Reaction of 2,3-Allenates with TsNBr₂ in the Presence of Base: A Facile Highly Stereoselective Synthesis of (1*E*,2*E*)-3-Bromo-4-Oxo-N'-Tosyl-2-Alkenoxyimidic Acid Ethyl Esters

Ruwei Shen^a, Xian Huang^{* a, b}

a. Department of Chemistry, Zhejiang University (Xixi Campus), Hangzhou 310028, P. R. China

b. State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, P. R. China

huangx@mail.hz.zj.cn

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1. Experiment procedures

All reactions were performed under an N₂ atmosphere. Dry solvents were distilled prior to use: THF and toluene were distilled from sodium-benzophenone; MeCN were distilled from P₂O₅; CH₂Cl₂ were distilled from CaH₂. Petroleum ether refers to the fraction with boiling point in the range 60–90 °C. All ¹H NMR and ¹³C NMR spectra were measured in CDCl₃ with TMS as the internal standard. Chemical shifts are expressed in ppm and J values are given in Hz. Melting points are uncorrected. 2,3-allenoates **1** were prepared according to the known method by treatment of the acid chlorides with the corresponding ethyl 2-(triphenylphoranylidene) propionate.¹

General Procedure. Synthesis of (1*E*,2*E*)-3-bromo-4-oxo-N'-tosyl-2-alkenoxyimidic acid ethyl esters **2**

Under an atmosphere of dry nitrogen, 2.0 equiv of K₂CO₃ (1.0 mmol) was added to a solution of TsNBr₂ (0.5 mmol) in 1 mL of dry CH₂Cl₂ at –65 °C (or –80 °C). Then a solution of 2,3-allenoate **1** (0.5 mmol) in 1 mL of CH₂Cl₂ was injected. After being stirred for 15 (or 20) min at –65 °C (or –80 °C), the cooling bath was removed and the reaction mixture was slowly warmed to room temperature and then kept stirring for another 30 min. The mixture was quenched with water and extracted with Et₂O (3 × 10ml). The combined organic layer was washed with saturated brine and dried over anhydrous MgSO₄. After filtration and removal of solvent in vacuo, the residues were purified on flash silica chromatography (petroleum ether - ethyl acetate 6:1 v/v) to afford **2**.

Reference:

1. Lang, R. W.; Hansen, H.-J. *Org. Synth.* **1984**, 62, 202.

2. Characterization data of 2a-2o and 7

(1E,2E)-3-bromo-4-oxo-N'-tosylpent-2-enoxylimidic acid ethyl ester (2a)

oil. ^1H NMR (400 MHz, CDCl_3): 7.80-7.83 (m, 2H), 7.31-7.29 (m, 3H), 4.18 (q, $J = 7.2$ Hz, 2H), 2.42 (s, 3H), 2.39 (s, 3H), 1.24 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 195.0, 163.3, 143.7, 138.2, 135.1, 129.4, 126.9, 123.2, 65.7, 27.4, 21.5, 13.0. IR (neat): 1701, 1631, 1570, 1311, 1159 cm^{-1} . MS: m/z (%) 375 [$\text{M}^+(\text{}^{81}\text{Br})$, 4], 373 [$\text{M}^+(\text{}^{79}\text{Br})$, 4], 294 [$\text{M}^+ - \text{Br}$, 80], 91 (100). Anal. Calcd for $\text{C}_{14}\text{H}_{16}\text{BrNO}_4\text{S}$ (374.25): C 44.93, H 4.31, N 3.74. Found: C 44.89, H 4.45, N 3.68.

(1E,2E)-2-benzyl-3-bromo-4-oxo-N'-tosylpent-2-enoxylimidic acid ethyl ester

(2b)

oil. ^1H NMR (400 MHz, CDCl_3): 7.77-7.75 (m, 2H), 7.28-7.26 (m, 7H), 4.06 (s, 2H), 3.95 (q, $J = 6.8$ Hz, 2H), 2.49 (s, 3H), 2.41 (s, 3H), 0.96 (t, $J = 6.8$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 193.9, 169.6, 143.29, 143.27, 138.5, 134.7, 129.7, 129.3, 128.4, 127.2, 126.9, 123.1, 65.5, 42.6, 29.1, 21.5, 13.1. IR (neat): 1704, 1607 1568 1311 1159 cm^{-1} . MS: m/z (%) 465 [$\text{M}^+(\text{}^{81}\text{Br})$, 7], 463 [$\text{M}^+(\text{}^{79}\text{Br})$, 7], 384 ($\text{M}^+ - \text{Br}$, 9), 91 (100). Anal. Calcd for $\text{C}_{21}\text{H}_{22}\text{BrNO}_4\text{S}$ (464.37): C 54.32, H 4.78, N 3.02. Found: C 54.41, H 4.66, N 3.15.

