Electronic Supplementary Material (ESI) for Chemical Communications

Electronic Supplementary Information

Modular synthesis of optically active lactones by Ru-catalyzed asymmetric allylic carboxylation and ring-closing metathesis reaction

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Experimental Procedures

General.

All reactions were carried out under argon atmosphere using standard Schlenk techniques, and the workup was performed in air. ^{1}H and ^{13}C NMR spectra were recorded on Varian Mercury 300, JEOL JNM-GSX 400, JEOL JNM-ECS 400 and JEOL JNM-ECA 500 spectrometers. Enantiomeric excess was determined by HPLC analysis using Hitachi L-2130/L-2455 and Shimadzu LC -10/SPD-10AV equipped with DAICEL Chiralcel OJ-H, OD-H, OB-H and Chiralpak AS-H columns. Optical rotation was measured on JASCO DIP-100. HRMS measurements were carried out on Thermo Fisher Scientific LTQ-Orbitrap XL.

Materials.

All solvents used for reactions were passed through purification columns just before use. Planar-chiral Cp'Ru complex **1a**-**1c** were synthesized as reported previously.[1,2] Cinnamyl chloride **2a** was purchased from TCI. Allylic chlorides **2b**-**2d**, **2f** and **2g** were prepared by Corey-Kim chlorination of the corresponding allylic alcohols,[3] whereas **2e** was prepared by the method according to that for analogous bromide.[4] All allylic chlorides were purified by distillation (**2a**, **2c**, **2f** and **2g**) or recrystallization from *n*-hexane (**2b**, **2d** and **2e**) prior to use. Unsaturated carboxylic acids were available from commercial source and used without any purification. Sodium carboxylate was synthesized by the reaction of the corresponding unsaturated carboxylic acid with Na_2CO_3 in acetone/water (*ca.* v/v = 1/1). 2nd generation Grubbs' catalyst (**G-II**) was purchased from Sigma-Aldrich.

Standard Procedure for Asymmetric Allylic Carboxylation.

Method A.

To a THF solution (1 mL) of (*R*)-**1c** (5 μmol, 1 mol%), allylic chloride (1.0 mmol) and Na_2CO_3 (1.5 mmol) was added a THF solution (1 mL) of unsaturated carboxylic acid (0.5 mmol), and the resulting mixture was stirred for 4 h at 25 °C. After dilution with *n*-hexane, the insoluble parts were filtered off through Celite and the filtrate was concentrated under reduced pressure. The residue was purified by SiO_2 column chromatography using a mixture of *n*-hexane and Et_2O (v/v = 20/1) as the eluent. Evaporation of the solvent gave branched allylic ester as colorless oil.

Method B.

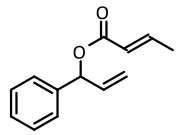
To a THF solution (1 mL) of (*R*)-**1c** (5 μmol, 1 mol%), allylic chloride (1.0 mmol) and Na_2CO_3 (1.5 mmol) was added a THF solution (1 mL) of unsaturated carboxylic acid (0.5 mmol), and the resulting mixture was stirred for 4 h at 25 °C. After dilution with *n*-hexane, the insoluble parts were filtered off through Celite and the filtrate was concentrated under reduced pressure. Acetone (5 mL) and diethylamine (5 mL) were added, and the mixture was stirred for 6 h at room temperature. After removal of the solvent under reduced pressure, the resulting crude material was purified by SiO_2 column chromatography to give branched allylic ester as colorless oil.

Method C.

To a THF solution (2 mL) of (*R*)-**1c** (15 μmol, 3 mol%) and allylic chloride (2.5 mmol) was added sodium carboxylate (0.5 mmol) portionwise, and the resulting mixture was stirred for 4 h at 25 °C. Workup was performed by the same method as **Method A**.

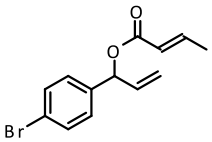
Characterization of Allylic Esters.

(*E*)-1-phenylallyl but-2-enoate (**4ab**, **Method A**)



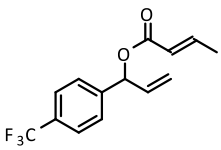
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.40–7.26 (m, 5H, Ph), 7.02 (dq, 1H, *J* = 15.5, 7.0 Hz, C*H*CH_3), 6.32 (ddd, 1H, *J* = 5.8, 1.4, 1.4 Hz, PhC*H*), 6.03 (ddd, 1H, *J* = 17.0, 10.3, 5.8 Hz, CH=), 5.91 (dq, 1H, *J* = 15.5, 1.7 Hz, COCH), 5.30 (ddd, 1H, *J* = 17.0, 1.4, 1.4 Hz, =CH_2), 5.24 (ddd, 1H, *J* = 10.3, 1.4, 1.4 Hz, =CH_2), 1.88 (dd, 3H, *J* = 7.0, 1.7 Hz, CH_3). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 165.3, 145.1, 139.1, 136.4, 128.5, 128.0, 127.1, 122.7, 116.7, 75.8, 17.9. HPLC analysis: Chiralcel OJ-H column, *n*-hexane/^{i}PrOH = 98/2 (v/v), 1.0 mL/min, 220 nm; major enantiomer *t* = 20.1 min, minor enantiomer *t* = 16.9 min, 97% ee. $[\alpha]_D^{28}$ = –38.2 (*c* 0.27, $CHCl_3$). HRMS (ESI): Calcd for $C_{13}H_{14}O_2Na$ [M+Na$^+$]: 225.0886, found: *m*/*z* = 225.0889.

(*E*)-1-(4-bromophenyl)allyl but-2-enoate (**4bb**, **Method A**)



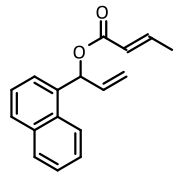
^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.50–7.43 (m, 2H, Ar), 7.26–7.21 (m, 2H, Ar), 7.02 (dq, *J* = 15.7, 6.9 Hz, =C*H*CH_3), 6.28–6.23 (m, 1H, ArC*H*), 5.98 (ddd, 1H, *J* = 17.7, 10.4, 5.8 Hz, CH=), 5.90 (dq, 1H, *J* = 15.7, 1.7 Hz, COCH=), 5.33-5.22 (m, 2H, =CH_2), 1.89 (dd, 3H, *J* = 6.9, 1.7 Hz, CH_3). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 147.6, 131.8, 125.8, 124.0, 120.6, 118.4, 113.3, 113.0, 109.1, 75.4, 29.7. HPLC analysis: Chiralcel OD-H column, *n*-hexane/^{i}PrOH = 100/1 (v/v), 0.5 mL/min, 220 nm; major enantiomer *t* = 13.2 min, minor enantiomer *t* = 12.2 min, 96% ee. $[\alpha]_D^{29}$ = –21.3 (*c* 0.19, $CHCl_3$). HRMS (ESI): Calcd for $C_{13}H_{13}O_2BrNa$ [M+Na$^+$]: 302.9991, found: *m*/*z* = 302.9996.

(*E*)-1-(4-trifluoromethylphenyl)allyl but-2-enoate (**4cb**, **Method A**)



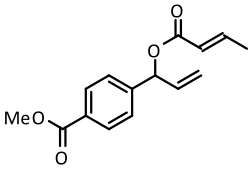
^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.61 (d, 2H, *J* = 8.3 Hz, Ar), 7.47 (d, 2H, *J* = 8.3 Hz, Ar), 7.04 (dq, *J* = 15.5, 6.9 Hz, =C*H*CH_3), 6.36–6.31 (m, 1H, ArC*H*), 6.00 (ddd, 1H, *J* = 17.3, 10.4, 5.9 Hz, CH=), 5.92 (dq, 1H, *J* = 15.5, 1.7 Hz, COCH=), 5.36–5.25 (m, 2H, =CH_2), 1.90 (dd, 3H, *J* = 6.9, 1.7 Hz, CH_3). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 166.2, 145.8, 143.1 (q, *J* = 1.3 Hz), 135.7, 130.2 (q, *J* = 32.5 Hz), 127.3, 125.5 (q, *J* = 3.8 Hz), 124.0 (q, *J* = 272.1 Hz), 122.3, 117.6, 75.2, 17.9. HPLC analysis: Chiralcel OD-H column, *n*-hexane/^{i}PrOH = 200/1 (v/v), 0.5 mL/min, 220 nm; major enantiomer *t* = 15.4 min, minor enantiomer *t* = 13.9 min, 98% ee. $[\alpha]_D^{30}$ = –11.9 (*c* 0.28, $CHCl_3$). HRMS (ESI): Calcd for $C_{14}H_{13}O_2F_3Na$ [M+Na$^+$]: 293.0765, found: *m*/*z* = 293.0760.

(*E*)-1-(naphthalene-1-yl)allyl but-2-enoate (**4db**, **Method B**)



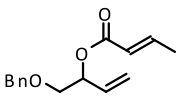
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.16–8.11 (m, 1H, Ar), 7.89–7.79 (m, 2H, Ar) 7.63–7.57 (m, 1H, Ar) 7.56–7.42 (m, 3H, Ar), 7.10–6.98 (m, 2H, ArC*H* and =C*H*CH_3), 6.21 (ddd, 1H, *J* = 17.0, 9.8, 5.2 Hz, CH=), 5.93 (dq, 1H, *J* = 15.6, 1.7 Hz, COCH=), 5.36–5.25 (m, 2H, =CH_2), 1.88 (dd, 3H, *J* = 6.9, 1.7 Hz, CH_3). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 147.7, 131.6, 124.2, 123.1, 122.5, 112.0, 118.5, 118.4, 116.3, 115.9, 115.6, 115.5, 114.4, 113.4, 109.0, 74.0, 29.7. HPLC analysis: Chiralcel OD-H column, *n*-hexane/^{i}PrOH =200/1 (v/v), 0.5 mL/min, 220 nm; major enantiomer *t* = 30.7 min, minor enantiomer *t* = 28.9 min, 96% ee. $[\alpha]_D^{29}$ = –24.4 (*c* 0.27, $CHCl_3$). HRMS (ESI): Calcd for $C_{17}H_{16}O_2Na$ [M+Na$^+$]: 275.1043, found: *m*/*z* = 275.1047.

(*E*)-methyl 4-(1-(but-2-enyloxy)allyl)benzoate (**4eb**, **Method B**)



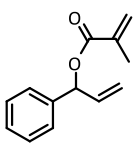
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.04–7.99 (m, 2H, Ar), 7.45–7.39 (m, 2H, Ar), 7.04 (dq, *J* = 15.7, 6.9 Hz, =C*H*CH_3), 6.37–6.31 (m, 1H, ArC*H*), 6.00 (ddd, 1H, *J* = 17.7, 10.4, 5.8 Hz, CH=), 5.92 (dq, 1H, *J* = 15.7, 1.7 Hz, COCH), 5.36–5.23 (m, 2H, =CH_2), 3.91 (s, 3H, OCH_3), 1.90 (dd, 3H, *J* = 6.9, 1.7 Hz, CH_3). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 148.7, 147.5, 131.9, 130.6, 124.0, 119.2, 116.9, 113.3, 109.4, 75.6, 57.0, 29.8, 16.2. HPLC analysis: Chiralcel OJ-H column, *n*-hexane/^{i}PrOH = 98/2 (v/v), 1.0 mL/min, 220 nm; major enantiomer *t* = 39.8 min, minor enantiomer *t* = 22.1 min. 95% ee. $[\alpha]_D^{29}$ = –18.4 (*c* 0.28, $CHCl_3$). HRMS (ESI): Calcd for $C_{15}H_{16}O_4Na$ [M+Na$^+$]: 283.0941, found: *m*/*z* = 283.0945.

(*E*)-1-(benzyloxy)but-3-en-2-yl but-2-enoate (**4fb**, **Method A**)



^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.37–7.25 (m, 5H, Ph), 7.01 (dq, 1H, *J* = 15.7, 6.9 Hz, =C*H*CH_3), 5.93–5.91 (m, 2H, COCH= and C*H*=), 5.57–5.50 (m, 1H, C*H*CH=), 5.32 (ddd, 1H, *J* =17.3, 1.5, 1.4 Hz, =CH_2), 5.23 (ddd, 1H, *J* = 10.7, 1.4, 1.2 Hz, =CH_2), 4.59 (d, 1H, *J* = 12.2 Hz, PhCH_2), 4.54 (d, 1H, *J* = 12.2 Hz, PhCH_2), 3.61 (dd, 1H, *J* = 10.7, 4.0 Hz, CH_2CH), 3.59 (dd, 1H, *J* = 10.7, 2.8 Hz, CH_2CH), 1.89 (dd, 3H, *J* = 6.9, 1.7 Hz, CH_3). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 165.5, 144.9, 137.9, 133.5, 128.3, 127.5, 122.6, 117.6, 73.1, 72.8, 71.3, 17.9. One carbon signal could not be detected probably due to overlapping. HPLC analysis: Chiralcel OJ-H column, 2% ^{i}PrOH, 1.0 mL/min, 220 nm; major enantiomer *t* = 18.3 min, minor enantiomer *t* = 15.1 min. 90% ee. $[\alpha]_D^{28}$ = –11.0 (*c* 0.22, $CHCl_3$). HRMS (ESI): Calcd for $C_{15}H_{18}O_3Na$ [M+Na$^+$]: 269.1148, found: *m*/*z* = 269.1152.

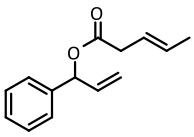
(*E*)-1-phenylallyl methacrylate (**4ac**, **Method A**)



^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.41–7.27 (m, 5H, Ph), 6.32 (ddd, 1H, *J* = 5.8, 1.7, 1.5 Hz, PhC*H*), 6.22–6.17 (m, 1H, =CH_2), 6.04 (ddd, 1H, *J* = 17.1, 10.5, 5.8 Hz, CH=), 5.59 (dq, 1H, *J* = 1.6, 1.6 Hz, CCH_3=CH_2), 5.32 (ddd, 1H, *J* = 17.1, 1.7, 1.4 Hz, CH=CH_2), 5.25 (ddd, 1H, *J* = 10.5, 1.5, 1.4 Hz, CH=CH_2), 1.98 (dd, 3H, *J* = 1.6, 0.9 Hz, CH_3). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 166.3, 139.1, 136.5, 136.4, 128.6, 128.1, 127.0, 125.7, 116.8, 76.3, 18.3. HPLC analysis: Chiralcel OJ-H column, *n*-hexane/^{i}PrOH = 100/2 (v/v), 0.5 mL/min, 220 nm; major enantiomer *t* = 15.6

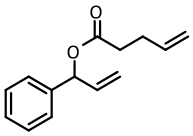
min, minor enantiomer t = 12.8 min, 94% ee. $[\alpha]_D^{29}$ = –25.4 (c 0.23, $CHCl_3$). HRMS (ESI): Calcd for $C_{14}H_{16}O_2Na$ [M+Na^+]: 225.0886, found: m/z = 225.0889.

(*E*)-1-phenylallyl pent-3-enoate (**4ad**, **Method C**)



^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.40–7.26 (m, 5H, Ph), 6.26 (ddd, 1H, J = 6.0, 1.7, 1.5 Hz, PhC*H*), 6.00 (ddd, 1H, J = 17.1, 10.4, 6.0 Hz, CH=), 5.64–5.49 (m, 2H, C*H*=C*H*), 5.28 (ddd, 1H, J = 17.1, 1.7, 1.3 Hz, =CH_2), 5.24 (ddd, 1H, J = 10.4, 1.5, 1.3 Hz, =CH_2), 3.10–3.05 (m, 2H, CH_2), 1.71–1.66 (m, 3H, CH_3). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 171.1, 138.9, 136.3, 129.6, 128.5, 128.1, 127.1, 122.5, 116.9, 76.2, 38.2, 17.9. HPLC analysis: Chiralcel OJ-H column, *n*-hexane/^{i}PrOH = 98/2 (v/v), 1.0 mL/min, 220 nm; major enantiomer t = 9.7 min, minor enantiomer t = 7.5 min, 97% ee. $[\alpha]_D^{29}$ = –27.2 (c 0.23, $CHCl_3$). HRMS (ESI): Calcd for $C_{14}H_{16}O_2Na$ [M+Na^+]: 239.1043, found: m/z = 239.1047.

1-phenylallyl pent-4-enoate (**4ae**, **Method C**)



^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.40–7.26 (m, 5H, Ph), 6.32 (ddd, 1H, J = 5.9, 1.6, 1.4 Hz, PhC*H*), 6.00 (ddd, 1H, J = 17.1, 10.4, 5.9 Hz, CH=), 5.86-5.76 (m, 1H, CH_2C*H*=), 5.30 (ddd, 1H, J = 17.1, 1.6, 1.3 Hz, =CH_2), 5.23 (ddd, 1H, J = 10.4, 1.4, 1.3 Hz, =CH_2), 5.09–4.90 (m, 2H, CH_2CH=CH_2), 2.51–2.35 (m, 4H, CH_2CH_2CH=). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 152.9, 126.5, 124.6, 124.4, 118.2, 117.8, 117.0, 108.9, 107.8, 76.3, 42.4, 38.4. HPLC analysis: Chiralcel OJ-H column, *n*-hexane/^{i}PrOH = 98/2 (v/v), 1.0 mL/min, 220 nm; major enantiomer t = 8.7 min, minor enantiomer t = 7.9 min. 95% ee. $[\alpha]_D^{29}$ = –34.7 (c 0.25, $CHCl_3$). HRMS (ESI): Calcd for $C_{14}H_{16}O_2Na$ [M+Na^+]: 239.1043, found: m/z = 239.1046.

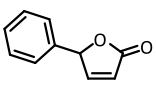
Standard Procedure for Ring-Closing Metathesis (RCM) of Branched Allylic Ester.

To a CH_2Cl_2 solution (5 mL) of **G-II** (6 μmol, 2 mol%) was added a CH_2Cl_2 solution (1 mL) of branched allylic ester (0.3 mmol) and the mixture was stirred for 16 h at 25 °C. After CH_2Cl_2 was removed under reduced pressure, the residue was purified by SiO_2 column chromatography using a mixture of *n*-hexane and AcOEt (v/v = 7/3) as the eluent. Concentration of the resulting solution gave optically active lactones.

For the syntheses of **6db**, **6fb**, **6ac**, and **7**, the reactions were performed under reflux, whereas **8** was prepared by the reaction at 30 ºC.

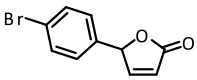
Charactarization of Lactones.

5-phenylfuran-2(5*H*)-one (**6ab**).[5]



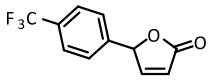
^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.52 (dd, 1H, J = 5.5, 2.0 Hz, CH=), 7.43–7.32 (m, 3H, Ph), 7.29–7.24 (m, 2H, Ph), 6.23 (dd, 1H, J = 5.5, 1.8 Hz, PhC*H*), 6.01 (dd, 1H, J = 2.0, 1.8 Hz, COCH=). ^{13}C NMR ($CDCl_3$, 76 MHz): δ 173.0, 155.8, 134.3, 129.3, 129.0, 126.5, 121.0, 84.3. HPLC analysis: Chiralcel OJ-H column, *n*-hexane/^{i}PrOH = 9/1 (v/v), 1.0 mL/min, 220 nm; major enantiomer t = 31.1 min, minor enantiomer t = 34.5 min, 97% ee.

5-(4-bromophenyl)furan-2(5*H*)-one (**6bb**).[5]



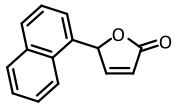
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.54 (d, 2H, *J* = 8.4 Hz, Ar), 7.50 (dd, 1H, *J* = 5.6, 1.8 Hz, CH=), 7.15 (d, 2H, *J* = 8.4 Hz, Ar), 6.24 (dd, 1H, *J* = 5.6, 2.2 Hz, ArC*H*), 5.97 (dd, 1H, *J* = 2.2, 1.8 Hz, COCH=). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 172.6, 155.2, 133.3, 132.3, 128.1, 123.4, 121.3, 83.5. HPLC analysis: Chiralpak AS-H column, *n*-hexane/^{i}PrOH = 4/1 (v/v), 0.4 mL/min, 220 nm; major enantiomer *t* = 62.7 min, minor enantiomer *t* = 60.9 min, 98% ee. $[\alpha]_D^{27}$ = –31.2 (*c* 0.28, $CHCl_3$).

5-(4-trifluoromethylphenyl)furan-2(5*H*)-one (**6cb**).



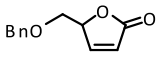
^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.67 (d, 2H, *J* = 8.2 Hz, Ar), 7.54 (dd, 1H, 7.6, 1.7 Hz, CH=), 7.42 (d, 2H, 8.2 Hz, Ar), 6.27 (dd, 1H, *J* = 7.6, 2.2 Hz, ArC*H*), 6.07 (br, 1H, COCH=). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 172.6, 155.2, 138.3 (q, *J* = 1.4 Hz), 131.5 (q, *J* = 32.8 Hz), 122.6, 126.0, (q, *J* = 3.8 Hz), 123.7 (q, *J* = 272.3 Hz), 121.3, 88.3. HPLC analysis: Chiralpak AS-H column, *n*-hexane/^{i}PrOH = 9/1 (v/v), 0.3 mL/min, 210 nm; major enantiomer *t* = 77.3 min, minor enantiomer *t* = 74.0 min, 95% ee. $[\alpha]_D^{29}$ = –140.6 (*c* 0.12, $CHCl_3$). HRMS (ESI): Calcd for $C_{11}H_7F_3O_2Na$ [M+Na$^+$]: 251.0290, found: *m*/*z* = 251.0293.

5-(naphthalene-1-yl)furan-2(5*H*)-one (**6db**)



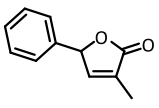
^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.91–7.81 (m, 3H, Ar), 7.79 (br, 1H Ar), 7.60 (dd, 1H, *J* = 5.6, 1.6 Hz, CH=), 7.57–7.48 (m, 2H, Ar), 7.31 (dd, 1H, *J* = 8.5, 1.7 Hz, Ar), 6.28 (br, 1H, *J* = 5.6, 2.0 Hz, COCH=), 6.18 (br, 1H, ArC*H*). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 173.1, 155.8, 133.6, 133.2, 131.6, 129.1, 128.0, 127.8, 126.9, 126.8, 126.2, 123.3, 121.2, 84.5. HPLC analysis: Chiralpak AS-H column, *n*-hexane/^{i}PrOH = 4/1 (v/v), 1.0 mL/min, 220 nm; major enantiomer *t* = 29.4 min, minor enantiomer *t* = 24.1 min, 99% ee. $[\alpha]_D^{30}$ = –196.6 (*c* 0.14, $CHCl_3$). HRMS (ESI): Calcd for $C_{14}H_{10}O_2Na$ [M+Na$^+$]: 233.0573, found: *m*/*z* = 233.0577.

5-(benzyloxymethyl)furan-2(5*H*)-one (**6fb**).[6]



^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.50 (dd, 1H, *J* = 5.8, 1.6 Hz, CH=), 7.41–7.27 (m, 5H, Ph), 6.17 (dd, 1H, *J* = 5.8, 2.1 Hz, CH=), 5.17 (tdd, 1H, *J* = 5.2, 2.1, 1.6 Hz, C*H*CH=), 4.57 (s, 2H, PhCH_2), 3.75 (dd, 1H, *J* = 10.4, 5.2, OCH_2CH) 3.67 (dd, 1H, *J* = 10.4, 5.2 Hz, OCH_2CH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 172.7, 153.8, 137.3, 128.5, 128.0, 127.7, 122.6, 82.1, 73.8, 69.5. HPLC analysis: Chiralpak AS-H column, *n*-hexane/^{i}PrOH = 4/1 (v/v), 1.0 mL/min, 220 nm; major enantiomer *t* = 24.1 min, minor enantiomer *t* = 20.4 min, 95% ee.

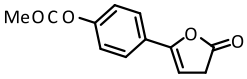
3-methyl-5-phenylfuran-2(5*H*)-one (**6ac**).[7]



^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.43–7.32 (m, 3H, Ph), 7.28–7.23 (m, 2H, Ph), 7.12 (dq, 1H, *J* = 1.7, 1.7 Hz, CH=), 5.88–5.83 (m, 1H, PhC*H*), 1.99 (dd, 3H, *J* = 1.7, 1.7 Hz, CH_3). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 174.3, 148.4, 135.4, 129.5, 129.0, 128.9, 126.4, 82.1, 10.6. HPLC analysis: Chiralpak AS-H column, *n*-hexane/^{i}PrOH = 9/1 (v/v), 1.0 mL/min, 220 nm;

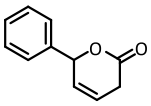
major enantiomer *t* = 21.7 min, minor enantiomer *t* = 17.1 min, 97% ee.

Methyl 4-(5-oxo-4,5-dihydrofuran-2-yl)benzoate (**7**).



^{1}H NMR ($CDCl_3$, 500 Hz): δ 8.08 (d, 2H, *J* = 8.7 Hz, Ar), 7.67 (d, 2H, *J* = 8.7 Hz, Ar), 5.93 (t, 1H, *J* = 2.8 Hz, =CH), 3.94 (s, 3H, Me), 3.46 (d, 2H, *J* = 2.8 Hz, CH_2). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 175.2, 166.4, 153.2, 132.3, 130.9, 130.0, 124.6, 100.2, 52.2, 34.7. HRMS (ESI) Calcd for $C_{12}H_{10}O_4Na$ [M+Na$^+$]: 241.0471, found: *m*/*z* = 241.0475.

6-phenyl-3,6-dihydro-2*H*-pyran-2-one (**8**).[8]

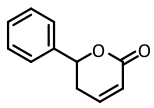


^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.43–7.31 (m, 5H, Ph), 6.07–5.97 (m, 3H, PhC*H* and C*H*=C*H*), 3.19–3.15 (m, 2H, CH_2). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 168.23, 138.20, 128.89, 126.83, 126.18, 121.80, 81.28, 29.91. One carbon signal could not be detected probably due to overlapping. HPLC analysis: Chiralpak AS-H column, *n*-hexane/^{i}PrOH = 4/1 (v/v), 1.0 mL/min, 220 nm; major enantiomer *t* = 27.6 min, minor enantiomer *t* = 25.1 min, 93% ee. $[\alpha]_D^{28}$ = –125.0 (*c* 0.12, $CHCl_3$).

Procedure for Tandem RCM－Olefin Isomerization.

To a 1,2-dichloroethane solution (5 mL) of **G-II** (6 μmol, 2 mol%) was added a 1,2-dichloroethane solution (1 mL) of **4ad** (0.3 mmol), and the mixture was stirred for 16 h at 50 °C. After addition of 2-propanol (0.2 mL), the mixture was refluxed for 48 h. After cooling to room temperature, the solvent was removed under reduced pressure and the residue was purified by SiO_2 column chromatography using a mixture of *n*-hexane and AcOEt (v/v = 7/3) as the eluent. Evaporation of the solvent under reduced pressure gave **9** as white solid.

6-phenyl-5,6-dihydro-2*H*-pyran-2-one (**9**).[9]

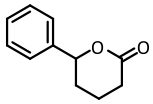


^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.46–7.30 (m, 5H, Ph), 6.97 (ddd, 1H, *J* = 9.7, 5.5, 3.0 Hz, CH_2C*H*=), 6.15 (ddd, 1H, *J* = 9.7, 2.4, 1.3 Hz, COCH=), 5.46 (dd, 1H, *J* =10.5, 5.5 Hz, 1H, PhC*H*), 2.76–2.54 (m, 2H, CH_2). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 164.0, 144.8, 138.5, 128.7, 128.6, 126.0, 121.7, 79.2, 31.6. HPLC analysis: Chiralpak AS-H column, *n*-hexane/^{i}PrOH = 4/1 (v/v), 1.0 mL/min, 220 nm; major enantiomer *t* = 31.1 min, minor enantiomer *t* = 20.4 min, 94% ee.

Procedure for Tandem RCM－Hydrogenation.

To a CH_2Cl_2 solution (5 mL) of **G-II** (6 μmol, 2 mol%) was added a CH_2Cl_2 solution (1 mL) of **4ad** (0.3 mmol). After stirring for 12 h at 30 °C, the mixture was transferred to autoclave through cannula, rinsing with CH_2Cl_2 (3 × 2 mL). The mixture was stirred for 24 h at 70 °C under H_2 pressure (1 MPa). After cooling to room temperature, CH_2Cl_2 was removed under reduced pressure, and the residue was purified by SiO_2 column chromatography using a mixture of *n*-hexane and AcOEt (v/v = 7/3) as the eluent. Recrystallization from *n*-hexane gave **10** as colorless needle.

6-phenyltetrahydro-2*H*-pyran-2-one (**10**).[10]



^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.41–7.27 (m, 5H, Ph), 5.36 (dd, 1H, *J* = 10.1, 3.4 Hz, PhC*H*), 2.79–2.49 (m, 2H, $COCH_2$), 2.25–2.11 (m, 1H, $CHCH_2$), 2.07–1.80 (m, 3H, $CHCH_2$ and $CH_2CH_2CH_2$). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 171.2, 139.7, 128.6, 128.2, 125.7, 81.5, 30.5, 29.5, 18.6. HPLC analysis: Chiralcel OB-H column, hexane/^{i}PrOH = 4/1 (v/v), 1.0 mL/min, 254 nm; major enantiomer *t* = 20.3 min, minor enantiomer *t* = 24.7 min, >99% ee.

Metathesis Reaction of 4ae.

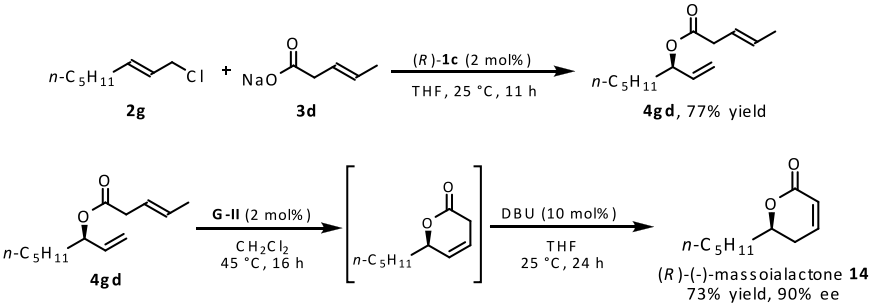
The reaction was performed according to the standard procedure. The resulting brown oil was analyzed by mass spectrometry.

ESI MS: m/z = 189.2(10.5), 399.3(100), 400.3(24.0), 427.3(15.2), 587.2(10.1), 775.3(9.6). Calcd for $C_{12}H_{12}O_2$ [**11**+H]$^+$, 189.1; $C_{24}H_{24}O_4Na$ [**12a**+Na]$^+$, 399.2; $C_{24}H_{24}O_4Na$ [**13**+Na]$^+$, 427.2; $C_{36}H_{36}O_6Na$ [**12b**+Na]$^+$, 587.2; $C_{48}H_{48}O_8Na$ [**12c**+Na]$^+$, 775.3.

One-Pot Sequential Asymmetric Allylic Carboxylation－Ring-Closing Metathesis.

