Supporting Information for Publication

Synthesis of 2,3,5,6-Tetrasubstituted Tetrahydropyrans via (3,5)-Oxonium-Ene Reaction

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1. General preparation of the starting materials B1-3 and 1a-d and their spectral data S2-S7
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S-1
**General Information:** All reagents are commercially obtained. BF$_3$Et$_2$O was distilled over CaH$_2$ prior to use. $^1$H NMR spectra were recorded in CDCl$_3$ on 400 MHz NMR spectrometer using TMS as internal standard. The $^{13}$C and $^{19}$F NMR spectra were recorded at 100 MHz and 376 MHz, respectively. For $^{13}$C and $^{19}$F NMR CDCl$_3$ and C$_6$F$_6$ were used as internal standard. IR spectra were recorded on FT-IR spectrometer. Melting points were measured in open capillary tubes and are uncorrected.

**Preparation of ethyl 2-(1-hydroxyalkyl/hydroxy(phenyl)methyl)-5-methylhex-4-enoate 1:** The ethyl 2-(1-hydroxyalkyl/hydroxy(phenyl)methyl)-5-methylhex-4-enoate 1 was synthesized starting from β-keto ester A and 1-bromo-3-methylbut-2-ene as shown in Scheme 1.$^1$ Thus the reaction of β-keto ester A with 1-bromo-3-methylbut-2-ene in presence of sodium hydride in THF afforded α-substituted β-keto ester B, which after reduction with sodium borohydride in methanol gives alcohol 1 in 78-84% yields.

The methyl and ethyl substituted β-keto esters B-1 and B-2 afforded two inseparable diastereomers$^2$ 1a,b whereas phenyl substituted β-ketoester B-3 gave two separable anti- and syn-diastereomers 1c and 1d, respectively. The structures of all compounds are determined.

![Scheme 1: Synthesis of ethyl 2-(1-hydroxyalkyl/hydroxy(phenyl)methyl)-5-methylhex-4-enoate](image-url)
from IR, $^1$H, $^{13}$C NMR and mass spectroscopy. The stereochemistry of compounds 1c and 1d are determined from coupling constants values.$^{2b,3}$

References:


**General procedure for the synthesis of α-alkyl-β-keto esters (B-1-3):** To a suspension of sodium hydride (15.69 mmol, 1 equiv.) in THF (15 mL) at 0 °C was added β-keto ester (15.69 mmol, 1 equiv.) dropwise via syringe. After 20 min, a solution of 3,3-dimethylallyl bromide (17.26 mmol, 1.1 equiv.) in THF (5 mL) was added and the mixture was left at room temperature overnight. The solvent was removed under reduced pressure, and the residue was dissolved in Et$_2$O (10 mL) and washed with brine (25 mL). The organic layer was dried and filtered, and the filtrate was concentrated under reduced pressure. The residue was purified by column chromatography over silica gel to afford the title compounds as colourless oil.

**Synthesis of Ethyl 2-acetyl-5-methylhex-4-enoate (B-1):** To a suspension of sodium hydride (0.378 g, 15.69 mmol) in THF (15 mL) at 0 °C was added ethyl acetoacetate (2 mL, 15.69 mmol) dropwise via syringe. After 20 min, a solution of 3,3-dimethylallyl bromide (2 mL, 17.26 mmol) in THF (5 mL) was added and the mixture was left at room temperature overnight. The solvent was removed under reduced pressure, and the residue was dissolved in Et$_2$O (10 mL) and washed with brine (25 mL). The organic layer was dried and filtered, and
chromatography over silica gel (10% EtOAc/hexane) to afford ethyl 2-acetyl-5-methylhex-4-enoate B-1 (2.97 g, 95%) as a colourless oil; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 1.27 (t, $J = 7.2$ Hz, 3 H), 1.63 (s, 3 H), 1.68 (s, 3 H), 2.22 (s, 3 H), 2.54 (t, $J = 7.2$ Hz, 2 H), 3.43 (t, $J = 7.6$ Hz, 1 H), 4.19 (q, $J = 7.2$ Hz, 2 H), 5.03 (dt, $J = 7.6$ and 1.6 Hz, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 14.1, 17.8, 25.8, 27.0, 29.1, 59.8, 61.3, 119.9, 134.7, 169.7, 203.2; IR (KBr, Neat): 2981, 2930, 1739, 1718, 1205, 1150 cm$^{-1}$. HRMS (APCI) cald. for C$_{11}$H$_{18}$O$_3$ (M+H)$^+$ requires 199.1334; found 199.1337. APCI-MS: m/z (relative intensity): 199.2 ((M+H)$^+$, 43%), 181.1 (100), 169.1 (55), 143.1 (17), 124.1 (43), 107.1 (73).