(1E,2E)-3-bromo-4-oxo-N'-tosylhex-2-enoxylimidic acid ethyl ester (2c)

oil. ^1H NMR (400 MHz, CDCl_3): 7.79-7.77 (m, 2H), 7.31 (s, 1H), 7.29-7.27 (m, 2H), 4.15 (q, $J = 7.2$ Hz, 2H), 2.72 (q, $J = 7.2$ Hz, 2H), 2.40 (s, 3H), 1.21 (t, $J = 7.2$ Hz, 3H), 1.10 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 198.0, 163.2, 143.6, 138.1, 135.2, 129.4, 126.8, 123.1, 65.6, 33.1, 21.5, 13.1, 7.2. IR (neat): 1698, 1611, 1570, 1311, 1159 cm^{-1} . MS: m/z (%) 389 [$\text{M}^+(\text{}^{81}\text{Br})$, 4], 387 [$\text{M}^+(\text{}^{79}\text{Br})$, 4], 308

([M⁺-Br, 85], 91 (100). Anal. Calcd for C₁₅H₁₈BrNO₄S (388.28): C 46.40, H 4.67, N 3.61. Found: C 46.55, H 4.56, N 3.60.

(1E,2E)-3-bromo-2-methyl-4-oxo-N'-tosylhex-2-enoxylimidic acid ethyl ester (2d)

oil. ¹H NMR (400 MHz, CDCl₃): 7.75-7.73 (m, 2H), 7.27-7.25 (m, 2H), 4.32 (q, *J* = 7.2 Hz, 2H), 2.82 (q, *J* = 7.2 Hz, 2H), 2.40 (s, 3H), 2.19 (s, 3H), 1.32 (t, *J* = 7.2 Hz, 3H), 0.99 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): 196.0, 171.4, 143.2, 140.2, 138.3, 129.2, 126.9, 123.3, 65.8, 34.1, 23.0, 21.4, 13.4, 7.8. IR (neat): 1698, 1613, 1577, 1301, 1157 cm⁻¹. MS: *m/z* (%) 403 [M⁺(⁸¹Br), 4], 401 [M⁺(⁷⁹Br), 4], 322 ([M⁺-Br, 100], 91 (88). Anal. Calcd for C₁₆H₂₀BrNO₄S (402.3): C 47.77, H 5.01, N 3.48. Found: C 47.61, H 5.10, N 3.45.

(1E,2E)-2-benzyl-3-bromo-4-oxo-N'-tosylhex-2-enoxylimidic acid ethyl ester (2e)

oil. ¹H NMR (400 MHz, CDCl₃): 7.76-7.42 (m, 2H), 7.28-7.24 (m, 7H), 4.07 (s, 2H), 3.98 (q, *J* = 7.2 Hz, 2H), 2.86 (q, *J* = 7.2 Hz, 2H), 2.40 (s, 3H), 1.00 (t, *J* = 7.2 Hz, 3H), 0.96 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): 196.2, 169.7, 143.1, 142.9, 138.4, 134.8, 129.6, 129.1, 128.3, 127.0, 126.8, 123.3, 65.4, 42.5, 34.4, 21.4, 13.0, 7.8. IR (neat): 1698, 1605, 1569, 1311, 1157 cm⁻¹. MS: *m/z* (%) 480 [M⁺(⁸¹Br)+1, 5], 478 [M⁺(⁷⁹Br)+1, 5], 398 (M⁺-Br, 10), 91 (100). Anal. Calcd for C₂₂H₂₄BrNO₄S (478.40): C 55.23, H 5.06, N 2.93. Found: C 55.14, H 5.16, N 2.95.

