

Synthesis of 1,2,4-triazolines and triazoles utilizing oxazolones

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Supporting information

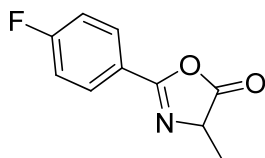
Contents

	Page #
Synthetic procedures and chemical characterization	S1-S6
X-ray structure of compound 14	S7
X-ray structure of compound 18	S8
^1H and ^{13}C spectrum of the compounds	S9-S48

General Information: The reagents and solvents were purchased from commercial suppliers and used without further purification. Anhydrous methylene chloride, benzene, acetonitrile, and tetrahydrofuran were dispensed from a delivery system which passes the solvents through a column packed with dry neutral alumina. Synthesis of azlactones was carried out in flame dried flask under nitrogen atmosphere while the cycloaddition reactions were carried out in 20mL disposable scintillation vials and during the reaction the vial caps were kept slightly loose. All reactions were magnetically stirred and monitored by TLC with 0.25 μ m pre-coated silica gel plates using UV light to visualize the compounds. Column chromatography was carried out on Silica Gel 60 (230-400 mesh). Yields refer to spectroscopically pure compounds obtained after acid-base extraction. ^1H , ^{13}C -NMR and DEPT spectra were recorded on a 500 MHz, 600MHz and 900MHz spectrometers. Chemical shifts are reported relative to the residue peaks of the solvent (CDCl_3 : 7.24 ppm for ^1H and 77.0 ppm for ^{13}C and CD_3OD : 3.30 ppm for ^1H and 49.0 ppm for ^{13}C). The following abbreviations are used to denote the nature of the carbon atoms: s =tertiary, d = secondary, t = primary, q = quartary. Melting points were obtained using a capillary melting point apparatus and are uncorrected.

Synthesis and characterization of starting oxazolones:

General procedure for synthesis of oxazolones:¹ 1.3 equivalents of TFAC was added to a suspension of N-benzoyl amino acid in anhydrous dichloromethane in a round bottom flask placed under nitrogen. The reaction mixture was stirred for 1.5 h at room temperature, after which the contents of the flask were poured into a separating funnel and washed with aqueous sodium bicarbonate solution three times to remove acid formed during the reaction. Subsequently, the reaction mixture was washed with brine, dried over sodium sulfate and placed on a rotary evaporator to evaporate the solvent. Residual solvent was removed under vacuum and previously reported oxazolones (**1**, **5**, **9**, **11**, **13**, **15**) were matched with their reported data².



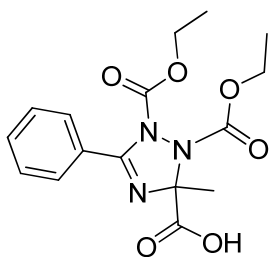
2-(4-fluorophenyl)-4-methyloxazol-5(4H)-one (7): ^1H NMR (500 MHz) (CDCl_3) δ : 7.93 (2H, m), 7.10 (2H, m), 4.38 (2H, q, J = 7 Hz), 1.51 (3H, d, J = 7 Hz). ^{13}C NMR/ DEPT (125 MHz) (CDCl_3) δ : 178.5, 166.4, 164.4, 164.3, 160.6, 130.2 (s), 130.1 (s), 128.1, 128.0, 122.1, 122.0, 116.1 (s), 116.0 (s), 60.9 (s), 16.7 (t). IR (NaCl, neat): 3290, 1734, 1705, 1631. MS (ES⁺) m/z : ($\text{M}+\text{H}$)⁺ 194.1 mp 128-130 $^\circ\text{C}$. HRMS (ES⁺) calcd. for $\text{C}_{10}\text{H}_9\text{NO}_2\text{F}$ ($\text{M}+\text{H}$)⁺: 194.0617 found: 194.0624.

General procedure for the cycloaddition reactions:

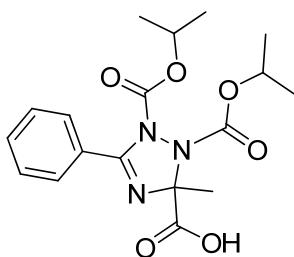
One equivalent of the azodicarboxylate was added to solution of oxazolone (0.5-0.8 mmol) in 10mL of acetonitrile in a 20mL scintillation vial. The reaction mixture was stirred at room temperature for 4-22 hours. The contents of the vial were transferred into a separating funnel containing aqueous sodium bicarbonate and dichloromethane. The product was extracted into the aqueous bicarbonate layer and the dichloromethane layer was discarded. The aqueous sodium bicarbonate layer was acidified with HCl, and the product extracted four times with 40mL of dichloromethane. The dichloromethane fractions were combined and dried over sodium sulfate. The organic solvent was removed using a rotary evaporator to provide the product, which was further dried over vacuum and analyzed.

¹ Kahlon, D. K.; Lansdell, T. A.; Fisk, J. S.; Hupp, C. D.; Friebe, T. L.; Hovde, S.; Jones, A. D.; Dyer, R. D.; Henry, R. W.; Tepe, J. J. *J. Med. Chem.* **2009**, 2, 1302-1309

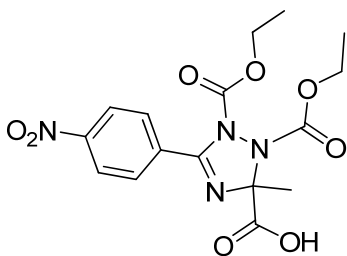
²a) Chen, F. M. F.; Kuroda, K.; Benoiton, N. L. *Synthesis*, **1978**, 12, 928. b) Peet, N. P.; Burkhart, J. P.; Angelastro, M. R.; Giroux, E. L.; Mehdi, S.; Bey, P.; Kolb, M.; Neises, B.; Schirlin D. *J. Med. Chem.* **1990**, 33, 394. c) Peddibhotla, S.; Tepe, J. J. *Synthesis*, **2003**, 9, 1433.



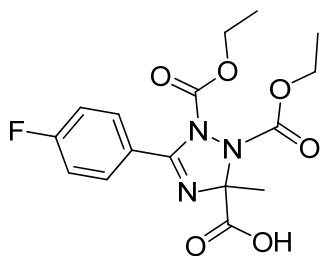
1,2-bis(ethoxycarbonyl)-3-methyl-5-phenyl-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (2): ^1H NMR (500 MHz) (CDCl_3) δ : 7.77 (2H, d, J = 7 Hz), 7.44 (1H, t, J = 7 Hz), 7.36 (2H, t, J = 7 Hz), 4.18 (2H, m), 4.10 (2H, m), 1.76 (3H, s), 1.21 (3H, t, J = 7 Hz), 1.02 (3H, t, J = 7 Hz). ^{13}C NMR/ DEPT (125 MHz) (CDCl_3) δ : 171.5, 158.8, 154.3, 152.8, 131.7 (s), 129.7 (s), 128.6, 127.7 (s), 90.2, 63.9 (d), 62.8 (d), 22.6 (t), 14.1 (t), 13.7 (t). IR (NaCl, neat): 1759, 1700, 1631. MS (ES+) m/z : $(\text{M}+\text{H})^+$ 350.1 HRMS (ES+) calcd. for $\text{C}_{16}\text{H}_{20}\text{N}_3\text{O}_6$ $(\text{M}+\text{H})^+$: 350.1352 found: 350.1354.



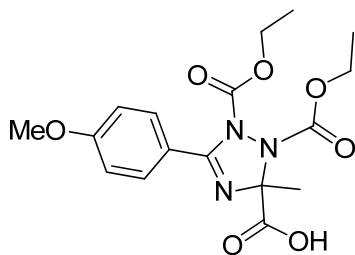
1,2-bis(isopropoxycarbonyl)-3-methyl-5-phenyl-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (3): ^1H NMR (500 MHz) (CDCl_3) δ : 7.78 (2H, d, J = 7 Hz), 7.45 (1H, t, J = 7 Hz), 7.37 (2H, t, J = 7 Hz), 4.98 (1H, m), 4.83 (1H, m), 1.78 (3H, s), 1.24 (3H, d, J = 6 Hz), 1.20 (3H, d, J = 6 Hz), 1.08 (3H, d, J = 6 Hz), 0.98 (3H, d, J = 6 Hz). ^{13}C NMR/ DEPT (125 MHz) (CDCl_3) δ : 172.3, 159.4, 153.5, 152.3, 131.7 (s), 129.8 (s), 128.7, 127.7 (s), 89.7, 72.4 (s), 71.2 (s), 22.6 (t), 21.9 (t), 21.5 (t), 21.4 (t), 21.2 (t). IR (NaCl, neat): 1761, 1705, 1653, 1630. MS (ES) m/z : $(\text{M}+\text{H})^+$ 378.1 m.p. 48°C. HRMS (ES+) calcd. for $\text{C}_{18}\text{H}_{24}\text{N}_3\text{O}_6$ $(\text{M}+\text{H})^+$: 378.1665 found: 378.1667.



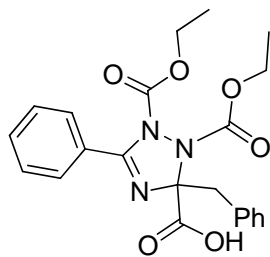
1,2-bis(ethoxycarbonyl)-3-methyl-5-(4-nitrophenyl)-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (6): ^1H NMR (500 MHz) (CDCl_3) δ : 8.19 (2H, d, J = 7 Hz), 7.95 (2H, d, J = 7 Hz), 4.22 (2H, q, J = 7 Hz), 4.12 (2H, q, J = 7 Hz), 1.78 (3H, s), 1.23 (3H, t, J = 7 Hz), 1.06 (3H, t, J = 7 Hz). ^{13}C NMR/ DEPT (125 MHz) (CDCl_3) δ : 171.7, 157.5, 154.1, 152.6, 149.6, 130.8 (s), 122.9 (s), 90.5, 64.5 (d), 63.2 (d), 22.5 (t), 14.1 (t), 13.8 (t). IR (NaCl, neat): 3100(br), 1761, 1653, 1599, 1527. MS (ES+) m/z : $(\text{M}+\text{H})^+$ 395.1 mp 134-136 °C. HRMS (ES+) calcd. for $\text{C}_{16}\text{H}_{19}\text{N}_4\text{O}_8$ $(\text{M}+\text{H})^+$: 395.1203 found: 395.1212.



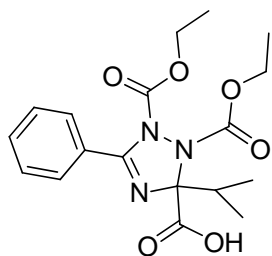
1,2-bis(ethoxycarbonyl)-5-(4-fluorophenyl)-3-methyl-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (8): ^1H NMR (500 MHz) (CDCl_3) δ : 7.80 (2H, m), 7.04 (2H, m), 4.20 (2H, q, $J = 7$ Hz), 4.12 (2H, q, $J = 7$ Hz), 7.73 (3H, s), 1.22 (2H, t, $J = 7$ Hz), 1.06 (2H, t, $J = 7$ Hz). ^{13}C NMR/ DEPT (125 MHz) (CDCl_3) δ : 171.7, 165.9, 163.9, 158.1, 154.0, 152.7, 132.2(s), 132.1 (s), 124.5, 124.4, 155.1 (s), 155.0 (s), 89.8, 64.1 (d), 62.9 (d), 22.4 (t), 14.1 (t), 13.7 (t). IR (NaCl, neat): 3200(br), 1759, 1633, 1604, 1510. MS (ES+) m/z : $(\text{M}+\text{H})^+$ 368.1 mp 46-48 °C. HRMS (ES+) calcd. for $\text{C}_{16}\text{H}_{19}\text{N}_3\text{O}_6\text{F}$ $(\text{M}+\text{H})^+$: 368.1258 found: 368.1264.



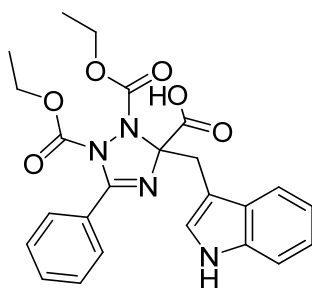
1,2-bis(ethoxycarbonyl)-5-(4-methoxyphenyl)-3-methyl-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (10): ^1H NMR (500 MHz) (CDCl_3) δ : 7.75 (2H, d, $J = 7$ Hz), 6.86 (2H, d, $J = 7$ Hz), 4.20 (2H, q, $J = 7$ Hz), 4.11 (2H, q, $J = 7$ Hz), 3.80 (3H, s), 1.76 (3H, s), 1.22 (3H, t, $J = 7$ Hz), 1.08 (3H, t, $J = 7$ Hz). ^{13}C NMR/ DEPT (125 MHz) (CDCl_3) δ : 171.8, 162.5, 158.7, 154.0, 153.0, 131(s), 120.4, 113.1 (s), 89.4, 63.9 (d), 62.8 (d), 55.3 (t), 22.4 (t), 14.1 (t), 13.7 (t). IR (NaCl, neat): 3200 (br), 1757, 1718, 1624, 1608, 1512. MS (ES+) m/z : $(\text{M}+\text{H})^+$ 380.1 mp 42-44 °C. HRMS (ES+) calcd. for $\text{C}_{17}\text{H}_{22}\text{N}_3\text{O}_7$ $(\text{M}+\text{H})^+$: 380.1458 found: 380.1461.



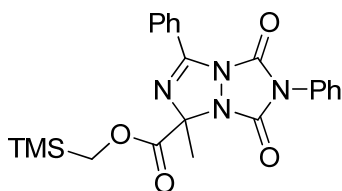
3-benzyl-1,2-bis(ethoxycarbonyl)-5-phenyl-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (12): ^1H NMR (500 MHz) (CDCl_3) δ : 7.67 (2H, d, $J = 7$ Hz), 7.45 (1H, t, $J = 7$ Hz), 7.35 (2H, t, $J = 7$ Hz), 7.29 (2H, d, $J = 7$ Hz), 7.21 (2H, , $J = 7$ Hz), 7.15 (1H, t, $J = 7$ Hz), 4.28 (2H, m), 3.73 (1H, m), 3.70 (1H, m), 3.64 (1H, d, $J = 14$ Hz), 3.44 (1H, d, $J = 14$ Hz), 1.29 (3H, t, $J = 7$ Hz), 0.91 (3H, t, $J = 7$ Hz). ^{13}C NMR/ DEPT (125 MHz) (CDCl_3) δ : 171.7, 159.8, 154.9, 151.4, 133.6, 131.5 (s), 131.2 (s), 129.2 (s), 128.8, 127.7 (s), 127.6 (s), 126.8 (s), 93.0, 63.4 (d), 63.0 (d), 40.7 (d), 14.1 (t), 13.4 (t). IR (NaCl, neat): 3200 (br), 1757, 1718, 1686, 1635. MS (ES+) m/z : $(\text{M}+\text{H})^+$ 426.2 mp 61-64°C. HRMS (ES+) calcd. for $\text{C}_{22}\text{H}_{24}\text{N}_3\text{O}_6$ $(\text{M}+\text{H})^+$: 426.1665 found: 426.1666.



1,2-bis(ethoxycarbonyl)-3-isopropyl-5-phenyl-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (14): ^1H NMR (500 MHz) (CDCl_3) δ : 7.77 (2H, d, J = 7 Hz), 7.45 (1H, t, J = 7 Hz), 7.35 (2H, t, J = 7 Hz), 4.15 (4H, m), 2.62 (1H, m), 1.20 (3H, t, J = 7 Hz), 1.05 (6H, q, J = 7 Hz), 0.91 (3H, d, J = 7 Hz) ^{13}C NMR/ DEPT (125 MHz) (CDCl_3) δ : 171.6, 158.7, 155.5, 152.4, 131.6 (s), 129.6 (s), 128.6, 127.7 (s), 96.1, 63.7 (d), 62.9 (d), 33.1 (s), 17.2 (t), 16.2 (t), 14.0 (t), 13.8 (t). IR (NaCl, neat): 1759, 1635. MS (ES+) m/z : $(\text{M}+\text{H})^+$ 378.1 mp 98-99°C. HRMS (ES+) calcd. for $\text{C}_{18}\text{H}_{24}\text{N}_3\text{O}_6$ $(\text{M}+\text{H})^+$: 378.1665 found: 378.1665.



3-((1H-indol-3-yl)methyl)-1,2-bis(ethoxycarbonyl)-5-phenyl-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (16): ^1H NMR (500 MHz) (CDCl_3) δ : 8.28 (1H, br), 7.68 (1H, d, J = 7 Hz), 7.60 (2H, d, J = 7 Hz), 7.37 (1H, t, J = 7 Hz), 7.27 (2H, d, J = 7 Hz), 7.15 (1H, d, J = 7 Hz), 7.06 (2H, m), 7.01 (1H, d, J = 2 Hz), 4.25 (2H, m), 3.69 (2H, s), 3.45 (1H, m), 2.96 (1H, m), 1.26 (3H, t, J = 7 Hz), 0.59 (3H, t, J = 8 Hz). ^{13}C NMR/ DEPT (125 MHz) (CDCl_3) δ : 171.2, 159.7, 154.8, 152.3, 135.6, 131.5 (s), 129.3 (s), 128.7, 128.4, 127.7 (s), 124.5 (s), 121.5 (s), 119.4 (s), 119.2 (s), 110.8 (s), 107.3, 93.4, 63.2 (d), 62.9 (d), 30.4 (d), 14.2 (t), 13.1 (t). IR (NaCl, neat): 3391, 2984, 1753, 1633, 1458, 1327, 1259. MS (ES+) m/z : $(\text{M}+\text{H})^+$ 465.2 mp 97-99°C. HRMS (ES+) calcd. for $\text{C}_{24}\text{H}_{25}\text{N}_4\text{O}_6$ $(\text{M}+\text{H})^+$: 465.1774 found: 465.1776.

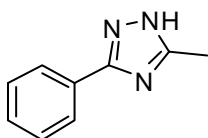


(Trimethylsilyl)methyl 1-methyl-5,7-dioxo-3,6-diphenyl-1,5,6,7-tetrahydro-[1,2,4]triazolo[1,2-a][1,2,4]triazole-1-carboxylate (17): 4-methyl-2-phenyloxazol-5(4H)-one (**1**, 175mg, 1 mmol) was dissolved in 10mL of acetonitrile in a 20mL scintillation vial and PTAD (175mg, 1 mmol) was added to the reaction mixture. The addition of PTAD turned the solution scarlet red in color. The reaction mixture was stirred for 4 hours, which lead to the disappearance of the color. At this point the reaction mixture was cooled to 0 °C and (trimethylsilyl)diazomethane (1.5 mL, 3 mmol) was added to the solution in drop wise manner. The reaction mixture was stirred for 15 minutes and then methanol (3mL) was added in drop wise manner. The reaction was further stirred at 0°C for 3 h and then the reaction temperature was allowed to come to ambient temperature. Subsequently, the reaction mixture was concentrated to minimal residue and purified by running silica-gel column using ethyl acetate: hexanes (1:4) to obtain (trimethylsilyl)methyl 1-methyl-5,7-dioxo-3,6-diphenyl-1,5,6,7-tetrahydro-[1,2,4]triazolo[1,2-a][1,2,4]triazole-1-carboxylate as viscous liquid (370 mg, 85%).

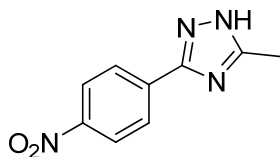
^1H NMR (500 MHz) (CDCl_3) δ : 8.06 (1H, d, J = 7 Hz), 7.58 (1H, t, J = 7 Hz), 7.46 (6H, m), 7.38 (1H, t, J = 7 Hz), 4.09 (1H, d, J = 14 Hz), 3.85 (1H, d, J = 14 Hz), 2.06 (3H, s), 0.07 (9H, s). ^{13}C NMR/ DEPT (150MHz, -10°C) (CD_3OD) δ : 167.5, 153.7, 153.5, 148.1, 133.3 (s), 130.9, 130.3 (s), 129.2 (s), 128.7 (s), 128.4 (s), 125.9 (s), 125.0, 90.8, 60.9 (d), 23.4 (t), -3.2 (t). IR (NaCl, neat): 1794, 1740, 1616, 1500, 1450, 1398, 1329, 1251. MS (ES) m/z : $(\text{M}+\text{H})^+$ 437.2 HRMS (ES+) calcd. for $\text{C}_{22}\text{H}_{25}\text{N}_4\text{O}_4\text{Si}$ $(\text{M}+\text{H})^+$: 437.1645 found: 437.1653.

General procedure for conversion of triazolines to triazoles:

The triazoline (0.5-1mmol) was dissolved in 25 mL ethanol in 100mL flask. Four equivalents of sodium hydroxide were added to this solution and the solution was heated to reflux for 2 hours. The temperature of the flask was allowed to cool down to room temperature. The excess base in the solution was neutralized with aqueous HCl. The ethanol was removed on a rotary evaporator and the residue was dissolved in ethyl acetate. The ethyl acetate solution was washed with brine and dried over sodium sulfate. Subsequently, the ethyl acetate was removed on a rotary evaporator and silica-gel column chromatography was performed using ethyl acetate to obtain the triazole³.

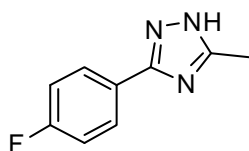


Synthesis of 5-methyl-3-phenyl-1H-1,2,4-triazole (18): ^1H NMR (500 MHz) (CDCl_3) δ : 7.95 (2H, d, J = 7 Hz), 7.42 (3H, m), 2.45 (3H, s) ^{13}C NMR/ DEPT (150MHz, -10°C) (CD_3OD) δ : 162.7, 155.5, 131.7, 130.6(s), 1129.8(s), 127.2 (s), 11.6 (t) IR (NaCl, neat): 3500 (br), 1700, 1720 MS (ES) m/z : $(\text{M})^+$ 159.1 m.p. 144-145 $^\circ\text{C}$ HRMS (ES+) calcd. for $\text{C}_9\text{H}_{10}\text{N}_3$ $(\text{M}+\text{H})^+$: 160.0875 found: 160.0880



5-methyl-3-(4-nitrophenyl)-1H-1,2,4-triazole(19):

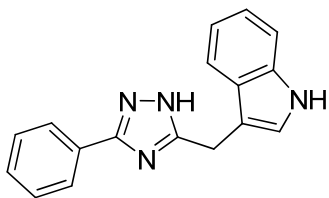
^1H NMR (600MHz) (CD_3OD) δ : 8.31 (2H, d, J = 9 Hz), 8.21 (2H, d, J = 9 Hz), 2.51 (3H, s). ^{13}C NMR/ DEPT (150 MHz) (CD_3OD) δ : 149.6, 138.0, 129.9, 128.0, 125.0, 11.7. IR (NaCl, neat): 3034(br), 1603, 1508. MS (ES+) m/z : $(\text{M}+\text{H})^+$ 205.1 mp 232-234 $^\circ\text{C}$. HRMS (ES+) calcd. for $\text{C}_9\text{H}_9\text{N}_4\text{O}_2$ $(\text{M}+\text{H})^+$: 205.0726 found: 205.0726.



