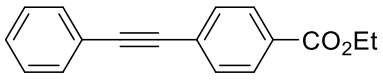
Supporting Information for Single Electron Transfer-Induced Coupling of Alkynylzinc Reagents with Aryl and Alkenyl Iodides

Keisho Okura, Hitomi Kawashima, Fumiko Tamakuni, Naoya Nishida, and Eiji Shirakawa

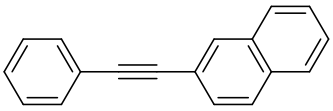
General Remarks. All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique or in a glove box under a nitrogen atmosphere. Nuclear magnetic resonance spectra were taken on a JEOL JNM LA500 spectrometer (^{1}H, 500 MHz and ^{13}C, 125 MHz), a JEOL JNM LA600 spectrometer (^{1}H, 600 MHz), or a JEOL JNM ECX500II spectrometer (^{1}H, 500 MHz). GC spectra were taken on Shimadzu GC-2014 equipped with capillary column ID-BPX5 (SGE). High-resolution mass spectra were obtained with a Bruker Daltonics microTOF-Q spectrometer (ESI and APCI). Preparative recycling gel permeation chromatography (GPC) was performed with JAI LC-908 equipped with JAIGEL-1H and -2H using chloroform as an eluent. Unless otherwise noted, reagents were commercially available and used without further purification. Toluene, tetrahydrofuran (THF) and diethyl ether (Et_2O) were purified by passing through an alumina/catalyst column system (GlassContour Co.). 2-Iodonaphthalene,[1] 2-(3-butenyl)phenyl iodide,[2] (*E*)-1-iodo-1-octene,[3] (*Z*)-1-iodo-1-octene,[4] and (*Z*)-β-iodostyrene[5] were prepared according to literature methods.

Preparation of Tris[4-(trifluoromethyl)phenyl]phosphine (L).[6] To a mixture of 4-bromo(trifluoromethyl)benzene (4.82 g, 21.4 mmol) and Et_2O (27.6 mL) in a 300 mL 3-necked round bottom flask was added dropwise a 1.6 M hexane solution of butyllithium (13.3 mL, 21.4 mmol) in Et_2O (18.4 mL) at 0 °C and the resulting mixture was stirred at 0 °C for 1.5 h. Dropwise addition of a solution of trichlorophosphine (0.98 g, 7.1 mmol) in Et_2O (30 mL) at 0 °C was followed by stirring at 0 °C for 1.5 h. After the temperature was raised to 23 °C, the reaction mixture was quenched with a saturated NH_4Cl aqueous solution (5.0 mL), extracted with Et_2O (10 mL x 3) and washed with brine (10 mL x 3). The combined organic layer was dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was subjected to silica gel chromatography (hexane) to give tris[4-(trifluoromethyl)phenyl]phosphine (**L**: 1.96 g, 58% yield) as a yellow solid. A part (1.00 g) of it was purified by GPC, cutting off the earlier and later fractions to obtain **L** (0.58 g, 58% yield) with a higher purity as a white solid. Phosphine **L** purchased from Wako Chemical Co. (product number 322-94901) was also used after the same GPC purification. Phosphine **L** that has not passed this purification process sometimes shows insufficient reproducibility on the coupling reaction of terminal alkynes with aryl and alkenyl iodides with the aid of diethylzinc. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (t, J = 7.8 Hz, 6 H), 7.63 (d, J = 8.0 Hz, 6 H).

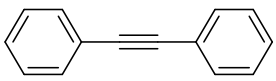
Coupling of Terminal Alkynes with Aryl and Alkenyl Iodides with the Aid of Diethylzinc (Table 2 and Scheme 1): Representative Procedure (Table 2, Entry 3). To a mixture of phenylethyne (**1a**: 30.6 mg, 0.300 mmol), ethyl 4-iodobenzoate (**2a**: 55.2 mg, 0.200 mmol) and tris[4-(trifluoromethyl)phenyl]phosphine (**L**: 9.3 mg, 0.020 mmol) in a 3 mL vial were added toluene (0.63 mL), THF (0.25 mL) and a 1.3 M toluene solution of Et_2Zn (0.12 mL, 0.15 mmol), and the resulting mixture was stirred at 110 °C for 24 h. After cooling, the reaction mixture was quenched with a saturated NH_4Cl aqueous solution (0.5 mL) and extracted with Et_2O (10 mL x 3). The combined organic layer was dried over $MgSO_4$, filtered and concentrated in vacuo. The residue was subjected to silica gel chromatography (hexane/ethyl acetate = 10/1, PTLC) to give ethyl 4-(phenylethynyl)benzoate (**3aa**: 48.2 mg, 96% yield).



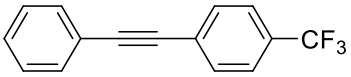
Ethyl 4-(phenylethynyl)benzoate (3aa).[7] An yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.41 (t, *J* = 7.1 Hz, 3 H), 4.39 (q, *J* = 7.1 Hz, 2 H), 7.34–7.40 (m, 3 H), 7.52–7.58 (m, 2 H), 7.59 (d, *J* = 8.1 Hz, 2 H), 8.03 (d, *J* = 8.1 Hz, 2 H).



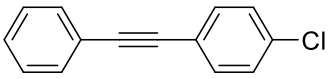
(2-Naphthyl)phenylethyne (3ab).[8] A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32–7.41 (m, 3 H), 7.46–7.54 (m, 2 H), 7.55–7.62 (m, 3 H), 7.79–7.86 (m, 3 H), 8.06 (s, 1 H).



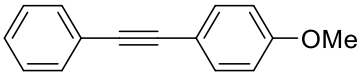
Diphenylethyne (3ac).[9] A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30–7.39 (m, 6 H), 7.50–7.58 (m, 4 H).



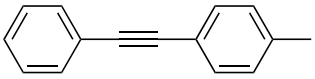
Phenyl[4-(trifluoromethyl)phenyl]ethyne (3ad).[10] A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34–7.41 (m, 3 H), 7.52–7.58 (m, 2 H), 7.61 (d, *J* = 8.7 Hz, 2 H), 7.63 (d, *J* = 8.7 Hz, 2 H).



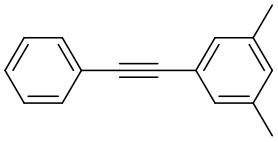
(4-Chlorophenyl)phenylethyne (3ae).[8] A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.7 Hz, 2 H), 7.33–7.39 (m, 3 H), 7.46 (d, *J* = 8.7 Hz, 2 H), 7.50–7.55 (m, 2 H).



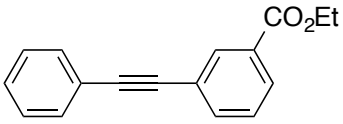
(4-Methoxyphenyl)phenylethyne (3af).[9] A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.83 (s, 3 H), 6.88 (d, *J* = 8.8 Hz, 2 H), 7.28–7.37 (m, 3 H), 7.47 (d, *J* = 8.8 Hz, 2 H), 7.49–7.55 (m, 2 H).



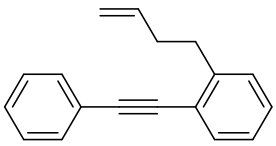
(4-Methylphenyl)phenylethyne (3ag).[9] A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.37 (s, 3 H), 7.16 (d, *J* = 8.6 Hz, 2 H), 7.29–7.37 (m, 3 H), 7.43 (d, *J* = 8.6 Hz, 2 H), 7.49–7.55 (m, 2 H).



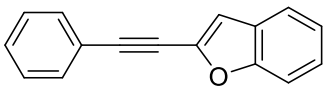
(3,5-Dimethylphenyl)phenylethyne (3ah).[10] An yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.32 (s, 6 H), 6.97 (s, 1 H), 7.18 (s, 2 H), 7.29–7.37 (m, 3 H), 7.49–7.55 (m, 2 H).



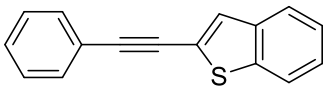
Ethyl 3-(phenylethynyl)benzoate (3ai).[11] A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.42 (t, *J* = 7.2 Hz, 3 H), 4.40 (q, *J* = 7.2 Hz, 2 H), 7.32–7.40 (m, 3 H), 7.43 (t, *J* = 7.7 Hz, 1 H), 7.52–7.58 (m, 2 H), 7.70 (dt, *J* = 8.0, 1.4 Hz, 1 H), 8.01 (dt, *J* = 7.5 Hz, 1.4 Hz, 1 H) 8.21 (t, *J* = 1.4 Hz, 1 H).



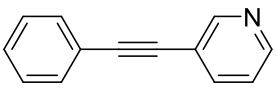
[2-(3-Butenyl)phenyl]phenylethyne (3aj). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.47 (q, *J* = 7.4 Hz, 2 H), 2.96 (t, *J* = 7.9 Hz, 2 H), 5.00 (d, *J* = 10.2 Hz, 1 H), 5.07 (d, *J* = 17.1 Hz, 1 H), 5.92 (ddt, *J* = 17.1, 10.2, 6.5 Hz, 1 H), 7.19 (t, *J* = 7.2 Hz, 1 H), 7.23 (d, *J* = 6.5 Hz, 1 H), 7.26 (t, *J* = 7.1 Hz, 1 H), 7.31–7.39 (m, 3 H), 7.49–7.56 (m, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 34.3, 34.7, 88.1, 93.1, 114.9, 122.6, 123.5, 125.9, 128.2, 128.4, 128.9, 131.5, 132.2, 138.2, 143.9. HRMS (APCI) Calcd for $C_{18}H_{16}$: $[M+H]^+$, 233.1325. Found: m/z 233.1330.



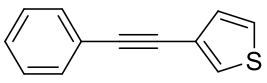
2-Benzofuranylphenylethyne (3ak).[12] An yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.01 (s, 1 H), 7.30 (t, *J* = 7.5 Hz, 1 H), 7.35 (t, *J* = 8.0 Hz, 1 H), 7.37–7.42 (m, 3 H), 7.48 (d, *J* = 8.3 Hz, 1 H), 7.55–7.62 (m, 3 H).



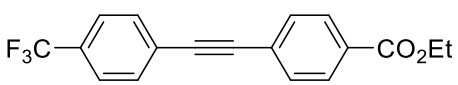
2-Benzothienylphenylethyne (3al).[13] A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34–7.42 (m, 5 H), 7.51 (s, 1 H), 7.53–7.59 (m, 2 H), 7.74–7.81 (m, 2 H).



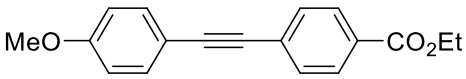
Phenyl(3-pyridyl)ethyne (3am).[7] A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (dd, *J* = 7.9, 5.0 Hz, 1 H), 7.34–7.42 (m, 3 H), 7.52–7.59 (m, 2 H), 7.81 (dt, *J* = 7.9, 1.8 Hz, 1 H), 8.55 (d, *J* = 4.5 Hz, 1 H), 8.77 (s, 1 H).



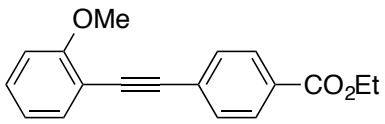
Phenyl(3-thienyl)ethyne (3an).[14] A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.20 (dd, *J* = 5.0, 1.1 Hz, 1 H), 7.30 (dd, *J* = 5.0, 3.0 Hz, 1 H), 7.31–7.38 (m, 3 H), 7.48–7.55 (m, 3 H).



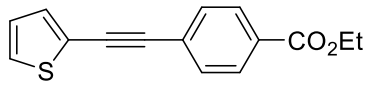
Ethyl 4-[4-(trifluoromethyl)phenylethynyl]benzoate (3ba).[15] An yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.41 (t, *J* = 7.1 Hz, 3 H), 4.40 (q, *J* = 7.1 Hz, 2 H), 7.61 (d, *J* = 8.6 Hz, 2 H), 7.63 (d, *J* = 8.7 Hz, 2 H), 7.65 (d, *J* = 8.7 Hz, 2 H), 8.05 (d, *J* = 8.6 Hz, 2 H).



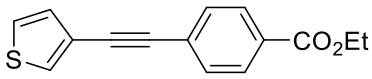
Ethyl 4-(4-methoxyphenylethynyl)benzoate (3ca).[10] A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.40 (t, *J* = 7.1 Hz, 3 H), 3.84 (s, 3 H), 4.39 (q, *J* = 7.1 Hz, 2 H), 6.89 (d, *J* = 8.9 Hz, 2 H), 7.49 (d, *J* = 8.9 Hz, 2 H), 7.56 (d, *J* = 8.1 Hz, 2 H), 8.01 (d, *J* = 8.1 Hz, 2 H).



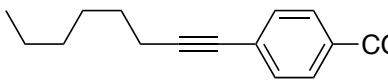
Ethyl 4-(2-methoxyphenylethynyl)benzoate (3da). An yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.41 (t, *J* = 7.1 Hz, 3 H), 3.93 (s, 3 H), 4.39 (q, *J* = 7.1 Hz, 2 H), 6.92 (d, *J* = 8.3 Hz, 1 H), 6.96 (t, *J* = 7.5 Hz, 1 H), 7.34 (td, *J* = 7.7, 2.0 Hz, 1 H), 7.51 (dd, *J* = 7.5, 2.0 Hz, 1 H), 7.61 (d, *J* = 8.1 Hz, 2 H), 8.02 (d, *J* = 8.1 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.3, 55.8, 61.1, 88.8, 92.7, 110.7, 111.9, 120.5, 128.2, 129.4, 129.6, 130.3, 131.5, 133.6, 160.1, 166.1. HRMS (ESI) Calcd for $C_{18}H_{16}O_3$: $[M+Na]^+$, 303.0992. Found: m/z 303.0997.



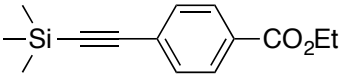
Ethyl 4-(2-thienylethynyl)benzoate (3ea). A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.41 (t, *J* = 7.1 Hz, 3 H), 4.39 (q, *J* = 7.1 Hz, 2 H), 7.03 (dd, *J* = 5.0, 3.5 Hz, 1 H), 7.32 (dd, *J* = 3.5, 1.5 Hz, 1 H), 7.34 (dd, *J* = 5.0, 1.5 Hz, 1 H), 7.56 (d, *J* = 8.7 Hz, 2 H), 8.02 (d, *J* = 8.7 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.3, 61.1, 85.5, 92.4, 122.7, 127.2, 127.5, 128.0, 129.5, 139.9, 131.2, 132.5, 166.0. HRMS (ESI) Calcd for $C_{15}H_{12}O_2S$: $[M+Na]^+$, 279.0450. Found: m/z 279.0452.



Ethyl 4-[3-thienylethynyl]benzoate (3fa). A brown solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.40 (t, *J* = 7.1 Hz, 3 H), 4.39 (q, *J* = 7.1 Hz, 2 H), 7.21 (dd, *J* = 5.0, 1.2 Hz, 1 H), 7.32 (dd, *J* = 5.0, 2.9 Hz, 1 H), 7.54–7.59 (m, 1 H) 7.56 (d, *J* = 8.7, 2 H), 8.02 (d, *J* = 8.7, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.3, 61.1, 87.4, 88.3, 121.8, 125.5, 127.8, 129.3, 129.5, 129.77, 129.79, 131.3, 166.0. HRMS (ESI) Calcd for $C_{15}H_{12}O_2S$: $[M+Na]^+$, 279.0450. Found: m/z 279.0448.

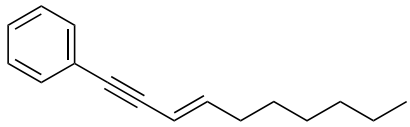


Ethyl 4-(1-octyn-1-yl)benzoate (3ga). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.91 (t, *J* = 6.5 Hz, 3 H), 1.27–1.40 (m, 4 H), 1.39 (t, *J* = 7.1 Hz, 3 H), 1.46 (quint, *J* = 7.4 Hz, 2 H), 1.61 (quint, *J* = 7.2 Hz, 2 H), 2.42 (t, *J* = 7.1 Hz, 2 H), 4.37 (q, *J* = 7.1 Hz, 2 H), 7.43 (d, *J* = 8.1 Hz, 2 H), 7.95 (d, *J* = 8.1 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 14.3, 19.5, 22.5, 28.5, 28.6, 31.3, 61.0, 80.1, 93.9, 128.8, 129.2, 129.3, 131.4, 166.2. HRMS (ESI) Calcd for $C_{17}H_{22}O_2$: $[M+Na]^+$, 281.1512. Found: m/z 281.1519.

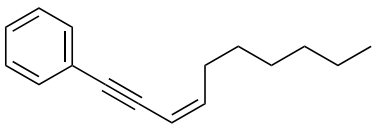


Ethyl 4-(trimethylsilylethynyl)benzoate (3ha).[16] An yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.26 (s, 9 H), 1.39 (t, *J* = 7.1 Hz, 3 H), 4.37 (q, *J* = 7.1 Hz, 2 H), 7.51 (d, *J* = 8.4 Hz, 2 H), 7.97 (d, *J* =

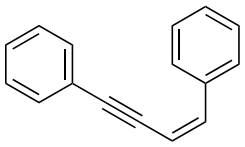
8.4 Hz, 2 H).



(*E*)-1-Phenyl-3-decen-1-yne ((*E*)-5aa).[17] An yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89 (t, *J* = 7.0 Hz, 3 H), 1.22–1.37 (m, 6 H), 1.43 (quint, *J* = 7.3 Hz, 2 H), 2.16 (qd, *J* = 7.3, 1.5 Hz, 2 H), 5.69 (dt, *J* = 15.7, 1.6 Hz, 1 H), 6.25 (dt, *J* = 15.7, 7.1 Hz, 1 H), 7.27–7.33 (m, 3 H), 7.38–7.46 (m, 2 H).



(*Z*)-1-Phenyl-3-decen-1-yne ((*Z*)-5aa).[17] An yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89 (t, *J* = 6.8 Hz, 3 H), 1.22–1.42 (m, 6 H), 1.49 (quint, *J* = 7.2 Hz, 2 H), 2.40 (q, *J* = 7.4 Hz, 2 H), 5.67 (d, *J* = 10.8 Hz, 1 H), 5.98 (dt, *J* = 10.8, 7.4 Hz, 1 H), 7.26–7.36 (m, 3 H), 7.39–7.48 (m, 2 H).



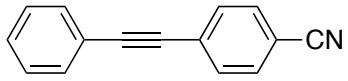
(*Z*)-1,4-Diphenyl-but-1-en-3-yne ((*Z*)-5ab).[18] An yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.93 (d, *J* = 12.0 Hz, 1 H), 6.71 (d, *J* = 12.0 Hz, 1 H), 7.28–7.45 (m, 6 H), 7.46–7.53 (m, 2 H), 7.93 (d, *J* = 8.4 Hz, 2 H).

Behavior of Organozinc Species toward Aryl Iodides (Table 3). To phenylethyne (**1a**: 30.6 mg, 0.300 mmol) in a 3 mL vial were added toluene (0.63 mL for entries 1 and 3; 0.51 mL for entry 2), THF (0.25 mL) and a 1.3 M toluene solution of Et_2Zn (0.12 mL, 0.15 mmol for entries 1 and 3; 0.24 mL, 0.30 mmol for entry 2), and the resulting mixture was stirred at 110 °C for 10 h. Addition of ethyl 4-iodobenzoate (**2a**: 55.2 mg, 0.200 mmol) and tris[4-(trifluoromethyl)phenyl]phosphine (**L**: 9.3 mg, 0.020 mmol) at 23 °C was followed by stirring at 110 °C for 24 h. After cooling, the reaction mixture was quenched with a saturated NH_4Cl aqueous solution (0.5 mL). For entry 1, Et_2Zn (0.12 mL, 0.15 mmol) in toluene (0.63 mL) and THF (0.25 mL) was treated with **2a** and **L** without treatment of **1a**. The conversion of **2a** and the yields of ethyl 4-(phenylethynyl)benzoate (**3aa**), ethyl 4-ethylbenzoate (**6a**), ethyl benzoate (**7a**) and diethyl biphenyl-4,4'-dicarboxylate (**8a**) were determined by GC using decane as an internal standard.

