Supporting Information

3,3-Diazidoenones – New Type of Highly Reactive Bis-Azides. Preparation and Synthetic Transformations

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Experimental part

General details

Ionic liquids were received from Merck. All solvents and reagents were of reagent grade and used without additional purification. Cyclooctyne was synthesized starting from cyclooctene in three stages and purified using the reagents and procedures described in[i]. All reactions were monitored by thin-layer chromatography carried out on silica gel plates (Silufol UV-254). Column chromatography was performed on silica gel (63–200 mesh). The NMR spectra were recorded on 400 MHz spectrometer at 400.1 MHz for ^{1}H NMR, at 100.6 MHz for ^{13}C NMR, at 376 MHz for ^{19}F NMR and at 162 MHz for ^{31}P NMR. Melting points were determined on glass capillary tubes and are uncorrected. IR spectra were recorded on IR spectrometer with Fourier transform. Registration of IR-spectra was carried out at a resolution of 4 cm^{-1}, the number of scans 20. All reactions with phosphorous (III) derivatives were carried out under an argon atmosphere in flame-dried glassware under positive pressure of argon using standard Schlenk techniques.

Caution! Organic azides may cause potentially damage and should be carefully handled. The maximum reaction scale was limited to 1.0 mmol. Temperatures above 35^{o}C are not recommended. Protective gear is needed with larger scales. Bear in mind that some of described reactions proceed with emission of gas so make sure that reactor has venting.

General method for preparation of dichlorovinylketones 1a-f. To a stirred suspension of aluminum chloride (5 mmol) in 10 ml of dichloromethane at -5°C a

solution of acyl chloride (5 mmol) in 10 ml dichloromethane was added dropwise. After 10 minutes of stirring at -5°C vinylidene chloride (6 mmol) was slowly added and the reaction mixture was allowed to reach the room temperature and was stirred at ambient temperature for another 6 hours. Then reaction mixture was poured into crushed ice (50 g). An organic layer was separated and a water layer was extracted with dichloromethane (3×25 ml). Combined dichloromethane layers were dried over $CaCl_2$ and concentrated. The residue was dissolved in diethyl ether (25 mL) and cooled to -5°C. Then triethylamine (5 mmol) was added dropwise. The reaction mixture was allowed to reach the room temperature, filtered and the solvent was removed to give crude product which was purified by column chromatography (chloroform).

1-(4-Bromophenyl)-3,3-dichloroprop-2-en-1-one **1a** was prepared according to the general method from 5 mmol (1.097 g) of 4-bromobenzoyl chloride, 5 mmol (0.667 g) of aluminum chloride, 6 mmol (0.582 g) of vinylidene chloride and 5 mmol (0.506) of triethylamine in a yield of 66% (0.924 mg) as yellow solid. Analytical data for **1a**: mp 67-69°C (lit.[ii] 73°C); ^{1}H NMR ($CDCl_3$, 400.13 MHz): δ 7.87 (d, $^3J_{H\text{-}H}$= 8.4 Hz, 2H), 7.48 (d, $^3J_{H\text{-}H}$= 8.4 Hz, 2H), 7.23 (s , 1H) ppm; ^{13}C NMR ($CDCl_3$, 100.61 Hz): δ 185.4, 140.3, 136.2, 135.3, 129.9, 129.2, 123.6; IR (ν, cm^{-1}): 3091, 3059, 2923, 1160 (C=O), 1575 (C=C), 1223; Elemental Analysis: Found C, 46.01; H, 2.13; Cl, 45.01%; Calculated for $C_9H_5Cl_3O$: C, 45.90; H, 2.14; Cl, 45.16.

3,3-Dichloro-1-(4-chlorophenyl)prop-2-en-1-one **1b** was prepared according to the general method from 5 mmol (0.875 g) of 4-chlorobenzoyl chloride, 5 mmol (0.667

g) of aluminum chloride, 6 mmol (0.582 g) of vinylidene chloride and 5 mmol (0.506) of triethylamine in a yield of 63% (0.742 mg) as yellowish solid. Analytical data for **1b**: mp 49-51°C (lit.[ii] 50°C); ^{1}H NMR ($CDCl_3$, 400.13 MHz): δ 7.87 (d, $^{3}J_{H-H}$= 8.4 Hz, 2H), 7.48 (d, $^{3}J_{H-H}$= 8.4 Hz, 2H), 7.23 (s, 1H); ^{13}C NMR ($CDCl_3$, 100.61 Hz): δ 185.4, 140.3, 136.2, 135.3, 129.9, 129.2, 123.6; IR (ν, cm^{-1}): 3096, 3061, 2923, 1663 (C=O), 1570 (C=C), 1221; Elemental Analysis: Found C, 46.01; H, 2.13; Cl, 45.01%; Calculated for $C_9H_5Cl_3O$: C, 45.90; H, 2.14; Cl, 45.16.

3,3-Dichloro-1-phenylprop-2-en-1-one **1c** was prepared according to the general method from 5 mmol (1.005 g) of benzoyl chloride, 5 mmol (0.667 g) of aluminum chloride, 6 mmol (0.582 g) of vinylidene chloride and 5 mmol (0.506) of triethylamine in a yield of 63% (0.742 g) as yellow liquid. Analytical data for **1c**: ^{1}H NMR ($CDCl_3$, 400.13 MHz): δ 7.92 (d, $^{3}J_{H-H}$= 7.7 Hz, 2H), 7.61-7.47 (m, 3H), 7.27 (s, 1H); ^{13}C NMR ($CDCl_3$, 100.61 Hz): δ 186.5, 136.8, 135.3, 133.6, 128.8, 128.4, 124.0; IR (ν, cm^{-1}): 3050, 2926, 1670 (C=O), 1568 (C=C); Elemental Analysis: Found C, 53.85; H, 2.99; Cl, 35.17%; Calculated for $C_9H_6Cl_2O$: C, 53.77; H, 3.01; Cl, 35.27.

3,3-Dichloro-1-(3-nitrophenyl)prop-2-en-1-one **1d** was prepared according to the general method from 5 mmol (0.928 g) of 3-nitrobenzoyl chloride, 5 mmol (0.667 g) of aluminum chloride, 6 mmol (0.582 g) of vinylidene chloride and 5 mmol (0.506) of triethylamine in a yield of 57% (0.701 mg) as yellow solid. Analytical data for **1d**: mp 96-97℃ (lit.[ii] 96°C); ^{1}H NMR ($CDCl_3$, 400.13 MHz): δ 8.74 (s, 1H) 8.47 (d, $^{3}J_{H-H}$= 8.2 Hz, 1H), 8.27 (d, $^{3}J_{H-H}$= 7.8 Hz, 1H), 7.74 (dd, $^{3}J_{H-H}$= 8.0, 7.8 Hz, 1H), 7.33 (s

, 1H); ^{13}C NMR ($CDCl_3$, 100.61 Hz): δ 184.2, 148.5, 1385, 138.3, 133.9, 130.2, 127.8, 123.2, 122.6; IR (ν, cm^{-1}): 3107, 3082, 1670 (C=O), 1599 (C=C), 1520 (NO_2), 1346 (NO_2); Elemental Analysis: Found C, 43.82; H, 2.06; Cl, 28.91; N, 5.67%; Calculated for $C_9H_5Cl_2NO_3$: C, 44.94; H, 2.05; Cl, 28.82; N, 5.69.

1,1-Dichlorohex-1-en-3-one **1e** was prepared according to the general method from 5 mmol (0.533 g) of butyryl chloride, 5 mmol (0.667 g) of aluminum chloride, 6 mmol (0.582 g) of vinylidene chloride and 5 mmol (0.506) of triethylamine in a yield of 75% (0.626 g) as yellow liquid. Analytical data for **1e**: bp 95°C (20 mm Hg) (lit.[10] 41°C (4 mm Hg)); ^{1}H NMR ($CDCl_3$, 400.13 MHz): δ 6.63 (s, 1H), 2.50 (t, $^3J_{H\text{-}H}$= 7.2 Hz, 2H), 1.63 (m , 2H), 0.92 (t, $^3J_{H\text{-}H}$= 7.4 Hz); ^{13}C NMR ($CDCl_3$, 100.61 Hz): δ 195.7, 134.7, 126.3, 46.0, 17.1, 13.5; IR (ν, cm^{-1}): 3047 (=CH), 2965, 2934, 2878, 1703 (C=O), 1575 (C=C); Elemental Analysis: Found C, 43.05; H, 4.81; Cl, 42.59%; Calculated for $C_6H_8Cl_2O$: C, 43.15; H, 4.83; Cl, 42.45.

1,1-Dichlorohept-1-en-3-one **1f** was prepared according to the general method from 5 mmol (0.603 g) of pentanoyl chloride, 5 mmol (0.667 g) of aluminum chloride, 6 mmol (0.582 g) of vinylidene chloride and 5 mmol (0.506) of triethylamine in a yield of 73% (0.661 g) as yellow liquid. Analytical data for **1f**: bp 102°C (20 mm Hg); ^{1}H NMR ($CDCl_3$, 400.13 MHz): δ 6.65 (s, 1H), 2.54 (t,$^3J_{H\text{-}H}$= 7.5 Hz, 2H), 1.60 (m, 2H), 1.34 (m, 2H), 0.92 (t, $^3J_{H\text{-}H}$= 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100.61 Hz): δ 195.9, 134.8, 126.4, 43.9, 25.7, 22.2, 13.8; IR (ν, cm^{-1}): 3046, 2958, 2934, 2870, 1702 (C=O), 1573 (C=C). Elemental Analysis: Found C, 46.34; H, 5.59; Cl, 39.27%; Calculated for $C_7H_{10}Cl_2O$: C, 46.44; H, 5.57; Cl, 39.16.

4-Bromo-1,1-dichloro-4-methylpent-1-en-3-one **1g** was prepared according to procedure described in[iii]. Analytical data is in accordance with[iii].

4,4-Dichloro-1,1,1-trifluoro-but-3-en-2-one **1h** was prepared according to procedure described in[iv]. Analytical data is in accordance with[iv].

5-(4-Bromo-phenyl)-3-chloro-isoxazole **4a**. To a stirred solution of **1a** (0.5 mmol, 140 mg) in 10 ml of acetonitrile at 70°C was slowly (for 2 h) added a solution of sodium azide (1.0 mmol, 65 mg) in 5 ml of distilled water. After that reaction mixture was stirred for 1 h at 70°C and was cooled down to room temperature then it was quenched with a solution of cyclooctyne (1.0 mmol, 108 mg) in 1 ml of dichloromethane, stirred for 1 h at ambient temperature and concentrated at reduced pressure to remove organic solvents. After that it was extracted with dichloromethane (2×25 ml), washed with water and brine, dried, concentrated and subjected to column chromatography on silicagel (eluent: hexane then hexane/ethyl acetate 9:1 mixture) to give isoxazole **4a** as pale-pink-yellow sweet-smelling plates in 37% yield (48 mg). Analytical data for **4a**: R_f = 0.58 (hexane/ethyl acetate = 9/1); mp 139–142°C (lit.[v] 141-142°C); ^{1}H NMR (DMSO-d6): δ 7.83 – 7.70 (m, 4H), 7.41 (s, 1H); ^{13}C NMR (DMSO-d_6): δ 170.7, 153.9, 132.8 (2C), 128.1 (2C), 125.5, 125.3, 102.1; IR (ν, cm^{-1}): 3165, 3132, 1604, 1487, 1433, 1404 (C=C, C=N, C–O–N), 1365, 1068, 1009, 968, 941, 825, 794; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_9H_6BrClNO$ 257.9321, found 257.9316.

General method A for preparation of azidoisoxazoles 5a-d. A solution of dichlorovinylketone **1a-d** (1 equiv.) in dry acetonitrile (5 ml per 1 mmol of

dichloride) was added dropwise at -5°C and a vigorous stirring to a suspension of carefully grounded sodium azide (4 equiv.) in dry acetonitrile (5 ml per 1 mmol). After that a reaction mixture was intensively stirred for 1.5-4.0 h (see Table 1) to complete the substitution reactions (TLC analyzes with ethyl acetate/n-hexane 1:9 mixture as an eluent) and allowed to warm slowly to ambient temperature. Then a reaction mixtures was left tonight at stirring to complete the cyclization. After that reaction mixture was filtered through the silica-gel pod, the solvent was evaporated at reduced pressure and corresponding isoxazole was purified by column chromatography on silica-gel using ethyl acetate/hexane mixture (1:4 vol.) as an eluent giving desired product in yields of 52 to 62%.

3-Azido-5-(4-bromo-phenyl)-isoxazole **5a** was prepared according to the **general method A** (see above) from 0.5 mmol (140.0 mg) of 3,3-dichloro-1-(4-bromo-phenyl)-propenone **1a** and 2.0 mmol (130.0 mg) of sodium azide in a yield of 62% (82.2 mg) as white sweet-smelling plates. Analytical data for **5a**: R_f = 0.60 (hexane/ethyl acetate = 9/1); mp 118–120°C; ^{1}H NMR ($CDCl_3$): δ 7.62 (s, 4H), 6.25 (s, 1H); ^{13}C NMR ($CDCl_3$): δ 170.6, 161.0, 132.3 (2C), 127.2 (2C), 125.8, 125.3, 93.5; IR (ν, cm^{-1}): 3134, 3090, 2160 (N_3), 2129 (N_3), 1607, 1488, 1457, 1445, 1397 (C=C, C=N, CON), 1223, 1140, 1073; 1009, 947, 826, 779; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_9H_6BrN_4O$ 264.9725, found 264.9719.

3-Azido-5-(4-chloro-phenyl)-isoxazole **5b** was prepared according to the **general method A** (see above) from 0.5 mmol (117.8 mg) of 3,3-dichloro-1-(4-chloro-phenyl)-propenone **1b** and 2.0 mmol (130.0 mg) of sodium azide in a yield of 56%

(57.4 mg) as yellowish sweet-smelling fibrous solid. Analytical data for **5b**: R_f = 0.60 (hexane/ethyl acetate = 9/1); mp 101–104°C; ^{1}H NMR ($CDCl_3$): δ 7.67 (d, J = 8.6 Hz, 2H), 7.45 (d, J = 8.6 Hz, 2H), 6.22 (s, 1H); ^{13}C NMR ($CDCl_3$): δ 170.6, 161.0, 136.9, 129.4 (2C), 127.0 (2C), 125.3, 93.4; IR (ν, cm^{-1}): 3136, 3093, 2161 (N_3), 2129 (N_3), 1608, 1563, 1493, 1458, 1445, 1403 (C=C, C=N, CON), 1223, 1140, 1094; 1013, 947, 829, 778; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_9H_6ClN_4O$ 221.0230, found 221.0225.

3-Azido-5-phenyl-isoxazole **5c** was prepared according to the **general method A** (see above) from 0.5 mmol (100.5 mg) of 3,3-dichloro-1-phenyl-propenone **1c** and 2.0 mmol (130.0 mg) of sodium azide in a yield of 52% (48.4 mg) as white sweet-smelling fibrous solid. Analytical data for **5c**: R_f = 0.60 (hexane/ethyl acetate = 9/1); mp 84–85°C (lit.[vi] 86-88°C); ^{1}H NMR ($CDCl_3$): δ 7.76–7.73 (m, 2H), 7.48–7.47 (m, 3H), 6.24 (s, 1H); ^{13}C NMR ($CDCl_3$): δ 171.8, 160.9, 130.8, 129.0 (2C), 126.9, 125.7 (2C), 93.0; IR (ν, cm^{-1}): 3148, 3056, 2160 (N_3), 2127 (N_3), 1614, 1572, 1499, 1467, 1453, 1434 (C=C, C=N, CON), 1240, 1146, 945, 800, 763, 685; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_9H_7N_4O$ 187.0620, found 187.0615.

3-Azido-5-(3-nitro-phenyl)-isoxazole **5d** was prepared according to the **general method A** (see above) from 0.5 mmol (123.0 mg) of 3,3-dichloro-1-(3-nitro-phenyl)-propenone **1d** and 2.0 mmol (130.0 mg) of sodium azide in a yield of 61% (70.5 mg) as yellowish amorphous solid. Analytical data for **5d**: R_f = 0.60 (hexane/ethyl acetate = 7/3); mp 175–178°C (with decomp.); ^{1}H NMR ($CDCl_3$): δ 8.58 (s, 1H), 8.33 (dd, J = 8.2, 1.1 Hz, 1H), 8.09 (d, J = 7.8 Hz, 1H), 7.71 (t, J = 8.0 Hz, 1H), 6.40 (s, 1H); ^{13}C

NMR (DMSO-d_6): δ 168.6, 161.2, 148.4, 131.6, 131.2, 127.7, 125.4, 120.3, 96.3; IR (ν, cm^{-1}): 3141, 3087, 3078, 2164 (N_3), 2133 (N_3), 1622, 1603, 1531 (NO_2), 1485, 1462, 1442, 1423 1434 (C=C, C=N, CON), 1352 (NO_2); 1231, 800, 737; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_9H_6BN_5O_3$ 232.0471, found 232.0465.

General method B for preparation of azidoisoxazoles 5a-c. To a solution of NaN_3 (2 mmol) in 2 ml of $[emim]^+[OTf]^-$ at 0°C and vigorous stirring a solution of dichlorovinylketone **1a-c** (0.5 mmol) in 1 ml of $[emim]^+[OTf]^-$ was added dropwise. After 3h of strirring at 0°C the reaction mixture was warmed to 25°C and stirred overnight. Then it was extracted with n-hexane (2×10 ml), combined hexane fractions were washed with water and brine, dried with Na_2SO_4 and evaporated to dryness giving NMR-pure azidoisoxazoles **5a-c** in yields of 74-75%.

General method for preparation of azidoisoxazoles 5e-h. A solution of dichlorovinylketone **1e-h** (1 equiv.) in acetone (5 ml per 1 mmol of dichloride) was added dropwise at -5°C and a vigorous stirring to a solution of sodium azide (4 equiv.) in acetone/water 1:1 vol. mixture (10 ml of the mixture per 1 mmol of NaN_3). After that a reaction mixture was stirred for 1 to 36 h to complete the substitution reactions (TLC analysis) and allowed to warm to ambient temperature. At the temperature of about 10-15°C nitrogen-gas evolution became visible. After the completion of cyclization the solvent was evaporated, the residue was extracted with CH_2Cl_2 (2×10 ml per 1 mmol of taken dichlorovinylketone), washed with water and brine, dried with Na_2SO_4, concentrated, and subjected to column chromatography on

silica-gel using ethyl acetate/hexane mixture (1:4 vol.) as an eluent giving **5e-h** in yields of 30 to 40%.

3-Azido-5-propyl-isoxazole **5e** was prepared according to the **general method** (see above) from 1.0 mmol (167.0 mg) of 1,1-dichloro-hex-1-en-3-one **1e** and 4.0 mmol (260.0 mg) of sodium azide in a yield of 30% (45.6 mg) as colorless transparent sweet-smelling oil. Analytical data for **5e**: R_f = 0.65 (hexane/ethyl acetate = 9/1); ^{1}H NMR ($CDCl_3$): δ 5.72 (s, 1H), 2.67 (t, J = 7.5 Hz, 2H), 1.78–1.62 (m, 3H), 0.98 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$): δ 175.5, 159.9, 94.3, 28.6, 20.4, 13.1; IR (ν, cm^{-1}): 2966, 2933, 2873, 2133 (N_3), 1603, 1473, 1442 (C=C, C=N, CON); HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_6H_9N_4O$ 153.0776, found 153.0772.

3-Azido-5-butyl-isoxazole **5f** was prepared according to the **general method** (see above) from 0.5 mmol (90.5 mg) of 1,1-dichloro-hept-1-en-3-one **1f** and 2.0 mmol (130.0 mg) of sodium azide in a yield of 36% (59.8 mg) as colorless transparent sweet-smelling oil. Analytical data for **5f**: R_f = 0.65 (hexane/ethyl acetate = 9/1); ^{1}H NMR ($CDCl_3$): δ 5.71 (s, 1H), 2.68 (t, J = 7.6 Hz, 2H), 1.68–1.61 (m, 2H), 1.43–1.31 (m, 2H), 0.92 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$): δ 175.7, 159.8, 94.2, 28.9, 26.4, 18.8, 13.2; IR (ν, cm^{-1}): 2960, 2933, 2873, 2133 (N_3), 1603, 1469, 1444 (C=C, C=N, CON); HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_7H_{11}N_4O$ 167.0933, found 167.0928.

3-Azido-5-(1-bromo-1-methyl-ethyl)-isoxazole **5g** was prepared according to the **general method** (see above) from 1.0 mmol (246.0 mg) of 4-bromo-1,1-dichloro-4-methyl-pent-1-en-3-one **1g** and 5.0 mmol (325.0 mg) of sodium azide in a yield of

36% (83.2 mg) as yellowish transparent oil. Analytical data for **5g**: R_f = 0.62 (hexane/ethyl acetate = 9/1); ^{1}H NMR ($CDCl_3$): δ 5.99 (s, 1H), 2.12 (s, 6H); ^{13}C NMR ($CDCl_3$): δ 176.9, 159.9, 93.8, 49.9, 32.2 (2C); IR (ν, cm^{-1}): 2133 (N_3), 1593, 1473, 1435 (C=C, C=N, CON), 1275, 1115; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_6H_8BrN_4O$ 230.9881, found 230.9876.

3-Azido-5-trifluoromethyl-isoxazole **5h**. Dichlorovinylketone **1h** (1.0 mmol, 193 mg) was involved in a reaction with sodium azide (4.0 mmol, 260 mg) in 10 ml of dry acetonitrile according to the **general method**. After completion of cyclization **5h** have been converted to **7h** and was not isolated in an individual state. Analytical data for **5h**: ^{1}H NMR (CD_3CN): δ 6.87 (q, J = 1.0 Hz, 1H); ^{19}F NMR (CD_3CN): δ -65.80 (bs).

1-(4-Bromophenyl)-3,3-bis(4,5,6,7,8,9-hexahydrocycloocta[d][1,2,3]triazol-1-yl)-prop-2-en-1-one **6a**. A solution of **1a** (0.5 mmol, 140.0 mg) in 2.5 ml of dry acetonitrile was added dropwise at -5°C and a vigorous stirring to a suspension of carefully grounded sodium azide (2 mmol., 130.0 mg) in 5 ml of dry acetonitrile. The reaction mixture was intensively stirred for 2 h at 0°C and then a solution of cyclooctyne (2 mmol, 216.4 mg) in 1 ml of acetonitrile was added. After that it was stirred for another 2 h at 0°C, warmed to room temperature and stirred for 1 h at ambient temperature to complete the reaction. The reaction mixture was diluted with 10 ml of hexane and the inorganic salts were filtered off, washed with hexane (2×10 ml). The organic solvents and partly an excess of cyclooctyne were removed under reduced pressure leaving yellowish oily residue. Pure **6a** was isolated by column

chromatography (hexane/ethyl acetate = 7/3 mixture as an eluent) in 88 % yield (225.0 mg) as yellowish oil which tends to solidify upon standing. Analytical data for **6a**: R_f= 0.35 (hexane/ethyl acetate = 7/3); ^{1}H NMR ($CDCl_3$): δ 7.74 (AA'BB', J=8.6 Hz, 2H), 7.57 (AA'BB', J =8.6 Hz, 2H), 7.36 (s, 1H), 2.98–2.80 (m, 4H), 2.48–2.30 (m, 4H), 1.82–1.66 (m, 4H), 1.63–1.53 (m, 2H), 1.51–1.34 (m, 10H); ^{13}C NMR ($CDCl_3$): δ 186.3, 146.3, 145.3, 135.3, 134.94, 1334.86, 131.6 (2C), 129.6 (2C), 128.7, 121.0, 29.3, 27.6, 27.2, 25.5, 25.42, 25.39, 25.1, 24.2, 24.0, 23.8, 21.1, 20.9; IR (ν, cm^{-1}): 3084, 3055, 3037, 2929, 2854, 1684 (C=O); 1651 (C=C), 1458, 1444, 1402, 1250, 1223, 1174, 1070, 1051, 1004, 941, 835, 806; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_{25}H_{30}BrN_6O$ 509.1664, found 509.1659.

1-(5-(4-Bromophenyl)isoxazol-3-yl)-4,5,6,7,8,9-hexahydro-1H-cycloocta[d][1,2,3]-triazole **7a**. To a solution of 0.47 mmol (125 mg) of **5a** in 8 ml of dichloromethane at room temperature and stirring in a dropwise manner was added a solution of 1.0 mmol (110.2 mg) of cyclooctine in 2 ml of dichloromethane. After that the reaction mixture was stirred for 2 h at room temperature, the solvent was removed at reduced pressure leaving yellowish solid residue which was washed several times with cold *n*-hexane and dried in vacuum. The residue was recrystallized from ethyl acetate/hexane/chloroform (1/2/2) yielding **7a** (93%, 163 mg) as a yellowish transparent needles. Analytical data for **7a**: R_f= 0.5 (hexane/ethyl acetate = 7/3); mp 201–205°C; ^{1}H NMR (400.1 MHz, $CDCl_3$): δ 7.67 (AA'BB', J = 8.2 Hz, 4H), 7.17 (s, 1H), 3.22 (t, J = 6.1 Hz, 2H), 2.98 (t, J = 6.1 Hz, 2H), 1.93 (bs, 2H), 1.79 (bs, 2H), 1.51 (bs, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 169.6, 158.9, 146.0, 134.5, 132.1

(2C), 126.9 (2C), 125.1, 125.0, 94.4, 28.4, 25.8, 25.7, 24.4, 24.2, 22.2; IR (ν, cm^{-1}): 3134, 2927, 2848, 1612, 1585, 1574, 1562, 1527, 1464, 1452, 1429, 1409, 1398, 1317, 1277, 1261, 1248, 1068, 1051, 1005, 947, 823; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_{17}H_{18}BrN_4O$ 373.0664, found 373.0661.

1-(5-(trifluoromethyl)isoxazol-3-yl)-4,5,6,7,8,9-hexahydro-1H-cycloocta[d][1,2,3]-triazole **7h**. To the reaction mixture from the synthesis of **5h** (see above) a solution of cyclooctine (2.0 mmol, 216.4 mg) in 1 ml of dry acetonitrile was added dropwise at 20°C. After 2 h of stirring the reaction mixture was concentrated in vacuum and a residue was subjected to column chromatography using CH_2Cl_2 as an eluent. After removing of the solvent pure **7h** was obtained in a yield of 40% (115 mg) as white powder. Analytical data for **7h**: R_f= 0.55 (CH_2Cl_2); mp 91–93°C; ^{1}H NMR ($CDCl_3$): δ 7.38 (q, J_{HF} = 0.9 Hz, 1H, isoxazole-C^{4}H), 3.24 – 3.16 (m, 2H), 3.00 – 2.92 (m, 2H), 1.97 – 1.87 (m, 2H), 1.84 – 1.74 (m, 2H), 1.54 – 1.44 (m, 4H); ^{13}C NMR ($CDCl_3$): δ 159.69 (q, J_{CF} = 43.5 Hz, isoxazole-C^5), 158.59 (isoxazole-C^3), 146.80 (triazole-C^4), 135.07 (triazole-C^5), 117.17 (q, J_{CF} = 271.0 Hz, CF_3), 100.89 (q, J_{CF} = 2.3 Hz, isoxazole-C^4), 28.62, 26.00, 25.97, 24.64, 24.51, 22.47 ($\underline{C}H_2$ carbons, 6C in total); ^{19}F NMR ($CDCl_3$): δ -64.56 (d, J_{HF} = 0.9 Hz); IR (ν, cm^{-1}): 3134, 2939, 2920, 2852, 1543, 1456, 1446; 1313, 1255, 1215, 1201, 1147 (CF_3), 1025, 960, 935, 858; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_{12}H_{14}F_3N_4O$ 287.1120, found 287.1122.

Experimental procedure for cosynthesis of 1,1-bis(4,5,6,7,8,9-hexahydrocycloocta[d]-[1,2,3]triazol-1-yl)hex-1-en-3-one **6e** and 1-chloro-1-(4,5,6,7,8,9-hexahydrocycloocta[d]-[1,2,3]triazol-1-yl)hex-1-en-3-one **8e**. To a

suspension of NaN_3 (4.0 mmol, 260 mg) in 10 ml of dry acetonitrile a solution of **1e** (1.0 mmol, 167 mg) in 1 ml of dry acetonitrile was added dropwise at 0°C. After stirring of the reaction mixture for 24 h at 0°C a solution of cyclooctine (3.0 mmol, 324.6 mg) in 1 ml of acetonitrile was added dropwise and the stirring was continued for additional 2 hours. After that reaction mixture was filtered, concentrated in vacuum and a residue was subjected to column chromatography on silica gel using EtOAc/hexane 4:1 mixture as an eluent yielding **6e** (258 mg, 65%) and **8e** (76 mg, 27%) as transparent yellowish oils.

Analytical data for **6e**: R_f = 0.53 (hexane/ethyl acetate = 7/3); ^{1}H NMR ($CDCl_3$): δ 6.74 (s, 1H), 3.01–2.87 (m, 2H), 2.87–2.73 (m, 2H), 2.25 (t, J = 7.2 Hz, 2H), 1.94–1.71 (m, 4H), 1.64–1.41 (m, 6H), 0.82 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$): δ 195.1, 144.3, 134.6, 133.0, 128.96, 44.52, 27.29, 25.6, 25.4, 24.26, 23.74, 21.37, 16.39, 13.03; IR (ν, cm^{-1}): 2960, 2634, 2868, 2856, 1709 (C=O), 1622 (C=C), 1458, 1442, 1385, 1244, 1051, 947, 852; 771, 762; HRMS (ESI-TOF) (m/z) $[M+H]^+$ calcd for $C_{14}H_{21}ClN_3O$ 282.1373 found 282.1370.

Analytical data for **8e**: R_f = 0.28 (hexane/ethyl acetate = 2/1); ^{1}H NMR ($CDCl_3$): δ 6.79 (bs, 1H), 2.86 (dt, J = 19.5, 6.5 Hz, 4H), 3.01–2.87 (m, 2H), 2.87–2.73 (m, 2H), 2.45–2.37 (m, 2H), 2.37–2.61 (m, 4H), 1.76–1.64 (m, 4H), 1.61–1.46 (m, 4H), 1.40 (bs, 6H), 1.32 (bs, 4H), 0.82 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$): δ 196.3, 146.1, 145.2, 135.3, 134.8, 132.9, 123.5, 44.9, 27.64, 27.3, 25.5, 25.3, 25.3, 25.2, 24.2, 24.1, 23.9, 23.8, 20.9, 20.8, 16.5, 13.1; IR (ν, cm^{-1}): 2931, 2856, 1709 (C=O), 1649 (C=C),

1458, 1444, 1400, 1250, 1053, 1036, 947, 756; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_{22}H_{33}N_6O$ 397.2716 found 397.2708.