³ a) Perez, M. A.; Dorado, C. A.; Soto, J. L. *Synthesis*, **1983**, 6, 483. b) Santus, M. *Pol. J. Chem.* **1980**, 54, 1067c) Konetzki, I.; Bouyssou, T.; Pestel, S.; Schnapp, A. *PCT Int. Appl.* **2008**, 105pp. d) Yeung, K.; Farkas, M.; E. Kadow;; J. F. Meanwell, N. A. *Tetrahedron Lett.* **2005**, 46, 3429

3-(4-fluorophenyl)-5-methyl-1H-1,2,4-triazole(20):

^1H NMR (600MHz) (CD_3OD) δ : 7.96 (2H, dd, J = 5 Hz, 7 Hz), 7.15 (2H, t, J = 9Hz), 2.49 (3H, s). ^{13}C NMR/ DEPT (150MHz) (CD_3OD) δ : 165.8, 164.1, 160.7, 156.9, 129.4, 129.3, 127.8, 127.7, 116.7, 116.6, 11.9. IR (NaCl, neat): 3055 (br), 1603, 1560, 1533, 1473, 1219. MS (ES) m/z : $(\text{M}+\text{H})^+$ 178.1 m.p. 279-283 $^\circ\text{C}$. HRMS (ES+) calcd. for $\text{C}_9\text{H}_9\text{FN}_3$ $(\text{M}+\text{H})^+$: 178.0781, found: 178.0783.



3-((3-phenyl-1H-1,2,4-triazol-5-yl)methyl)-1H-indole(21):

^1H NMR (600MHz) (CD_3OD) δ : 7.98 (2H, d, J = 7 Hz), 7.44 (4H, m), 7.34 (1H, d, J = 8 Hz), 7.17 (1H, s), 7.08 (1H, t, J = 7 Hz), 6.98 (1H, t, J = 7 Hz), 4.30 (2H, s). ^{13}C NMR/ DEPT (226MHz) (CD_3OD) δ : 160.8, 138.3, 131.2, 130.8, 129.8, 128.3, 127.4, 124.5, 122.6, 120.0, 119.2, 112.4, 110.5, 24.2. IR (NaCl, neat): 3333, 3128 (br), 1558, 1471. MS (ES+) m/z : $(\text{M}+\text{H})^+$ 275.1 mp 241-243 $^\circ\text{C}$. HRMS (ES+) calcd. for $\text{C}_{17}\text{H}_{15}\text{N}_4$ $(\text{M}+\text{H})^+$: 275.1297 found: 275.1307.

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FIGURE 2. X-ray crystal structure of 14

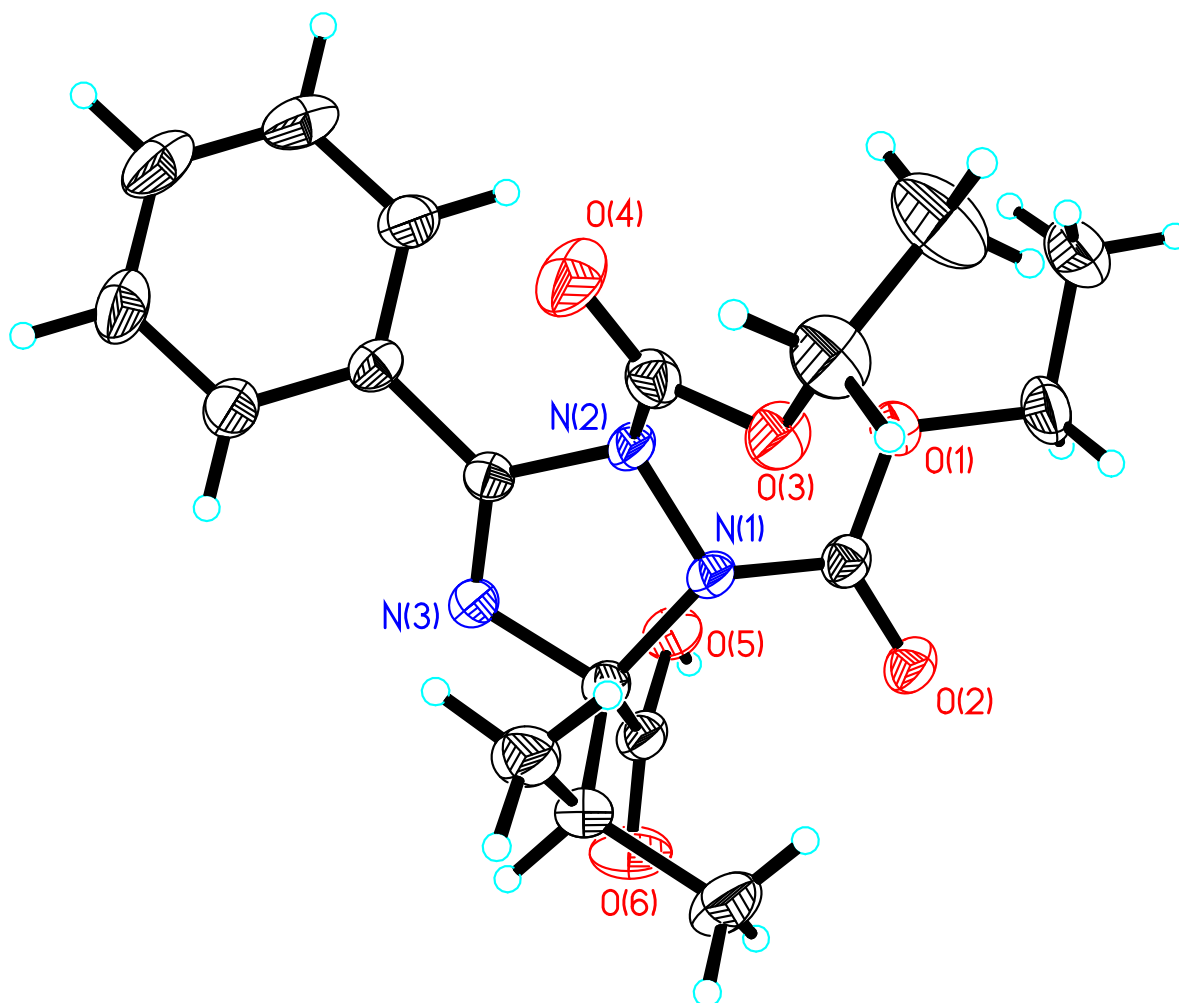
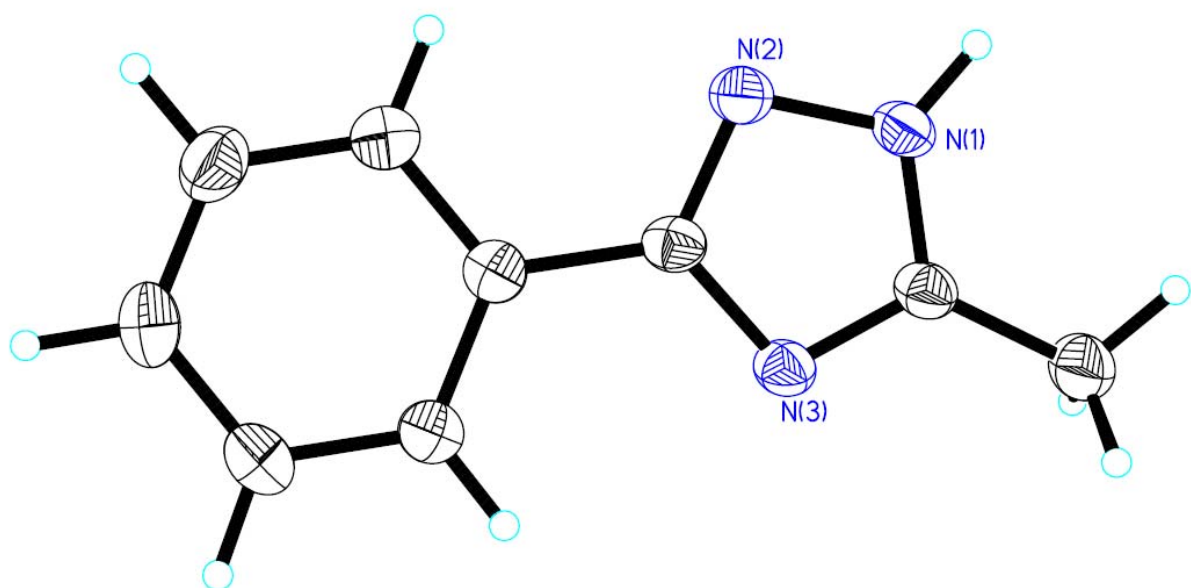
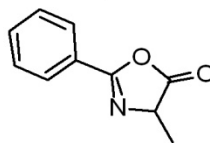
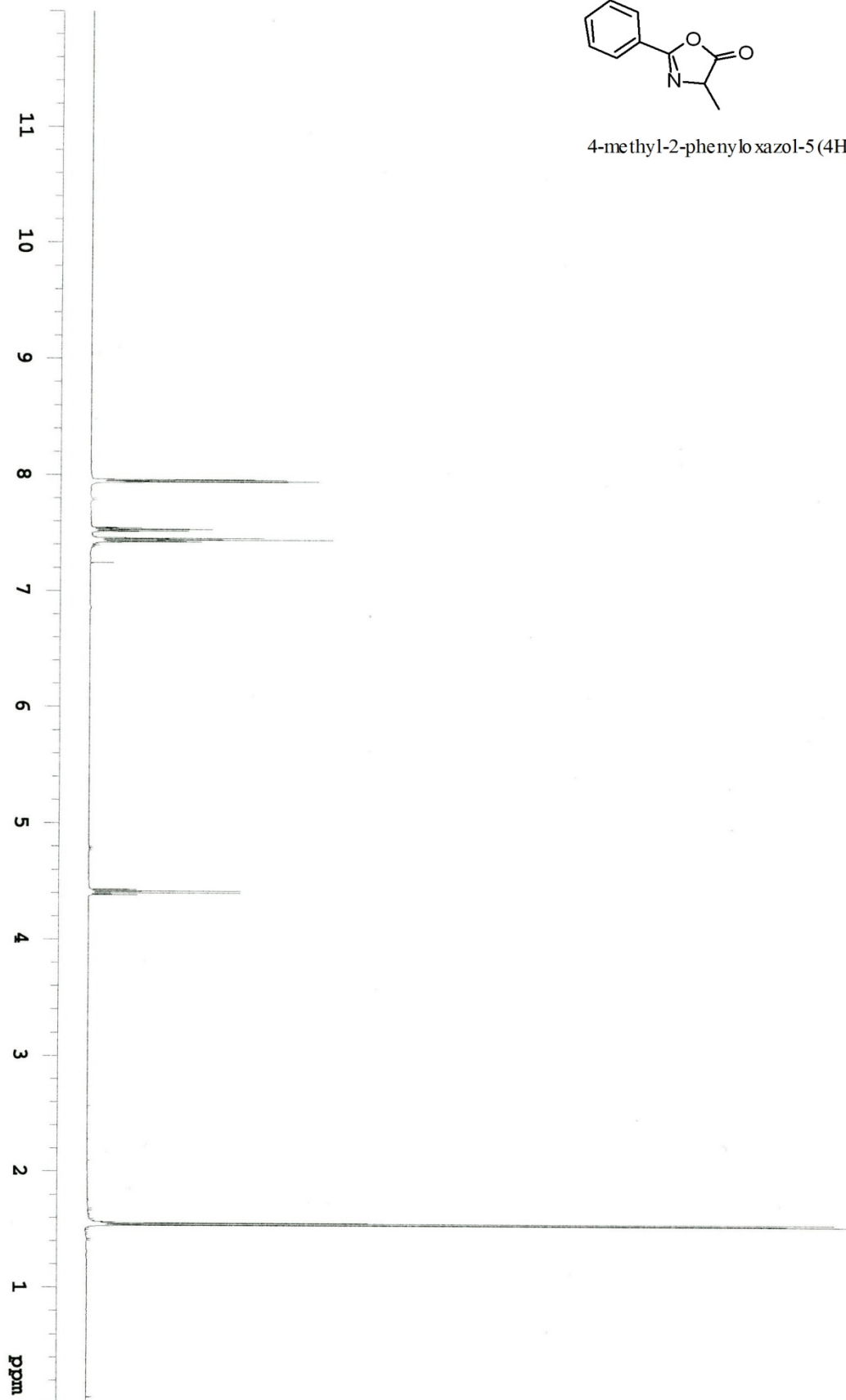


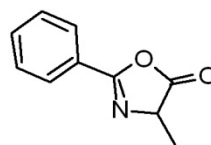
FIGURE 3. X-ray crystal structure of **18**



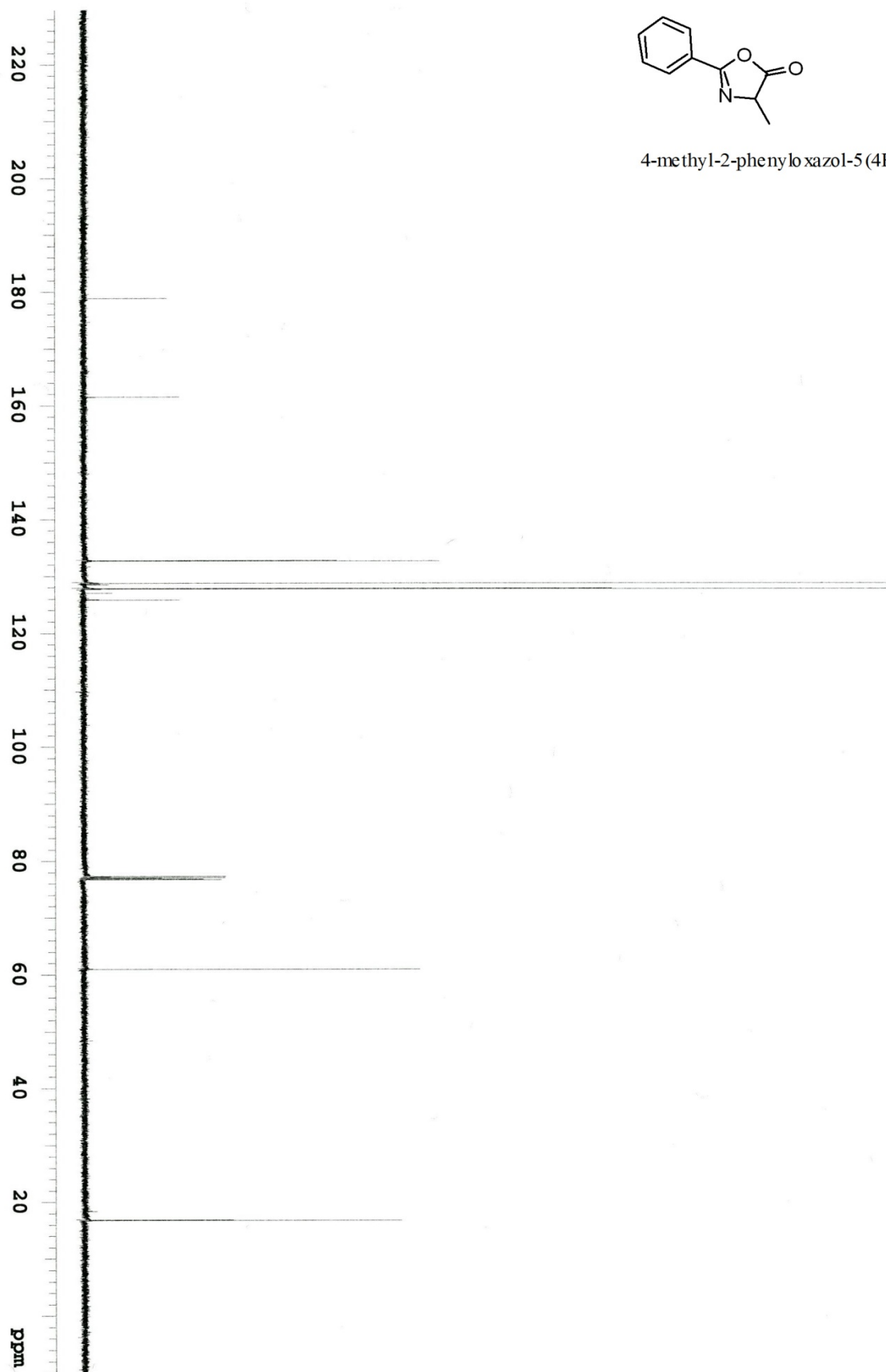


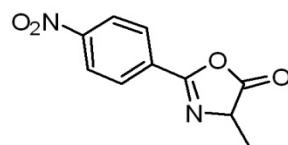
4-methyl-2-phenyl oxazol-5(4H)-one (1)



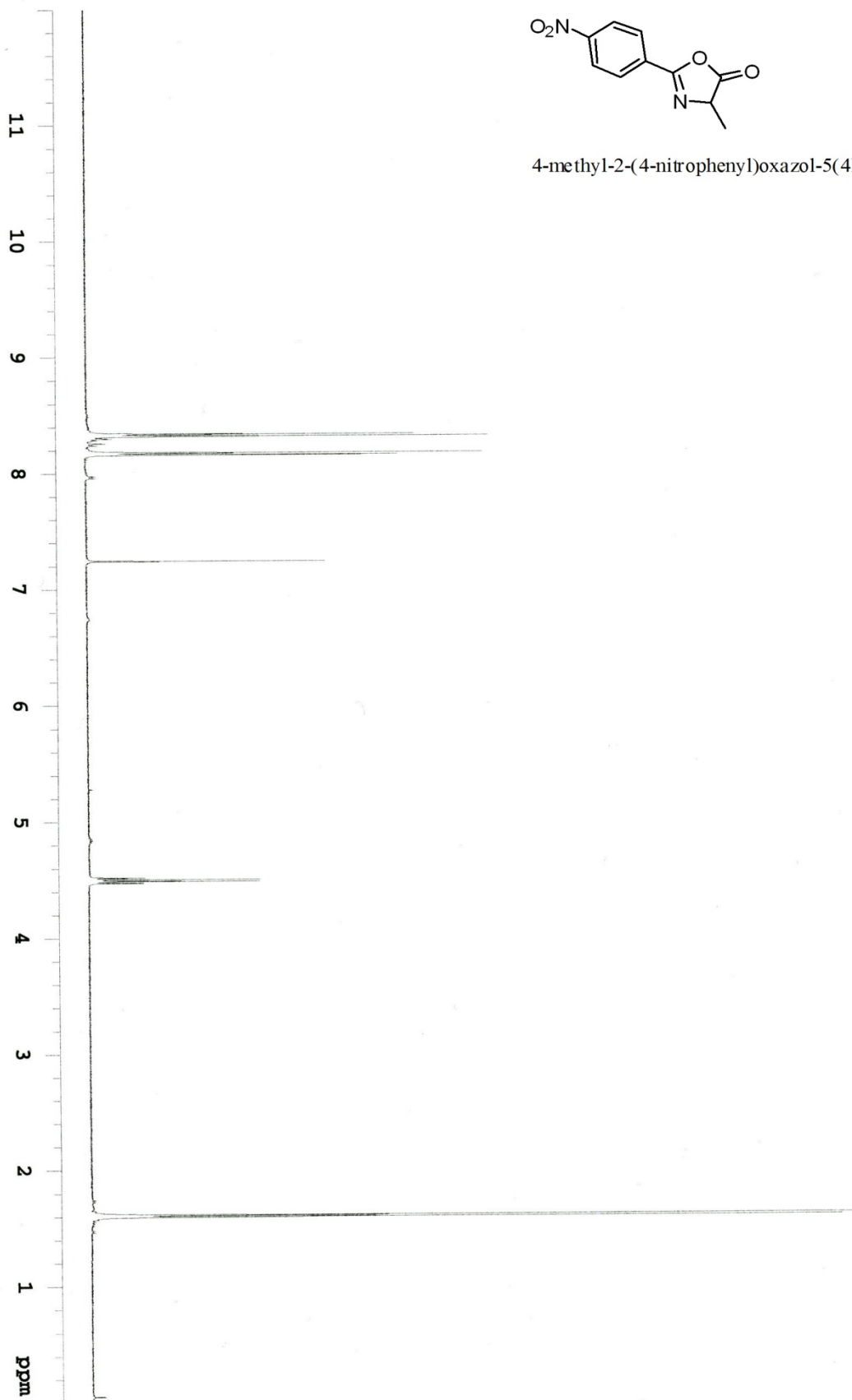


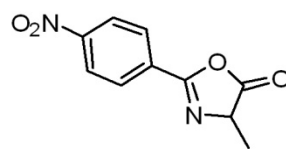
4-methyl-2-phenyloxazol-5(4H)-one (1)



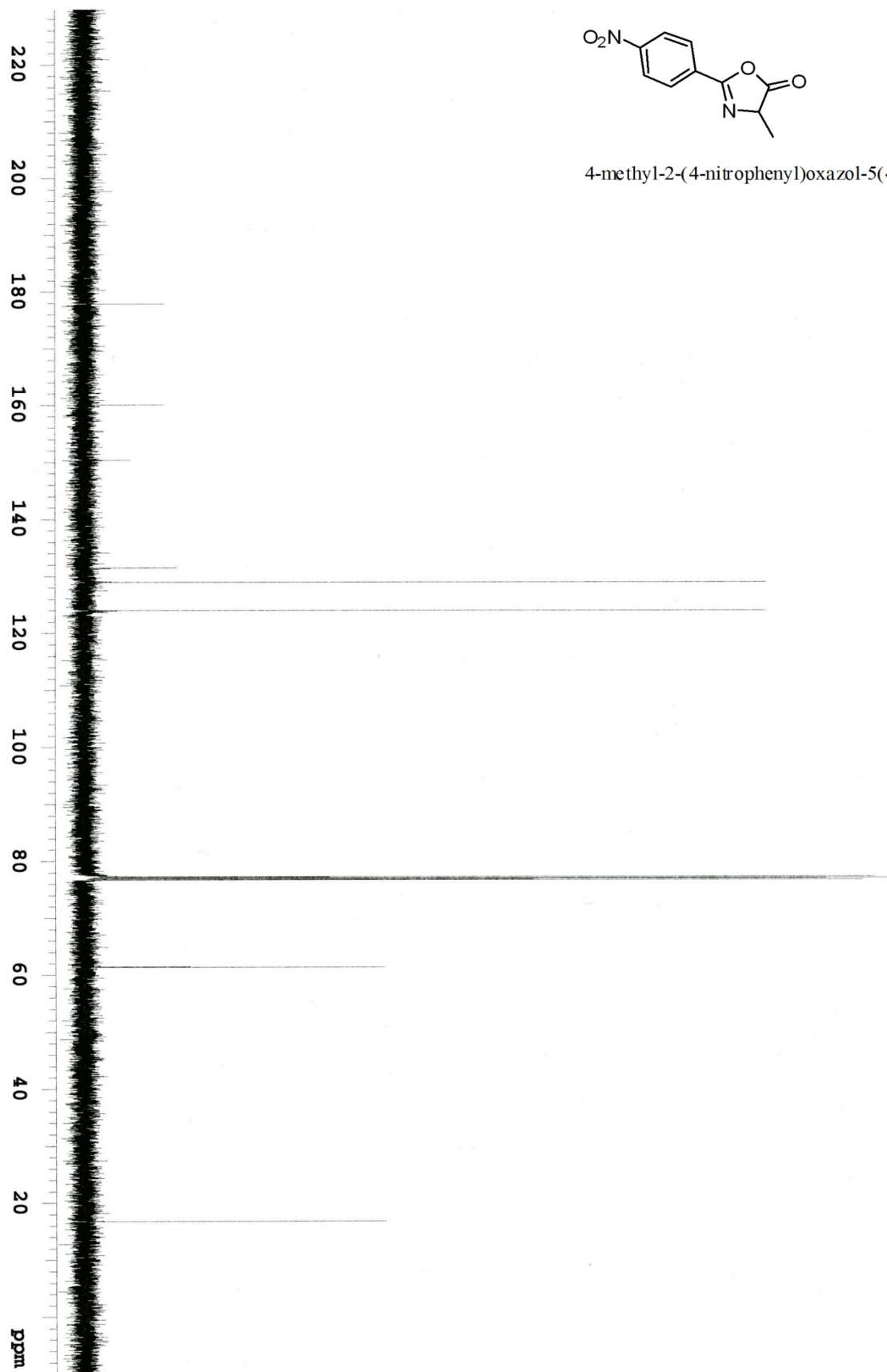


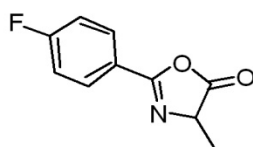
4-methyl-2-(4-nitrophenyl)oxazol-5(4H)-one (5)