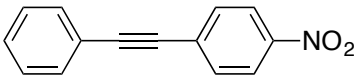
NMR Experiment Pursuing Transformation of the Et–Zn Moieties to PhC≡C–Zn Moieties (Scheme 2). To a solution of phenylethyne (**1a**: 30.6 mg, 0.300 mmol) in toluene-d_8 (0.63 mL) and THF-d_8 (0.25 mL) in a 3 mL vial was added a 1.3 M toluene-d_8 solution of Et_2Zn (0.12 mL, 0.15 mmol). The resulting mixture was stirred at 110 °C for a time specified in Scheme 2, and a aliquot (0.15 mL) of the solution was subjected to ^{1}H NMR analysis after dilution with 0.45 mL of toluene-d_8. The peaks concerning the ethyl units of Et_2Zn (**A**) and PhC≡CZnEt (**B**) in toluene-d_8–THF-d_8 (15:1) appear as follows: for **A**,œ 0.20–0.30 (m, 4 H), 1.20–1.35 (m, 6 H); for **B**, 0.35–0.45 (m, 2 H), 1.35–1.50 (m, 3 H). The peaks concerning the phenylethynyl units of PhC≡CZnEt (**B**) and $(PhC≡C)_2Zn$ (**C**) in toluene-d_8–THF-d_8 (15:1) are

not well divided and appear as follows: 6.95–7.03 (m, 1 or 2 H), 7.05–7.10 (m, 2 or 4 H), 7.60–7.66 (m, 2 or 4 H).

Coupling of Alkynylzinc Halides, Prepared by Transmetalation between an Alkynyllithium and a Zinc Halide, with Aryl Iodides (Scheme 3): Representative Procedure (Synthesis of 3aa by Use of ZnI_2). To a solution of phenylethyne (**1a**: 32.7 mg, 0.320 mmol) in THF (0.25 mL) in a 15 mL pressure-resistant tube (Ace Pressure Tube, Ace Glass 864804) was added a 1.6 M hexane solution of butyllithium (0.19 mL, 0.30 mmol) at –78 °C. After stirring for 3 h at 0 °C, a THF solution (0.5 mL) of ZnI_2 (95.8 mg, 0.300 mmol, 99.999% trace metals basis, Aldrich Co., product number 409278) was added to the resulting mixture at 0 °C and stirring was continued for 1 h. Addition of ethyl 4-iodobenzoate (**2a**: 55.2 mg, 0.200 mmol), tris[4-(trifluoromethyl)phenyl]phosphine (**L**: 9.3 mg, 0.020 mmol) and toluene (0.25 mL) at 23 °C was followed by stirring at 110 ºC for 24 h. After cooling, the reaction mixture was quenched with a saturated NH_4Cl aqueous solution (0.5 mL) and extracted with Et_2O (10 mL x 3). The combined organic layer was dried over $MgSO_4$, filtered and concentrated in vacuo. The residue was subjected to silica gel chromatography (hexane/ethyl acetate = 10/1, PTLC) to give ethyl 4-(phenylethynyl)benzoate (**3aa**: 45.4 mg, 91% yield).



(4-Cyanophenyl)phenylethyne (3ao).[7] An orange solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35–7.41 (m, 3 H), 7.51–7.57 (m, 2 H), 7.61 (d, *J* = 8.3 Hz, 2 H), 7.64 (d, *J* = 8.3 Hz, 2 H).



(4-Nitrophenyl)phenylethyne (3ap).[8] An yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35–7.44 (m, 3 H), 7.52–7.60 (m, 2 H), 7.67 (d, *J* = 8.9 Hz, 2 H), 8.23 (d, *J* = 8.9 Hz, 2 H).

Acceleration of the Reaction by Addition of a Single Electron Donor (Scheme 5). To a solution of ethyl 4-iodobenzoate (**2a**: 55.2 mg, 0.200 mmol) in THF (0.060 mL) in a 3 mL vial were added a 0.50 M THF solution of lithium 4,4'-di-*tert*-butylbiphenylide (LDBB: 0.040 mL, 0.020 mmol for the second and third entries) or THF (0.040 mL for the first entry). Addition of a solution of bis(phenylethynyl)zinc, prepared from phenylethyne (**1a**: 30.6 mg, 0.300 mmol) and a 1.3 M toluene solution of Et_2Zn (0.12 mL, 0.15 mmol) in toluene (0.18 mL) and THF (0.15 mL) at 110 °C for 10 h in the presence or absence of tris[4-(trifluoromethyl)phenyl]phosphine (**L**: 9.3 mg, 0.020 mmol), was followed by stirring at 80 ºC for 24 h. After cooling, the reaction mixture was quenched with a saturated NH_4Cl aqueous solution (0.5 mL). The conversion of **2a** and the yield of ethyl 4-(phenylethynyl)benzoate (**3aa**) were determined by GC using decane as an internal standard.

References

1 T. Satoh, K. Itoh, M. Miura and M. Nomura, *Bull. Chem. Soc. Jpn.*, 1993, **66**, 2121–2123.

2 E. Negishi, C. Copéret, S. Ma, T. Mita, T. Sugihara and J. M. Tour, *J. Am. Chem. Soc.*, 1996, **118**, 5904–5918.

3 B. M. Trost and M. T. Rudd, *Org. Lett.*, 2003, **5**, 4599–4602.

4 H. C. Brown, T. Hamaoka, N. Ravindran, C. Subrahmanyam, V. Somayaji and N. G. Bhat, *J. Org. Chem.*, 1989, **54**, 6075–6079.

5 F. Chen and X. Zhang, *Chem. Lett.*, 2011, **40**, 978–979.

6 P. Suomalainen, H. K. Reinius, H. Riihimäki, R. H. Laitinen, S. Jääskeläinen, M. Haukka, J. T. Pursiainen, T. A. Pakkanen and A. O. Krause, *J. Mol. Catal. A: Chem.*, 2001, **169**, 67–78.

7 H. Kim and P. H. Lee, *Adv. Synth. Catal.*, 2009, **351**, 2827–2832.

8 J. Liu, F. Dai, Z. Yang, S. Wang, K. Xie, A. Wang, X. Chen and Z. Tan, *Tetrahedron Lett.*, 2012, **53**, 5678–5683.

9 V. Mouriès, R. Waschbüsch, J. Carran and P. Savignac, *Synthesis*, 1998, 271–274.

10 E. Fager-Jokela, M. Muuronen, M. Patzschke and J. Helaja, *J. Org. Chem.*, 2012, **77**, 9134–9147.

11 H. Kim and P. H. Lee, *Adv. Synth. Catal.*, 2009, **351**, 2827–2832.

12 L. Lu, H. Yan, P. Sun, Y. Zhu, H. Yang, D. Liu, G. Rong and J. Mao, *Eur. J. Org. Chem.*, 2013, 1644–1648.

13 A. R. Katritzky, A. A. A. Abdel-Fattah and M. Wang, *J. Org. Chem.*, 2002, **67**, 7526–7529.

14 C. Torborg, J. Huang, T. Schulz, B. Schäffner, A. Zapf, A. Spannenberg, A. Börner and M. Beller, *Chem. Eur. J.*, 2009, **15**, 1329–1336.

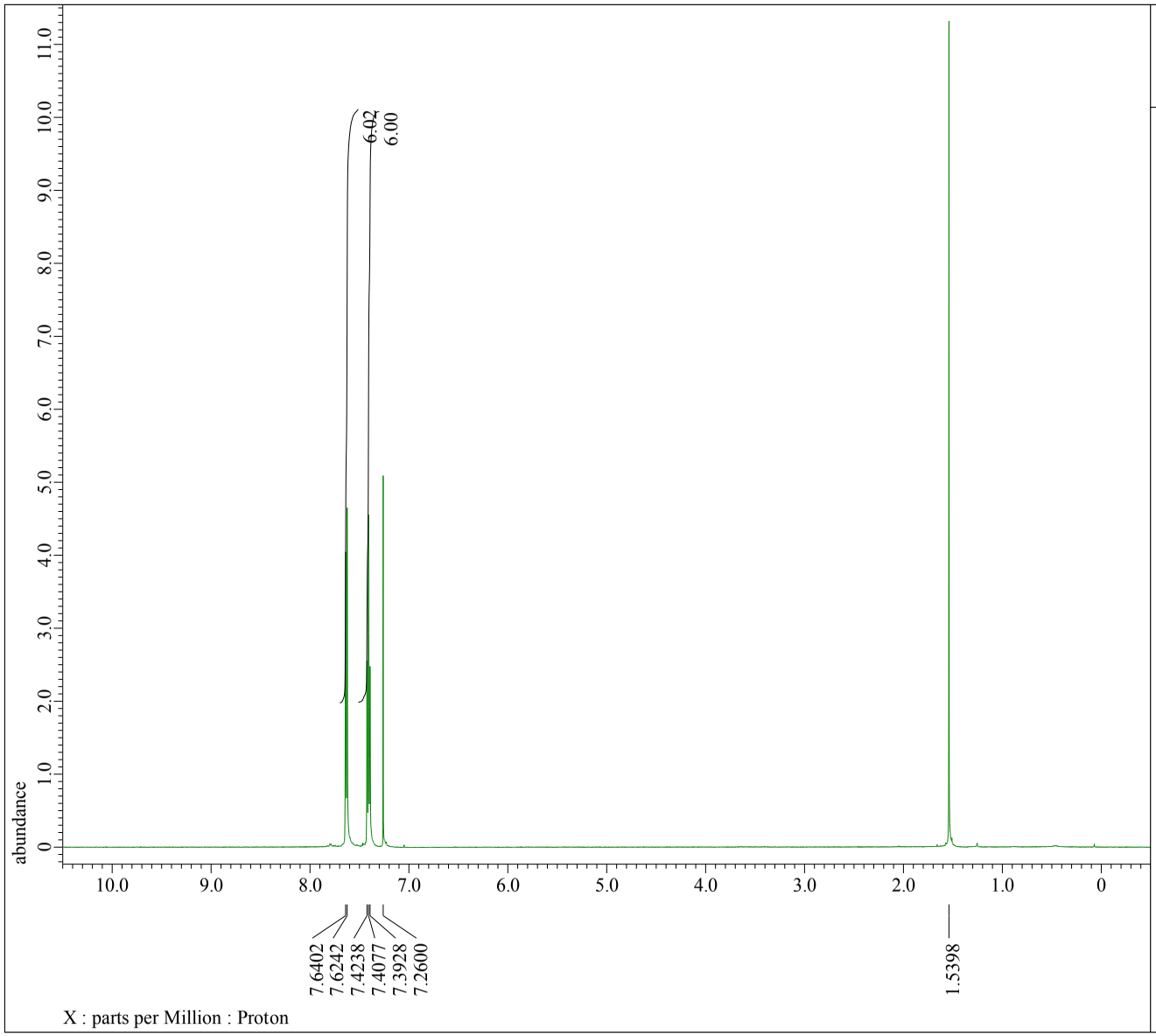
15 L. Melzig, A. Metzger and P. Knochel, *Chem. Eur. J.*, 2011, **17**, 2948–2956.

16 Y.-L. Zhao, L. Liu, W. Zhang, C.-H. Sue, Q. Li, O. Š. Miljanić, O. M. Yaghi and J. F. Stoddart, *Chem. Eur. J.*, 2009, **15**, 13356–13380.

17 M. Hoshi and K. Shirakawa, *Synlett*, 2002, 1101–1104.

18 T. Gehrmann, S. A. Scholl, J. L. Fillol, H. Wadepohl and L. H. Gade, *Chem. Eur. J.*, 2012, **18**, 3925–3941.

NMR Spectra of the Products



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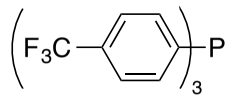

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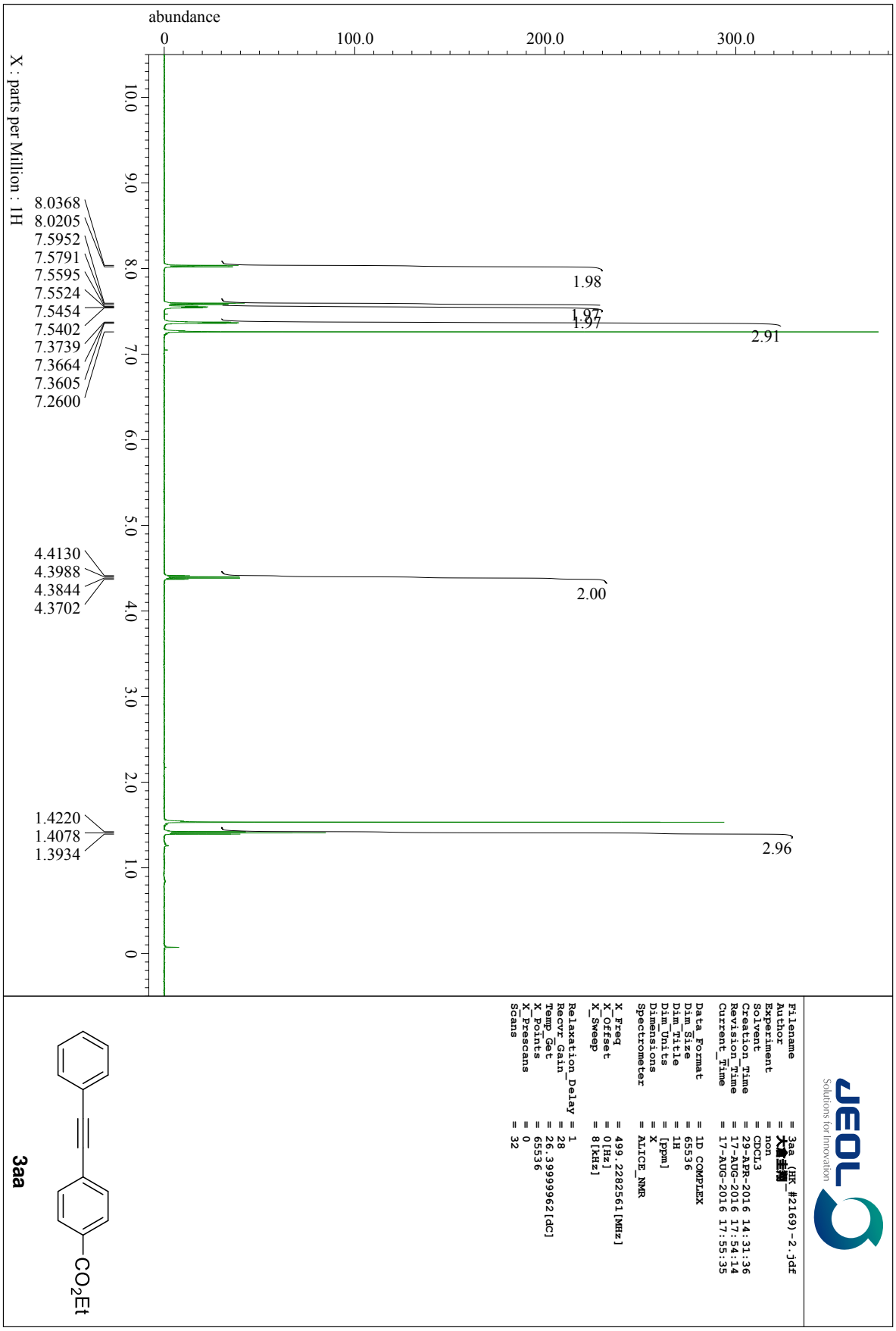
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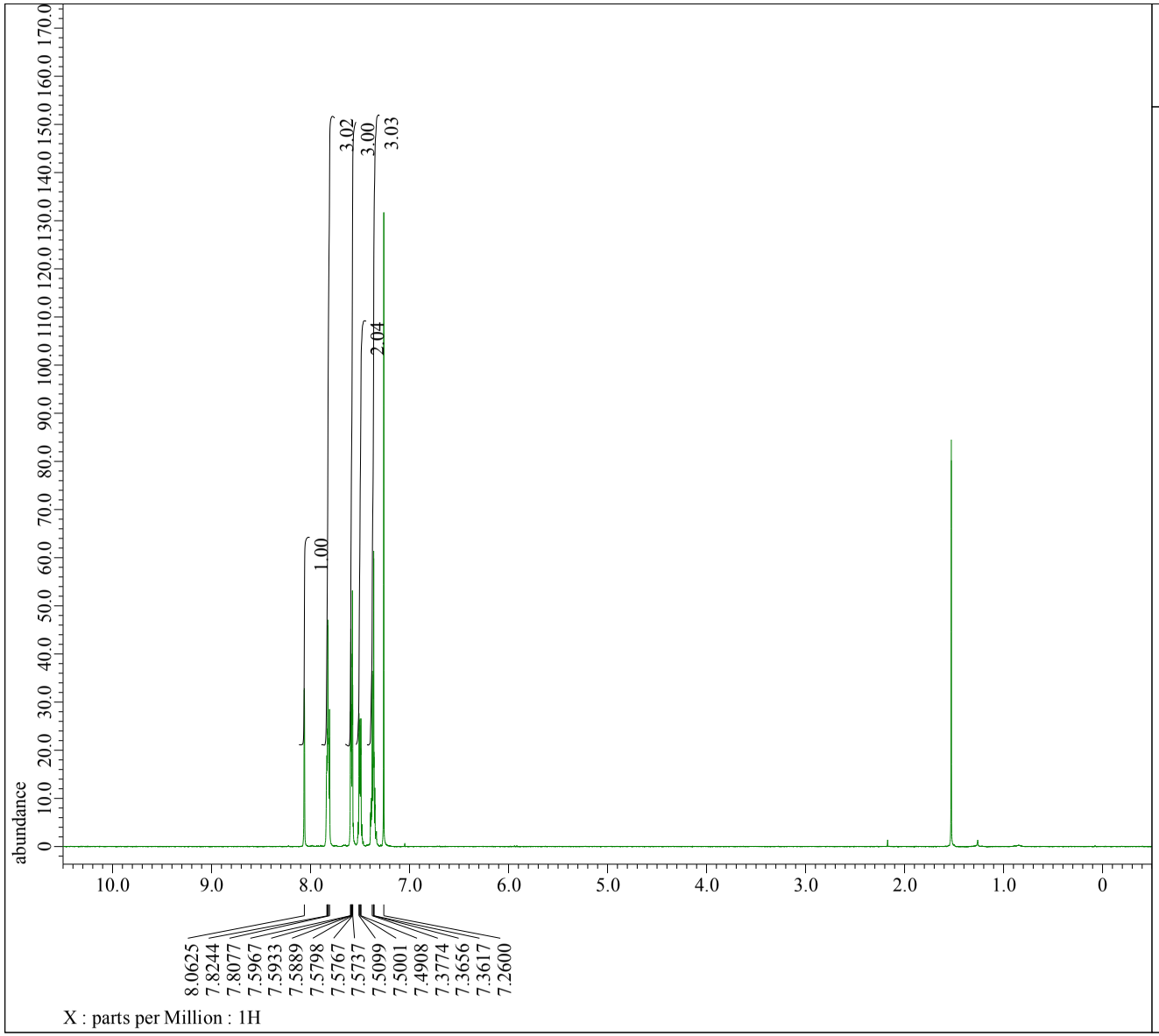

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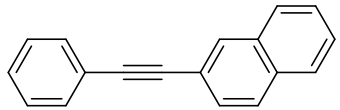

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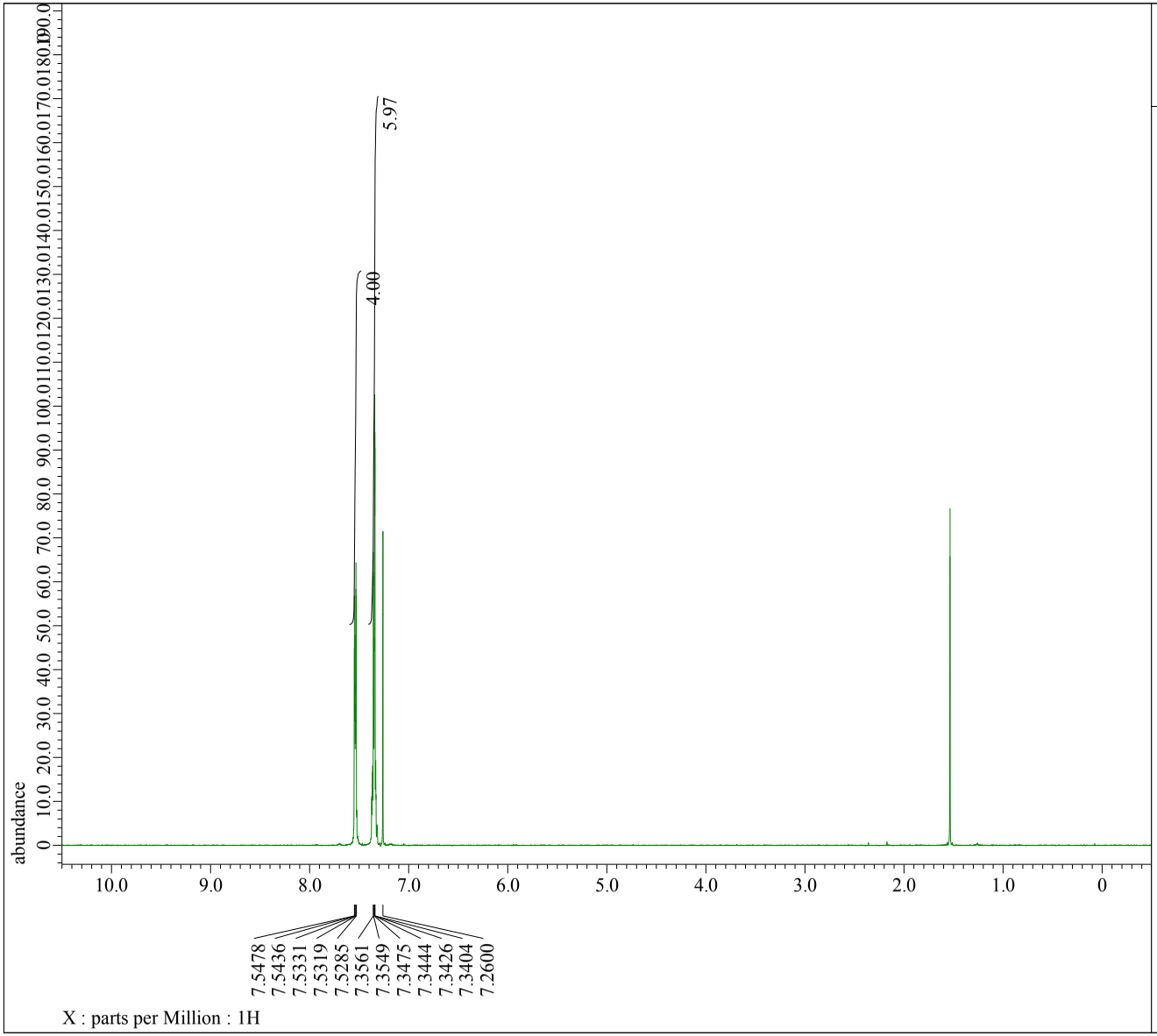

