Supporting Information

NHC-Gold(I) Complexes as Effective Catalyst for Carboxylative Cyclization of Propargylamines with Carbon Dioxide

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General information

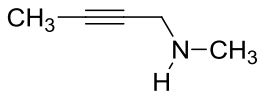
Solvents were purchased from Kanto Chemical Co., Inc. or Nacalai Tesque, Inc., and dried by refluxing over sodium benzophenone ketyl (toluene, THF, ether), P_2O_5 (CH_2Cl_2, CH_3CN) or CaH_2 (methanol, 2-propanol, *tert*-butyl alcohol) and distilled under argon. Carbon dioxide (99.999%) was purchased from Showa Tansan. Other reagents were used as delivered. ^{1}H(300.40 and 399.78 MHz), ^{13}C(100.53 MHz) NMR were recorded with JEOL JNM-LA300 and JNM-ECX400 spectrometers. The NMR chemical shifts were referenced to $SiMe_4$ by using residual protio impurities in the deuterated solvent. Abbreviations for ^{1}H NMR are as follows: s = singlet, d = doublet, t = triplet, q = quartet, sept = septet, m = multiplet, or br = broad. Elemental analyses were carried out using a PE2400 Series II CHNS/O Analyzer (Parkin Elmer). IR spectra were recorded on a JASCO FT/IR-610 spectrometer. Mass spectra (MS) were obtained with a JEOL JMS-SX102A instrument at Material Analysis Suzukake-dai Center, Technical Department, Tokyo Institute of Technology. Recycling preparative HPLC was performed on a Japan Analytical Industry LC-918 system connected to RI and UV detectors.

AuCl(IPr),[1] AuCl(IMes),[2] AuCl(I^{t}Bu),[3] AuCl[P(C_6H_5)$_3$],[4] AuCl[P(OC_2H_5)$_3$],[4] AuCl[P(C_2H_5)$_3$],[4] AgCl(IPr),[2] CuCl(IPr),[5] and AuOH(IPr)[6] were prepared according to literatures.

Terminal propargylamine (**1e**) was purchased from Sigma-Aldrich, and used after purification by distillation under argon. Aliphatic internal propargylamines (**1a**-**1d** and **1f**-**1i**) were prepared by propargylation of primary amines by treatment with the corresponding propargyl chlorides or bromides in the presence of base. Aromatic internal propargylamines (**1j**-**1k**) were prepared by Sonogashira reaction of the corresponding aryl halides and terminal propargylamines.[7]

Characterization data for amine substrates 1.

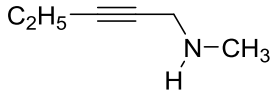
1-Methylamino-2-butyne (1a)[8]



Colorless liquid; ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.10 (brs, 1H, N*H*), 1.79 (t, $^5J_{HH}$ = 2.4 Hz, 3H, CH_3C), 2.42 (s, 3H, NCH_3), 3.30 (q, $^5J_{HH}$ = 2.4 Hz, 2H, CH_2N); ^{13}C{^{1}H} NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 3.5, 35.4, 40.4, 77.3, 79.0.

HRMS (ESI) Calcd for C_5H_9N 83.0735 (M^+); Found 83.0735.

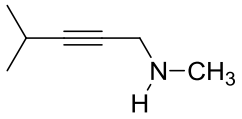
1-Methylamino-2-pentyne (1b)



Colorless liquid; ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 0.99 (brs, 1H, N*H*), 1.06 (t, $^3J_{HH}$ = 7.4 Hz, 3H, CH_3CH$_2$), 2.13 (qt, $^3J_{HH}$ = 7.4 Hz, $^5J_{HH}$ = 2.2 Hz, 2H, CH$_3$CH_2), 2.38 (s, 3H, NCH_3), 3.27 (t, $^5J_{HH}$ = 2.2 Hz, 2H, CH_2N); ^{13}C{^{1}H} NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 12.3, 14.1, 35.3, 40.4, 77.3, 85.0.

HRMS (ESI) Calcd for $C_6H_{12}N$ 98.0964 ($M + H^+$); Found 98.0965.

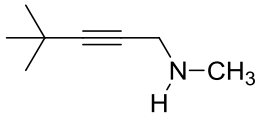
1-Methylamino-4-methyl-2-pentyne (1c)



Colorless liquid; ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.14 (d, $^{3}J_{HH}$ = 6.8 Hz, 6H, $(CH_3)_2CH$), 1.27 (brs, 1H, N*H*), 2.43 (s, 3H, NCH_3), 2.54 (t of sept, $^{3}J_{HH}$ = 6.8 Hz, $^{5}J_{HH}$ = 1.6 Hz, 1H, $(CH_3)_2CH$), 3.33 (d, $^{5}J_{HH}$ = 1.6 Hz, 2H, CH_2N); $^{13}C\{^{1}H\}$ NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 20.6, 23.3, 35.3, 40.4, 77.0, 89.5.

HRMS (ESI) Calcd for $C_7H_{14}N$ ($M + H^+$) 112.1121; Found 112.1118.

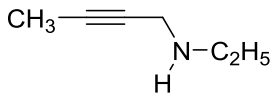
1-Methylamino-4,4-dimethyl-2-pentyne (1d)



Colorless liquid; ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.19 (s, 9H, $(CH_3)_3C$), 2.42 (s, 3H, NCH_3), 3.33 (s, 2H, CH_2N), 4.20 (s, 1H, NH); $^{13}C\{^{1}H\}$ NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 27.4, 31.3 , 35.3, 40.5, 76.3, 92.3.

HRMS (ESI) Calcd for $C_8H_{16}N$ ($M + H^+$) 126.1277; Found 126.1280.

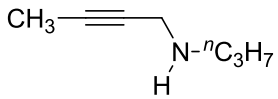
1-Ethylamino-2-butyne (1f)



Colorless liquid; ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.08 (t, $^{3}J_{HH}$ = 7.1 Hz, 3H, CH_2CH_3), 1.49 (brs, 1H, N*H*), 1.79 (t, $^{5}J_{HH}$ = 2.4 Hz, 3H, $CH_3C{\equiv}C$), 2.69 (q, $^{3}J_{HH}$ = 7.1 Hz, 2H, CH_2CH_3), 3.34 (q, $^{5}J_{HH}$ = 2.4 Hz, 2H, CH_2N); $^{13}C\{^{1}H\}$ NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 3.5, 15.0, 38.4, 43.0 , 77.3, 78.8.

HRMS (ESI) Calcd for $C_6H_{12}N$ ($M + H^+$) 98.0964; Found 98.0965.

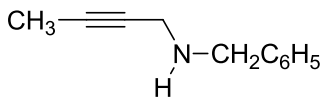
1-Propylamino-2-butyne (1g)



Colorless liquid; ^{1}H NMR (399.78 MHz, $C_2D_2Cl_4$, rt, δ/ppm): 0.88 (t, $^{3}J_{HH}$ = 7.3 Hz, 3H, CH_2CH_3), 1.25 (brs, 1H, N*H*), 1.47 (tq, $^{3}J_{HH}$ = 7.3 Hz, $^{3}J_{HH}$ = 7.3 Hz, 2H, CH_2CH_3), 1.78 (t, $^{5}J_{HH}$ = 2.4 Hz, 3H, $CH_3C{\equiv}C$), 2.59 (t, $^{3}J_{HH}$ = 7.3 Hz, 2H, NCH_2), 3.33 (q, $^{5}J_{HH}$ = 2.4 Hz, 2H, CH_2N); $^{13}C\{^{1}H\}$ NMR (100.53 MHz, $C_2D_2Cl_4$, rt, δ/ppm): 3.80, 12.1, 23.2, 38.7, 50.9, 77.9, 79.2.

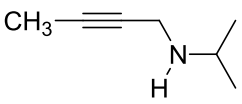
HRMS (ESI) Calcd for $C_7H_{14}N$ ($M + H^+$) 112.1121; Found 112.1120.

1-Benzylamino-2-butyne (1h)[9]



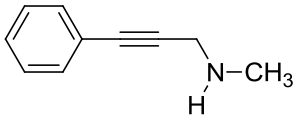
Colorless liquid; ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.62 (brs, 1H, N*H*), 1.83 (t, $^{5}J_{HH}$ = 2.2 Hz, 3H, $CH_3C{\equiv}C$), 3.36 (q, $^{5}J_{HH}$ = 2.2 Hz, 2H, CH_2N), 3.84 (s, 2H, NCH_2), 7.24-7.34 (m, 5H, C_6H_5); $^{13}C\{^{1}H\}$ NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 3.6, 38.0, 52.7, 77.3, 79.2, 127.1, 128.45, 128.47, 139.9.

1-Isopropylamino-2-butyne(1i) [10]



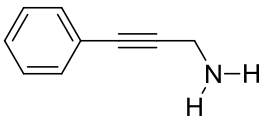
Colorless liquid; ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.02 (d, $^{3}J_{HH}$ = 6.2 Hz, 6H, NCH(CH_3)$_2$), 1.35 (brs, 1H, N*H*), 1.79 (t, $^{5}J_{HH}$ = 2.4 Hz, 3H, CH_3C≡C), 2.95 (sept, $^{3}J_{HH}$ = 6.2 Hz, 1H, NC*H*(CH_3)$_2$), 3.34 (q, $^{5}J_{HH}$ = 2.4 Hz, 2H, CH_2N); ^{13}C{^{1}H} NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 3.5, 22.6, 36.1, 47.1, 77.4, 78.6.

1-Methylamino-3-phenyl-2-propyne (1j)[11]



Colorless liquid; ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.21 (brs, 1H, N*H*), 2.53 (s, 3H, NHCH_3), 3.60 (s, 2H, CH_2N), 7.28-7.29 (m, 3H, Ar), 7.41-7.43 (m, 2H, Ar); ^{13}C{^{1}H} NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 35.5, 40.9, 83.6, 87.6, 123.4, 128.1, 128.3, 131.7.

1-Amino-3-phenyl-2-propyne (1k)[12]



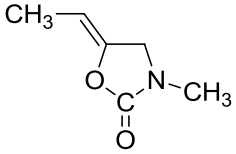
Colorless liquid; ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.44 (s, 2H, NH_2), 3.64 (s, 2H, CH_2N), 7.28-7.30 (m, 3H, Ar), 7.40-7.42 (m, 2H, Ar); ^{13}C{^{1}H} NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 32.3, 82.5, 90.3, 123.3, 128.1, 128.4, 131.6.

Carboxylative cyclization of 1-methylamino-2-butyne with mixed gases.

1-Methylamino-2-butyne (2 mmol) was added to a methanol (2 mL) suspension of AuCl(IPr) (0.04 mmol) in a 20 mL Schlenk flask under Ar atomosphere. The flask was charged with 2.0 L of mixed gas and stirred at 40 ºC. After evaporation of the resulting mixture under reduced pressure, the product yield was determined by ^{1}H NMR using durene as an internal standard.

Characterization data for 2.

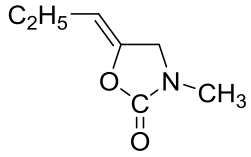
(Z)-3-Methyl-5-ethylidene-1,3-oxazolidin-2-one (2a)



White powder; mp 73.3-75.2 ºC. ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.66 (dt, $^{3}J_{HH}$ = 7.0 Hz, $^{5}J_{HH}$ = 2.2 Hz, 3H, CH_3CH), 2.90 (s, 3H, NCH_3), 4.09 (dq, $^{4}J_{HH}$ = 2.2 Hz, $^{5}J_{HH}$ = 2.2 Hz, 2H, CCH_2N), 4.59 (qt, $^{3}J_{HH}$ = 7.0 Hz, $^{4}J_{HH}$ = 2.2 Hz, 1H, CH_3C*H*); ^{13}C{^{1}H} NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 9.8, 30.5, 49.8, 97.2, 141.5, 156.1(C=O). IR (cm^{-1}, KBr): 1770(C=O).

Anal. Calcd for $C_6H_9NO_2$: C, 56.68; H, 7.13; N, 11.02. Found: C, 56.61; H, 7.16; N, 11.03.

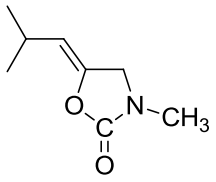
(Z)-3-Methyl-5-propylidene-1,3-oxazolidin-2-one (2b)



Pale yellow liquid; ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 0.97 (t, $^{3}J_{HH}$ = 7.5 Hz, 3H, C*H*$_3$CH$_2$), 2.15 (dqt, $^{3}J_{HH}$ = 7.5 Hz, $^{3}J_{HH}$ = 7.5 Hz, $^{5}J_{HH}$ = 1.7 Hz, 2H, CH$_3$C*H*$_2$), 2.90 (s, 3H, NC*H*$_3$), 4.09 (dt, $^{4}J_{HH}$ = 2.0 Hz, $^{5}J_{HH}$ = 1.7 Hz, 2H, CC*H*$_2$N), 4.57 (tt, $^{3}J_{HH}$ = 7.5 Hz, $^{4}J_{HH}$ = 2.0 Hz, 1H, CH$_2$C*H*); ^{13}C{^{1}H} NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 14.1, 18.3, 30.6, 49.9, 104.8, 140.6, 156.3(C=O). IR (cm^{-1}, $CHCl_3$): 1778(C=O).

HRMS (ESI) Calcd for $C_7H_{11}NNaO_2$ (M + Na^{+}) 164.0682; Found 164.0684.

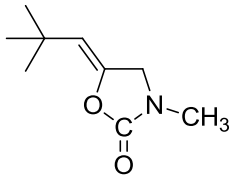
(Z) -3-Methyl-5-(2'-methylpropylidene)-1,3-oxazolidin-2-one (2c)



Pale yellow viscous liquid; ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 0.96 (d, $^{3}J_{HH}$ = 6.7 Hz, 6H, (C*H*$_3$)$_2$CH), 2.75 (qt, $^{3}J_{HH}$ = 6.7 Hz, $^{4}J_{HH}$ =1.5 Hz, 1H, (CH$_3$)$_2$C*H*), 2.89 (s, 3H, NC*H*$_3$), 4.06 (d, $^{4}J_{HH}$ = 1.5 Hz, 2H, CC*H*$_2$N), 4.42 (dt, $^{3}J_{HH}$ = 6.7 Hz, $^{4}J_{HH}$ = 1.5 Hz, 1H, CH$_2$C*H*); ^{13}C{^{1}H} NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 23.0, 25.0, 30.6, 49.9, 110.4, 139.4, 156.3 (C=O). IR (cm^{-1}, $CHCl_3$): 1752(C=O).

Anal. Calcd for $C_8H_{13}NO_2$: C, 61.91; H, 8.44; N, 9.03. Found: C, 61.85; H, 8.40; N, 8.80.

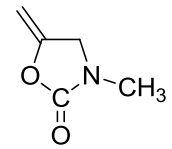
(Z) -3-Methyl-5-(2', 2'-dimethylpropylidene)-1,3-oxazolidin-2-one (2d)



White powder; mp 87.0-88.4 ºC. ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.14 (s, 9H, (C*H*$_3$)$_3$C), 2.90 (s, 3H, NC*H*$_3$), 4.06 (d, $^{4}J_{HH}$ = 2.2 Hz, 2H, CC*H*$_2$N), 4.49 (t, $^{4}J_{HH}$ = 2.2 Hz, 1H, (CH$_3$)$_3$CC*H*); ^{13}C{^{1}H} NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 30.4, 30.5, 31.7, 50.6, 112.8, 139.1, 156.4 (C=O). IR (cm^{-1}, KBr): 1712(C=O).

Anal. Calcd for $C_9H_{15}NO_2$: C, 63.88; H, 8.93; N, 8.28. Found: C, 63.80; H, 8.95; N, 8.19.

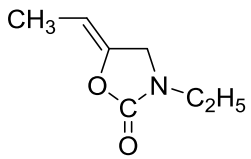
3-Methyl-5-methylene-1,3-oxazolidin-2-one (2e)[13]



^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 2.91 (s, 3H, NC*H*$_3$), 4.16 (dd, $^{4}J_{HH}$ = 2.4 Hz, $^{4}J_{HH}$ = 2.4 Hz, 2H, CC*H*$_2$N), 4.28 (dt, $^{2}J_{HH}$ = 3.1 Hz, $^{4}J_{HH}$ = 2.4 Hz, 1H), 4.72 (dt, $^{2}J_{HH}$ = 3.1 Hz, $^{4}J_{HH}$ = 2.4 Hz, 1H).

