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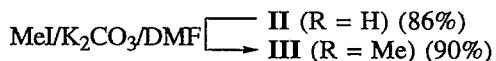
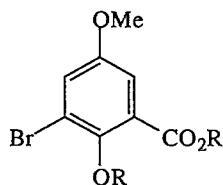
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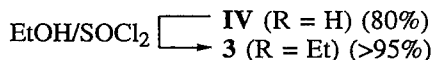
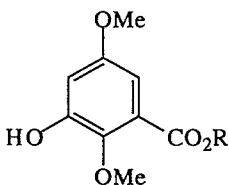
S1

Supporting Information: Synthesis of the Eneidyne Aglycone (\pm)-Calicheamicinone



3-Bromo-2-hydroxy-5-methoxybenzoic acid II. IR (thin film) 2858, 1661, 1607, 1430, 1234, 1044, 860 and 786 cm⁻¹. ¹H NMR (300 MHz, DMSO-d⁶) δ 7.47 (1H, d, J = 3.5 Hz), 7.29 (1H, d, J = 3.5 Hz), 3.73 (3H, s). ¹³C NMR (75 MHz, DMSO-d⁶) δ 171.3, 152.0, 151.5, 125.4, 113.8, 113.1, 110.5 and 55.9. HRMS (+CI) calcd for C₈H₈O₄Br (M+1⁺) 246.9606, found 246.9603.

3-Bromo-2,5-dimethoxybenzoic acid methyl ester III. ¹H NMR (300 MHz, DMSO-d⁶) δ 7.44 (1H, d, J = 3.5 Hz), 7.20 (1H, d, J = 3.5 Hz), 3.84 (3H, s), 3.77 (3H, s), 3.74 (3H, s).

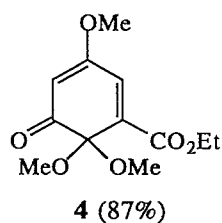


2,5-Dimethoxy-3-hydroxybenzoic acid IV. Mp 168-170 °C (H₂O). IR (thin film) 3194, 2925, 1713, 1598, 1455, 1409, 1349, 1287, 1169, 1051, 969, 848, 784 cm⁻¹. ¹H NMR (300 MHz, DMSO-d⁶) δ 12.77 (1H, br s), 9.61 (1H, s), 6.57 (2H, m), 3.67 (3H, s), 3.66 (3H, s). ¹³C NMR (75 MHz, DMSO-d⁶) δ 167.5, 155.1, 151.8, 141.0, 127.1, 106.0, 104.1, 60.9, 55.3. HRMS (+CI) calcd for C₉H₁₀O₅ (M⁺) 198.0528, found 198.0525.

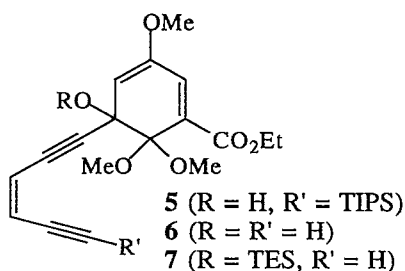
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S2

2,5-Dimethoxy-3-hydroxybenzoic acid ethyl ester 3. IR (thin film) 3418, 2981, 2841, 1714, 1616, 1591, 1494, 1470, 1430, 1371, 1344, 1307, 1273, 1222, 1155, 1048, 1028, 996, 851, 791 cm^{-1} . ^1H NMR (300 MHz, CDCl_3) δ 6.91 (1H, d, $J = 3.0$ Hz), 6.71 (1H, d, $J = 3.0$ Hz), 6.01 (1H, br s), 4.38 (2H, q, $J = 7.0$ Hz), 3.85 (3H, s), 3.78 (3H, s), 1.39 (3H, t, $J = 7.0$ Hz). ^{13}C NMR (75 MHz, CDCl_3) δ 165.4, 155.8, 150.5, 141.1, 124.0, 106.8, 106.0, 62.4, 61.2, 55.6, 14.1. HRMS (+CI) calcd for $\text{C}_{11}\text{H}_{15}\text{O}_5$ ($\text{M}+1$) 227.0919, found 227.0921.



