Electronic Supplementary Information

Development of a Highly Efficient Single-Mode Microwave Applicator with a Resonant Cavity and its Application to Continuous Flow Syntheses

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1. Details of our consideration of a MW boundary condition to find suitable helical tube reactors

In a resonator, the electric field is vibrating in the upper and lower direction. By arranging a reaction tube in the direction parallel to the electric field, the microwave penetrates through the tube and heats a solution. When the permittivity of the solution is much different from that of air, reflection increases and the energy of the MW can scarcely be used to heat the solution. To avoid this problem, the diameter of the reaction tube must be lessened, which forces us to decrease the flow rate to maintain the necessary residence time and makes the large-scale production per unit time difficult. To solve these issues, we have adopted a helical tubular glass reactor, whose helical turn is piled up several times, based on the following consideration of a MW boundary condition: In a helical tube reactor, a solution flows in a direction nearly orthogonal to the electric field. When assuming the whole of the helical tube as a single large circular cylinder, an apparent permittivity of the side face of the cylinder can be brought close to that of air by choosing a suitable interval of the turn of the reactor in accordance to the permittivity of solutions. Thereby, the reflection can be reduced and the inner diameter of the tube can be enlarged.

We have intensively examined heating profiles of some organic solvents by using our MW applicators equipped with helical borosilicate glass tube reactors with different turn intervals and have disclosed widely applicable tube reactors with the inner diameter of 3.6 mm (see: Fig. S2). With this apparatus, the microwave energy was sufficiently supplied to solvents as well as solutions to heat them rapidly. We could finally conduct highly energy-efficient MW-assisted continuous flow syntheses under the conditions of the flow rate up to 20 mL/min and achieved continuous synthesis of products at a scale of 100 g/h or more.

2. General description of the microwave applicator

2.1. Apparatus for MW-assisted flow synthesis

The microwave applicator is consisted of three major apparatus, viz., MW generator & resonant cavity, pumping system and control device, and PC (Fig. S1).

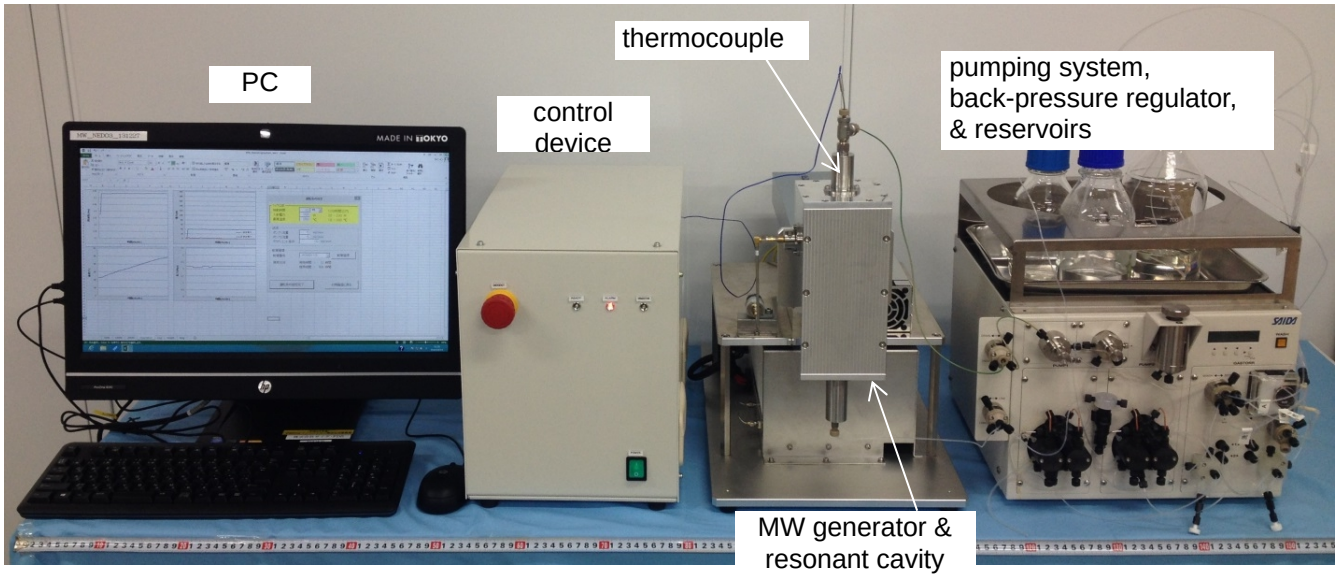


Fig. S1 Entire apparatus for MW-assisted flow synthesis

2.2. MW generator and resonant cavity

2.2.1. MW generator

MW is generated by an oscillator and transmitted through a variable attenuator and a high power amplifier to amplify the MW power to a certain level. All these devices are made of solid-state devices for telecommunication applications. This system produces up to 200 W output in a frequency range covering 2.4 to 2.5 GHz. The MW input power and the reflected power are monitored by power monitors, installed in the generator.

2.2.2. Supply of MW power to the cavity

MW, generated and amplified by the MW generator, is transmitted to a resonant cavity via coaxial cable and waveguide.

2.2.3. Resonant cavity

The resonant cavity focuses the MW power on a custom-designed reactor which is placed along with the central axis. The cavity size is designed to keep the frequency at resonance in between 2.4 and 2.5 GHz while flowing a reaction mixture through a helical tube reactor.

2.2.4. Design of the resonant cavity and the tube reactor

The cavity size, shape of the helical tube reactor, and the placement of the tube reactor in the cavity are well engineered by using computer simulation technology to optimize Q value to generate resonance.