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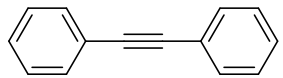

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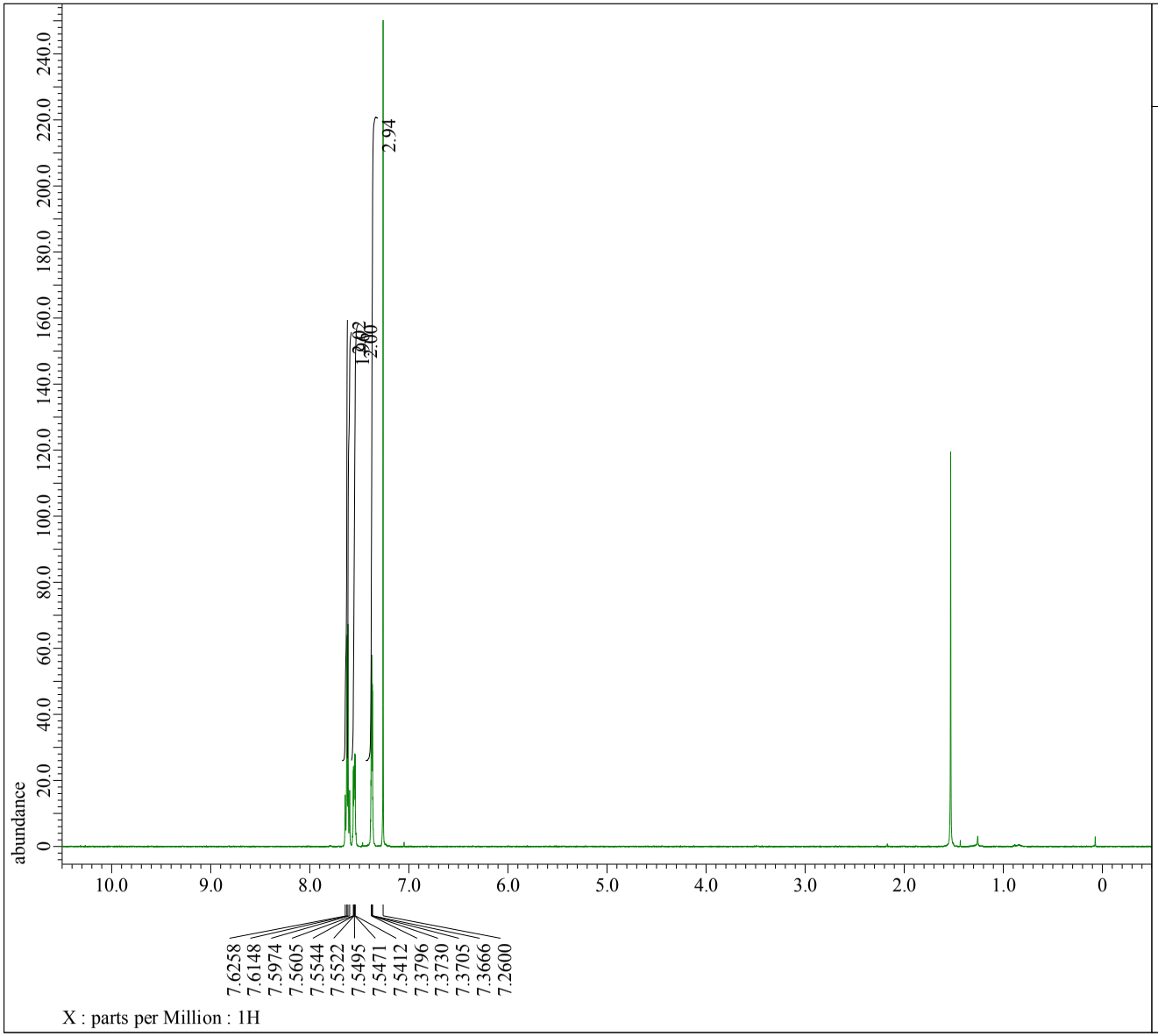

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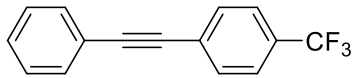

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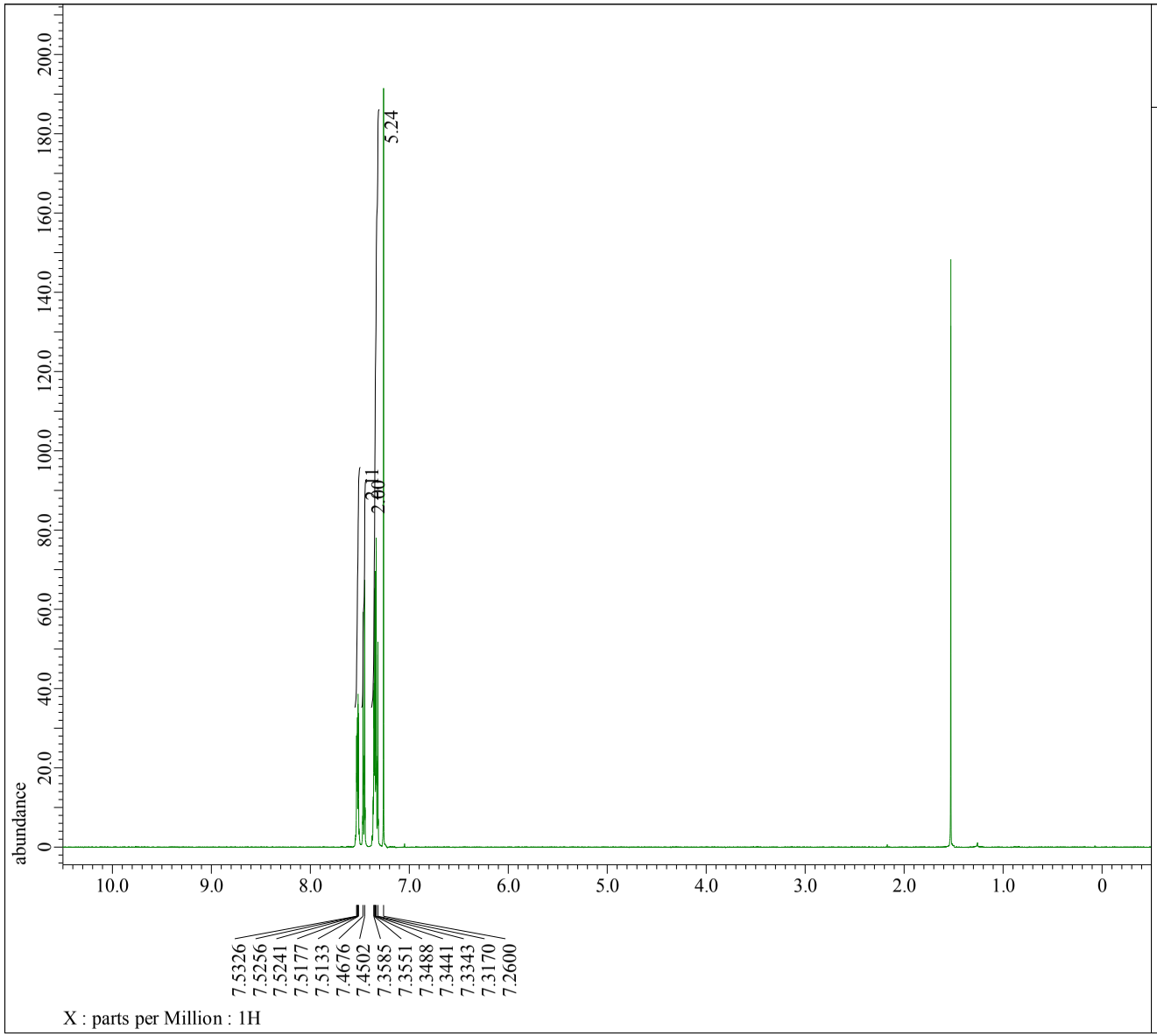

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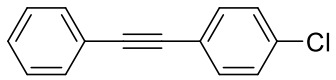

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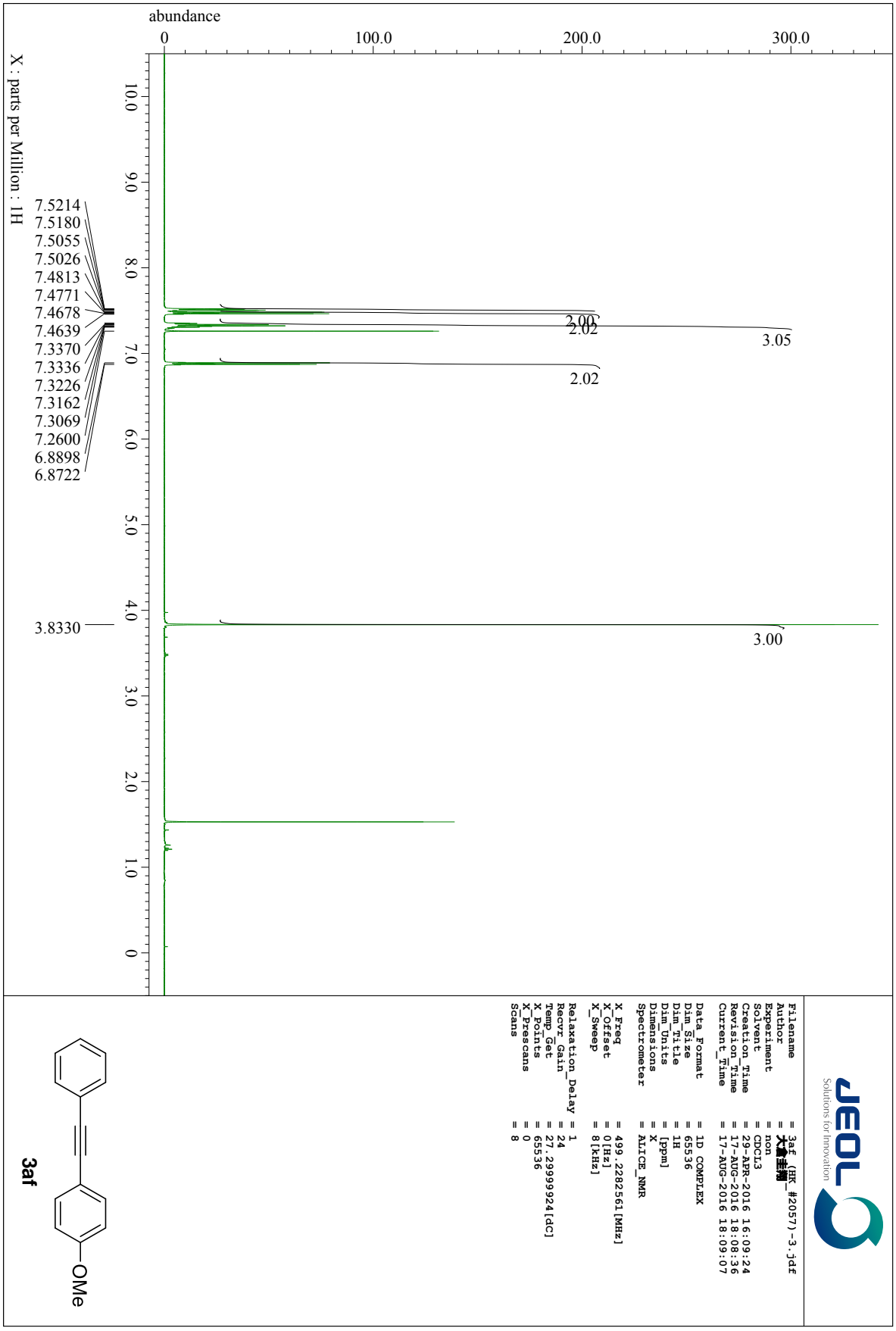
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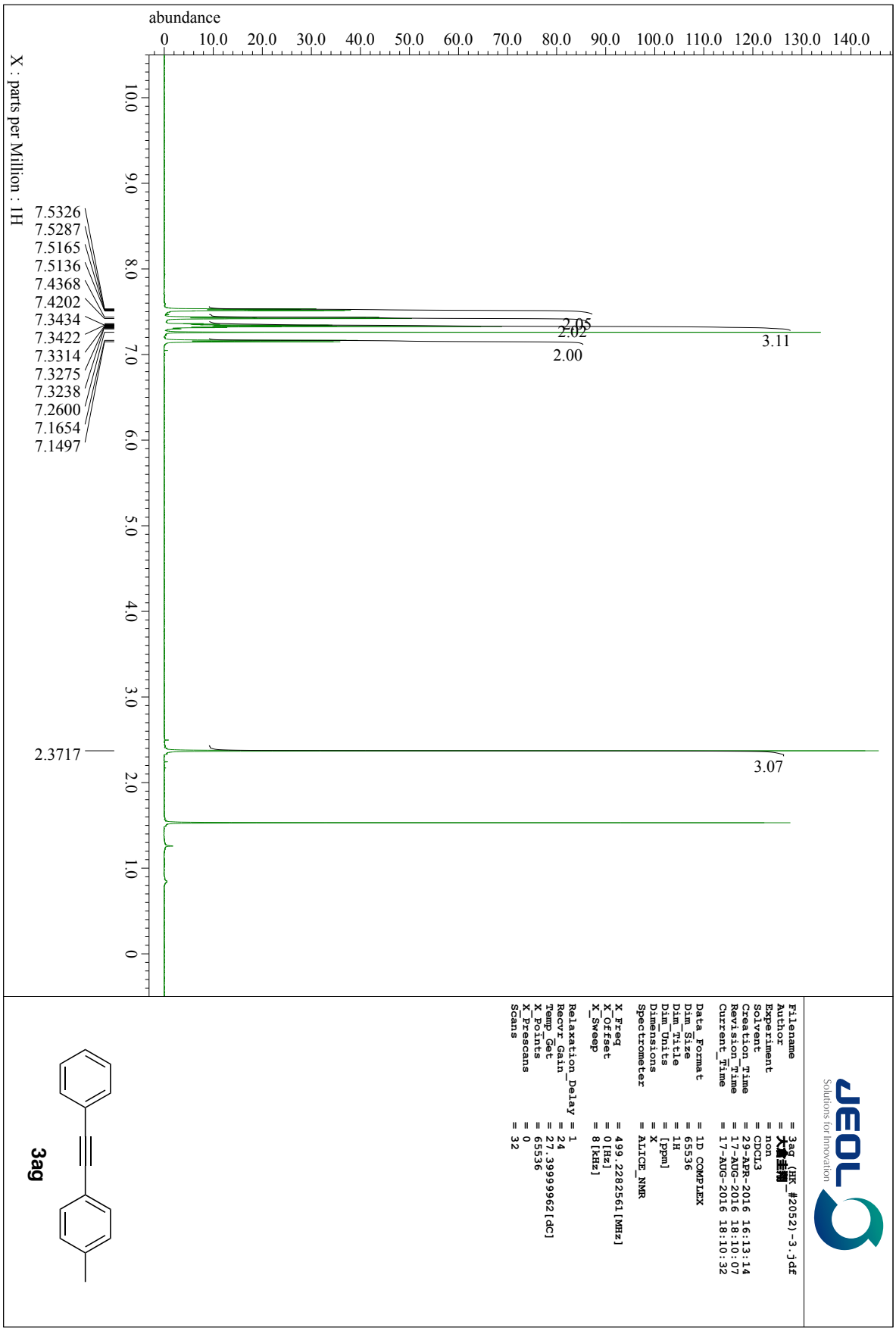
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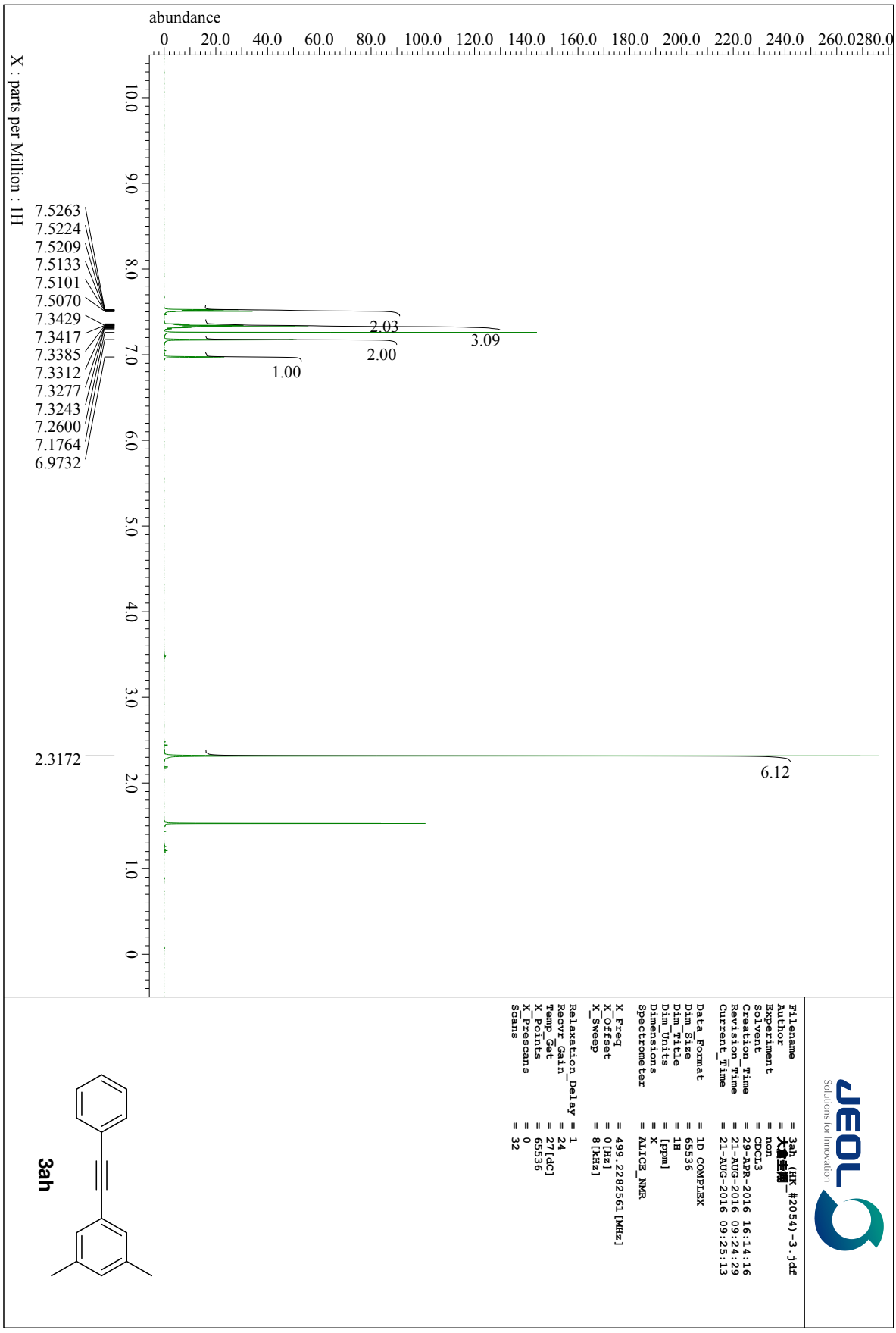
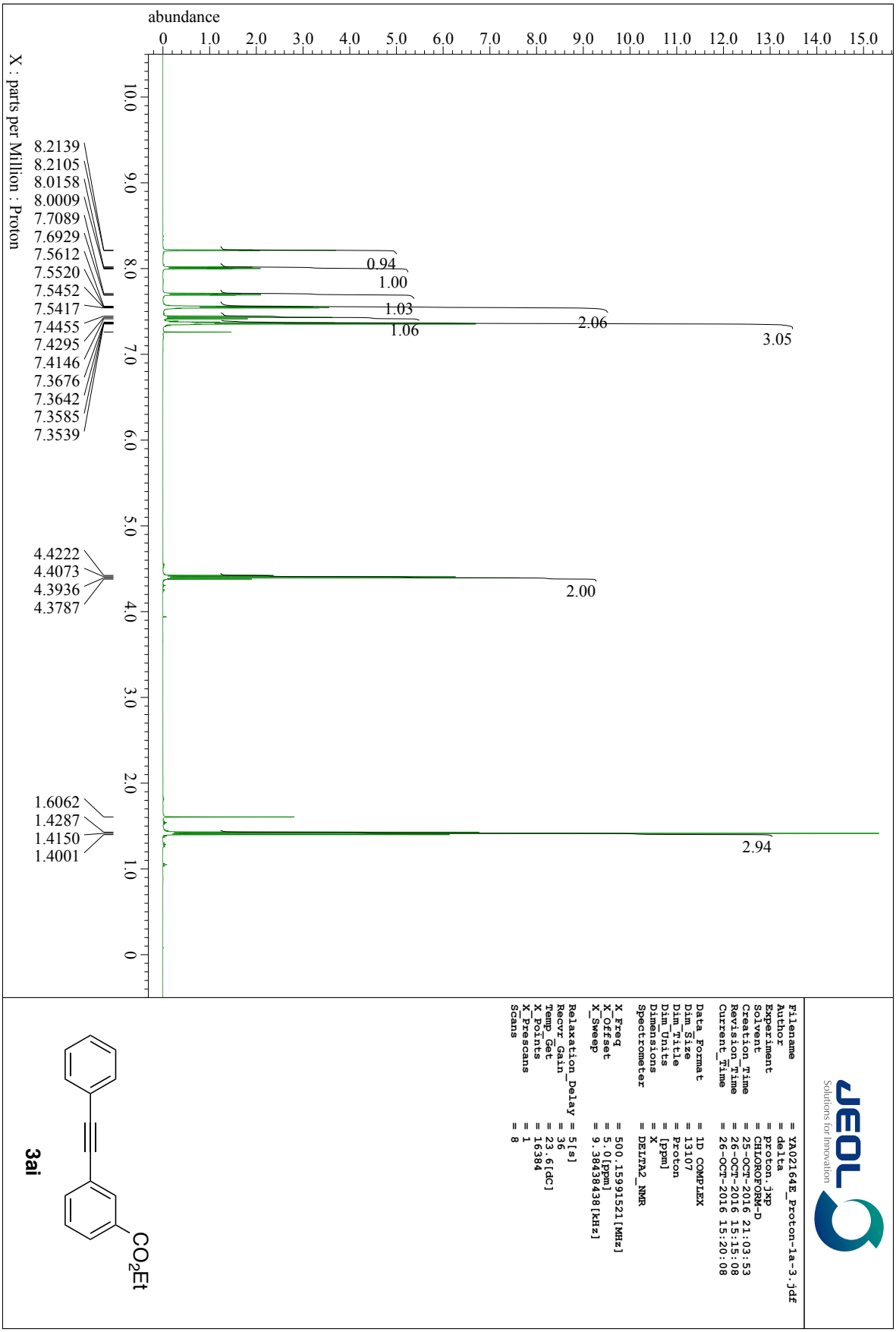
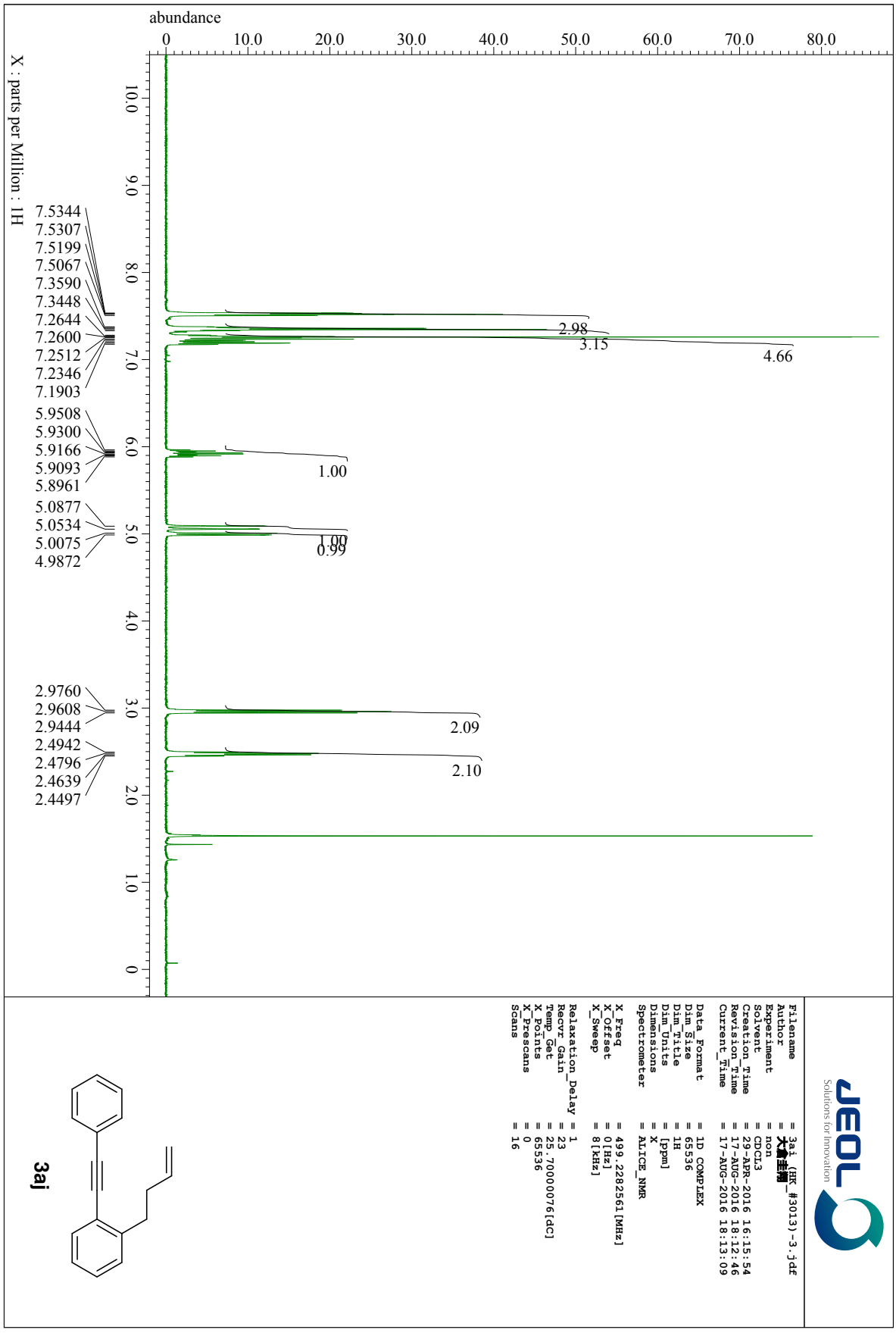
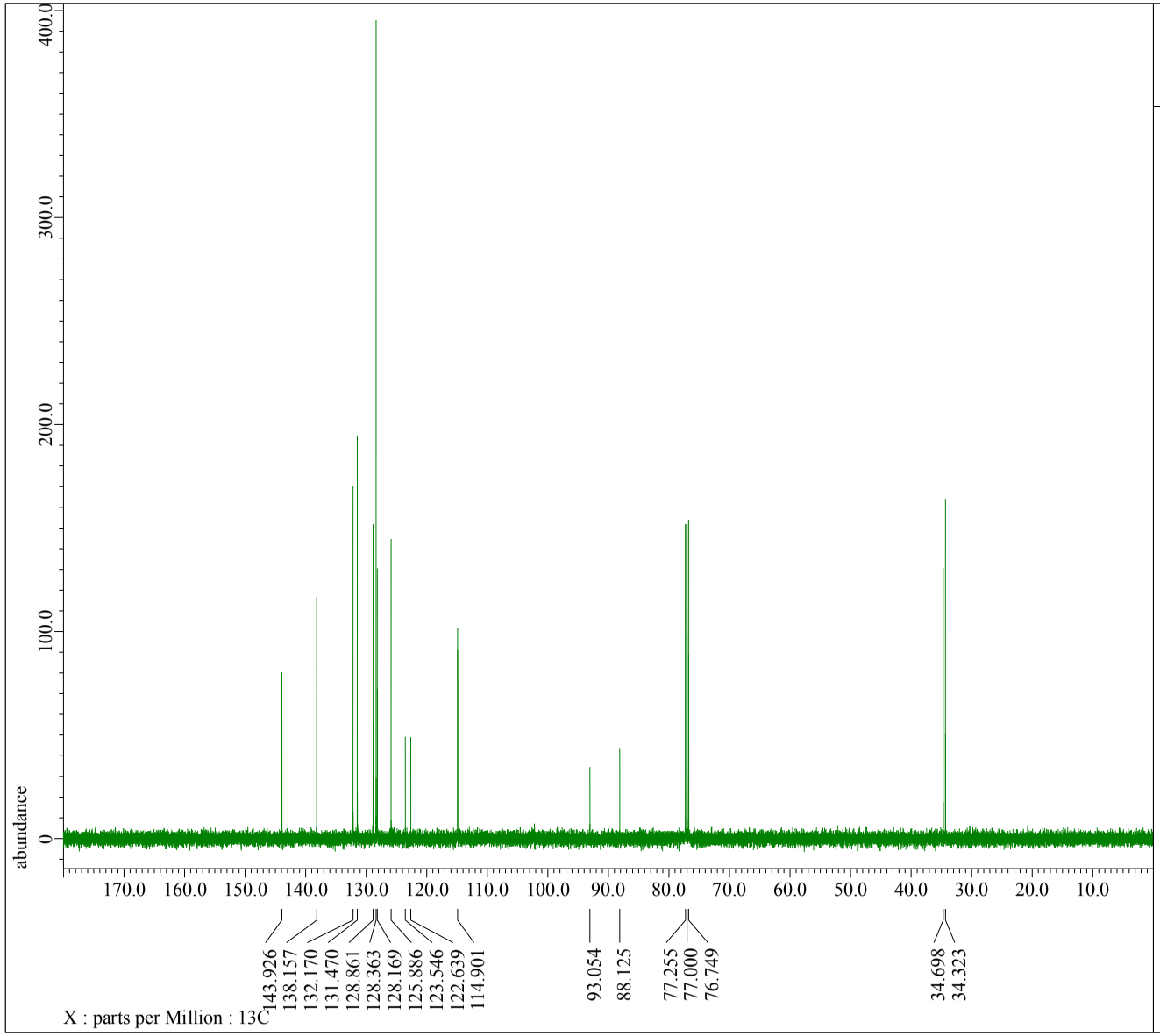
Scans = 32