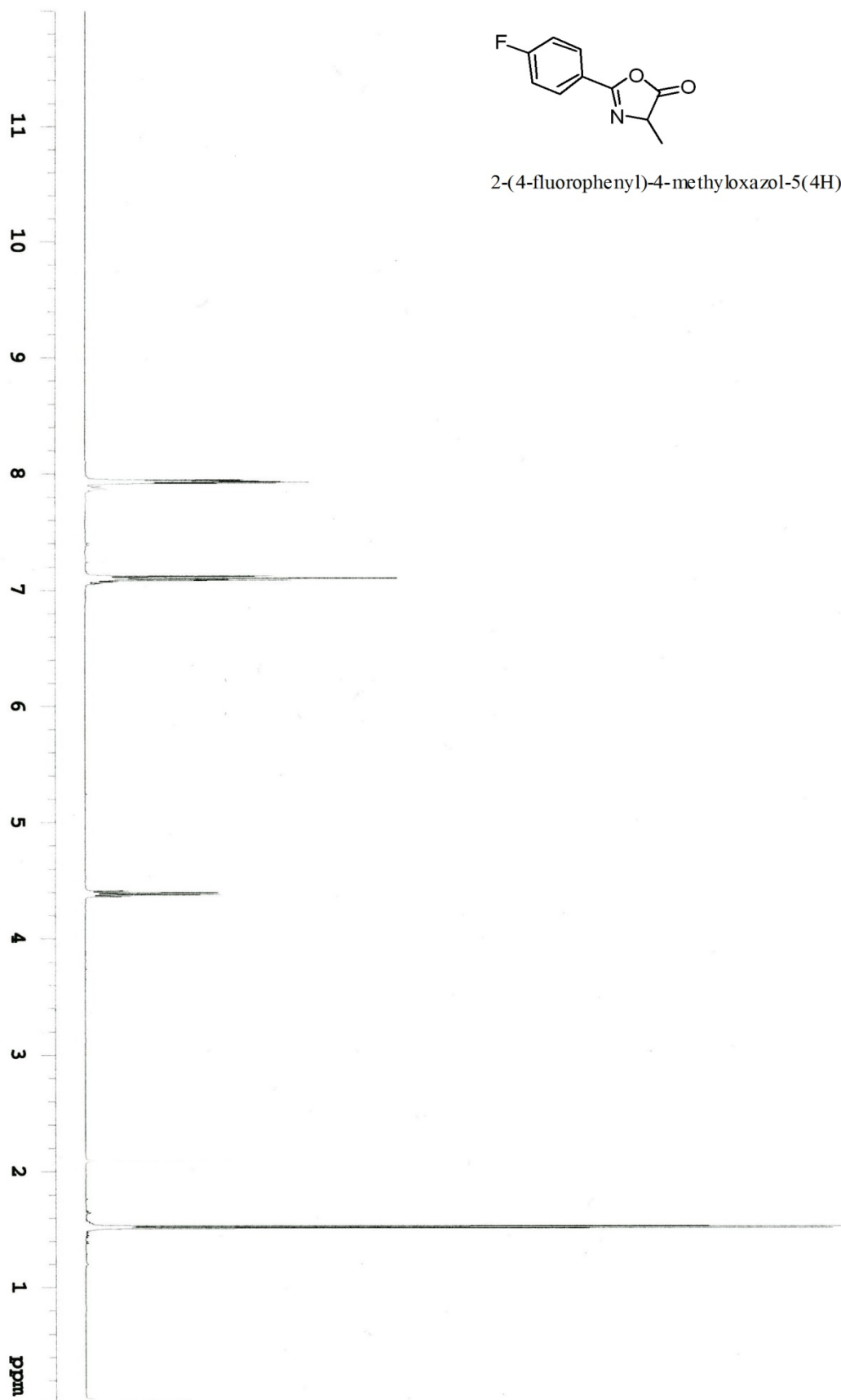


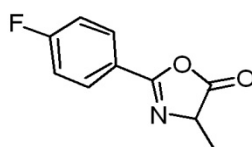
4-methyl-2-(4-nitrophenyl)oxazol-5(4H)-one (5)



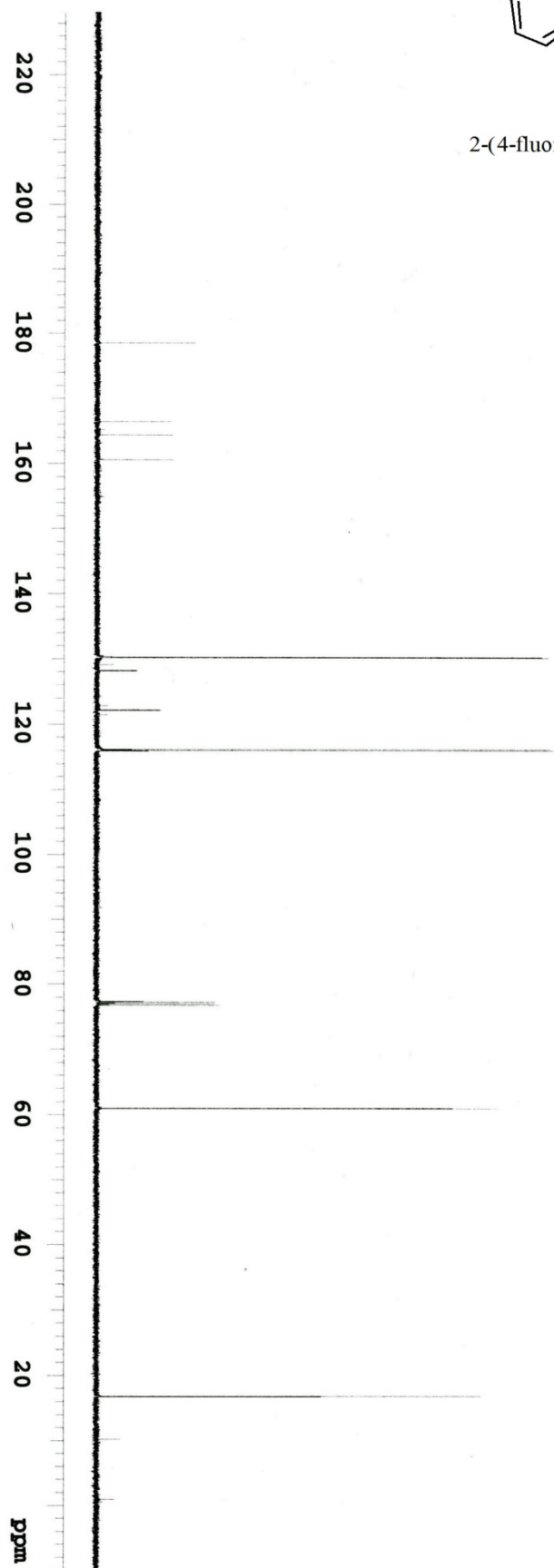


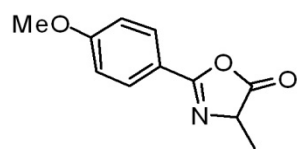
2-(4-fluorophenyl)-4-methyloxazol-5(4H)-one (7)



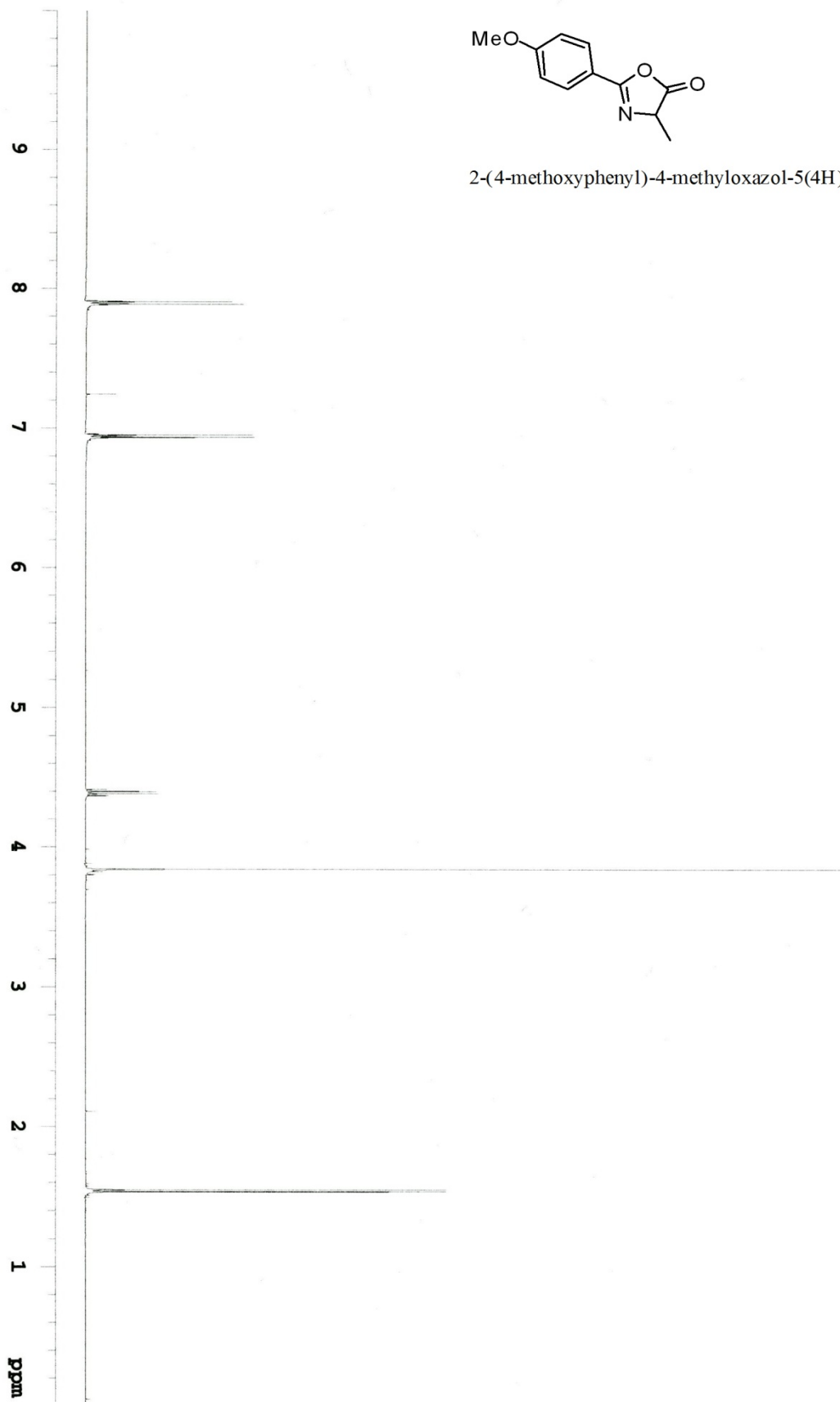


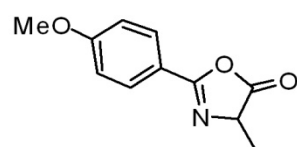
2-(4-fluorophenyl)-4-methyloxazol-5(4H)-one (7)



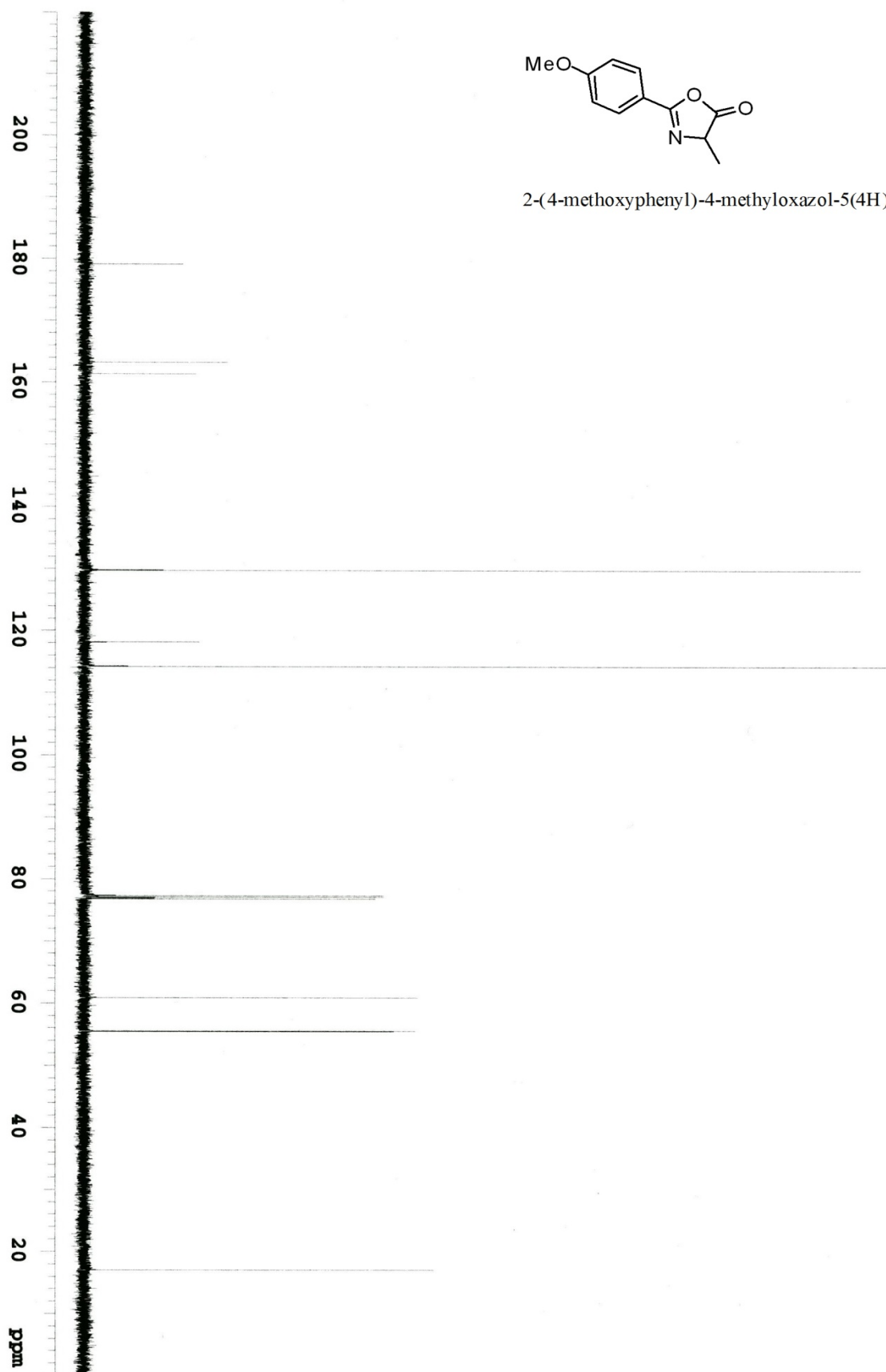


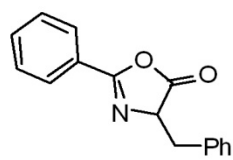
2-(4-methoxyphenyl)-4-methyloxazol-5(4H)-one (9)



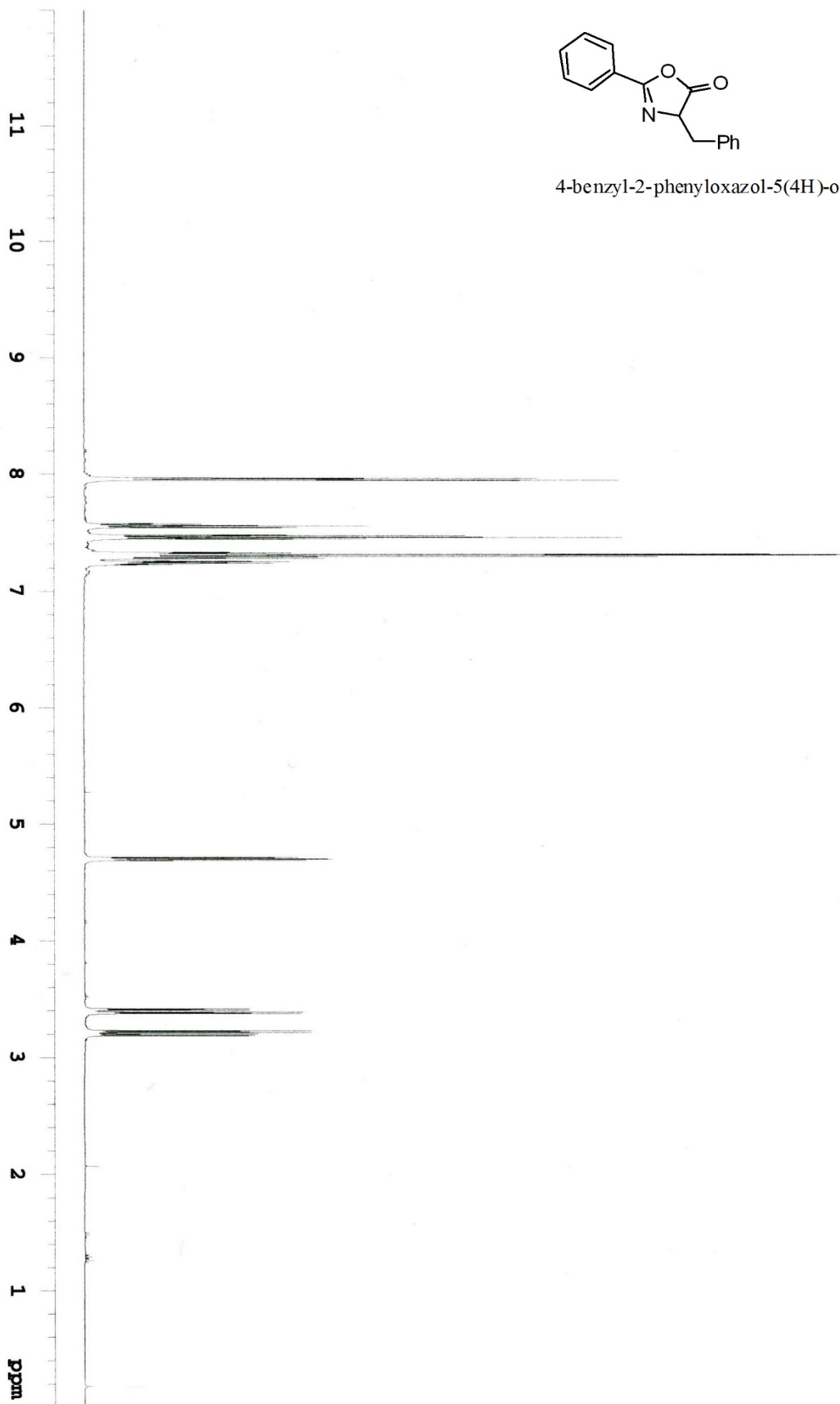


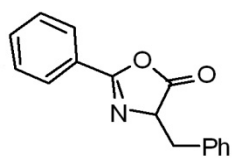
2-(4-methoxyphenyl)-4-methyloxazol-5(4H)-one (9)



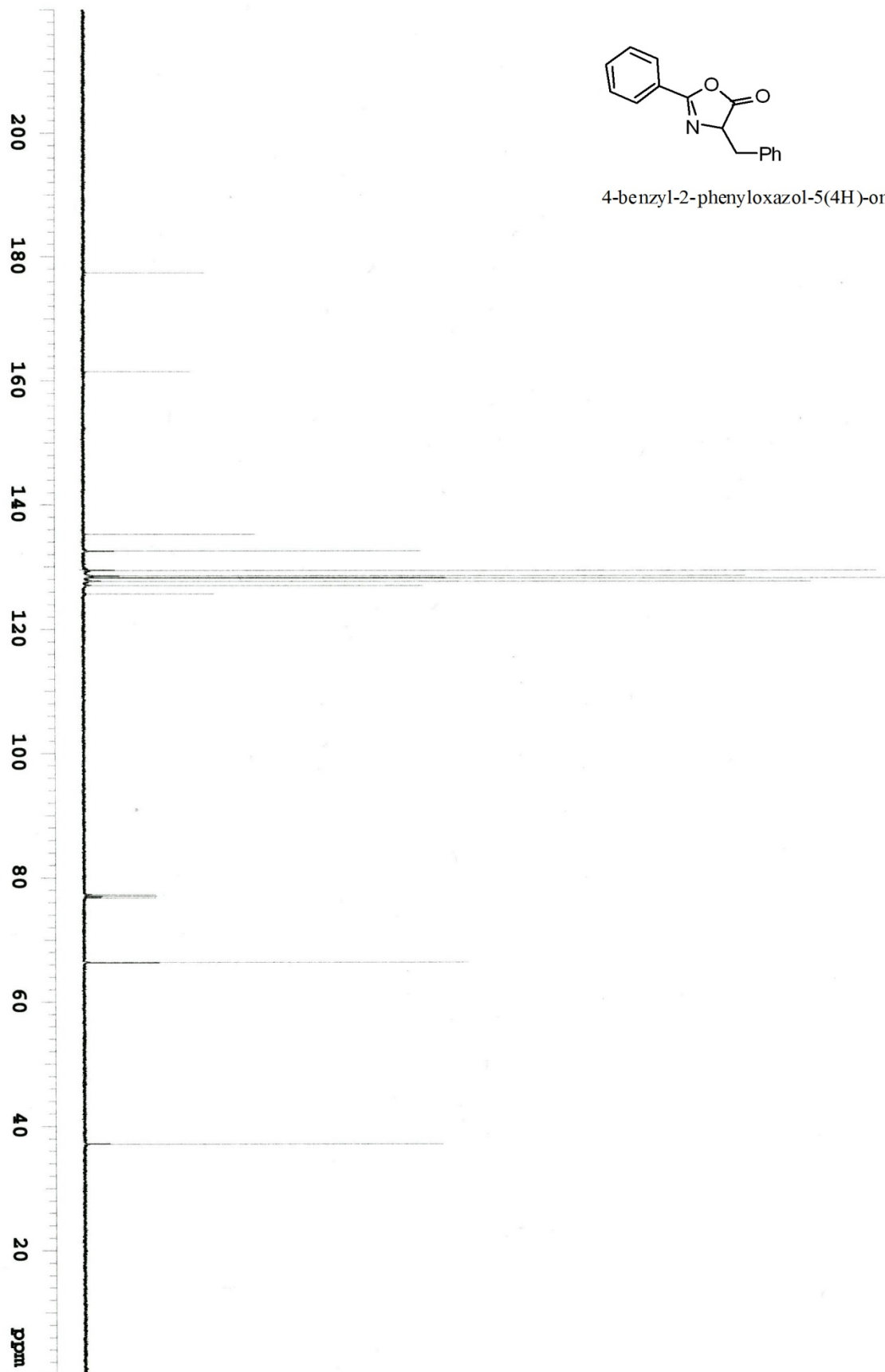


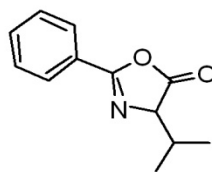
4-benzyl-2-phenyloxazol-5(4H)-one (11)



