

Direct conversion of aromatic 1,3-dioxanes to hydroxypropyl esters with
pyridinium hydrobromide perbromide and sodium acetate in water

Shinsei Sayama*

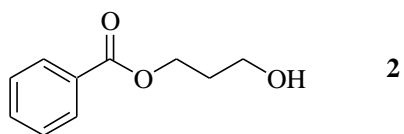
Supporting Information

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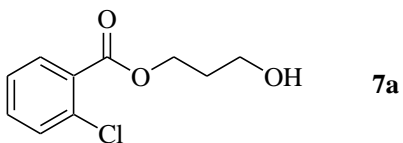
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General Information:

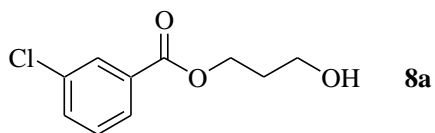
IR spectra were recorded on a Perkin Elmer Spectrum One FT-IR spectrometer. ^1H and ^{13}C NMR spectra were recorded with a JEOL JNM-EX270 spectrometer at room temperature and the chemical shifts (δ) were given relative to internal standard SiMe_4 in CDCl_3 .



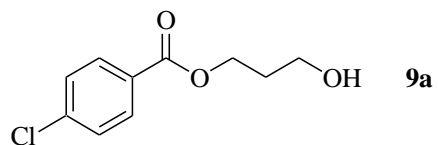
IR (KBr, cm^{-1}) 3420, 2961, 2890, 1716, 1602, 1584, 1452, 1390, 1358, 1316, 1278, 1177, 1120, 1070, 1052, 1027, 971, 927, 711. ^1H NMR(CDCl_3) δ 2.01 (2H, quint, $J=5.4$ Hz), 3.77 (2H, t, $J=5.4$ Hz), 4.48 (2H, t, $J=5.4$ Hz), 7.43 (2H, t, $J=8.1$ Hz), 7.56 (1H, t, $J=8.1$ Hz), 8.03 (2H, d, $J=8.1$ Hz). ^{13}C NMR (CDCl_3) δ 31.80, 59.08, 61.79, 128.33, 129.54, 130.04, 132.99, 166.97.



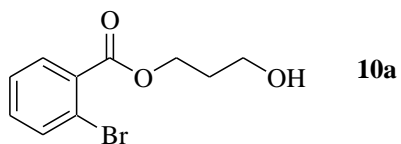
IR (neat, cm^{-1}) 3401, 2961, 2892, 1727, 1592, 1571, 1472, 1436, 1389, 1358, 1295, 1253, 1163, 1138, 1123, 1051, 969, 924, 844, 792, 748, 719, 690, 649. ^1H NMR (CDCl_3) δ 2.02 (2H, quint, $J=5.9$ Hz), 3.80 (2H, t, $J=5.9$ Hz), 4.50 (2H, t, $J=5.9$ Hz), 7.28-7.34 (1H, m), 7.38-7.47 (2H, m), 7.82 (1H, dd, $J=6.8, 2.0$ Hz). ^{13}C NMR(CDCl_3) δ 31.66, 59.28, 62.51, 126.59, 130.20, 131.05, 131.35, 132.56, 133.58, 166.16.



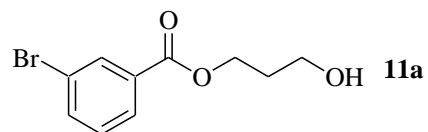
IR (neat, cm^{-1}) 3401, 3072, 2961, 2889, 1722, 1597, 1574, 1471, 1427, 1390, 1358, 1325, 1292, 1259, 1133, 1085, 1073, 1052, 972, 930, 900, 860, 810, 748, 674, 656. ^1H NMR (CDCl_3) δ 2.04 (2H, quint, $J=6.4$ Hz), 3.77 (2H, t, $J=6.4$ Hz), 4.50 (2H, t, $J=6.4$ Hz), 7.37 (1H, t, $J=7.8$ Hz), 7.53 (1H, dd, $J=7.8, 1.3$ Hz), 7.92 (1H, dd, $J=7.8, 1.3$ Hz), 8.01 (1H, brs). ^{13}C NMR (CDCl_3) δ 31.79, 59.12, 62.19, 127.72, 129.66, 129.72, 131.86, 133.06, 134.57, 166.10.



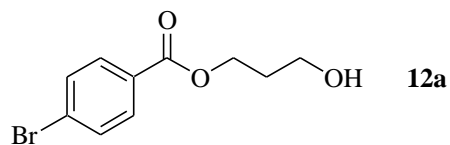
IR (neat, cm^{-1}) 3416, 2960, 2888, 1720, 1595, 1488, 1469, 1402, 1359, 1325, 1275, 1172, 1121, 1105, 1092, 1052, 1015, 970, 926, 850, 759, 725, 685. ^1H NMR (CDCl_3) δ 2.01 (2H, quint, $J=6.2$ Hz), 3.77 (2H, t, $J=6.2$ Hz), 4.48 (2H, t, $J=6.2$ Hz), 7.41 (2H, d, $J=8.6$ Hz), 7.97 (2H, d, $J=8.6$ Hz). ^{13}C NMR (CDCl_3) δ 31.80, 59.10, 62.03, 128.53, 128.73, 130.96, 139.49, 166.09.



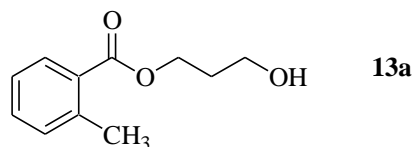
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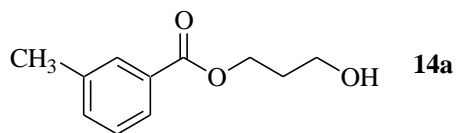
IR (neat, cm^{-1}) 3405, 3069, 2960, 2889, 1721, 1593, 1569, 1471, 1424, 1390, 1358, 1325, 1291, 1259, 1165, 1128, 1101, 1067, 1051, 1000, 971, 928, 901, 854, 810, 746, 715, 672, 650. ^1H NMR (CDCl_3) δ 2.01 (2H, quint, $J=6.4$ Hz), 3.78 (2H, t, $J=6.4$ Hz), 4.49 (2H, t, $J=6.4$ Hz), 7.32 (1H, t, $J=7.8$ Hz), 7.68 (1H, dd, $J=7.8, 1.3$ Hz), 7.96 (1H, dd, $J=7.8, 1.3$ Hz), 8.16 (1H, brs). ^{13}C NMR(CDCl_3) δ 31.80, 59.12, 62.19, 122.48, 128.19, 129.97, 132.59, 135.97, 165.60.



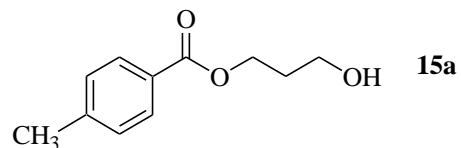
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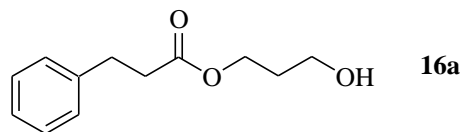
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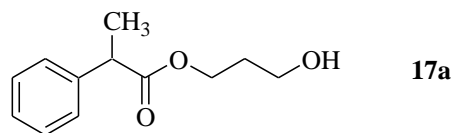
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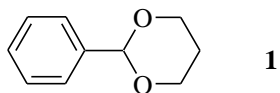
IR (neat, cm^{-1}) 3419, 3036, 2959, 2888, 1715, 1612, 1577, 1509, 1455, 1407, 1389, 1358, 1310, 1277, 1208, 1178, 1112, 1052, 1020, 971, 927, 841, 754, 691, 637. ^1H NMR (CDCl_3) δ 2.00 (2H, quint, $J=6.2$ Hz), 2.40 (3H, s), 3.77 (2H, t, $J=6.2$ Hz), 4.37 (2H, t, $J=6.2$ Hz), 7.25 (2H, d, $J=7.8$ Hz), 7.92 (2H, d, $J=7.8$ Hz). ^{13}C NMR (CDCl_3) δ 21.63, 31.93, 59.17, 61.56, 127.31, 129.09, 129.61, 143.73, 167.09.



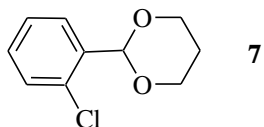
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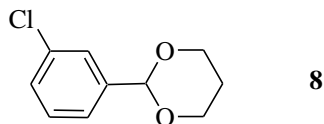
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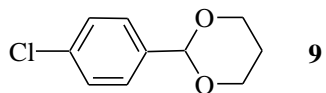
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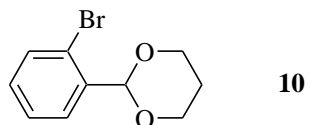
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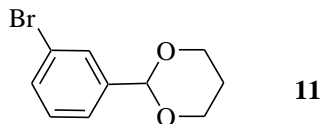
IR (neat, cm^{-1}) 2969, 2855, 1603, 1578, 1468, 1430, 1377, 1278, 1237, 1210, 1146, 1109, 1018, 996, 961, 873, 856, 785, 726, 686. ^1H NMR (CDCl_3) δ 1.45 (1H, d, $J=13.5$ Hz), 2.21 (1H, m), 3.97 (2H, t, $J=13.5$ Hz), 4.26 (2H, m), 5.46 (1H, s), 7.31 (3H, m), 7.50 (1H, brs). ^{13}C NMR (CDCl_3) δ 25.62, 67.31, 100.49, 124.19, 126.33, 128.81, 129.48, 134.13, 140.55.



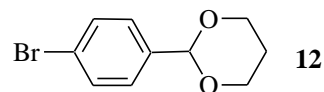
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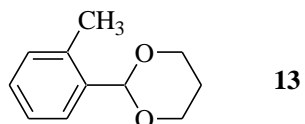
IR (neat, cm^{-1}) 3086, 2976, 2923, 2864, 1594, 1570, 1477, 1458, 1435, 1421, 1386, 1379, 1370, 1341, 1289, 1269, 1234, 1208, 1158, 1145, 1120, 1096, 1045, 1027, 1007, 988, 959, 948, 925, 894, 877, 863, 842, 761, 682, 642. ^1H NMR (CDCl_3) δ 1.43 (1H, d, $J=13.5$ Hz), 2.23 (1H, m), 4.01 (2H, t, $J=13.5\text{Hz}$), 4.27 (2H, m), 5.76 (1H, s), 7.18 (1H, t, $J=8.1$ Hz), 7.33 (1H, t, $J=8.1$ Hz), 7.52 (1H, d, $J=8.1$ Hz), 7.69 (1H, d, $J=8.1$ Hz). ^{13}C NMR (CDCl_3) δ 25.62, 67.50, 100.82, 122.23, 127.47, 128.00, 130.24, 132.52, 137.42.



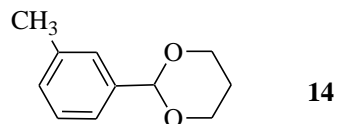
IR (neat, cm^{-1}) 2968, 2854, 1572, 1469, 1428, 1376, 1277, 1237, 1207, 1146, 1107, 1070, 1018, 993, 960, 894, 868, 853, 782, 706, 679, 640. ^1H NMR (CDCl_3) δ 1.43 (1H, d, $J=13.5$ Hz), 2.20 (1H, m), 3.96 (2H, t, $J=13.5\text{Hz}$), 4.25 (2H, m), 5.45 (1H, s), 7.22 (1H, t, $J=8.1$ Hz), 7.39 (1H, d, $J=8.1$ Hz), 7.45 (1H, d, $J=8.1$ Hz), 7.65 (1H, brs). ^{13}C NMR (CDCl_3) δ 25.62, 67.32, 100.41, 122.31, 124.67, 129.24, 129.77, 131.76, 140.77.



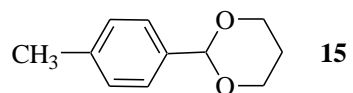
IR (neat, cm^{-1}) 2969, 2954, 2927, 2864, 1595, 1575, 1491, 1470, 1463, 1399, 1384, 1340, 1277, 1238, 1211, 1150, 1108, 1066, 1012, 991, 966, 944, 929, 898, 847, 804, 755, 638. ^1H NMR (CDCl_3) δ 1.42 (1H, d, $J=13.5$ Hz), 2.19 (1H, m), 3.96 (2H, t, $J=13.5$ Hz), 4.25 (2H, m), 5.44 (1H, s), 7.35 (2H, d, $J=8.1$ Hz), 7.48 (2H, d, $J=8.1$ Hz). ^{13}C NMR (CDCl_3) δ 25.62, 67.30, 100.73, 122.73, 127.76, 131.43, 137.72.



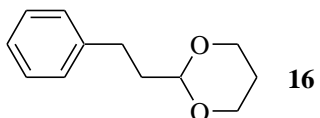
IR (neat, cm^{-1}) 3029, 2964, 2927, 2851, 2724, 1609, 1489, 1464, 1428, 1394, 1377, 1345, 1277, 1236, 1186, 1147, 1121, 1098, 1051, 1036, 1010, 988, 960, 947, 926, 893, 865, 848, 790, 755, 722, 643. ^1H NMR (CDCl_3) δ 1.43 (1H, d, $J=13.5$ Hz), 2.23 (1H, m), 2.39 (3H, s), 3.97 (2H, t, $J=13.5$ Hz), 4.26 (2H, m), 5.60 (1H, s), 7.12-7.24 (3H, m), 7.57 (1H, dd, $J=5.4, 2.7$ Hz). ^{13}C NMR (CDCl_3) δ 18.73, 25.74, 67.46, 100.13, 125.78, 125.80, 128.59, 130.34, 135.55, 136.46.



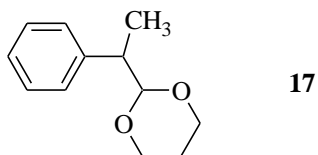
IR (neat, cm^{-1}) 2964, 2923, 2852, 2723, 1613, 1467, 1428, 1377, 1277, 1237, 1163, 1146, 1106, 1026, 999, 960, 934, 900, 860, 793, 767, 698. ^1H NMR (CDCl_3) δ 1.43 (1H, d, $J=13.5$ Hz), 2.23 (1H, m), 2.35 (3H, s), 3.97 (2H, t, $J=13.5$ Hz), 4.26 (2H, m), 5.47 (1H, s), 7.14 (1H, m), 7.25 (2H, m), 7.32 (1H, brs). ^{13}C NMR (CDCl_3) δ 21.34, 25.73, 67.36, 101.75, 123.06, 126.48, 128.14, 129.51, 137.93, 138.56.



IR (neat, cm^{-1}) 2964, 2925, 2852, 2724, 1619, 1519, 1468, 1428, 1379, 1311, 1277, 1237, 1218, 1178, 1153, 1104, 1021, 990, 947, 926, 893, 872, 849, 810, 777. ^1H NMR (CDCl_3) δ 1.42 (1H, d, $J=13.5$ Hz), 2.20 (1H, m), 2.33 (3H, s), 3.96 (2H, t, $J=13.5$ Hz), 4.24 (2H, m), 5.46 (1H, s), 7.15 (2H, d, $J=8.1$ Hz), 7.35 (2H, d, $J=8.1$ Hz). ^{13}C NMR (CDCl_3) δ 21.18, 25.72, 67.30, 101.66, 125.82, 128.84, 135.87, 138.43.

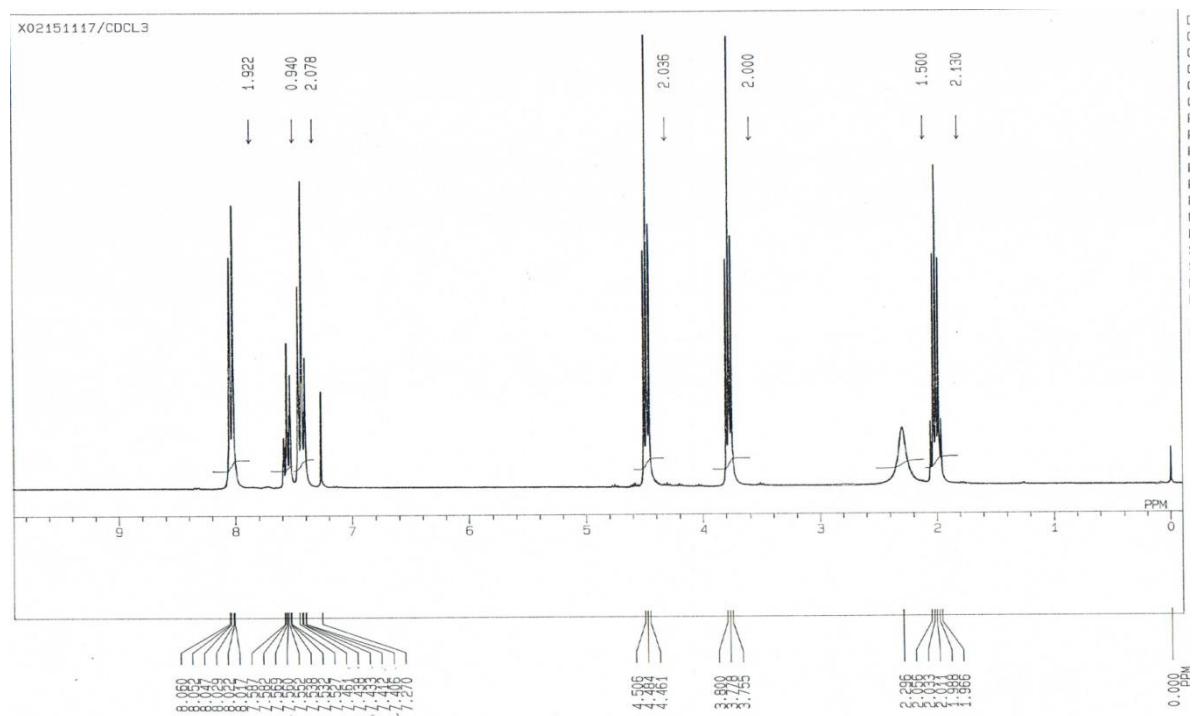
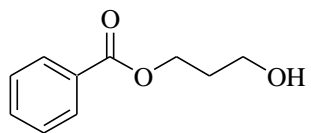


IR (neat, cm^{-1}) 3085, 3062, 3026, 2959, 2928, 2851, 2730, 2664, 1603, 1496, 1469, 1455, 1430, 1404, 1378, 1286, 1258, 1240, 1216, 1195, 1133, 1087, 1031, 1004, 970, 947, 926, 910, 884, 851, 814, 748, 700. ^1H NMR (CDCl_3) δ 1.33 (1H, d, $J=13.5$ Hz), 1.91 (2H, m), 2.09 (1H, m), 2.72 (2H, t, $J=8.1$ Hz), 3.74 (2H, t, $J=13.5$ Hz), 4.11 (2H, m), 4.51 (1H, t, $J=5.4$ Hz), 7.18-7.30 (5H, m). ^{13}C NMR (CDCl_3) δ 25.81, 30.07, 36.60, 66.84, 101.42, 125.77, 128.32, 128.42, 141.69.

