Supporting Information for
A New Approach to Arylhydrazides via the Reaction of Mitsunobu Reagent with Arynes: Further Application to Access Diverse Nitrogen-containing Heterocycles in One Pot

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1. General

All isolated compounds were characterized on Bruker 400 and JEOL 400 MHz spectrometers in the CDCl$_3$, CD$_2$OD or (CD$_3$)$_2$CO. Chemical shifts are reported as δ values relative to internal chloroform (δ 7.26 for $^1$H NMR and 77.16 for $^{13}$C NMR), methanol (δ 3.31 for $^1$H NMR) and acetone (δ 2.05 for $^1$H NMR and 29.84 for $^{13}$C NMR). High resolution mass spectra (HRMS) were obtained on a 4G mass spectrometer by using electrospray ionization (ESI) analyzed by quadrupole time-of-flight (QToF). All melting points were measured with the samples after column chromatography and uncorrected. Column chromatography was performed on silica gel. Anhydrous THF, PhMe were distilled over sodium benzophenone ketyl under Ar. All other solvents and reagents were used as obtained from commercial sources without further purification.

2. General Experimental Procedure

2.1 General Procedure for the Preparation of Arylhydrazines 3a-3j

To a solution of benzyne precursor 1a (149 mg, 0.500 mmol) and diisopropyl azodicarboxylate (2a, 212 mg, 1.05 mmol) in CH$_3$CN (2.5 mL) were added 18-Crown-6 (330 mg, 1.25 mmol) and Ph$_3$P (275 mg, 1.05 mmol) at rt. After 10 mins, CH$_3$CN (2.5 mL, containing H$_2$O 4.32 g/L) and CsF (152 mg, 1.00 mmol) were added successively. The mixture was heated to 50 °C and kept for 4 h. The solvent was removed under reduced pressure and the residue was diluted with EtOAc (50 mL) and washed with H$_2$O three times. The organic layer was dried with MgSO$_4$, filtered, and concentrated in vacuo. The resulting mixture was purified by flash chromatography to respectively give 3a (116 mg, 83%) as a light yellow oil and 4a as a light yellow oil (4 mg, 3%).

Other Arylhydrazines were prepared following the similar method: 1 (0.5 mmol), azodicarboxylate/PPh$_3$ (2.1 equiv.), CsF (2.0 equiv.), 18-Crown-6 (2.5 equiv.), H$_2$O (1.2 equiv.), solvent (5 mL), 50 °C.

2.2 General Procedure for the Preparation of Heterocycles 5a-5c

5a & 5c To a solution of benzyne precursor 1a (149 mg, 0.500 mmol) and azodicarboxylate 2c (242 mg, 1.05 mmol) in CH$_3$CN (2.5 mL) were added 18-Crown-6 (330 mg, 1.25 mmol) and Ph$_3$P (275 mg, 1.05 mmol) at rt. After 10 min, CH$_3$CN (2.5 mL, containing H$_2$O 4.32 g/L) and CsF (152 mg, 1.00 mmol) were added successively. The mixture was heated to 50 °C and kept for 4 h. The solvent was removed under reduced pressure.

To a solution of the above residue and cycloheptanone (2.0 equiv.) or acetylacetone (2.0 equiv.) in AcOH (5.0 mL) was added ZnCl$_2$ (68 mg, 0.5 mmol) at rt. The mixture was heated to 130 °C and kept for 12 h. The solvent was removed under reduced pressure and the residue was diluted with EtOAc and washed with H$_2$O three times. The organic layer was dried with MgSO$_4$, filtered, and concentrated in vacuo. The residue was purified by flash chromatography to respectively give 5a (52 mg, 56% for 3 steps) as a white solid or 5c (55 mg, 64% for 3 steps) as a yellow oil.