**Ethyl 5-methyl-2-propionylhex-4-enoate (B-2):** Colourless oil (3.13 g, 94%); $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 1.06 (t, $J = 7.2$ Hz, 3 H), 1.26 (t, $J = 7.2$, 3 H), 1.62 (s, 3 H), 1.67 (s, 3 H), 2.47-2.60 (m, 4 H), 3.45 (t, $J = 7.2$ Hz, 1 H), 4.17 (q, $J = 7.2$ Hz, 2 H), 5.02 (dt, $J =7.6$ and 1.6 Hz, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 7.8, 14.2, 17.9, 25.9, 27.2, 35.6, 58.9, 61.4, 120.1, 134.8, 169.9, 206.0; IR (KBr, Neat): 2980, 2937, 1741, 1716, 1198, 1156 cm$^{-1}$. HRMS (APCI) cald. for C$_{12}$H$_{20}$O$_3$ (M$^+$) requires 212.1412; found 212.1408. APCI-MS: m/z (relative intensity): 212.2 ((M+H)$^+$, 100%), 194.7 (65), 168.7 (27), 123.8 (22).

**Ethyl 2-benzoyl-5-methylhex-4-enoate (B-3):** Colourless oil (3.88 g, 95%); $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 1.67 (t, $J = 7.2$ Hz, 3 H), 1.62 (s, 3 H), 1.65 (s, 3 H), 2.61-2.78 (m, 2 H), 4.14 (q, $J = 7.2$ Hz, 2 H), 4.30 (t, $J = 7.2$ Hz, 1 H), 5.11 (dt, $J = 7.2$ and 1.6 Hz, 1 H), 7.44-7.50 (m, 2 H), 7.55-7.61 (m, 1 H), 7.97-8.00 (m, 2 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 14.1, 17.9, 25.8, 27.8, 54.6, 61.4, 120.3, 128.7, 128.8, 133.5, 134.7, 136.4, 169.9, 195.2; IR (KBr, Neat): 2979, 2929, 1737, 1688, 1448, 1379, 1241, 1153 cm$^{-1}$. HRMS (APCI) cald. for C$_{16}$H$_{20}$O$_3$ (M+H)$^+$ requires 261.1490; found 261.1494. APCI-MS: m/z (relative intensity): 261.2 ((M+H)$^+$, 57%), 215.1 (21), 193.1 (23), 169.1 (12), 124.1 (100).
General procedure for the synthesis of Ethyl 2-(1-hydroxyalkyl/hydroxy(phenyl)methyl)-5-methylhex-4-enoate (1a-d): To a solution of α-alkyl-β-keto esters (14.98 mmol, 1 equiv.) in dry MeOH (15 mL) at 0 °C, was added sodium borohydride (39.25 mmol, 2.62 equiv.) in small portions. The reaction mixture was stirred in between 0 °C to 5 °C for 1.5 h. The progress of the reaction was monitored by TLC with ethyl acetate and hexane as eluents. After completion of the reaction, the product was extracted with ethyl acetate (30 mL) and then washed with water (15 mL) and brine (15 mL). The organic layer was dried (Na$_2$SO$_4$) and evaporated to leave the crude product, which was purified by column chromatography over silica gel to give the title compounds.