abundance
X : parts per Million : 1H
7.5263
7.5224
7.5209
7.5133
7.5101
7.5070
7.3429
7.3417
7.3385
7.3312
7.3277
7.3243
7.2600
7.1764
6.9732
2.3172
2.03
3.09
2.00
1.00
6.12
Filename = 3ah_(HK_#20554)-3.jdf
Author = 大倉圭翔
Experiment = non
Solvent = CDCl3
Creation_Time = 29-APR-2016 16:14:16
Revision_Time = 21-AUG-2016 09:24:29
Current_Time = 21-AUG-2016 09:25:13
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561 [MHz]
X_Offset = 0 [Hz]
X_Sweep = 8 [kHz]
Relaxation_Delay = 1
Recvr_Gain = 24
Temp_Get = 27[dC]
X_Points = 65536
X_Prescans = 0
Scans = 32
3ah

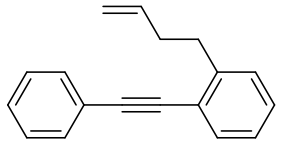

abundance
X : parts per Million : Proton
8.2139
8.2105
8.0158
8.0009
7.7089
7.6929
7.5612
7.5520
7.5452
7.5417
7.4455
7.4295
7.4146
7.3676
7.3642
7.3585
7.3539
4.4222
4.4073
4.3936
4.3787
1.6062
1.4287
1.4150
1.4001
0.94
1.00
1.03
1.06
2.06
3.05
2.00
2.94
Filename = YA02164E_Proton-1a-3.jdf
Author = delta
Experiment = proton.jxp
Solvent = CHLOROFORM-D
Creation_Time = 25-OCT-2016 21:03:53
Revision_Time = 26-OCT-2016 15:15:08
Current_Time = 26-OCT-2016 15:20:08
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = DELTA2_NMR
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Sweep = 9.38438438[kHz]
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 23.6[dC]
X_Points = 16384
X_Prescans = 1
Scans = 8
3ai
CO2Et


abundance
X : parts per Million : 1H
7.5344
7.5307
7.5199
7.5067
7.3590
7.3448
7.2644
7.2600
7.2512
7.2346
7.1903
5.9508
5.9300
5.9166
5.9093
5.8961
5.0877
5.0534
5.0075
4.9872
2.9760
2.9608
2.9444
2.4942
2.4796
2.4639
2.4497
2.98
3.15
4.66
1.00
1.00
0.99
2.09
2.10
3aj
Filename = 3aj_(HK_#3013)-3.jdf
Author = 大島圭翔
Experiment = non
Solvent = CDCL3
Creation_time = 29-APR-2016 16:15:54
Revision_Time = 17-AUG-2016 18:12:46
Current_Time = 17-AUG-2016 18:13:09
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561[MHz]
X_Offset = 0[Hz]
X_Sweep = 8[kHz]
Relaxation_Delay = 1
Recvr_Gain = 23
Temp_Get = 25.70000076[dC]
X_Points = 65536
X_Prescans = 0
Scans = 16

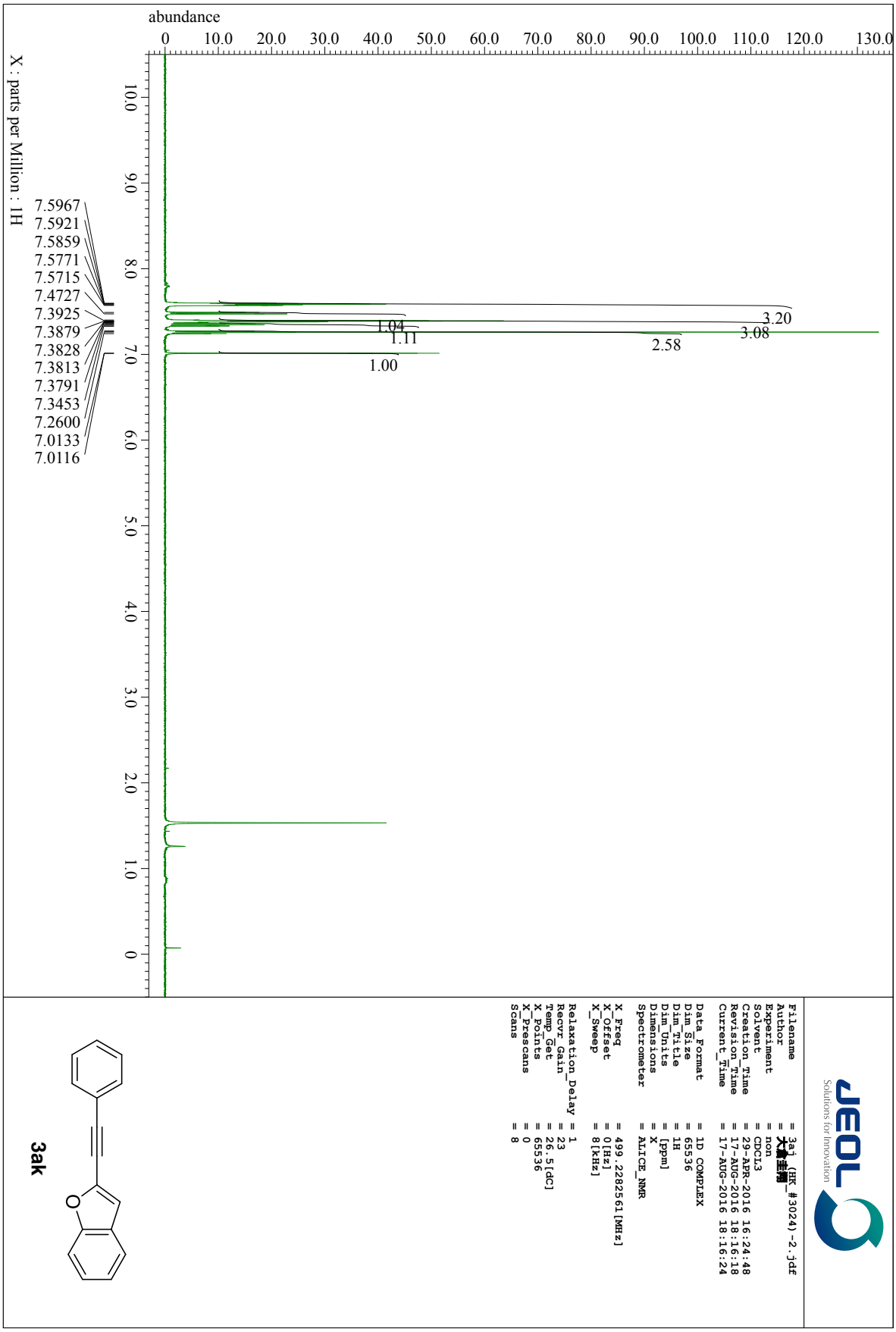


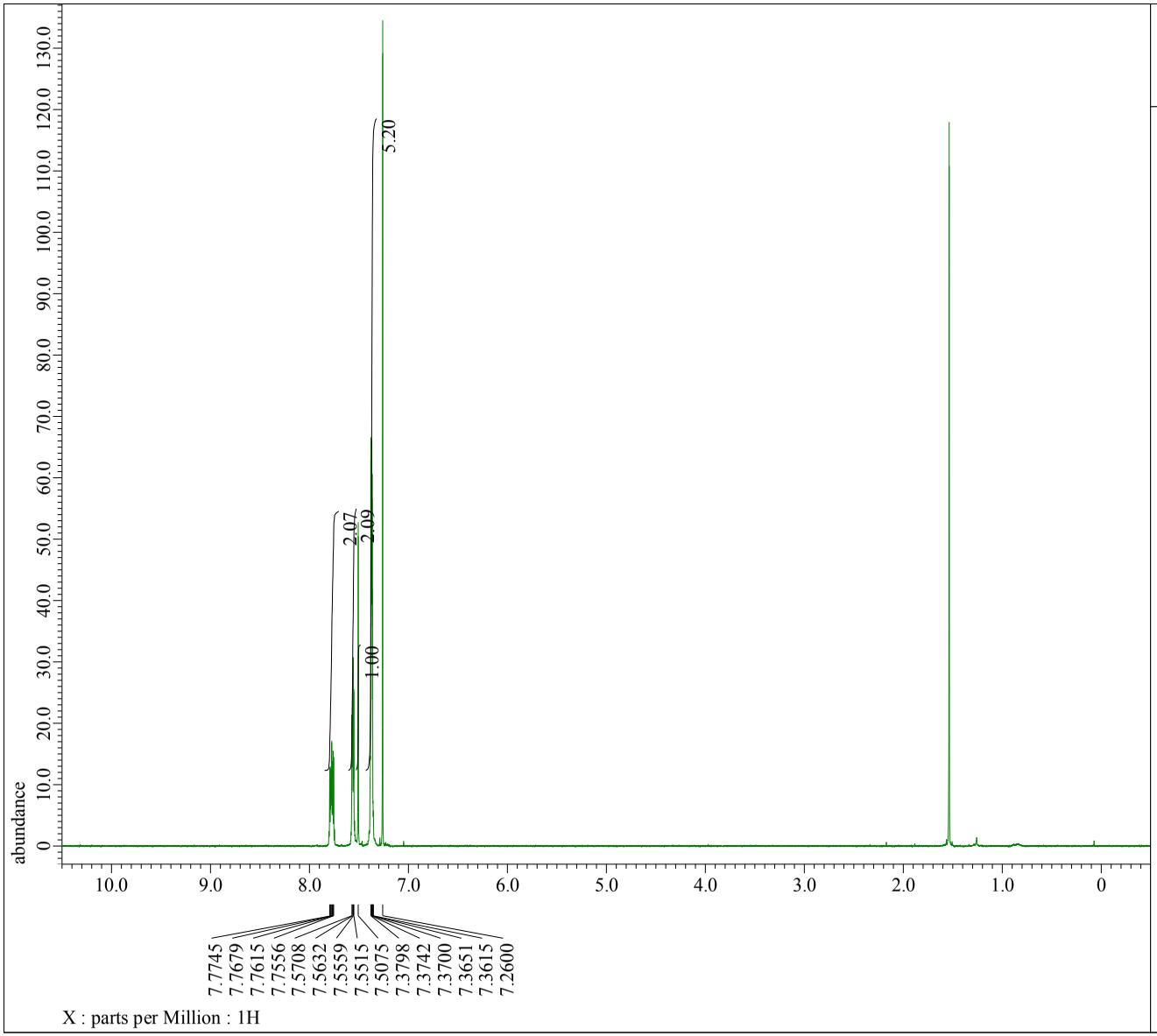
JEOL
Solutions for Innovation

```
Filename          = 3ai-13C (HK_#3013)-2.jdf
Author            = 大倉圭翔
Experiment        = bcm
Solvent           = CDCL3
Creation_Time     = 29-APR-2016 16:24:10
Revision_Time     = 29-APR-2016 19:37:11
Current_Time      = 29-APR-2016 19:40:58

Data_Format       = 1D COMPLEX
Dim_Size          = 65536
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = ALICE_NMR

X_Freq            = 125.54304253[MHz]
X_Offset          = 0[Hz]
X_Sweep           = 33.89830469[kHz]

Relaxation_Delay = 1.06700003
Recvr_Gain        = 26
Temp_Get          = 27.5[dC]
X_Points          = 65536
X_Prescans        = 2
Scans             = 128
```