2.3. Pumping system

2.3.1. Design of the pumping system

Most of the components of the pumping system are developed by diverting HPLC devices. We have also developed software to control pumps and to handle signals from detectors linked with controller devices to work as one integrated system. Two units of a double-plunger pump are installed to send reaction solutions into the tube reactor each at the flow rate of up to 10 mL/min under the pressure of up to 20 MPa.

2.3.2. Flow route

A solution of reactant(s) in an organic solvent is supplied from a reservoir and pumped into the helical tube reactor placed in the resonant cavity via a degasser and a pressure gauge. The pressure gauge also works as a mixer when two pumps are used. The reaction mixture comes out from the tube reactor and flows out after passing another pressure gauge and a back-pressure regulator.

2.3.3. Tubing and connection

A thermocouple and an inlet/outlet tube are connected to a stainless steel reducing union and tee. The PEEK inlet tube (o.d. 1/16") from the pump system is connected to the reducing union at bottom of the reactor by a PEEK screw type fitting. A thermocouple (o.d. 1/16") and an outlet tube are connected to the top and side of the tee at the top of the reactor. Both ends of the helical glass tube reactor are welded to the metal tube (o.d. 6 mm) which is resisted to a tight fitting by a cap nut and ferrule. Total swept volume of the entire system is about 8.3 mL, which includes the glass tube reactor volume (7 mL that includes 6 mL inside the resonant cavity and 1.2 mL outside the cavity), pump volume (0.4 mL), and the tubing volume (0.7 mL).

2.3.4. Pressure gauge

Two pressure gauges are installed before and after the tube reactor to detect clogging in the tube reactor, and the pressure difference between these two pressure gauges is always monitored.

2.4. Control system

2.4.1. Software

The programmable logic controller (PLC) produced by Mitsubishi Electric Corporation is used and the control-sequence is developed by the special language on the PLC. Microsoft Excel is used for the interface on the PC, and PLC makes a PC and the equipment cooperate.

2.4.2. Tuning control

System software to automatically tune the MW frequency to the best resonance is installed in the control device. Because the frequency at resonance changes according to dielectric property inside the cavity, which depends on the kind of reagent(s) and solvent(s) as well as their temperature, tuning is periodically made.

2.4.3. MW power control

MW input power is always monitored by a power monitor installed in the MW generator and automatically controlled at a certain level by changing the condition of the attenuator.

2.4.4. Safety Systems

Various kinds of safety systems are installed, which includes the following: the detection of a tube reactor in the cavity, constant monitoring of the pressure and exit temperature of a reaction solution, constant monitoring of the pressure difference between two pressure gauges, and an emergency shutdown system quickly responding to changes of the pressure, temperature, and pressure difference. Three apparatus are linked as network, and the entire system will immediately shut down if one unit detects any system trouble.

2.5. PC Interface

2.5.1. Setting of MW power conditions

All conditions of MW irradiation are set by PC.

2.5.2. Displaying current status and logs

The real time status data about MW irradiation and a reaction solution are transmitted from control devices to PC. These data are shown up in the PC display as graphical format and are also logged in PC. Such data are easily available for reporting as an Excel format.

2.5.3. Recording of error data

An error message is immediately shown up in the PC display, if the system detects any error or trouble. Such error data are automatically recorded in PC and are easily referred for analysis.

2.6. Tube reactors

2.6.1. Tube reactors

The tube reactor is consisted of a helical borosilicate glass tube, metal tubes, fittings and a thermocouple (Fig. S2). The outside diameter and the internal diameter of the glass tube are 6 mm

and 3.6 mm, respectively. The glass tube is coiled up with outside diameter of 20 mm and its internal volume is 5.5–6.0 mL. The glass tube is covered by a heat shrink tube made of polytetrafluoroethylene tubular film to prevent from scattering of glass pieces in case of breakage of the reactor. The tube reactor is easily installed in the resonant cavity from its top (see: Fig. S1) and removed (Fig. S3).

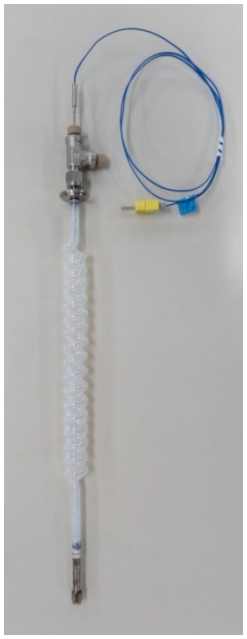


Fig. S2 A tube reactor

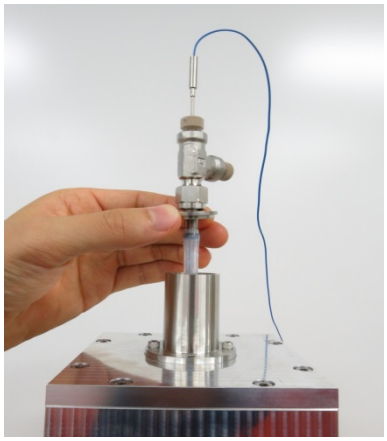


Fig. S3 Installation and removal of the reactor

2.6.2. Safety

As the tube reactor should have long time reliability, an accelerated pressure test is implemented under the conditions of giving pressure from 0.1 MPa to 5 MPa repeatedly over 1,000 times. A software is developed and installed in PC to record history of the usage of a specific tube reactor. Such historical data are easily checked, which will help users to decide when is the right timing to change the reactor to a new one, as the life of tube reactors may be different according to the conditions of reactions.

3. The exit temperature profile of MeCN by using the continuous flow MW apparatus under various conditions

MeCN was pumped into the apparatus at the flow rate of 5 mL/min with the constant irradiation power of 50 W under the back-pressure of 2.5 MPa. The time-course of the exit temperature of MeCN, directly measured by the thermocouple set inside the reactor, after starting the MW irradiation is shown in Fig. S4. The exit temperature reached a steady state at 160 °C after

6 min. By a similar method, the exit temperature, after reaching a steady state, was measured under several conditions changing the irradiation power (50–200 W) and the flow rate (5–20 mL/min) and is summarized in Table S1.