1-[5-(4-Chloro-phenyl)-isoxazol-3-yl]-4-phenyl-1H-[1,2,3]triazole **9b**. To a solution of 0.5 mmol (110.3 mg) of **5b** in 8 ml of dichloromethane a solution of 0.525 mmol (53.6 mg) of freshly distilled phenyl-acetylene in 2 ml of dichloromethane was added. After that to the reaction mixture at room temperature and stirring was added a solution of $CuSO_4*5H_2O$ (10 % mol.) in 1 ml of distilled water and a solution of sodium ascorbate (40 % mol.) in 1 ml of distilled water. The reaction mixture was refluxed at 40°C for 12 h, cooled, washed with 10 ml of ammonia hydroxide solution (5 weight % in water), distilled water (2x25 ml) and brine, dried over Na_2SO_4 and evaporated to dryness giving **9b** as a yellowish solid in 81 % yield (131 mg). Analytical data for **9b**: mp 221–223°C (with decomp.); ^{1}H NMR (DMSO-d6): δ 9.38 (s, 1H, triazole-C^{5}H), 8.09–7.95 (m, 4H, Ar), 7.90 (s, 1H, isoxazole-C^{4}H), 7.70 – 7.62 (m, 2H, Ar), 7.54 – 7.43 (m, 2H, Ar), 7.43 – 7.34 (m, 1H, Ar); ^{13}C NMR (DMSO-d_6): 171.08 (isoxazole-C^5), 158.51 (isoxazole-C^3), 148.18 (triazole-C^4), 136.56 (C-Cl), 130.00 (2C, 4-Cl-Ph-C^{3,5}), 129.75 (triazole-Ph-C^4), 129.49 (2C, triazole-Ph-C^{2,6}), 129.21 (triazole-Ph-C^1), 128.18 (2C, 4-Cl-Ph-C^{2,6}), 126.16 (2C, triazole-Ph-C^{3,5}), 125.25 (4-Cl-Ph-C-isoxazole), 120.07 (triazole-C^5), 95.09 (isoxazole-C^4); IR (ν, cm^{-1}): 3136, 1616, 1541, 1481, 1466, 1450, 1406, 124, 1093, 1043, 1012, 947, 935, 835, 810; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_{17}H_{12}ClN_4O$ 323.0700, found 323.0694.

5-Amino-1-[5-(4-chloro-phenyl)-isoxazol-3-yl]-1H-[1,2,3]triazole-4-carbonitrile

10b. Sodium hydride (0.525 mmol, 21 mg of 60 weight % suspension in mineral oil) was carefully added to 10 ml of absolute ethanol. To a resulted solution of sodium ethoxide a solution of malononitrile (0.525 mmol, 35 mg) in 1 ml of ethanol was added dropwise and the resulted mixture was chilled to -20°C. After that a solution of azidoisoxazole **5b** (0.5 mmol, 110 mg) in 5 ml of ethanol was added dropwise. The reaction mixture was stirred for 30 min at -20°C and allowed to reach the room temperature. Then it was acidified with acetic acid to pH 6 and was warmed at 60°C for 2 h. After cooling down an off-white precipitate was filtered off, washed with fresh ethanol and ether. A combined filtrate was concentrated and was worked up giving in total 113 mg (79%) of **10b** as an off-white powder. Analytical data for **10b**: decomp. > 250°C; ^{1}H NMR (DMSO-d6): δ 9.06 (bs, 1H, NH_2), 7.84 (AB, d, *J* = 8.6 Hz, 2H, Ar), 7.56 (AB, d, *J* = 8.6 Hz, 2H, Ar), 6.91 (s, 1H, isoxazole-C^{4}H), 3.36 (bs, 1H, NH_2); ^{13}C NMR (DMSO-d_6): δ 166.44 (isoxazole-C^5), 162.91 (isoxazole-C^3), 149.36 (triazole-C^5), 134.97 (C-Cl), 129.64 (2C, Ar-C^{3,5}), 127.55 (2C, Ar-C^{2,6}), 126.68 (Ar-C^4), 117.84 (-CN), 105.04 (triazole-C^4), 94.27 (isoxazole-C^4); IR (ν, cm^{-1}): 3419, 3305 (NH_2), 3143 (aromatic C-H stretchings), 2241, 2227 (C≡N), 1649, 1614, 1543, 1468 (C=C, C=N, CON), 1095, 831, 802, 775; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_{12}H_8ClN_6O$ 287.0448, found 287.0444.

General method for a preparation of iminophosphoranes 11a-c. To a solution of 0.5 mmol (135 mg) of triphenyphosphine in 2 ml of distilled dichloromethane at 5°C and stirring a solution of azidoisoxazole **5a-c** (0.5 mmol) in 2 ml of dichloromethane

was added dropwise. The reaction mixture was stirred for another 30 minutes at this temperature and allowed to reach the room temperature. After that the reaction mixture was left overnight, the solvent was evaporated at a reduced pressure, the residue was treated with small amount of *n*-hexane, filtered and dried to give the desired NMR-pure iminophosphorane **11a-c**.

3-Imino-(triphenyl)-phosphorano-5-(4-bromophenyl)-isoxazole **11a** was obtained starting from 0.5 mmol (132.5 mg) of **5a** according to the **general method** above in 94% yield (235 mg) as off-white powder, which after recrystallization from ethylacetate/hexane (1/5) gave yellowish transparent crystals. Analytical data for **11a**: R_f = 0.36 (hexane/ethyl acetate = 7/3); mp 192–194°C; ^{1}H NMR ($CDCl_3$): δ 7.89–7.76 (m, 6H), 7.59–7.44 (m, 13H), 6.28 (d, J_{HP} = 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$): δ 167.1, 166.4, 132.6 (d, J_{CP} = 10.0 Hz, 6C), 131.7 (d, J_{CP} = 2.6 Hz, 3C), 131.4 (2C), 128.8 (d, J_{CP} = 100.0 Hz, 3C), 128.2 (d, J_{CP} = 12.0 Hz, 6C), 127.3, 126.4 (2C), 122.8, 98.5 (d, J_{CP} = 20.5 Hz); ^{31}P-{^{1}H} NMR ($CDCl_3$): δ 15.3; IR (ν, cm^{-1}): 3076, 3051; 1616, 1489, 1469, 1437, 1404, 1387 (C=C, C=N, CON, P=N), 1219, 1111 (P-N-C), 1076, 1009, 955, 833, 787; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_{27}H_{21}BrIN_2OP$ 499.0575, found 499.0571.

3-Imino-(triphenyl)-phosphorano-5-(4-chlorophenyl)-isoxazole **11b** was obtained starting from 0.5 mmol (110.3 mg) of **5b** according to the **general method** above in 96% yield (218 mg) as off-white powder, which after recrystallization from ethylacetate/hexane (1/5) gave yellowish transparent crystals. Analytical data for **11b**: R_f = 0.36 (hexane/ethyl acetate = 7/3); mp 195–197 ℃; ^{1}H NMR ($CDCl_3$): δ

7.89–7.76 (m, 6H), 7.64–7.52 (m, 5H), 7.52–7.44 (m, 6H), 7.35 (d, J = 1.8 Hz, 2H), 6.27 (d, J_{HP} = 0.8 Hz, 1H); ^{13}C NMR ($CDCl_3$): δ 167.1, 166.4, 134.5, 132.6 (d, J_{CP} = 10.0 Hz, 6C), 131.7 (d, J_{CP} = 2.6 Hz, 3C), 128.8 (d, J_{CP} = 100.0 Hz, 3C), 128.5 (2C), 128.2 (d, J_{CP} = 12.0 Hz, 6C), 126.9, 126.2 (2C), 98.5 (d, J_{CP} = 20.5 Hz); ^{31}P-{^{1}H} NMR ($CDCl_3$): δ 15.3; IR (ν, cm^{-1}): 3078, 3053; 1616, 1495, 1469, 1436, 1389 (C=C, C=N, CON, P=N), 1217, 1110 (P-N-C), 1093, 1070, 954, 787; HRMS (ESI-TOF) (*m*/*z*) [M+H]$^+$ calcd for $C_{27}H_{21}ClN_2OP$ 455.1080, found 455.1076.

3-Imino-(triphenyl)-phosphorano-5-phenylisoxazole **11c** was obtained starting from 0.5 mmol (93.1 mg) of **5c** according to the **general method** above in 91% yield (191 mg) as off-white powder, which after recrystallization from ethylacetate/hexane (1/5) gave colorless transparent crystals. Analytical data for **11c**: R_f = 0.24 (hexane/ethyl acetate = 7/3); mp 135–138 ℃; ^{1}H NMR ($CDCl_3$): δ 7.79–7.86 (m, 6H), 7.68 (dd, J = 8.2, 1.3 Hz, 2H), 7.55 (td, J = 7.2, 1.4 Hz, 3H), 7.50–7.45 (m, 6H), 7.40–7.30 (m, 3H), 6.28 (bs, 1H); ^{13}C NMR ($CDCl_3$): δ 167.5, 167.1, 167.0, 132.6 (d, J_{CP} = 10.0 Hz, 6C), 131.6 (d, J_{CP} = 2.6 Hz, 3C), 128.9 (d, J_{CP} = 100.0 Hz, 3C), 128.7 (1C), 128.4 (1C), 128.2 (2C), 128.2 (d, J_{CP} = 12.0 Hz, 6C), 124.9 (2C), 98.1 (d, J_{CP} = 20.2 Hz); ^{31}P-{^{1}H} NMR ($CDCl_3$): δ 15.3; HRMS (ESI-TOF) (*m*/*z*) [M+H]$^+$ calcd for $C_{27}H_{22}N_2OP$ 421.1470, found 421.1765.

5-(4-Chloro-phenyl)-isoxazol-3-ylamine **12b**. A suspension of **11b** (0.11 mmol, 50 mg) in a mixture of 1 ml of EtOH and 1 ml of distilled water was heated at 100°C and intensive stirring for 24 h in a sealed vial. After that the reaction mixture was concentrated and the product was isolated by column chromatography (eluent hexane

/ ethyl acetate 3:2 vol.). After evaporation of the eluent the aminoisoxazole **12b** was obtained in 75% yield (16 mg) as white solid. Analytical data for **12b**: R_f = 0.27 (hexane/ethyl acetate 3:2 vol.); mp 136-139°C (lit. 137–138°C[vii]); ^{1}H NMR ($CDCl_3$): δ 7.64 (d, J = 8.7 Hz, 2H), 7.41 (d, J = 8.7 Hz, 2H), 6.08 (s, 1H), 4.06 (br. s, 2H) ($CDCl_3$); ^{13}C NMR ($CDCl_3$): δ 168.0, 163.3, 135.7, 128.8 (2C), 128.4, 126.5 (2C), 91.8; HRMS (ESI-TOF) (m/z) $[M+H]^+$ calcd. for $C_9H_8ClN_2O$ 195.0325, found 195.0319. Analytical data is in accordance with[26].

[5-(4-Chloro-phenyl)-isoxazol-3-yl]-(4-nitro-benzylidene)-amine **13b**. A suspension of 0.2 mmol (91 mg) of **11b** and 0.21 mmol (32 mg) of 4-nitro-benzaldehyde in 2 ml of toluene was subjected to heating at 120°C and stirring in a sealed vial for a period of 8 h. Then it was cooled to an ambient temperature, precipitate was collected by filtration, it was washed with 5 ml of a mixture of hexane/ethyl acetate (1/1) and dried giving **13b** as an off-white solid in 81% yield (53 mg). Analytical data for **13b**: mp 198–201 ℃; ^{1}H NMR (DMSO-d_6) δ 9.12 (s, 1H, -N=CH-), 8.37 (d, J = 8.8 Hz, 2H, 4-NO_2-Ph-C^{3,5}H), 8.22 (d, J = 8.8 Hz, 2H, 4-NO_2-Ph-C^{2,6}H), 7.88 (d, J = 8.6 Hz, 2H, 4-Cl-Ph-C^{2,6}H), 7.63 (d, J = 8.6 Hz, 2H, 4-Cl-Ph-C^{3,5}H), 7.41 (s, 1H, isoxazole-C^{4}H); ^{13}C NMR (DMSO-d_6) δ 170.41 (isoxazole-C^3), 169.57 (isoxazole-C^5), 166.32 (imine -N=CH-), 150.11 (C-NO_2), 140.63 (4-NO_2-Ph-C^1), 135.81 (C-Cl), 130.88 (4-NO_2-Ph-C^{2,6}), 129.94 (4-Cl-Ph-C^{2,6}), 127.67 (4-Cl-Ph-C^{3,5}), 126.05 (4-Cl-Ph-C^1-isoxazole), 124.64 (4-NO_2-Ph-C^{3,5}), 94.94 (isoxazole-C^4); IR (ν, cm^{-1}): 3118, 3099, 1600 (C=N), 1543 (NO_2), 1616, 1493, 1419, 1396, (C=C, C=N, CON), 1350 (NO_2),

1311 1220, 1108, 1093, 1012, 858, 847, 833, 808; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_{16}H_{11}ClN_3O_3$ 328.0489, found 328.0486.

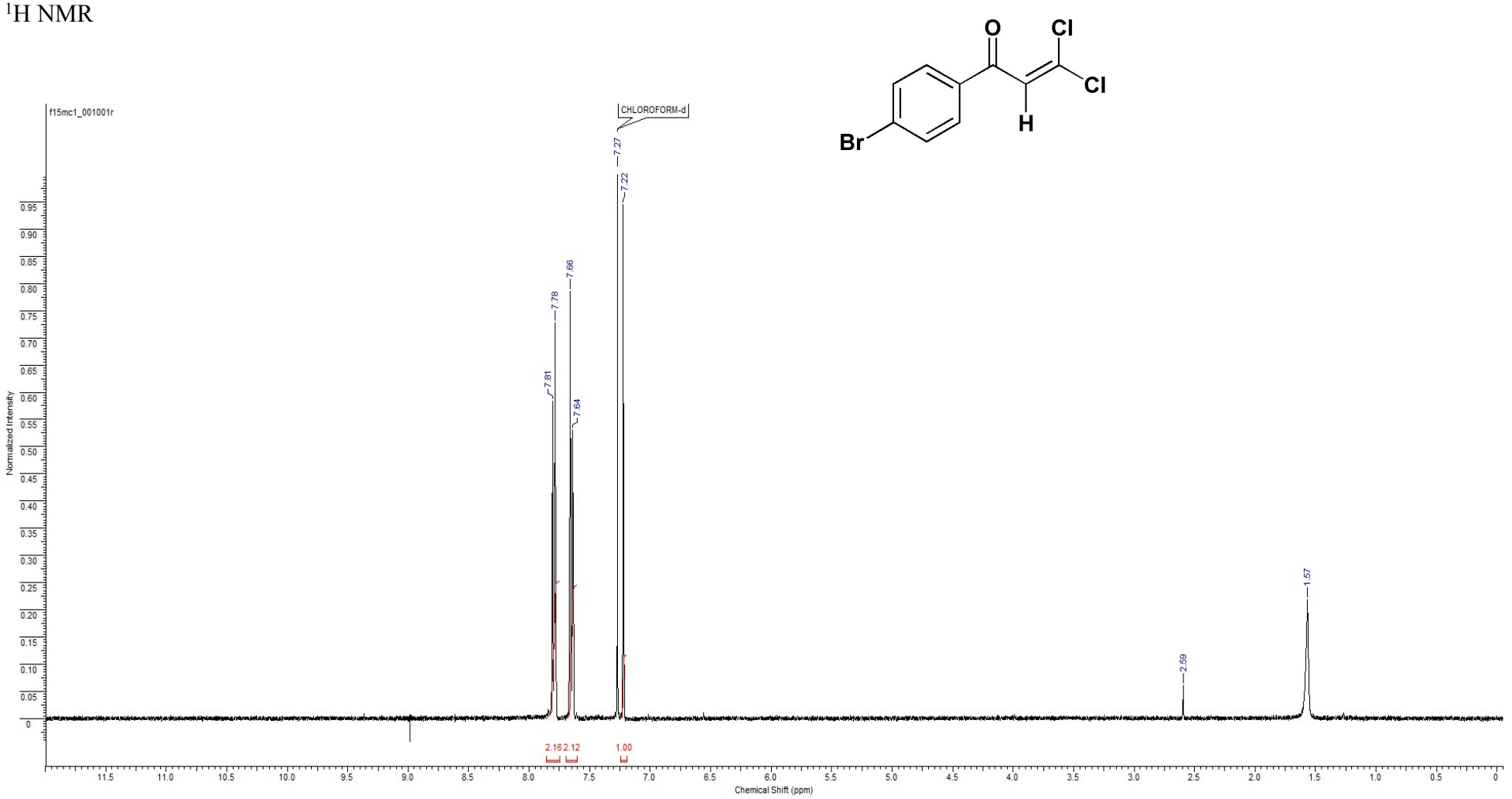
5-(4-Chloro-phenyl)-isoxazol-3-yl-phosphorimidic acid triethyl ester 14b may be isolated in 96% yield as an individual product (see procedure for **15b**). Analytical data for *14b*: R_f = 0.47 (hexane/ethyl acetate = 3/2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, J = 8.5 Hz, 2H), 7.37 (d, J = 8.5 Hz, 2H), 6.11 (d, J_{HP} = 1.0 Hz, 1H), 4.26–4.15 (m, 6H), 1.36 (td, J = 7.1, 0.5 Hz, 9H); ^{13}C NMR ($CDCl_3$): δ 167.1, 164.9 (d, J_{CP} = 4.7 Hz), 134.9, 128.6 (2C), 126.6 (1C), 126.2 (2C), 97.0 (d, J_{CP} = 19.7 Hz), 64.1 (d, J_{CP} = 5.1 Hz, 3C), 15.7 (d, J_{CP} = 6.9 Hz, 3C); ^{31}P-{^{1}H} NMR: δ 5.8.

[5-(4-Chloro-phenyl)-isoxazol-3-yl]-phosphoramidic acid diethyl ester **15b**. To a solution of 0.5 mmol (110.3 mg) of **5b** in 4 ml of dichloromethane at 5°C (ice bath cooling) and stirring a solution of 0.5 mmol (83 mg) of triethylphosphite in 1 ml of dichloromethane was added dropwise. After addition of triethylphosphite the reaction mixture was allowed to reach the room temperature and was stirred at ambient temperature for another 1 h.[viii] Then to the reaction mixture 2 ml of 5% HCl had been added dropwise at vigorous stirring. After 1 h of stirring at ambient temperature **15b** was isolated extraction from the reaction mixture with dichloromethane (3x10 ml) and following standard work-up procedures in 94% yield (155 mg) as an off-white solid. Analytical data for **15b**: R_f = 0.17 (hexane/ethyl acetate = 3/2); mp 152–155 ℃; ^{1}H NMR (400.1 MHz, $CDCl_3$): δ 7.83 (d, J_{HP} = 8.3 Hz, 1H, NH), 7.68 (d, J = 8.6 Hz, 2H, Ar), 7.42 (d, J = 8.6 Hz, 2H, Ar), 6.54 (s, 1H, isoxazole-C^{4}H), 4.32–4.05 (m, 4H, CH_2), 1.36 (t, J = 7.1 Hz, 6H, CH_3); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 168.4 (1C,

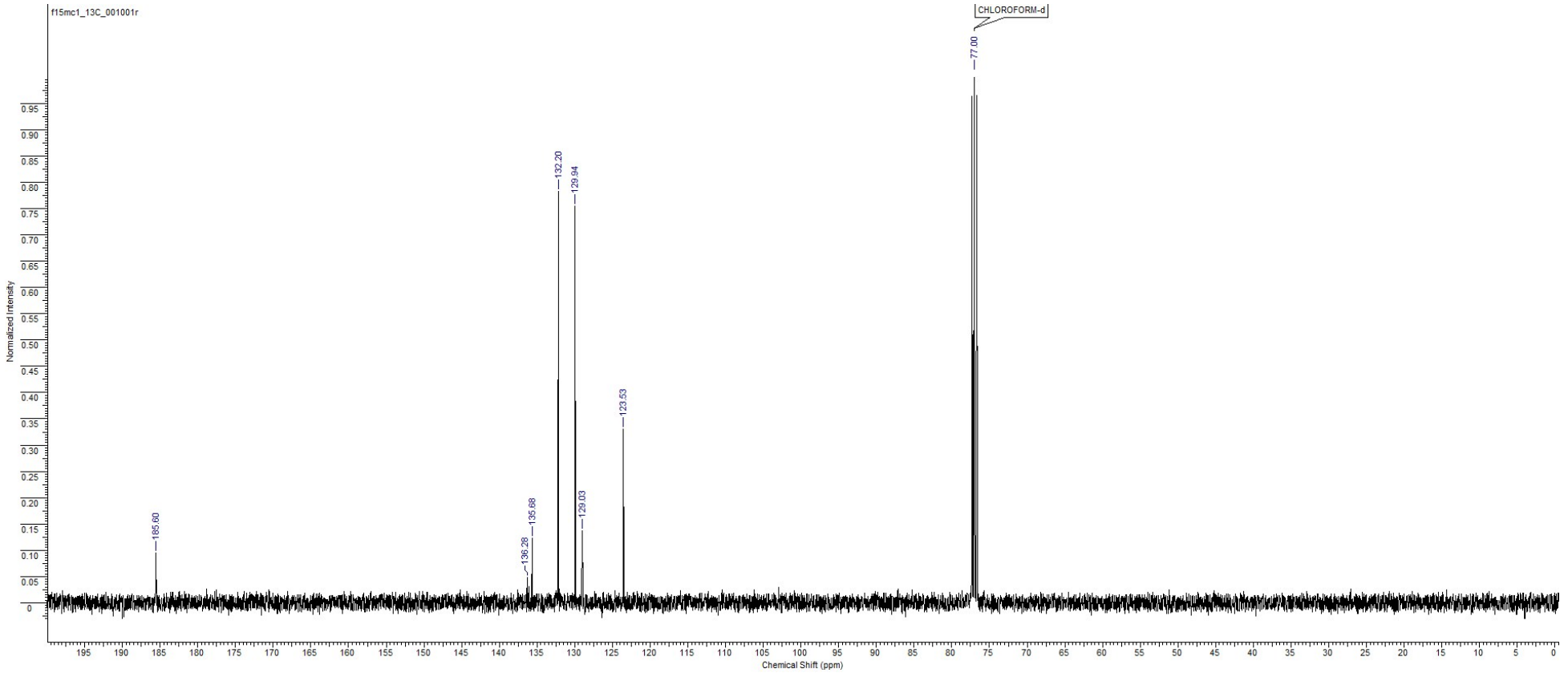
isoxazole-C^5), 159.3 (d, J_{CP} = 4.7 Hz, 1C, isoxazole-C^3), 135.9 (1C, Cl-Ar-C^4), 128.8 (2C, Cl-Ar-$C^{3,5}$), 126.6 (2C, Cl-Ar-$C^{2,6}$), 125.4 (1C, Ar-C^1), 92.3 (1C, isoxazole-C^4), 63.2 (d, J_{CP} = 5.1 Hz, 2C, CH_2), 15.7 (d, J_{CP} = 6.9 Hz, 2C, CH_3); ^{31}P-{^{1}H} NMR ($CDCl_3$): δ 0.4; IR (ν, cm^{-1}): 3155, 3136, 3099, 2987, 2929, 2910, 2858, (CH and NH stretchings), 1618, 1522, 1506, 1406 (C=C, C=N, CON), 1234 (P=O), 1192, 1091, 1049, 1030, 974, 945 (P-O-Alk, P-N), 910, 837, 798; HRMS (ESI-TOF) (*m*/*z*) $[M+H]^+$ calcd for $C_{13}H_{17}ClN_2O_4P$ 331.0614, found 331.0610.

1-(4-bromophenyl)-3,3-dichloroprop-2-en-1-one **1a**

^{1}H NMR



^{13}C NMR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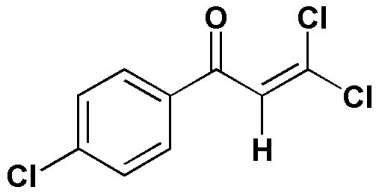
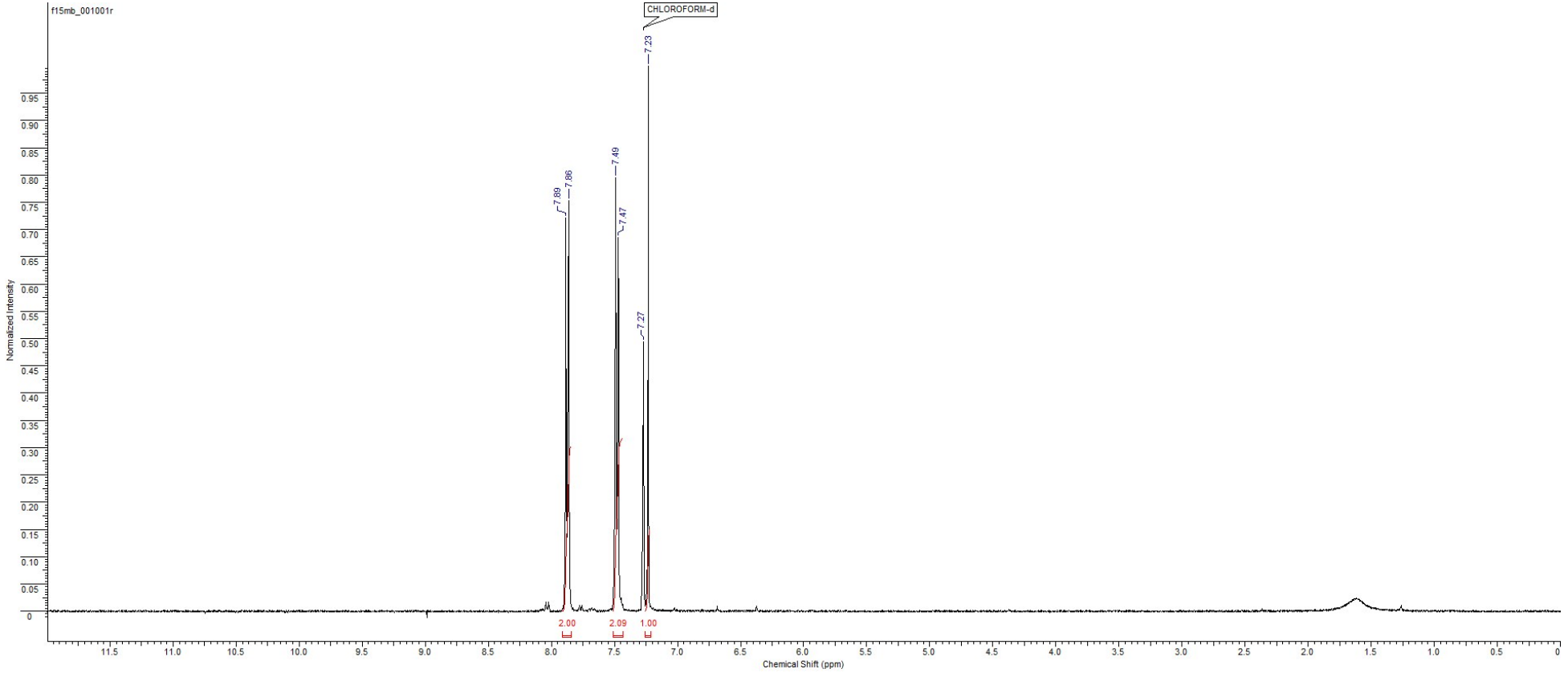
Normalized Intensity
Chemical Shift (ppm)

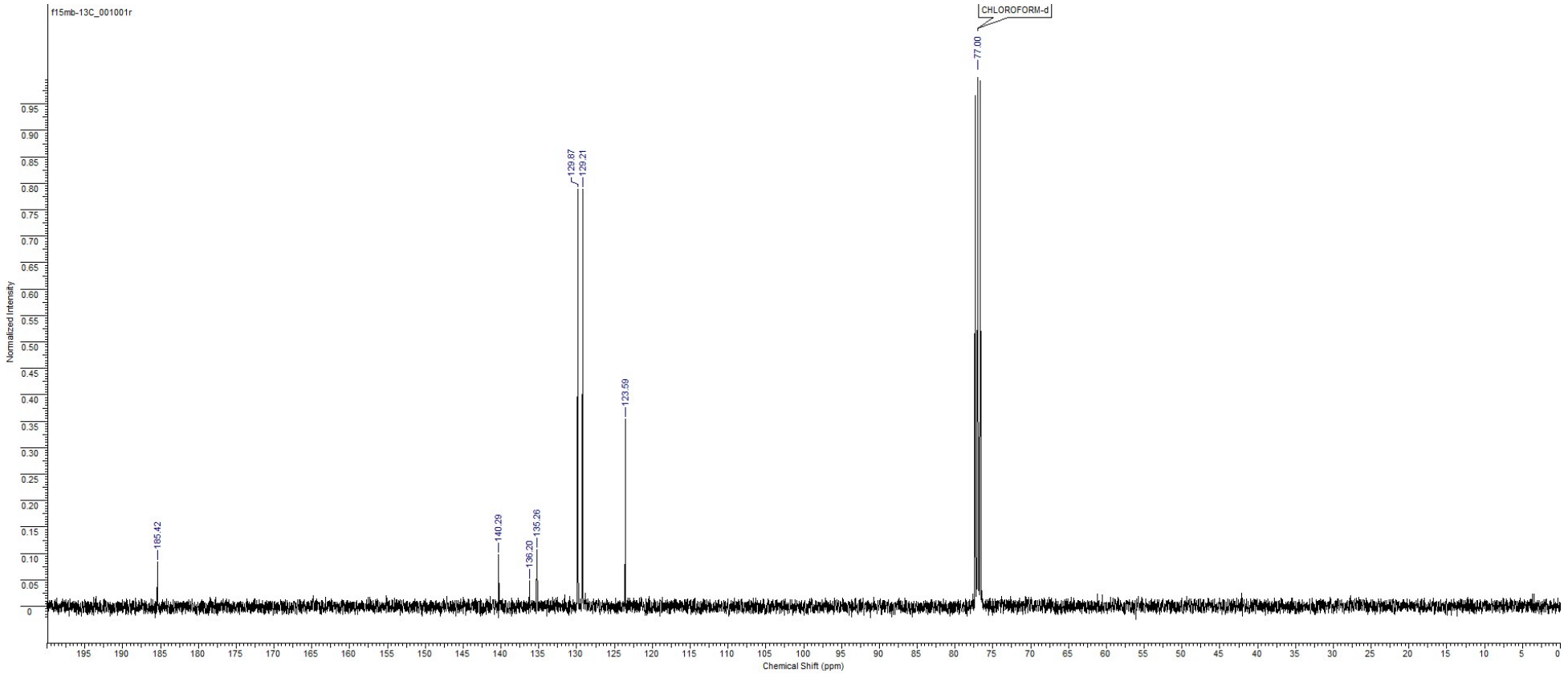
3,3-dichloro-1-(4-chlorophenyl)prop-2-en-1-one **1b**