To a THF solution (1 mL) of (*R*)-**1c** (5 μmol, 1 mol%), allylic chloride (1.0 mmol) and Na_2CO_3 (1.5 mmol) was added a 1 mL of THF solution of crotonic acid **3b** (0.5 mmol), and the resulting mixture was stirred for 4 h at 25 °C. **G-II** (10 μmol, 2 mol%) and CH_2Cl_2 (8 mL) were added, and the mixture was stirred for 16 h. After removal of the solvent under reduced pressure, the residue was purified by SiO_2 column chromatography using a mixture of *n*-hexane and AcOEt (v/v = 7/3) as the eluent.

Enantioselective Synthesis of (*R*)-(–)-Massoialactone (14).

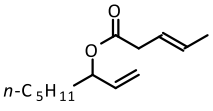


To a THF solution (2 mL) of (*R*)-**1c** (10 μmol, 2 mol%) and allylic chloride **2g** (1.0 mmol) was added sodium pent-3-enoate **3d** (0.5 mmol), and the resulting mixture was stirred for 11 h at 25 °C. After dilution with hexane, insoluble parts were removed by filtration. The filtrate was concentrated under reduced pressure, and the residue was purified by flash column chromatography on SiO_2 using the mixture of *n*-hexane and Et_2O (v/v = 20/1) as the eluent. Evaporation of the solvent gave the ester **4gd** as colorless oil (81 mg, 77% yield).

G-II (6 μmol, 2 mol%) was added to a CH_2Cl_2 solution (6 mL) of **4gd** (63 mg, 0.3 mmol), and the mixture was stirred for 16 h at 45 °C. After removal of the solvent under reduced pressure, a THF

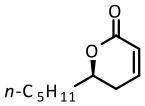
solution (2 mL) of 1,8-diazabicyclo-[5,4,0]-undec-7-ene (30 μmol, 10 mol%) was added and the mixture was stirred for 24 h at 25 °C. The resulting mixture was poured into a mixture of Et_2O (20 mL) and NH_4Cl aq., and the organic layer was washed with NH_4Cl aq., water and brine successively. The solution was dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by SiO_2 column chromatography using a mixture of *n*-hexane and AcOEt (v/v = 7/3) as the eluent to give **14** as colorless oil (36.8 mg, 73% yield).

(*E*)-oct-1-en-3-yl pent-3-enoate (4gd).



^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.77 (ddd, 1H, *J* = 17.0, 10.6, 6.4 Hz, CHC*H*=), 5.65–5.50 (m, 2H, $COCH_2CH$=C*H*), 5.27–5.19 (m, 2H, OCH and $=CH_2$), 5.15 (ddd, 1H, *J* = 10.6, 1.3, 1.3Hz, $=CH_2$), 3.04–2.99 (m, 2H, $COCH_2$), 1.69 (d, 3H, *J* = 4.9 Hz, =CHC*H*$_3$), 1.67–1.54 (m, 1H, CHC*H*$_2$CH$_2$), 1.38–1.21 (m, 7H, C*H*$_2$CH$_2$), 0.88 (t, 3H, *J* = 6.9 Hz, CH_2CH_3). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 171.4, 136.7, 129.3, 122.8, 116.5, 74.9, 38.3, 34.1, 31.5, 24.7, 22.5, 17.9, 13.9. $[\alpha]_D^{26}$ = +4.1 (*c* 0.07, $CHCl_3$). HRMS (ESI): Calcd for $C_{13}H_{22}O_2Na$ [M+Na$^+$]: 233.1512, found: *m*/*z* = 233.1516.

(*R*)-(-)-6-pentyl-5,6-dihydro-2*H*-pyran-2-one, (*R*)-(–)-massoialactone (14).[11]



^{1}H NMR ($CDCl_3$, 500 MHz): δ 6.89–6.83 (m, 1H, CH_2CH=), 6.01 (ddd, 1H, *J* = 9.7, 2.4, 1.4 Hz, COC*H*=), 4.45–4.38 (m, 1H, OC*H*), 2.39–2.28 (m, 2H, C*H*$_2$CH=), 1.86–1.74 (m, 1H, C*H*$_2$CH), 1.69–1.59 (m, 1H, C*H*$_2$CH), 1.54–1.47 (m, 1H, CHC*H*$_2$CH$_2$), 1.46–1.25 (m, 5H, C*H*$_2$CH$_2$), 0.90 (t, 3H, *J* = 7.0 Hz, CH_3). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 164.5, 144.9, 121.5, 78.0, 34.8, 31.5, 29.4, 24.5, 22.4, 13.9. HPLC analysis: Chiralpak AS-H column, hexane/^{i}PrOH = 9/1 (v/v), 1.0 mL/min, 210 nm; major enantiomer *t* = 18.8 min, minor enantiomer *t* = 22.4 min. 90% ee. $[\alpha]_D^{27}$ = –103.7 (*c* 0.20, $CHCl_3$).

References

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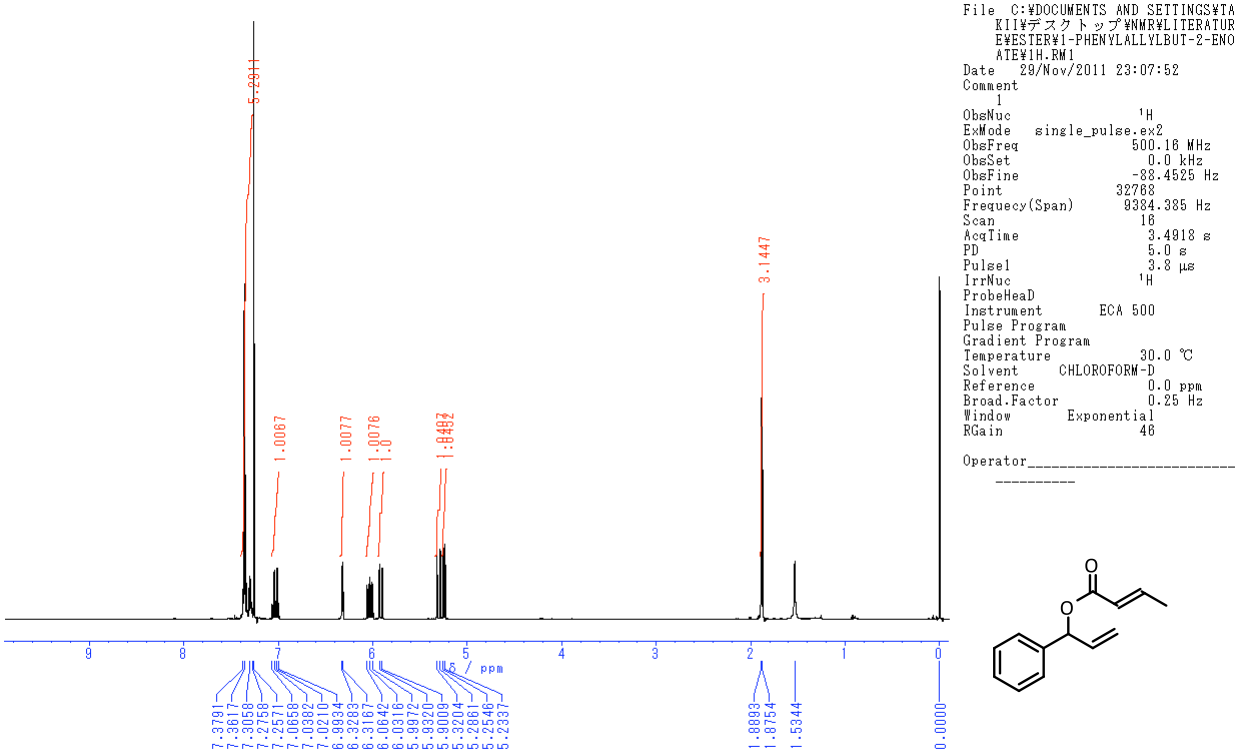
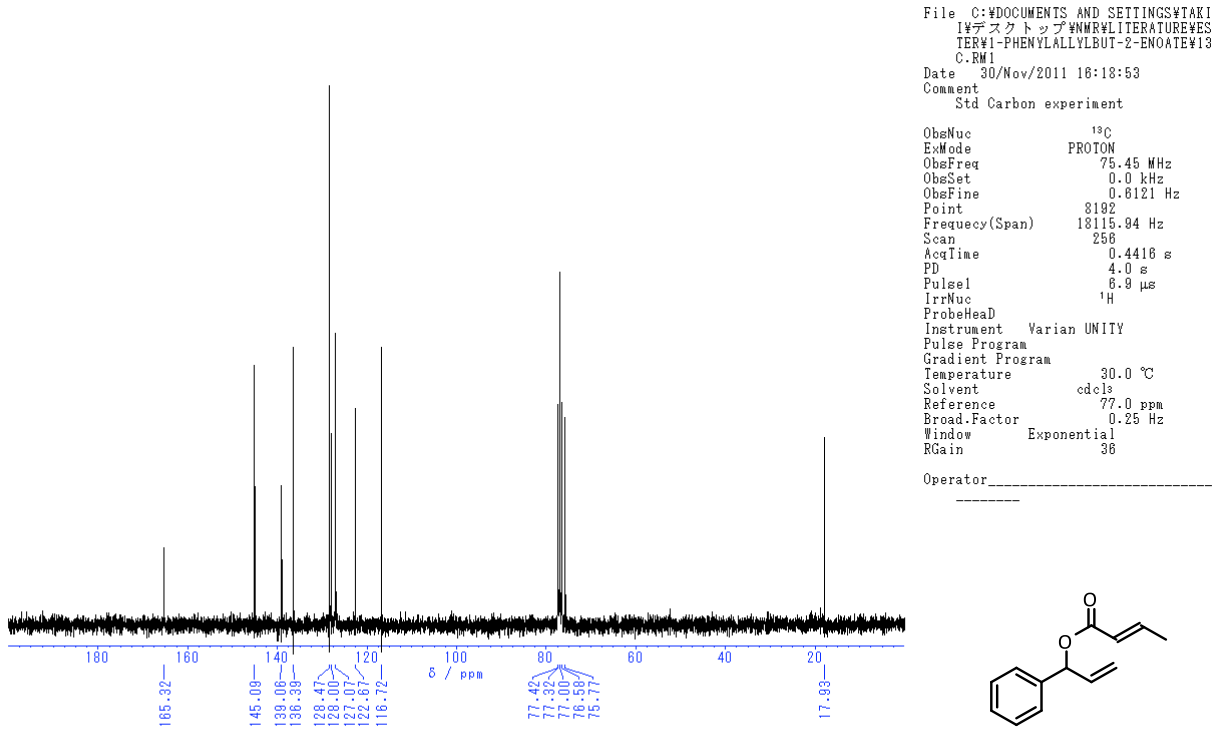
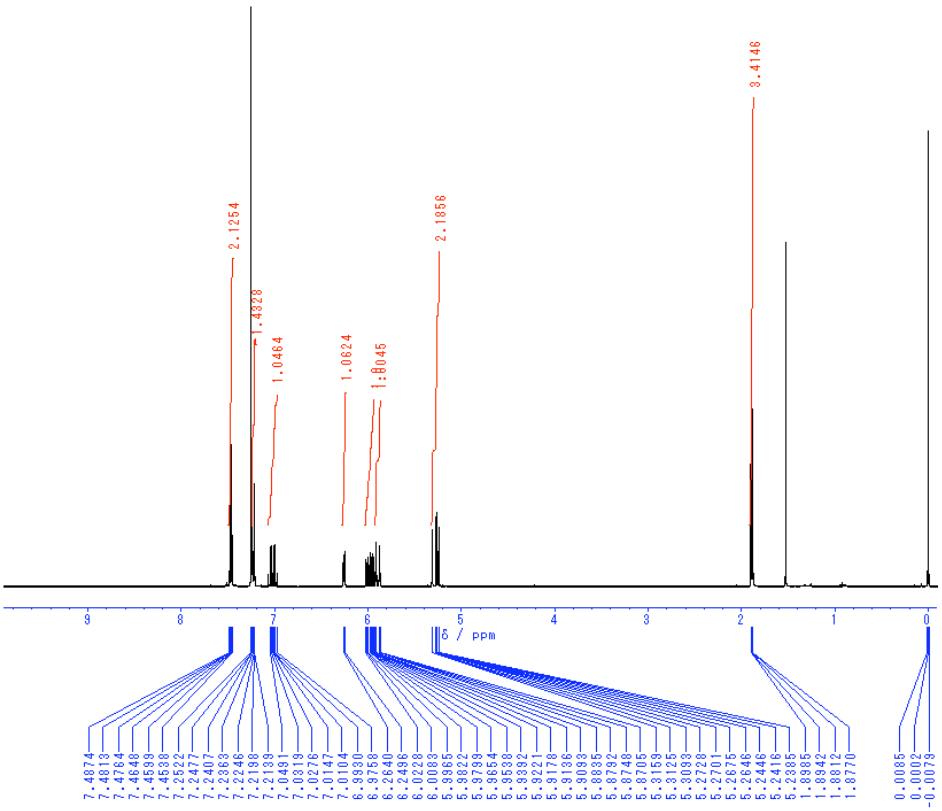
NMR Spectra

(*E*)-1-phenylallyl but-2-enoate (4ab)





(*E*)-1-(4-bromophenyl)allyl but-2-enoate (4bb).

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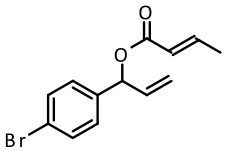

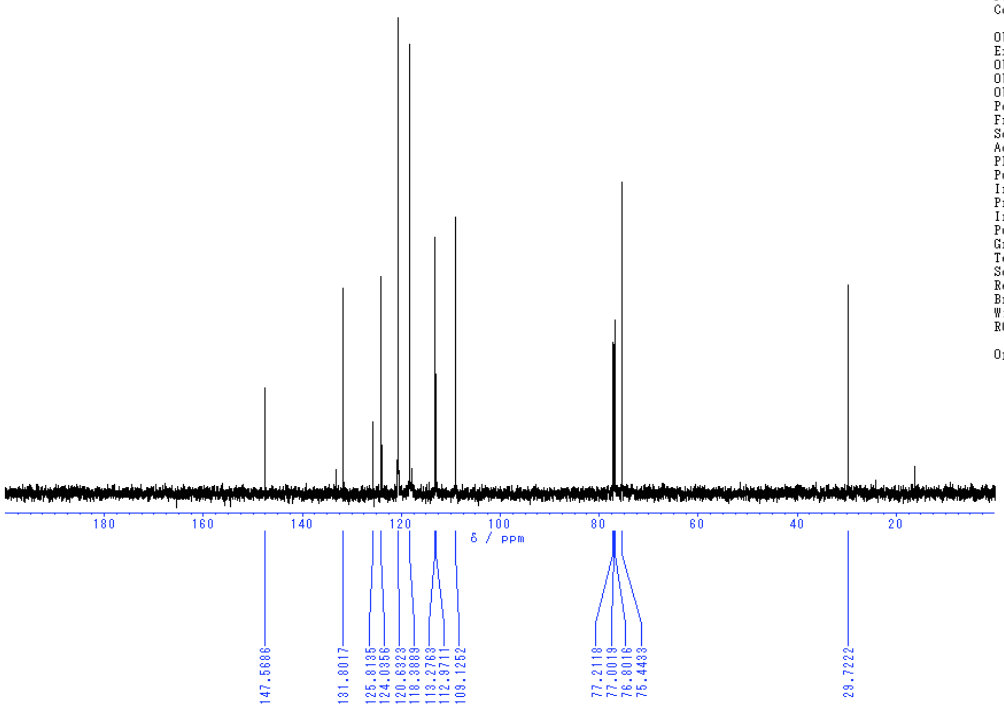


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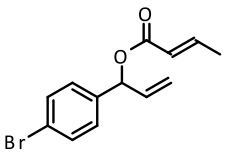
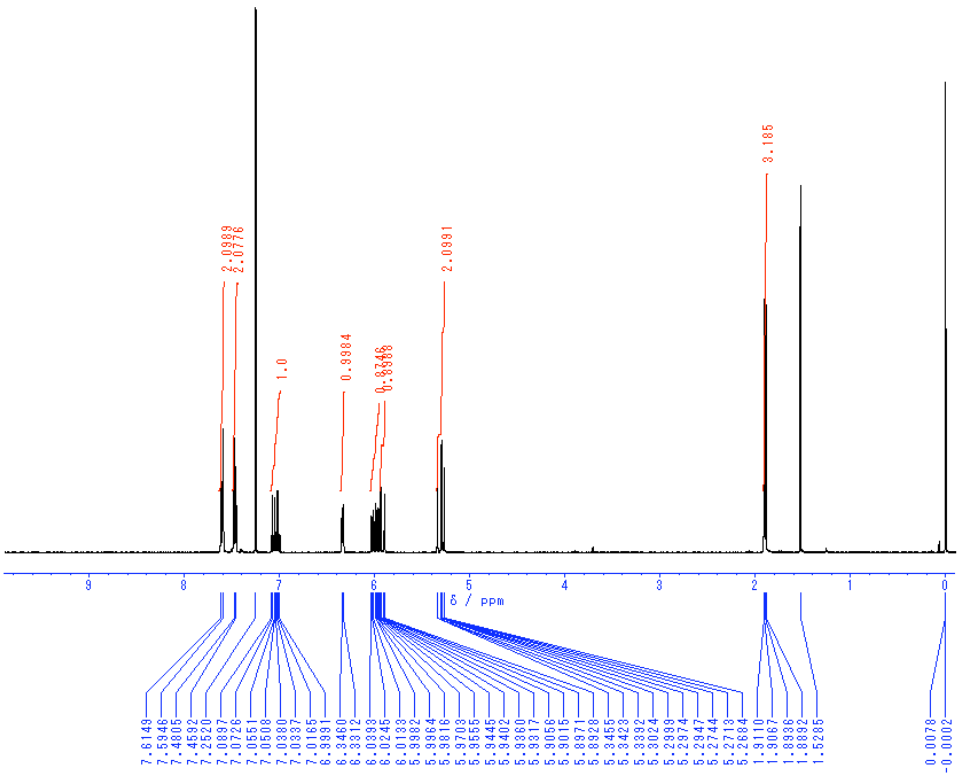


(*E*)-1-(4-trifluoromethylphenyl)allyl but-2-enoate (**4cb**).

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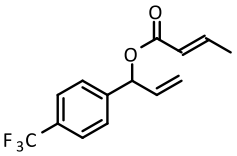

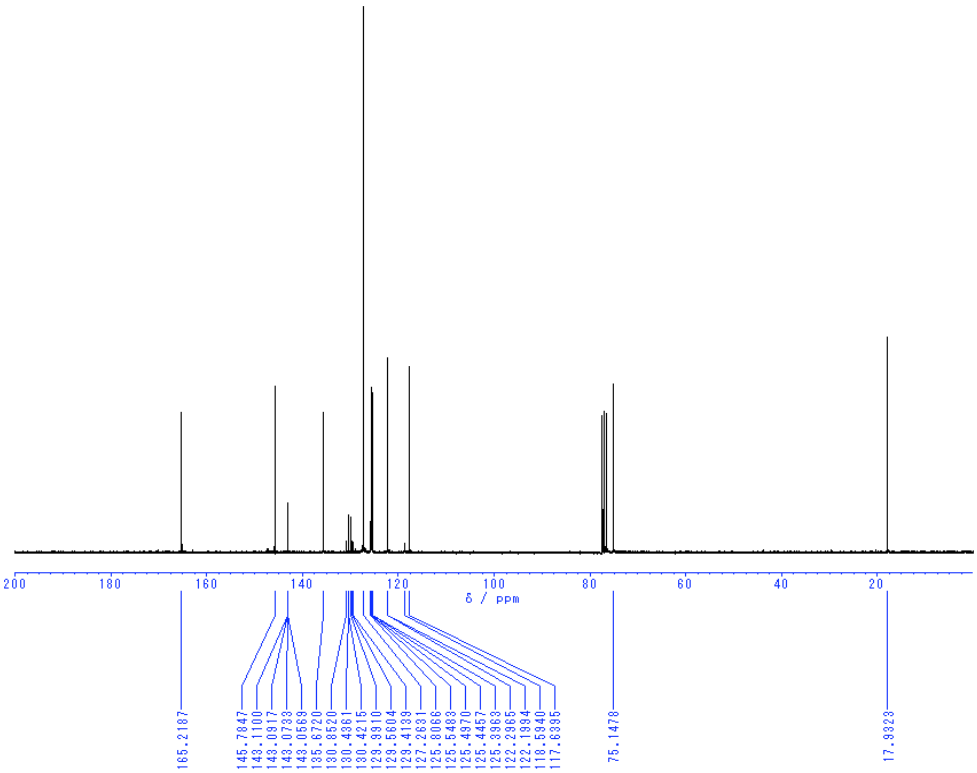


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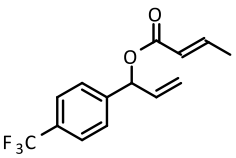
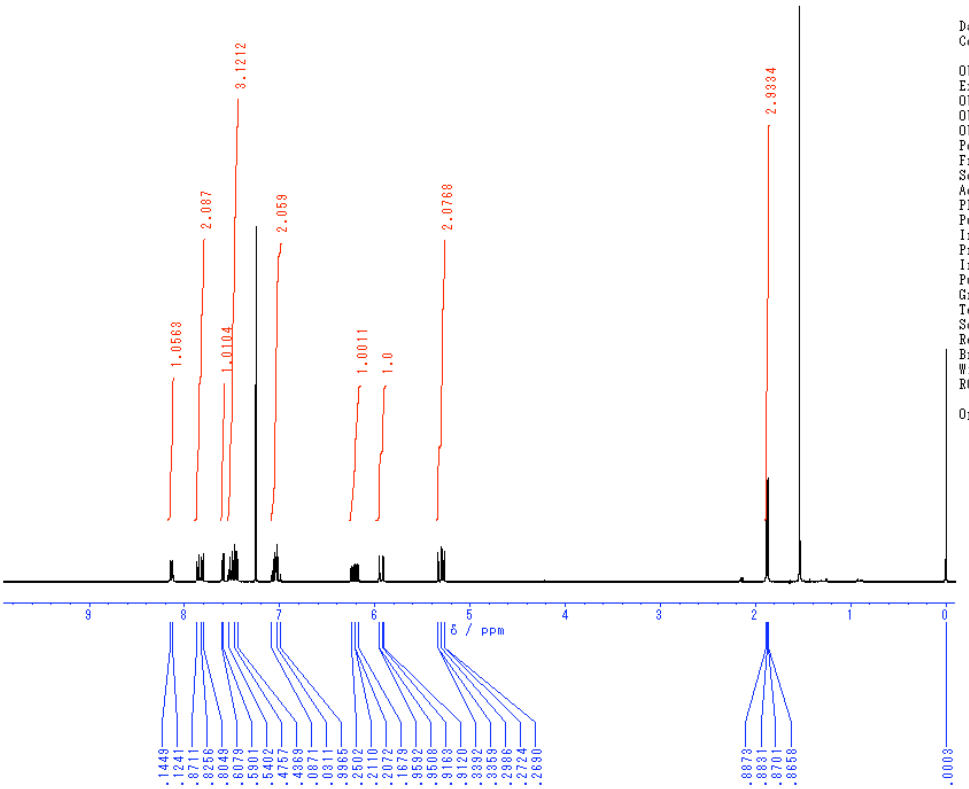
File  C:¥DOCUMENTS AND SETTINGS¥T
    AKII¥デスクトップ¥NMR¥LITERAT
    URE¥ESTER¥(E)-1-(4-(TRIFLUORO
    METHYL)PHENYL)ALLYL BUT-2-ENO
    ATE¥13C.RM1
Date    01/Jul/2011 00:01:38
Comment
    Std Carbon experiment

ObsNuc                 13C
ExMode              PROTON
ObsFreq             75.45 MHz
ObsSet               -1.0 kHz
ObsFine           998.4988 Hz
Point               65536
Frequecy(Span)    18115.94 Hz
Scan                 4096
AcqTime               2.0 s
PD                    4.0 s
Pulse1                6.9 µs
IrrNuc                  1H
ProbeHeaD
Instrument    Varian UNITY
Pulse Program
Gradient Program
Temperature          30.0 ℃
Solvent             cdcl3
Reference           76.9982 ppm
Broad.Factor         0.1382 Hz
Window       Exponential
RGain                   36

Operator_____________________
    __________
```

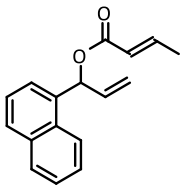


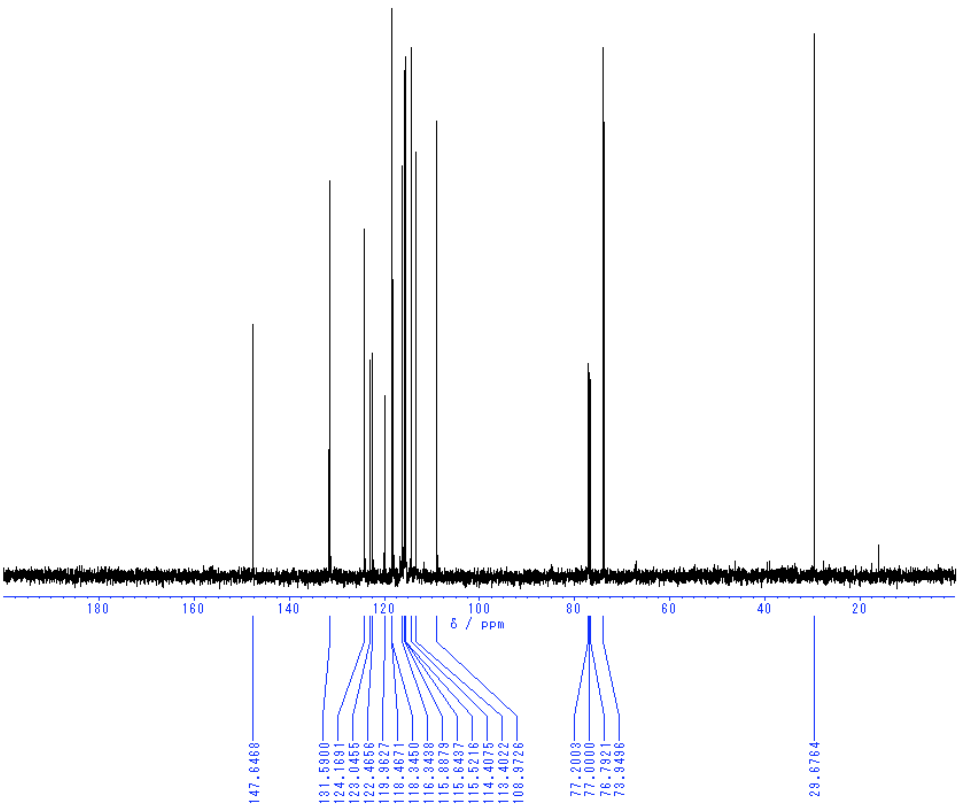
(*E*)-1-(naphthalene-1-yl)allyl but-2-enoate (**4db**).



File C:¥DOCUMENTS AND SETTINGS¥TAKII¥デスクトップ¥NMR¥LITERATURE¥ESTER¥(E)-1-(NAPHTHALEN-1-YL)ALLYL BUT-2-ENOATE¥1H.RM1
Date 01/Oct/2010 13:15:32
Comment
101001-1
ObsNuc 1H
ExMode NON
ObsFreq 399.65 MHz
ObsSet 124.0 kHz
ObsFine 10500.0 Hz
Point 32768
Frequecy(Span) 7993.6 Hz
Scan 4
AcqTime 4.0993 s
PD 2.901 s
Pulse1 5.7 μs
IrrNuc 1H
ProbeHeaD
Instrument ALICE
Pulse Program
Gradient Program
Temperature 30.0 ℃
Solvent CDCL3
Reference 0.0000 ppm
Broad.Factor 0.122 Hz
Window Exponential
RGain 23

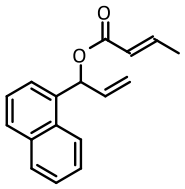
Operator_____



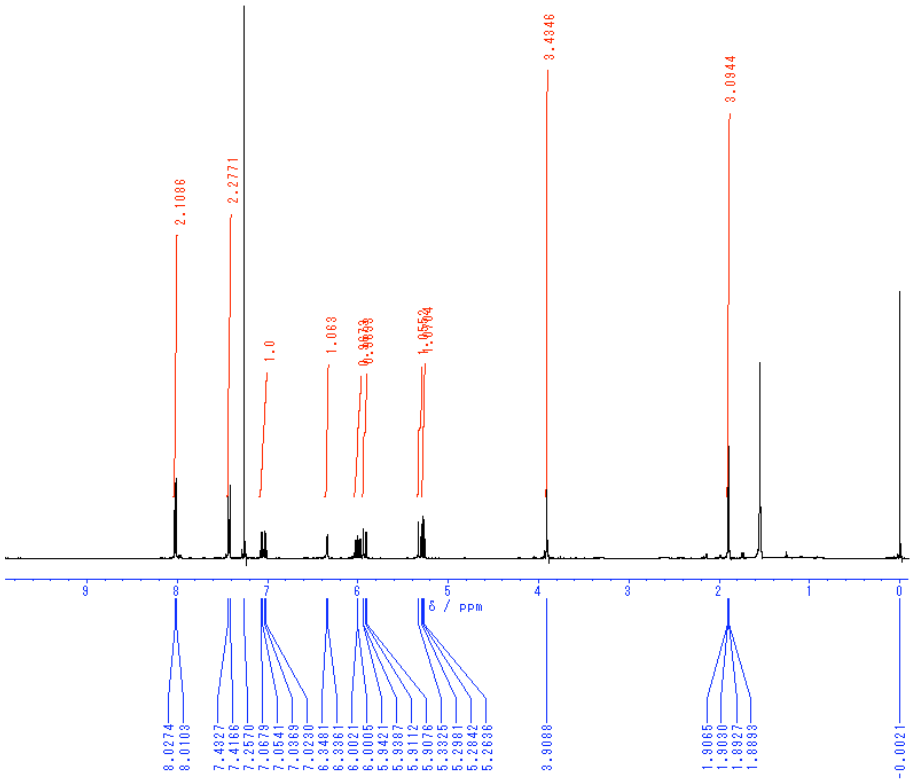


File C:¥DOCUMENTS AND SETTINGS¥TAKII¥デスクトップ¥NMR¥LITERATURE¥ESTER¥(E)-1-(NAPHTHALEN-1-YL)ALLYL BUT-2-ENOATE¥13C.RM1
Date 10/Oct/2011 22:11:07
Comment
1
ObsNuc 13C
ExMode single_pulse_dec
ObsFreq 125.77 MHz
ObsSet -5.0 kHz
ObsFine 301.0403 Hz
Point 16384
Frequecy(Span) 31443.66 Hz
Scan 128
AcqTime 0.5211 s
PD 2.0 s
Pulse1 5.0 μs
IrrNuc 1H
ProbeHeaD
Instrument ECA 500
Pulse Program
Gradient Program
Temperature 30.0 ℃
Solvent CHLOROFORM-D
Reference 77.0 ppm
Broad.Factor 0.25 Hz
Window Exponential
RGain 58

Operator_____

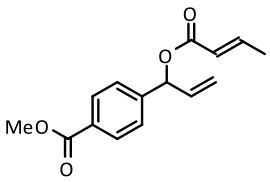


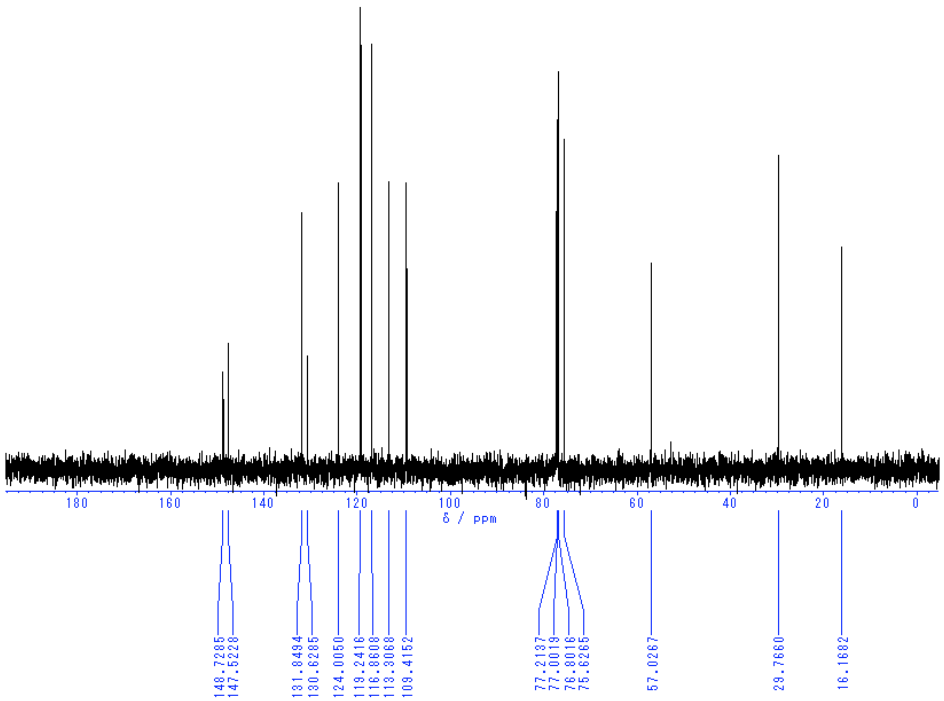
(*E*)-methyl 4-(1-(but-2-enyloxy)allyl)benzoate (4eb)




