

Supporting information-Rhodium-catalysed conjugate addition of arylboronic acids to enantiopure dehydroamino acid derivatives

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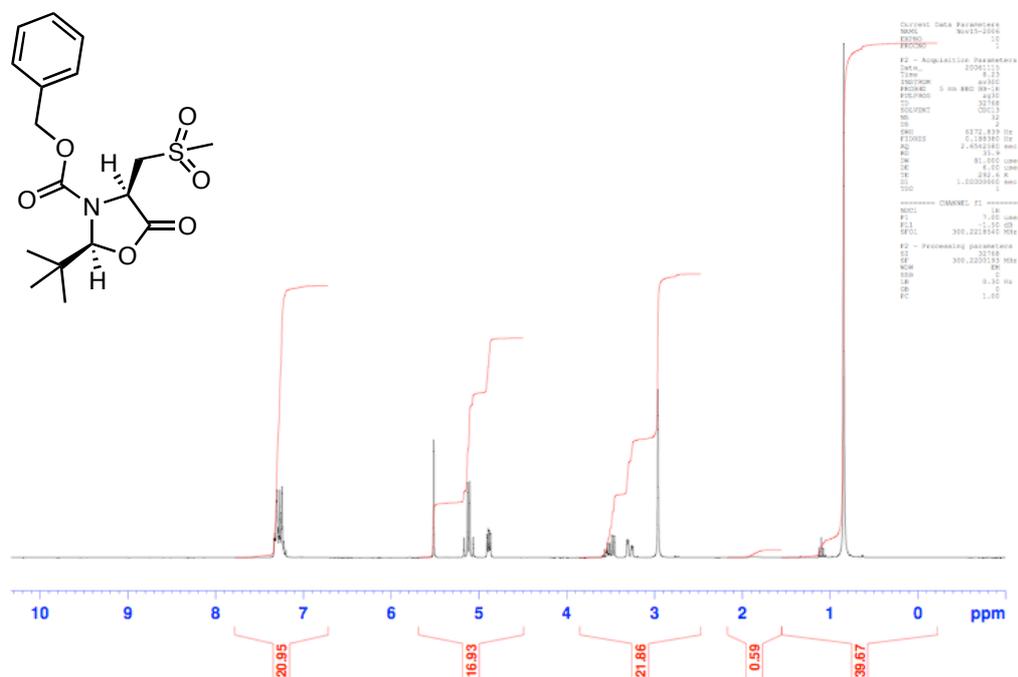
^b Pfizer Limited, Ramsgate Road, Sandwich, Kent, CT13 9NJ, UK.

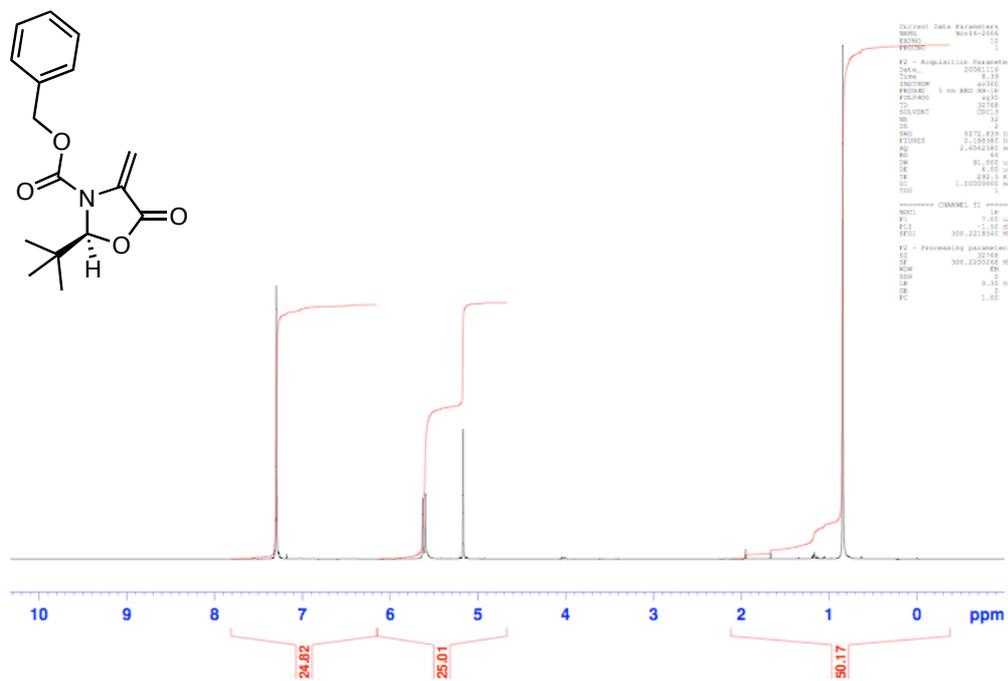
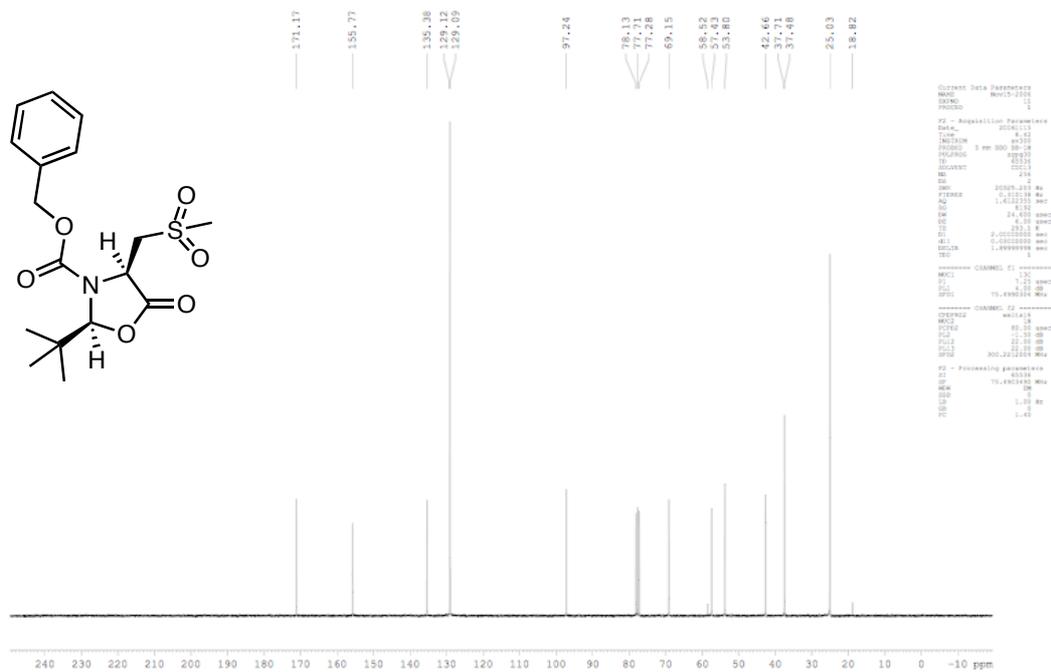
General experimental:

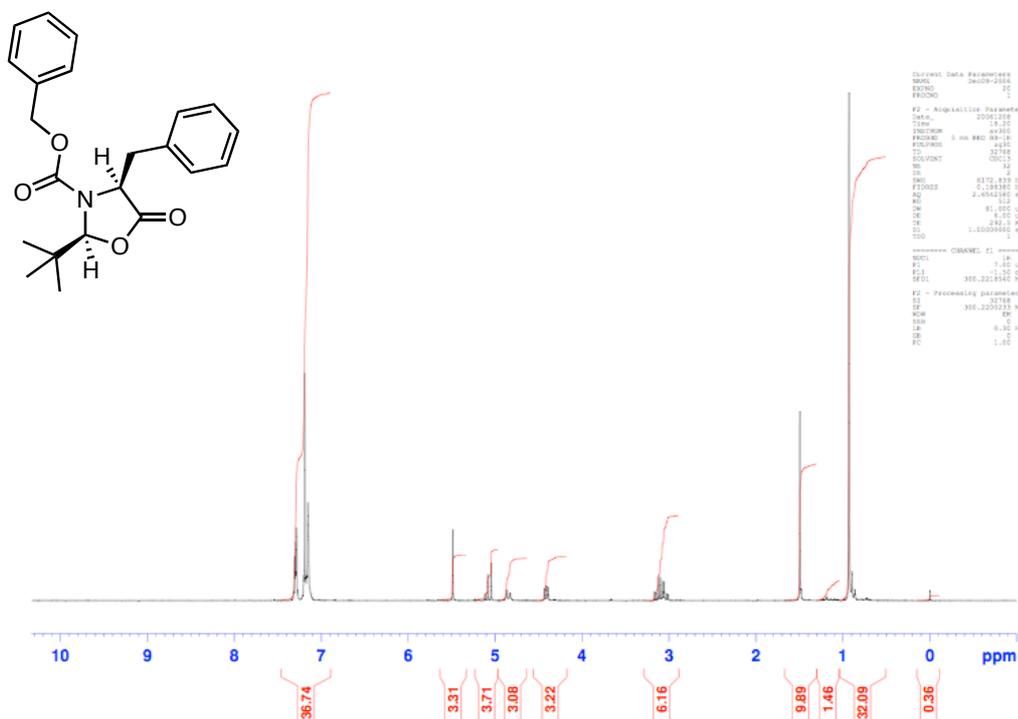
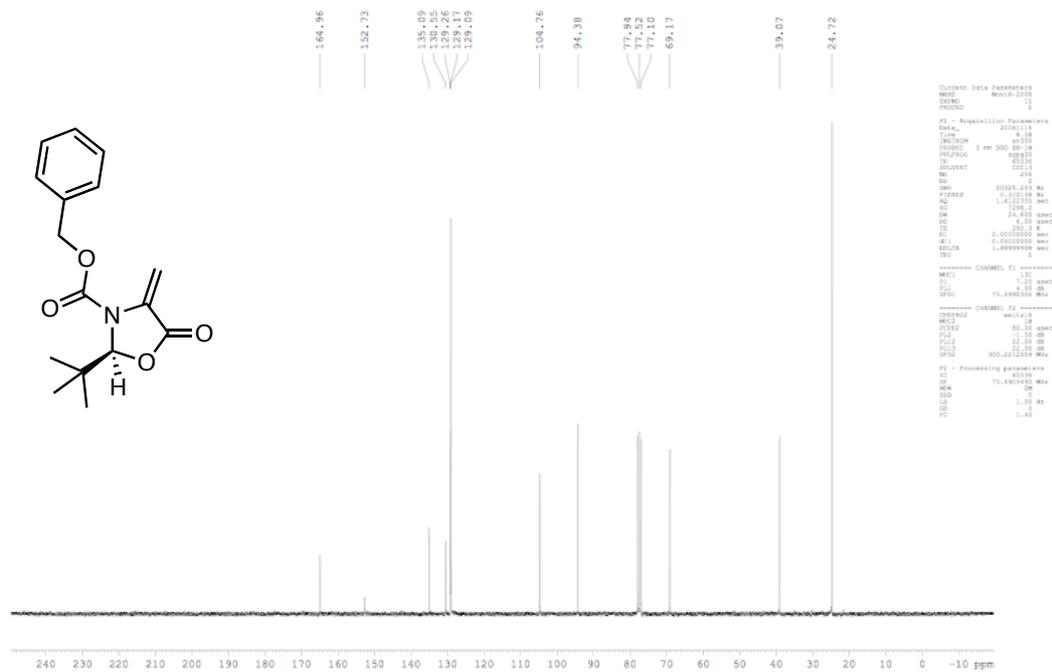
IR spectra were recorded on a Perkin-Elmer 1600 FT IR spectrophotometer, using NaCl discs. ¹H NMR spectra were obtained on a Bruker Avance 300 spectrometer operating at 300 MHz, unless otherwise noted, with tetramethylsilane as an internal standard. *J* values are given in Hz. ¹³C NMR spectra were obtained on a Bruker Avance 300 spectrometer operating at 75 MHz, unless otherwise noted. All dry solvents were freshly distilled under nitrogen prior to use. Mass spectra were obtained on a Bruker Time-of-Flight mass spectrometer (ESI-TOF). Enantiomeric excesses were determined using HPLC (see data see individual compounds details) with a UV detector at 254 nm. Tetrahydrofuran was distilled over alumina column. Petroleum ether refers to that fraction obtained between 40-60 °C. All other reagents were obtained from commercial suppliers and used as received. All glassware used under anhydrous conditions was dried in an oven and allowed to cool under nitrogen prior to use. All reactions were carried out under argon unless otherwise stated. Flash chromatography was conducted under medium pressure, using matrix 60 silica.