5b 1-tetralone (2.0 equiv.) was used as the ketone. In accordance with the above operation, for the second step, the mixture was stirred under O$_2$, heated at 130 °C and kept for 24 h. The solvent was removed under reduced pressure and the residue was diluted with EtOAc and washed with H$_2$O three times. The organic layer was dried with MgSO$_4$, filtered, and concentrated in vacuo. The residue was purified by flash chromatography to give 5b (58 mg, 53% for 4 steps) as a white solid.
3. Characterization Data of the Products

**4a** (4 mg, Y = 3%, *R*<sub>f</sub> = 0.64 (PE:EA = 5:1)) as a light yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.03 (d, \(J = 6.4\) Hz, 1H), 7.66 (d, \(J = 7.6\) Hz, 1H), 7.50 (t, \(J = 7.6\) Hz, 1H), 7.25 (t, \(J = 7.2\) Hz, 1H), 5.36 (q, \(J = 6.0\) Hz, 1H), 5.29 (q, \(J = 6.0\) Hz, 1H), 1.49 (d, \(J = 6.4\) Hz, 6H), 1.46 (d, \(J = 6.0\) Hz, 6H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 159.1, 150.7, 141.0, 129.7, 123.1, 120.2, 118.1, 114.8, 72.6, 71.6, 22.2, 22.1; ESI-HRMS m/z Calcd. for C\(_{14}\)H\(_{18}\)N\(_2\)O\(_3\) + Na (M+Na): 285.1210, found 285.1214.

**3a** (116 mg, Y = 83%, *R*<sub>f</sub> = 0.32 (PE:EA = 5:1) as a light yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.41 (br s, 2H), 7.33-7.29 (m, 2H), 7.18-7.15 (m, 2H), 5.05-4.94 (m, 2H), 1.26 (d, \(J = 6.0\) Hz, 12H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 156.2, 154.5, 141.9, 128.6, 126.0, 124.0, 70.9, 70.1, 22.0, 22.0; ESI-HRMS m/z Calcd. for C\(_{14}\)H\(_{20}\)N\(_2\)O\(_4\) + Na (M+Na): 303.1315, found 303.1314.

**3b** (97 mg, Y = 77%, *R*<sub>f</sub> = 0.16 (PE:EA = 5:1)) as a light yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.42 (d, \(J = 8.0\) Hz, 2H), 7.35-7.31 (m, 2H), 7.20 (t, \(J = 7.2\) Hz, 1H), 7.15 (br s, 1H), 4.28-4.18 (m, 4H), 1.27 (t, \(J = 6.8\) Hz, 6H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 156.5, 155.1, 141.8, 128.8, 126.5, 124.4, 63.1, 62.4, 14.6; ESI-HRMS m/z Calcd. for C\(_{12}\)H\(_{16}\)N\(_2\)O\(_4\) + Na (M+Na): 275.1002, found 275.1003.

**3c** (111 mg, Y = 72%, *R*<sub>f</sub> = 0.49 (PE:EA = 5:1)) as a light yellow solid. m.p. 105-106 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.40 (br s, 2H), 7.30 (t, \(J = 7.6\) Hz, 2H), 7.14 (t, \(J = 7.2\) Hz, 1H), 6.91-6.74 (m, 1H), 1.49 (s, 18H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 155.5, 153.7, 142.2, 128.8, 126.5, 124.4, 63.1, 62.4, 14.6; ESI-HRMS m/z Calcd. for C\(_{16}\)H\(_{24}\)N\(_2\)O\(_4\) + Na (M+Na): 331.1628, found 331.1629.