^{1}H NMR





^{13}C NMR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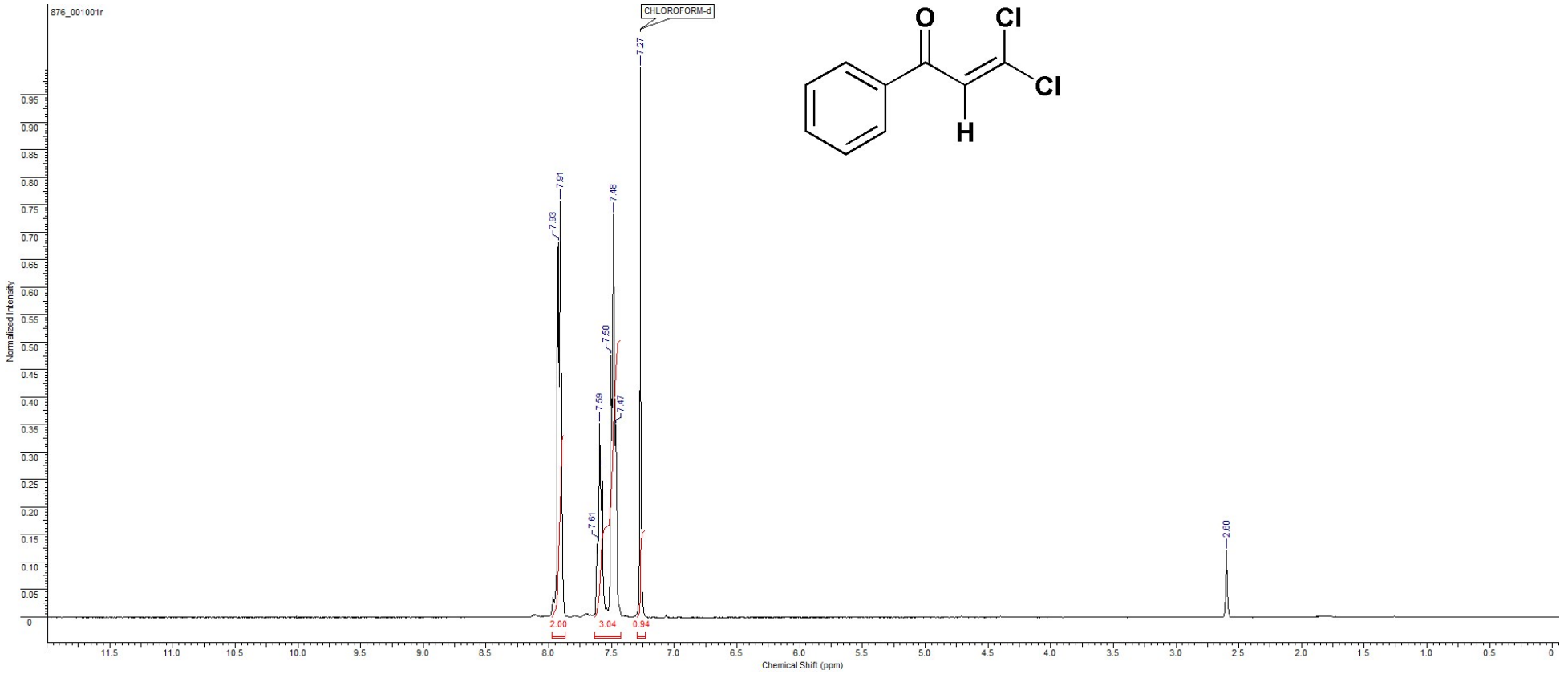
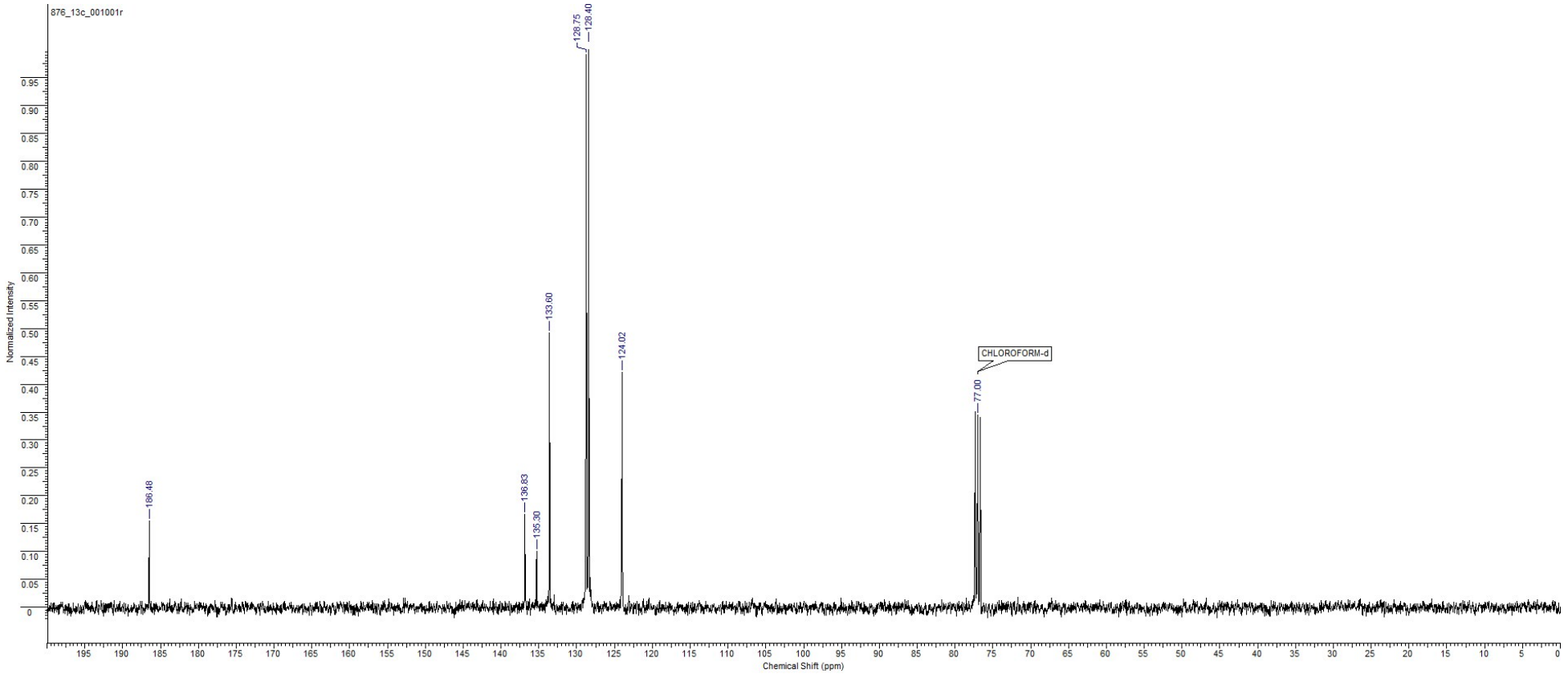
Normalized Intensity
Chemical Shift (ppm)

3,3-dichloro-1-phenylprop-2-en-1-one **1c**

^{1}H NMR

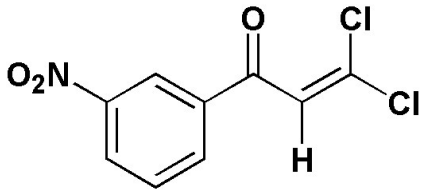


^{13}C NMR

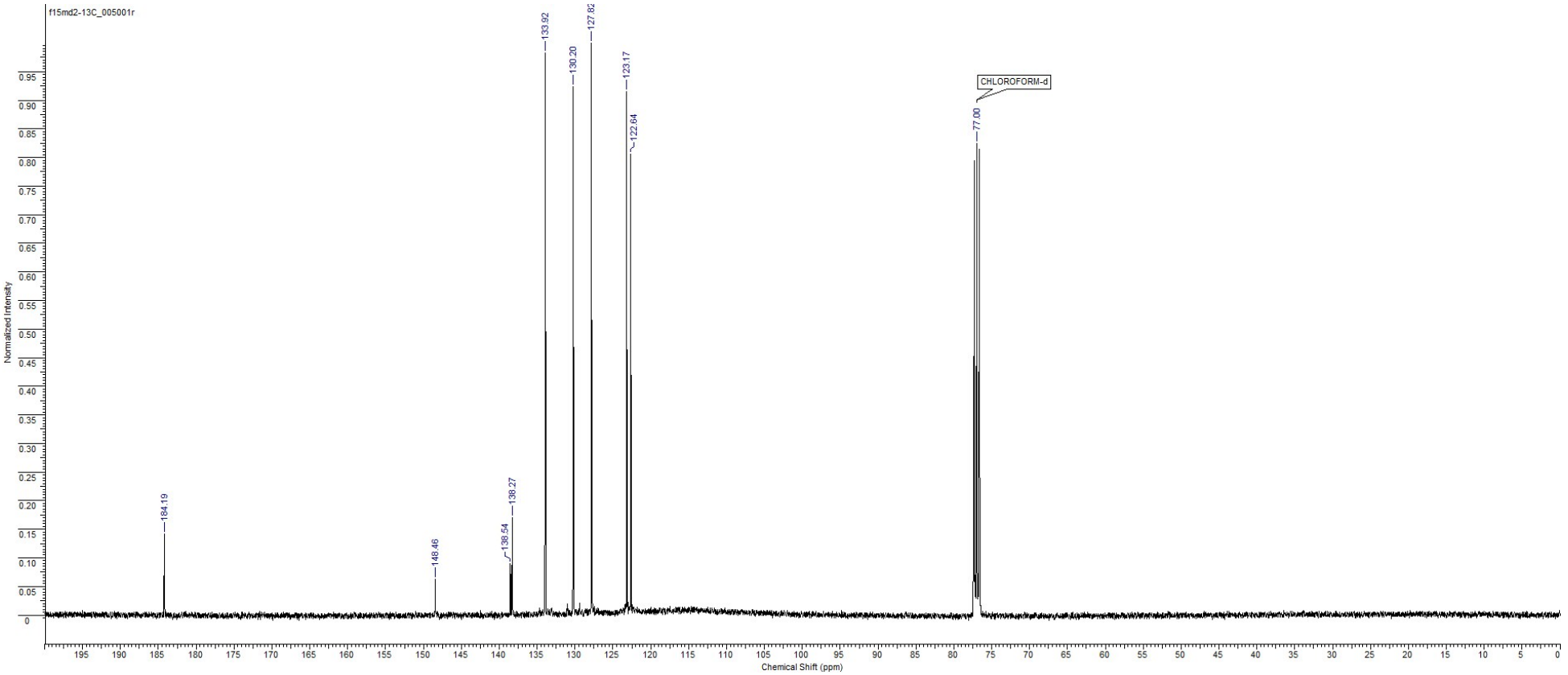

876_13c_001001r
Normalized Intensity
Chemical Shift (ppm)
186.48
136.83
135.30
133.60
128.75
128.40
124.02
77.00
CHLOROFORM-d

3,3-dichloro-1-(3-nitrophenyl)prop-2-en-1-one **1d**

^{1}H NMR

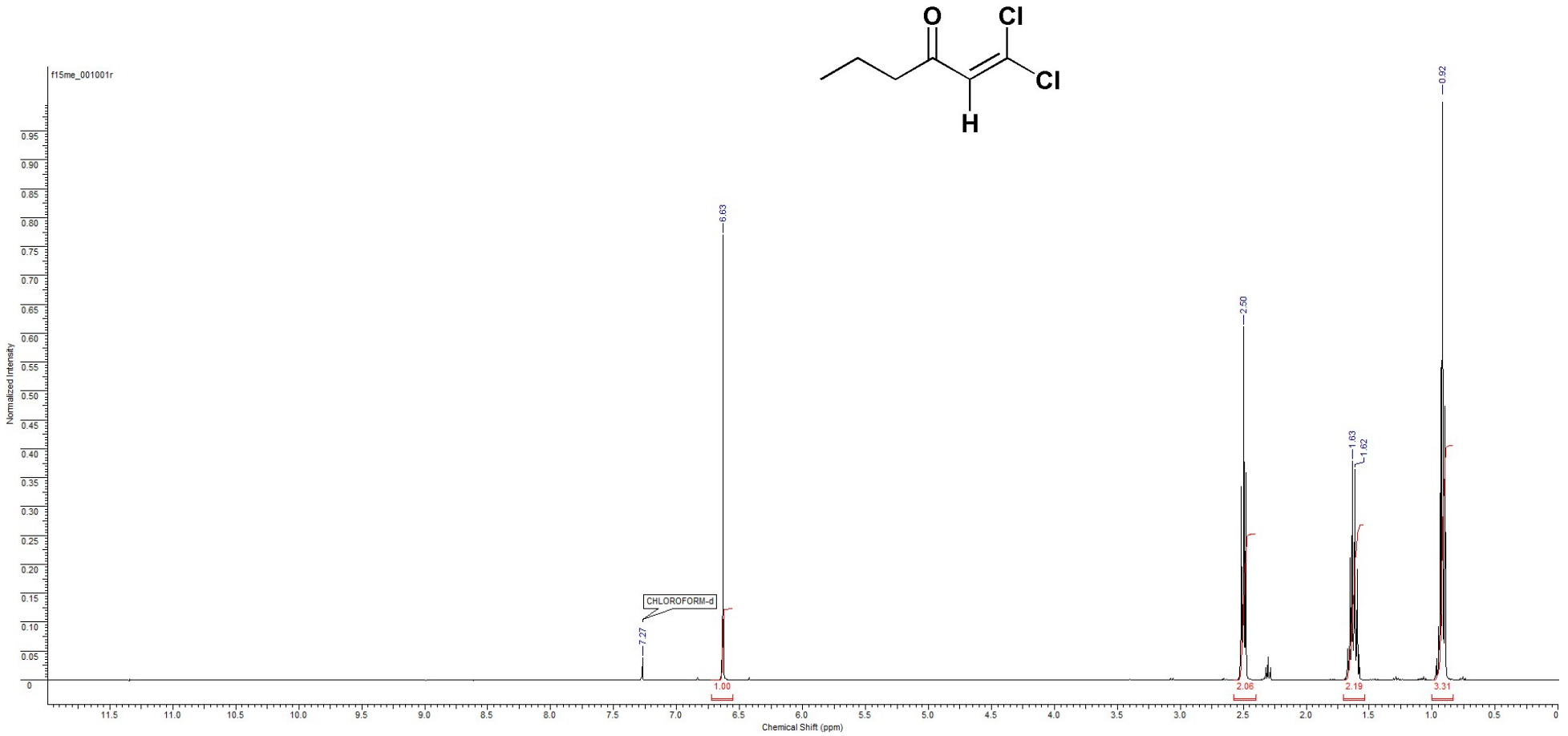


^{13}C NMR

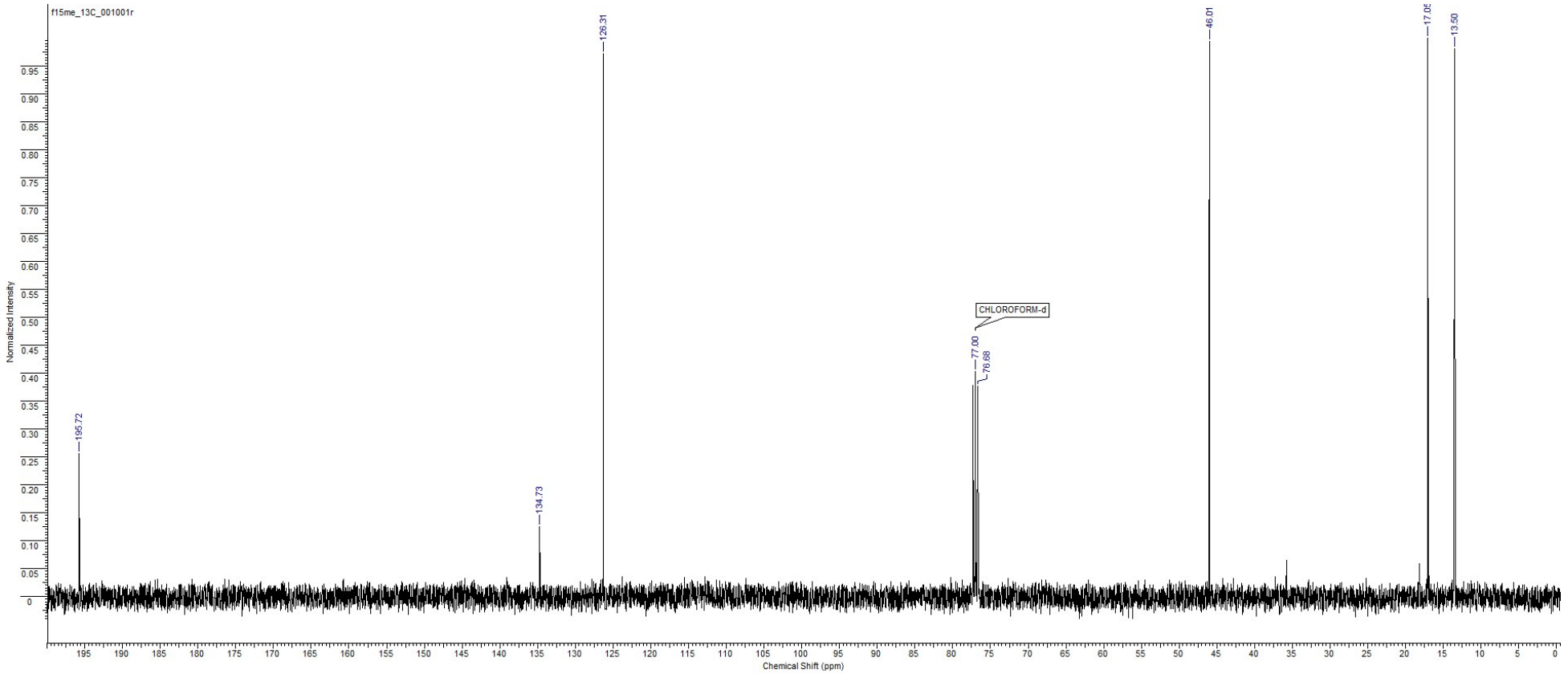

f15md2-13C_005001r
Normalized Intensity
Chemical Shift (ppm)
184.19
148.46
138.54
138.27
133.92
130.20
127.82
123.17
122.64
77.00
CHLOROFORM-d

1,1-dichlorohex-1-en-3-one **1e**

^{1}H NMR

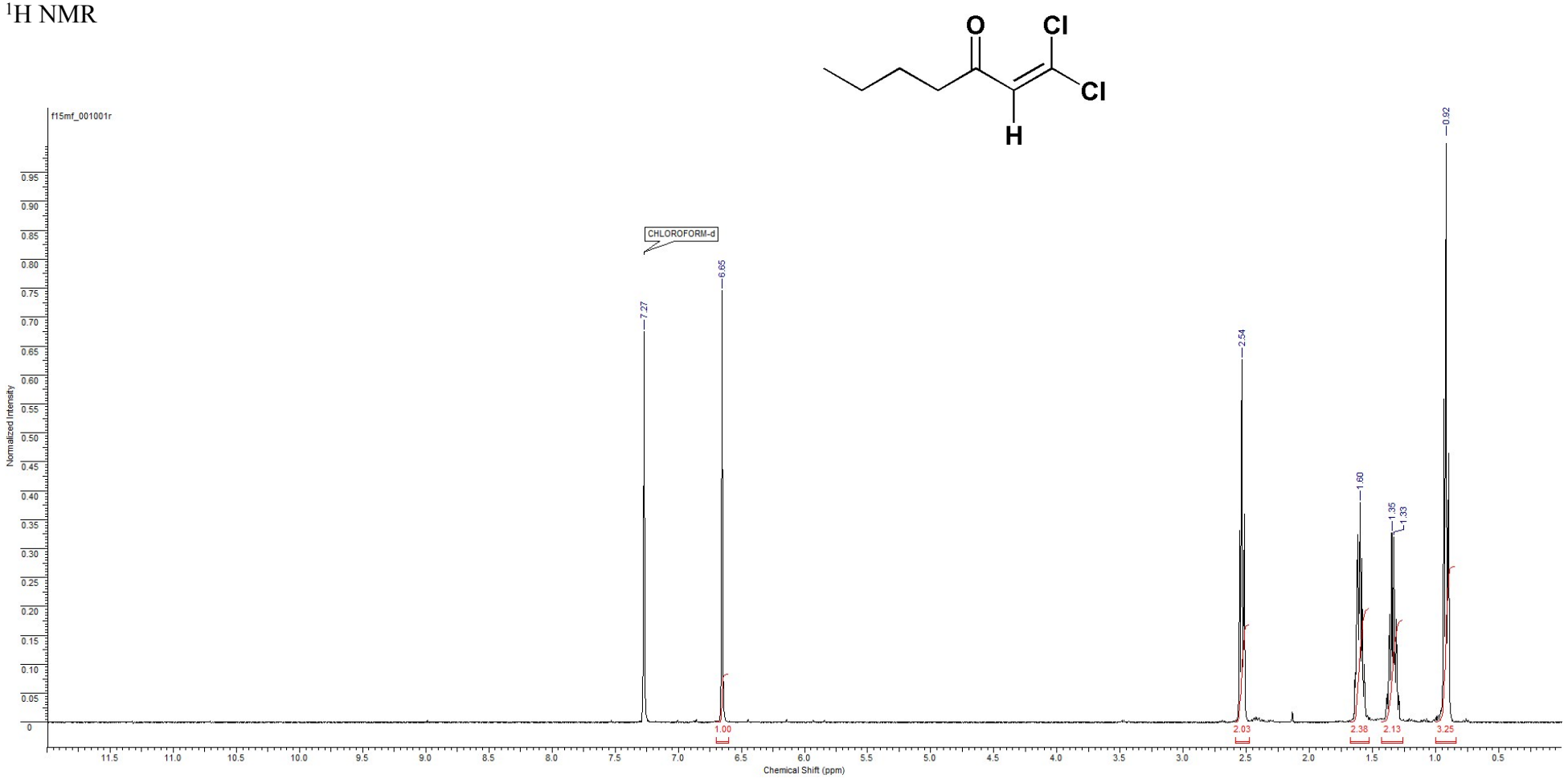


^{13}C NMR

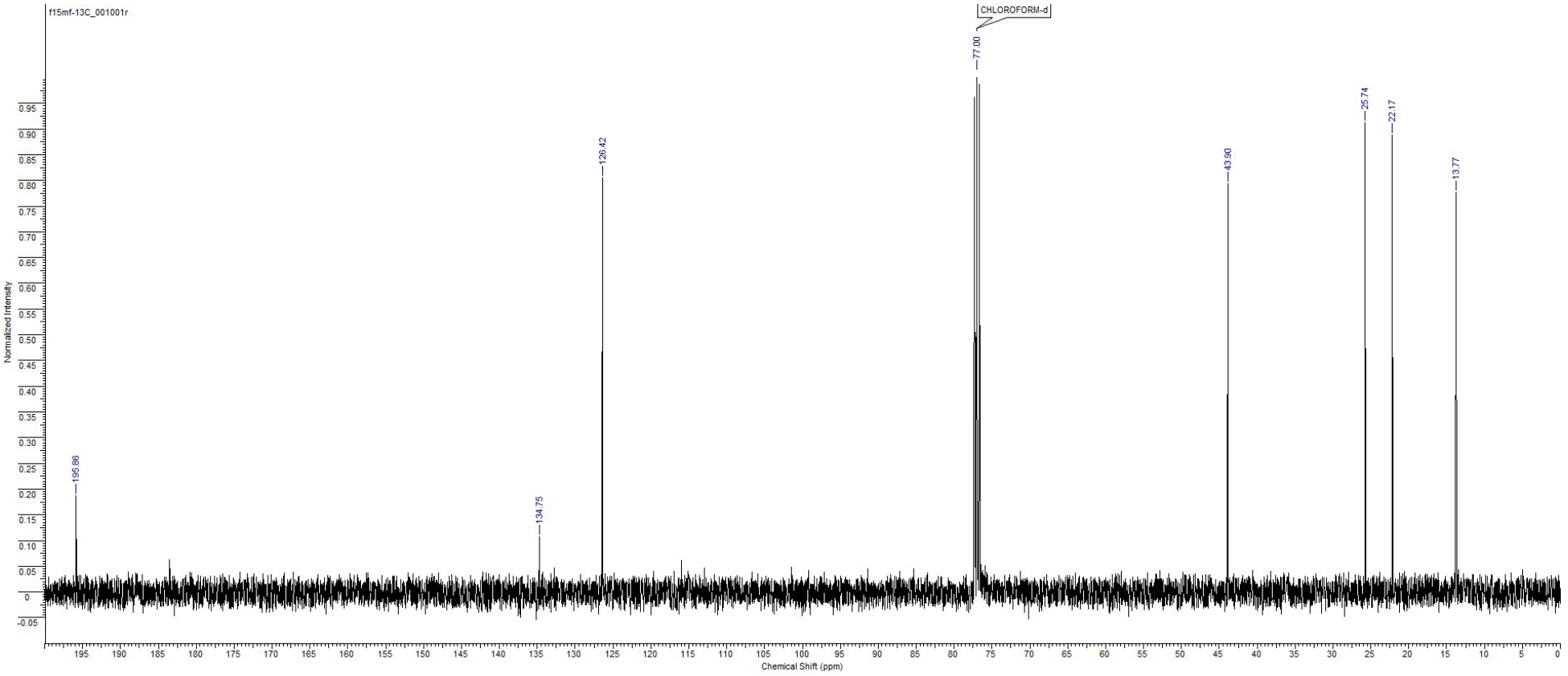

f15me_13C_001001r
Normalized Intensity
Chemical Shift (ppm)
195.72
134.73
126.31
77.00
76.68
CHLOROFORM-d
46.01
17.05
13.50

1,1-dichlorohept-1-en-3-one **1f**

^{1}H NMR

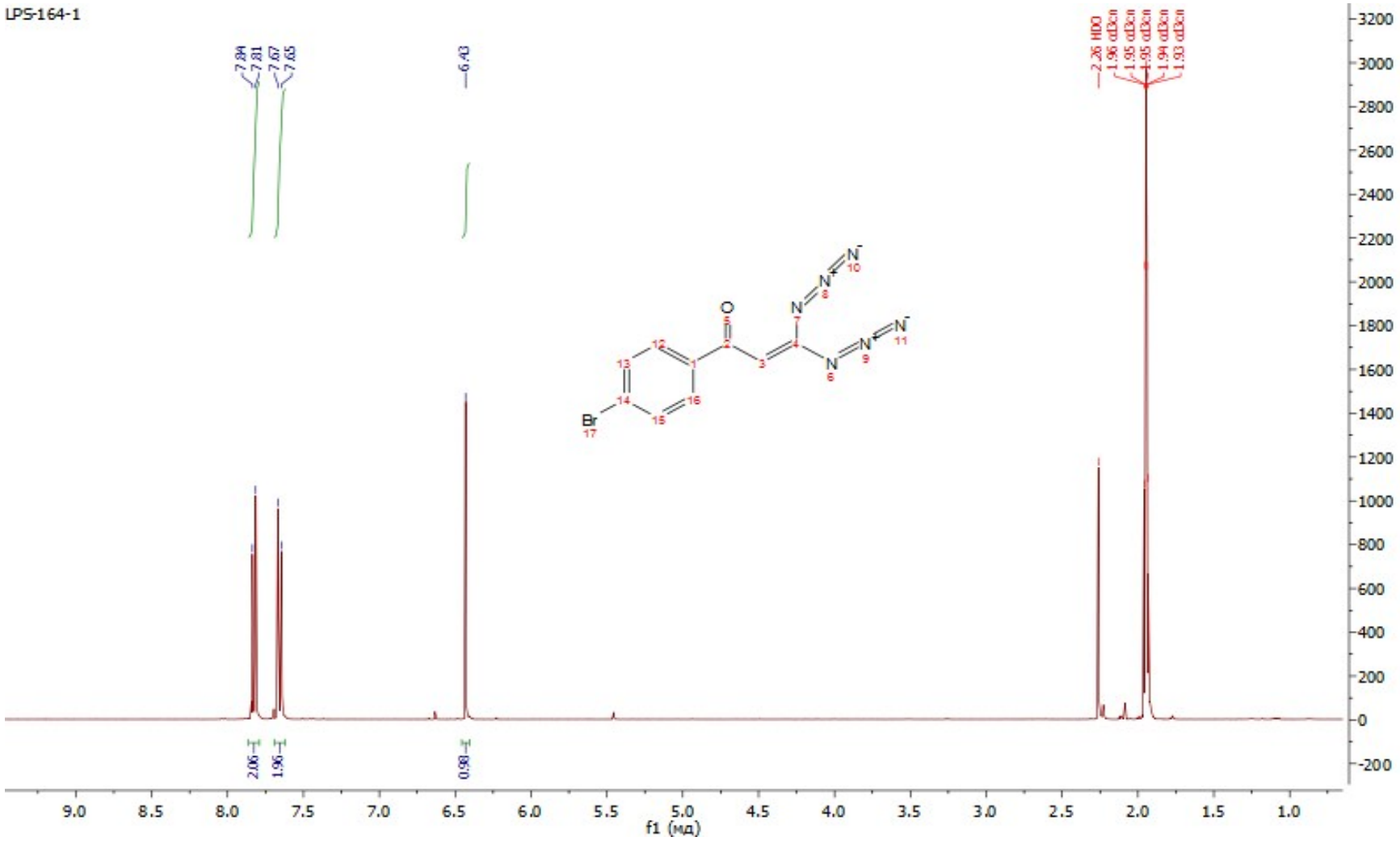


^{13}C NMR

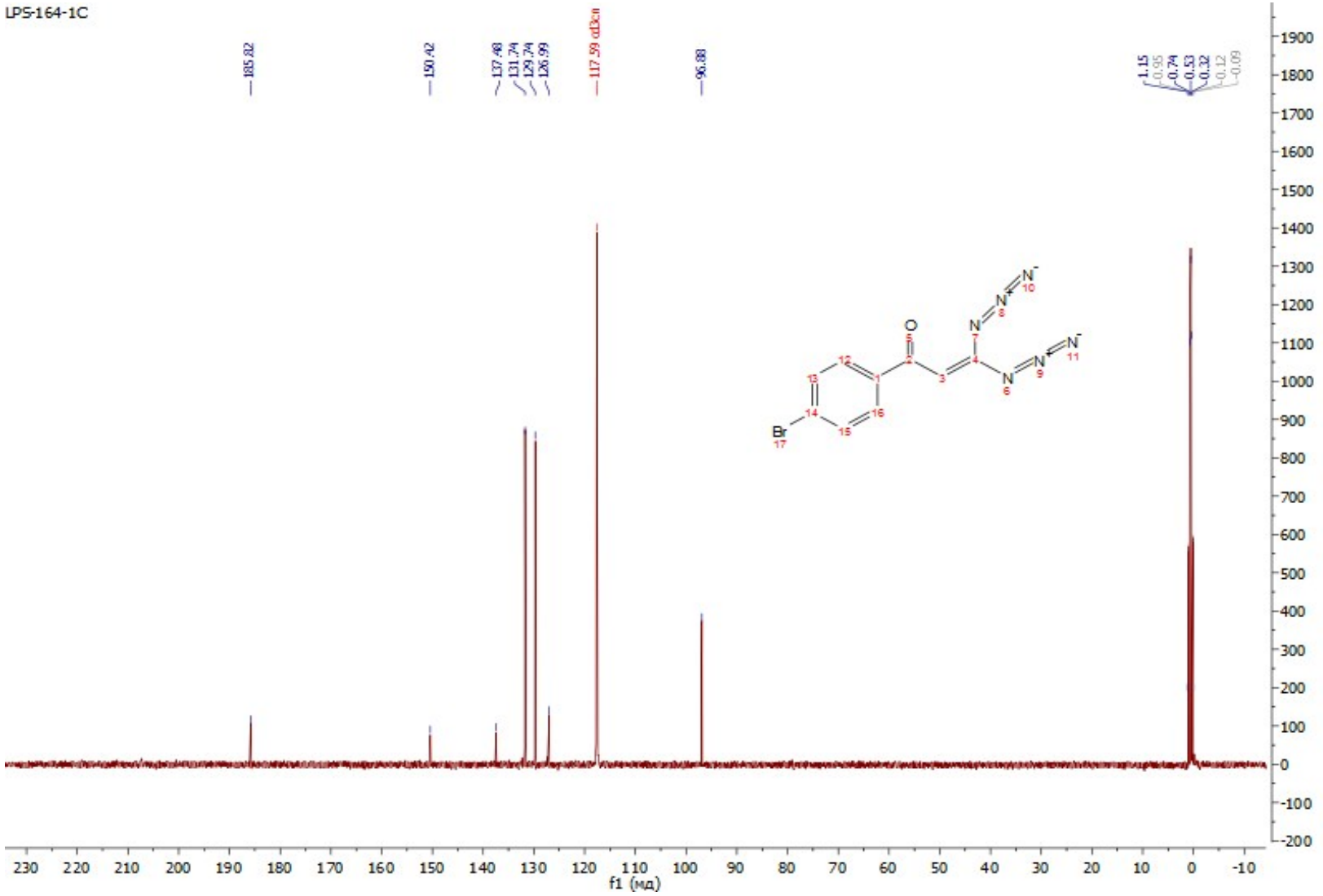

f15mf-13C_001001r
CHLOROFORM-d
77.00
126.42
43.90
25.74
22.17
13.77
195.86
134.75
Normalized Intensity
Chemical Shift (ppm)

