 20 (TBST) buffer, the film was incubated in a solution of Immunopure® Streptavidin-HRP Conjugated (Thermo Fishcher Scientific Inc.) in MilliQ water (diluted 1/15,000) at room temperature for 1 h, and the film was washed with 1x TBST buffer. A solution of luminol and H_2O_2 (Immobilon™ Western Chemluminescent HRP substrate) was added to the film, and luminescence was monitored with an ATTO Ez-Capture (diaphragm: 0.95, exposure time: 1 min).

Isolation of bovine red blood cells[S12]

Bovine whole blood (defibrinated, 4.0 mL), purchased from COSMO BIO Co., Ltd., was mixed with EDTA (3.0 mg) and saline (0.9%(w/v), 4mL). This blood sample was centrifuged at 5,000 g for 5 min, and the heaviest red phase was collected as purified RBCs.

CA-II labeling in living RBCs

Living RBCs (2 μL) in 1x HEPES-buffered saline (100 μL) were incubated with/without compound **1** at room temperature for 30 min. Probe (**2**, **P3**) was then added, and the samples were incubated at room temperature for 30 min. They were then irradiated at 365 nm (5 min, 0 ºC), and 20 μL of lysis buffer (50 mM Tris buffer (pH 7.4), 150 mM NaCl, 2.5 mM EDTA and 1% Nonidet P-40) was added. After centrifugation at 15,000 g for 10 min, the supernatants were collected and mixed with 5x SDS-PAGE loading buffer. The samples were heated at 98 ºC for 20 min and subjected to SDS-PAGE. Bands were visualized with an in-gel fluorescence imager (λ_{em} >540 nm diaphragm: 3.0, exposure time: 5 s) equipped with WSE-5500 VariRays.

5. Mass Spectrometric Analysis

Mass spectrometric analysis of whole protein

CA-II (1 μM) was photo-irradiated (365 nm) in phosphate buffer (pH = 7.0) with/without **P3** (1 μM) at 0 ºC for 5 min. The photo-irradiated samples were then concentrated by using Amicon centrifugal filter, and the resultants were briefly purified using ZipTip (C18) eluting with 50% MeCN aq. containing 0.1% TFA. For these samples, the mass spectra were obtained using a BRUKER micrOTOF II mass spectrometer.

In-solution digestion

To each sample (100 ng/μL, 10 μL) was added 10 μL of 2,2,2-trifluoroethanol (TFE) and after incubation for 30 min, 80 μL of 50 mM Tris-HCl (pH 8.5) was further added.[S13] Trypsin (Promega, Sequencing Grade Modified Trypsin, V5113) was then added in a ratio of 1:10 (enzyme/protein), and incubation was continued for overnight at 37 ºC.

Mass spectrometry (LC-MS/MS analysis)

Mass spectra were acquired using an LTQ Orbitrap XL equipped with an electrospray ionization (ESI) source (Thermo Fisher Scientific). For representative LC-MS/MS analysis with data-dependent acquisition (DDA), full MS scans were acquired with an *m/z* range of 350-1600 in the Orbitrap mass analyzer (resolution 60,000 at *m/z* 400) and MS/MS spectra were acquired in the linear ion trap mass analyzer. Fragmentation was performed with collision-induced dissociation (CID). For detailed MS/MS acquisition of the probe-modified peptides, CID or higher-energy collision dissociation (HCD) was applied in the Orbitrap (resolution 15,000 at *m/z* 400).

In the nano-flow HPLC system (UltiMate 3000 nanoLC system, Thermo Fisher Scientific), a spray tip column (NTCC-360/75-3 Nikkyo Technos) and a μ-precolumn (PepMap100 C18, 300 μm i. d. x 5 mm, Thermo Fisher) were used as analytical and trap columns, respectively. For the analytical column, solvent A (distilled water/MeCN (100:4) containing 0.1% formic acid) and solvent B (MeCN containing 0.1% formic acid) were used.

Data analysis

LC-MS/MS data were analyzed with Proteome Discoverer 1.3 (Thermo Scientific) using the SEQUEST

search algorithm. The following parameters were used: precursor mass tolerance 5 ppm, fragment mass tolerance 0.8 Da (MS2 in LTQ), enzyme was trypsin, maximum number of missed cleavage sites was 3, dynamic modifications were N-terminal acetylation (+42.010565 Da) and oxidation on methionine (+15.994915 Da), and chemical modification on any amino acid (+474.120920 Da). UniProt bovine CA-II amino acid sequence (P00921 without the initiator methionine) was used for a searching. Peptide assignments were filtered to a false discovery rate (FDR) below 1% using Peptide Validator in Proteome Discoverer version 1.3. Simulation of *m/z* values and product ion peaks was performed using ProteinProspector (http://prospector.ucsf.edu/prospector/cgi-bin/msform.cgi?form=msproduct). For in-silico fragmentation of the probe-modified peptide, the sum of the elemental compositions of amino acids and the probe ($C_{21}H_{22}N_4O_7S$) was used.

6. Supplementary References

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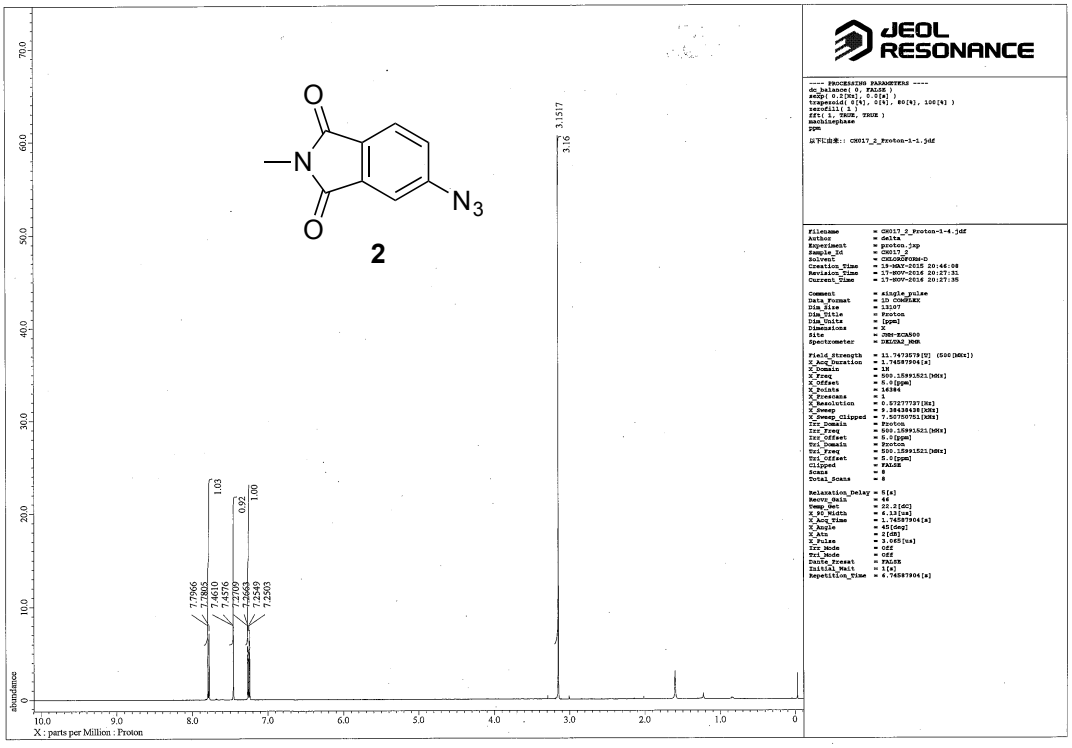
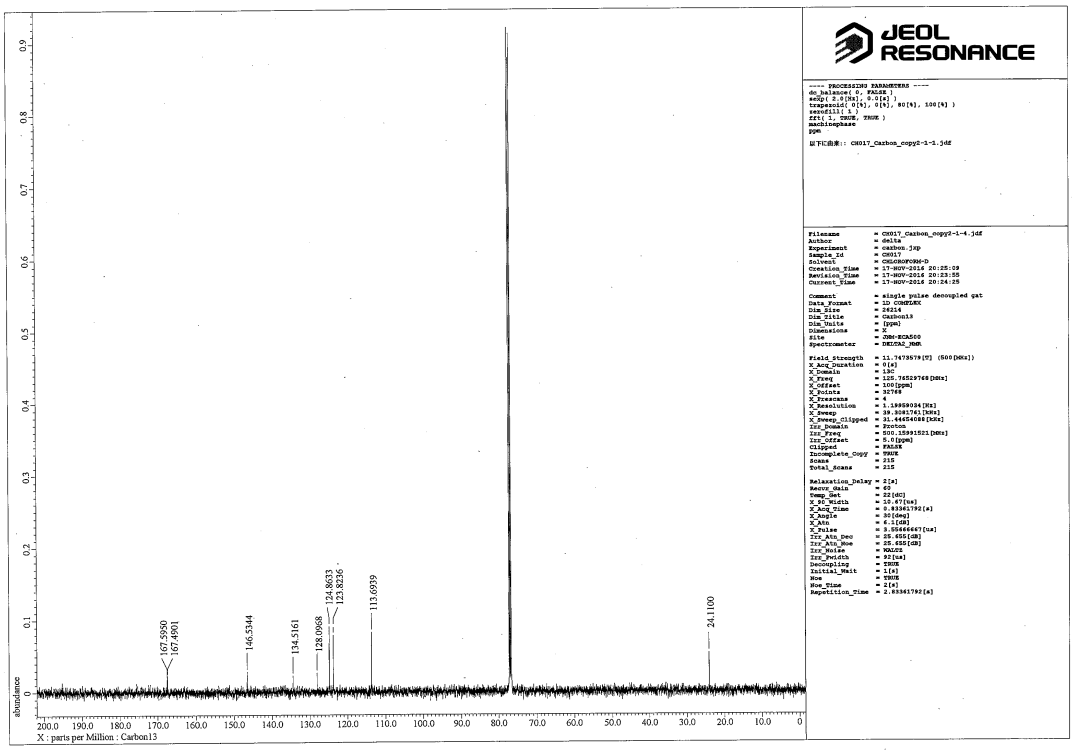
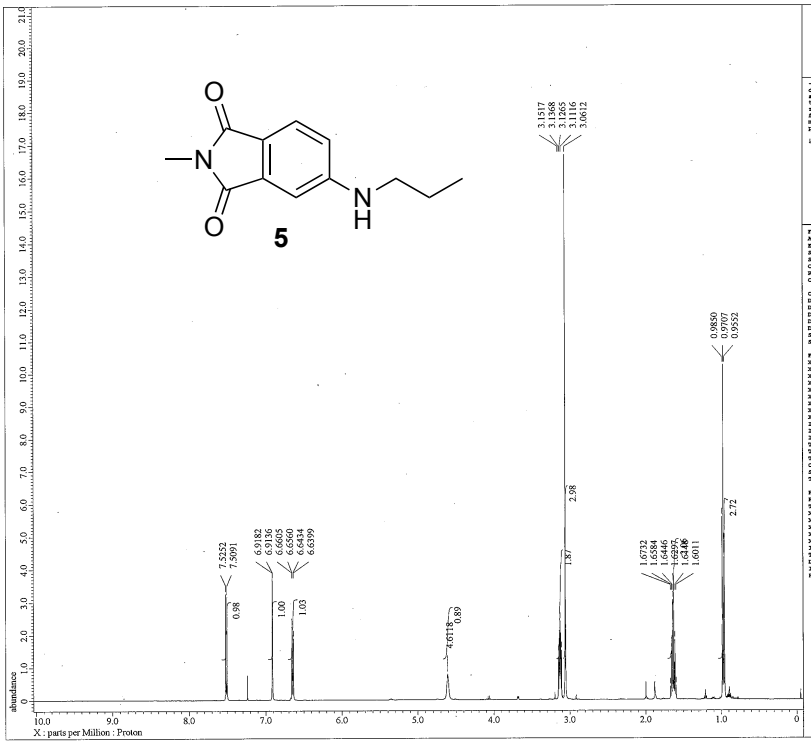
7. NMR spectra