(1E,2E)-3-bromo-4-oxo-N'-tosyloct-2-enoxylimidic acid ethyl ester (2f)

oil. ¹H NMR (400 MHz, CDCl₃): 7.79-7.77 (m, 2H), 7.30 (s, 1H), 7.29-7.27 (m, 2H), 4.16 (q, *J* = 7.2 Hz, 2H), 2.68 (t, *J* = 7.6 Hz, 2H), 2.40 (s, 3H), 1.61-1.53 (m, 2H), 1.38-1.28 (m, 2H), 1.21 (t, *J* = 7.2 Hz, 3H), 0.90 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): 197.4, 163.3, 143.6, 138.1, 135.2, 129.4, 126.8, 123.2, 65.6, 39.4, 25.2, 84

22.0, 21.5, 13.7, 13.1. IR (neat): 1718, 1621, 1306, 1162 cm^{-1} . MS: m/z (%) 417 [$\text{M}^+(\text{}^{81}\text{Br})$, 5], 415 [$\text{M}^+(\text{}^{79}\text{Br})$, 5], 336 (M^+-Br , 56), 91 (100). Anal. Calcd for $\text{C}_{17}\text{H}_{22}\text{BrNO}_4\text{S}$ (416.33): C 49.04, H 5.33, N 3.36. Found: C 49.15, H 5.41, N 3.25.

(1E,2E)-3-bromo-2-methyl-4-oxo-N'-tosyloct-2-enoxylimidic acid ethyl ester (2g)

oil. ^1H NMR (400 MHz, CDCl_3): 7.76-7.74 (m, 2H), 7.27-7.25 (m, 2H), 4.31 (q, $J = 7.2$ Hz, 2H), 2.79 (t, $J = 7.2$ Hz, 2H), 2.40 (s, 3H), 2.20 (s, 3H), 1.51-1.44 (m, 2H), 1.36-1.31 (m, 2H), 1.31 (t, $J = 7.2$ Hz, 3H), 0.91 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 195.5, 171.3, 143.2, 140.4, 138.3, 129.2, 126.9, 123.5, 65.8, 40.4, 25.8, 23.2, 22.0, 21.5, 13.8, 13.4. IR (neat): 1698, 1616, 1577, 1304, 1160 cm^{-1} . MS: m/z (%) 430 [$\text{M}^+(\text{}^{81}\text{Br})-1$, 5], 428 [$\text{M}^+(\text{}^{79}\text{Br})-1$, 5], 350 (M^+-Br , 100), 91 (99). Anal. Calcd for $\text{C}_{18}\text{H}_{24}\text{BrNO}_4\text{S}$ (430.36): C 50.24, H 5.62, N 3.25. Found: C 50.15, H 5.57, N 3.21.

(1E,2E)-2-benzyl-3-bromo-4-oxo-N'-tosyloct-2-enoxylimidic acid ethyl ester (2h)

oil. ^1H NMR (400 MHz, CDCl_3): 7.27-7.47 (m, 2H), 7.28-7.24 (m, 5H), 4.07 (s, 2H), 3.97 (q, $J = 7.2$ Hz, 2H), 2.84 (t, $J = 7.2$ Hz, 2H), 2.41 (s, 3H), 1.52-1.44 (m, 2H), 1.37-1.30 (m, 2H), 0.95 (t, $J = 7.2$ Hz, 3H), 0.91 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 195.8, 169.7, 143.2, 143.1, 138.4, 134.9, 129.7, 129.2, 128.3, 127.1, 126.9, 123.4, 65.5, 42.6, 40.7, 25.8, 22.0, 21.5, 13.9, 13.1. IR (neat): 1701, 1617, 1159 cm^{-1} . MS: m/z (%) 507 [$\text{M}^+(\text{}^{81}\text{Br})$, 16], 505 [$\text{M}^+(\text{}^{79}\text{Br})$, 17], 426 (M^+-Br , 10), 91 (100). Anal. Calcd for $\text{C}_{24}\text{H}_{28}\text{BrNO}_4\text{S}$ (506.45): C 56.92, H 5.57, N 2.77. Found: C 57.01, H 5.51, N 2.69.

