8c
4-Benzyloxy-3-oxobutanoic acid

10b

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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4-Benzyloxy-3-oxobutanoic acid

**NMR Data**

**PMR**
- **Channel 1**
  - Comp: 150 ppm
  - **SF1**: 8.60 dB
  - **EPD1**: 103.4083033 MHz
- **Channel 2**
  - Comp: 4162 ppm
  - **SF2**: 89.59 ppm
  - **SF3**: 12.80 dB
  - **EPD2**: 403.3016001 MHz
  - **SF4**: 37.60 dB
  - **EPD3**: 103.605113 MHz

**Mass**
- **Mn**: 283.25884683

**Reference**
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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4-(tert-butoxy)-3-oxobutanoic acid

\[
\text{t-BuO} \quad \text{O} \quad \text{c} \quad \text{H}
\]

10c

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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4-((tert-butoxy)-3-oxobutanoic acid
4-(benzyloxy)-2-methyl-3-oxobutanoic acid

10e

NMR@CHEM.OX
4-(benzyloxy)-2-methyl-3-oxobutanoic acid

**10e**

Instrument: DQX400
Chemist: ykschau
Group: mgm
SC:115 crude
c13aqm, CDC13 [C13-NMR] mgmgrp 46

---

NMR@CHEM.CX

NAME: May03-2010-18
SPINOS: 2
PREDCON: 1
DATE: 20140539
TIME: 12:11
DEXTUM: xuv40
PROBES: 5 Nq ONP 1W/1.3
PHASE: 000000
TE: 127.16
SOLVENT: CDC13
NS: 236
DS: 4
TH: 26171.010 Hz
P1: 0.714440 As
NQ: 0.675130 sec
NS: 127.16
CH: 19.120 ussec
DC: 7.30 ussec
T: 100.0 K
D1: 1.00010040 sec
D11: 0.60010030 sec
T90: 1

******* CHANNEL f1 *******
NHC1: 1C
P1: 9.50 ussec
P11: 0.40 As
SF01: 101.644031 MHz

******* CHANNEL f2 *******
CPDPQ2: wait,16
NHC2: 1M
CPD2: 4.00 ussec
P21: 0.30 As
P212: 29.30 As
P213: 15.30 As
SFQ2: 401.201603 MHz
D1: 207.19
DR: 101.631371 MHz
DOR: 0
SR: 1.40 Hz
JR: 0
PC: 1.40
4-(tert-butoxy)-2-methyl-3-oxobutanoic acid

Instrument DPX200
Chemist ykschau
Group mgm
SC112 crude
h@aco.uk CDCl3 (C:\NMR) mgngp 20

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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4-\((\text{tert-butoxy})\)-2-methyl-3-oxobutanoic acid

\[
\text{t-BuO} \quad 4 \\
\text{C} \quad 3 \\
\text{O} \quad 2 \\
\text{OH} \quad 1
\]

10f

Instrument DPX200
Chemist ykschu:
Group ngm
5C112 crude
cl3aco512.au CDC13 (C\:\NMR) nomarep 20

\begin{verbatim}
Current Data Parameters
NAME     May05-2010-26
DECAY    4
POPCNO   1

F2 - Acquisition Parameters
FREQ   2010525
INSTR   DPX200
FRES    5mm Dual 13C/\nSOLVENT CDCl3
SC      512
ES      12077.28 Hz
FIDRES  0.368578 Hz
AQ      1.356652 sec
BG      1224
DM      41.4036 ussec
EE      30.95 usec
TE      400.5 kHz
E1      1.00300035 sec
E11     0.03200035 sec
DELTA   0.89999999 sec
TD1     |

====== CHANNEL f1 ======
MAX1   13C
F1     7.38 usec
F1L1   0.36 db
STEP1  50.3277413 MHz

====== CHANNEL f2 ======
CP0852  70.3616 MHz
RINC2   18
FID02   110.00 ussec
FLS     -3.06 db
FL12    25.06 db
FL13    25.06 db
STEP2   200.13860035 MHz

F2 - Processing parameters
ST      10768
IT      50.3270936 MHz
DOM     18
SNB     8
LB      2.00 Hz
BC      8
PC      1.40
\end{verbatim}

NMR@CHEM.ox
Benzyl 4-(benzyloxy)-3-oxobutanoate
Benzyl 4-(benzyloxy)-3-oxobutanoate

Instrument DQX400
Chemical SC
Group MGM
SC194
e13aq.au CDCl3 (C:NMR) mgmgrp 26
Ethyl 4-(\textit{tert}-butoxy)-3-oxobutanoate

$$\text{HOOC} - \text{CH} - \text{CH}_2 - \text{CH}_2 - \text{CH}_2 - \text{OEt}$$