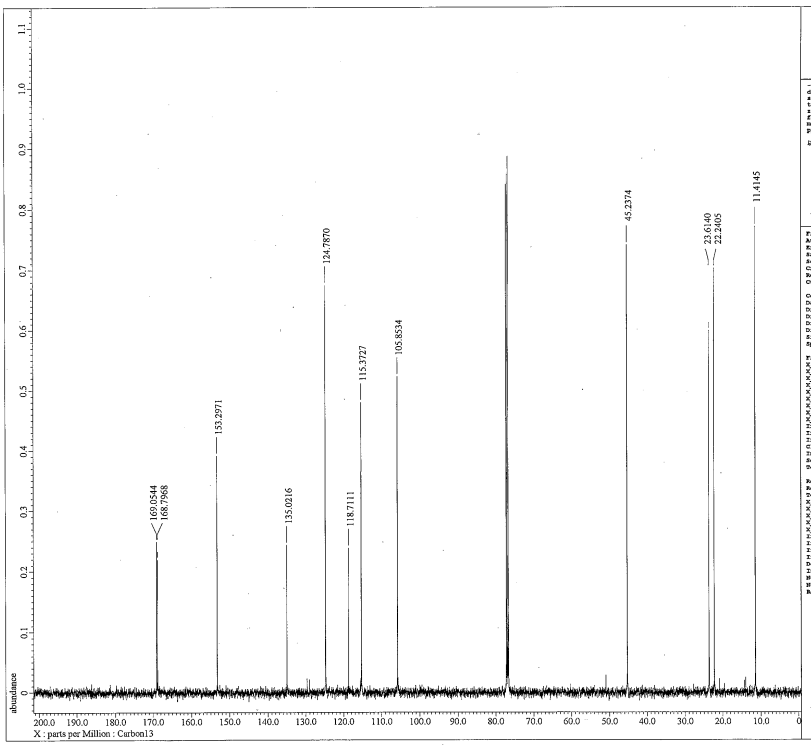




JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
expf( 0.2[Hz], 0.5[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: CH026_p_Proton-1-1.jdf
```

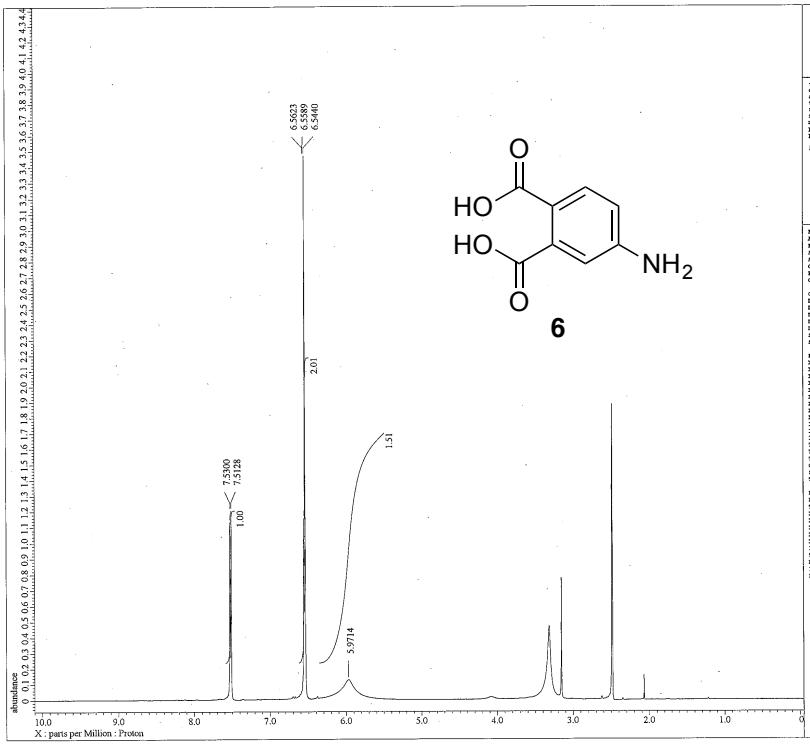
```
Filename          = CH026_p_Proton-1-8.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = CH026_p
Solvent           = CHLOROFORM-D
Creation_Time     =  2-JUN-2015 20:54:14
Revision_Time     = 16-NOV-2016 19:57:16
Current_Time      = 16-NOV-2016 19:57:42

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13197
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T]  (500[MHz])
X_Acq_Duration    = 1.74587904[s]
X_Domain          = 1H
X_Freq            = 500.15991521[MHz]
X_Offset          = 5.0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.57277737[Hz]
X_Sweep           = 9.38438438[KHz]
X_Sweep_Clipped   = 7.50750751[KHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Trt_Domain        = Proton
Trt_Freq          = 500.15992521[MHz]
Trt_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 30
Temp_Set          = 21.8[dC]
X_90_Width        = 6.13[us]
X_Acq_Time        = 1.74587904[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 3.065[us]
Irr_Mode          = off
Trt_Mode          = off
Dante_Presat      = FALSE
Initial_Wait      = 5[s]
Repetition_Time   = 6.74587904[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
expf( 1.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: CH026_p_Carbon_copy2-1-1.jdf
```

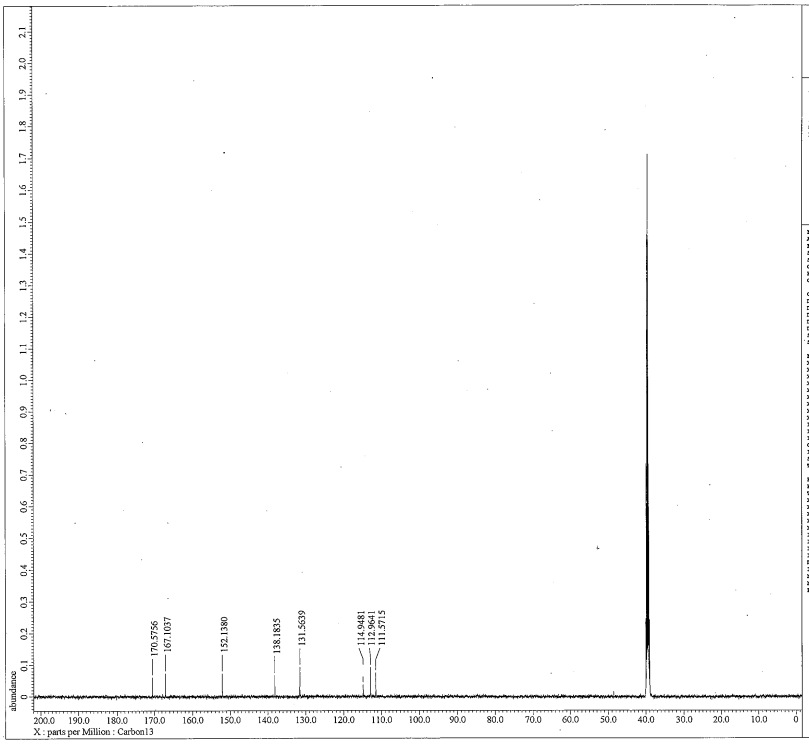
```
Filename          = CH026_p_Carbon_copy2-1-4.j
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = CH026_p
Solvent           = CHLOROFORM-D
Creation_Time     =  2-JUN-2015 20:59:23
Revision_Time     = 16-NOV-2016 19:58:48
Current_Time      = 16-NOV-2016 19:59:06

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T]  (500[MHz])
X_Acq_Duration    = 0[s]
X_Domain          = 13C
X_Freq            = 125.74525768[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.19898034[Hz]
X_Sweep           = 39.3081761[KHz]
X_Sweep_Clipped   = 31.4465408[KHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Clipped           = FALSE
Incomplete_Copy   = TRUE
Scans             = 184
Total_Scans       = 184

Relaxation_Delay = 2[s]
Recvr_Gain        = 40
Temp_Set          = 22[dC]
X_90_Width        = 10.8[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 6.1[dB]
X_Pulse           = 3.43333333[us]
Irr_Atn_Dec       = 25.52?[dB]
Irr_Atn_Noe       = 25.52?[dB]
Irr_Noise         = WALTZ6
Irr_Width         = 92[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.83361792[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
do_baseline( 0, FALSE )
setp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 5, 0, 50[%] )

ｵﾘｼﾞﾅﾙﾌｧｲﾙ名:: CH003_Proton-1-1.jdf
```

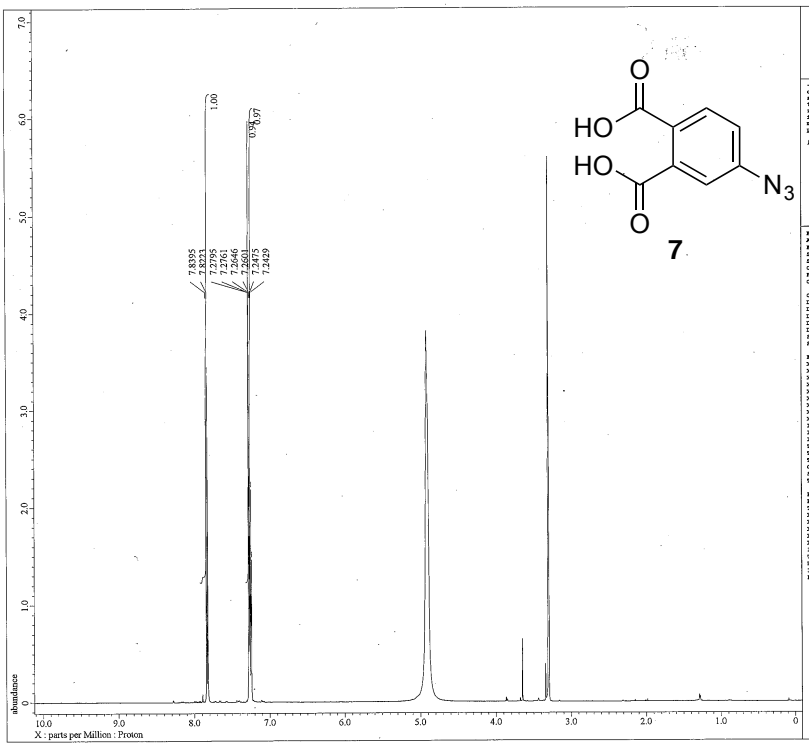
```
Filename          = CH003_Proton-1-5.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = CH003
Solvent           = DMSO-D6
Creation_Time     = 14-APR-2015 15:08:47
Revision_Time     = 17-NOV-2016 19:53:08
Current_Time      = 17-NOV-2016 19:53:34

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 1.74587904[s]
X_Domain          = 1H
X_Freq            = 500.13991521[MHz]
X_Offset          = 5.0[ppm]
X_Points          = 14384
X_Prescans        = 1
X_Resolution      = 0.57277737[Hz]
X_Sweep           = 8.38438438[kHz]
X_Sweep_Clipped   = 7.56730751[kHz]
Irr_Domain        = Proton
Irr_Freq          = 500.13991521[MHz]
Irr_Offset        = 5.0[ppm]
Tri_Domain        = Proton
Tri_Freq          = 500.13991521[MHz]
Tri_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recyc_Calc        = 46
Temp_Set          = 22.6[DC]
X_90_Width        = 4.15[us]
X_Acq_Time        = 1.74587904[s]
X_Angle           = 45[deg]
X_Atn             = 3.00[dB]
X_Pulse           = 2.08[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Preset      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.74587904[s]
```

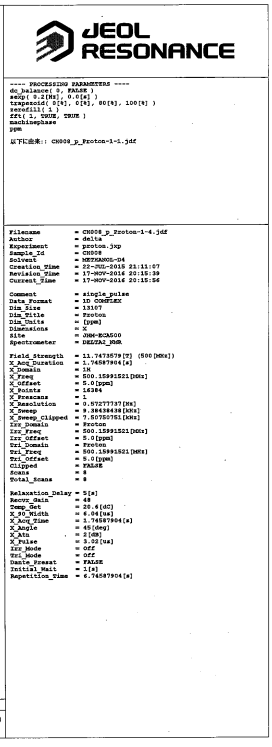


JEOL RESONANCE

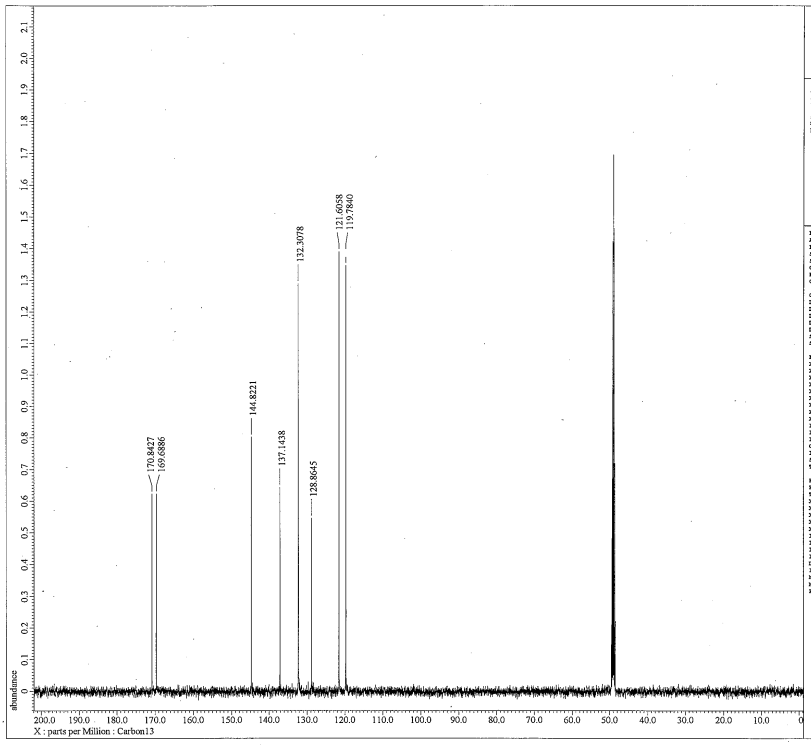
```
---- PROCESSING PARAMETERS ----
do_baseline( 0, FALSE )
setp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

ｵﾘｼﾞﾅﾙﾌｧｲﾙ名:: CH003_Carbon_copy1-1-1.jdf
```

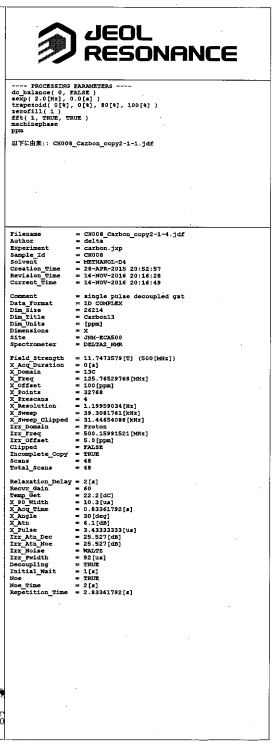
```
Filename          = CH003_Carbon_copy1-1-5.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = CH003
Solvent           = DMSO-D6
Creation_Time     = 14-APR-2015 15:14:57
Revision_Time     = 16-NOV-2016 20:11:57
Current_Time      = 16-NOV-2016 20:12:16

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 0[s]
X_Domain          = 13C
X_Freq            = 125.76529768[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.19869204[Hz]
X_Sweep           = 39.3581761[kHz]
X_Sweep_Clipped   = 31.44654088[kHz]
Irr_Domain        = Proton
Irr_Freq          = 500.13991521[MHz]
Irr_Offset        = 5.0[ppm]
Clipped           = FALSE
Incomplete_Copy   = TRUE
Scans             = 407
Total_Scans       = 407