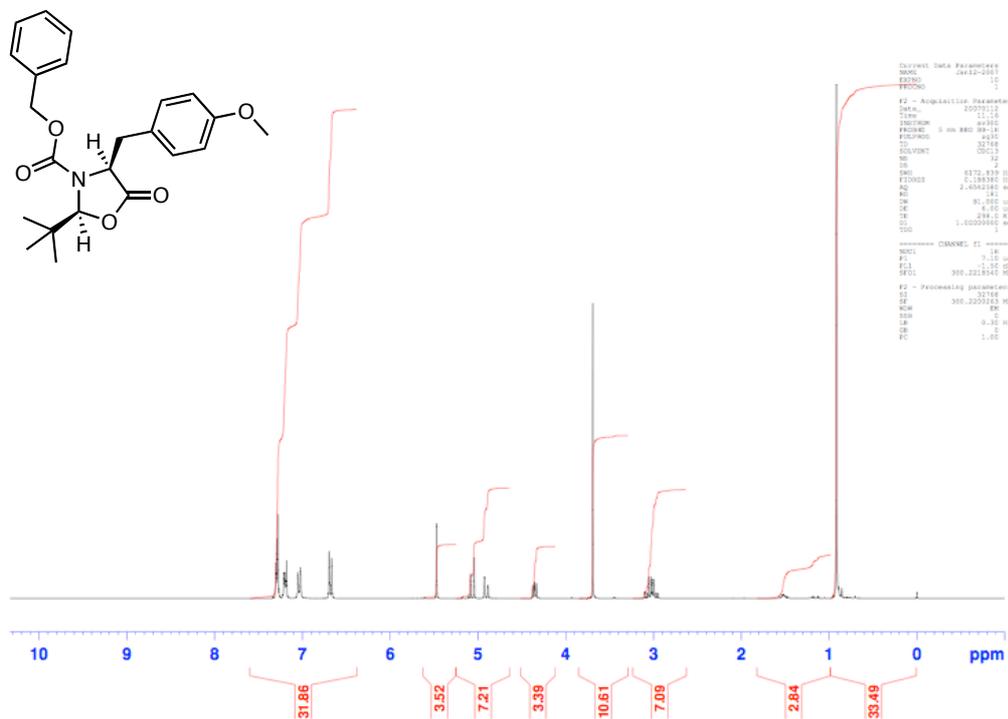
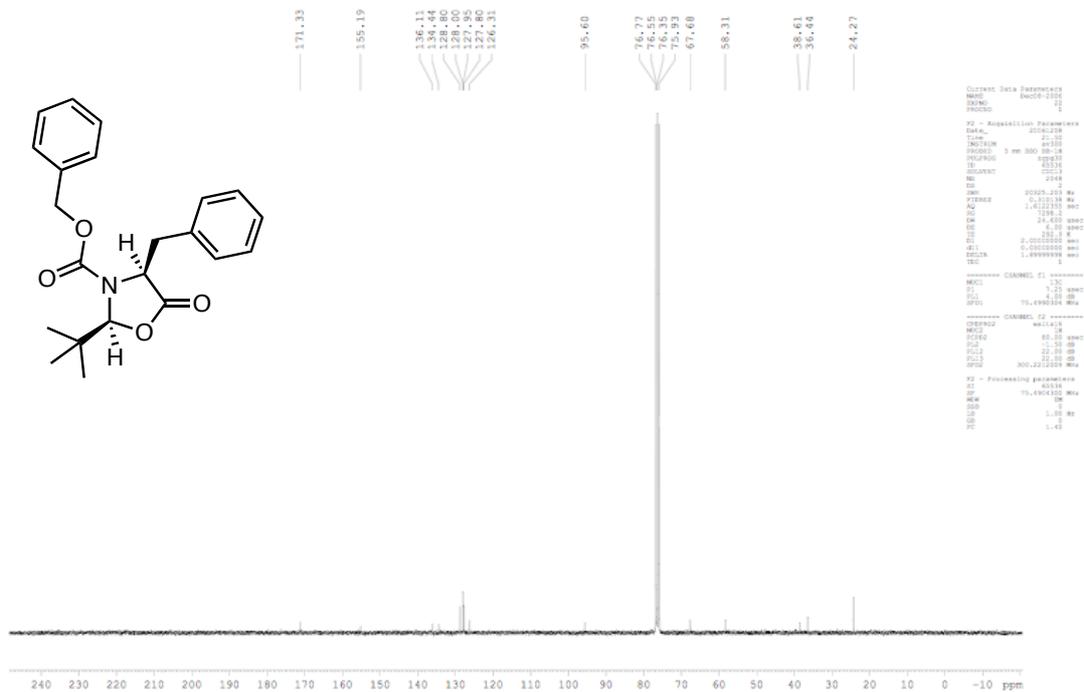
CCDC 776776 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

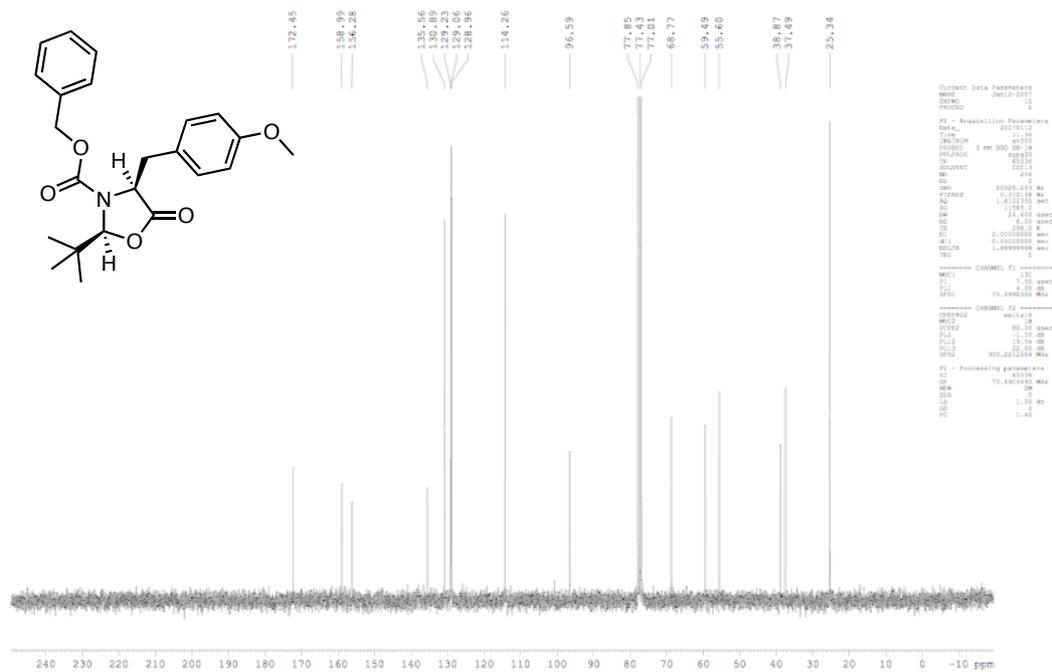
¹H and ¹³C NMR Spectra:



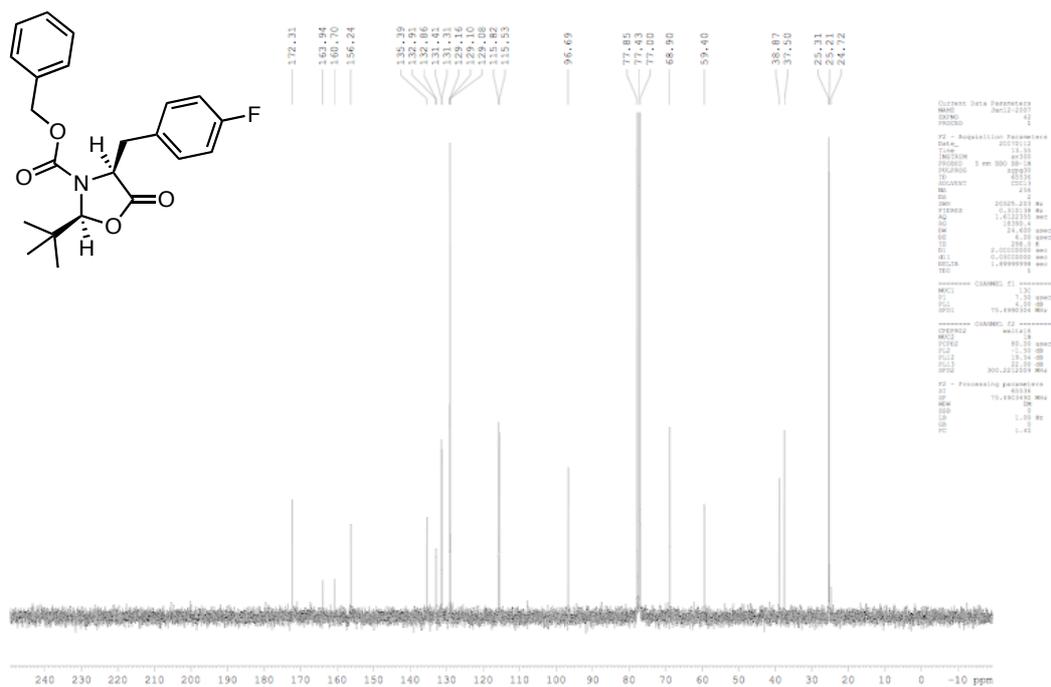
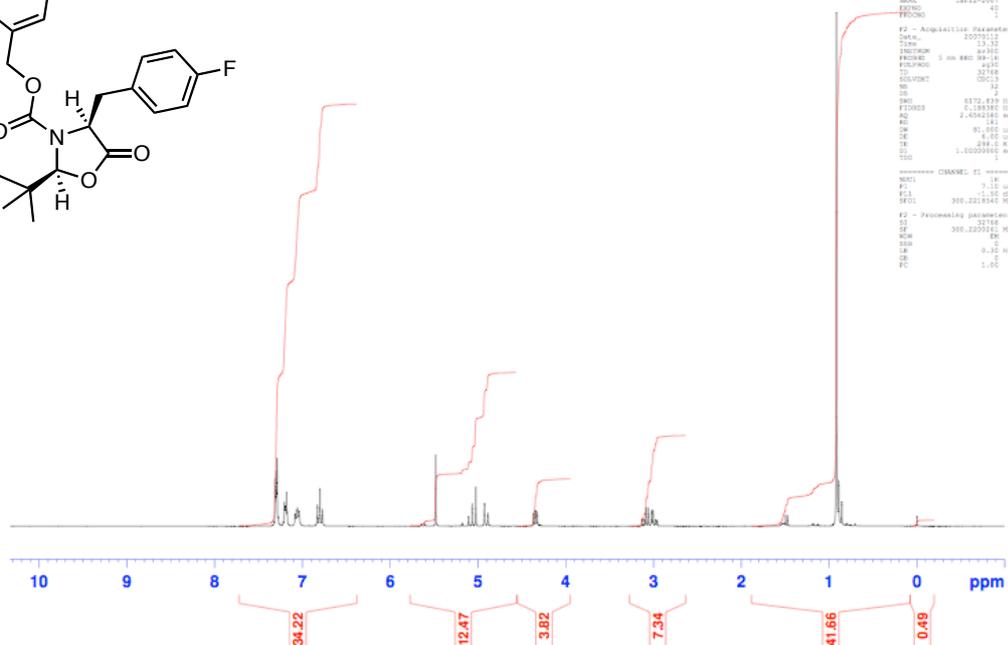
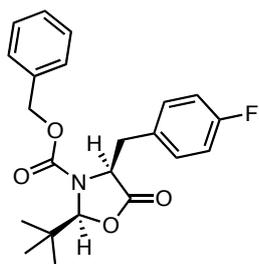