ortho-Quinone ketal 4. Mp 65-66 $^{\circ}\text{C}$. IR (thin film) 2944, 1733, 1660, 1594, 1377, 1210, 1077 cm^{-1} . ^1H NMR (300 MHz, C_6D_6) δ 7.09 (1H, d, $J = 2.5$ Hz), 5.24 (1H, d, $J = 2.5$ Hz), 4.04 (2H, q, $J = 7.0$ Hz), 3.42 (6H, s), 2.73 (3H, s), 0.94 (3H, t, $J = 7.0$ Hz). ^{13}C NMR (75 MHz, C_6D_6) δ 192.8, 166.8, 163.1, 140.3, 133.7, 102.1, 93.6, 61.1, 55.4, 51.2, 14.0. HRMS (+CI) calcd for $\text{C}_{12}\text{H}_{16}\text{O}_6$ (M^+) 256.0946, found 256.0948.



Triisopropylsilylacetylene 5. IR (thin film) 3488, 2944, 2866, 1727, 1655, 1610, 1460, 1366, 1260, 1215, 1143, 1077, 882, 677 cm^{-1} . ^1H NMR (300 MHz, C_6D_6) δ 6.85 (1H, d, $J = 2.5$ Hz), 5.51 (1H, d, $J = 11.0$ Hz), 5.49 (1H, d, $J = 11.0$ Hz), 5.11 (1H, d, $J = 2.5$ Hz), 3.96-3.85 (2H, m), 3.59 (3H, s), 3.58 (3H, s), 3.32 (1H, s), 3.04 (3H, s), 1.21-1.14 (21H, m), 0.94-0.84 (3H, m). ^{13}C NMR (75 MHz, C_6D_6) δ 170.2, 165.0, 151.5, 133.5, 120.3, 119.7,

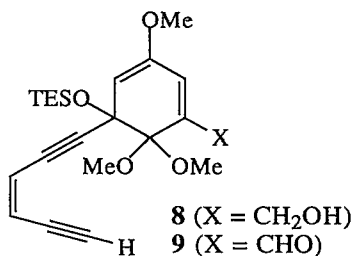
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S3

106.0, 104.5, 100.4, 99.3, 98.1, 82.3, 73.9, 60.8, 54.5, 52.4, 51.9, 18.9, 14.0, 11.6. HRMS (+CI) calcd for $C_{27}H_{40}O_6Si$ (M^+) 488.2594, found 488.2583.

Terminal enediyne 6. IR (thin film) 3472, 3272, 2978, 2943, 2837, 2097, 1719, 1661, 1613, 1455, 1367, 1255, 1220, 1138, 1067, 1026, 997, 967 cm^{-1} . 1H NMR (300 MHz, C_6D_6) δ 6.84 (1H, d, $J = 2.5$ Hz), 5.52 (1H, dd, $J = 11.0, 1.0$ Hz), 5.34 (1H, dd, $J = 11.0, 2.5$ Hz), 5.09 (1H, d, $J = 2.5$ Hz), 3.96-3.87 (2H, m), 3.60 (3H, s), 3.58 (3H, s), 3.38 (1H, s), 2.98 (1H, m), 2.96 (3H, s), 0.93-0.84 (3H, m). ^{13}C NMR (75 MHz, C_6D_6) δ 165.1, 151.5, 133.7, 133.4, 121.4, 119.2, 106.2, 102.1, 100.4, 98.0, 85.5, 81.9, 74.0, 60.8, 54.5, 52.4, 52.0, 13.9. HRMS (+CI) calcd for $C_{18}H_{20}O_6$ (M^+) 332.1259, found 332.1271.