Relaxation_Delay  = 2[s]
Recyc_Calc        = 40
Temp_Set          = 22.9[DC]
X_90_Width        = 10.2[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 6.1[dB]
X_Pulse           = 3.43333333[us]
Irr_Atn_Dec       = 25.62[dB]
Irr_Atn_Noe       = 25.62[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 92[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.83361792[s]
```



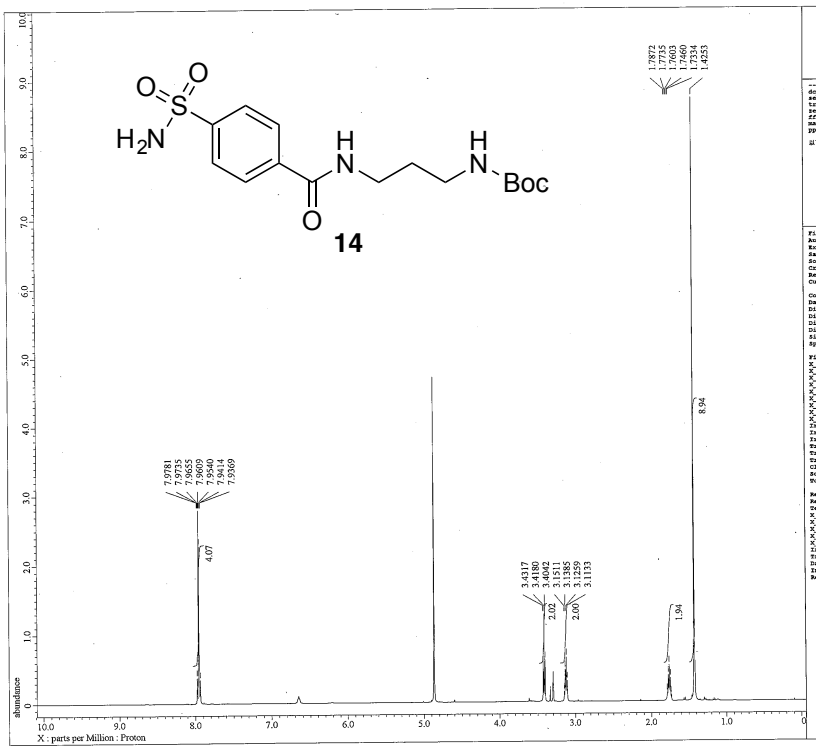


```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
expf( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に出来:: CH008_p_Proton-1-1.jdf

Filename        = CH008_p_Proton-1-4.jdf
Author          = delta
Experiment      = proton.jxp
Sample_Id       = CH008
Solvent         = METHANOL-D4
Creating_Time   = 22-JUL-2015 21:11:07
Revision_Time   = 17-NOV-2016 20:15:39
Current_Time    = 17-NOV-2016 20:15:56

Comment         = single_pulse
Data_Format     = 1D COMPLEX
Dim_Size        = 13107
Dim_Title       = Proton
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DELTA2_NMR

Field_Strength  = 11.7473579[T]  (500[MHz])
X_Acq_Duration  = 1.74587904[s]
X_Domain        = 1H
X_Freq          = 500.15891521[MHz]
X_Offset        = 5.0[ppm]
X_Points        = 16384
X_Prescans      = 1
X_Resolution    = 0.57277737[Hz]
X_Sweep         = 9.38658438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15891521[MHz]
Irr_Offset      = 5.0[ppm]
Dri_Domain      = Proton
Dri_Freq        = 500.15891521[MHz]
Dri_Offset      = 5.0[ppm]
Clipped         = FALSE
Scans           = 8
Total_Scans     = 8

Relaxation_Delay = 5[s]
Recvr_Gain      = 48
Temp_Set        = 20.6[dC]
X_90_Width      = 6.06[us]
X_Acq_Time      = 1.74587904[s]
X_Angle         = 45[deg]
X_Atn           = 2[dB]
X_Pulse         = 3.03[us]
Irr_Mode        = Off
Dri_Mode        = Off
Dumie_Presat    = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 6.74587904[s]
```





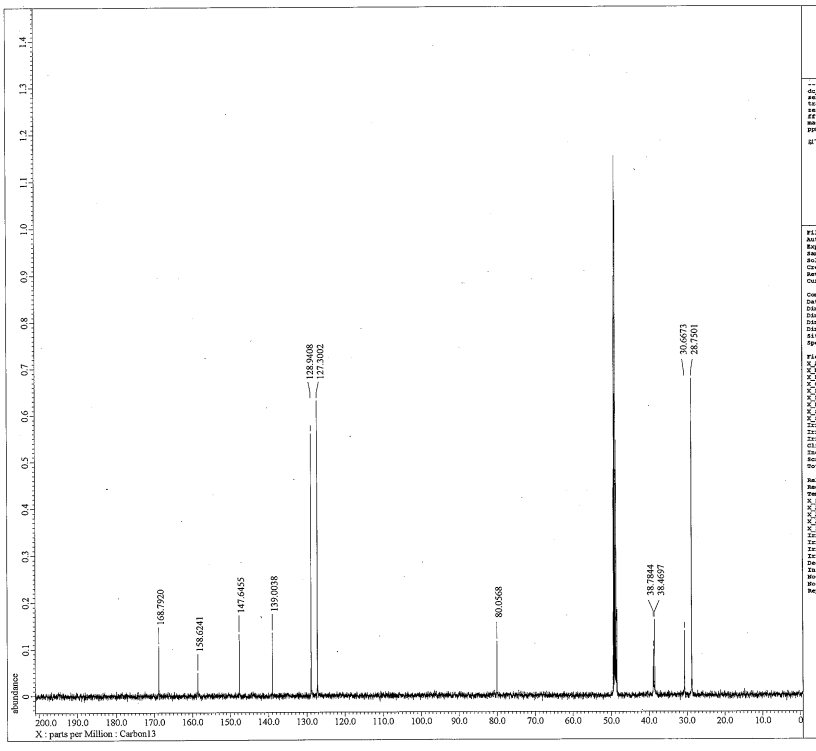
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
expf( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に出来:: CH008_Carbon_copy2-1-1.jdf

Filename        = CH008_Carbon_copy2-1-4.jdf
Author          = delta
Experiment      = carbon.jxp
Sample_Id       = CH008
Solvent         = METHANOL-D4
Creating_Time   = 28-APR-2015 20:52:57
Revision_Time   = 16-NOV-2016 20:14:28
Current_Time    = 16-NOV-2016 20:16:49

Comment         = single pulse decoupled gat
Data_Format     = 1D COMPLEX
Dim_Size        = 26214
Dim_Title       = Carbon13
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DELTA2_NMR

Field_Strength  = 11.7473579[T]  (500[MHz])
X_Acq_Duration  = 0[s]
X_Domain        = 13C
X_Freq          = 125.74529768[MHz]
X_Offset        = 100[ppm]
X_Points        = 32768
X_Prescans      = 4
X_Resolution    = 1.13939034[Hz]
X_Sweep         = 39.3081761[kHz]
X_Sweep_Clipped = 31.4465408[kHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15891521[MHz]
Irr_Offset      = 5.0[ppm]
Clipped         = FALSE
Incomplete_Copy = TRUE
Scans           = 48
Total_Scans     = 48

Relaxation_Delay = 2[s]
Recvr_Gain      = 40
Temp_Set        = 22.2[dC]
X_90_Width      = 10.2[us]
X_Acq_Time      = 0.83361792[s]
X_Angle         = 30[deg]
X_Atn           = 6.1[dB]
X_Pulse         = 3.43333333[us]
Irr_Atn_Dec     = 26.527[dB]
Irr_Atn_Noe     = 26.527[dB]
Irr_Noise       = WALTZ
Irr_Pwidth      = 92[us]
Decoupling      = TRUE
Initial_Wait    = 1[s]
Noe             = TRUE
Noe_Time        = 2[s]
Repetition_Time = 2.83361792[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sedp( 0.2[Hz], 0.5[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下元由来:: CH090_p_Proton-1-1.jdf
```

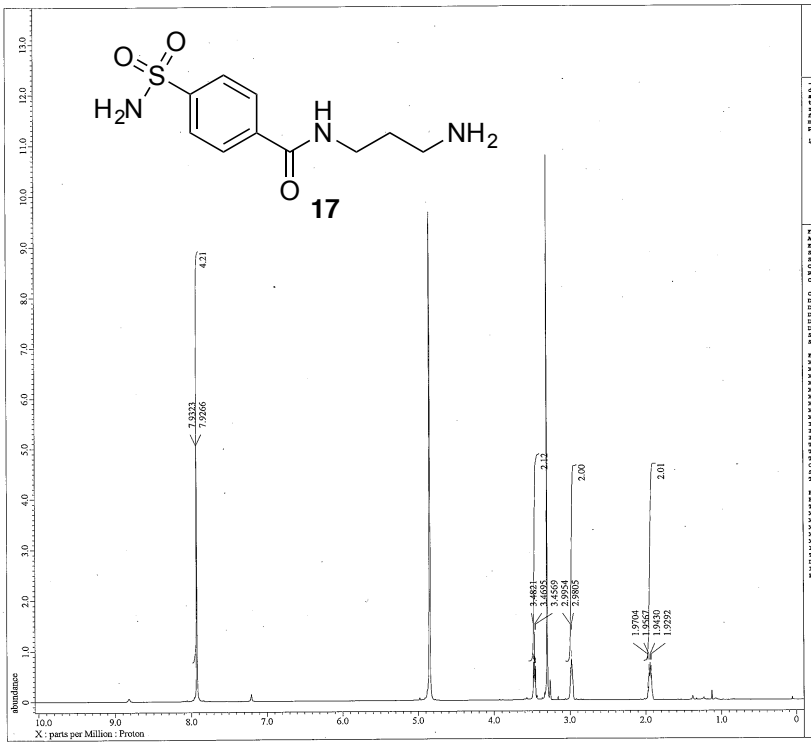
```
Filename        = CH090_p_Proton-1-5.jdf
Author          = delta
Experiment      = proton.jxp
Sample_Id       = CH090_p
Solvent         = METHANOL-D4
Creation_Time   = 28-JAN-2016 10:46:10
Revision_Time   = 16-NOV-2016 21:03:15
Current_Time    = 16-NOV-2016 21:03:18

Comment         = single_pulse
Data_Format     = 1D COMPLEX
Dim_Size        = 13107
Dim_Title       = Proton
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DELTA2_NMR

Field_Strength  = 11.7473579[T]  (500[MHz])
X_Acq_Duration  = 1.74587904[s]
X_Domain        = 1H
X_Freq          = 500.15991521[MHz]
X_Offset        = 5.0[ppm]
X_Points        = 24384
X_Prescans      = 1
X_Resolution    = 0.57277737[Hz]
X_Sweep         = 9.38438438[KHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15991521[MHz]
Irr_Offset      = 5.0[ppm]
Wrl_Domain      = Proton
Wrl_Freq        = 500.15991521[MHz]
Wrl_Offset      = 5.0[ppm]
Clipped         = FALSE
Scans           = 8
Total_Scans     = 8

Relaxation_Delay = 5[s]
Recvr_Gain      = 30
Temp_Set        = 19.8[dC]
X_90_Width      = 4.84[us]
X_Acq_Time      = 1.74587904[s]
X_Angle         = 45[deg]
X_Atn           = 2[dB]
X_Pulse         = 3.02[us]
Irr_Mode        = Off
Wrl_Mode        = Off
Dmfe_Mrgst      = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 6.74587904[s]
```