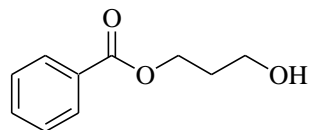


IR (neat, cm^{-1}) 3086, 3062, 3029, 2967, 2934, 2850, 2728, 1600, 1494, 1453, 1429, 1379, 1265, 1238, 1216, 1144, 1101, 1026, 1008, 975, 919, 892, 858, 762, 700. ^1H NMR (CDCl_3) δ 1.26-1.31 (4H, m), 2.04 (1H, m), 2.92 (1H, quint, $J=5.4$ Hz), 3.70 (2H, q $J=13.5$ Hz), 4.07 (2H, m), 4.58 (1H, d, $J=5.4$ Hz), 7.20-7.32 (5H, m). ^{13}C NMR (CDCl_3) δ 16.13, 25.77, 44.75, 67.02, 67.05, 104.94, 126.33, 128.14, 142.92.

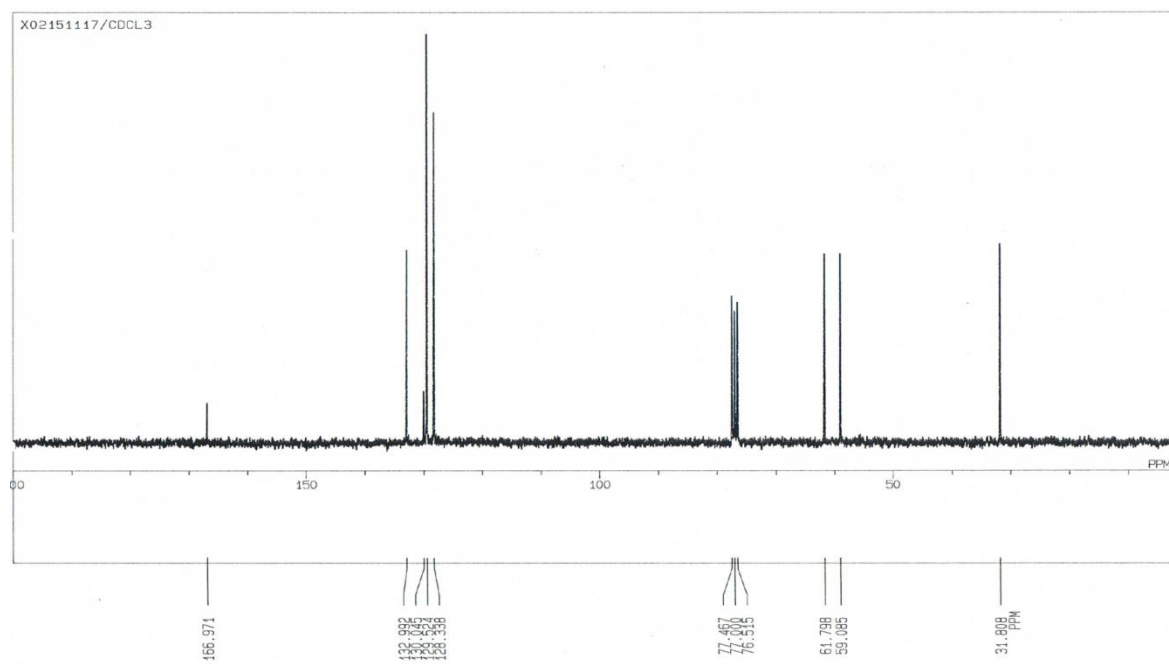
^1H NMR (CDCl_3 , 270.05 MHz)



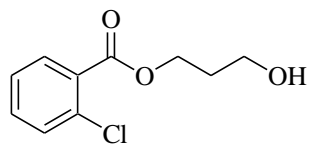
^{13}C NMR (CDCl_3 , 67.80 MHz)



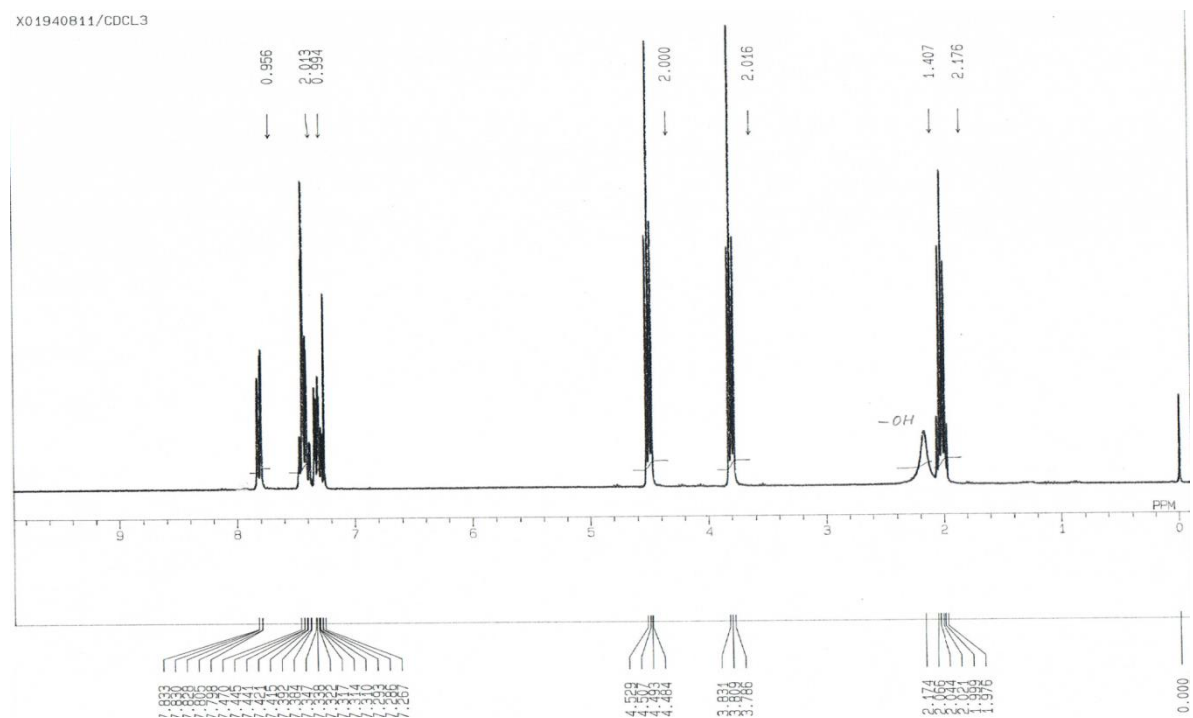
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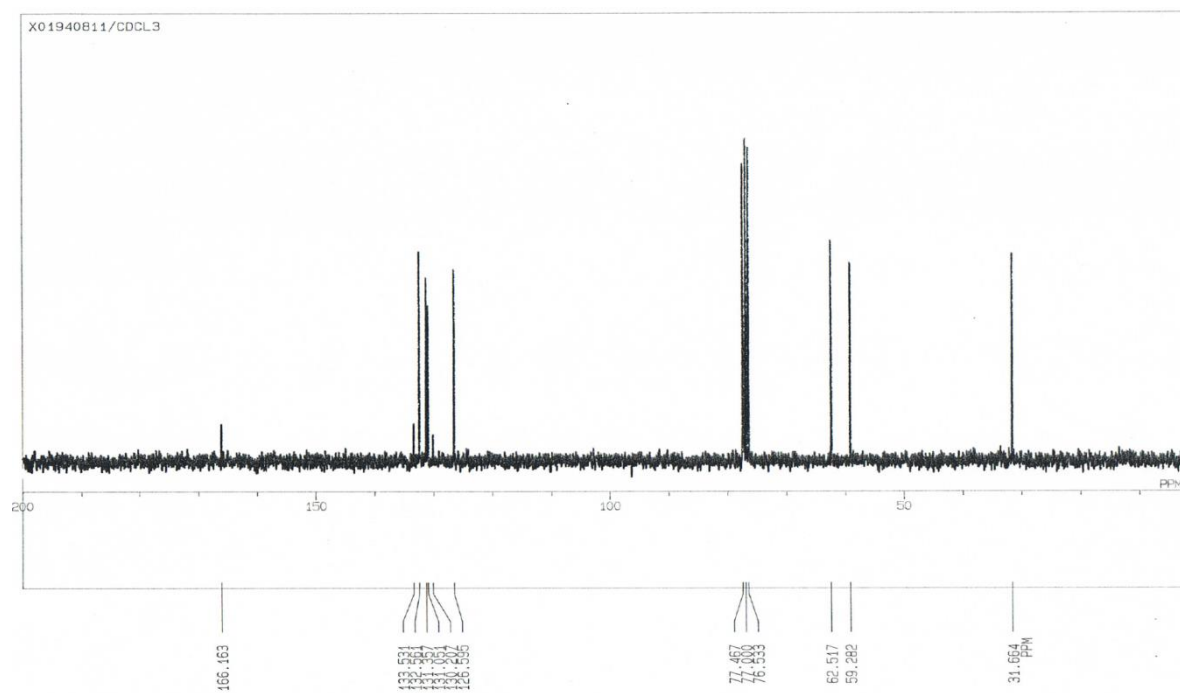
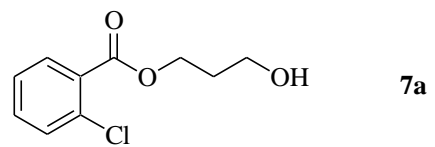
^1H NMR (CDCl_3 , 270.05 MHz)



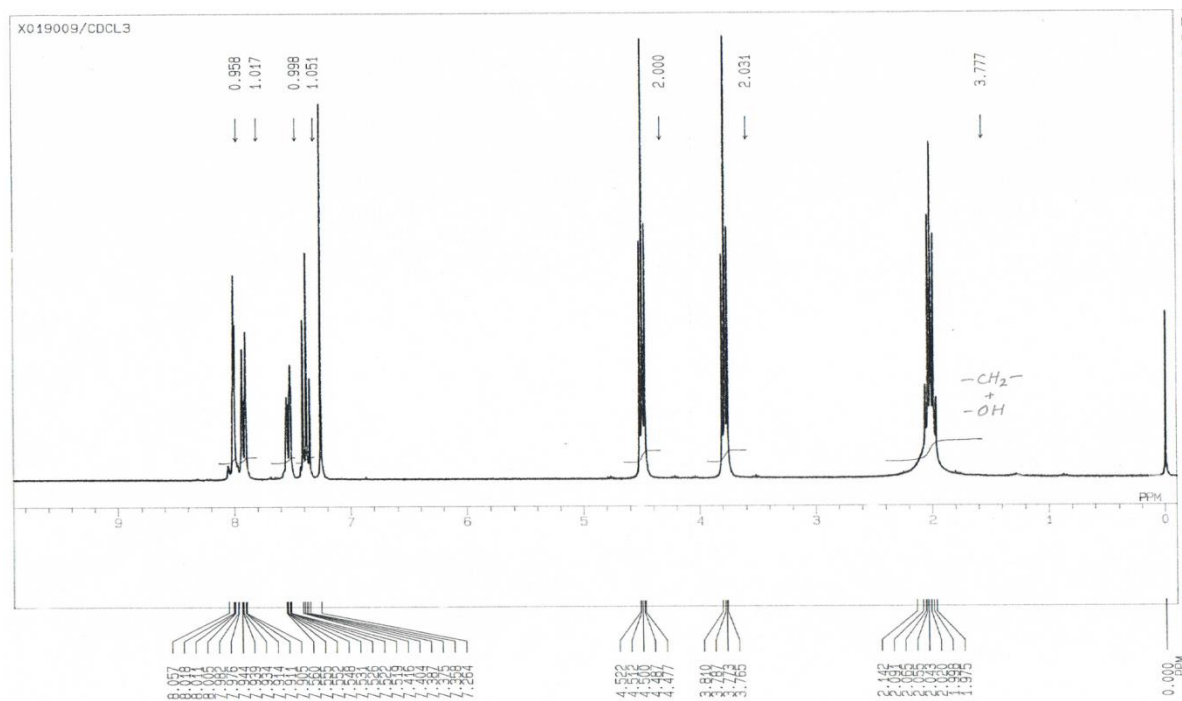
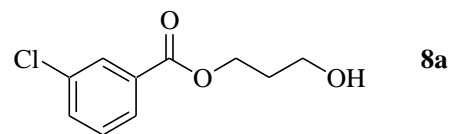
7a



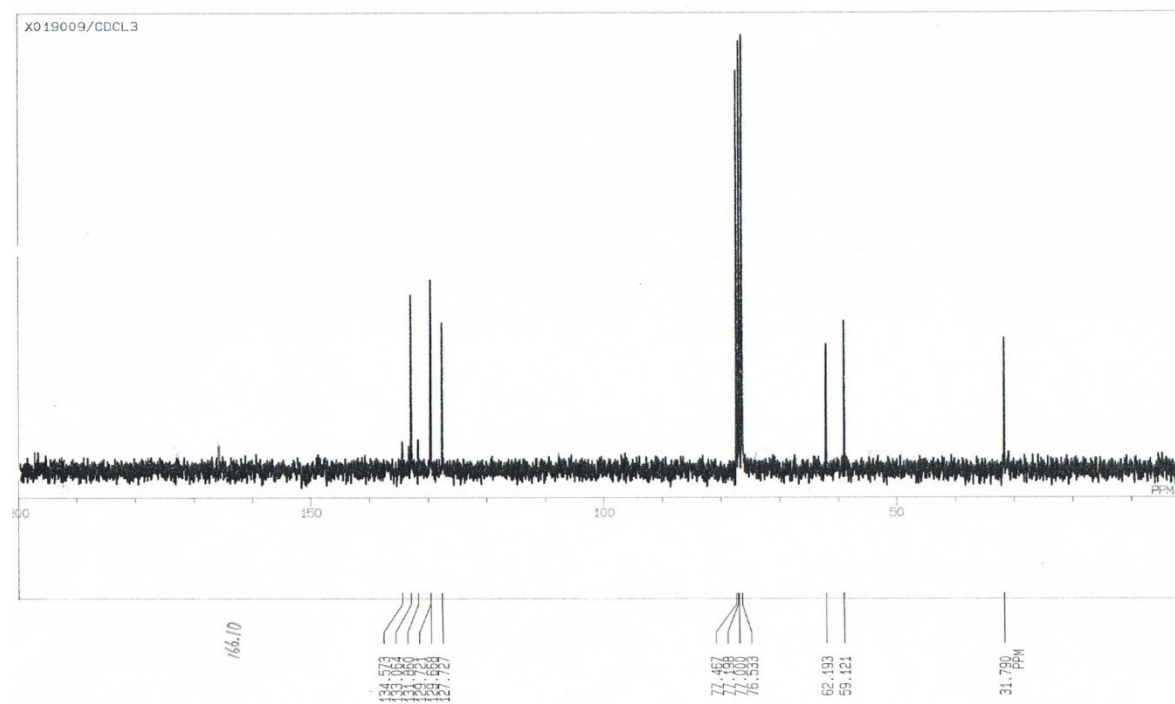
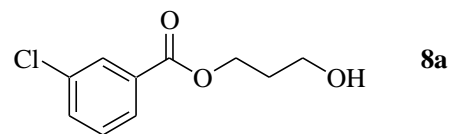
^{13}C NMR (CDCl_3 , 67.80 MHz)



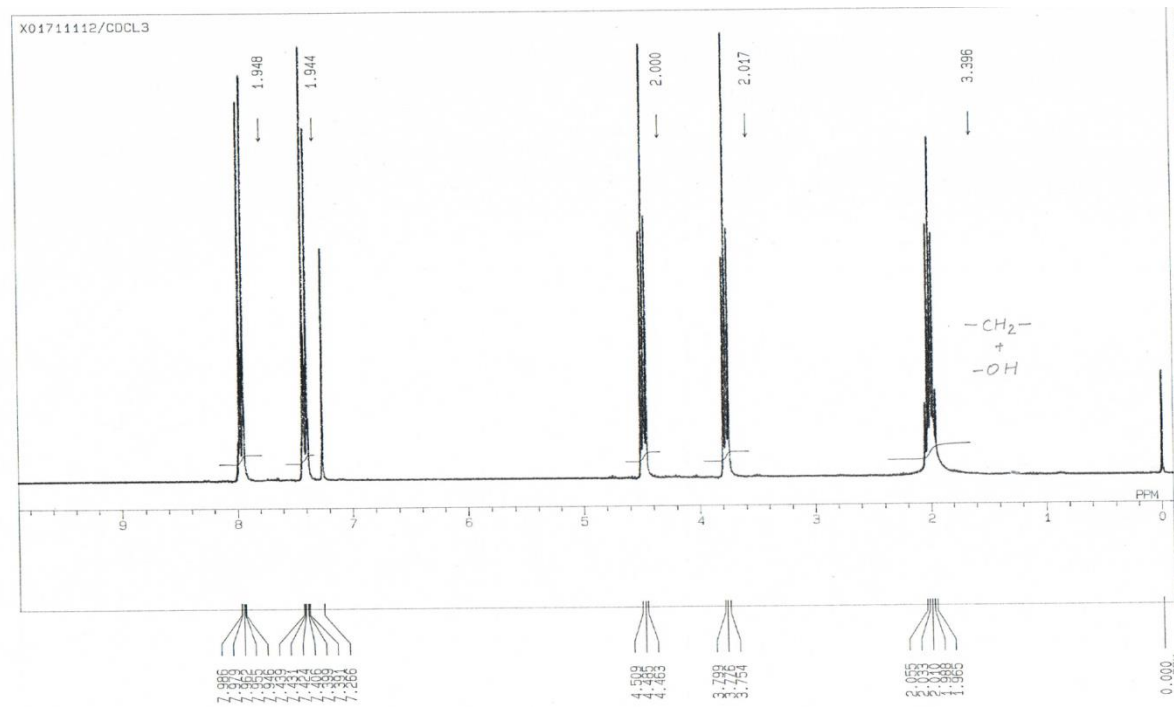
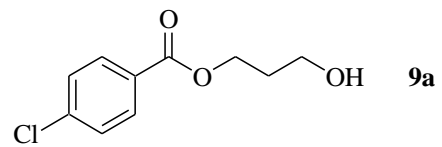
^1H NMR (CDCl_3 , 270.05 MHz)