Triethylsilyl ether 7. IR (thin film) 2943, 1727, 1655, 1366, 1266, 1210, 1138, 1082, 1038, 993 cm^{-1} . 1H NMR (300 MHz, C_6D_6) δ 6.70 (1H, d, $J = 2.5$ Hz), 5.57 (1H, dd, $J = 11.0, 1.0$ Hz), 5.36 (1H, dd, $J = 11.0, 2.5$ Hz), 5.21 (1H, d, $J = 2.5$ Hz), 3.96-3.89 (2H, m), 3.82 (3H, s), 3.61 (3H, s), 3.05 (3H, s), 2.90 (1H, m), 1.11 (9H, t, $J = 8.0$ Hz), 0.94-0.79 (9H, m). ^{13}C NMR (75 MHz, C_6D_6) δ 165.4, 152.6, 136.6, 130.7, 121.4, 118.8, 105.6, 101.2, 100.9, 84.9, 83.1, 81.2, 72.9, 60.7, 54.4, 52.5, 51.4, 14.0, 7.3, 6.8. HRMS (+CI) calcd for $C_{24}H_{35}O_6Si$ ($M+1$) 447.2202, found 447.2200.



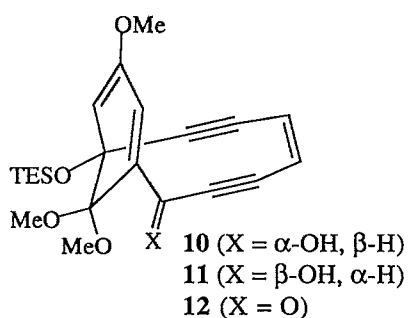
Alcohol 8. 1H NMR (300 MHz, C_6D_6) δ 6.11 (1H, m), 5.57 (1H, dd, $J = 11.0, 1.0$ Hz), 5.35 (1H, dd, $J = 11.0, 2.5$ Hz), 5.00 (1H, d, $J = 2.5$ Hz), 4.42 (1H, ddd, $J = 14.0, 4.5, 1.5$ Hz), 4.10 (1H, ddd, $J = 14.0, 8.0, 1.5$ Hz), 3.65 (3H, s), 3.10 (3H, s), 3.09 (3H, s), 2.91

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S4

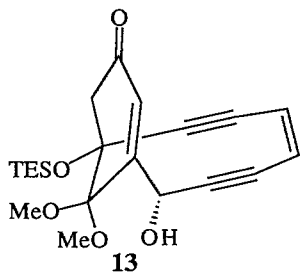
(1H, dd, $J = 2.5, 1.0$ Hz), 2.02 (1H, dd, $J = 8.0, 4.5$ Hz), 1.18-1.06 (9H, m), 0.92-0.76 (6H, m). HRMS (+CI) calcd for $C_{22}H_{33}O_5Si$ ($M+1$) 405.2097, found 405.2100.

Aldehyde 9. 1H NMR (300 MHz, C_6D_6) δ 10.11 (1H, s), 7.07 (1H, d, $J = 2.5$ Hz), 5.54 (1H, dd, $J = 11.0, 1.0$ Hz), 5.35 (1H, dd, $J = 11.0, 2.5$ Hz), 5.22 (1H, d, $J = 2.5$ Hz), 3.68 (3H, s), 3.01 (3H, s), 2.98 (3H, s), 2.91 (1H, m), 1.04 (9H, t, $J = 8.0$ Hz), 0.82-0.69 (6H, m). HRMS (+CI) calcd for $C_{22}H_{31}O_5Si$ (MH^+) 403.1941, found 403.1950.

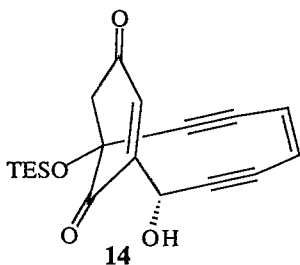


12-(S)-Alcohol 10. 1H NMR (300 MHz, acetone- d_6) δ 6.13 (1H, d, $J = 10.0$ Hz), 5.98 (1H, dd, $J = 10.0, 1.5$ Hz), 5.84 (1H, dd, $J = 2.0, 0.5$ Hz), 5.26 (1H, m), 4.78 (1H, d, $J = 2.0$ Hz), 3.57 (3H, s), 3.52 (3H, s), 3.27 (3H, s), 2.82 (1H, s), 0.98-0.90 (9H, m), 0.82-0.72 (6H, m).