Synthesis of Ethyl 2-(1-hydroxyethyl)-5-methylhex-4-enoate (1a): To a solution of ethyl 2-acetyl-5-methylhex-4-enoate (2.97 g, 14.98 mmol) in dry MeOH (15mL) at 0 °C, was added sodium borohydride (1.485 g, 39.25 mmol) in small portions. The reaction mixture was stirred in between 0 °C to 5 °C for 1.5 h. The progress of the reaction was monitored by TLC with ethyl acetate and hexane (3:22) as eluents. After completion of the reaction, the product was extracted with ethyl acetate (30 mL) and then washed with water (15 mL) and brine (15 mL). The organic layer was dried (Na$_2$SO$_4$) and evaporated to leave the crude products, which were purified by column chromatography over silica gel to give an inseparable mixture of two diastereomers 1a (2.40 g, 80% overall yield) as a colourless oil; $^1$H NMR (400 MHz, CDCl$_3$): δ 1.19-1.29 (m, 6 H), 1.62 (s, 3 H), 1.69 (s, 3 H), 2.29-2.47 (m, 3 H), 2.51 (brs 0.5 H), 2.69 (brs, 0.5 H), 3.86-3.96 (m, 0.5 H), 3.98-4.06 (m, 0.5 H), 4.08-4.24 (m, 2 H), 5.04-5.14 (m, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 14.1(2C), 17.5(2C), 20.5, 21.1, 25.6(2C), 26.7, 27.7, 53.0, 53.1, 60.2, 60.3, 67.8, 67.9, 120.5, 121.1, 133.3, 133.6, 174.7, 175.0; IR (KBr, Neat): 3441, 2972, 2929, 1732, 1640, 1182, 1155 cm$^{-1}$. HRMS (APCI) calcd. for C$_{11}$H$_{20}$O$_3$ (M+H)$^+$ requires 201.1490; found 201.1498. APCI-MS: m/z (relative intensity):
201.2 ((M+H)+, 30%), 137.1 (4), 125.1 (11), 124.1 (100), 123.1 (14), 82.0 (24).

**Ethyl 2-(1-hydroxypropyl)-5-methylhex-4-enoate (1b):** Colourless oil (2.50 g, 78%); \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 0.98 (t, \(J = 7.6\) Hz, 3 H), 1.25 (t, \(J = 7.2\) Hz, 1.5 H), 1.26 (t, \(J = 7.2\) Hz, 1.5 H), 1.45-1.54 (m, 2H), 1.61 (s, 1.5 H), 1.62 (s, 1.5 H), 1.69 (s, 3 H), 2.28-2.50 (m, 3 H), 2.67 (brs, 1 H), 3.56-3.64 (m, 0.5 H), 3.70-3.78 (m,0.5 H), 4.08-4.22 (m, 2 H), 5.04-5.16 (m, 1 H); \(^13\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) 10.2, 10.3, 14.3(2C), 17.8(2C), 25.8, 26.1, 27.4(2C), 28.3, 28.6, 50.7, 51.2, 60.5(2C), 73.3, 73.4, 120.7, 121.3, 133.6, 134.1, 175.3, 175.5; IR (KBr, Neat): 3456, 2968, 2932, 1731, 1642 , 1183, 1159 cm\(^{-1}\). HRMS (APCI) calcd. for C\(_{12}\)H\(_{22}\)O\(_3\) (M+H)+ requires 215.1647; found 215.1653. APCI-MS: m/z (relative intensity): 215.2 ((M+H)+, 100%), 197.2 (6), 169.1 (8), 151.1 (7), 124.1 (35), 123.1 (14), 82.0 (7).

**Synthesis of anti/syn-Ethyl 2-(hydroxy(phenyl)methyl)-5-methylhex-4-enoate (1c & 1d):**
To a solution of ethyl 2-benzoyl-5-methylhex-4-enoate (3.90 g, 14.98 mmol) in dry MeOH (15 mL) at 0 \(^\circ\)C, was added sodium borohydride (1.485 g, 39.25 mmol) in small portions. The reaction mixture was stirred in between 0 \(^\circ\)C to 5 \(^\circ\)C for 1.5 h. The progress of the reaction was monitored by TLC with ethyl acetate and hexane (7:43) as eluents. After completion of the reaction, the product was extracted with ethyl acetate (30 mL) and then washed with water (15 mL) and brine (15 mL). The organic layer was dried (Na\(_2\)SO\(_4\)) and evaporated to leave the crude products, which were separated by column chromatography over silica gel to give 1c (1.73 g, 44%) and 1d (1.58 g, 40%) as a colourless oil.

