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Supporting Information

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Contact Ion Pair Directed Lewis-Acid Catalysis: Asymmetric Formation of *trans*-Configured β-Lactones

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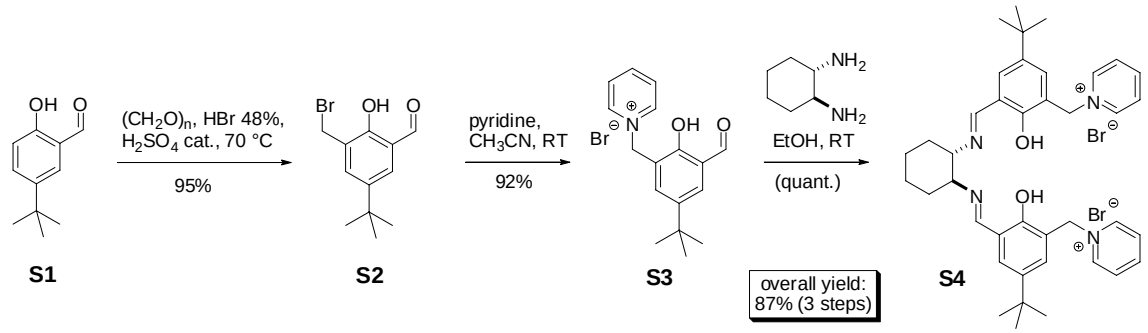
Experimental

Except as otherwise indicated, all reactions were carried out in oven or flame dried glassware under a positive pressure of argon. Dichloromethane was purified by distillation and dried by a passage over activated alumina under nitrogen atmosphere. All aldehydes and *N*,*N*-diisopropylethylamine (Acros, >99.5%) were distilled from CaH_2 under nitrogen. 5-Hexenal was prepared from the corresponding commercially available alcohol according to published procedures.[1] Enantiomerically pure (+)-(*S*,*S*)-1,2-diaminocyclohexane was generously donated by *Reuter Chemische Apparatebau KG* (RCA; Freiburg, Germany). Propionyl bromide (Aldrich, 98%), *n*-Valeroyl bromide (TCI Europe., >98%), 5-*tert*-butyl-2-hydroxy-benzaldehyde (Aldrich, 98%), pyridine (Fluka, >99.8%), anhydrous pentane (Fluka, over molecular sieve, >99.5%), *n*-hexane (Fluka, UV quality), cyclohexane (Thommen & Furler), ethyl acetate (Thommen & Furler), diethyl ether (Fluka) and triethylamine (Fluka, >99.5%) were used as purchased. All other laboratory chemicals were purchased from *ABCR*, *Acros*, *Aldrich*, *Fluka*, *J.T. Baker* or *Merck* and were used without purification. For work-up procedures and flash chromatography, distilled technical grade solvents were used. Unless otherwise indicated, all liquids were added *via* syringe, solids were added neat against an argon flow. Solvents were removed at a heating bath temperature of 40 °C and 800 - 30 mbar pressure by rotary evaporation. Non-volatile compounds were dried *in vacuo* at 0.01 mbar. Except as otherwise indicated, reactions were magnetically stirred and monitored either by ^{1}H-NMR spectra or thin layer chromatography (TLC) using silica gel plates from *Merck* (silica gel 60 F_{254}). Visualization occurred by fluorescence quenching under UV light and by staining with aqueous $KMnO_4$ / NaOH. Purification by flash chromatography was performed on silica gel 60 Å, 32-62, provided by *Fluka,* using a forced flow of eluent at 0.2-0.4 bar pressure. NMR-spectra were recorded on a *Varian Gemini 300*, a *Varian Mercury 300* and a *Bruker ARX300* spectrometer operating at 300 MHz (^{1}H) and 75 MHz (^{13}C). Chemical shifts δ are referred in terms of ppm and *J*-coupling

constants are given in Hz. Abbreviations for multiplicity are as follows: *s* (singulet), *d* (doublet), *t* (triplet), *q* (quadruplet), *m* (multiplet), *b* (broad signal). IR-spectra were recorded on a *Perkin Elmer Spectrum One FT-IR* with a *Universal ATR Sampling Accessory* and the signals are given by wave numbers (cm^{-1}). Optical rotation was measured on a *Jasco DIP-100 digital polarimeter* operating at the sodium D line with a 100 mm path length cell. Melting points were measured using a *Büchi 535* melting point apparatus in open glass capillaries and are uncorrected. Mass spectra were obtained from the ETH Zürich MS Service. High resolution EI mass spectra were performed on a *Micromass AutoSpec Ultima* and were calibrated with perfluorotributylamine (PFTBA) prior to data acquisition. High resolution ESI mass spectra were performed on an *Ion Spec Ultima 2 FTICR*. ESI mass spectra were performed on a *Finnigan TSQ7000*. Combustion analysis was performed by the Mikroelementaranalytisches Laboratorium at ETH Zürich. Analytical gas chromatography (GC) was performed on a *Hewlett Packard HP6890 Series gas chromatograph* and a *ThermoFinnigan TraceGC gas chromatograph* with a flame ionization detector using a *Chrompac Chirasil-Dex CB silica capillary column* (25 m x 0.25 mm x 0.25 μm film thickness). Hydrogen was used as the carrier gas at the indicated pressure. Analytical high performance liquid chromatography (HPLC) was performed on a *Hitachi LaChrom Elite liquid chromatograph* equipped with a variable wavelength UV detector (deuterium lamp, 190-600 nm), using a *Daicel Chiralcel^{TM} OD-H column* (25 x 0.46 cm). HPLC grade isopropanol and hexanes were used as the eluting solvents.

Synthesis of salen ligand S4



1-(5-*tert*-Butyl-3-formyl-2-hydroxybenzyl)pyridinium bromide (S3)[2]

3-Bromomethyl-5-*tert*-butyl-2-hydroxy-benzaldehyde (**S2**)[3] (6.07 g, 22.4 mmol) was dissolved in dry acetonitrile (80 mL) and pyridine (1.90 mL, 23.5 mmol) was added. The suspension was stirred for 14 h at ambient temperature. Et_2O (50 mL) was added and the mixture was filtered. After washing with Et_2O (50 mL) the product was dried *in vacuo* to give **S3** as a white solid (7.20 g, 20.6 mmol, yield: 92%). No further purification was necessary.

$C_{17}H_{20}BrNO_2$, **MW:** 350.25 g/mol. **Mp:** >250 °C. **^{1}H NMR (300 MHz, DMSO, 21 °C):** δ = 11.16 (*s*, 1 H, $C_{Ar}OH$); 10.05 (*s*, 1 H, C*H*O); 9.14 (*d*, *J* = 5.5, 2 H, *o*-CH_{Pyr}); 8.60 (*tt*, *J* = 7.6, 1.3, 1 H, *p*-CH_{Pyr}); 8.15 (*m*, 2 H, *m*-CH_{Pyr}); 8.09 (*d*, *J* = 2.6, 1 H, CH_{Ar}); 7.88 (*d*, *J* = 2.6, 1 H, CH_{Ar}); 5.89 (*s*, 2 H, $C_{Ar}CH_2$); 1.22 (*s*, 9 H, $C(CH_3)_3$). **^{13}C NMR (75 MHz, DMSO, 21 °C):** δ = 196.3, 156.7, 145.9, 145.0, 142.8, 136.3, 130.7, 128.1, 121.9, 121.2, 58.9, 34.1, 30.9. **HRMS (ESI) *m/z*:** Calc. for $[M\text{-}Br]^+$: 270.1489. Found: 270.1489. **Anal. Calcd. for $C_{17}H_{20}BrNO_2$:** C, 58.30; H, 5.76; N, 4.00; O, 9.14; Br, 22.81. Found: C, 58.36; H, 5.69; N, 4.06.

(*S*,*S*)-(+)-N,N'-Bis(3-*tert*-butyl-5-(pyridinium-1-ylmethyl)salicylidene)-1,2-cyclohexane-diamine dibromide (S4)

To a solution of (1*S*,2*S*)-(+)-1,2-diaminocyclohexane (0.20 g, 1.78 mmol, 1 equiv.) in ethanol (20 mL) at ambient temperature aldehyde **S3** (1.26 g, 3.56 mmol, 2 equiv.) and molecular sieves

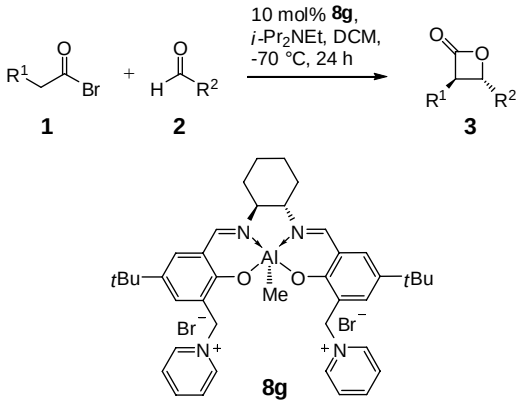
(4Å) were added. The mixture was stirred for 24 h at ambient temperature. After filtration, EtOH was removed *in vacuo*. Subsequent repetitive azeotropic removal of residual EtOH with DCM gave **S4** as an orange solid (1.38 g, 1.78 mmol, yield: 100%). No further purification was necessary.

$C_{40}H_{50}Br_2N_4O_2$, **MW:** 778.66 g/mol. **Mp:** decomposition above 195 °C. **$[\alpha]_D^{24.3°C}$** (c = 1.100, acetonitrile) = +320.5 ± 0.1. **^{1}H NMR (300 MHz, CD_2Cl_2, 21 °C):** δ = 14.16 (*bs*, 2 H, $C_{Ar}OH$); 9.55 (*d*, *J* = 5.5, 4 H, *o*-CH_{Pyr}); 8.46 (*t*, *J* = 7.8, 2 H, *p*-CH_{Pyr}); 8.38 (*s*, 2 H, N=CHC_{Ar}); 8.22 (*d*, *J* = 2.5, 2 H, CH_{Ar}); 7.99 (*m*, 4 H, *m*-CH_{Pyr}); 7.32 (*d*, *J* = 2.5, 2 H, CH_{Ar}); 6.13 (*dd*, 4 H, $C_{Ar}CH_2$); 3.43 (*d*, *J* = 9.5, 2 H, $(CH)_{ring}$-N); 1.90 (*m*, 4 H, $(CH_2)_{ring}$); 1.67 (*m*, 2 H, $(CH_2)_{ring}$); 1.47 (*m*, 2 H, $(CH_2)_{ring}$); 1.27 (*s*, 18 H, $C(CH_3)_3$). **^{13}C NMR (75 MHz, CD_2Cl_2, 21 °C):** δ = 164.9, 159.1, 145.4, 145.3, 142.0, 133.0, 130.3, 128.1, 120.7, 118.2, 71.6, 60.1, 34.6, 33.2, 31.6, 24.5. **HRMS (ESI) *m/z*:** Calc. for $[M^{2+}\text{-}Br^-]^+$: 697.3112. Found: 697.3114.

Formation of the active catalyst 8g

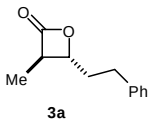
To a solution of diimine **S4** (0.16 g, 0.21 mmol, 1 equiv.) in DCM (3.5 mL) a solution of Me_3Al in toluene (2 M, 0.10 mL, 0.21 mmol, 1 equiv.) was added. The mixture was stirred for 3 h at ambient temperature. Pentane (5 mL) was added to fully precipitate complex **8g** and the mixture was filtered under nitrogen. Washing the filter cake with an additional amount of pentane (5 mL) and drying *in vacuo* afforded the active catalyst as bright yellow powder in quantitative yield. To avoid decomposition, the complex was synthesized at the same day as the catalysis was carried out.

General procedure for the formation of β-lactones 3



To a mixture of complex **8g** (0.075 mmol, 0.1 equiv.) in DCM (3 mL) was successively added at −70 °C aldehyde **2** (0.75 mmol, 1 equiv.), acid bromide **1** (4.5 mmol, 6 equiv.) and diisopropylethylamine (1.875 mmol, 2.5 equiv.). The resulting heterogeneous mixture was stirred at −70 °C for 24 h. The reaction mixture was poured into aqueous 1 M HCl (30 mL) and extracted with DCM (2 x 20 mL). The combined organic phase was dried over $MgSO_4$ and filtered through a short plug of silica gel. DCM was subsequently removed *in vacuo*. For β-lactones **3a**, **3c**, **3e** and **3h** the yield was determined by ^{1}H-NMR using acetophenone as internal standard. The crude product mixtures of all other β-lactones were purified by flash chromatography.

(3*R*,4*R*)-*trans*-3-Methyl-4-(2-phenylethyl)oxetan-2-one (3a)

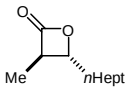


β-Lactone **3a** (0.41 mmol, yield: 82%, *ee* = 88% *dr* = 97:3) was prepared from propionylbromide (**1a**) and 3-phenylpropionaldehyde (**2a**) according to the general procedure, but using 0.50 mmol of **2a** in 2 mL of DCM. The *dr* value was determined by ^{1}H-NMR and the *ee* value by HPLC

(Chiralcel OD-H, 97:3 *n*-hexane/*i*PrOH, 1.0 mL/min, 210 nm). An analytically pure sample was obtained as colorless oil by flash chromatography (pentane / diethyl ether 20:1).

$C_{12}H_{14}O_2$, **MW:** 190.24 g/mol. Spectral data for the racemate have been reported earlier.[4] **[α]** $_D^{25.9°C}$ (c = 1.280, $CHCl_3$) = +67.4 ± 0.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 7.34-7.17 (*m*, 5 H, CH_{Ar}); 4.16 (*ddd*, *J* = 7.5, 5.9, 4.0, 1 H, C*H*-O); 3.20 (*qd*, *J* = 7.5, 4.0, 1 H, C*H*-C(O)); 2.77 (*m*, 2 H, $CH_2CH_2C_{Ar}$); 2.13 (*m*, 2 H, $CH_2CH_2C_{Ar}$); 1.32 (*d*, *J* = 7.5, 3 H, CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 171.6, 139.9, 128.5, 128.2, 126.3, 78.6, 50.8, 35.6, 31.4, 12.5. **IR (ATR):** ν = 2935, 1816, 1603, 1124, 840, 698. **HRMS (EI) *m/z*:** Calc. for [M^+]: 190.0988. Found: 190.0989. **Anal. Calcd. for $C_{12}H_{14}O_2$**: C, 75.76; H, 7.42; O, 16.82. Found: C, 75.85; H, 7.53.

(3*R*,4*R*)-*trans*-3-Methyl-4-heptyloxetan-2-one (3b)



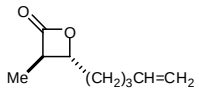
3b

β-Lactone **3b** was prepared from propionylbromide (**1a**) and octanal according to the general procedure. Purification by flash chromatography (pentane / diethyl ether 20:1) gave **3b** as colorless oil (107 mg, 0.58 mmol, 77%, *ee* = 87% *dr* = 96:4). The *dr* value was determined by ^{1}H-NMR and the *ee* value by GC (ThermoFinnigan TraceGC, Chrompac Chirasil-Dex CB column 25 m x 0.25 mm, flow rate 1.1 mL/min, method: 60 °C, ramp @ 5 °C/min to 180 °C, hold 20 min).

$C_{11}H_{20}O_2$, **MW:** 184.28 g/mol. Spectral data for the racemate has been reported earlier.[4] **[α]** $_D^{26.4°C}$ (c = 1.175, $CHCl_3$) = +45.6 ± 0.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 4.17 (*td*, *J* = 6.6, 4.0, 1 H, C*H*-O); 3.21 (*qd*, *J* = 7.5, 4.0, 1 H, C*H*-C(O)); 1.93-1.68 (*m*, 2 H, CH_2CH_2CH-O); 1.39 (*d*, *J* = 7.5, 3 H, $CHCH_3$) 1.49-1.28 (*m*, 10 H, $(CH_2)_5CH_3$); 0.88 (*t*, *J* = 6.6, 3 H, CH_2CH_3).

^{13}C NMR (75 MHz, $CDCl_3$, 21 °C): δ = 171.9, 79.5, 50.7, 34.2, 31.7, 29.2, 29.1, 25.0, 22.6, 14.1, 12.6. **IR (ATR):** ν = 2927, 1821, 1123. **HRMS (EI) *m/z*:** Calc. for $[M-H]^+$: 183.1380. Found: 183.1382. **Anal. Calcd. for $C_{11}H_{20}O_2$**: C, 71.70; H, 10.94; O, 17.36. Found: C, 71.55; H, 10.80.

(3*R*,4*R*)-*trans*-3-Methyl-4-(4-pentenyl)oxetan-2-one (3c)

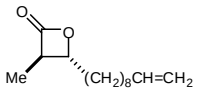


3c

β-Lactone **3c** (0.37 mmol, yield: 74%, *ee* = 88%, *dr* = 96:4) was prepared from propionylbromide (**1a**) and 5-hexenal according to the general procedure, but using 0.50 mmol of aldehyde in 2 mL DCM The *dr* value was determined by ^{1}H-NMR and the *ee* value by GC (Hewlett Packard HP6890, Chrompac Chirasil-Dex CB column 25 m x 0.25 mm, flow rate 1.1 mL/min, method: 60 °C for 2 min, ramp @ 5 °C/min to 150 °C, hold 0.75 min, ramp @ 8 °C/min to 200 °C). An analytically pure sample was obtained as colorless oil by flash chromatography (pentane / diethyl ether 20:1).

$C_9H_{14}O_2$, **MW:** 154.21 g/mol. Spectral data for the racemate has been reported earlier.[4] **[α]** $_D^{21.8°C}$ (c = 1.025, $CHCl_3$) = +58.7 ± 0.3. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 5.78 (*ddt*, *J* = 16.9, 10.2, 6.7, 1 H, CH_2CH=CH_2); 5.07-4.97 (*m*, 2 H, CH=CH_2); 4.18 (*ddd*, *J* = 7.3, 6.1, 4.0, 1 H, C*H*-O); 3.23 (*qd*, *J* = 7.5, 4.0, 1 H, C*H*-C(O)); 2.12 (*m*, 2 H, CH_2CH=CH_2); 1.93-1.71 (*m*, 2 H, CH_2CH_2CH-O); 1.64-1.42 (*m*, 2 H, $CH_2CH_2CH_2$); 1.39 (*d*, *J* = 7.5, 3 H, $CHCH_3$). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 171.9, 137.6, 115.3, 79.3, 50.7, 33.4, 33.1, 24.1, 12.4. **IR (ATR):** ν = 2936, 1817, 1641, 1123. **HRMS (EI) *m/z*:** Calc. for $[M^+]$: 154.0989. Found: 154.0991.

(3*R*,4*R*)-*trans*-3-Methyl-4-(9-decenyl)oxetan-2-one (3d)

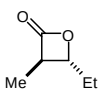


3d

β-Lactone **3d** was prepared from propionylbromide (**1a**) and undecylenic aldehyde according to the general procedure. Purification by flash chromatography (pentane / diethyl ether 20:1) gave **3d** as colorless oil (104 mg, 0.47 mmol, 62%, *ee* = 87%, *dr* = 94:6). The *dr* value was determined by ^{1}H-NMR and the *ee* value by GC (ThermoFinnigan TraceGC, Chrompac Chirasil-Dex CB column 25 m x 0.25 mm, flow rate 1.1 mL/min, method: 60 °C, ramp @ 5 °C/min to 180 °C, hold 20 min).

$C_{14}H_{24}O_2$, MW: 224.34 g/mol. **[α]** $_{D}^{27.0°C}$ (c = 1.245, $CHCl_3$) = +40.5 ± 0.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 5.81 (*ddt*, *J* = 16.8, 10.1, 6.7, 1 H, CH_2CH=CH_2); 5.03-4.91 (*m*, 2 H, CH=CH_2); 4.17 (*ddd*, *J* = 7.1, 6.3, 4.0, 1 H, C*H*-O); 3.22 (*qd*, *J* = 7.5, 4.0, 1 H, C*H*-C(O)); 2.04 (*m*, 2 H, CH_2CH=CH_2); 1.93-1.68 (*m*, 2 H, CH_2CH_2CH-O); 1.45-1.25 (*m*, 12 H, $CH_2(CH_2)_6CH_2$); 1.39 (*d*, *J* = 7.5, 3H, CHCH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 171.9, 139.0, 114.1, 79.5, 50.7, 34.2, 33.8, 29.4, 29.3, 29.2, 29.1, 28.9, 25.9, 12.6. **IR (ATR):** ν = 2926, 1821, 1640, 1122. **HRMS (EI) *m/z*:** Calc. for [M^+]: 224.1776. Found: 224.1776. **Anal. Calcd. for $C_{14}H_{24}O_2$**: C, 74.95; H, 10.78; O, 14.26. Found: C, 75.06; H, 10.56.

(3*R*,4*R*)-*trans*-3-Methyl-4-ethyloxetan-2-one (3e)



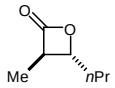
3e

β-Lactone **3e** (0.76 mmol, yield: 76%, *ee* = 87% *dr* = 95:5) was prepared from propionylbromide (**1a**) and propanal according to the general procedure, but using 1.00 mmol of aldehyde in 4 mL of DCM. The *dr* value was determined by ^{1}H-NMR and the *ee* value by GC (ThermoFinnigan

TraceGC, Chrompac Chirasil-Dex CB column 25 m x 0.25 mm, flow rate 1.1 mL/min, method: 60 °C, ramp @ 5 °C/min to 180 °C, hold 20 min). An analytically pure sample was obtained as light-yellow oil by flash chromatography (pentane / diethyl ether 20:1).

$C_6H_{10}O_2$, MW: 114.14 g/mol. **$[\alpha]_D^{25.5°C}$** (c = 1.625, $CHCl_3$) = +55.0 ± 0.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 4.13 (*td*, *J* = 6.6, 4.0, 1 H, C*H*-O); 3.23 (*qd*, *J* = 7.5, 4.0, 1 H, C*H*-C(O)); 1.97-1.72 (*m*, 2 H, CH_3CH_2CH-O); 1.39 (*d*, *J* = 7.5, 3 H, $CHCH_3$); 1.02 (*t*, *J* = 7.5, 3 H, CH_2CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 171.8, 80.5, 50.3, 27.3, 12.7, 9.1. **IR (ATR):** ν = 2937, 1816, 1125, 848. **HRMS (EI) *m/z*:** Calc. for $[M-H]^+$: 113.0597. Found: 113.0592.

(3*R*,4*R*)-*trans*-3-Methyl-4-propyloxetan-2-one (3f)

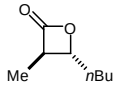


3f

β-Lactone **3f** was prepared from propionylbromide (**1a**) and butanal according to the general procedure. Purification by flash chromatography (pentane / diethyl ether 20:1) gave **3f** as colorless oil (64 mg, 0.50 mmol, 67%, *ee* = 93%, *dr* = 97:3). The *dr* value was determined by ^{1}H-NMR and the *ee* value by GC (Hewlett Packard HP6890, Chrompac Chirasil-Dex CB column 25 m x 0.25 mm, flow rate 1.1 mL/min, method: 60 °C for 2 min, ramp @ 5 °C/min to 150 °C, hold 0.75 min, ramp @ 8 °C/min to 200 °C).

$C_7H_{12}O_2$, MW: 128.17 g/mol. **$[\alpha]_D^{23.2°C}$** (c = 1.005, $CHCl_3$) = +65.5 ± 0.1. Spectral data for **3f** has been reported earlier.[5] **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 4.19 (*ddd*, *J* = 7.4, 6.1, 4.0, 1 H, C*H*-O); 3.23 (*qd*, *J* = 7.5, 4.0, 1 H, C*H*-C(O)); 1.92-1.68 (*m*, 2 H, CH_2CH_2CH-O); 1.55-1.35 (*m*, 2 H, CH_2CH_3); 1.39 (*d*, *J* = 7.5, 3H, $CHCH_3$); 0.99 (*t*, *J* = 7.4, 3 H, CH_2CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 171.8, 79.3, 50.7, 36.1, 18.4, 13.7, 12.5. **IR (ATR):** ν = 2962, 1817, 1124, 872, 814. **HRMS (EI) *m/z*:** Calc. for $[M^+]$: 128.0832. Found: 128.0832.