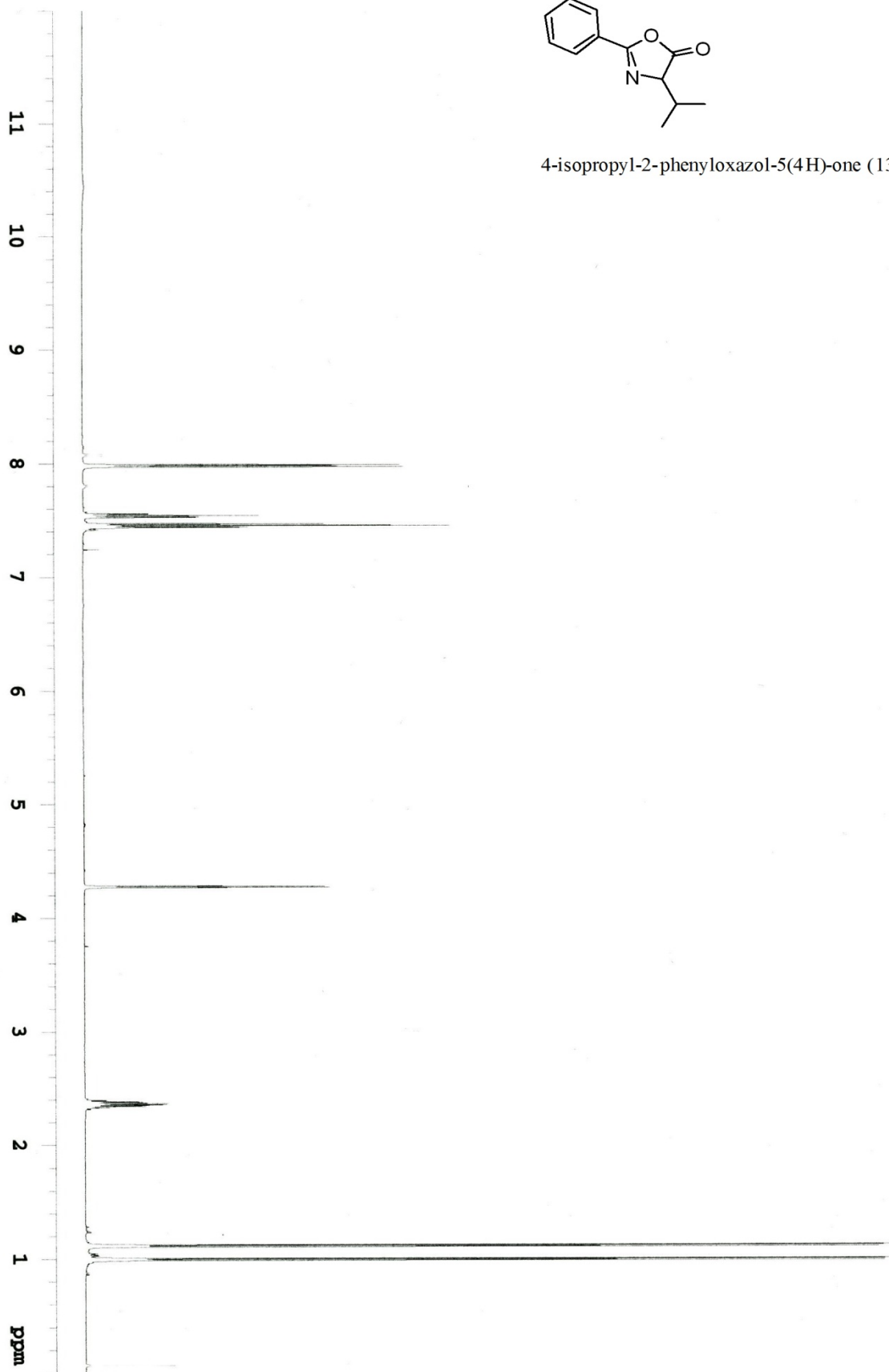


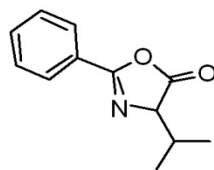
4-benzyl-2-phenyloxazol-5(4H)-one (11)



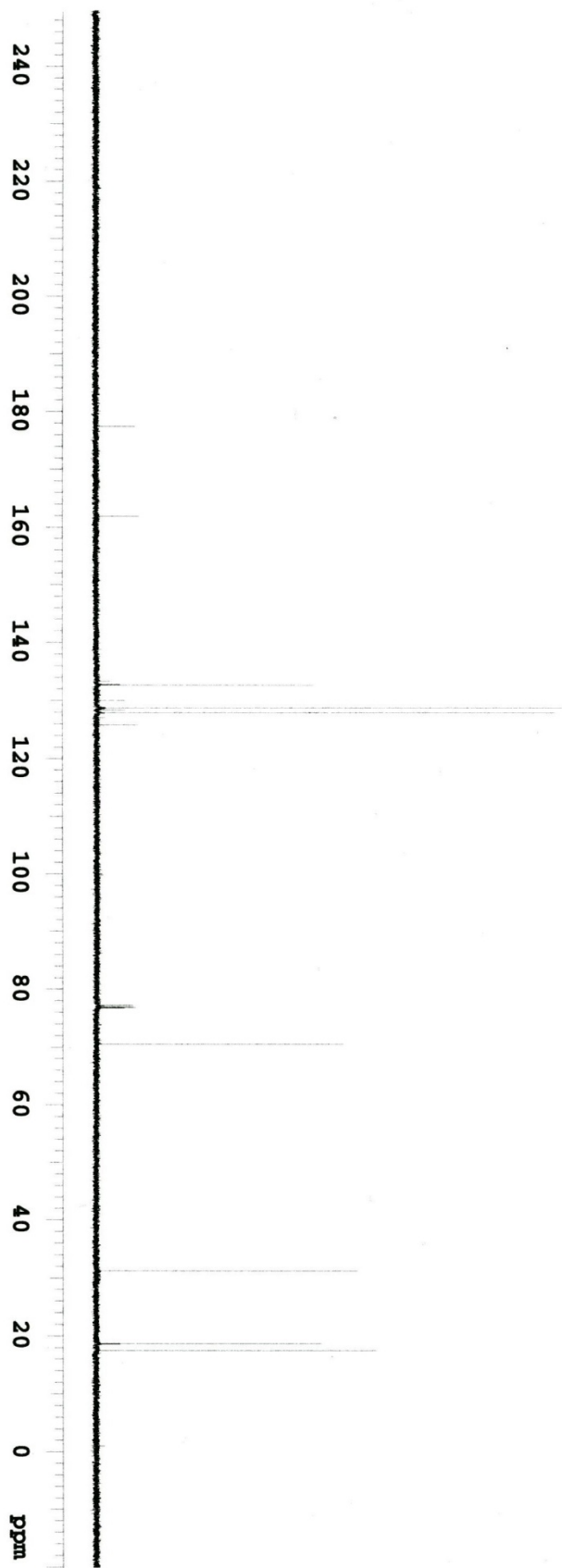


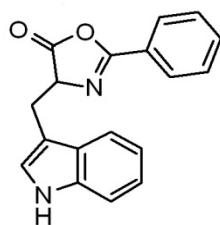
4-isopropyl-2-phenyloxazol-5(4H)-one (13)



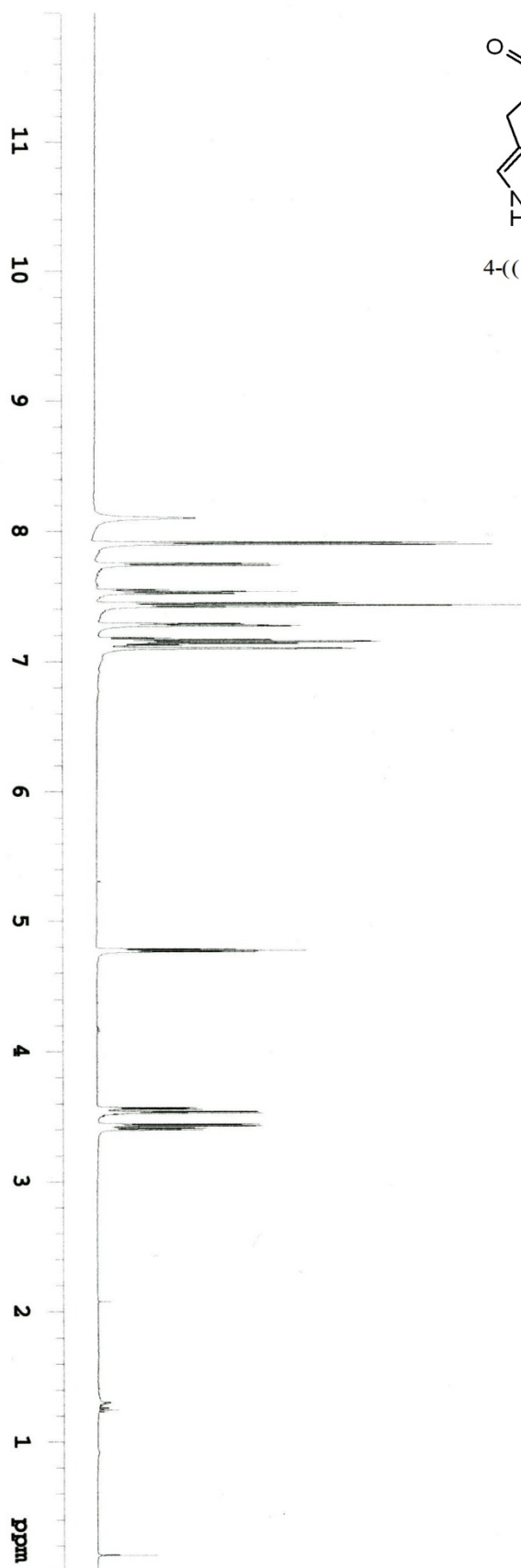


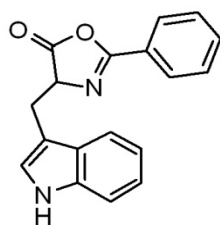
4-isopropyl-2-phenyloxazol-5(4H)-one (13)



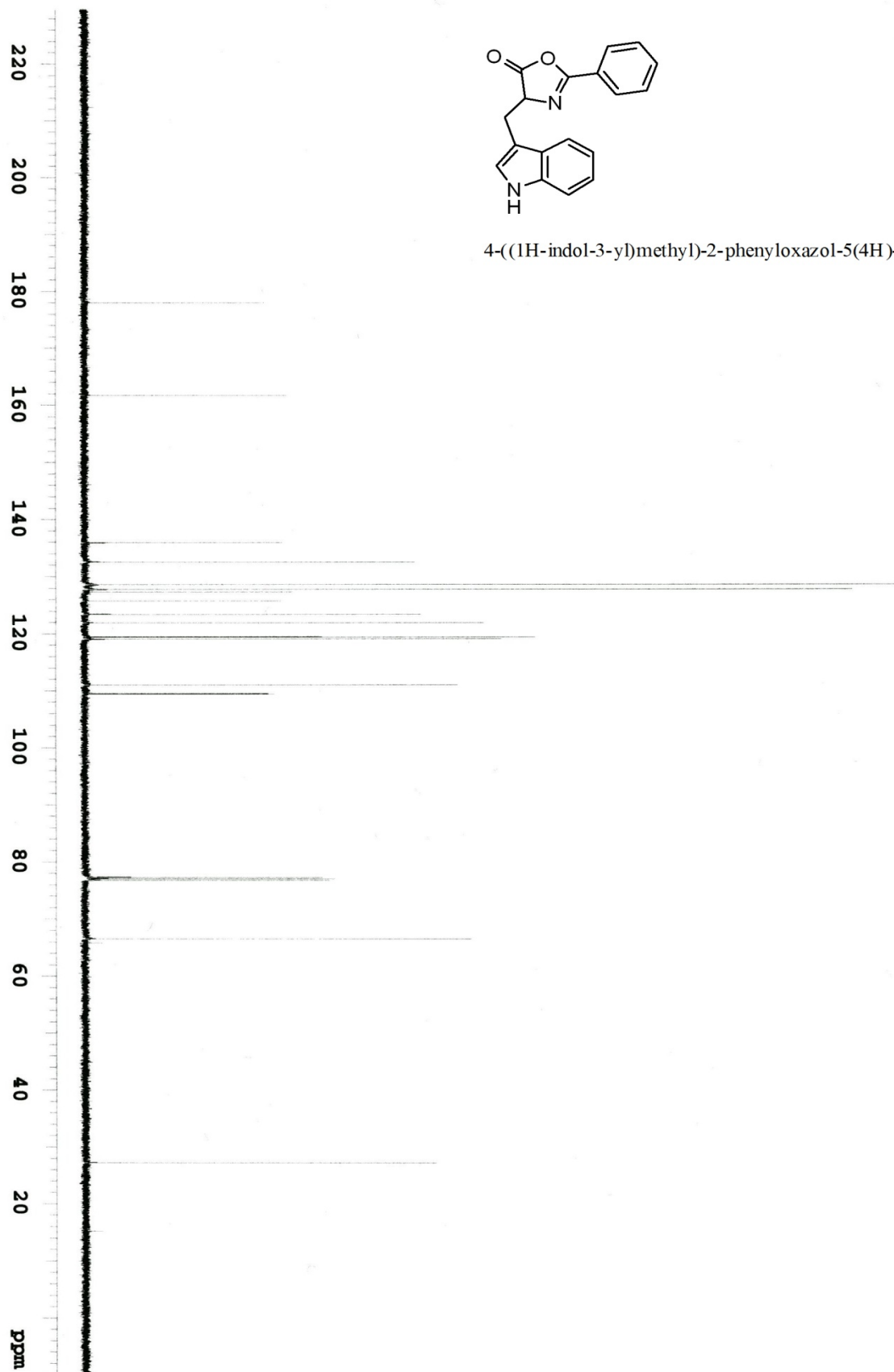


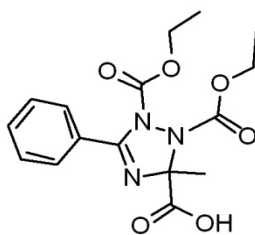
4-((1H-indol-3-yl)methyl)-2-phenyloxazol-5(4H)-one (15) :



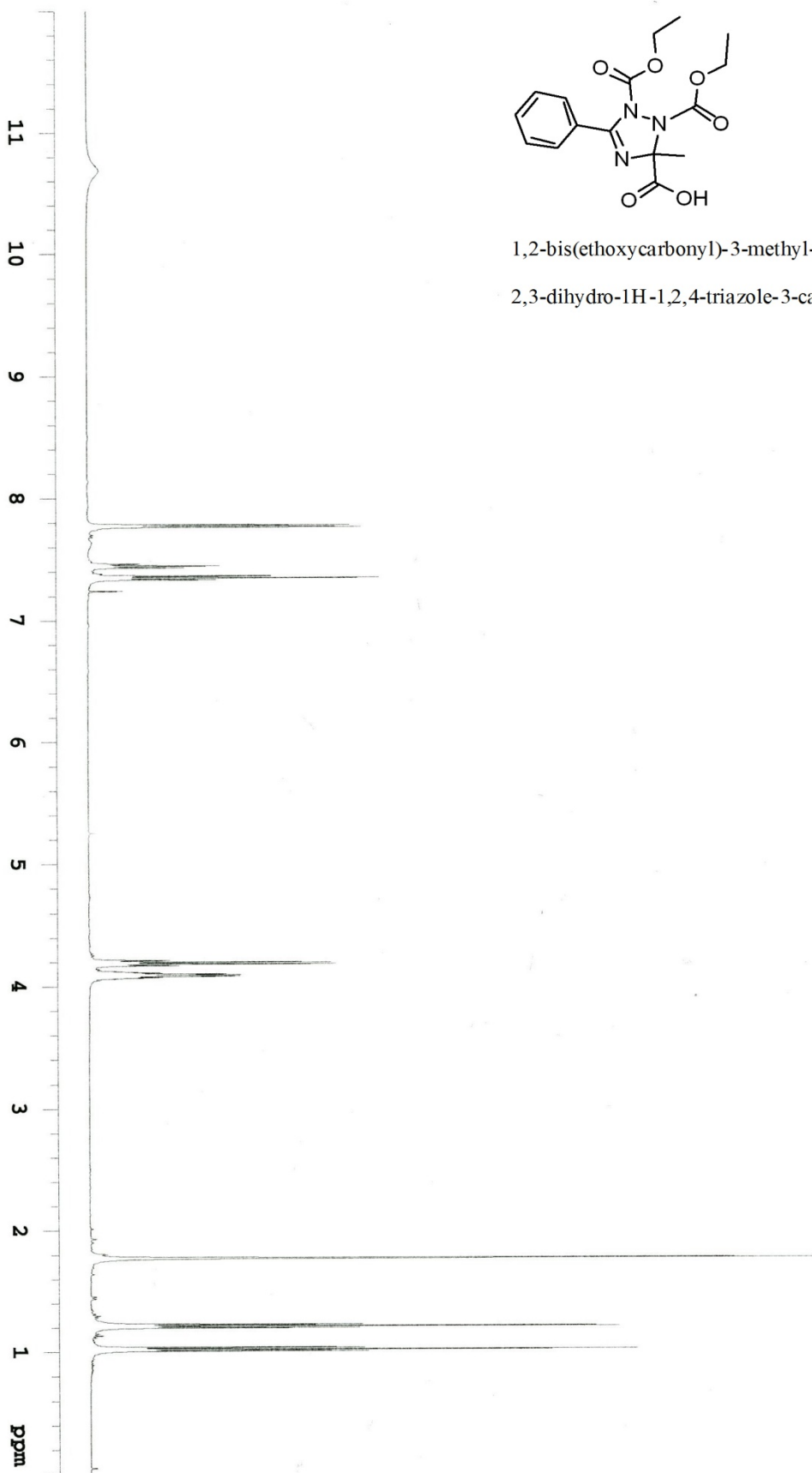


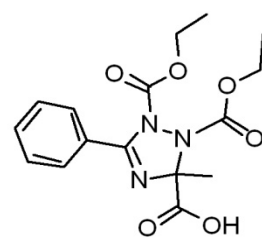
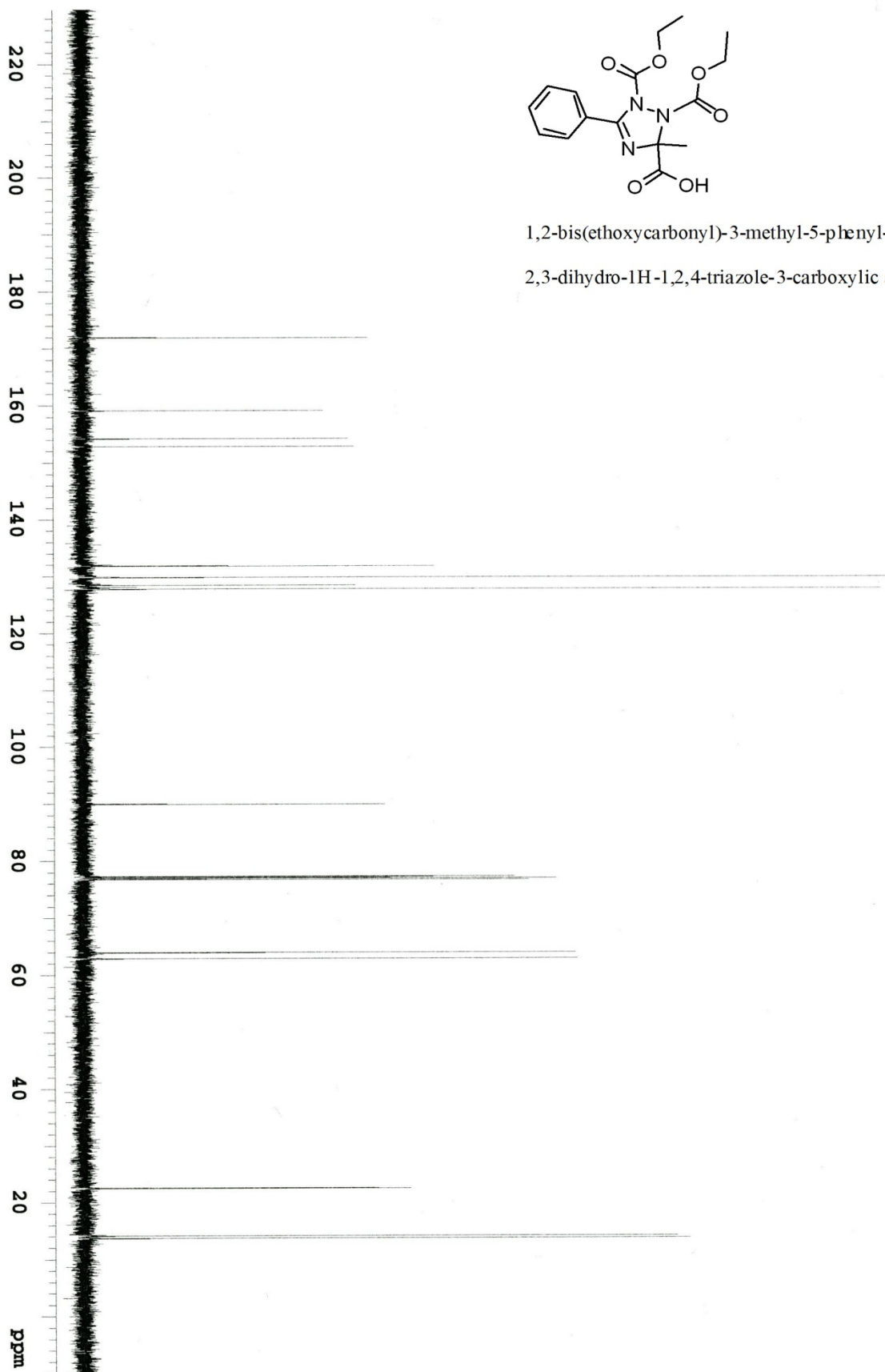
4-((1H-indol-3-yl)methyl)-2-phenyloxazol-5(4H)-one (15) :