(1E,2E)-3-bromo-2-butyl-4-oxo-N'-tosyloct-2-enoxylimidic acid ethyl ester (2i)

oil. ^1H NMR (400 MHz, CDCl_3): 7.76-7.74 (m, 2H), 7.27-7.25 (m, 2H), 4.31 (q, $J = 7.2$ Hz, 2H), 2.79 (t, $J = 7.2$ Hz, 2H), 2.62 (t, $J = 7.2$ Hz, 2H), 2.41 (s, 3H), 1.50-1.29 (m, 8H), 1.30 (t, $J = 7.2$ Hz, 3H), 0.93 (t, $J = 7.2$ Hz, 3H), 0.91 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 195.9, 170.9, 144.6, 143.2, 138.4, 129.2, 126.9, 123.2, 65.8, 40.5, 36.8, 28.1, 25.9, 22.6, 22.1, 21.5, 13.9, 13.7, 13.6. IR (neat): 1696, 1609, 1572, 1310, 1159 cm^{-1} . MS: m/z (%) 473 [$\text{M}^+(\text{}^{81}\text{Br})$, 12], 471 [$\text{M}^+(\text{}^{79}\text{Br})$, 12], 392 ($\text{M}^+ - \text{Br}$, 83), 91 (100). Anal. Calcd for $\text{C}_{21}\text{H}_{30}\text{BrNO}_4\text{S}$ (472.44): C 53.39, H 6.40, N 2.96. Found: C 53.45, H 6.36, N 2.91.

(1E,2E)-3-bromo-4-oxo-N'-tosylundec-2-enoxylimidic acid ethyl ester (2j)

oil. ^1H NMR (400 MHz, CDCl_3): 7.81-7.79 (m, 2H), 7.32 (s, 1H), 7.31-7.29 (m, 2H), 4.18 (q, $J = 7.2$ Hz, 2H), 2.70 (t, $J = 7.2$ Hz, 2H), 2.42 (s, 3H), 1.64-1.57 (m, 2H), 1.31-1.29 (m, 8H), 1.23 (t, $J = 7.2$ Hz, 3H), 0.88 (t, $J = 6.8$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 197.4, 163.3, 143.6, 138.1, 135.2, 129.4, 126.8, 123.2, 65.6, 39.8, 31.5, 28.9, 28.8, 23.1, 22.5, 21.5, 13.9, 13.1. IR (neat): 1717, 1624, 1569, 1305, 1160 cm^{-1} . MS: m/z (%) 459 [$\text{M}^+(\text{}^{81}\text{Br})$, 5], 457 [$\text{M}^+(\text{}^{79}\text{Br})$, 5], 378 ($\text{M}^+ - \text{Br}$, 68), 91 (100). Anal. Calcd for $\text{C}_{20}\text{H}_{28}\text{BrNO}_4\text{S}$ (458.41): C 52.40, H 6.16, N 3.06. Found: C 52.57, H 6.07, N 3.11.

(1E,2E)-3-bromo-2-methyl-4-oxo-N'-tosylundec-2-enoxylimidic acid ethyl ester (2k)

(2k)

oil. ^1H NMR (400 MHz, CDCl_3): 7.76-7.74 (m, 2H), 7.27-7.25 (m, 2H), 4.31 (q, $J = 7.2$ Hz, 2H), 2.78 (t, $J = 7.2$ Hz, 2H), 2.40 (s, 3H), 2.20 (s, 3H), 1.52-1.47 (m, 2H), 1.31 (t, $J = 7.2$ Hz, 3H), 1.31-1.25 (m, 8H), 0.88 (t, $J = 6.8$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 195.5, 171.3, 143.2, 140.3, 138.3, 129.2, 126.9, 123.4, 65.8, 40.7, 31.6, 29.0, 28.8, 23.7, 23.2, 22.5, 21.5, 14.0, 13.4. IR (neat): 1718, 1615, 1580, 1280, 1159 cm^{-1} . MS: m/z (%) 472 [$\text{M}^+(\text{}^{81}\text{Br}) - 1$, 7], 470 [$\text{M}^+(\text{}^{79}\text{Br}) - 1$, 7], 392 ($\text{M}^+ - \text{Br}$, 100), 91 (74). Anal. Calcd for $\text{C}_{21}\text{H}_{30}\text{BrNO}_4\text{S}$ (472.44): C 53.39, H 6.40, N 2.96. Found: C 53.24, H 6.36, N 2.85.