3,3-diazido-1-(4-bromophenyl)prop-2-en-1-one **3a**

^{1}H NMR (CD_3CN), T = 0°C

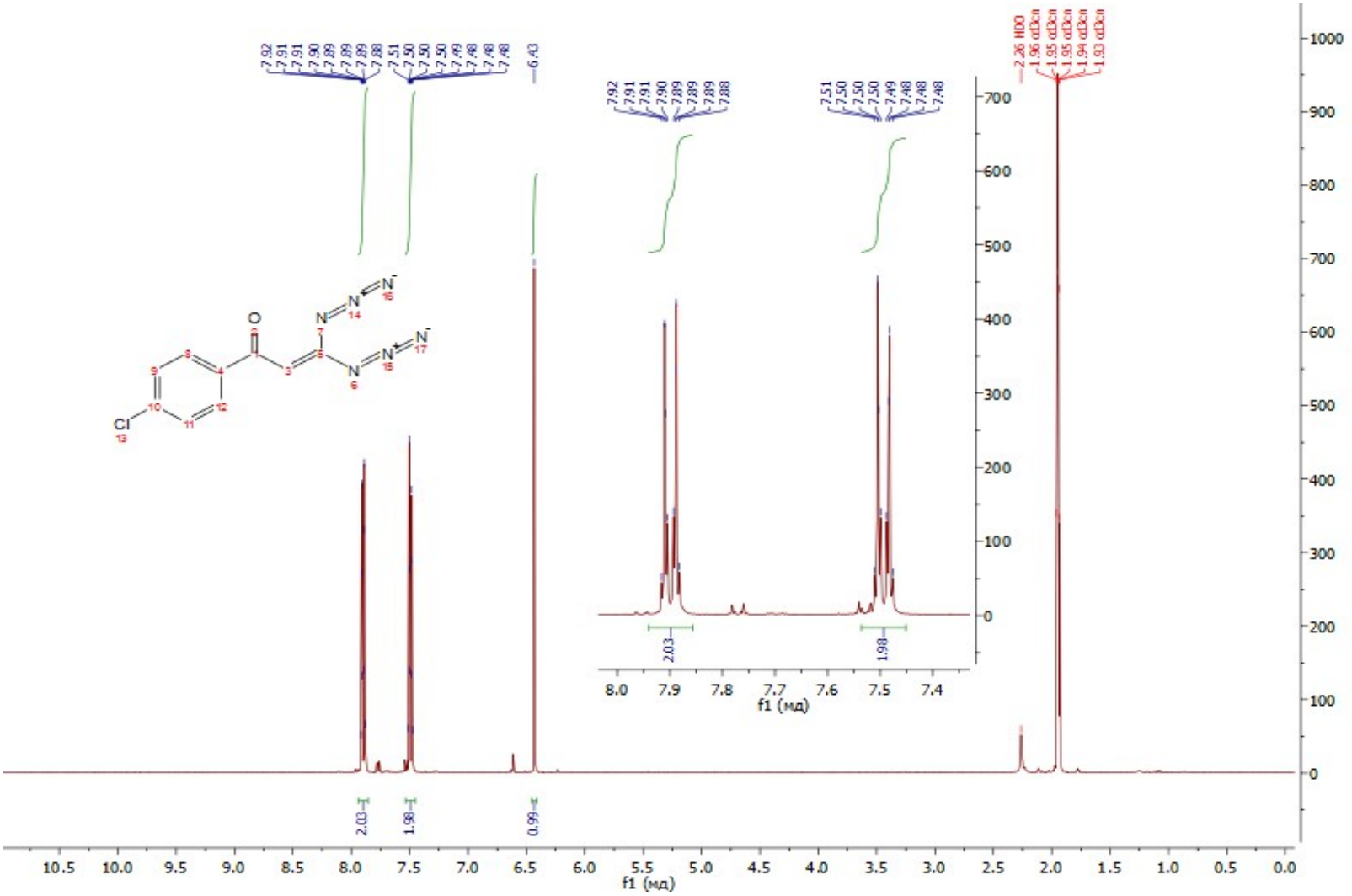


^{13}C NMR (CD_3CN), T = 0°C

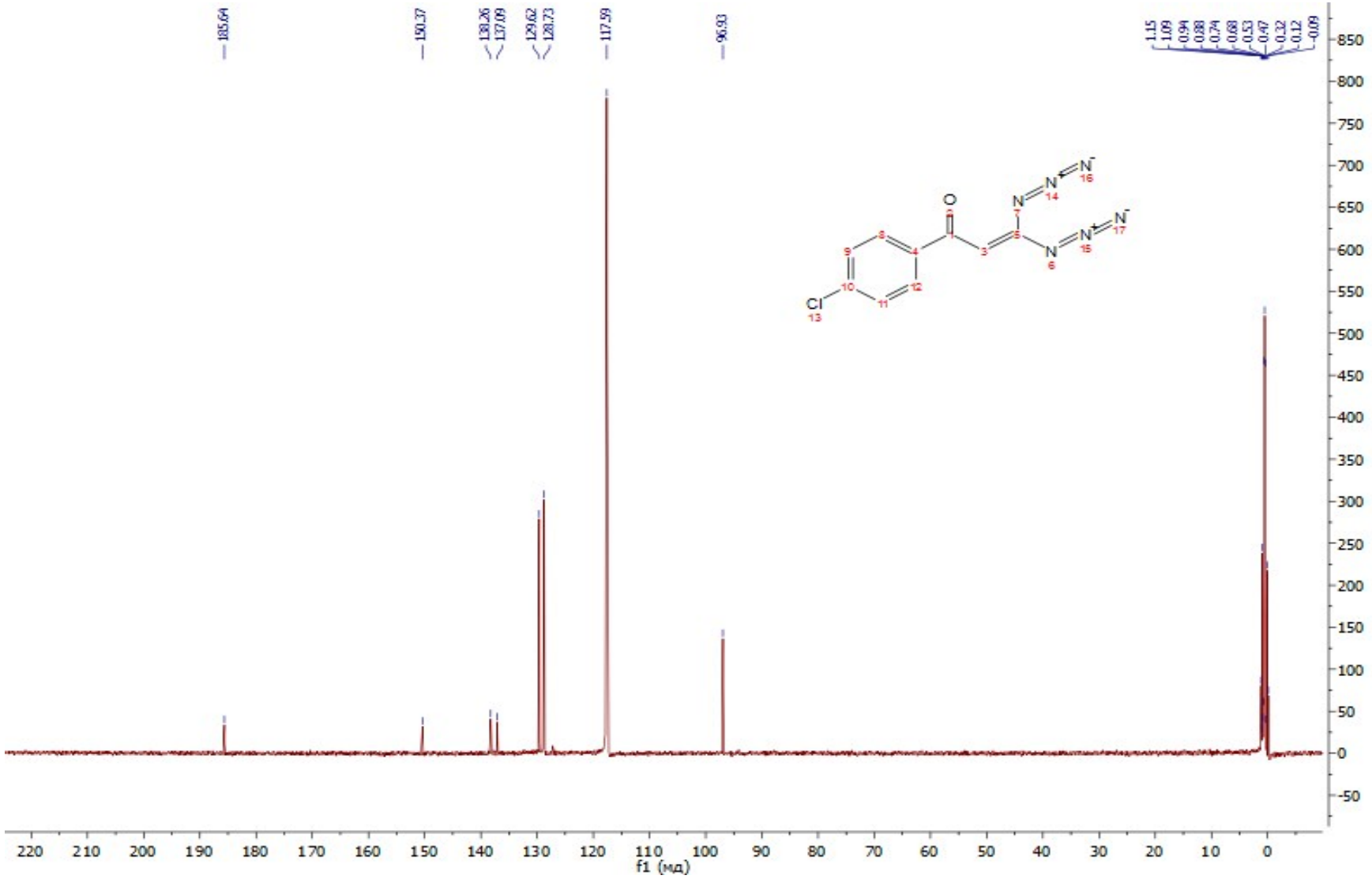


3,3-diazido-1-(4-chlorophenyl)prop-2-en-1-one **3b**

^{1}H NMR (CD_3CN), T = 0°C

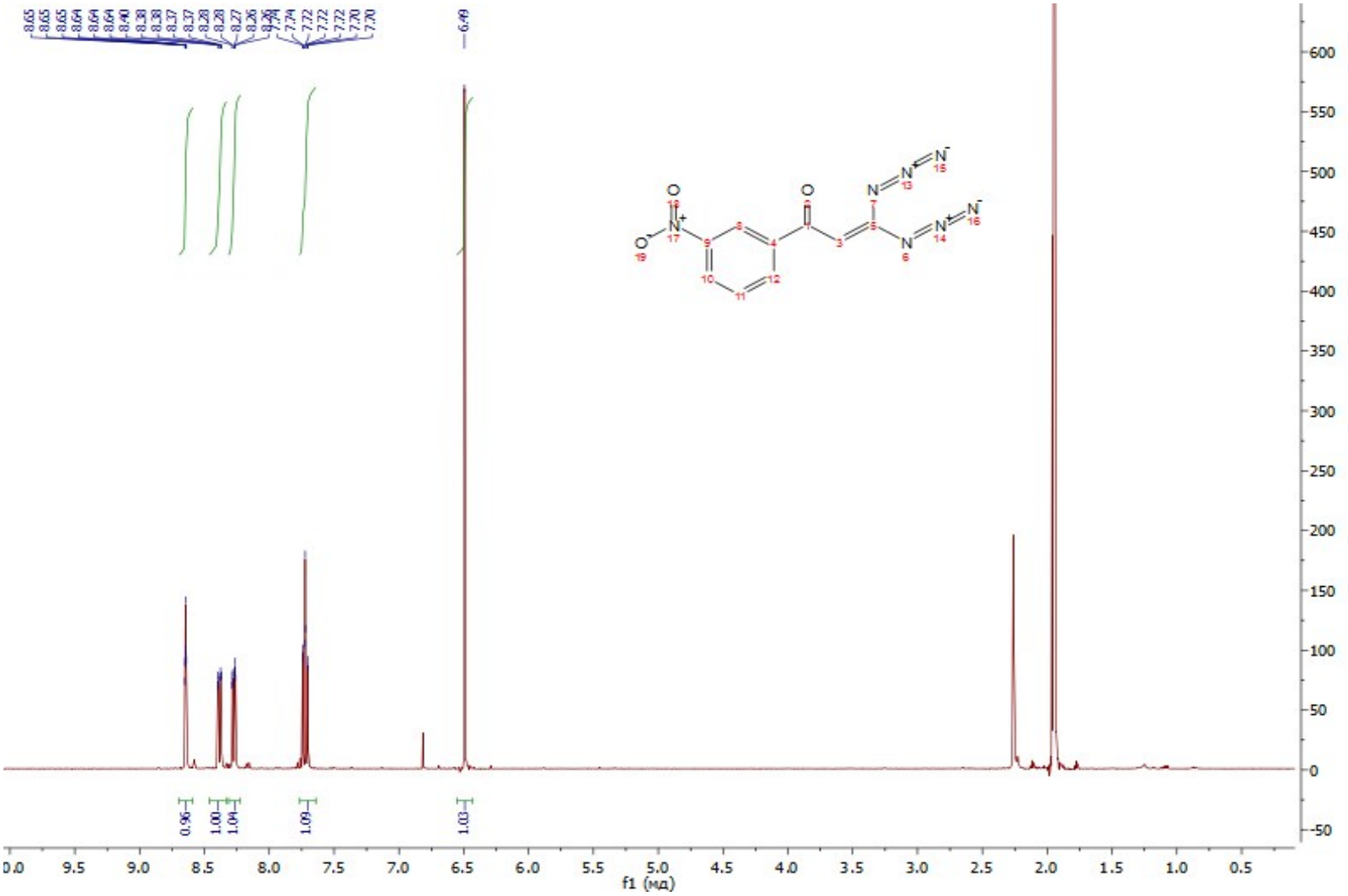


^{13}C NMR (CD_3CN), T = 0°C

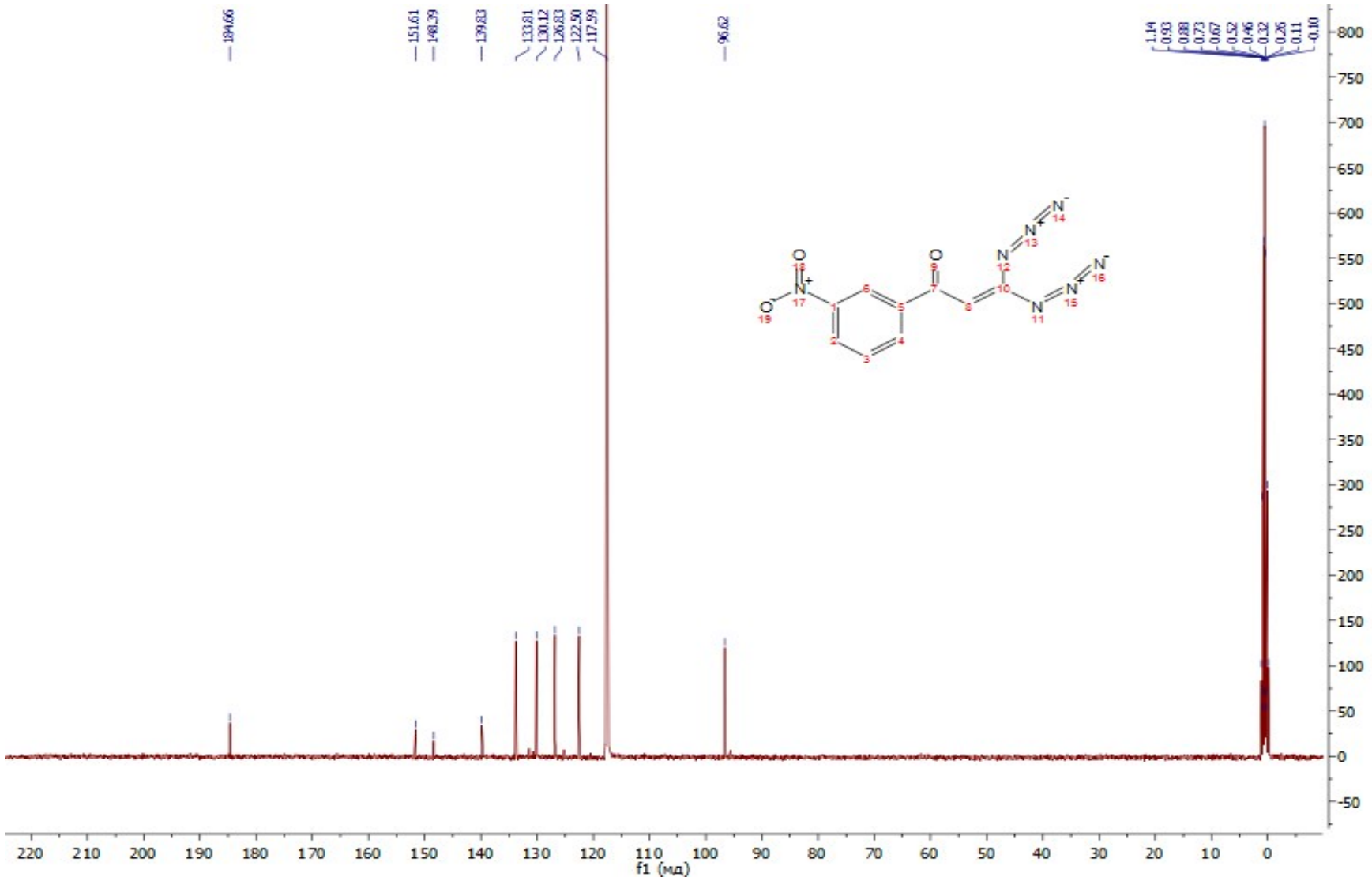


3,3-diazido-1-(3-nitrophenyl)prop-2-en-1-one **3d**

^{1}H NMR (CD_3CN, 0^{o}C)

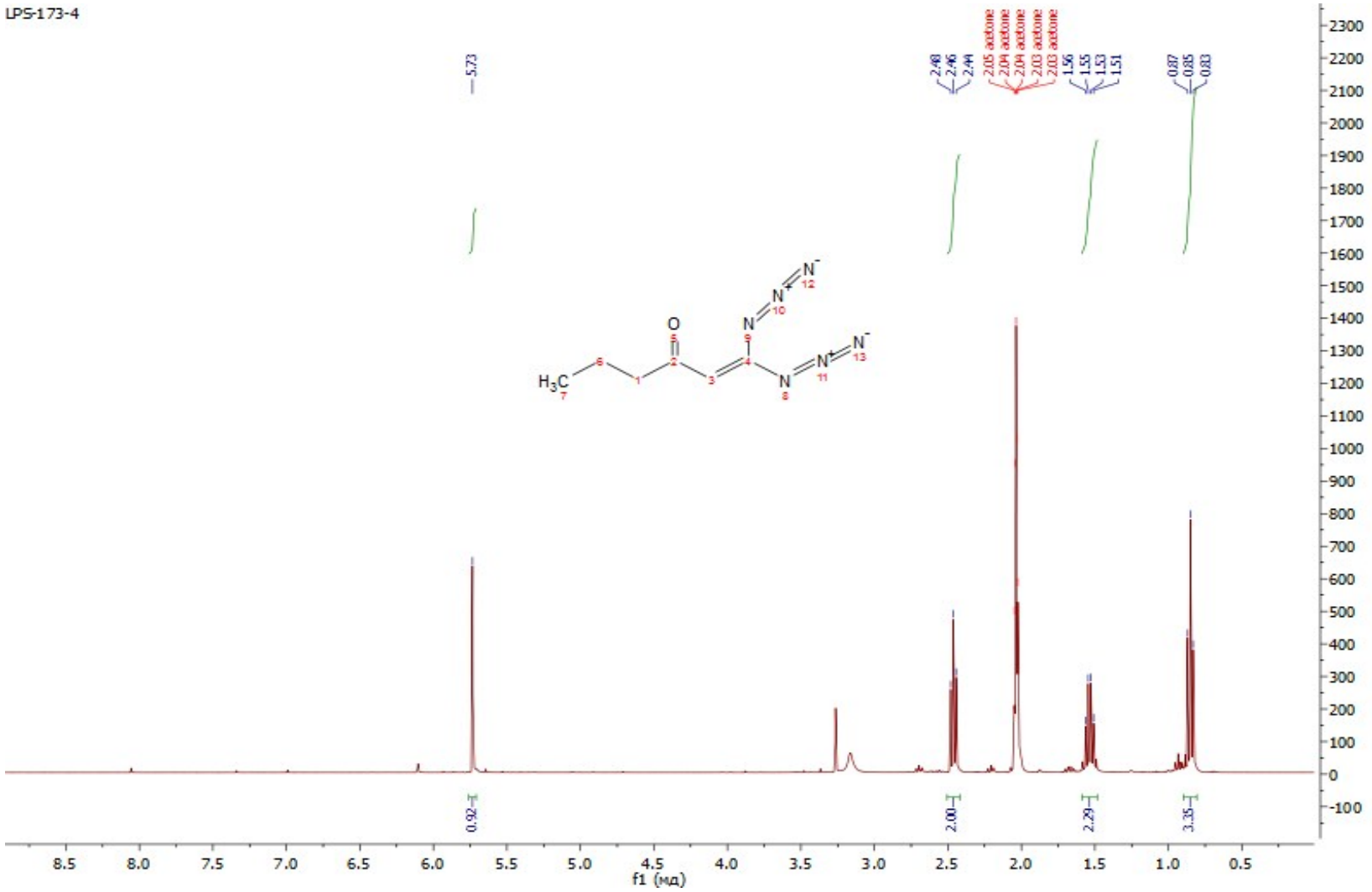


^{13}C NMR (CD_3CN, 0°C)