**anti-Ethyl 2-(hydroxy(phenyl)methyl)-5-methylhex-4-enoate (1c):** Colourless oil (1.73 g, 44%); \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 1.18 (t, \(J = 7.2\) Hz, 3 H), 1.50 (s, 3 H), 1.65 (s, 3 H), 2.05 (ddd, \(J = 13.2, 7.6\) and 5.6 Hz, 1 H), 2.25 (ddd, \(J = 14.8, 8.0\) and 6.8 Hz, 1 H), 2.75 (ddd, \(J = 12.8, 8.8, 5.2\) Hz, 1 H), 3.16 (d, \(J = 5.2\) Hz, 1H), 4.11 (q, \(J = 7.2\) Hz, 2 H), 4.80 (dd,
$J = 6.4$ and $4.4$ Hz, $1H$), $5.03$ (dt, $J = 6.8$ and $1.2$ Hz, $1H$), $7.26-7.37$ (m, $5H$); $^{13}C$ NMR (100 MHz, CDCl$_3$): $\delta$ 14.3, 17.8, 25.9, 28.4, 53.3, 60.7, 74.8, 120.2, 126.6, 128.0, 128.6, 134.3, 142.2, 175.2; IR (KBr, Neat): 3461, 2978, 2929, 1729, 1452, 1377, 1180, 1037, 766, 702 cm$^{-1}$. HRMS (APCI) cald. for C$_{16}$H$_{22}$O$_3$ (M$^+$) requires 262.1569; found 262.1573. APCI-MS: m/z (relative intensity): 262.2 (M$^+$, 3%), 244.6 (15), 177.7 (15), 176.7 (100), 123.8 (22).

**syn-Ethyl 2-(hydroxy(phenyl)methyl)-5-methylhex-4-enoate (1d):** Colourless oil (1.58 g, 40%); $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 1.11 (t, $J = 7.2$ Hz, $3H$), 1.54 (s, $3H$), 1.64 (s, $3H$), 2.29 (ddd, $J = 14.4$, 8.8 and 5.8 Hz, $1H$), 2.46 (ddd, $J = 14.4$, 8.8 and 5.6 Hz, $1H$), 2.72 (ddd, $J = 10.4$, 5.6 and 4.4 Hz, $1H$), 3.03 (brs, $1H$), 4.02 (q, $J = 7.2$ Hz, $2H$), 4.95 (d, $J = 5.6$ Hz, $1H$), 5.04 (t, $J = 6.8$ Hz, $1H$), 7.24-7.39 (m, $5H$); $^{13}C$ NMR (100 MHz, CDCl$_3$): $\delta$ 14.2, 17.8, 25.9, 26.1, 53.3, 60.7, 74.2, 121.1, 126.4, 127.8, 128.4, 134.0, 141.7, 175.0; IR (KBr, Neat): 3460, 2979, 2930, 1728, 1453, 1375, 1180, 1026, 767, 701 cm$^{-1}$. HRMS (APCI) cald. for C$_{16}$H$_{22}$O$_3$ (M+H)$^+$ requires 263.1647; found 263.1652. APCI-MS: m/z (relative intensity): 263.2 ((M+H)$^+$, 19%), 192.1 (5), 178.1 (15), 177.1 (100), 171.1 (21), 131.1 (13), 124.1 (75).
$^1\text{H} \text{NMR spectra of B-1}$

![NMR Spectra](image)

S-8
$^{13}$C NMR spectra of B-1

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\(^1\)H NMR spectra of B-2
$^{13}$C NMR spectra of B-2
$^1$H NMR spectra of B-3
$^{13}$C NMR spectra of B-3

![NMR Spectra Image]

S-13
$^1$H NMR spectra of 1a
\(^{13}\)C NMR spectra of 1a
$^1$H NMR spectra of 1b

H$_3$C – OH

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CO$_2$Et

S-16
$^{13}$C NMR spectra of 1b

![NMR Spectra of 1b](image)

S-17
$^1$H NMR spectra of 1c

![NMR Spectrum Image]