2l

(1E,2E)-2-benzyl-3-bromo-4-oxo-N'-tosylundec-2-enoxylimidic acid ethyl ester

(2l)

oil. ^1H NMR (400 MHz, CDCl_3): 7.79-7.77 (m, 2H), 7.30-7.26 (m, 5H), 4.09 (s, 2H), 3.98 (q, $J = 7.2$ Hz, 2H), 2.85 (t, $J = 7.2$ Hz, 2H), 2.42 (s, 3H), 1.55-1.48 (m, 2H), 1.31-1.27 (m, 8H), 0.97 (t, $J = 7.2$ Hz, 3H), 0.90 (t, $J = 6.8$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 195.8, 169.6, 143.1, 143.0, 138.4, 134.8, 129.6, 129.1, 128.3, 127.1, 126.8, 123.4, 65.4, 42.6, 40.9, 31.6, 29.0, 28.8, 23.7, 22.5, 21.4, 14.0, 13.0. IR (neat): 1697, 1606, 1579, 1312, 1158 cm^{-1} . MS: m/z (%) 550 [$\text{M}^+(\text{}^{81}\text{Br})+1$, 17], 548 [$\text{M}^+(\text{}^{79}\text{Br})+1$, 16], 398 (M^+-Br , 9), 91 (100). Anal. Calcd for $\text{C}_{27}\text{H}_{34}\text{BrNO}_4\text{S}$ (548.53): C 59.12, H 6.25, N 2.55. Found: C 59.01, H 6.36, N 2.53.

2m

(1E,2E)-3-bromo-4-oxo-4-phenyl-N'-tosylbut-2-enoxylimidic acid ethyl ester

(2m)

m.p. 114-116 $^{\circ}\text{C}$. ^1H NMR (400 MHz, CDCl_3): 7.89-7.87 (m, 2H), 7.82-7.80 (m, 2H), 7.79 (s, 1H), 7.63-7.59 (m, 1H), 7.50-7.46 (m, 2H), 7.32-7.29 (m, 2H), 3.83 (q, $J = 6.8$ Hz, 2H), 2.42 (s, 3H), 0.70 (t, $J = 6.8$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 188.7, 161.8, 143.7, 138.3, 136.3, 134.4, 132.7, 129.5, 129.4, 129.0, 126.8, 123.1, 65.3, 21.5, 12.3. IR (KBr): 1678, 1627, 1571, 1303, 1155 cm^{-1} . MS: m/z (%) 437 [$\text{M}^+(\text{}^{81}\text{Br})$, 6], 435 [$\text{M}^+(\text{}^{79}\text{Br})$, 6], 356 (M^+-Br , 46), 105 (100). Anal. Calcd for $\text{C}_{19}\text{H}_{18}\text{BrNO}_4\text{S}$ (436.32): C 52.30, H 4.16, N 3.21. Found: C 52.21, H 4.29, N 3.25.

2n

(1E,2E)-2-benzyl-3-bromo-4-oxo-4-phenyl-N'-tosylbut-2-enoxylimidic acid ethyl ester (2n)

low m.p. solid. ^1H NMR (400 MHz, CDCl_3): 7.92-7.90 (m, 2H), 7.81-7.79 (m, 2H), 7.55-7.53 (m, 1H), 7.46-7.42 (m, 2H), 7.40-7.37 (m, 2H), 7.35-7.31 (m, 2H), 7.28-7.25 (m, 3H), 4.32 (s, 2H), 3.74 (q, $J = 7.2$ Hz, 2H), 2.40 (s, 3H), 0.77 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 189.8, 166.5, 143.3, 141.2, 138.4, 135.4, 134.7, 133.5, 129.9, 129.22, 129.19, 128.5, 128.3, 127.0, 126.8, 123.9, 65.5, 41.4, 21.5, 12.7. IR (KBr): 1670, 1607, 1590, 1313, 1158 cm^{-1} . MS: m/z (%) 528 [$\text{M}^+(\text{}^{81}\text{Br})+1$, 4], 526 [$\text{M}^+(\text{}^{79}\text{Br})+1$, 4], 446 (M^+-Br , 7), 91 (100). Anal. Calcd for $\text{C}_{26}\text{H}_{24}\text{BrNO}_4\text{S}$ (526.44): C 59.32, H 4.60, N 2.66. Found: C 59.21, H 4.51, N 2.60.