Ketone 12. 1H NMR (300 MHz, C_6D_6) δ 5.88 (1H, d, $J = 2.0$ Hz), 5.57 (1H, d, $J = 10.0$ Hz), 5.26 (1H, d, $J = 10.0$ Hz), 4.82 (1H, d, $J = 2.0$ Hz), 3.44 (3H, s), 3.24 (3H, s), 3.01 (3H, s), 1.09 (9H, t, $J = 8.0$ Hz), 0.88-0.72 (6H, m). HRMS (+CI) calcd for $C_{22}H_{28}O_5Si$ (M^+) 400.1706, found 400.1703.



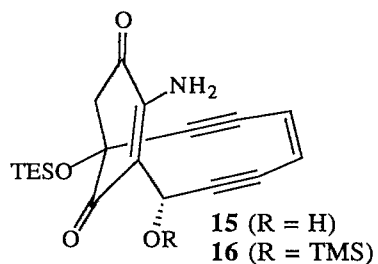
Enone 13. IR (thin film) 3499, 2960, 2870, 1677, 1458, 1407, 1296, 1251, 1161, 1122, 1071, 992, 947 cm^{-1} . ^1H NMR (300 MHz, acetone- d_6) δ 6.08 (1H, s), 6.07 (1H, d, $J = 9.5$ Hz), 5.97 (1H, dd, $J = 9.5, 1.5$ Hz), 5.38 (1H, m), 4.39 (1H, d, $J = 12.0$ Hz), 3.72 (3H, s), 3.39 (3H, s), 3.00 (1H, dd, $J = 17.0, 0.5$ Hz), 2.83 (1H, br s), 2.71 (1H, dd, $J = 17.0, 1.0$ Hz), 1.08-0.97 (9H, m), 0.82-0.71 (6H, m). ^{13}C NMR (75 MHz, acetone- d_6) δ 195.6, 161.1, 127.6, 124.4, 123.9, 104.2, 102.5, 99.6, 88.6, 86.1, 78.5, 68.0, 52.9, 52.2, 50.0, 7.1, 6.6. HRMS (+CI) calcd for $\text{C}_{21}\text{H}_{29}\text{O}_5\text{Si}$ (M+1) 389.1784, found 389.1774.



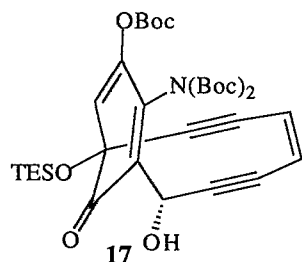
Enedione 14. Mp 101-104 $^{\circ}\text{C}$ (dec). IR (thin film) 3508, 2953, 1709, 1674, 1403, 1244, 1038, 956, 742 cm^{-1} . ^1H NMR (300 MHz, C_6D_6) δ 5.64 (1H, d, $J = 1.5$ Hz), 5.14 (1H, dd, $J = 10.0, 1.5$ Hz), 5.09 (1H, d, $J = 10.0$ Hz), 4.92 (1H, dd, $J = 11.0, 1.5$ Hz), 4.54 (1H, d, $J = 11.0$ Hz), 3.06 (1H, dd, $J = 17.0, 1.5$ Hz), 2.47 (1H, d, $J = 17.0$ Hz), 1.04 (9H, t, $J = 7.5$ Hz), 0.79 (6H, m). ^{13}C NMR (75 MHz, C_6D_6) δ 194.7, 192.5, 147.7, 132.2, 124.4, 122.6, 100.2, 96.0, 93.0, 89.1, 75.4, 67.7, 50.4, 7.1, 6.4. HRMS (+CI) calcd for $\text{C}_{19}\text{H}_{23}\text{O}_4\text{Si}$ (M+1) 343.1367, found 343.1356.