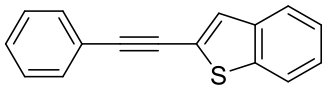
3aj


abundance
X : parts per Million : 1H
7.5967
7.5921
7.5859
7.5771
7.5715
7.4727
7.3925
7.3879
7.3828
7.3813
7.3791
7.3453
7.2600
7.0133
7.0116
3.20
3.08
2.58
1.04
1.11
1.00
Filename = 3aj_(HK_#3024)-2.jdf
Author = 大森圭陽
Experiment = non
Solvent = CDCl3
Creation_time = 29-APR-2016 16:24:48
Revision_time = 17-AUG-2016 18:16:18
Current_Time = 17-AUG-2016 18:16:24
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561[MHz]
X_Offset = 0[Hz]
X_Sweep = 8[kHz]
Relaxation_Delay = 1
Recvr_Gain = 23
Temp_Get = 26.5[dC]
X_Points = 65536
X_Prescans = 0
Scans = 8
JEOL
Solutions for Innovation
3ak

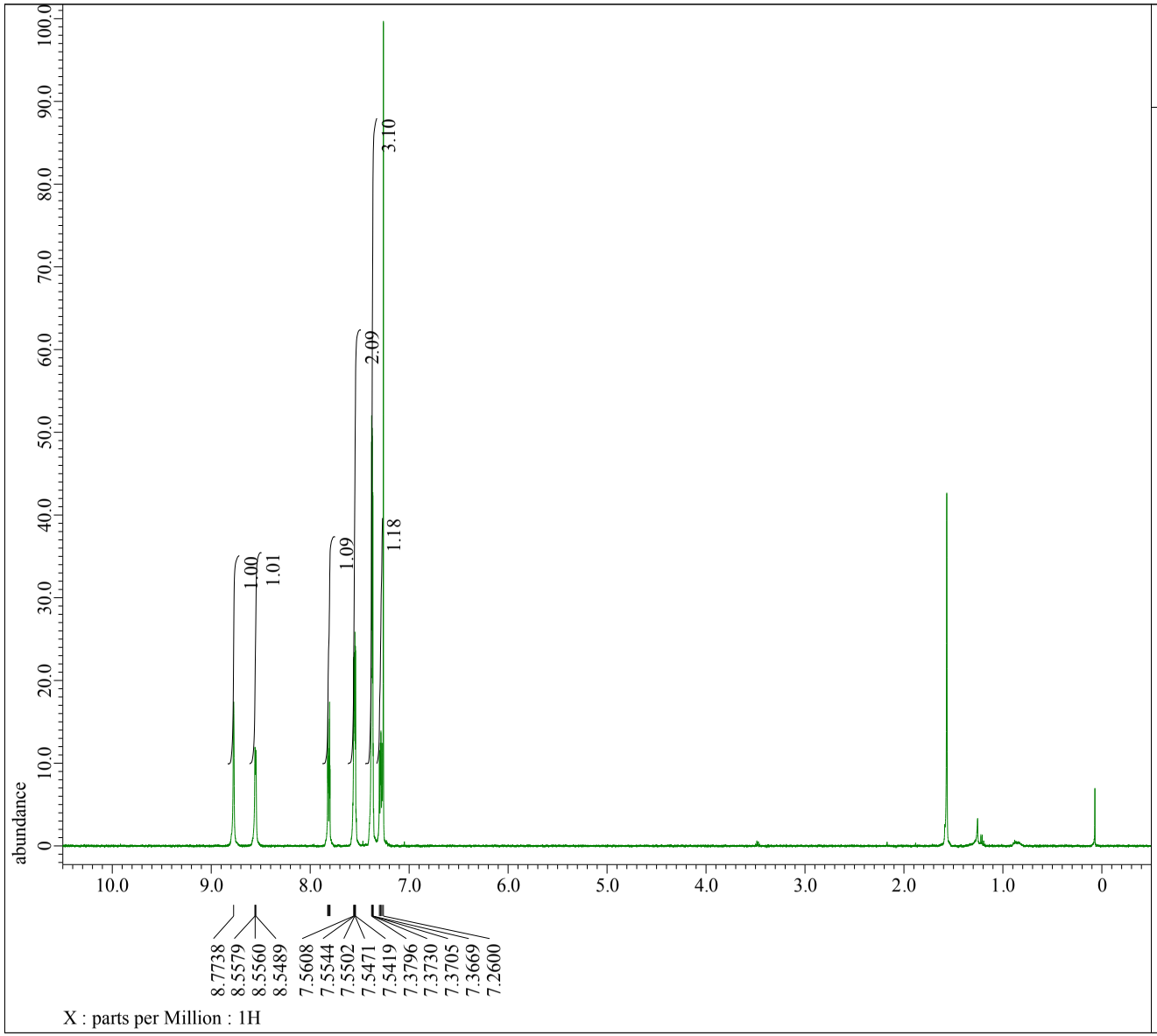


JEOL
Solutions for Innovation

```
Filename          = 3ak_(HK_#3008)-2.jdf
Author            = 大倉圭翔
Experiment        = non
Solvent           = CDCL3
Creation_Time     = 29-APR-2016 16:26:54
Revision_Time     = 17-AUG-2016 18:17:30
Current_Time      = 17-AUG-2016 18:17:53

Data_Format       = 1D COMPLEX
Dim_Size          = 65536
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = ALICE_NMR

X_Freq            = 499.2282561[MHz]
X_Offset          = 0[Hz]
X_Sweep           = 8[kHz]

Relaxation_Delay  = 1
Recvr_Gain        = 23
Temp_Get          = 25.29999924[dC]
X_Points          = 65536
X_Prescans        = 0
Scans             = 16
```

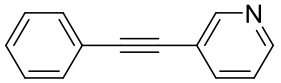


3al

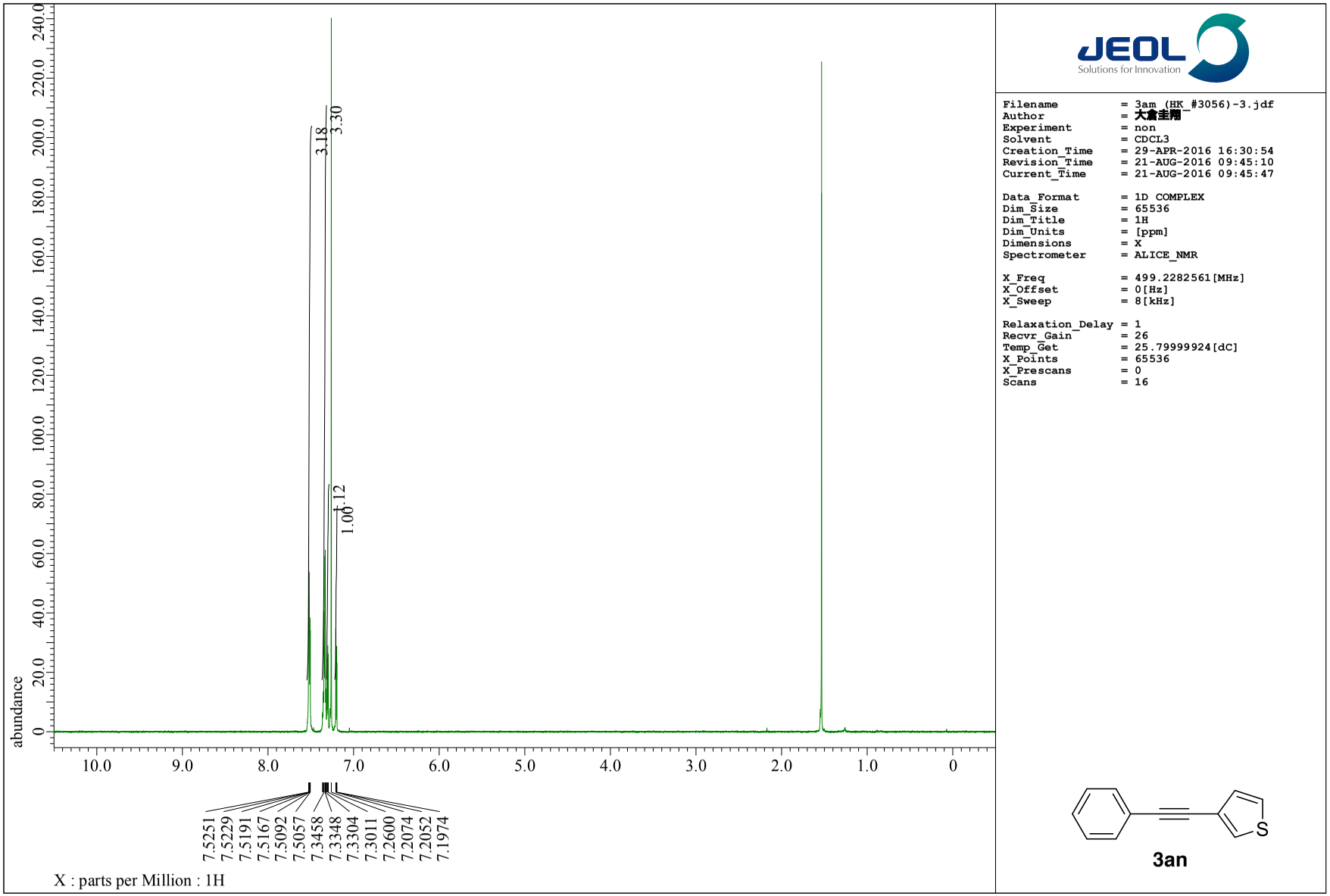


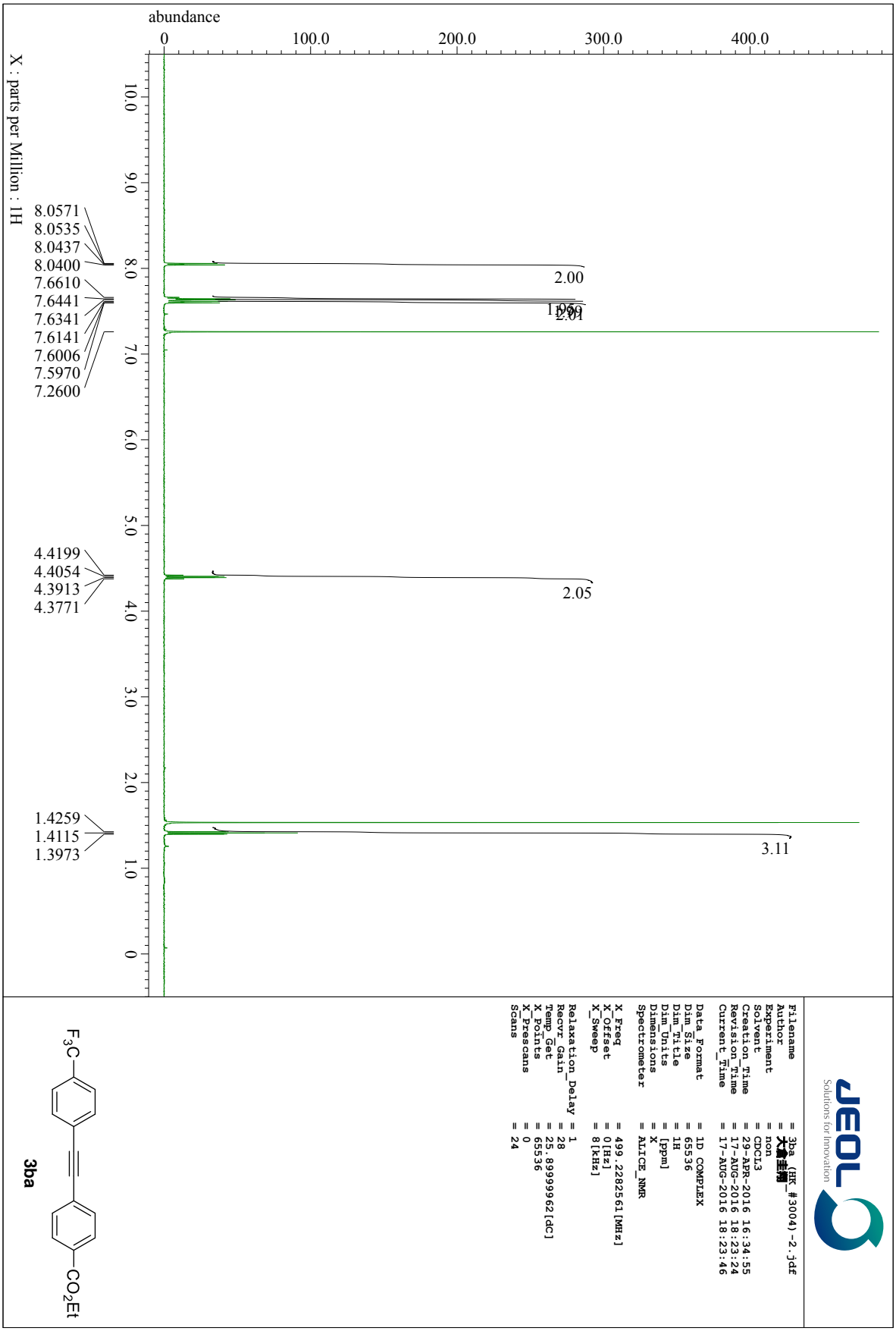
JEOL
Solutions for Innovation

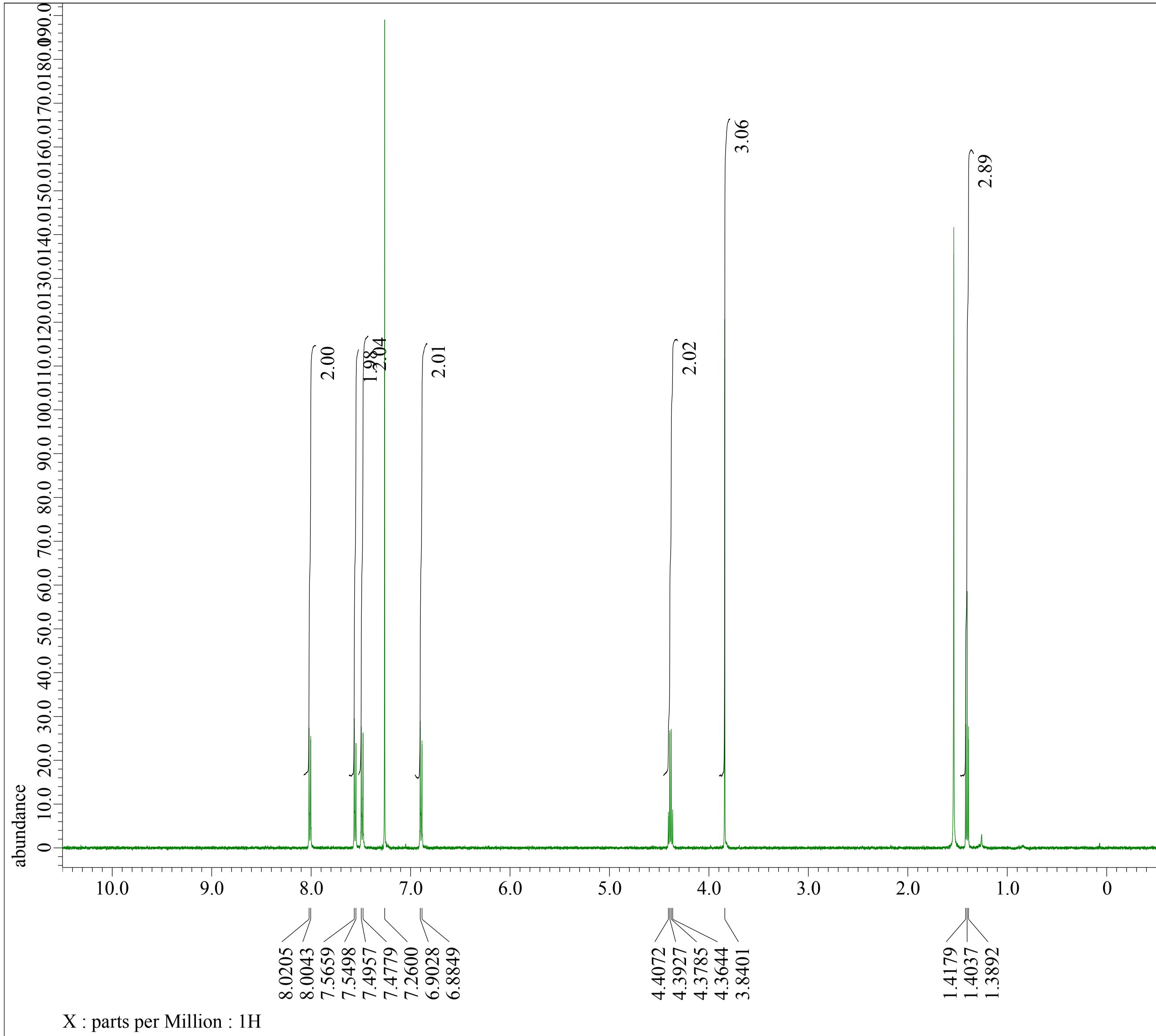
```
Filename          = 3al (HK #3009)-3.jdf
Author            = 大倉圭翔
Experiment        = non
Solvent           = CDCL3
Creation_Time     = 29-APR-2016 16:28:21
Revision_Time     = 17-AUG-2016 18:18:49
Current_Time      = 17-AUG-2016 18:19:09

Data_Format       = 1D COMPLEX
Dim_Size          = 65536
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = ALICE_NMR

X_Freq            = 499.2282561[MHz]
X_Offset          = 0[Hz]
X_Sweep           = 8[kHz]

Relaxation_Delay  = 1
Recvr_Gain        = 23
Temp_Get          = 25.5[dC]
X_Points          = 65536
X_Prescans        = 0
Scans             = 16
```



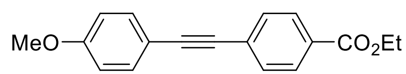
3am


abundance
240.0
220.0
200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0
3.18
3.30
1.12
1.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.5251
7.5229
7.5191
7.5167
7.5092
7.5057
7.3458
7.3348
7.3304
7.3011
7.2600
7.2074
7.2052
7.1974
X : parts per Million : 1H
JEOL
Solutions for Innovation
Filename = 3am (HK #3056)-3.jdf
Author = 大倉圭翔
Experiment = non
Solvent = CDCL3
Creation_Time = 29-APR-2016 16:30:54
Revision_Time = 21-AUG-2016 09:45:10
Current_Time = 21-AUG-2016 09:45:47
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561[MHz]
X_Offset = 0[Hz]
X_Sweep = 8[kHz]
Relaxation_Delay = 1
Recvr_Gain = 26
Temp_Get = 25.79999924[dC]
X_Points = 65536
X_Prescans = 0
Scans = 16
S
3an