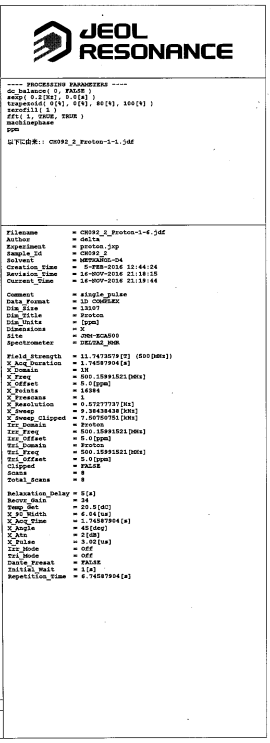


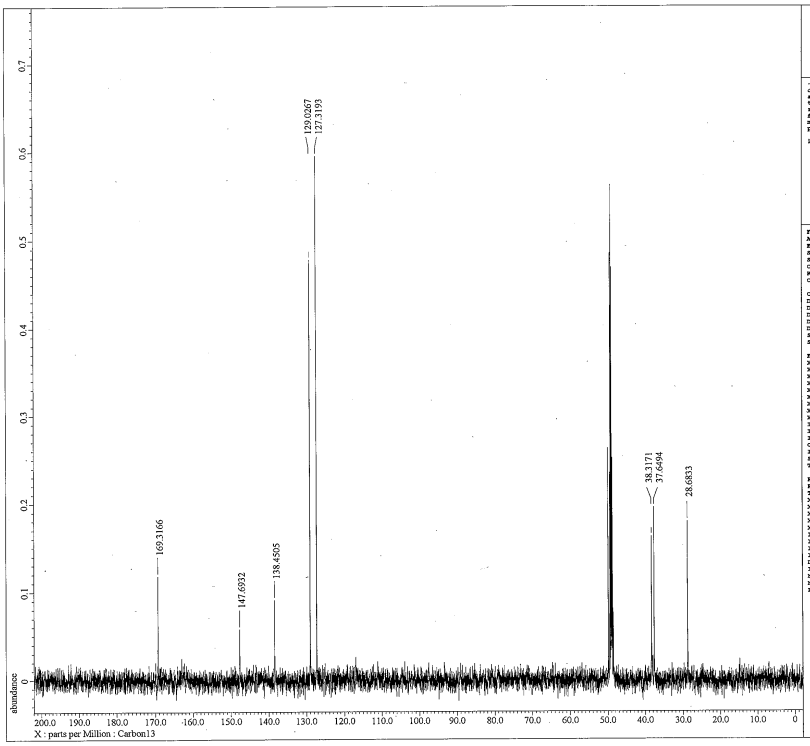
JEOL RESONANCE

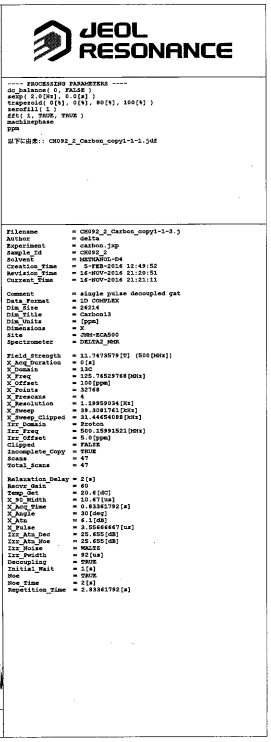
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sedp( 1.0[Hz], 0.5[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下元由来:: CH090_p_Carbon_copy4-1-1.jdf
```

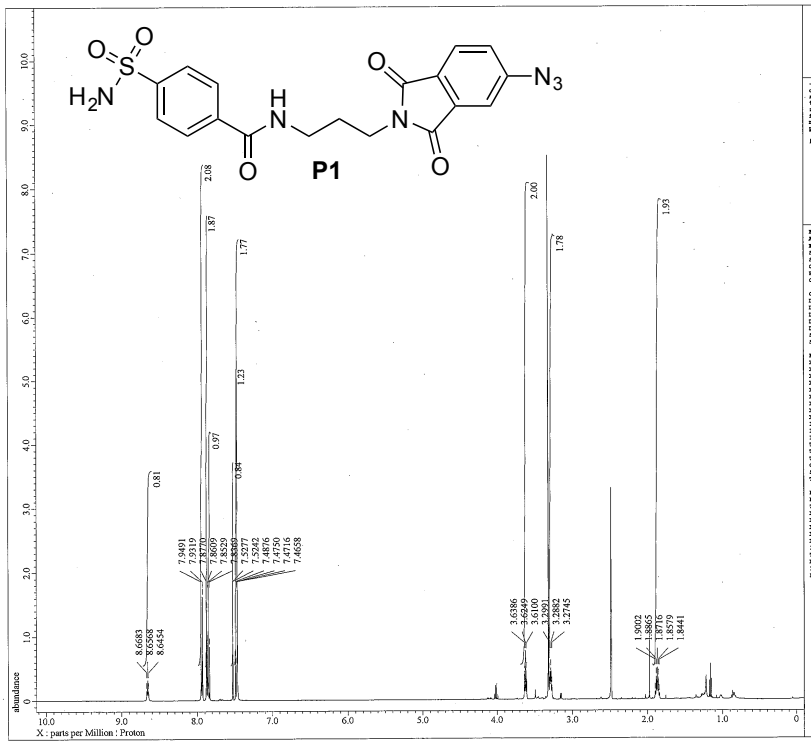
```
Filename        = CH090_p_Carbon_copy4-1-4.j
Author          = delta
Experiment      = carbon.jxp
Sample_Id       = CH090_p
Solvent         = METHANOL-D4
Creation_Time   = 26-JAN-2016 10:54:48
Revision_Time   = 16-NOV-2016 21:04:43
Current_Time    = 16-NOV-2016 21:04:52

Comment         = single pulse decoupled gat
Data_Format     = 1D COMPLEX
Dim_Size        = 26214
Dim_Title       = Carbon13
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DELTA2_NMR

Field_Strength  = 11.7473579[T]  (500[MHz])
X_Acq_Duration  = 0[s]
X_Domain        = 13C
X_Freq          = 125.76529768[MHz]
X_Offset        = 100[ppm]
X_Points        = 32768
X_Prescans      = 4
X_Resolution    = 1.19826034[Hz]
X_Sweep         = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15991521[MHz]
Irr_Offset      = 5.0[ppm]
Clipped         = FALSE
Incomplete_Copy = TRUE
Scans           = 229
Total_Scans     = 229

Relaxation_Delay = 2[s]
Recvr_Gain      = 40
Temp_Set        = 20.2[dC]
X_90_Width      = 10.67[us]
X_Acq_Time      = 0.83361792[s]
X_Angle         = 30[deg]
X_Atn           = 4.1[dB]
X_Pulse         = 3.55666667[us]
Irr_Atn_Dec     = 25.65[dB]
Irr_Atn_Noe     = 25.65[dB]
Irr_Noise       = WALTZ5
Irr_Width       = 92[us]
Decoupling      = TRUE
Initial_Wait    = 1[s]
Noe             = TRUE
Noe_Time        = 2[s]
Repetition_Time = 2.83361792[s]
```


17
4.21
7.9321
7.9266
2.12
2.00
2.01
3.4821
3.4695
3.4569
3.4534
2.9805
1.9704
1.9567
1.9430
1.9292
abundance
X : parts per Million : Proton


JEOL RESONANCE
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sedp(0.2[Hz], 0.5[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来:: CH092_2_Proton-1-1.jdf
Filename = CH092_2_Proton-1-4.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = CH092_2
Solvent = METHANOL-D4
Creation_Time = 5-FEB-2016 12:44:24
Revision_Time = 16-NOV-2016 21:18:15
Current_Time = 16-NOV-2016 21:19:44
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 14984
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[KHz]
X_Sweep_Clipped = 7.50750751[KHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Wrl_Domain = Proton
Wrl_Freq = 500.15991521[MHz]
Wrl_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recyr_Gain = 36
Temp_Set = 20.5[dC]
X_90_Width = 6.84[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 3.92[us]
Irr_Mode = off
Wrl_Mode = off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]


129.0267
127.3193
169.3166
147.6932
138.4505
38.3171
37.6494
26.6833
abundance
X : parts per Million : Carbon13

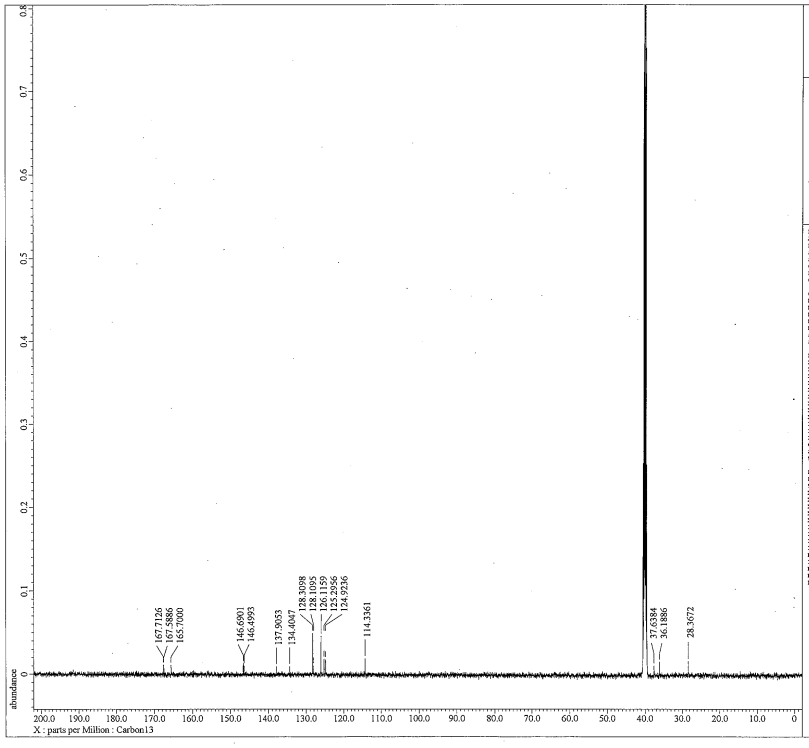

JEOL RESONANCE
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sedp(2.0[Hz], 0.2[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来:: CH092_2_Carbon_copy1-1-1.jdf
Filename = CH092_2_Carbon_copy1-1-3.j
Author = delta
Experiment = carbon.jxp
Sample_Id = CH092_2
Solvent = METHANOL-D4
Creation_Time = 5-FEB-2016 12:49:52
Revision_Time = 16-NOV-2016 21:20:51
Current_Time = 16-NOV-2016 21:21:11
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19859034[Hz]
X_Sweep = 39.3281761[KHz]
X_Sweep_Clipped = 31.4625408[KHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Incomplete_Copy = TRUE
Scans = 47
Total_Scans = 47
Relaxation_Delay = 2[s]
Recyr_Gain = 60
Temp_Set = 20.6[dC]
X_90_Width = 10.67[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 6.1[dB]
X_Pulse = 3.55466667[us]
Irr_Atn_Dec = 25.65[dB]
Irr_Atn_Noe = 25.65[dB]
Irr_Noise = WALTZ16
Irr_Width = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.93361792[s]



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
do_balance( 0, FALSE )
expf( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に出来:: CH098_3_Proton-1-1.jdf
```

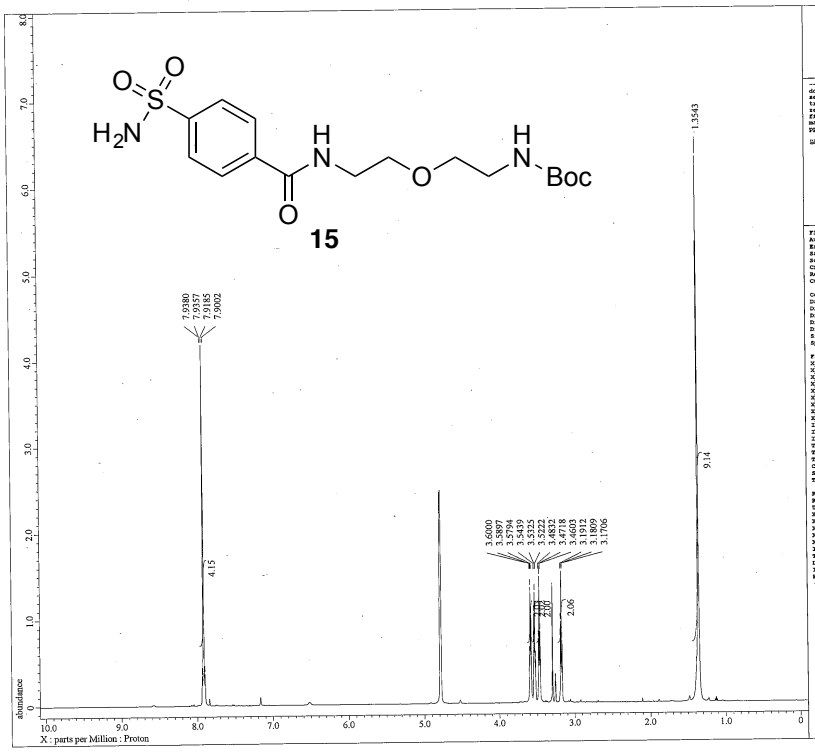
```
Filename          = CH098_3_Proton-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = CH098_3
Solvent           = DMSO-D6
Creation_Time     = 16-FEB-2016 21:19:58
Revision_Time     = 17-NOV-2016 20:31:43
Current_Time      = 17-NOV-2016 20:31:44

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T]  (500[MHz])
X_Acq_Duration    = 1.74587904[s]
X_Domain          = 1H
X_Freq            = 500.15991521[MHz]
X_Offset          = 6.0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.57277737[Hz]
X_Sweep           = 9.38439438[kHz]
X_Sweep_Clipped   = 7.99792791[kHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 6.0[ppm]
Wrt_Domain        = Proton
Wrt_Freq          = 500.15991521[MHz]
Wrt_Offset        = 6.0[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recyc_Gain        = 50
Temp_Set          = 21[dC]
X_90_Width        = 4.06[us]
X_Acq_Time        = 1.74587904[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 3.01[us]
Irr_Mode          = Off
Wrt_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.74587904[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
do_balance( 0, FALSE )
expf( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に出来:: CH098_3_Carbon-1-1.jdf
```

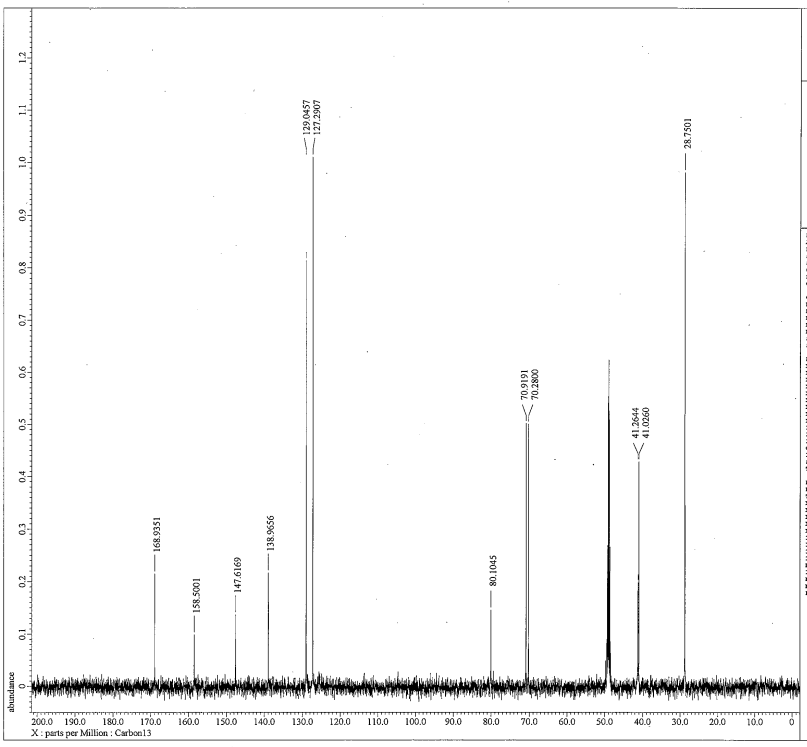
```
Filename          = CH098_3_Carbon-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = CH098_3
Solvent           = DMSO-D6
Creation_Time     = 16-FEB-2016 21:24:13
Revision_Time     = 17-NOV-2016 20:32:40
Current_Time      = 17-NOV-2016 20:34:17

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T]  (500[MHz])
X_Acq_Duration    = 0.83361792[s]
X_Domain          = 13C
X_Freq            = 125.76529768[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.19958036[Hz]
X_Sweep           = 39.3245741[kHz]
X_Sweep_Clipped   = 31.44654088[kHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recyc_Gain        = 40
Temp_Set          = 21.4[dC]
X_90_Width        = 10.67[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 6.1[dB]
X_Pulse           = 3.55666667[us]
Irr_Atn_Dec       = 25.45[dB]
Irr_Atn_Noe       = 20.45[dB]
Irr_Mode          = Noise
Irr_Pwidth        = 90[us]
Decoupling        = Pulse
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.83361792[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
expl( 2.0[Hz], 0[%] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

出力ﾌｧｲﾙ名:: CH12S_3_Proton-2-1.jdf
```