S-18
$^{13}\text{C}$ NMR spectra of 1c
$^1$H NMR spectra of 1d
$^{13}$C NMR spectra of 1d
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$^{13}$C NMR spectra of 3a
$^1$H NMR spectra of 3b
$^{13}$C NMR spectra of 3b

**Sample Information**
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- Temp: Not used
- Solvent: CDCl3
- Spin: Not used
- Acquisition: alfa 20.000
- Acquisition flags: 25125.6

**Chemical Shifts (ppm)**
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$^1$H NMR spectra of 3c
$\text{C NMR spectra of 3c}$

![C NMR spectrum of 3c](image)

S-27
$^{1}$H NMR spectra of 3d

S-28
$^{13}$C NMR spectra of 3d

![C NMR spectra of 3d](image-url)
\(^1\)H NMR spectra of 3e
$^{13}$C NMR spectra of 3e

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S-31
$^{19}$F NMR spectra of 3e
$^1$H NMR spectra of 3f
$^{13}$C NMR spectra of 3f

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$^1$H NMR spectra of 3g
$^{13}$C NMR spectra of 3g
$^1$H NMR spectra of 3h

![NMR Spectrum Image]

S-37
$^{13}$C NMR spectra of 3h
$^1$H NMR spectra of 3i
$^{13}$C NMR spectra of 3i
$^1$H NMR spectra of 3j
$^{13}$C NMR spectra of 3j

![C NMR spectra of 3j](image)
$^3$H NMR spectra of 3k
$^{13}$C NMR spectra of 3k

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$^1$H NMR spectra of 3l

![H NMR spectra of 3l](image-url)
$^{13}$C NMR spectra of 3l

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$^1$H NMR spectra of 3m

![NMR Spectra Image]
$^{13}$C NMR spectra of 3m

![C NMR spectra of 3m](image-url)
$^1$H NMR spectra of 3n
$^{13}$C NMR spectra of 3n
$^1$H NMR spectra of 3o
$^{13}\text{C}$ NMR spectra of 3o
$^1$H NMR spectra of 3p

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$^{13}$C NMR spectra of 3p
$^1$H NMR spectra of 4a
$^{13}$C NMR spectra of 4a
$^1$H NMR spectra of 4b
$^{13}$C NMR spectra of 4b

![NMR Spectrum Image]
$^1$H NMR spectra of 4c
$^{13}$C NMR spectra of 4c
$^1$H NMR spectra of 4d

![H NMR spectrum of 4d](image)

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$^{13}$C NMR spectra of 4d
$^1$H NMR spectra of 4e

![NMR Spectra Image]

S-63
$^{13}$C NMR spectra of 4e
$^{19}$F NMR spectra of 4e
H NMR spectra of 4f

O₂N

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$^{13}$C NMR spectra of 4f
$^1$H NMR spectra of 4g
$^{13}$C NMR spectra of 4g

![NMR spectrum image]

S-69
$^1$H NMR spectra of 4h
13C NMR spectra of 4h

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$^1$H NMR spectra of 4i
$^{13}$C NMR spectra of 4i
**$^1$H NMR spectra of 4j**

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$^{13}$C NMR spectra of 4j

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$^1$H NMR spectra of 4k
$^{13}$C NMR spectra of 4k

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S-77
\(^1\)H NMR spectra of 41

\[ \text{H}_3C - \text{CH}_3 \]

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\(^{13}\)C NMR spectra of 4l
$^1$H NMR spectra of 4m
$^{13}$C NMR spectra of 4m

![Chemical Structure Image]

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\(^1\)H NMR spectra of 4n

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$^{13}$C NMR spectra of 4n

S-83
$^1$H NMR spectra of 4o
$^{13}$C NMR spectra of 4o
\( ^1\text{H} \text{NMR spectra of 4p} \)
$^{13}$C NMR spectra of 4p
$^1$H NMR spectra of 11a
$^{13}$C NMR spectra of 11a
$^1$H NMR spectra of 11b
$^{13}$C NMR spectra of 11b
$^1$H NMR spectrta of 11c

![NMR Spectra Image]