```
File  C:¥DOCUMENTS AND SETTINGS¥TAKII¥
    デスクトップ¥NMR¥LITERATURE¥ESTER¥(
    E)-METHYL 4-(1-(BUT-2-ENOYLOXY)ALLY
    L)BENZOATE¥1H.RM1
Date    11/Oct/2011 09:47:02
Comment
      1
ObsNuc                  1H
ExMode    single_pulse.ex2
ObsFreq               500.18 MHz
ObsSet                  0.0 kHz
ObsFine               -88.4525 Hz
Point                 32768
Frequecy(Span)        9384.385 Hz
Scan                    16
AcqTime                 3.4918 s
PD                      5.0 s
Pulse1                  3.8 µs
IrrNuc                  1H
ProbeHeaD
Instrument        ECA 500
Pulse Program
Gradient Program
Temperature             30.0 ℃
Solvent          CHLOROFO
Reference               5.0042 ppm
Broad.Factor            0.1432 Hz
Window        Exponential
RGain                   48

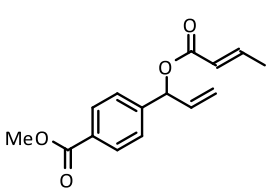
Operator_________________________________
     -----
```



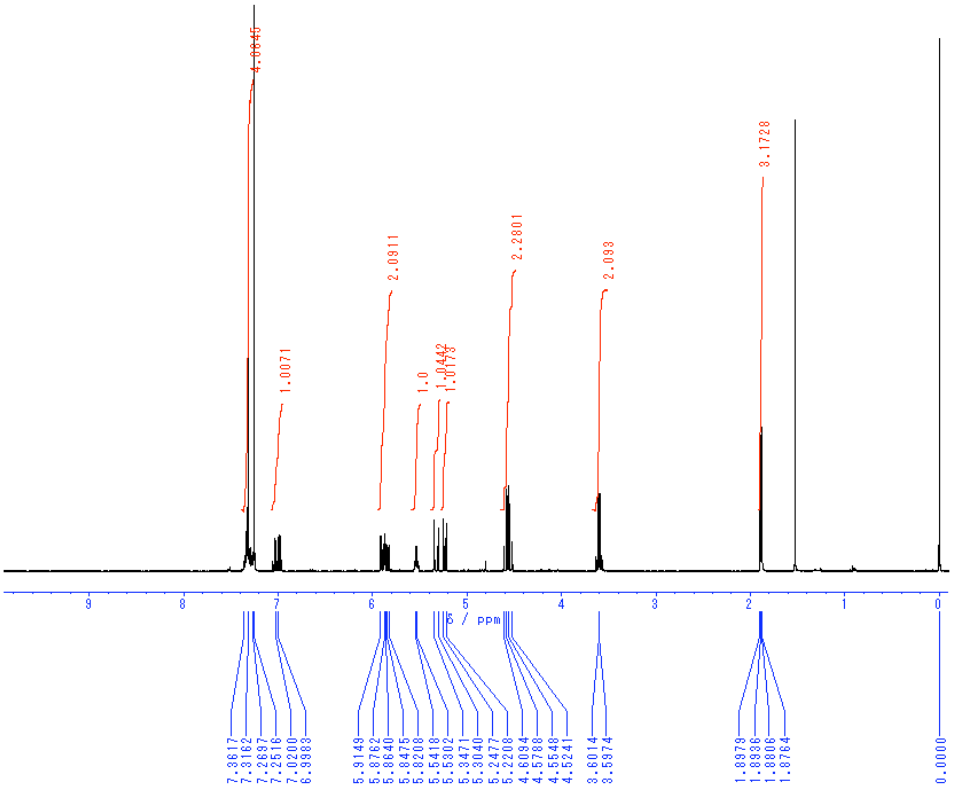



```
File  C:¥DOCUMENTS AND SETTINGS¥TAK
    II¥デスクトップ¥NMR¥LITERATURE¥
    ESTER¥(E)-METHYL 4-(1-(BUT-2-EN
    OYLOXY)ALLYL)BENZOATE¥13C.RM1
Date    10/Oct/2011 22:25:03
Comment
      1
ObsNuc                  13C
ExMode    single_pulse_dec
ObsFreq               125.77 MHz
ObsSet                 -5.0 kHz
ObsFine               301.0403 Hz
Point                 16384
Frequecy(Span)        31443.66 Hz
Scan                    64
AcqTime                 0.5211 s
PD                      2.0 s
Pulse1                  5.0 µs
IrrNuc                  1H
ProbeHeaD
Instrument        ECA 500
Pulse Program
Gradient Program
Temperature             30.0 ℃
Solvent          CHLOROFO
Reference              76.9381 ppm
Broad.Factor            0.9596 Hz
Window        Exponential
RGain                   58

Operator_________________________________
     ---------
```

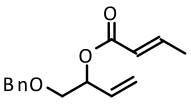


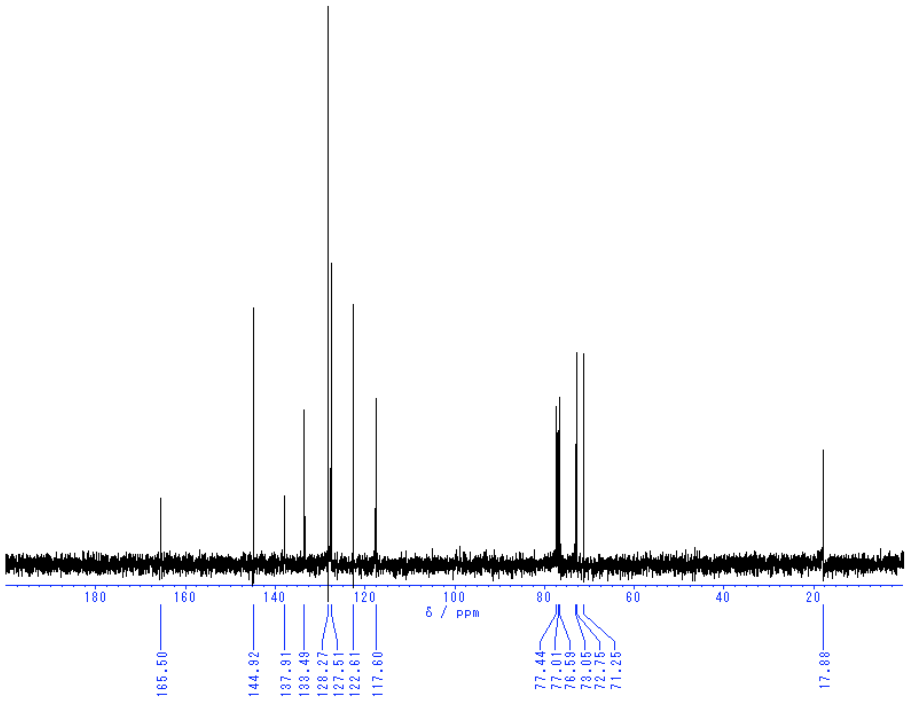
(*E*)-1-(benzyloxy)but-3-en-2-yl but-2-enoate (4fb).




```
File  C:¥DOCUMENTS AND SETTINGS¥T
    AKII¥デスクトップ¥NMR¥LITERAT
    URE¥ESTER¥BNO¥1H.RM1
Date   13/Nov/2010 19:36:00
Comment
    101113-2
ObsNuc                  1H
ExMode                NON
ObsFreq           399.65 MHz
ObsSet             124.0 kHz
ObsFine          10500.0 Hz
Point               32768
Frequecy(Span)     7993.6 Hz
Scan                    8
AcqTime            4.0993 s
PD                 2.901 s
Pulse1             5.7 μs
IrrNuc                  1H
ProbeHeaD
Instrument          ALICE
Pulse Program
Gradient Program
Temperature          30.4 ℃
Solvent             CDCl3
Reference            0.0 ppm
Broad.Factor       0.122 Hz
Window       Exponential
RGain                  23

Operator_____________________
    __________
```

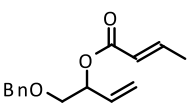




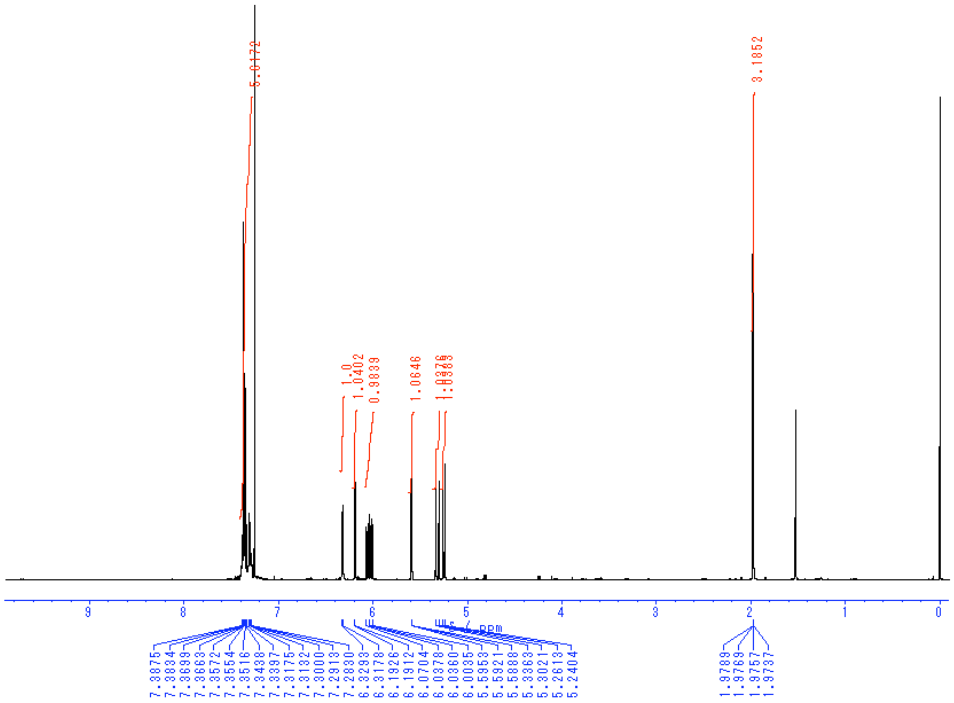

```
File  C:¥DOCUMENTS AND SETTINGS¥TAKII¥
    デスクトップ¥NMR¥LITERATURE¥ESTER¥
    BNO¥13C.RM1
Date   14/Dec/2011 18:42:49
Comment
    Std Carbon experiment

ObsNuc                 13C
ExMode             PROTON
ObsFreq          75.45 MHz
ObsSet             0.0 kHz
ObsFine          0.6121 Hz
Point               8192
Frequecy(Span)   18115.94 Hz
Scan                  64
AcqTime            0.4416 s
PD                 4.0 s
Pulse1             6.8 μs
IrrNuc                  1H
ProbeHeaD
Instrument   Varian UNITY
Pulse Program
Gradient Program
Temperature          30.0 ℃
Solvent            cdcl3
Reference            77.0 ppm
Broad.Factor       0.25 Hz
Window       Exponential
RGain                  36

Operator_____________________
    ______
```

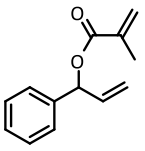


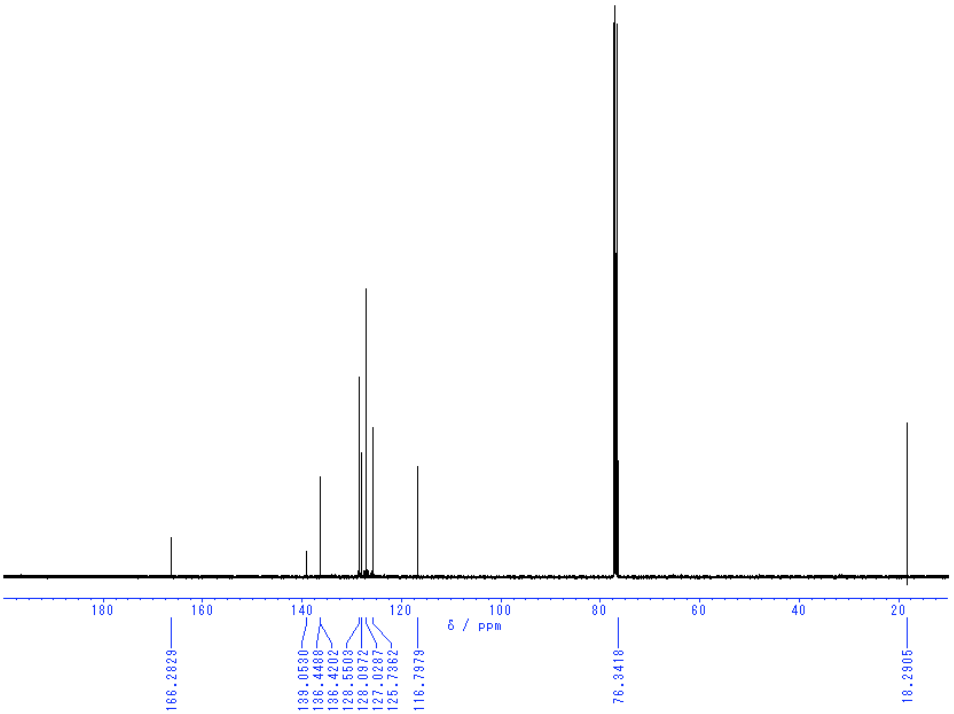
(*E*)-1-phenylallyl methacrylate (**4ac**).




```
File  C:¥DOCUMENTS AND SETTINGS¥T
    AKII¥デスクトップ¥NMR¥LITERAT
    URE¥ESTER¥1-PHENYLALLYL METHA
    CRYLATE¥1H.RM1
Date   26/Apr/2011 23:54:39
Comment
    1
ObsNuc                  1H
ExMode   single_pulse.ex2
ObsFreq              500.16 MHz
ObsSet                 0.0 kHz
ObsFine              -88.4525 Hz
Point                65536
Frequecy(Span)     9384.385 Hz
Scan                      8
AcqTime                6.9835 s
PD                     5.0 s
Pulse1                 6.25 µs
IrrNuc                 1H
ProbeHeaD
Instrument       ECA 500
Pulse Program
Gradient Program
Temperature           30.0 ℃
Solvent         CHLOROFO
Reference             -0.0001 ppm
Broad.Factor           0.0716 Hz
Window       Exponential
RGain                  48

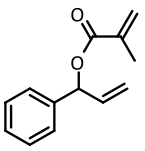
Operator_____________________________
    __________
```



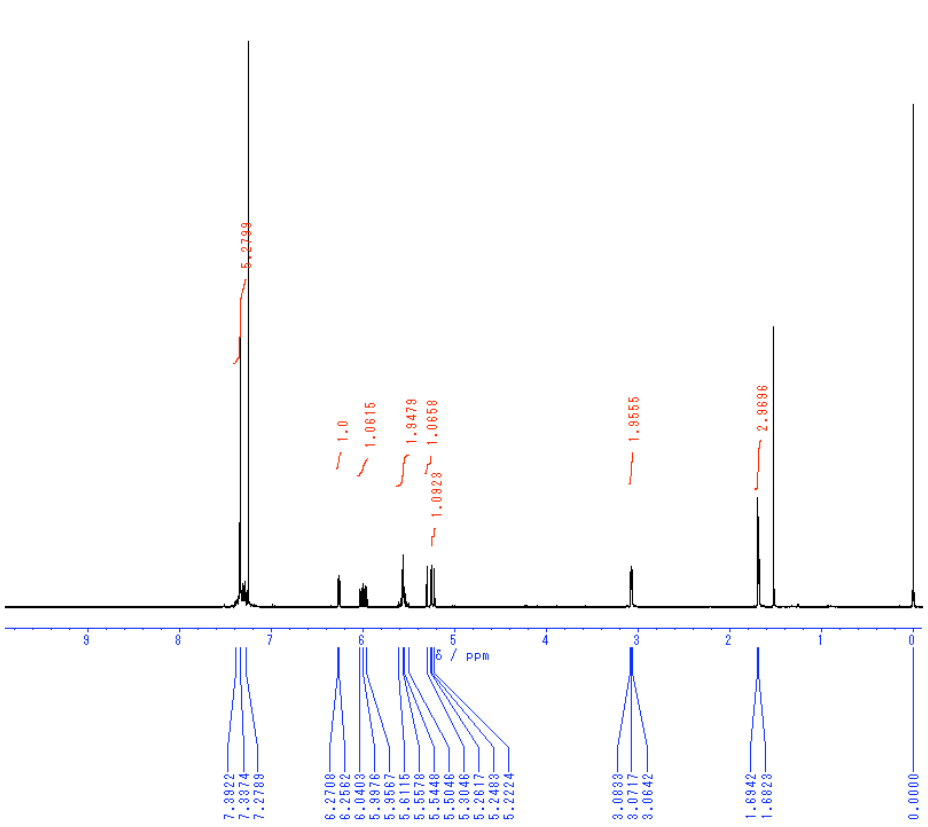



```
File  C:¥DOCUMENTS AND SETTINGS¥TAK
    II¥デスクトップ¥NMR¥LITERATURE¥
    ESTER¥1-PHENYLALLYL METHACRYLAT
    E¥13C.RM1
Date   27/Apr/2011 08:06:00
Comment
    1
ObsNuc                 13C
ExMode   single_pulse_dec
ObsFreq              125.77 MHz
ObsSet                -5.0 kHz
ObsFine            301.0403 Hz
Point                65536
Frequecy(Span)     33808.18 Hz
Scan                  7967
AcqTime                1.6672 s
PD                     2.0 s
Pulse1                 4.55 µs
IrrNuc                 1H
ProbeHeaD
Instrument       ECA 500
Pulse Program
Gradient Program
Temperature           30.0 ℃
Solvent         CHLOROFO
Reference             77.0 ppm
Broad.Factor           0.2999 Hz
Window       Exponential
RGain                  58

Operator_____________________________
    _________
```

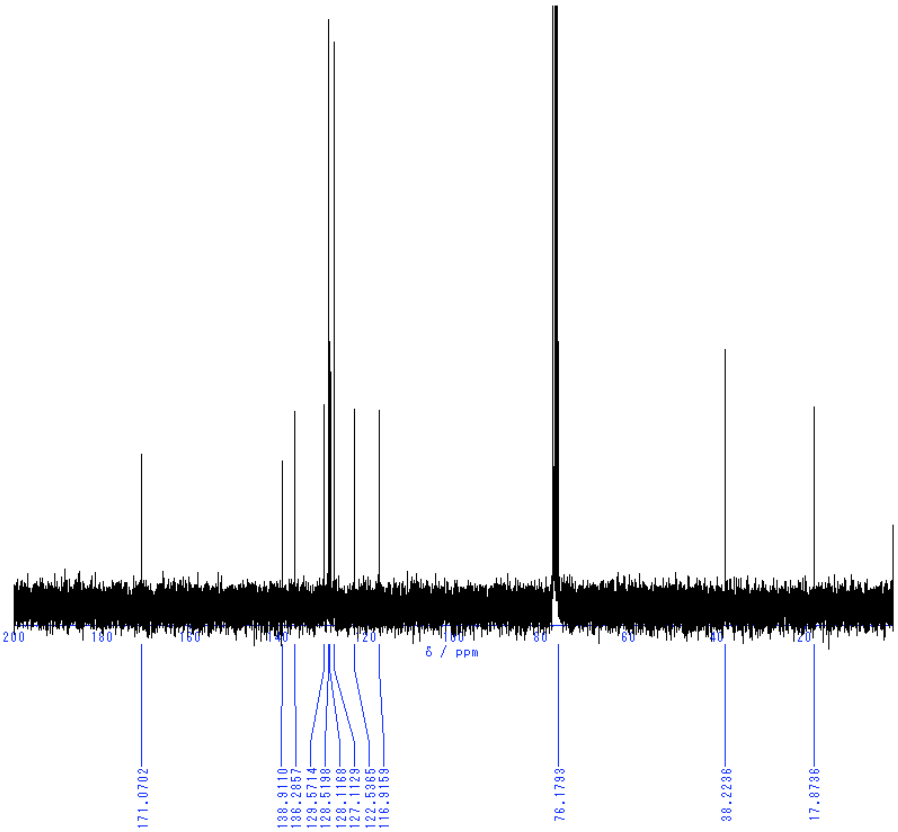


(*E*)-1-phenylallyl pent-3-enoate (**4ad**).




```
File  C:¥DOCUMENTS AND SETTINGS¥TAKI
    I¥デスクトップ¥NMR¥LITERATURE¥ES
    TER¥1-PHENYLALLYLPENT-3-ENOATE¥1
    H.RM1
Date    28/Jun/2010 09:55:16
Comment
    100628-2
ObsNuc                  1H
ExMode                 NON
ObsFreq              399.65 MHz
ObsSet               124.0 kHz
ObsFine             10500.0 Hz
Point                 32768
Frequecy(Span)       7993.6 Hz
Scan                     8
AcqTime                4.0993 s
PD                     2.901 s
Pulse1                 5.7 μs
IrrNuc                 1H
ProbeHeaD
Instrument           ALICE
Pulse Program
Gradient Program
Temperature           30.4 ℃
Solvent              CDCL3
Reference              0.0 ppm
Broad.Factor           0.122 Hz
Window       Exponential
RGain                  23

Operator_______________________________
    ________
```

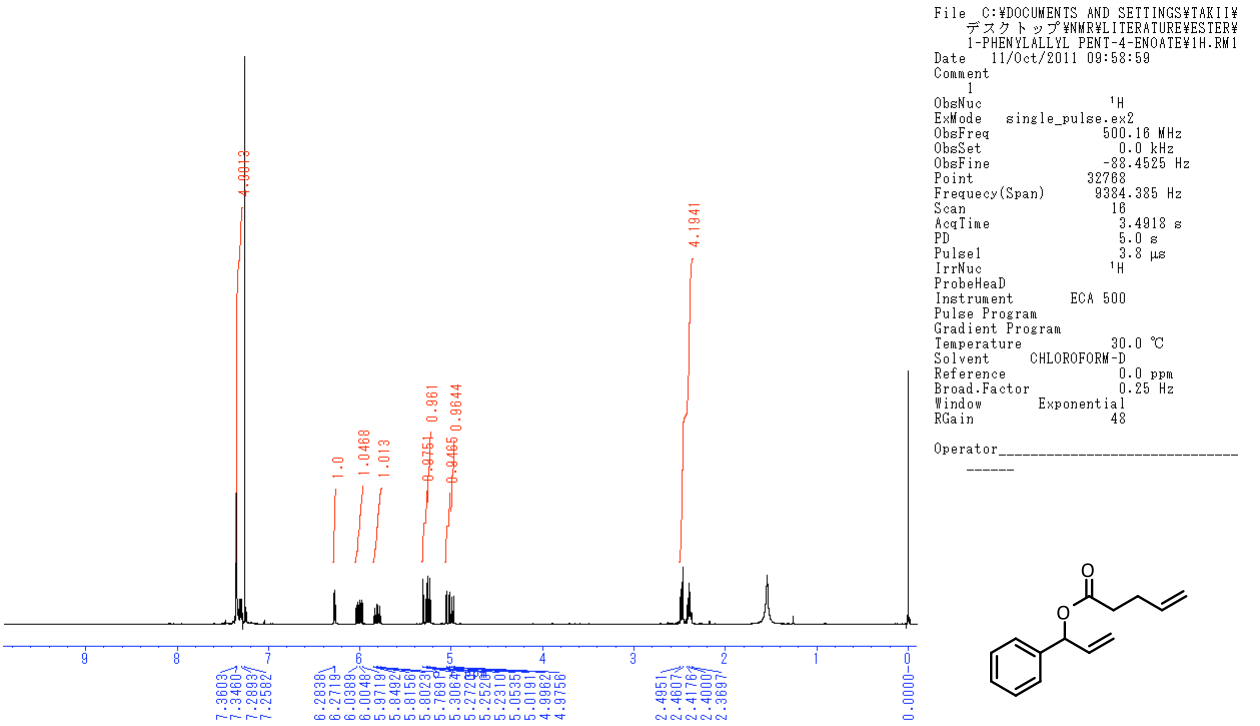


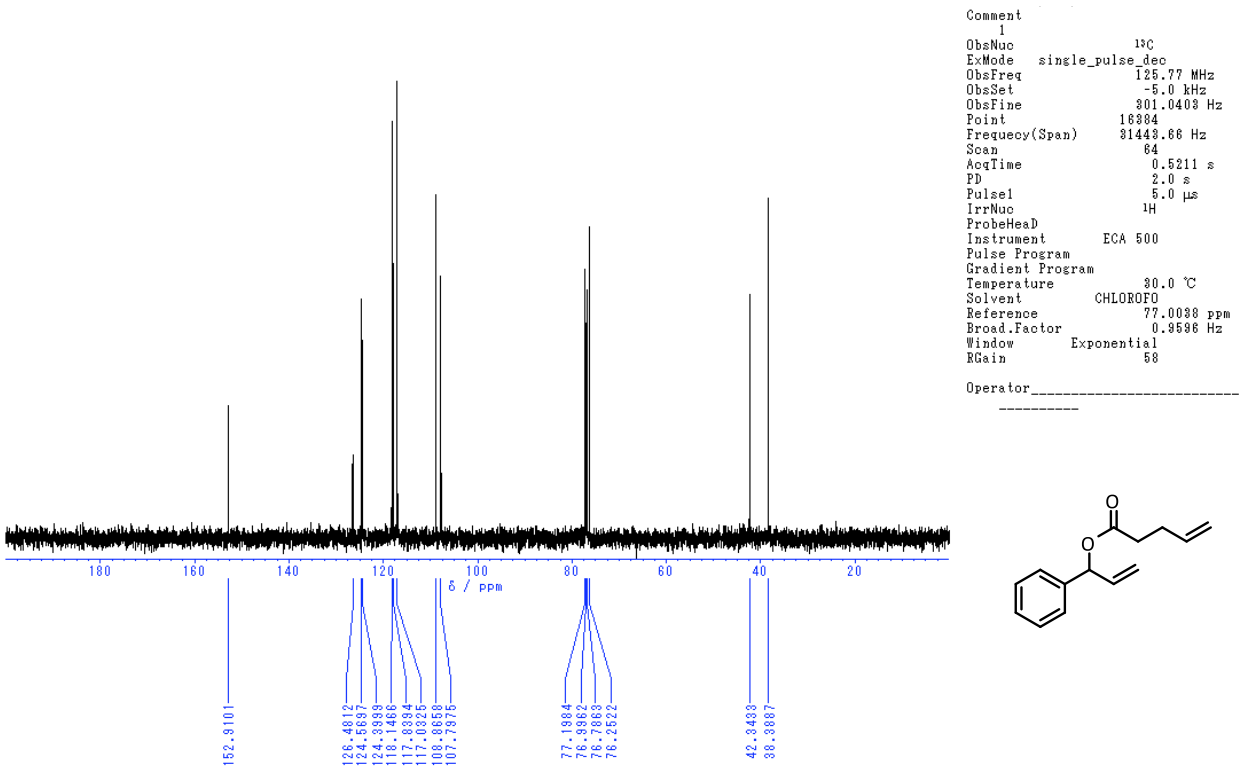

```
File  C:¥DOCUMENTS AND SETTINGS¥TAKII¥デ
    スクトップ¥NMR¥LITERATURE¥ESTER¥1-PH
    ENYLALLYLPENT-3-ENOATE¥13C-2.RM1
Date    21/Jul/2011 01:51:13
Comment
    Std Carbon experiment

ObsNuc                 13C
ExMode              PROTON
ObsFreq              75.45 MHz
ObsSet               -1.0 kHz
ObsFine            998.4988 Hz
Point                65536
Frequecy(Span)     18115.94 Hz
Scan                  4400
AcqTime                2.0 s
PD                     4.0 s
Pulse1                 6.9 μs
IrrNuc                 1H
ProbeHeaD
Instrument    Varian UNITY
Pulse Program
Gradient Program
Temperature           30.0 ℃
Solvent             cdcl3
Reference             77.0 ppm
Broad.Factor          0.1382 Hz
Window       Exponential
RGain                  36

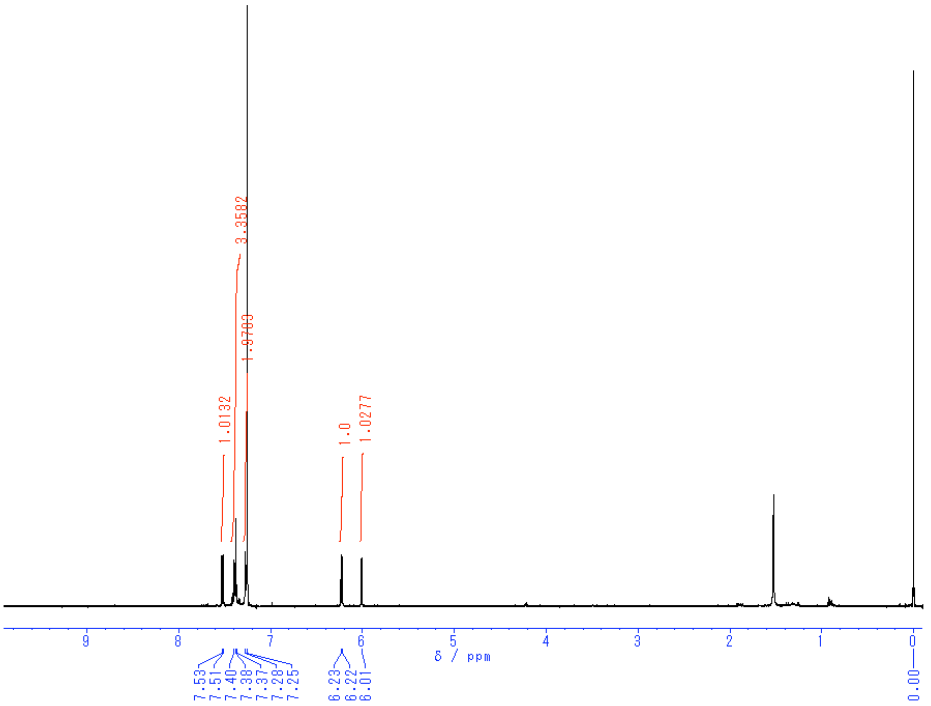
Operator_______________________________
    ____
```

1-phenylallyl pent-4-enoate (**4ae**).



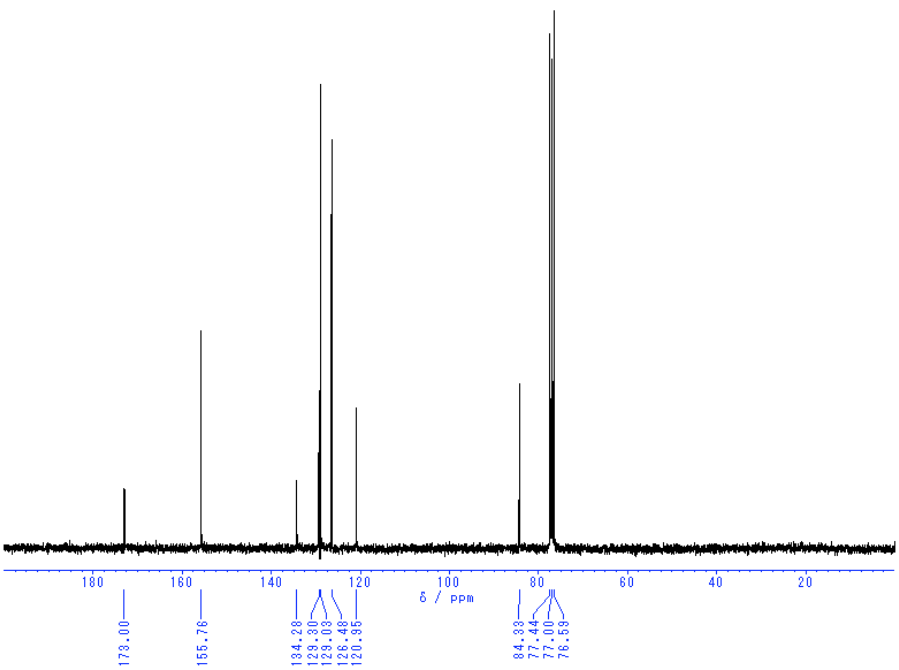


5-phenylfuran-2(5*H*)-one (6ab).