**3d** (133 mg, Y = 71%, *R*<sub>f</sub> = 0.27 (PE:EA = 5:1)) as a light yellow oil. \(^1\)H NMR (300 MHz, CD\(_3\)OD) \(\delta\) 7.32-7.21 (br s, 15H), 5.18 (s, 2H), 5.12 (s, 2H); \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 7.32-7.26 (br s, 16H), 5.21 (s, 2H), 5.18 (s, 2H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 156.3, 154.8, 141.5, 135.7, 135.6, 128.8, 128.7, 128.6, 128.4, 128.3, 128.0, 126.6, 124.5, 68.5, 68.0; ESI-HRMS m/z Calcd. for C\(_{22}\)H\(_{30}\)N\(_2\)O\(_4\) + Na (M+Na): 399.1315, found 399.1316.
3e (142 mg, Y = 83%, Rf = 0.30 (PE:EA = 2:1)) as a yellow oil. ¹H NMR (300 MHz, CDCl₃) δ 7.03 (br s, 1H), 6.95-6.93 (m, 2H), 6.81-6.79 (m, 1H), 5.04-4.96 (m, 2H), 3.86 (s, 3H), 3.85 (s, 3H), 1.28-1.25 (m, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 156.1, 154.8, 148.6, 147.5, 135.2, 116.8, 110.7, 109.2, 70.6, 69.9, 56.0, 55.8, 22.0, 22.0; ESI-HRMS m/z Calcd. for C₁₆H₂₄N₂O₆ + Na (M+Na): 363.1527, found 363.1528.

3f (113 mg, Y = 70%, Rf = 0.24 (PE:EA = 5:1)) as a yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 6.99 (br s, 1H), 6.94 (br s, 1H), 6.86 (br s, 1H), 6.74-6.72 (m, 1H), 5.95 (s, 2H), 5.03-4.93 (m, 2H), 1.27-1.23 (m, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 156.1, 154.7, 147.5, 146.1, 136.0, 118.4, 107.7, 106.9, 101.5, 70.8, 69.9, 22.0, 21.9; ESI-HRMS m/z Calcd. for C₁₅H₂₀N₂O₆ + Na (M+Na): 347.1214, found 347.1213.

3g (The regioselectivity was confirmed through comparison of the ¹³C-NMR of 3g with analogous m-methoxylarylhydrazide. See Fig. S35) (94 mg, Y = 60%, Rf = 0.33 (PE:EA = 5:1)) as a yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.20 (t, J = 8.0 Hz, 1H), 7.09-7.02 (m, 3H), 6.72 (d, J = 7.6 Hz, 1H), 5.02-4.97 (m, 2H), 3.77 (s, 3H), 1.26 (d, J = 5.6 Hz, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 159.8, 156.1, 154.3, 143.0, 129.2, 116.0, 111.8, 109.7, 70.9, 70.1, 55.4, 22.0, 22.0; ESI-HRMS m/z Calcd. for C₁₅H₂₂N₂O₅ + Na (M+Na): 333.1421, found 333.1420.

3h (131 mg, Y = 85%, Rf = 0.33 (PE:EA = 5:1)) as a yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.20 (br s, 1H), 7.12 (br s, 2H), 7.08-7.06 (m, 1H), 5.05-4.96 (m, 2H), 2.23 (s, 3H), 2.22 (s, 3H), 1.26 (d, J = 6.4 Hz, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 159.8, 156.1, 154.3, 143.0, 129.2, 116.0, 111.8, 109.7, 70.9, 70.1, 55.4, 22.0, 22.0; ESI-HRMS m/z Calcd. for C₁₅H₂₄N₂O₅ + Na (M+Na): 331.1628, found 331.1629.

3i (141 mg, Y = 88%, Rf = 0.32 (PE:EA = 5:1)) as a yellow light yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.28 (br s, 1H), 7.18 (br s, 1H), 7.15 (br s, 2H), 5.03-4.96 (m, 2H), 2.87 (q, J = 7.2 Hz, 4H), 2.10-2.02 (m, 2H), 1.26 (d, J = 6.4 Hz, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 156.1, 154.8, 144.7, 142.4, 140.1, 124.1, 122.7, 120.8, 70.6, 69.8, 32.9, 32.4, 25.6, 22.0, 22.0; ESI-HRMS m/z Calcd. for C₁₆H₂₄N₂O₄ + Na (M+Na): 343.1628, found 343.1630.