(3*R*,4*R*)-*trans*-3-Methyl-4-butyloxetan-2-one (3g)

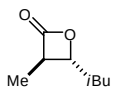


3g

β-Lactone **3g** was prepared from propionylbromide (**1a**) and pentanal according to the general procedure, but using 1.00 mmol of aldehyde in 4 mL of DCM. Purification by flash chromatography (pentane / diethyl ether 20:1) gave **3g** as colorless oil (91 mg, 0.64 mmol, 64%, *ee* = 89%, *dr* = 97:3). The *dr* value was determined by ^{1}H-NMR and the *ee* value by GC (ThermoFinnigan TraceGC, Chrompac Chirasil-Dex CB column 25 m x 0.25 mm, flow rate 1.1 mL/min, method: 60 °C, ramp @ 5 °C/min to 180 °C, hold 20 min).

$C_8H_{14}O_2$, **MW:** 142.20 g/mol. Spectral data for the racemate has been reported earlier.[6] **$[\alpha]_D^{28.3°C}$** (c = 1.080, $CHCl_3$) = +58.0 ± 0.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 4.18 (*ddd*, *J* = 7.1, 6.3, 4.0, 1 H, C*H*-O); 3.22 (*qd*, *J* = 7.5, 4.0, 1 H, C*H*-C(O)); 1.94-1.69 (*m*, 2 H, CH_2CH_2CH-O); 1.48-1.30 (*m*, 2 H, CH_2CH_3); 1.39 (*d*, *J* = 7.5, 3 H, $CHCH_3$); 0.93 (*t*, *J* = 7.1, 3 H, CH_2CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 171.9, 79.5, 50.7, 33.8, 27.1, 22.4, 13.9, 12.6. **IR (ATR):** ν = 2934, 1818, 1124, 840. **HRMS (EI) *m/z*:** Calc. for $[M-H]^+$: 141.0910. Found: 141.0911. **Anal. Calcd. for $C_8H_{14}O_2$**: C, 67.57; H, 9.92; O, 22.50. Found: C, 67.41; H, 9.72.

(3*R*,4*R*)-*trans*-3-Methyl-4-*iso*-butyloxetan-2-one (3h)



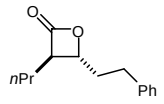
3h

β-Lactone **3h** (0.38 mmol, yield: 76%, *ee* = 87%, *dr* = 94:6) was prepared from propionylbromide (**1a**) and isovaleraldehyde according to the general procedure, but using 0.50 mmol of aldehyde in 2 mL of DCM. The *dr* value was determined by ^{1}H-NMR and the *ee* value

by GC (Hewlett Packard HP6890, Chrompac Chirasil-Dex CB column 25 m x 0.25 mm, flow rate 1.1 mL/min, method: 60 °C for 2 min, ramp @ 5 °C/min to 150 °C, hold 0.75 min, ramp @ 8 °C/min to 200 °C). An analytically pure sample was obtained as colorless oil by flash chromatography (pentane / diethyl ether 20:1).

$C_8H_{14}O_2$, MW: 142.20 g/mol. **$[\alpha]_D^{27.4°C}$** (c = 1.000, $CHCl_3$) = +59.9 ± 0.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 4.25 (*ddd*, *J* = 7.5, 5.9, 4.0, 1 H, C*H*-O); 3.21 (*qd*, *J* = 7.5, 4.0, 1 H, C*H*-C(O)); 1.85-1.71 (*m*, 2 H, CHCH_2CH-O); 1.67-1.56 (*m*, 1 H, C*H*$(CH_3)_2$); 1.39 (*d*, *J* = 7.5, 3 H, CHCH_3); 0.97 (*d*, *J* = 6.5, 3 H, CH_2CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 171.9, 78.5, 51.2, 43.0, 25.4, 22.8, 22.3, 12.5. **IR (ATR):** ν = 2959, 1817, 1120, 882. **HRMS (EI) *m/z*:** Calc. for [M^+]: 142.0988. Found: 142.0988.

(3*R*,4*R*)-*trans*-3-Propyl-4-(2-phenylethyl)oxetan-2-one (3i)



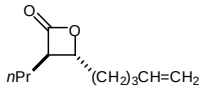
3i

β-Lactone **3i** was prepared from valeroylbromide and 3-phenylpropionaldehyde according to the general procedure. Purification by flash chromatography (DCM / pentane 3:1, then pentane / diethyl ether 40:1) gave **3i** as colorless oil (149 mg, 0.68 mmol, 91%, *ee* = 94%, *dr* = 98:2). The *dr* value was determined by ^{1}H-NMR and the *ee* value by HPLC (Chiralcel OD-H, 97:3 *n*-hexane/*i*PrOH, 1.0 mL/min, 210 nm).

$C_{14}H_{18}O_2$, MW: 218.29 g/mol. **$[\alpha]_D^{26.4°C}$** (c = 1.300, $CHCl_3$) = +59.2 ± 0.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 7.33-7.16 (*m*, 5 H, CH_{Ar}); 4.22 (*ddd*, *J* = 7.9, 5.5, 4.0, 1 H, C*H*-O); 3.19 (*ddd*, *J* = 8.4, 6.9, 4.0, 1 H, C*H*-C(O)); 2.75 (*m*, 2 H, $CH_2CH_2C_{Ar}$); 2.11 (*m*, 2 H, $CH_2CH_2C_{Ar}$); 1.70 (*m*, 2 H, CH_2CH-C(O)); 1.41 (*m*, 2 H, $CH_3CH_2CH_2$); 0.92 (*t*, *J* = 7.3, 3 H, CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 171.1, 140.0, 128.5, 128.2, 126.2, 77.1, 56.0, 36.2, 31.4, 29.8,

20.3, 13.8. **IR (ATR):** ν = 2931, 1813, 1603, 1114, 838, 748, 698. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 218.1301. Found: 218.1300. **Anal. Calcd. for $C_{14}H_{18}O_2$**: C, 77.03; H, 8.31; O, 14.66. Found: C, 77.22; H, 8.38.

(3*R*,4*R*)-*trans*-3-Propyl-4-(2-phenylethyl)oxetan-2-one (3j)

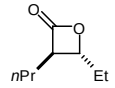


3j

β-Lactone **3j** was prepared from valeroylbromide and 5-hexenal according to the general procedure. Purification by flash chromatography (pentane / diethyl ether 40:1) gave **3j** as colorless oil (131 mg, 0.72 mmol, 96%, *ee* = 95%, *dr* = 98:2). The *dr* value was determined by ^{1}H-NMR and the *ee* value by GC (ThermoFinnigan TraceGC, Chrompac Chirasil-Dex CB column 25 m x 0.25 mm, flow rate 1.1 mL/min, method: 60 °C, ramp @ 5 °C/min to 180 °C, hold 20 min).

$C_{11}H_{18}O_2$, MW: 182.26 g/mol. **$[\alpha]_D^{22.6°C}$** (c = 1.280, $CHCl_3$) = +38.6 ± 0.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 5.78 (*ddt*, *J* = 16.9, 10.2, 6.7, 1 H, CH_2CH=CH_2); 5.07-4.97 (*m*, 2 H, CH=CH_2); 4.23 (*ddd*, *J* = 7.5, 5.8, 4.0, 1 H, C*H*-O); 3.19 (*ddd*, *J* = 8.7, 6.6, 4.0, 1 H, C*H*-C(O)); 2.12 (*m*, 2 H, CH_2CH=CH_2); 1.92-1.69 (*m*, 4 H, CH_2CH_2CH-O/ CH_2CH-C(O)); 1.64-1.37 (*m*, 4 H, $CH_2CH_2CH_2$/ $CH_3CH_2CH_2$); 0.96 (*t*, *J* = 7.3, 3 H, CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 171.3, 137.6, 115.3, 77.9, 56.0, 33.8, 33.2, 30.0, 24.3, 20.4, 13.9. **IR (ATR):** ν = 2933, 1814, 1641, 1120. **HRMS (EI) *m/z*:** Calc. for $[M-H]^+$: 182.1301. Found: 182.1304. **Anal. Calcd. for $C_{11}H_{18}O_2$**: C, 72.49; H, 9.95; O, 17.56. Found: C, 72.71; H, 9.98.

(3*R*,4*R*)-*trans*-3-Propyl-4-ethyloxetan-2-one (3k)

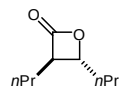


3k

β-Lactone **3k** was prepared from valeroylbromide and propanal according to the general procedure, but using 0.50 mmol of aldehyde in 2 mL of DCM. Purification by flash chromatography (pentane / diethyl ether 40:1) gave **3k** as light-yellow oil (45 mg, 0.32 mmol, 63%, *ee* = 94%, *dr* = 97:3). The *dr* value was determined by ^{1}H-NMR and the *ee* value by GC (ThermoFinnigan TraceGC, Chrompac Chirasil-Dex CB column 25 m x 0.25 mm, flow rate 1.1 mL/min, method: 60 °C, ramp @ 5 °C/min to 180 °C, hold 20 min).

$C_8H_{14}O_2$, **MW:** 142.20 g/mol. Spectral data for the racemate has been reported earlier.[7] **$[\alpha]_D^{24.5°C}$** (c = 1.215, $CHCl_3$) = +25.8 ± 0.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 4.18 (*td*, *J* = 6.6, 4.0, 1 H, C*H*-O); 3.19 (*ddd*, *J* = 8.8, 6.6, 4.0, 1 H, C*H*-C(O)); 1.96-1.64 (*m*, 4 H, CH_3CH_2CH-O/ CH_2CH-C(O)); 1.54-1.36 (*m*, 2 H, $CH_3CH_2CH_2$); 1.02 (*t*, *J* = 7.5, 3 H, CH_3); 0.96 (*t*, *J* = 7.3, 3 H, CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 171.4, 79.1, 55.5, 30.0, 27.5, 20.4, 13.8, 9.2. **IR (ATR):** ν = 2964, 1813, 1125, 849. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 142.0988. Found: 142.0988. **Anal. Calcd. for $C_8H_{14}O_2$**: C, 67.57; H, 9.92; O, 22.50. Found: C, 67.85; H, 9.88.

(3*R*,4*R*)-*trans*-3-Propyl-4-propyloxetan-2-one (3l)



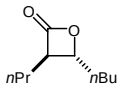
3l

β-Lactone **3l** was prepared from valeroylbromide and butanal according to the general procedure. Purification by flash chromatography (pentane / diethyl ether 40:1) gave **3l** as colorless oil (109 mg, 0.70 mmol, 93%, *ee* = 95%, *dr* = 98:2). The *dr* value was determined by

^{1}H-NMR and the *ee* value by GC (ThermoFinnigan TraceGC, Chrompac Chirasil-Dex CB column 25 m x 0.25 mm, flow rate 1.1 mL/min, method: 60 °C, ramp @ 5 °C/min to 180 °C, hold 20 min).

$C_9H_{16}O_2$, MW: 156.22 g/mol. **$[\alpha]_D^{22.3°C}$** (c = 1.630, $CHCl_3$) = +36.8 ± 0.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 4.22 (*ddd*, *J* = 7.4, 5.9, 4.0, 1 H, C*H*-O); 3.18 (*ddd*, *J* = 8.7, 6.6, 4.0, 1 H, C*H*-C(O)); 1.90-1.63 (*m*, 4 H, CH_2CH_2CH-O/ CH_2CH-C(O)); 1.53-1.34 (*m*, 4 H, 2x $CH_3CH_2CH_2$); 0.96 (*m*, 6 H, 2x CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 171.4, 77.9, 55.9, 36.5, 29.9, 20.3, 18.4, 13.8. **IR (ATR):** ν = 2960, 1813, 1125, 818. **HRMS (EI) *m/z*:** Calc. for $[M-C_2H_5]^+$: 127.0754. Found: 127.0754. **Anal. Calcd. for $C_9H_{16}O_2$**: C, 69.19; H, 10.32; O, 20.48. Found: C, 69.26; H, 10.29.

(3*R*,4*R*)-*trans*-3-Propyl-4-butyloxetan-2-one (3m)



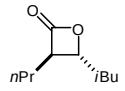
3m

β-Lactone **3m** was prepared from valeroylbromide and pentanal according to the general procedure, but using 0.50 mmol of aldehyde in 2 mL of DCM. Purification by flash chromatography (pentane / diethyl ether 40:1) gave **3m** as colorless oil (78 mg, 0.46 mmol, 92%, *ee* = 93%, *dr* = 96:4). The *dr* value was determined by ^{1}H-NMR and the *ee* value by GC (ThermoFinnigan TraceGC, Chrompac Chirasil-Dex CB column 25 m x 0.25 mm, flow rate 1.1 mL/min, method: 60 °C, ramp @ 5 °C/min to 180 °C, hold 20 min).

$C_{10}H_{18}O_2$, MW: 170.25 g/mol. **$[\alpha]_D^{26.9°C}$** (c = 1.135, $CHCl_3$) = +30.5 ± 0.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 4.22 (*ddd*, *J* = 7.3, 6.1, 4.0, 1 H, C*H*-O); 3.18 (*ddd*, *J* = 8.7, 6.6, 4.0, 1 H, C*H*-C(O)); 1.93-1.61 (*m*, 4 H, CH_2CH_2CH-O/ CH_2CH-C(O)); 1.52-1.29 (*m*, 6 H, $CH_3CH_2CH_2CH$/ $CH_3CH_2CH_2CH_2$); 0.94 (*m*, 6 H, 2x CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21**

°C): δ = 171.4, 78.1, 55.9, 34.1, 30.0, 27.1, 22.4, 20.4, 13.9, 13.8. **IR (ATR):** ν = 2958, 1814, 1125, 837. **HRMS (EI) *m/z*:** Calc. for $[M-H]^+$: 169.1223. Found: 169.1224. **Anal. Calcd. for $C_{10}H_{18}O_2$**: C, 70.55; H, 10.66; O, 18.80. Found: C, 70.71; H, 10.49.

(3*R*,4*R*)-*trans*-3-Propyl-4-*iso*-butyloxetan-2-one (3n)



3n

β-Lactone **3n** was prepared from valeroylbromide and isovaleraldehyde according to the general procedure, but using 0.50 mmol of aldehyde in 2 mL of DCM. Purification by flash chromatography (pentane / diethyl ether 40:1) gave **3n** as colorless oil (65 mg, 0.38 mmol, 76%, *ee* = 94%, *dr* = 96:4). The *dr* value was determined by ^{1}H-NMR and the *ee* value by GC (ThermoFinnigan TraceGC, Chrompac Chirasil-Dex CB column 25 m x 0.25 mm, flow rate 1.1 mL/min, method: 60 °C, ramp @ 5 °C/min to 180 °C, hold 20 min).

$C_{10}H_{18}O_2$, MW: 170.25 g/mol. **[α]** $_D^{24.9°C}$ (c = 1.600, $CHCl_3$) = +47.4 ± 0.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 4.30 (*ddd*, *J* = 8.0, 5.2, 4.0, 1 H, C*H*-O); 3.17 (*ddd*, *J* = 8.4, 6.9, 4.0, 1 H, C*H*-C(O)); 1.88-1.36 (*m*, 6 H, CHCH_2CH-O/ $CH_3C H_2C H_2$CH-C(O)); 0.93 (*m*, 6 H, 2x CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 171.4, 76.9, 56.4, 43.5, 29.9, 25.4, 22.8, 22.4, 20.3, 13.8. **IR (ATR):** ν = 2958, 1815, 1116. **HRMS (EI) *m/z*:** Calc. for $[M-C_4H_9]^+$: 113.0597. Found: 113.0596. **Anal. Calcd. for $C_{10}H_{18}O_2$**: C, 70.55; H, 10.66; O, 18.80. Found: C, 70.71; H, 10.59.