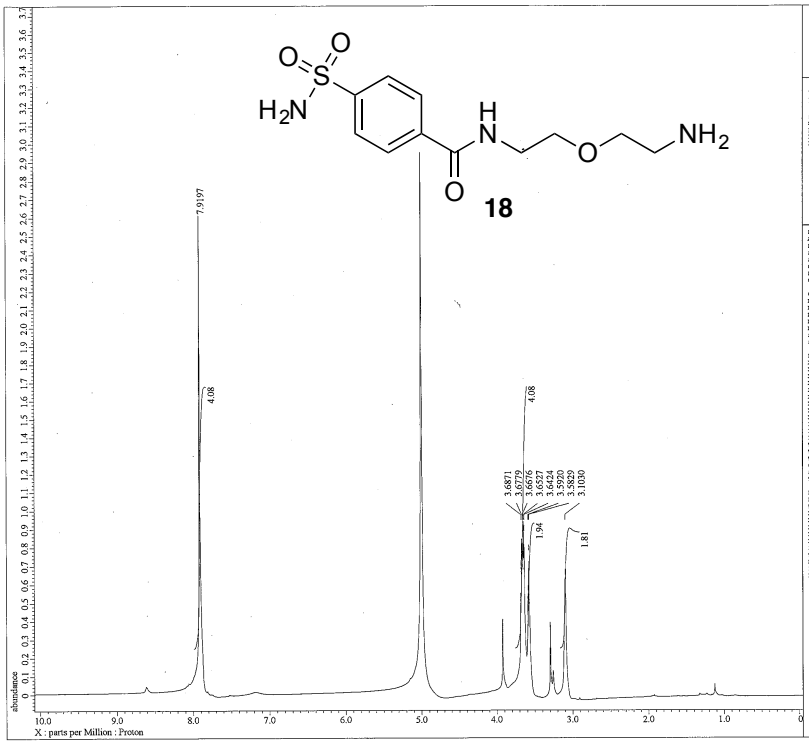
```
Filename          = CH12S_3_Proton-2-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = CH12S_3
Solvent           = METHANOL-D4
Creation_Time     = 19-APR-2016 22:26:49
Revision_Time     = 16-NOV-2016 21:23:44
Current_Time      = 16-NOV-2016 21:23:57

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 19107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = Jmn-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T]  (500[MHz])
X_Acq_Duration    = 1.74587904[s]
X_Domain          = 1H
X_Freq            = 500.15991521[MHz]
X_Offset          = 5.0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.57277737[Hz]
X_Sweep           = 9.38438438[KHz]
X_Sweep_Clipped   = 7.50750751[KHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Wtr_Domain        = Proton
Wtr_Freq          = 500.15991521[MHz]
Wtr_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 32
Temp_Set          = 23.4[dC]
X_90_Width        = 4.04[us]
X_Acq_Time        = 1.74587904[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 2.02[us]
Irr_Mode          = Off
Wtr_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 5[s]
Repetition_Time   = 6.74587904[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
expl( 2.0[Hz], 0[%] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

出力ﾌｧｲﾙ名:: CH12S_3_Carbon_copy1-1-1.jdf
```

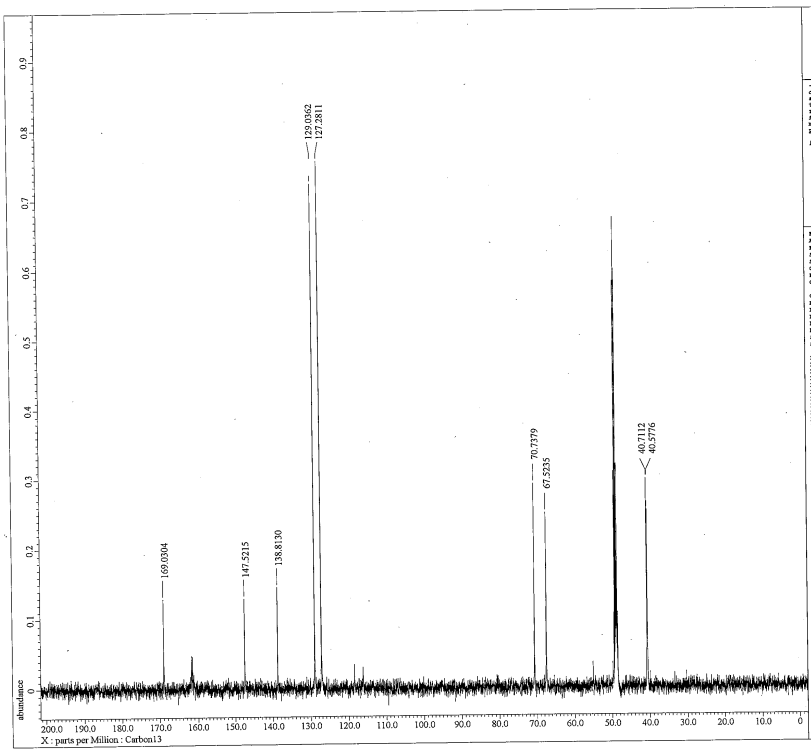
```
Filename          = CH12S_3_Carbon_copy1-1-4.j
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = CH12S_3
Solvent           = METHANOL-D4
Creation_Time     = 19-APR-2016 22:28:30
Revision_Time     = 16-NOV-2016 21:26:40
Current_Time      = 16-NOV-2016 21:27:00

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 24234
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = Jmn-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T]  (500[MHz])
X_Acq_Duration    = 2[s]
X_Domain          = 13C
X_Freq            = 125.76523768[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.19858634[Hz]
X_Sweep           = 39.3951761[KHz]
X_Sweep_Clipped   = 31.4465408[KHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Clipped           = FALSE
Incomplete_Copy   = TRUE
Scans             = 45
Total_Scans       = 45

Relaxation_Delay  = 2[s]
Recvr_Gain        = 40
Temp_Set          = 22.5[dC]
X_90_Width        = 10.67[us]
X_Acq_Time        = 0.83261792[s]
X_Angle           = 30[deg]
X_Atn             = 6.1[dB]
X_Pulse           = 3.55666667[us]
Irr_Atn_Dec       = 25.65[dB]
Irr_Atn_Noe       = 25.65[dB]
Irr_Noise         = WALTZ
Irr_Width         = 92[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.83261792[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
do_balance( 0, FALSE )
setp( 0.2[%], 0.5[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -3, 5, 50[%] )
phase( -7, 0, 50[%] )

以下に由来：: CH128_Proton-3-1.jdf
```

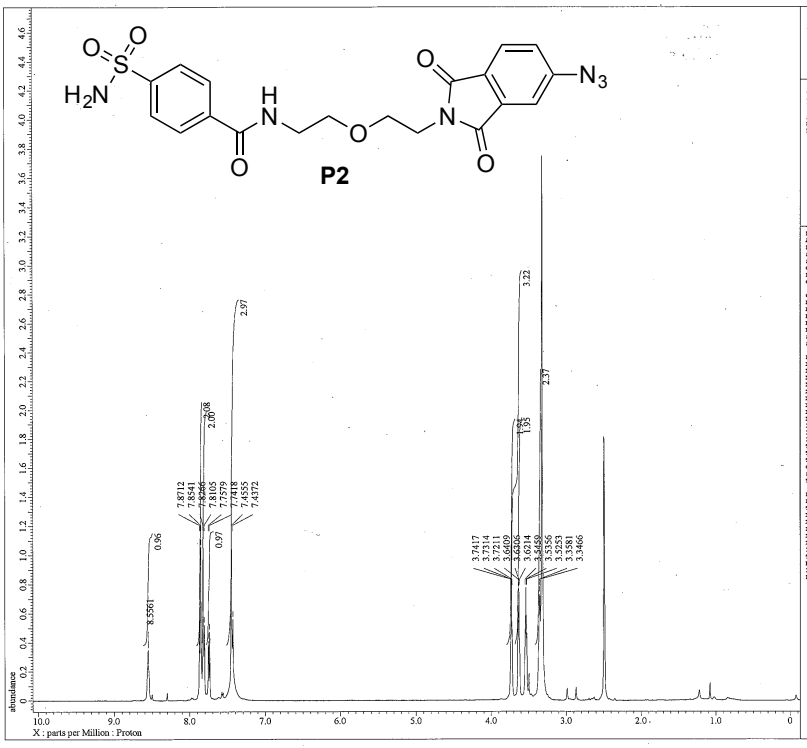
```
Filename        = CH128_Proton-3-18.jdf
Author          = delta
Experiment      = proton.jxp
Sample_Id       = CH128
Solvent         = METHANOL-D4
Creation_Time   = 21-APR-2016 14:25:57
Revision_Time   = 14-NOV-2016 21:32:28
Current_Time    = 14-NOV-2016 21:32:47

Comment         = single_pulse
Data_Format     = 1D COMPLEX
Dim_Size        = 19207
Dim_Title       = Proton
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DELTA2_NMR

Field_Strength  = 11.7473579[T] (500[MHz])
X_Acq_Duration  = 1.74587904[s]
X_Domain        = 1H
X_Freq          = 500.15991521[MHz]
X_Offset        = 5.0[ppm]
X_Points        = 16384
X_Prescans      = 1
X_Resolution    = 0.57277797[Hz]
X_Sweep         = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15991521[MHz]
Irr_Offset      = 5.0[ppm]
Wrt_Domain      = Proton
Wrt_Freq        = 500.15991521[MHz]
Wrt_Offset      = 5.0[ppm]
Clipped         = FALSE
Scans           = 8
Total_Scans     = 8

Relaxation_Delay = 5[s]
Recov_Gain      = 30
Temp_Get        = 21.8[dC]
X_90_Width      = 6.04[us]
X_Acq_Time      = 1.74587904[s]
X_Angle         = 45[deg]
X_Atn           = 2[dB]
X_Pulse         = 3.02[us]
Irr_Mode        = Off
Wrt_Mode        = Off
Dante_Presat    = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 6.74587904[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
do_balance( 0, FALSE )
setp( 0.2[%], 0.5[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来：: CH128_Carbon_copy1-1-1.jdf
```

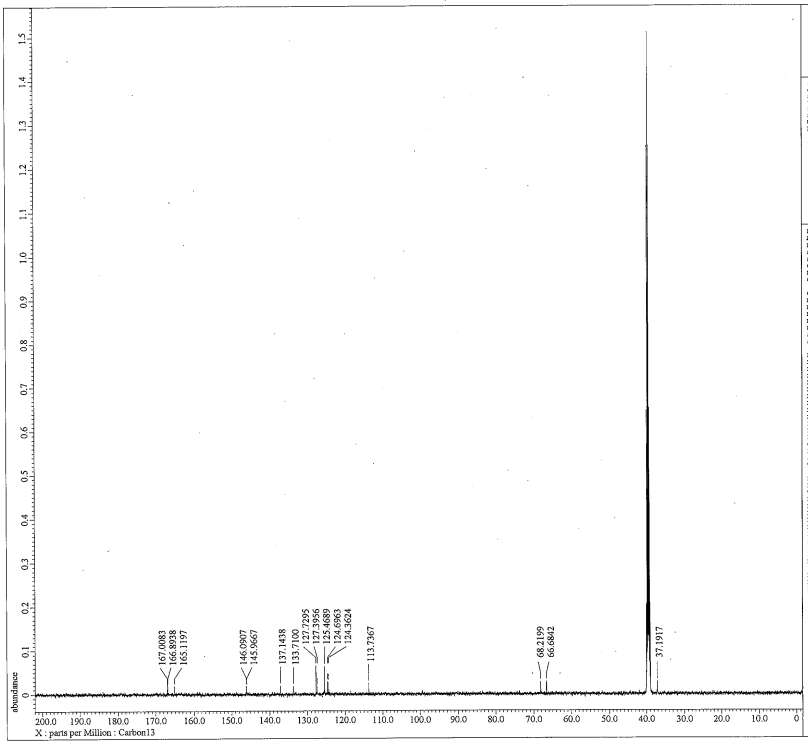
```
Filename        = CH128_Carbon_copy1-1-1.jdf
Author          = delta
Experiment      = carbon.jxp
Sample_Id       = CH128
Solvent         = METHANOL-D4
Creation_Time   = 21-APR-2016 14:18:01
Revision_Time   = 14-NOV-2016 21:37:05
Current_Time    = 14-NOV-2016 21:37:49

Comment         = single pulse decoupled gat
Data_Format     = 1D COMPLEX
Dim_Size        = 26214
Dim_Title       = Carbon13
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DELTA2_NMR

Field_Strength  = 11.7473879[T] (500[MHz])
X_Acq_Duration  = 0[s]
X_Domain        = 13C
X_Freq          = 125.76529768[MHz]
X_Offset        = 100[ppm]
X_Points        = 32768
X_Prescans      = 4
X_Resolution    = 1.19959034[Hz]
X_Sweep         = 39.3081761[kHz]
X_Sweep_Clipped = 31.44454988[kHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15991521[MHz]
Irr_Offset      = 5.0[ppm]
Clipped         = FALSE
Incomplete_Copy = TRUE
Scans           = 86
Total_Scans     = 86

Relaxation_Delay = 2[s]
Recov_Gain      = 40
Temp_Get        = 22.4[dC]
X_90_Width      = 10.47[us]
X_Acq_Time      = 0.83361792[s]
X_Angle         = 90[deg]
X_Atn           = 4.1[dB]
X_Pulse         = 5.55666667[us]
Irr_Atn_Dec     = 25.455[dB]
Irr_Atn_Noe     = 25.455[dB]
Irr_Noise       = WALTZ
Irr_Width       = 82[us]
Decoupling      = TRUE
Initial_Wait    = 1[s]
Noe             = TRUE
Noe_Time        = 2[s]
Repetition_Time = 2.83361792[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
exp( 0.2[Hz], 0.5[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

出下元由来：: CH133_Proton-1-1.jdf
```