3j (67 mg, Y = 42%, Rf = 0.29 (PE:EA = 5:1)) as a yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.35 (br s, 1H), 7.18 (br s, 1H), 7.13-7.06 (m, 1H), 6.87-6.75 (m, 1H), 5.05-4.97 (m, 2H), 1.28 (d, J =
6.4 Hz, 12H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 156.1, 154.1, 151.4 ($J = 13.5$ Hz), 148.1 ($J = 13.6$ Hz), 138.3 (dd, $J = 7.5$, 3.0 Hz), 119.5, 116.8 ($J = 18.0$ Hz), 113.5, 71.5, 70.6, 22.0, 22.0; ESI-HRMS m/z Calcd. for C$_{14}$H$_{18}$F$_2$N$_2$O$_4$ + Na (M+Na): 339.1127, found 339.1128.

3k (The regioselectivity was confirmed through comparison of the $^{13}$C-NMR of 3k with analogous m-alkoxyaryldiazide like 3g) (108 mg, Y = 62%, R$_f$ = 0.22 (PE:EA = 5:1)) as a light yellow oil. $^1$H NMR (300 MHz, CDCl$_3$) δ 7.22 (d, $J = 8.4$ Hz, 1H), 7.08 (br s, 2H), 6.85-6.64 (m, 2H), 5.06-4.96 (m, 2H), 4.63 (q, $J = 2.1$ Hz, 2H), 1.86 (t, $J = 2.1$ Hz, 3H), 1.28 (d, $J = 6.0$ Hz, 12H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 158.1, 156.1, 154.3, 143.0, 129.3, 116.5, 112.4, 110.7, 84.0, 74.0, 71.1, 70.2, 56.7, 22.1, 22.1, 3.8; ESI-HRMS m/z Calcd. for C$_{18}$H$_{24}$N$_2$O$_5$ + Na (M+Na): 371.1577, found 371.1574.

3l (ratio of $\alpha$:m = 1:4 according to the integrals of allylic hydrogens. The minor isomer was further confirmed through the transformation of E1cB eliminative cleavage of the N-N′-bond reported by Magnus (Org. Lett. 2009, 11, 5646), and the crude $^1$H-NMR of which could be found in Fig. S36. It could be easily compared with known compounds. See Fig. S37 and Fig. S38) (112 mg, Y = 70%, R$_f$ = 0.33 (PE:EA = 5:1)) as a yellow oil. $^1$H NMR (300 MHz, (CD$_3$)$_2$CO) δ 8.83 (s, 0.80H), 8.50 (s, 0.20H), 7.59-7.20 (m, 4H), 7.07-6.97 (m, 1H), 6.04-5.90 (m, 1H), 3.51 (d, $J = 6.0$ Hz, 0.40H) 3.39 (d, $J = 6.0$ Hz, 1.6H), 1.42-1.09 (m, 12H); $^{13}$C NMR (75 MHz, (CD$_3$)$_2$CO) δ 156.8, 154.8, 144.7, 141.2, 129.3, 123.6, 141.0, 138.2, 129.1, 127.6 (minor isomer), 126.3, 124.4, 121.9, 116.1, 40.6, 22.2, 22.2; ESI-HRMS m/z Calcd. for C$_{17}$H$_{24}$N$_2$O$_4$ + Na (M+Na): 343.1628, found 343.1622.