References

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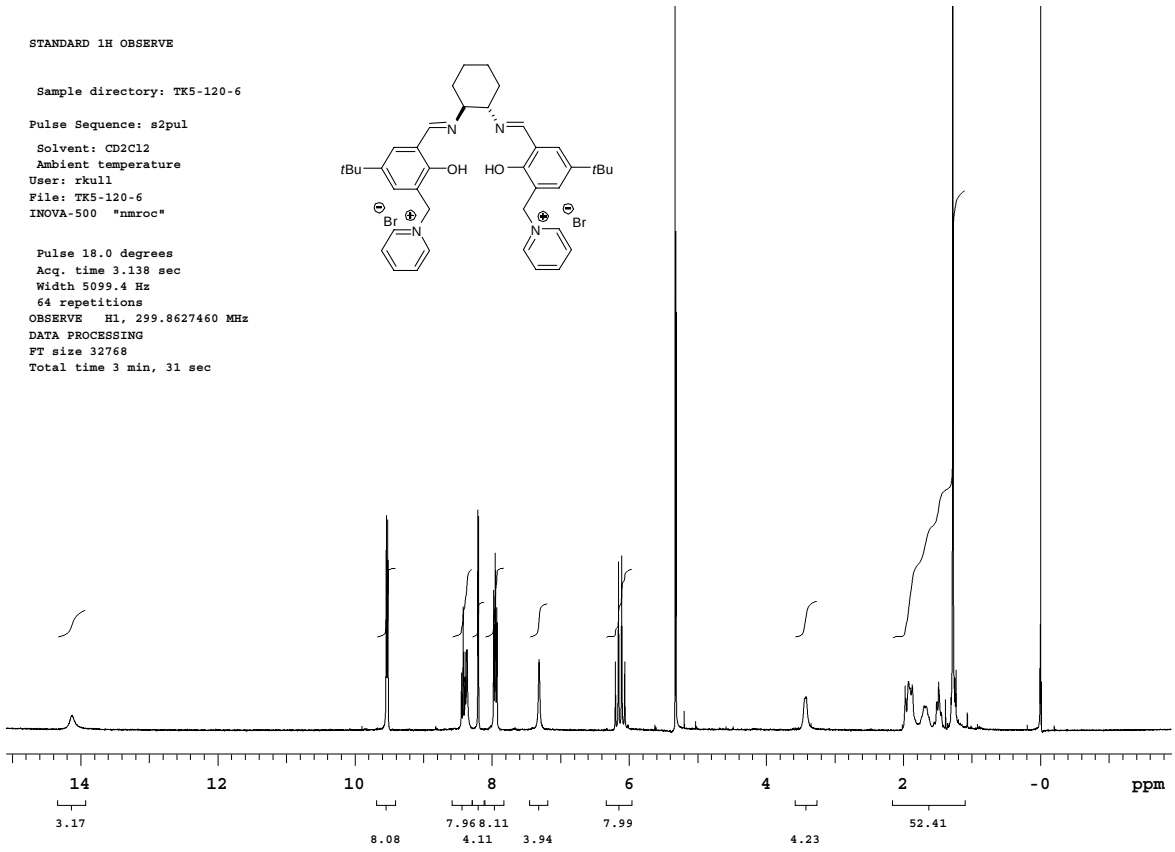
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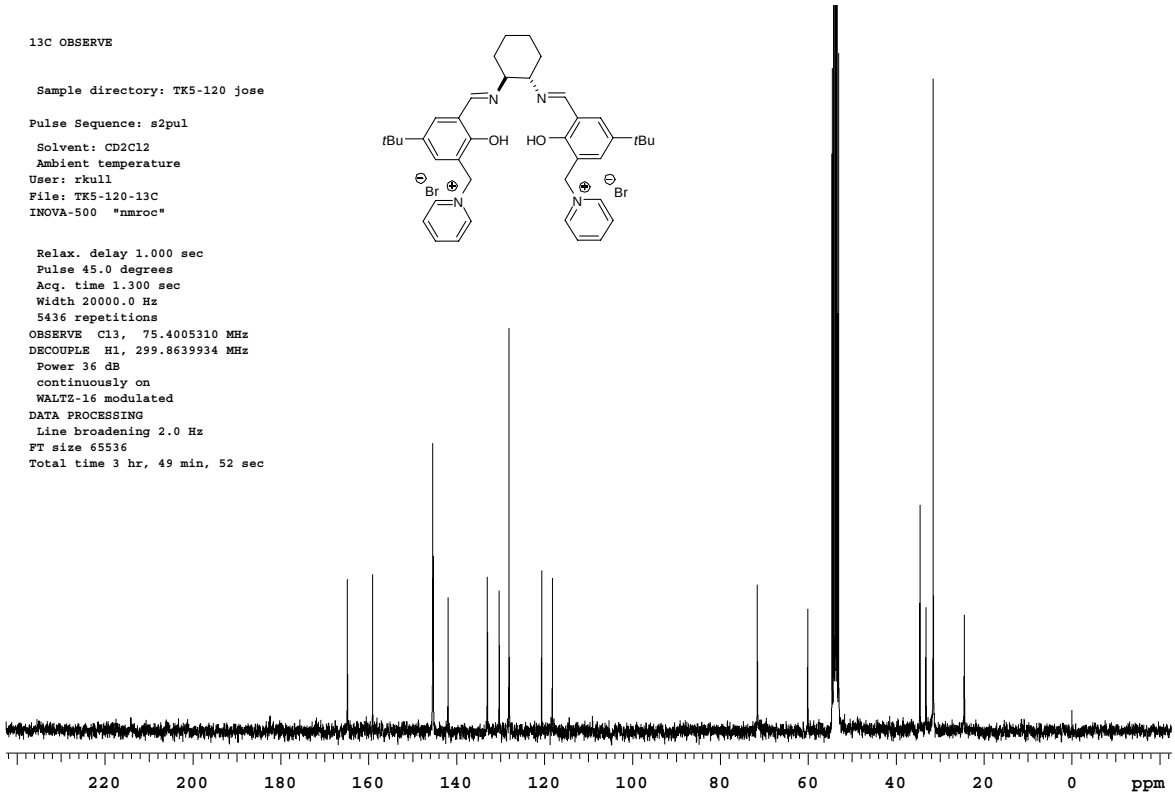
Acq. time 3.138 sec
Width 5099.4 Hz
64 repetitions
OBSERVE H1, 299.8627460 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec
tBu
OH
HO
tBu
Br
Br
14
12
10
8
6
4
2
-0
ppm
3.17
8.08
7.96
8.11
4.11
3.94
7.99
4.23
52.41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Solvent: CD2Cl2
Ambient temperature
User: rkull
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INOVA-500 "nmroc"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
5436 repetitions
OBSERVE C13, 75.4005310 MHz
DECOUPLE H1, 299.8639934 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 3 hr, 49 min, 52 sec
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HO
tBu
Br
Br
220
200
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120
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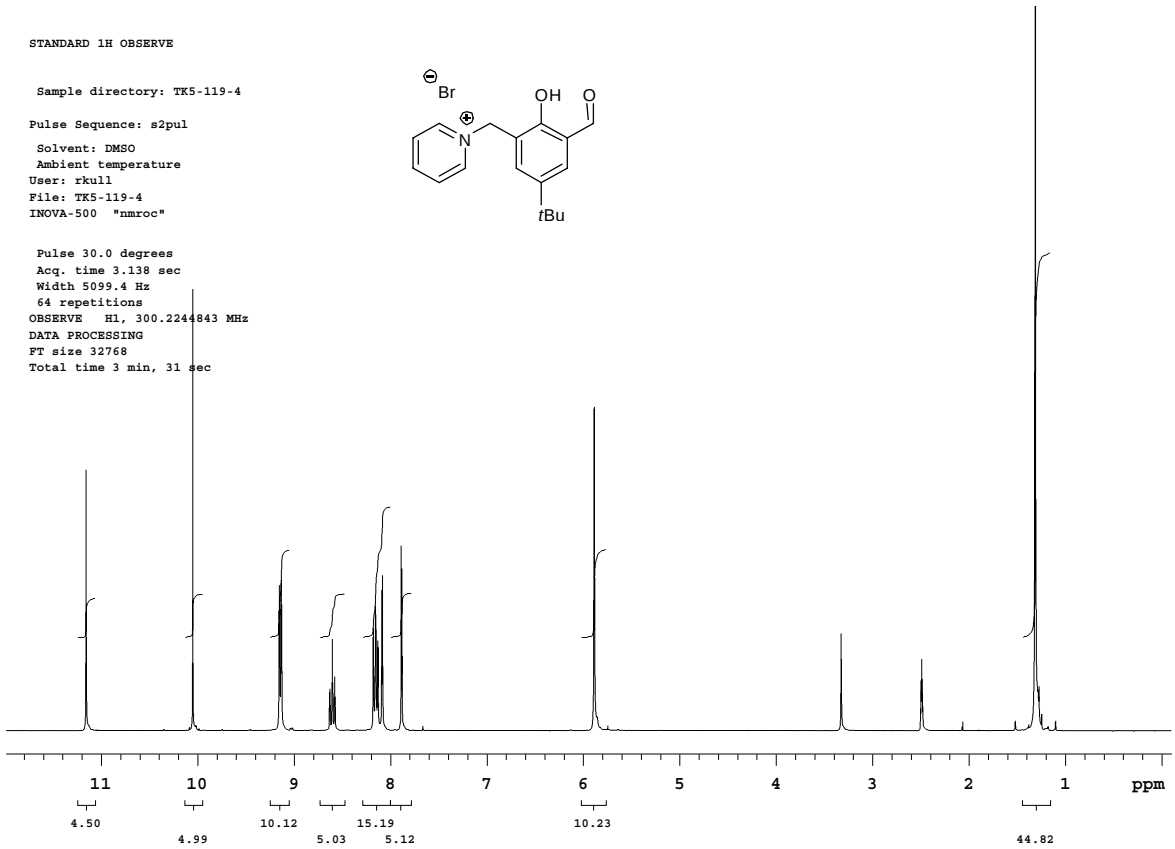
0
ppm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Ambient temperature
User: rkull
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INOVA-500 "nmroc"
Pulse 30.0 degrees
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Width 5099.4 Hz
64 repetitions
OBSERVE H1, 300.2244843 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec
Br
OH
O
N
tBu
11
10
9
8
7
6
5
4
3
2
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ppm
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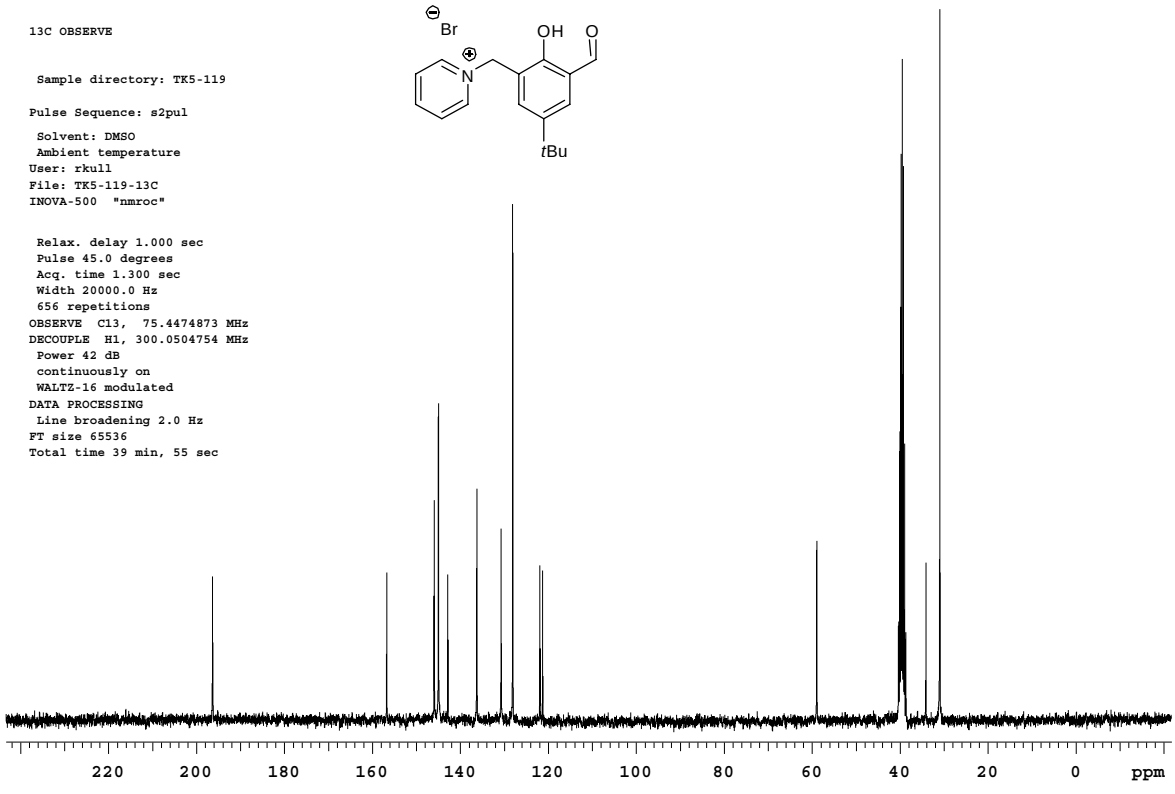
5.12
10.23
44.82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Ambient temperature
User: rkull
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DECOUPLE H1, 300.0504754 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
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Total time 39 min, 55 sec
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tBu
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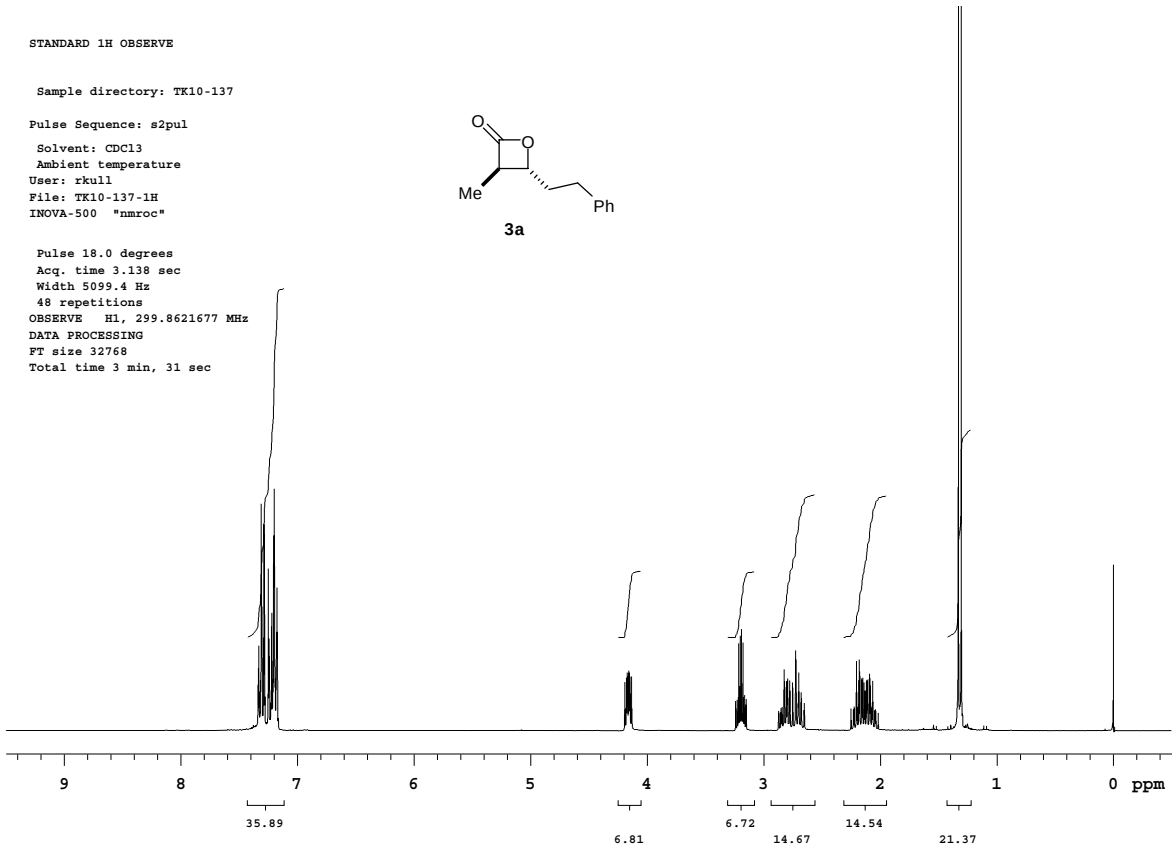
ppm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Ambient temperature
User: rkull
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INOVA-500 "nmroc"
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48 repetitions
OBSERVE H1, 299.8621677 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec
O
O
Me
Ph
3a
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8
7
6
5
4
3
2
1
0
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14.54
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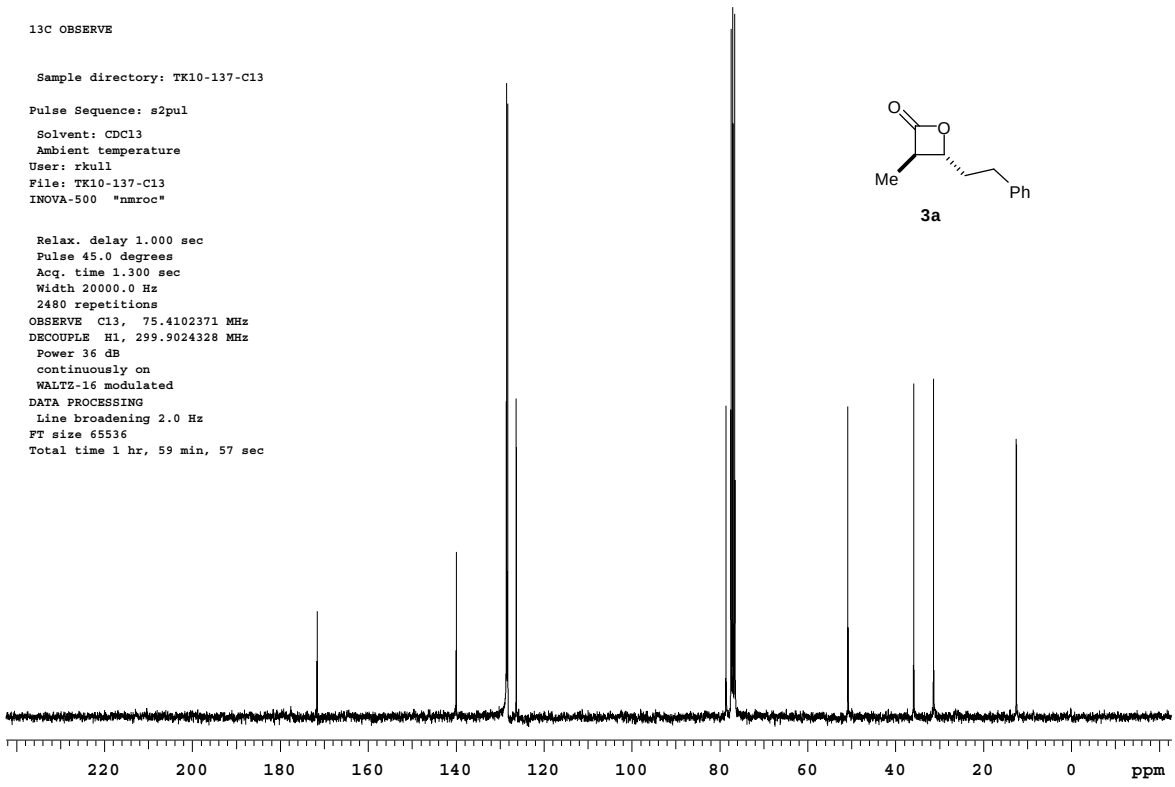

13C OBSERVE
Sample directory: TK10-137-C13
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-137-C13
INOVA-500 "nmroc"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
2480 repetitions
OBSERVE C13, 75.4102371 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 59 min, 57 sec
O
O
Me
Ph
3a
220
200
180
160
140
120
100
80
60
40
20
0
ppm

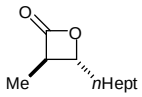
STANDARD 1H OBSERVE

Sample directory: TK10-258chrom

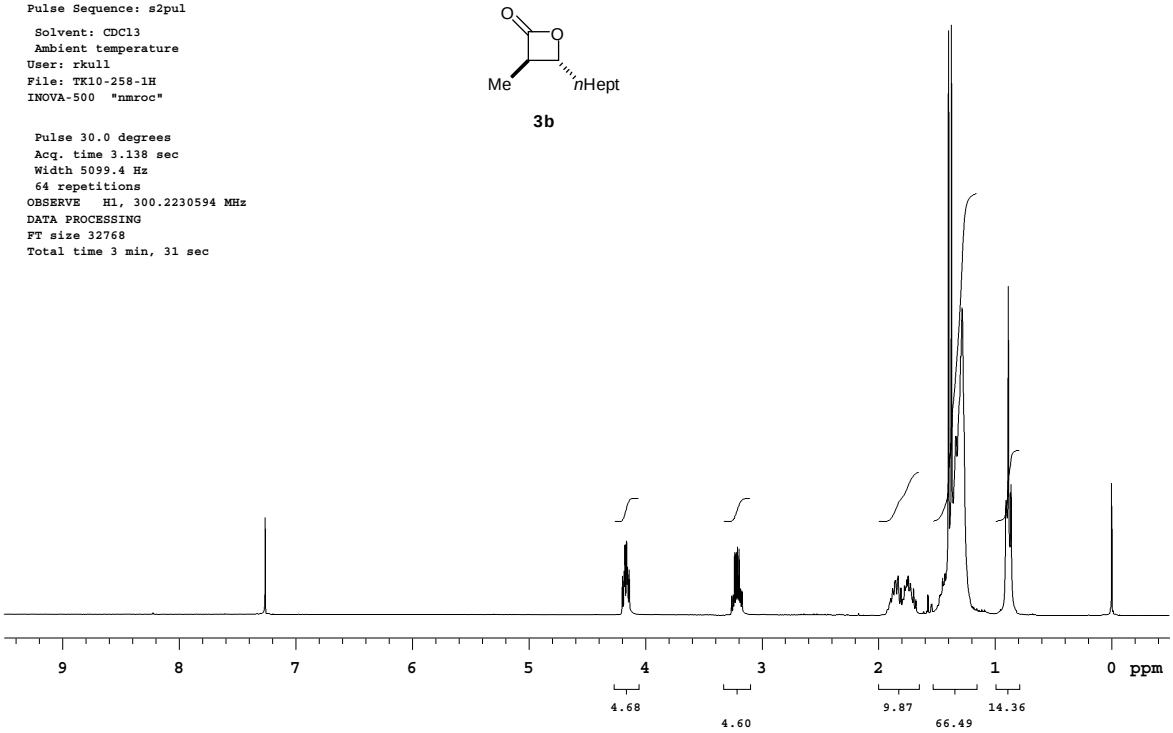
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-258-1H
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
64 repetitions
OBSERVE H1, 300.2230594 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec



3b



13C OBSERVE

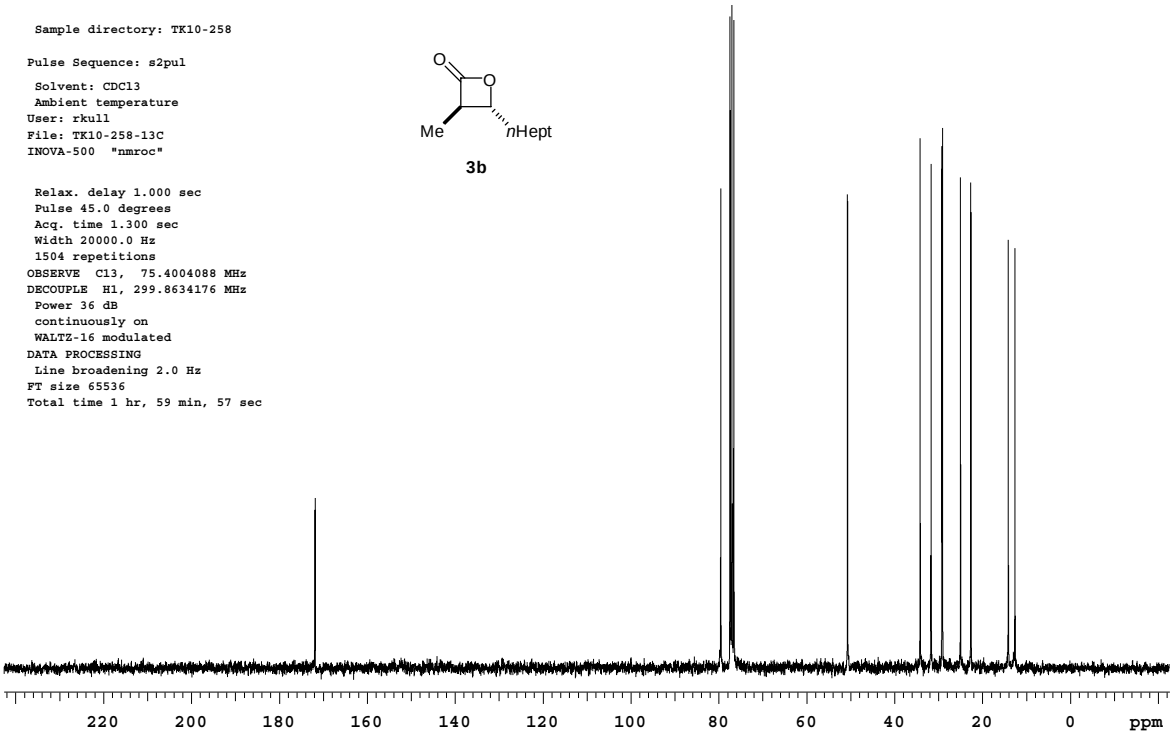
Sample directory: TK10-258

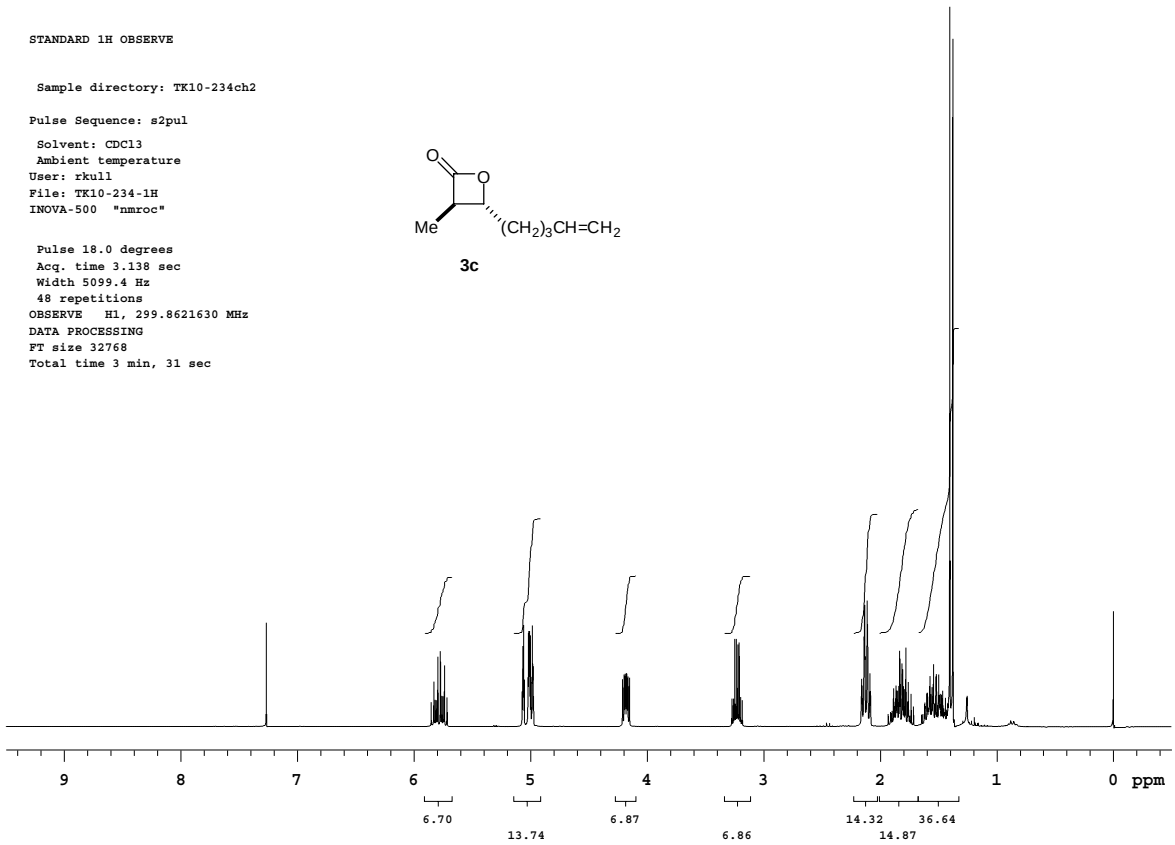
Pulse Sequence: s2pul

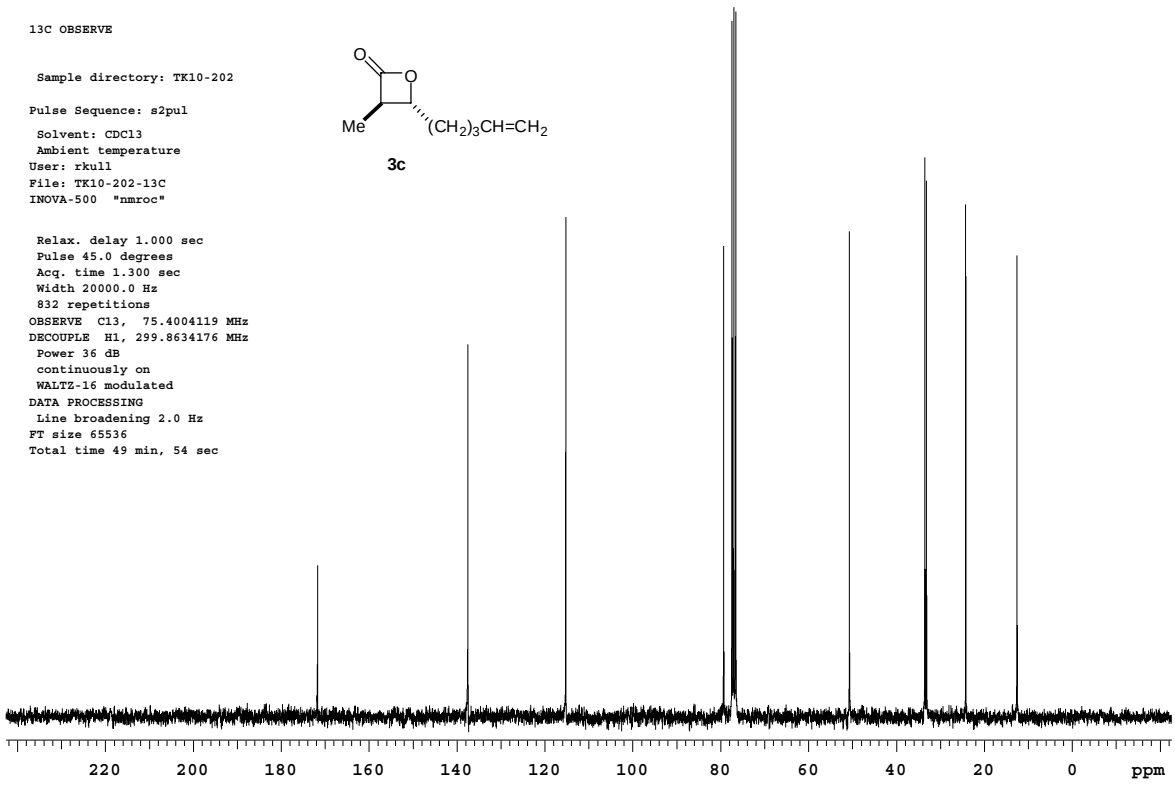
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-258-13C
INOVA-500 "nmroc"

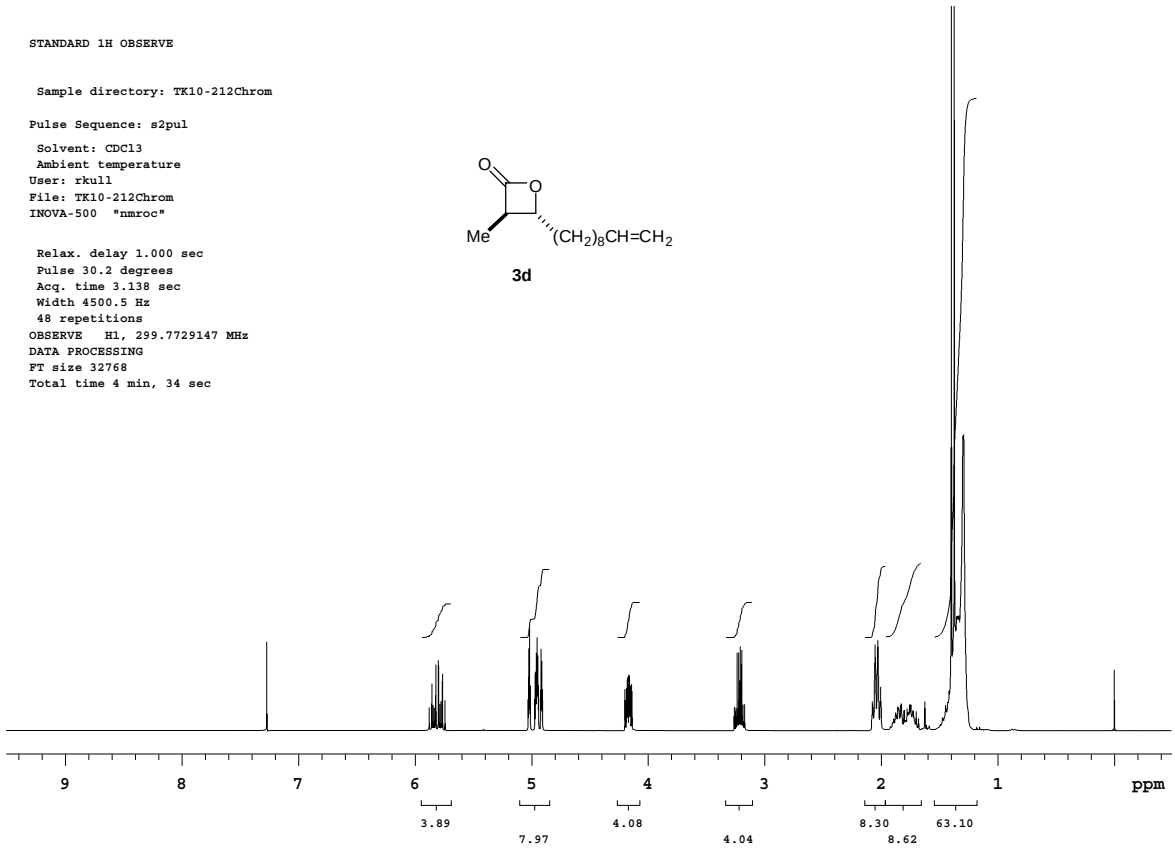
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1504 repetitions
OBSERVE C13, 75.4004088 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 59 min, 57 sec