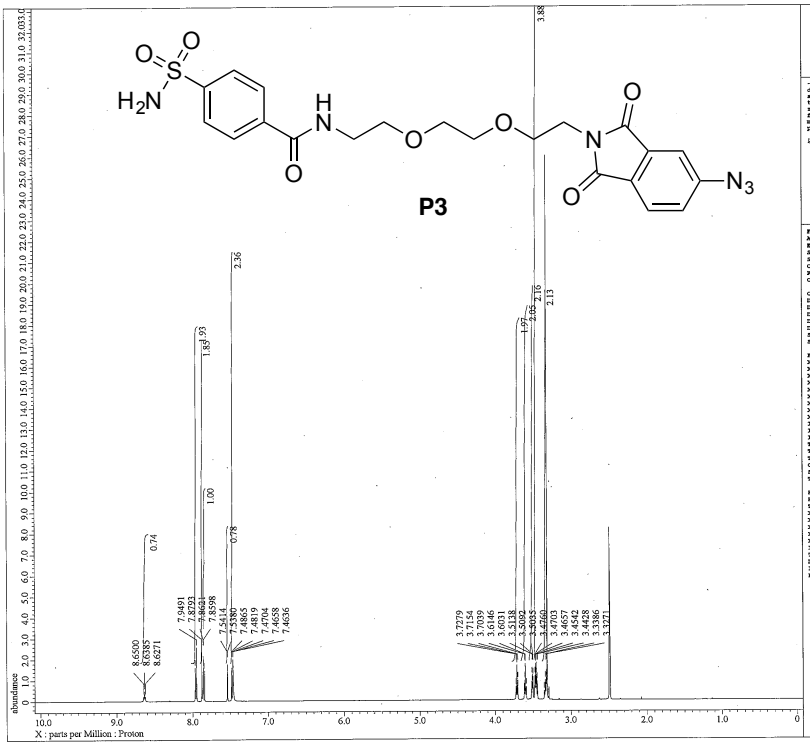
```
Filename        = CH133_Proton-1-4.jdf
Author          = delta
Experiment      = proton.jxp
Sample_Id       = CH133
SolventC        = DMSO-D6
Creation_Time   = 15-MAY-2016 19:59:28
Revision_Time   = 17-NOV-2016 20:39:09
Current_Time    = 17-NOV-2016 20:39:11

Comment         = single_pulse
Data_Format     = 1D COMPLEX
Dim_Size        = 13107
Dim_Title       = Proton
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DELTA2_NMR

Field_Strength  = 11.7473579[T] (500[MHz])
X_Acq_Duration  = 1.74587904[s]
X_Domain        = 1H
X_Freq          = 500.15891521[MHz]
X_Offset        = 5.0[ppm]
X_Points        = 16384
X_Prescans      = 1
X_Resolution    = 0.57277737[Hz]
X_Sweep         = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15891521[MHz]
Irr_Offset      = 5.0[ppm]
Tri_Domain      = Proton
Tri_Freq        = 500.15891521[MHz]
Tri_Offset      = 5.0[ppm]
Clipped         = FALSE
Scans           = 8
Total_Scans     = 8

Relaxation_Delay = 5[s]
Recyc_Gain      = 50
Temp_Set        = 20.2[dC]
X_90_Width      = 6.04[us]
X_Acq_Time      = 1.74587904[s]
X_Angle         = 45[deg]
X_Atn           = 2[dB]
X_Pulse         = 3.02[us]
Irr_Mode        = off
Tri_Mode        = off
Data_Format     = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 6.74587904[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
exp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

出下元由来：: CH133_Carbon_copy3-1-1.jdf
```

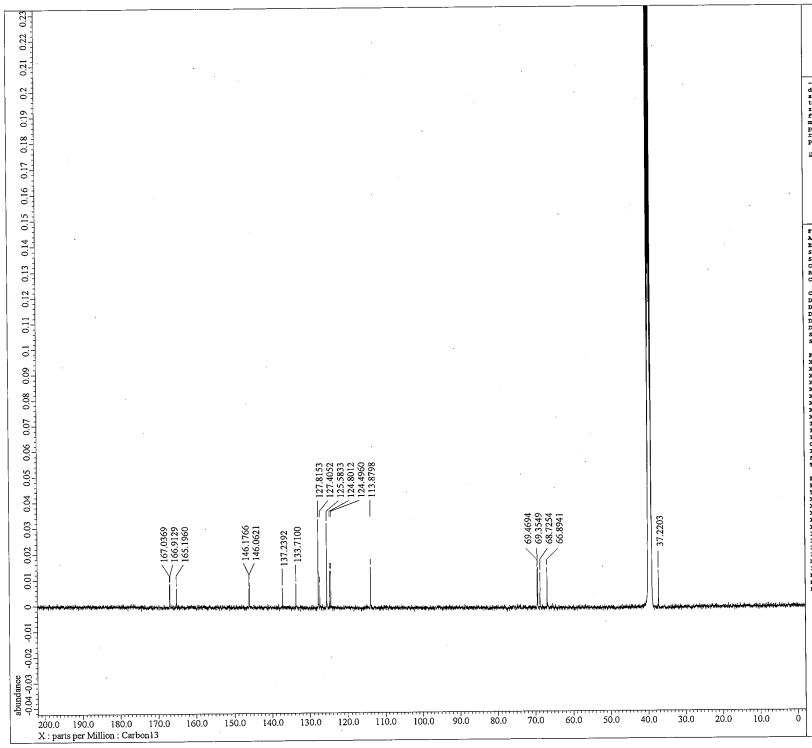
```
Filename        = CH133_Carbon_copy3-1-4.jdf
Author          = delta
Experiment      = carbon.jxp
Sample_Id       = CH133
SolventC        = DMSO-D6
Creation_Time   = 11-MAY-2016 20:04:11
Revision_Time   = 17-NOV-2016 20:40:34
Current_Time    = 17-NOV-2016 20:41:44

Comment         = single pulse decoupled gat
Data_Format     = 1D COMPLEX
Dim_Size        = 26214
Dim_Title       = Carbon13
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DELTA2_NMR

Field_Strength  = 11.7473579[T] (500[MHz])
X_Acq_Duration  = 0[s]
X_Domain        = 13C
X_Freq          = 125.76529768[MHz]
X_Offset        = 100[ppm]
X_Points        = 32768
X_Prescans      = 4
X_Resolution    = 1.19869804[Hz]
X_Sweep         = 39.3985761[kHz]
X_Sweep_Clipped = 31.64854088[kHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15891521[MHz]
Irr_Offset      = 5.0[ppm]
Clipped         = FALSE
Complete_Copy   = TRUE
Scans           = 591
Total_Scans     = 591

Relaxation_Delay = 2[s]
Recyc_Gain      = 40
Temp_Set        = 21.6[dC]
X_90_Width      = 10.67[us]
X_Acq_Time      = 0.83361792[s]
X_Angle         = 30[deg]
X_Atn           = 4.1[dB]
X_Pulse         = 3.55666667[us]
Irr_Atn_Dec     = 25.65[dB]
Irr_Atn_Hse     = 25.65[dB]
Irr_Noise       = WALTZ16
Irr_Pwidth      = 92[us]
Decoupling      = TRUE
Initial_Wait    = 1[s]
Hse             = TRUE
Hse_Time        = 2[s]
Repetition_Time = 2.83361792[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sedp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
manthingphase
ppm

以下に由来:: CH096_2_Proton-1-1.jdf
```

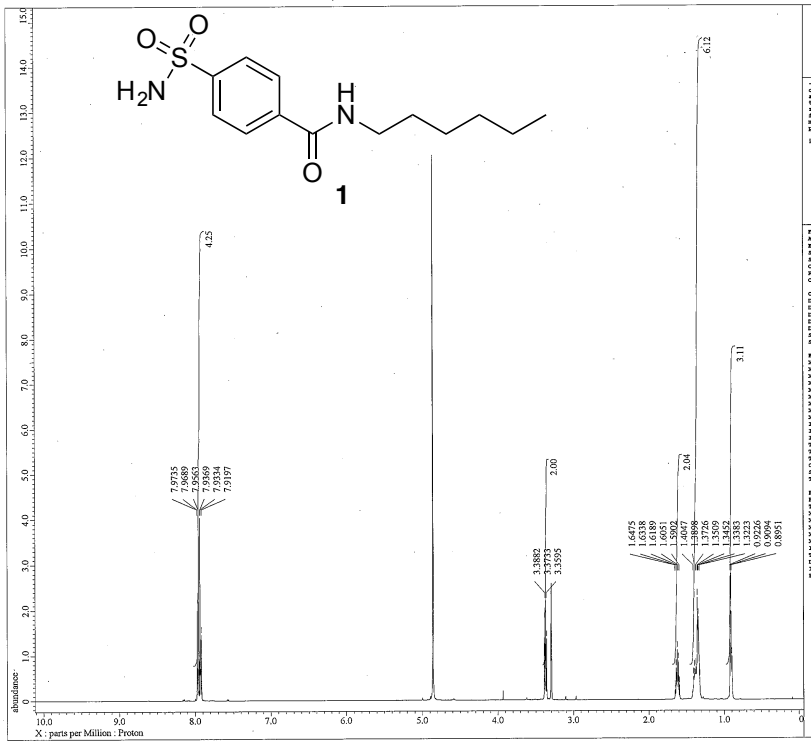
```
Filename        = CH096_2_Proton-1-4.jdf
Author          = delta
Experiment      = proton.jxp
Sample_Id       = CH096_2
Solvent         = DMSO-D6
Creation_Time   = 15-FEB-2016 20:14:14
Revision_Time   = 17-NOV-2016 20:11:34
Current_Time    = 17-NOV-2016 20:11:20

Comment         = single_pulse
Data_Format     = 1D COMPLEX
Dim_Size        = 13107
Dim_Title       = Proton
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DEUTA2_NMR

Field_Strength  = 11.7473579[T] (500[MHz])
X_Acq_Duration  = 1.74587904[s]
X_Domain        = 1H
X_Freq          = 500.15891521[MHz]
X_Offset        = 5.0[ppm]
X_Points        = 16384
X_Prescans      = 1
X_Resolution    = 0.57277737[Hz]
X_Sweep         = 9.38438438[KHz]
X_Sweep_Clipped = 7.50758751[KHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15891521[MHz]
Irr_Offset      = 5.0[ppm]
Tri_Domain      = Proton
Tri_Freq        = 500.15891521[MHz]
Tri_Offset      = 5.0[ppm]
Clipped         = FALSE
Scans           = 8
Total_Scans     = 8

Relaxation_Delay = 5[s]
Recvr_Gain      = 36
Temp_Set        = 25.6[dC]
X_90_Width      = 4.04[us]
X_Acq_Time      = 1.74587904[s]
X_Angle         = 45[deg]
X_Atn           = 3[dB]
X_Pulse         = 3.02[us]
Irr_Mode        = Off
Tri_Mode        = Off
Data_Format     = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 6.74587904[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sedp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
manthingphase
ppm
reference( 40.04209[ppm], 39.5[ppm] )
phase( 0, 0, 80[%] )

以下に由来:: CH096_2_Carbon_copy1-1-1.jdf
```

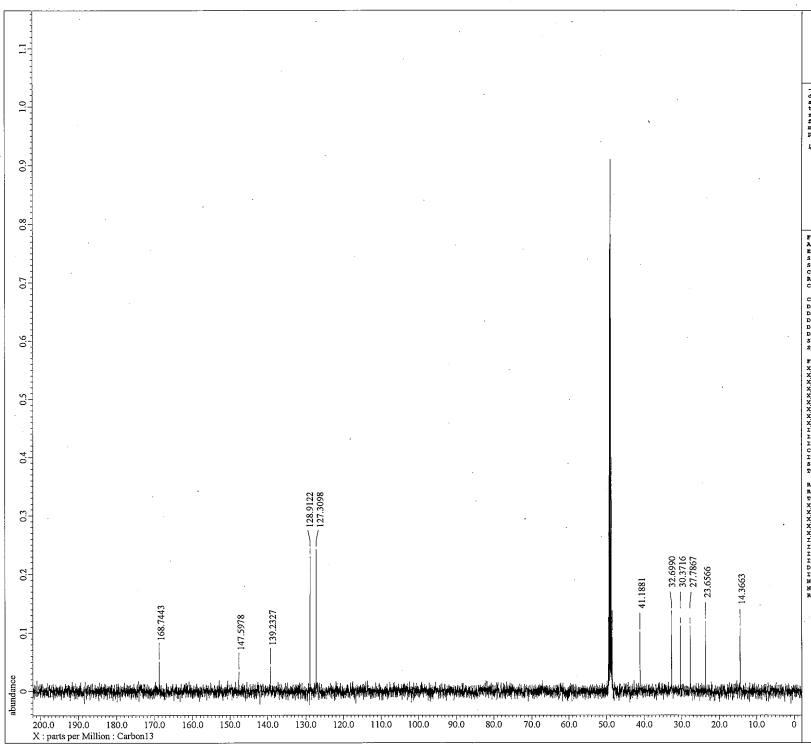
```
Filename        = CH096_2_Carbon_copy1-1-6.j
Author          = delta
Experiment      = carbon.jxp
Sample_Id       = CH096_2
Solvent         = DMSO-D6
Creation_Time   = 15-FEB-2016 21:46:35
Revision_Time   = 14-NOV-2016 22:05:27
Current_Time    = 14-NOV-2016 22:06:27

Comment         = single pulse decoupled gat
Data_Format     = 1D COMPLEX
Dim_Size        = 26214
Dim_Title       = Carbon13
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DEUTA2_NMR

Field_Strength  = 11.7473579[T] (500[MHz])
X_Acq_Duration  = 0.83[s]
X_Domain        = 13C
X_Freq          = 125.76529768[MHz]
X_Offset        = 120[ppm]
X_Points        = 32768
X_Prescans      = 4
X_Resolution    = 1.19959034[Hz]
X_Sweep         = 39.3082761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15891521[MHz]
Irr_Offset      = 5.0[ppm]
Clipped         = FALSE
Incomplete_Copy = TRUE
Scans           = 15927
Total_Scans     = 15927

Relaxation_Delay = 2[s]
Recvr_Gain      = 60
Temp_Set        = 25[dC]
X_90_Width      = 16.67[us]
X_Acq_Time      = 0.83361792[s]
X_Angle         = 30[deg]
X_Atn           = 4.1[dB]
X_Pulse         = 3.05666667[us]
Irr_Atn_Dec     = 25.65[dB]
Irr_Atn_Noe     = 25.65[dB]
Irr_Noise       = WALTZ
Irr_Pwidth      = 92[us]
Decoupling      = TRUE
Initial_Wait    = 1[s]
Noe             = TRUE
Noe_Time        = 2[s]
Repetition_Time = 2.83361792[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
expr( 0.5[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: CH099_Proton-1-1.jdf
```