3m (The regioselectivity was readily observed from the $^1$H-NMR of 3m) (DIAD/PPh$_3$ (4.2 equiv.) were used instead, 50 mg, Y = 27%, R$_f$ = 0.37 (PE:EA = 5:1)) as a yellow oil. $^1$H NMR (300 MHz, (CD$_3$)$_2$CO) δ 8.71 (s, 1H), 7.39 (s, 1H), 7.18 (s, 1H), 7.05 (s, 1H), 4.85-4.74 (m, 2H), 2.19 (s, 3H), 1.13 (d, $J = 3.9$ Hz, 6H), 1.11 (d, $J = 4.2$ Hz, 6H); $^{13}$C NMR (75 MHz, (CD$_3$)$_2$CO) δ 156.8, 154.5, 144.7, 141.2, 129.3, 123.6, 123.0, 121.8, 71.1, 70.0, 71.1, 70.0, 22.2, 22.2, 21.2; ESI-HRMS m/z Calcd. for C$_{15}$H$_{23}$BrN$_2$O$_4$ + Na (M+Na): 395.0577, found 395.0575.
**5a** (52 mg, Y = 56%, R<sub>t</sub> = 0.66 (PE:EA = 5:1)) as a white solid. m.p. 133-134 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.68 (s, 1H), 7.48-7.45 (m, 1H), 7.27-7.24 (m, 1H), 7.09-7.06 (m, 2H), 2.85-2.80 (m, 4H), 1.90-1.87 (m, 2H), 1.80-1.77 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 137.5, 134.4, 129.4, 120.7, 119.1, 117.8, 113.8, 110.3, 31.9, 29.6, 28.8, 27.6, 24.8; ESI-HRMS m/z Calcd. for C<sub>13</sub>H<sub>12</sub>N + H (M+H): 186.1277, found 186.1278.

![Structure of 5a](image)

**5b** (58 mg, Y = 53%, R<sub>t</sub> = 0.51 (PE:EA = 5:1)) as a white solid. m.p. 222-223 °C. <sup>1</sup>H NMR (400 MHz, (CD<sub>3</sub>)<sub>2</sub>CO) δ 11.28 (s, 1H), 8.49 (d, J = 8.4 Hz, 1H), 8.21 (d, J = 8.4 Hz, 1H), 8.18 (d, J = 8.0 Hz, 1H), 8.04 (d, J = 8.0 Hz, 1H), 7.68-7.59 (m, 3H), 7.56-7.52 (m, 1H), 7.41 (td, J = 7.2, 1.2 Hz, 1H), 7.28-7.24 (m, 1H); <sup>13</sup>C NMR (100 MHz, (CD<sub>3</sub>)<sub>2</sub>CO) δ 140.1, 136.3, 133.4, 129.6, 126.3, 126.0, 125.5, 124.8, 122.5, 122.3, 120.5, 120.3, 120.2, 118.9, 112.2; ESI-HRMS m/z Calcd. for C<sub>16</sub>H<sub>11</sub>N + H (M+H): 218.0964, found 218.0965.

![Structure of 5b](image)

**5c** (55 mg, Y = 64%, R<sub>t</sub> = 0.53 (PE:EA = 5:1)) as a yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.43-7.42 (m, 4H), 7.34-7.32 (m, 1H), 5.99 (s, 1H), 2.30 (s, 3H), 2.29 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 149.0, 140.0, 139.4, 129.0, 127.3, 124.8, 107.0, 13.6, 12.4; ESI-HRMS m/z Calcd. C<sub>11</sub>H<sub>12</sub>N<sub>2</sub> + H (M+H): 173.1073, found 173.1074.
4. NMR spectra

Fig. S1. $^1$H NMR of compound 4a (400 MHz, CDCl$_3$).

Fig. S2. $^{13}$C NMR of compound 4a (100 MHz, CDCl$_3$).
Fig. S3. $^1$H NMR of compound 3a (400 MHz, CDCl$_3$).

Fig. S4. $^{13}$C NMR of compound 3a (100 MHz, CDCl$_3$).
Fig. S5. $^1$H NMR of compound 3b (400 MHz, CDCl$_3$).

Fig. S6. $^{13}$C NMR of compound 3b (100 MHz, CDCl$_3$).
**Fig. S7.** $^1$H NMR of compound 3c (400 MHz, CDCl$_3$).