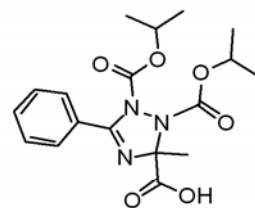


1,2-bis(ethoxycarbonyl)-3-methyl-5-phenyl-
2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (2)

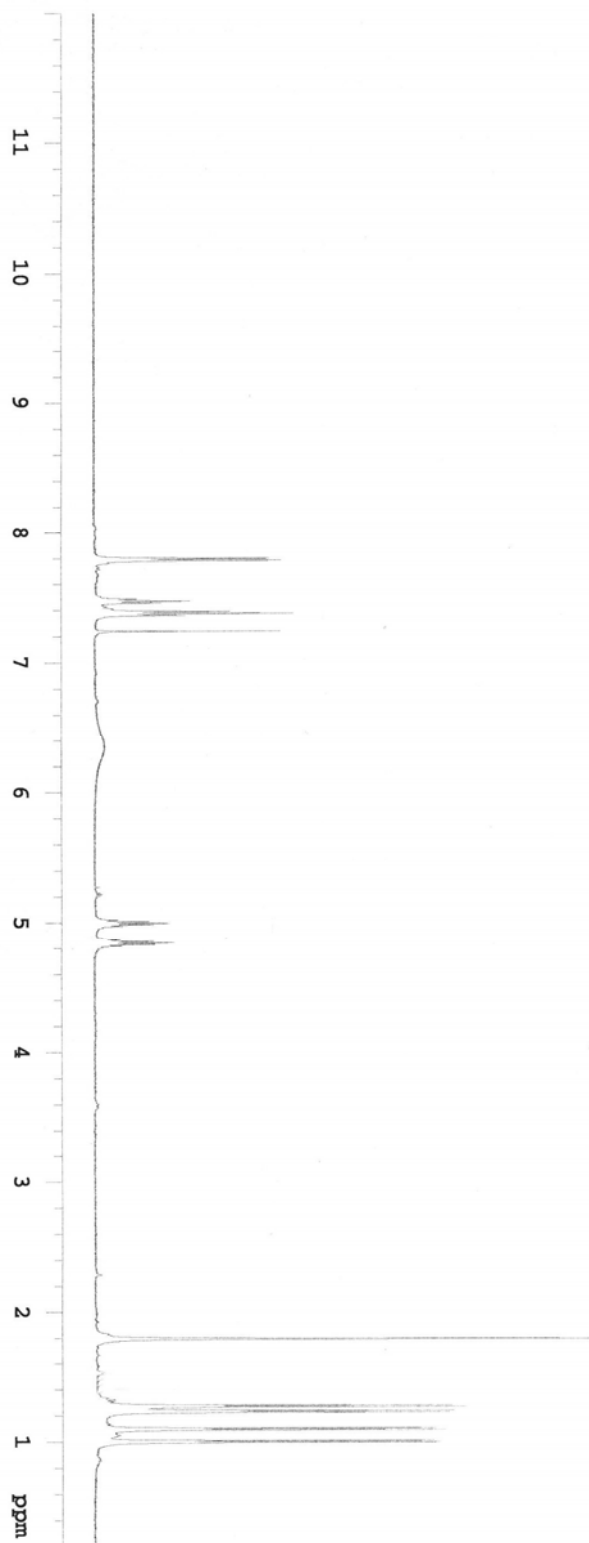


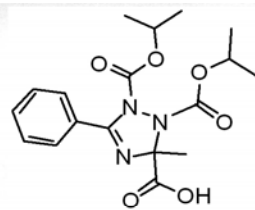


1,2-bis(ethoxycarbonyl)-3-methyl-5-phenyl-
2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (2)

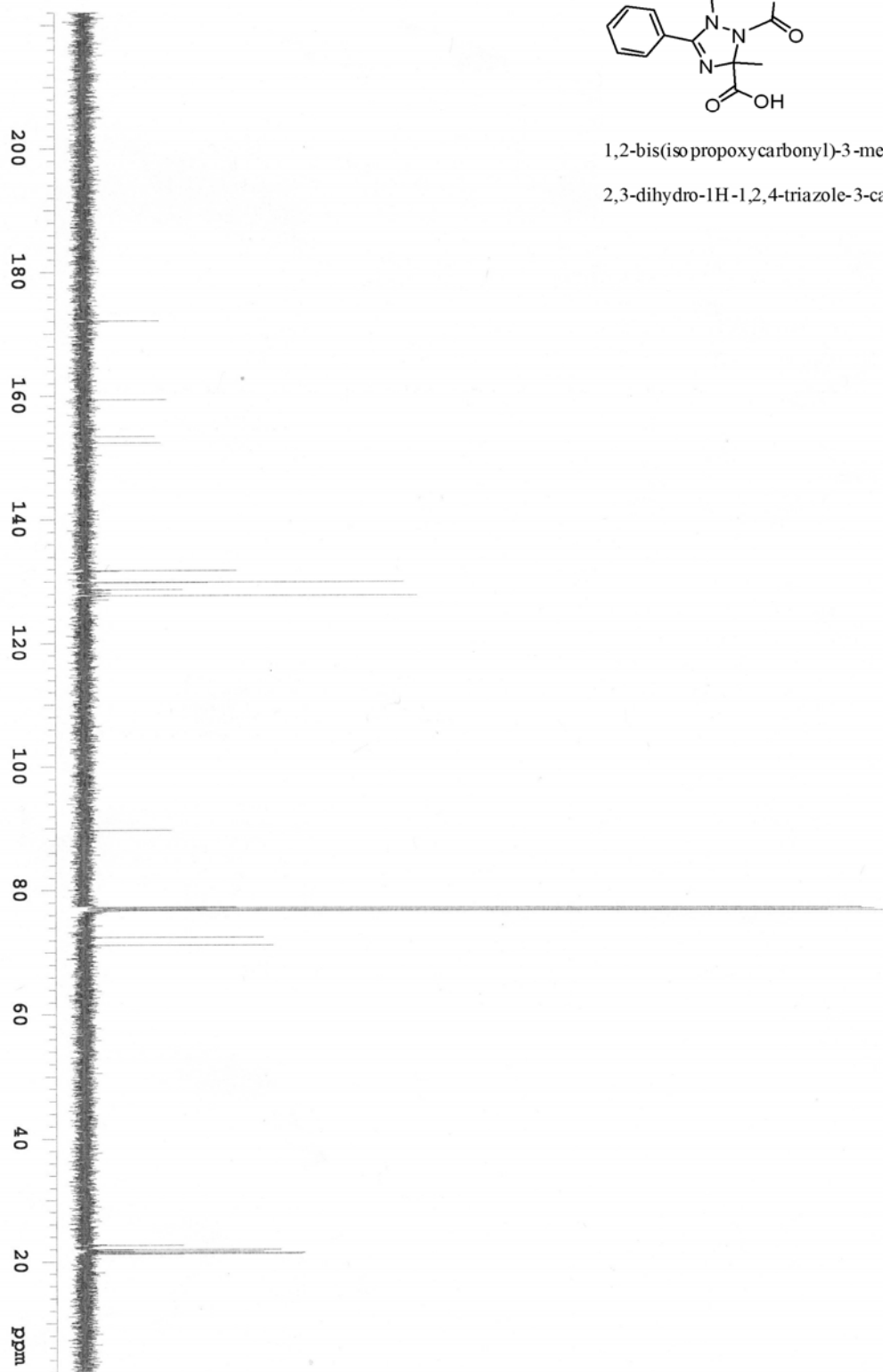


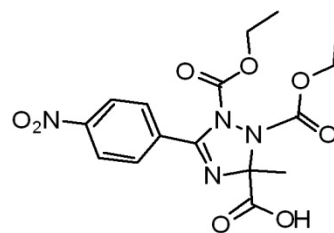
1,2-bis(isopropoxycarbonyl)-3-methyl-5-phenyl-
2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (3)



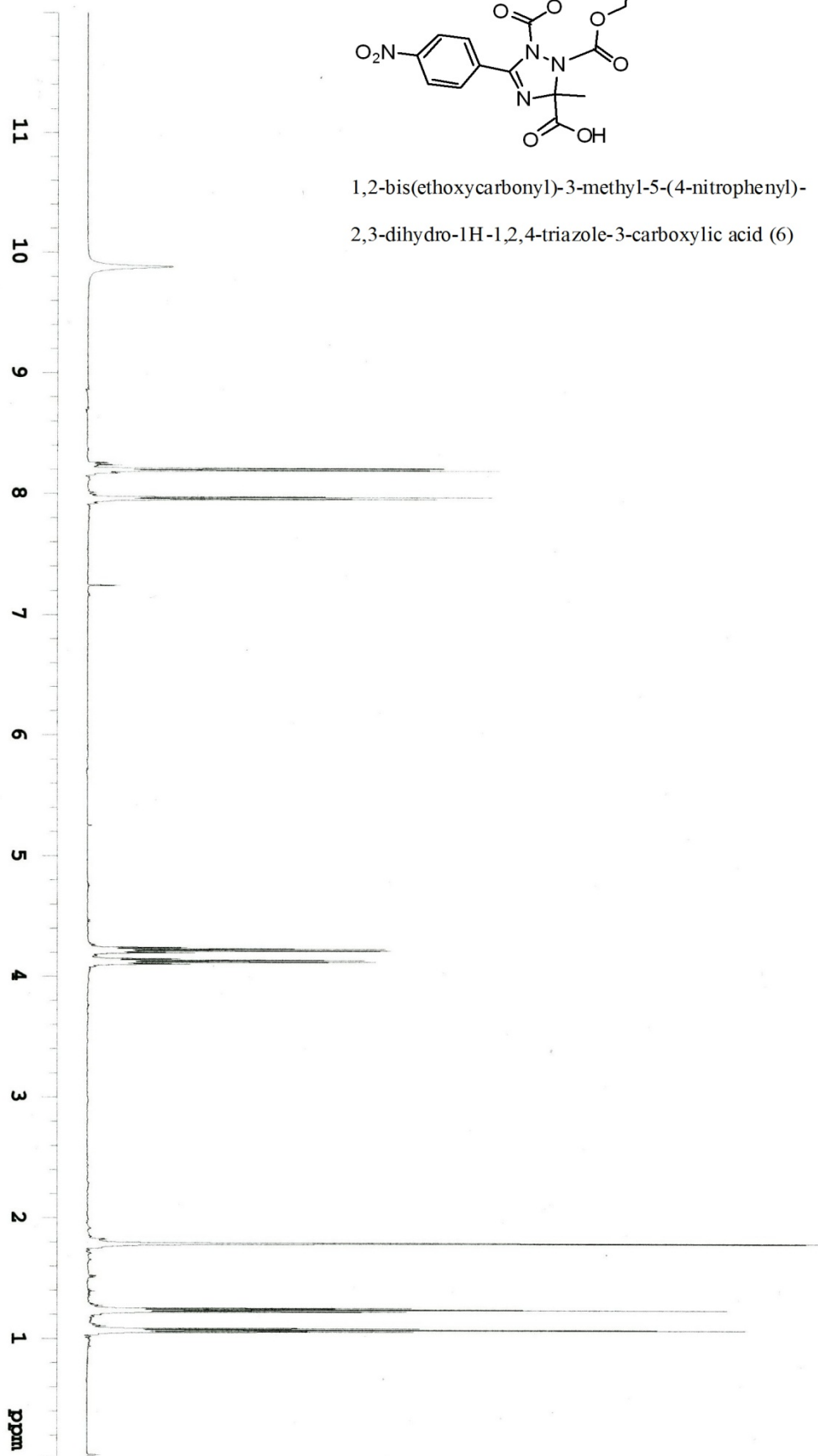


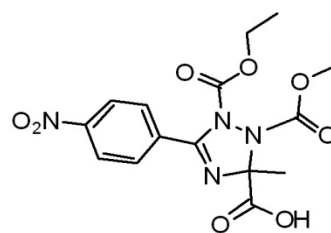
1,2-bis(isopropoxycarbonyl)-3-methyl-5-phenyl-
2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (3)



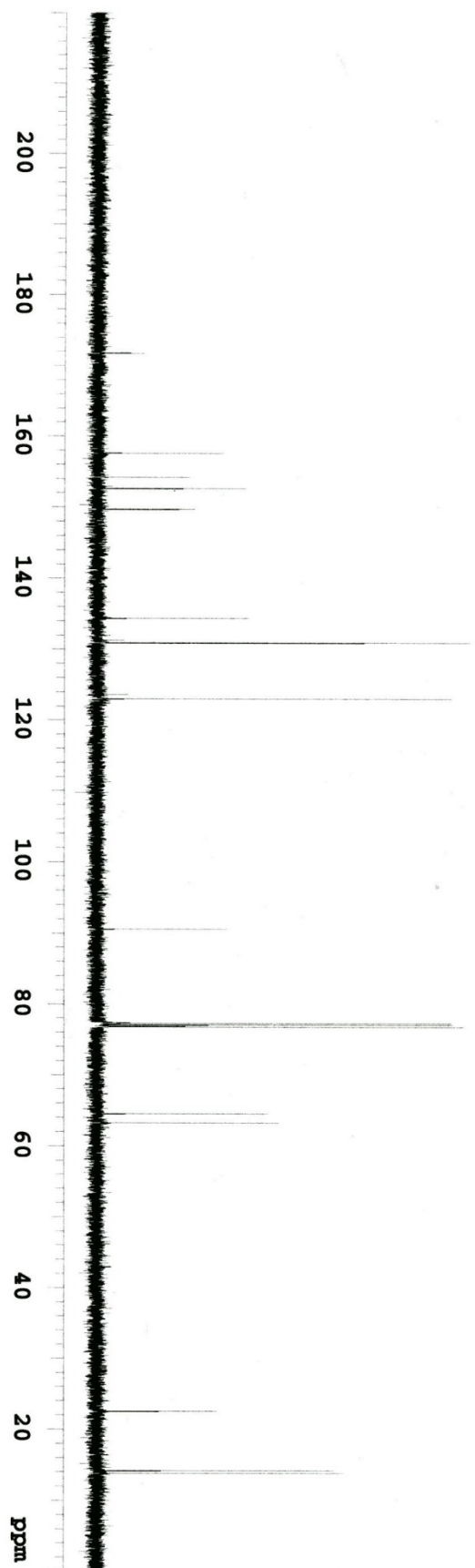


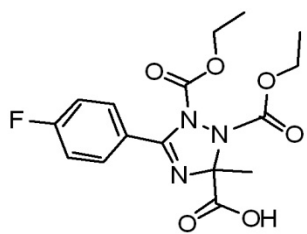
1,2-bis(ethoxycarbonyl)-3-methyl-5-(4-nitrophenyl)-
2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (6)





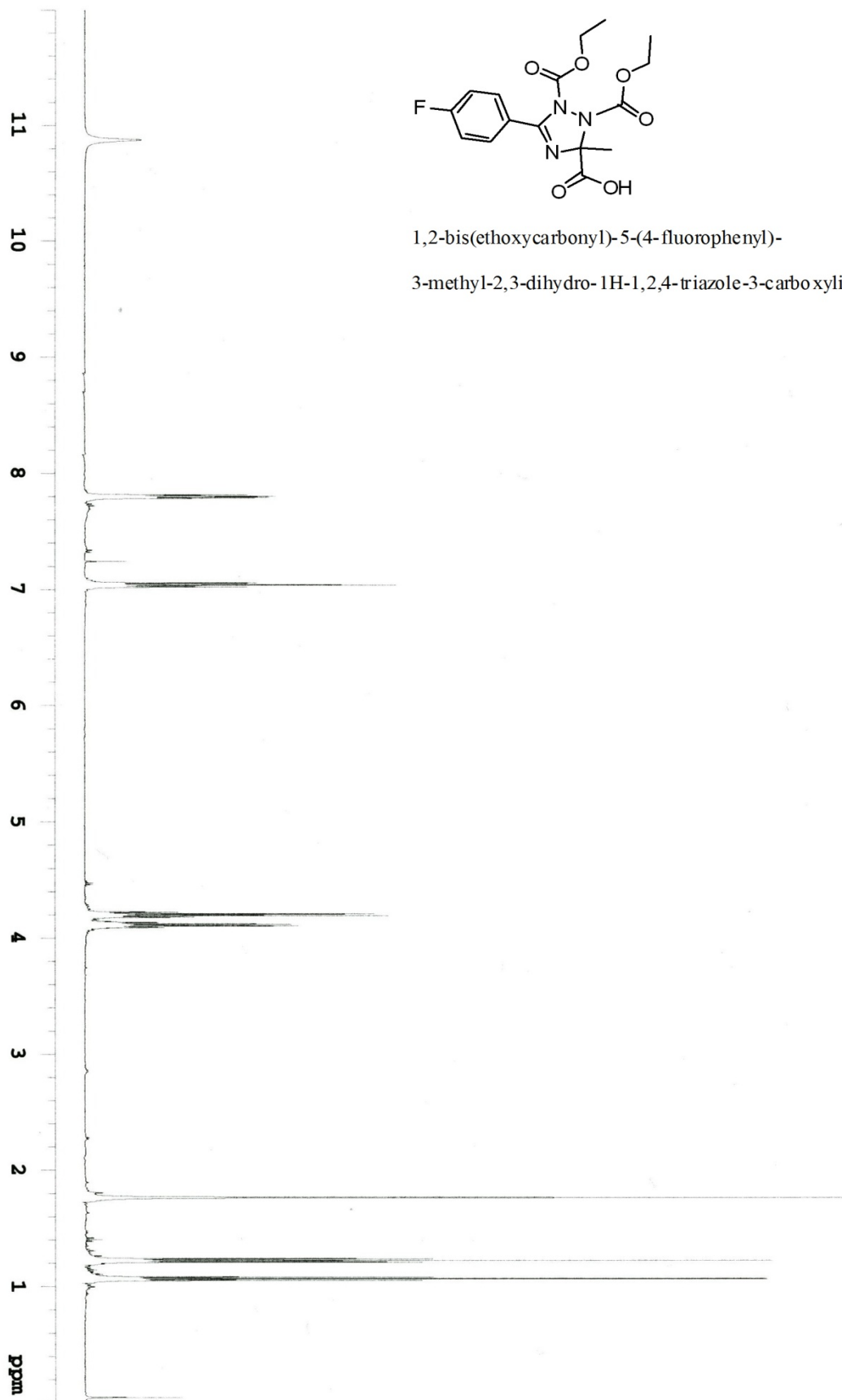
1,2-bis(ethoxycarbonyl)-3-methyl-5-(4-nitrophenyl)-
2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (6)

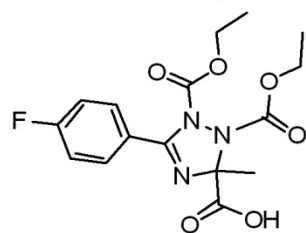




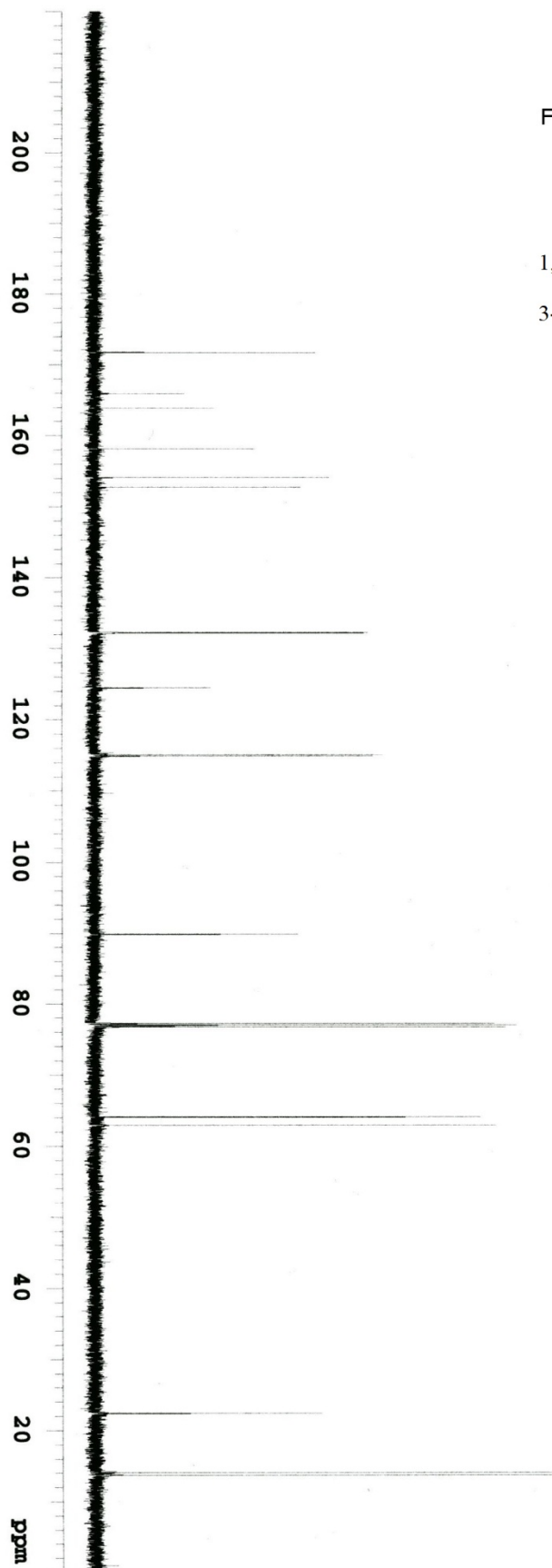
1,2-bis(ethoxycarbonyl)-5-(4-fluorophenyl)-

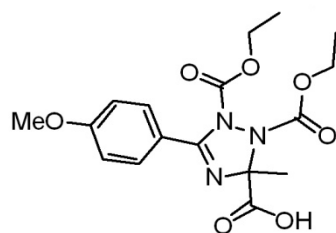
3-methyl-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (8)