```
File  C:¥DOCUMENTS AND SETTINGS¥TAKI
    I¥デスクトップ¥NMR¥LITERATURE¥LA
    CTONE¥5-PHENYLFURAN-2(5H)-ONE¥1H
    .RM1
Date    05/Jan/2011 13:41:19
Comment
    110105-2
ObsNuc                  1H
ExMode                 NON
ObsFreq            399.65 MHz
ObsSet              124.0 kHz
ObsFine            10500.0 Hz
Point                 32768
Frequecy(Span)      7993.6 Hz
Scan                      4
AcqTime              4.0993 s
PD                   2.901 s
Pulse1               5.7 µs
IrrNuc                  1H
ProbeHeaD
Instrument           ALICE
Pulse Program
Gradient Program
Temperature          31.2 ℃
Solvent             CDCL3
Reference             0.0 ppm
Broad.Factor          0.122 Hz
Window       Exponential
RGain                  25

Operator_______________________________
    --------
```

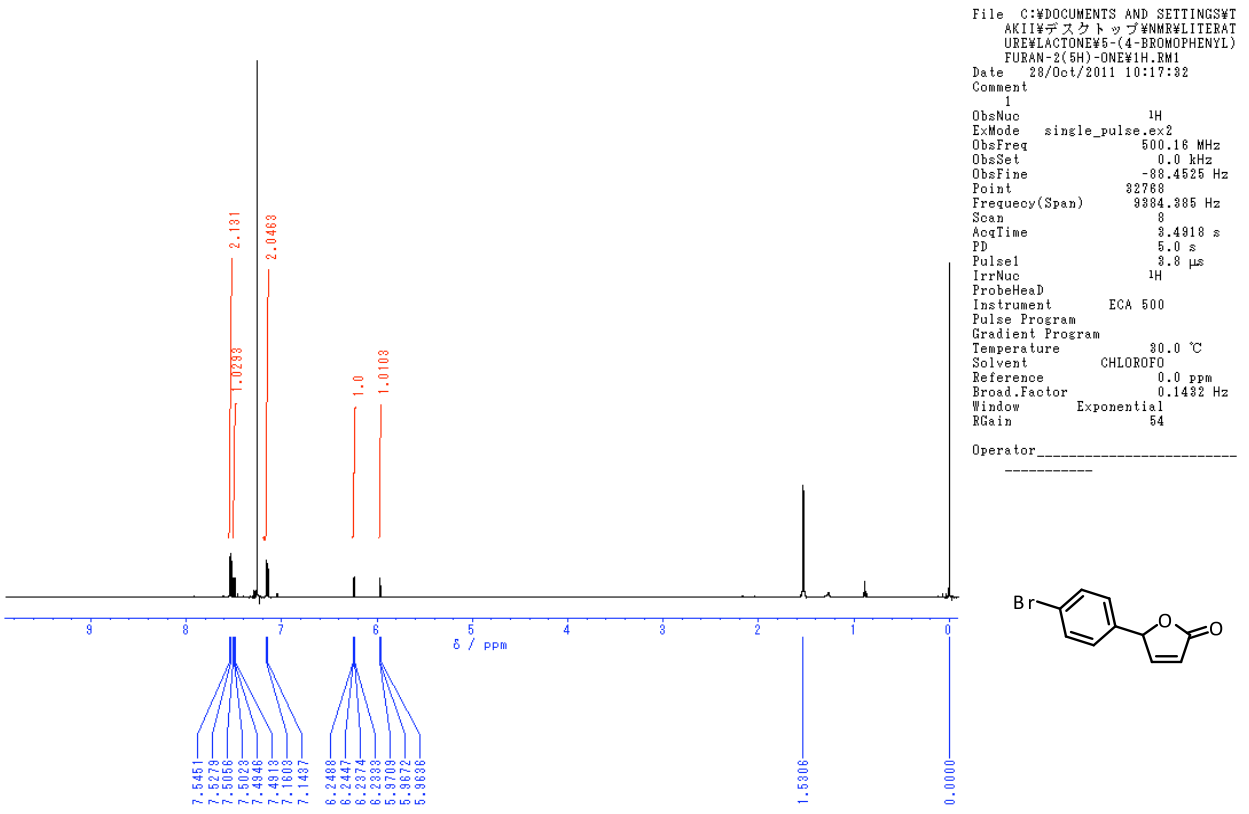


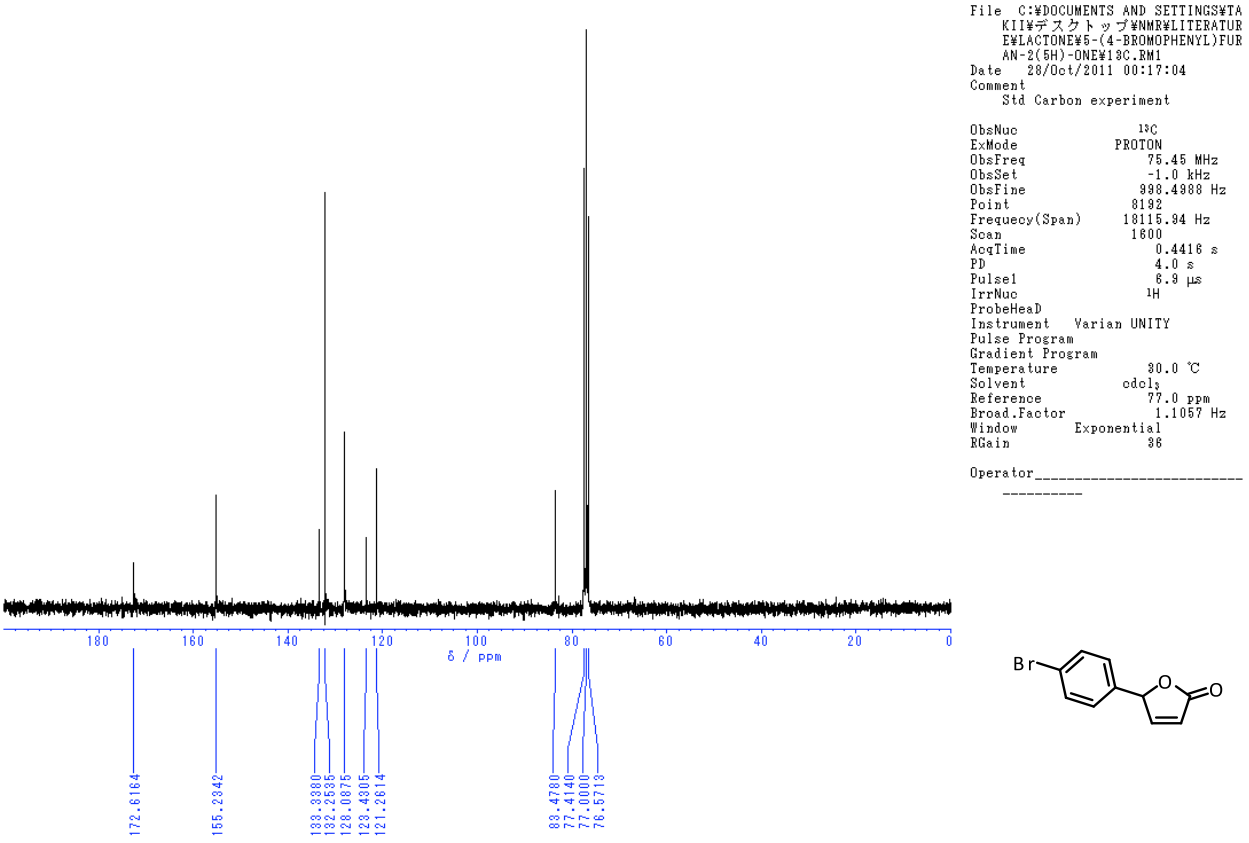

```
File  C:¥DOCUMENTS AND SETTINGS¥TAKII¥
    デスクトップ¥NMR¥LITERATURE¥LACTON
    E¥5-PHENYLFURAN-2(5H)-ONE¥13C.RM1
Date    22/Dec/2011 01:48:07
Comment
    Std Carbon experiment

ObsNuc                 13C
ExMode             PROTON
ObsFreq            75.45 MHz
ObsSet               0.0 kHz
ObsFine              0.6121 Hz
Point                8192
Frequecy(Span)     18115.94 Hz
Scan                 2260
AcqTime              0.4416 s
PD                   4.0 s
Pulse1               6.9 µs
IrrNuc                  1H
ProbeHeaD
Instrument   Varian UNITY
Pulse Program
Gradient Program
Temperature          30.0 ℃
Solvent             cdcl3
Reference            77.0 ppm
Broad.Factor         0.25 Hz
Window       Exponential
RGain                  86

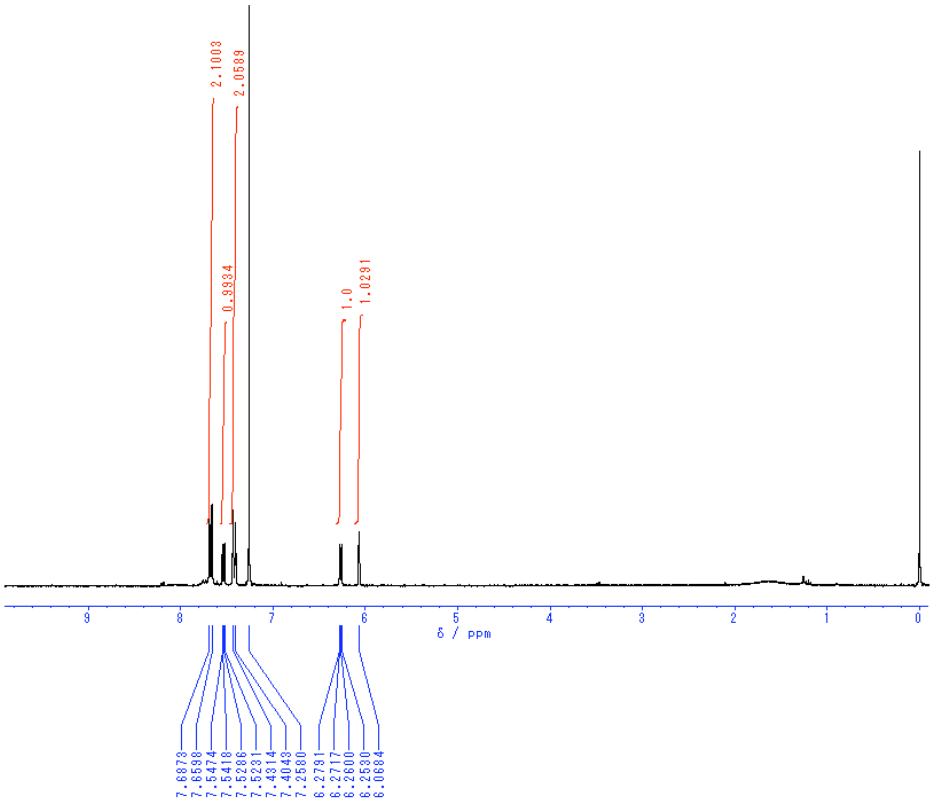
Operator_______________________________
    ------
```

5-(4-bromophenyl)furan-2(5*H*)-one (6bb).





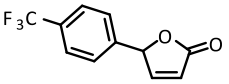
5-(4-trifluoromethylphenyl)furan-2(5*H*)-one (6cb).

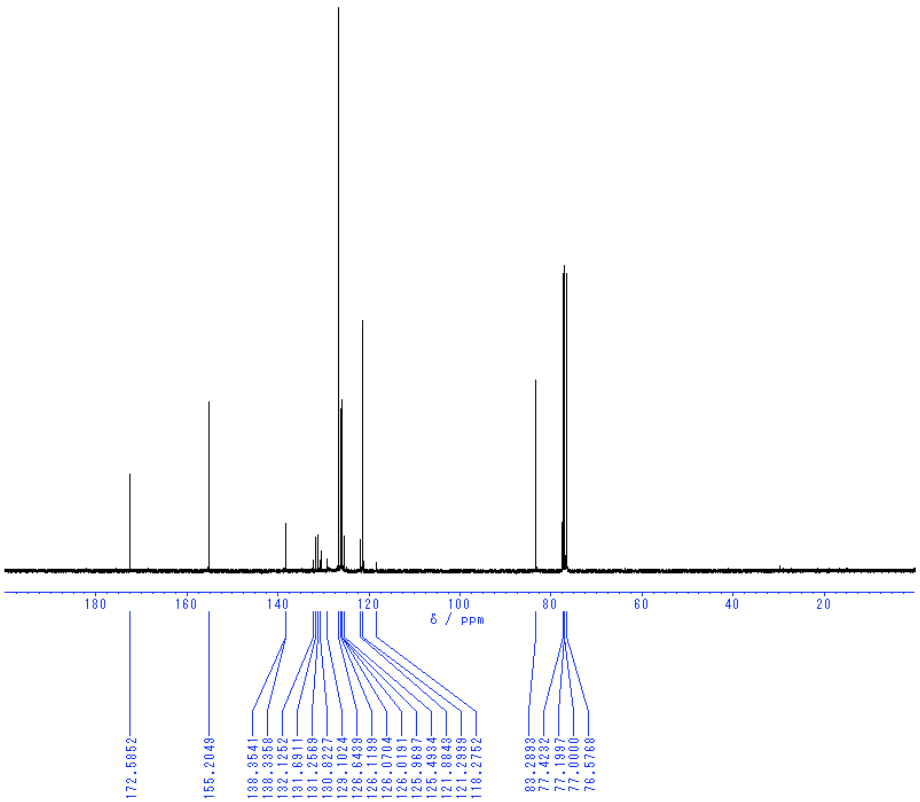



```
File  C:¥DOCUMENTS AND SETTINGS¥TAKI
    I¥デスクトップ¥NMR¥LITERATURE¥LA
    CTONE¥5-(4-TRIFLUOROMETHYLPHENYL
    )FURAN-2(5H)-ONE¥1H.RM1
Date   05/Jul/2011 17:40:49
Comment
    Std Proton parameters

ObsNuc                  1H
ExMode                 NON
ObsFreq            300.04 MHz
ObsSet              -1.0 kHz
ObsFine            994.528 Hz
Point                16384
Frequecy(Span)     4803.074 Hz
Scan                     8
AcqTime              3.4095 s
PD                   6.5872 s
Pulse1               4.7 µs
IrrNuc                 13C
ProbeHeaD
Instrument    Varian UNITY
Pulse Program
Gradient Program
Temperature          30.0 ℃
Solvent             cdcl3
Reference           0.0004 ppm
Broad.Factor         0.1466 Hz
Window        Exponential
RGain                  38

Operator_______________________________
    --------
```

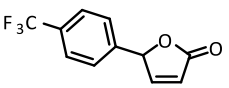




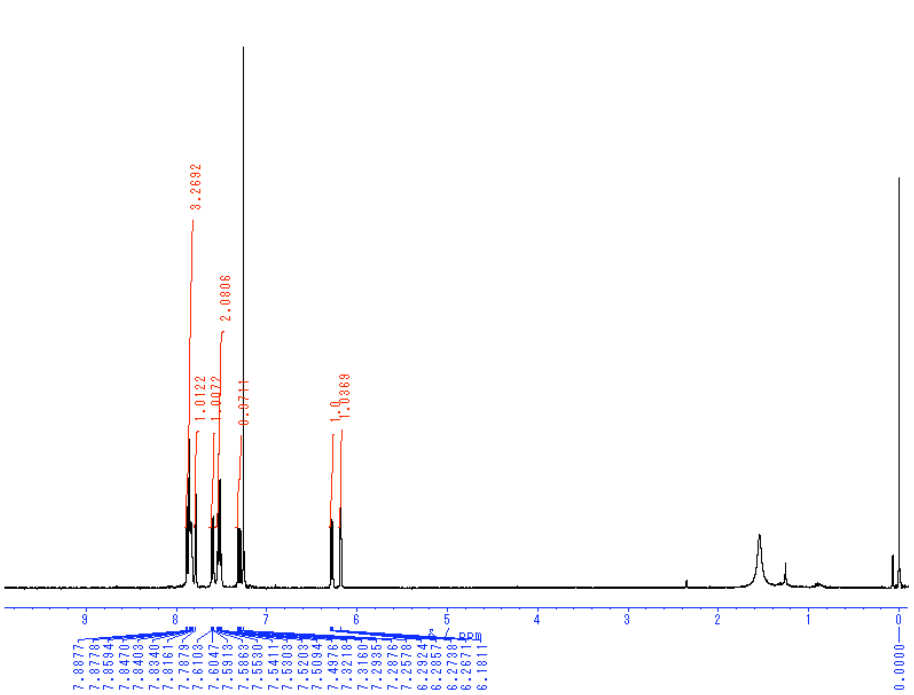

```
File  C:¥DOCUMENTS AND SETTINGS¥TAKII¥
    デスクトップ¥NMR¥LITERATURE¥LACTON
    E¥5-(4-TRIFLUOROMETHYLPHENYL)FURAN
    -2(5H)-ONE¥13C.RM1
Date   06/Jul/2011 22:28:05
Comment
    Std Carbon experiment

ObsNuc                 13C
ExMode             PROTON
ObsFreq            75.45 MHz
ObsSet              -1.0 kHz
ObsFine            998.4988 Hz
Point                65536
Frequecy(Span)     18115.94 Hz
Scan                  6400
AcqTime              2.0 s
PD                   4.0 s
Pulse1               6.9 µs
IrrNuc                 1H
ProbeHeaD
Instrument    Varian UNITY
Pulse Program
Gradient Program
Temperature          30.0 ℃
Solvent             cdcl3
Reference           77.0 ppm
Broad.Factor         0.1382 Hz
Window        Exponential
RGain                  36