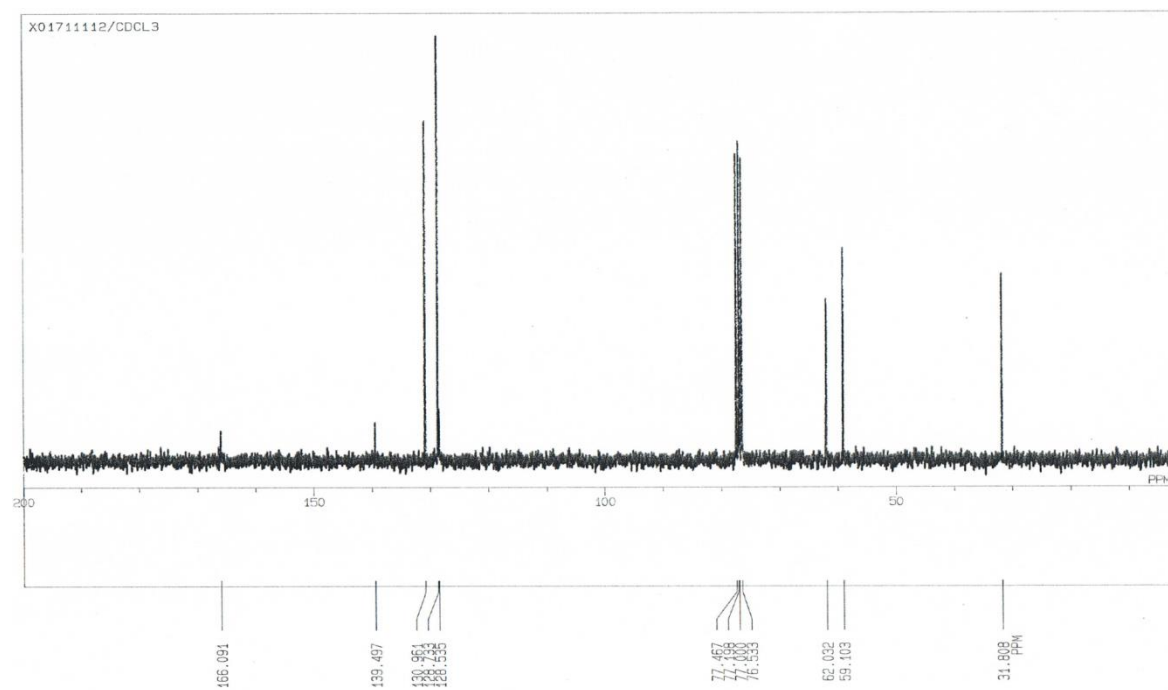
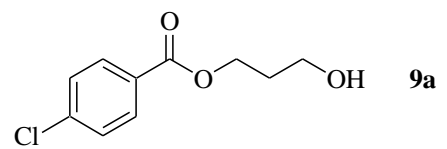
^{13}C NMR (CDCl_3 , 67.80 MHz)



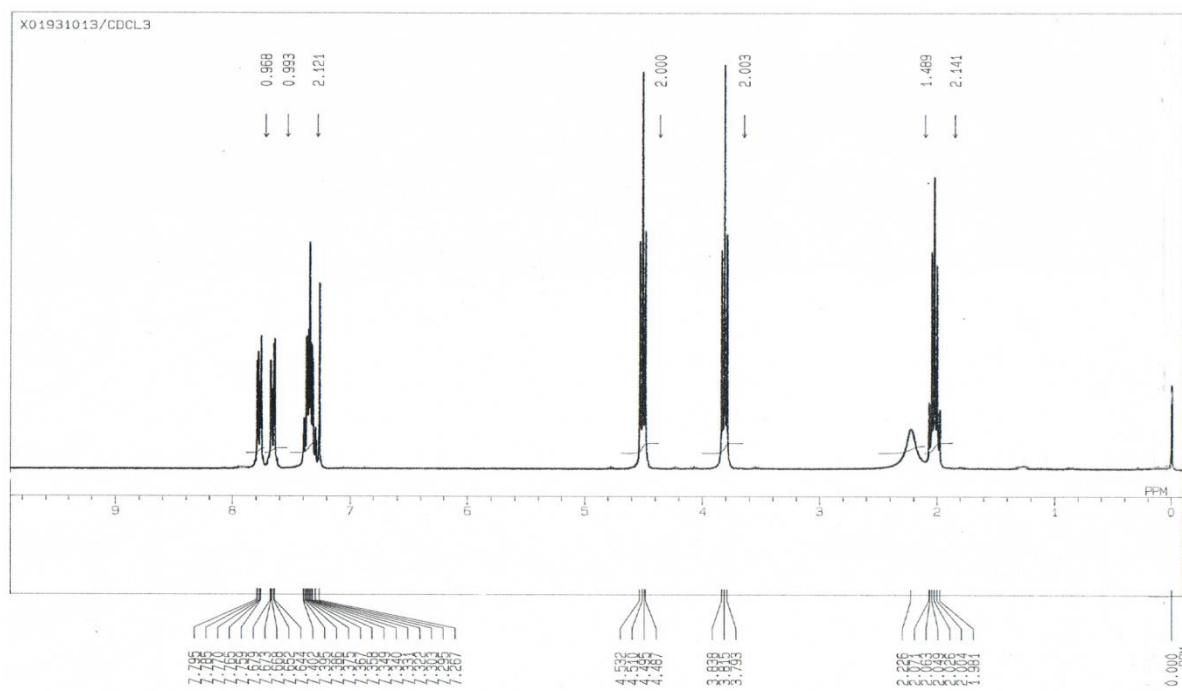
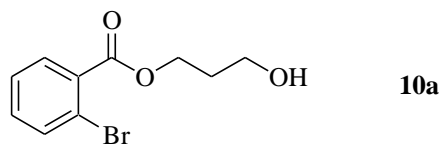
^1H NMR (CDCl_3 , 270.05 MHz)



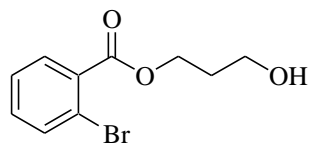
^{13}C NMR (CDCl_3 , 67.80 MHz)



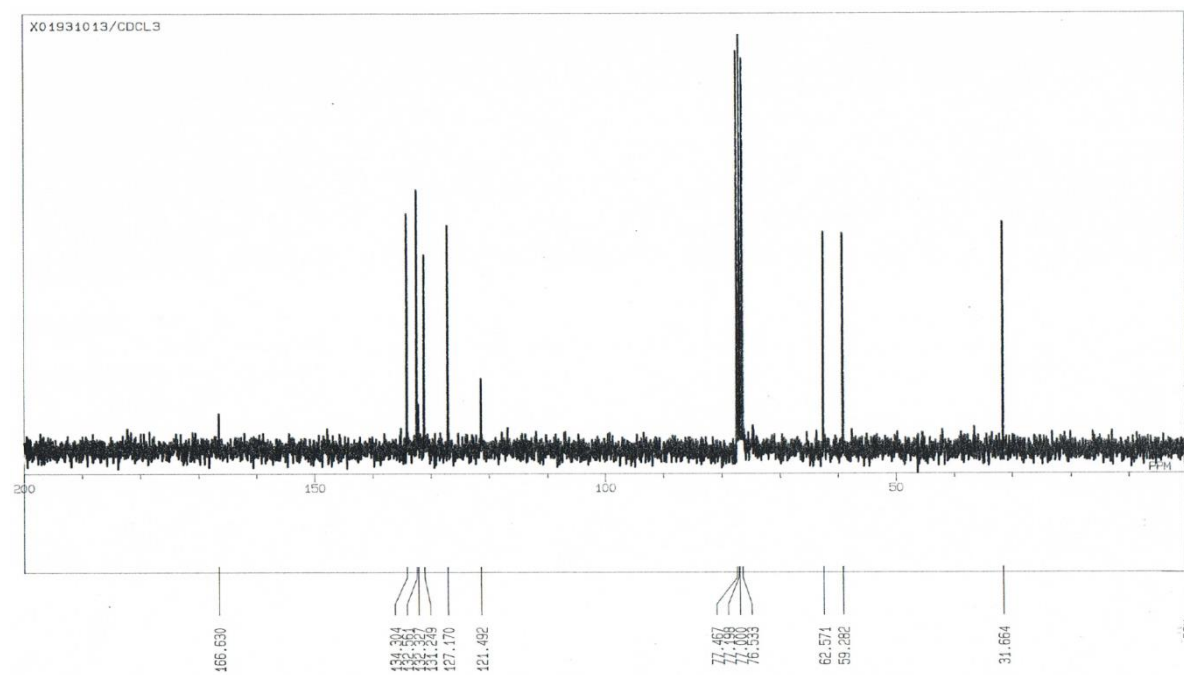
^1H NMR (CDCl_3 , 270.05 MHz)



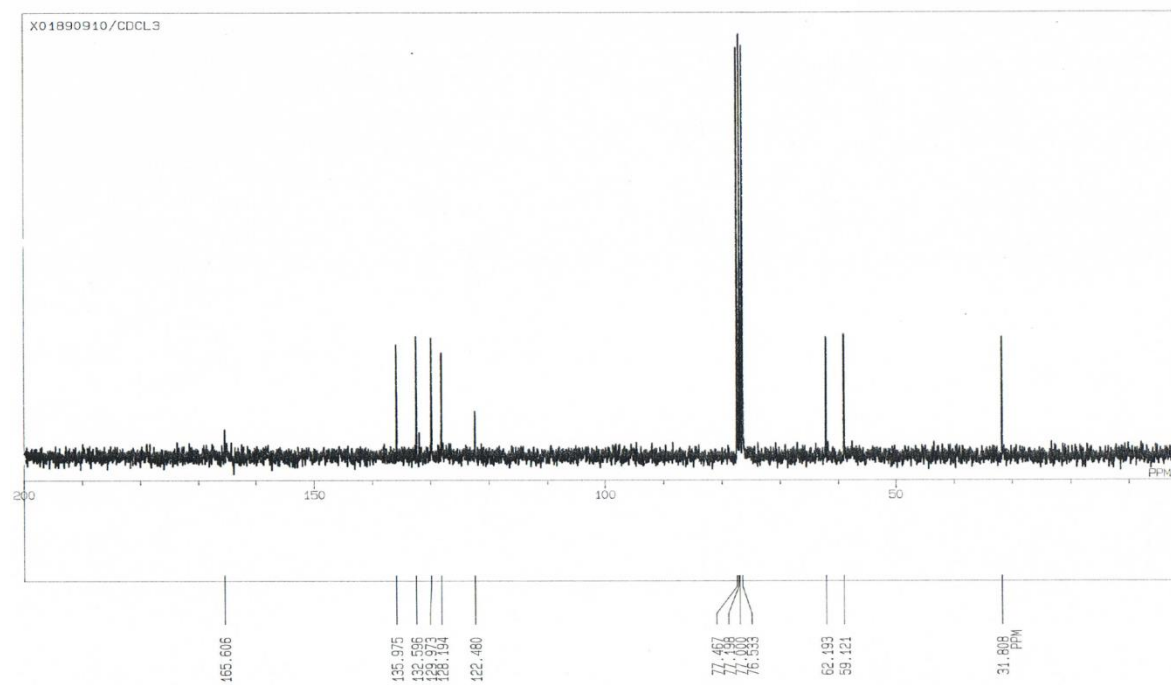
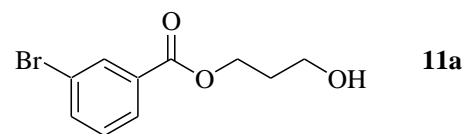
^{13}C NMR (CDCl_3 , 67.80 MHz)



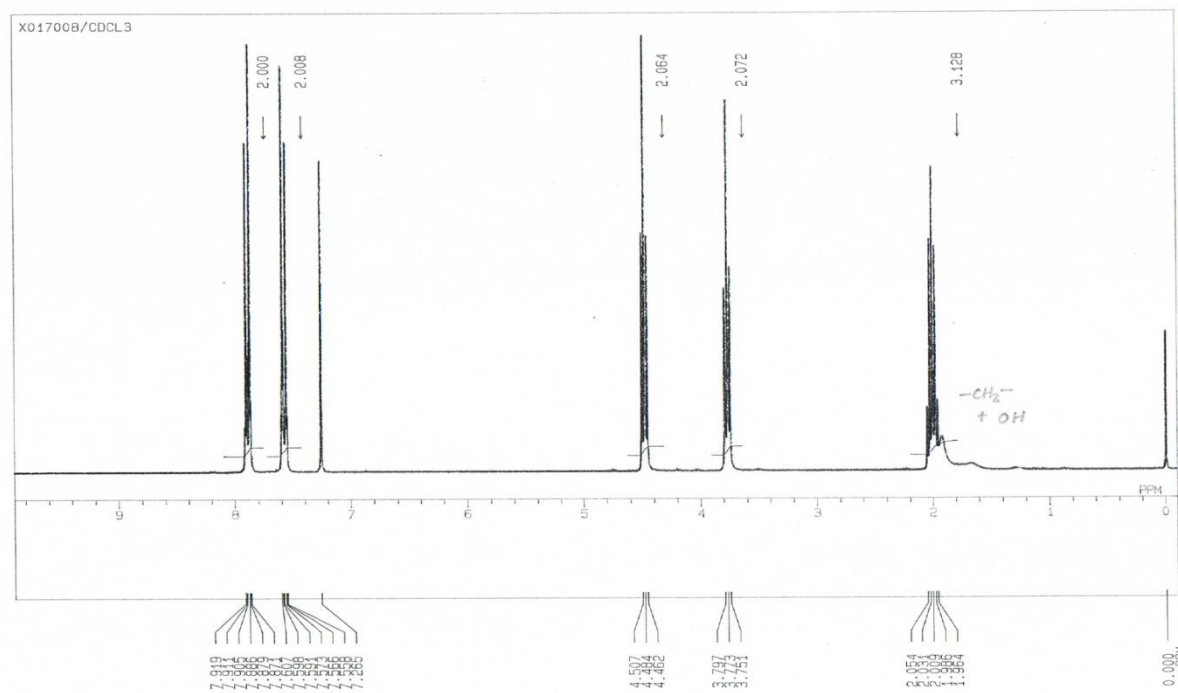
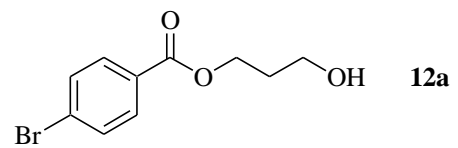
10a



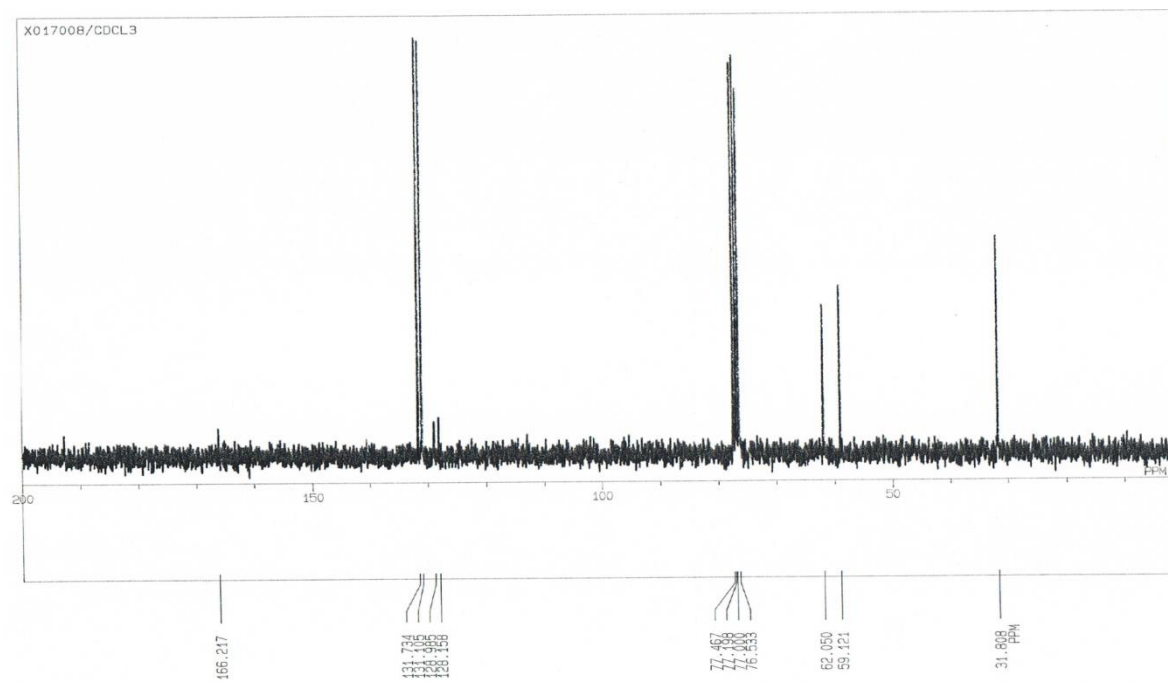
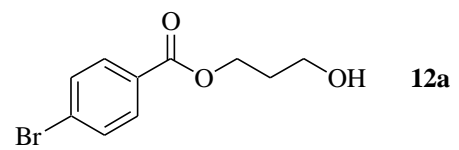
^{13}C NMR (CDCl_3 , 67.80 MHz)