^{13}C{^{1}H} NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 30.6, 50.6, 86.6, 148.9, 155.9.

(Z)-3-Ethyl-5-ethylidene-1,3-oxazolidin-2-one (2f)

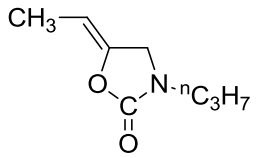


Pale yellow liquid; ^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.12 (t, $^{3}J_{HH}$ = 7.3 Hz, 3H, NCH$_2$C*H*$_3$), 1.63 (dt, $^{3}J_{HH}$ = 6.8 Hz, $^{5}J_{HH}$ = 2.3 Hz, 3H, C*H*$_3$CH), 3.31 (q, $^{3}J_{HH}$ = 7.3 Hz, 2H, NC*H*$_2$CH$_3$), 4.06 (dq, $^{4}J_{HH}$ = 2.2 Hz, $^{5}J_{HH}$ = 2.3 Hz, 2H, NC*H*$_2$C=CH), 4.57 (qt, $^{3}J_{HH}$ = 6.8 Hz, $^{4}J_{HH}$ = 2.2 Hz, 1H,

CH_3CH); $^{13}C\{^1H\}$ NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 10.0, 12.5, 38.5, 47.1, 97.2, 141.9, 155.6 (C=O). IR (cm^{-1}, $CHCl_3$): 1774(C=O).

HRMS (ESI) Calcd for $C_7H_{11}NNaO_2$ ($M + Na^+$) 164.0682; Found 164.0678.

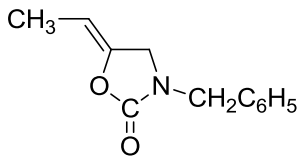
(Z)-3-Propyl-5-ethylidene-1,3-oxazolidin-2-one (2g)



Pale yellow liquid; ^1H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 0.89 (t, $^3J_{HH}$ = 7.5 Hz, 3H, CH_2CH_3), 1.54 (tq, $^3J_{HH}$ = 7.5 Hz, $^3J_{HH}$ = 7.2 Hz, 2H, CH_2CH_3), 1.64 (dt, $^3J_{HH}$ = 7.0 Hz, $^5J_{HH}$ = 2.2 Hz, 3H, CH_3CH), 3.22 (t, $^3J_{HH}$ = 7.2 Hz, 2H, NCH_2CH_2), 4.06 (dq, $^4J_{HH}$ = 2.2 Hz, $^5J_{HH}$ = 2.2 Hz, 2H, $NCH_2C{=}CH$), 4.57 (qt, $^3J_{HH}$ = 7.0 Hz, $^4J_{HH}$ = 2.2 Hz, 1H, CH_3CH); $^{13}C\{^1H\}$ NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 10.0, 11.1, 20.6, 45.4, 47.7, 97.3, 141.9, 156.2 (C=O). IR (cm^{-1}, $CHCl_3$): 1765(C=O).

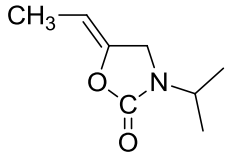
HRMS (ESI) Calcd for $C_8H_{13}NNaO_2$ ($M + Na^+$) 178.0835; Found 178.0838.

(Z)-3-Benzyl-5-ethylidene-1,3-oxazolidin-2-one (2h)[14]



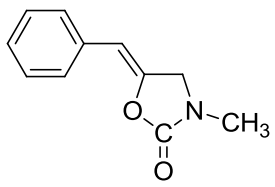
White powder; mp 39.5-41.5 ºC. ^1H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.67 (dt, $^3J_{HH}$ = 7.0 Hz, $^5J_{HH}$ = 2.2 Hz, 3H, CH_3CH), 3.95 (dq, $^4J_{HH}$ = 2.2 Hz, $^5J_{HH}$ = 2.2 Hz, 2H, CCH_2N), 4.45 (s, 2H, $NCH_2C_6H_5$), 4.55 (qt, $^3J_{HH}$ = 7.0 Hz, $^4J_{HH}$ = 2.2 Hz, 1H, CH_3CH), 7.26-7.38 (m, 5H, Ar); $^{13}C\{^1H\}$ NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 10.0, 47.2, 48.0, 97.7, 128.2, 129.0, 135.3, 141.7, 156.1 (C=O). IR (cm^{-1}, KBr): 1776(C=O).

(Z)-3-Isopropyl-5-ethylidene-1,3-oxazolidin-2-one (2i)



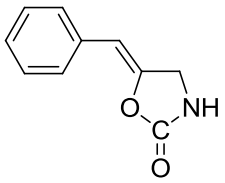
^1H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 1.17 (d, $^3J_{HH}$ = 6.7 Hz, 6H, $CH(CH_3)_2$), 1.67 (dt, $^3J_{HH}$ = 7.0 Hz, $^5J_{HH}$ = 2.2 Hz, 3H, CH_3CH), 4.04 (dq, $^4J_{HH}$ = 2.2 Hz, $^5J_{HH}$ = 2.2 Hz, 2H, CCH_2N), 4.15 (septet, $^3J_{HH}$ = 6.7 Hz, 1H, $CH(CH_3)_2$), 4.59 (qt, $^3J_{HH}$ = 7.0 Hz, $^4J_{HH}$ = 2.2 Hz, 1H, CH_3CH); $^{13}C\{^1H\}$ NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 10.0, 19.8, 43.0, 44.7, 97.2, 142.2, 155.3 (C=O).

(Z)-3-Methyl-5-benzylidene-1,3-oxazolidin-2-one (2j)[13]



White powder; mp 140.0-142.6 ºC. ^1H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 2.96 (s, 3H, NCH_3), 4.29 (d, $^4J_{HH}$ = 2.1 Hz, 2H, NCH_2C), 5.50 (t, $^4J_{HH}$ = 2.1 Hz, 1H, C_6H_5CH), 7.20 (t, $^3J_{HH}$ = 7.3 Hz, 1H, Ar), 7.32 (dd, $^3J_{HH}$ = 7.3 Hz, $^3J_{HH}$ = 7.6 Hz, 2H, Ar), 7.56 (d, $^3J_{HH}$ = 7.6 Hz, 1H, Ar); $^{13}C\{^1H\}$ NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 30.6 51.0, 103.0 126.9, 128.3, 128.6, 133.6, 141.8, 155.8 (C=O). IR (cm^{-1}, KBr): 1781(C=O).

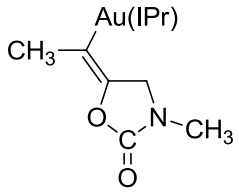
(Z) -5-Benzylidene-1,3-oxazolidin-2-one (2k)



White powder; mp 156.0-157.8 ºC.^{1}H NMR (399.78 MHz, $CDCl_3$, rt, δ/ppm): 4.40 (d, $^{4}J_{HH}$ = 2.0 Hz, 2H, CH_2), 5.52 (t, $^{4}J_{HH}$ = 2.0 Hz, 1H, C_6H_5CH), 7.21 (dd, $^{3}J_{HH}$ = 7.5 Hz, $^{3}J_{HH}$ = 7.5 Hz, *p*-Ar), 7.33 (t, $^{3}J_{HH}$ = 7.5 Hz, *m*-Ar), 7.56 (d, $^{3}J_{HH}$ = 7.5 Hz, *o*-Ar); $^{13}C\{^{1}H\}$ NMR (100.53 MHz, $CDCl_3$, rt, δ/ppm): 45.2, 103.4, 127.0, 128.3, 128.6, 133.5, 143.9, 157.2 (C=O). IR (cm^{-1}, KBr): 1763(C=O).
Anal. Calcd for $C_{10}H_9NO_2$: C, 68.56; H, 5.18; N, 8.00. Found: C, 68.73; H, 5.12; N, 7.79.

Synthesis of 3a.

1-Methylamino-2-butyne (500 μmol) was added to a THF (7 mL) solution of AuOH(IPr) (500 μmol) in a 20 mL Schlenk flask under Ar atomosphere. The Schlenk flask was charged with CO_2 and stirred at 40 ºC for 15 h. The resulting mixture was evaporated and washed with ether then recrystallized from THF and *n*-pentane.



Colorless crystals; ^{1}H NMR (399.78 MHz, THF-d_8, rt, δ/ppm): 1.20 (d, $^{3}J_{HH}$ = 7.0 Hz, 12H, (CH_3)$_2$CH), 1.33 (d, $^{3}J_{HH}$ = 7.0 Hz, 12H, (CH_3)$_2$CH), 1.41(t, 3H, $^{5}J_{HH}$ = 1.8 Hz, CH_3C=C), 2.56 (s, 3H, NCH_3), 2.64 (sept, $^{3}J_{HH}$ = 7.0 Hz, 12H, (CH_3)$_2$CH), 3.27 (q, $^{5}J_{HH}$ = 1.8 Hz, 2H, CH_2), 7.32 (d, $^{3}J_{HH}$ = 7.7 Hz, 4H, Ar), 7.47 (t, $^{3}J_{HH}$ = 7.7 Hz, 2H, Ar), 7.51 (s, 2H, NC*H*=C*H*N); $^{13}C\{^{1}H\}$ NMR (100.53 MHz, THF-d_8, rt, δ/ppm): 18.7, 23.1, 23.9, 28.7, 29.3, 51.0, 123.4, 123.6, 129.9, 130.4, 135.1, 139.5, 145.9, 156.9 (N*C*O_2), 196.6(N*C*N). IR (cm^{-1}, KBr): 1750(C=O).
Anal. Calcd for $C_{33}H_{44}N_3O_2Au$: C, 55.69; H, 6.23; N, 5.90. Found: C, 55.53; H, 6.03; N, 5.72.

X-ray structure determination for 2a and 3a.

All measurements were made on a Rigaku Saturn CCD area detector equipped with graphite-monochromated Mo-$K\alpha$ radiation (λ = 0.71070 Å) under nitrogen stream at 123 K. Indexing was performed from eighteen images. The crystal-to-detector distance was 45.05 mm. The data were collected to a maximum 2θ value of 55.0°. A total of 720 oscillation images were collected. A sweep of data was carried out using ω scans from –110.0 to 70.0° in 0.5° steps, at χ = 45.0° and ϕ = 0.0°. A second sweep was performed using ω scans from –110.0 to 70.0° in 0.5° steps, at χ = 45.0° and ϕ = 90.0°. Intensity data were collected for Lorentz-polarization effects as well as absorption. Structure solution and refinements were performed with the CrystalStructure program package. The heavy atom positions were determined by Direct methods (SIR2002), and the remaining non-hydrogen atoms were found by subsequent Fourier techniques. An empirical absorption correction based on equivalent reflections was applied to all data. All non-hydrogen atoms other than solvent molecules were refined anisotropically by full-matrix least-square techniques based on F^2. All hydrogen atoms were constrained to ride on their parent atom. Relevant crystallographic data are compiled in Table S1.

Table S1. Crystallographic data for **2a** and **3a**

	2a	**3a**
Empirical Formula	$C_6H_9NO_2$	$C_{33}H_{44}AuN_3O_2$
Formula Weight	127.14	711.70
Crystal Color, Habit	colorless, prism	colorless, prism
Crystal System	monoclinic	monoclinic
Space Group	$P2_1/c$ (#14)	$P2_1$ (#4)
Lattice Parameters	a = 5.687(3) Å	a = 12.931(2) Å
	b = 17.147(9) Å	b = 20.981(2) Å
	c = 6.926(4) Å	c = 24.074(3) Å
	β = 110.433(8) °	β = 104.552(5) °
	V = 633.0(6) Å^3	V = 6322.0(11) Å^3
Z value	4	8
D_{calc}	1.334 g/cm^3	1.495 g/cm^3
F_{000}	272.00	2864.00
μ(MoKα)	1.005 cm^{-1}	47.023 cm^{-1}
Exposure Rate	32.0 sec./º	6.0 sec./º
No. of Reflections Measured	5073	70865
No. of unique reflections	1413	28647
No. Variables	118	1581
$R1$ (I>2.00σ(I))	0.0632	0.0398
w$R2$ (All reflections)	0.1507	0.1010
GOF on F^2	1.000	1.000

$R1 = \Sigma \, ||F_o| - |F_c|| \, / \, \Sigma \, |F_o|$, w$R2 = [\Sigma (w (F_o^2 - F_c^2)^2) / \Sigma \, w(F_o^2)^2]^{1/2}$

References

[1] M. R. Fructos, T. R. Belderrain, P. de Frémont, N. M. Scott, S. P. Nolan, M. M. Díaz-Requejo, P. J. Pérez, *Angew. Chem.***2005,** *117*, 5418-5422. *Angew. Chem. Int. Ed.* **2005,** *44*, 5284-5288.

[2] P. de Frémont, N. M. Scott, E. D. Stevens, T. Ramnial, O. C. Lightbody, C. L. B. Macdonald, J. A. C. Clyburne, C. D. Abernethy, S. P. Nolan, *Organometallics* **2005,** *24*, 6301-6309.

[3] M. R. L. Furst, C. S. J. Cazin, *Chem. Commun.* **2010,** *46*, 6924.

[4] J. Vicente, P. González-Herrero, Y. García-Sánchez, P. G. Jones, M. Bardají, *Inorg. Chem.* **2004,** *43*, 7516-7531.

[5] L. Zhang, J. Cheng, B. Carry, Z. Hou, *J. Am. Chem. Soc.* **2012,** *134*, 14314-14317.

[6] A. Gómez-Suárez, R. S. Ramón, A. M. Z. Slawin, S. P. Nolan, *Dalton Trans.* **2012,** *41*, 5461-5463.

[7] K. M. Miller, C. Molinaro, T. F. Jamison, *Tetrahedron Asymmetry* **2003,** *14*, 3619-3625.

[8] R. A. Walsh, A. T. Bottini, *J. Org. Chem.* **1970,** *35*, 1086–1092.

[9] Y. Hirata, T. Yukawa, N. Kashihara, Y. Nakao, T. Hiyama, *J. Am. Chem. Soc.* **2009,** *131*, 10964–10973.

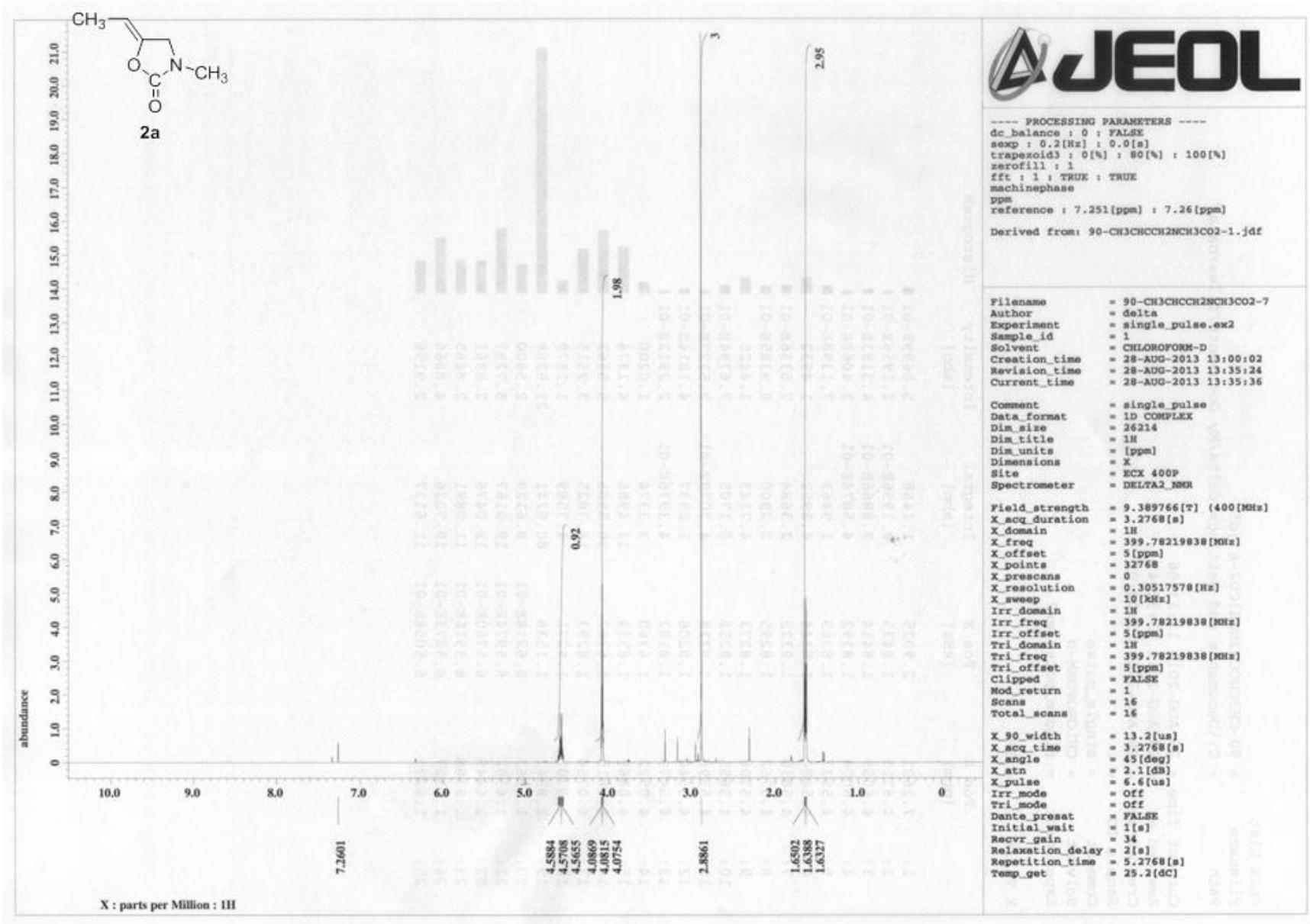
[10] M. L. G. Borst, N. van der Riet, R. H. Lemmens, F. J. J. de Kanter, M. Schakel, A. W. Ehlers, A. M. Mills, M. Lutz, A. L. Spek, K. Lammertsma, *Chem. Eur. J.* **2005,** *11*, 3631–3642.