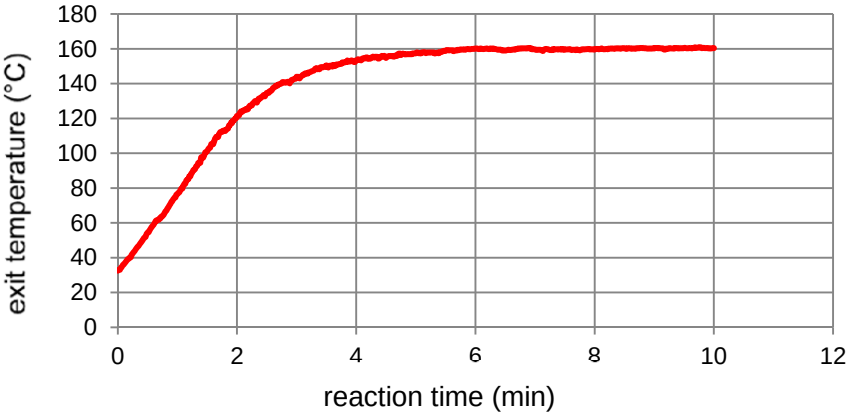


Fig. S4 Time-course of the exit temperature of MeCN after starting the MW irradiation at the flow rate of 5 mL/min with the irradiation power of 50 W.

Table S1 The exit temperature of MeCN under various conditions[a]

irradiation power (W) / flow rate (mL/min) and residence time[b]	50	100	150	200
5 (62 s)	160	–[c]	–[c]	–[c]
10 (31 s)	133	197	–[c]	–[c]
15 (21 s)	112	170	206	231
20 (17 s)	98	151	185	209

[a] MeCN at room temperature was pumped into the reactor at the given flow rate with the given irradiation power of MW under the back-pressure of 2.5 MPa. The exit temperature of MeCN was directly measured by a thermocouple set at the exit of the reactor, and the temperature after reaching a steady state is shown. [b] The residence time in the reactor in the resonant cavity is shown in the parenthesis. [c] Not available due to boiling of MeCN.

4. The surface temperature of the helical tube reactor

The surface temperature of the entire helical tube reactor was directly monitored by a thermal imaging infrared camera through a slit of a specially prepared resonant cavity (Fig. S5a). A tube reactor without the film was employed for more precise analysis of the surface temperature, although a tube reactor, covered with a polytetrafluoroethylene tubular film for safety and insulation reasons, was used for the continuous flow synthesis (Fig. S5b). The temperatures of 15 points of the reactor were measured at once (Fig. S5a) and were linearly corrected so that the temperature of the

15th point corresponded to the temperature directly measure by a thermocouple set inside the reactor‘s exit. Acetonitrile was pumped into the reactor at the given flow rate with the given MW irradiation power, and the temperatures after 10 min, while it reached a steady state, were shown in Figs. S5c–S5f. In all cases, it was found that the temperature continuously increased from the entrance of the reactor up to two thirds of its length and remained almost the same on the last third.

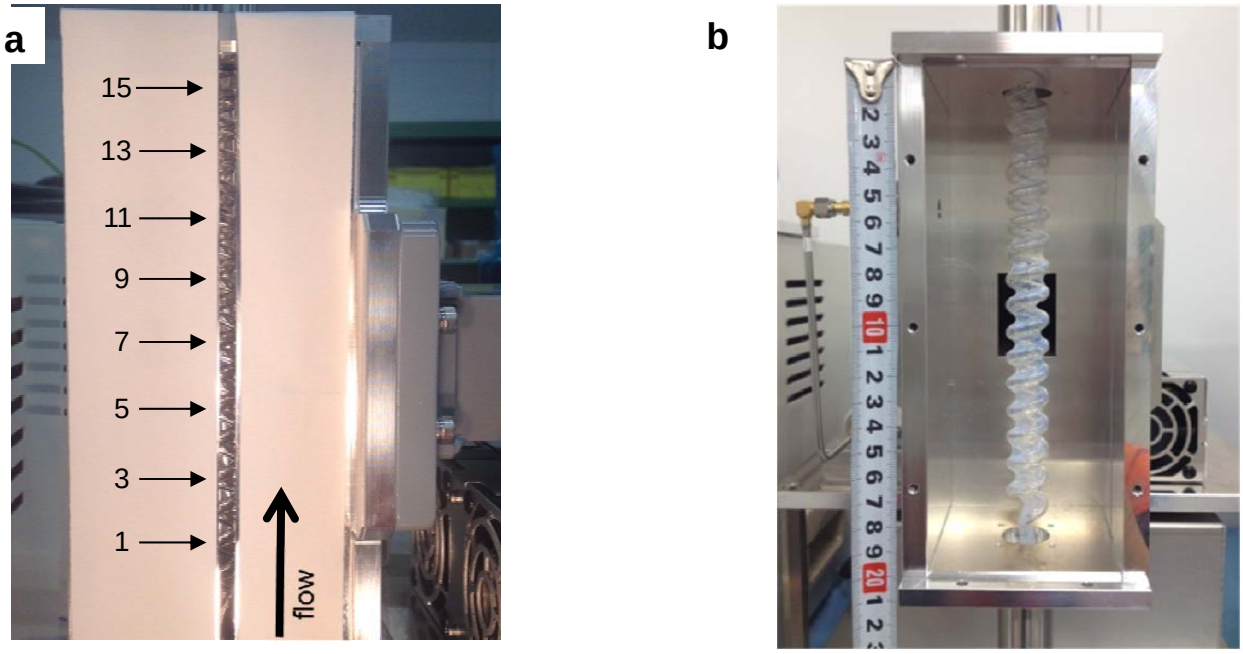


Fig. S5 The temperature of 15 measuring points of the tube reactor, while MeCN was pumped into it at the given flow rate with the given MW irradiation power. (a) The tube reactor set in a specially prepared resonant cavity with a slit. The 15 measuring points are indicated by arrows. (b) The standard helical glass tube reactor for the continuous flow synthesis set in a resonant cavity. The tube is covered with a polytetrafluoroethylene tubular film for safety and insulation reasons.