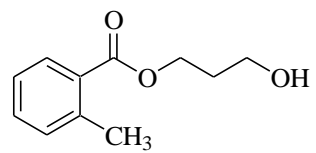
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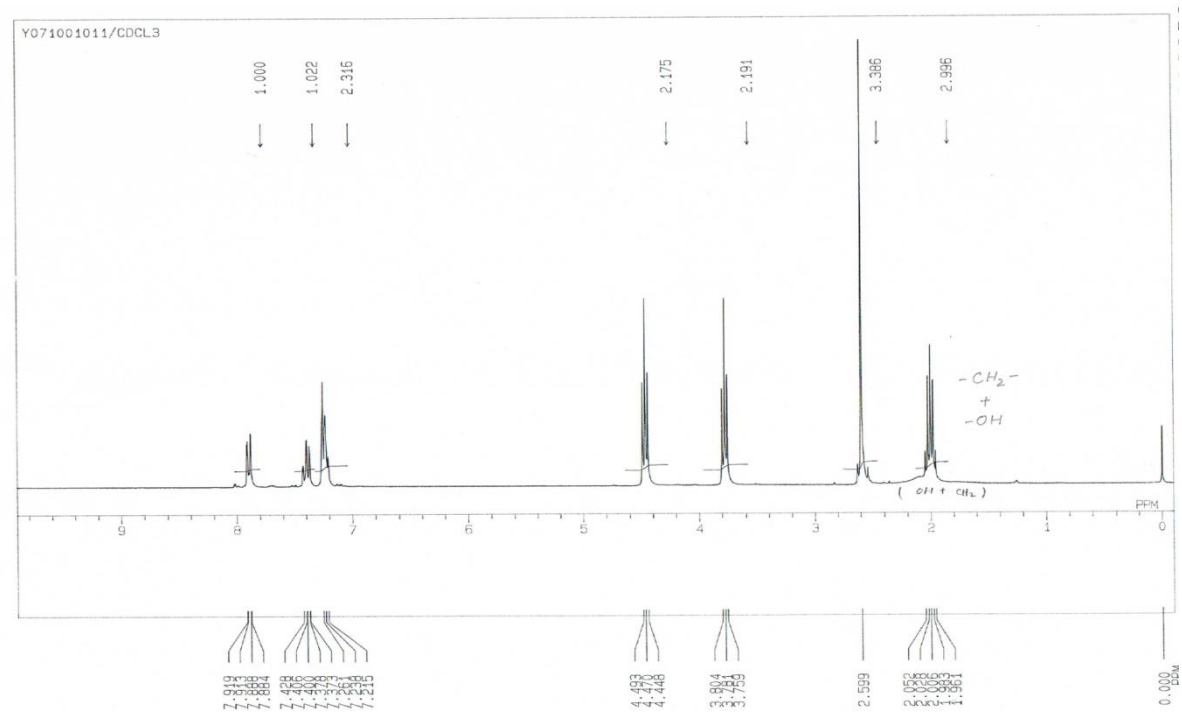
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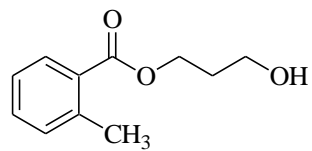
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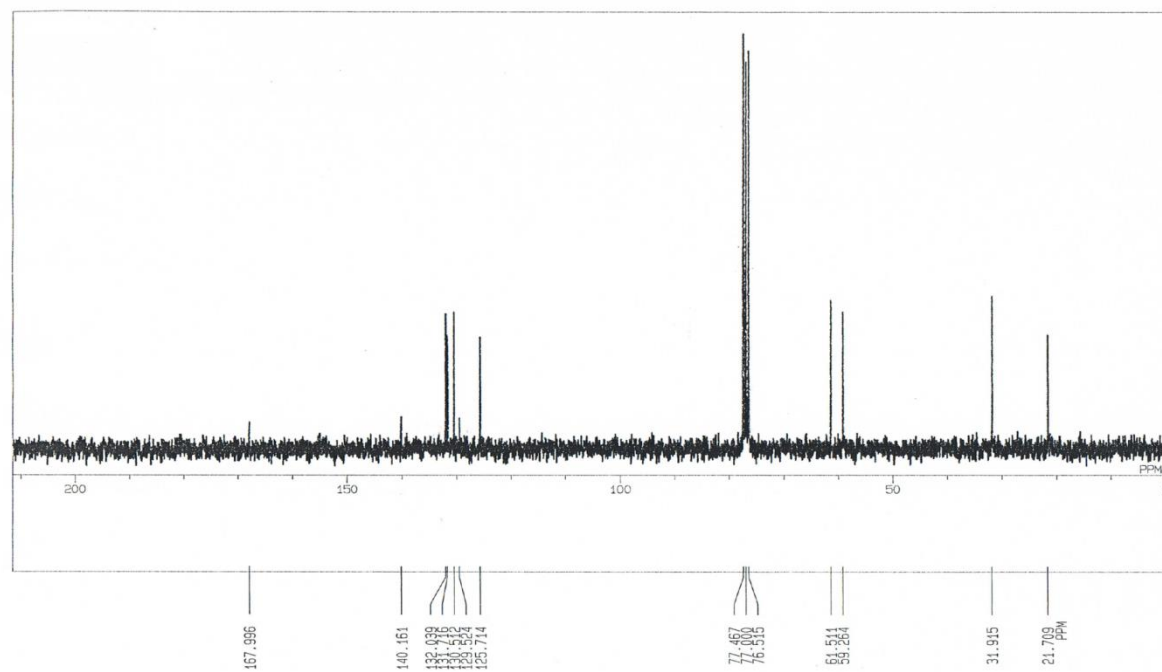
13a



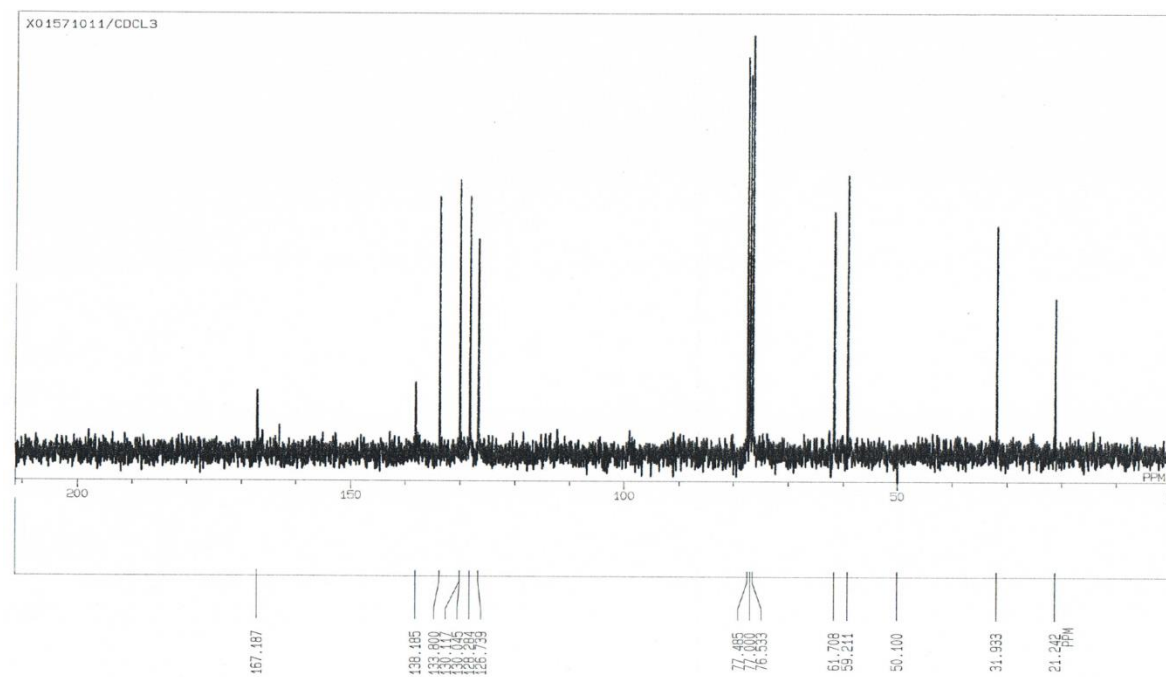
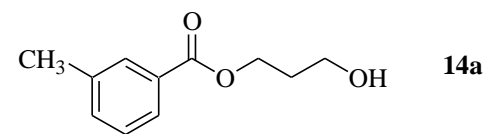
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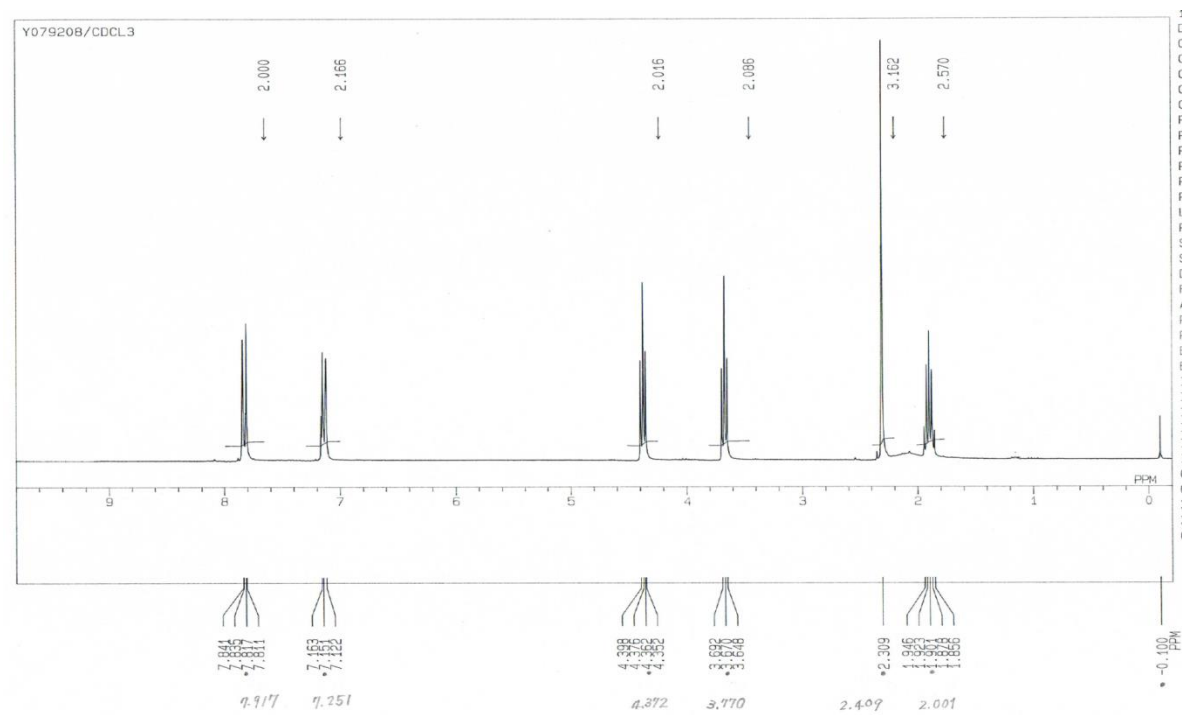
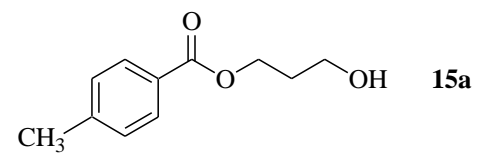
13a



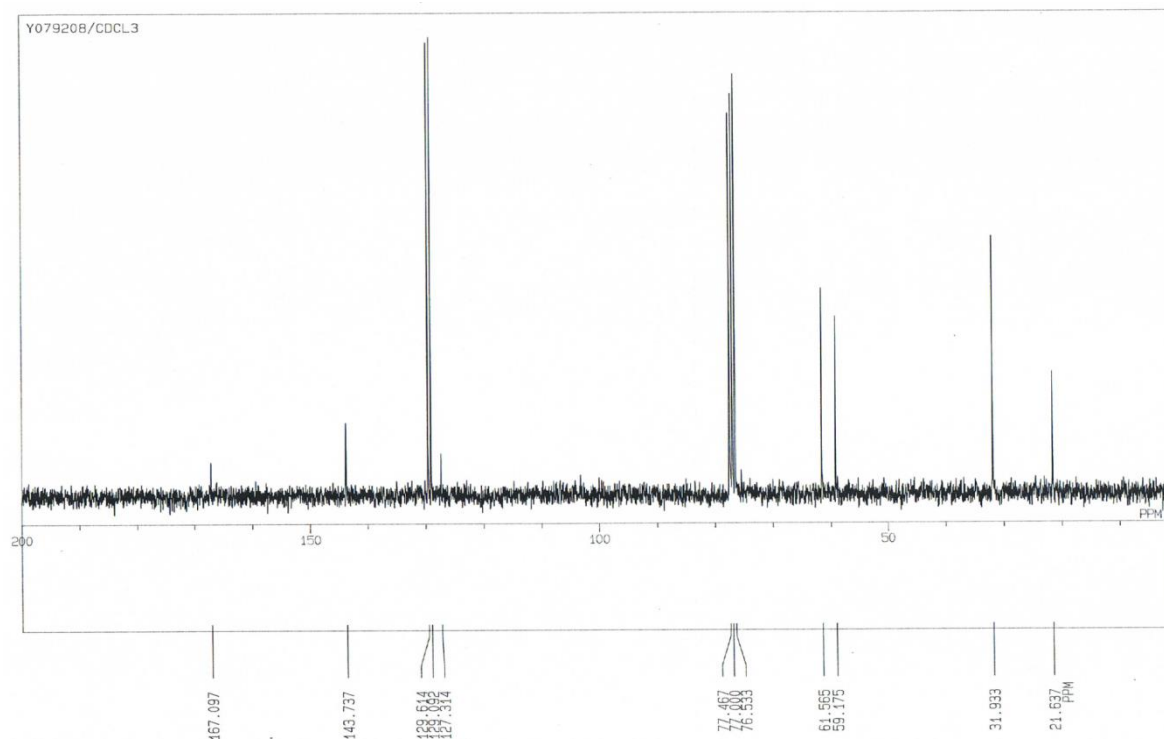
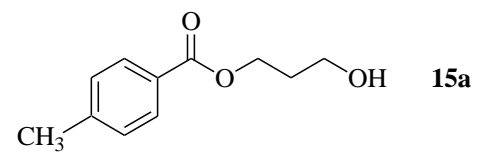
^{13}C NMR (CDCl_3 , 67.80 MHz)



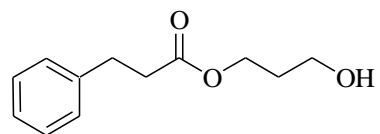
^1H NMR (CDCl_3 , 270.05 MHz)