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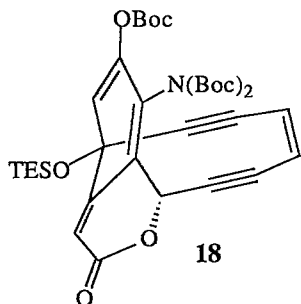
S6



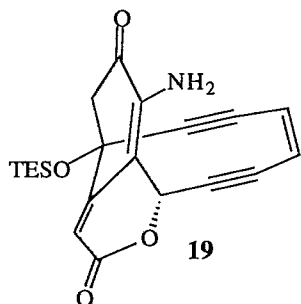
Aminoenedione 15. Mp 124-125 °C (dec). IR (thin film) 3450, 3354, 2956, 2877, 2818, 1697, 1660, 1633, 1455, 1384, 1252, 1159, 999, 952, 819, 740 cm^{-1} . ^1H NMR (300 MHz, C_6D_6) δ 6.11 (1H, d, $J = 10.0$ Hz), 5.19 (1H, dd, $J = 8.0, 1.5$ Hz), 5.11 (1H, d, $J = 8.0$ Hz), 5.02 (1H, dd, $J = 10.0, 1.5$ Hz), 3.87 (2H, br s), 3.10 (1H, d, $J = 18.5$ Hz), 2.51 (1H, d, $J = 18.5$ Hz), 1.10 (9H, t, $J = 8.0$ Hz), 0.89 (6H, m). ^{13}C NMR (75 MHz, CDCl_3) δ 193.6, 190.3, 142.1, 124.3, 123.2, 114.3, 99.9, 96.8, 91.1, 85.0, 74.2, 63.1, 48.9, 6.9, 6.0. HRMS (+CI) calcd for $\text{C}_{19}\text{H}_{24}\text{NO}_4\text{Si}$ (M+1) 358.1475, found 358.1472.



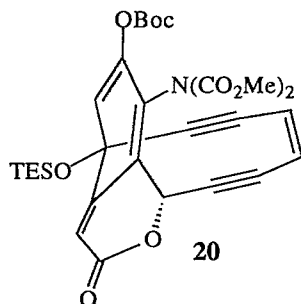
Tris (tert-butoxycarbonyl) protected secondary alcohol 17. Mp 135-137 °C (dec). IR (thin film) 3509, 2959, 1800, 1770, 1698, 1456, 1371, 1252, 1115, 743 cm^{-1} . ^1H NMR (300 MHz, C_6D_6) δ 6.24 (1H, s), 5.73 (1H, dd, $J = 10.0, 2.0$ Hz), 5.31 (2H, m), 4.66 (1H, d, $J = 10.0$ Hz), 1.43 (9H, s), 1.37 (9H, s), 1.27 (9H, s), 1.07 (9H, t, $J = 8.0$ Hz), 0.84 (6H, m). ^{13}C NMR (75 MHz, C_6D_6) δ 196.6, 150.6, 148.3, 148.2, 142.3, 136.1, 132.7, 127.9, 126.0, 123.5, 101.3, 94.5, 92.6, 87.8, 84.1, 83.9, 83.3, 75.2, 63.5, 27.4, 26.9, 26.8, 6.9, 6.0. HRMS (+CI) calcd for $\text{C}_{34}\text{H}_{48}\text{NO}_{10}\text{Si}$ (M+1) 658.3048, found 658.3043.