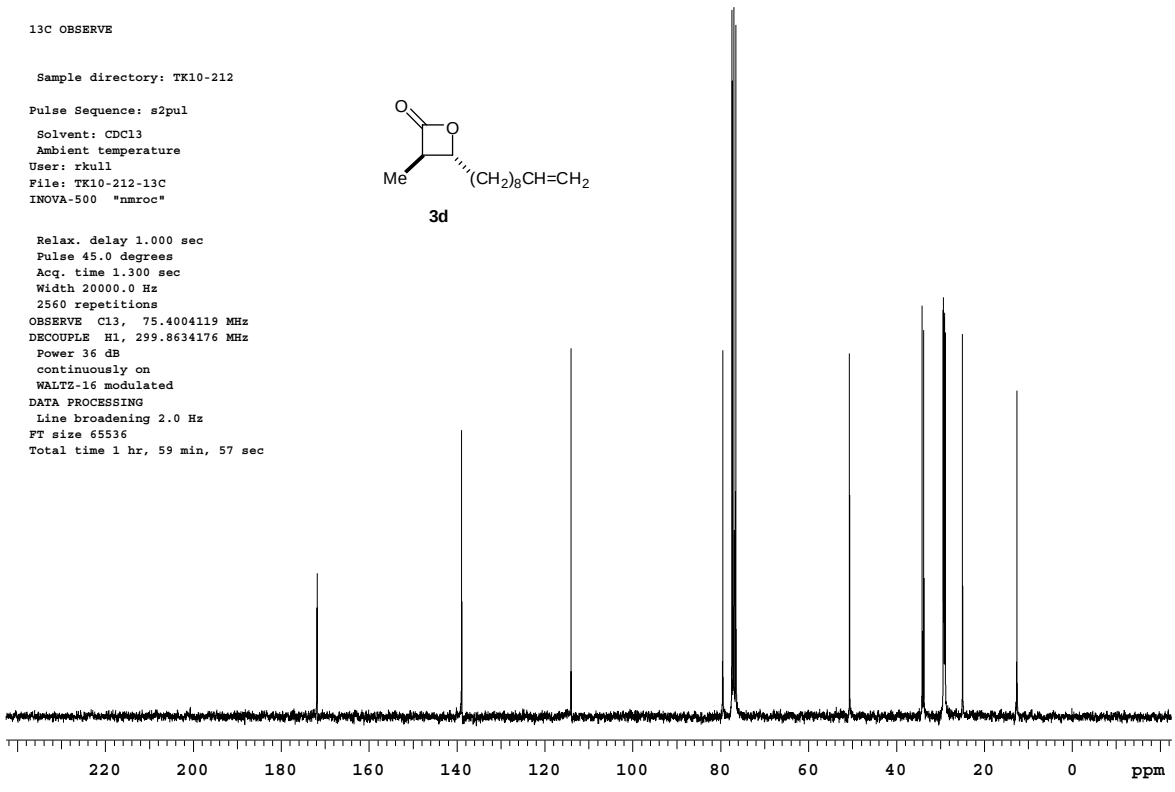
3b

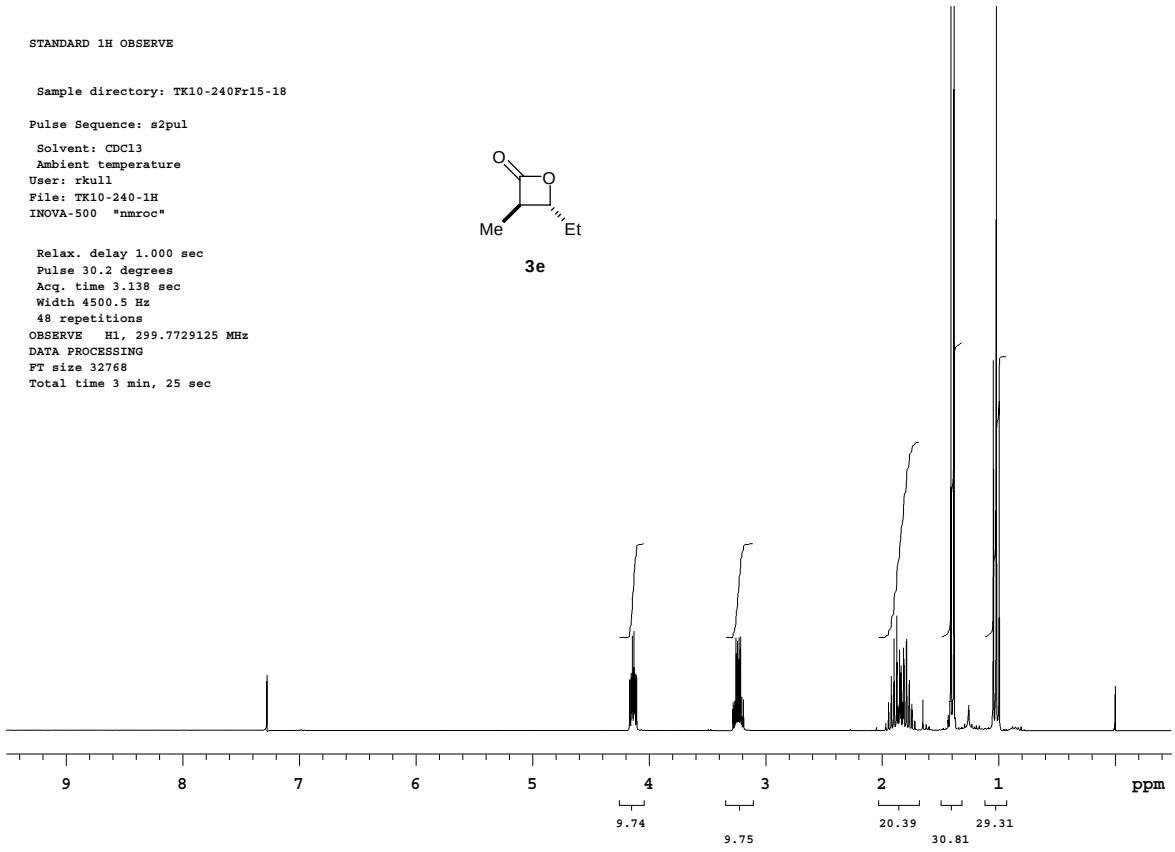


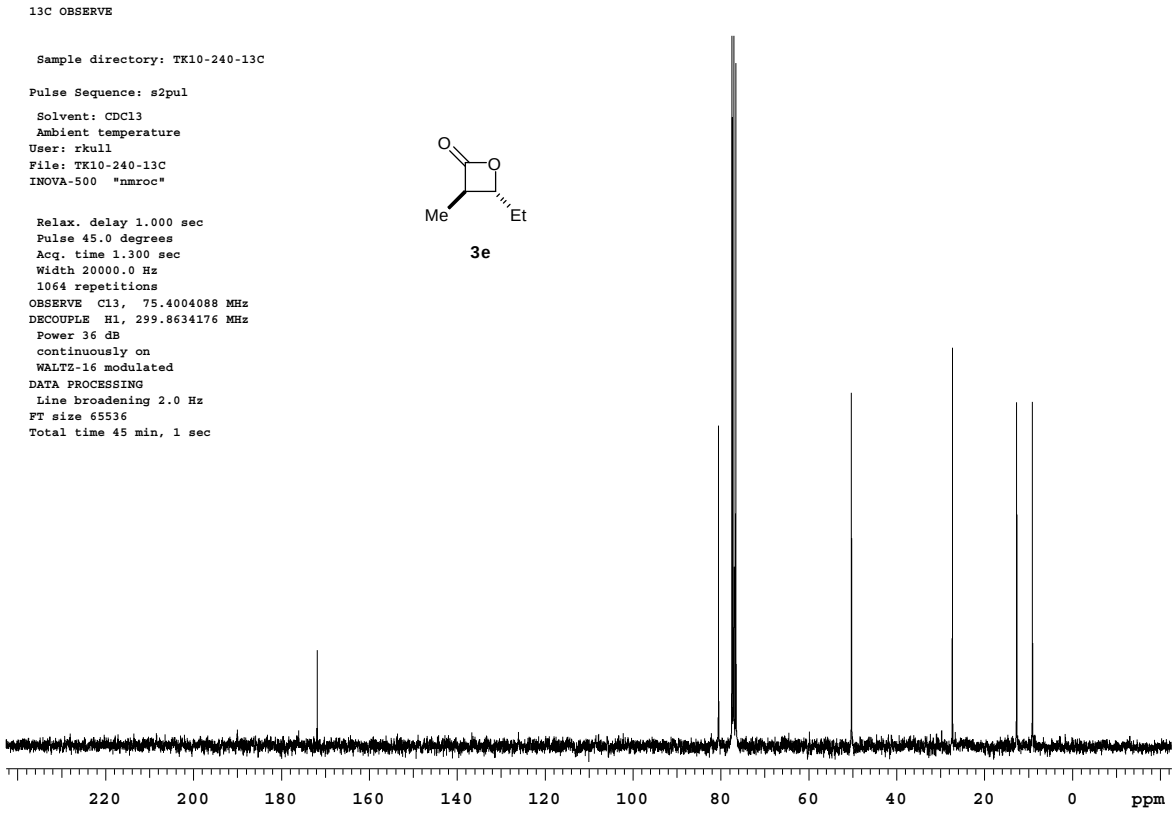

STANDARD 1H OBSERVE
Sample directory: TK10-234ch2
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-234-1H
INOVA-500 "nmroc"
Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
48 repetitions
OBSERVE H1, 299.8621630 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec
O
O
Me
(CH2)3CH=CH2
3c
9
8
7
6
5
4
3
2
1
0 ppm
6.70
13.74
6.87
6.86
14.32
36.64
14.87


13C OBSERVE
Sample directory: TK10-202
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-202-13C
INOVA-500 "nmroc"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
832 repetitions
OBSERVE C13, 75.4004119 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 49 min, 54 sec
O
O
Me
(CH2)3CH=CH2
3c
220
200
180
160
140
120
100
80
60
40
20
0
ppm


STANDARD 1H OBSERVE
Sample directory: TK10-212Chrom
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-212Chrom
INOVA-500 "nmroc"
Relax. delay 1.000 sec
Pulse 30.2 degrees
Acq. time 3.138 sec
Width 4500.5 Hz
48 repetitions
OBSERVE H1, 299.7729147 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 34 sec
O
O
Me
(CH2)8CH=CH2
3d
9
8
7
6
5
4
3
2
1
ppm
3.89
7.97
4.08
4.04
8.30
8.62
63.10


13C OBSERVE
Sample directory: TK10-212
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-212-13C
INOVA-500 "nmroc"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
2560 repetitions
OBSERVE C13, 75.4004119 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 59 min, 57 sec
O
O
Me
(CH2)8CH=CH2
3d
220
200
180
160
140
120
100
80
60
40
20
0
ppm


STANDARD 1H OBSERVE
Sample directory: TK10-240Fr15-18
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-240-1H
INOVA-500 "nmroc"
Relax. delay 1.000 sec
Pulse 30.2 degrees
Acq. time 3.138 sec
Width 4500.5 Hz
48 repetitions
OBSERVE H1, 299.7729125 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 25 sec
O
O
Me
Et
3e
9
8
7
6
5
4
3
2
1
ppm
9.74
9.75
20.39
30.81
29.31


13C OBSERVE
Sample directory: TK10-240-13C
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-240-13C
INOVA-500 "nmroc"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1064 repetitions
OBSERVE C13, 75.4004088 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 45 min, 1 sec
O
O
Me
Et
3e
220
200
180
160
140
120
100
80
60
40
20
0
ppm

STANDARD 1H OBSERVE

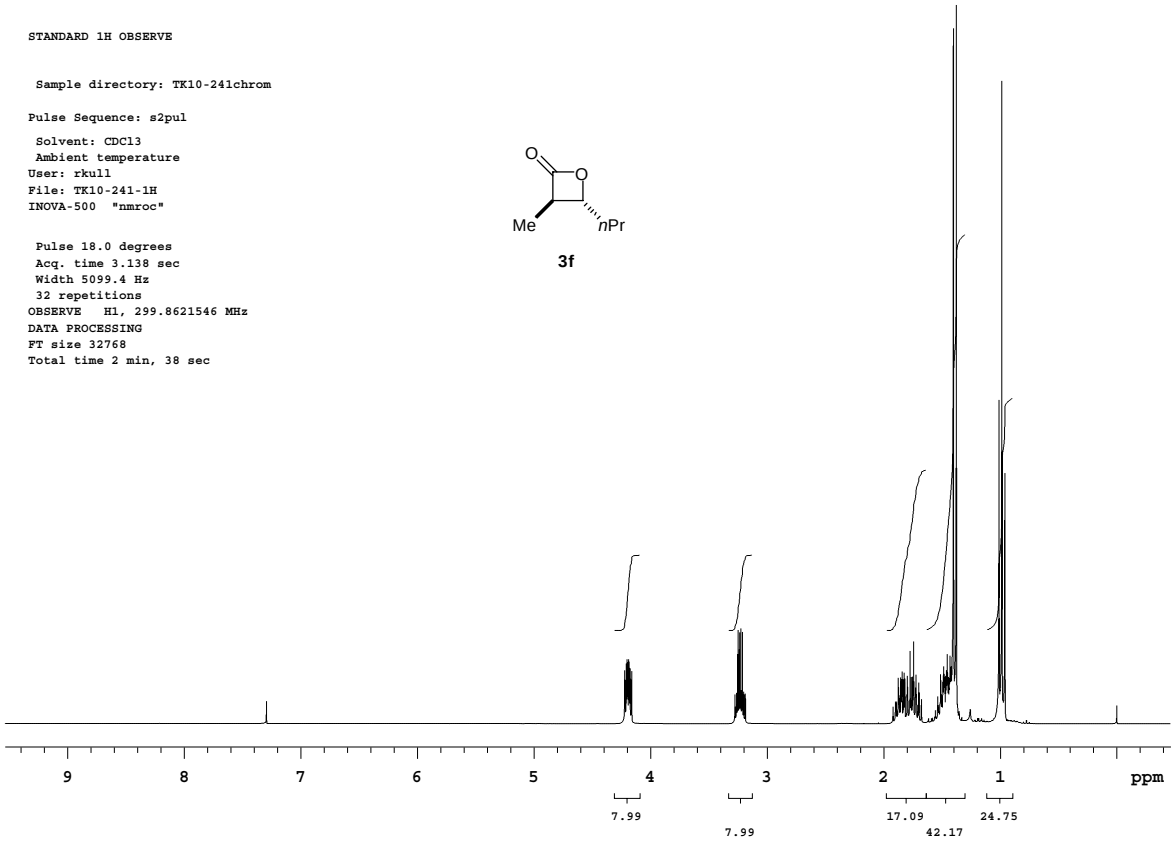
Sample directory: TK10-241chrom

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-241-1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
32 repetitions
OBSERVE H1, 299.8621546 MHz
DATA PROCESSING
FT size 32768
Total time 2 min, 38 sec

3f



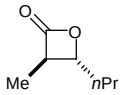
13C OBSERVE

Sample directory: TK10-241Chrom

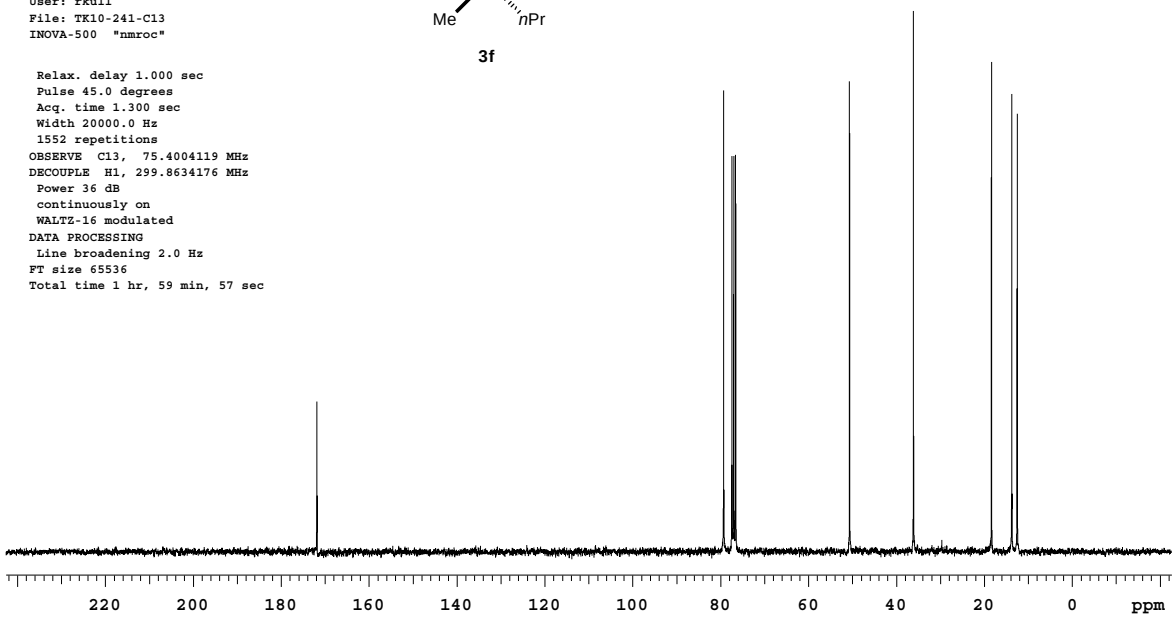
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-241-C13
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1552 repetitions
OBSERVE C13, 75.4004119 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 59 min, 57 sec



3f



STANDARD 1H OBSERVE

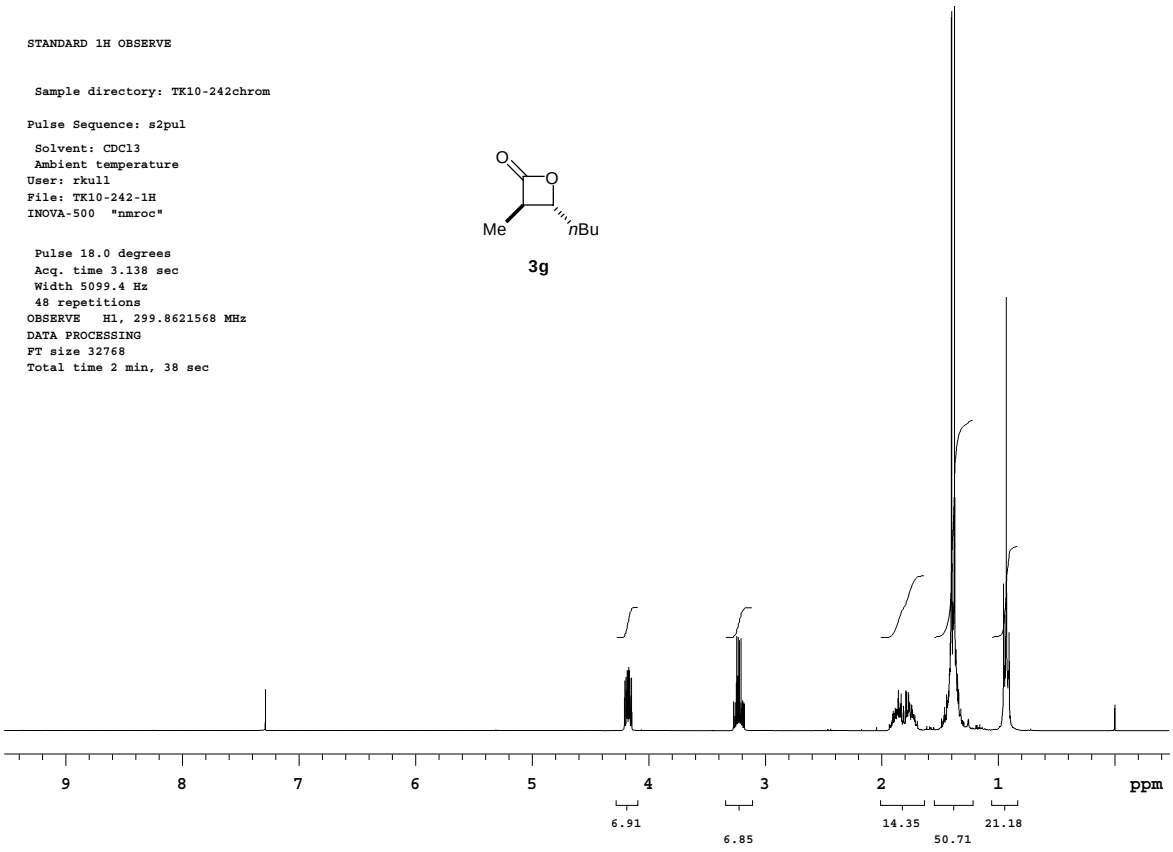
Sample directory: TK10-242chrom

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-242-1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
48 repetitions
OBSERVE H1, 299.8621568 MHz
DATA PROCESSING
FT size 32768
Total time 2 min, 38 sec

3g



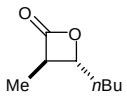
13C OBSERVE

Sample directory: TK10-242Chrom

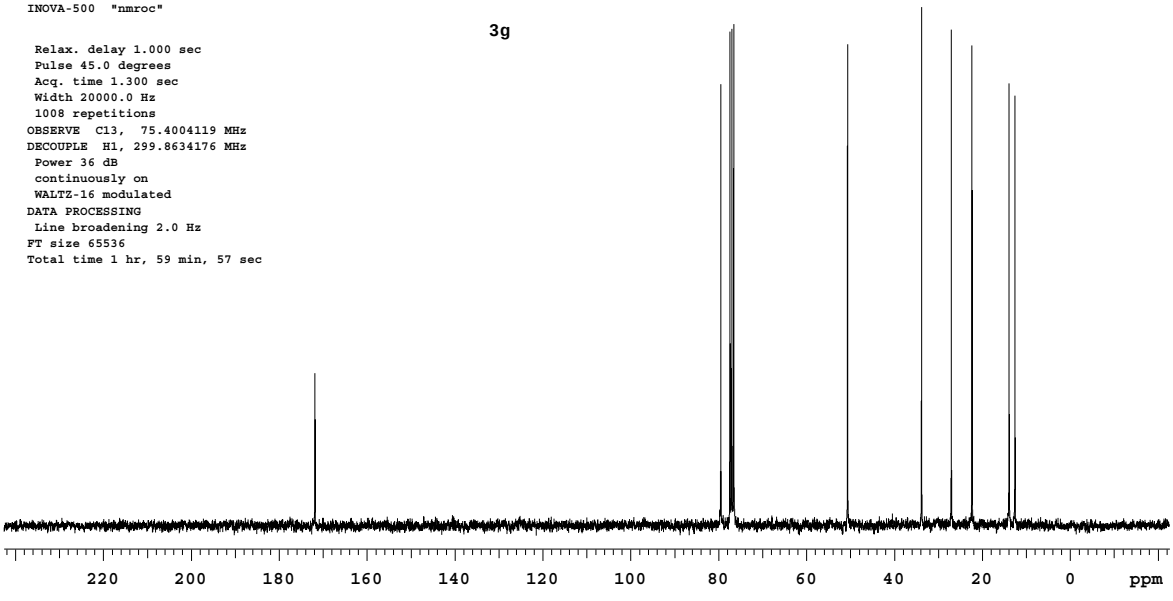
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-242-C13
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1008 repetitions
OBSERVE C13, 75.4004119 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 59 min, 57 sec