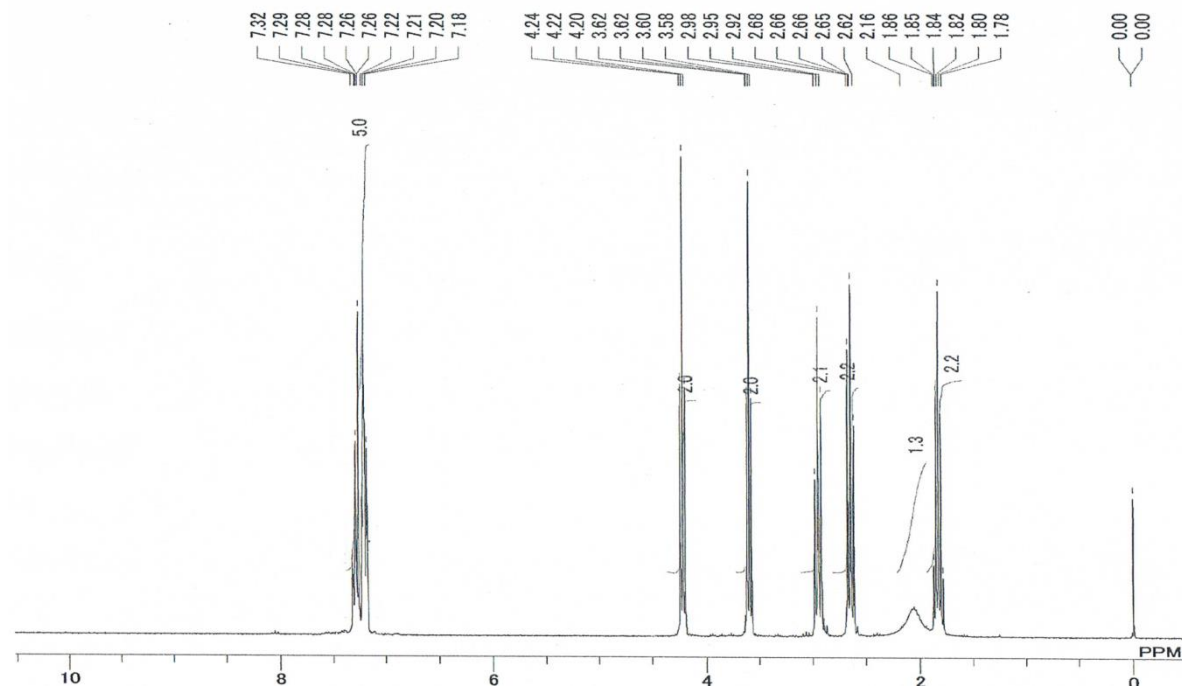
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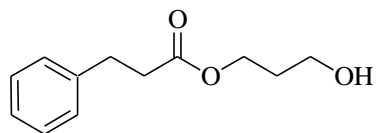
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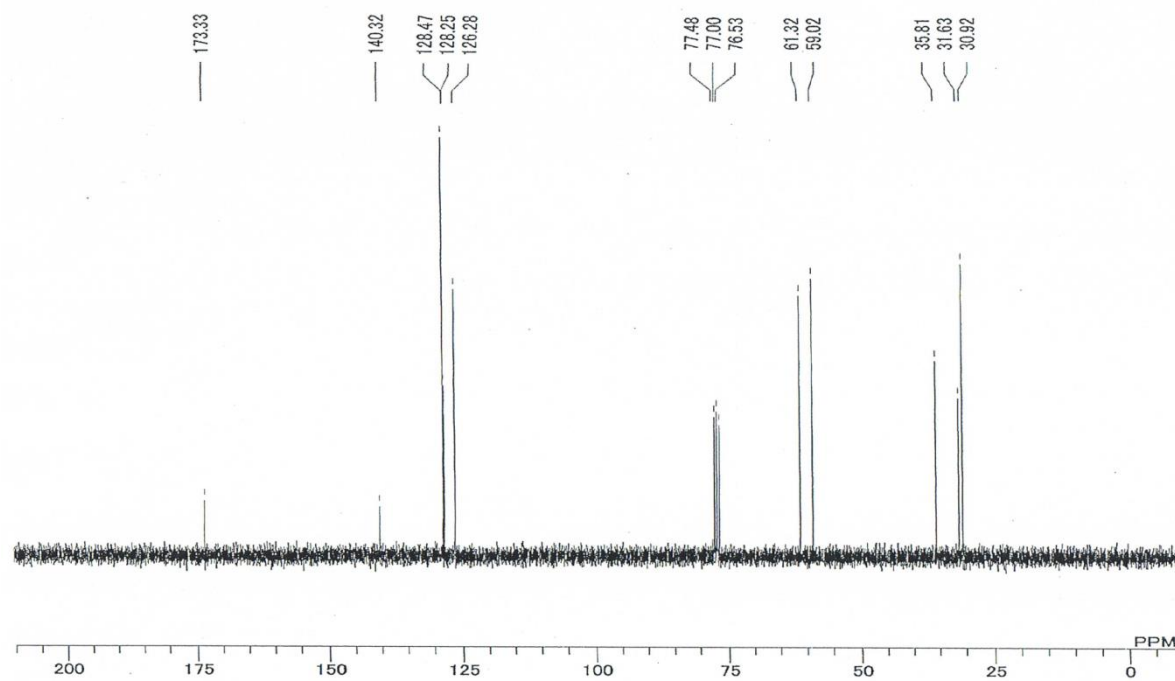
16a



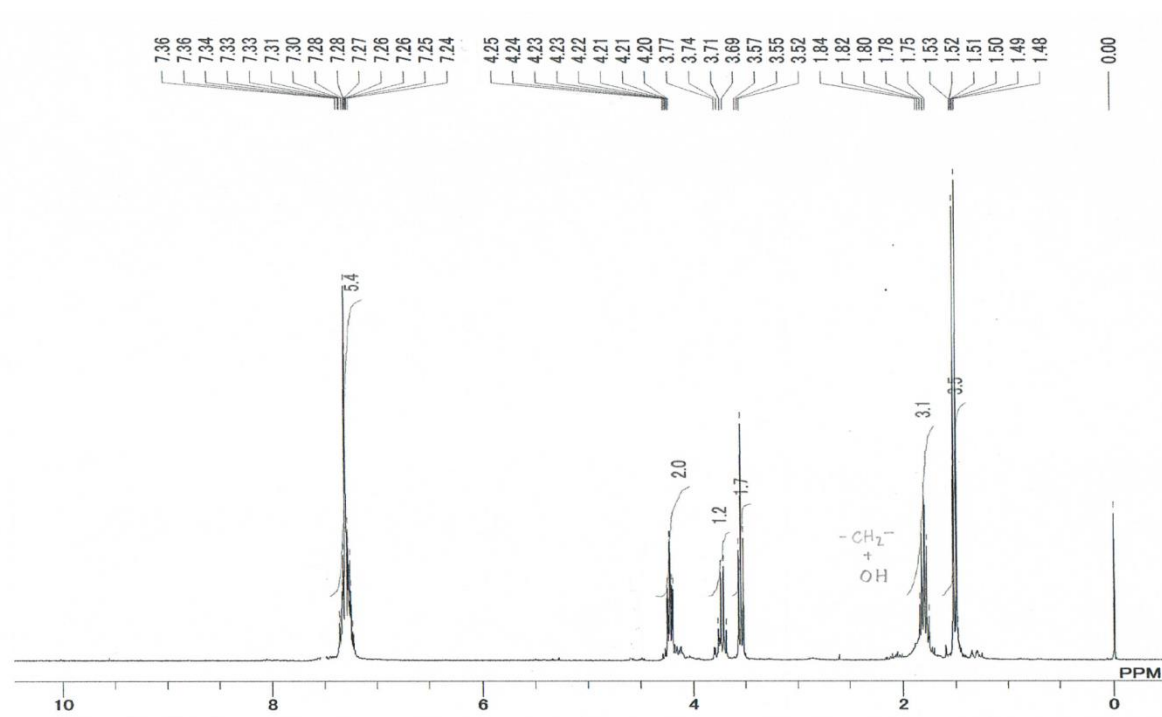
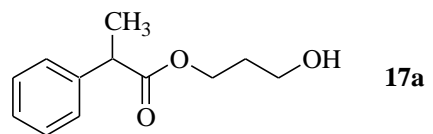
^{13}C NMR (CDCl_3 , 67.80 MHz)



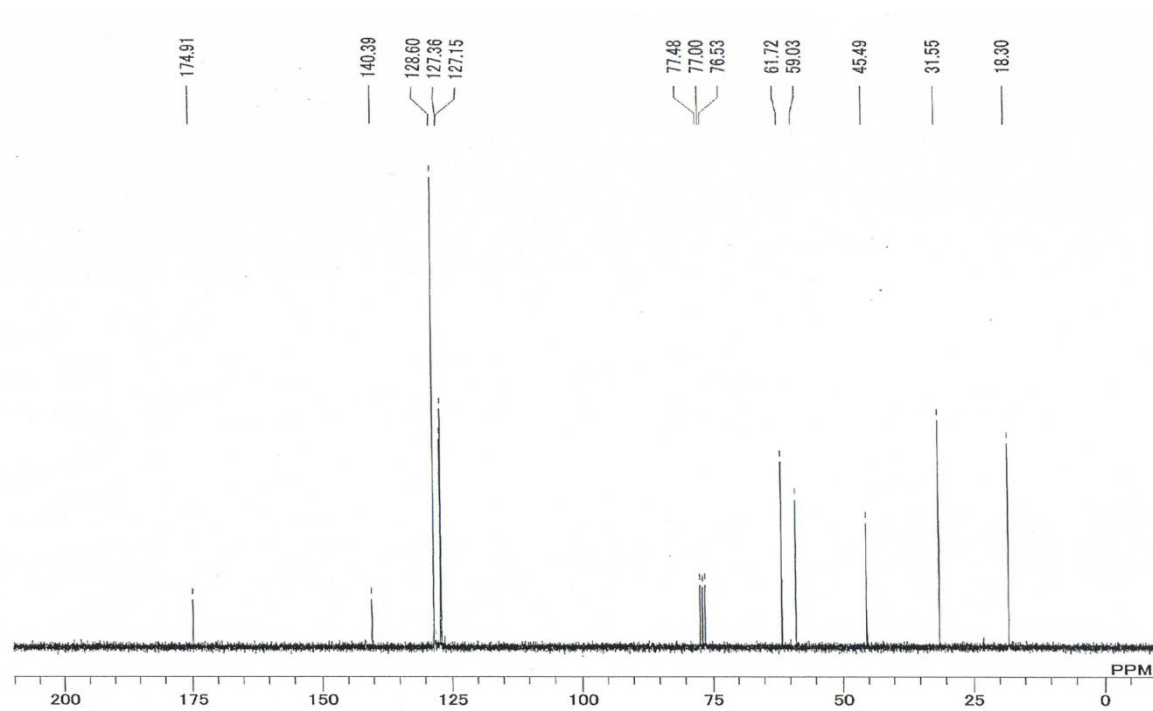
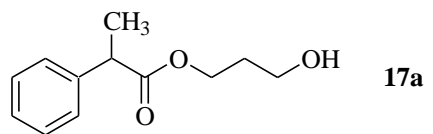
16a



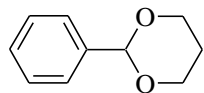
$^1\text{H NMR}$ (CDCl_3 , 270.05 MHz)



^{13}C NMR (CDCl_3 , 67.80 MHz)

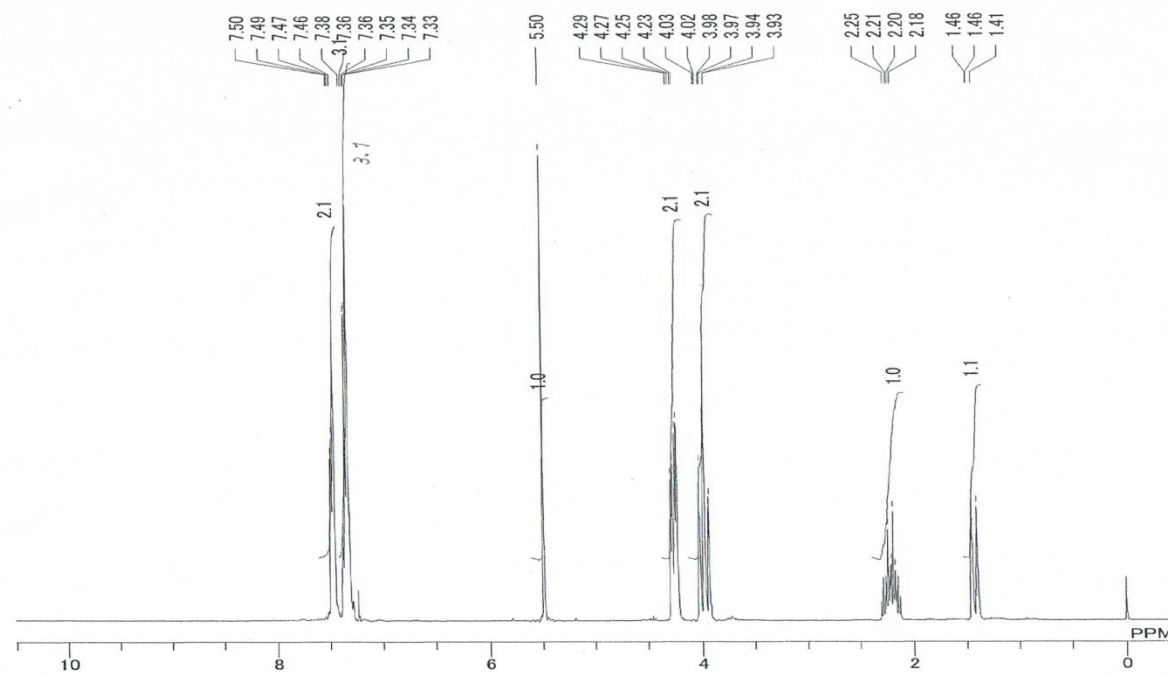


^1H NMR (CDCl_3 , 270.05 MHz)

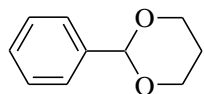


1

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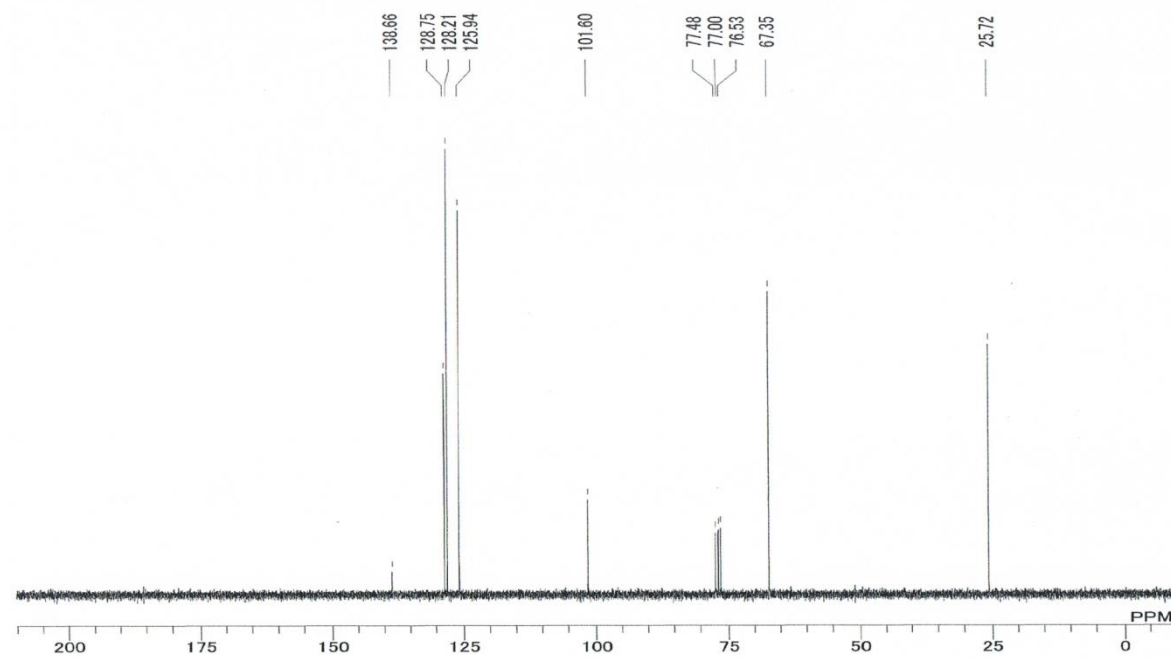


^{13}C NMR (CDCl_3 , 67.80 MHz)

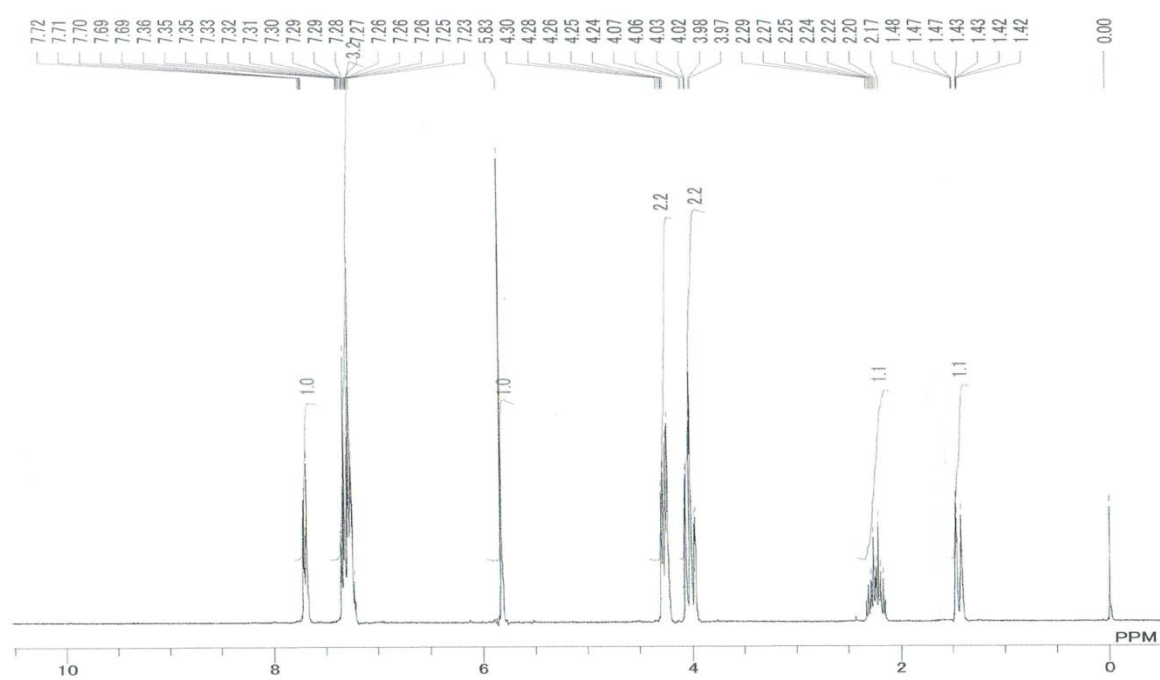
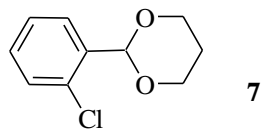