Operator_______________________________
    ------
```



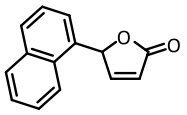
5-(naphthalene-1-yl)furan-2(5*H*)-one (6db).

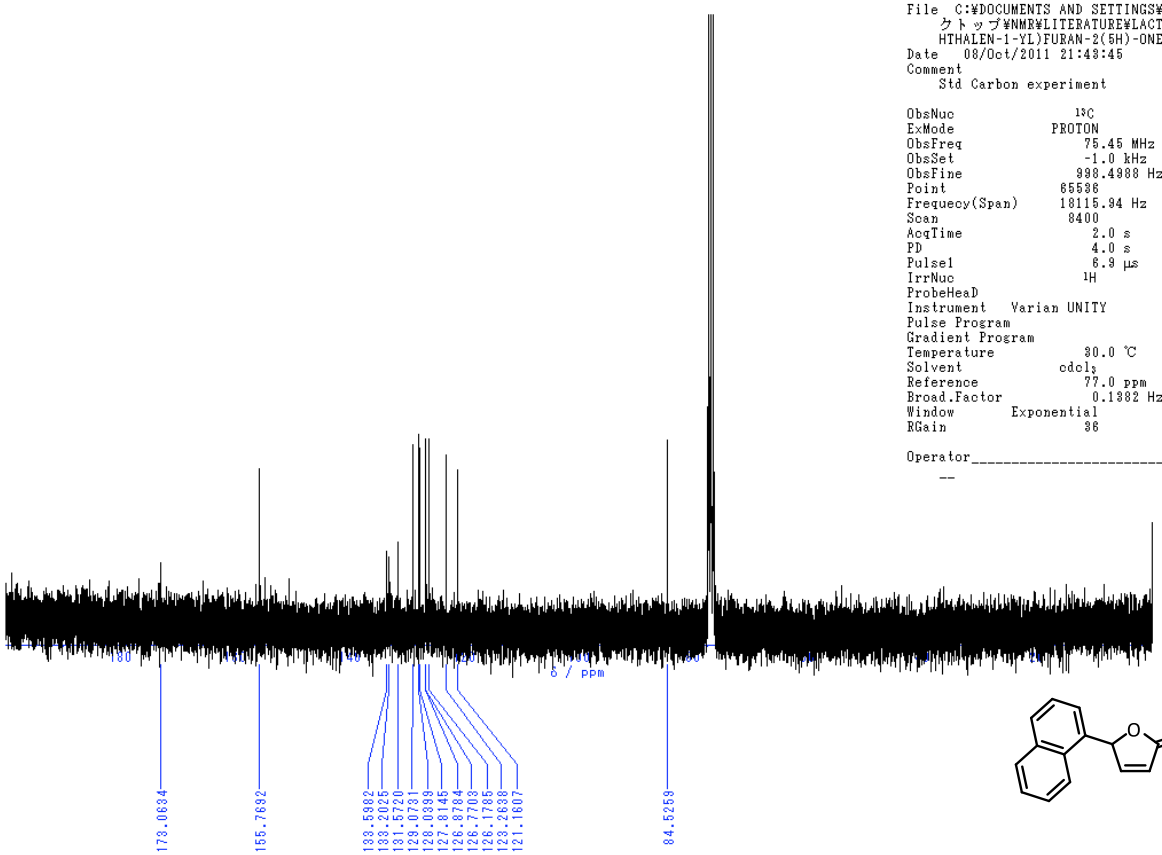


File C:¥DOCUMENTS AND SETTINGS¥TAKI I¥デスクトップ¥NMR¥LITERATURE¥LA CTONE¥5-(NAPHTHALEN-1-YL)FURAN-2 (5H)-ONE¥1H.RM1
Date 14/Jun/2011 15:29:11
Comment
Std Proton parameters

ObsNuc	1H
ExMode	NON
ObsFreq	300.04 MHz
ObsSet	-1.0 kHz
ObsFine	994.528 Hz
Point	16384
Frequecy(Span)	4808.074 Hz
Scan	8
AcqTime	3.4095 s
PD	6.5872 s
Pulse1	4.7 μs
IrrNuc	13C
ProbeHeaD	
Instrument	Varian UNITY
Pulse Program	
Gradient Program	
Temperature	30.0 ℃
Solvent	cdcl3
Reference	0.0 ppm
Broad.Factor	0.1466 Hz
Window	Exponential
RGain	38

Operator_____



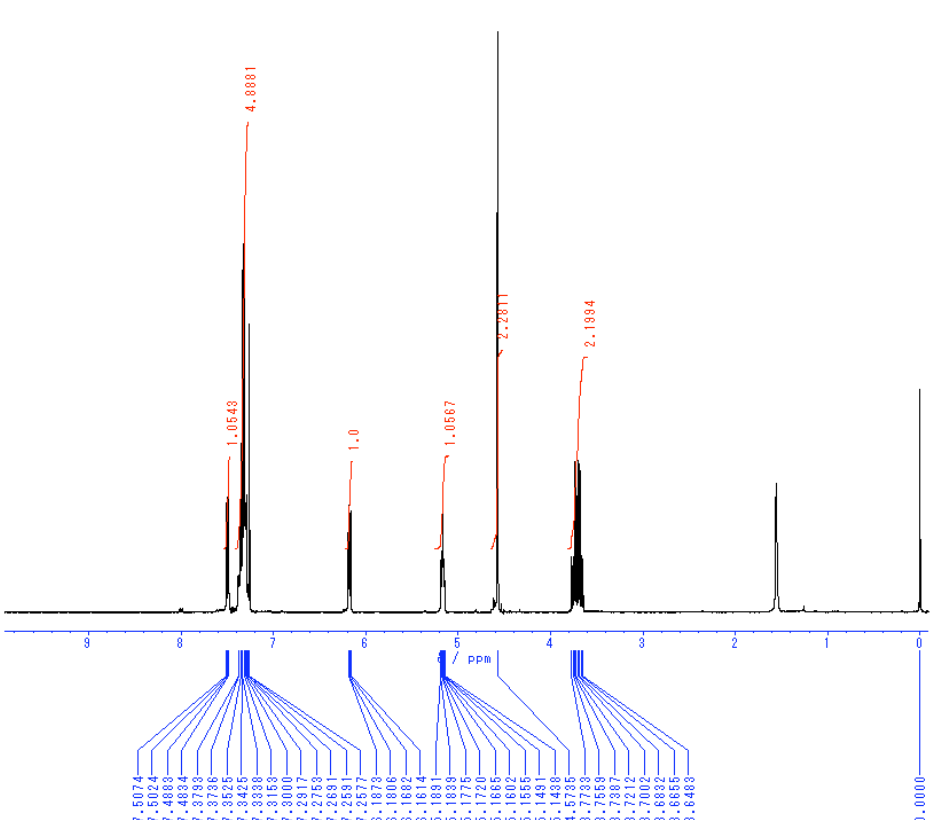


File C:¥DOCUMENTS AND SETTINGS¥TAKII¥デス クトップ¥NMR¥LITERATURE¥LACTONE¥5-(NAP HTHALEN-1-YL)FURAN-2(5H)-ONE¥13C-1.RM1
Date 08/Oct/2011 21:43:45
Comment
Std Carbon experiment

ObsNuc	13C
ExMode	PROTON
ObsFreq	75.45 MHz
ObsSet	-1.0 kHz
ObsFine	998.4988 Hz
Point	65536
Frequecy(Span)	18115.94 Hz
Scan	8400
AcqTime	2.0 s
PD	4.0 s
Pulse1	6.9 μs
IrrNuc	1H
ProbeHeaD	
Instrument	Varian UNITY
Pulse Program	
Gradient Program	
Temperature	30.0 ℃
Solvent	cdcl3
Reference	77.0 ppm
Broad.Factor	0.1382 Hz
Window	Exponential
RGain	36

Operator_____

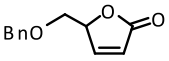
5-(benzyloxymethyl)furan-2(5*H*)-one (**6fb**).

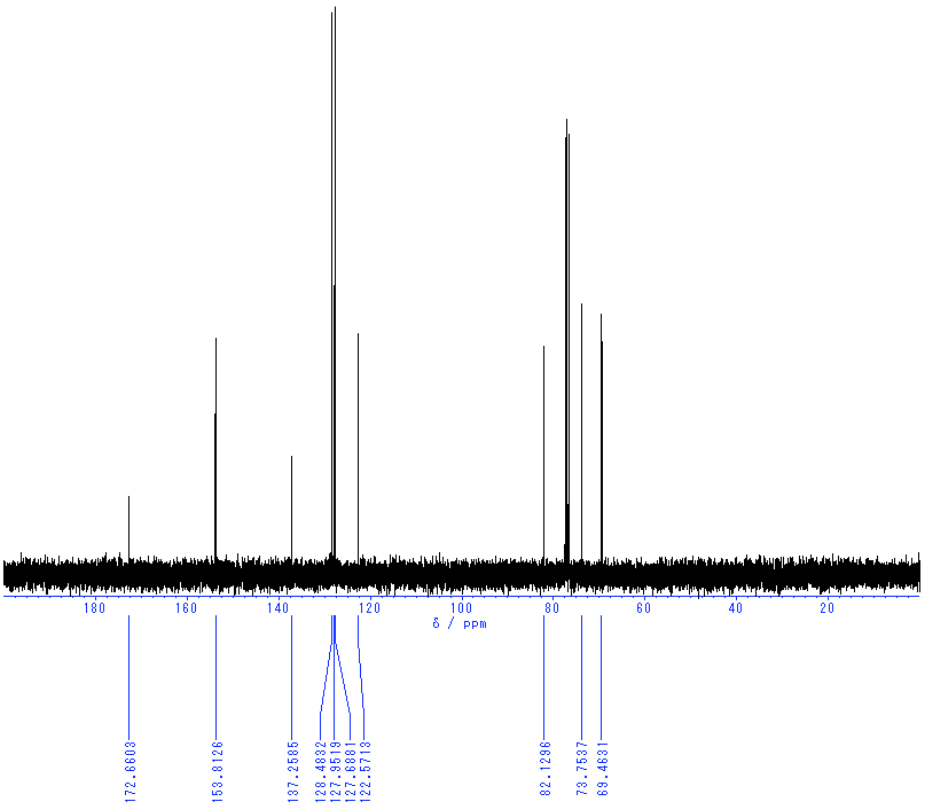


File C:¥DOCUMENTS AND SETTINGS ¥TAKII¥デスクトップ¥NMR¥LIT ERATURE¥LACTONE¥5-(BENZYLOX YMETHYL)FURAN-2(5H)-ONE¥1H. RM1
Date 11/Jun/2011 17:52:41
Comment
Std Proton parameters

ObsNuc	^{1}H
ExMode	NON
ObsFreq	300.04 MHz
ObsSet	-1.0 kHz
ObsFine	994.528 Hz
Point	16384
Frequecy(Span)	4803.074 Hz
Scan	8
AcqTime	3.4095 s
PD	6.5872 s
Pulse1	4.7 μs
IrrNuc	^{13}C
ProbeHeaD	
Instrument	Varian UNITY
Pulse Program	
Gradient Program	
Temperature	30.0 ℃
Solvent	cdcl$_3$
Reference	0.0 ppm
Broad.Factor	0.1466 H z
Window	Exponential
RGain	32

Operator_____

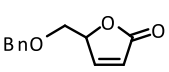




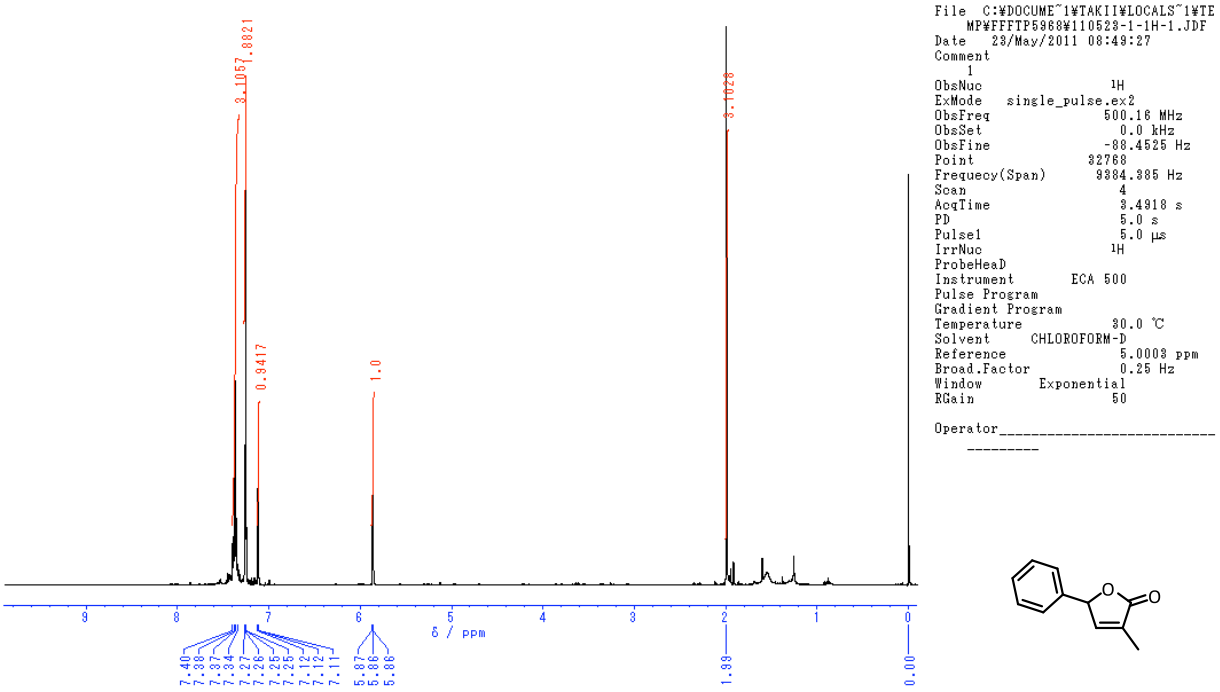
File C:¥DOCUMENTS AND SETTINGS¥TAKII ¥デスクトップ¥NMR¥LITERATURE¥LACT ONE¥5-(BENZYLOXYMETHYL)FURAN-2(5H)-ONE¥13C.RM1
Date 14/Jun/2011 11:59:54
Comment
Std Carbon experiment

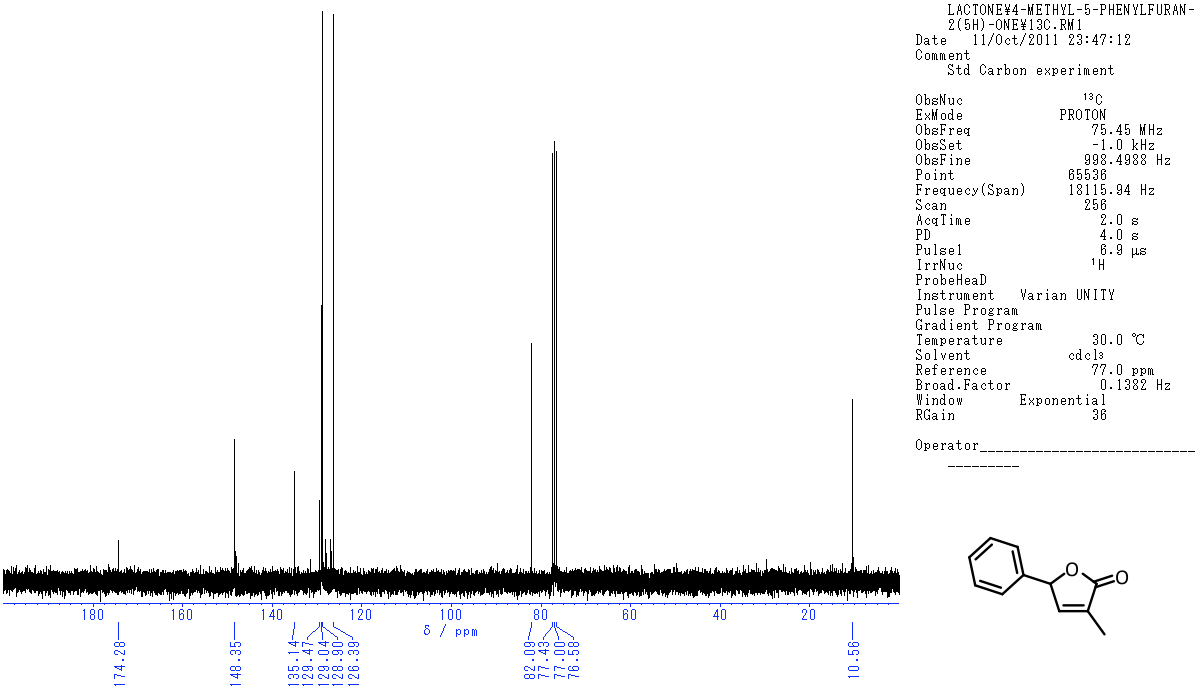
ObsNuc	^{13}C
ExMode	PROTON
ObsFreq	75.45 MHz
ObsSet	-1.0 kHz
ObsFine	998.4988 Hz
Point	65536
Frequecy(Span)	18115.94 Hz
Scan	128
AcqTime	2.0 s
PD	4.0 s
Pulse1	6.9 μs
IrrNuc	^{1}H
ProbeHeaD	
Instrument	Varian UNITY
Pulse Program	
Gradient Program	
Temperature	30.0 ℃
Solvent	cdcl$_3$
Reference	77.0 ppm
Broad.Factor	0.1382 Hz
Window	Exponential
RGain	36

Operator_____

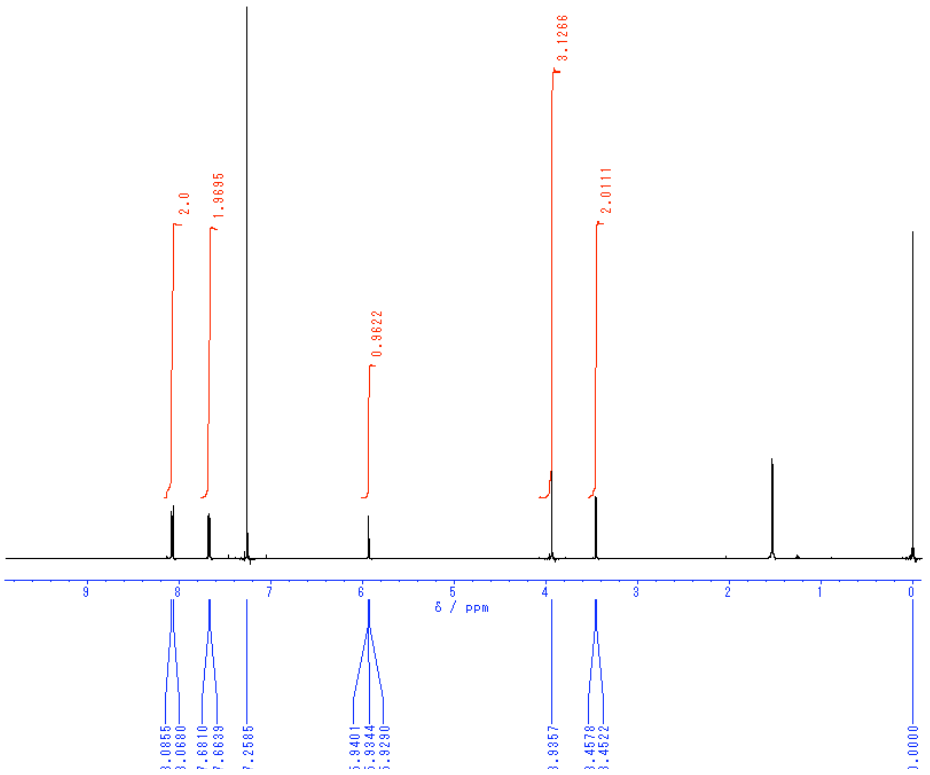


3-methyl-5-phenylfuran-2(5*H*)-one (6ac).



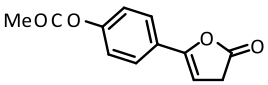


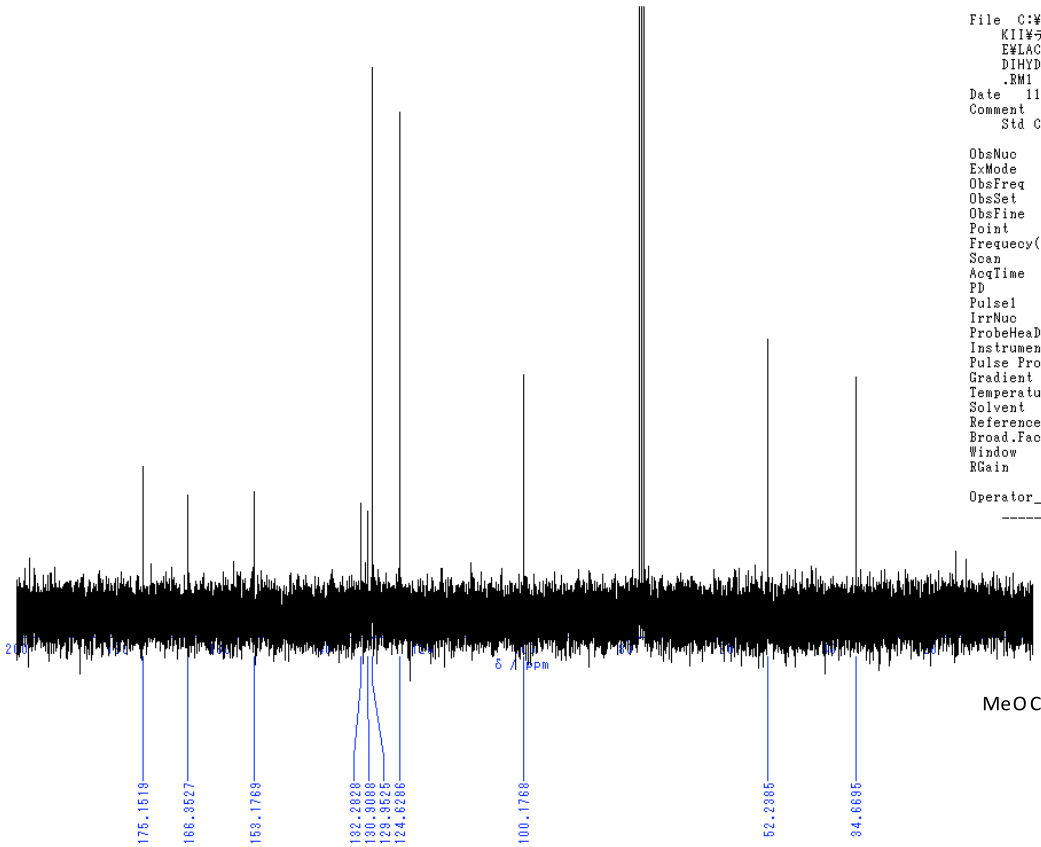
Methyl 4-(5-oxo-4,5-dihydrofuran-2-yl)benzoate (**7**).




```
File  C:¥DOCUMENTS AND SETTINGS¥TAKI
    I¥デスクトップ¥NMR¥LITERATURE¥LA
    CTONE¥METHYL 4-(5-OXO-4,5-DIHYDR
    OFURAN-2-YL)BENZOATE¥1H-1.RM1
Date   28/Oct/2011 10:28:06
Comment
    1
ObsNuc                  1H
ExMode    single_pulse.ex2
ObsFreq              500.16 MHz
ObsSet                 0.0 kHz
ObsFine               -88.4525 Hz
Point                32768
Frequecy(Span)       9384.385 Hz
Scan                    16
AcqTime                 3.4918 s
PD                      5.0 s
Pulse1                  3.8 µs
IrrNuc                  1H
ProbeHeaD
Instrument       ECA 500
Pulse Program
Gradient Program
Temperature            30.1 ℃
Solvent          CHLOROFO
Reference               0.0 ppm
Broad.Factor            0.1432 Hz
Window        Exponential
RGain                   50

Operator_______________________________
    ________
```

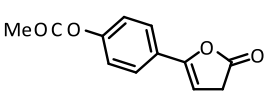




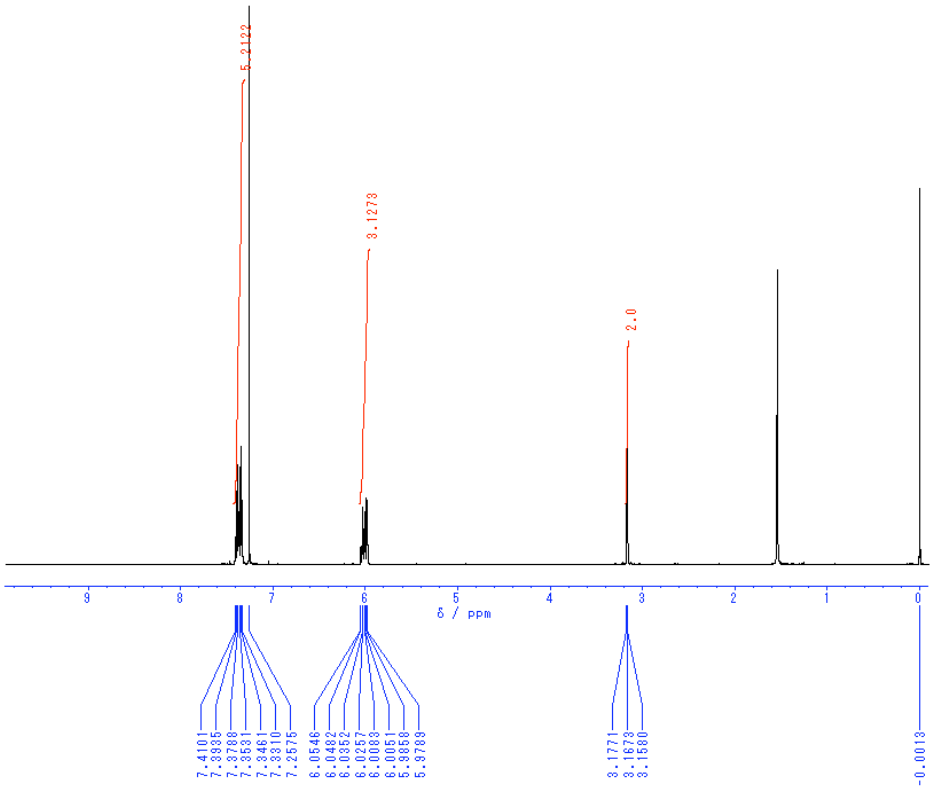

```
File  C:¥DOCUMENTS AND SETTINGS¥TA
    KII¥デスクトップ¥NMR¥LITERATUR
    E¥LACTONE¥METHYL 4-(5-OXO-4,5-
    DIHYDROFURAN-2-YL)BENZOATE¥13C
    .RM1
Date   11/Oct/2011 23:32:04
Comment
    Std Carbon experiment

ObsNuc                 13C
ExMode             PROTON
ObsFreq              75.45 MHz
ObsSet               -1.0 kHz
ObsFine             998.4988 Hz
Point              65536
Frequecy(Span)     18115.94 Hz
Scan                   64
AcqTime                2.0 s
PD                     4.0 s
Pulse1                 6.9 µs
IrrNuc                 1H
ProbeHeaD
Instrument   Varian UNITY
Pulse Program
Gradient Program
Temperature            30.0 ℃
Solvent            cdcl3
Reference              77.0 ppm
Broad.Factor            0.1382 Hz
Window        Exponential
RGain                  36

Operator_______________________________
    __________
```

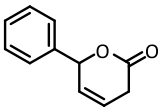


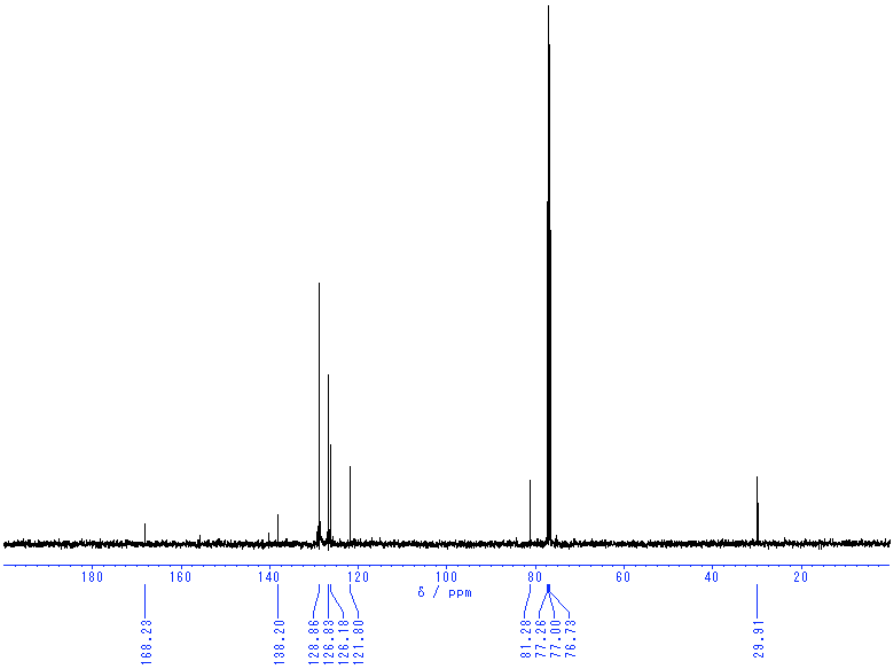
6-phenyl-3,6-dihydro-2*H*-pyran-2-one (8).




```
File  C:¥DOCUMENTS AND SETTINGS¥TA
    KII¥デスクトップ¥NMR¥LITERATUR
    E¥LACTONE¥6-PHENYL-3,6-DIHYDRO
    -2H-PYRAN-2-ONE¥1H.RM1
Date    02/Sep/2011 17:52:28
Comment
    1
ObsNuc                  1H
ExMode   single_pulse.ex2
ObsFreq               500.16 MHz
ObsSet                  0.0 kHz
ObsFine               -88.4525 Hz
Point                 65536
Frequecy(Span)        9384.385 Hz
Scan                      8
AcqTime                 6.9835 s
PD                      5.0 s
Pulse1                  6.2 µs
IrrNuc                  1H
ProbeHeaD
Instrument        ECA 500
Pulse Program
Gradient Program
Temperature           30.0 ℃
Solvent          CHLOROFO
Reference               5.0042 ppm
Broad.Factor            0.0716 Hz
Window        Exponential
RGain                   46

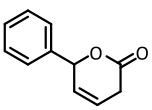
Operator_____________________________
    _________
```



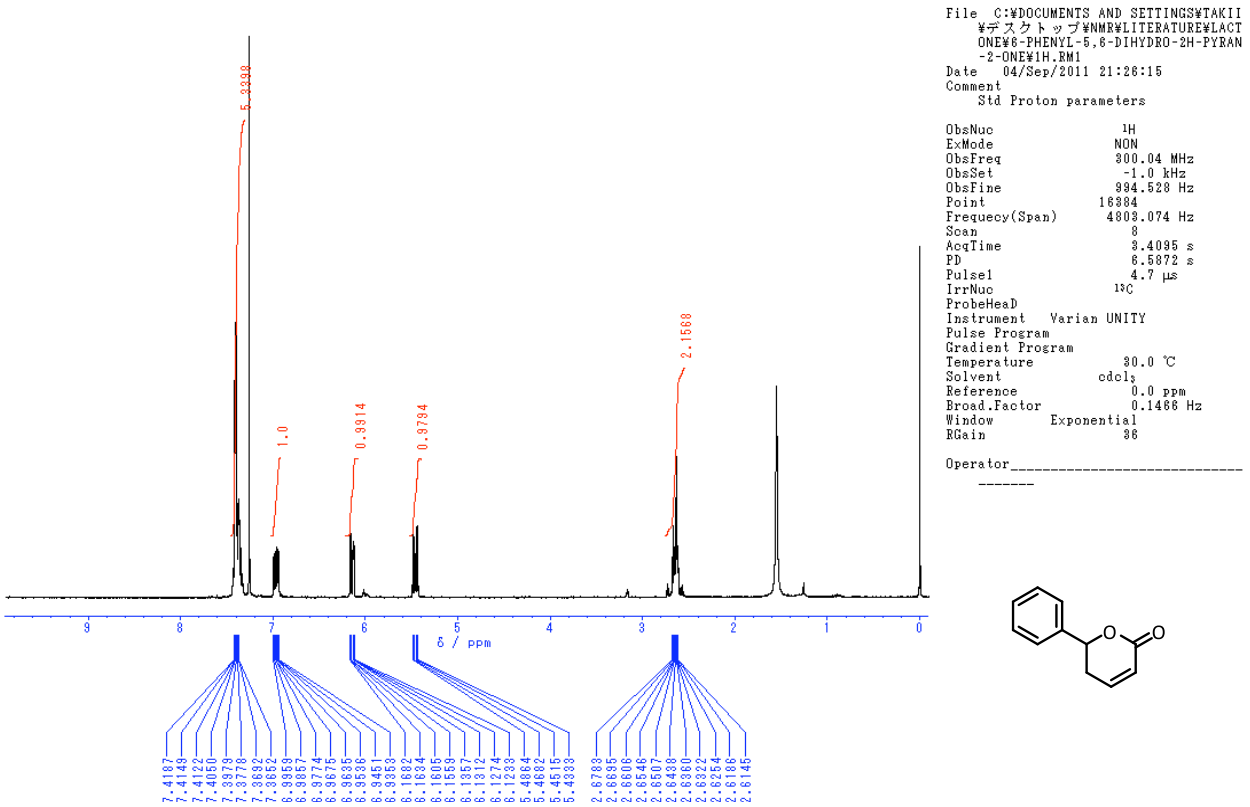


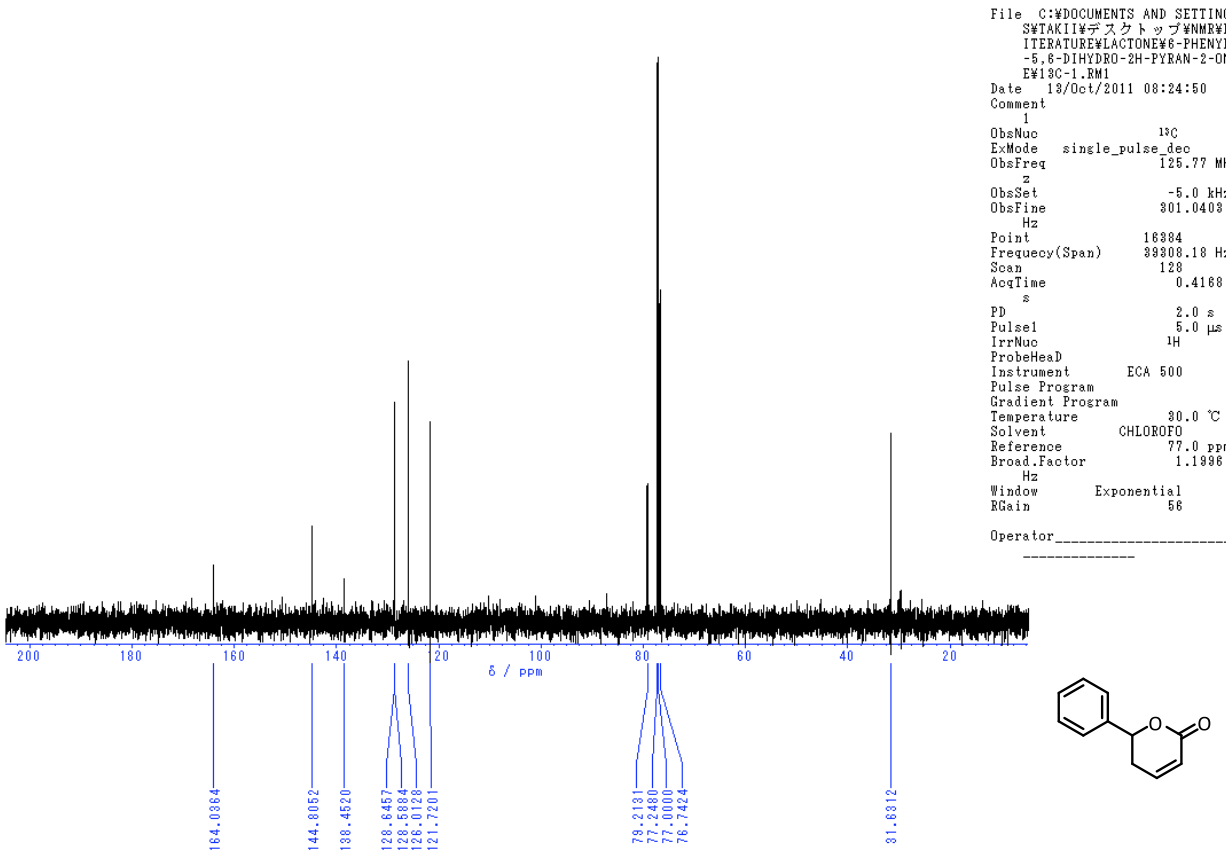

```
File  C:¥DOCUMENTS AND SETTINGS¥TAKII¥
    デスクトップ¥NMR¥LITERATURE¥LACTONE
    ¥6-PHENYL-3,6-DIHYDRO-2H-PYRAN-2-ON
    E¥13C.RM1
Date    28/Dec/2011 08:40:57
Comment
    1
ObsNuc                  13C
ExMode   single_pulse_dec
ObsFreq               125.77 MHz
ObsSet                 -5.0 kHz
ObsFine               301.0403 Hz
Point                 8192
Frequecy(Span)        39308.18 Hz
Scan                  3051
AcqTime                 0.2084 s
PD                      2.0 s
Pulse1                  5.0 µs
IrrNuc                  1H
ProbeHeaD
Instrument        ECA 500
Pulse Program
Gradient Program
Temperature           30.0 ℃
Solvent      CHLOROFORM-D
Reference             77.0 ppm
Broad.Factor           0.25 Hz
Window        Exponential
RGain                   50

Operator_____________________________
    _____
```

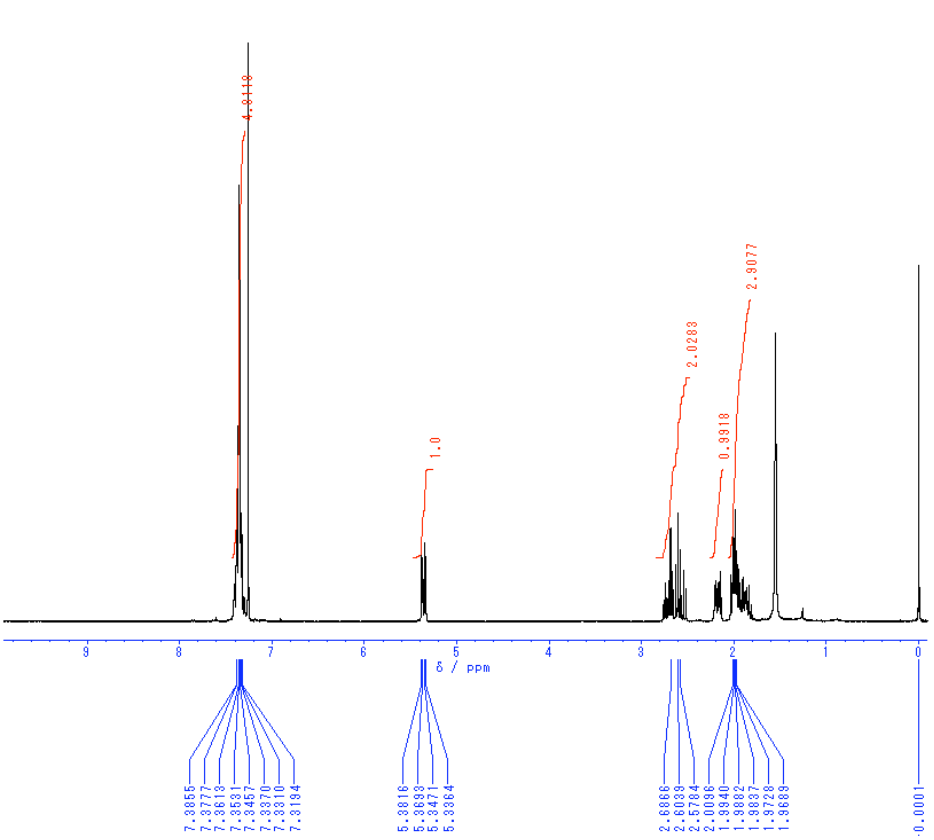


6-phenyl-5,6-dihydro-2*H*-pyran-2-one (9).





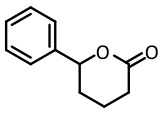
6-phenyltetrahydro-2*H*-pyran-2-one (10).

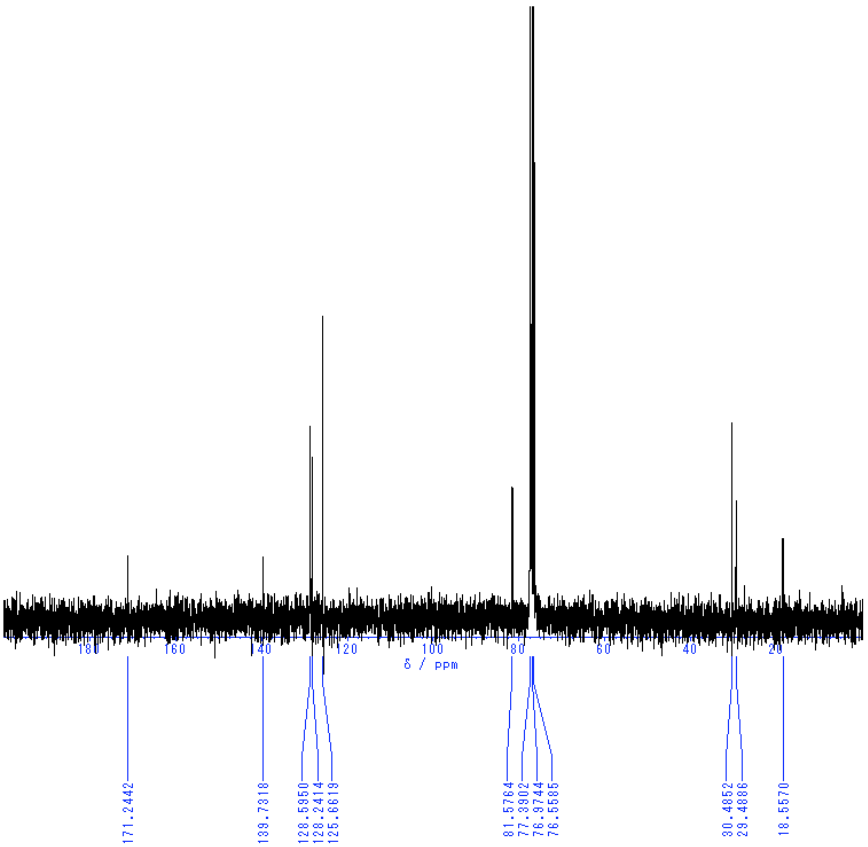