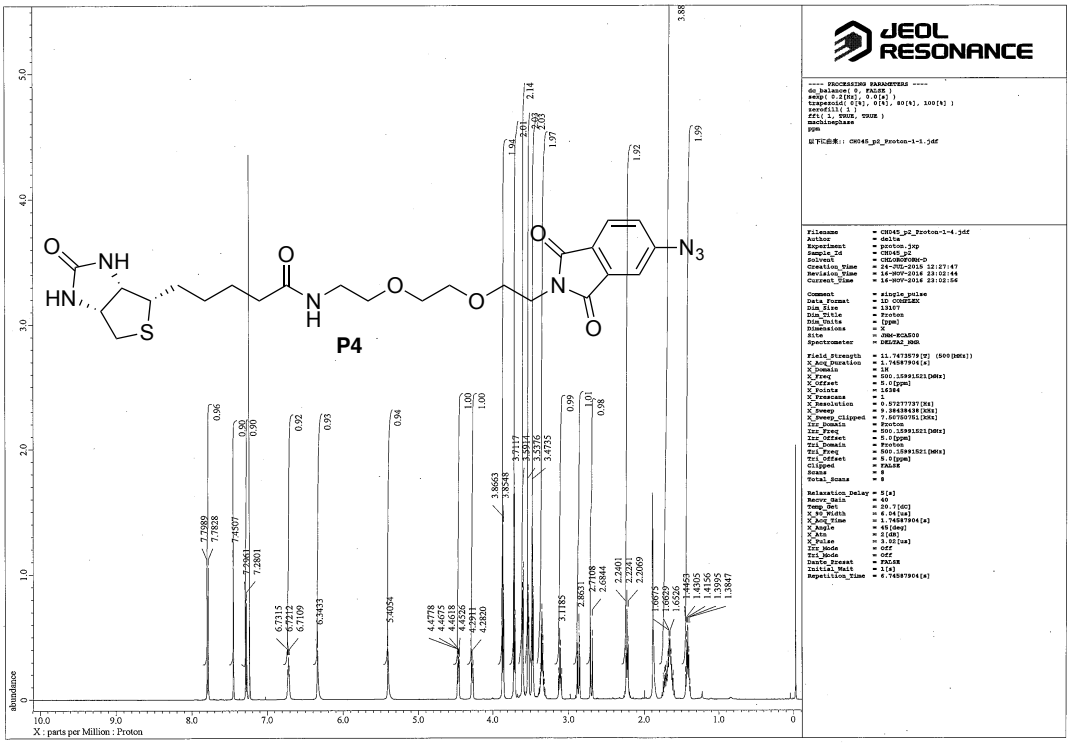
```
Filename        = CH099_Proton-1-5.jdf
Author          = delta
Experiment      = proton.jxp
Sample_Id       = CH099
Solvent         = METHANOL-D4
Creation_Time   = 22-FEB-2016 17:55:41
Revision_Time   = 14-NOV-2016 22:09:51
Current_Time    = 14-NOV-2016 22:10:01

Comment         = single_pulse
Data_Format     = 1D COMPLEX
Dim_Size        = 13107
Dim_Title       = Proton
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DELTA2_NMR

Field_Strength  = 11.7473579[T]  (500[MHz])
X_Acq_Duration  = 1.74587904[s]
X_Domain        = 1H
X_Freq          = 500.15991521[MHz]
X_Offset        = 6.0[ppm]
X_Points        = 16384
X_Prescans      = 1
X_Resolution    = 0.57277737[Hz]
X_Sweep         = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15991521[MHz]
Irr_Offset      = 6.0[ppm]
Tri_Domain      = Proton
Tri_Freq        = 500.15991521[MHz]
Tri_Offset      = 6.0[ppm]
Clipped         = FALSE
Scans           = 8
Total_Scans     = 8

Relaxation_Delay = 5[s]
Recyc_Gain      = 40
Temp_Set        = 25.8[dC]
X_90_Width      = 6.04[us]
X_Acq_Time      = 1.74587904[s]
X_Angle         = 30[deg]
X_Atn           = 25[dB]
X_Pulse         = 2.00[us]
Irr_Mode        = Off
Tri_Mode        = Off
Decpls_Preset   = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 6.74587904[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
expr( 0.5[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: CH099_Carbon_copy1-1-1.jdf
```

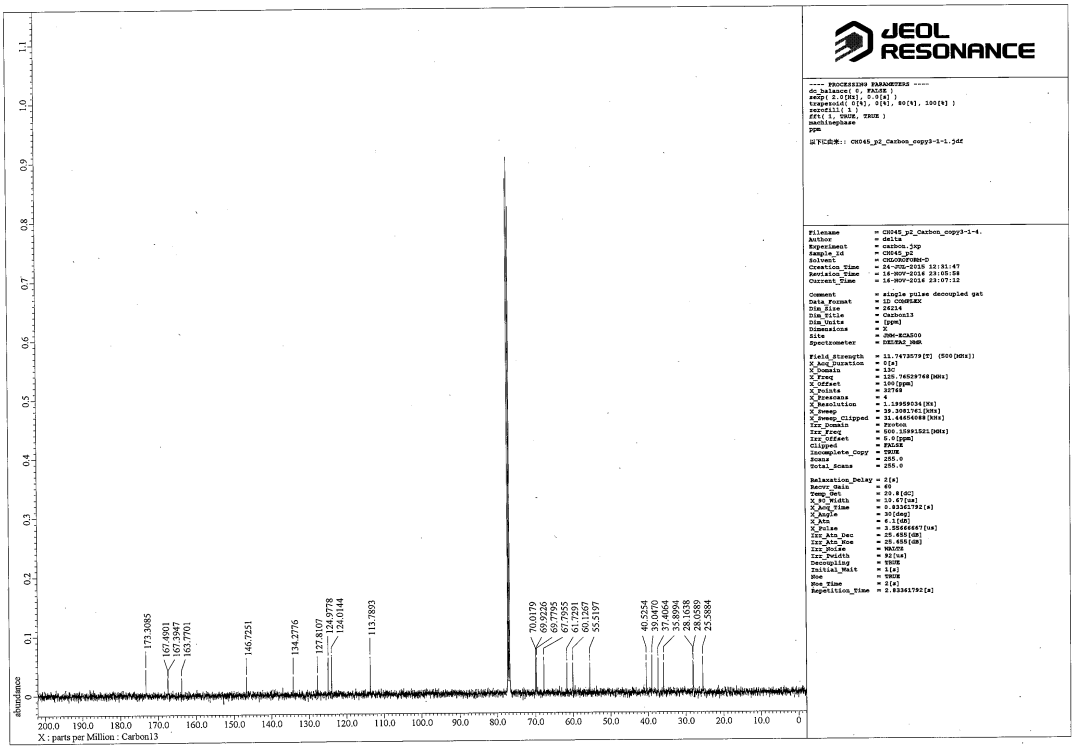
```
Filename        = CH099_Carbon_copy1-1-4.jdf
Author          = delta
Experiment      = carbon.jxp
Sample_Id       = CH099
Solvent         = METHANOL-D4
Creation_Time   = 22-FEB-2016 18:00:46
Revision_Time   = 14-NOV-2016 22:10:32
Current_Time    = 14-NOV-2016 22:11:22

Comment         = single pulse decoupled gat
Data_Format     = 1D COMPLEX
Dim_Size        = 26214
Dim_Title       = Carbon13
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DELTA2_NMR

Field_Strength  = 11.7473579[T]  (500[MHz])
X_Acq_Duration  = 1.[s]
X_Domain        = 13C
X_Freq          = 125.76529768[MHz]
X_Offset        = 100[ppm]
X_Points        = 32768
X_Prescans      = 4
X_Resolution    = 1.19859034[Hz]
X_Sweep         = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15991521[MHz]
Irr_Offset      = 6.0[ppm]
Clipped         = FALSE
Incomplete_Copy = TRUE
Scans           = 72
Total_Scans     = 72

Relaxation_Delay = 2[s]
Recyc_Gain      = 40
Temp_Set        = 21.9[dC]
X_90_Width      = 10.67[us]
X_Acq_Time      = 0.83361792[s]
X_Angle         = 30[deg]
X_Atn           = 4.1[dB]
X_Pulse         = 3.55666667[us]
Irr_Atn_Dec     = 25.65[dB]
Irr_Atn_Noe     = 28.65[dB]
Irr_Mode        = WALTZ5
Irr_Width       = 92[us]
Decoupling      = TRUE
Initial_Wait    = 1[s]
Noe             = TRUE
Noe_Time        = 2[s]
Repetition_Time = 2.83361792[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0 , FALSE )
exp[ 2.0[Hz], 0.5[s] ]
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, THUR, THUR )
machinephase
ppm

以下に由来:: CH045_p2_Proton-1-1.jdf
```

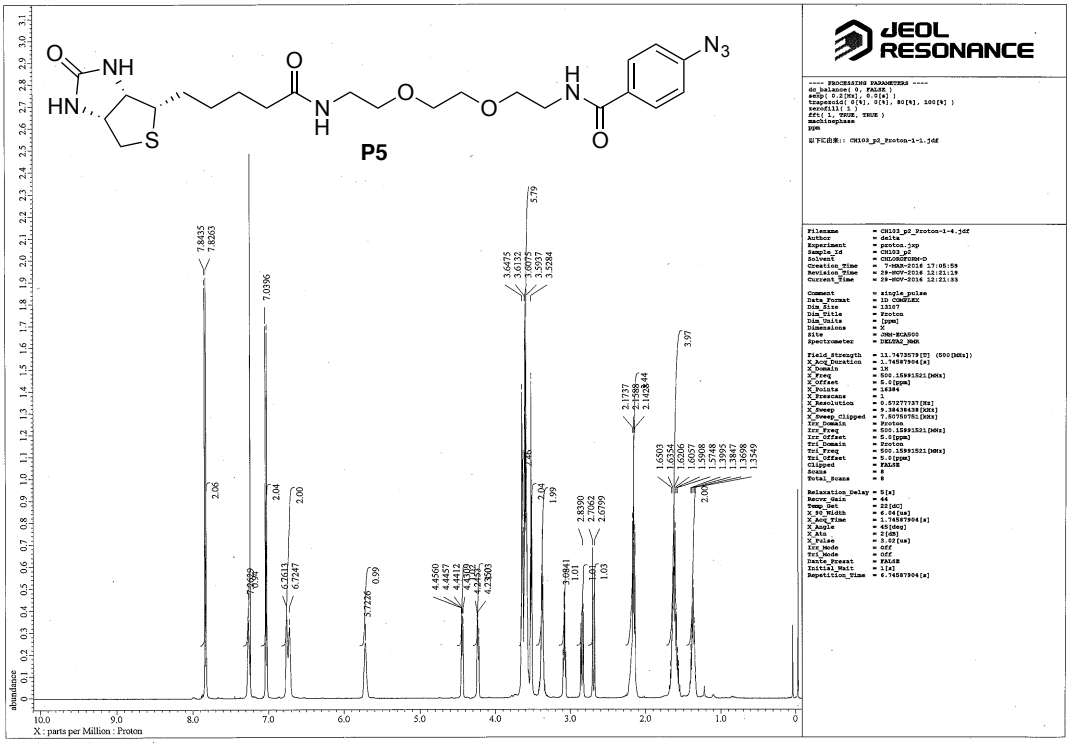
```
Filename          = CH045_p2_Proton-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = CH045_p2
Solvent           = CHLOROFORM-D
Creation_Time     = 24-JUL-2015 12:27:47
Revision_Time     = 14-NOV-2014 23:02:44
Current_Time      = 14-NOV-2014 23:02:56

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 1.74587904[s]
X_Domain          = 1H
X_Freq            = 500.15991521[MHz]
X_Offset          = 5.0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.57277737[Hz]
X_Sweep           = 9.38498438[KHz]
X_Sweep_Clipped   = 7.50790751[KHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Tri_Domain        = Proton
Tri_Freq          = 500.15991521[MHz]
Tri_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Set          = 20.7[dC]
X_90_Width        = 6.04[us]
X_Acq_Time        = 1.74587904[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 3.02[us]
Irr_Mode          = Off
Tri_Mode          = Off
DataFe_Preset     = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.74587904[s]
```