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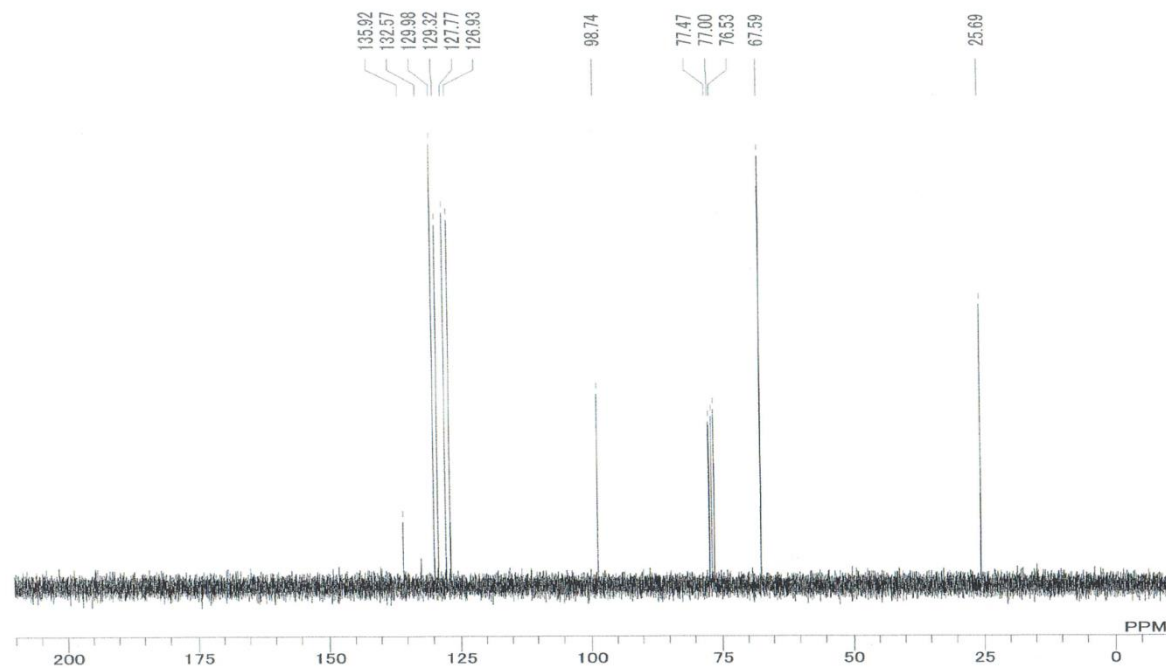
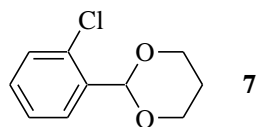
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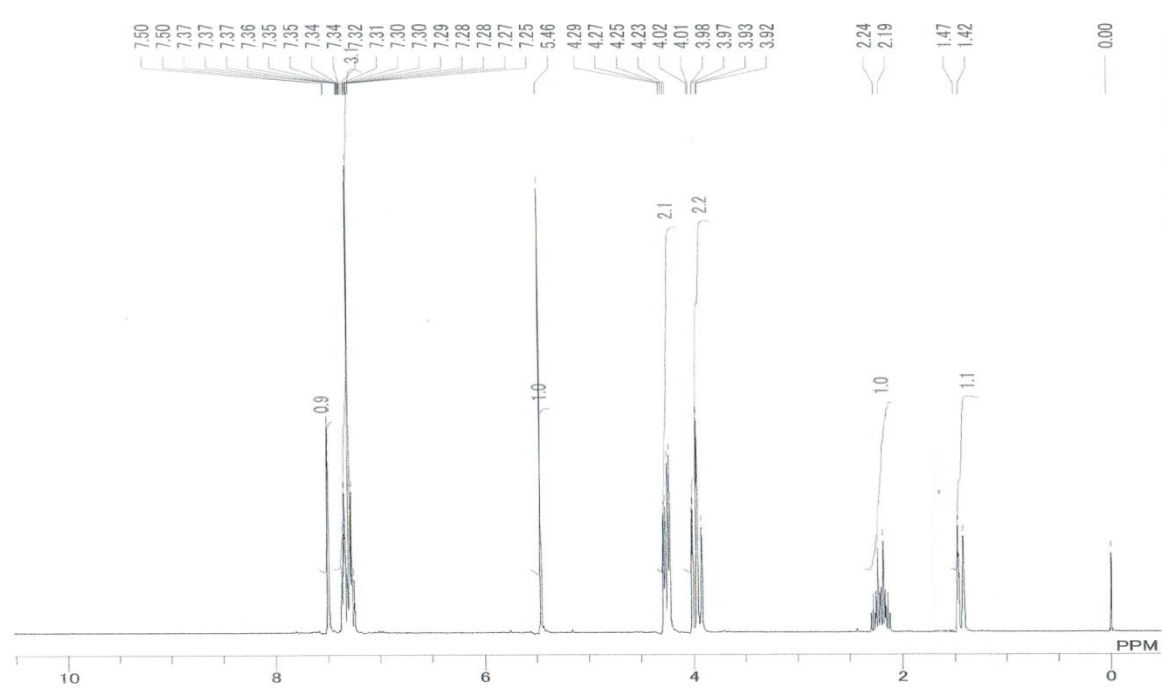
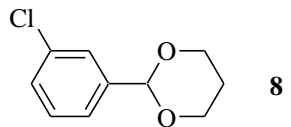
^1H NMR (CDCl_3 , 270.05 MHz)



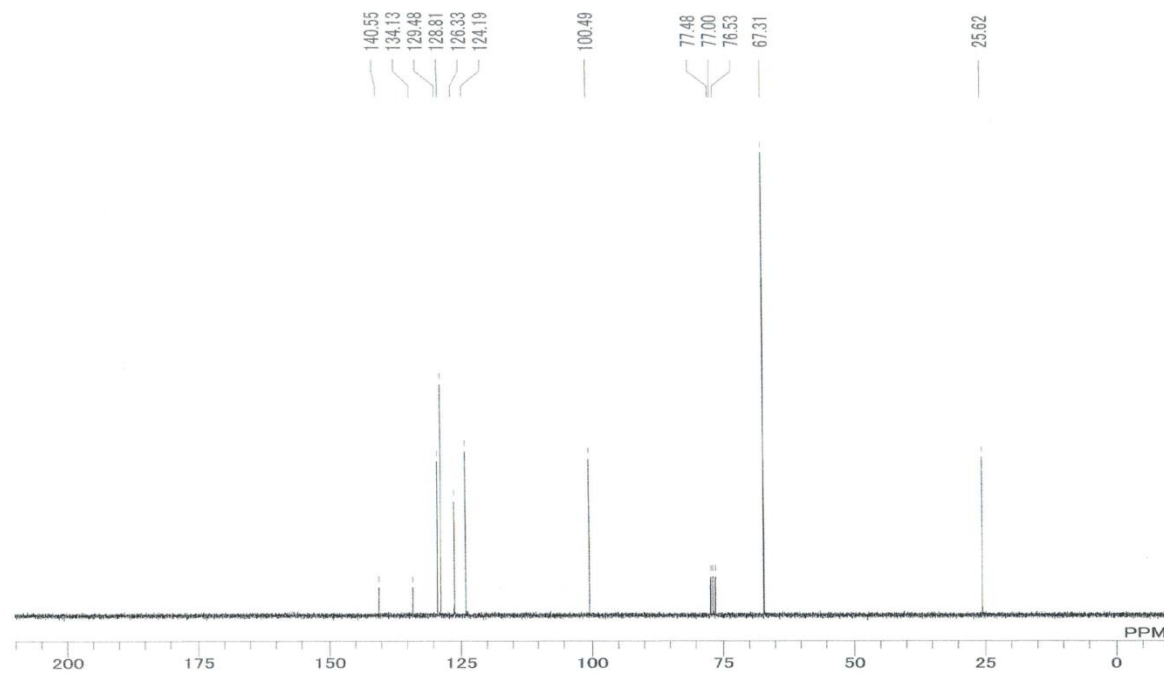
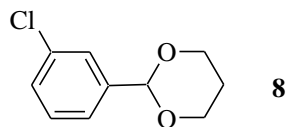
^{13}C NMR (CDCl_3 , 67.80 MHz)



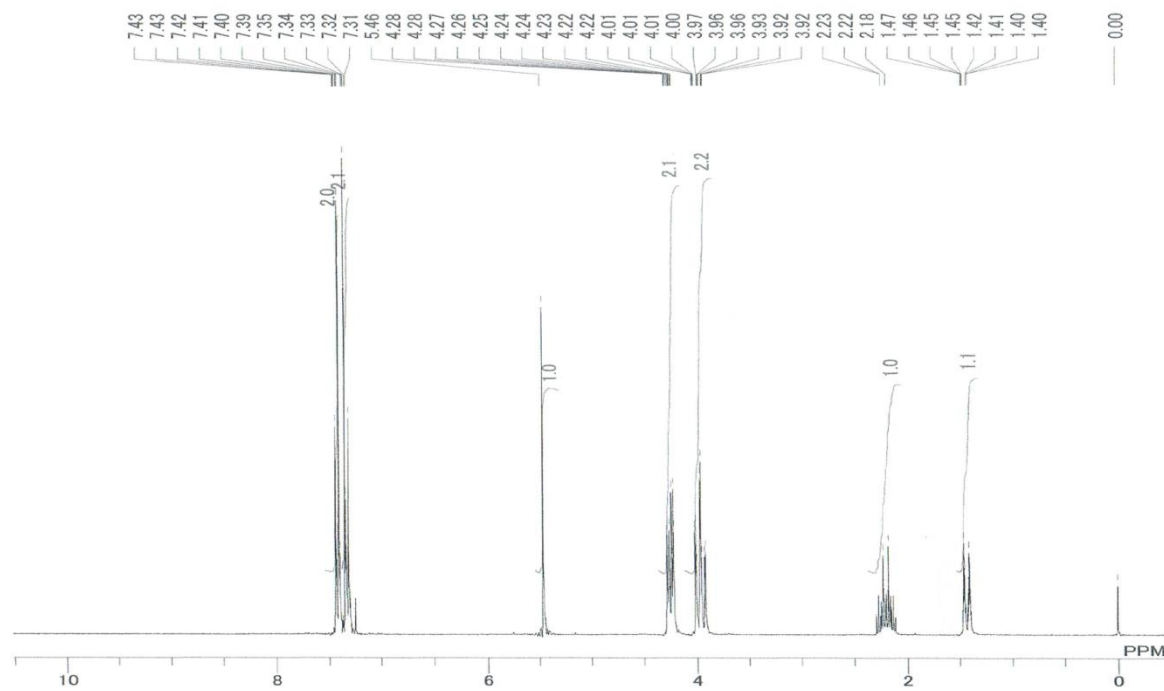
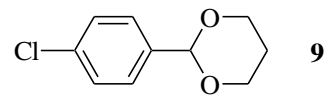
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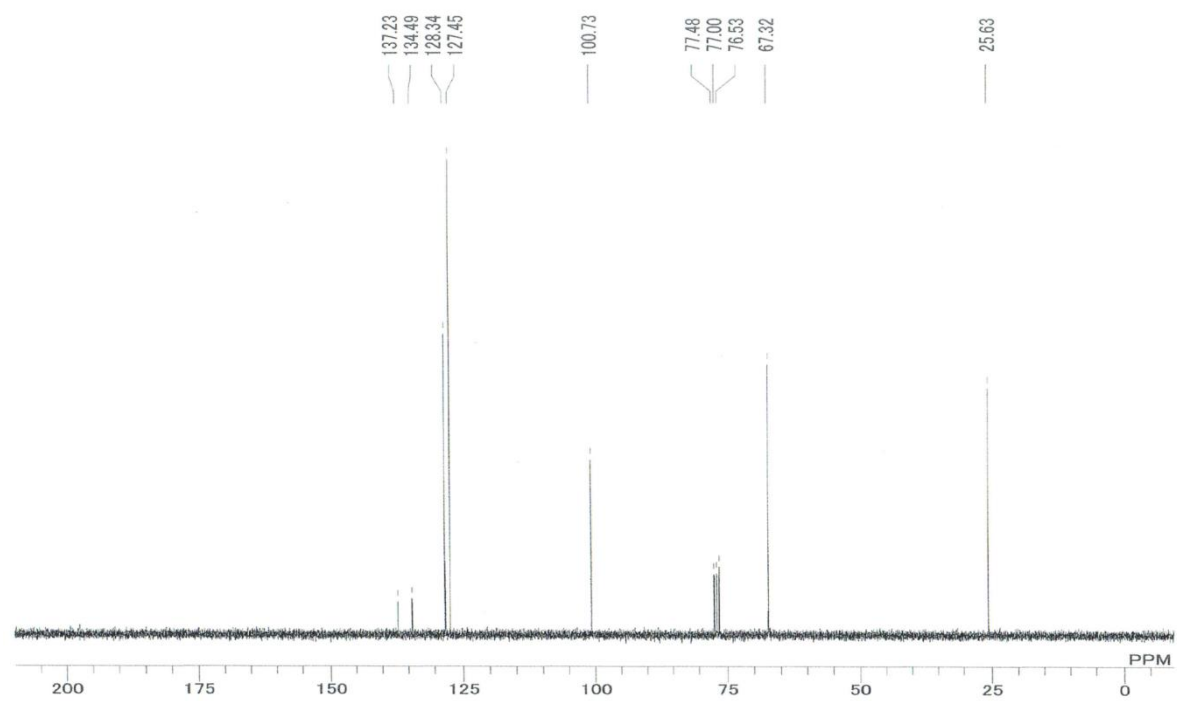
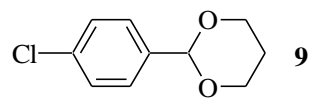
^{13}C NMR (CDCl_3 , 67.80 MHz)