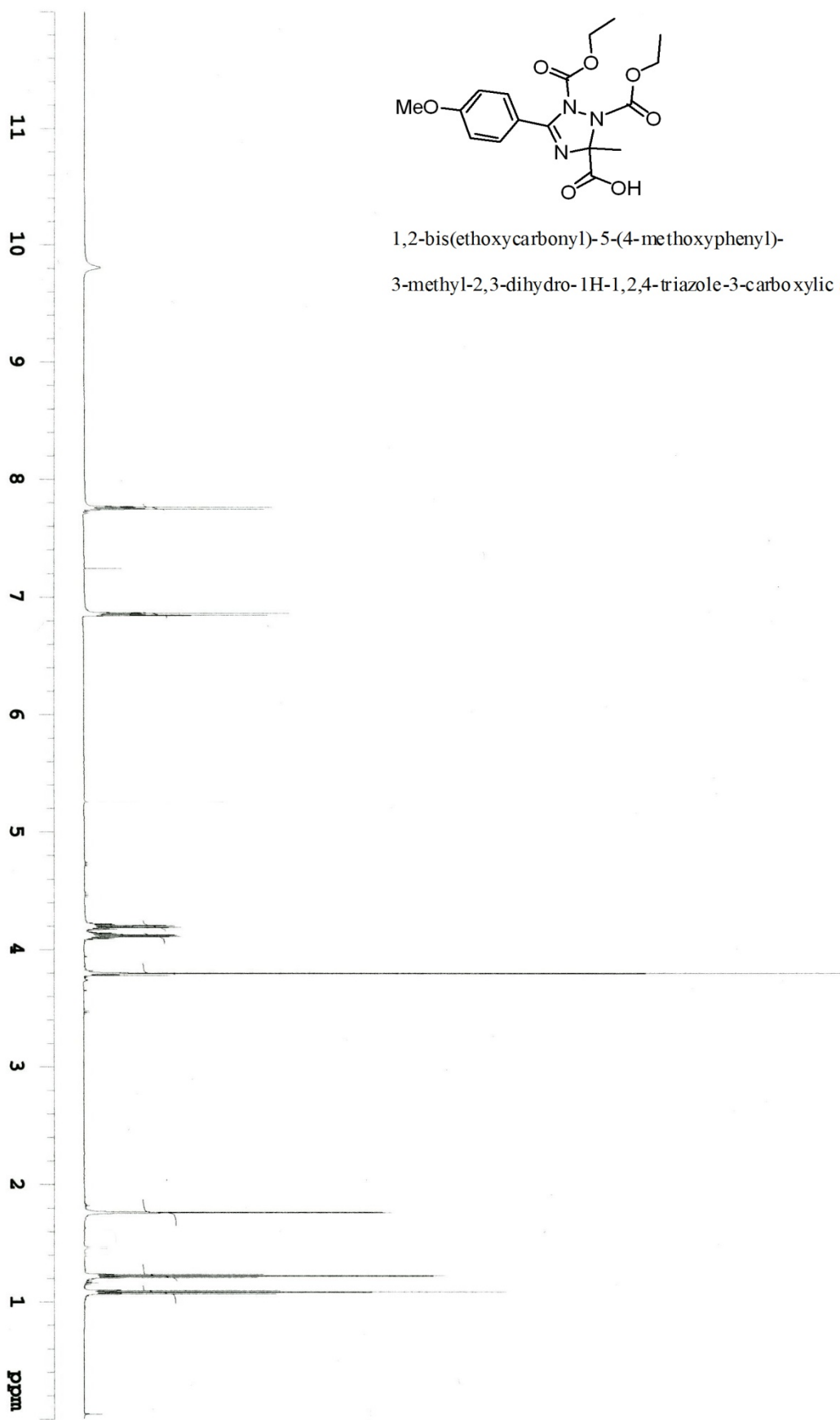


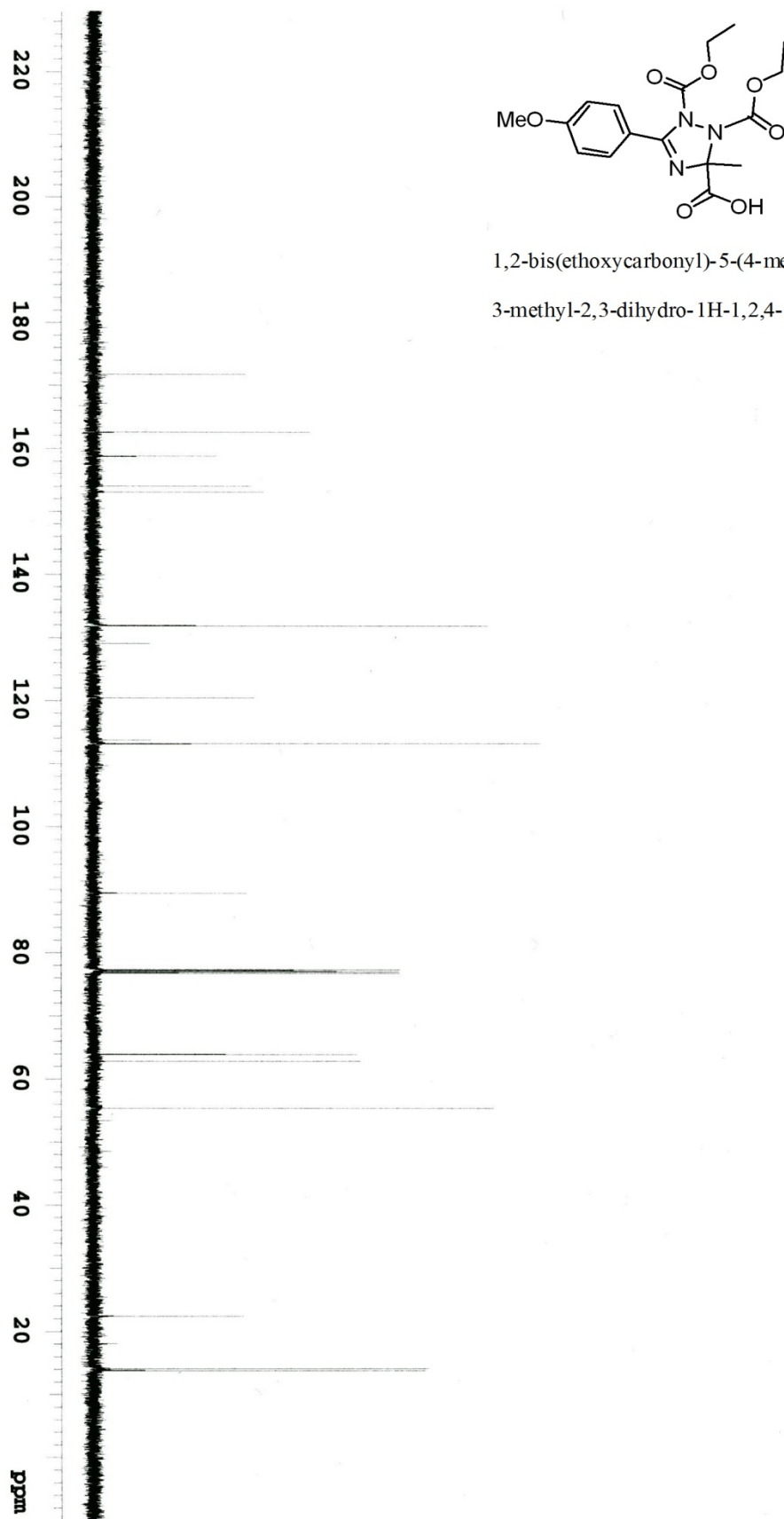
1,2-bis(ethoxycarbonyl)-5-(4-fluorophenyl)-
3-methyl-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (8)



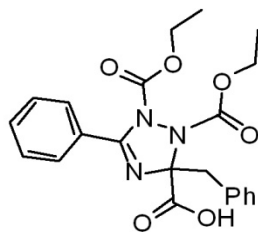


1,2-bis(ethoxycarbonyl)-5-(4-methoxyphenyl)-
3-methyl-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (10)



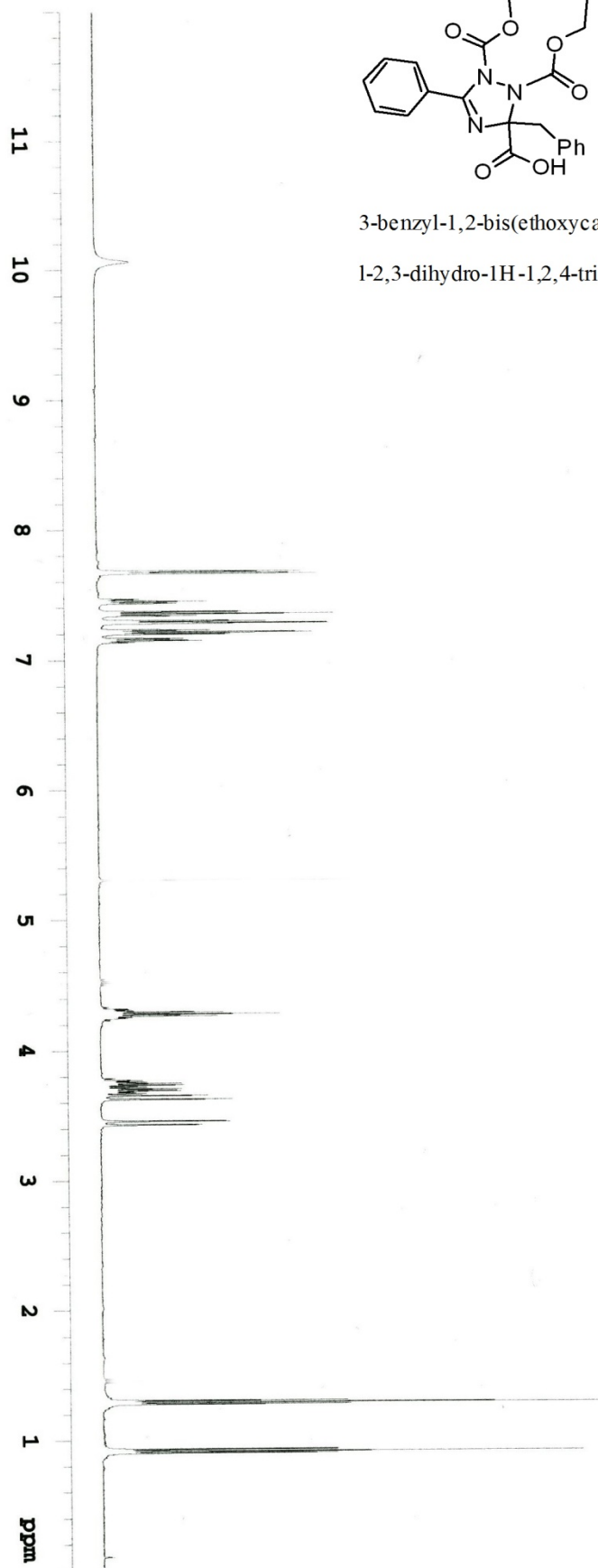


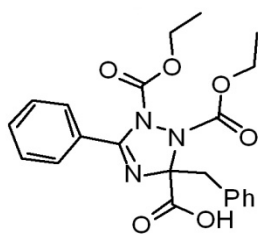
1,2-bis(ethoxycarbonyl)-5-(4-methoxyphenyl)-
3-methyl-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (10)



3-benzyl-1,2-bis(ethoxycarbonyl)-5-phenyl

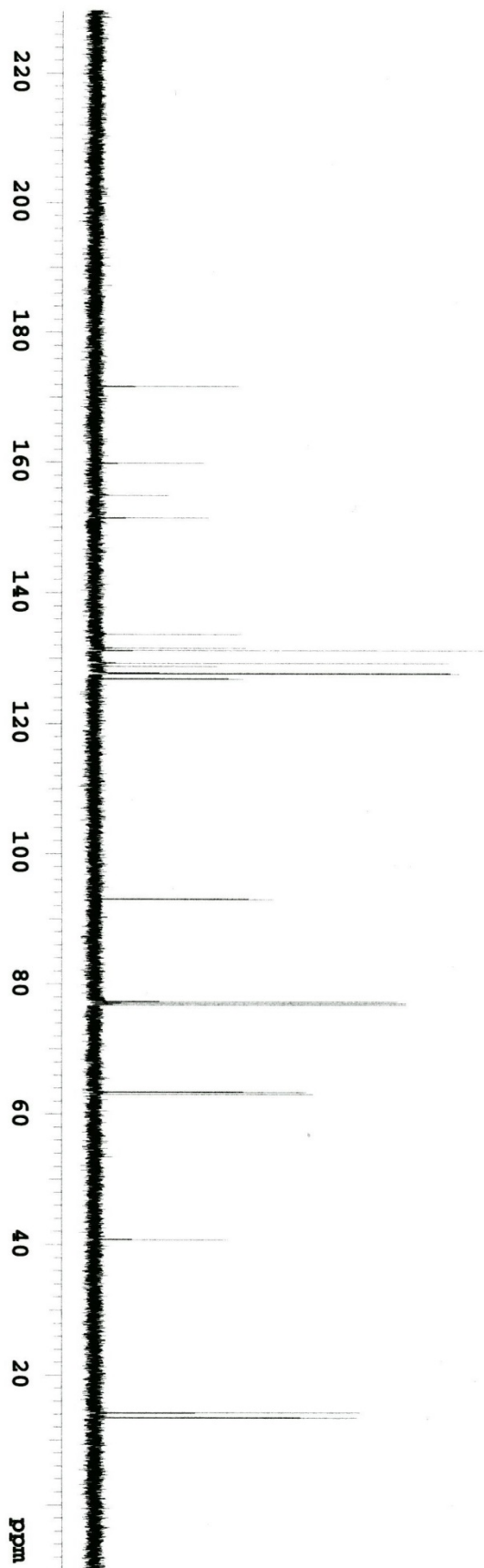
1-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (12):

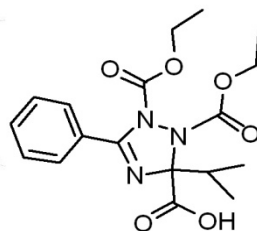




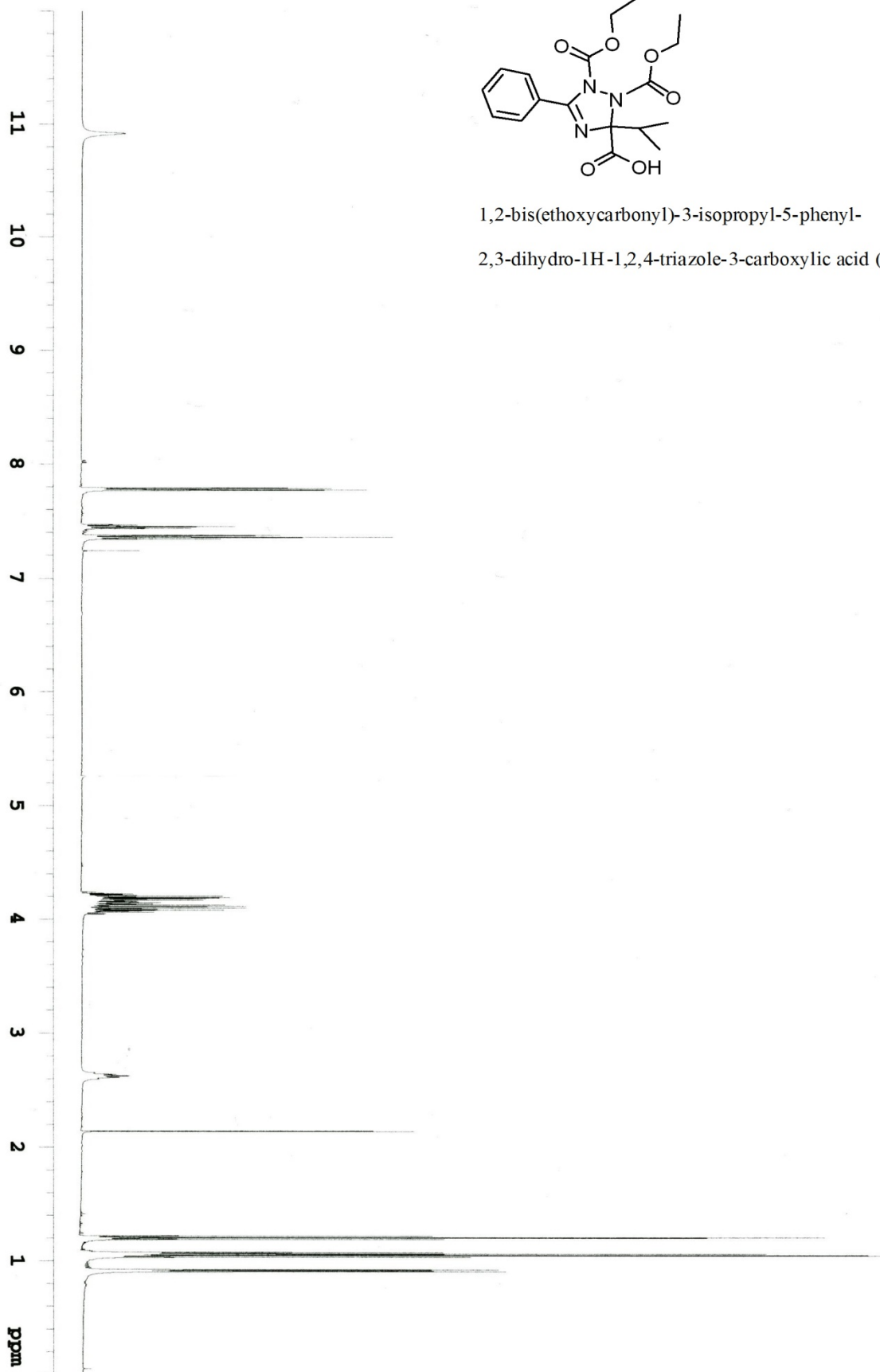
3-benzyl-1,2-bis(ethoxycarbonyl)-5-phenyl

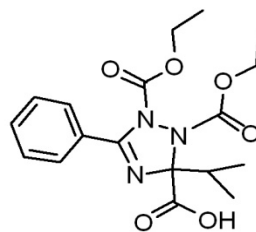
1-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (12):



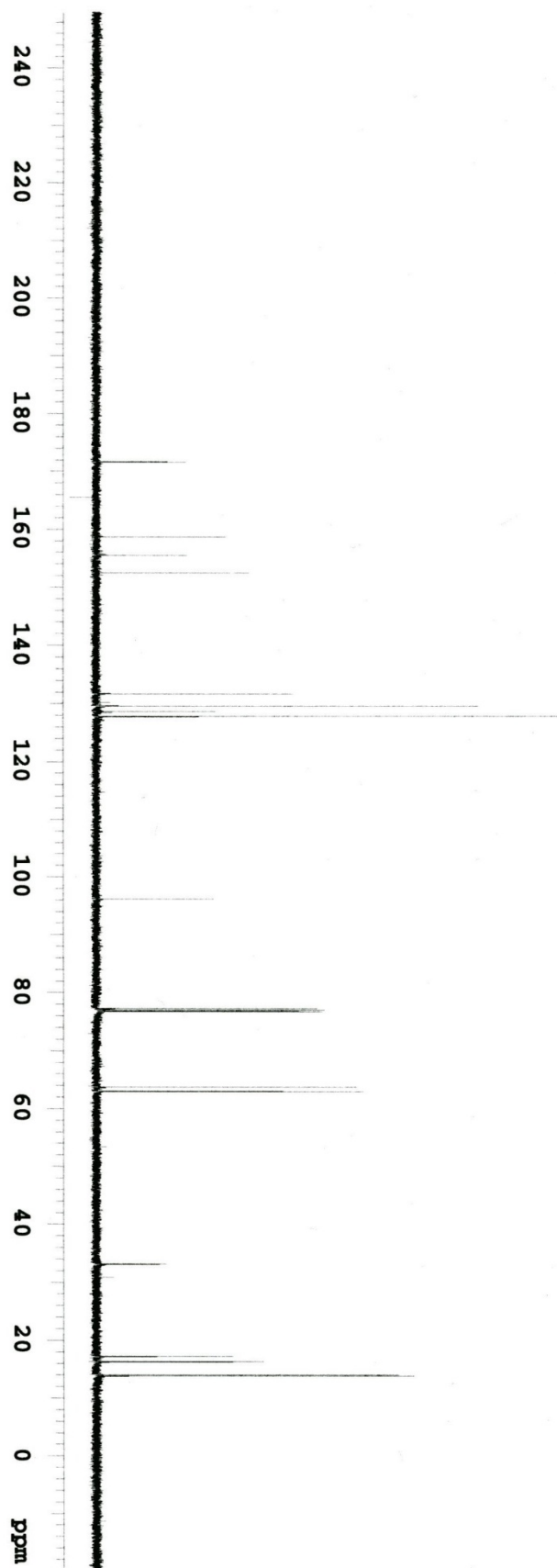


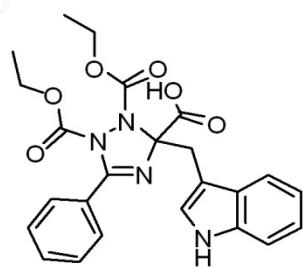
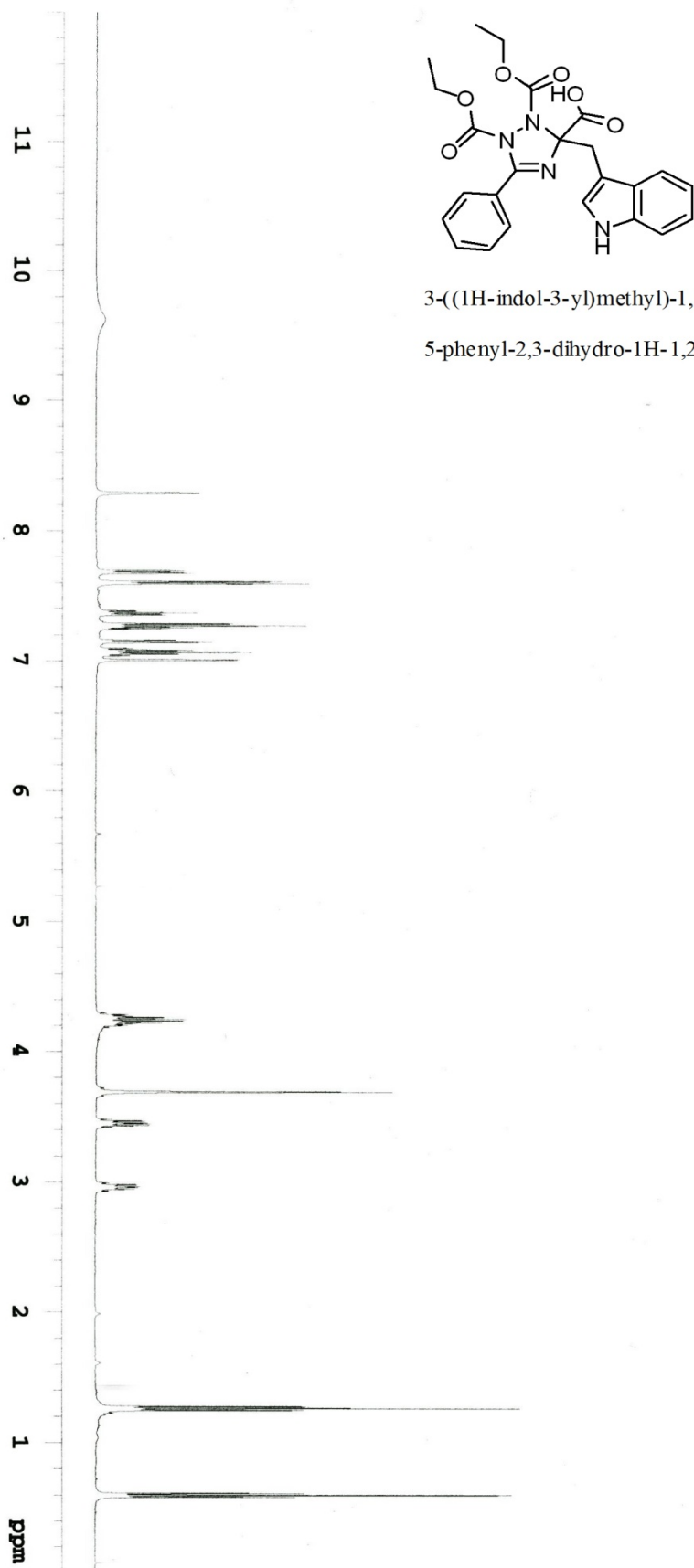
1,2-bis(ethoxycarbonyl)-3-isopropyl-5-phenyl-
2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (14)