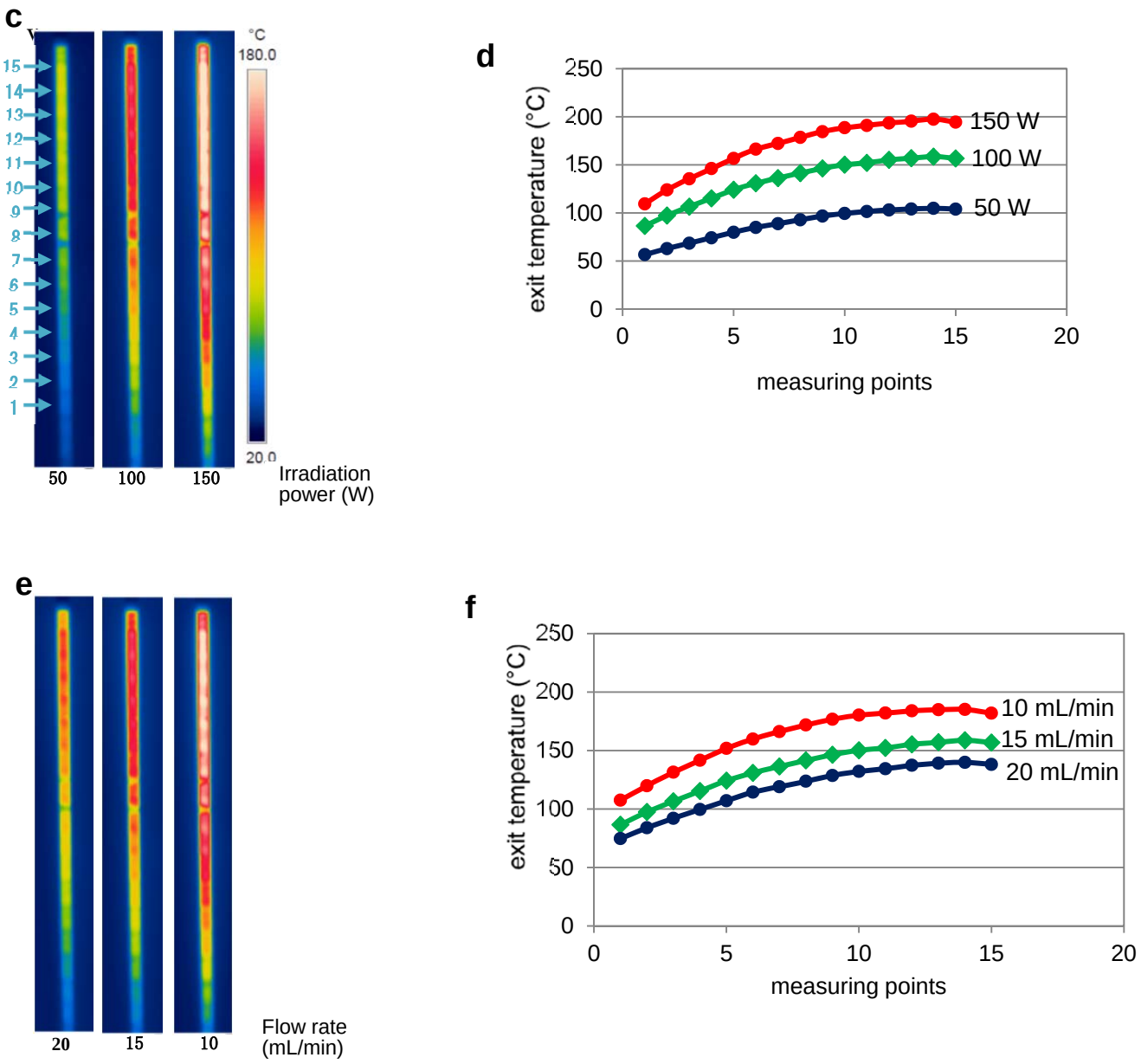


Fig. S5 (continued) (c) and (d) The corrected temperature distribution of the tube reactor while MeCN was pumped into it at the fixed flow rate of 15 mL/min with three different MW irradiation powers (50 W, 100 W, and 150 W). (e) and (f) The corrected temperature distribution of the tube reactor while MeCN was pumped into it with the fixed irradiation power of 100 W at three different flow rates (10 mL/min, 15 mL/min, and 20 mL/min).

5. The continuous preparation of 1,2,3,4-tetrahydro-1*H*-carbazole (4) during 60 min using the continuous flow MW apparatus