2o

(1E,2E)-2-(1-bromo-2-oxo-2-phenylethylidene)-N'-tosylhexanoxylimidic acid ethyl ester (2o)

oil. ^1H NMR (400 MHz, CDCl_3): 7.90-7.88 (m, 2H), 7.79-7.77 (m, 2H), 7.56-7.53 (m, 1H), 7.45-7.41 (m, 2H), 7.26-7.24 (m, 2H), 3.99 (q, $J = 7.2$ Hz, 2H), 2.89 (t, $J = 8.0$ Hz, 2H), 2.40 (s, 3H), 1.63-1.55 (m, 2H), 1.51-1.41 (m, 2H), 1.03 (t, $J = 7.2$ Hz, 3H), 0.98 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 190.1, 167.6, 143.3, 142.8, 138.5, 134.9, 133.4, 129.9, 129.2, 128.2, 126.9, 123.4, 65.8, 35.6, 28.8, 22.5, 21.5, 13.8, 13.1. IR (neat): 1670, 1608, 1594, 1320, 1159 cm^{-1} . MS: m/z (%) 494 [$\text{M}^+(\text{}^{81}\text{Br})+1$, 33], 492 [$\text{M}^+(\text{}^{79}\text{Br})+1$, 32], 412 (M^+-Br , 96), 91 (100). Anal. Calcd for $\text{C}_{23}\text{H}_{26}\text{BrNO}_4\text{S}$ (492.43): C 56.10, H 5.32, N 2.84. Found: C 56.01, H 5.36, N 2.75.

4-bromo-5-ethoxy-3-methyl-5-(naphthalen-1-yl)-1-tosyl-1H-pyrrol-2(5H)-one (7)

m.p. 146-148 °C. ^1H NMR (400 MHz, CDCl_3): 8.52-8.50 (m, 1H), 7.94-7.92 (m, 1H), 7.82-7.80 (m, 1H), 7.66-7.62 (m, 1H), 7.28-7.24 (m, 1H), 7.07-7.05 (m, 2H), 6.94-6.92 (m, 1H), 6.88-6.84 (m, 1H), 6.74-6.72 (m, 2H), 3.79-3.72 (m, 1H), 3.50-3.42 (m, 1H), 2.20 (s, 3H), 2.04 (s, 3H), 1.47 (t, $J = 6.8$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): 166.5, 143.4, 143.5, 135.0, 134.6, 133.7, 130.9, 129.4, 129.34, 129.29, 128.53, 128.47, 127.8, 126.3, 125.2, 124.6, 121.3, 96.3, 57.9, 21.4, 14.6, 10.3. IR (KBr): 1732, 1380, 1179 cm^{-1} . MS: m/z (%) 501 [$\text{M}^+(\text{}^{81}\text{Br})$, 17], 499 [$\text{M}^+(\text{}^{79}\text{Br})$, 16], 91 (100). 394.1070.

3. ^1H NMR and ^{13}C NMR Spectra of 2a-2o and 7

7.788
7.768
7.303
7.291
7.271
7.260

4.185
4.168
4.150
4.132

2.703
2.684
2.665
2.401

1.605
1.586
1.568
1.549
1.530
1.376
1.358
1.339
1.320
1.302
1.284
1.222
1.205
1.186
0.919
0.901
0.883

7.755
7.735
7.265
7.249

4.338
4.320
4.303
4.284

2.807
2.789
2.771
2.635
2.621
2.600
2.405
2.178
1.503
1.479
1.461
1.441
1.421
1.401
1.383
1.364
1.345
1.322
1.304
1.286
0.948
0.929
0.925
0.912
0.906
0.888

7.810
7.789
7.323
7.310
7.291

4.206
4.187
4.170
4.151

2.717
2.698
2.680
2.421

1.638
1.619
1.602
1.584
1.565
1.305
1.291
1.278
1.242
1.225
1.207
0.898
0.880
0.863

7.756
7.737
7.273
7.269
7.248

4.338
4.320
4.302
4.285

2.799
2.781
2.763
2.402
2.201

1.520
1.504
1.486
1.468
1.325
1.307
1.288
1.281
1.273
1.252
0.894
0.878
0.859

The crystal structure of **2m**

The crystal structure of **7**