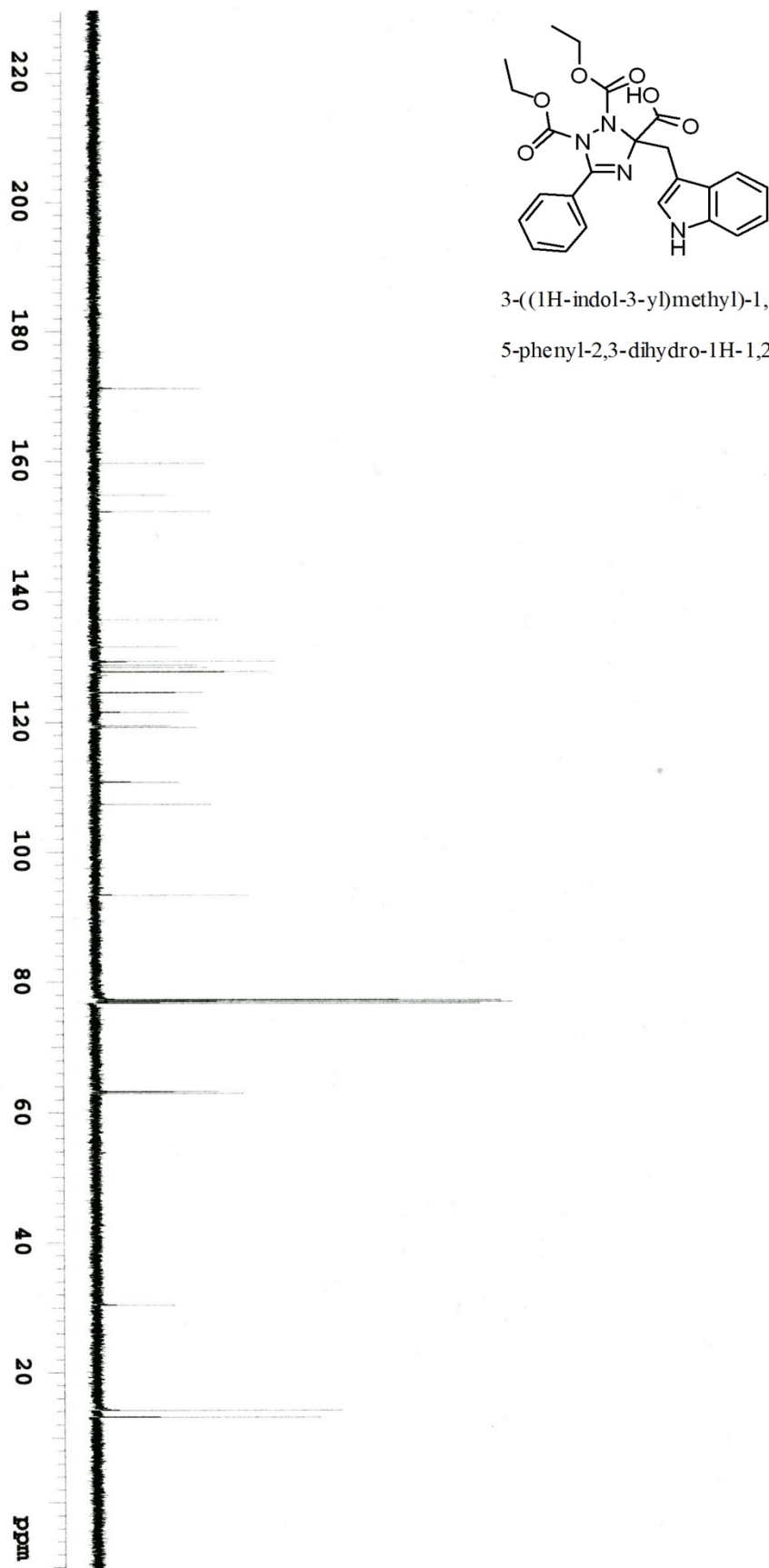


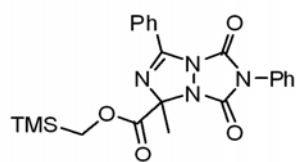
1,2-bis(ethoxycarbonyl)-3-isopropyl-5-phenyl-
2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (14)



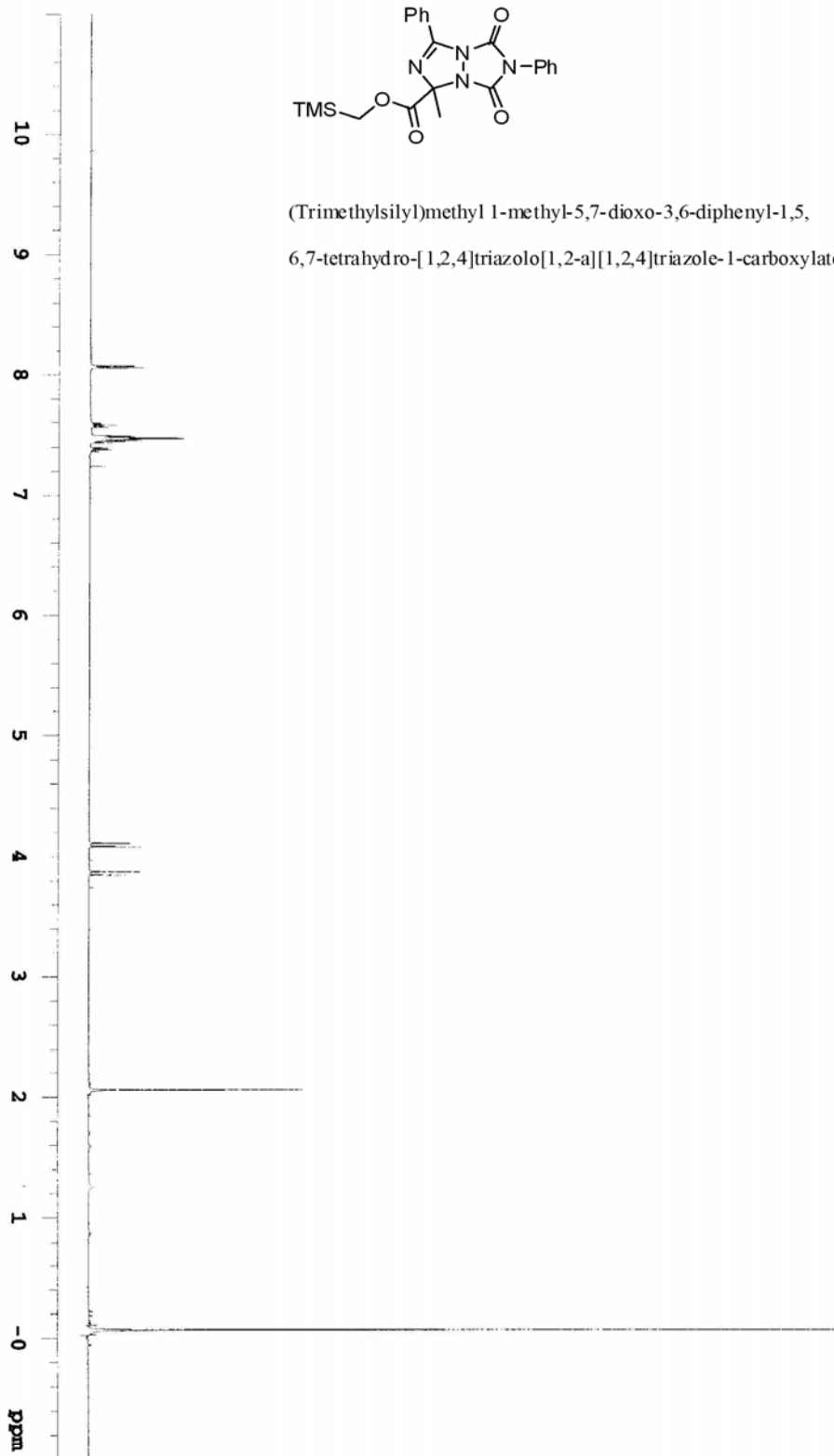


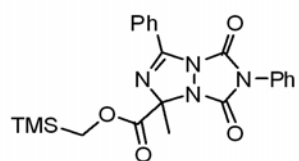
3-((1H-indol-3-yl)methyl)-1,2-bis(ethoxycarbonyl)-
5-phenyl-2,3-dihydro-1H-1,2,4-triazole-3-carboxylic acid (16):



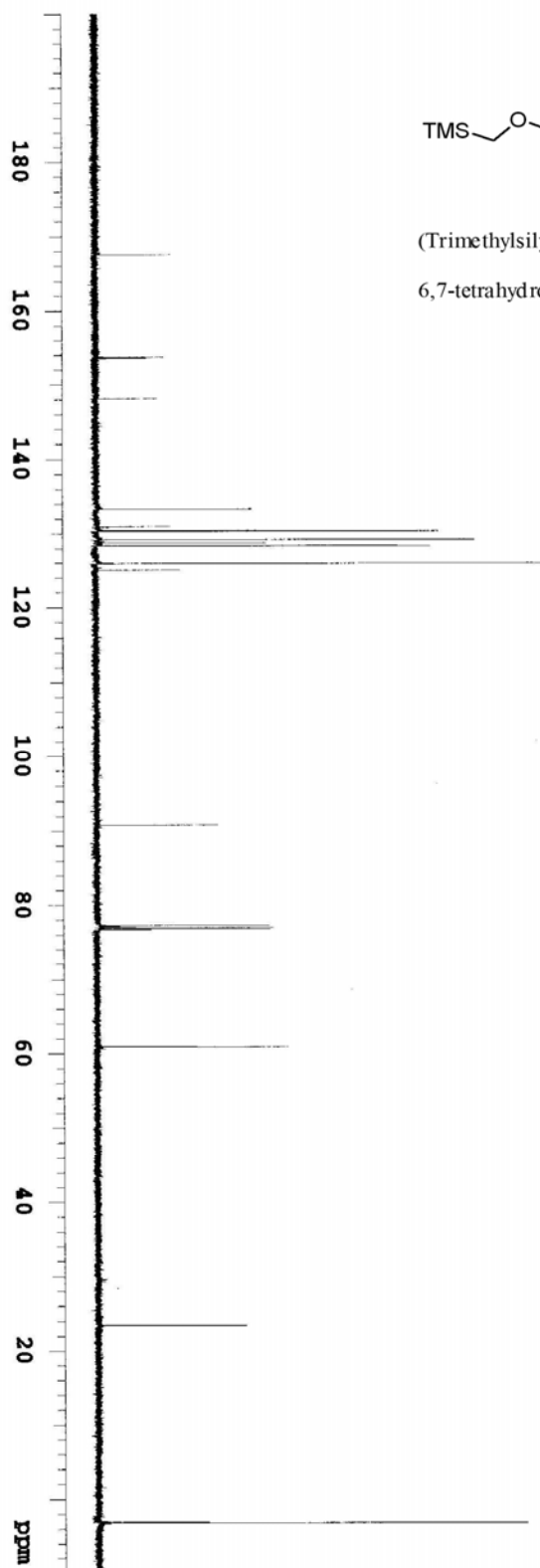


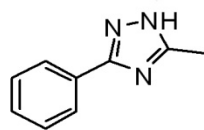
(Trimethylsilyl)methyl 1-methyl-5,7-dioxo-3,6-diphenyl-1,5,6,7-tetrahydro-[1,2,4]triazolo[1,2-a][1,2,4]triazole-1-carboxylate (17):



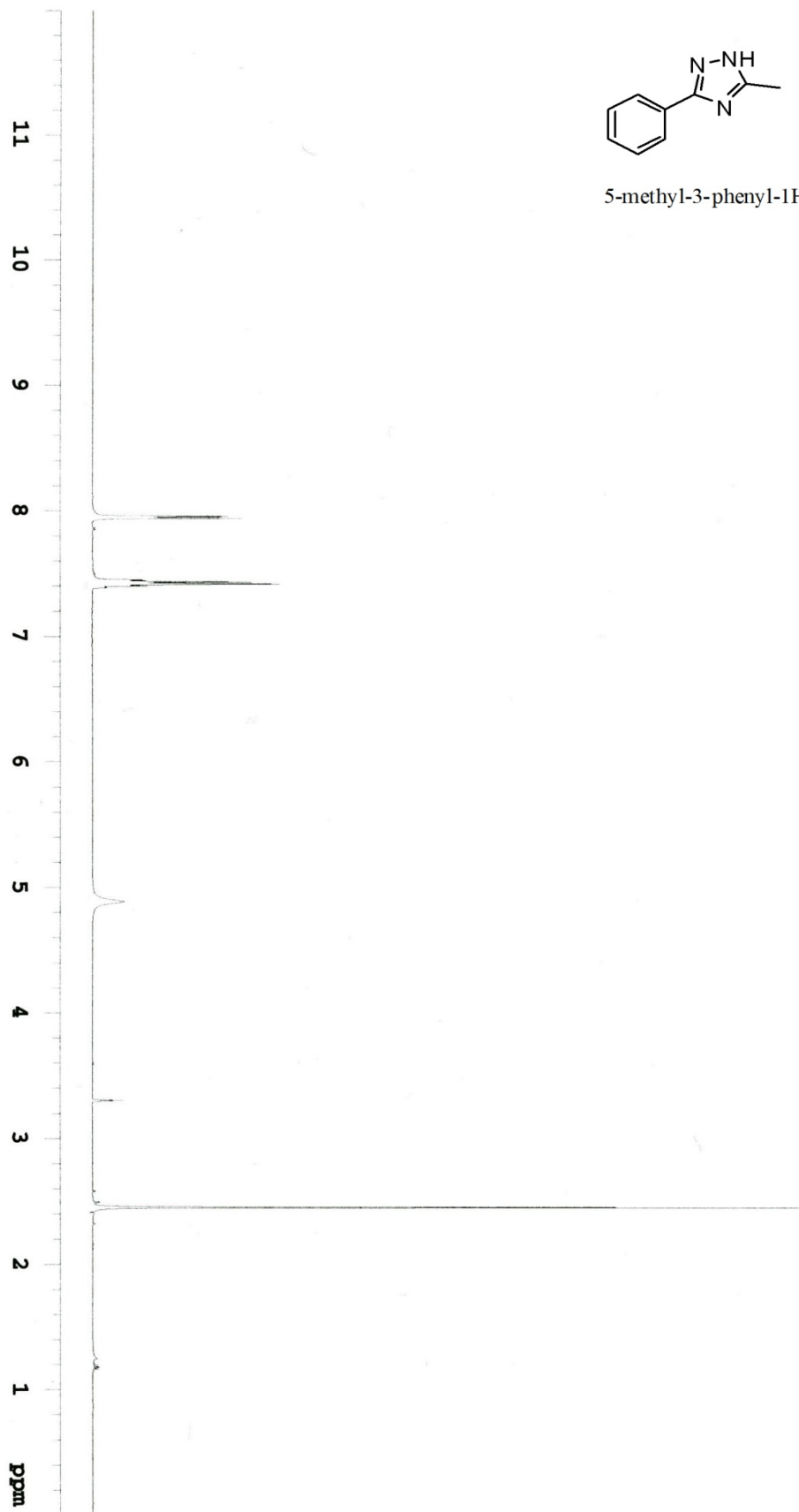


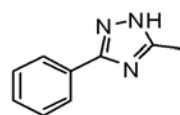
(Trimethylsilyl)methyl 1-methyl-5,7-dioxo-3,6-diphenyl-1,5,
6,7-tetrahydro-[1,2,4]triazolo[1,2-a][1,2,4]triazole-1-carboxylate (17):



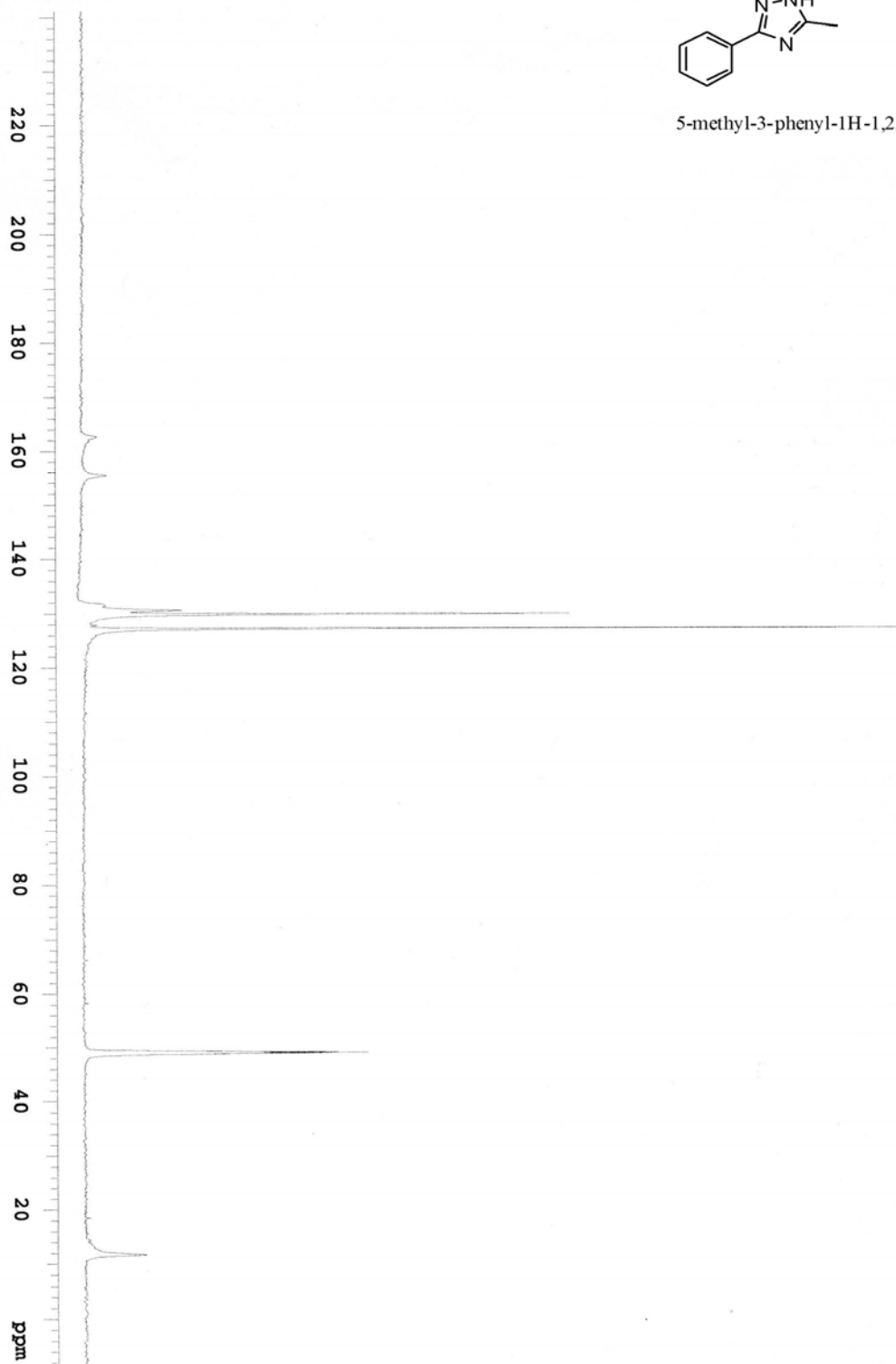


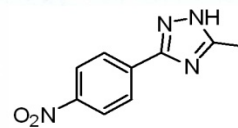
5-methyl-3-phenyl-1H-1,2,4-triazole (18)



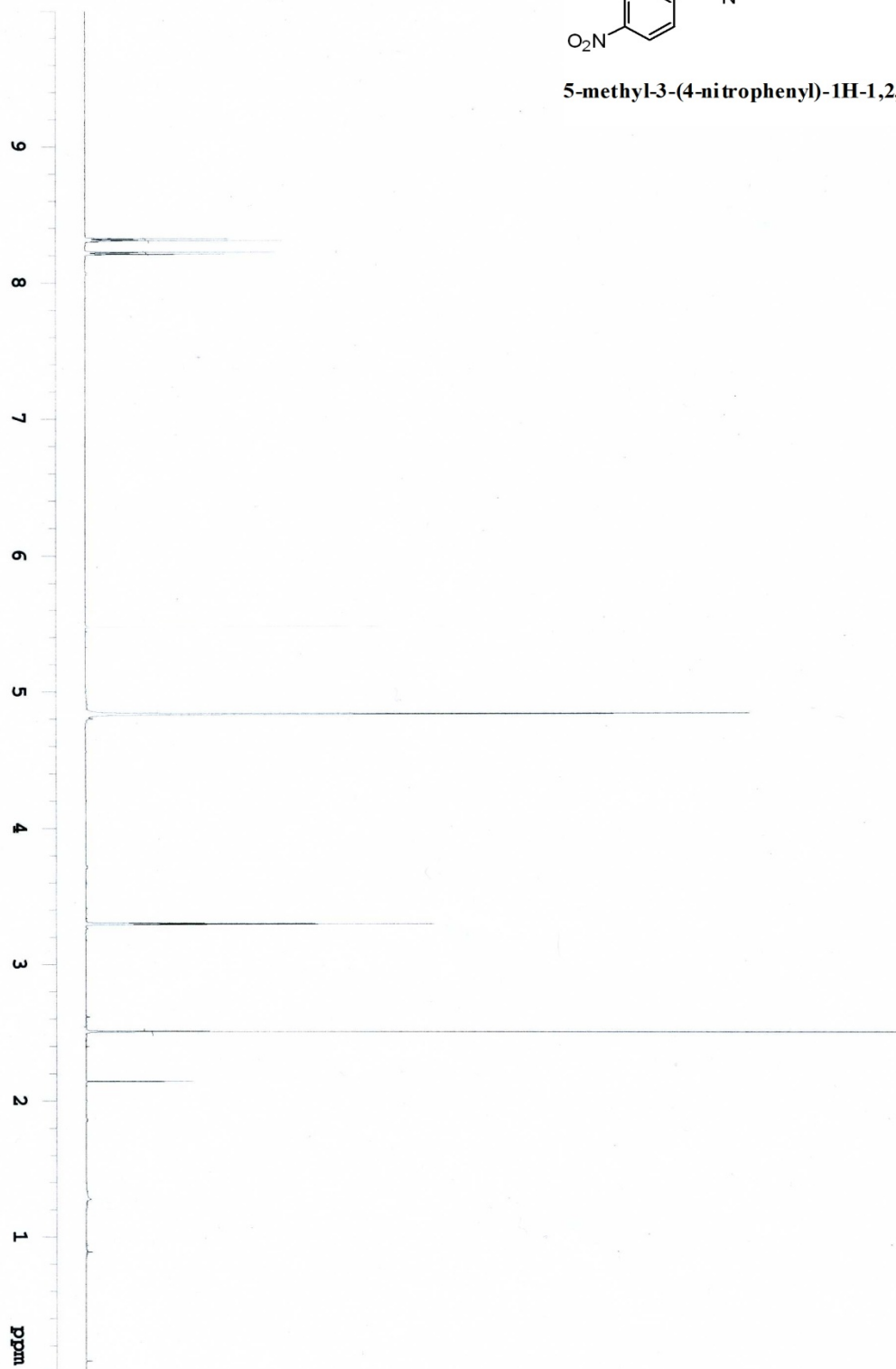


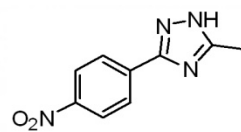
5-methyl-3-phenyl-1H-1,2,4-triazole (18)