11b

NMR spectrum of Ethyl 4-(\textit{tert}-butoxy)-3-oxobutanoate
Ethyl 4-(tert-butoxy)-3-oxobutanoate

![Chemical Structure](image)

**11b**

Instrument: DQX400
Chemical yield: 63%
Group name: SC655
distilled

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Benzyl 4-(benzyloxy)-2-methyl-3-oxobutanoate

\[
\begin{align*}
\text{Ph} & \quad \text{O} \\
\text{O} & \quad \text{C} \quad \text{Ph} \\
\text{Me} & \quad \text{O} \\
\text{O} & \quad \text{C}
\end{align*}
\]

Current Data Parameters
- **NAN**: May 03 2016 13
- **FRODO**: 1

**F2 - Acquisition Parameters**
- **Date**: 20161003
- **Time**: 13:56
- **INSTRUM**: DPX200
- **FREQ**: 5 mm Dual 125/
- **FID**: 256
- **SOLVENT**: CDCl3
- **RG**: 15
- **SN**: 1795.42 Hz
- **FIDRES**: 0.176680 Hz
- **AQ**: 0.294560 saq
- **BG**: 456.1
- **SW**: 178.800 USec
- **DR**: 6.00 USec
- **TD**: 816.0 K
- **DI**: 1.10036000 Asec

**--------- CHANNEL f1 --------**
- **NEXC**: 1
- **FL**: 1.00 wsec
- **FF**: -1.00 US
- **SPDW**: 200.131000 MHz

**F2 - Processing Parameters**
- **S1**: 32768
- **SP**: 200.1310125 MHz
- **GRW**: 0
- **D1**: 630 Hz
- **GB**: 0
- **FC**: 1.05
Benzyl 4-(benzyloxy)-2-methyl-3-oxobutanoate

11c

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Ethyl 4-(tert-butoxy)-2-methyl-3-oxobutanoate

**Chemist:** ykochau
**Group:** mpm
**SC 181 fff**

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Ethyl 4-(tert-butoxy)-2-methyl-3-oxobutanoate

Instrument DOX 300
Chemist ykschau
Group mgm
SC-101 H
c13accxau CDCI3 (D2NMR) mgmgrp 3

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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(2R, 5S)-methyl 1’-(4’-(benzyloxy)-3’-oxobutanoyl)-2-(tert-butyl)-5-carboxylate oxazolidine

$\text{Ph} \overset{\text{O}}{\text{O}} \overset{\text{O}}{\text{N}} \overset{\text{CO}_3\text{Me}}{\text{O}} \overset{\text{tBu}}{\text{O}}$

13b
(2R, 5S)-methyl 1’-(4’-(benzyloxy)-3’-oxobutanoyl)-2-(tert-butyl)-5-carboxylate oxazolidine

**NMRCHEM.OX**

**NAME** Nmr22-2G10-40
**SOLVENT** CDCl3
**CAS** 13acq.au
**S/N** 40

**Spectrum:**

- **Chemical Shifts:**
  - 220 ppm: 13c
  - 9.16 ppm: 13c
  - 0.90 ppm: 13c

- **Resonance Frequencies:**
  - 103.600593 MHz
  - 403.221609 MHz

**Parameters:**
- **Field Strength:** 10.023 MHz
- **Temperature:** 298 K
- **Sensitivity:** 1.40
(2R, 5S)-methyl-1’-(4’-(tert-butoxy)-3’-oxobutanoyl)-2-(tert-butyl)-5-carboxylate oxazolidine

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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(2R, 5S)-methyl-1’-(4’-(tert-butoxy)-3’-oxobutanoyl)-2-(tert-butyl)-5-carboxylate oxazolidine

13c

Instrument DQX400
Chemical shift
Group ppm
SC 99I column
c13acqau GC13 (D2/NMR) mgmgrp 11

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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14a/15a
Instrument DQX400
Chemist PA
Group MGM
PA176B
h1acq.au CDCl3 [E\NMR2011] mgmgp 11

14b

1.73
1.80
2.11
2.69
3.08
6.41
31.08
(2R, 5R, 6S)-1-Aza-2-(tert-butyl)-6-hydroxy-6-(tert-butoxymethyl)-5-methoxycarbonyl-3-oxabicyclo[3.3.0]octane