```
File  C:¥DOCUMENTS AND SETTINGS¥TAKII
    ¥デスクトップ¥NMR¥LITERATURE¥LACT
    ONE¥6-PHENYLTETRAHYDRO-2H-PYRAN-2
    -ONE¥1H.RM1
Date   04/Sep/2011 00:47:34
Comment
    Std Proton parameters

ObsNuc                  1H
ExMode                 NON
ObsFreq            300.04 MHz
ObsSet              -1.0 kHz
ObsFine            994.528 Hz
Point                16384
Frequecy(Span)     4803.074 Hz
Scan                     8
AcqTime             3.4095 s
PD                  6.5872 s
Pulse1               4.7 µs
IrrNuc                 13C
ProbeHeaD
Instrument   Varian UNITY
Pulse Program
Gradient Program
Temperature          30.0 ℃
Solvent             cdcl3
Reference          0.0001 ppm
Broad.Factor       0.1466 Hz
Window       Exponential
RGain                  36

Operator_____________________________
    _______
```

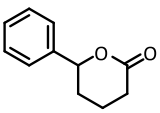




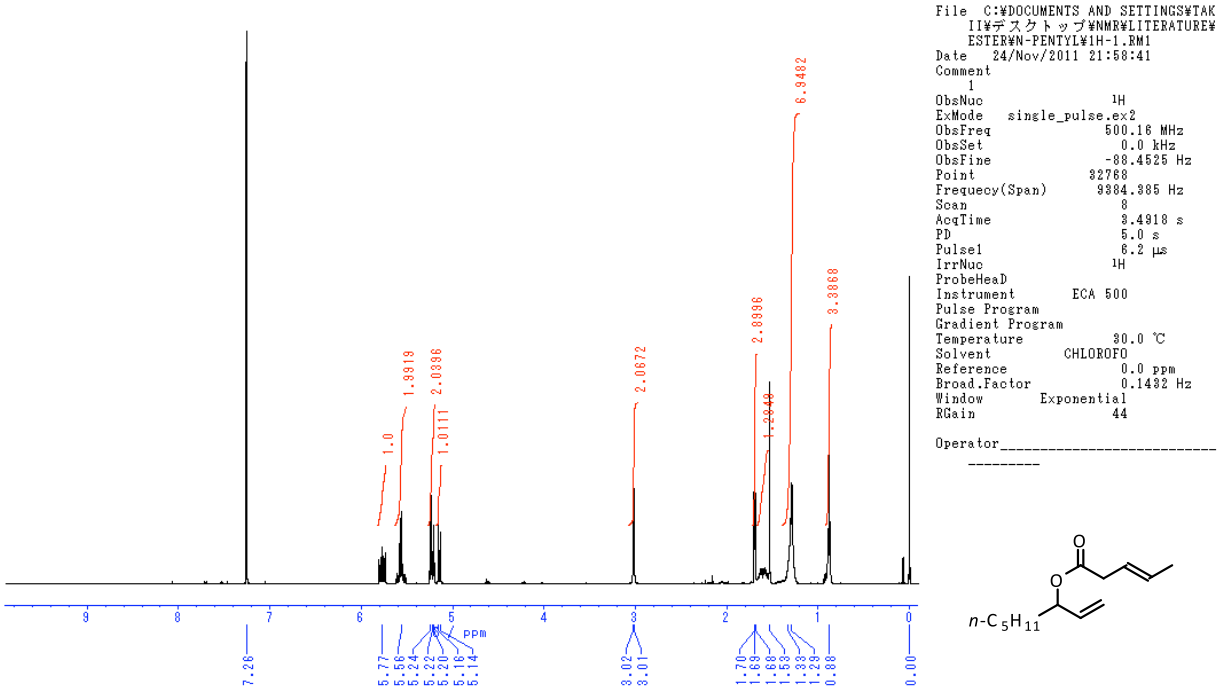

```
File  C:¥DOCUMENTS AND SETTINGS¥TAKII¥デス
    クトップ¥NMR¥LITERATURE¥LACTONE¥6-PHENY
    LTETRAHYDRO-2H-PYRAN-2-ONE¥13C.RM1
Date   13/Oct/2011 23:45:15
Comment
    Std Carbon experiment

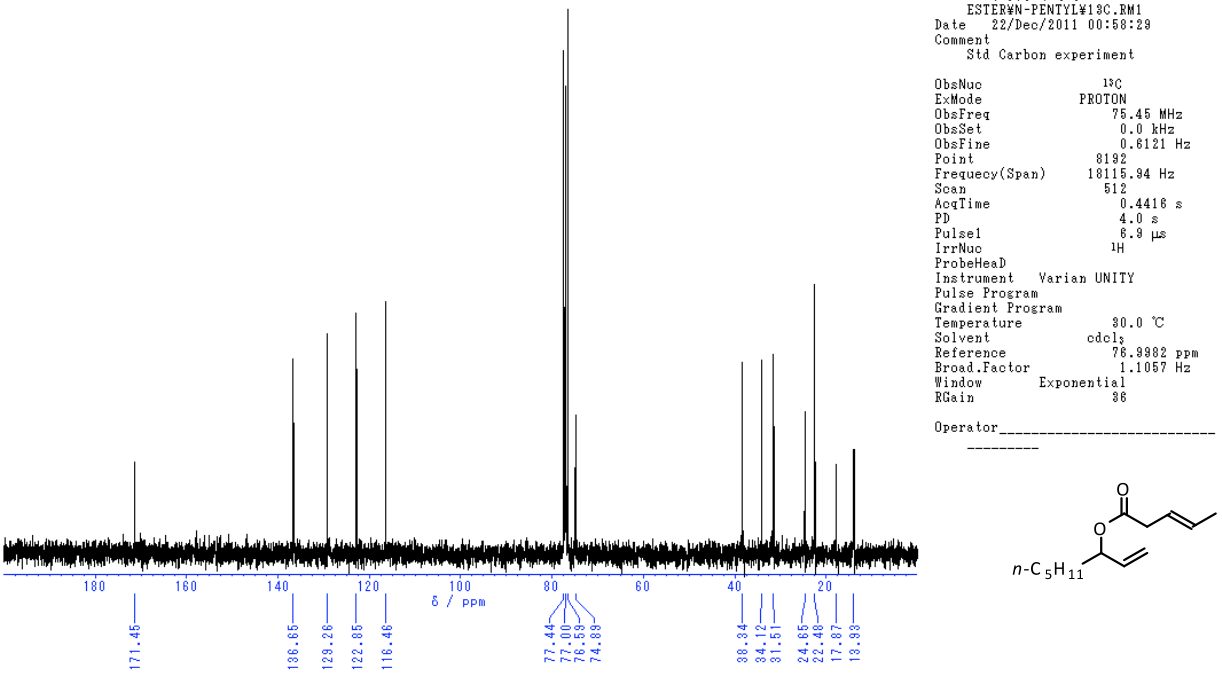
ObsNuc                 13C
ExMode             PROTON
ObsFreq           75.45 MHz
ObsSet              -1.0 kHz
ObsFine           998.4988 Hz
Point                8192
Frequecy(Span)    18115.94 Hz
Scan                 7800
AcqTime            0.4416 s
PD                   4.0 s
Pulse1               6.9 µs
IrrNuc                 1H
ProbeHeaD
Instrument   Varian UNITY
Pulse Program
Gradient Program
Temperature          30.0 ℃
Solvent             cdcl3
Reference         105.0261 ppm
Broad.Factor        1.1057 Hz
Window       Exponential
RGain                  36

Operator_____________________________
    _
```

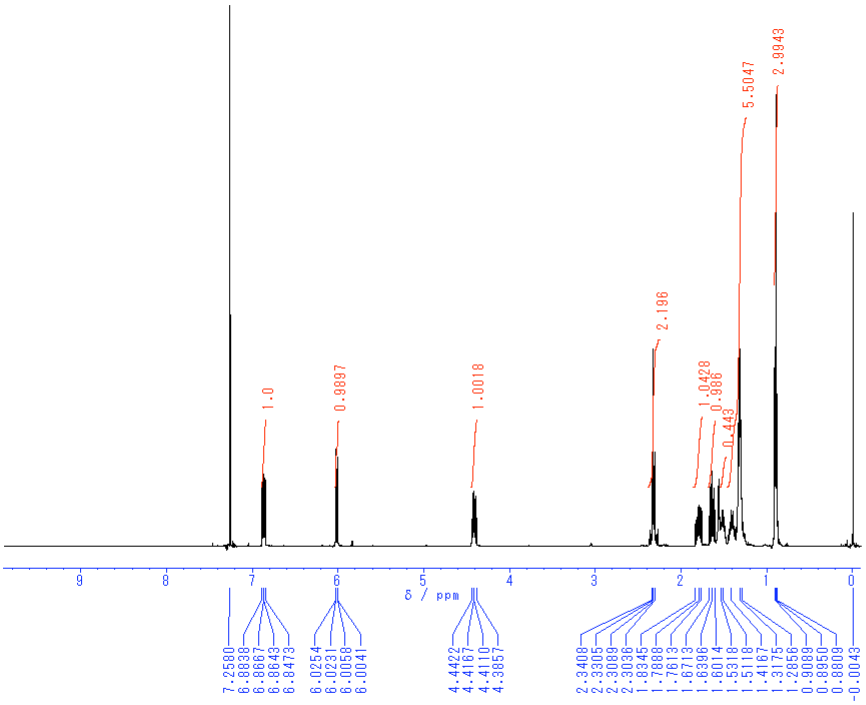


(*E*)-oct-1-en-3-yl pent-3-enoate (4gd).



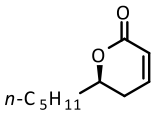


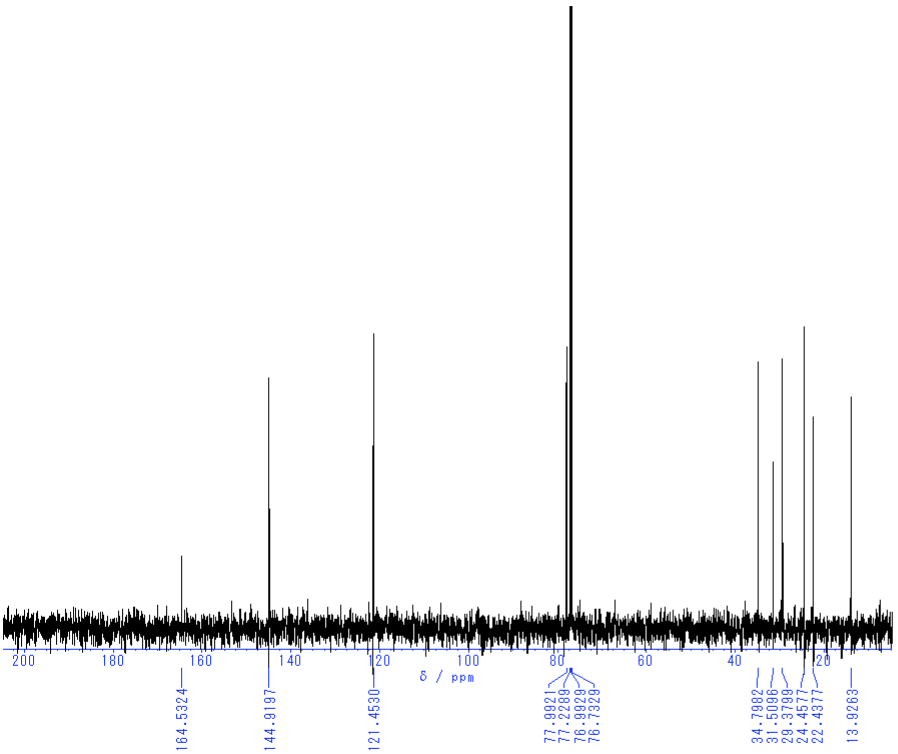
(*R*)-6-pentyl-5,6-dihydro-2H-pyran-2-one ((*R*)-(-)-massoialactone) (**14**).




```
File  C:¥DOCUMENTS AND SETTINGS¥TAKII¥
    デスクトップ¥NMR¥LITERATURE¥LACTONE
    ¥MASSOIA LACTONE¥1H.RM1
Date   29/Nov/2011 16:57:50
Comment
    1
ObsNuc                  ¹H
ExMode   single_pulse.ex2
ObsFreq              500.16 MHz
ObsSet                 0.0 kHz
ObsFine              -88.4525 Hz
Point                32768
Frequecy(Span)       9384.385 Hz
Scan                    16
AcqTime                3.4918 s
PD                     5.0 s
Pulse1                 3.8 µs
IrrNuc                  ¹H
ProbeHeaD
Instrument       ECA 500
Pulse Program
Gradient Program
Temperature           30.0 ℃
Solvent         CHLOROFO
Reference              5.0051 ppm
Broad.Factor           0.1432 Hz
Window       Exponential
RGain                  42

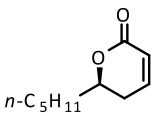
Operator_____________________________
    _____
```





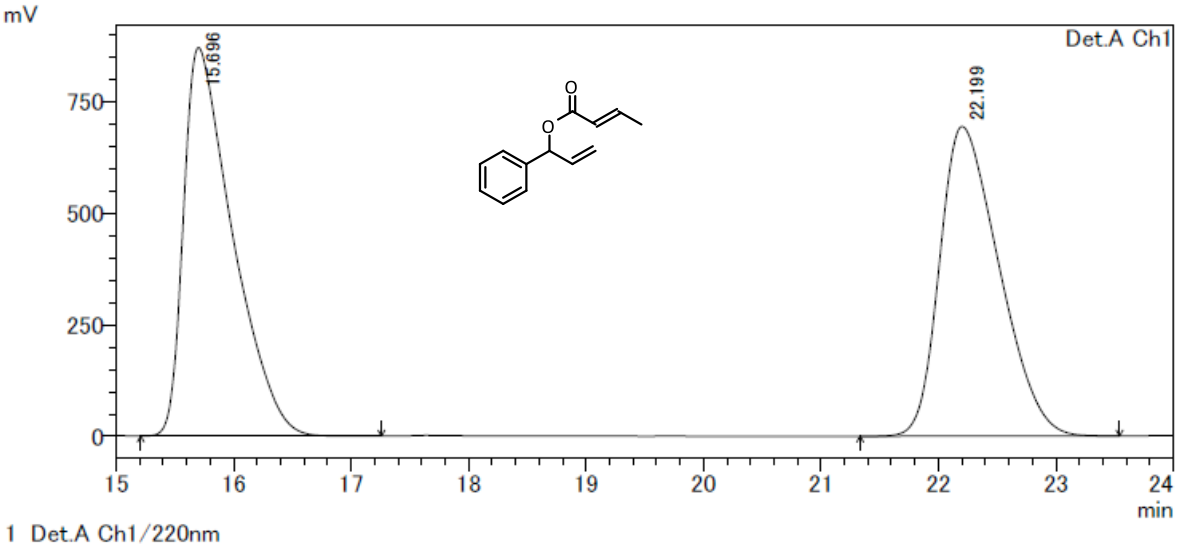

```
File  C:¥DOCUMENTS AND SETTINGS¥TAKI
    I¥デスクトップ¥NMR¥LITERATURE¥LA
    CTONE¥MASSOIA LACTONE¥13C.RM1
Date   30/Nov/2011 09:04:54
Comment
    1
ObsNuc                 ¹³C
ExMode   single_pulse_dec
ObsFreq              125.77 MHz
ObsSet                -5.0 kHz
ObsFine              301.0403 Hz
Point                8192
Frequecy(Span)       39308.18 Hz
Scan                    710
AcqTime                0.2084 s
PD                     2.0 s
Pulse1                 5.0 µs
IrrNuc                  ¹H
ProbeHeaD
Instrument       ECA 500
Pulse Program
Gradient Program
Temperature           30.0 ℃
Solvent         CHLOROFO
Reference             77.0072 ppm
Broad.Factor           2.3992 Hz
Window       Exponential
RGain                  50