JEOL RESONANCE

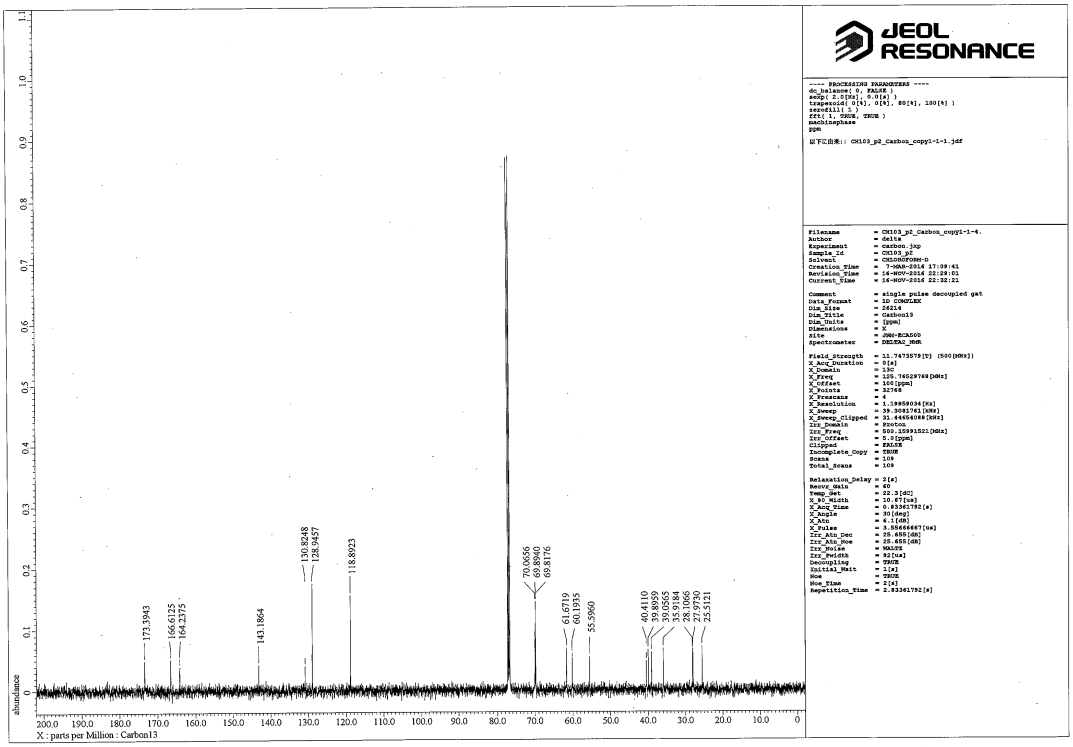
```
---- PROCESSING PARAMETERS ----
dc_balance( 0 , FALSE )
exp[ 2.0[Hz], 0.5[s] ]
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, THUR, THUR )
machinephase
ppm

以下に由来:: CH045_p2_Carbon_copy3-1-1.jdf
```

```
Filename          = CH045_p2_Carbon_copy3-1-4.
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = CH045_p2
Solvent           = CHLOROFORM-D
Creation_Time     = 24-JUL-2015 12:31:47
Revision_Time     = 14-NOV-2014 23:05:58
Current_Time      = 14-NOV-2014 23:07:12

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 24214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.74735579[T] (500[MHz])
X_Acq_Duration    = 0[s]
X_Domain          = 13C
X_Freq            = 125.76529768[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 1.19959034[Hz]
X_Sweep           = 39.3081361[KHz]
X_Sweep_Clipped   = 31.4465488[KHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Clipped           = FALSE
Incomplete_Copy   = TRUE
Scans             = 255.0
Total_Scans       = 255.0

Relaxation_Delay  = 2[s]
Recvr_Gain        = 40
Temp_Set          = 20.8[dC]
X_90_Width        = 10.67[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 4.2[dB]
X_Pulse           = 3.55666667[us]
Irr_Atn_Dec       = 25.65[dB]
Irr_Atn_Noe       = 25.65[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 92[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.83361792[s]
```



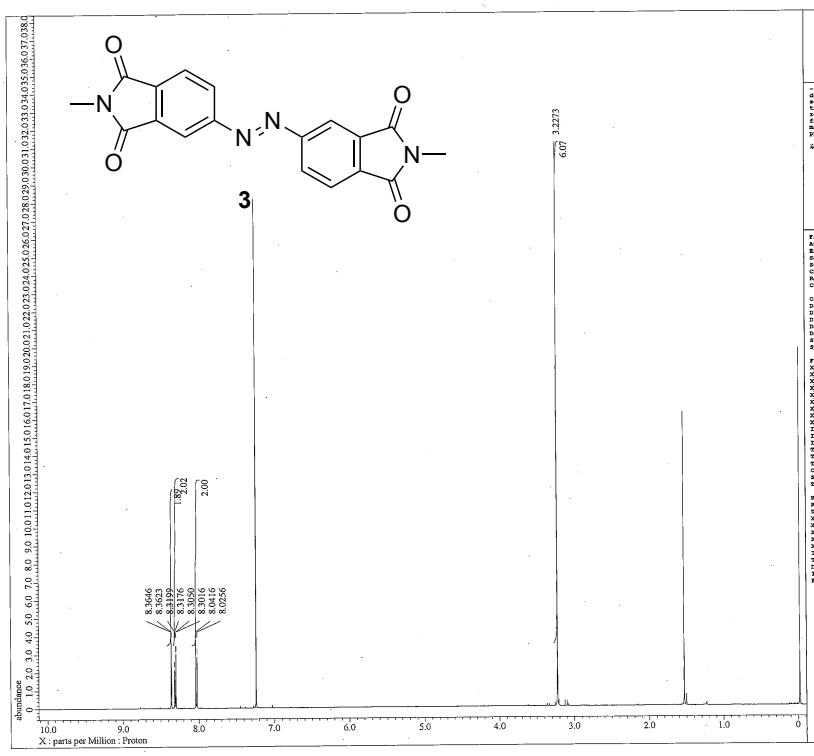

```
---- PROCESSING PARAMETERS ----
do_balance( 0, FALSE )
setQ( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下応用来:: CH103_p2_Proton-1-1.jdf
```

```
Filename          = CH103_p2_Proton-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = CH103_p2
Solvent           = CHLOROFORM-D
Creation_Time     = 7-NOA-2016 17:05:59
Revision_Time     = 29-NOV-2016 12:21:19
Current_Time      = 29-NOV-2016 12:21:33

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 1.74587904[s]
X_Domain          = 1H
X_Freq            = 500.15991521[MHz]
X_Offset          = 6.0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.57277737[Hz]
X_Sweep           = 9.38418438[KHz]
X_Sweep_Clipped   = 7.50750781[KHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 6.0[ppm]
Tri_Domain        = Proton
Tri_Freq          = 500.15991521[MHz]
Tri_Offset        = 9.0[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Set          = 22[dC]
X_90_Width        = 6.04[us]
X_Acq_Time        = 1.74587904[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 3.02[us]
Irr_Mode          = off
Tri_Mode          = off
Dads_Preset       = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.74587904[s]
```




```
---- PROCESSING PARAMETERS ----
do_balance( 0, FALSE )
setQ( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下応用来:: CH103_p2_Carbon_copy1-1-1.jdf
```

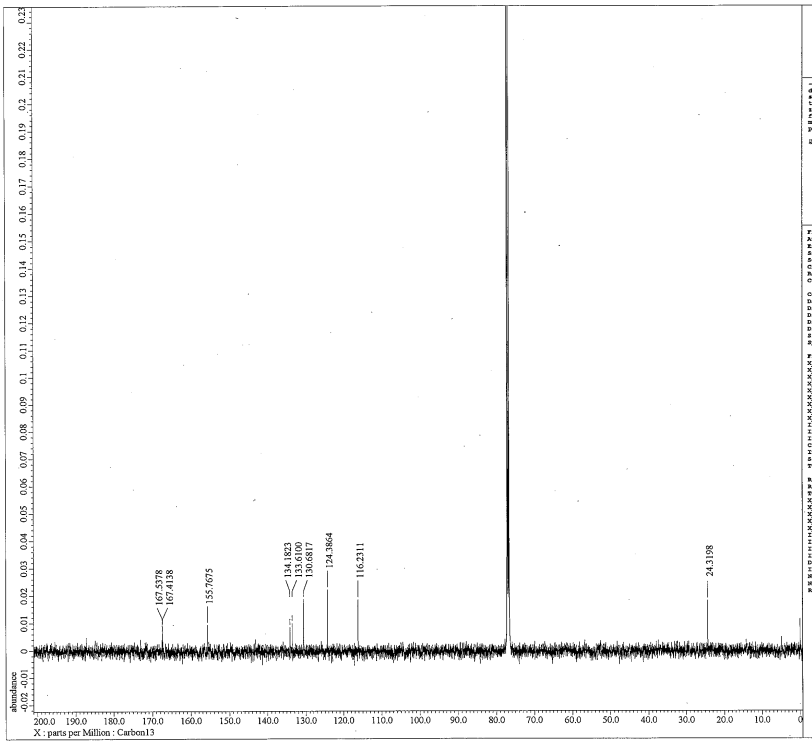
```
Filename          = CH103_p2_Carbon_copy1-1-4.
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = CH103_p2
Solvent           = CHLOROFORM-D
Creation_Time     = 7-NOA-2016 17:09:41
Revision_Time     = 14-NOV-2016 22:29:01
Current_Time      = 14-NOV-2016 22:32:21

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 0[s]
X_Domain          = 13C
X_Freq            = 125.76529798[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.19989034[Hz]
X_Sweep           = 39.3681761[KHz]
X_Sweep_Clipped   = 31.4465498[KHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 6.0[ppm]
Clipped           = FALSE
Decoupled_Copy    = TRUE
Scans             = 109
Total_Scans       = 109

Relaxation_Delay  = 2[s]
Recvr_Gain        = 40
Temp_Set          = 22.2[dC]
X_90_Width        = 10.67[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 4.1[dB]
X_Pulse           = 3.55666667[us]
Irr_Atn_Dec       = 25.65[dB]
Irr_Atn_Noe       = 25.65[dB]
Irr_Noise         = WALTZ
Irr_Width         = 82[ua]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.83361792[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
exp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下元素来:: CH080_p2_Proton-1-1.jdf
```

```
Filename        = CH080_p2_Proton-1-4.jdf
Author          = delta
Experiment      = proton.jxp
Sample_Id       = CH080_p2
Solvent         = CHLOROFORM-D
Creation_Time   = 15-DEC-2015 15:19:56
Revision_Time   = 17-NOV-2016 19:57:40
Current_Time    = 17-NOV-2016 19:57:51

Comment         = single_pulse
Data_Format     = 1D COMPLEX
Dim_Size        = 13107
Dim_Title       = Proton
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DELTA3_NMR

Field_Strength  = 11.7473579[T] (500[MHz])
X_Acq_Duration  = 1.74587904[s]
X_Domain        = 1H
X_Freq          = 500.15991521[MHz]
X_Offset        = 5.0[ppm]
X_Points        = 14984
X_Prescans      = 1
X_Resolution    = 0.57277737[Hz]
X_Sweep         = 8.58438438[KHz]
X_Sweep_Clipped = 7.50798751[KHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15991521[MHz]
Irr_Offset      = 5.0[ppm]
Trf_Domain      = Proton
Trf_Freq        = 500.15991521[MHz]
Trf_Offset      = 5.0[ppm]
Clipped         = FALSE
Scans           = 8
Total_Scans     = 8

Relaxation_Delay = 5[s]
Recyc_Gain      = 60
Temp_Set        = 22.4[dC]
X_90_Width      = 4.04[us]
X_Acq_Time      = 1.74587904[s]
X_Angle         = 45[deg]
X_Atn           = 2[dB]
X_Pulse         = 3.02[us]
Trf_Mode        = off
Trl_Mode        = off
Dmfe_Fixsat     = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 6.74587904[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
exp( 2.5[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下元素来:: CH080_Carbon_copy4-1-1.jdf
```

```
Filename        = CH080_Carbon_copy4-1-4.jdf
Author          = delta
Experiment      = carbon.jxp
Sample_Id       = CH080_13C
Solvent         = CHLOROFORM-D
Creation_Time   = 16-DEC-2015 21:26:27
Revision_Time   = 16-NOV-2016 20:04:18
Current_Time    = 16-NOV-2016 20:05:21

Comment         = single pulse decoupled gat
Data_Format     = 1D COMPLEX
Dim_Size        = 26214
Dim_Title       = Carbon13
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA500
Spectrometer    = DELTA2_NMR

Field_Strength  = 11.7473579[T] (500[MHz])
X_Acq_Duration  = 0[s]
X_Domain        = 13C
X_Freq          = 125.76529768[MHz]
X_Offset        = 100[ppm]
X_Points        = 32768
X_Prescans      = 4
X_Resolution    = 1.15958034[Hz]
X_Sweep         = 39.3082761[KHz]
X_Sweep_Clipped = 31.4465408[KHz]
Irr_Domain      = Proton
Irr_Freq        = 500.15991521[MHz]
Irr_Offset      = 5.0[ppm]
Clipped         = FALSE
Incomplete_Copy = TRUE
Scans           = 1475
Total_Scans     = 1475

Relaxation_Delay = 2[s]
Recyc_Gain      = 60
Temp_Set        = 22.8[dC]
X_90_Width      = 10.67[us]
X_Acq_Time      = 0.83361792[s]
X_Angle         = 30[deg]
X_Atn           = 6.1[dB]
X_Pulse         = 3.55666667[us]
Irr_Atn_Dec     = 28.65[dB]
Irr_Atn_Noe     = 28.65[dB]
Irr_Noise       = WALTZ
Irr_Width       = 92[us]
Decoupling      = TRUE
Initial_Wait    = 1[s]
Noe             = TRUE
Noe_Time        = 2[s]
Repetition_Time = 2.83361792[s]
```