A solution of cyclohexanone (**1**) in AcOH (2.0 M) and a solution of phenylhydrazine (**2**) in MeCN (2.2 M) were pumped into the reactor at the same flow rate (total flow rate of 15 mL/min) with the MW irradiation of 145 W. After 5 min, the exit temperature reached a steady state at 240 °C. The collection of the crude reaction mixture into a flask was started at this time and continued during the following 60 min, while the exit temperature kept at around 240 °C by adjusting the irradiation power in a range of 130–150 W to avoid boiling the reaction mixture. The time-course of the exit temperature and the irradiation power are shown in Fig. S6. The crude reaction mixture was placed in a refrigerator (ca. 4 °C) for 3 h, and the precipitate was filtered to give 52.6 g of 1,2,3,4-tetrahydro-1*H*-carbazole (**4**). The filtrate was concentrated and the residue was dissolved in EtOAc and washed with brine. The organic layer was separated, dried with $MgSO_4$, and concentrated *in vacuo*. The residue was purified by recrystallization from MeCN to provide 47.1 g of **4**. The mother liquor was concentrated *in vacuo* and purified by column chromatography on silica gel (*n*-hexane/EtOAc = 10:1) to give 15.3 g of **4**. Each product was obtained as a pale brown solid, and its purity was examined by ^{1}H NMR analysis to be more than 95%. Thus, 115 g (75% yield based on **1**) of **4** was obtained in total. Mp 112–115 °C (lit.[1] 114–116 °C). δ_H (500 MHz; $CDCl_3$; Me_4Si) 1.87–1.95 (m, 4H, C(2)-H_2 and C(3)-H_2), 2.71–2.75 (m, 4H, C(1)-H_2 and C(4)-H_2), 7.06–7.13 (m, 2H, C(6)-H and C(7)-H), 7.28 (d, J = 8 Hz, 1H, C(8)-H), 7.46 (d, J = 8 Hz, 1H, C(5)-H), 7.66 (br s, 1H, NH). δ_C (125 MHz; $CDCl_3$) 20.9, 23.19, 23.21, 23.3, 110.1, 110.3, 117.7, 119.1, 120.9, 127.8, 134.0, 135.6.

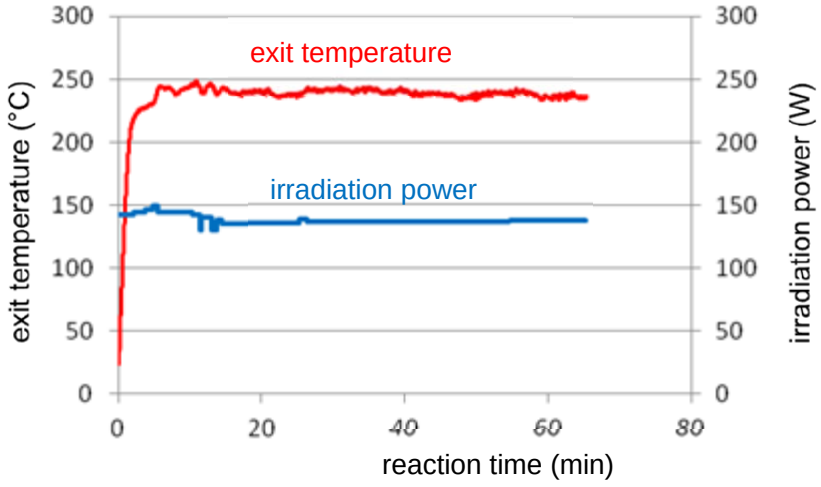
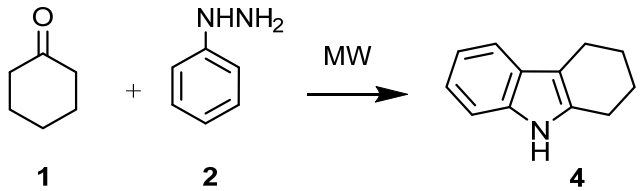


Fig. S6 The time-course of the exit temperature and the irradiation power during the continuous preparation of 1,2,3,4-tetrahydrocarbazole (**4**) for 60 min.

6. Comparison of our continuous preparation of 1,2,3,4-tetrahydro-1*H*-carbazole (4) for 60 min with the related reactions using MW-assisted continuous flow reactors and a MW-assisted batch reactor



1 **2** **4**

Ref.	Reactor	Substrates and reaction conditions	Product **4** yield	Productivity per hour (mmol/h)
This work	MW-assisted flow reactor	**1**:**2**＝1:1.1, AcOH–MeCN (1:1, 2.0 M), 240 °C, 140 W, 15 mL/min, 60 min	115 g, 75%	**673**
Ref.1	MW-assisted flow reactor filled with sand	**1**:**2**＝1:1.1, AcOH (0.5 M) 150 °C, 150 W, 0.5 mL/min, 80 min	3.1 g, 91%	**14**
Ref.2	MW-assisted flow reactor	**1**:**2**＝1.1:1, AcOH–*i*-PrOH (3:1, 1 M) 230 °C, 2.1 mL/min	90% (estimated 9.8 g/h)	**estimated 57**
Footnote 10 of ref.1	MW-assisted batch reactor	**1**:**2**＝1:1.1, AcOH (0.5 M) 150 °C, 150 W, 10 min	0.21 g, 62%	**estimated 7.4**