Operator_____________________________
    ________
```



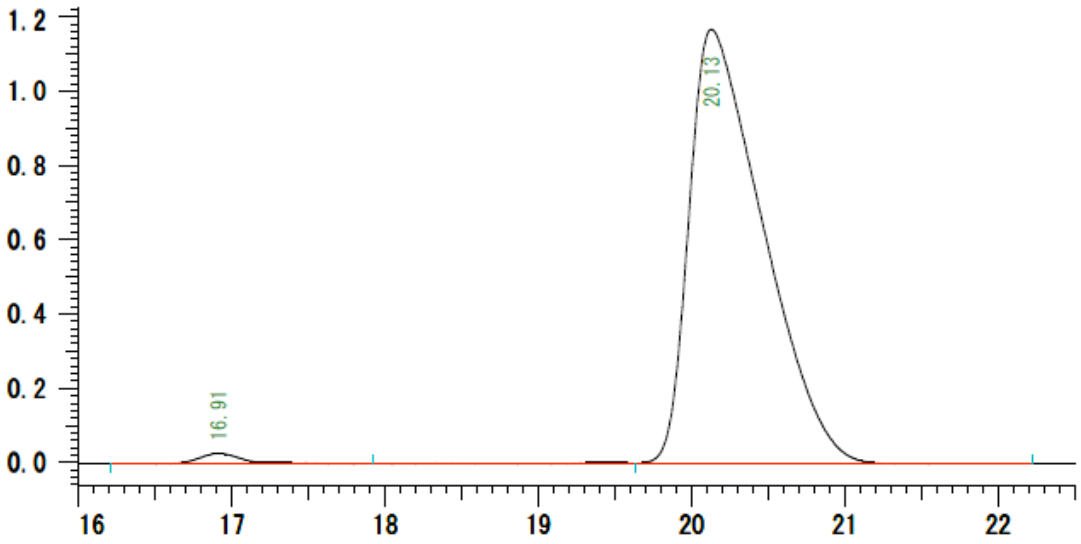
HPLC data

(*E*)-1-phenylallyl but-2-enoate (4ab)



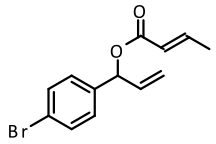
検出器A Ch1 220nm

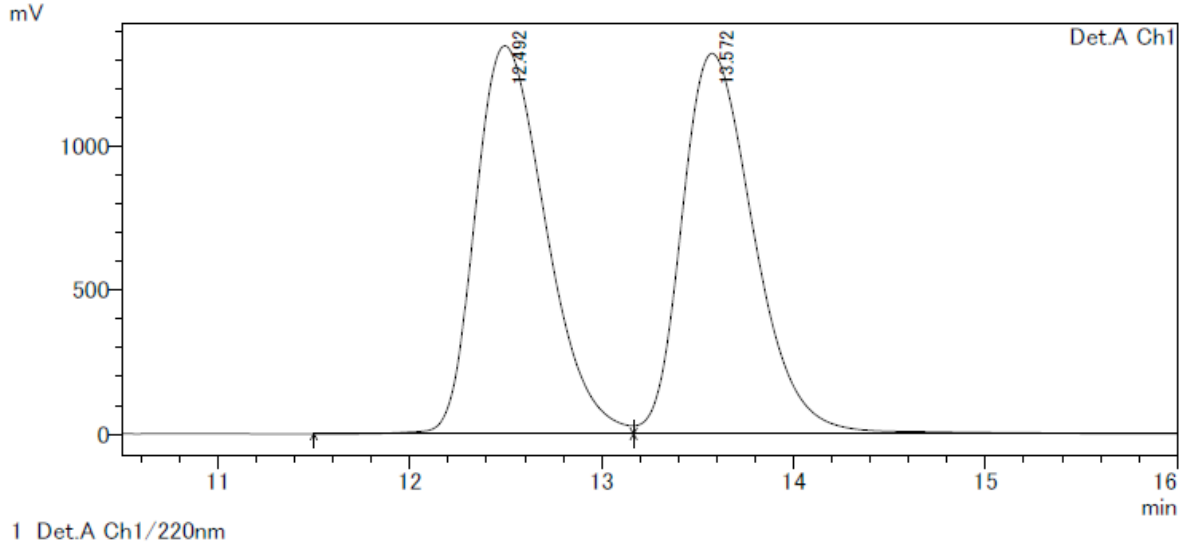
Peak	Retention Time	Area	Area%
1	15.696	25277961	50.841
2	22.199	24441514	49.159
合計		49719475	100.000



	Retention Time	Area	Area%
1	16.91	319441	1.600
2	20.13	19639764	98.400
		19959205	100.000

(*E*)-1-(4-bromophenyl)allyl but-2-enoate (4bb).

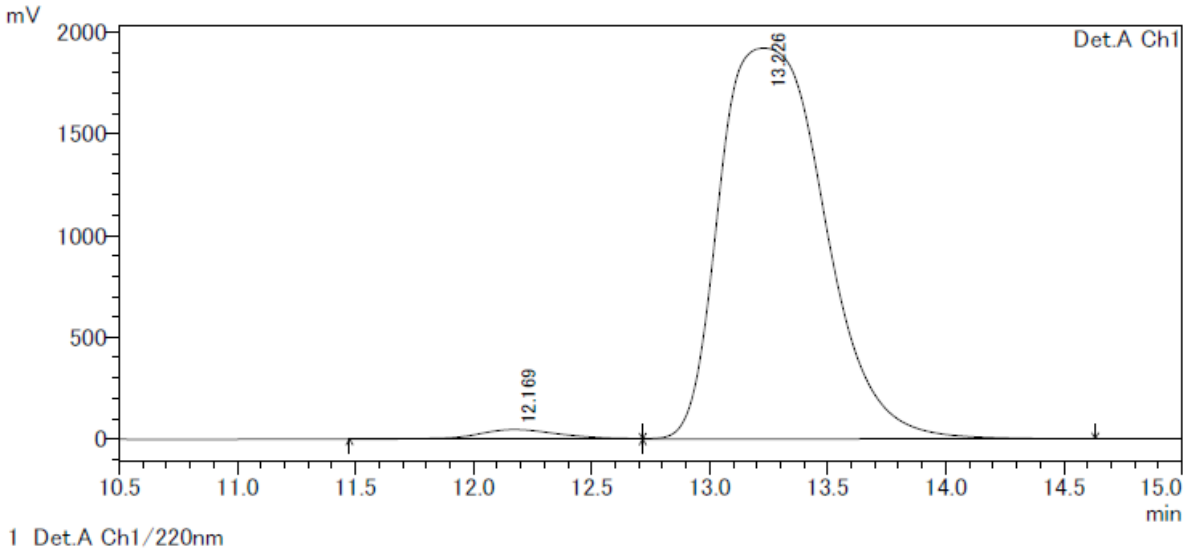




1 Det.A Ch1/220nm

検出器A Ch1 220nm

Peak	Retention Time	Area	Area%
1	12.492	34361132	49.776
2	13.572	34670327	50.224
合計		69031459	100.000

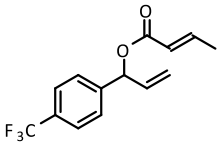


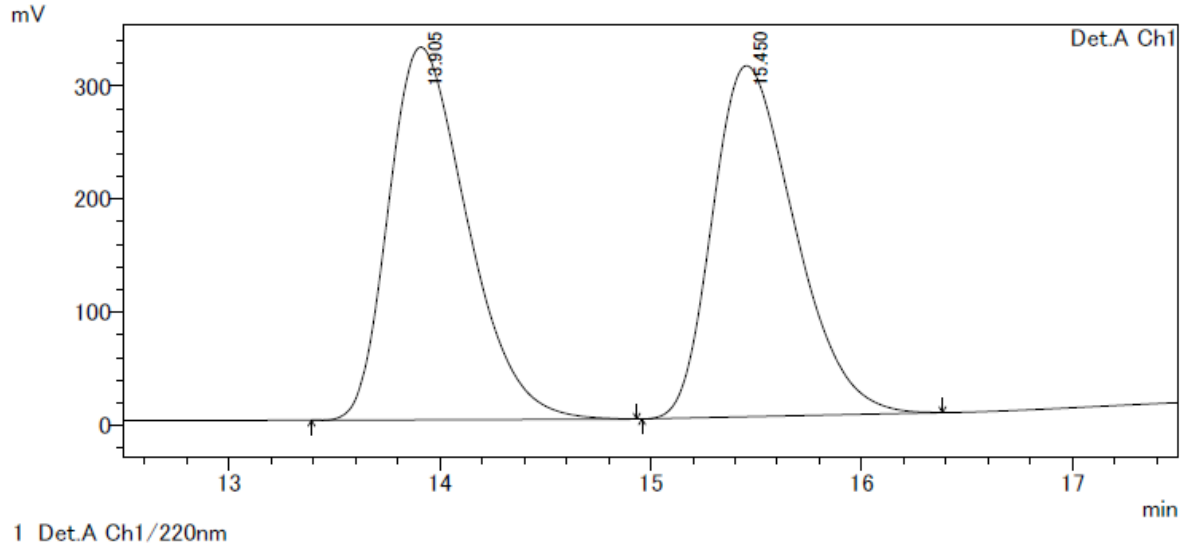
1 Det.A Ch1/220nm

検出器A Ch1 220nm

Peak	Retention Time	Area	Area%
1	12.169	1113180	1.841
2	13.226	59364144	98.159
合計		60477324	100.000

(*E*)-1-(4-trifluoromethylphenyl)allyl but-2-enoate (**4cb**).

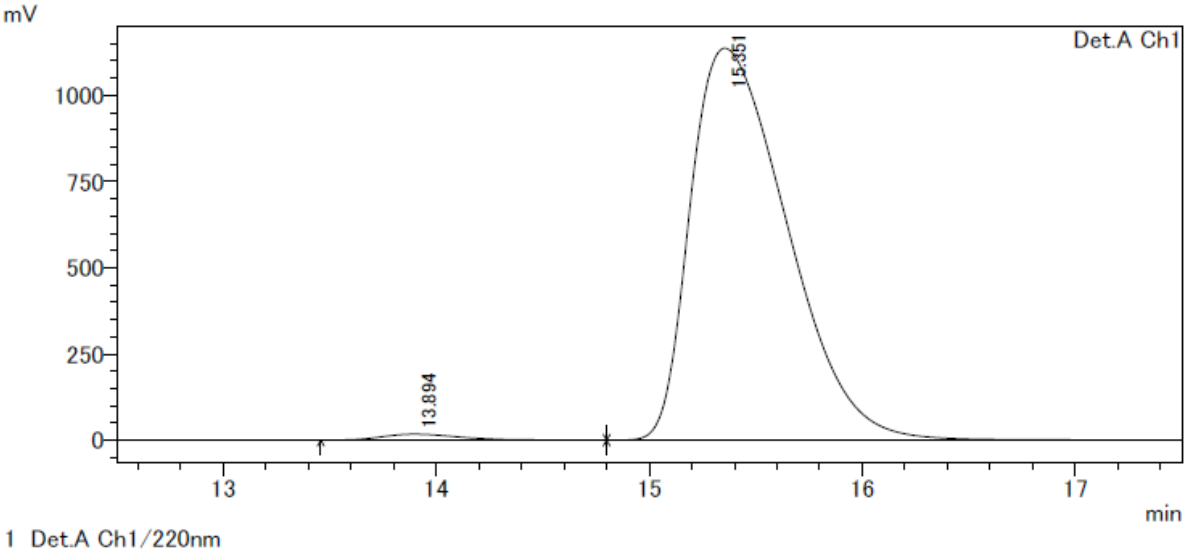




1 Det.A Ch1/220nm

検出器A Ch1 220nm

Peak	Retention Time	Area	Area%
1	13.905	8574623	50.389
2	15.450	8442226	49.611
合計		17016849	100.000

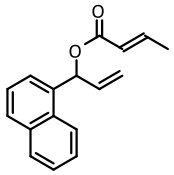


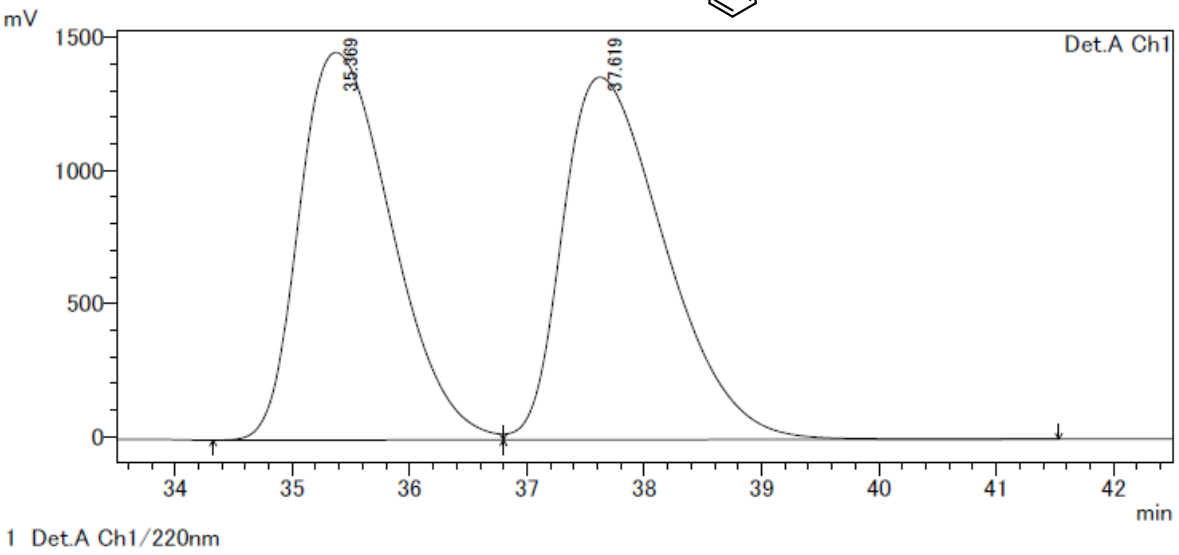
1 Det.A Ch1/220nm

検出器A Ch1 220nm

Peak	Retentin Time	Area	Area%
1	13.894	427370	1.181
2	15.351	35762861	98.819
合計		36190232	100.000

(*E*)-1-(naphthalene-1-yl)allyl but-2-enoate (**4db**).

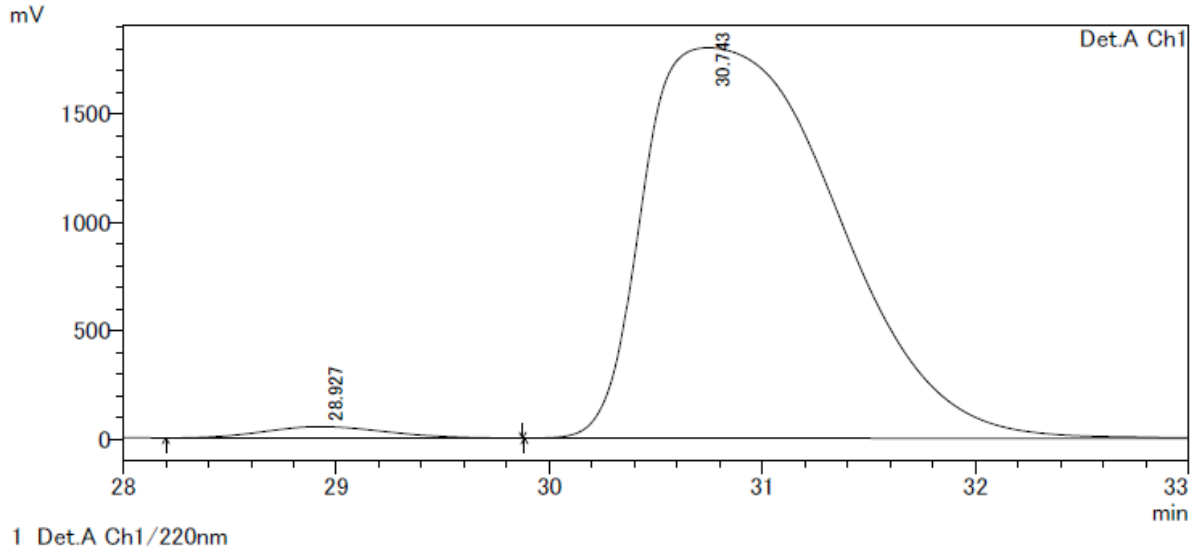




1 Det.A Ch1/220nm

検出器A Ch1 220nm

Peak	Retention Time	Area	Area%
1	35.369	79658155	49.102
2	37.619	82572388	50.898
合計		162230544	100.000

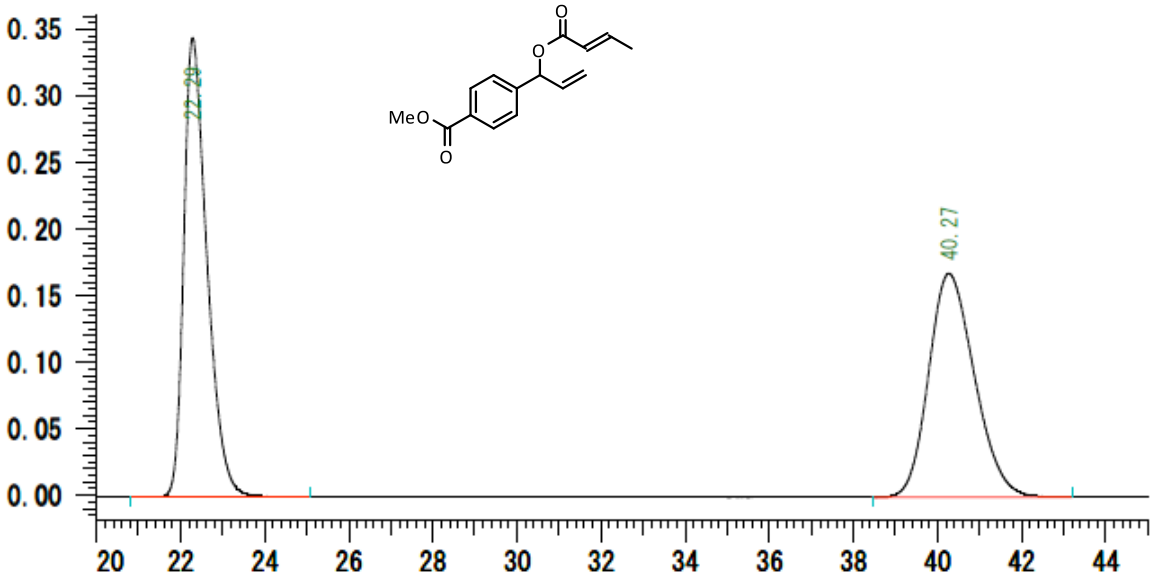


1 Det.A Ch1/220nm

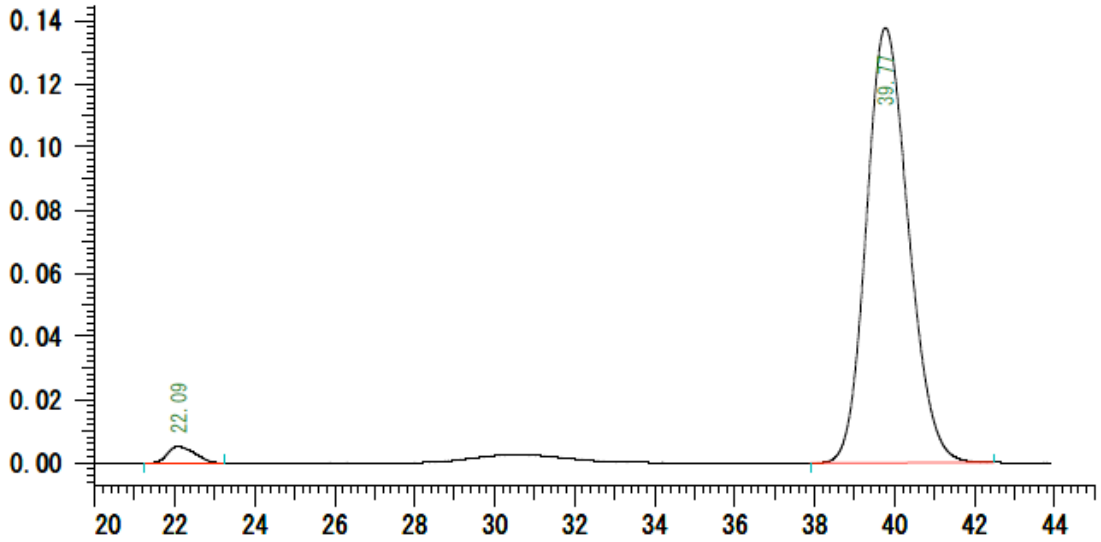
検出器A Ch1 220nm

Peak	Retention Time	Area	Area%
1	28.927	2075457	1.804
2	30.743	112965443	98.196
合計		115040900	100.000

(*E*)-methyl 4-(1-(but-2-enyloxy)allyl)benzoate (4eb)

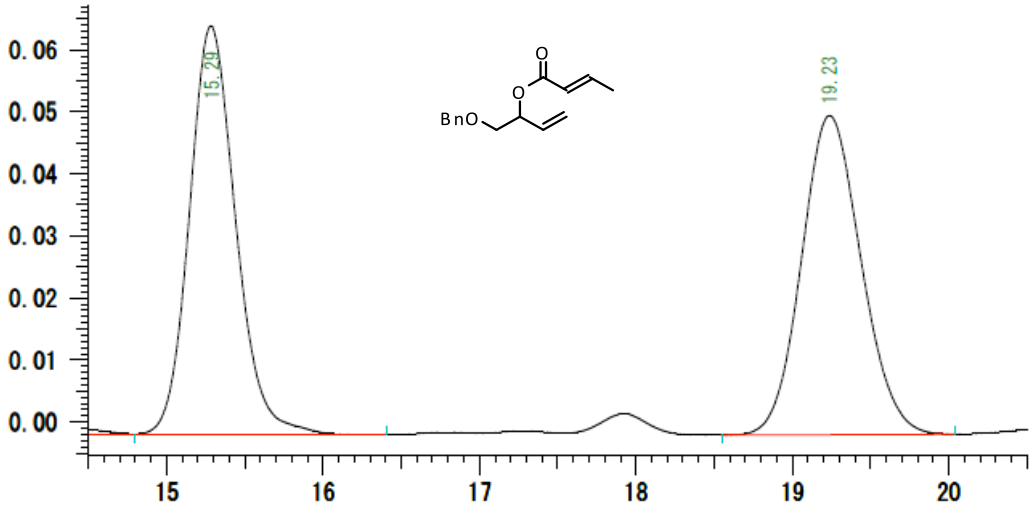


	Retention Time	Area	Area%
1	22.29	6587865	50.788
2	40.27	6383555	49.212
		12971420	100.000

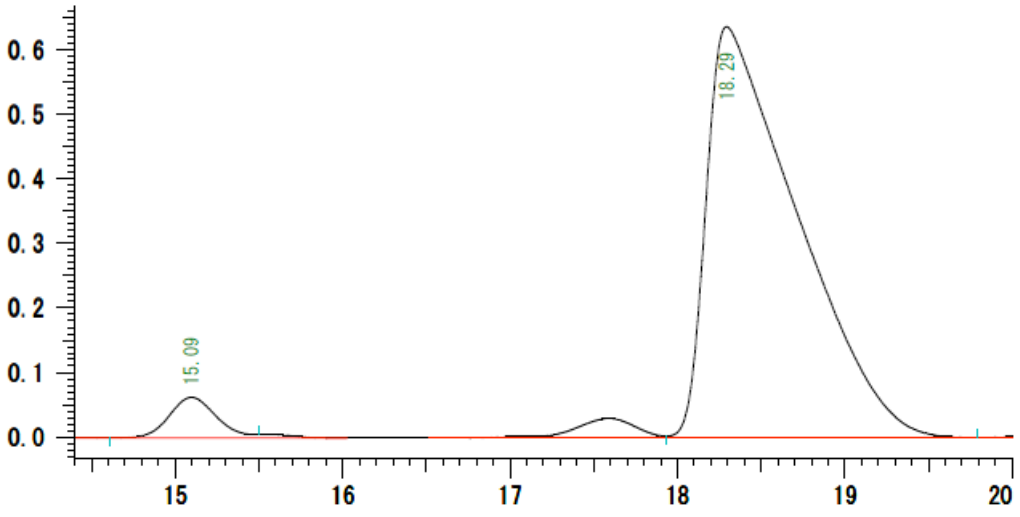


	Retention Time	Area	Area%
1	22.09	129310	2.495
2	39.77	5053089	97.505
		5182399	100.000

(*E*)-1-(benzyloxy)but-3-en-2-yl but-2-enoate (**4fb**).

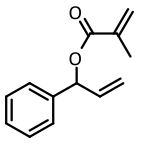


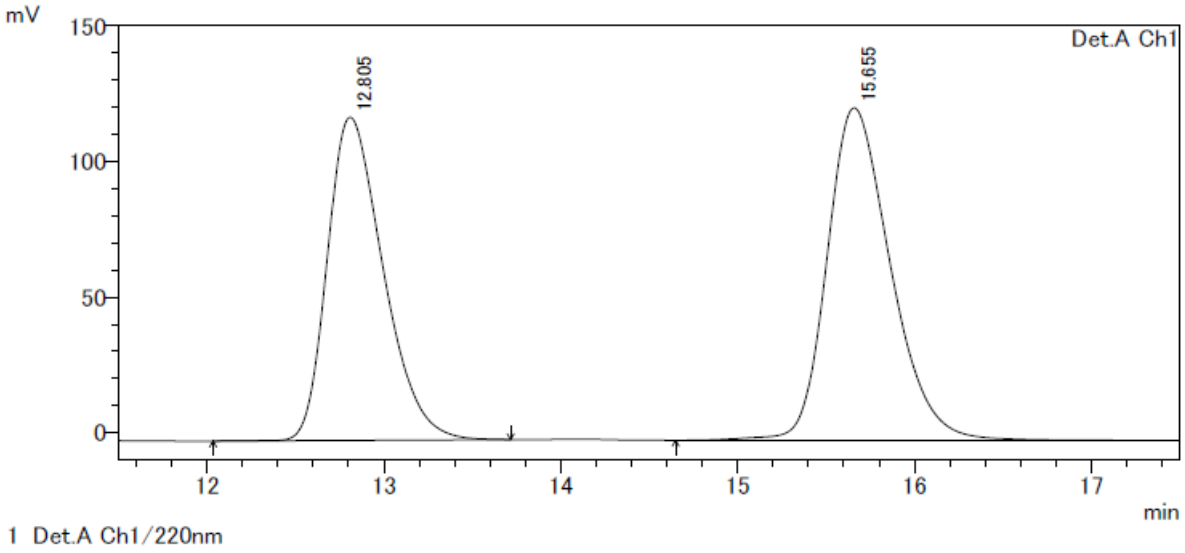
	Retention Time	Area	Area%
1	15.29	679055	49.930
2	19.23	680972	50.070
		1360027	100.000



	Retention Time	Area	Area%
1	15.09	619329	4.874
2	18.29	12087422	95.126
		12706751	100.000

(*E*)-1-phenylallyl methacrylate (**4ac**).

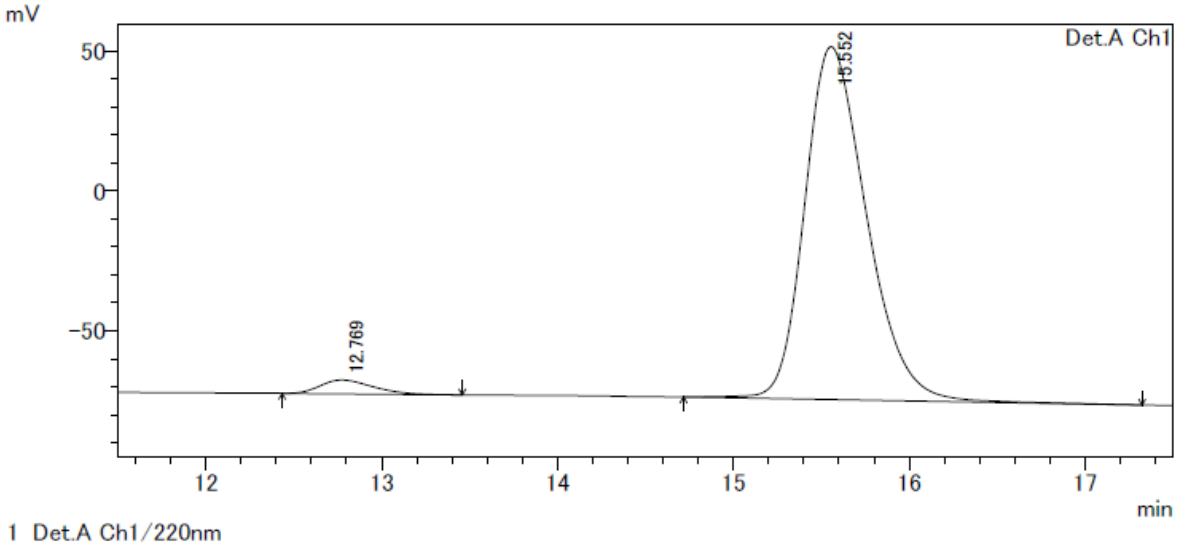




1 Det.A Ch1/220nm

UV-Vis Ch1 220nm

Peak	Retention Time	Area	Area%
1	12.805	2619011	46.047
2	15.655	3068721	53.953
合計		5687732	100.000

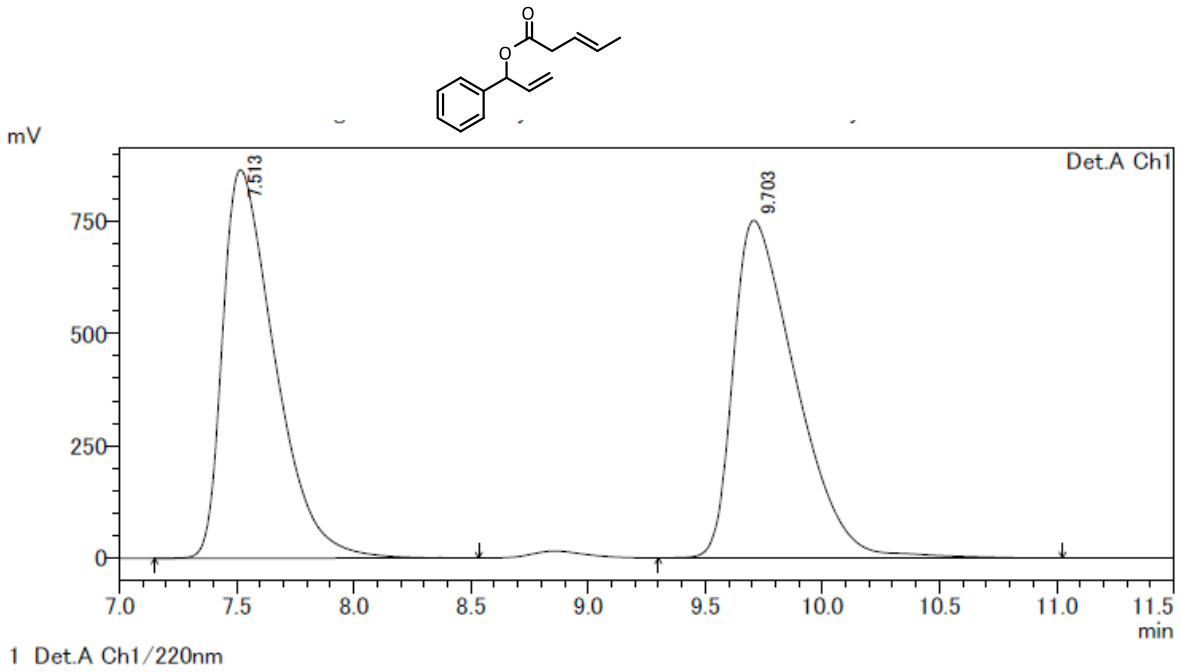


1 Det.A Ch1/220nm

UV-Vis Ch1 220nm

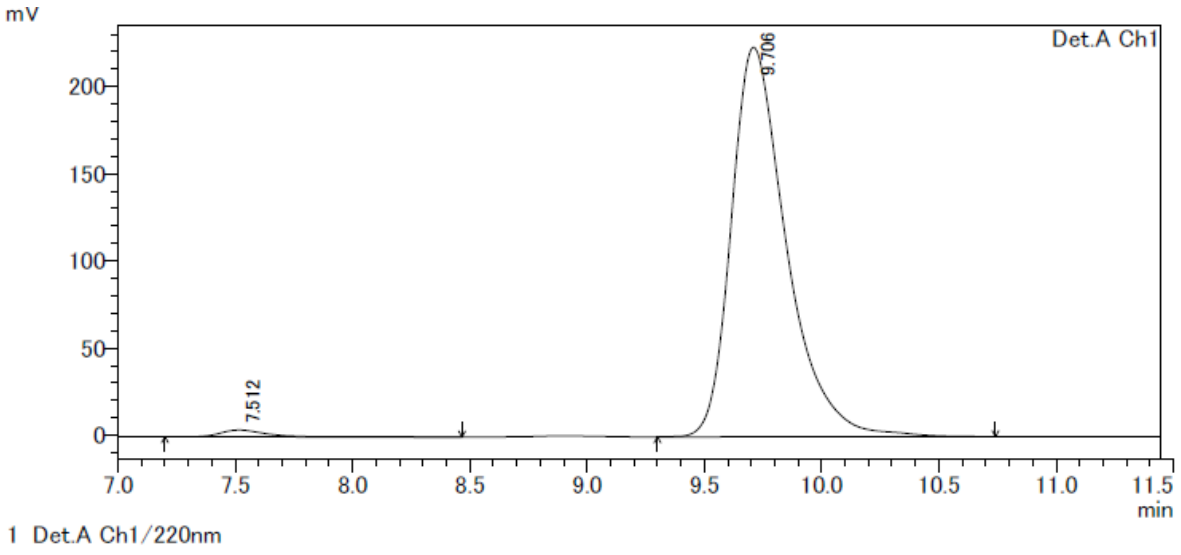
Peak	Retention Time	Area	Area%
1	12.769	103542	3.245
2	15.552	3087444	96.755
合計		3190986	100.000

(*E*)-1-phenylallyl pent-3-enoate (4ad).



検出器A Ch1 220nm

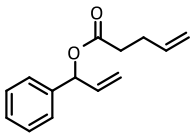
Peak	Retention Time	Area	Area%
1	7.513	13362232	48.855
2	9.703	13988448	51.145
合計		27350680	100.000

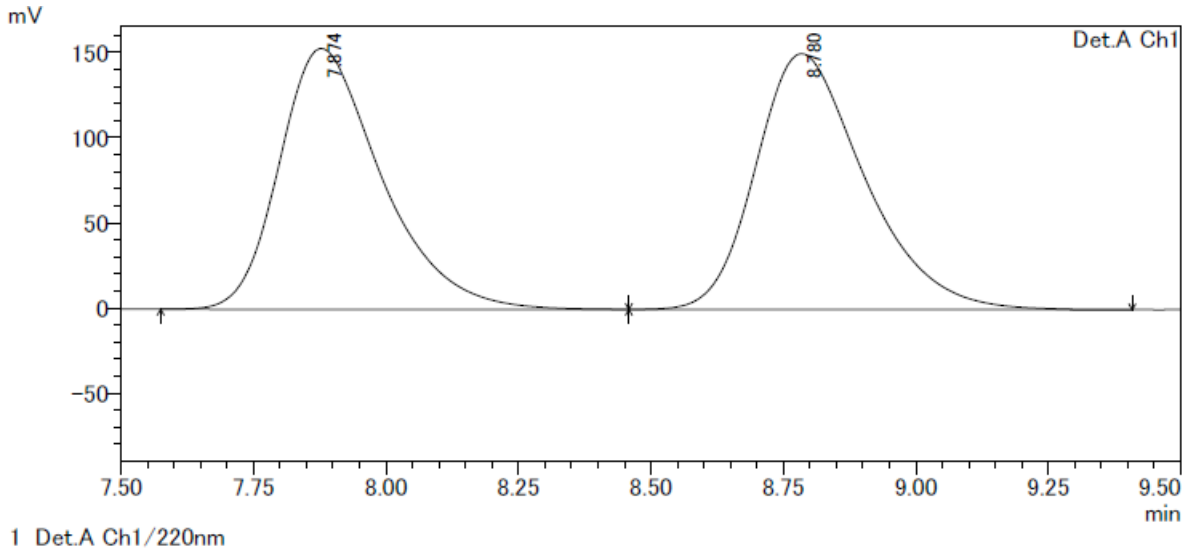


検出器A Ch1 220nm

Peak	Retention Time	Area	Area%
1	7.512	53148	1.435
2	9.706	3650678	98.565
合計		3703826	100.000

1-phenylallyl pent-4-enoate (**4ae**).

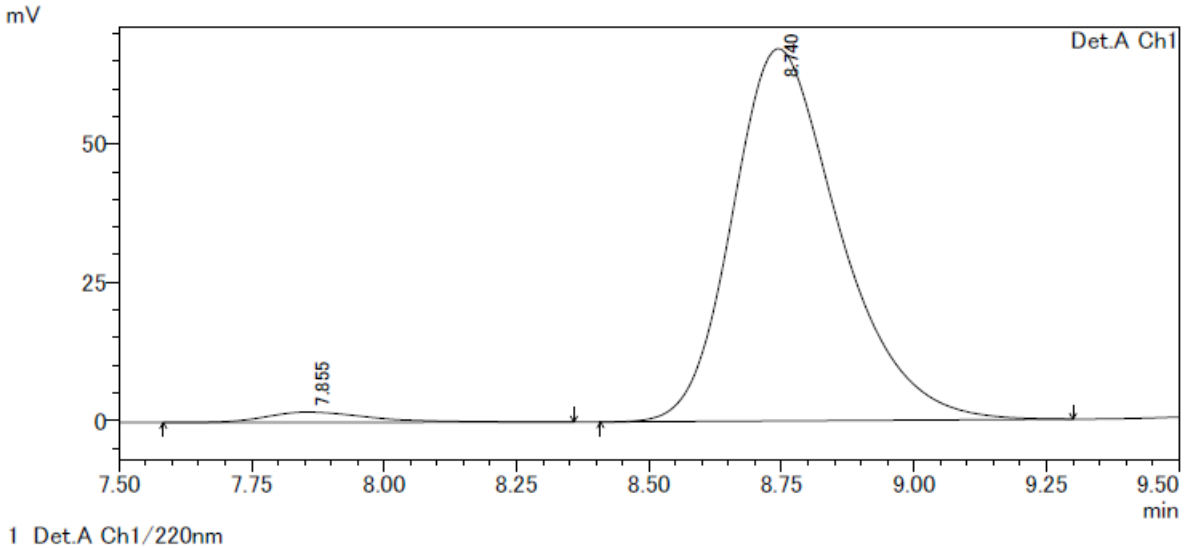




1 Det.A Ch1/220nm

検出器A Ch1 220nm