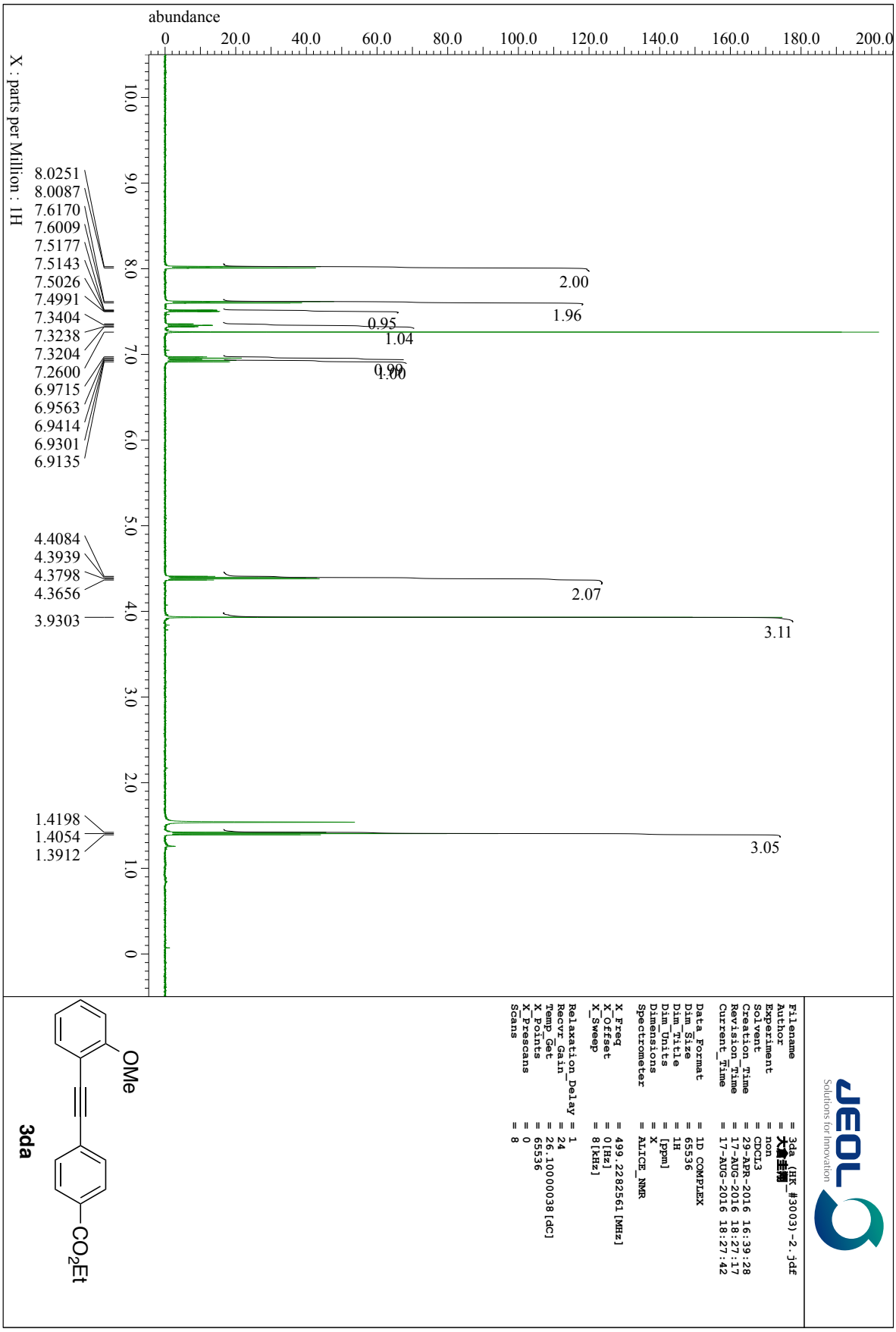

abundance
0
100.0
200.0
300.0
400.0
X : parts per Million : 1H
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.0571
8.0535
8.0437
8.0400
7.6610
7.6441
7.6341
7.6141
7.6006
7.5970
7.2600
4.4199
4.4054
4.3913
4.3771
1.4259
1.4115
1.3973
2.00
2.01
2.05
3.11
Filename = 3ba_(HK_#3004)-2.jdf
Author = 大倉圭翔
Experiment = non
Solvent = CDCl3
Creation_Time = 29-APR-2016 16:34:55
Revision_Time = 17-AUG-2016 18:23:24
Current_Time = 17-AUG-2016 18:23:46
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561[MHz]
X_Offset = 0[Hz]
X_Sweep = 8[kHz]
Relaxation_Delay = 1
Recvr_Gain = 28
Temp_Get = 25.8999962[dC]
X_Points = 65536
X_Prescans = 0
Scans = 24
JEOL
Solutions for Innovation
F3C
CO2Et
3ba

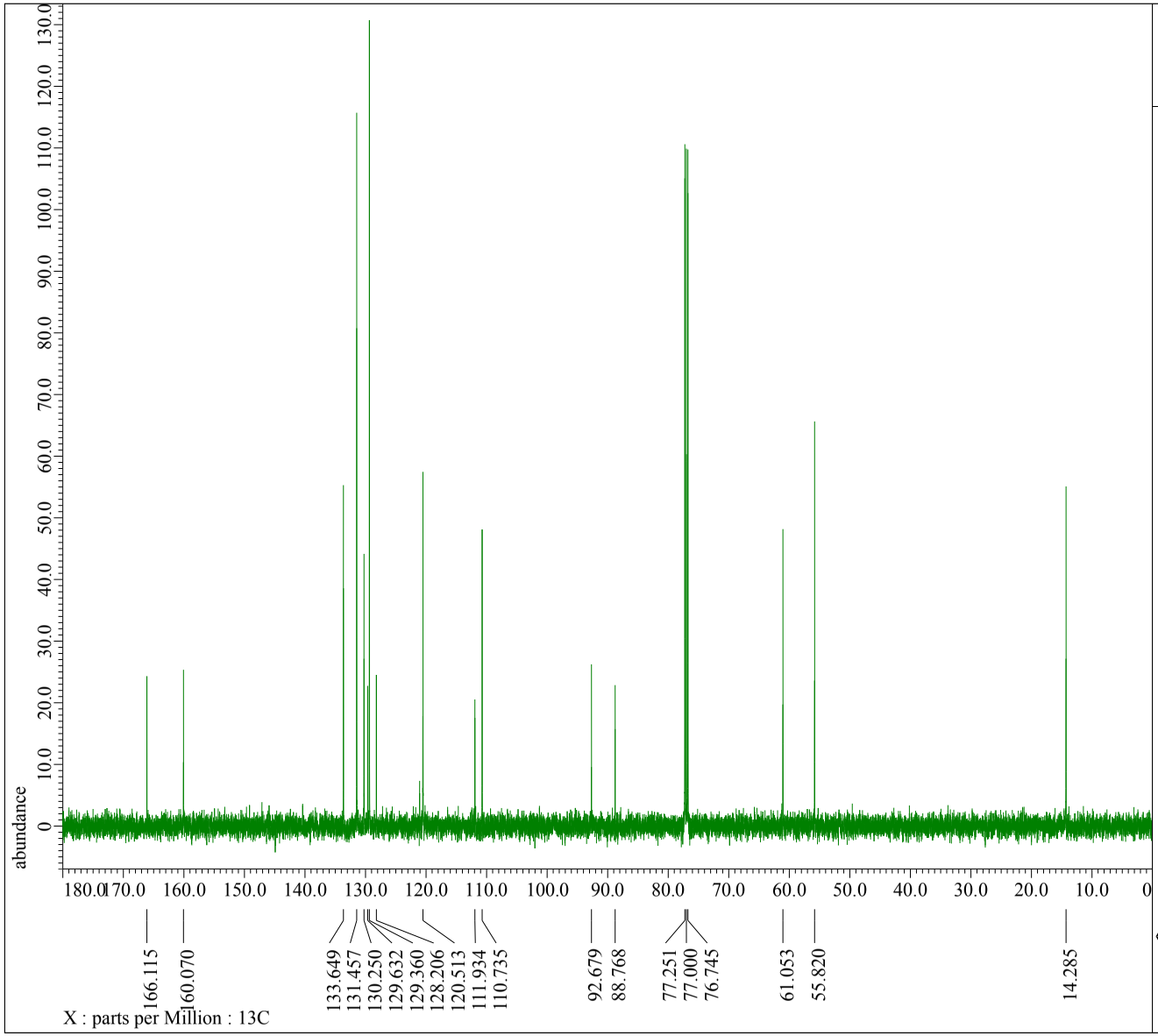



```
Filename          = 3ca (HK #3063)-3.jdf
Author            = 大倉圭翔
Experiment        = non
Solvent           = CDCL3
Creation_Time     = 29-APR-2016 16:38:27
Revision_Time     = 17-AUG-2016 18:25:58
Current_Time      = 17-AUG-2016 18:26:19

Data_Format       = 1D COMPLEX
Dim_Size          = 65536
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = ALICE_NMR

X_Freq            = 499.2282561[MHz]
X_Offset          = 0[Hz]
X_Sweep           = 8[kHz]

Relaxation_Delay  = 1
Recvr_Gain        = 25
Temp_Get          = 26.29999924[dC]
X_Points          = 65536
X_Prescans        = 0
Scans             = 8
```



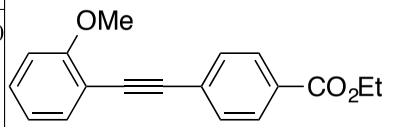
3ca


abundance
X : parts per Million : 1H
8.0251
8.0087
7.6170
7.6009
7.5177
7.5143
7.5026
7.4991
7.3404
7.3238
7.3204
7.2600
6.9715
6.9563
6.9414
6.9301
6.9135
4.4084
4.3939
4.3798
4.3656
3.9303
1.4198
1.4054
1.3912
2.00
1.96
0.95
1.04
0.99
2.07
3.11
3.05
OMe
CO2Et
3da
Filename = 3da_(HK_#3003)-2.jdf
Author = 大●圭●
Experiment = non
Solvent = CDCl3
Creation_time = 29-APR-2016 16:39:28
Revision_Time = 17-AUG-2016 18:27:17
Current_Time = 17-AUG-2016 18:27:42
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561 [MHz]
X_Offset = 0 [Hz]
X_Sweep = 8 [kHz]
Relaxation_Delay = 1
Recvr_Gain = 24
Temp_Get = 26.10000038 [dC]
X_Points = 65536
X_Prescans = 0
Scans = 8

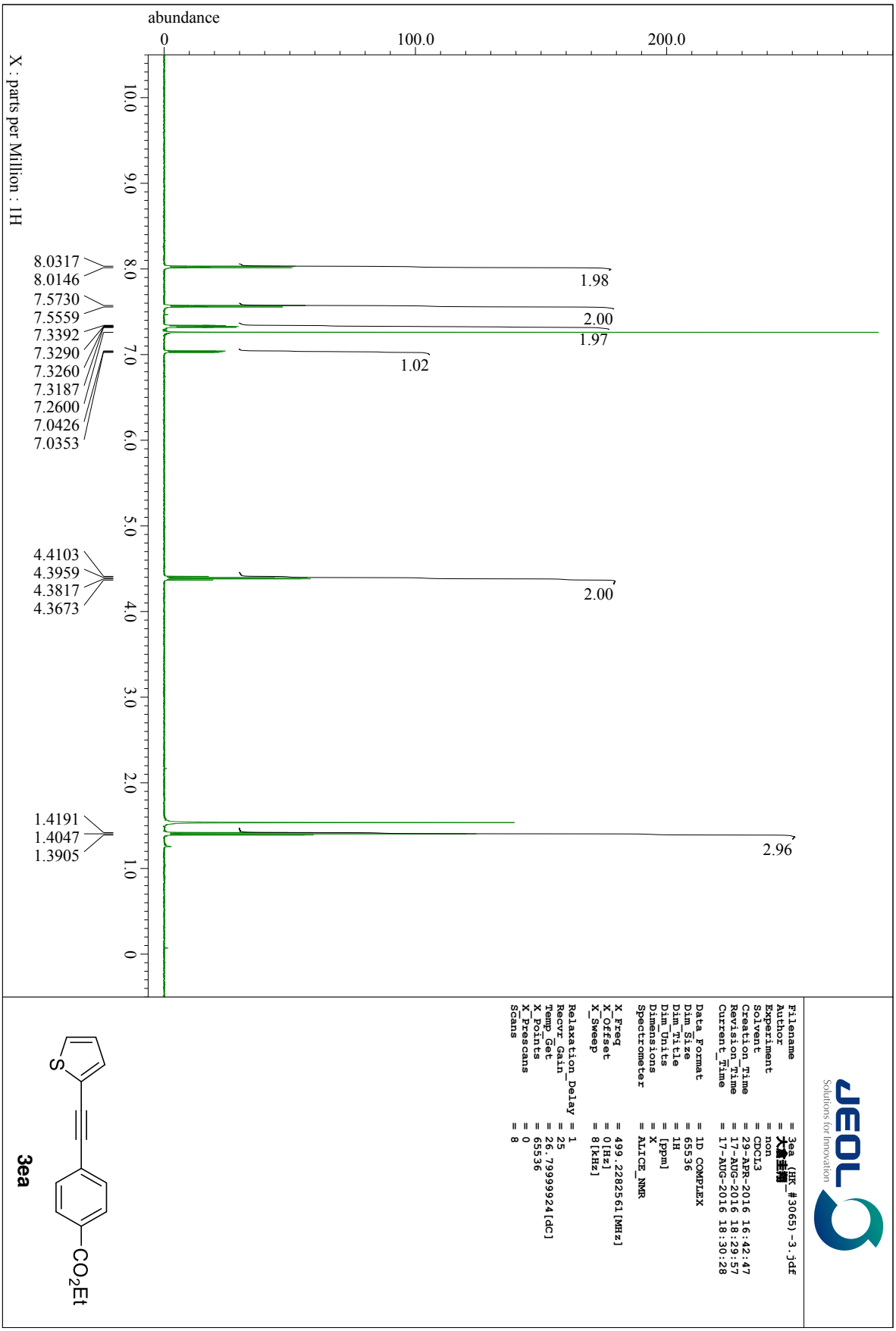


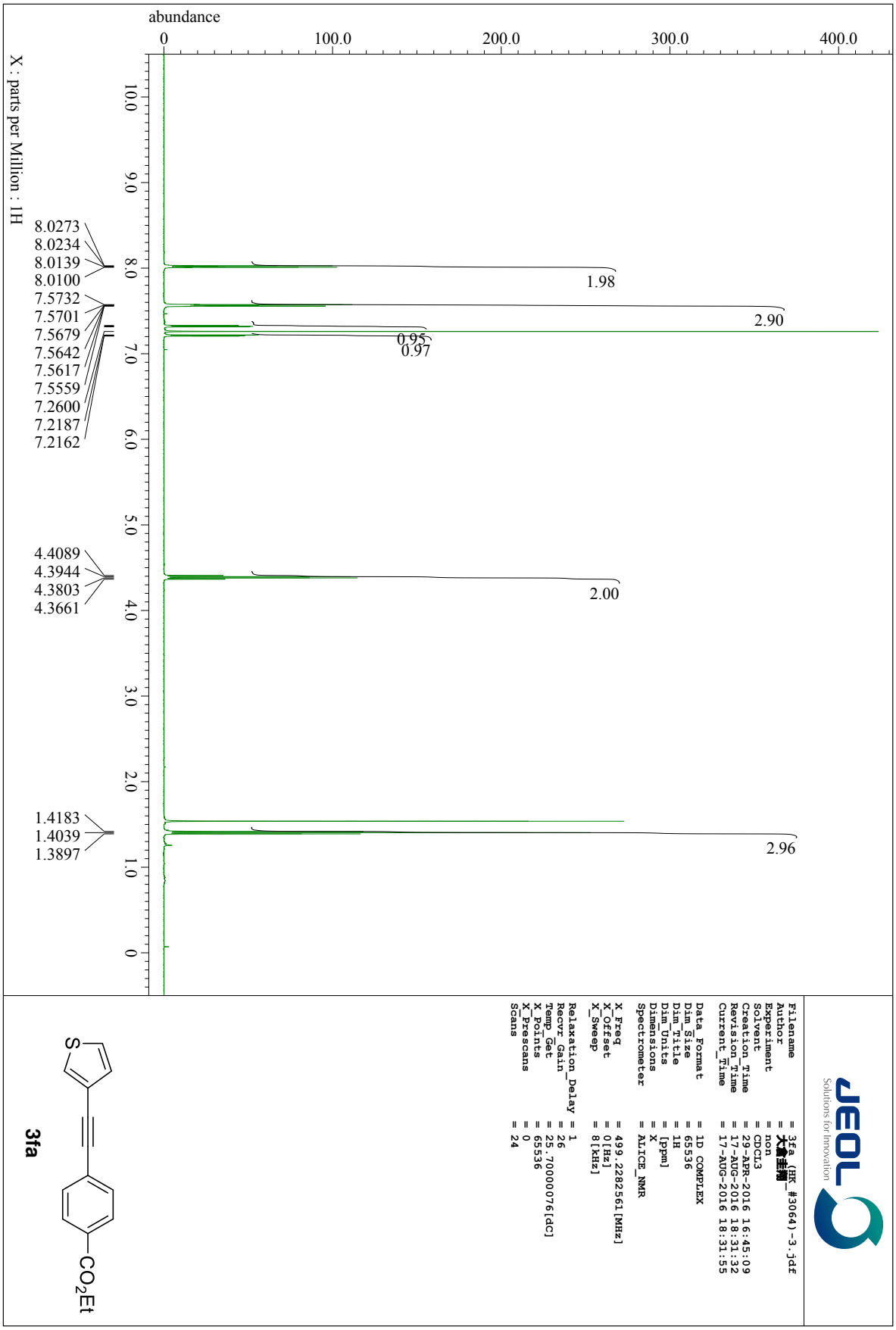
JEOL Solutions for Innovation

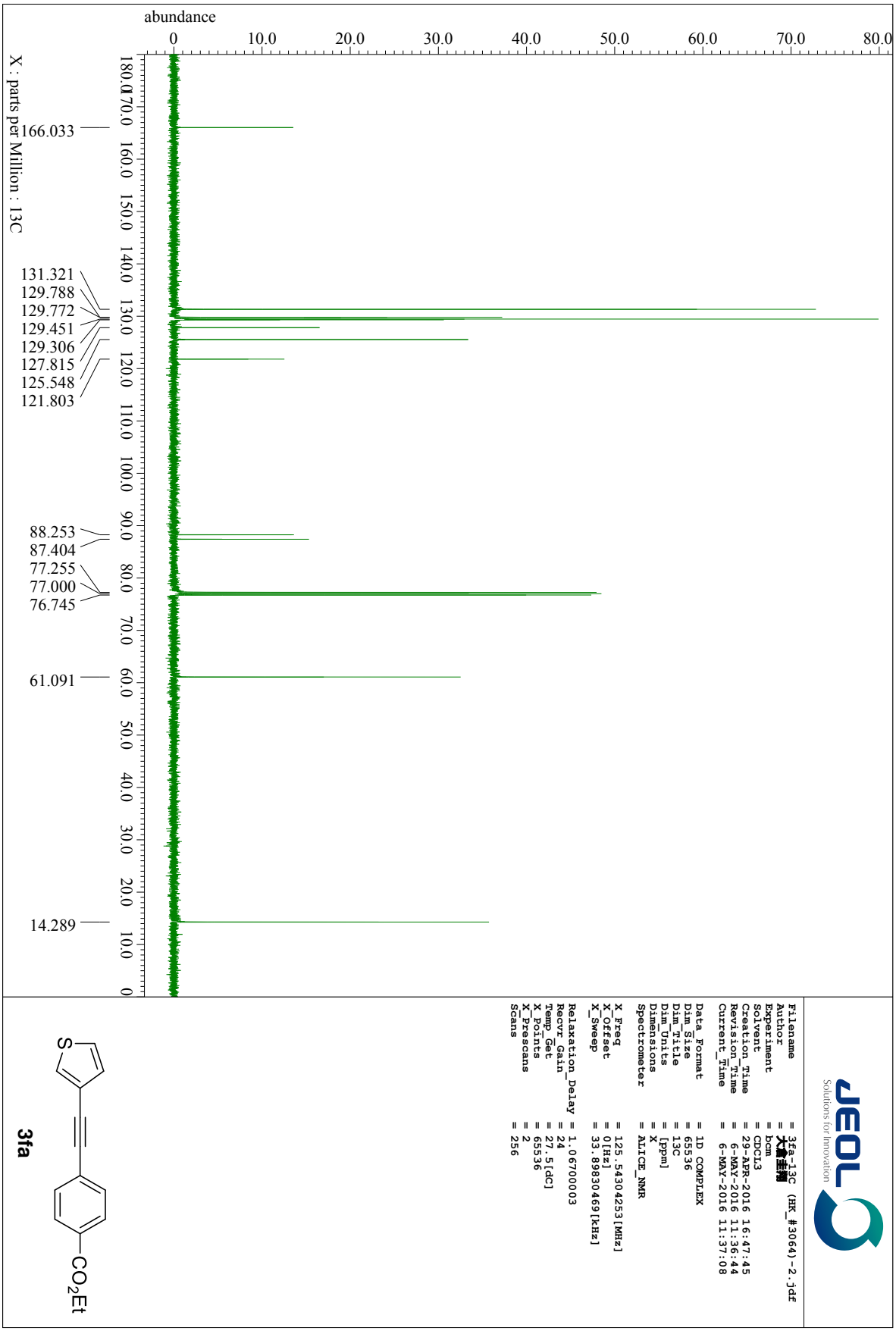
```
Filename          = 3da-13C (HK_#3003)-2.jdf
Author            = 大倉圭翔
Experiment        = bcm
Solvent           = CDCL3
Creation_Time     = 29-APR-2016 16:41:57
Revision_Time     = 29-APR-2016 21:39:03
Current_Time      = 29-APR-2016 21:39:32

Data_Format       = 1D COMPLEX
Dim_Size          = 65536
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = ALICE_NMR

X_Freq            = 125.54304253[MHz]
X_Offset          = 0[Hz]
X_Sweep           = 33.89830469[kHz]

Relaxation_Delay = 1.06700003
Recvr_Gain        = 26
Temp_Get          = 27.60000038[dC]
X_Points          = 65536
X_Prescans        = 2
Scans             = 64
```