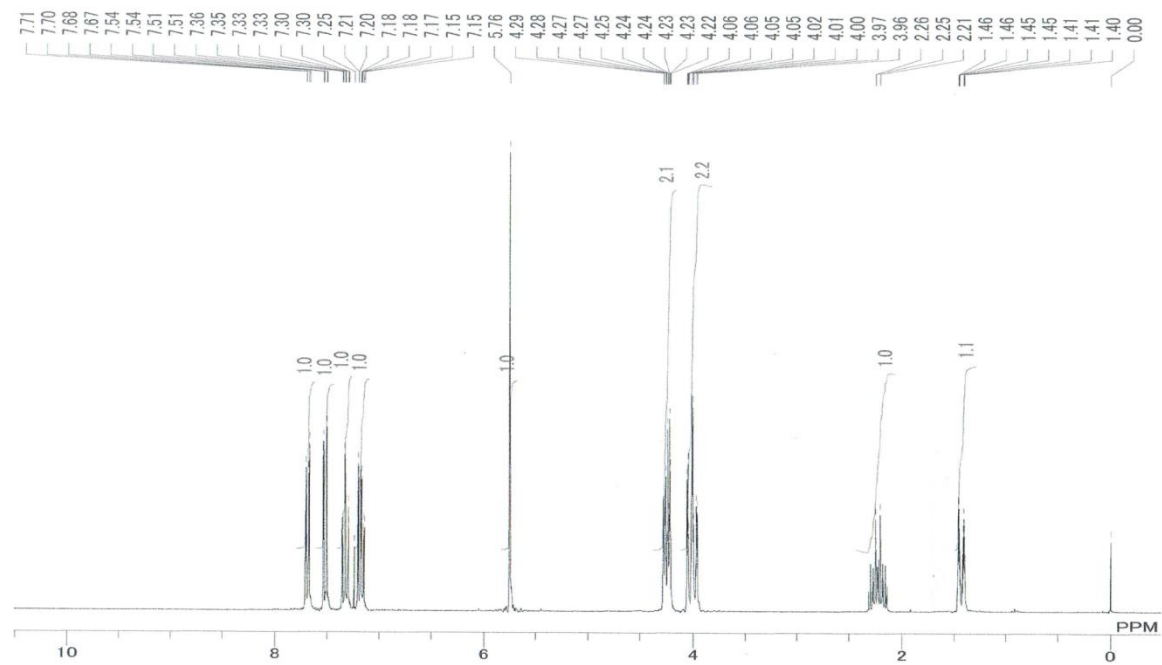
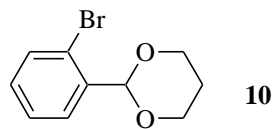
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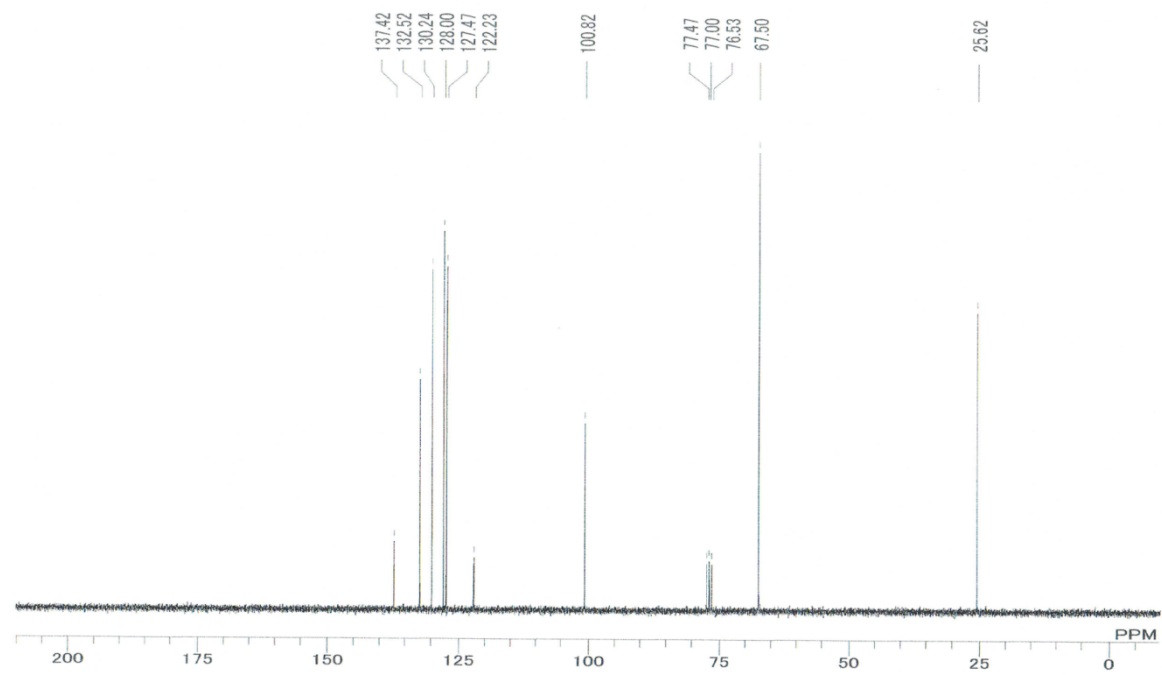
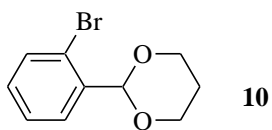
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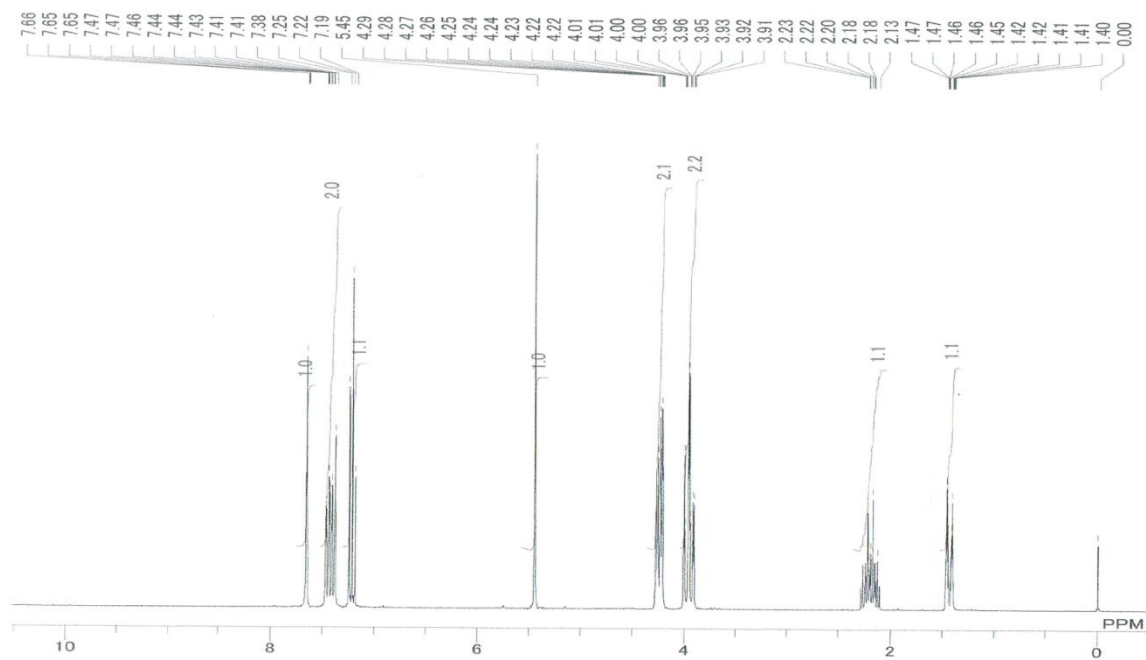
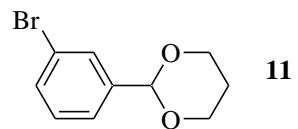
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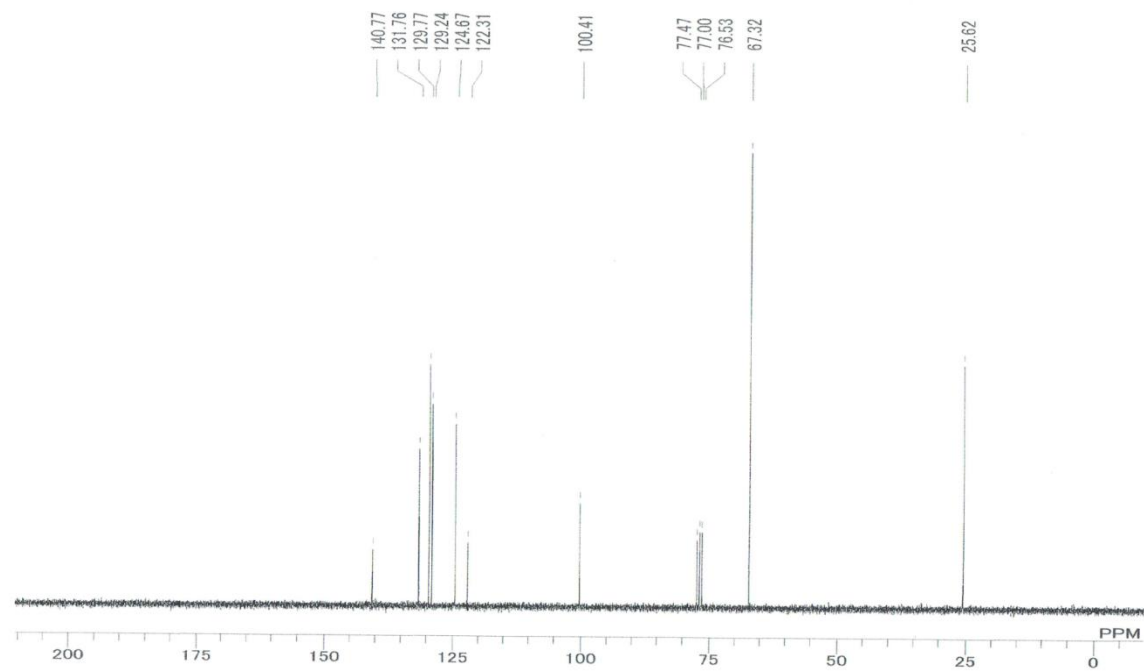
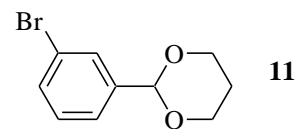
^{13}C NMR(CDCl_3 , 67.80 MHz)



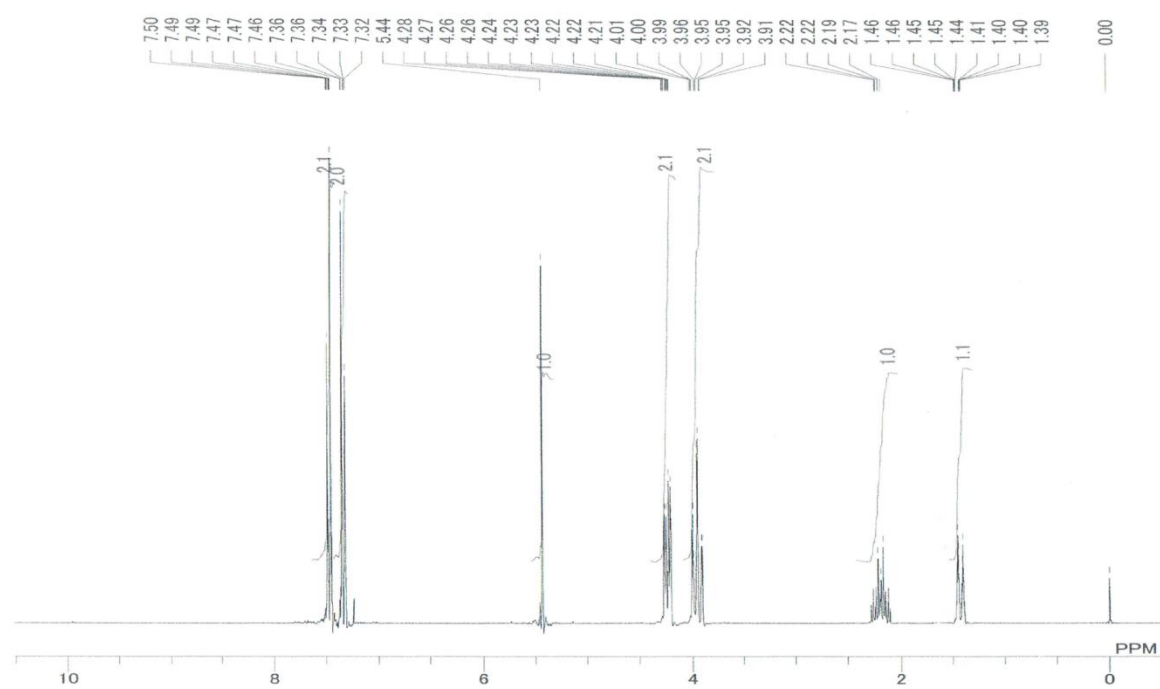
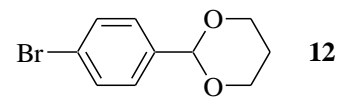
$^1\text{H NMR}(\text{CDCl}_3, 270.05 \text{ MHz})$



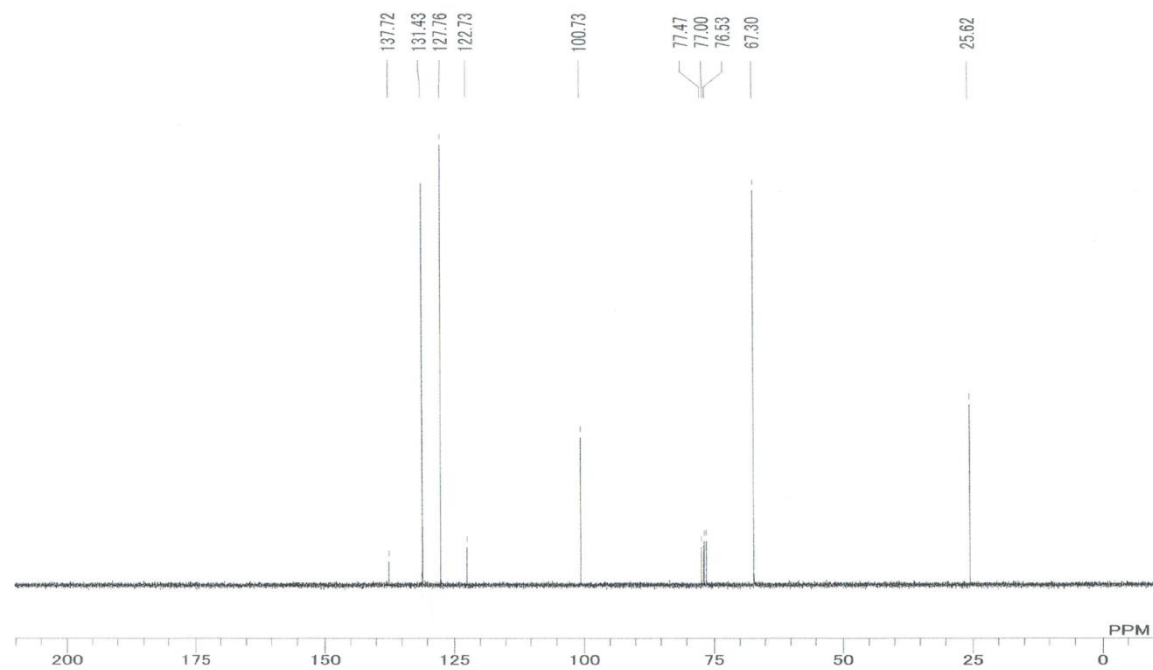
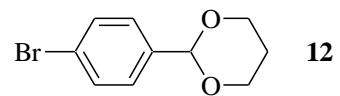
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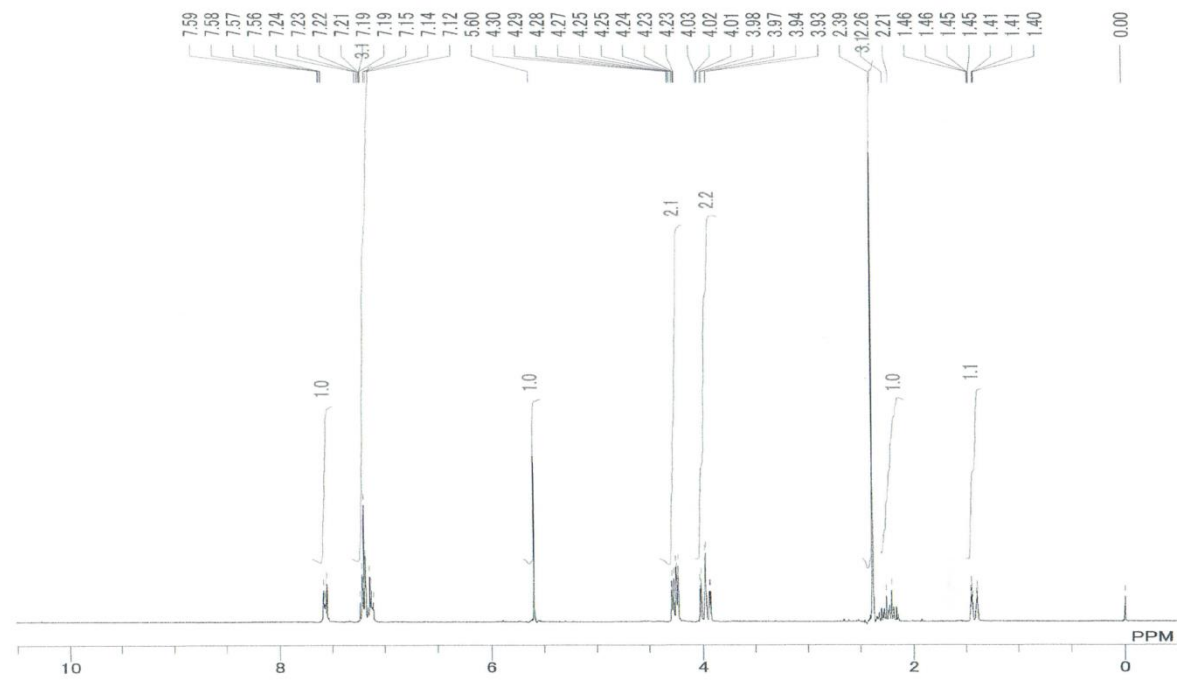
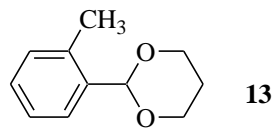
$^1\text{H NMR}$ (CDCl_3 , 270.05 MHz)



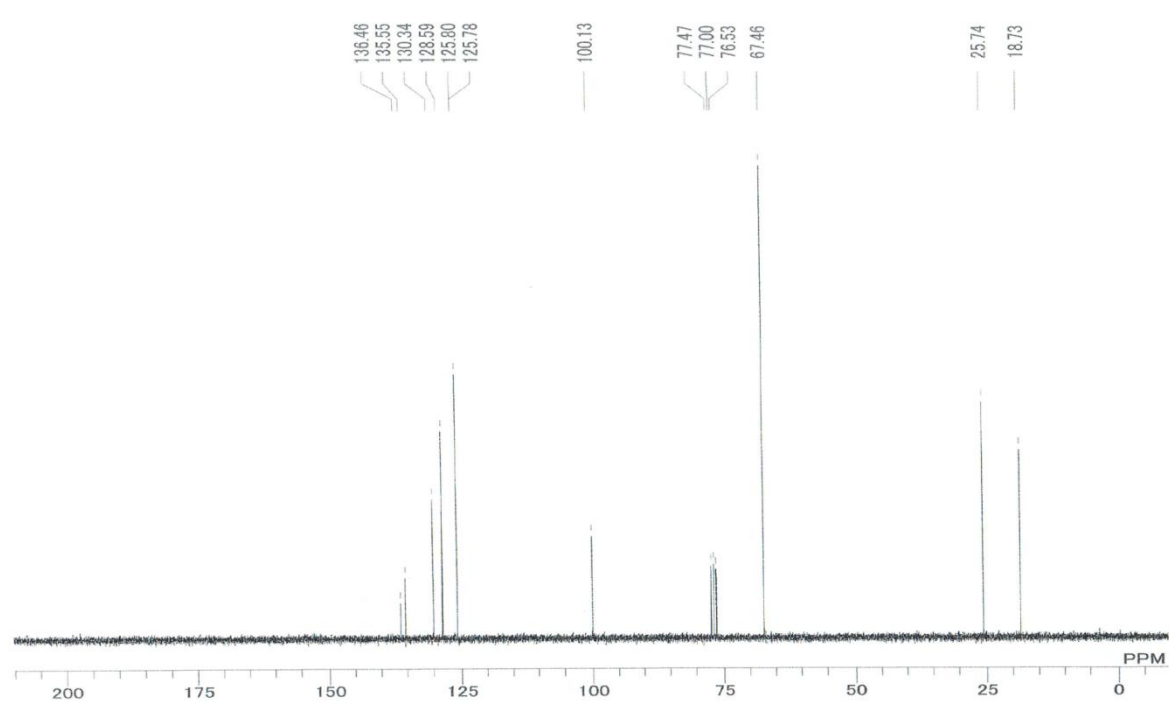
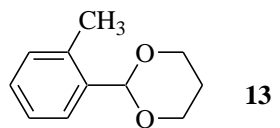
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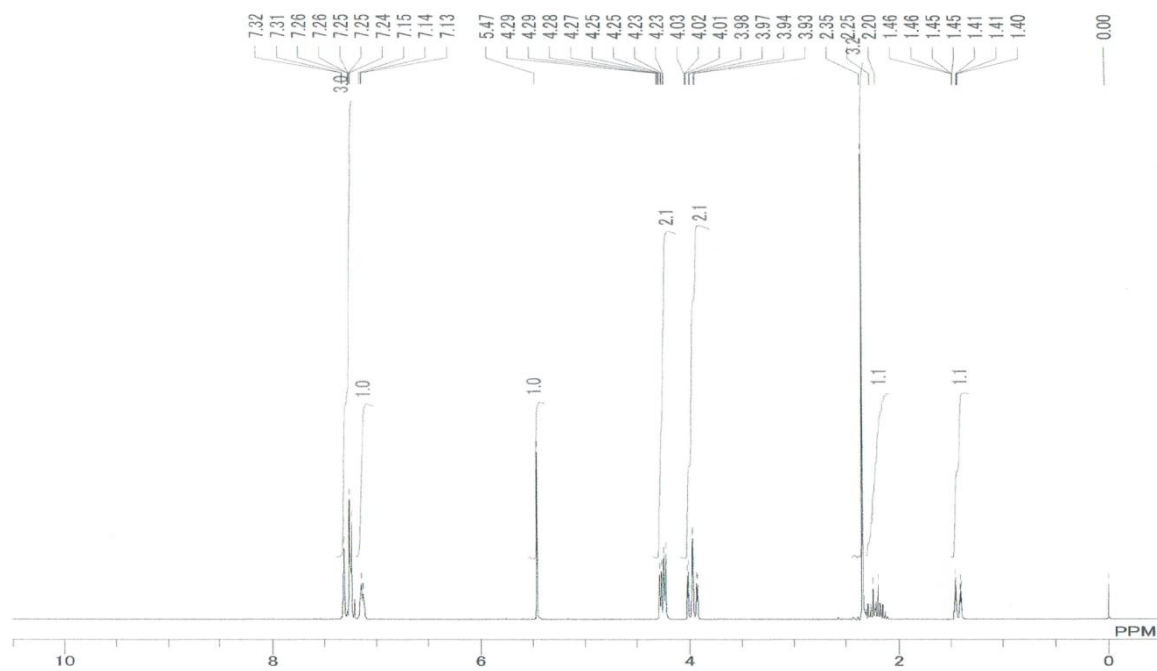
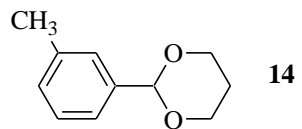
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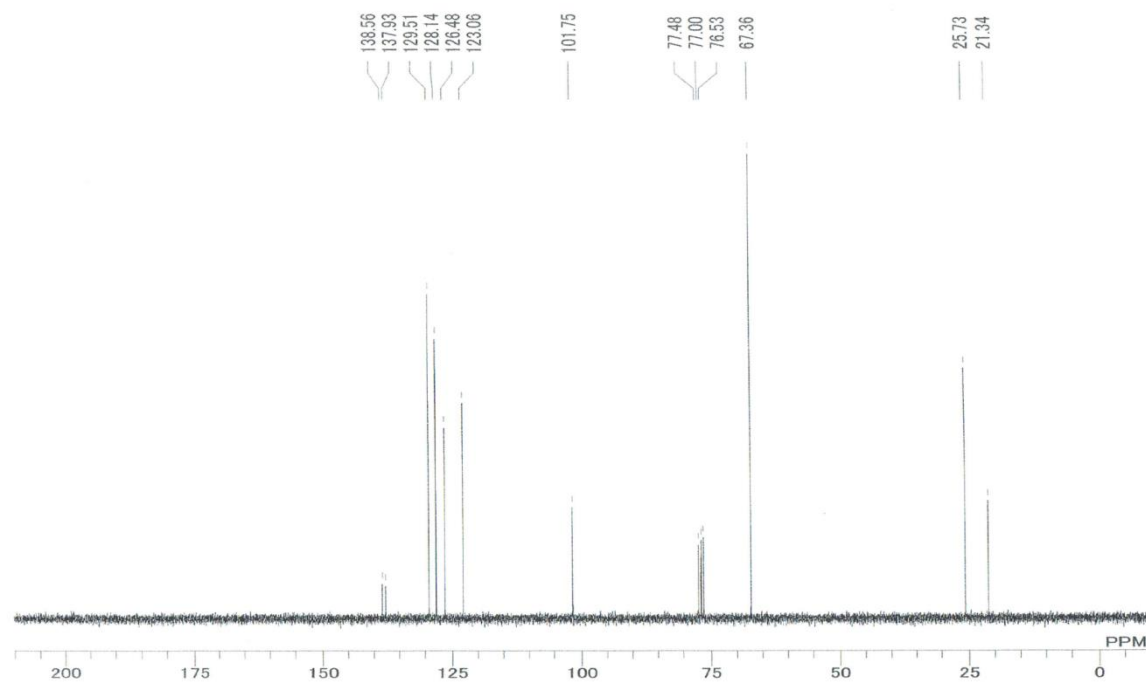
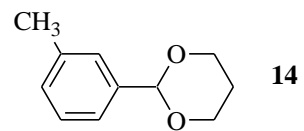
^{13}C NMR(CDCl_3 , 67.80 MHz)