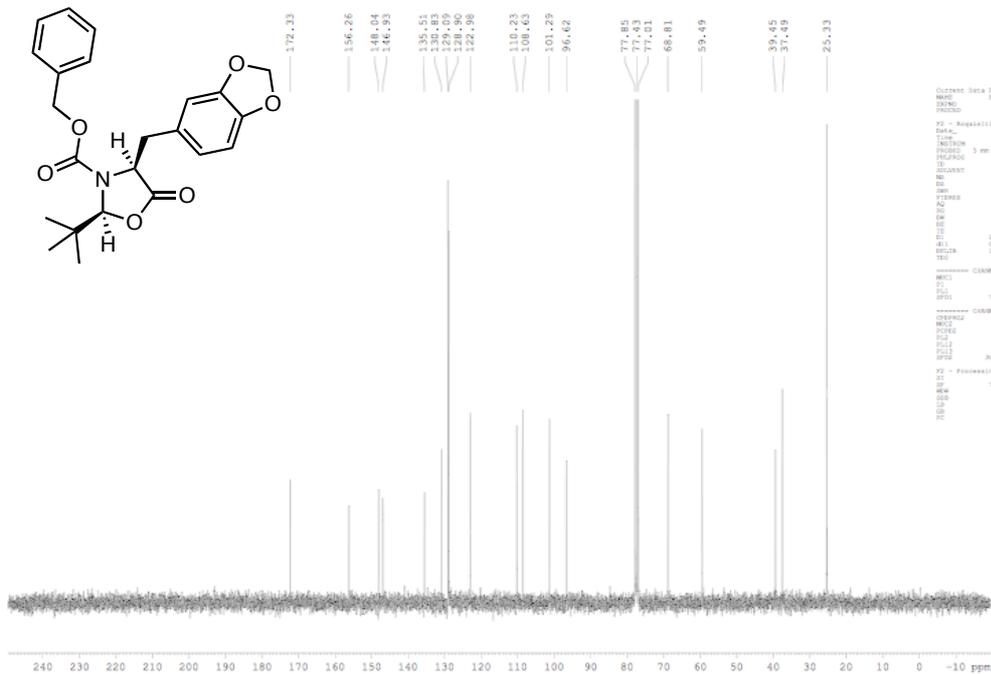
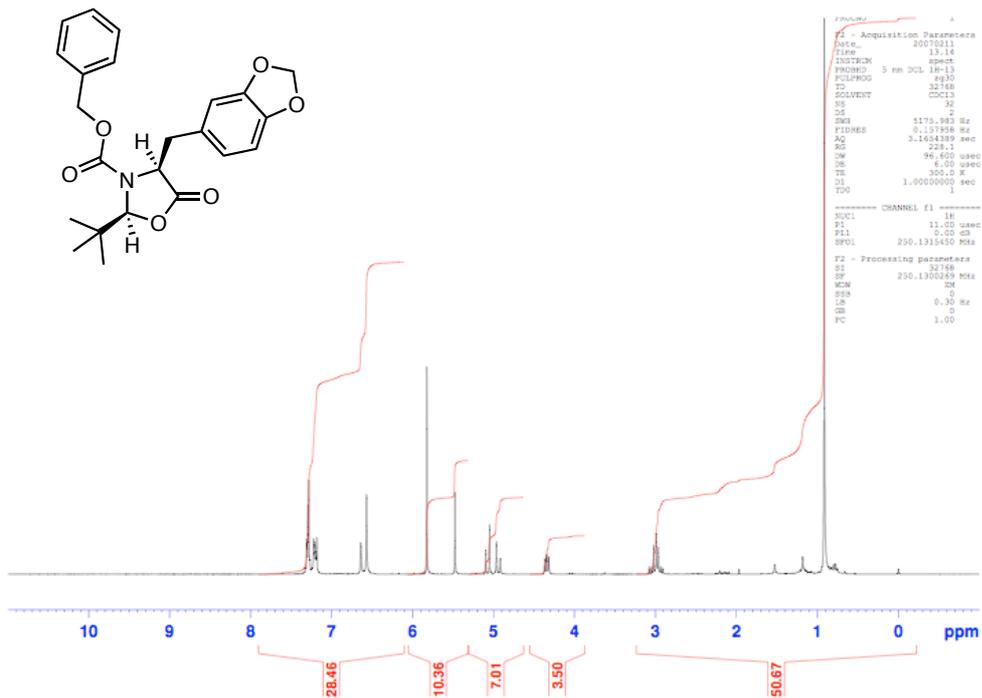