[11] D. Labrecque, K. T. Nwe, T. H. Chan, *Organometallics* **1994,** *13*, 332–335.

[12] N. Sakai, R. Kanada, M. Hirasawa, T. Konakahara, *Tetrahedron* **2005,** *61*, 9298–9304.

[13] Y. Kayaki, M. Yamamoto, T. Suzuki, T. Ikariya, *Green Chem.* **2006,** *8*, 1019–1021.

[14] S. Yoshida, K. Fukui, S. Kikuchi, T. Yamada, *Chem. Lett.* **2009,** *38*, 786–787.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CH3
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O
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4.0
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8.0
9.0
10.0
11.0
12.0
13.0
14.0
15.0
16.0
17.0
18.0
19.0
20.0
21.0
3
2.95
1.98
0.92
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
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1.6502
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X : parts per Million : 1H
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ppm
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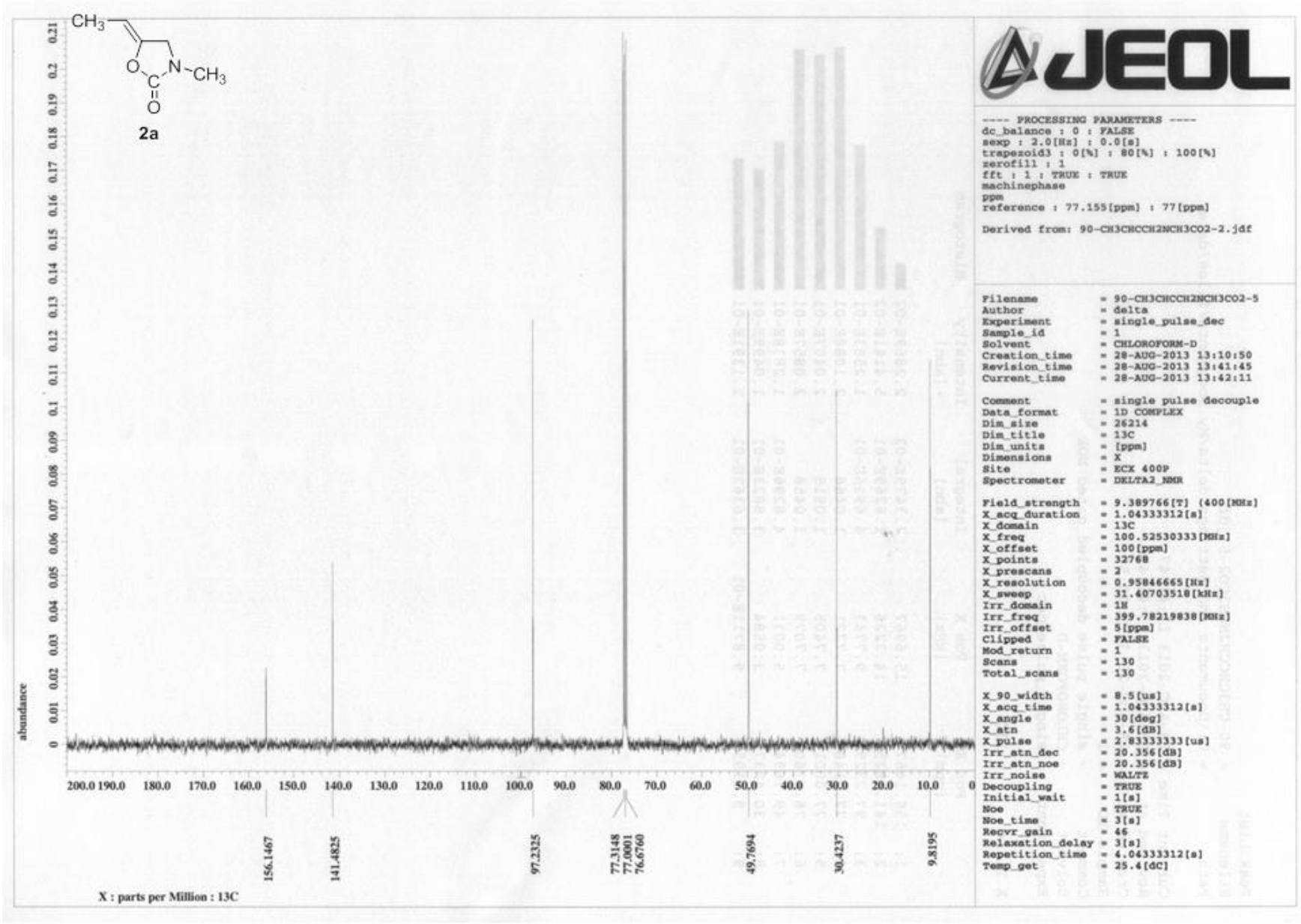
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CH3
C
O
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0.14
0.13
0.12
0.11
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
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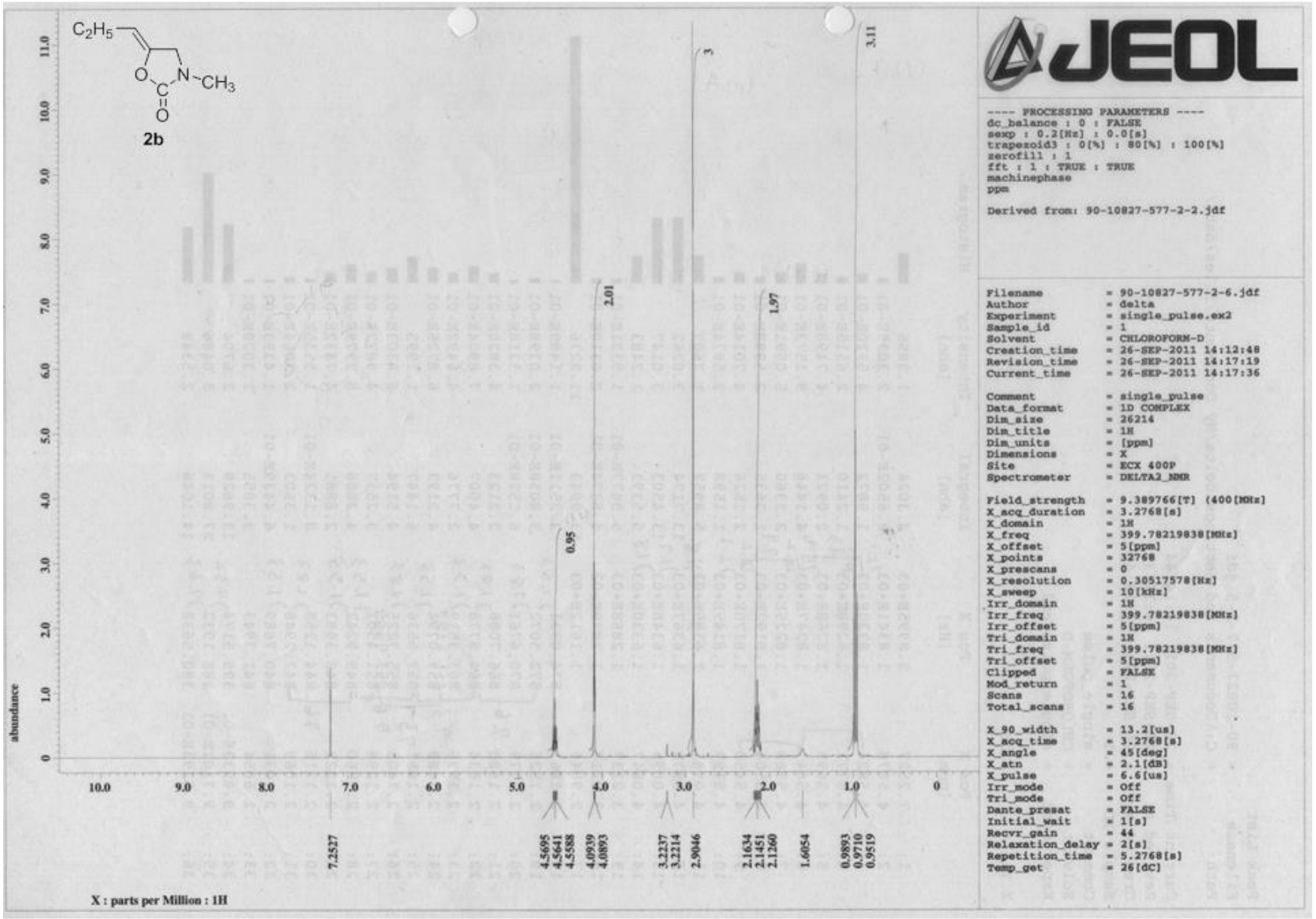
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CH3
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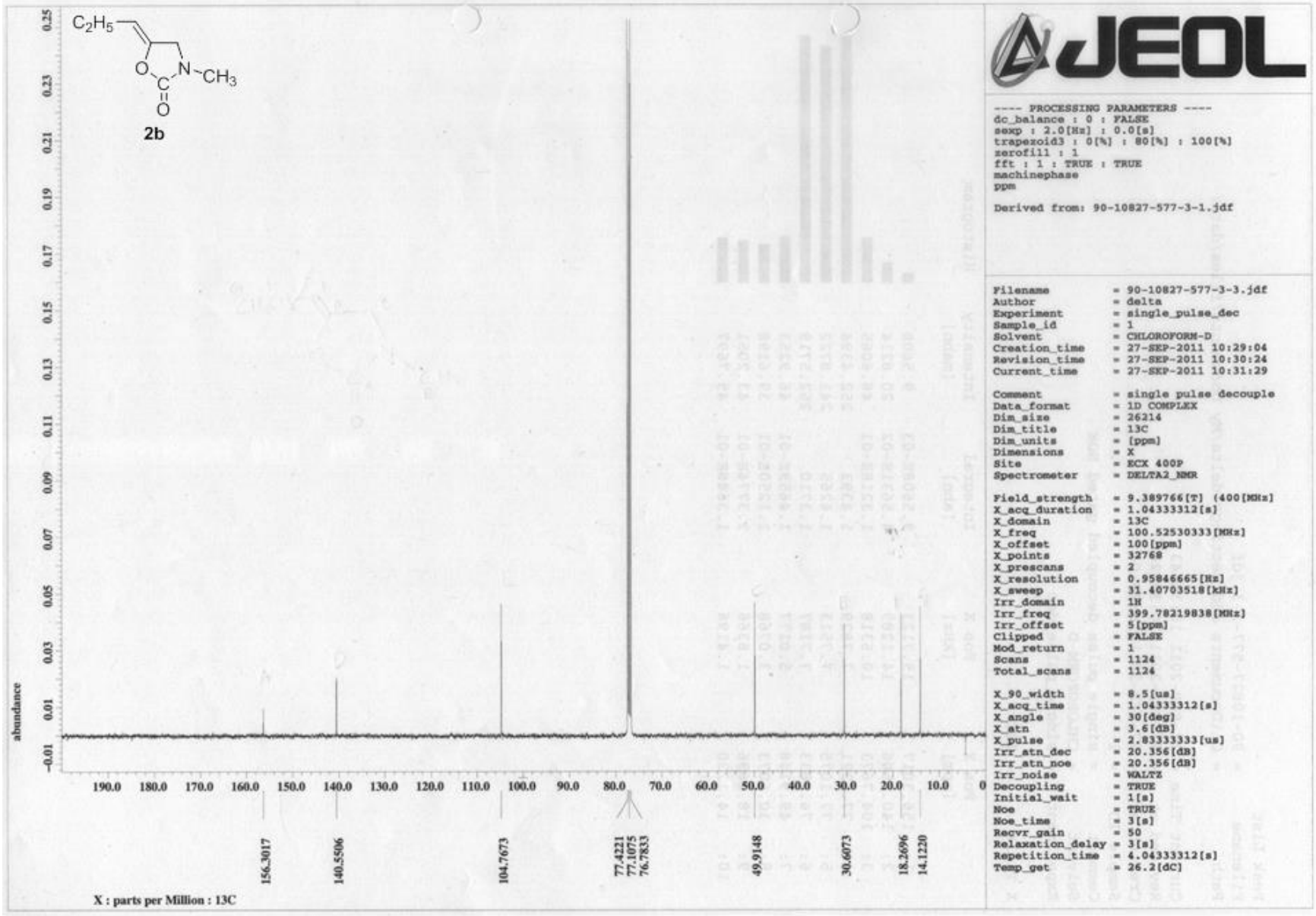
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0.01
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X : parts per Million : 13C
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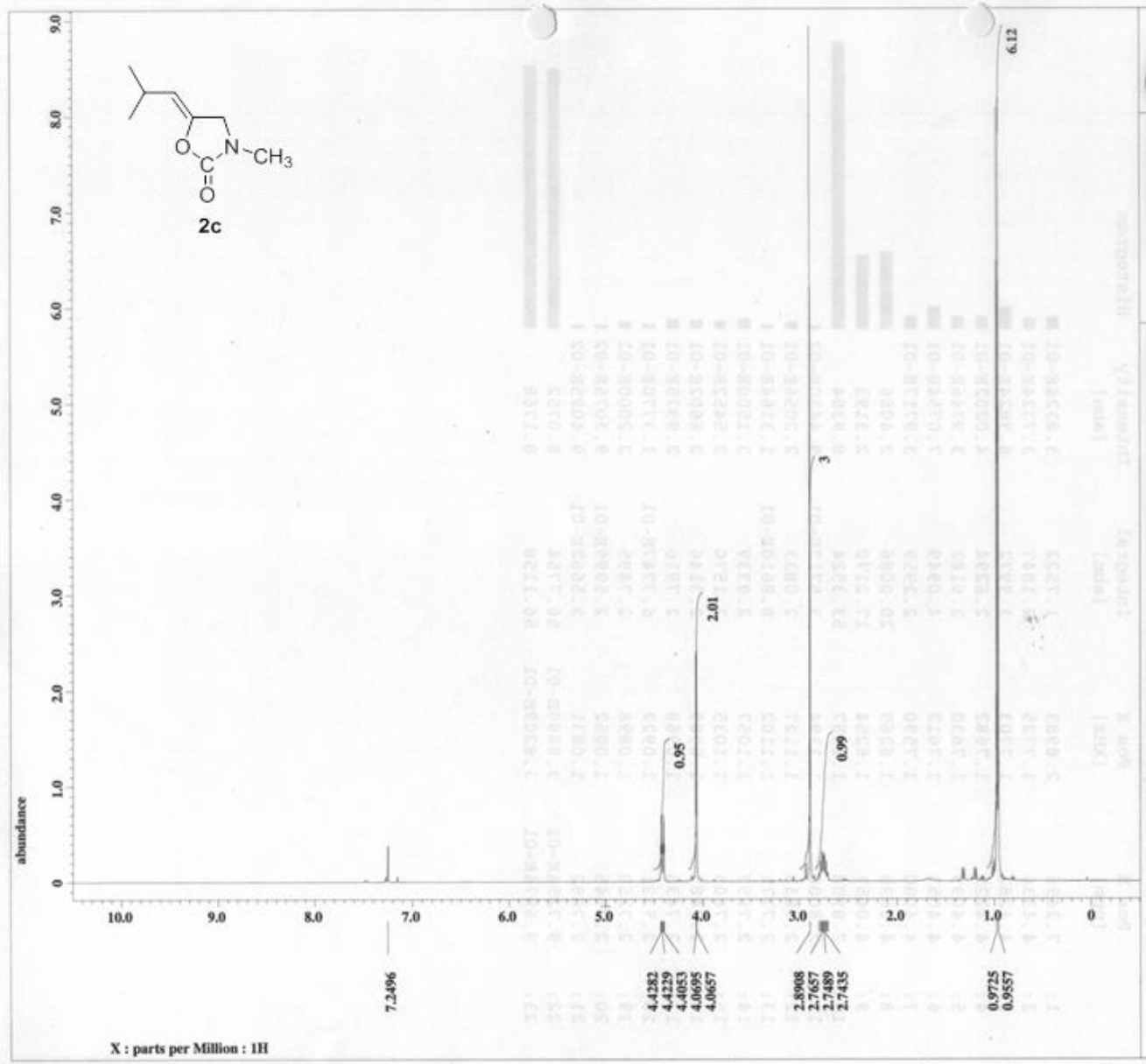
X_angle = 30[deg]
X_atn = 3.6[dB]
X_pulse = 2.83333333[us]
Irr_atn_dec = 20.356[dB]
Irr_atn_noe = 20.356[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 4.04333312[s]
Temp_get = 26.2[dC]