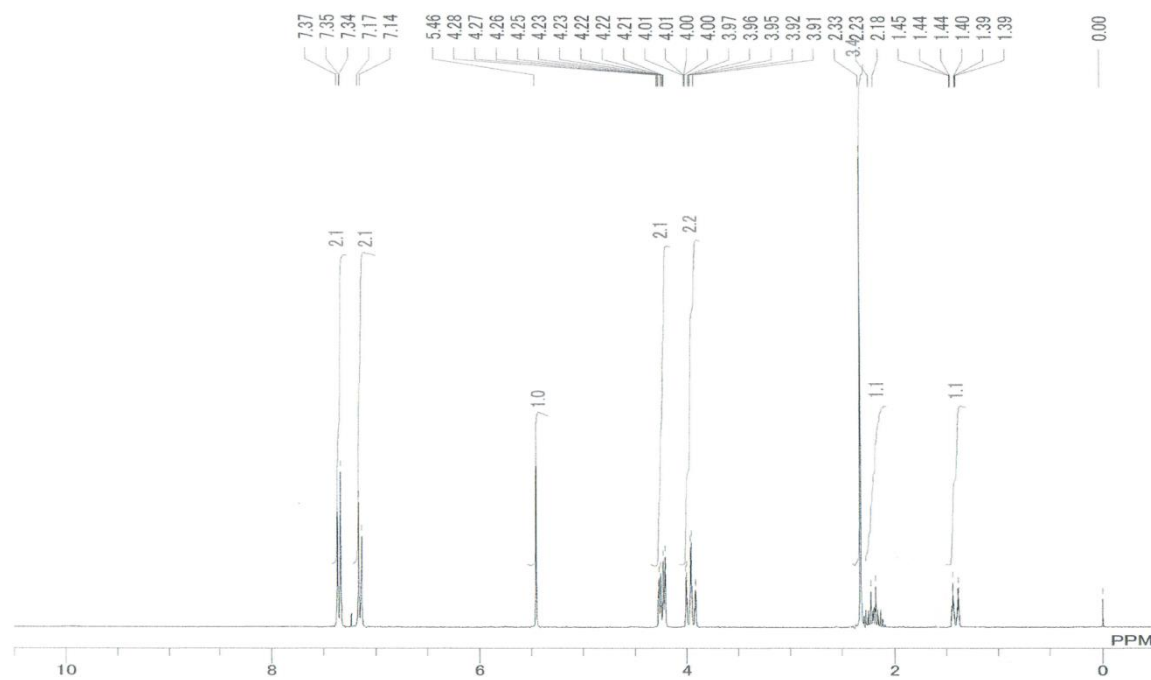
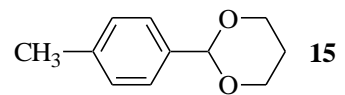
$^1\text{H NMR}(\text{CDCl}_3, 270.05 \text{ MHz})$



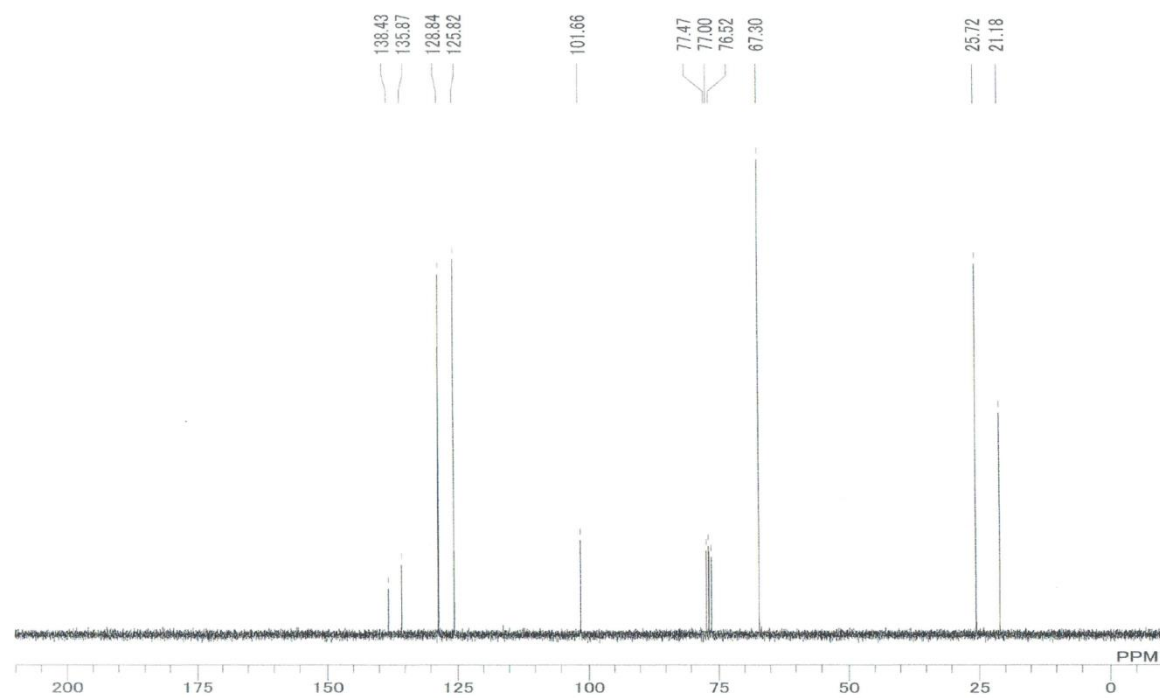
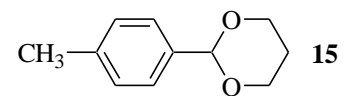
^{13}C NMR(CDCl_3 , 67.80 MHz)



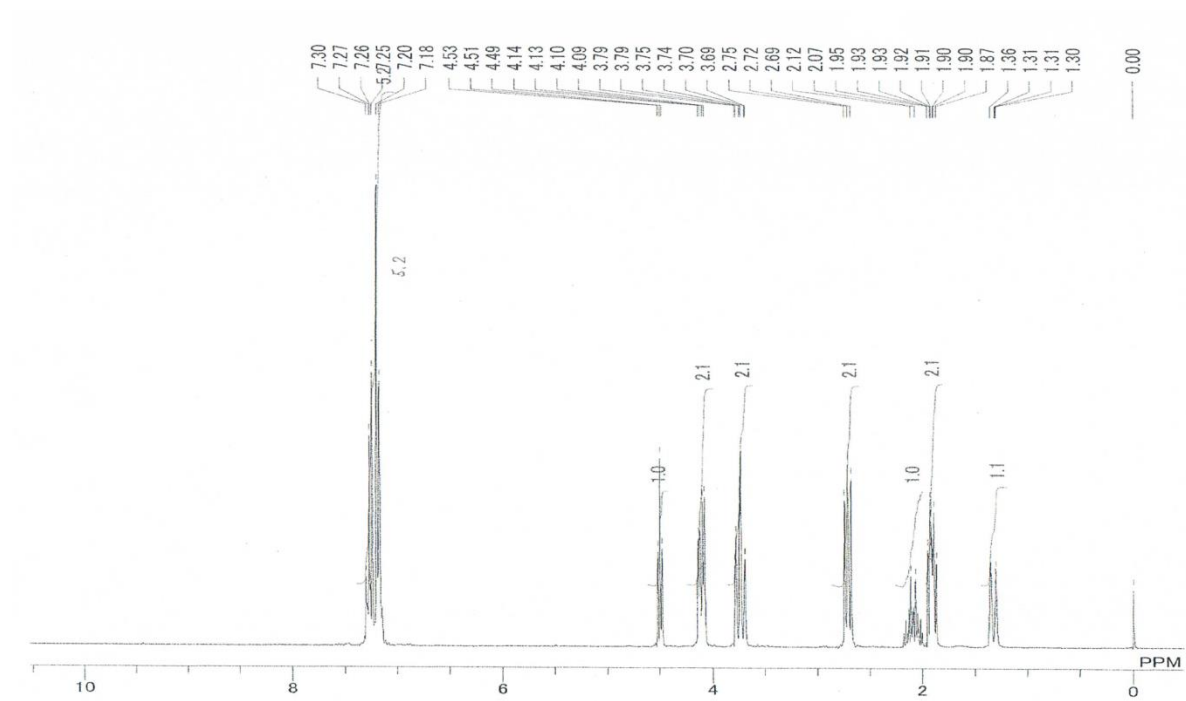
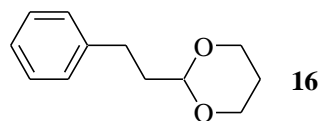
^1H NMR(CDCl_3 , 270.05 MHz)



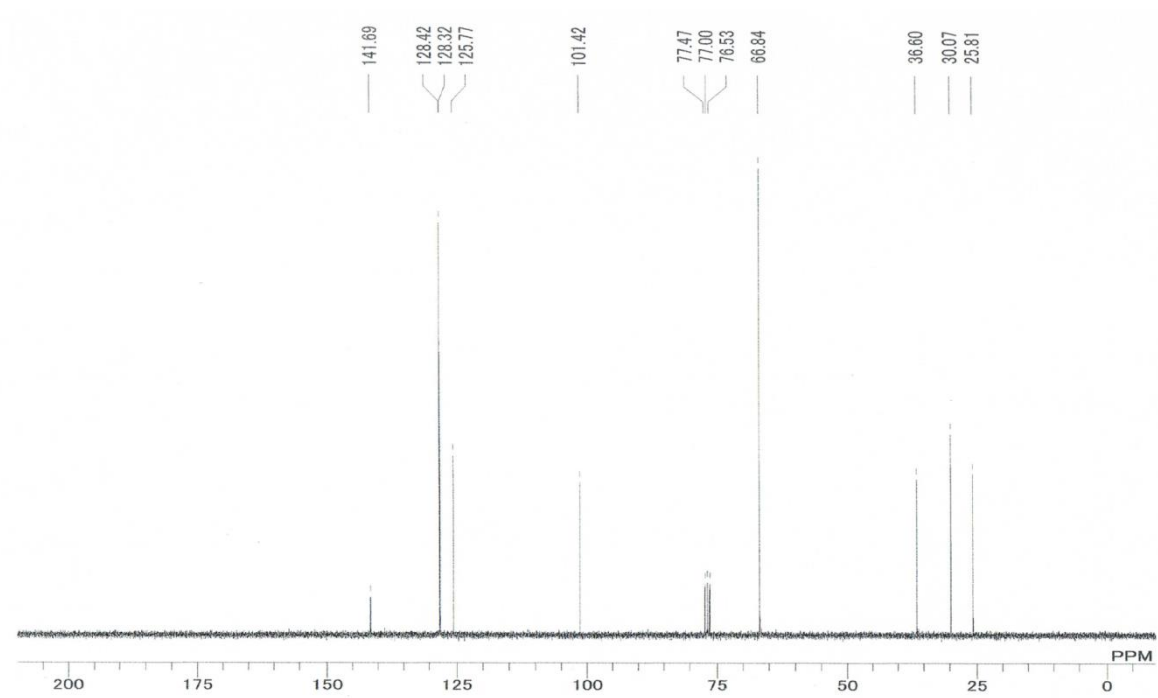
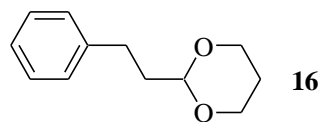
^{13}C NMR(CDCl_3 , 67.80 MHz)



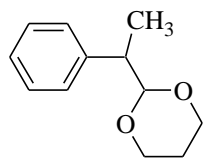
$^1\text{H NMR}$ (CDCl_3 , 270.05 MHz)



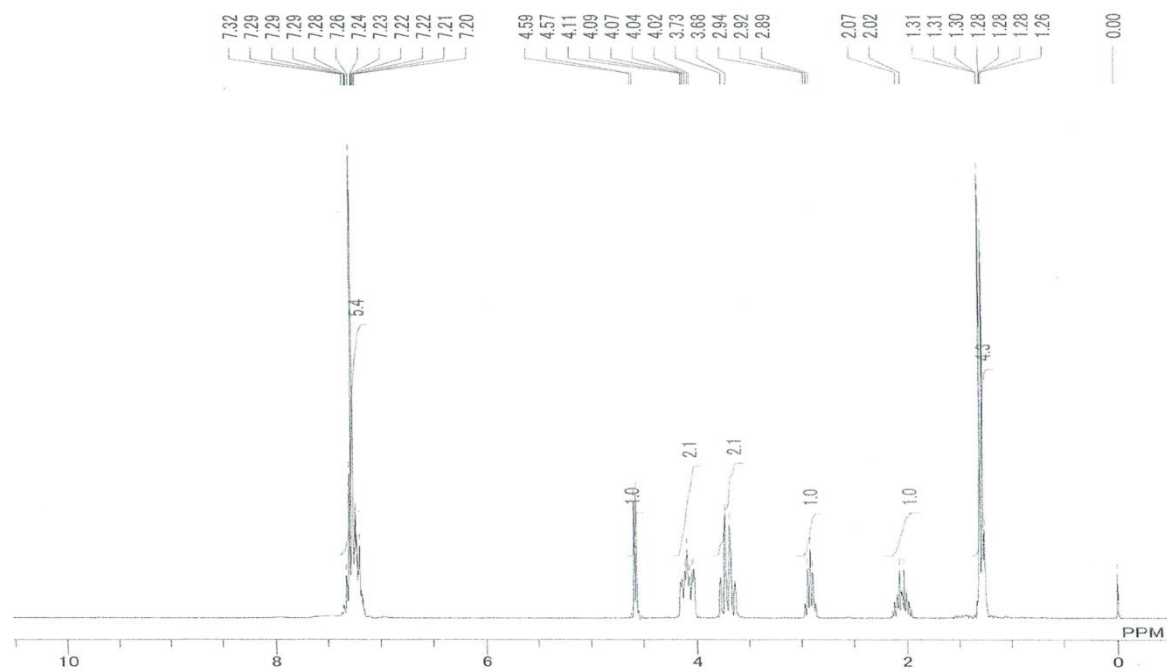
^{13}C NMR(CDCl_3 , 67.80 MHz)



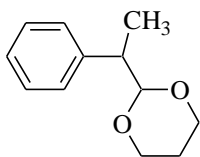
$^1\text{H NMR}(\text{CDCl}_3, 270.05 \text{ MHz})$



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^{13}C NMR(CDCl_3 , 67.80 MHz)



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