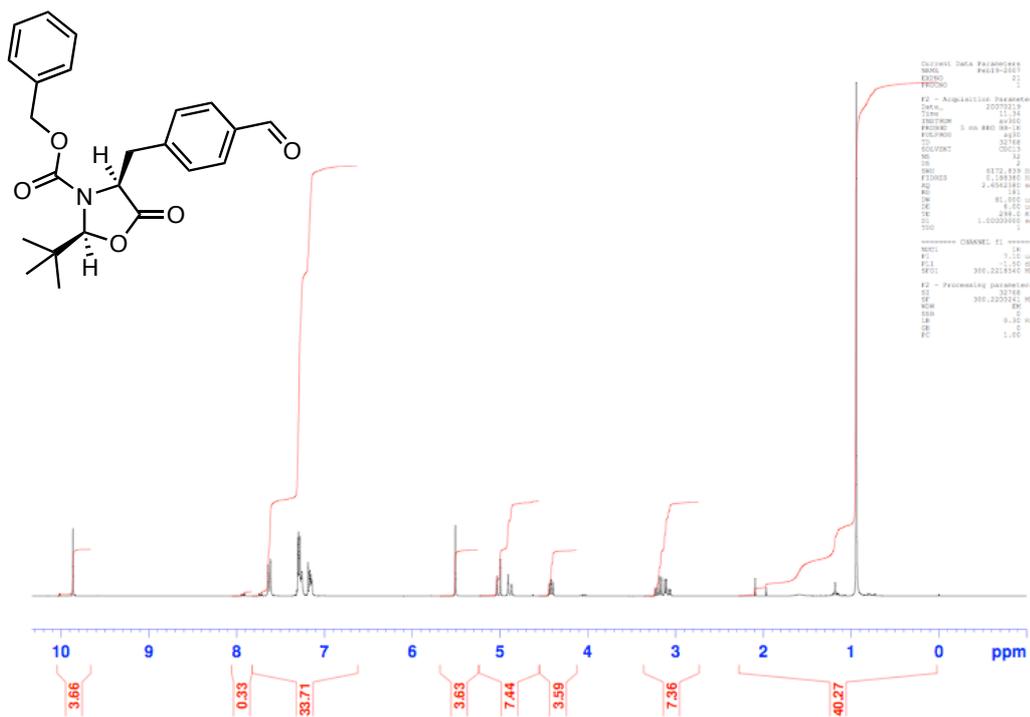




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Date_ 21:34
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PCPDPRG2
SOLVENT CDCl3
NS 204
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SWH 20321.428 Hz
FIDRES 0.102338 Hz
AQ 1.612210 sec
RG 1185.1
AQ 34.40 sec
RG 288.8 Hz
SI 2.0000000 sec
WDW 0.0000000 sec
SSB 0.0000000 sec
GB 0
TEC 0
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P1 1.20 sec
SFO 101.62538 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 13C
P2 1.20 sec
SFO2 101.62538 MHz
F2 - Processing parameters
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SFO2 101.62538 MHz
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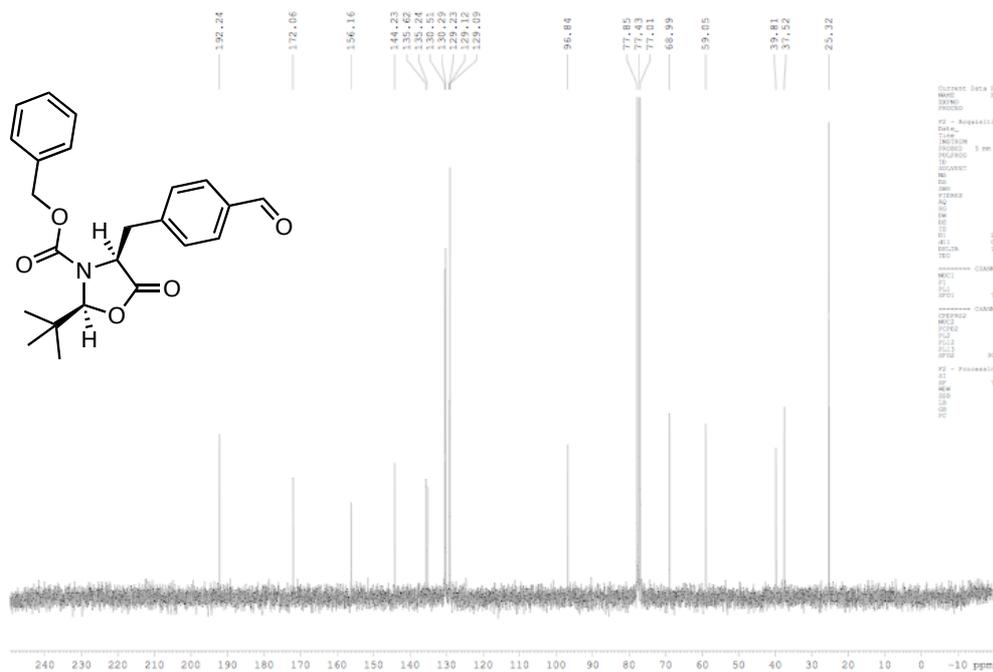