3g



STANDARD 1H OBSERVE

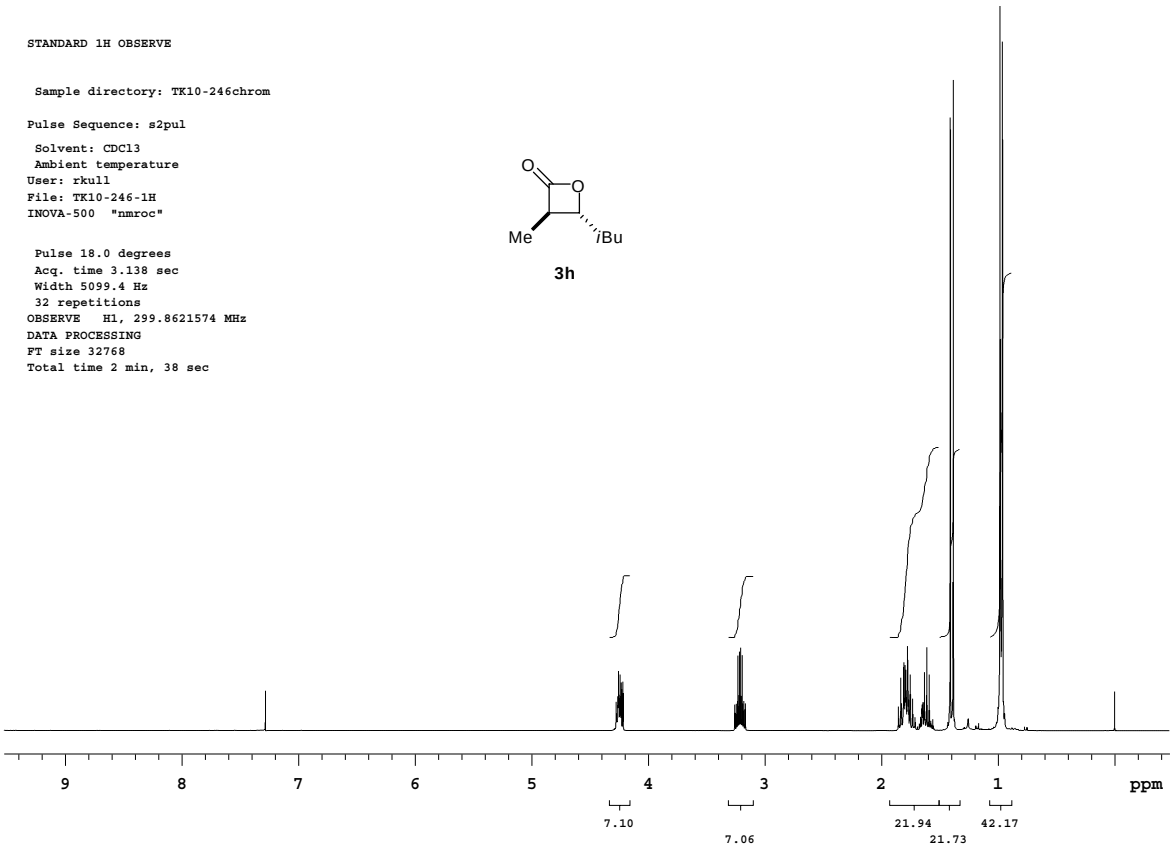
Sample directory: TK10-246chrom

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-246-1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
32 repetitions
OBSERVE H1, 299.8621574 MHz
DATA PROCESSING
FT size 32768
Total time 2 min, 38 sec

3h



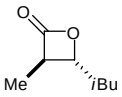
13C OBSERVE

Sample directory: TK10-246chrom

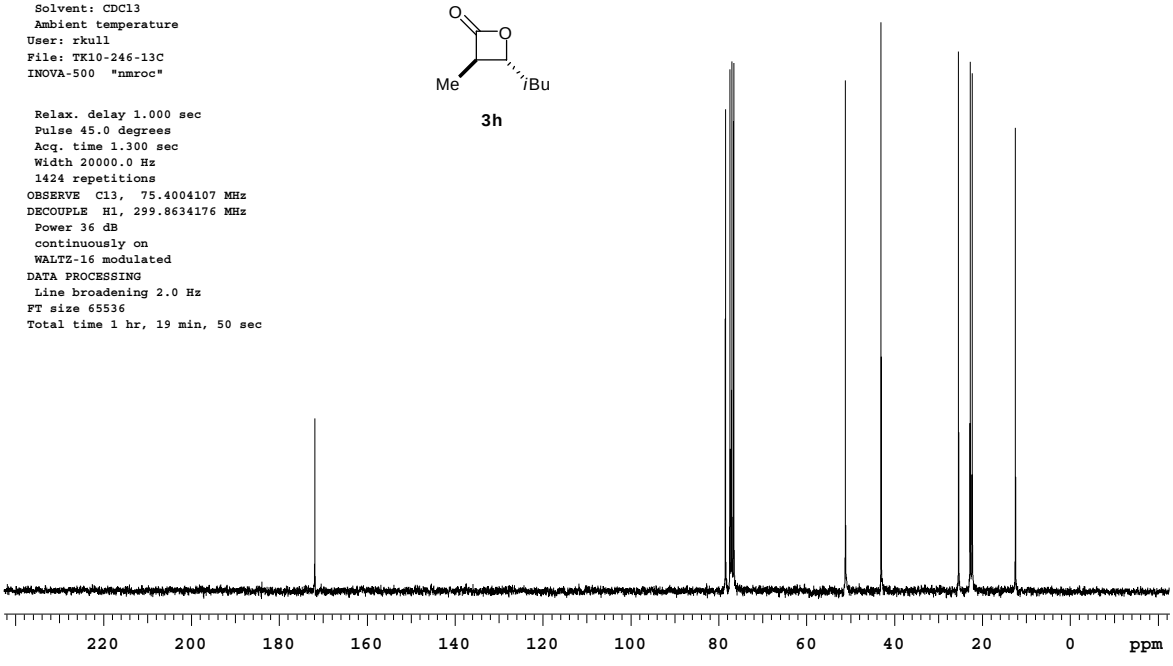
Pulse Sequence: s2pul

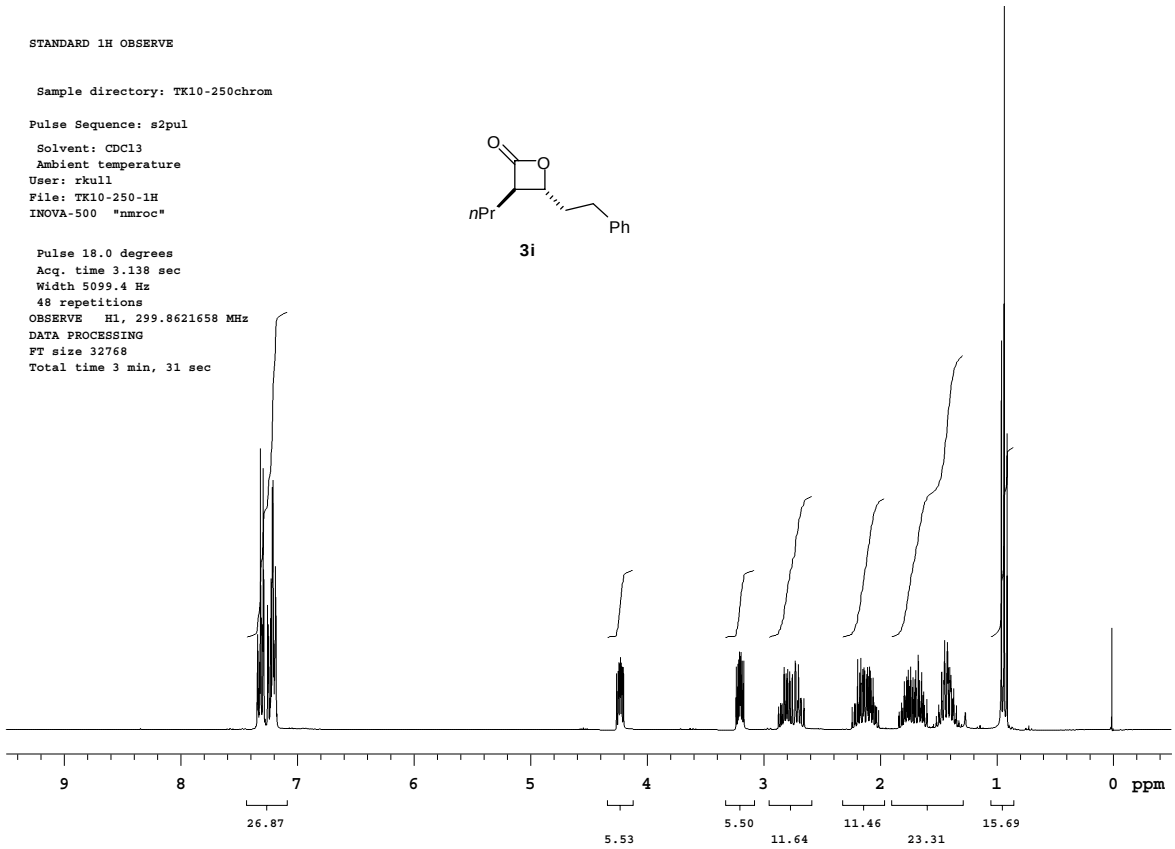
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-246-13C
INOVA-500 "nmroc"

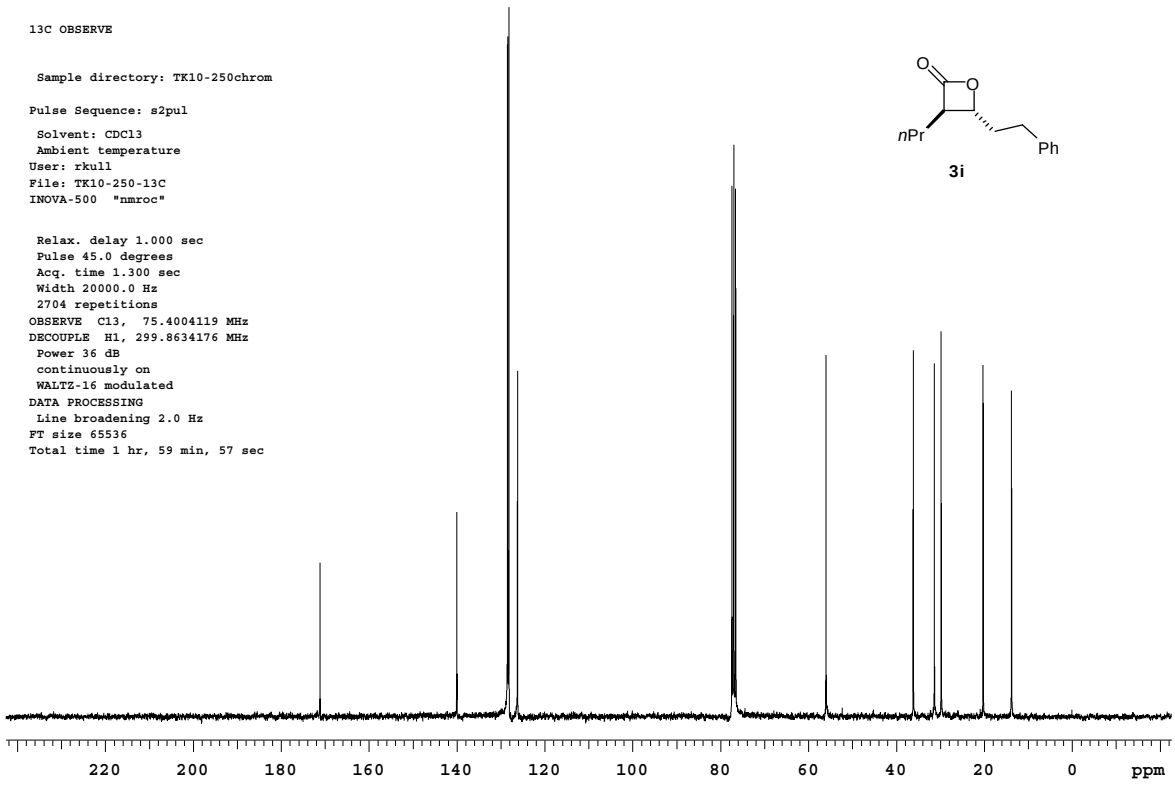
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1424 repetitions
OBSERVE C13, 75.4004107 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 19 min, 50 sec

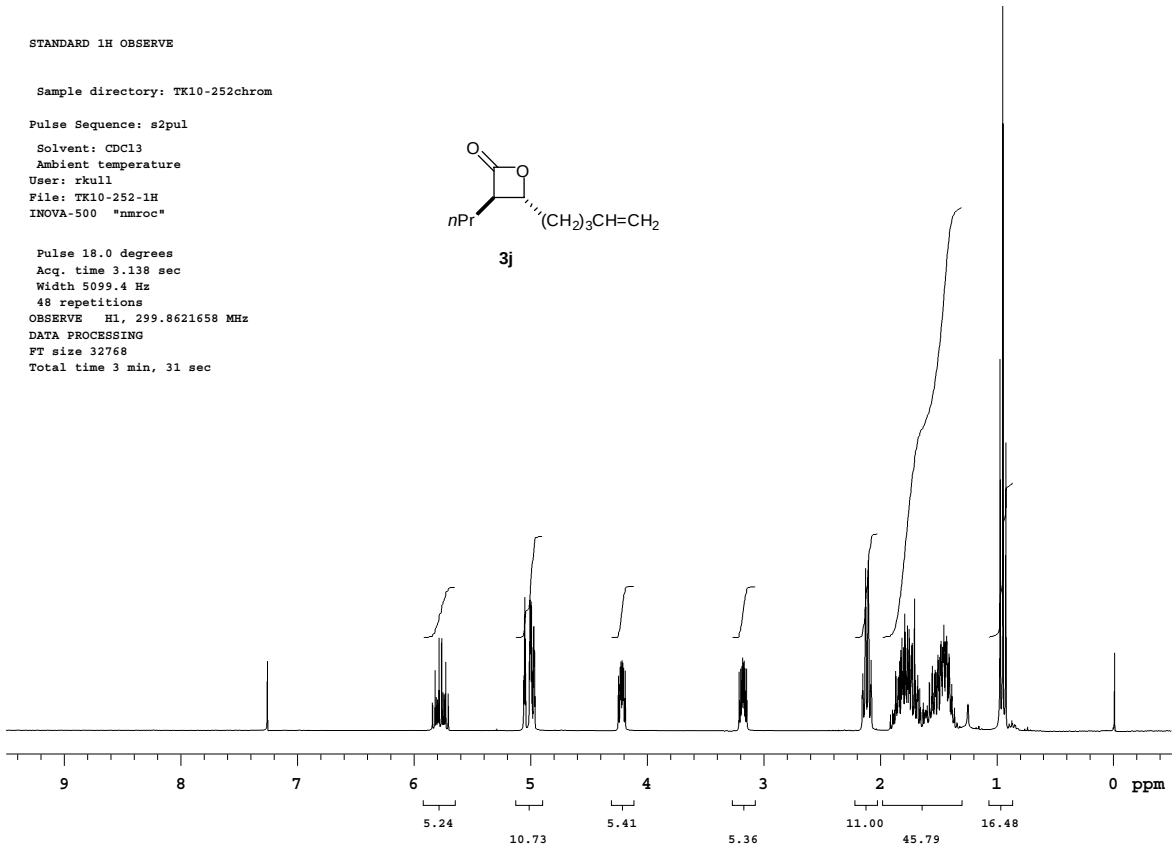


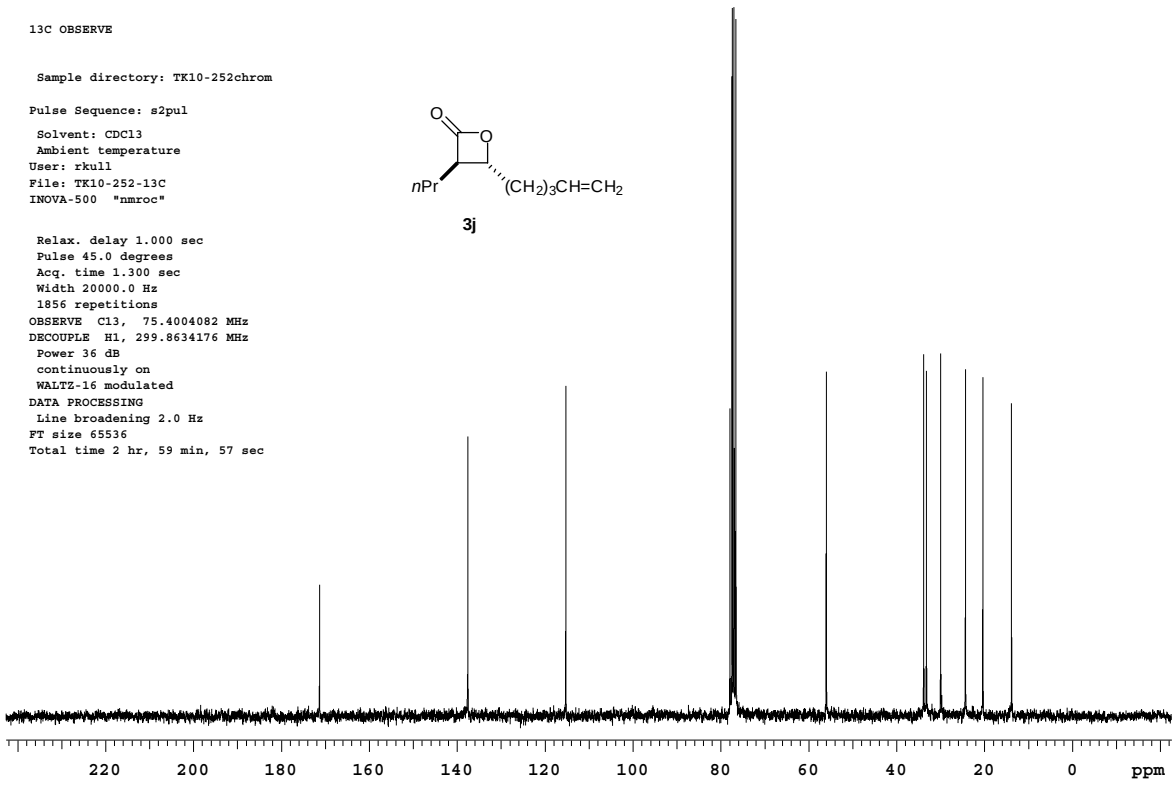
3h




STANDARD 1H OBSERVE
Sample directory: TK10-250chrom
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-250-1H
INOVA-500 "nmroc"
Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
48 repetitions
OBSERVE H1, 299.8621658 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec
O
O
nPr
Ph
3i
9
8
7
6
5
4
3
2
1
0 ppm
26.87
5.53
5.50
11.64
11.46
23.31
15.69


13C OBSERVE
Sample directory: TK10-250chrom
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-250-13C
INOVA-500 "nmroc"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
2704 repetitions
OBSERVE C13, 75.4004119 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 59 min, 57 sec
O
O
nPr
Ph
3i
220
200
180
160
140
120
100
80
60
40
20
0 ppm


STANDARD 1H OBSERVE
Sample directory: TK10-252chrom
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-252-1H
INOVA-500 "nmroc"
Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
48 repetitions
OBSERVE H1, 299.8621658 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec
nPr
(CH2)3CH=CH2
3j
9
8
7
6
5
4
3
2
1
0
ppm
5.24
10.73
5.41
5.36
11.00
45.79
16.48


13C OBSERVE
Sample directory: TK10-252chrom
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-252-13C
INOVA-500 "nmroc"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1856 repetitions
OBSERVE C13, 75.4004082 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 2 hr, 59 min, 57 sec
nPr
(CH2)3CH=CH2
3j
220
200
180
160
140
120
100
80
60
40
20
0
ppm

STANDARD 1H OBSERVE

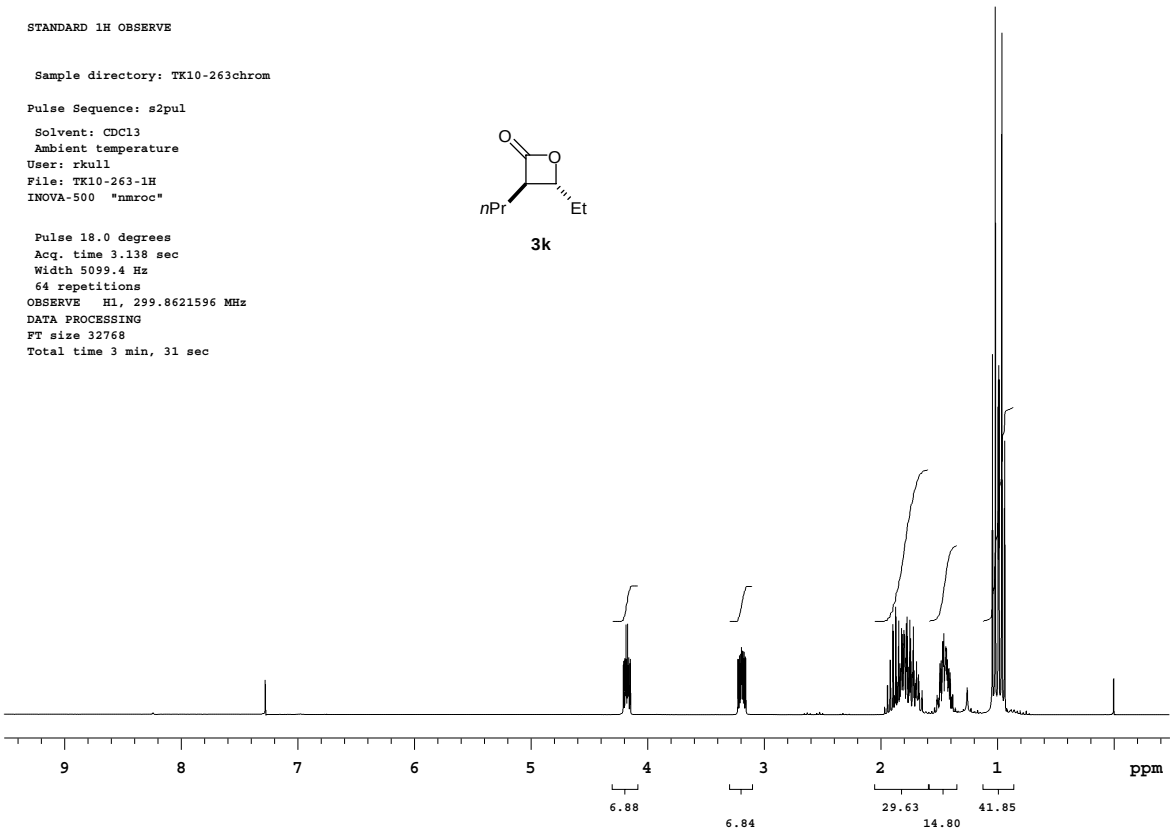
Sample directory: TK10-263chrom

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-263-1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
64 repetitions
OBSERVE H1, 299.8621596 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec