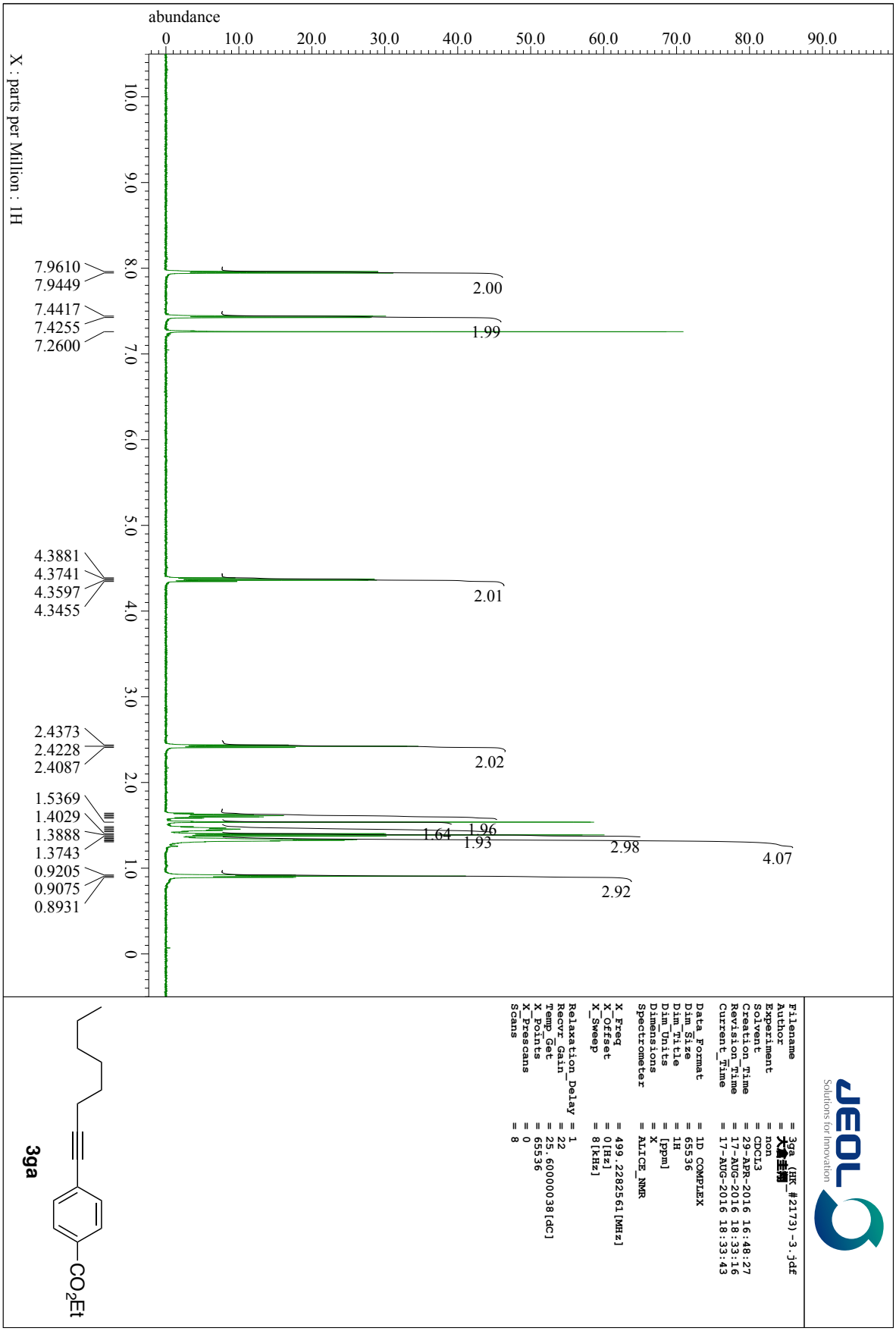


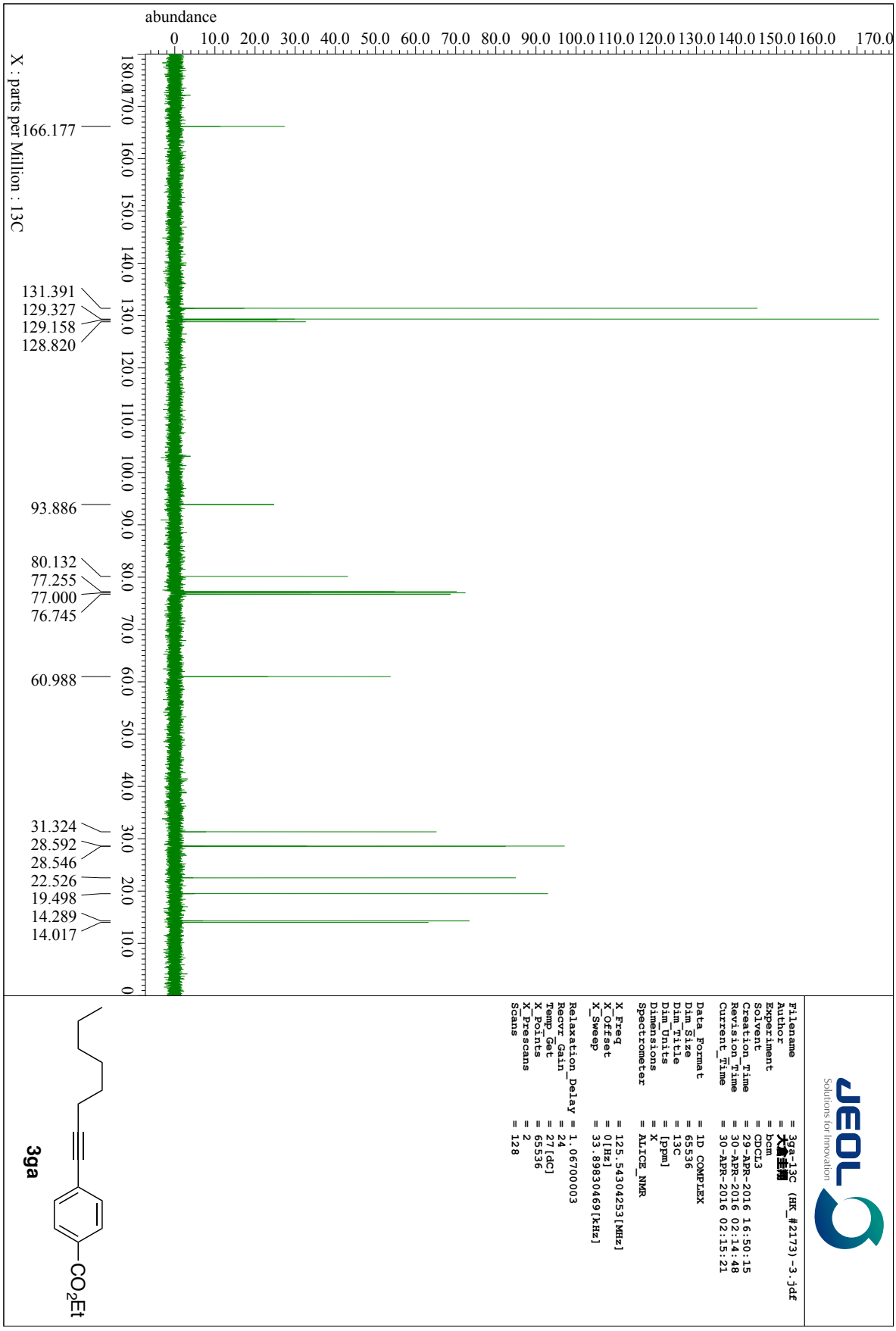
3da

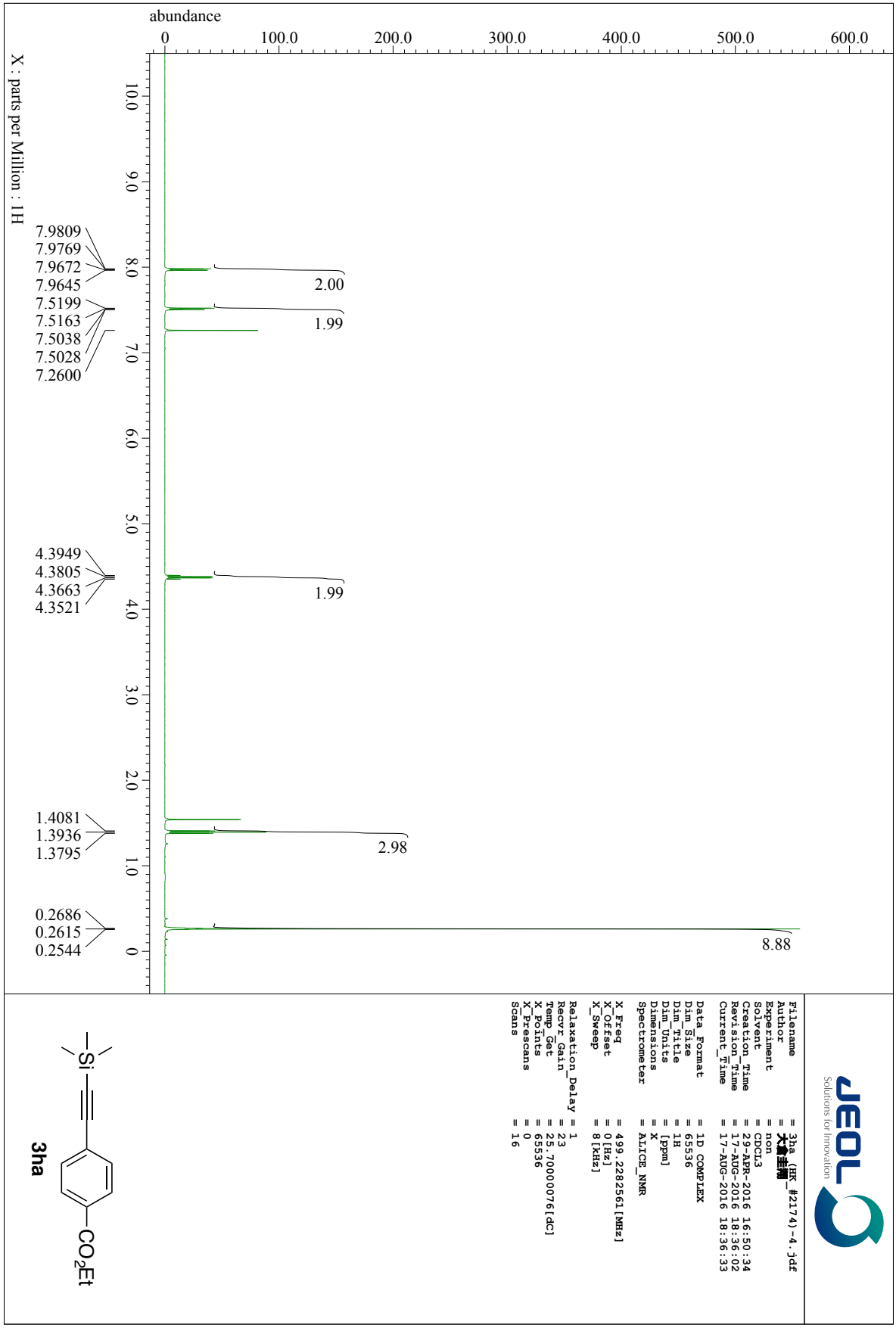

abundance
0
100.0
200.0
X : parts per Million : 1H
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.0317
8.0146
7.5730
7.5559
7.3392
7.3290
7.3260
7.3187
7.2600
7.0426
7.0353
4.4103
4.3959
4.3817
4.3673
1.4191
1.4047
1.3905
1.98
2.00
1.97
1.02
2.00
2.96
JEOL
Solutions for Innovation
Filename = 3ea_(HK_#3065)-3.jdf
Author = 大森佳翔_
Experiment = non
Solvent = CDCl3
Creation_Time = 29-APR-2016 16:42:47
Revision_Time = 17-AUG-2016 18:29:57
Current_Time = 17-AUG-2016 18:30:28
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561[MHz]
X_Offset = 0[Hz]
X_Sweep = 8[kHz]
Relaxation_Delay = 1
Recvr_Gain = 25
Temp_Get = 26.79999924[dC]
X_Points = 65536
X_Prescans = 0
Scans = 8
3ea
S
CO2Et

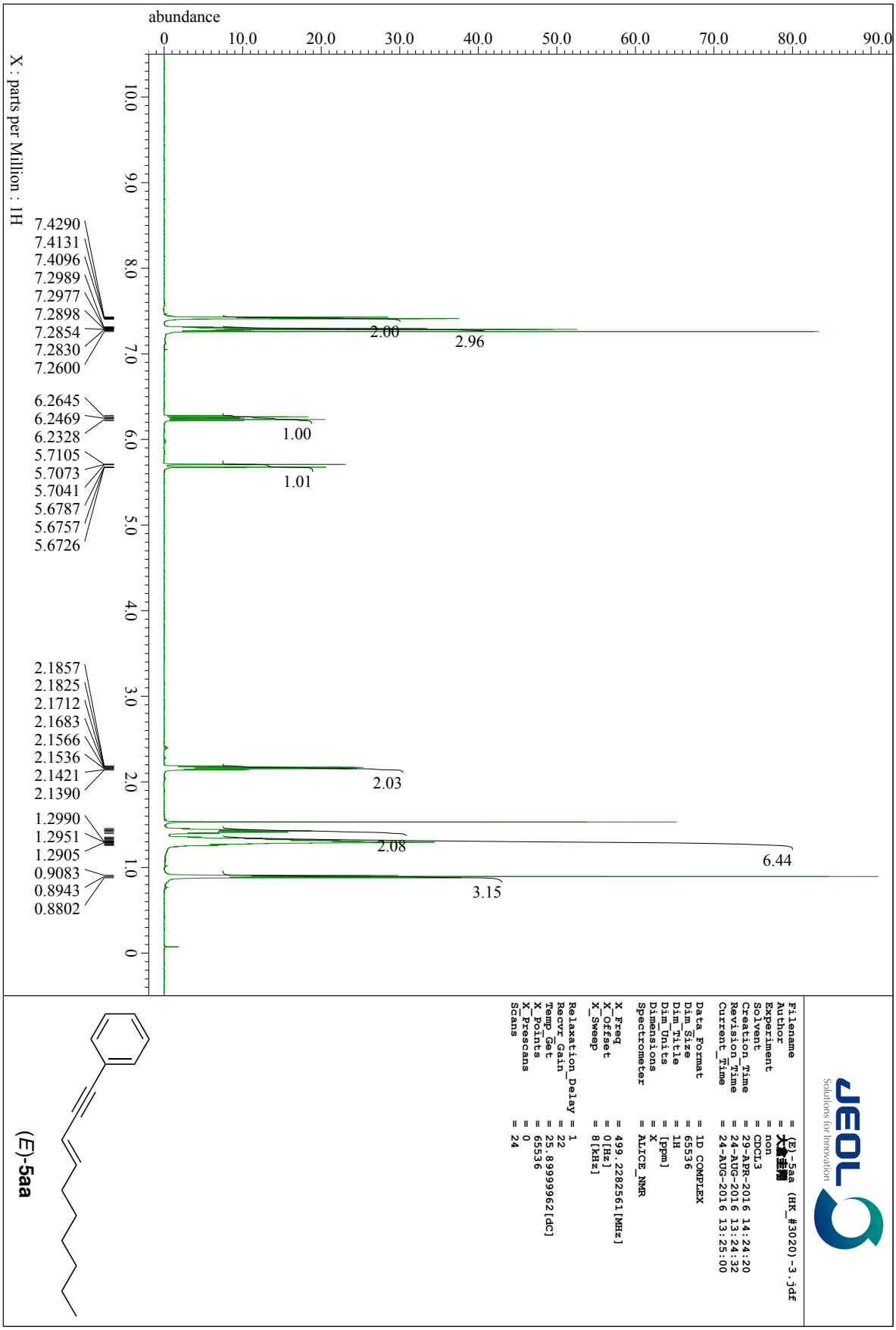

abundance
0
100.0
200.0
300.0
400.0
X : parts per Million : 1H
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.0273
8.0234
8.0139
8.0100
7.5732
7.5701
7.5679
7.5642
7.5617
7.5559
7.2600
7.2187
7.2162
4.4089
4.3944
4.3803
4.3661
1.4183
1.4039
1.3897
1.98
2.90
0.95
0.97
2.00
2.96
Filename = 3fa_(HK_#3064)-3.jdf
Author = 大倉圭翔
Experiment = non
Solvent = CDCl3
Creation_Time = 29-APR-2016 16:45:09
Revision_Time = 17-AUG-2016 18:31:32
Current_Time = 17-AUG-2016 18:31:55
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561[MHz]
X_Offset = 0[Hz]
X_Sweep = 8[kHz]
Relaxation_Delay = 1
Recvr_Gain = 26
Temp_Get = 25.70000076[dc]
X_Points = 65536
X_Prescans = 0
Scans = 24
JEOL
Solutions for Innovation
3fa
S
CO2Et

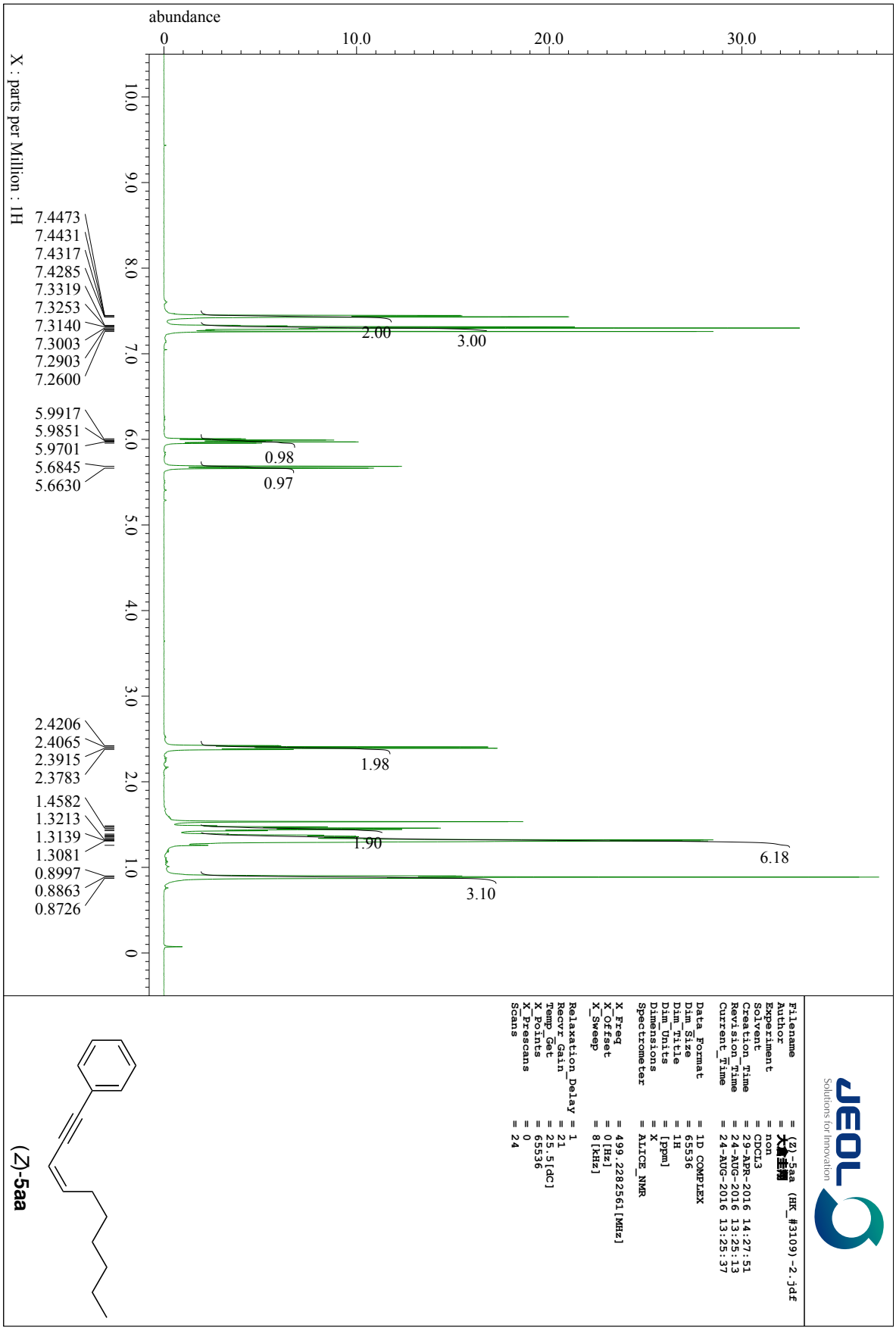

abundance
X : parts per Million : 13C
166.033
131.321
129.788
129.772
129.451
129.306
127.815
125.548
121.803
88.253
87.404
77.255
77.000
76.745
61.091
14.289
Filename = 3fa-13C (HK_#3064)-2.jdf
Author = 大森佳翔
Experiment = bcm
Solvent = CDCl3
Creation_Time = 29-APR-2016 16:47:45
Revision_Time = 6-MAY-2016 11:36:44
Current_Time = 6-MAY-2016 11:37:08
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 125.54304253[MHz]
X_Offset = 0[Hz]
X_Sweep = 33.89830469[kHz]
Relaxation_Delay = 1.06700003
Recvr_Gain = 24
Temp_Get = 27.5[dC]
X_Points = 65536
X_Prescans = 2
Scans = 256
3fa
S
CO2Et