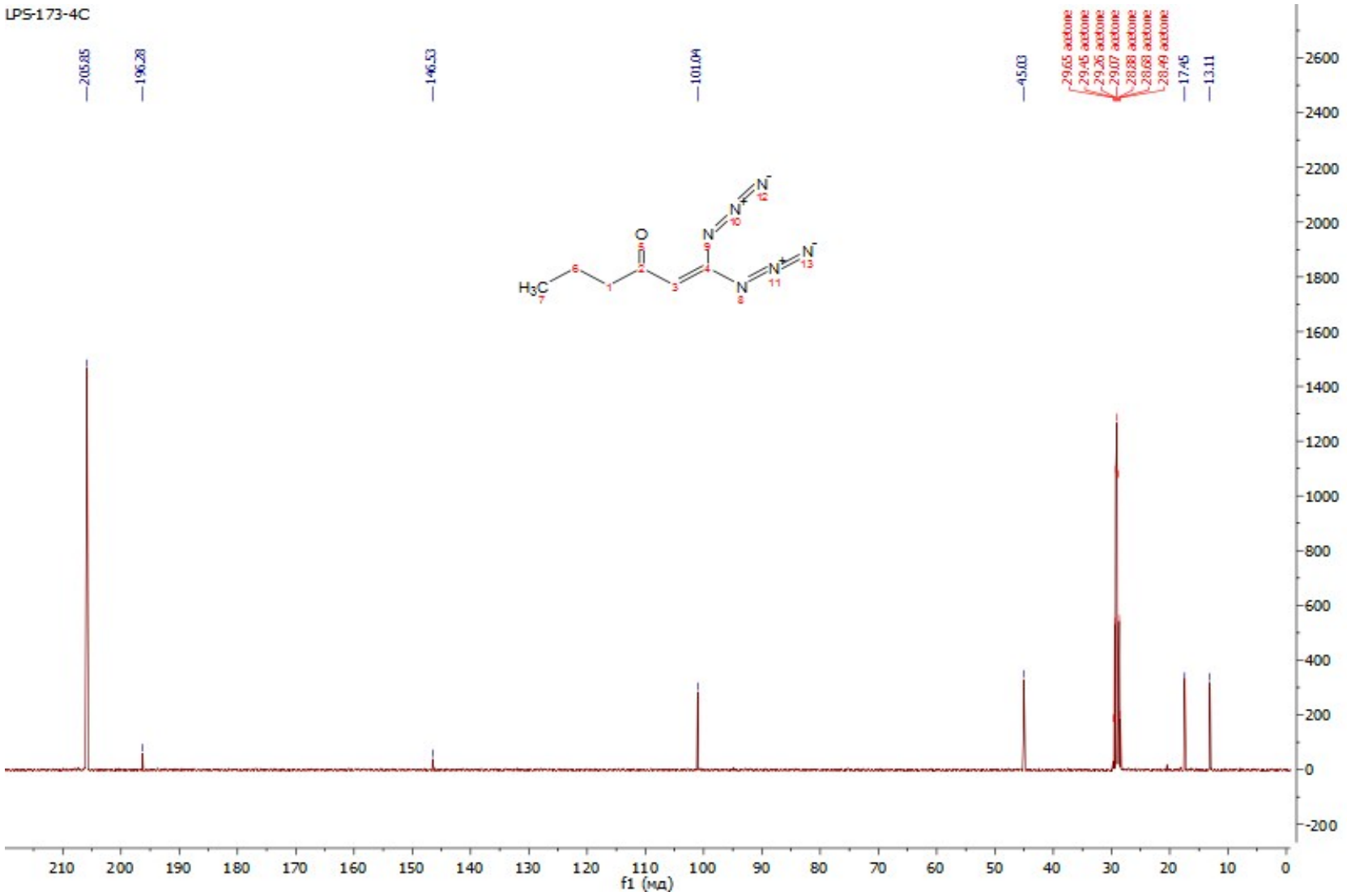


1,1-diazidohex-1-en-3-one **3e**

^{1}H NMR (solvent $(CD_3)_2CO$), T = 0^{o}C

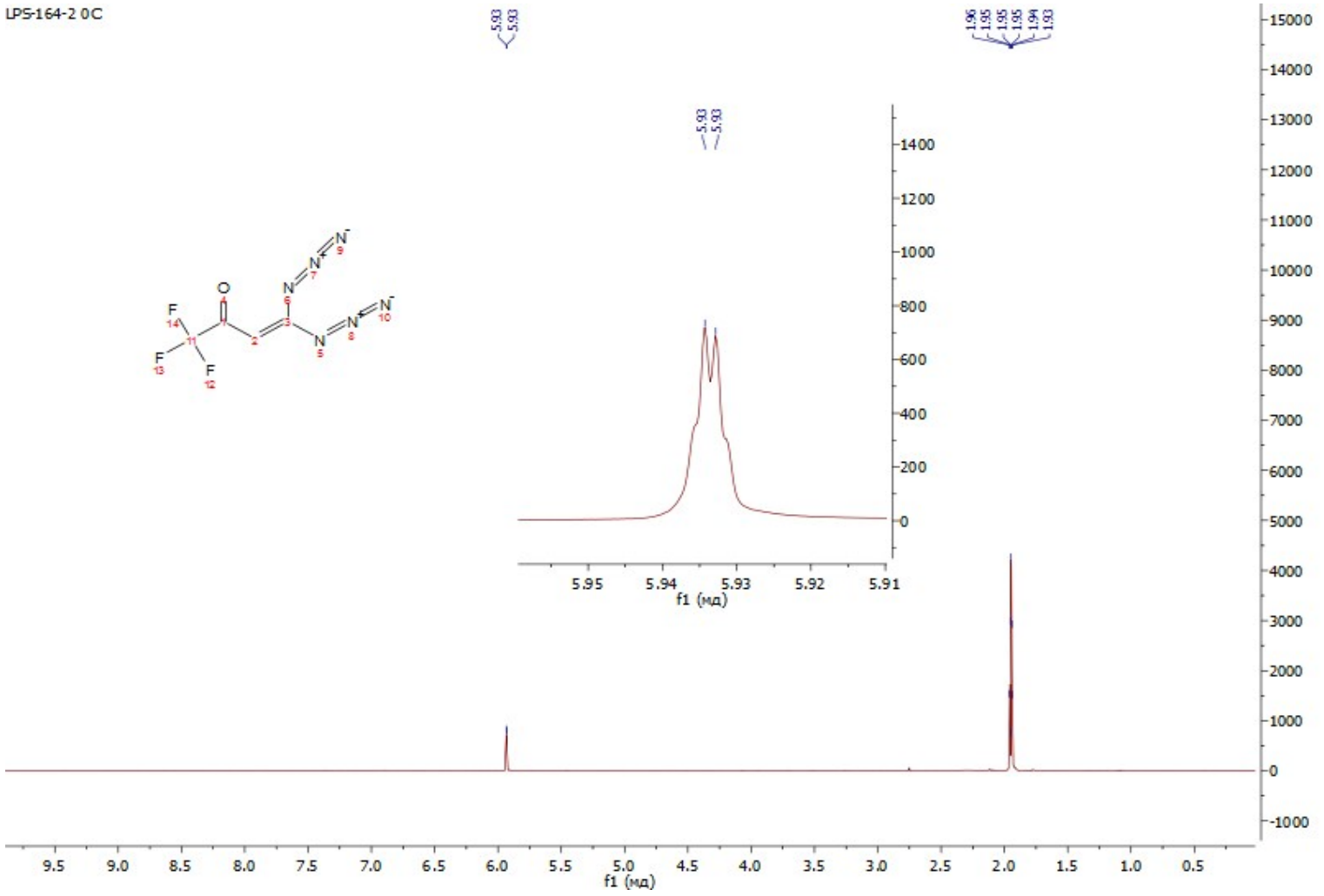


^{13}C NMR (solvent $(CD_3)_2CO$), T = 0°C

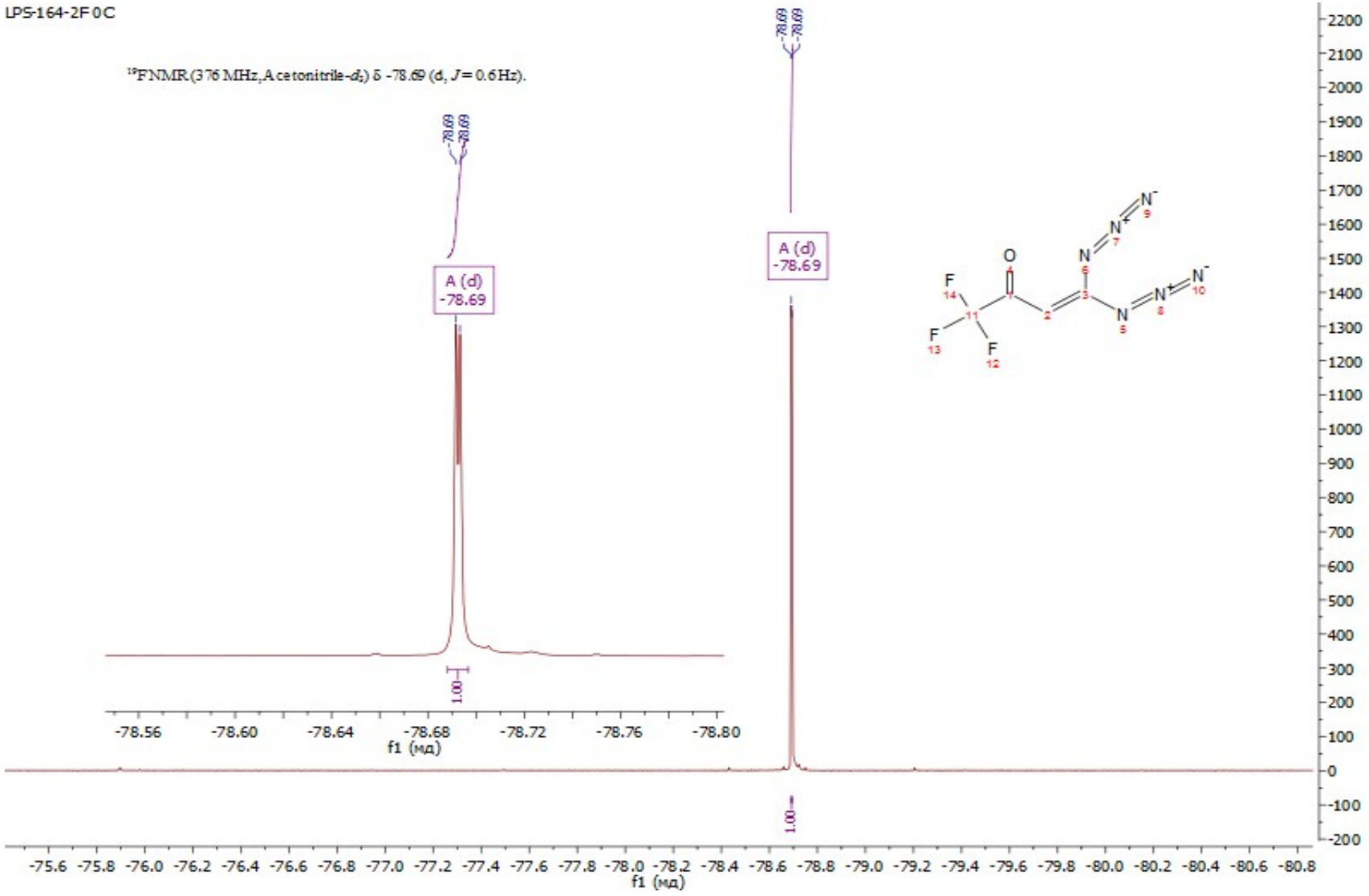


4,4-diazido-1,1,1-trifluorobut-3-en-2-one **3h**

^{1}H NMR (CD_3CN), T = 0^{o}C

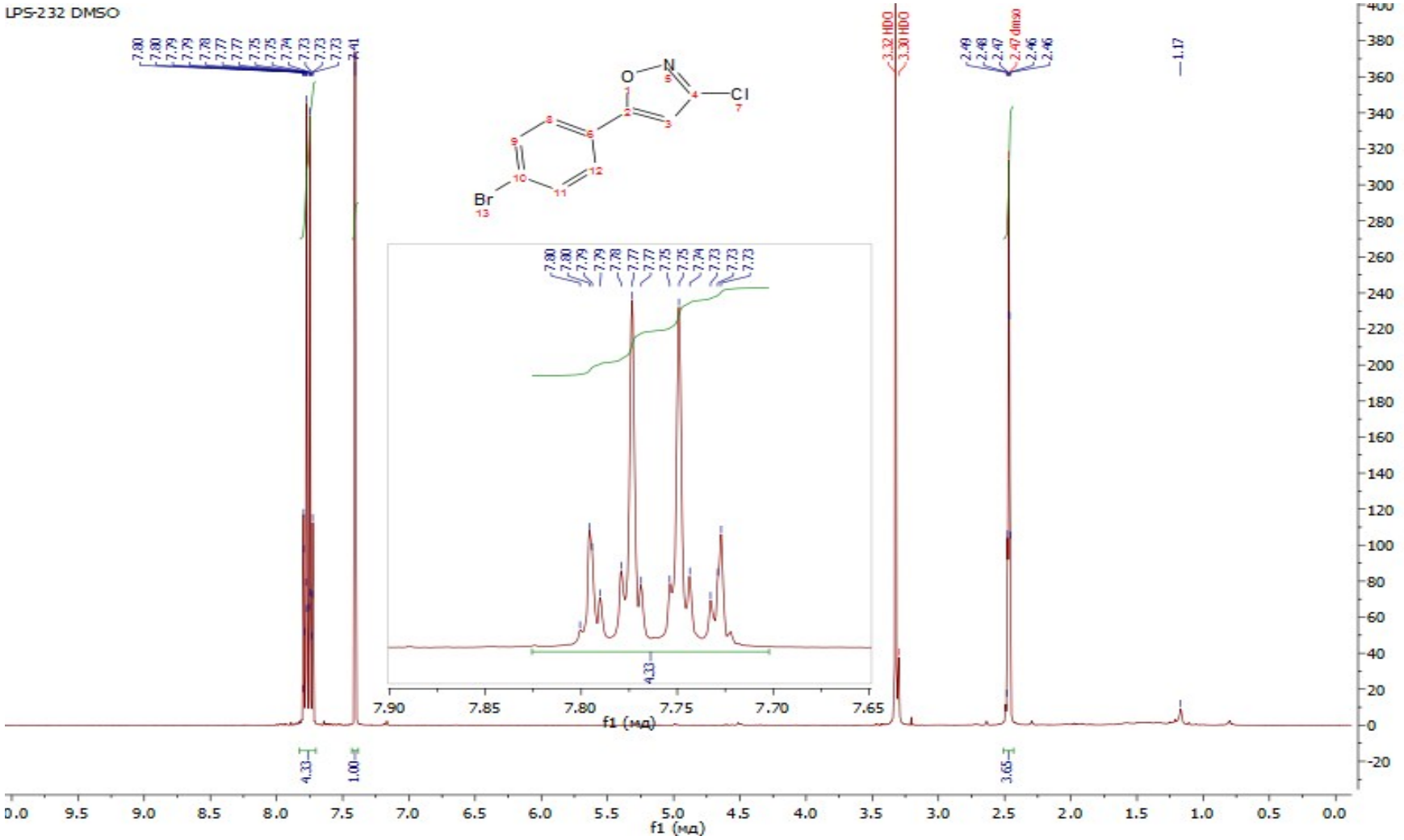


^{19}F NMR (CD_3CN), T = 0°C

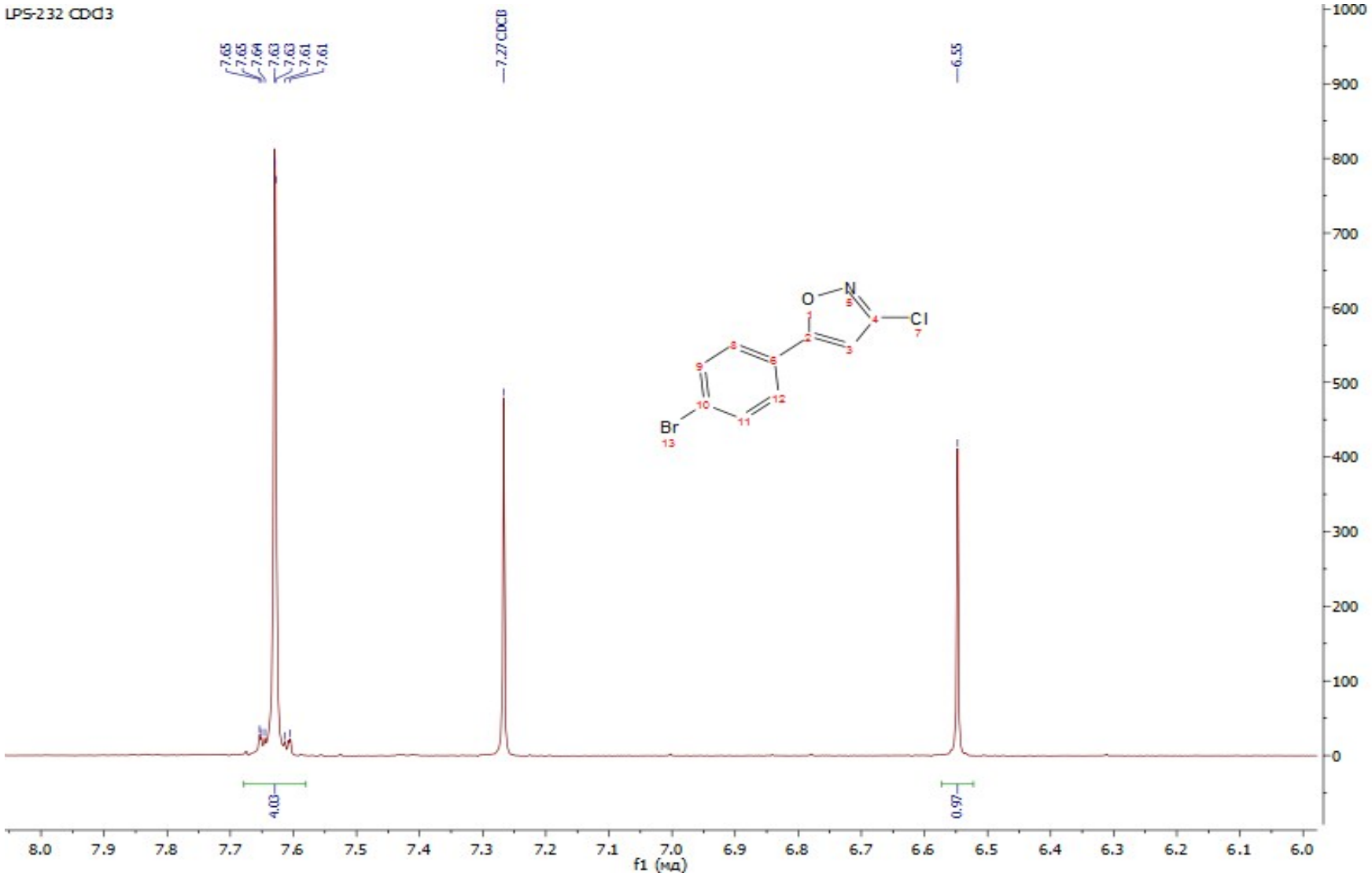


5-(4-bromophenyl)-3-chloroisoxazole **4a**

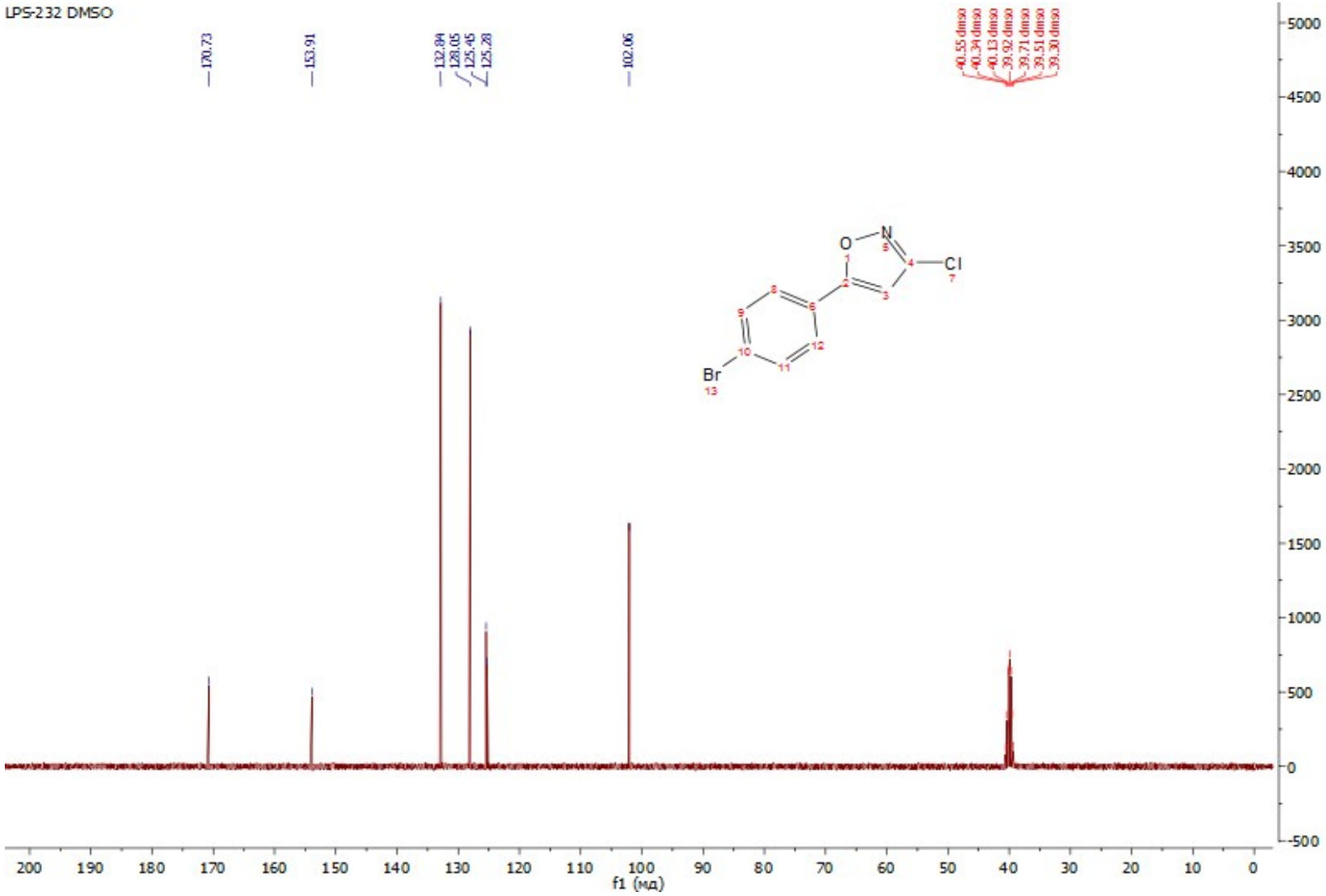
^{1}H NMR (DMSO-d_6)



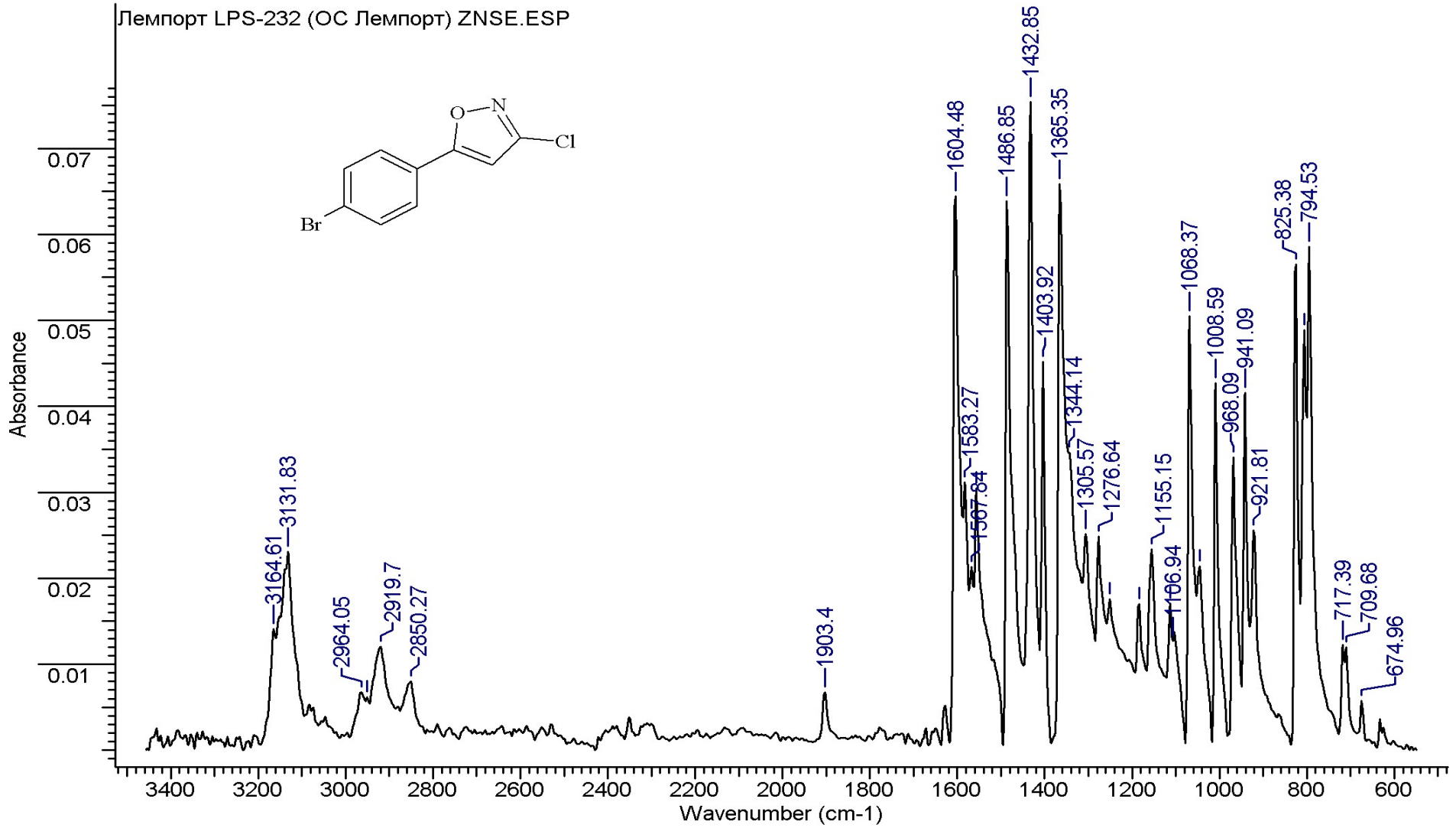
^{1}H ($CDCl_3$): region from 8.0 to 6.0 ppm



^{13}C NMR (DMSO-d_6)

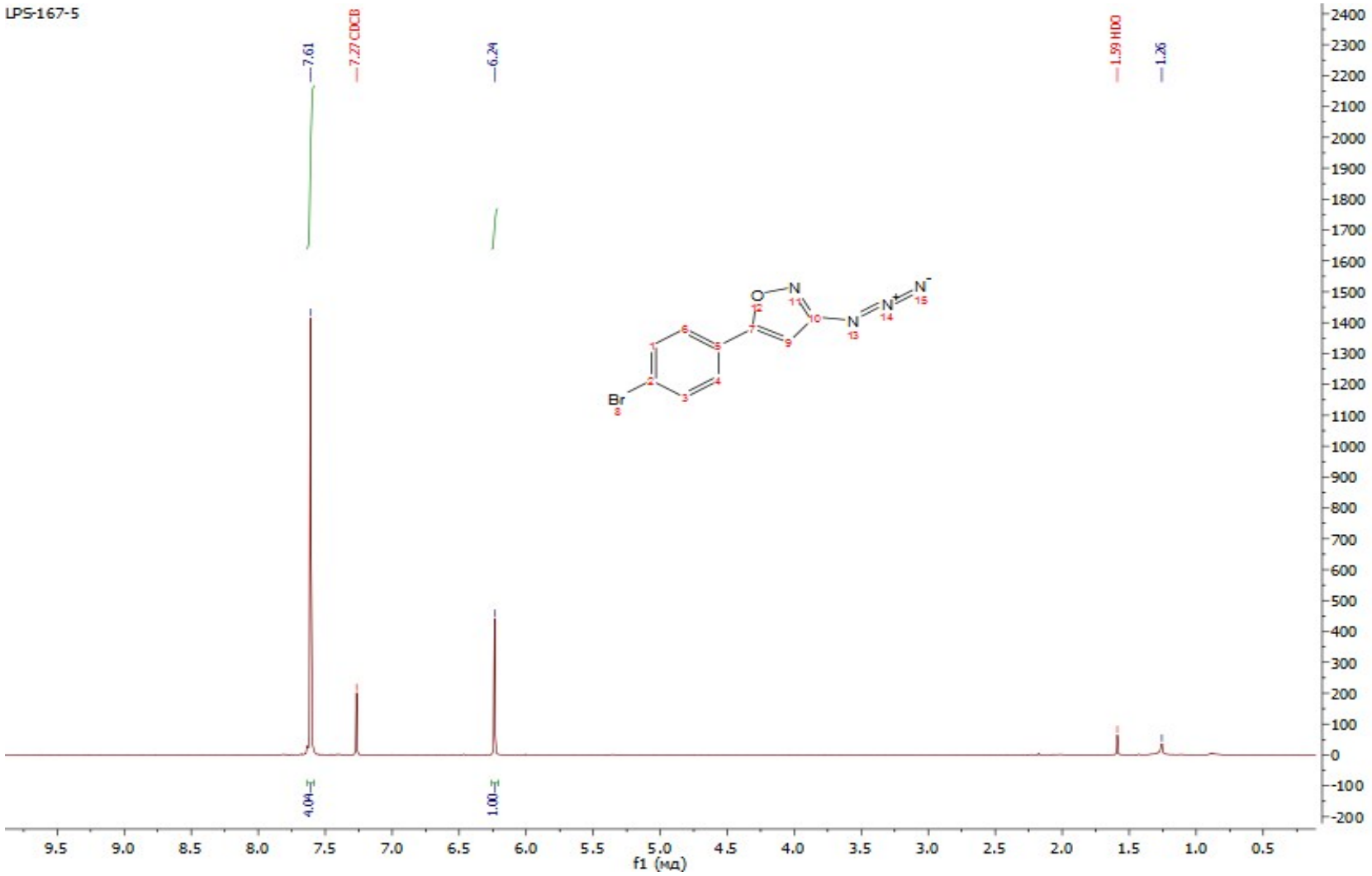


IR

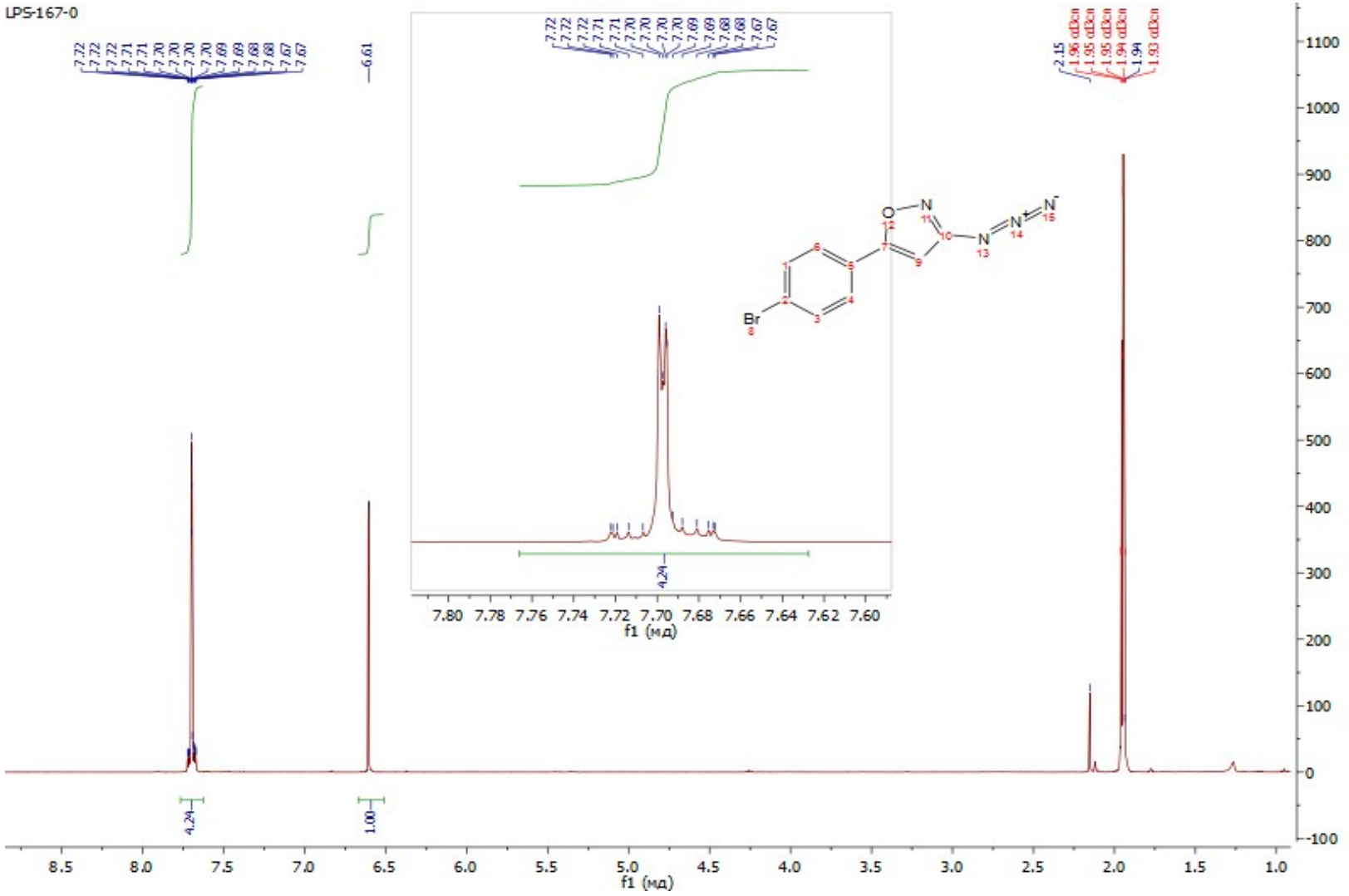

Лемпорт LPS-232 (ОС Лемпорт) ZNSE.ESP
Absorbance
0.07
0.06
0.05
0.04
0.03
0.02
0.01
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
Wavenumber (cm-1)
3164.61
3131.83
2964.05
2919.7
2850.27
1903.4
1604.48
1583.27
1507.84
1486.85
1432.85
1403.92
1365.35
1344.14
1305.57
1276.64
1155.15
1106.94
1068.37
1008.59
968.09
941.09
921.81
825.38
794.53
717.39
709.68
674.96
O
N
Cl
Br

3-azido-5-(4-bromophenyl)isoxazole **5a**

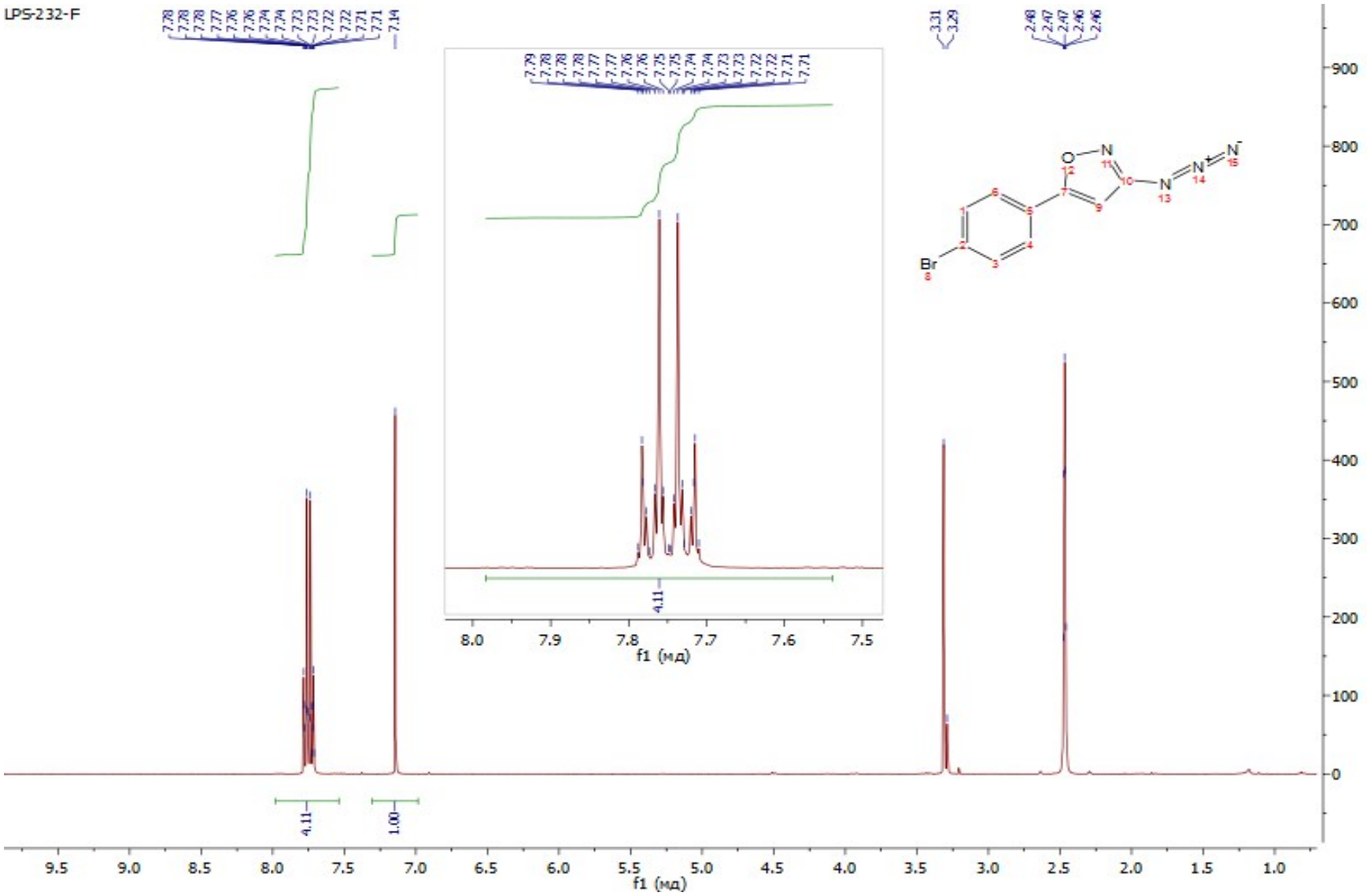
^{1}H NMR ($CDCl_3$)



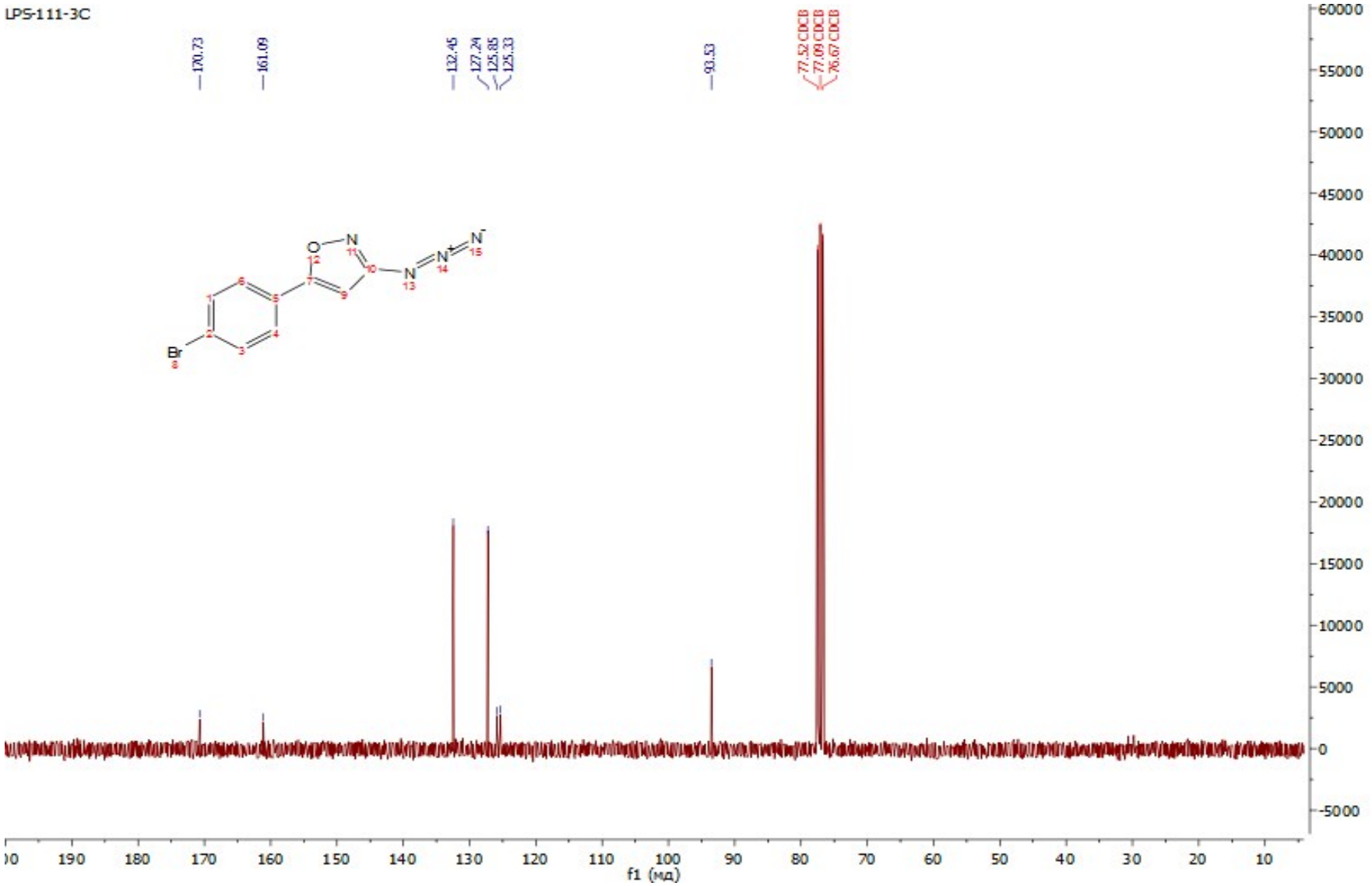
^{1}H NMR (CD_3CN)