3k



13C OBSERVE

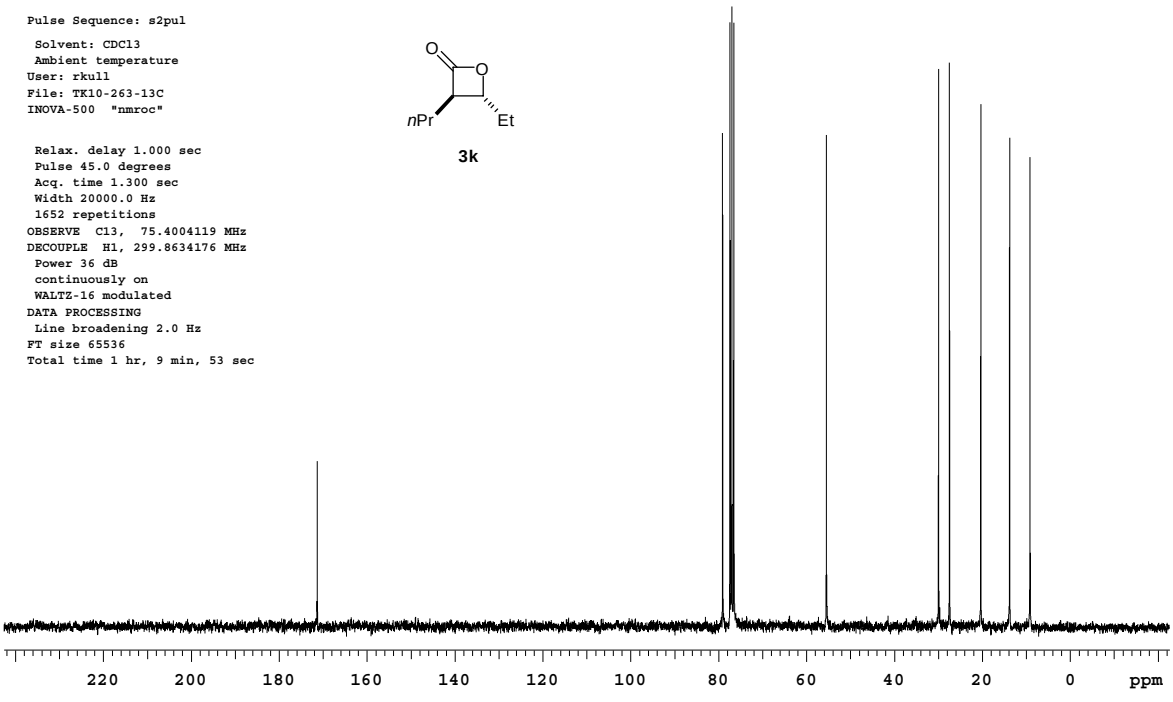
Sample directory: TK10-263

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-263-13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1652 repetitions
OBSERVE C13, 75.4004119 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 9 min, 53 sec

3k



STANDARD 1H OBSERVE

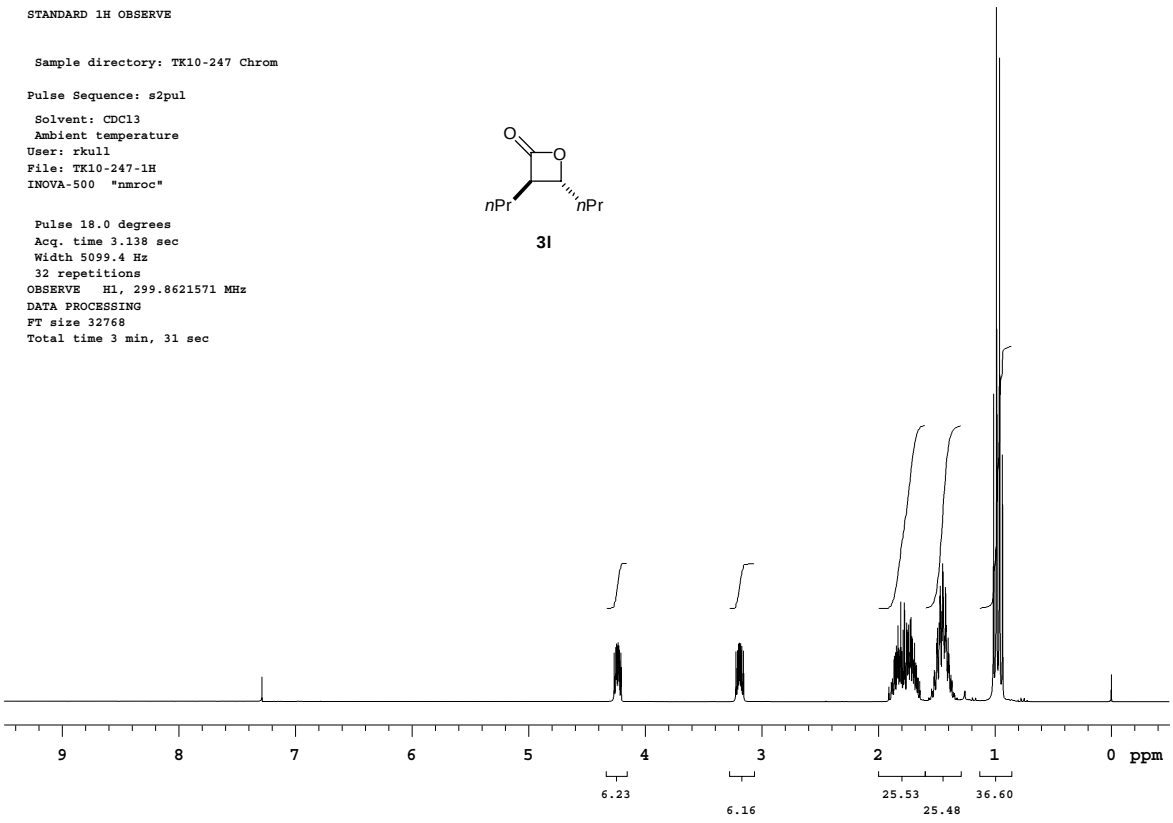
Sample directory: TK10-247 Chrom

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-247-1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
32 repetitions
OBSERVE H1, 299.8621571 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec

3l



13C OBSERVE

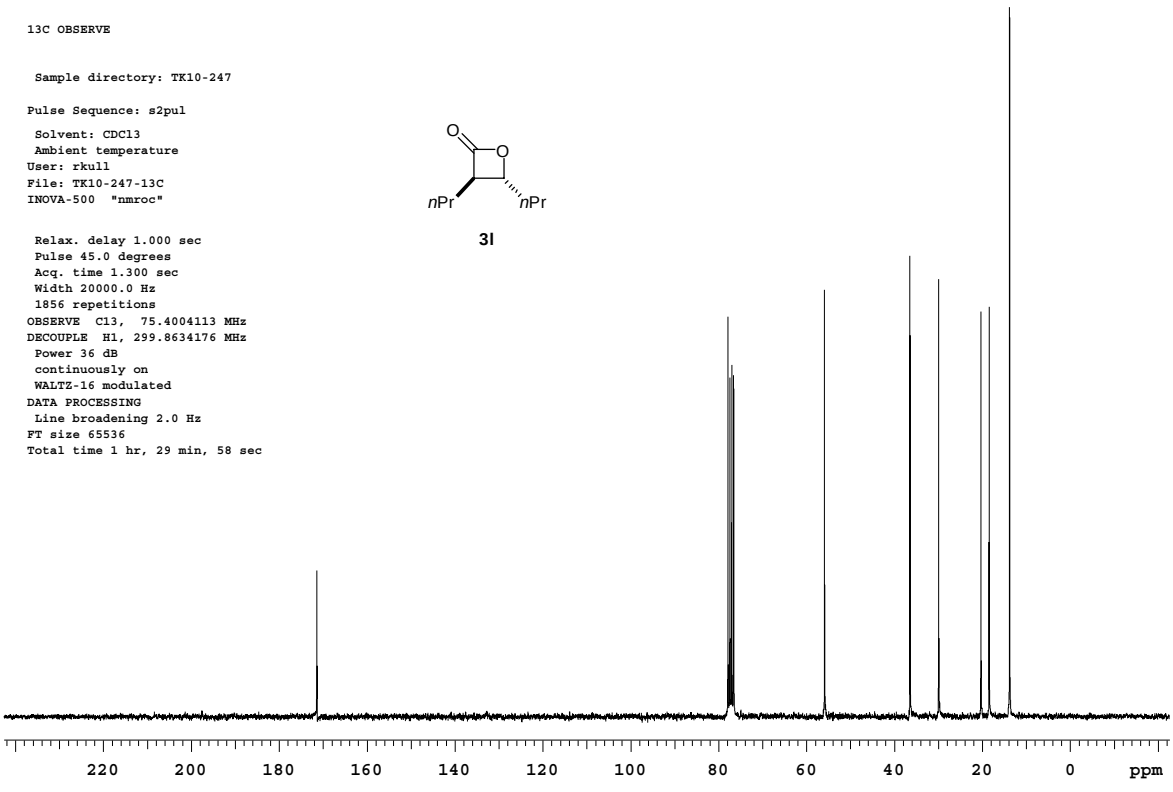
Sample directory: TK10-247

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-247-13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1856 repetitions
OBSERVE C13, 75.4004113 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 29 min, 58 sec

3l



STANDARD 1H OBSERVE

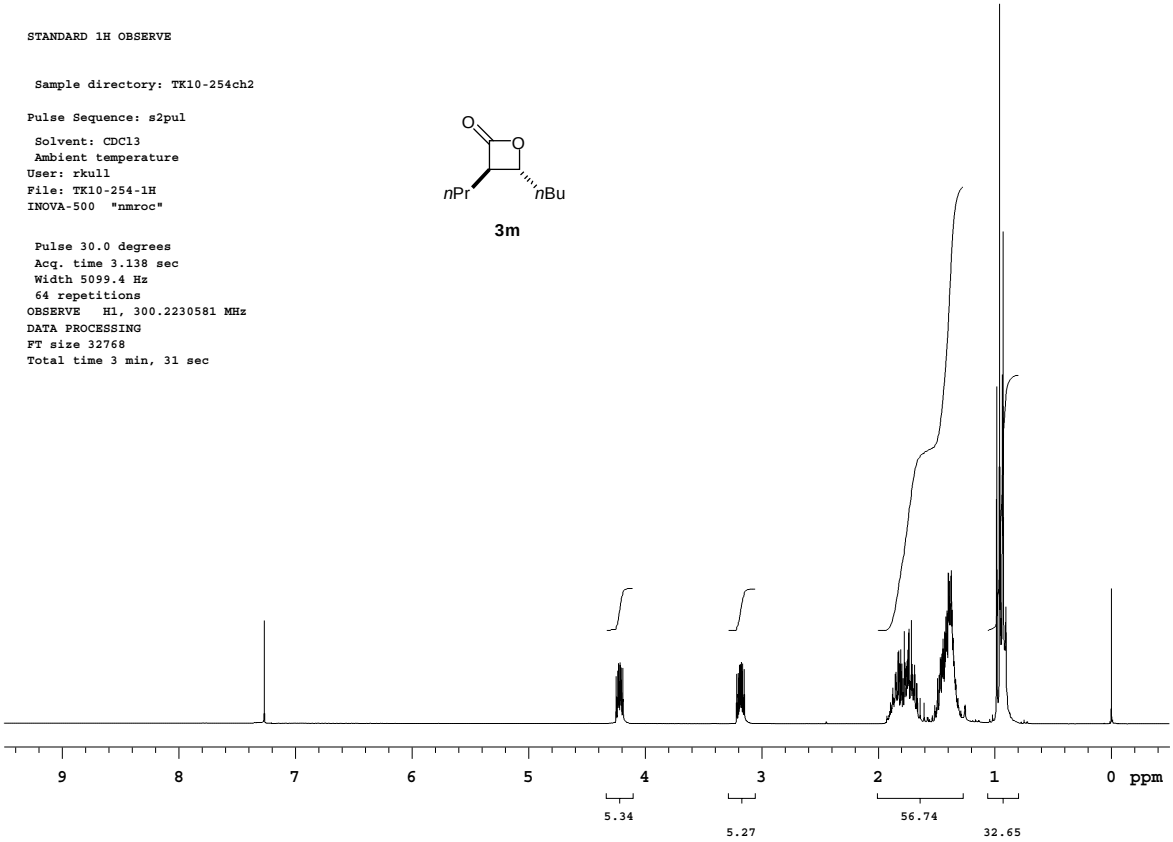
Sample directory: TK10-254ch2

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-254-1H
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
64 repetitions
OBSERVE H1, 300.2230581 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec

3m



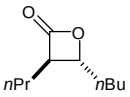
13C OBSERVE

Sample directory: TK10-254chrom

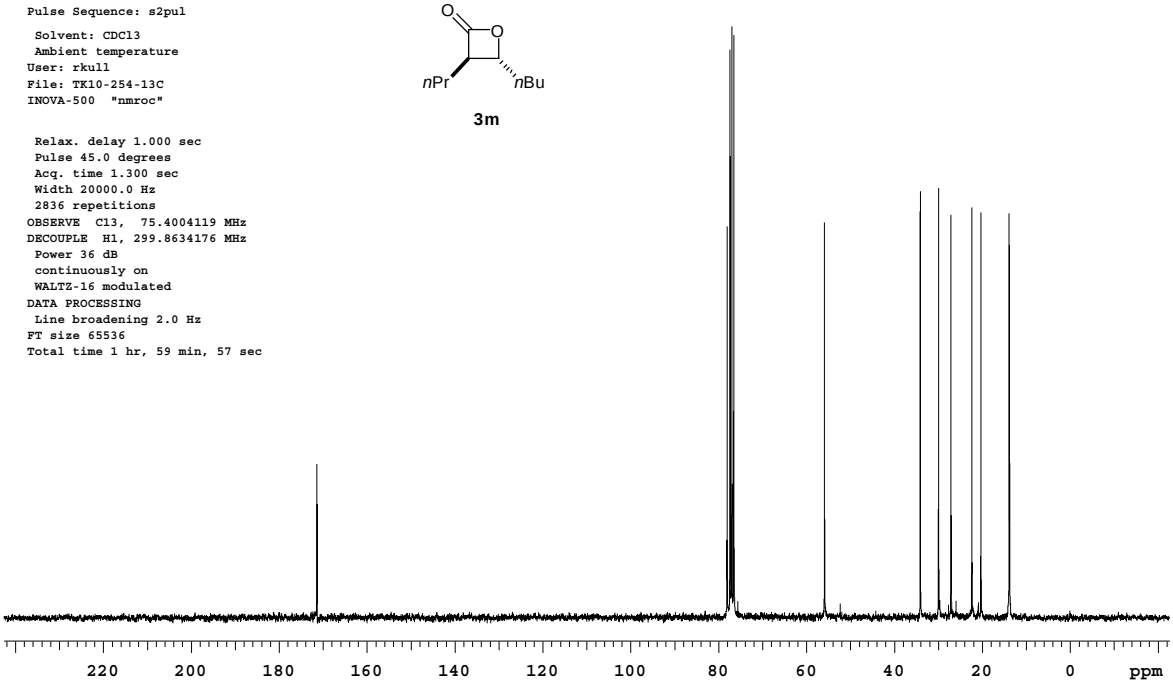
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-254-13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
2836 repetitions
OBSERVE C13, 75.4004119 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 59 min, 57 sec



3m



STANDARD 1H OBSERVE

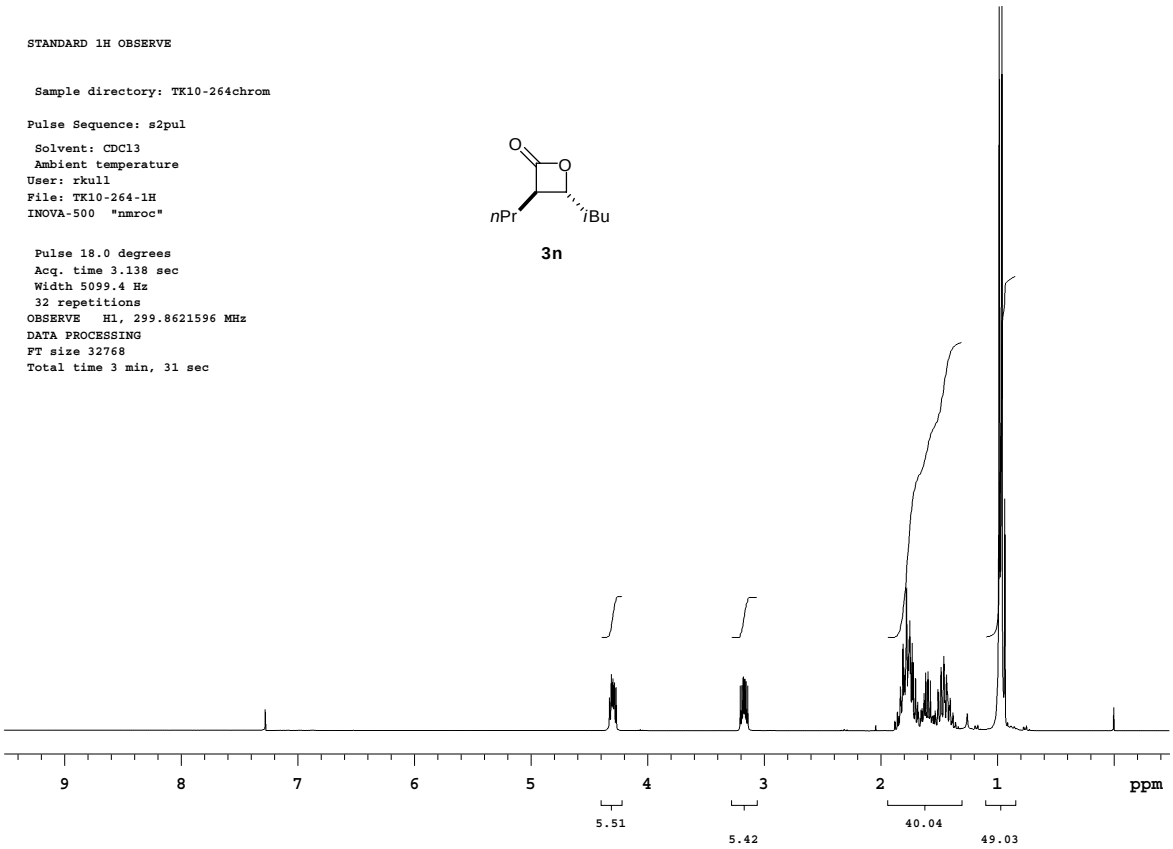
Sample directory: TK10-264chrom

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-264-1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
32 repetitions
OBSERVE H1, 299.8621596 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec

3n



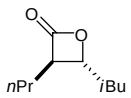
13C OBSERVE

Sample directory: TK10-264

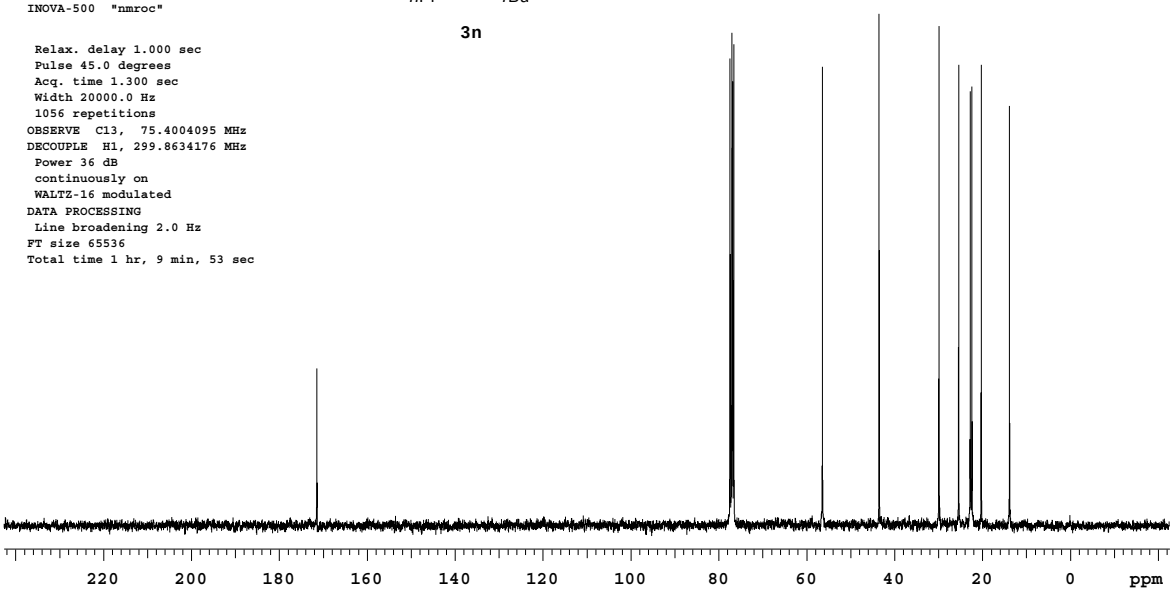
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkull
File: TK10-264-13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1056 repetitions
OBSERVE C13, 75.4004095 MHz
DECOUPLE H1, 299.8634176 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 9 min, 53 sec

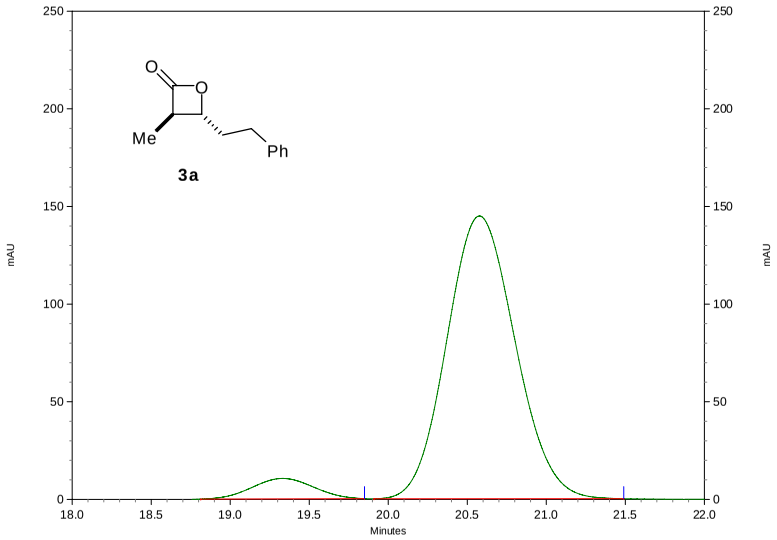


3n



Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\lactone\Data\TK10-22497-3-210-40min.met3-7-2008 6-48-25 PM.dat
Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ADH 90-10 EtOH 80min.met
Acquired: 3/7/2008 7:55:54 PM
Printed: 3/7/2008 8:14:22 PM

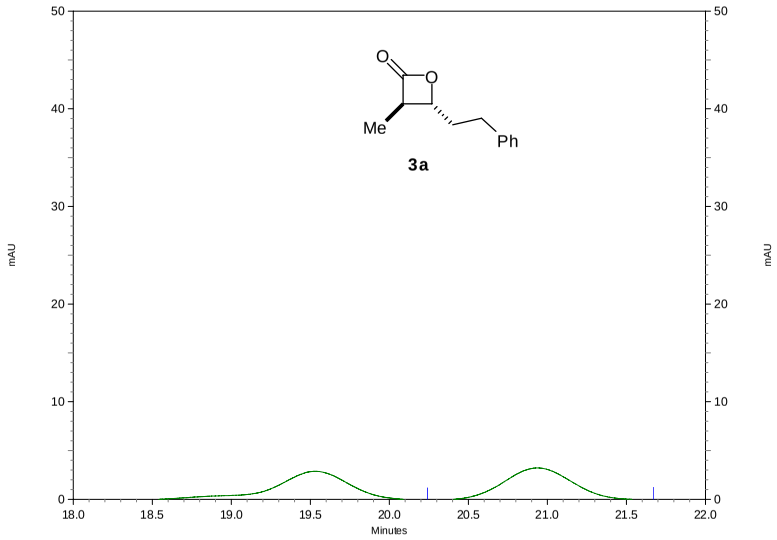


UV Results

Retention Time	Area	Area Percent	Height	Height Percent
19.333	1133462	6.106	41861	6.740
20.577	17430871	93.894	579234	93.260
Totals	18564333	100.000	621095	100.000

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\lactone\Data\TK10-13897-3-210-40min.met3-7-2008 7-29-14 PM.dat
Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ADH 90-10 EtOH 80min.met
Acquired: 3/7/2008 8:08:39 PM
Printed: 3/7/2008 8:10:29 PM

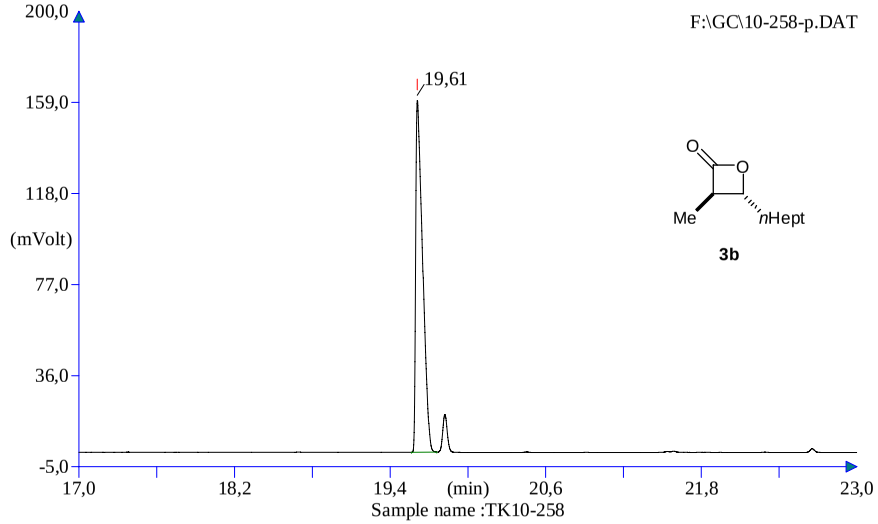


UV Results

Retention Time	Area	Area Percent	Height	Height Percent
19.530	391824	50.112	11707	47.314
20.940	390078	49.888	13036	52.686
Totals	781902	100.000	24743	100.000