**Fig. S8.** $^{13}$C NMR of compound 3c (100 MHz, CDCl$_3$).
Fig. S9. $^1$H NMR of compound 3d (300 MHz, CD$_3$OD).

Fig. S9'. $^1$H NMR of compound 3d (300 MHz, CDCl$_3$).
Fig. S10. $^{13}$C NMR of compound 3d (100 MHz, CDCl$_3$).

Fig. S11. $^1$H NMR of compound 3e (300 MHz, CDCl$_3$).
Fig. S12. $^{13}$C NMR of compound 3e (100 MHz, CDCl$_3$).

Fig. S13. $^1$H NMR of compound 3f (400 MHz, CDCl$_3$).
Fig. S14. $^{13}$C NMR of compound 3f (100 MHz, CDCl$_3$).

Fig. S15. $^1$H NMR of compound 3g (400 MHz, CDCl$_3$).
Fig. S16. $^{13}$C NMR of compound 3g (100 MHz, CDCl$_3$).

Fig. S17. $^1$H NMR of compound 3h (400 MHz, CDCl$_3$).
Fig. S18. $^{13}$C NMR of compound 3h (100 MHz, CDCl$_3$).

Fig. S19. $^1$H NMR of compound 3i (400 MHz, CDCl$_3$).
**Fig. S20.** $^{13}$C NMR of compound 3i (100 MHz, CDCl$_3$).

**Fig. S21.** $^1$H NMR of compound 3j (400 MHz, CDCl$_3$).
Fig. S22. $^{13}$C NMR of compound 3j (75 MHz, CDCl$_3$).

Fig. S23. $^1$H NMR of compound 3k (300 MHz, CDCl$_3$).
Fig. S24. $^{13}$C NMR of compound 3k (100 MHz, CDCl$_3$).

Fig. S25. $^1$H NMR of compound 3l (300 MHz, (CD$_3$)$_2$CO).
Fig. S26. $^{13}$C NMR of compound 3l (75 MHz, (CD$_3$)$_2$CO).

Fig. S27. $^1$H NMR of compound 3m (300 MHz, (CD$_3$)$_2$CO).
Fig. S28. $^{13}$C NMR of compound 3m (75 MHz, (CD$_3$)$_2$CO).

Fig. S29. $^1$H NMR of compound 5a (300 MHz, CDCl$_3$).
Fig. S30. $^{13}$C NMR of compound 5a (100 MHz, CDCl$_3$).

Fig. S31. $^1$H NMR of compound 5b (400 MHz, (CD$_3$)$_2$CO).
**Fig. S32.** $^{13}$C NMR of compound 5b (100 MHz, (CD$_3$)$_2$CO).

**Fig. S33.** $^1$H NMR of compound 5c (400 MHz, CDCl$_3$).
**Fig. S34.** $^{13}$C NMR of compound 5c (100 MHz, CDCl$_3$).

**Fig. S35.** $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 159.4, 155.2, 153.3, 143.1, 126.8, 115.6, 111.0, 109.1, 82.0, 81.2, 55.0, 28.0, 27.9.
Fig. S36. $^1$H NMR (300 MHz, CDCl$_3$)

Fig. S37. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.25-7.15 (m, 3H), 6.88 (d, $J = 7.2$ Hz, 1H), 6.44 (s, 1H),
5.96 (ddt, $J = 16.9, 10.1, 6.7$ Hz, 1H), 5.17-4.97 (m, 2H), 3.37 (d, $J = 6.7$ Hz, 2H), 1.52 (s, 9H).

Fig. S38. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.81 (d, $J = 7$Hz, 1H), 7.26 (t, $J = 8$Hz, 1H), 7.17 (d, $J = 7$Hz, 1H), 7.07 (t, $J = 7$Hz, 1H), 6.48 (s (br), 1H), 5.99 (ddt, $J = 17, 10, 6$ Hz, 1H), 5.20-5.17 (m, 1H), 5.11-5.07 (m, 1H), 3.39 (d, $J = 6$ Hz, 2H), 1.54 (s, 9H).