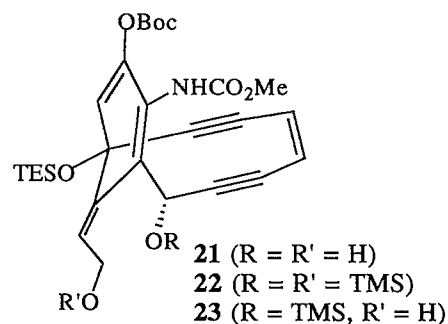
Tris (*tert*-Butyloxycarbonyl) protected lactone 18. IR (thin film) 2979, 1800, 1762, 1736, 1370, 1250, 1149, 1109, 745 cm^{-1} . ^1H NMR (300 MHz, C_6D_6) δ 6.45 (1H, s), 6.15 (1H, s), 6.00 (1H, d, $J = 2.0$ Hz), 5.47 (1H, d, $J = 9.5$ Hz), 5.35 (1H, dd, $J = 9.5, 2.0$ Hz), 1.34 (9H, s), 1.33 (9H, s), 1.27 (9H, s), 0.90 (9H, t, $J = 8.0$ Hz), 0.67 (6H, m). ^{13}C NMR (75 MHz, C_6D_6) δ 160.8, 154.4, 150.7, 148.7, 148.4, 144.0, 125.6, 125.0, 123.3, 119.9, 114.9, 98.2, 97.1, 95.2, 90.3, 84.1, 83.8, 83.1, 70.3, 67.9, 27.6, 27.5, 27.4, 7.0, 6.2. HRMS (+CI) calcd for $\text{C}_{36}\text{H}_{48}\text{NO}_{10}\text{Si}$ ($\text{M}+1$) 682.3048, found 682.3060.



Enamine lactone 19. Mp 146-150 $^{\circ}\text{C}$ (dec). IR (thin film) 3458, 3340, 2956, 2877, 1696, 1622, 1593, 1416, 1372, 1289, 1150, 1125, 1080, 1019, 850, 728 cm^{-1} . ^1H NMR (300 MHz, CDCl_3) δ 6.13 (1H, s), 5.99 (1H, d, $J = 1.5$ Hz), 5.90 (1H, d, $J = 9.5$ Hz), 5.81 (1H, dd, $J = 9.5, 1.5$ Hz), 4.67 (2H, br s), 3.14 (1H, d, $J = 16.5$ Hz), 2.97 (1H, d, $J = 16.5$ Hz), 1.02 (9H, t, $J = 8.0$ Hz), 0.79 (6H, m). ^{13}C NMR (75 MHz, C_6D_6) δ 189.6, 162.7, 154.5, 133.8, 125.1, 123.5, 113.0, 107.5, 99.1, 95.6, 88.8, 86.9, 70.8, 67.3, 49.1, 6.9, 5.8. HRMS (+CI) calcd for $\text{C}_{21}\text{H}_{24}\text{NO}_4\text{Si}$ ($\text{M}+1$) 382.1474, found 382.1464.



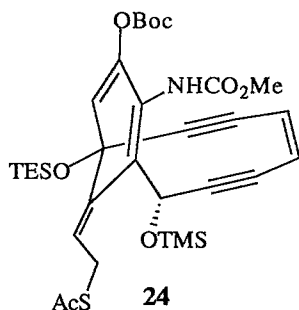
Enol carbonate 20. Mp 140-141 °C (dec). IR (thin film) 2958, 1808, 1770, 1732, 1273, 1110 cm^{-1} . ^1H NMR (300 MHz, CDCl_3) δ 6.22 (1H, d, $J = 10.0$ Hz), 6.21 (1H, s), 6.04 (3H, m), 3.86 (3H, s), 3.59 (3H, s), 1.49 (9H, s), 1.04 (9H, t, $J = 8.0$ Hz), 0.82 (6H, m). ^{13}C NMR (75 MHz, CDCl_3) δ 161.0, 153.2, 150.9, 150.4, 150.0, 142.2, 125.8, 124.3, 123.3, 121.8, 114.9, 97.1, 95.3, 94.3, 90.5, 84.7, 67.0, 54.5, 54.0, 27.4, 6.7, 5.7 (one coincidence). HRMS (+CI) calcd for $\text{C}_{30}\text{H}_{36}\text{NO}_{10}\text{Si}$ ($M+1$) 598.2109, found 598.2103.