GC-REPORT

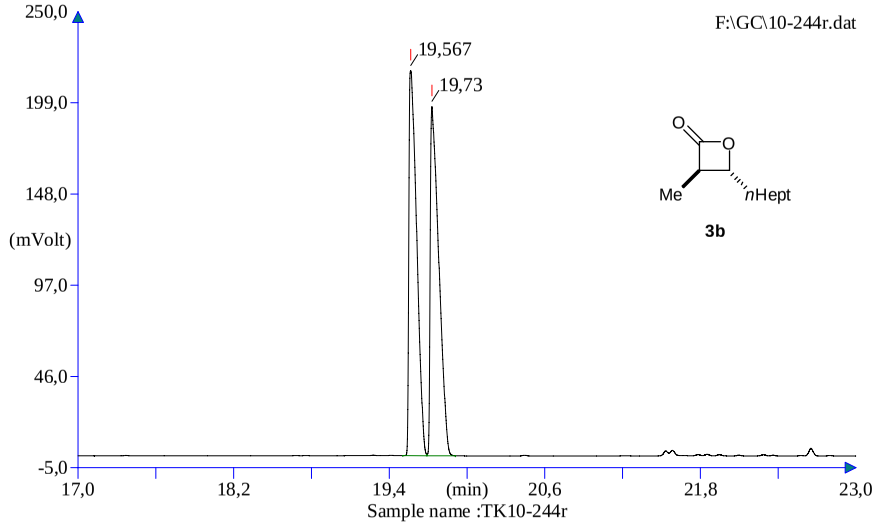
Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	01.03.2008 02:32
GC method:	
Sampler method:	
Sample ID:	TK10-258
Chromatogram filename:	F:\GC\10-258-p.DAT



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
19.610	5846638	93.378
19.823	414608	6.622
	6261246	

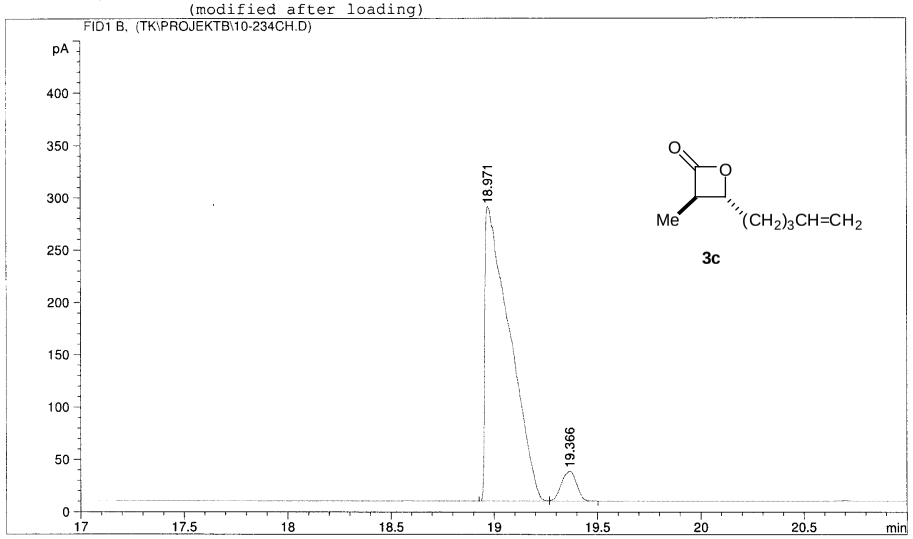
GC-REPORT

Method filename:	F:\GC\24.02.08a.mth
Method name:	Sven
Analysed:	26.02.2008 01:42
GC method:	
Sampler method:	
Sample ID:	TK10-244r
Chromatogram filename:	F:\GC\10-244r.dat



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
19.567	8977787	50.043
19.730	8962495	49.957
	17940280	

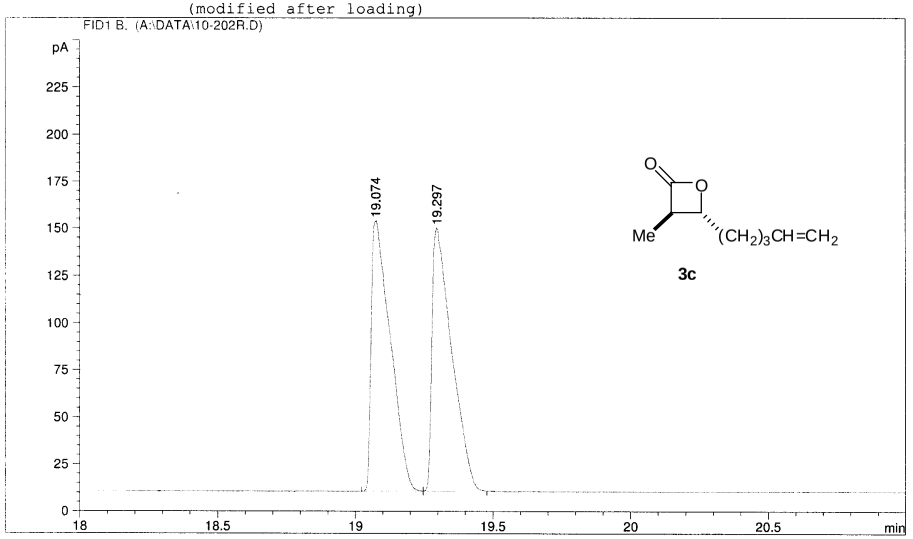
```
Injection Date  : 04.02.08 17:47:42          Seq. Line :   1
Sample Name     : TK10-234Chrom               Location : Vial 6
Acq. Operator   : Bert                           Inj :   1
Acq. Instrument : GC 4                     Inj Volume : 1 µl
Acq. Method     : D:\HPCHEM\2\METHODS\TK_ALL.M
Last changed    : 18.01.2008 16:13:06  by Bert
Analysis Method : C:\HPCHEM\1\METHODS\SLEEP2.M
Last changed    : 21.02.2008 14:23:32  by TK
                  (modified after loading)
```




```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 B,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  18.971 BB    0.1059 2330.17749  279.91925 94.15490
   2  19.366 BB    0.0725  144.65665   28.28961  5.84510

Totals :                  2474.83414  308.20886

 Results obtained with enhanced integrator!

                        *** End of Report ***
```



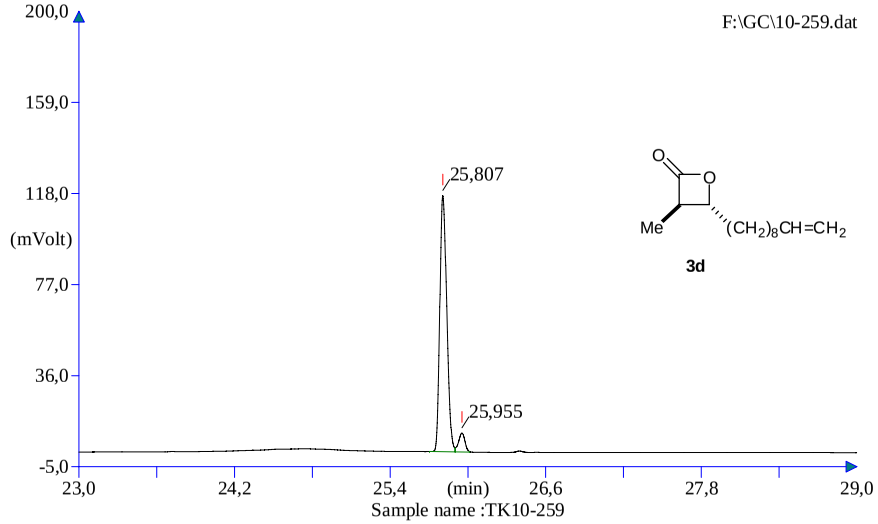
```
Injection Date  : 09.01.08 10:47:54          Seq. Line :   1
Sample Name     : 10-202r                     Location : Vial 6
Acq. Operator   : Fatos                          Inj :   1
Acq. Instrument : GC 4                     Inj Volume : 1 µl
Acq. Method     : D:\HPCHEM\2\METHODS\FH_MIX.M
Last changed    : 09.01.2008 10:43:26  by Fatos
Analysis Method : C:\HPCHEM\1\METHODS\SLEEP2.M
Last changed    : 10.01.2008 14:19:11  by TK
                  (modified after loading)
```




```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 B,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  19.074 BB    0.0662  694.56091  143.20177 49.97597
   2  19.297 BB    0.0662  695.22876  139.33026 50.02403

Totals :                  1389.78967  282.53203

 Results obtained with enhanced integrator!

                        *** End of Report ***
```

GC-REPORT

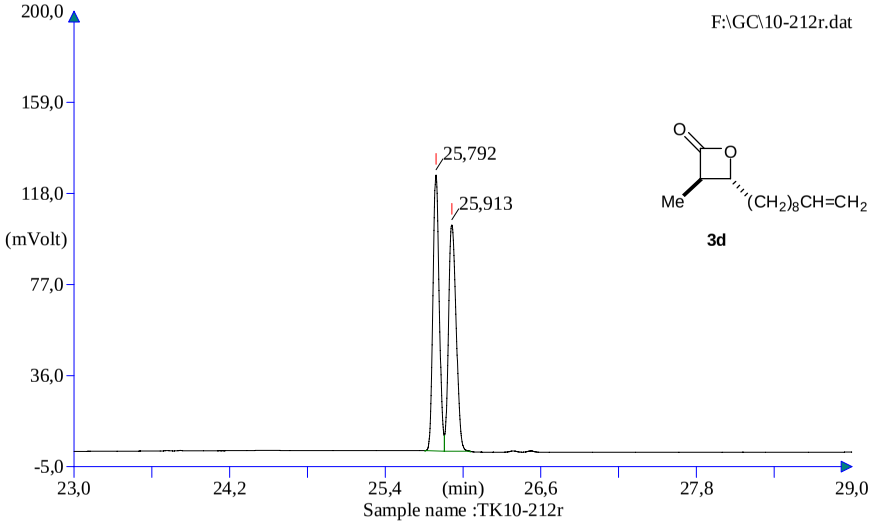
Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	01.03.2008 03:37
GC method:	
Sampler method:	
Sample ID:	TK10-259
Chromatogram filename:	F:\GC\10-259.dat



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
25.807	4323128	93.692
25.955	291070	6.308
	4614197	

GC-REPORT

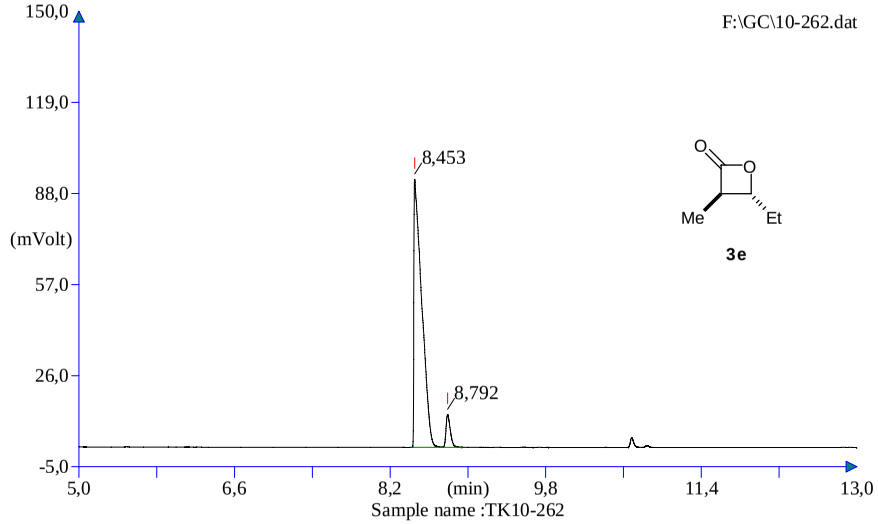
Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	25.02.2008 22:27
GC method:	
Sampler method:	
Sample ID:	TK10-212r
Chromatogram filename:	F:\GC\10-212r.dat



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
25.792	4198837	50.066
25.913	4187823	49.934
	8386660	

GC-REPORT

Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	01.03.2008 00:22
GC method:	
Sampler method:	
Sample ID:	TK10-262
Chromatogram filename:	F:\GC\10-262.dat

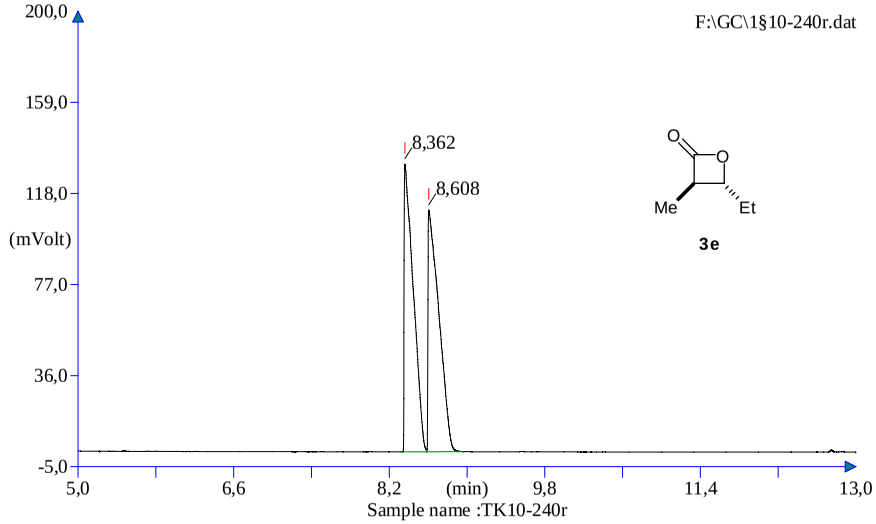


3e

Retention Time (min)	Area (.1*uV*sec)	Area % (%)
8.453	5108084	93.715
8.792	342586	6.285
	5450670	

GC-REPORT

Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	23.02.2008 22:54
GC method:	
Sampler method:	
Sample ID:	TK10-240r
Chromatogram filename:	F:\GC\1§10-240r.dat

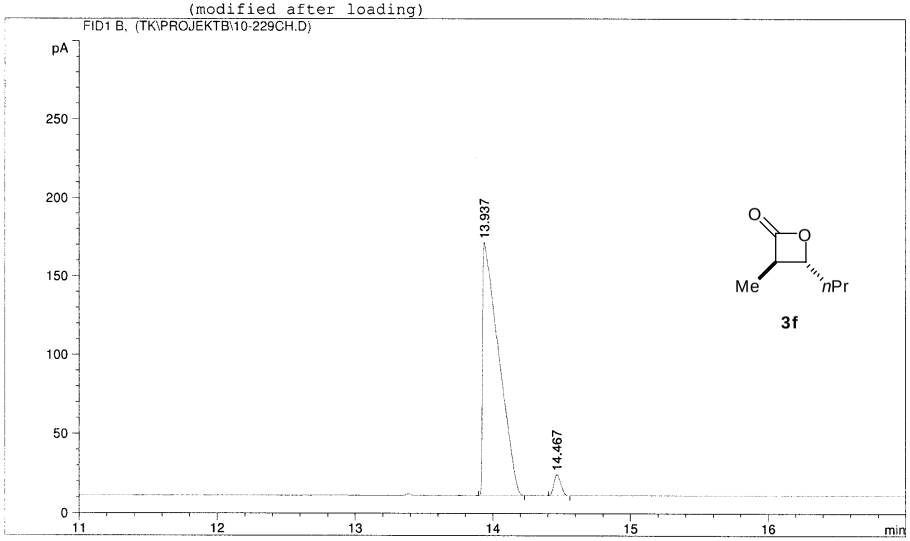


3e

Retention Time (min)	Area (.1*uV*sec)	Area % (%)
8.362	8463826	49.946
8.608	8482073	50.054
	16945900	

Data File D:\HPCHEM\1\DATA\TK\PROJEKTB\10-229CH.D Sample Name: TK10-229Chrom

```
Injection Date  : 31.01.08 15:46:04          Seq. Line :   1
Sample Name     : TK10-229Chrom               Location : Vial 6
Acq. Operator   : Bert                           Inj :   1
Acq. Instrument : GC 4                     Inj Volume : 1 µl
Acq. Method     : D:\HPCHEM\2\METHODS\TK_ALL.M
Last changed    : 18.01.2008 16:13:06  by Bert
Analysis Method : C:\HPCHEM\1\METHODS\SLEEP2.M
Last changed    : 16.02.2008 11:40:39  by TK
                  (modified after loading)
```



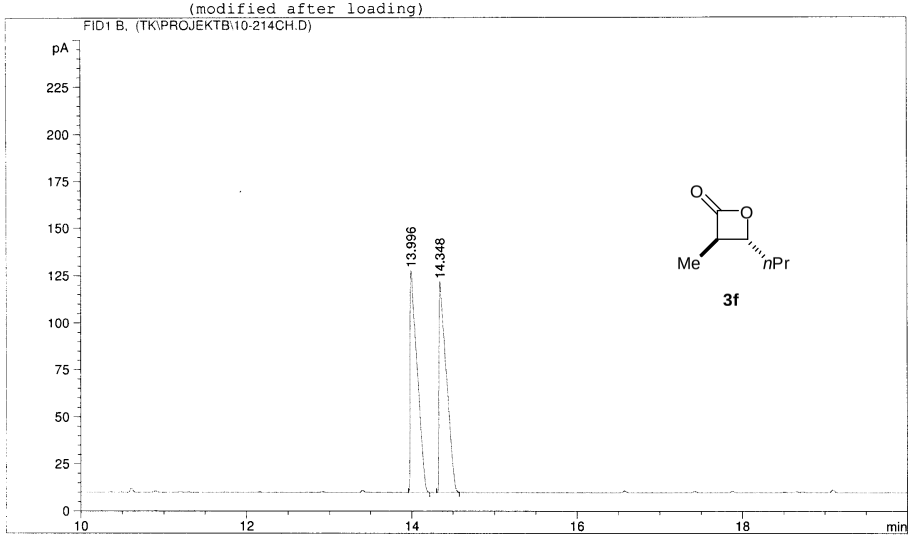
Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 B,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  13.937 PB    0.1049 1308.58936  160.16525 96.56572
   2  14.467 BB    0.0548   46.53884   13.18479  3.43428

Totals :                  1355.12819  173.35004

 Results obtained with enhanced integrator!
```

*** End of Report ***

Data File D:\HPCHEM\1\DATA\TK\PROJEKTB\10-214CH.D Sample Name: TK10-214Chrom

```
Injection Date  : 26.01.08 15:55:50          Seq. Line :   2
Sample Name     : TK10-214Chrom               Location : Vial 7
Acq. Operator   : Bert                           Inj :   1
Acq. Instrument : GC 4                     Inj Volume : 1 µl
Acq. Method     : D:\HPCHEM\2\METHODS\TK_ALL.M
Last changed    : 18.01.2008 16:13:06  by Bert
Analysis Method : C:\HPCHEM\1\METHODS\SLEEP2.M
Last changed    : 21.02.2008 14:20:46  by TK
                  (modified after loading)
```



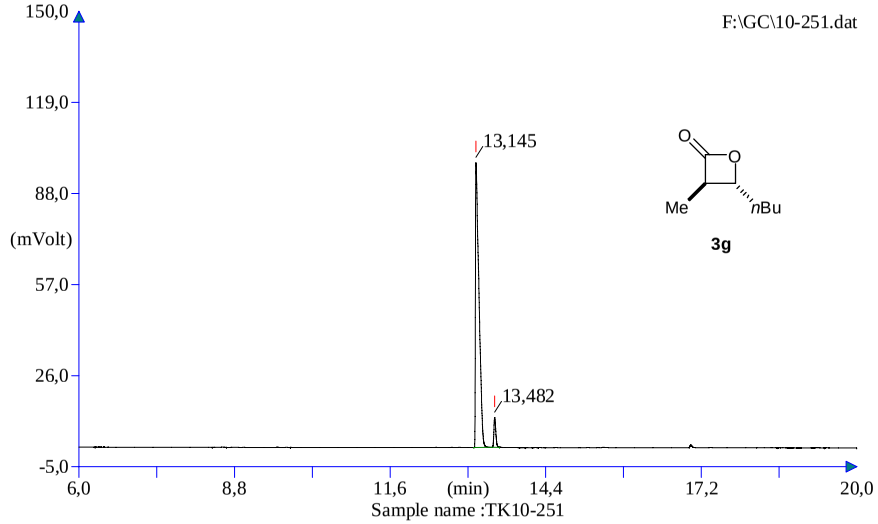
Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 B,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  13.996 PB    0.0780  696.38397  117.46735 49.99379
   2  14.348 PB    0.0801  696.55707  111.44951 50.00621

Totals :                  1392.94104  228.91686

 Results obtained with enhanced integrator!
```

*** End of Report ***

GC-REPORT

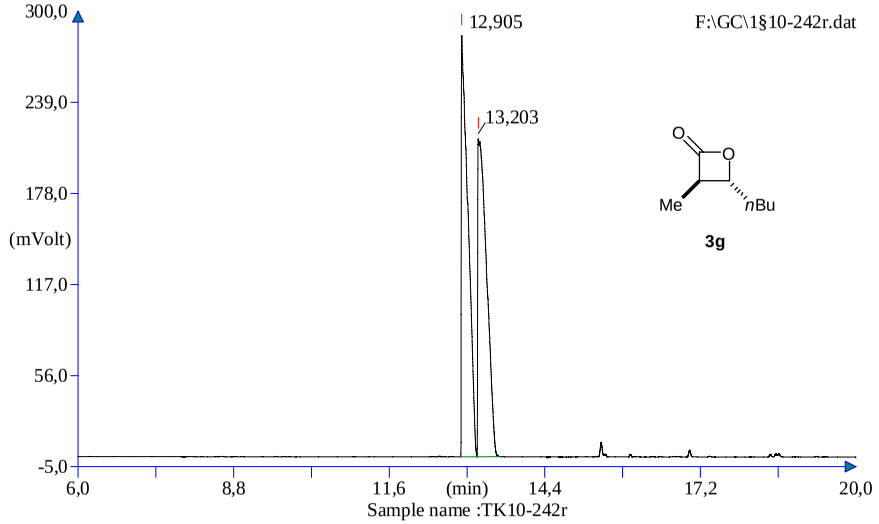
Method filename: F:\GC\23.02.08d.mth
Method name: Sven
Analysed: 01.03.2008 01:27
GC method:
Sampler method:
Sample ID: TK10-251
Chromatogram filename: F:\GC\10-251.dat



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
13.145	4266344	94.662
13.482	240579	5.338
	4506923	

GC-REPORT

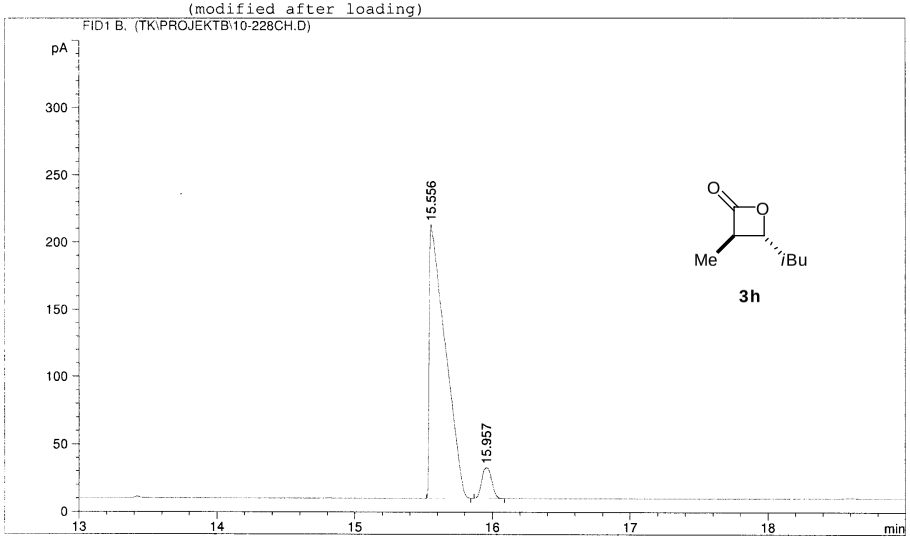
Method filename: F:\GC\23.02.08d.mth
Method name: Sven
Analysed: 23.02.2008 23:49
GC method:
Sampler method:
Sample ID: TK10-242r
Chromatogram filename: F:\GC\1§10-242r.dat