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: 90-11207-735-5.jdf
```

Filename	= 90-11207-735-10.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 13-DEC-2011 22:47:44
Revision_time	= 13-DEC-2011 22:54:29
Current_time	= 13-DEC-2011 22:55:03
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz]
X_acq_duration	= 3.2768[s]
X_domain	= 1H
X_freq	= 399.78219838[MHz]
X_offset	= 5[ppm]
X_points	= 32768
X_prescans	= 0
X_resolution	= 0.30517578[Hz]
X_sweep	= 10[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 399.78219838[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 14[us]
X_acq_time	= 3.2768[s]
X_angle	= 45[deg]
X_atn	= 2.1[dB]
X_pulse	= 7[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 36
Relaxation_delay	= 2[s]
Repetition_time	= 5.2768[s]
Temp_get	= 403.2[dC]

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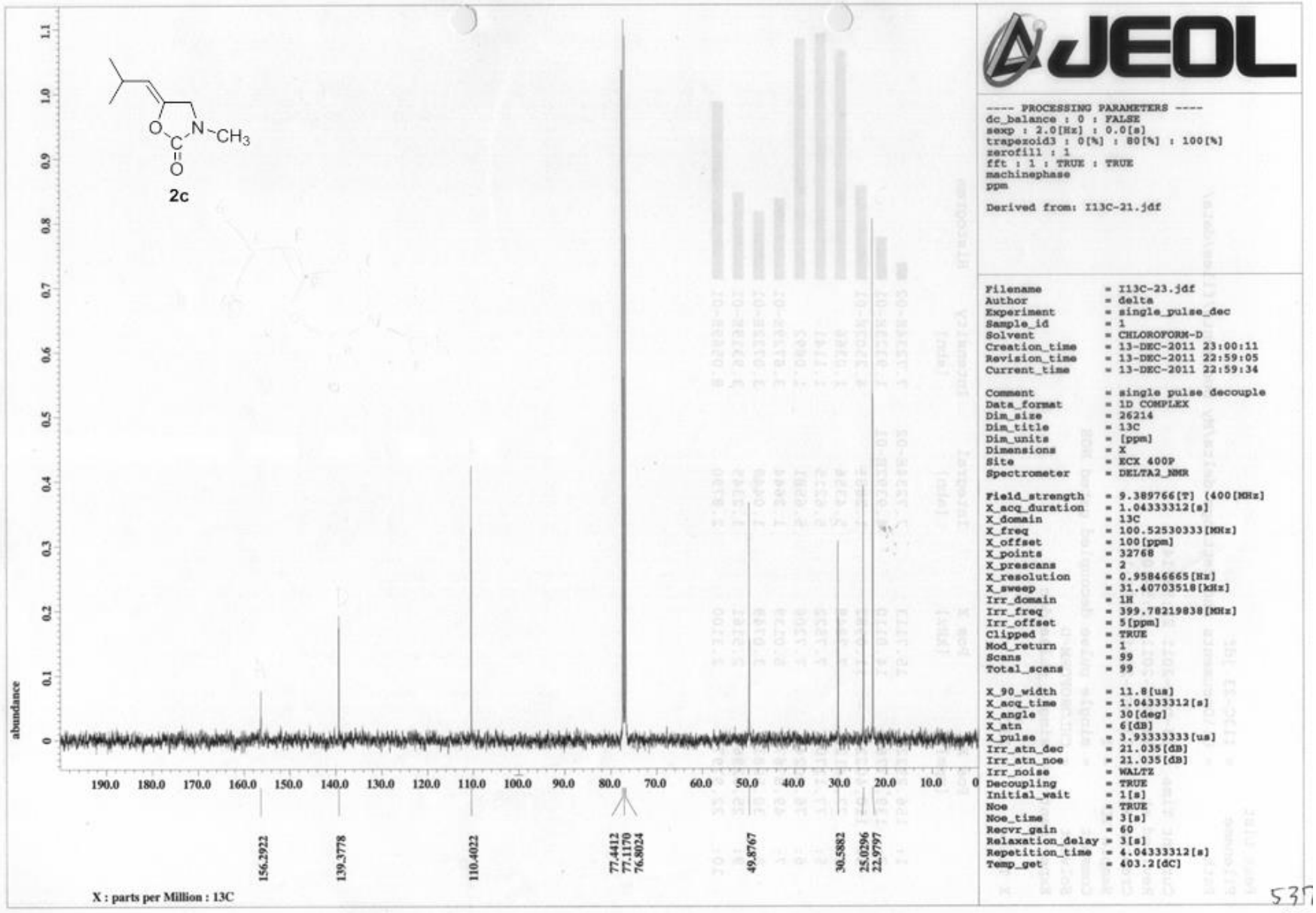

JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: I13C-21.jdf
```

Filename	= I13C-23.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 13-DEC-2011 23:00:11
Revision_time	= 13-DEC-2011 22:59:05
Current_time	= 13-DEC-2011 22:59:34
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz]
X_acq_duration	= 1.04333312[s]
X_domain	= 13C
X_freq	= 100.52530333[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 2
X_resolution	= 0.95846665[Hz]
X_sweep	= 31.40703518[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Clipped	= TRUE
Mod_return	= 1
Scans	= 99
Total_scans	= 99
X_90_width	= 11.8[us]
X_acq_time	= 1.04333312[s]
X_angle	= 30[deg]
X_atn	= 6[dB]
X_pulse	= 3.93333333[us]
Irr_atn_dec	= 21.035[dB]
Irr_atn_noe	= 21.035[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 3[s]
Recvr_gain	= 60
Relaxation_delay	= 3[s]
Repetition_time	= 4.04333312[s]
Temp_get	= 403.2[dC]

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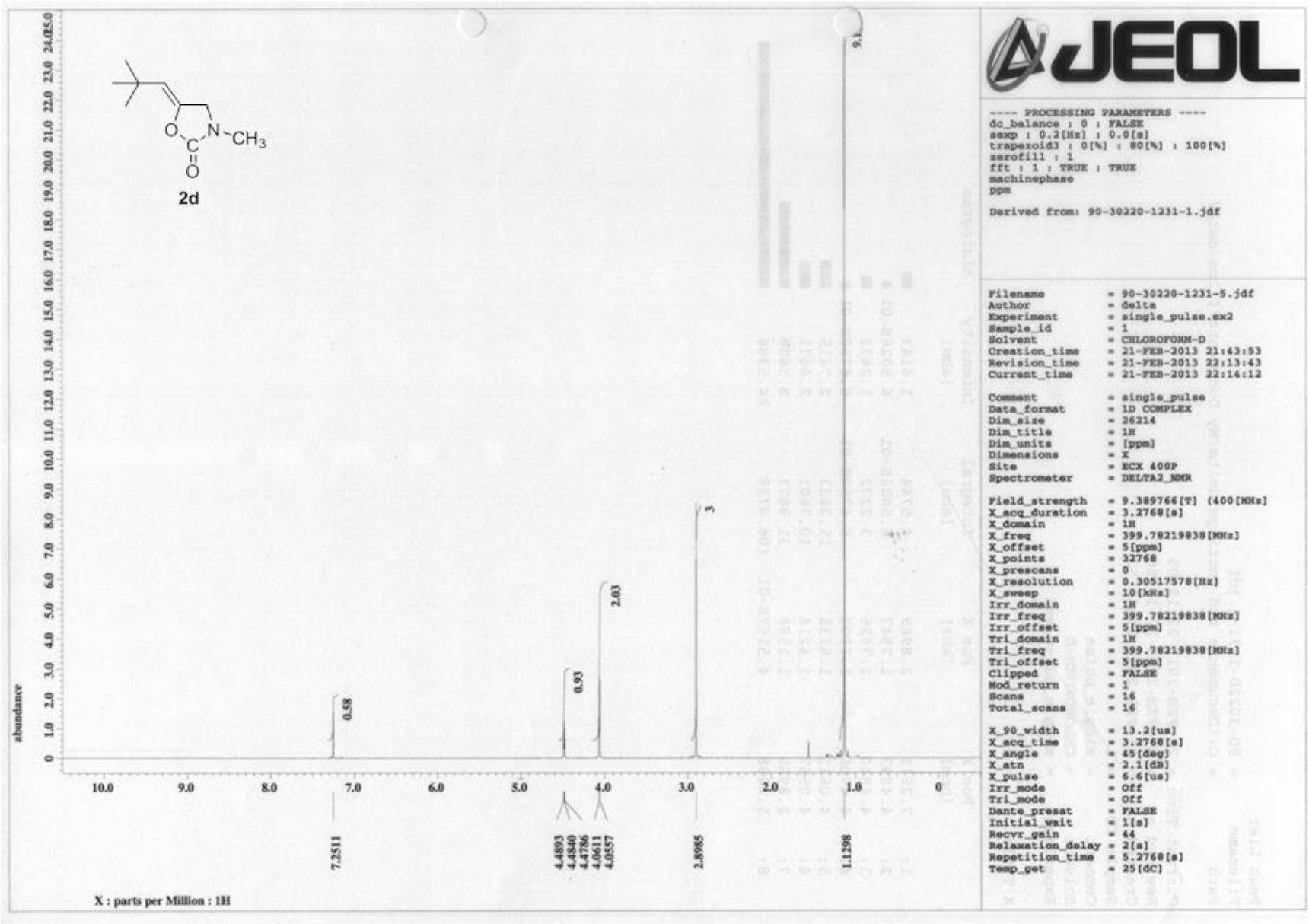
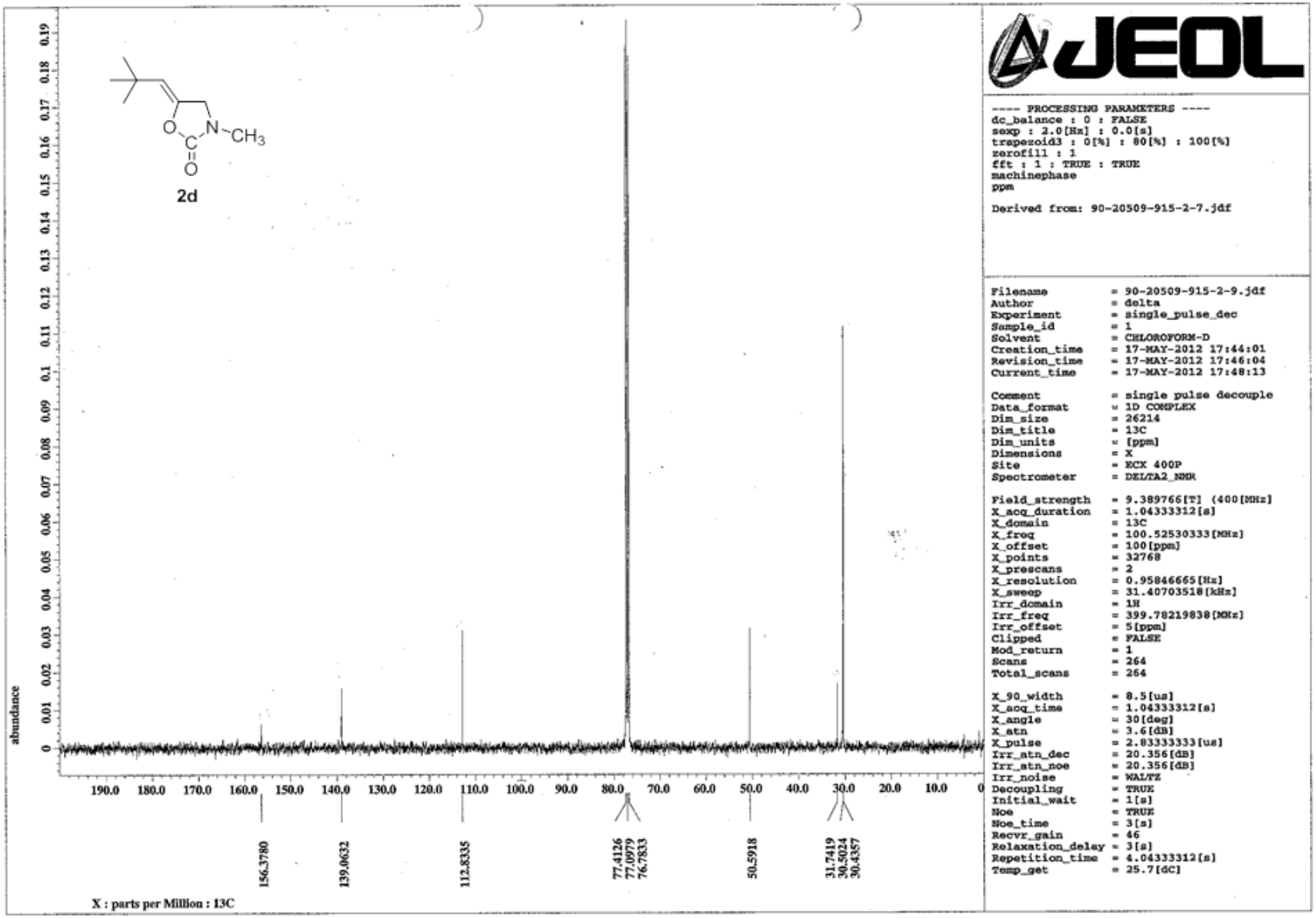
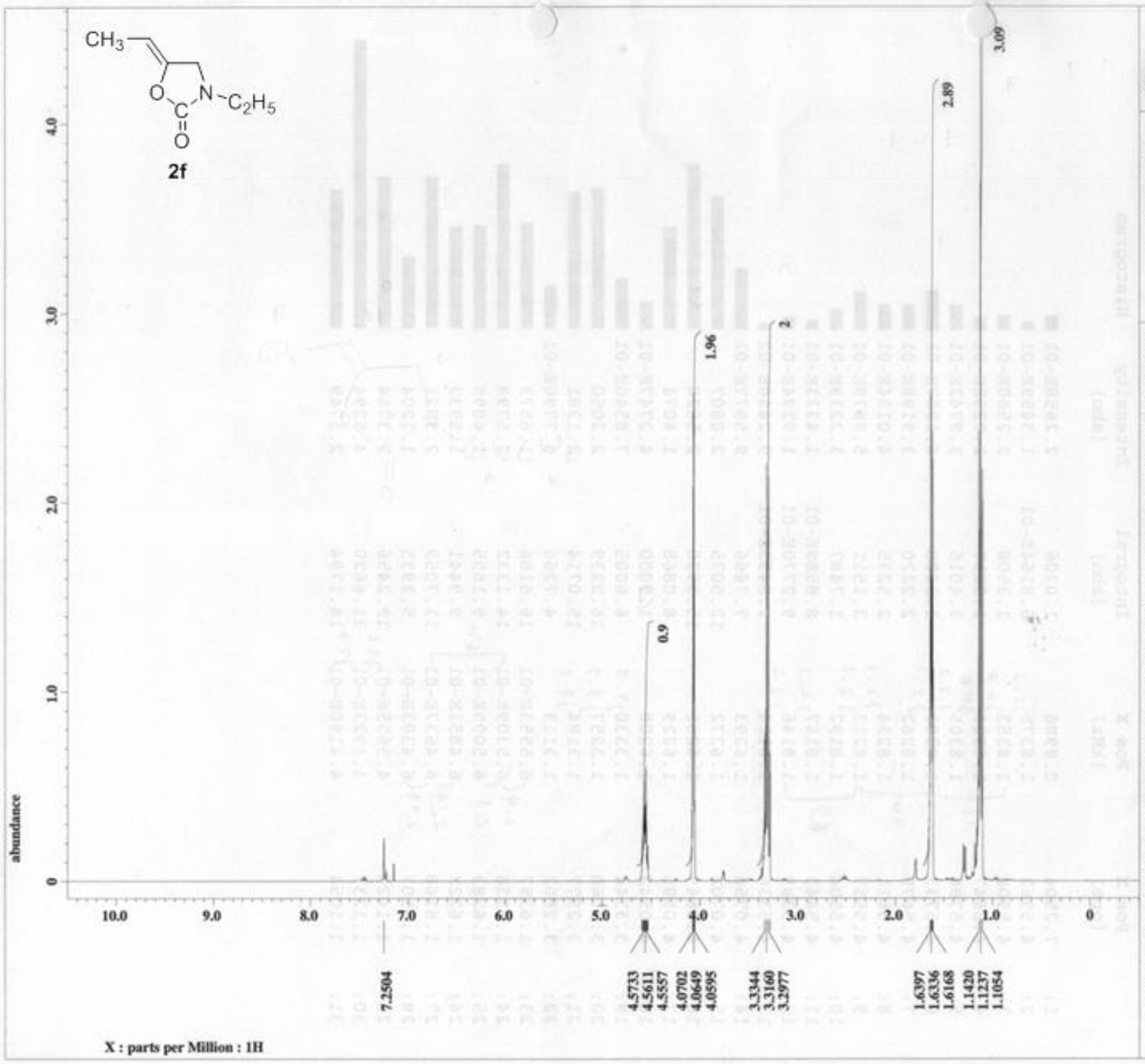