Peak	Retention Time	Area	Area%
1	7.874	2055447	48.861
2	8.780	2151307	51.139
合計		4206753	100.000

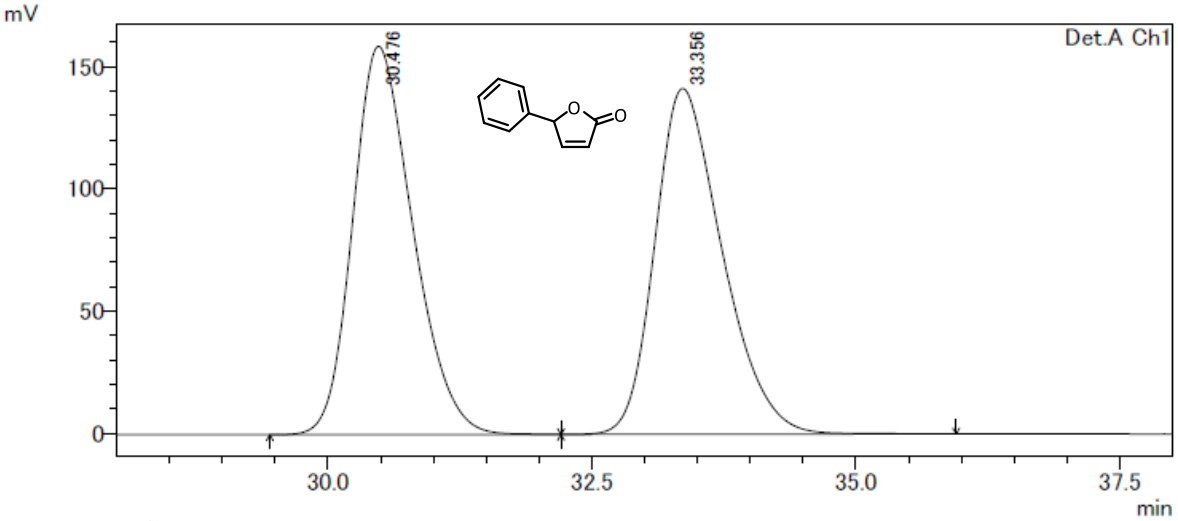


1 Det.A Ch1/220nm

検出器A Ch1 220nm

Peak	Retention Time	Area	Area%
1	7.855	25338	2.604
2	8.740	947785	97.396
合計		973123	100.000

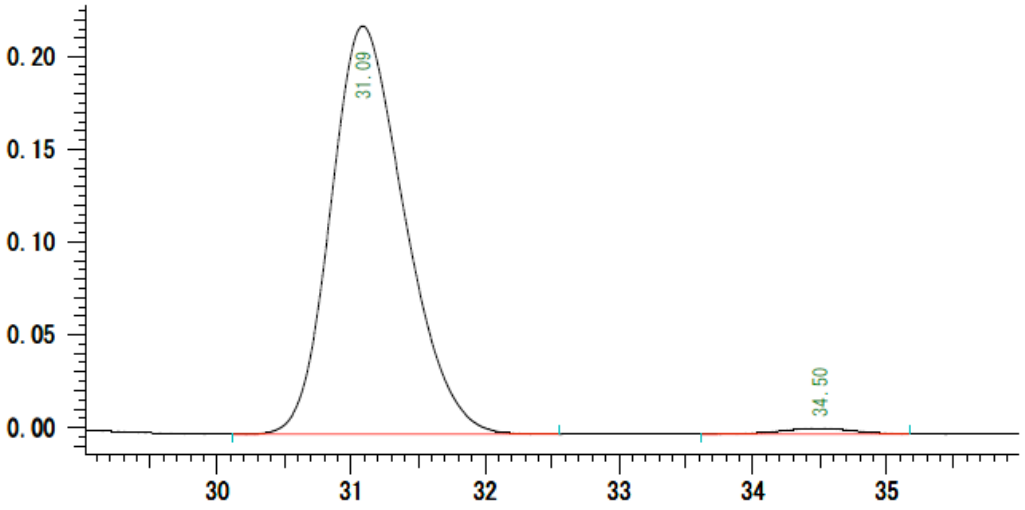
5-phenylfuran-2(5*H*)-one (6ab).



1 Det.A Ch1/220nm

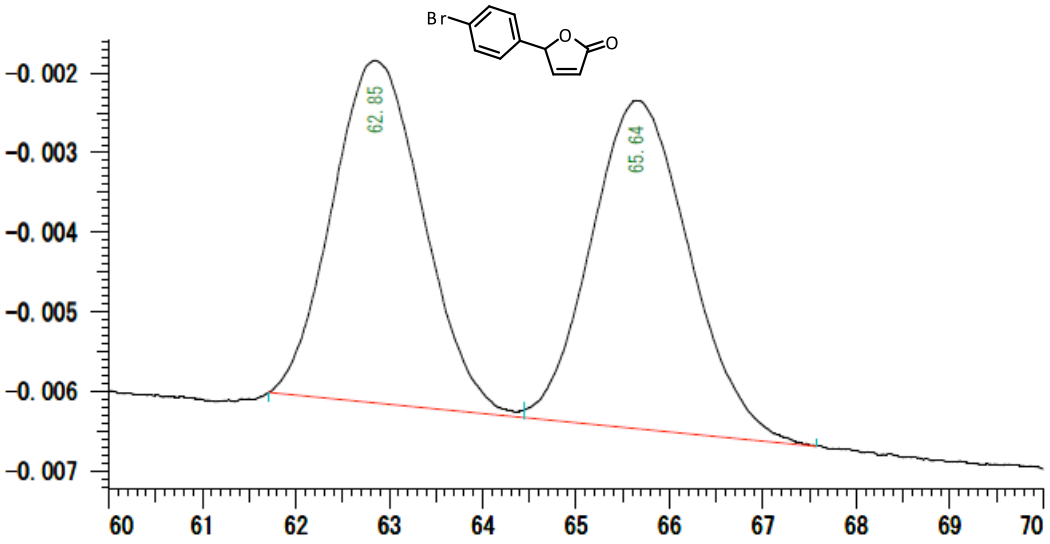
検出器A Ch1 220nm

Peak	Retention Time	Area	Area%
1	30.476	6285087	50.013
2	33.356	6281706	49.987
合計		12566794	100.000

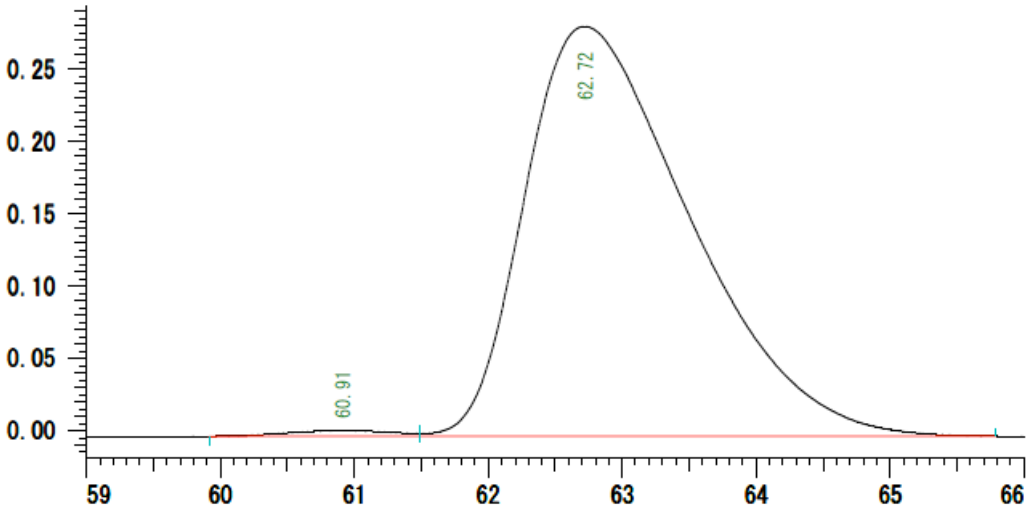


	Retention Time	Area	Area%
1	31.09	4217922	98.584
2	34.50	60568	1.416
		4278490	100.000

5-(4-bromophenyl)furan-2(5*H*)-one (**6bb**).

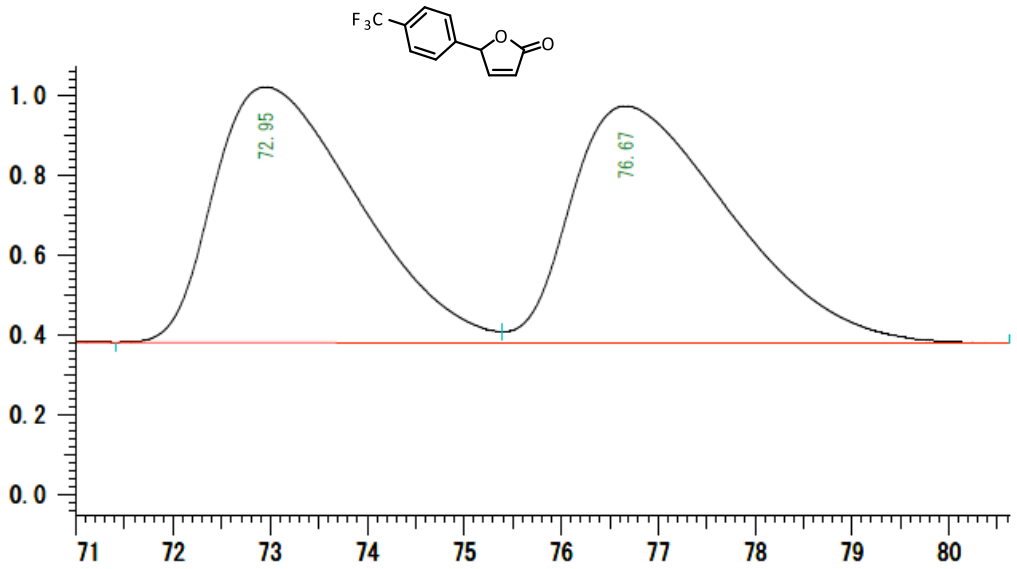


	Retention Time	Area	Area%
1	62.85	146131	49.121
2	65.64	151360	50.879
		297491	100.000

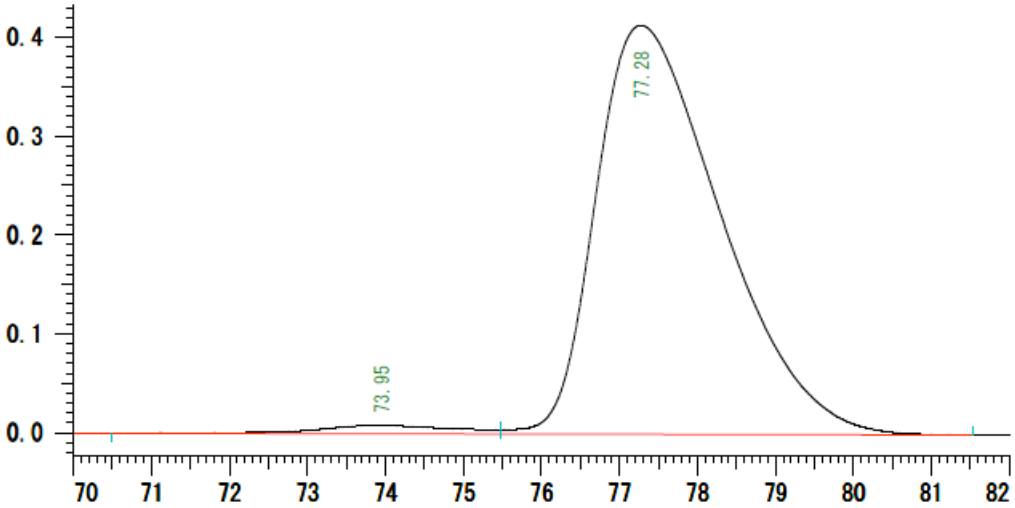


	Retention Time	Area	Area%
1	60.91	111406	0.911
2	62.72	12117867	99.089
		12229273	100.000

5-(4-trifluoromethylphenyl)furan-2(5*H*)-one (6cb).

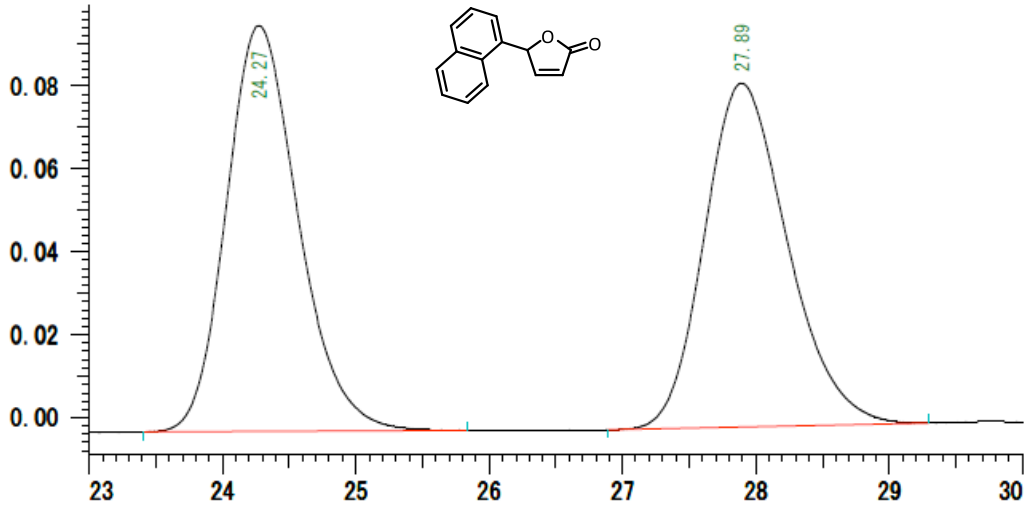


	Retention Time	Area	Area%
1	72.95	33209661	49.408
2	76.67	34005603	50.592
		67215264	100.000

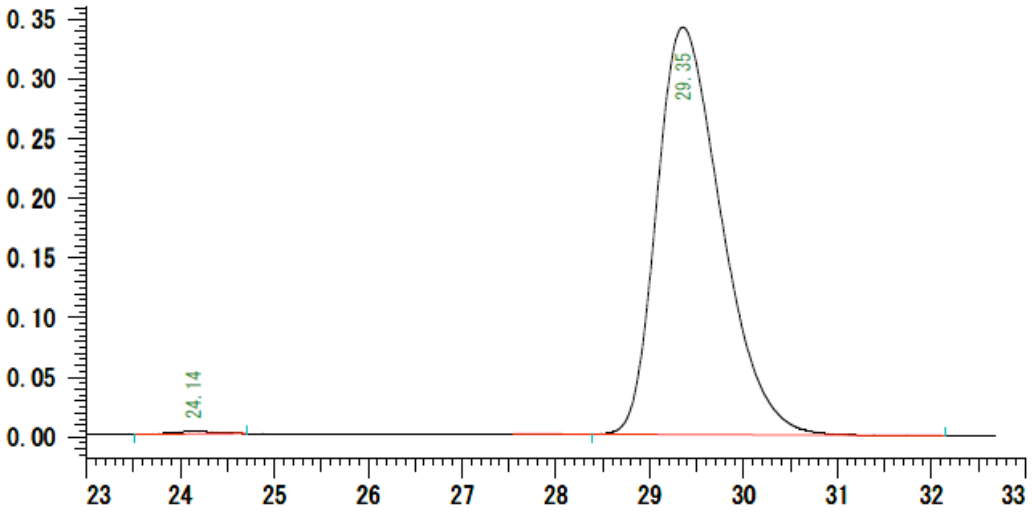


	Retention Time	Area	Area%
1	73.95	546325	2.323
2	77.28	22970352	97.677
		23516677	100.000

5-(naphthalene-1-yl)furan-2(5*H*)-one (6db).

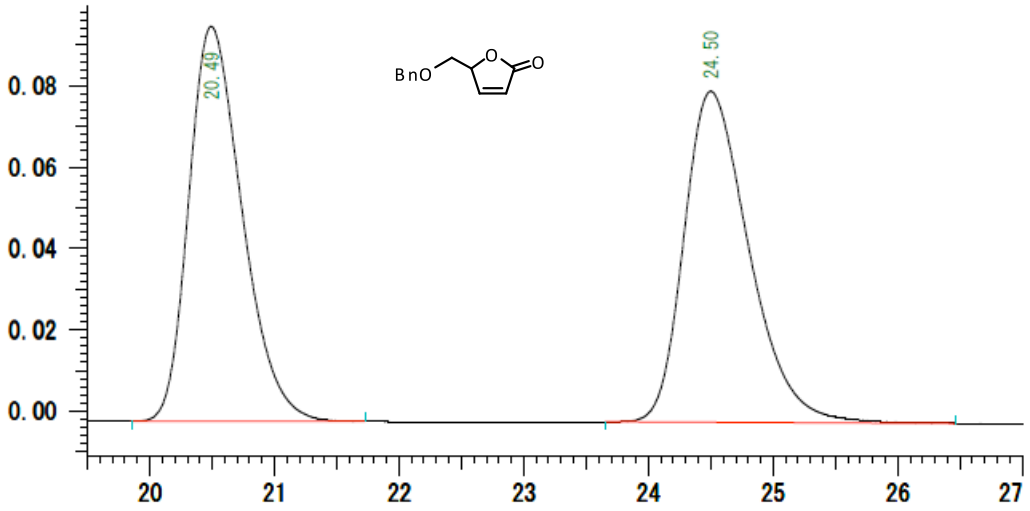


	Retention Time	Area	Area%
1	24.27	1833037	50.460
2	27.89	1799647	49.540
		3632684	100.000

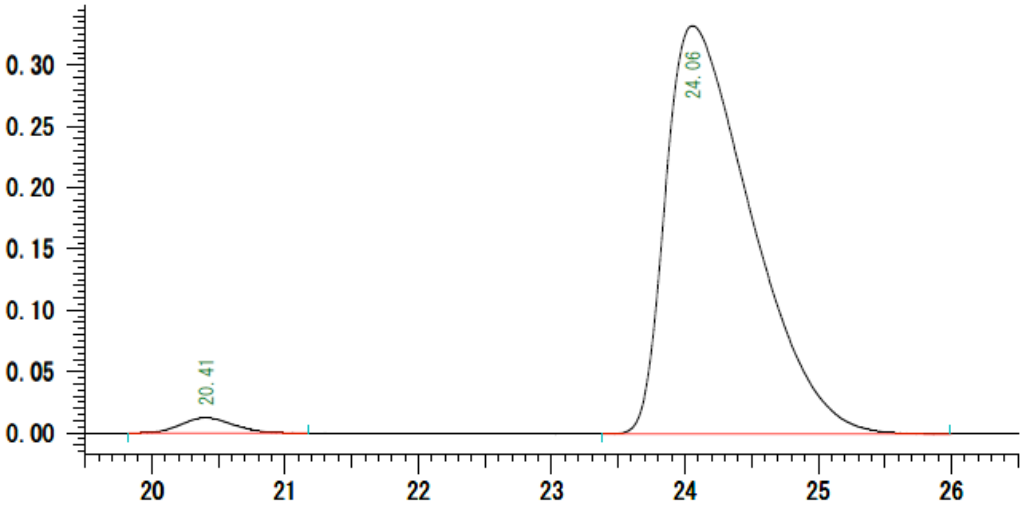


	Retention Time	Area	Area%
1	24.14	31891	0.389
2	29.35	8157308	99.611
		8189199	100.000

5-(benzyloxymethyl)furan-2(5*H*)-one (6fb).

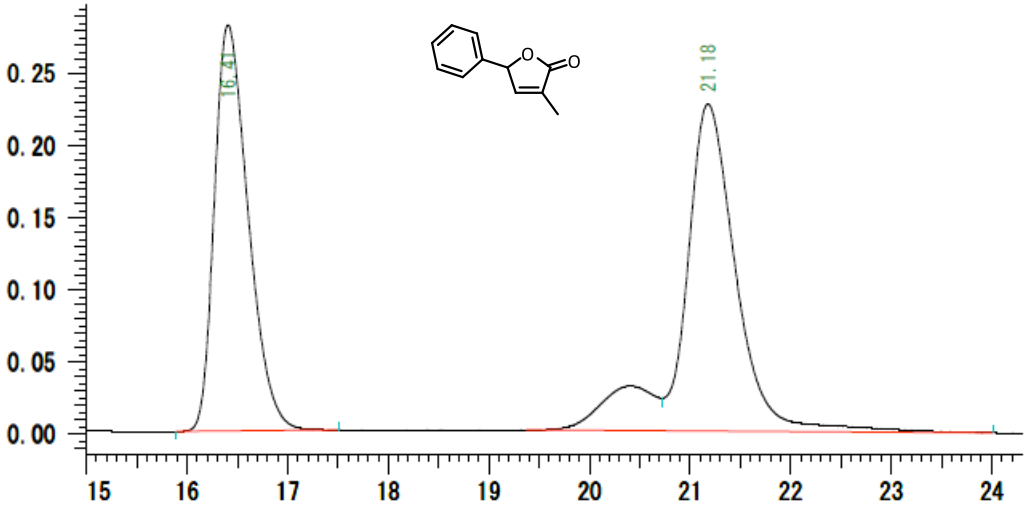


	Retention Time	Area	Area%
1	20.49	1445719	49.454
2	24.50	1477660	50.546
		2923379	100.000

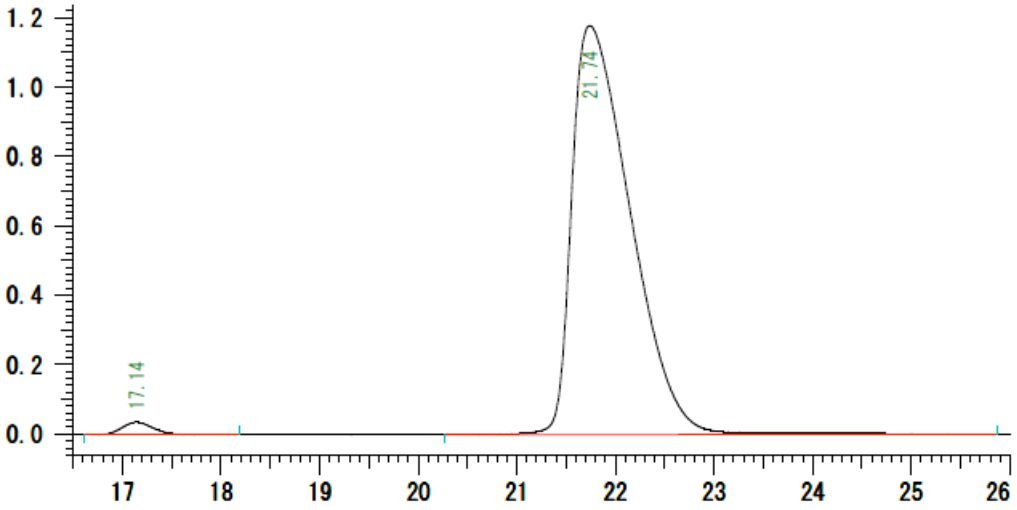


	Retention Time	Area	Area%
1	20.41	173573	2.287
2	24.06	7415562	97.713
		7589135	100.000

3-methyl-5-phenylfuran-2(5*H*)-one (**6ac**).

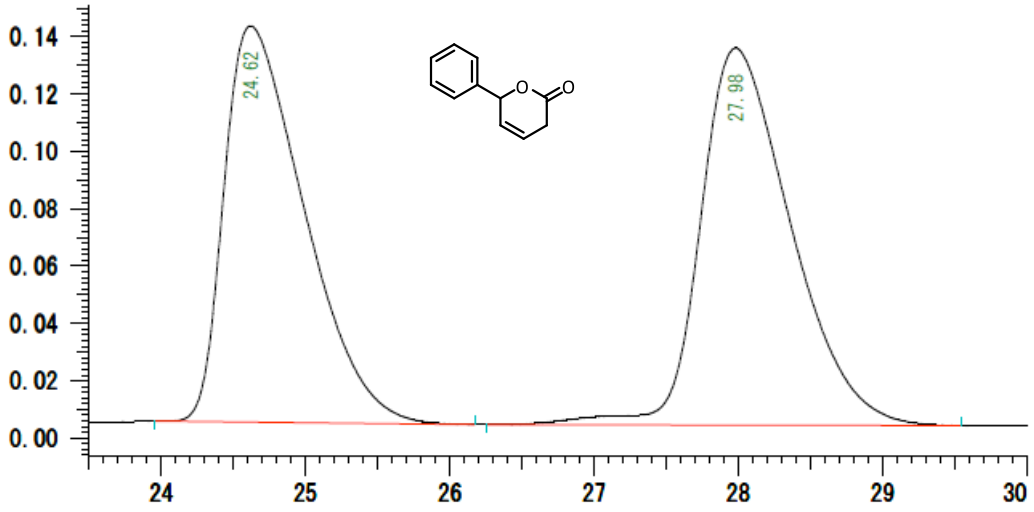


	Retention Time	Area	Area%
1	16.41	3289069	46.868
2	21.18	3728611	53.132
		7017680	100.000

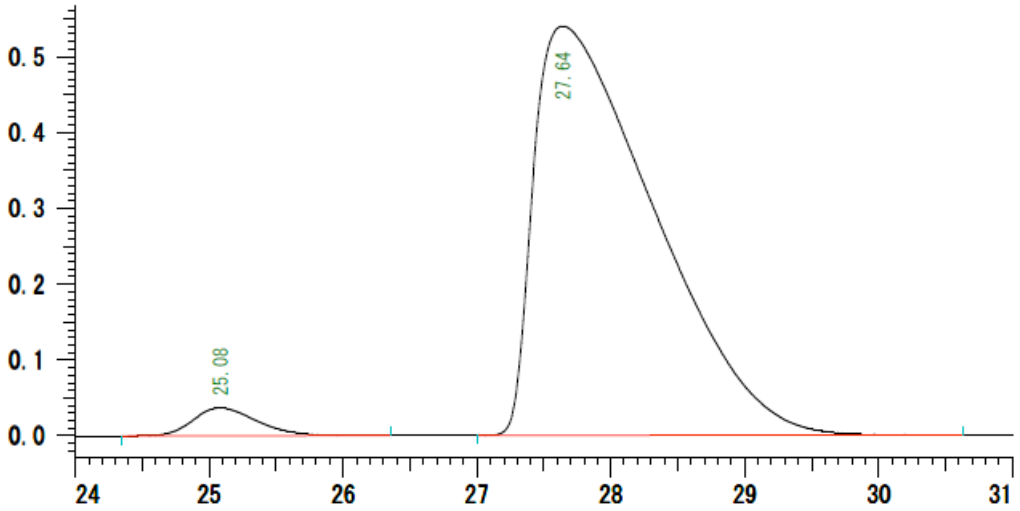


	Retention Time	Area	Area%
1	17.14	416218	1.674
2	21.74	24442188	98.326
		24858406	100.000

6-phenyl-3,6-dihydro-2*H*-pyran-2-one (8).

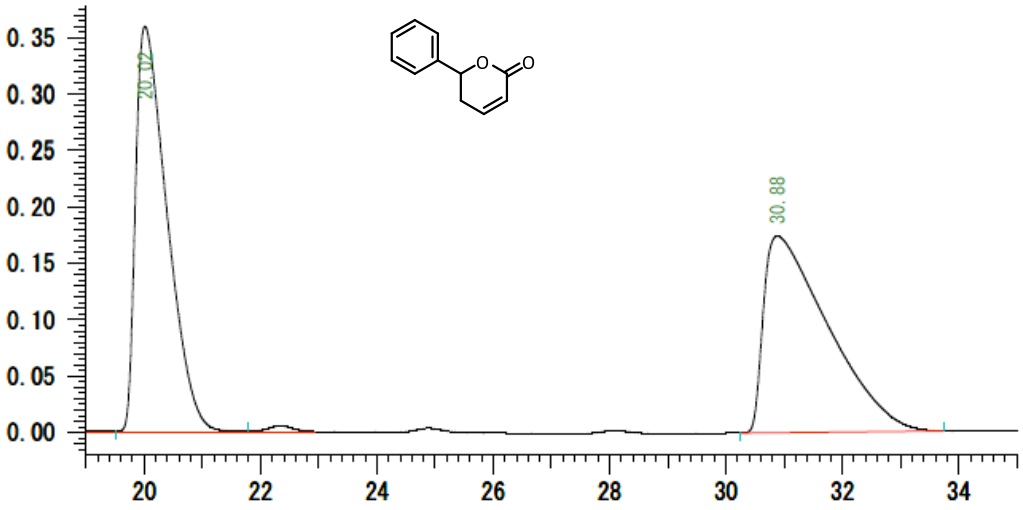


	Retention Time	Area	Area%
1	24.62	2661193	48.161
2	27.98	2864400	51.839
		5525593	100.000

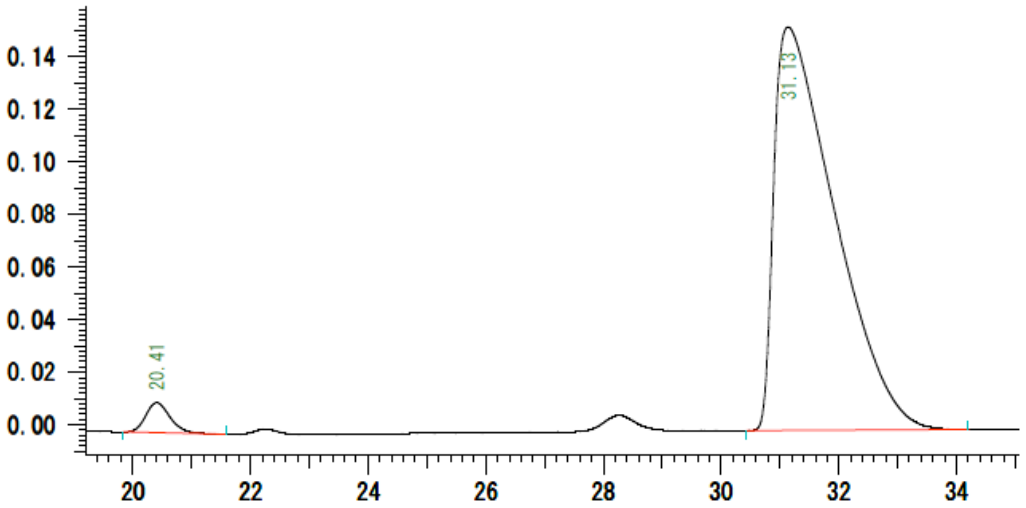


	Retention Time	Area	Area%
1	25.08	608772	3.490
2	27.64	16835038	96.510
		17443810	100.000

6-phenyl-5,6-dihydro-2*H*-pyran-2-one (**9**).

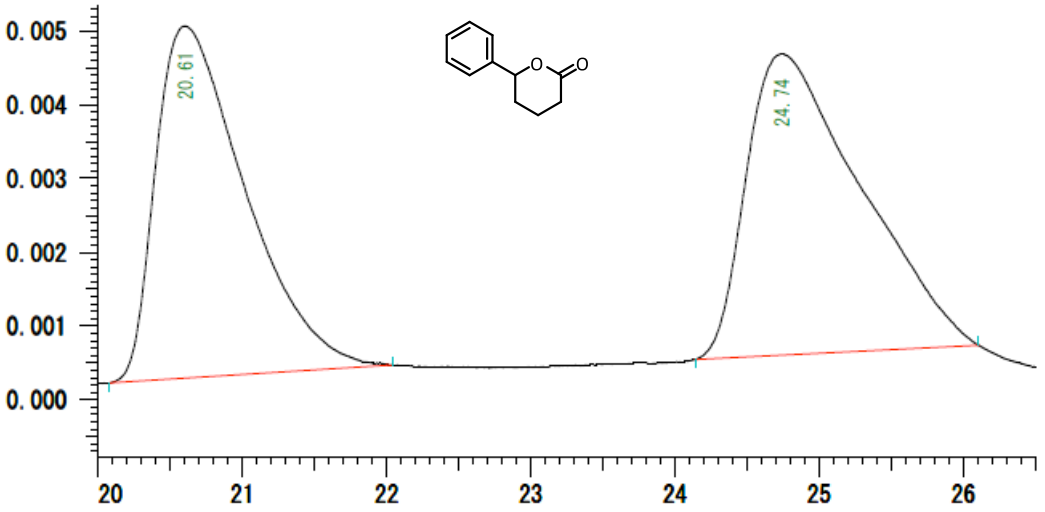


	Retention Time	Area	Area%
1	20.02	6714908	50.040
2	30.88	6704306	49.960
		13419214	100.000

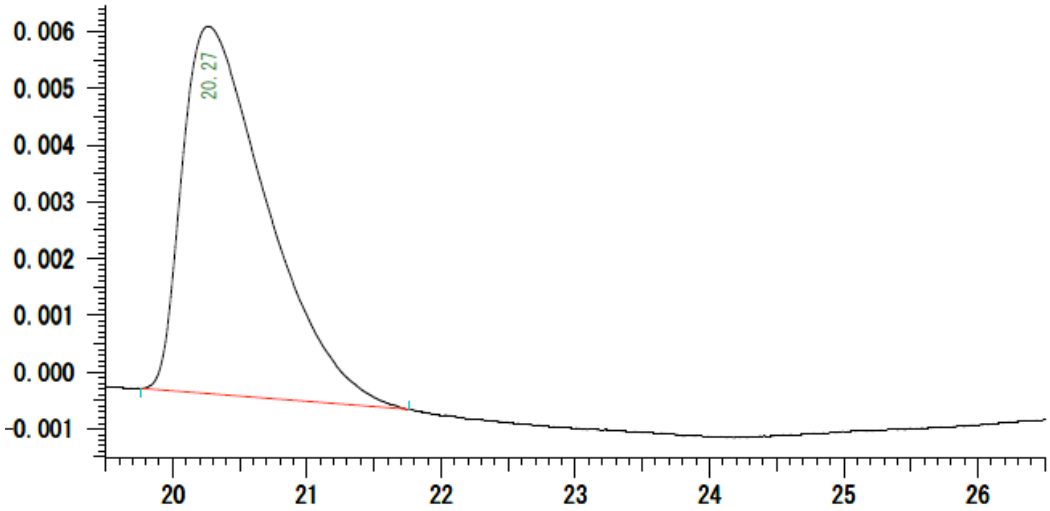


	Retention Time	Area	Area%
1	20.41	164561	2.911
2	31.13	5487714	97.089
		5652275	100.000

6-phenyltetrahydro-2*H*-pyran-2-one (10).

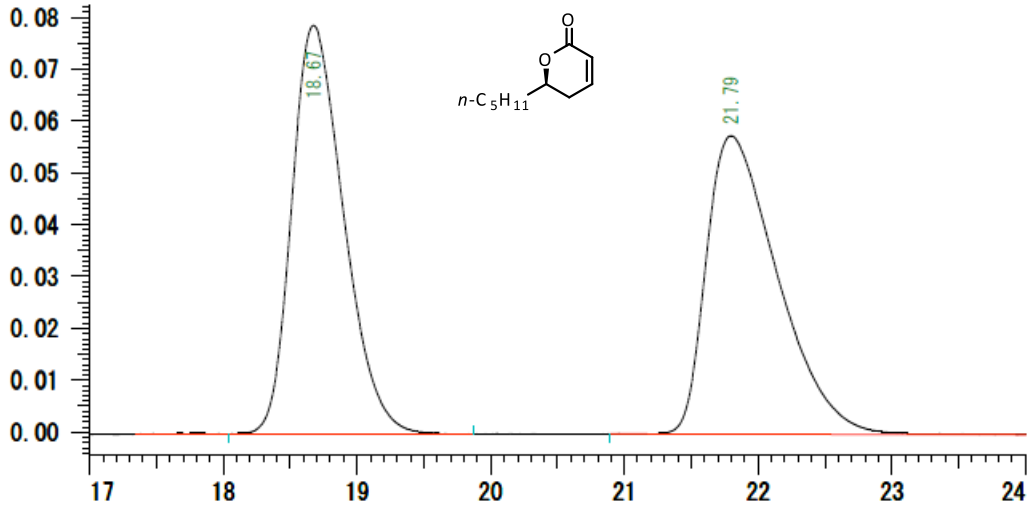


	Retention Time	Area	Area%
1	20.61	102700	47.868
2	24.74	111846	52.132
		214546	100.000

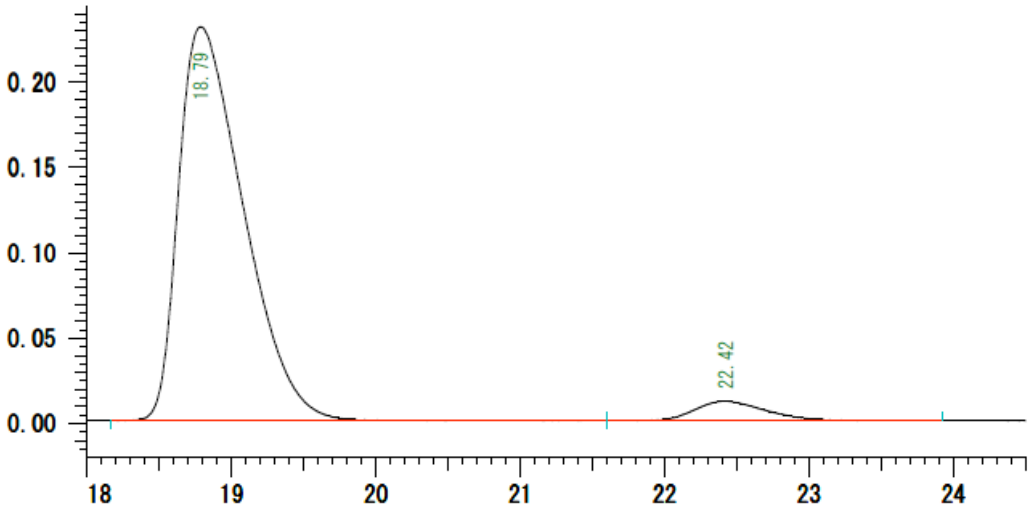


	Retention Time	Area	Area%
1	20.27	142881	100.000
		142881	100.000

(*R*)-6-pentyl-5,6-dihydro-2H-pyran-2-one, (*R*)-(–)-massoialactone, (14).



	Retention Time	Area	Area%
1	18.67	1069022	50.008
2	21.79	1068667	49.992
		2137689	100.000



	Retention Time	Area	Area%
1	18.79	3601218	94.980
2	22.42	190327	5.020
		3791545	100.000