LPS-167-0
7.72
7.72
7.72
7.71
7.71
7.70
7.70
7.70
7.70
7.69
7.69
7.68
7.68
7.67
7.67
6.61
2.15
1.96 cd3cn
1.95 cd3cn
1.95 cd3cn
1.94 cd3cn
1.94
1.93 cd3cn
4.24
1.00
f1 (мд)
Br
O
N
N
N
N

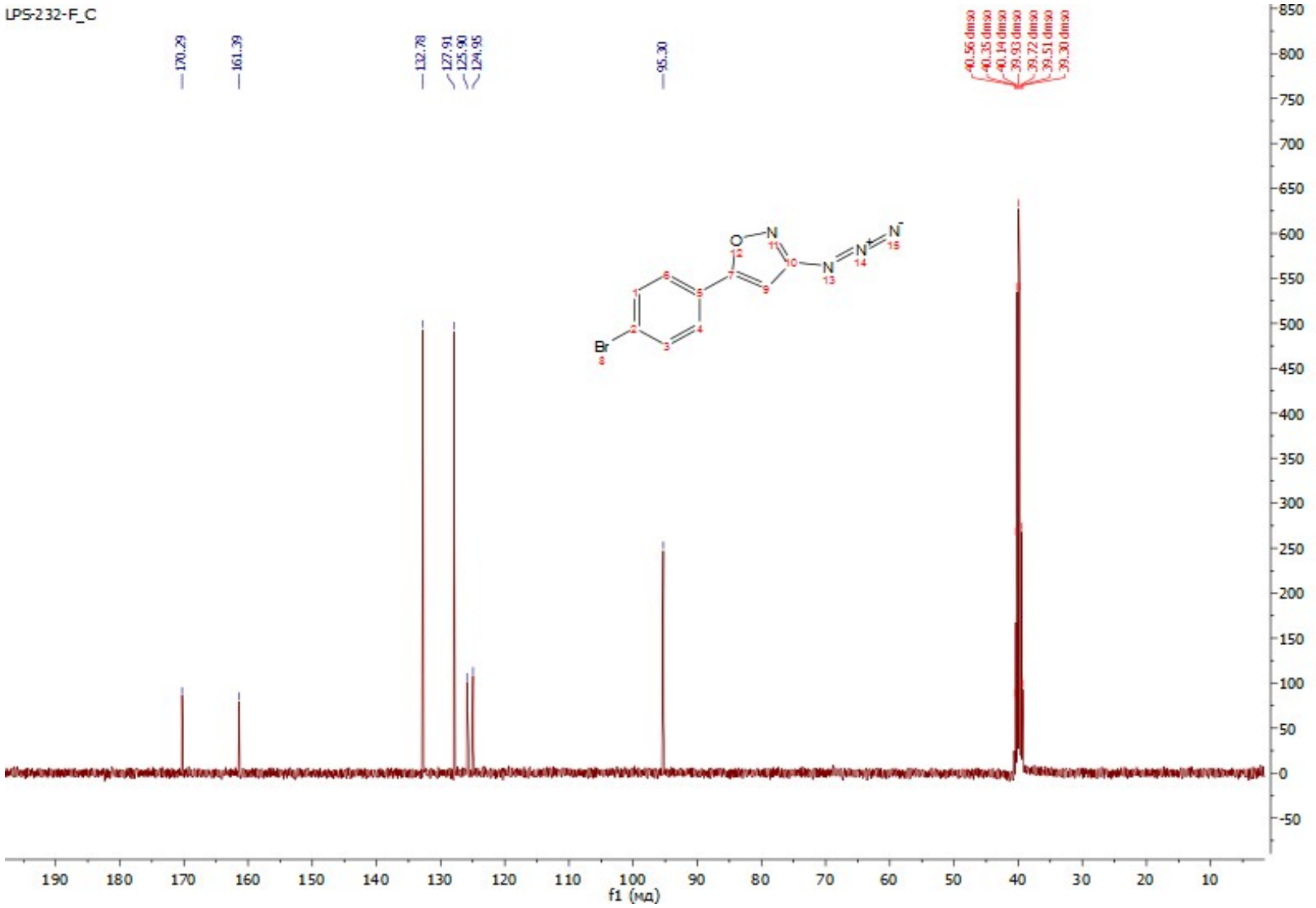
^{1}H NMR (DMSO-d_6)



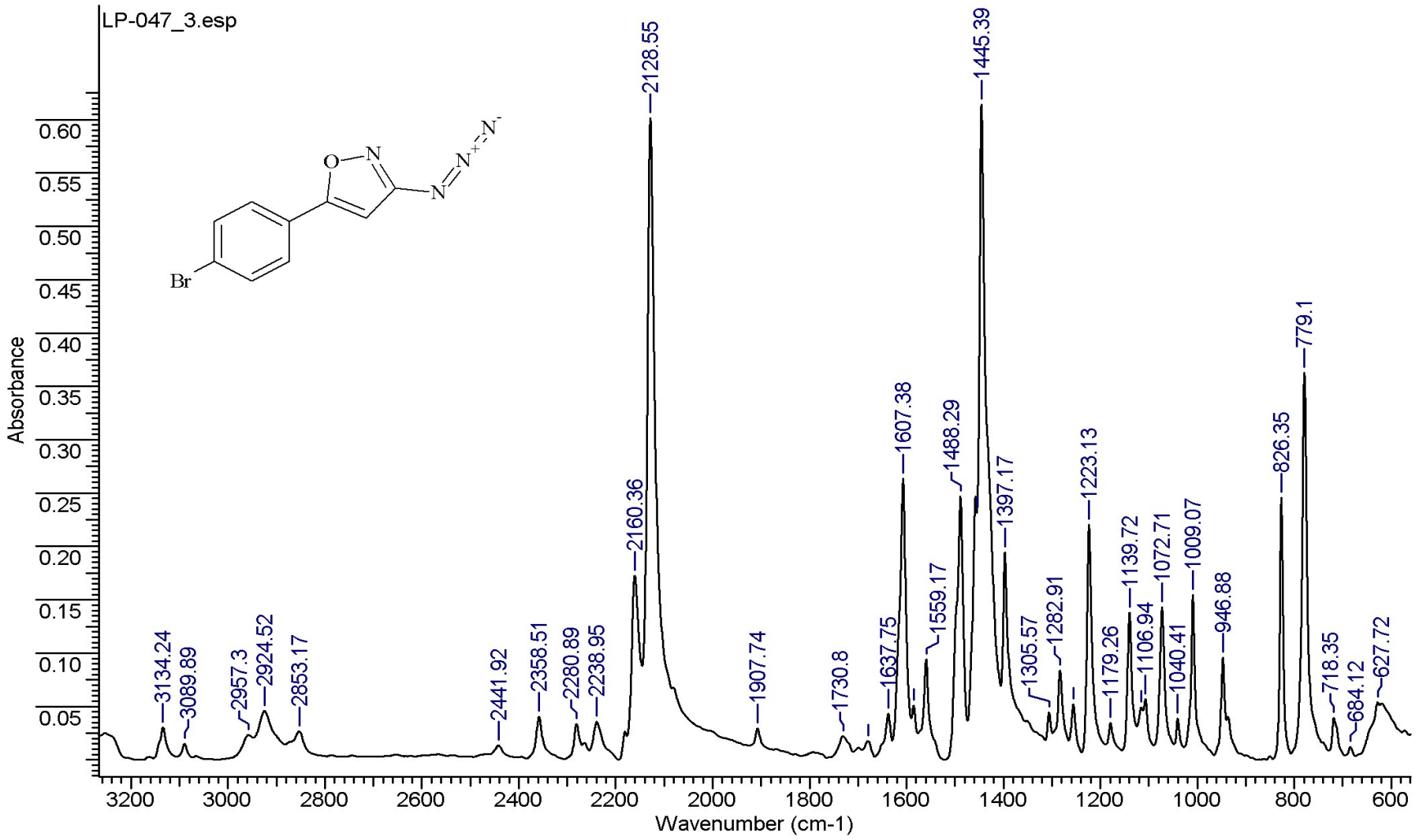
^{13}C NMR ($CDCl_3$)



^{13}C NMR (DMSO-d_6)

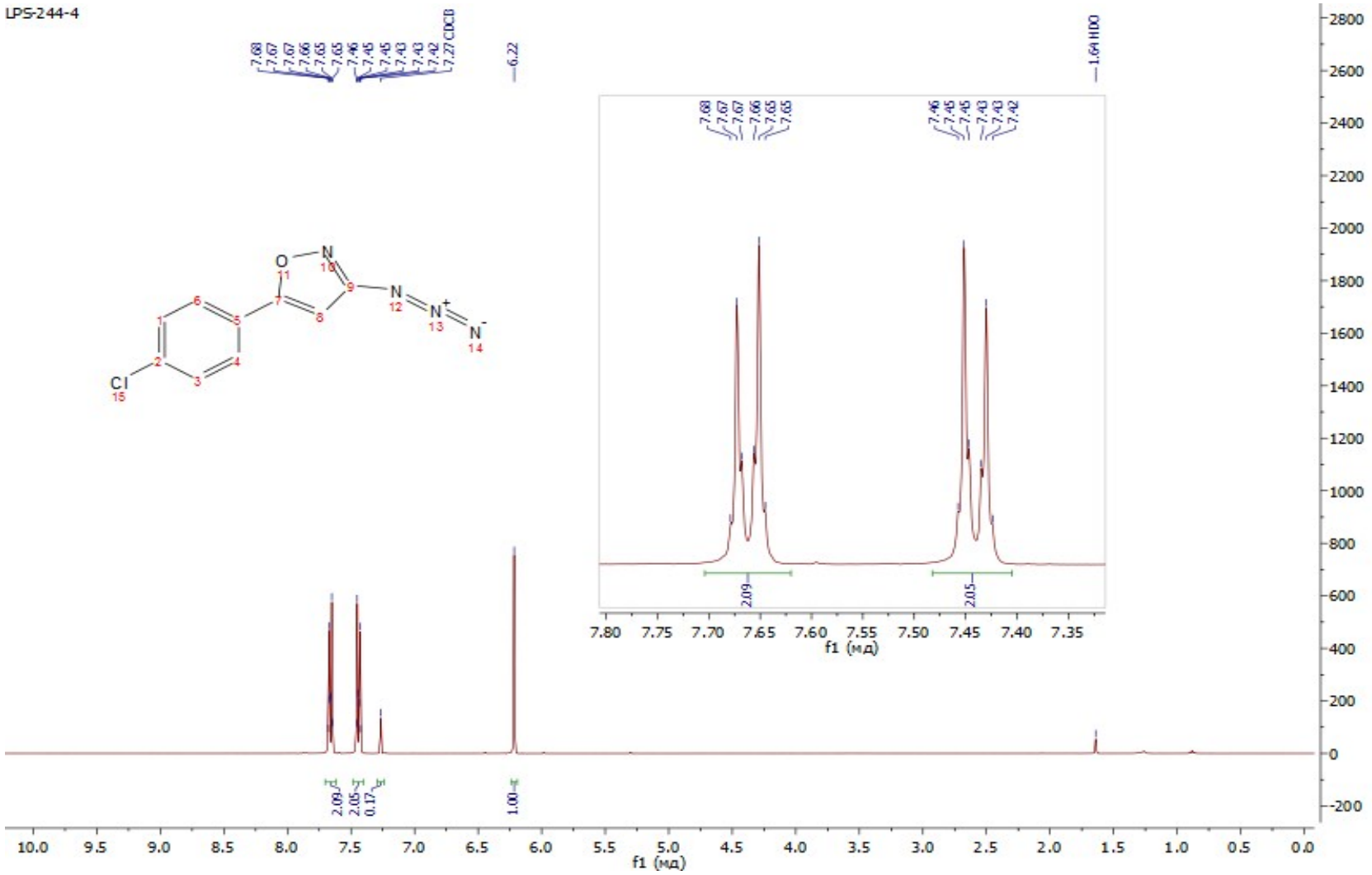


IR

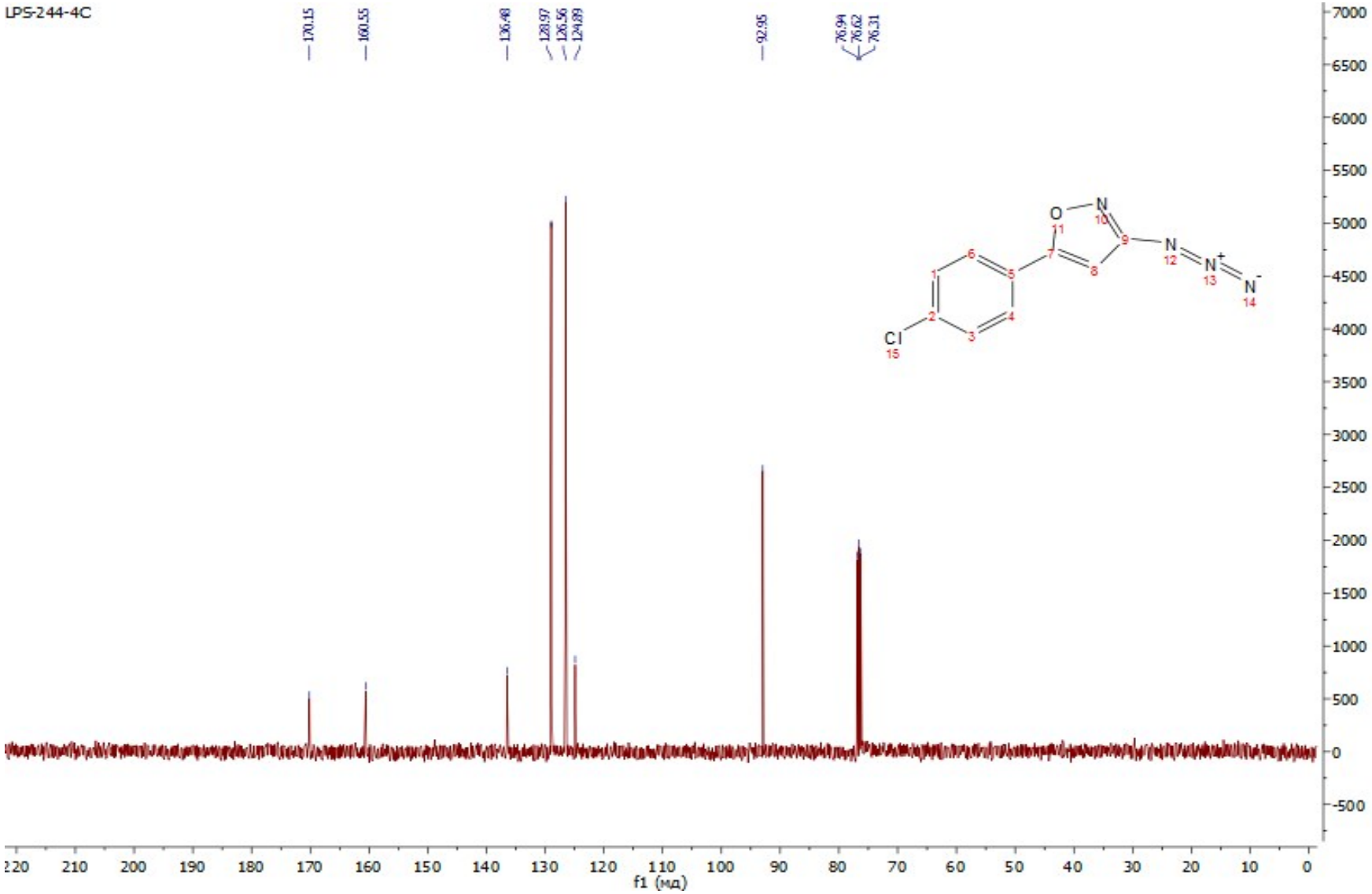

LP-047_3.esp
Absorbance
Wavenumber (cm-1)
3134.24
3089.89
2957.3
2924.52
2853.17
2441.92
2358.51
2280.89
2238.95
2160.36
2128.55
1907.74
1730.8
1637.75
1607.38
1559.17
1488.29
1445.39
1397.17
1305.57
1282.91
1223.13
1179.26
1139.72
1106.94
1072.71
1040.41
1009.07
946.88
826.35
779.1
718.35
684.12
627.72
Br

3-azido-5-(4-chlorophenyl)isoxazole **5b**

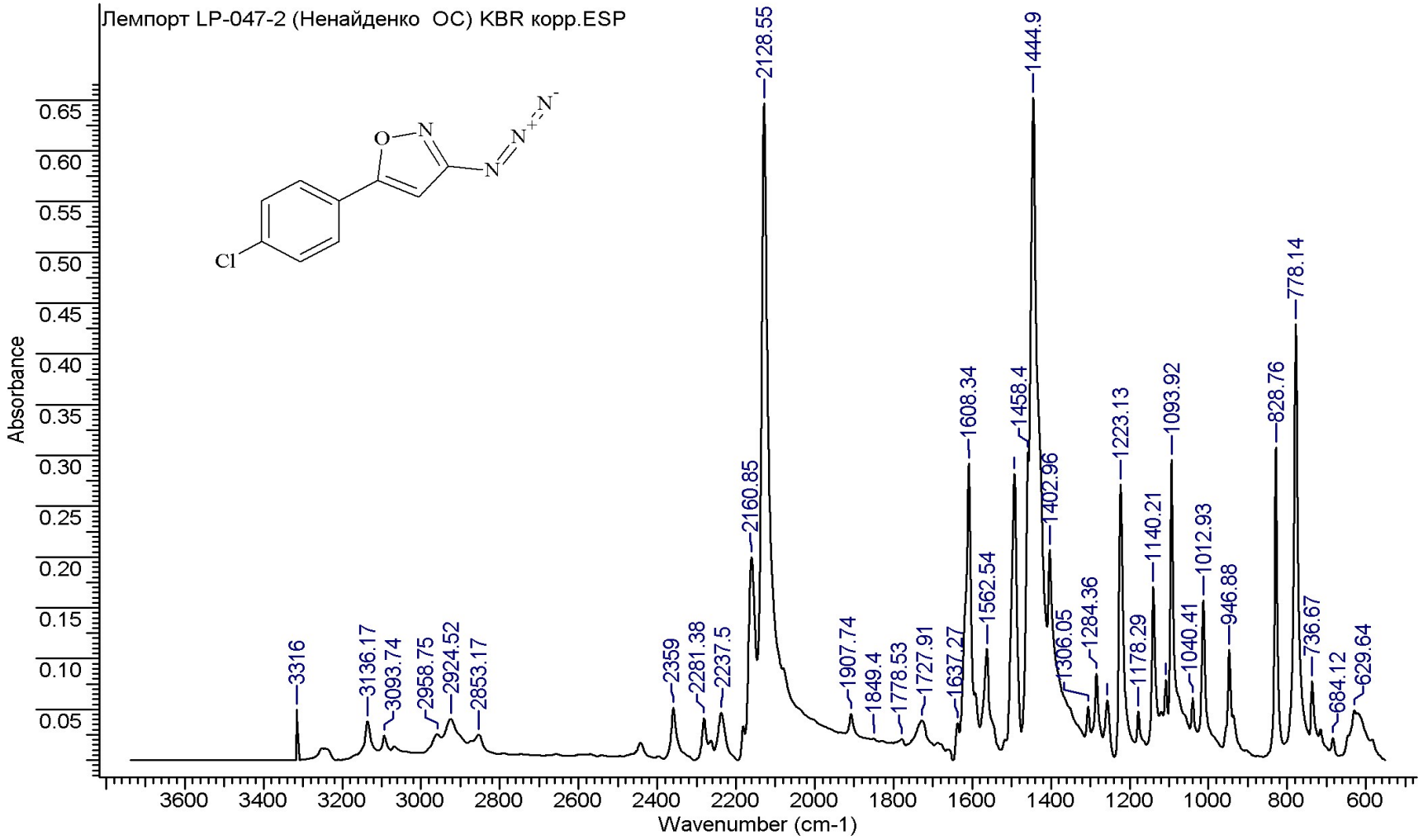
^{1}H NMR ($CDCl_3$)



^{13}C NMR ($CDCl_3$)

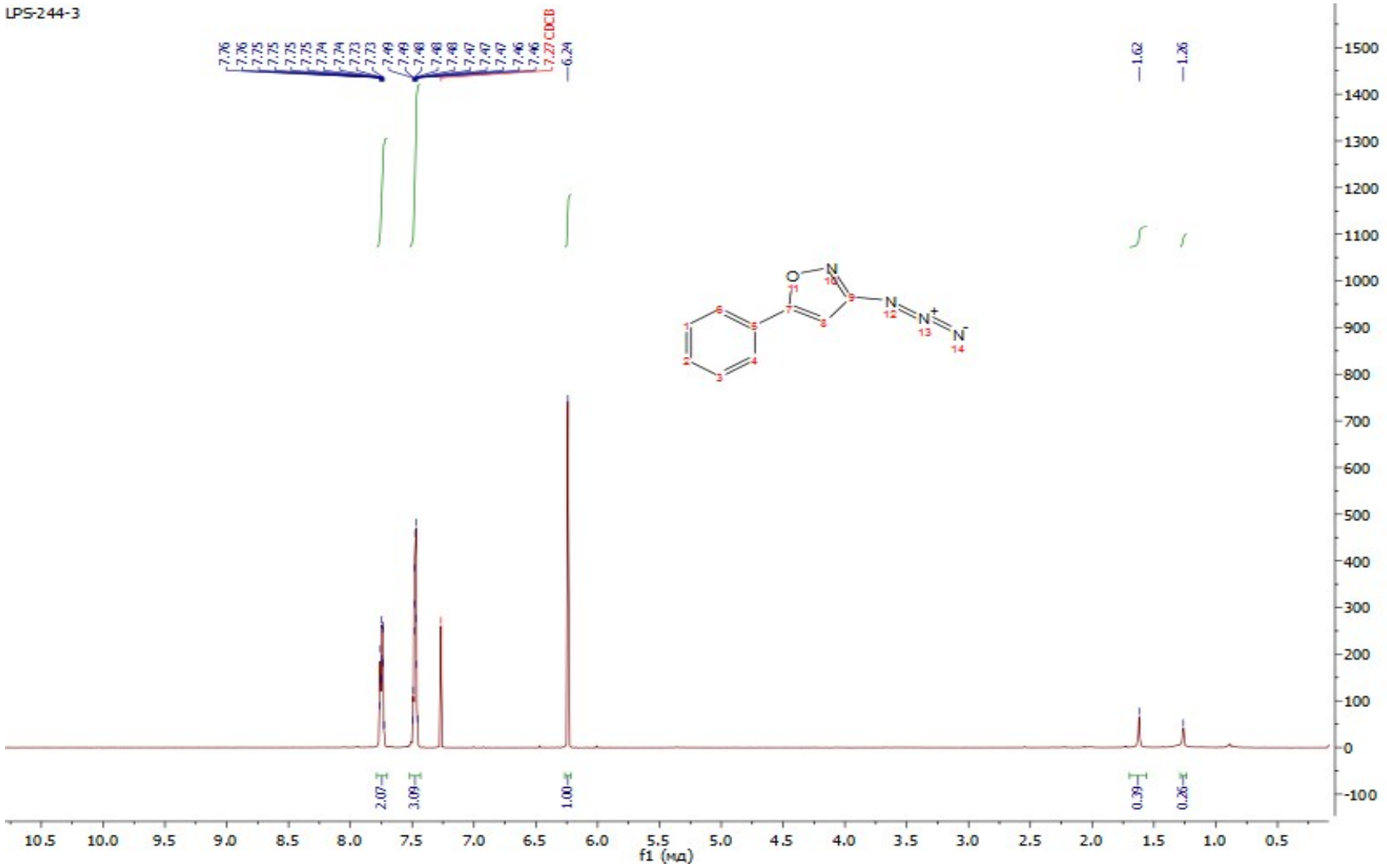


IR

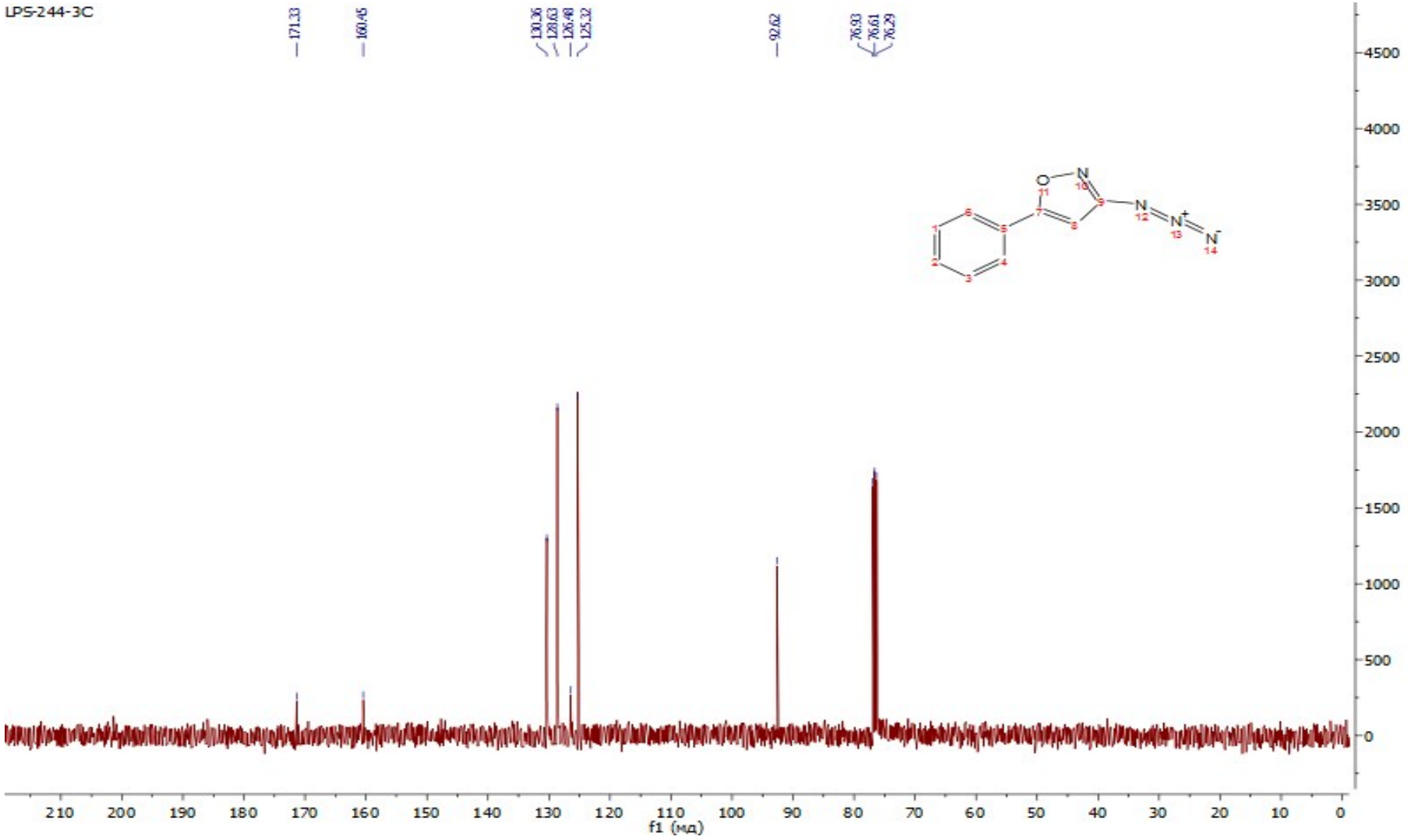

Лемпорт LP-047-2 (Ненайденко ОС) KBR корр.ESP
Absorbance
Wavenumber (cm-1)
3316
3136.17
3093.74
2958.75
2924.52
2853.17
2359
2281.38
2237.5
2160.85
2128.55
1907.74
1849.4
1778.53
1727.91
1637.27
1608.34
1562.54
1458.4
1444.9
1402.96
1306.05
1284.36
1223.13
1178.29
1140.21
1093.92
1040.41
1012.93
946.88
828.76
778.14
736.67
684.12
629.64
Cl

3-azido-5-phenylisoxazole **5c**

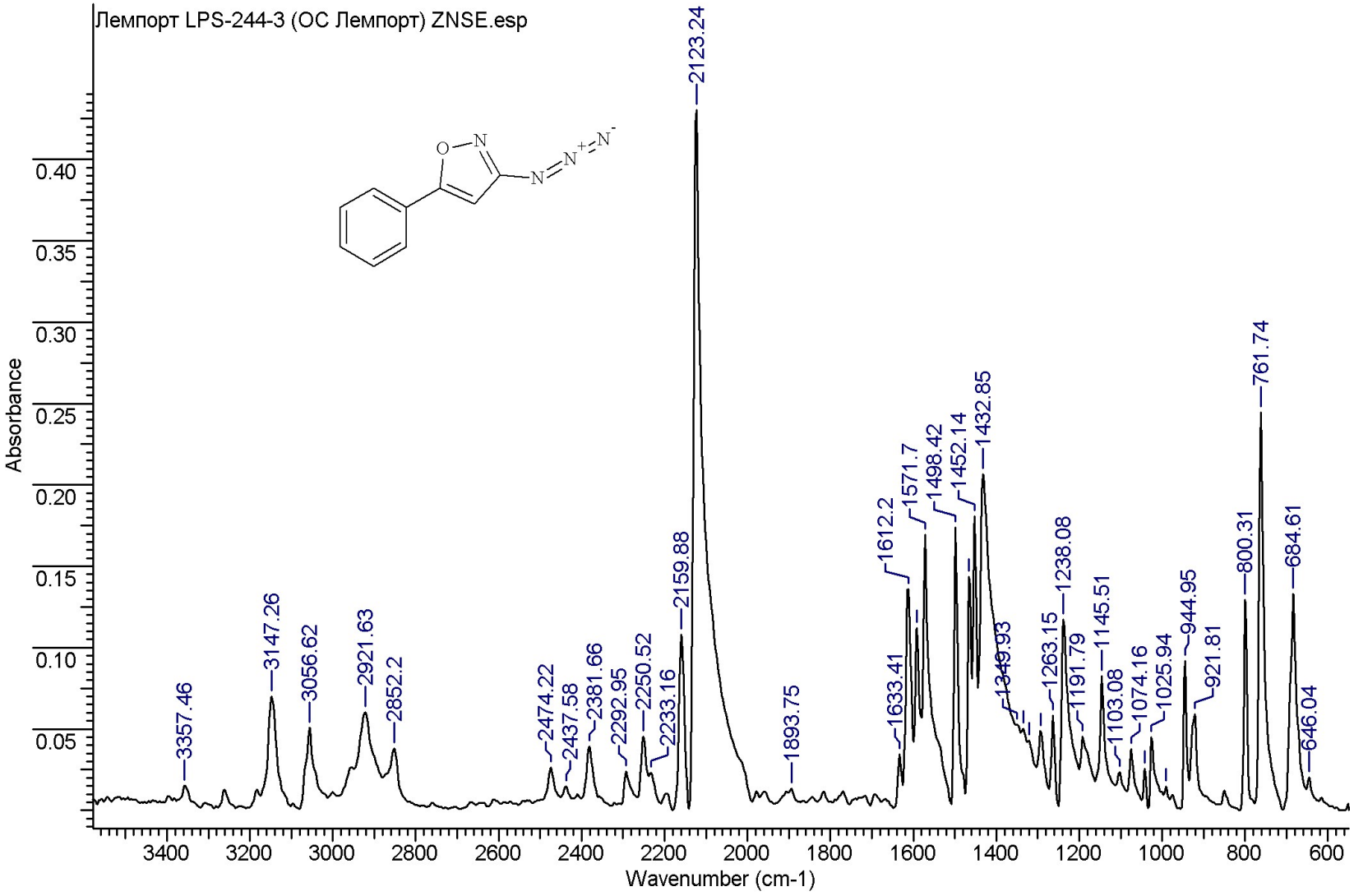
^{1}H NMR ($CDCl_3$)