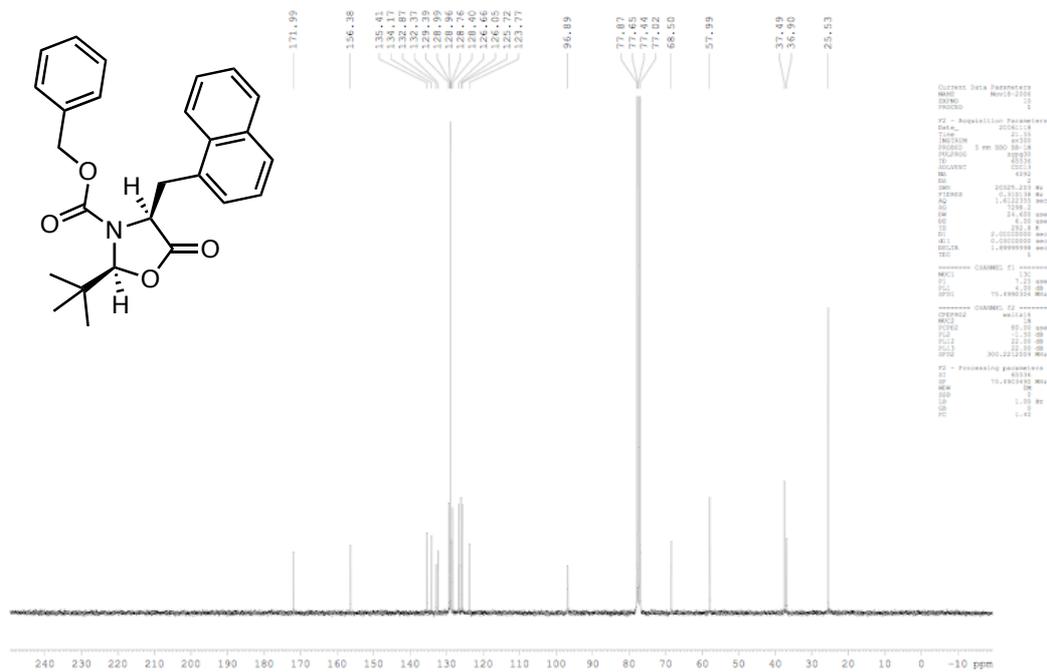
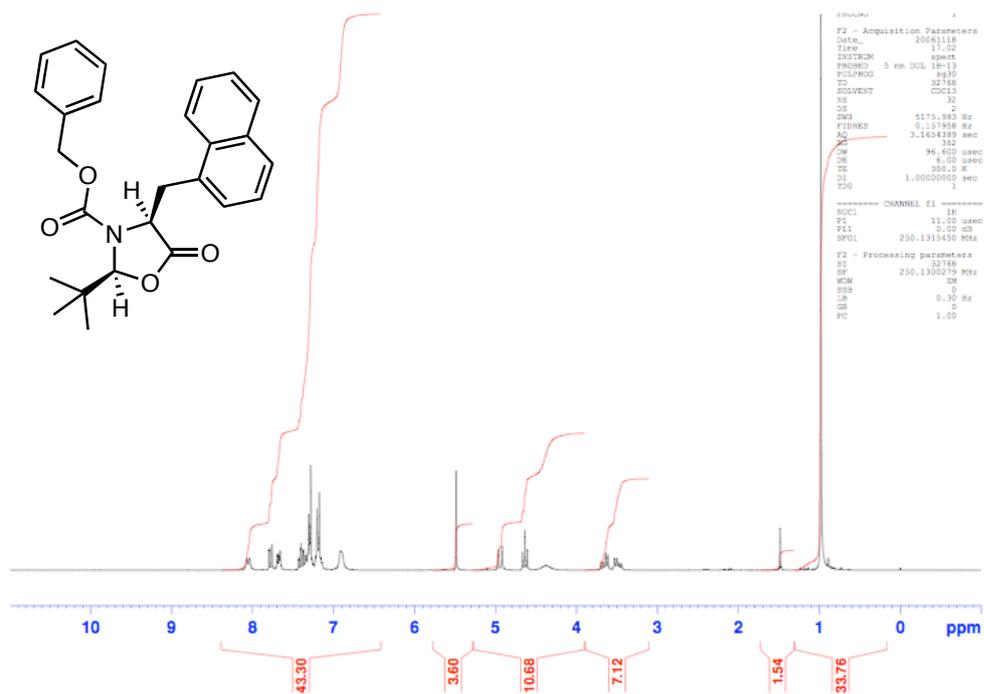
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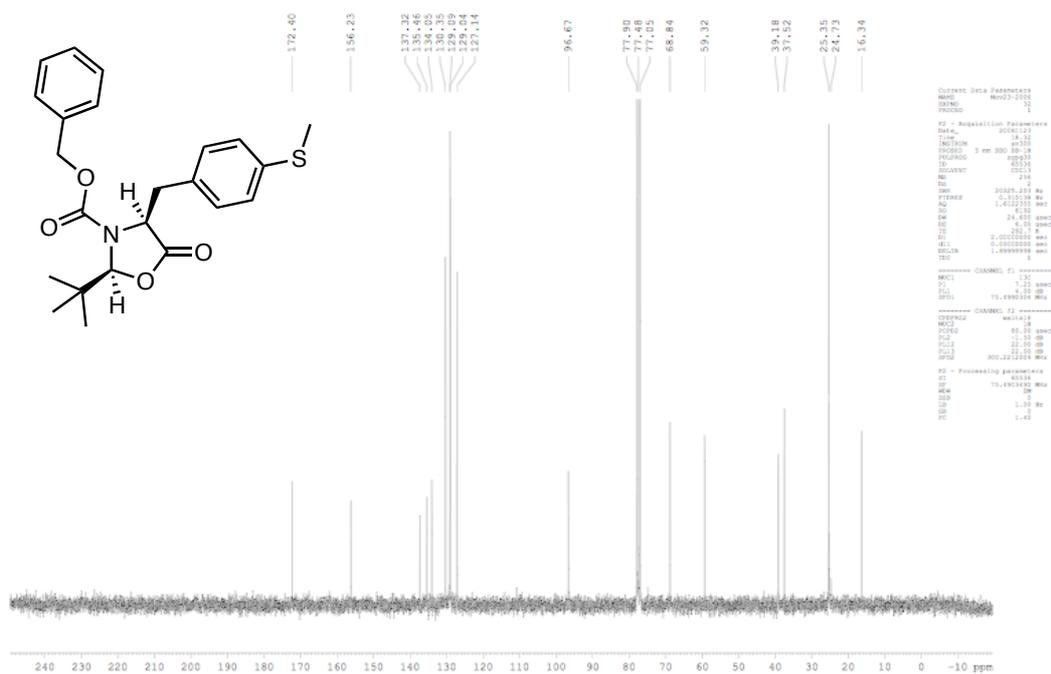
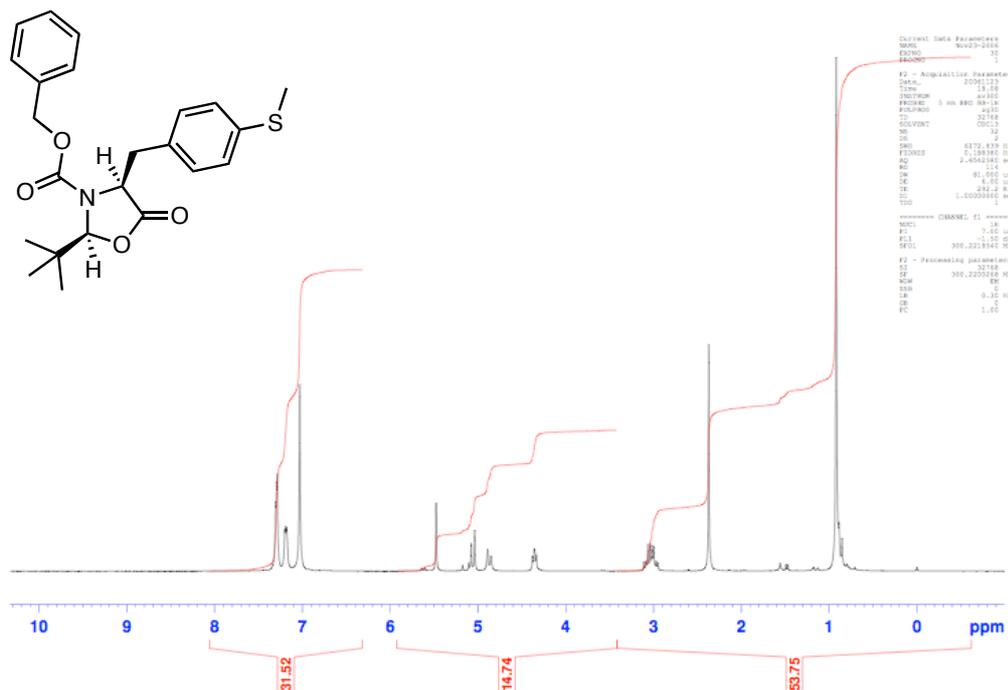
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PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 2048
DS: 4
SWH: 6172.819 Hz
FIDRES: 0.189360 Hz
AQ: 2.656260 sec
RG: 68
AQ2: 8.000000 sec
SFO: 500.136000 MHz
DE: 0.100000 mm
TE: 298.2 K
SI: 1.0000000000000000
SFO2: 1.0000000000000000
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NUC1: 13C
P1: 1.30 sec
PL1: -1.50 dB
SFO1: 101.6261250 MHz
===== CHANNEL f2 =====
NUC2: 13C
P2: 1.30 sec
PL2: -1.50 dB
SFO2: 101.6261250 MHz
F2 - Processing parameters
SI: 32768
SF: 300.2200000 MHz
WDW: EM
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LB: 0.30 Hz
GB: 0
PC: 1.00
    