7. The Diels–Alder reaction of diethyl acetylenedicarboxylate (5) and furan (6) by the continuous flow MW apparatus

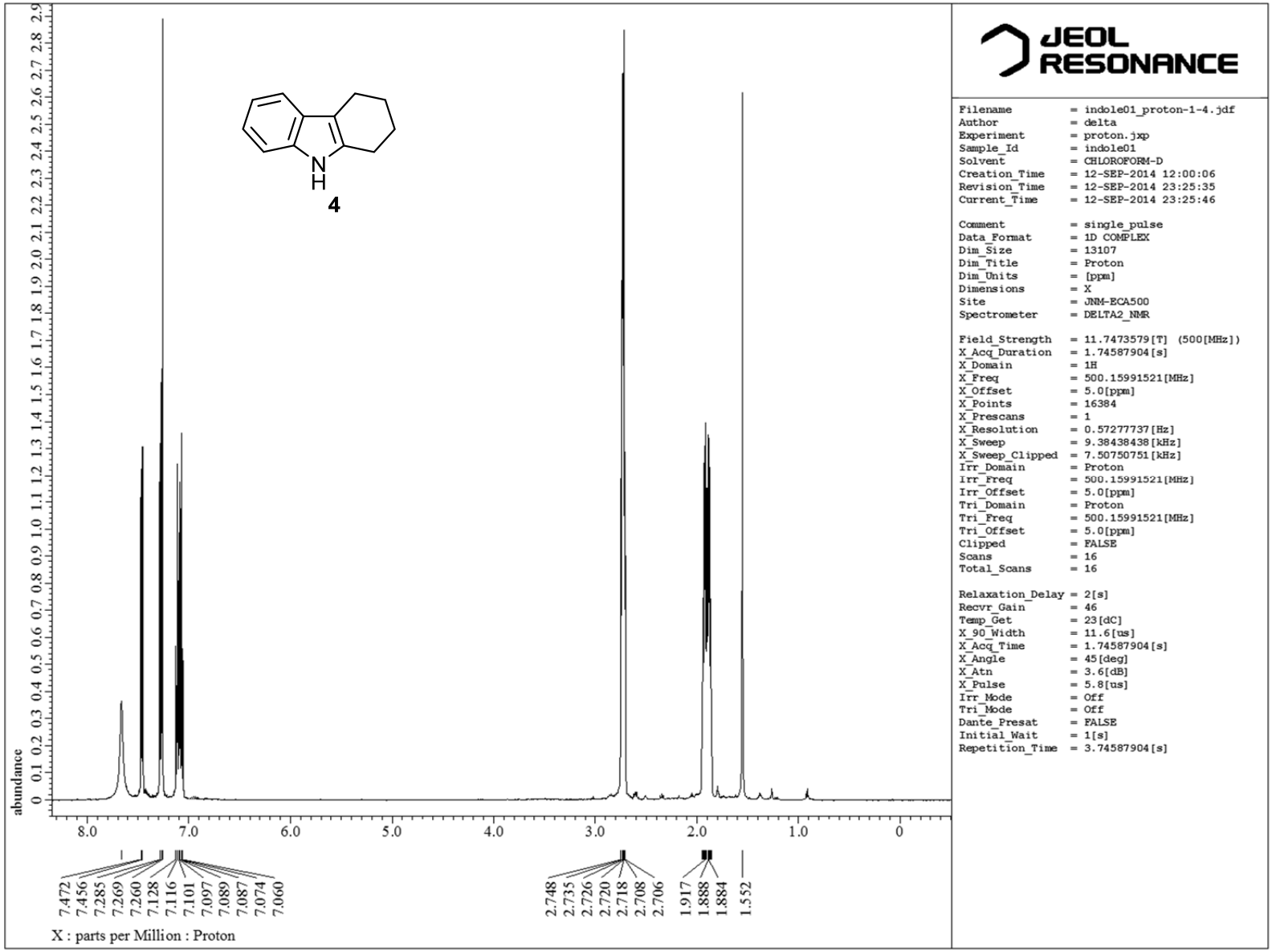
A solution of diethyl acetylenedicarboxylate (**5**) (1.1 M) and furan (**6**) (4.4 M) in *n*-PrOH was pumped into the reactor at the flow rate of 5.0 mL/min with the MW irradiation of 60 W. After 5 min, the exit temperature reached a steady state at 194 °C. The collection of the crude reaction mixture was started at this time and continued during the following 5 min, while the exit temperature kept at 194 °C. The crude product was concentrated *in vacuo*, and the residue was purified by column chromatography on silica gel (*n*-hexane /EtOAc = 10:1 to 3:1) to give diethyl 7-oxabicyclo[2.2.1]hepta-2,5-diene-2,3-dicarboxylate (**7**)[3] (4.91 g, 76%) as a slightly yellowish oil. δ_H (500 MHz; $CDCl_3$; Me_4Si) 1.32 (t, J = 7 Hz, 6H, CH_3 x 2), 4.27 (q, J = 7 Hz, 4H, CH_2 x 2), 5.68 (s, 2H, CH-O x 2), 7.22 (br s, 2H, vinyl-H x 2). δ_C (125 MHz; $CDCl_3$) 14.1 (*C*H3), 61.4 (*C*H2), 85.1 (*C*-O), 143.2 (vinyl-*C*H), 152.7 (vinyl-*C*), 163.1 (*C*=O).

References

1. M. C. Bagley, R. L. Jenkins, M. C. Lubinu, C. Mason, R. Wood, *J. Org. Chem.*, 2005, **70**, 7003.

2. P. Öhrngren, A. Fardost, F. Russo, J. S. Schanche, M. Fagrell, M. Larhed, *Org. Process Res. Dev.*, 2012, **16**, 1053.