2d
abundance
0.58
0.93
2.03
3
9.1
7.2511
4.4893
4.4840
4.4786
4.0611
4.0557
2.8985
1.1298
X : parts per Million : 1H
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: 90-30220-1231-1.jdf
Filename = 90-30220-1231-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 21-FEB-2013 21:43:53
Revision_time = 21-FEB-2013 22:13:43
Current_time = 21-FEB-2013 22:14:12
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 3.2760[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 5[ppm]
X_points = 32768
X_prescans = 0
X_resolution = 0.30517578[Hz]
X_sweep = 10[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 399.78219838[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 13.2[us]
X_acq_time = 3.2768[s]
X_angle = 45[deg]
X_atn = 2.1[dB]
X_pulse = 6.6[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 2[s]
Repetition_time = 5.2768[s]
Temp_get = 25[dC]

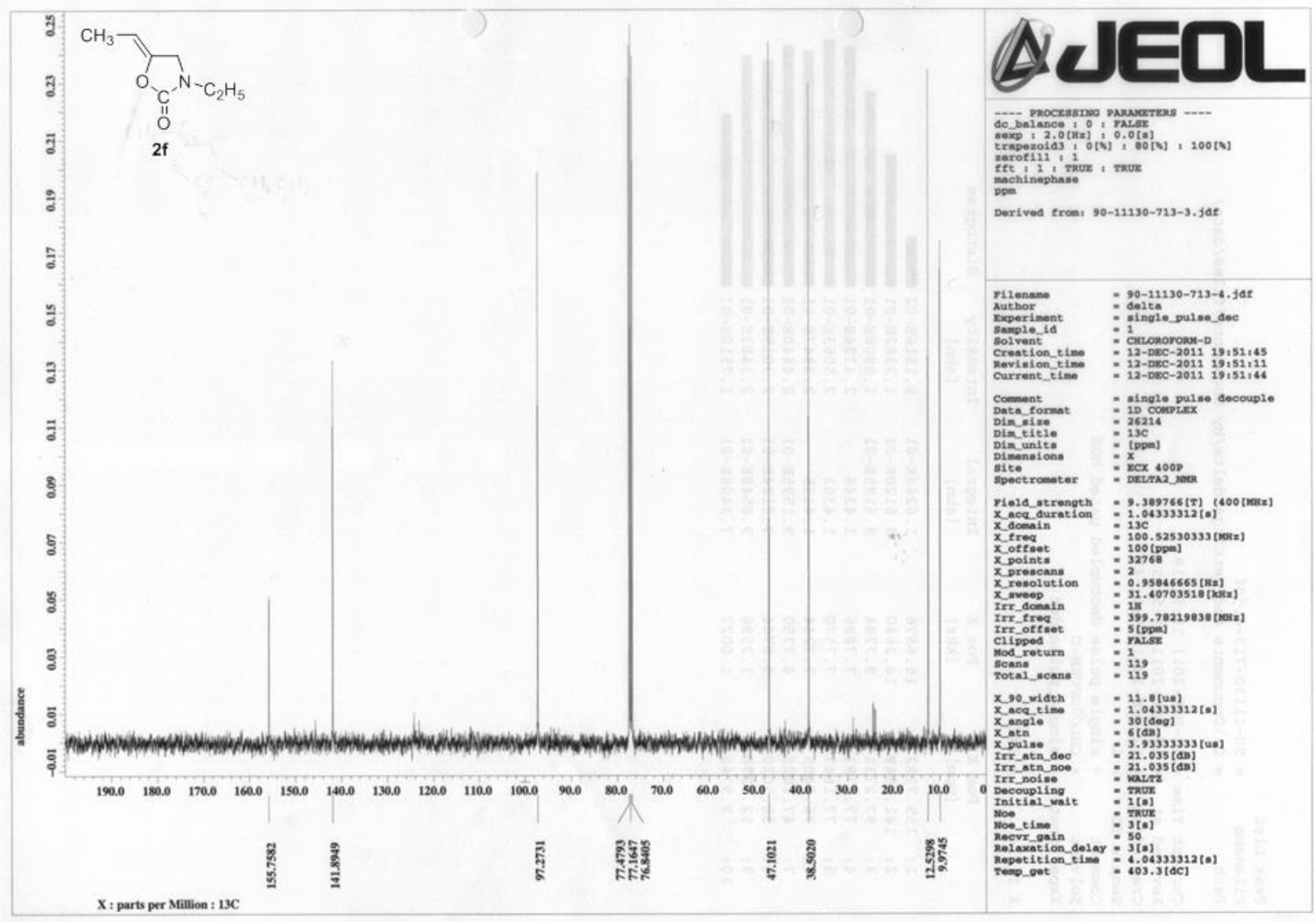

2d
abundance
X : parts per Million : 13C
156.3780
139.0632
112.8335
77.4126
77.0979
76.7833
50.5918
31.7419
30.5024
30.4357
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: 90-20509-915-2-7.jdf
Filename = 90-20509-915-2-9.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 17-MAY-2012 17:44:01
Revision_time = 17-MAY-2012 17:46:04
Current_time = 17-MAY-2012 17:48:13
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 2
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 264
Total_scans = 264
X_90_width = 8.5[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 3.6[dB]
X_pulse = 2.83333333[us]
Irr_atn_dec = 20.356[dB]
Irr_atn_noe = 20.356[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 46
Relaxation_delay = 3[s]
Repetition_time = 4.04333312[s]
Temp_get = 25.7[dC]