S-92
$^{13}$C NMR spectra of 11c
$^1$H NMR spectra of 11d

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$^{13}$C NMR spectra of 11d
$^{19}$F NMR spectra of 11d
$^1\text{H}$ NMR spectra of 11e
$^{13}$C NMR spectra of 11e

![NMR spectra graph](image)
$^1$H NMR spectra of 11f

![NMR Spectra Image]
$^{13}$C NMR spectra of 11f

![NMR Spectra](image-url)
$^1$H NMR spectra of 11g

![NMR Spectra Image]
$^{13}$C NMR spectra of 11g

![C NMR spectrum image]
$^1$H NMR spectra of 11h

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$^{13}$C NMR spectra of 11h
$^1$H NMR spectra of 12a

![NMR Spectrum Image]

S-105
$^{13}$C NMR spectra of 12a

![Chemical Structure](image)
$^1$H NMR spectra of 12b
$^{13}$C NMR spectra of 12b
$^1$H NMR spectra of 12c
$^{13}$C NMR spectra of 12c
\(^1\)H NMR spectra of 12d
$^{13}$C NMR spectra of 12d
$^1$H NMR spectra of 12e

![NMR spectrum image]

S-113
$^{13}$C NMR spectra of 12e
$^1$H NMR spectra of 12f

![NMR Spectra](image)

S-115
$^{13}$C NMR spectra of 12f
$^1$H NMR spectra of 13
$^{13}$C NMR spectra of 13
$^1$H NMR spectra of 14
$^{13}$C NMR spectra of 14

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The crystal parameters of compound 4g

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Figure 1: ORTEP diagram of 4g
The crystal parameters of compound 11f

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</tr>
<tr>
<td>Space group</td>
<td>P 21/c</td>
</tr>
<tr>
<td>a/Å</td>
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</tr>
<tr>
<td>b/Å</td>
<td>15.3970(4)</td>
</tr>
<tr>
<td>c/Å</td>
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</tr>
<tr>
<td>α/°</td>
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</tr>
<tr>
<td>β/°</td>
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</tr>
<tr>
<td>γ/°</td>
<td>90.00</td>
</tr>
<tr>
<td>V/Å³</td>
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<td>Z</td>
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<tr>
<td>Abs. Coeff./mm^{-1}</td>
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<td>GOF on F²</td>
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<tr>
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<td>R1 = 0.0615</td>
</tr>
<tr>
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<td>wR2 = 0.1806</td>
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<tr>
<td>R indices [all data]</td>
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<tr>
<td></td>
<td>wR2 = 0.2203</td>
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![Figure 2: ORTEP diagram of 11f](image-url)
The crystal parameters of compound 13

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<th>13-CCDC 819364</th>
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<td>Formula</td>
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<tr>
<td>Formula weight</td>
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</tr>
<tr>
<td>T/K</td>
<td>296(2)</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P 21/n</td>
</tr>
<tr>
<td>a/Å</td>
<td>8.9913(7)</td>
</tr>
<tr>
<td>b/Å</td>
<td>19.6654(14)</td>
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<td>c/Å</td>
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<td>β°</td>
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<td>R indices [all data]</td>
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Figure 3: ORTEP diagram of 13

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# The crystal parameters of compound 14

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<th>Parameter</th>
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<tr>
<td>Formula weight</td>
<td>366.44</td>
</tr>
<tr>
<td>T/K</td>
<td>296(2)</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P 21/c</td>
</tr>
<tr>
<td>a/Å</td>
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</tr>
<tr>
<td>b/Å</td>
<td>8.7884(6)</td>
</tr>
<tr>
<td>c/Å</td>
<td>18.2013(12)</td>
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<tr>
<td>α°</td>
<td>90.00</td>
</tr>
<tr>
<td>β°</td>
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<tr>
<td>γ°</td>
<td>90.00</td>
</tr>
<tr>
<td>V/Å³</td>
<td>1997.6(2)</td>
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<tr>
<td>Z</td>
<td>4</td>
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<td>Multi-scan</td>
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</tr>
<tr>
<td>R indices [all data]</td>
<td>(R_I = 0.0967)</td>
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</table>

![Figure 4: ORTEP diagram of 14](image-url)

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