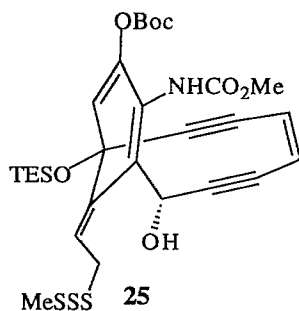
Diol 21. IR (thin film) 3354, 2972, 1762, 1718, 1257, 1151, 1087 cm^{-1} . ^1H NMR (300 MHz, CDCl_3) δ 6.49 (1H, dd, $J = 8.0, 6.0$ Hz), 5.97 (4H, m), 5.80 (1H, s), 4.39 (2H, m), 4.21 (1H, br t, $J = 5.0$ Hz), 3.71 (3H, br s), 1.50 (9H, s), 0.98 (9H, t, $J = 8.0$ Hz), 0.78 (6H, m). ^{13}C NMR (75 MHz, CDCl_3) δ 154.8, 150.6, 128.9, 124.7, 124.5, 124.3, 100.7, 99.3, 88.2, 88.1, 87.0, 84.2, 81.9, 71.2, 63.1, 59.9, 53.0, 27.4, 6.4, 5.6. HRMS (-CI) calcd for $\text{C}_{28}\text{H}_{37}\text{NO}_8\text{Si}$ 543.2288, found 543.2277.

Primary alcohol 23. IR (thin film) 3381, 3263, 2956, 1760, 1710, 1454, 1370, 1253, 1151, 1097, 845, 736 cm^{-1} . ^1H NMR (300 MHz, C_6D_6) δ 6.82 (1H, t, $J = 6.5$ Hz), 6.19 (1H, d, 1.5 Hz), 6.17 (1H, s), 5.56 (1H, d, $J = 9.5$ Hz), 5.48 (1H, br s), 5.47 (1H, dd, $J = 9.5, 1.5$

Hz), 4.45 (1H, br dd, $J = 14.0, 6.5$ Hz), 4.32 (1H, br dd, $J = 14.0, 6.5$ Hz), 3.24 (3H, br s), 1.90 (1H, br s), 1.24 (9H, s), 1.05 (9H, t, $J = 7.5$ Hz), 0.84 (6H, m), 0.25 (9H, s). ^{13}C NMR (75 MHz, CDCl_3) δ 150.6, 142.9, 134.6, 130.6, 129.0, 124.8, 124.2, 101.1, 101.0, 99.5, 87.8, 86.7, 84.0, 71.2, 63.2, 60.3, 52.8, 27.3, 7.0, 6.0, 0.0. HRMS (+CI) calcd for $\text{C}_{31}\text{H}_{46}\text{NO}_8\text{Si}_2$ ($\text{M}+1$) 616.2762, found 616.2776.



Thioacetate 24. Mp 114-114.5 °C (dec). IR (thin film) 3304, 2956, 2877, 1760, 1710, 1694, 1456, 1371, 1253, 1149, 1097, 874, 846, 744 cm^{-1} . ^1H NMR (300 MHz, C_6D_6) δ 6.74 (1H, dd, $J = 8.5, 8.0$ Hz), 6.22 (1H, d, $J = 1.0$ Hz), 5.53 (1H, d, $J = 9.5$ Hz), 5.46 (1H, dd, $J = 9.5, 1.0$ Hz), 5.31 (1H, s), 4.21 (2H, m), 3.30 (3H, br s), 1.82 (3H, s), 1.24 (9H, s), 1.06 (9H, t, $J = 8.0\text{Hz}$), 0.89 (6H, m), 0.32 (9H, s). ^{13}C NMR (75 MHz, CDCl_3) δ 195.6, 150.6, 142.9, 134.9, 126.8, 124.7, 124.6, 124.4, 124.1, 99.4, 87.8, 86.7, 84.0, 71.3, 63.4, 52.8, 30.3, 29.6, 27.4, 6.9, 6.1, 0.1. HRMS (+CI) calcd for $\text{C}_{33}\text{H}_{47}\text{NO}_8\text{Si}_2\text{S}$ 673.2561, found 673.2562.