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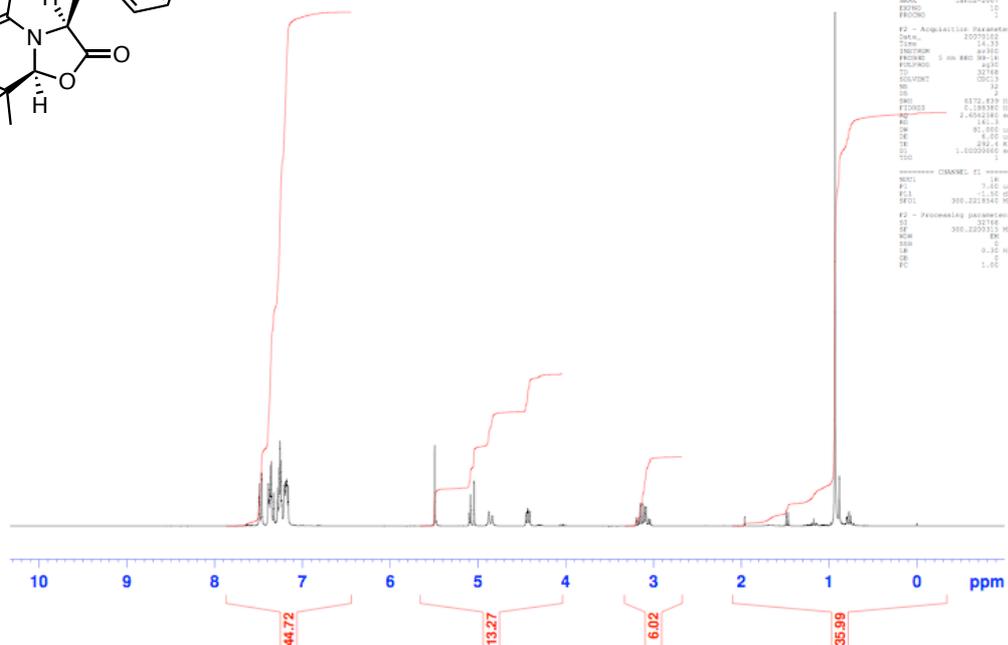
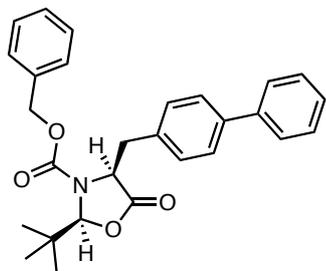


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Current Data Parameters
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EXPNO: 1
PROCNO: 1
F2 - Acquisition Parameters
Date_: 20070219
Time: 11:30
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PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 2048
DS: 4
SWH: 20261.253 Hz
FIDRES: 0.12018 Hz
AQ: 1.6122100 sec
RG: 128
AQ2: 8.000000 sec
SFO: 500.136000 MHz
DE: 0.100000 mm
TE: 298.2 K
SI: 1.0000000000000000
SFO2: 1.0000000000000000
===== CHANNEL f1 =====
NUC1: 13C
P1: 1.30 sec
PL1: 1.50 dB
SFO1: 101.6261250 MHz
===== CHANNEL f2 =====
NUC2: 13C
P2: 1.30 sec
PL2: 1.50 dB
SFO2: 101.6261250 MHz
F2 - Processing parameters
SI: 32768
SF: 101.6261250 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00
    
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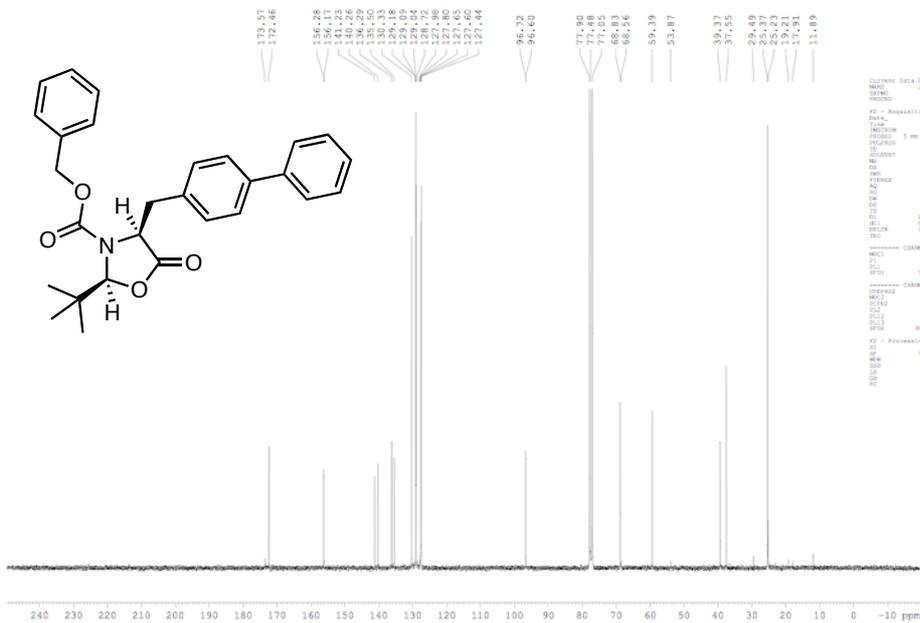






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PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 512
DS: 4
SWH: 6172.8190 Hz
FIDRES: 0.189340 Hz
AQ: 1.4541810 sec
RG: 141.1
IN: 81.000 umax
DE: 6.00 umax
TE: 292.2 K
SI: 1.0000000 sec
SFO: 300.1350600 MHz
===== CHANNEL f1 =====
NUC1: 13C
P1: 1.50000000
PC1: -1.50000000
SFO1: 300.2200000 MHz
F2 - Processing parameters
SI: 32768
SF: 300.2200000 MHz
WDW: EM
SSB: 0
GB: 0.30 Hz
PC: 1.00
    
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Current Data Parameters
Name: 0402-0401
EXPNO: 2
PROCNO: 1
F2 - Acquisition Parameters
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PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 512
DS: 4
SWH: 20165.205 Hz
FIDRES: 0.21210 Hz
AQ: 1.4522310 sec
RG: 141.1
IN: 81.000 umax
DE: 6.00 umax
TE: 292.2 K
SI: 1.0000000 sec
SFO: 300.2200000 MHz
===== CHANNEL f1 =====
NUC1: 13C
P1: 1.50000000
PC1: -1.50000000
SFO1: 300.2200000 MHz
F2 - Processing parameters
SI: 32768
SF: 300.2200000 MHz
WDW: EM
SSB: 0
GB: 0.30 Hz
PC: 1.00
    
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