^{13}C NMR ($CDCl_3$)

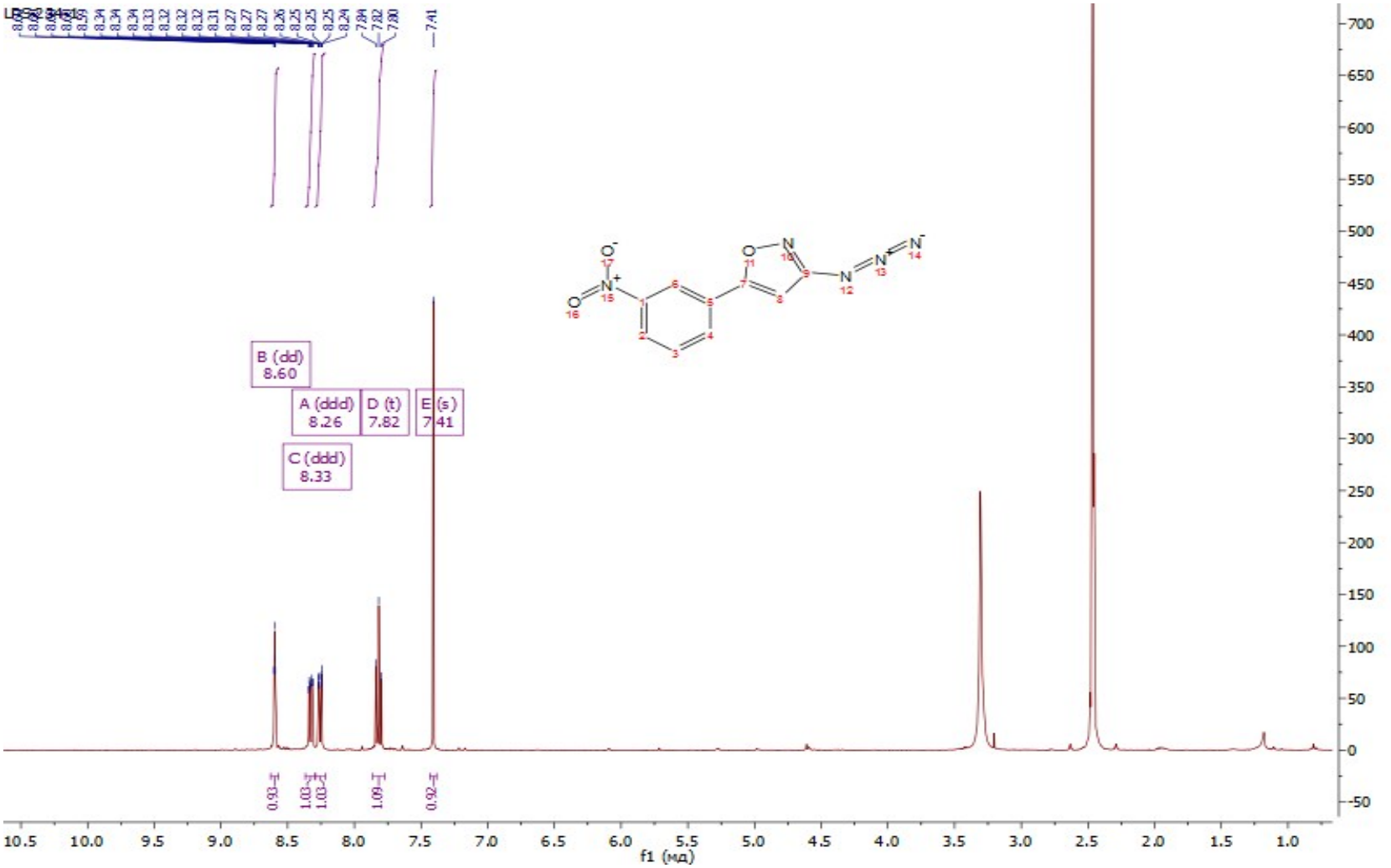


IR

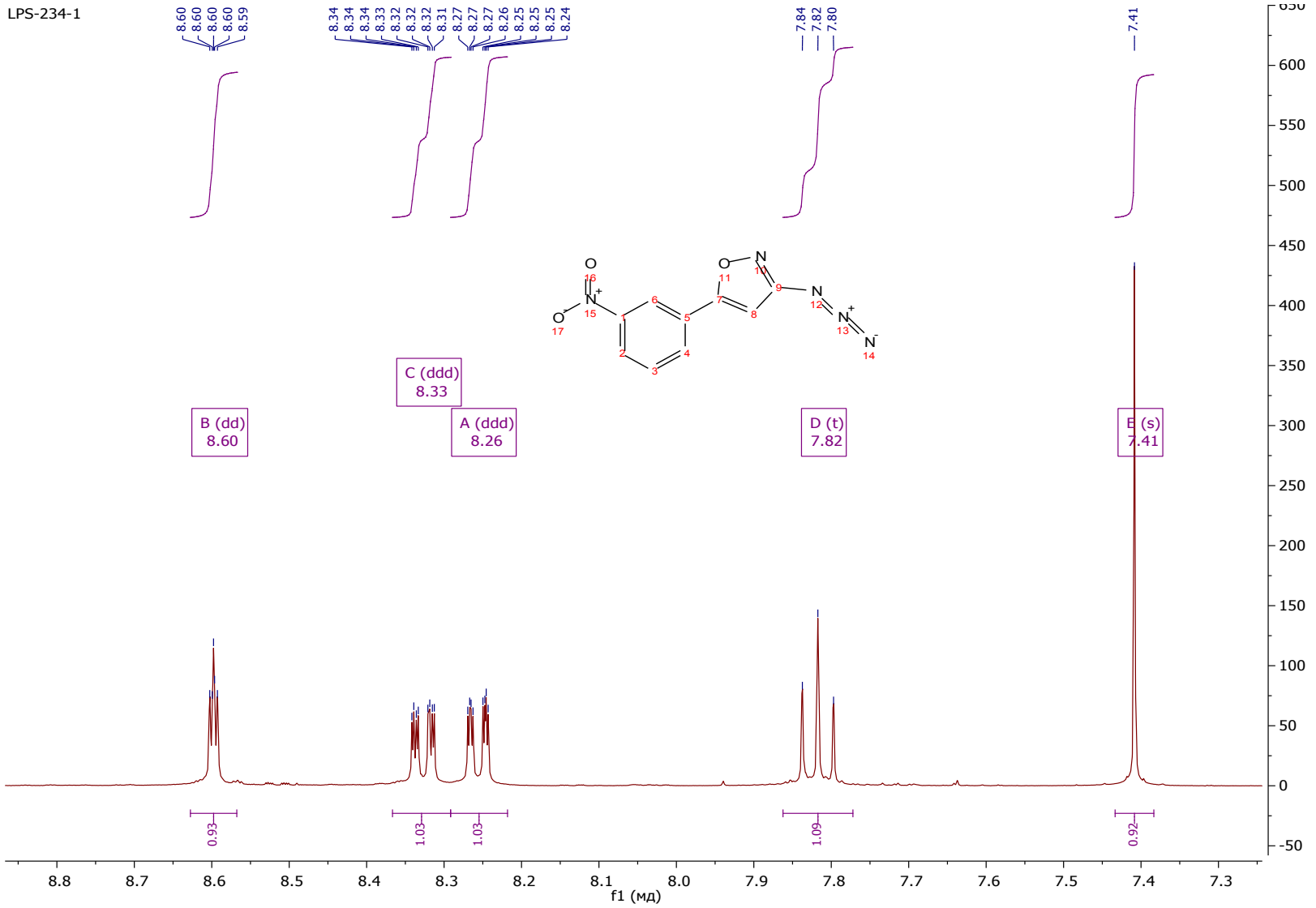

Лемпорт LPS-244-3 (ОС Лемпорт) ZNSE.esp
Absorbance
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
Wavenumber (cm-1)
3357.46
3147.26
3056.62
2921.63
2852.2
2474.22
2437.58
2381.66
2292.95
2250.52
2233.16
2159.88
2123.24
1893.75
1633.41
1612.2
1571.7
1498.42
1452.14
1432.85
1349.93
1263.15
1238.08
1191.79
1145.51
1103.08
1074.16
1025.94
944.95
921.81
800.31
761.74
684.61
646.04

3-azido-5-(3-nitrophenyl)isoxazole **5d**

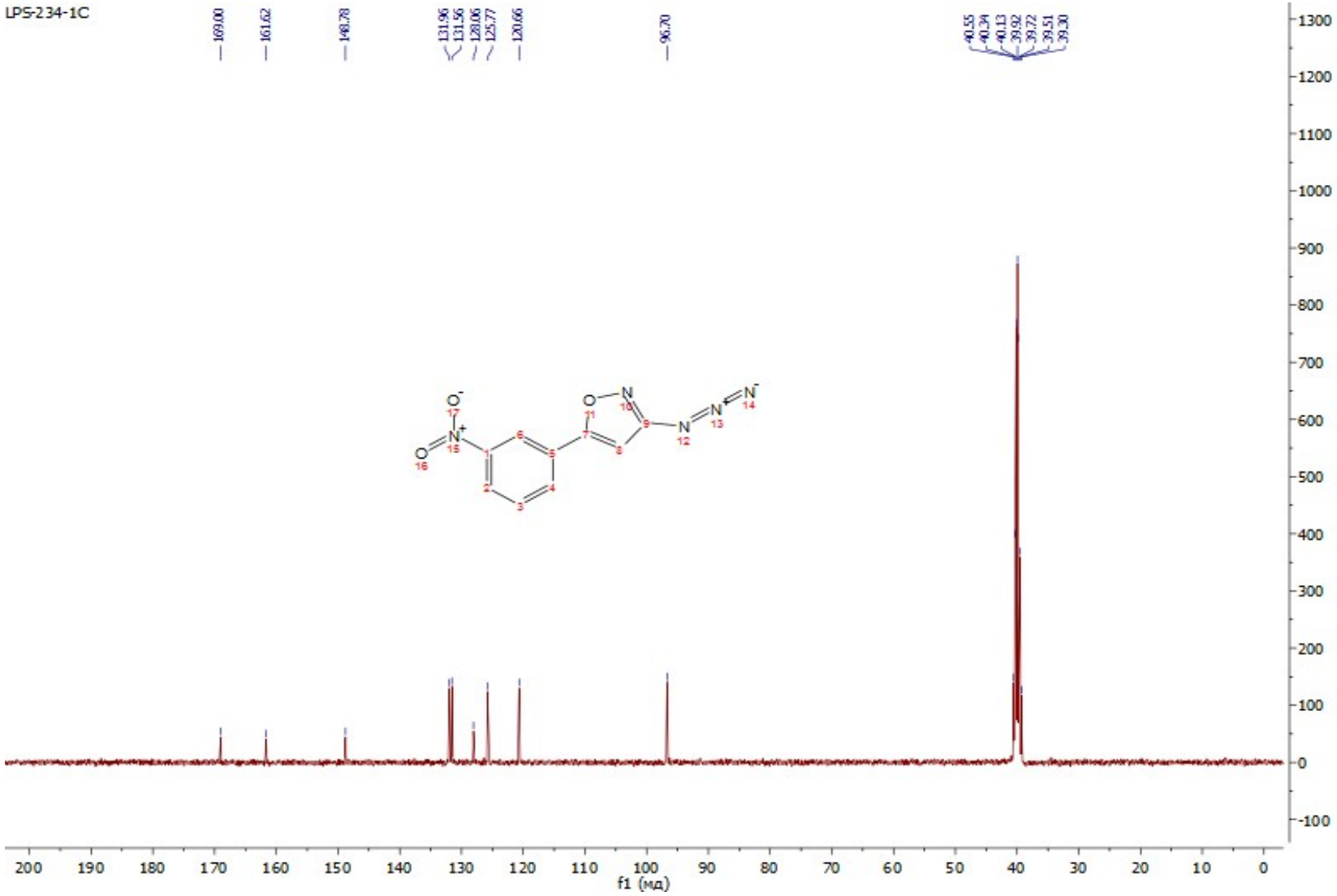
^{1}H NMR (DMSO-d_6)



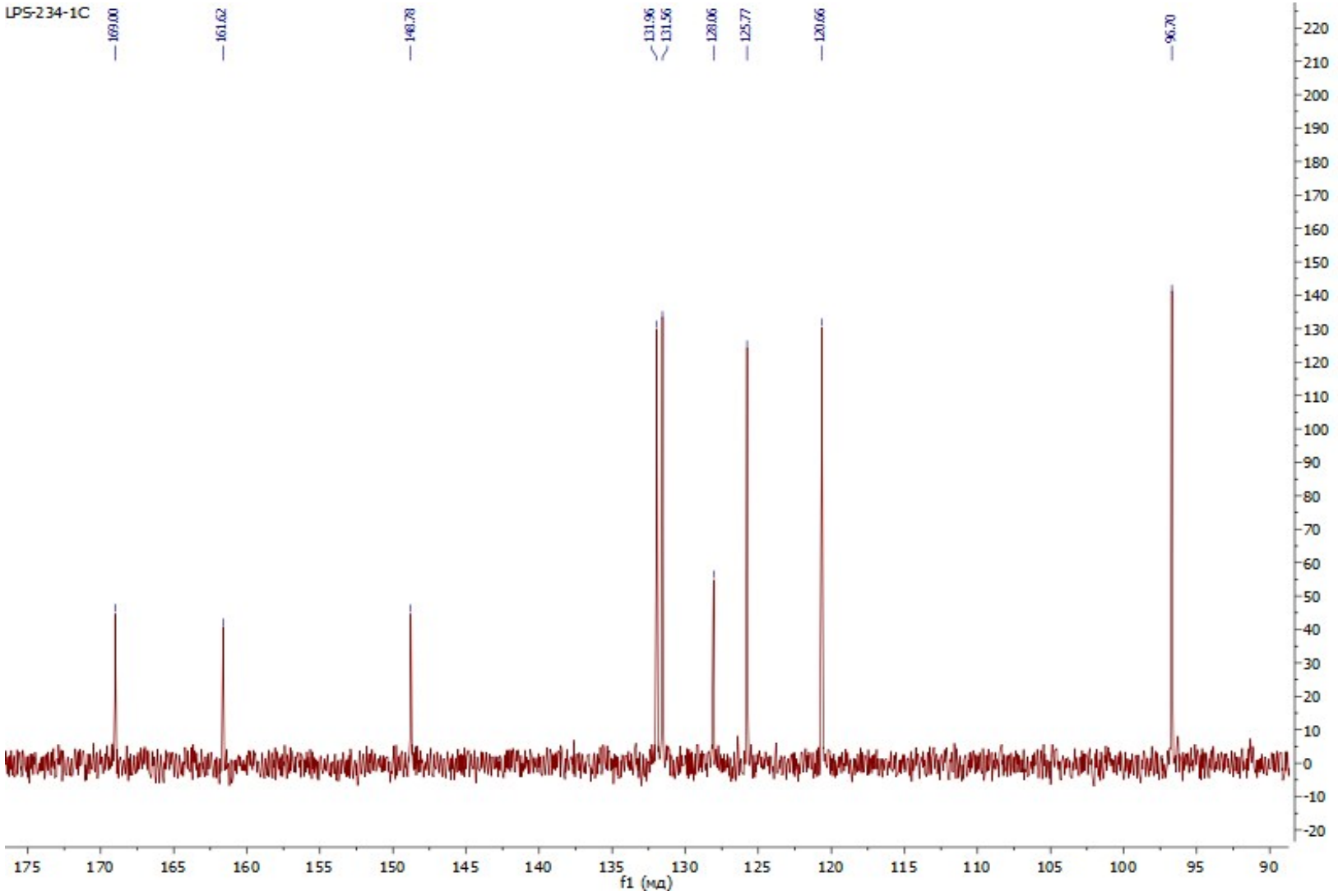
^{1}H NMR (DMSO-d_6): region from 8.8 to 7.3 ppm



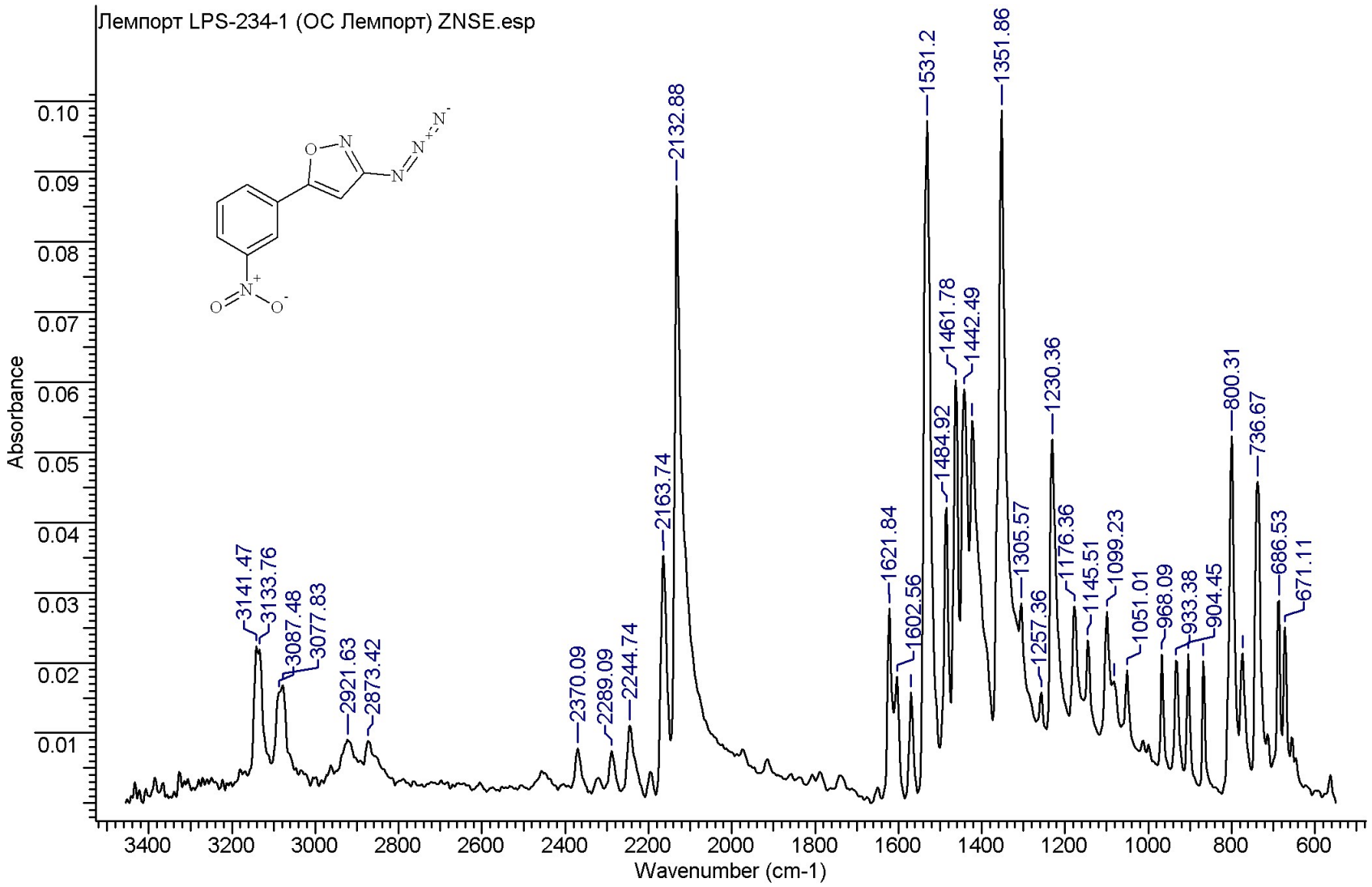
^{13}C NMR (DMSO-d_6)



^{13}C NMR (DMSO-d_6): region from 175 to 90 ppm

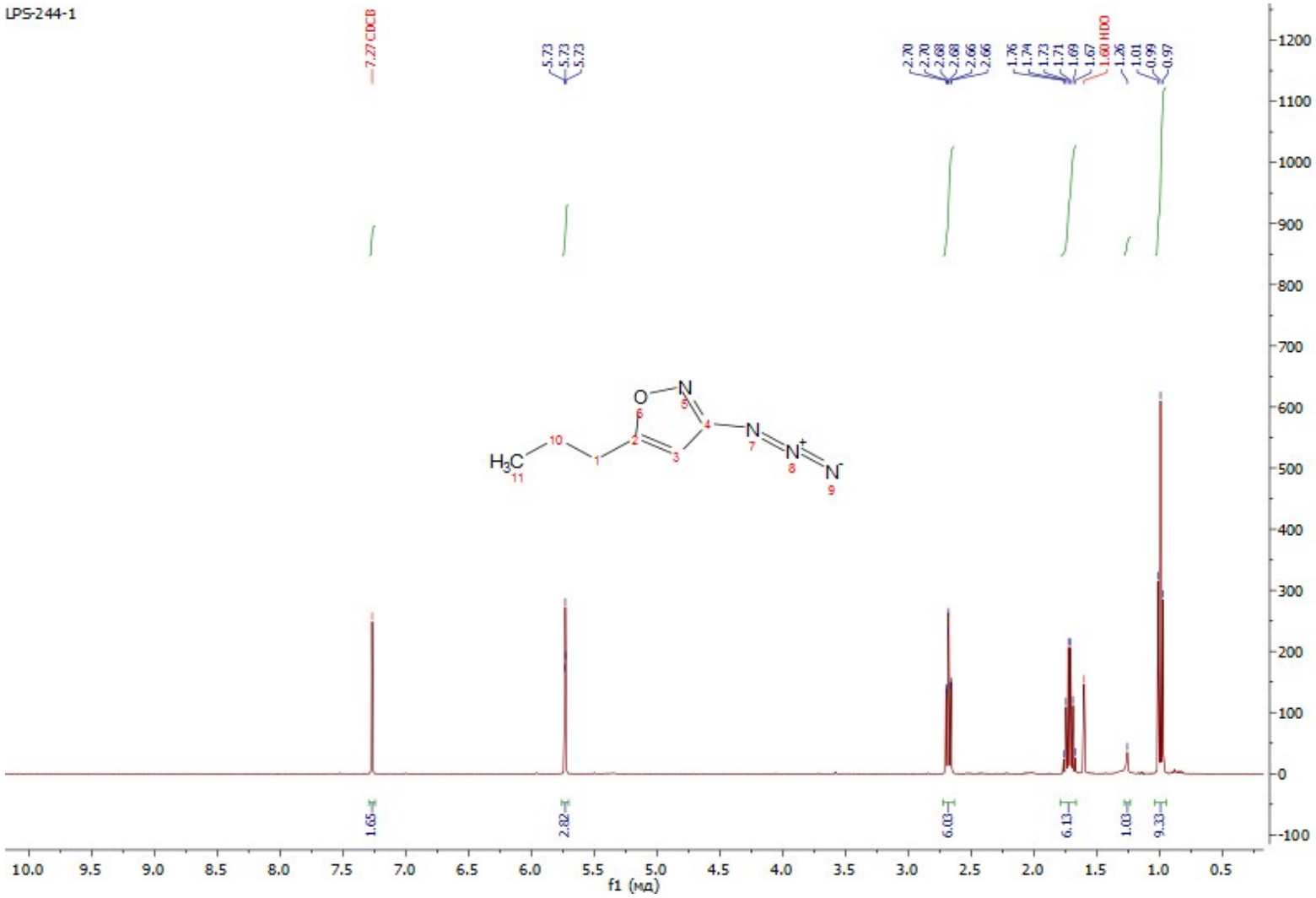


IR

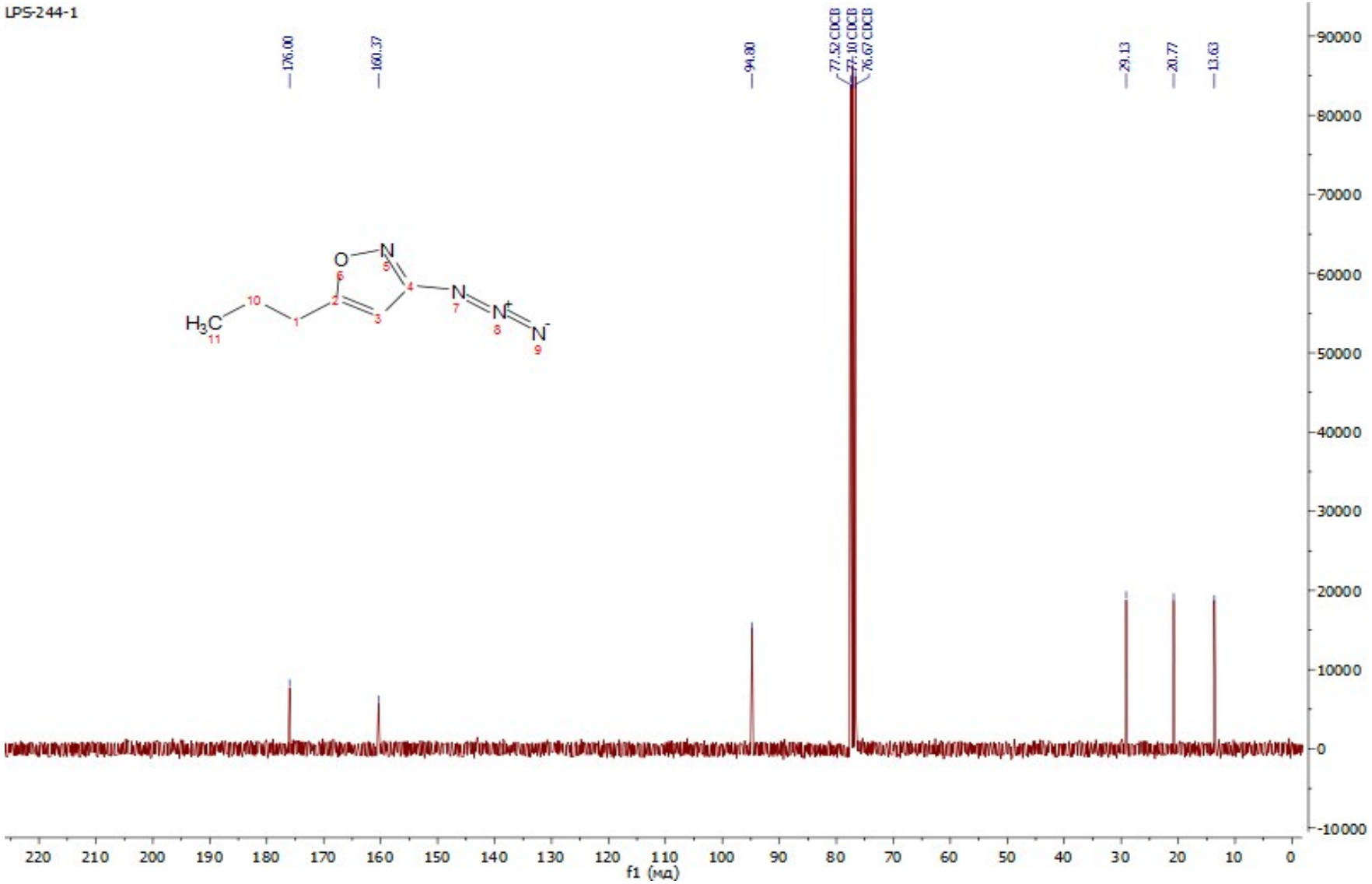

Лемпорт LPS-234-1 (ОС Лемпорт) ZNSE.esp
Absorbance
Wavenumber (cm-1)
3141.47
3133.76
3087.48
3077.83
2921.63
2873.42
2370.09
2289.09
2244.74
2163.74
2132.88
1621.84
1602.56
1531.2
1484.92
1461.78
1442.49
1351.86
1305.57
1257.36
1230.36
1176.36
1145.51
1099.23
1051.01
968.09
933.38
904.45
800.31
736.67
686.53
671.11

3-azido-5-propylisoxazole **5e**

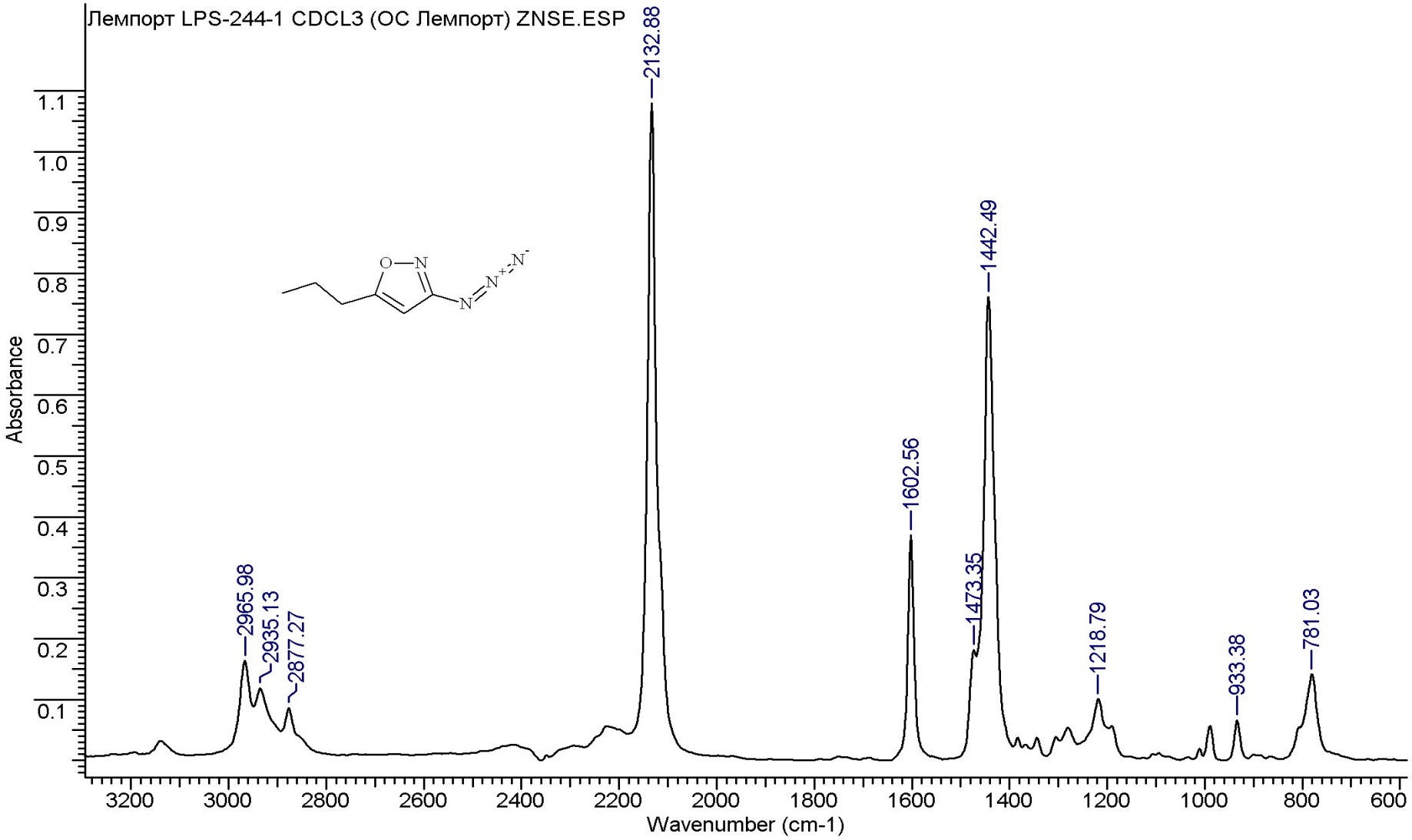
^{1}H NMR ($CDCl_3$)