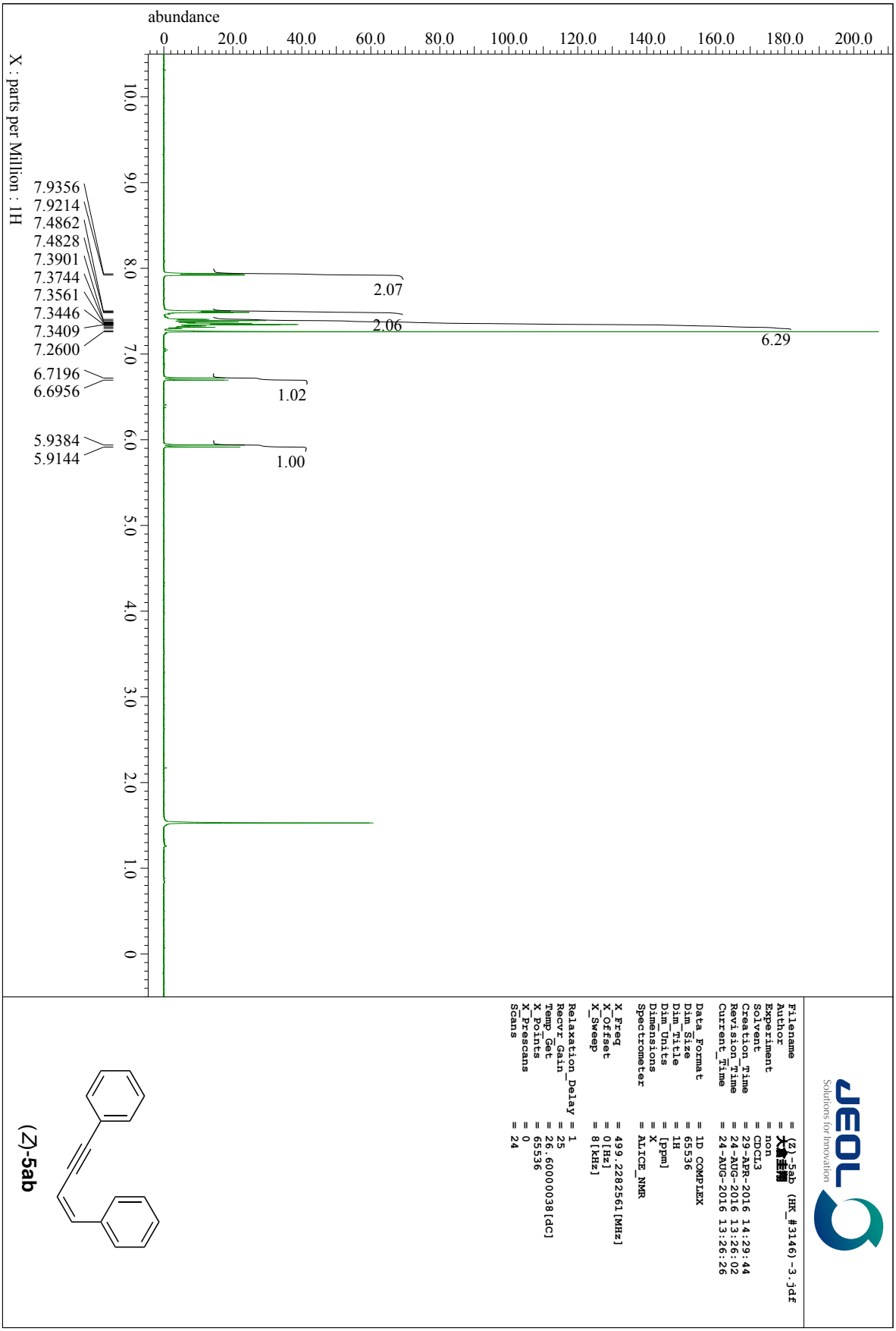

abundance
X : parts per Million : 1H
7.9610
7.9449
7.4417
7.4255
7.2600
4.3881
4.3741
4.3597
4.3455
2.4373
2.4228
2.4087
1.5369
1.4029
1.3888
1.3743
0.9205
0.9075
0.8931
2.00
1.99
2.01
2.02
1.96
1.64
1.93
2.98
4.07
2.92
Filename = 3ga_(HK_#2173)-3.jdf
Author = 大●佳●_
Experiment = non
Solvent = CDCl3
Creation_Time = 29-APR-2016 16:48:27
Revision_Time = 17-AUG-2016 18:33:16
Current_Time = 17-AUG-2016 18:33:43
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561[MHz]
X_Offset = 0[Hz]
X_Sweep = 8[kHz]
Relaxation_Delay = 1
Recvr_Gain = 22
Temp_Get = 25.60000038[dC]
X_Points = 65536
X_Prescans = 0
Scans = 8
JEOL
Solutions for Innovation
3ga
CO2Et

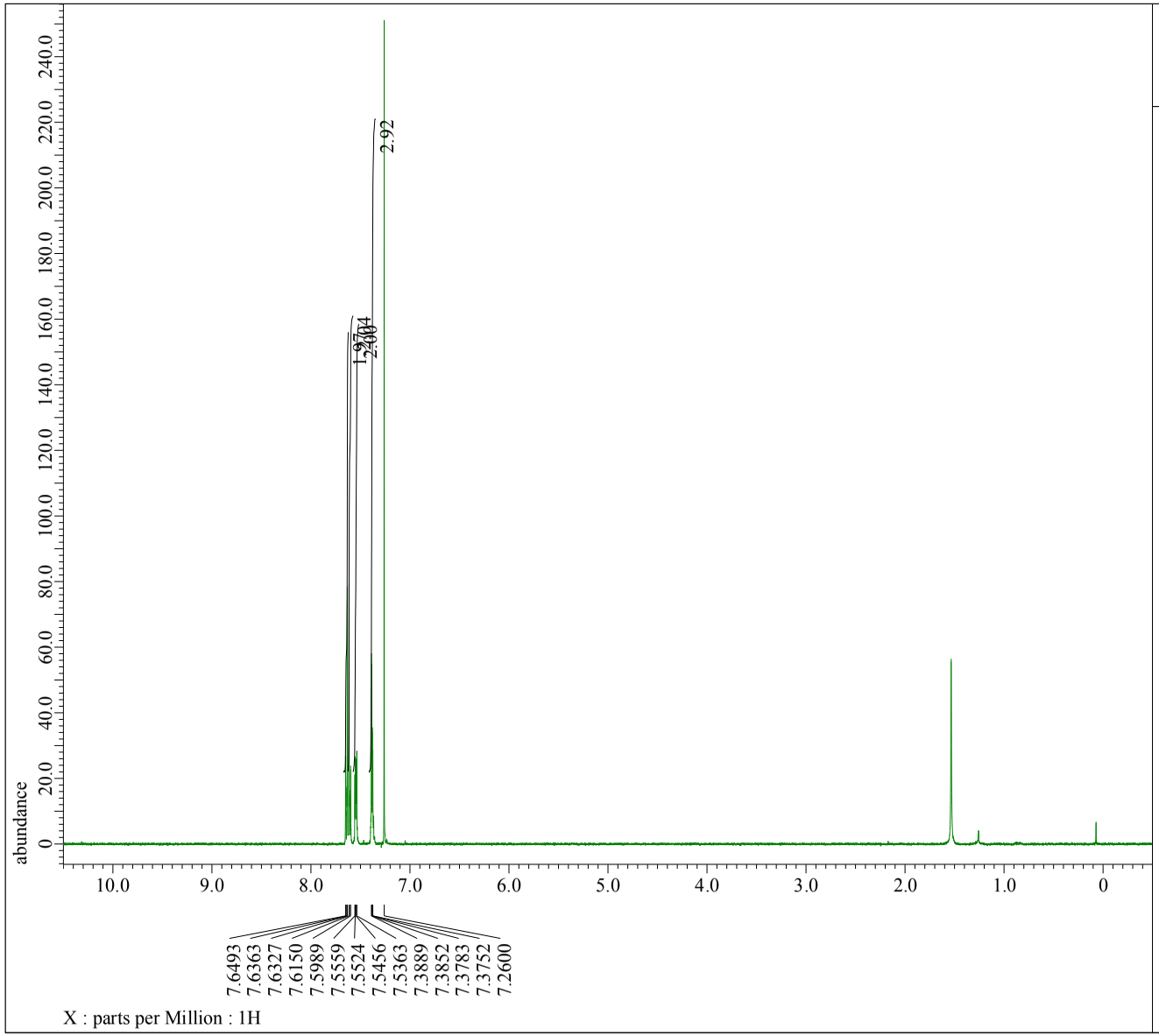

abundance
X : parts per Million : 13C
166.177
131.391
129.327
129.158
128.820
93.886
80.132
77.255
77.000
76.745
60.988
31.324
28.592
28.546
22.526
19.498
14.289
14.017
Filename = 3ga-13C (HK_#2173)-3.jdf
Author = 大森圭輔
Experiment = bcm
Solvent = CDCl3
Creation_Time = 29-APR-2016 16:50:15
Revision_Time = 30-APR-2016 02:14:48
Current_Time = 30-APR-2016 02:15:21
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 125.54304253[MHz]
X_Offset = 0[Hz]
X_Sweep = 33.89830469[kHz]
Relaxation_Delay = 1.06700003
Recvr_Gain = 24
Temp_Get = 27[dC]
X_Points = 65536
X_Prescans = 2
Scans = 128
JEOL
Solutions for Innovation
3ga
CO2Et


abundance
0
100.0
200.0
300.0
400.0
500.0
600.0
X : parts per Million : 1H
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.9809
7.9769
7.9672
7.9645
7.5199
7.5163
7.5038
7.5028
7.2600
4.3949
4.3805
4.3663
4.3521
1.4081
1.3936
1.3795
0.2686
0.2615
0.2544
2.00
1.99
1.99
2.98
8.88
Filename = 3ha_(HK_#2174)-4.jdf
Author = 大●圭翔
Experiment = non
Solvent = CDCl3
Creation_Time = 29-APR-2016 16:50:34
Revision_Time = 17-AUG-2016 18:36:02
Current_Time = 17-AUG-2016 18:36:33
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561[MHz]
X_Offset = 0[Hz]
X_Sweep = 8[kHz]
Relaxation_Delay = 1
Recvr_Gain = 23
Temp_Get = 25.700000076[dC]
X_Points = 65536
X_Prescans = 0
Scans = 16
Si
CO2Et
3ha


abundance
X : parts per Million : 1H
7.4290
7.4131
7.4096
7.2989
7.2977
7.2898
7.2854
7.2830
7.2600
6.2645
6.2469
6.2328
5.7105
5.7073
5.7041
5.6787
5.6757
5.6726
2.1857
2.1825
2.1712
2.1683
2.1566
2.1536
2.1421
2.1390
1.2990
1.2951
1.2905
0.9083
0.8943
0.8802
2.00
2.96
1.00
1.01
2.03
2.08
6.44
3.15
JEOL
Solutions for Innovation
Filename = (E)-5aa (HK_#3020)-3.jdf
Author = 大●圭爾
Experiment = non
Solvent = CDCl3
Creation_Time = 29-APR-2016 14:24:20
Revision_Time = 24-AUG-2016 13:24:32
Current_Time = 24-AUG-2016 13:25:00
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561[MHz]
X_Offset = 0[Hz]
X_Sweep = 8[kHz]
Relaxation_Delay = 1
Recvr_Gain = 22
Temp_Get = 25.89999962[dC]
X_Points = 65536
X_Prescans = 0
Scans = 24
(E)-5aa


abundance
X : parts per Million : 1H
7.4473
7.4431
7.4317
7.4285
7.3319
7.3253
7.3140
7.3003
7.2903
7.2600
5.9917
5.9851
5.9701
5.6845
5.6630
2.4206
2.4065
2.3915
2.3783
1.4582
1.3213
1.3139
1.3081
0.8997
0.8863
0.8726
2.00
3.00
0.98
0.97
1.98
1.90
6.18
3.10
Filename = (Z)-5aa (HK_#3109)-2.jdf
Author = 大倉圭輔
Experiment = non
Solvent = CDCl3
Creation_time = 29-APR-2016 14:27:51
Revision_time = 24-AUG-2016 13:25:13
Current_Time = 24-AUG-2016 13:25:37
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561[MHz]
X_Offset = 0[Hz]
X_Sweep = 8[kHz]
Relaxation_Delay = 1
Recvr_Gain = 21
Temp_Get = 25.5[dC]
X_Points = 65536
X_Prescans = 0
Scans = 24
(Z)-5aa


abundance
0
20.0
40.0
60.0
80.0
100.0
120.0
140.0
160.0
180.0
200.0
X : parts per Million : 1H
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.9356
7.9214
7.4862
7.4828
7.3901
7.3744
7.3561
7.3446
7.3409
7.2600
6.7196
6.6956
5.9384
5.9144
2.07
2.06
6.29
1.02
1.00
JEOL
Solutions for Innovation
Filename = (Z)-5ab (HK_#3146)-3.jdf
Author = 大倉圭輔
Experiment = non
Solvent = CDCl3
Creation_Time = 29-APR-2016 14:29:44
Revision_Time = 24-AUG-2016 13:26:02
Current_Time = 24-AUG-2016 13:26:26
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561[MHz]
X_Offset = 0[Hz]
X_Sweep = 8[kHz]
Relaxation_Delay = 1
Recvr_Gain = 25
Temp_Get = 26.60000038[dC]
X_Points = 65536
X_Prescans = 0
Scans = 24
(Z)-5ab



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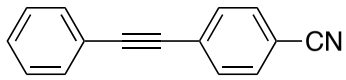
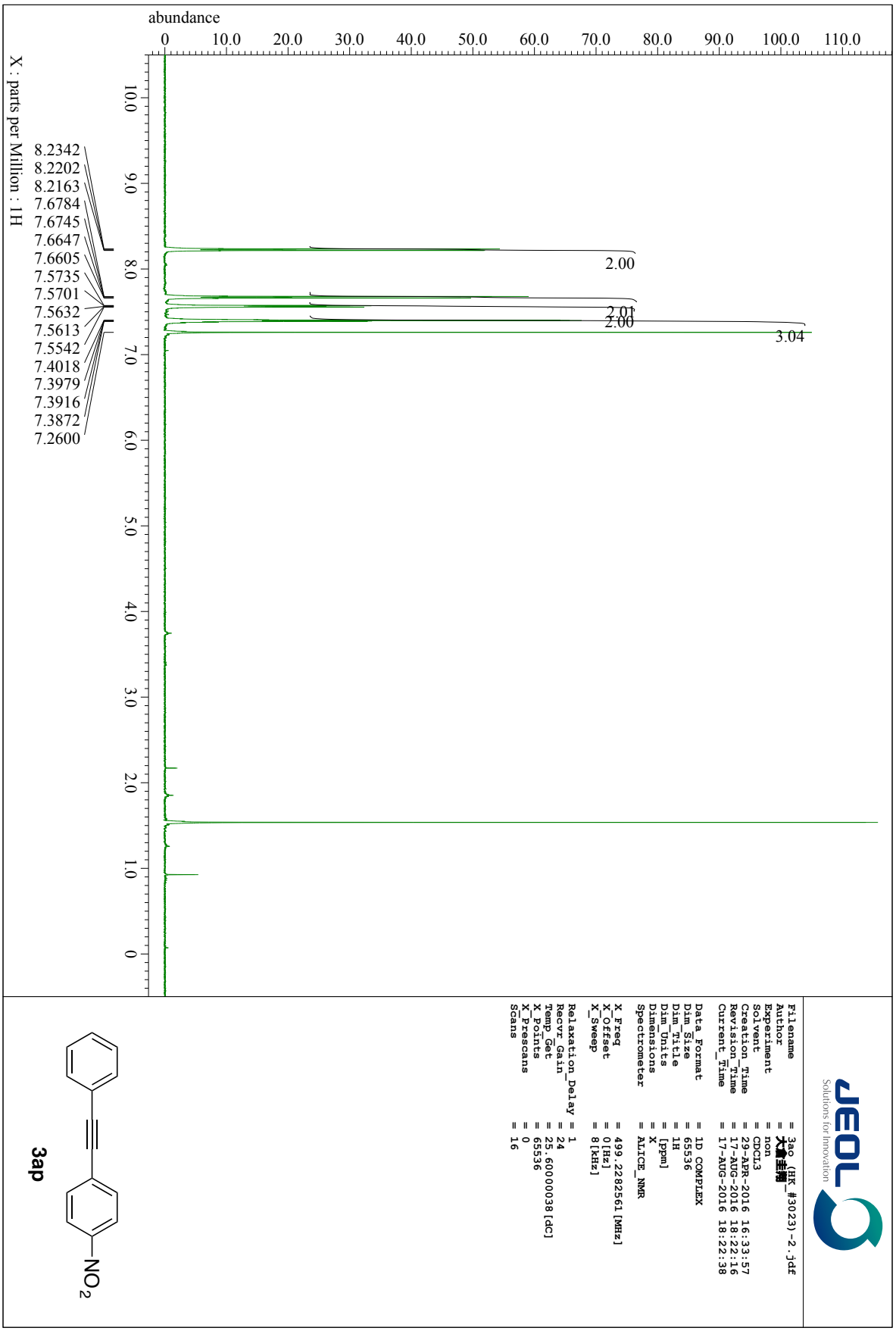
```
Filename          = 3an (HK #3018)-3.jdf
Author            = 大倉圭翔
Experiment        = non
Solvent           = CDCL3
Creation_Time     = 29-APR-2016 16:32:25
Revision_Time     = 17-AUG-2016 18:21:17
Current_Time      = 17-AUG-2016 18:21:40

Data_Format       = 1D COMPLEX
Dim_Size          = 65536
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = ALICE_NMR

X_Freq            = 499.2282561[MHz]
X_Offset          = 0[Hz]
X_Sweep           = 8[kHz]

Relaxation_Delay  = 1
Recvr_Gain        = 25
Temp_Get          = 26.5[dC]
X_Points          = 65536
X_Prescans        = 0
Scans             = 8
```



3ao


abundance
X : parts per Million : 1H
8.2342
8.2202
8.2163
7.6784
7.6745
7.6647
7.6605
7.5735
7.5701
7.5632
7.5613
7.5542
7.4018
7.3979
7.3916
7.3872
7.2600
2.00
2.01
2.00
3.04
Filename = 3ao_(HK_#3023)-2.jdf
Author = 大●圭爾
Experiment = non
Solvent = CDCL3
Creation_Time = 29-APR-2016 16:33:57
Revision_Time = 17-AUG-2016 18:22:16
Current_Time = 17-AUG-2016 18:22:38
Data_Format = 1D COMPLEX
Dim_Size = 65536
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Freq = 499.2282561 [MHz]
X_Offset = 0 [Hz]
X_Sweep = 8 [kHz]
Relaxation_Delay = 1
Recvr_Gain = 24
Temp_Get = 25.60000038 [dC]
X_Points = 65536
X_Prescans = 0
Scans = 16
3ap
NO2