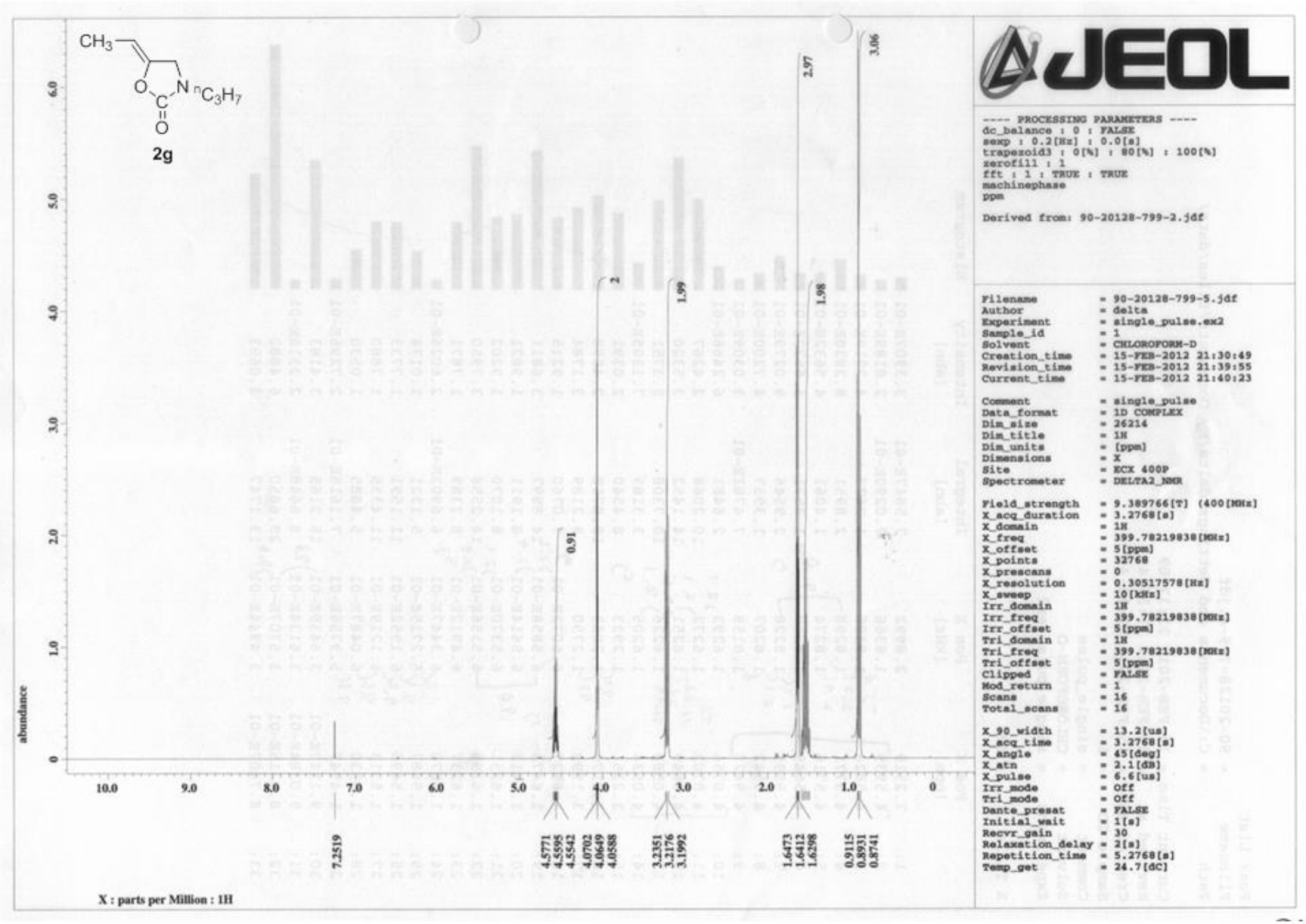


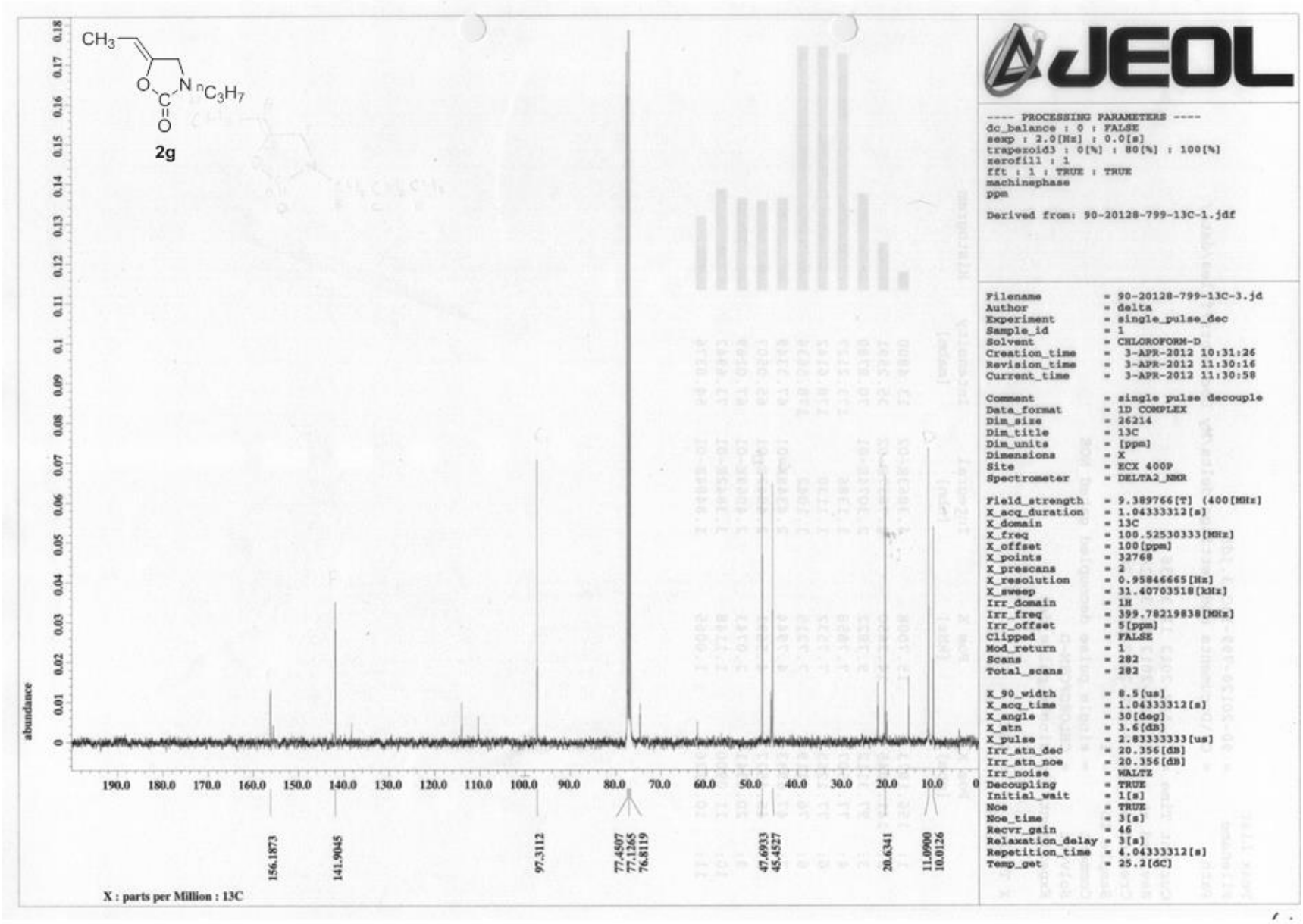
JEOL

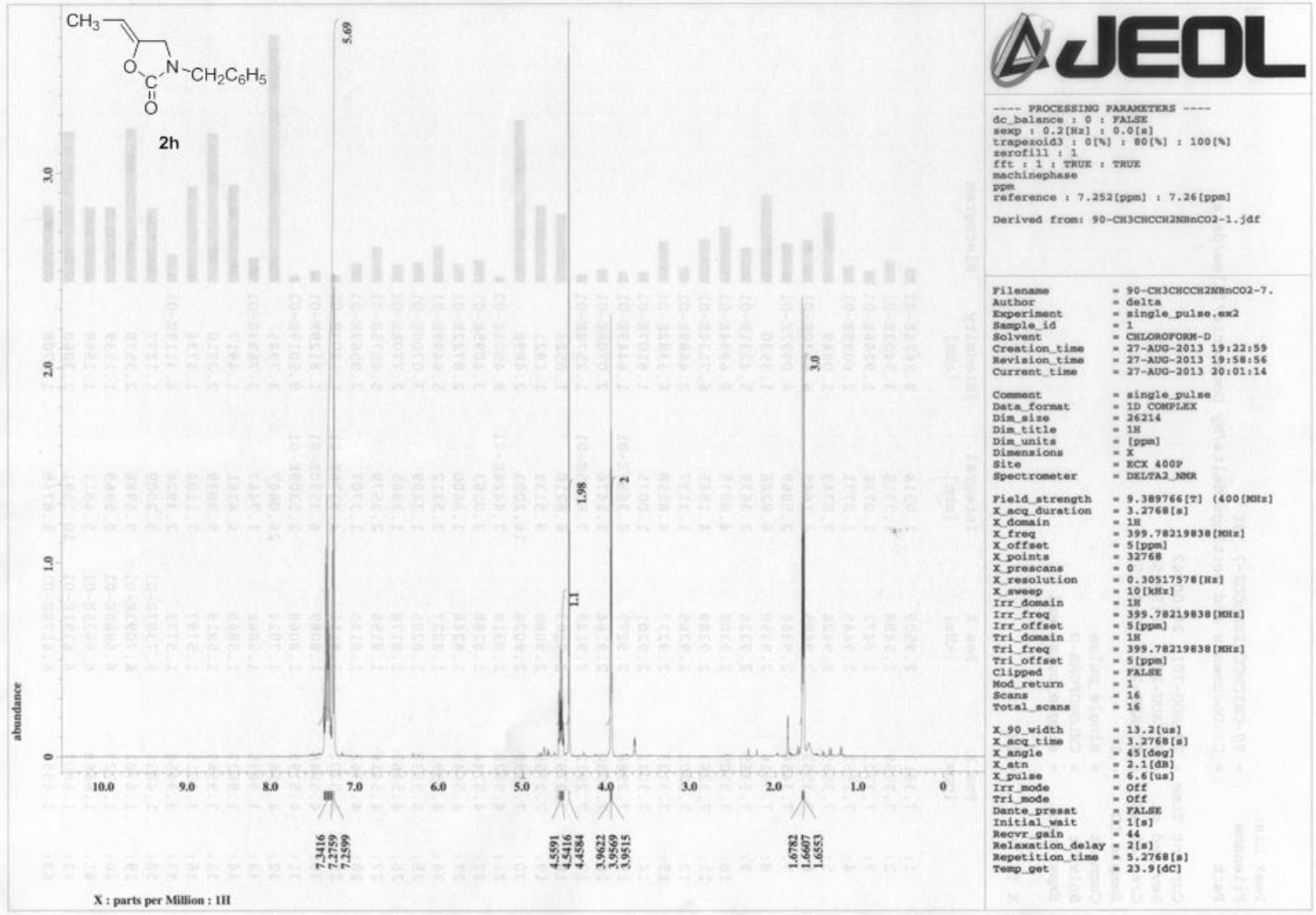
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: 90-11130-713-2.jdf
```

```
Filename          = 90-11130-713-7.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 12-DEC-2011 19:37:46
Revision_time     = 12-DEC-2011 19:46:07
Current_time      = 12-DEC-2011 19:46:26

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 3.2768[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 32768
X_prescans        = 0
X_resolution      = 0.30517578[Hz]
X_sweep           = 10[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 14[us]
X_acq_time        = 3.2768[s]
X_angle           = 45[deg]
X_atn             = 2.1[dB]
X_pulse           = 7[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 30
Relaxation_delay  = 2[s]
Repetition_time   = 5.2768[s]
Temp_get          = 403.3[dC]
```


CH3
O
N
C2H5
C
O
2f
abundance
0.25
0.23
0.21
0.19
0.17
0.15
0.13
0.11
0.09
0.07
0.05
0.03
0.01
-0.01
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
155.7582
141.8949
97.2731
77.4793
77.1647
76.8405
47.1021
38.5020
12.5298
9.9745
X : parts per Million : 13C
JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: 90-11130-713-3.jdf
Filename = 90-11130-713-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 12-DEC-2011 19:51:45
Revision_time = 12-DEC-2011 19:51:11
Current_time = 12-DEC-2011 19:51:44
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 2
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 119
Total_scans = 119
X_90_width = 11.8[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.93333333[us]
Irr_atn_dec = 21.035[dB]
Irr_atn_noe = 21.035[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 4.04333312[s]
Temp_get = 403.3[dC]


CH3
O
N
nC3H7
C
O
2g
abundance
X : parts per Million : 1H
2.97
3.06
2
1.99
1.98
0.91
7.2519
4.5771
4.5595
4.5542
4.0702
4.0649
4.0588
3.2351
3.2176
3.1992
1.6473
1.6412
1.6298
0.9115
0.8931
0.8741
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: 90-20128-799-2.jdf
Filename = 90-20128-799-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 15-FEB-2012 21:30:49
Revision_time = 15-FEB-2012 21:39:55
Current_time = 15-FEB-2012 21:40:23
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 3.2768[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 5[ppm]
X_points = 32768
X_prescans = 0
X_resolution = 0.30517578[Hz]
X_sweep = 10[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 399.78219838[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 13.2[us]
X_acq_time = 3.2768[s]
X_angle = 45[deg]
X_atn = 2.1[dB]
X_pulse = 6.6[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 30
Relaxation_delay = 2[s]
Repetition_time = 5.2768[s]
Temp_get = 24.7[dC]

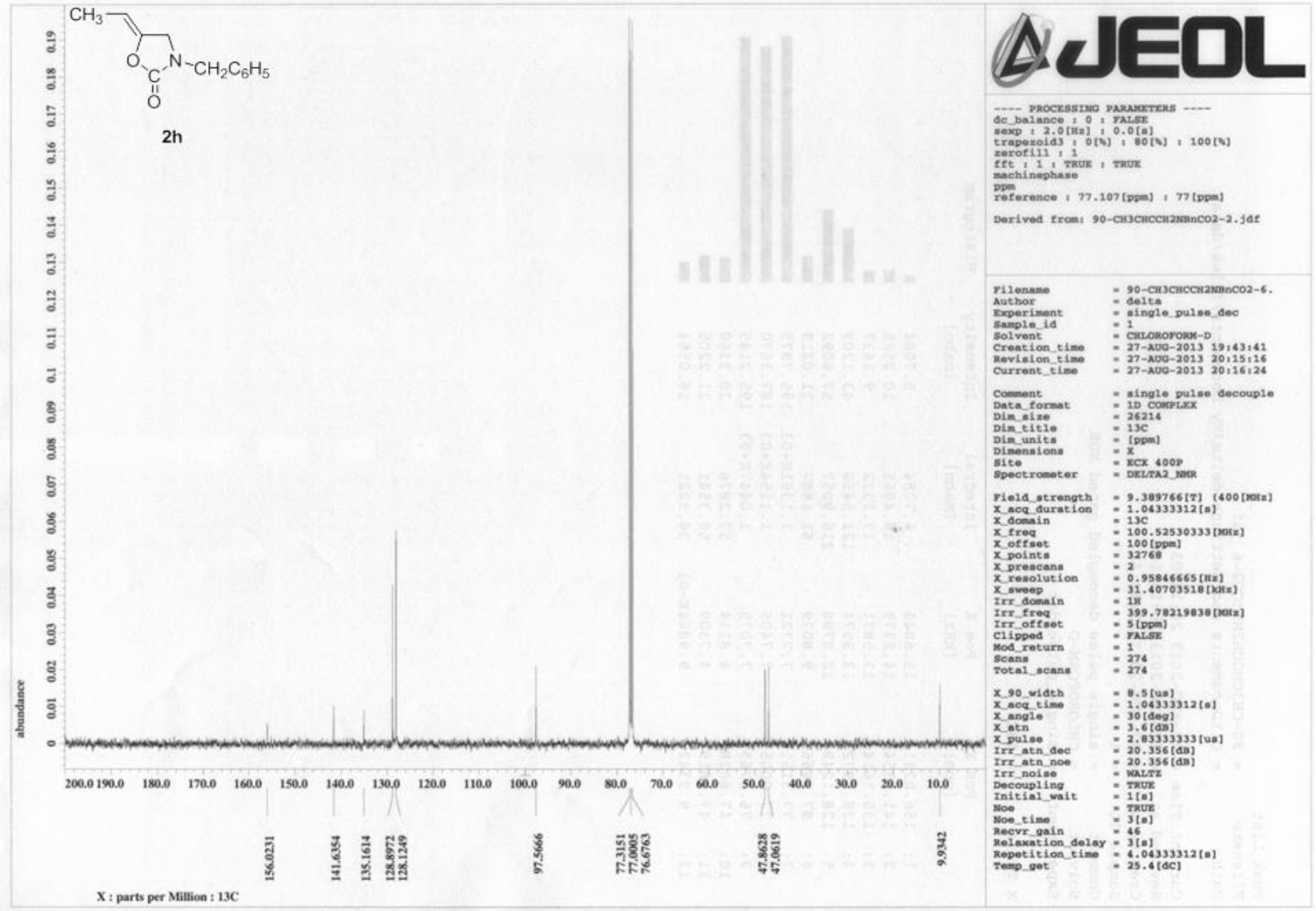

CH3
O
C
O
N
nC3H7
2g
abundance
0.18
0.17
0.16
0.15
0.14
0.13
0.12
0.11
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
156.1873
141.9045
97.3112
77.4507
77.1265
76.8119
47.6933
45.4527
20.6341
11.0900
10.0126
X : parts per Million : 13C
JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: 90-20128-799-13C-1.jdf
Filename = 90-20128-799-13C-3.jd
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 3-APR-2012 10:31:26
Revision_time = 3-APR-2012 11:30:16
Current_time = 3-APR-2012 11:30:58
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 2
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 282
Total_scans = 282
X_90_width = 8.5[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 3.6[dB]
X_pulse = 2.83333333[us]
Irr_atn_dec = 20.356[dB]
Irr_atn_noe = 20.356[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 46
Relaxation_delay = 3[s]
Repetition_time = 4.04333312[s]
Temp_get = 25.2[dC]

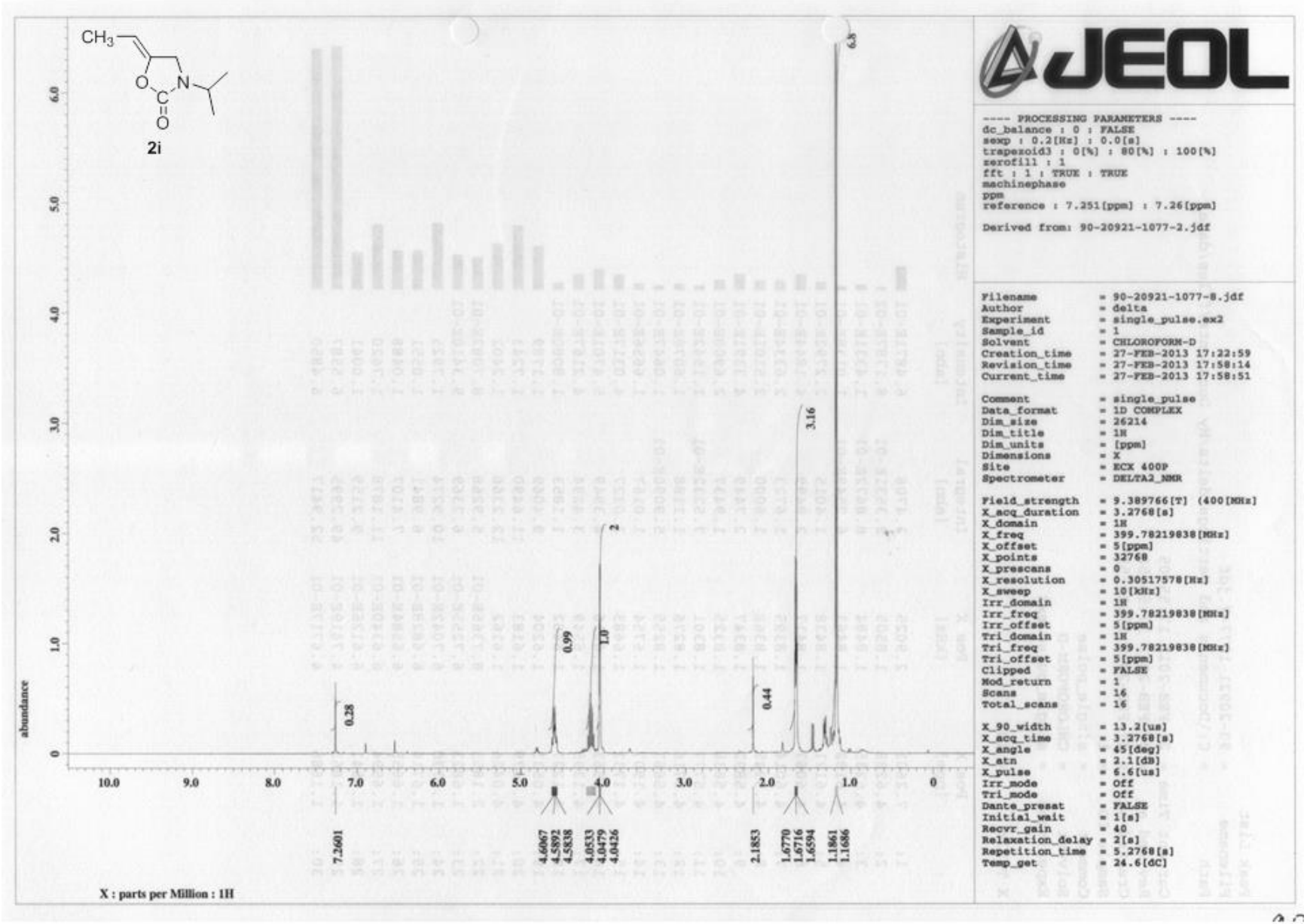


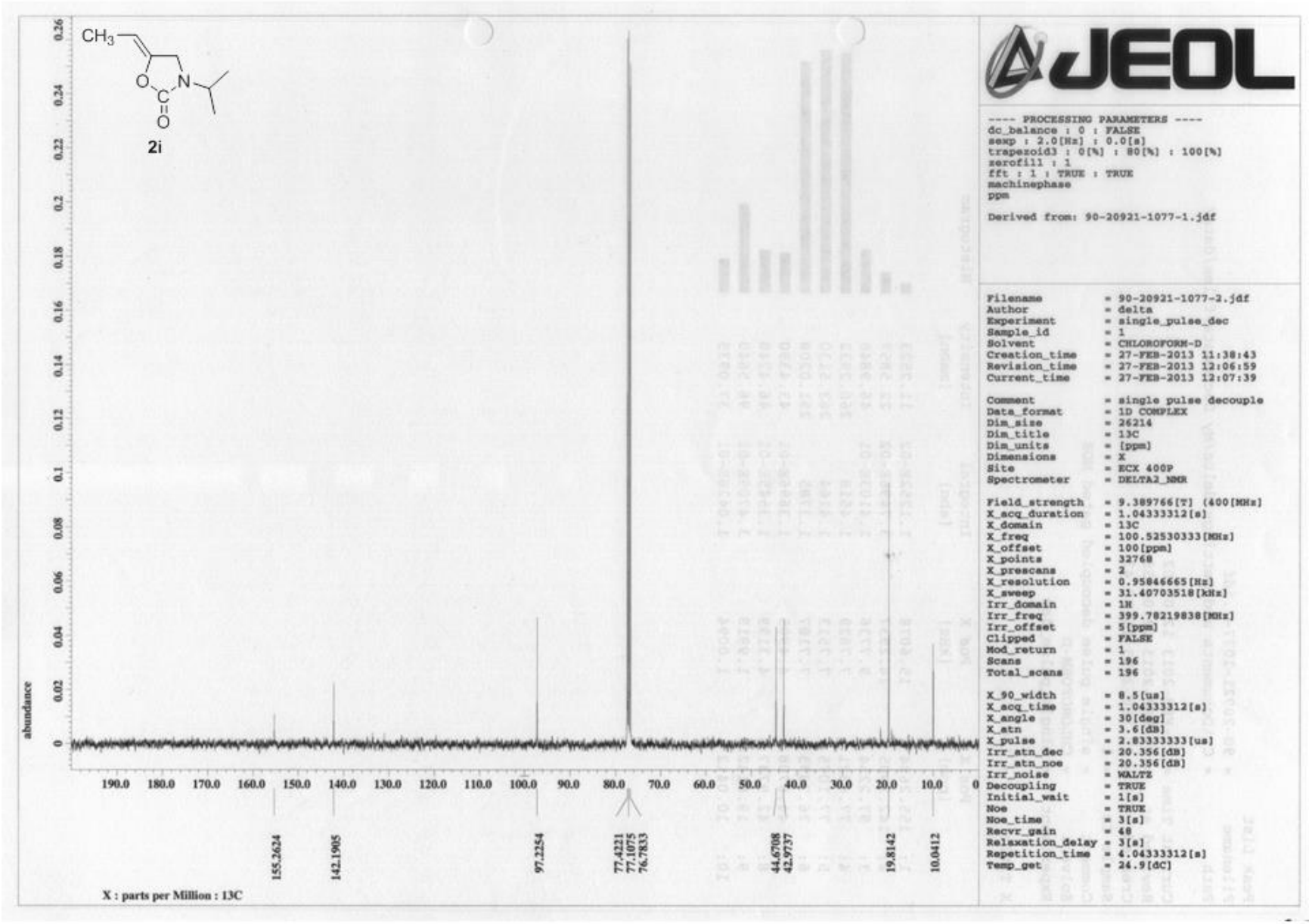
JEOL

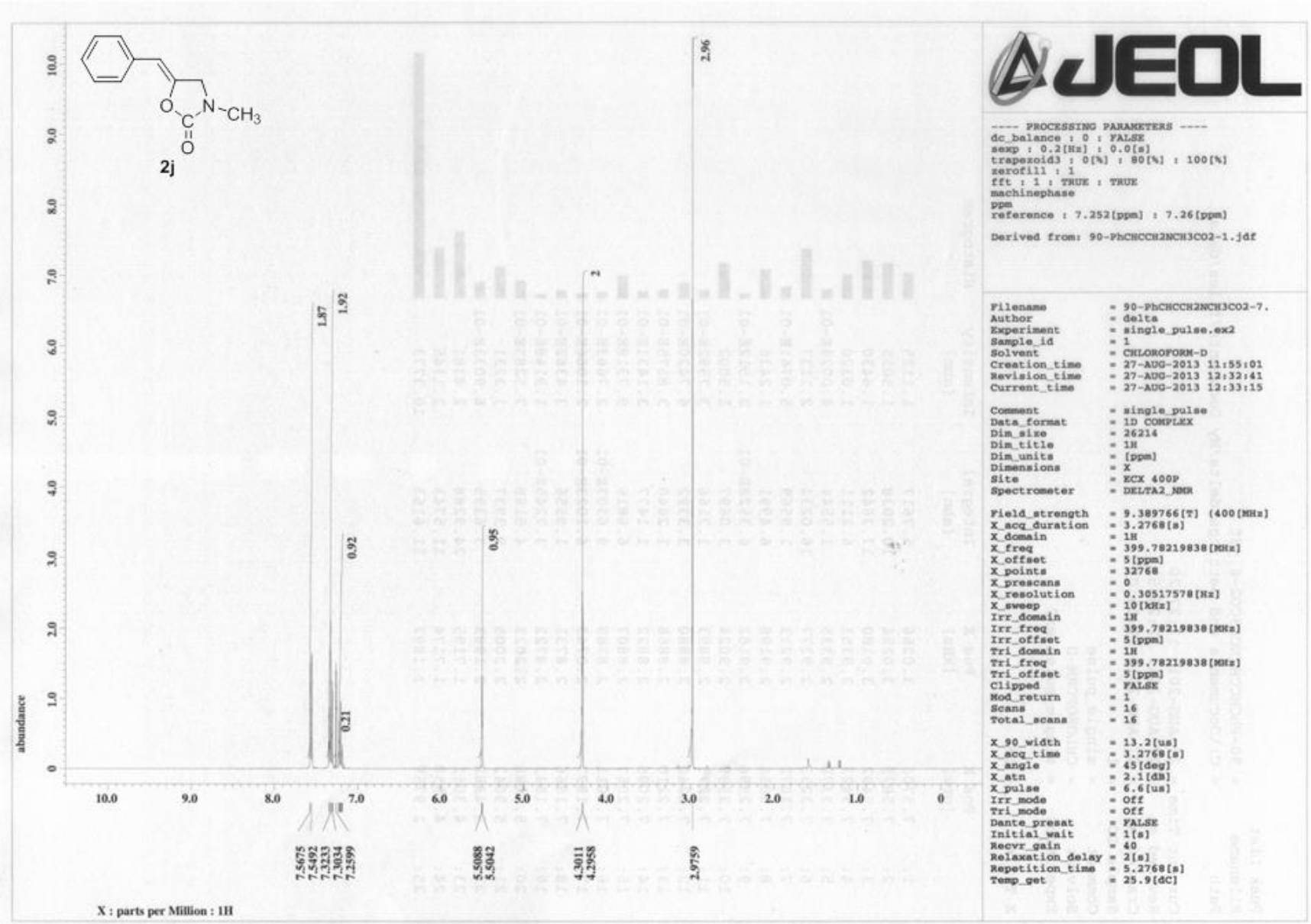
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
reference : 7.252[ppm] : 7.26[ppm]

Derived from: 90-CH3CHCCH2NBnCO2-1.jdf
```