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
12.905	23581610	49.978
13.203	23602830	50.022
	47184440	

Data File D:\HPCHEM\1\DATA\TK\PROJEKTB\10-228CH.D Sample Name: TK10-228chrom

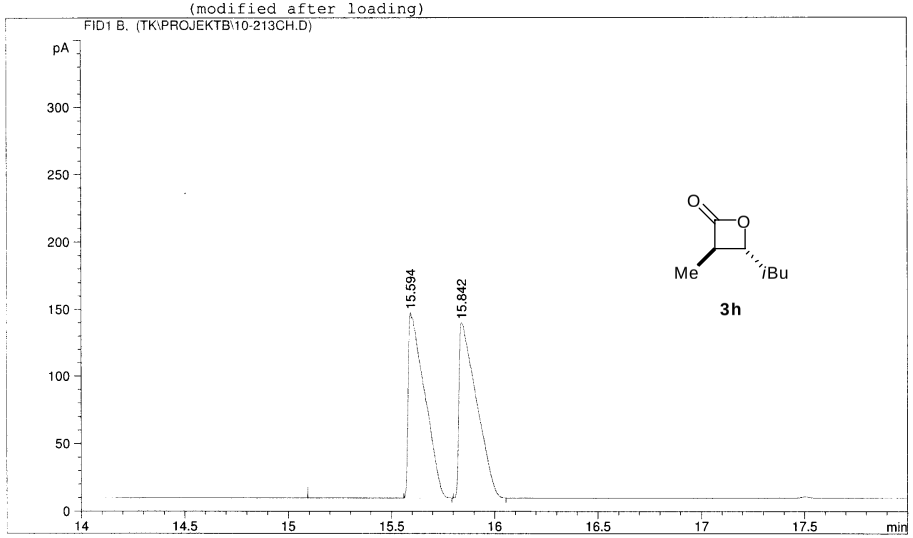
```
==================================================================
Injection Date  : 01.02.08 19:09:59          Seq. Line :   1
Sample Name     : TK10-228chrom               Location : Vial 6
Acq. Operator   : Bert                           Inj :   1
Acq. Instrument : GC 4                    Inj Volume : 1 µl
Acq. Method     : D:\HPCHEM\2\METHODS\TK_ALL.M
Last changed    : 18.01.2008 16:13:06  by Bert
Analysis Method : C:\HPCHEM\1\METHODS\SLEEP2.M
Last changed    : 21.02.2008 14:15:53  by TK
                  (modified after loading)
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 B,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  15.556 PP    0.0987 1617.41284  202.61157 93.51834
   2  15.957 BB    0.0697  112.10123   22.66980  6.48166

Totals :                  1729.51407  225.28138

 Results obtained with enhanced integrator!
==================================================================
                      *** End of Report ***
```


Data File D:\HPCHEM\1\DATA\TK\PROJEKTB\10-213CH.D Sample Name: TK10-213Chrom

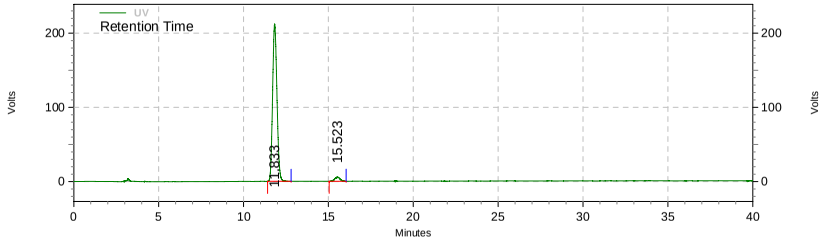
```
==================================================================
Injection Date  : 26.01.08 15:20:41          Seq. Line :   1
Sample Name     : TK10-213Chrom               Location : Vial 6
Acq. Operator   : Bert                           Inj :   1
Acq. Instrument : GC 4                    Inj Volume : 1 µl
Acq. Method     : D:\HPCHEM\2\METHODS\TK_ALL.M
Last changed    : 18.01.2008 16:13:06  by Bert
Analysis Method : C:\HPCHEM\1\METHODS\SLEEP2.M
Last changed    : 21.02.2008 14:12:13  by TK
                  (modified after loading)
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 B,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  15.594 BB    0.0694  761.51361  137.52124 49.98992
   2  15.842 BB    0.0763  761.82074  130.06361 50.01008

Totals :                  1523.33435  267.58485

 Results obtained with enhanced integrator!
==================================================================
                      *** End of Report ***
```

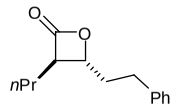
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\lactone\Data\TK10-25097-3-210-40min.met3-7-2008 9-15-03 AM.dat
Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ADH 90-10 EtOH 80min.met
Acquired: 3/7/2008 9:15:49 AM
Printed: 3/7/2008 4:11:14 PM



UV Results

Retention Time	Area	Area %	Height	Height %
11.833	15144812	96.77	847893	97.46
15.523	505215	3.23	22083	2.54
Totals	15650027	100.00	869976	100.00

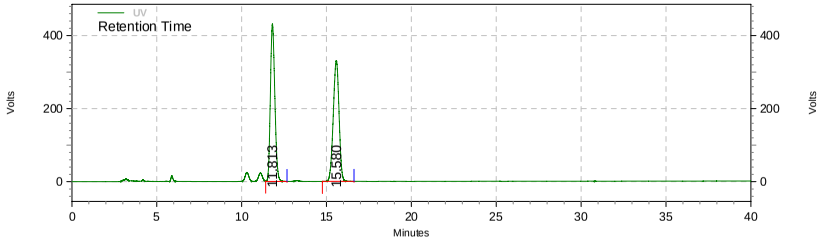


3i

Page 1 of 1

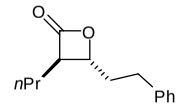
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\lactone\Data\TK10-24897-3-210-40min.met3-7-2008 8-34-01 AM.dat
Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ADH 90-10 EtOH 80min.met
Acquired: 3/7/2008 8:34:55 AM
Printed: 3/7/2008 4:08:12 PM



UV Results

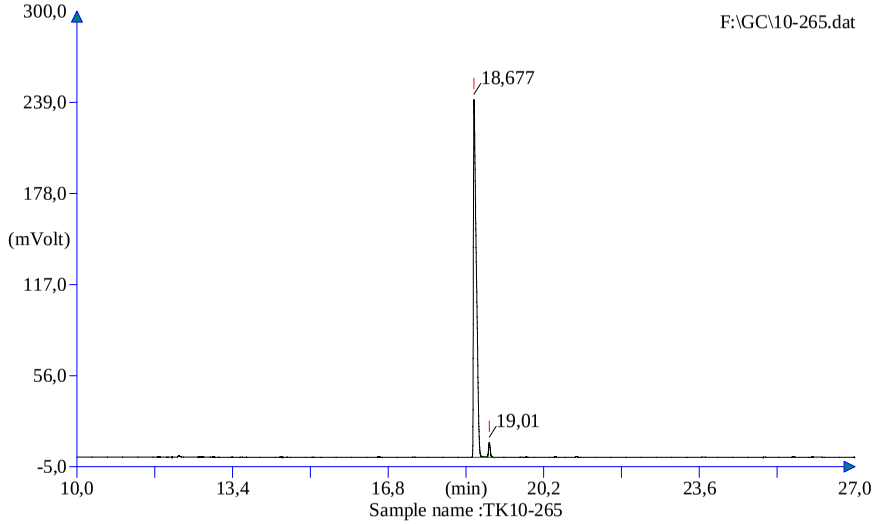
Retention Time	Area	Area %	Height	Height %
11.813	31289414	49.95	1724248	56.55
15.580	31350659	50.05	1324606	43.45
Totals	62640073	100.00	3048854	100.00



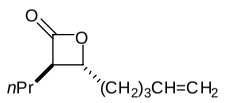
3i

GC-REPORT

Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	05.03.2008 22:05
GC method:	
Sampler method:	
Sample ID:	TK10-265
Chromatogram filename:	F:\GC\10-265.dat



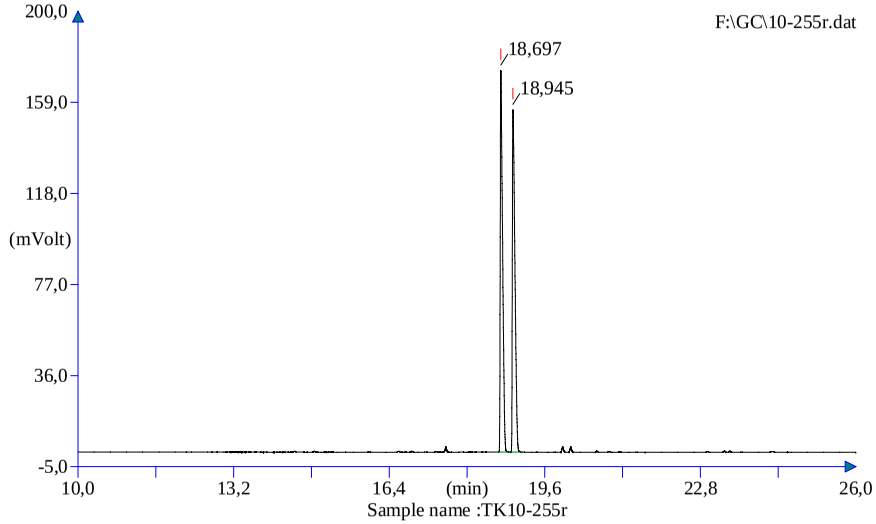
Retention Time (min)	Area (.1*uV*sec)	Area % (%)
18.677	10488490	97.698
19.010	247187	2.302
	10735680	



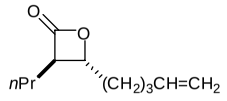
3j

GC-REPORT

Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	27.02.2008 12:38
GC method:	
Sampler method:	
Sample ID:	TK10-255r
Chromatogram filename:	10-255r.dat



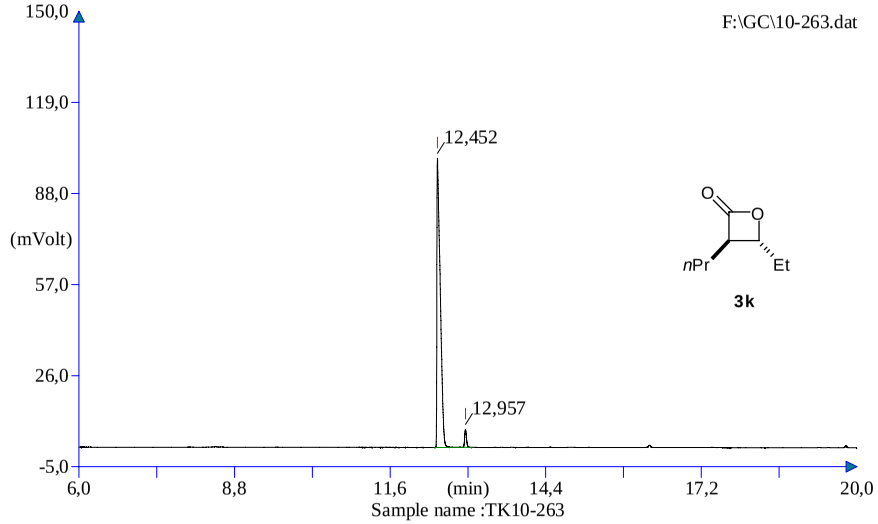
Retention Time (min)	Area (.1*uV*sec)	Area % (%)
18.697	5699592	50.072
18.945	5683316	49.929
	11382910	



3j

GC-REPORT

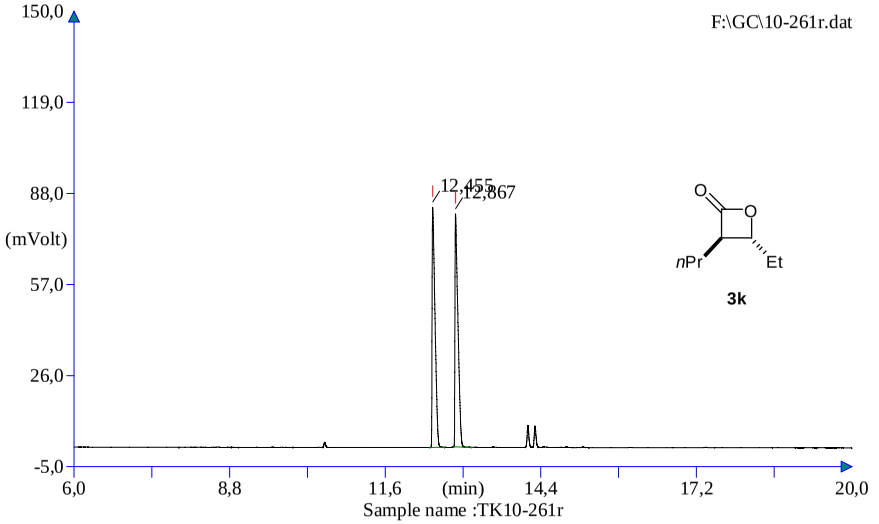
Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	01.03.2008 14:26
GC method:	
Sampler method:	
Sample ID:	TK10-263
Chromatogram filename:	F:\GC\10-263.dat



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
12.452	4132551	96.766
12.957	138093	3.234
	4270645	

GC-REPORT

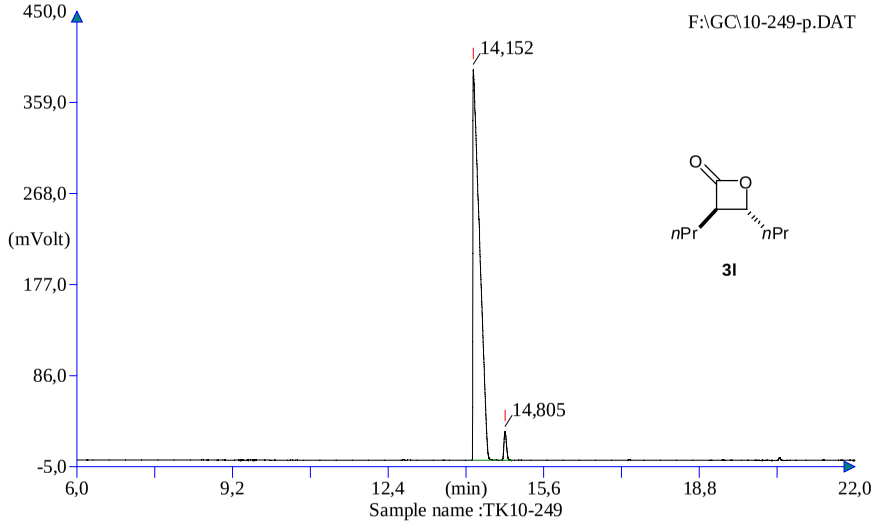
Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	01.03.2008 04:42
GC method:	
Sampler method:	
Sample ID:	TK10-261r
Chromatogram filename:	F:\GC\10-261r.dat



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
12.455	2967073	50.124
12.867	2952421	49.876
	5919494	

GC-REPORT

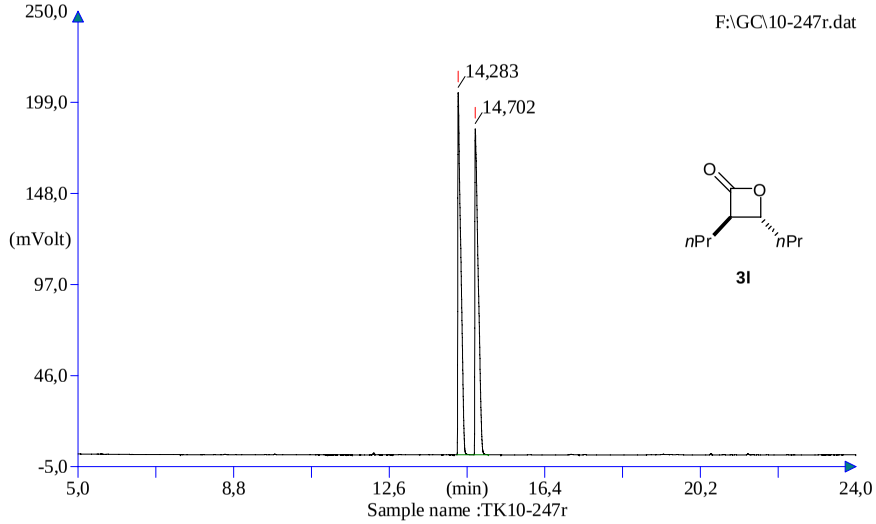
Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	23.02.2008 21:59
GC method:	
Sampler method:	
Sample ID:	TK10-249
Chromatogram filename:	F:\GC\10-249-p.DAT



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
14.152	37299920	97.333
14.805	1021991	2.667
	38321910	

GC-REPORT

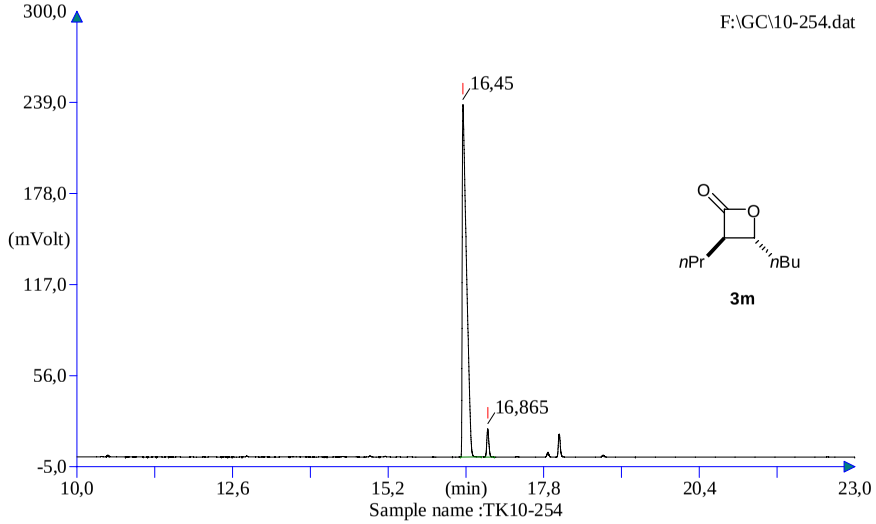
Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	22.02.2008 18:21
GC method:	
Sampler method:	
Sample ID:	TK10-247r
Chromatogram filename:	F:\GC\10-247r.dat



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
14.283	10493010	50.036
14.702	10478070	49.964
	20971080	

GC-REPORT

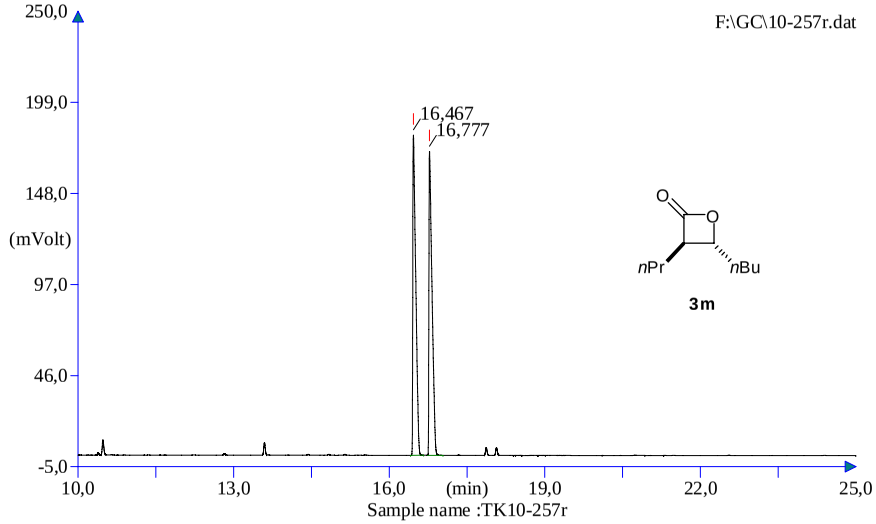
Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	27.02.2008 11:33
GC method:	
Sampler method:	
Sample ID:	TK10-254
Chromatogram filename:	F:\GC\10-254.dat



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
16.450	11636430	96.300
16.865	447081	3.700
	12083510	

GC-REPORT

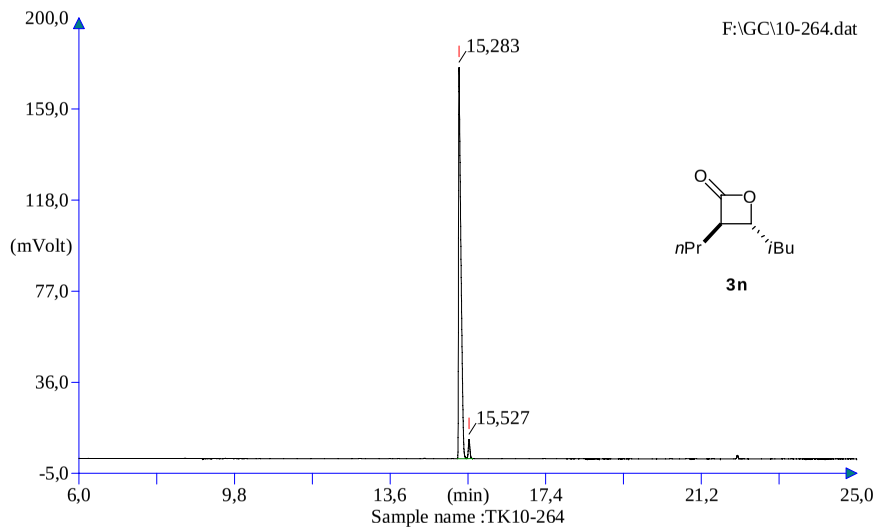
Method filename:	F:\GC\23.02.08d.mth
Method name:	Sven
Analysed:	27.02.2008 10:28
GC method:	
Sampler method:	
Sample ID:	TK10-257r
Chromatogram filename:	F:\GC\10-257r.dat



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
16.467	7073775	50.068
16.777	7054613	49.932
	14128390	

GC-REPORT

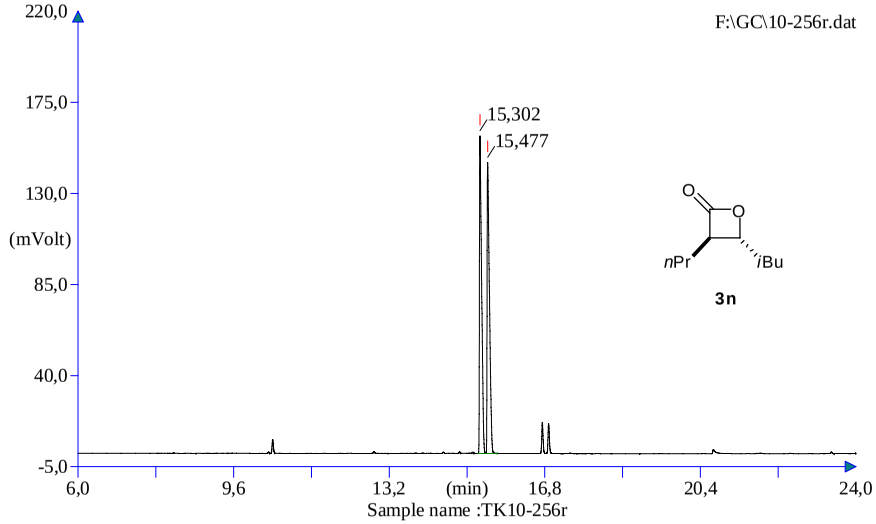
Method filename: F:\GC\23.02.08d.mth
Method name: Sven
Analysed: 01.03.2008 15:31
GC method:
Sampler method:
Sample ID: TK10-264
Chromatogram filename: F:\GC\10-264.dat



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
15.283	7160409	96.852
15.527	232701	3.148
	7393110	

GC-REPORT

Method filename: F:\GC\23.02.08d.mth
Method name: Sven
Analysed: 29.02.2008 20:43
GC method:
Sampler method:
Sample ID: TK10-256r
Chromatogram filename: F:\GC\10-256r.dat



Retention Time (min)	Area (.1*uV*sec)	Area % (%)
15.302	5394498	50.008
15.477	5392816	49.992
	10787310	