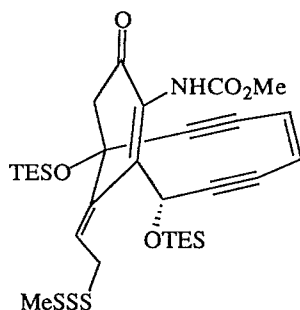


Triethylsilyl-tert-butyloxycarbonyl calicheamicinone 25. IR (thin film) 3355, 2955, 1757, 1714, 1504, 1371, 1252, 1147, 1099, 737 cm^{-1} . ^1H NMR (300 MHz, CDCl_3) δ 6.57 (1H, t, $J = 8.0$ Hz), 6.31 (1H, br s), 5.95 (3H, m), 5.80 (1H, s), 4.03 (2H, m), 3.72 (3H,

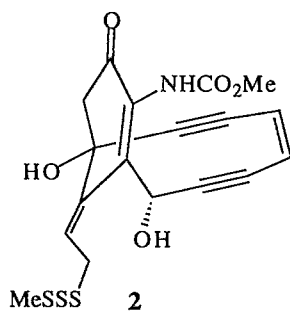
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br s), 2.56 (3H, s), 1.49 (9H, s), 1.02 (9H, t, $J = 7.5$ Hz), 0.82 (6H, m). ^{13}C NMR (75 MHz, CDCl_3) δ HRMS (+CI) calcd for $\text{C}_{29}\text{H}_{40}\text{NO}_7\text{SiS}_3$ 638.1736, found 638.1728.



Bis(triethylsilyl)calicheamicinone. IR (thin film) 3355, 2956, 1741, 1674, 1506, 1234, 1121 cm^{-1} . ^1H NMR (300 MHz, CDCl_3) δ 6.57 (1H, br s), 6.40 (1H, dd, $J = 11.0, 4.5$ Hz), 6.12 (1H, d, $J = 1.5$ Hz), 5.93 (1H, d, $J = 9.5$ Hz), 5.84 (1H, dd, $J = 9.5, 1.5$ Hz), 4.01 (1H, dd, $J = 14.5, 11.0$ Hz), 3.78 (3H, s), 3.63 (1H, dd, $J = 14.5, 4.5$ Hz), 2.55 (3H, s), 1.00 (18H, m), 0.79 (12H, m). HRMS (+CI) calcd for $\text{C}_{30}\text{H}_{45}\text{NO}_5\text{Si}_2\text{S}_3$ 651.1998, found 651.1999.



(±)-Calicheamicinone 2. Mp 93-95 °C (dec). IR (thin film) 3351, 2921, 1713, 1674, 1504, 1248, 1074 cm^{-1} . ^1H NMR (300 MHz, CDCl_3) δ 6.95 (1H, br s), 6.48 (1H, dd, $J = 9.0, 6.5$ Hz), 6.03 (1H, d, $J = 6.5$ Hz), 5.92 (1H, d, $J = 9.5$ Hz), 5.90 (1H, d, $J = 9.5$ Hz), 4.12 (1H, dd, $J = 14.0, 9.5$ Hz), 3.87 (1H, dd, $J = 14.0, 6.5$ Hz), 3.79 (3H, s), 3.26 (1H, d, $J = 6.5$ Hz), 3.21 (1H, d, $J = 17.0$ Hz), 2.85 (1H, d, $J = 17.0$ Hz), 2.73 (1H, s), 2.54 (3H, s). HRMS (+CI) calcd for $\text{C}_{18}\text{H}_{18}\text{NO}_5\text{S}_3$ (M+1) 424.0347, found 424.0338.