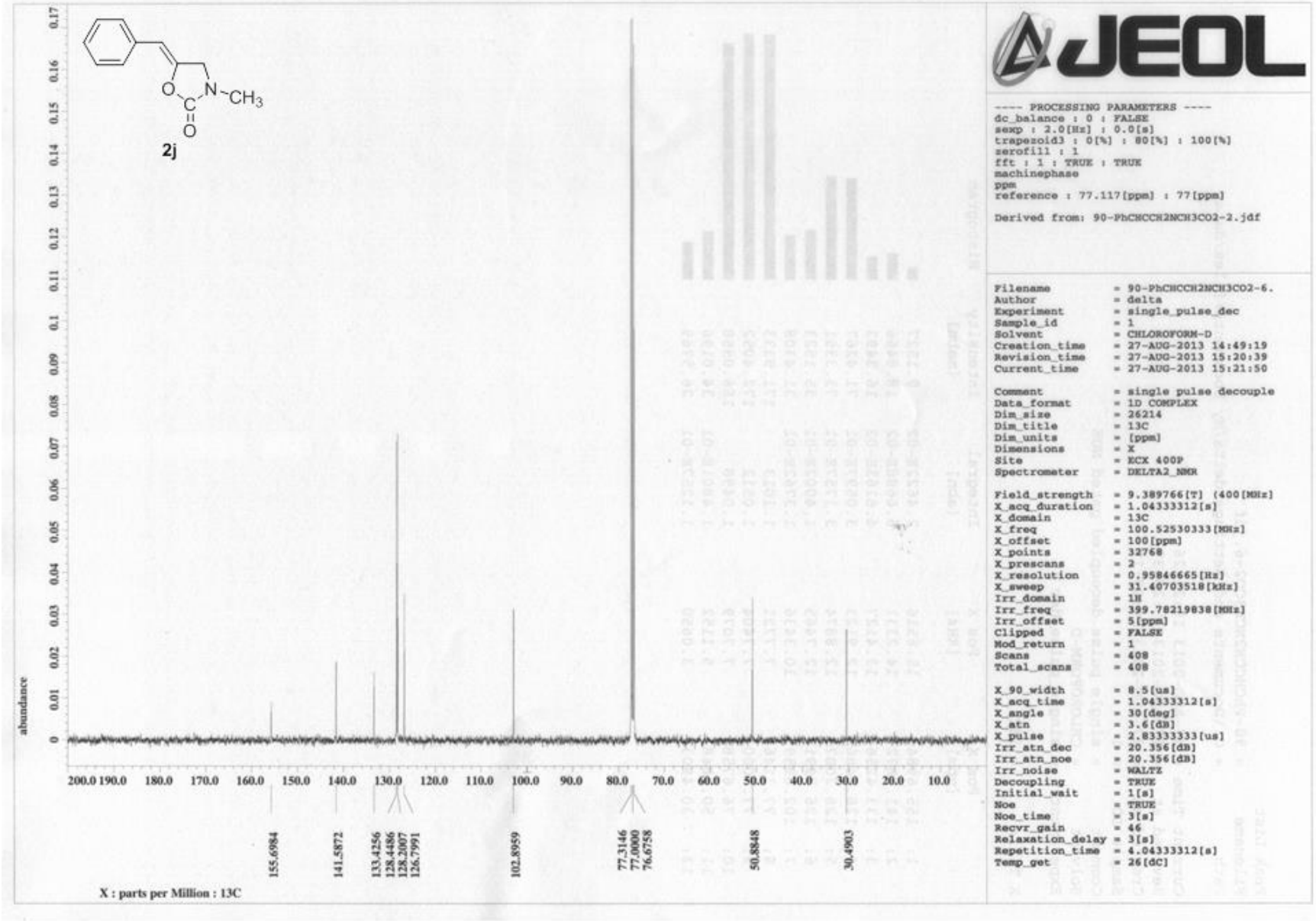
Filename	= 90-CH3CHCCH2NBnCO2-7.
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 27-AUG-2013 19:22:59
Revision_time	= 27-AUG-2013 19:58:56
Current_time	= 27-AUG-2013 20:01:14
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz]
X_acq_duration	= 3.2768[s]
X_domain	= 1H
X_freq	= 399.78219838[MHz]
X_offset	= 5[ppm]
X_points	= 32768
X_prescans	= 0
X_resolution	= 0.30517578[Hz]
X_sweep	= 10[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 399.78219838[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 13.2[us]
X_acq_time	= 3.2768[s]
X_angle	= 45[deg]
X_atn	= 2.1[dB]
X_pulse	= 6.6[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 44
Relaxation_delay	= 2[s]
Repetition_time	= 5.2768[s]
Temp_get	= 23.9[dC]

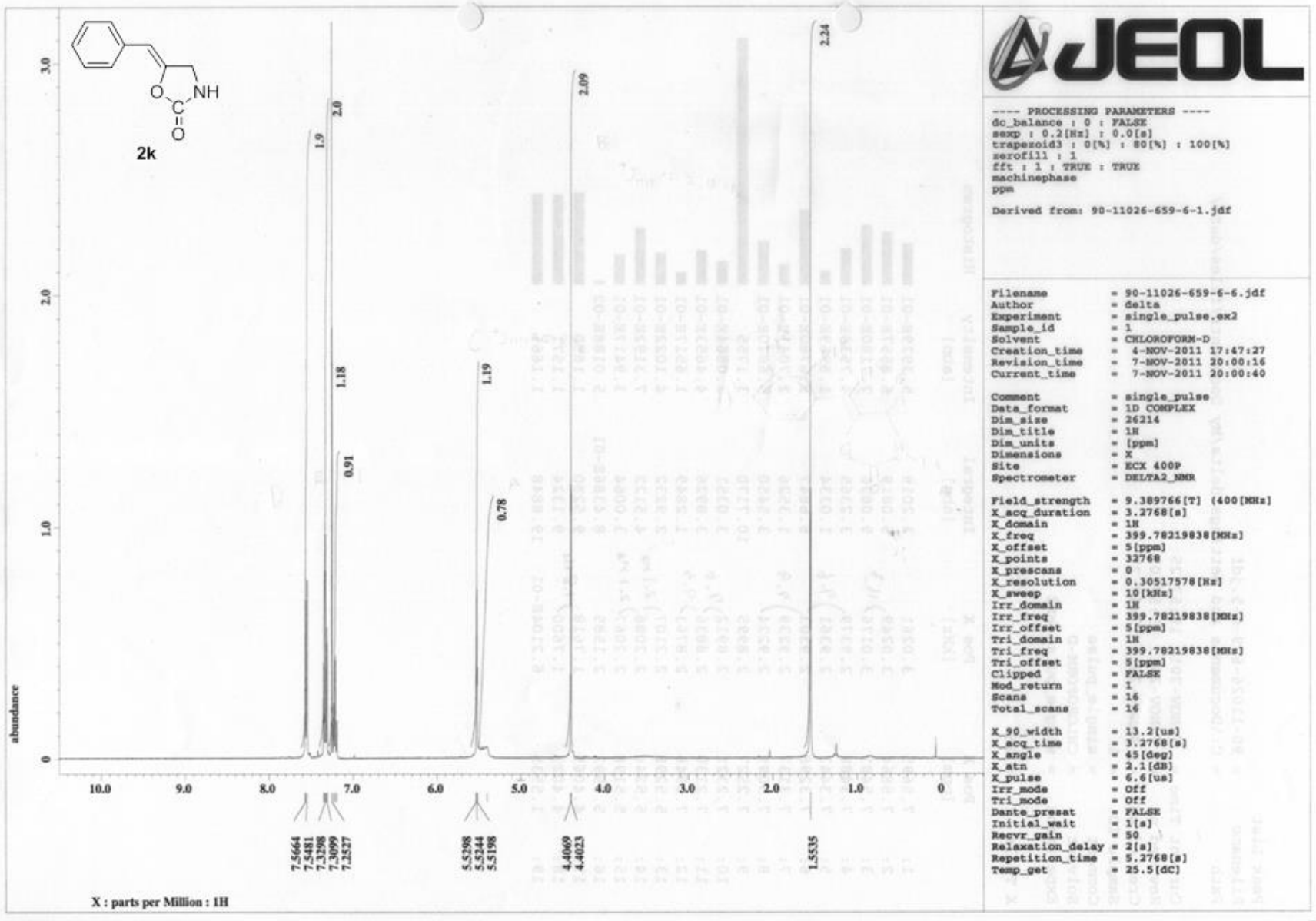

CH3
O
N
CH2C6H5
C
O
2h
abundance
0 0.01 0.02 0.03 0.04 0.05 0.06 0.07 0.08 0.09 0.1 0.11 0.12 0.13 0.14 0.15 0.16 0.17 0.18 0.19
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
156.0231
141.6354
135.1614
128.8972
128.1249
97.5666
77.3151
77.0005
76.6763
47.8628
47.0619
9.9342
X : parts per Million : 13C
JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
reference : 77.107[ppm] : 77[ppm]
Derived from: 90-CH3CHCCH2NBnCO2-2.jdf
Filename = 90-CH3CHCCH2NBnCO2-6.
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 27-AUG-2013 19:43:41
Revision_time = 27-AUG-2013 20:15:16
Current_time = 27-AUG-2013 20:16:24
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 2
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 274
Total_scans = 274
X_90_width = 8.5[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 3.6[dB]
X_pulse = 2.83333333[us]
Irr_atn_dec = 20.356[dB]
Irr_atn_noe = 20.356[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 46
Relaxation_delay = 3[s]
Repetition_time = 4.04333312[s]
Temp_get = 25.4[dC]