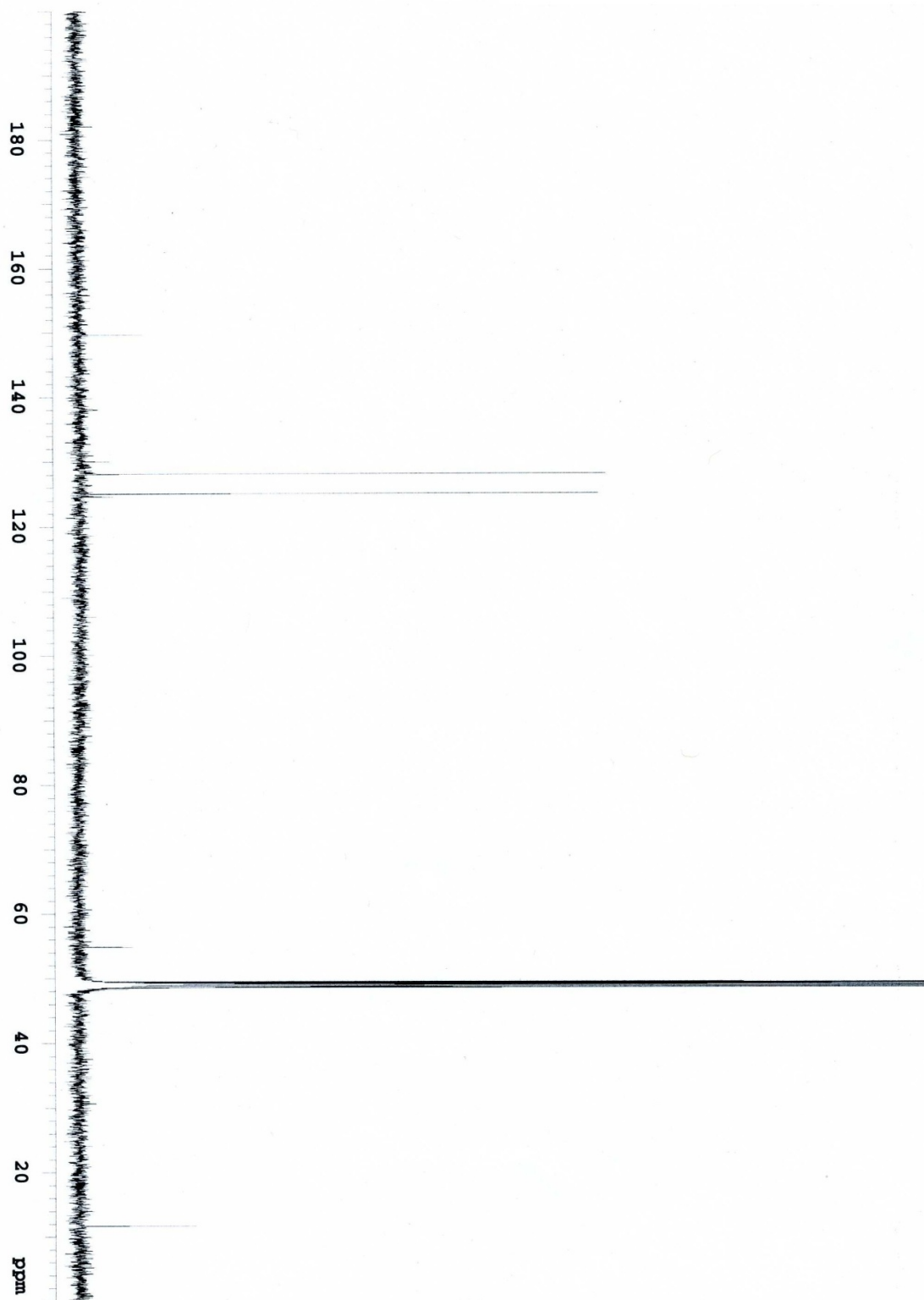


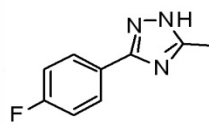
5-methyl-3-(4-nitrophenyl)-1H-1,2,4-triazole(19)



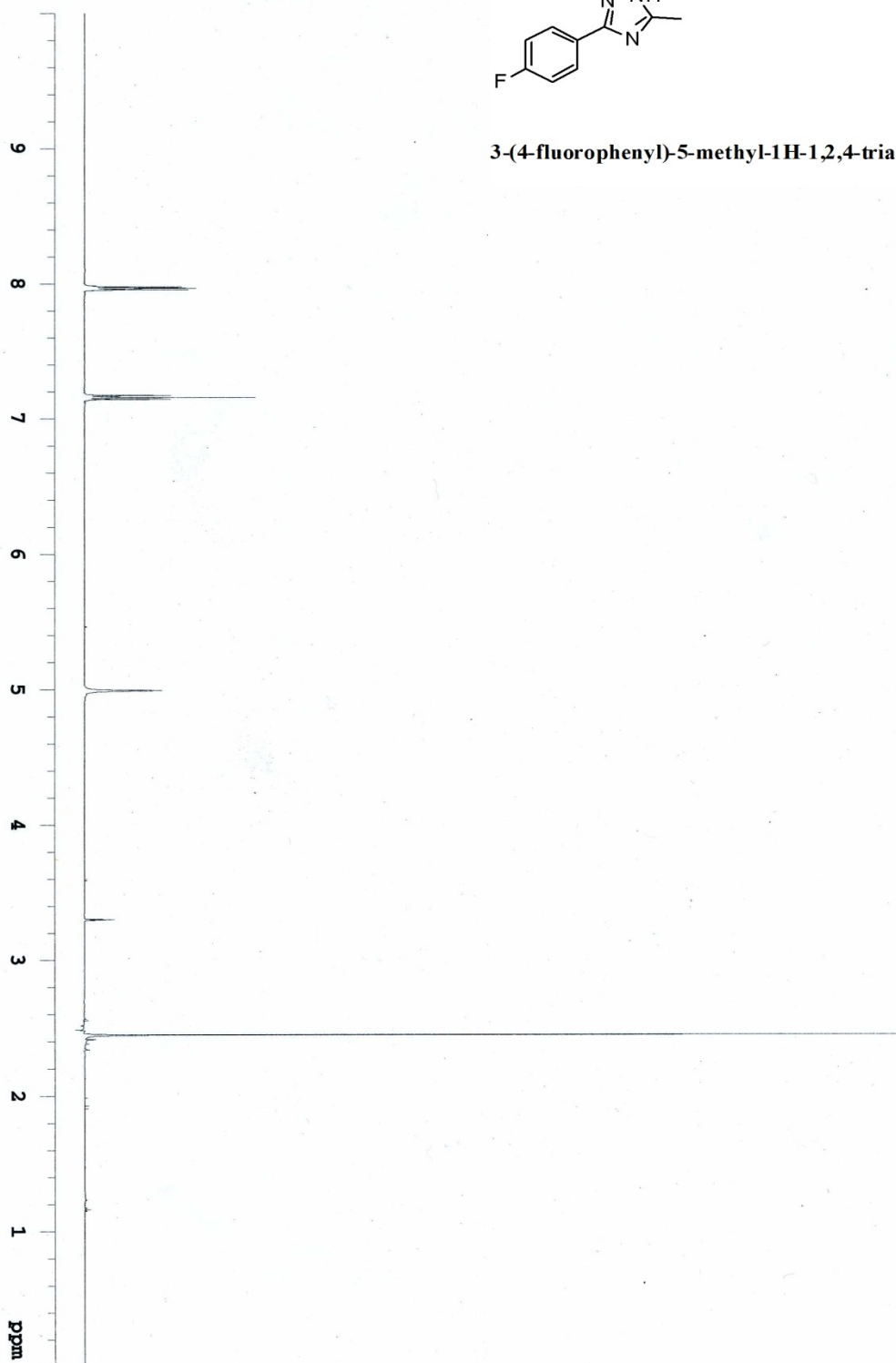


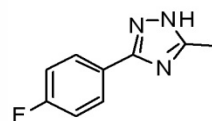
5-methyl-3-(4-nitrophenyl)-1H-1,2,4-triazole(19)



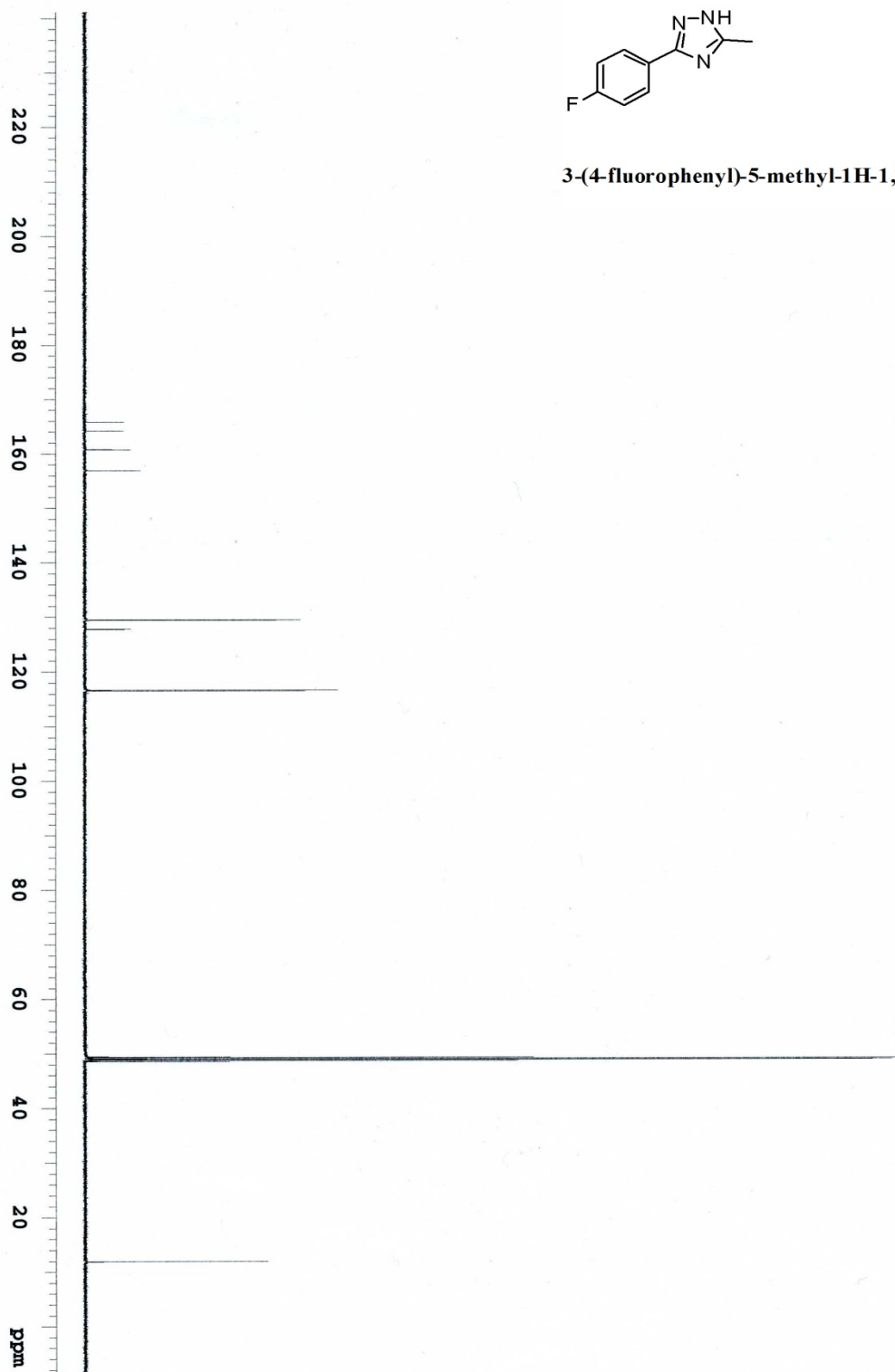


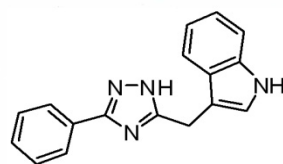
3-(4-fluorophenyl)-5-methyl-1H-1,2,4-triazole(20)



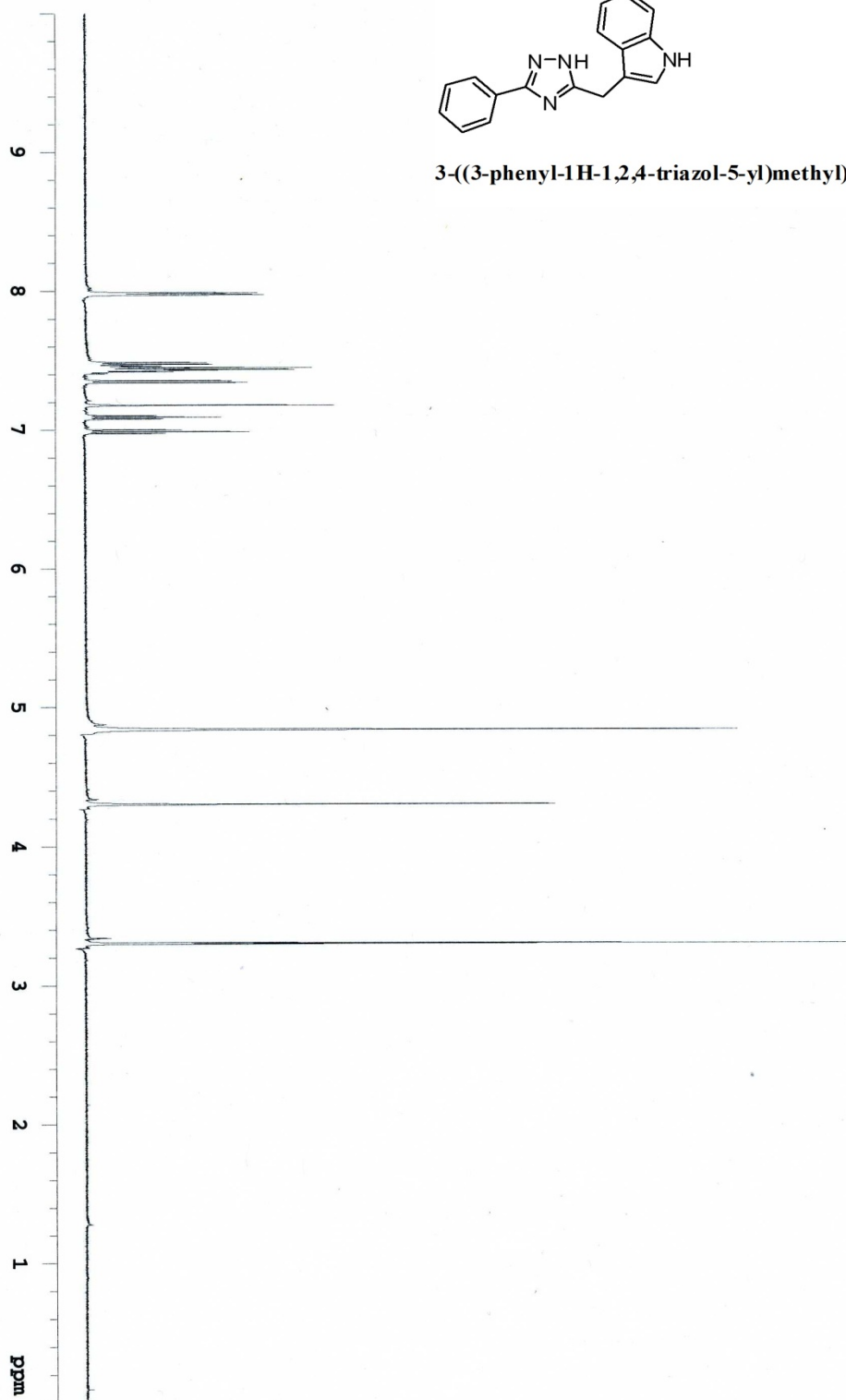


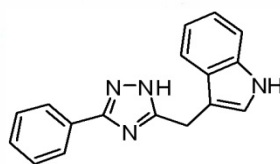
3-(4-fluorophenyl)-5-methyl-1H-1,2,4-triazole(20)



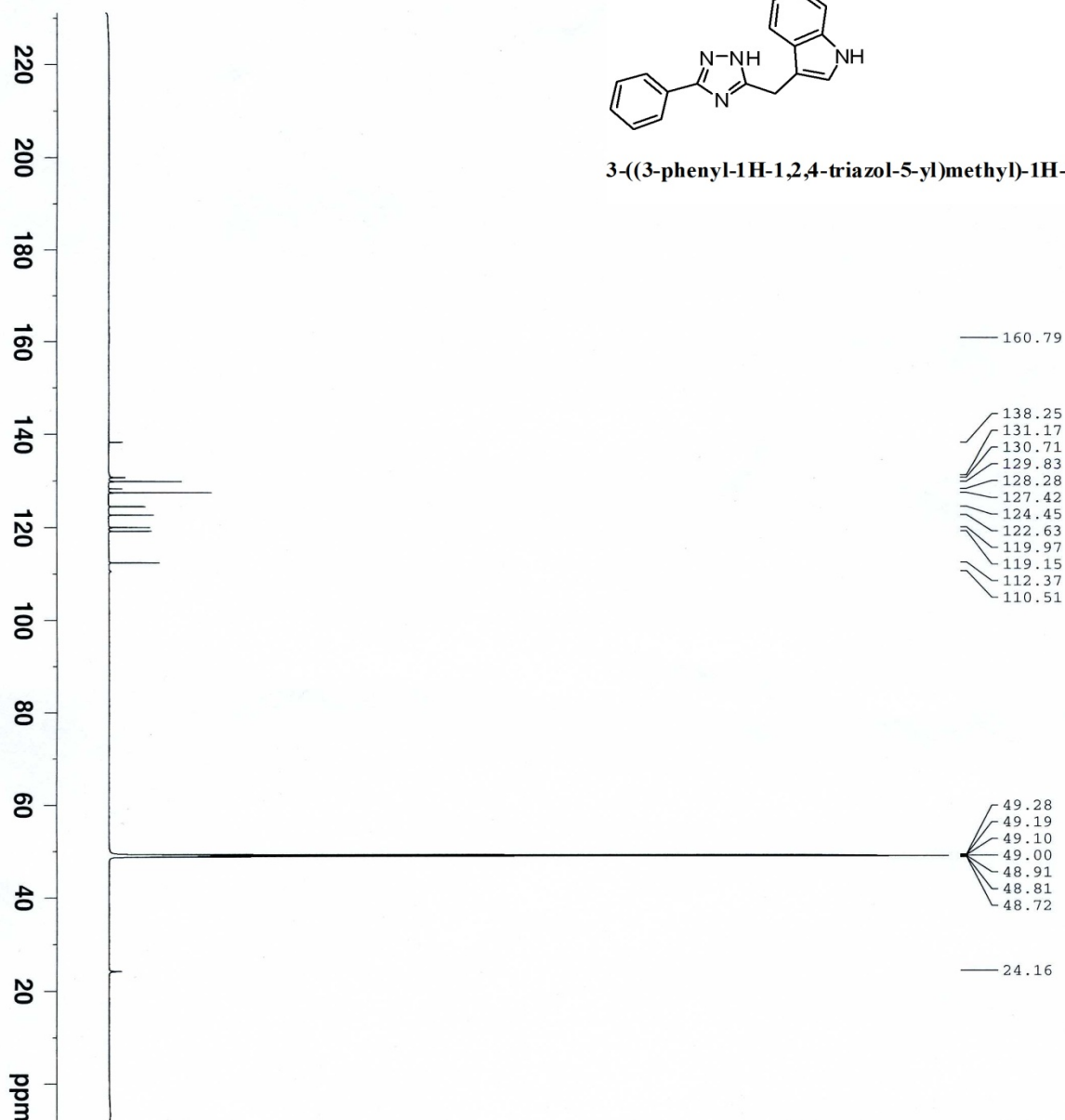


3-((3-phenyl-1H-1,2,4-triazol-5-yl)methyl)-1H-indole(21)





3-((3-phenyl-1H-1,2,4-triazol-5-yl)methyl)-1H-indole(21)



NAME	ros07018
EXPNO	7
PROCNO	1
Date_	20100330
Time	8.32
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg
TD	130006
SOLVENT	MeOD
NS	6668
DS	2
SWH	54200.543 Hz
FIDRES	0.416908 Hz
AQ	1.1993645 se
RG	11585.2
DW	9.225 us
DE	20.00 us
TE	298.0 K
D1	1.5000000 se
D11	0.03000000 se
TD0	1

===== CHANNEL f1 =====	
NUC1	13C
P1	13.00 us
PL1	-3.50 dB
PL1W	137.21128845 W
SFO1	226.3112125 MHz

===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	55.00 us
PL2	3.00 dB
PL12	18.00 dB
PL13	16.00 dB
PL2W	9.74408245 W
PL12W	0.30813494 W
PL13W	0.48836097 W
SFO2	899.9285997 MHz
SI	131072
SF	226.2860046 MHz
WDW	EM
SSB	0
LB	5.00 Hz
GB	0
PC	1.40