**14c**

Instrument: DQX400
Chemical shift: ppm
Group: ppm
Detector: ppm
Nucleus: ppm

**Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry**
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(2R, 5R, 6R)-1-Aza-2-(tert-butyl)-6-hydroxy-6-(tert-butoxymetyl)-5-methoxycarbonyl-3-oxabicyclo[3.3.0]octane
(2R, 5R, 6R)-1-Aza-2-(tert-butyl)-6-hydroxy-6-(tert-butoxymethyl)-5-methoxycarbonyl-3-oxabicyclo[3.3.0]octane

15c

Instrument: DQX460
Chemist: ykschu
Group: mgm
SC 0H (1) columned
$^{13}$C{H} (50 MHz, 200 Hz)

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Methyl 2-hydroxymethyl-3-hydroxy-3-(benzyloxymethyl) pyroglutamate and 2-amino-2-(methoxycarbonyl)-3-hydroxy-3-(benzyloxymethyl)-δ-lactone
Methyl 2-hydroxymethyl-3-hydroxy-3-(benzyloxymethyl) pyroglutamate and 2-amino-2-(methoxycarbonyl)-3-hydroxy-3-(benzyloxymethyl)-δ-lactone

17b

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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1-Aza-2-(hydroxymethyl)-3-oxo-4-oxa-6-hydroxy-8-oxo[3.3.0]octane
1-Aza-2-(hydroxymethyl)-3-oxo-4-oxa-6-hydroxy-8-oxo[3.3.0]octane
(2R, 5S)-methyl-1-(4’-(benzyloxy)-2’-methyl-3’-oxobutanoyl)-2-(tert-butyl)-5-carboxylate oxazolidine

**Instrument** DQX-400
**Chemical ysheau**
**Group** mgm
**SC117**

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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(2R, 5S)-methyl-1-(4'-(benzyloxy)-2'-methyl-3'-oxobutanoyl)-2-(tert-butyl)-5-carboxylate oxazolidine

**20b**

**Instrument** DQX400

**Chemist** yscbou

**Group** mpm

**SC117/3**

c13@acq.acn CDC13 (C 1H) mgmgrp 3
(2R, 5S)-methyl 1-(4’-(tert-butoxy)-2’-methyl-3’-oxobutanoyl)-2-(tert-butyl)-5-carboxylate oxazolidine
(2R, 5S)-methyl 1-(4’-(tert-butoxy)-2’-methyl-3’-oxobutanoyl)-2-(tert-butyl)-5-carboxylate oxazolidine

20c

Instrument DQX400
Chemical yield: 80%
Group ppm
3C119f
c36cc.eu CDC13 (C, NMR) mg3kgp 1
21a

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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(2R, 5R, 6R, 7S)-1-Aza-2-(tert-butyl)-6-hydroxy-7-methyl-6-(benzyloxymethyl)-5-methoxycarbonyl-3-oxabicyclo[3.3.0]octane
(2R, 5R, 6R, 7S)-1-Aza-2-(tert-butyl)-6-hydroxy-7-methyl-6-(benzyloxymethyl)-5-methoxycarbonyl-3-oxabicyclo[3.3.0]octane

21c
(2R, 5R, 6S, 7R)-1-Aza-2-(tert-butyl)-6-hydroxy-7-methyl-6-(benzyloxymethyl)-5-methoxycarbonyl-3-oxabicyclo[3.3.0]octane
(2R, 5R, 6S, 7R)-1-Aza-2-(tert-butyl)-6-hydroxy-7-methyl-6-(benzyloxymethyl)-5-methoxycarbonyl-3-oxabicyclo[3.3.0]octane
(2R, 5R, 6S, 7R)-1-Aza-2-(tert-butyl)-6-hydroxy-7-methyl-6-(benzyloxymethyl)-5-methoxycarbonyl-3-oxabicyclo[3.3.0]octane

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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(2R, 5R, 6S, 7R)-1-Aza-2-(tert-butyl)-6-hydroxy-7-methyl-6-(benzyloxymethyl)-5-methoxycarbonyl-3-oxabicyclo[3.3.0]octane

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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(2R, 5R, 6S, 7S)-1-Aza-2-(tert-butyl)-6-hydroxy-7-methyl-6-(benzyloxymethyl)-5-methoxycarbonyl-3-oxabicyclo[3.3.0]octane
(2R, 5R, 6S, 7S)-1-Aza-2-(tert-butyl)-6-hydroxy-7-methyl-6-(benzyloxymethyl)-5-methoxycarbonyl-3-oxabicyclo[3.3.0]octane
Instrument DQX400
Chemist PA
Group MGM
PA164B
c13acq.au CDCls (Et2NMR2011) mgmgrp 57

25b