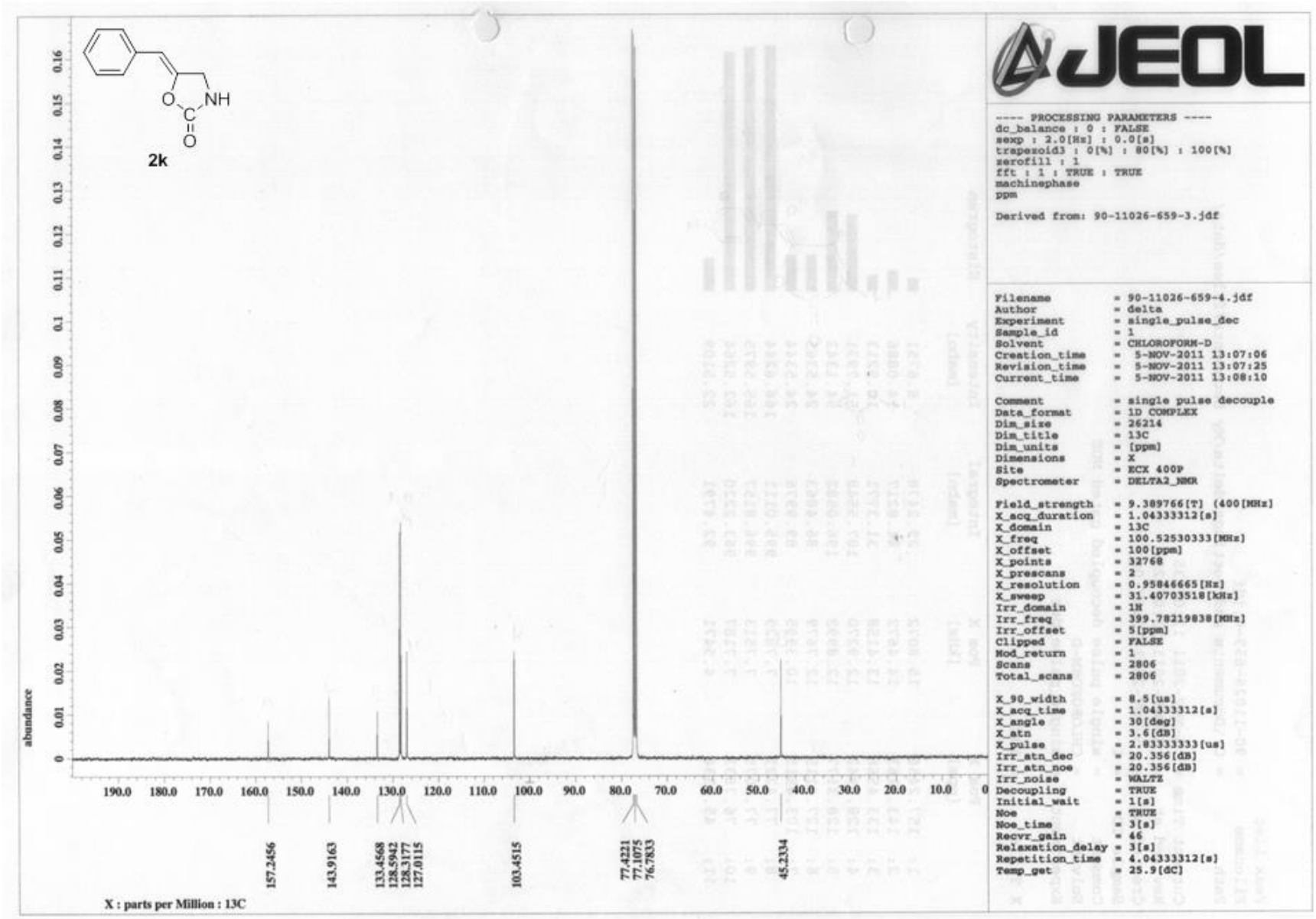

CH3
O
N
C
O
2i
abundance
X : parts per Million : 1H
7.2601
4.6067
4.5892
4.5838
4.0533
4.0479
4.0426
2.1853
1.6770
1.6716
1.6594
1.1861
1.1686
0.28
0.99
1.0
2
0.44
3.16
6.8
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
reference : 7.251[ppm] : 7.26[ppm]
Derived from: 90-20921-1077-2.jdf
Filename = 90-20921-1077-8.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 27-FEB-2013 17:22:59
Revision_time = 27-FEB-2013 17:58:14
Current_time = 27-FEB-2013 17:58:51
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 3.2768[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 5[ppm]
X_points = 32768
X_prescans = 0
X_resolution = 0.30517578[Hz]
X_sweep = 10[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 399.78219838[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 13.2[us]
X_acq_time = 3.2768[s]
X_angle = 45[deg]
X_atn = 2.1[dB]
X_pulse = 6.6[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 2[s]
Repetition_time = 5.2768[s]
Temp_get = 24.6[dC]

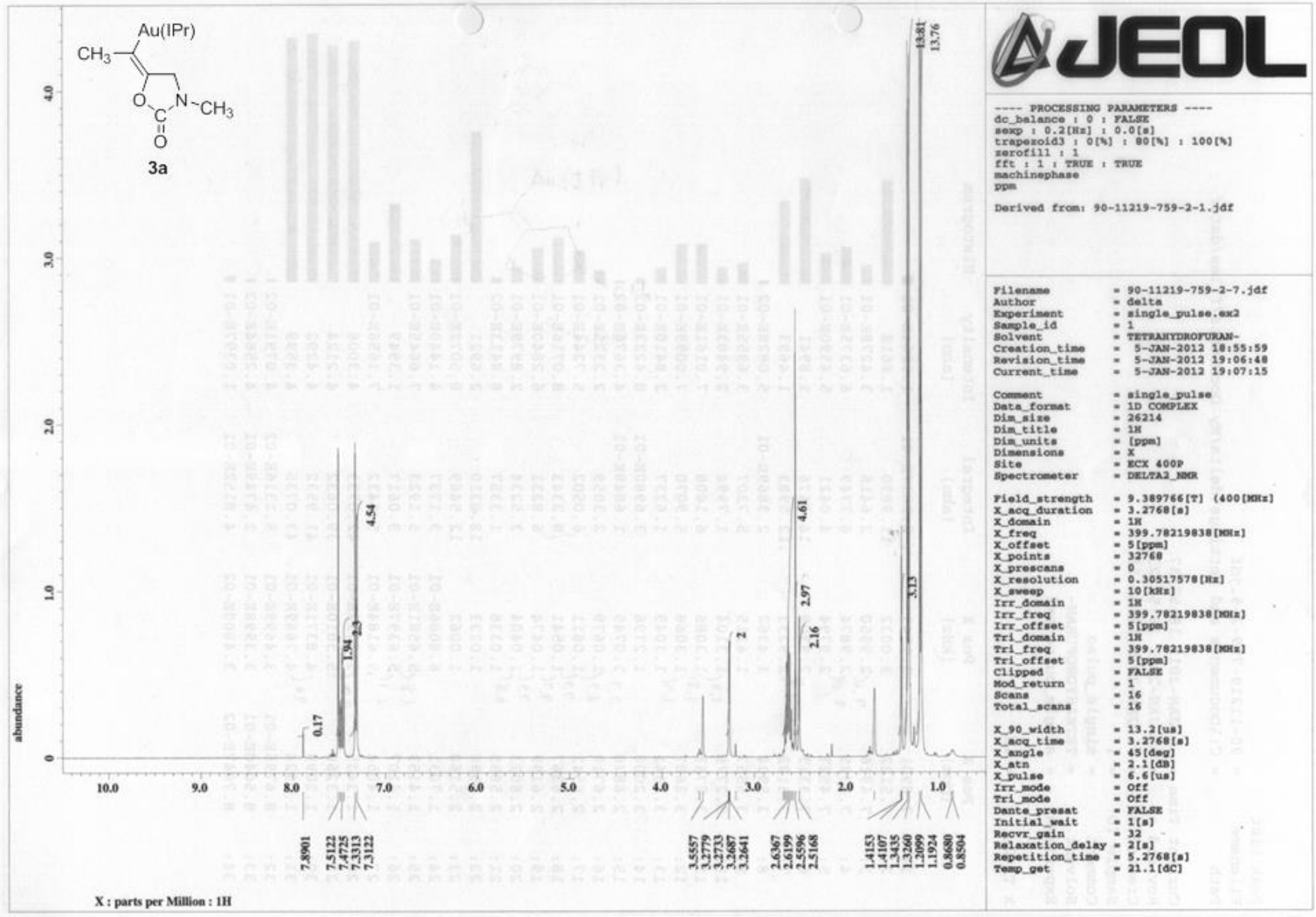

CH3
O
N
C
O
2i
abundance
0.26
0.24
0.22
0.2
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
155.2624
142.1905
97.2254
77.4221
77.1075
76.7833
44.6708
42.9737
19.8142
10.0412
X : parts per Million : 13C
JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: 90-20921-1077-1.jdf
Filename = 90-20921-1077-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 27-FEB-2013 11:38:43
Revision_time = 27-FEB-2013 12:06:59
Current_time = 27-FEB-2013 12:07:39
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 2
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 196
Total_scans = 196
X_90_width = 8.5[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 3.6[dB]
X_pulse = 2.83333333[us]
Irr_atn_dec = 20.356[dB]
Irr_atn_noe = 20.356[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 48
Relaxation_delay = 3[s]
Repetition_time = 4.04333312[s]
Temp_get = 24.9[dC]

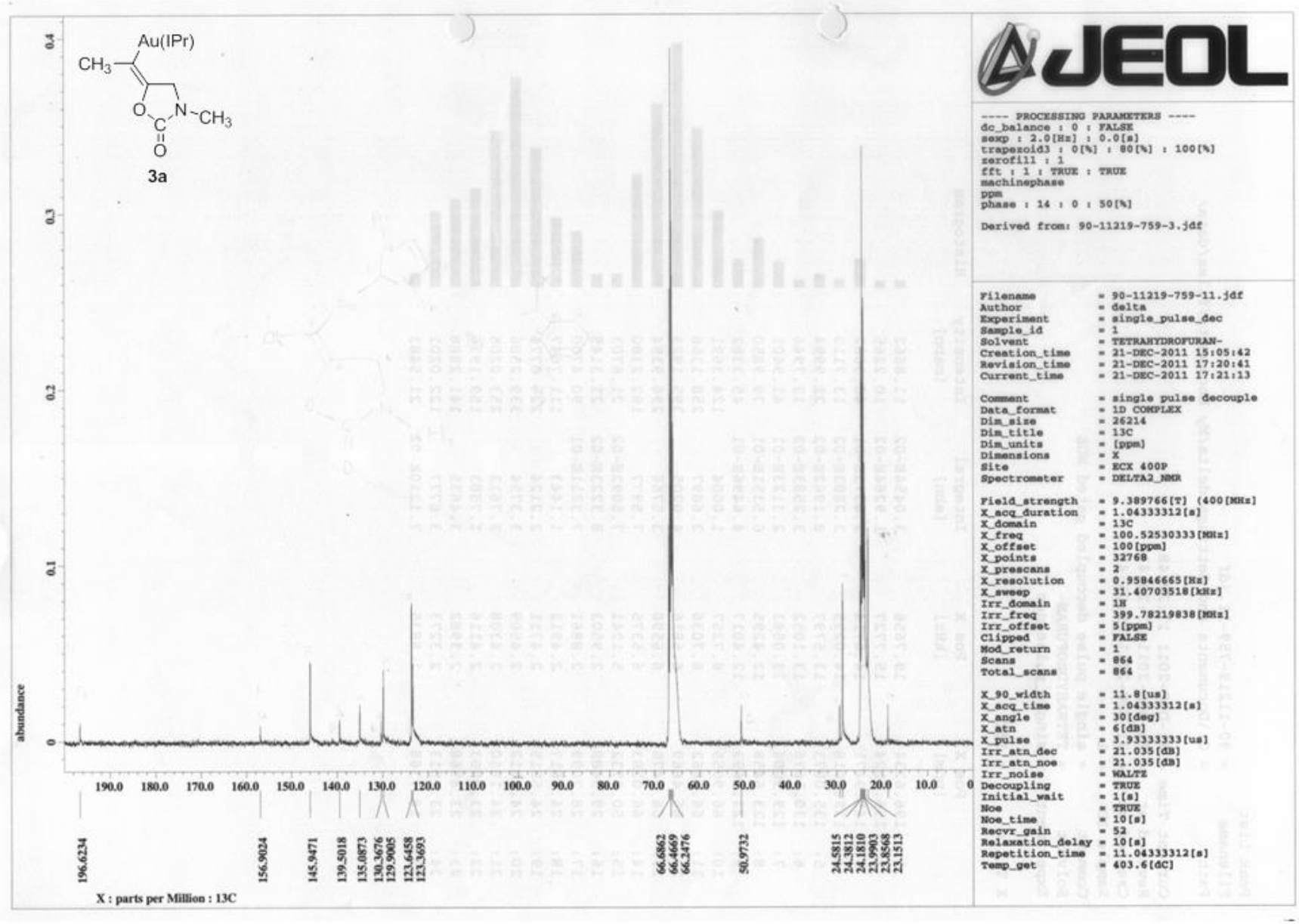

2j
abundance
X : parts per Million : 1H
1.87
1.92
0.92
0.95
0.21
2.96
2
7.5675
7.5492
7.3233
7.3034
7.2599
5.5088
5.5042
4.3011
4.2958
2.9759
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
reference : 7.252[ppm] : 7.26[ppm]
Derived from: 90-PhCHCCH2NCH3CO2-1.jdf
Filename = 90-PhCHCCH2NCH3CO2-7.
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 27-AUG-2013 11:55:01
Revision_time = 27-AUG-2013 12:32:41
Current_time = 27-AUG-2013 12:33:15
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 3.2768[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 5[ppm]
X_points = 32768
X_prescans = 0
X_resolution = 0.30517578[Hz]
X_sweep = 10[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 399.78219838[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 13.2[us]
X_acq_time = 3.2768[s]
X_angle = 45[deg]
X_atn = 2.1[dB]
X_pulse = 6.6[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 2[s]
Repetition_time = 5.2768[s]
Temp_get = 25.9[dC]


2j
abundance
X : parts per Million : 13C
155.6984
141.5872
133.4256
128.4486
128.2007
126.7991
102.8959
77.3146
77.0000
76.6758
50.8848
30.4903
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
reference : 77.117[ppm] : 77[ppm]
Derived from: 90-PhCHCCH2NCH3CO2-2.jdf
Filename = 90-PhCHCCH2NCH3CO2-6.
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 27-AUG-2013 14:49:19
Revision_time = 27-AUG-2013 15:20:39
Current_time = 27-AUG-2013 15:21:50
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 2
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 408
Total_scans = 408
X_90_width = 8.5[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 3.6[dB]
X_pulse = 2.83333333[us]
Irr_atn_dec = 20.356[dB]
Irr_atn_noe = 20.356[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 46
Relaxation_delay = 3[s]
Repetition_time = 4.04333312[s]
Temp_get = 26[dC]


2k
abundance
X : parts per Million : 1H
1.9
2.0
1.18
0.91
1.19
0.78
2.09
2.24
7.5664
7.5481
7.3298
7.3099
7.2527
5.5298
5.5244
5.5198
4.4069
4.4023
1.5535
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: 90-11026-659-6-1.jdf
Filename = 90-11026-659-6-6.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 4-NOV-2011 17:47:27
Revision_time = 7-NOV-2011 20:00:16
Current_time = 7-NOV-2011 20:00:40
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 3.2768[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 5[ppm]
X_points = 32768
X_prescans = 0
X_resolution = 0.30517578[Hz]
X_sweep = 10[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 399.78219838[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 13.2[us]
X_acq_time = 3.2768[s]
X_angle = 45[deg]
X_atn = 2.1[dB]
X_pulse = 6.6[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 5.2768[s]
Temp_get = 25.5[dC]


2k
abundance
0
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.1
0.11
0.12
0.13
0.14
0.15
0.16
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
157.2456
143.9163
133.4568
128.5942
128.3177
127.0115
103.4515
77.4221
77.1075
76.7833
45.2334
X : parts per Million : 13C
JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: 90-11026-659-3.jdf
Filename = 90-11026-659-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 5-NOV-2011 13:07:06
Revision_time = 5-NOV-2011 13:07:25
Current_time = 5-NOV-2011 13:08:10
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 2
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 2806
Total_scans = 2806
X_90_width = 8.5[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 3.6[dB]
X_pulse = 2.83333333[us]
Irr_atn_dec = 20.356[dB]
Irr_atn_noe = 20.356[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 46
Relaxation_delay = 3[s]
Repetition_time = 4.04333312[s]
Temp_get = 25.9[dC]


Au(IPr)
CH3
O
C
O
N
CH3
3a
abundance
X : parts per Million : 1H
13.81
13.76
4.61
4.54
3.13
2.97
2.16
2
2.3
1.94
0.17
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: 90-11219-759-2-1.jdf
Filename = 90-11219-759-2-7.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = TETRAHYDROFURAN-
Creation_time = 5-JAN-2012 18:55:59
Revision_time = 5-JAN-2012 19:06:48
Current_time = 5-JAN-2012 19:07:15
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 3.2768[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 5[ppm]
X_points = 32768
X_prescans = 0
X_resolution = 0.30517578[Hz]
X_sweep = 10[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 399.78219838[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 13.2[us]
X_acq_time = 3.2768[s]
X_angle = 45[deg]
X_atn = 2.1[dB]
X_pulse = 6.6[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 32
Relaxation_delay = 2[s]
Repetition_time = 5.2768[s]
Temp_get = 21.1[dC]


Au(IPr)
CH3
O
C
N
CH3
O
3a
abundance
0.4
0.3
0.2
0.1
0
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
196.6234
156.9024
145.9471
139.5018
135.0873
130.3676
129.9005
123.6458
123.3693
66.6862
66.4669
66.2476
50.9732
24.5815
24.3812
24.1810
23.9903
23.8568
23.1513
X : parts per Million : 13C
JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : 14 : 0 : 50[%]
Derived from: 90-11219-759-3.jdf
Filename = 90-11219-759-11.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = TETRAHYDROFURAN-
Creation_time = 21-DEC-2011 15:05:42
Revision_time = 21-DEC-2011 17:20:41
Current_time = 21-DEC-2011 17:21:13
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 2
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 864
Total_scans = 864
X_90_width = 11.8[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.93333333[us]
Irr_atn_dec = 21.035[dB]
Irr_atn_noe = 21.035[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 10[s]
Recvr_gain = 52
Relaxation_delay = 10[s]
Repetition_time = 11.04333312[s]
Temp_get = 403.6[dC]