3. M. J. Karney, K. A. Porter, E. K. Barnhardt, G. S. Vanier, *RSC Adv*., 2013, **3**, 7106.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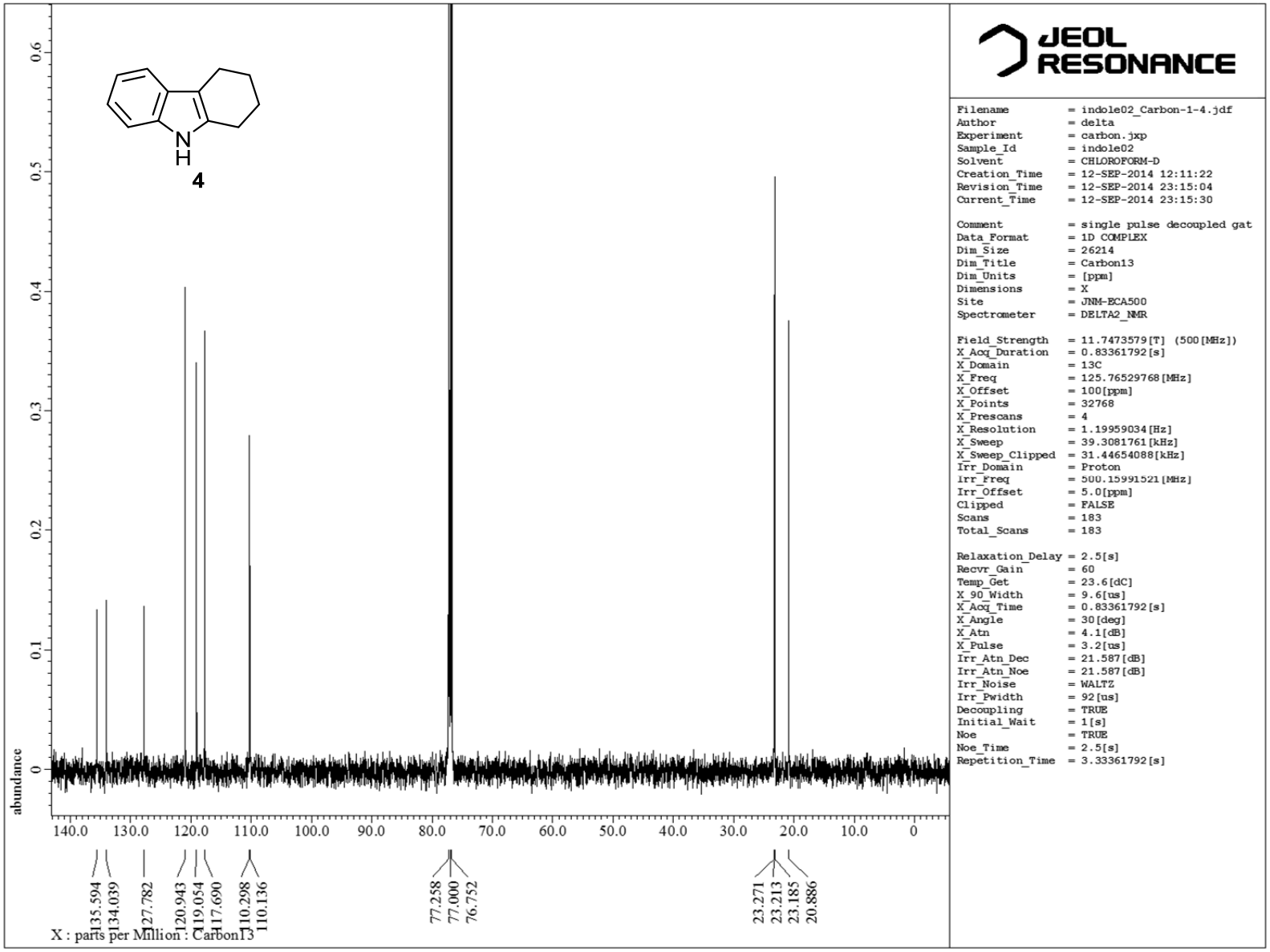
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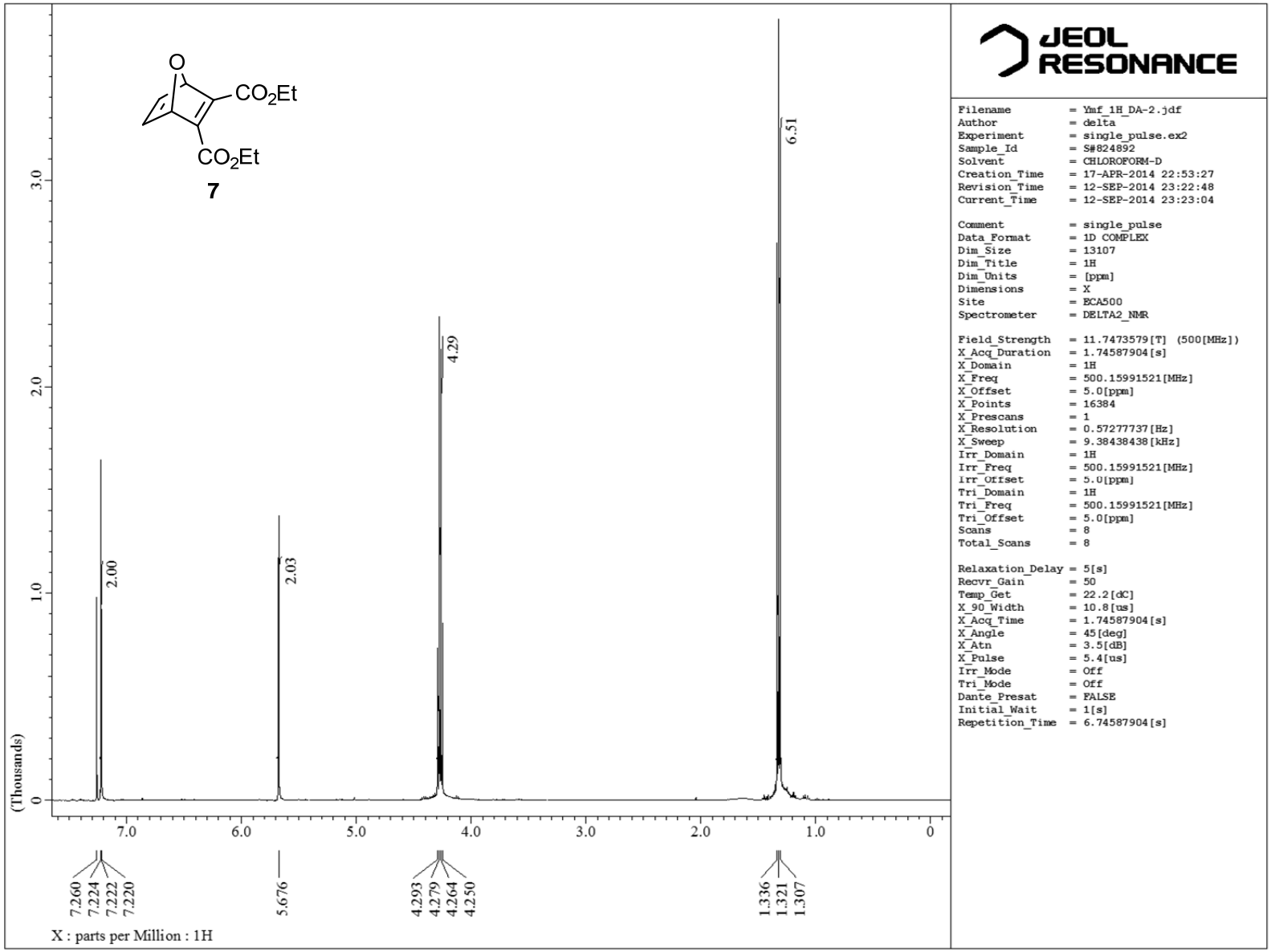

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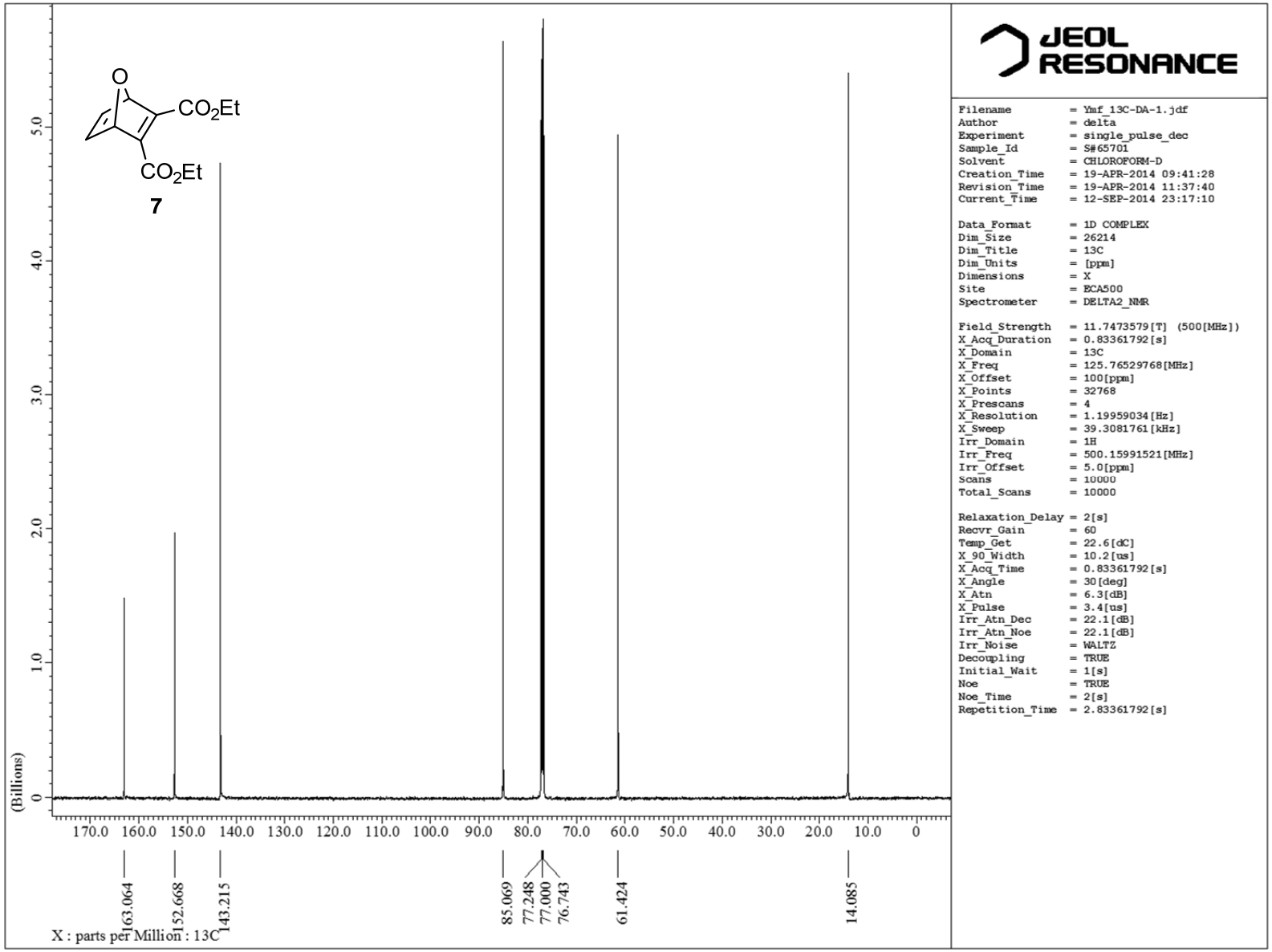
O
CO2Et
CO2Et
7
(Billions)
5.0
4.0
3.0
2.0
1.0
0
170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
163.064
152.668
143.215
85.069
77.248
77.000
76.743
61.424
14.085
X : parts per Million : 13C
JEOL RESONANCE
Filename = Ymf_13C-DA-1.jdf
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#65701
Solvent = CHLOROFORM-D
Creation_Time = 19-APR-2014 09:41:28
Revision_Time = 19-APR-2014 11:37:40
Current_Time = 12-SEP-2014 23:17:10
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = BCA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
Irr_Domain = 1H
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Scans = 10000
Total_Scans = 10000
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 22.6[dC]
X_90_Width = 10.2[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 6.3[dB]
X_Pulse = 3.4[us]
Irr_Atn_Dec = 22.1[dB]
Irr_Atn_Noe = 22.1[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.83361792[s]