^{13}C NMR ($CDCl_3$)

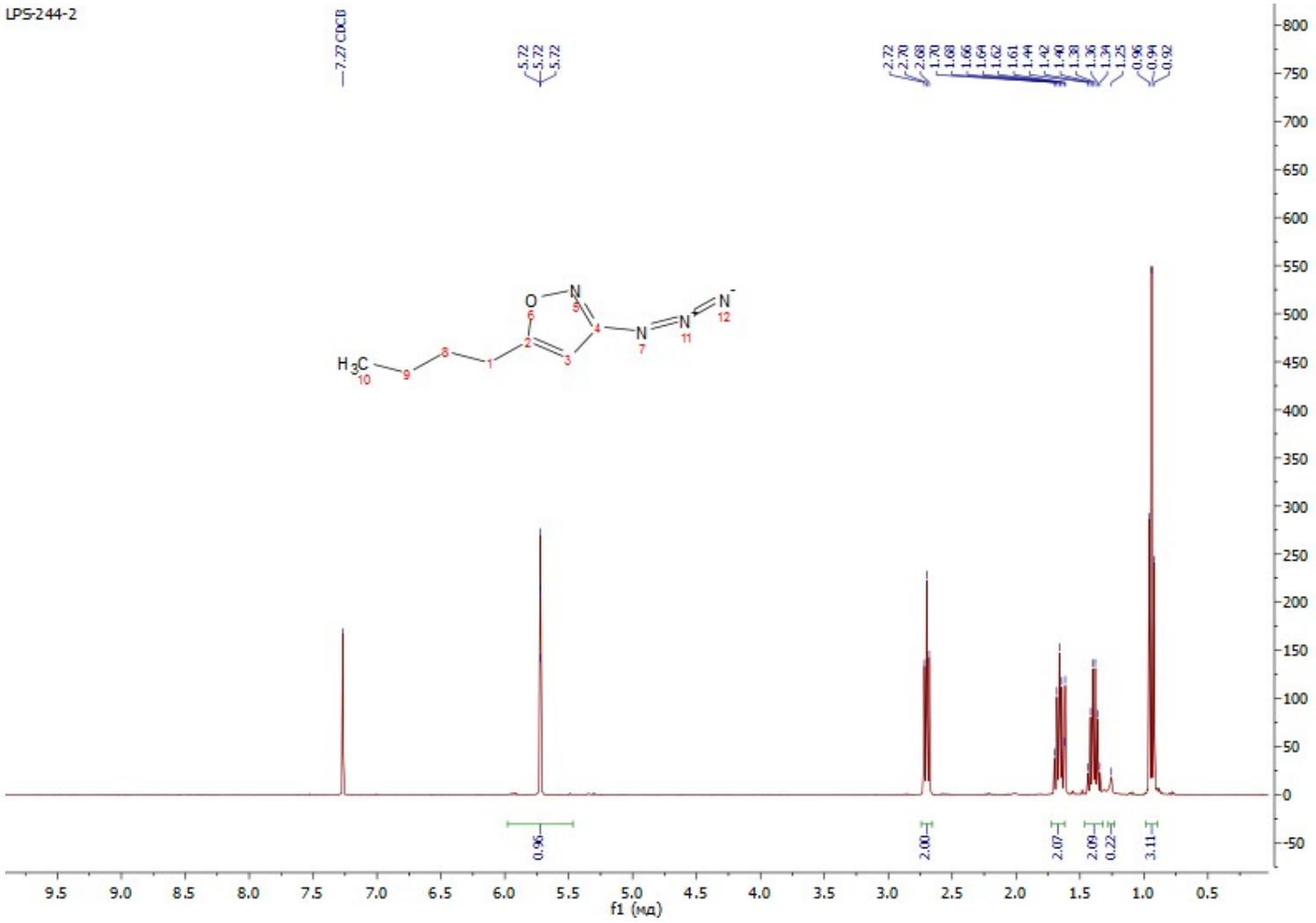


IR

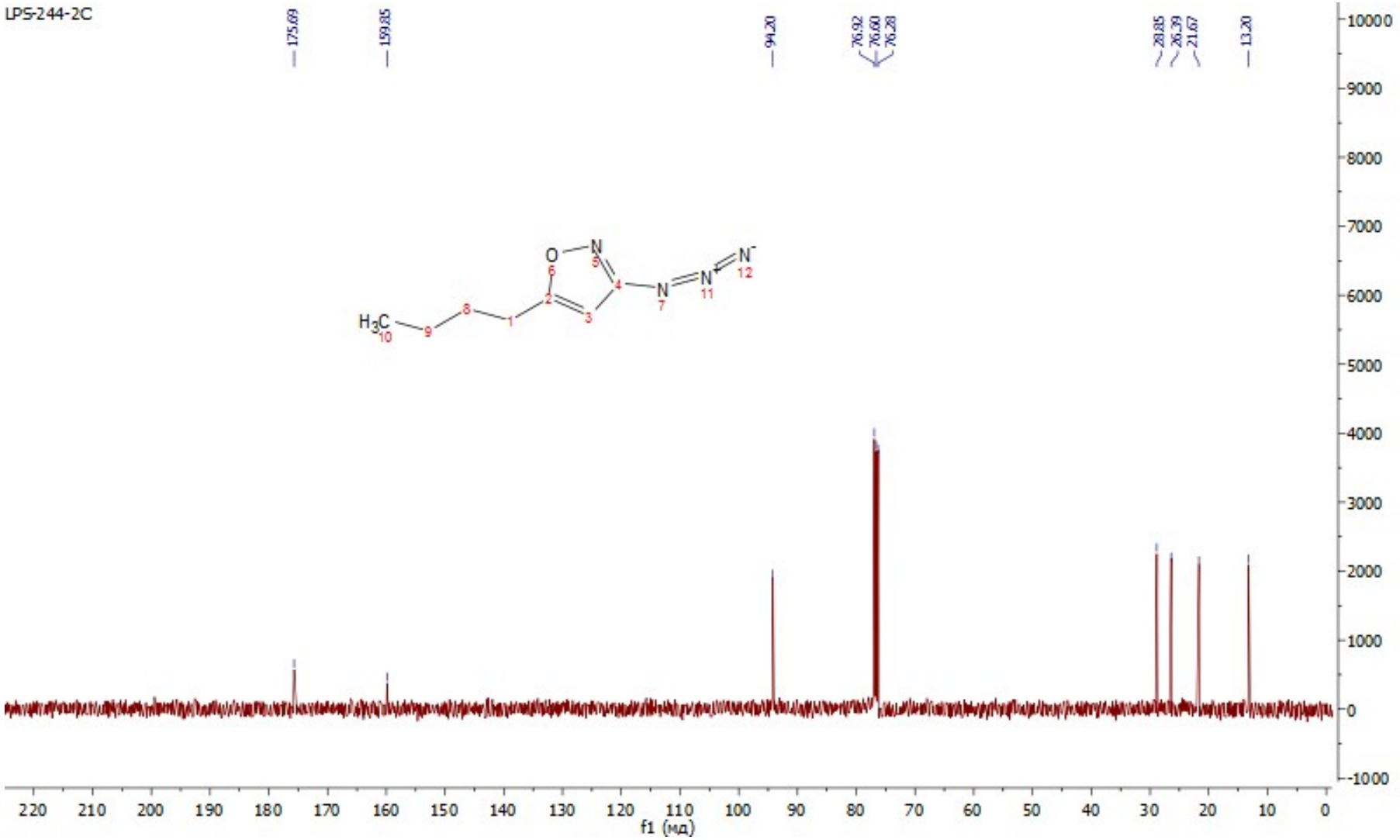

Лемпорт LPS-244-1 CDCL3 (ОС Лемпорт) ZNSE.ESP
2132.88
1442.49
1602.56
1473.35
1218.79
933.38
781.03
2965.98
2935.13
2877.27
Absorbance
Wavenumber (cm-1)

3-azido-5-butylisoxazole **5f**

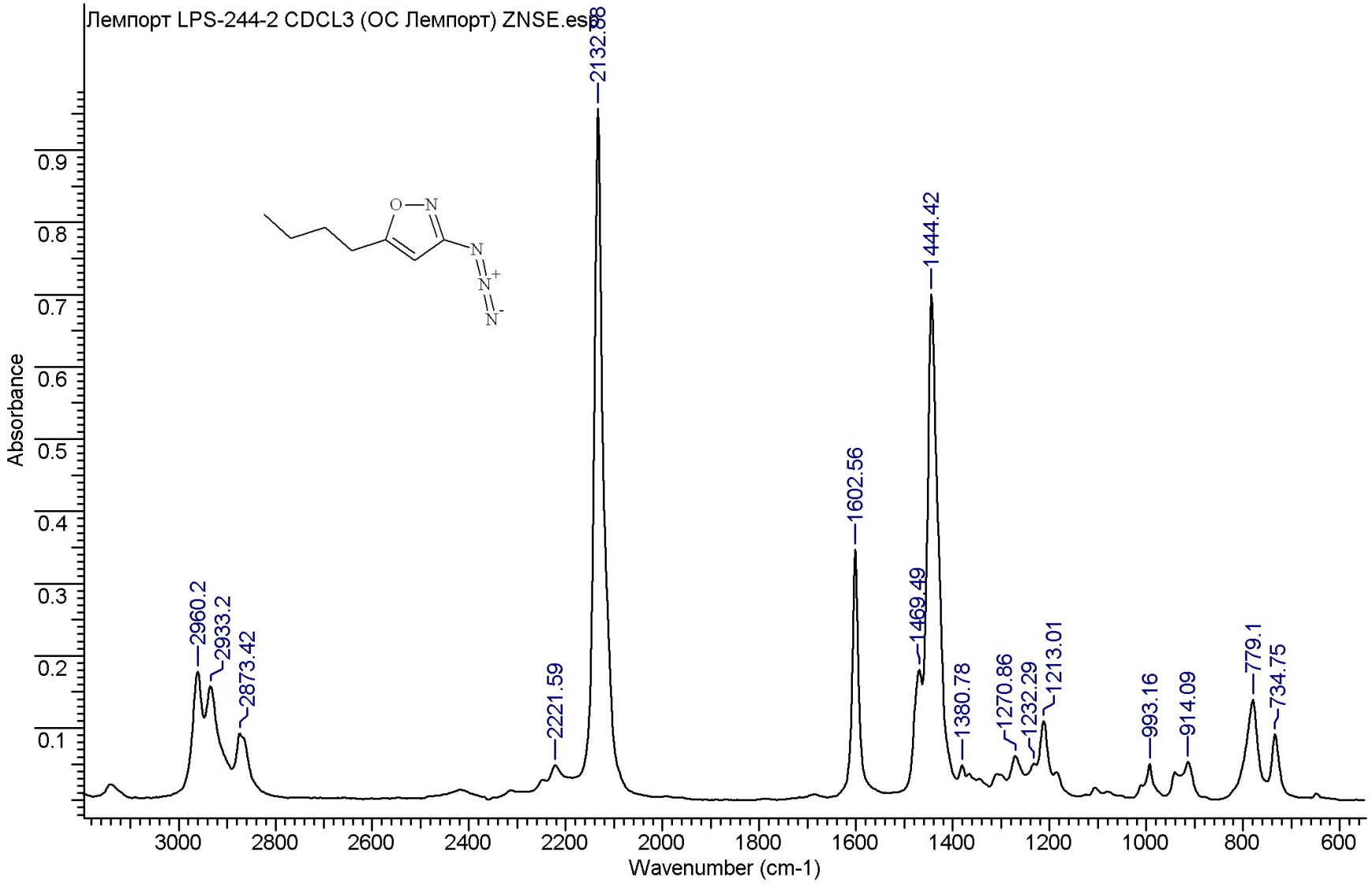
^{1}H NMR ($CDCl_3$)



^{13}C NMR ($CDCl_3$)

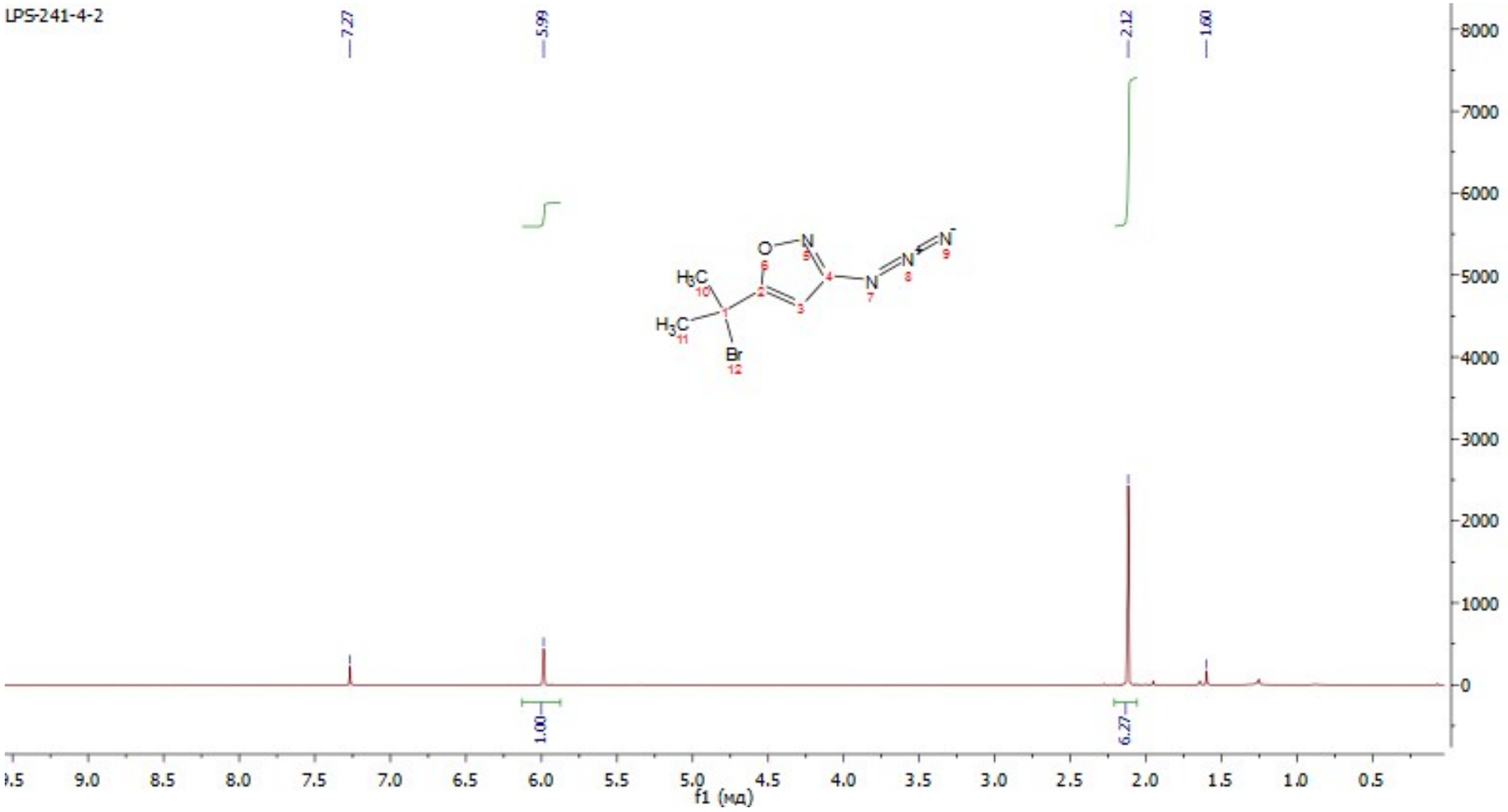


IR

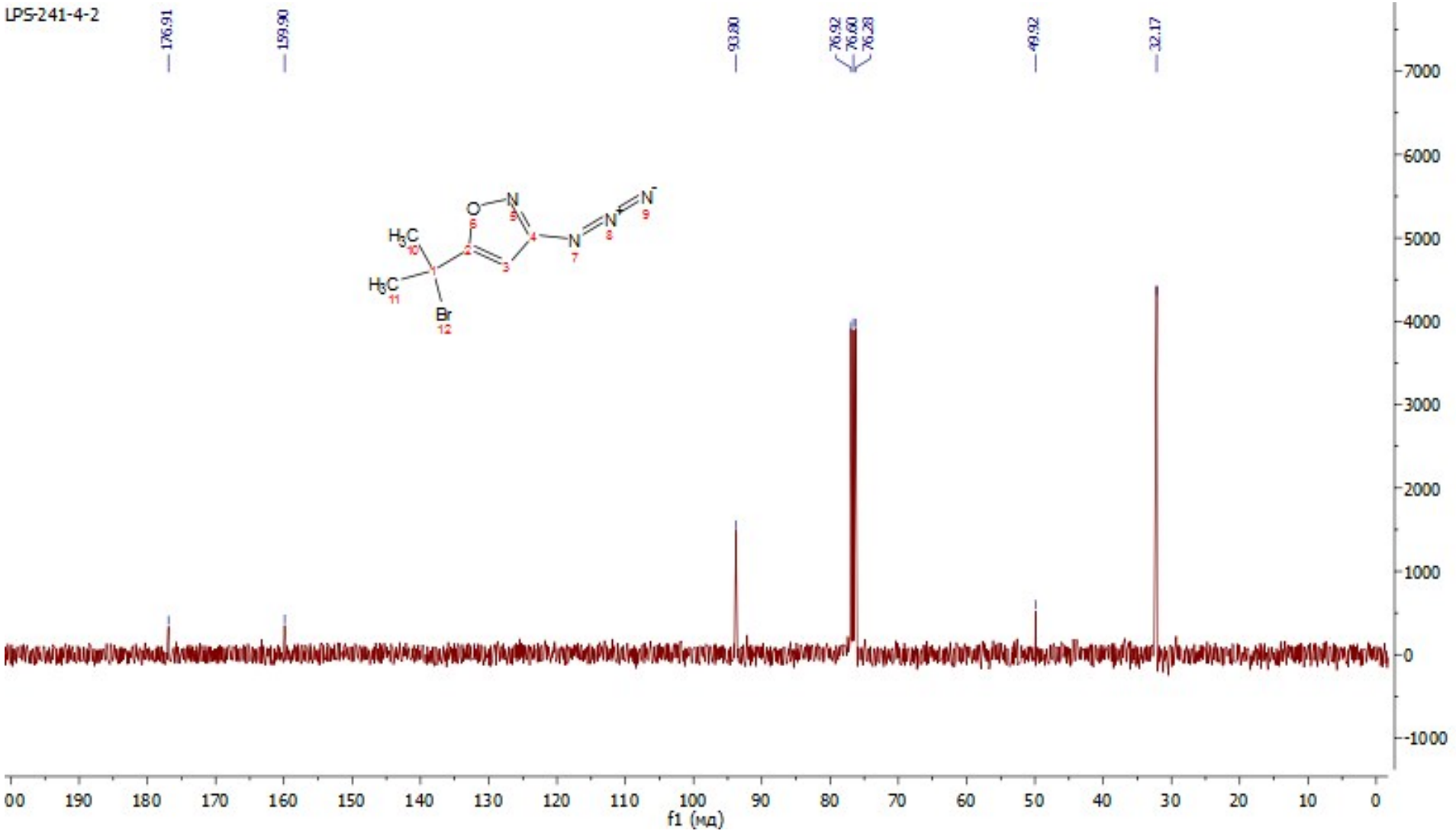

Лемпорт LPS-244-2 CDCL3 (ОС Лемпорт) ZNSE.esp
Absorbance
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
2960.2
2933.2
2873.42
2221.59
2132.88
1602.56
1469.49
1444.42
1380.78
1270.86
1232.29
1213.01
993.16
914.09
779.1
734.75
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
Wavenumber (cm-1)

3-azido-5-(2-bromopropan-2-yl)isoxazole **5g**

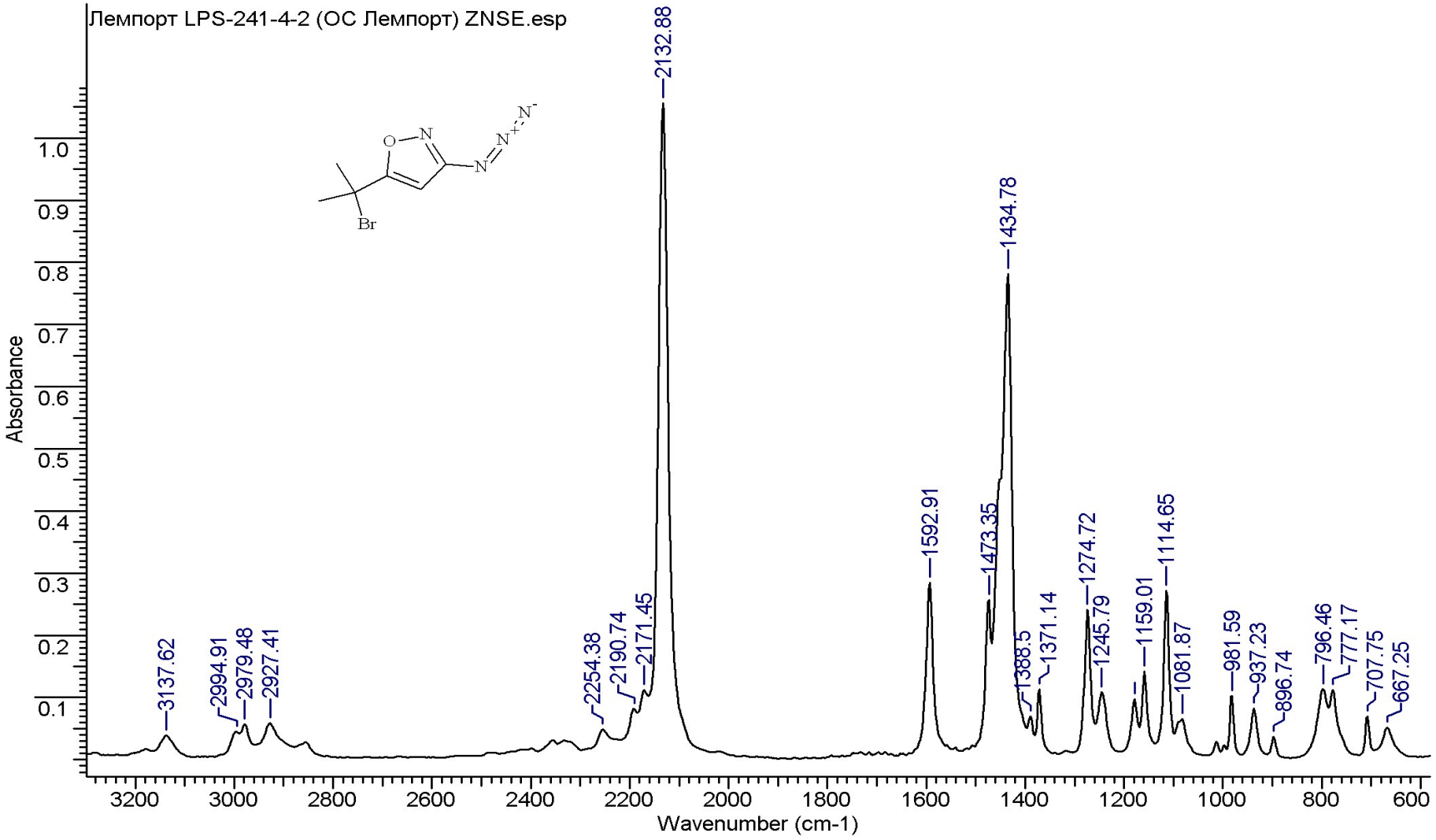
^{1}H NMR ($CDCl_3$)



^{13}C NMR ($CDCl_3$)

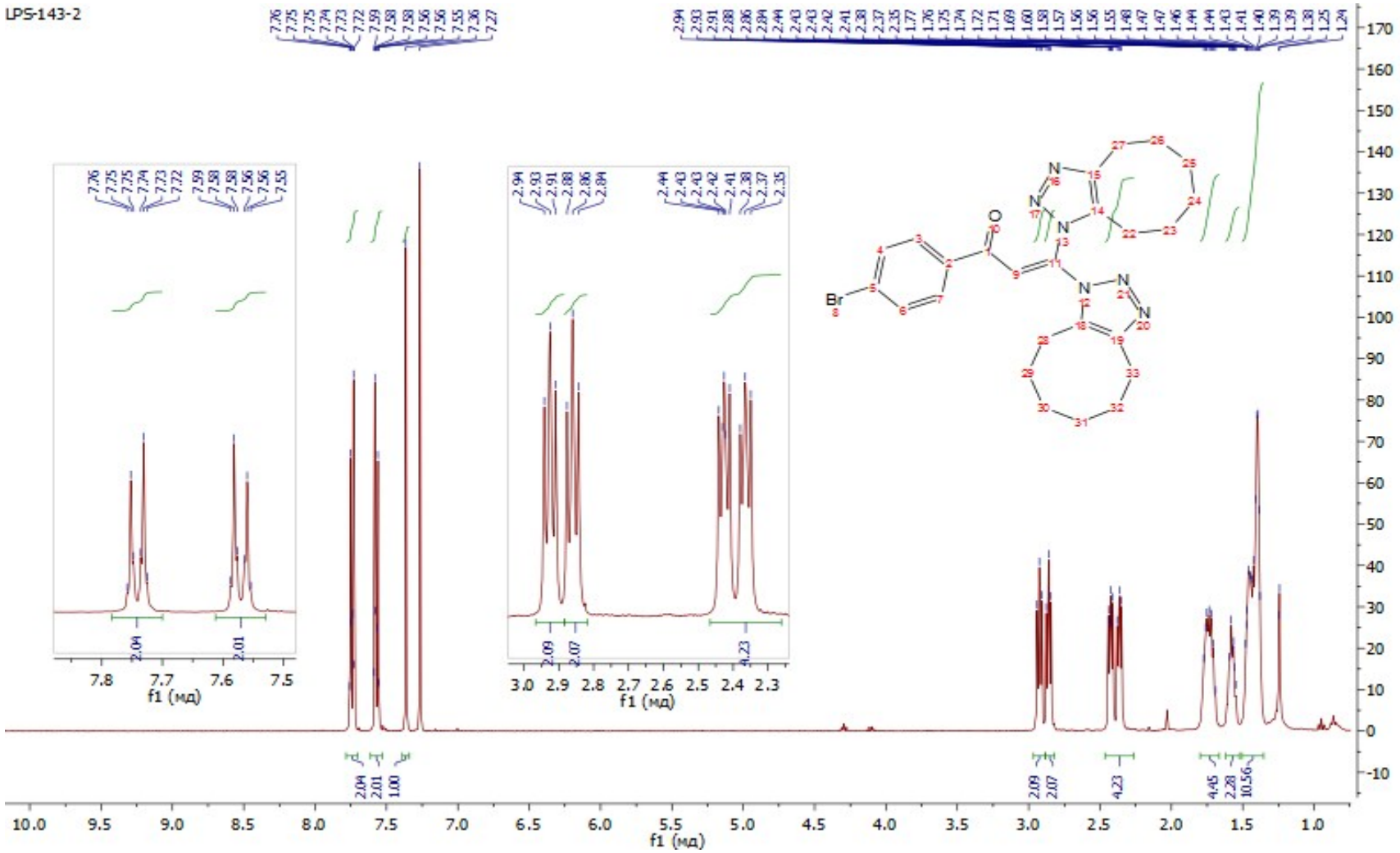


IR

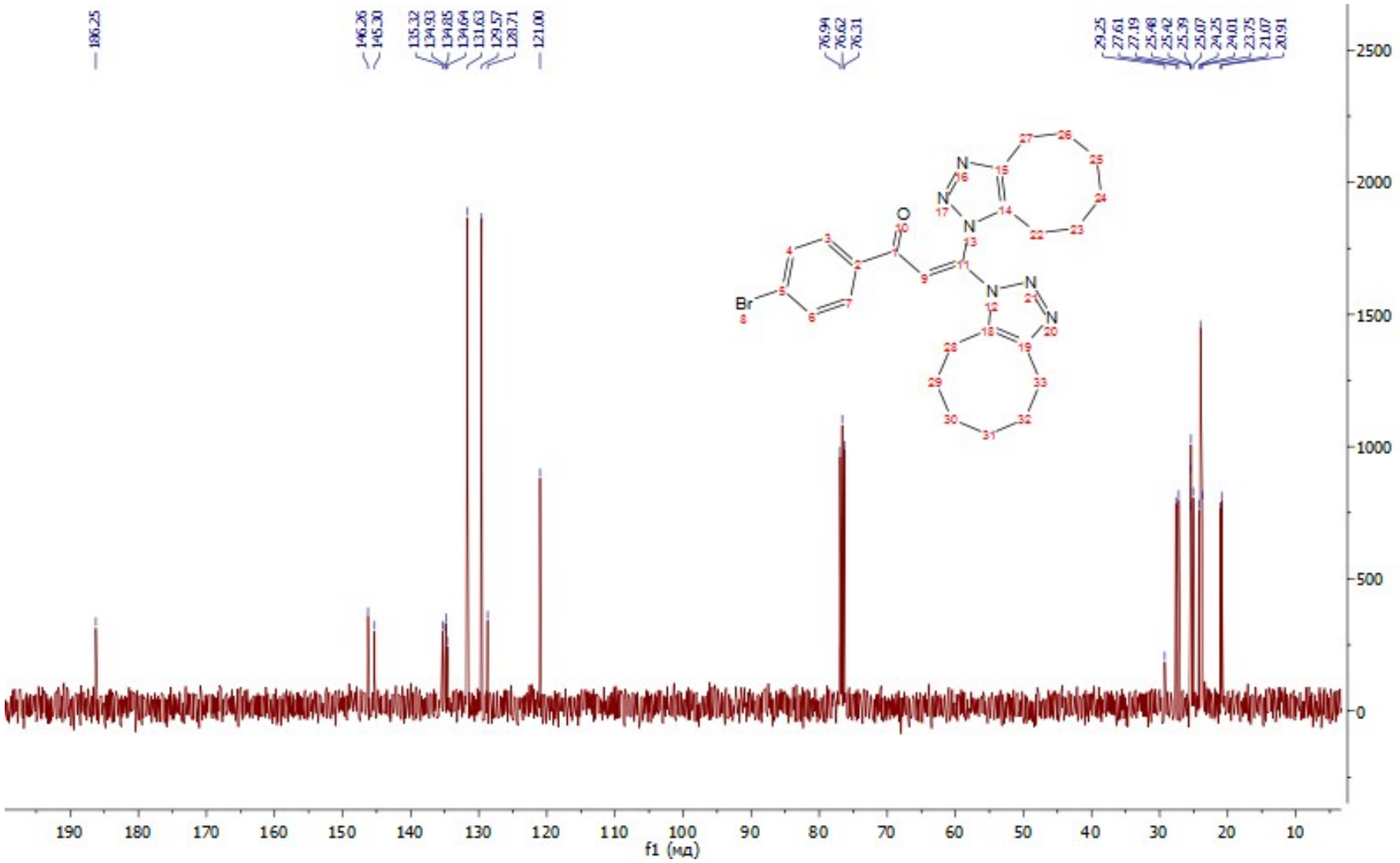

Лемпорт LPS-241-4-2 (ОС Лемпорт) ZNSE.esp
Absorbance
Wavenumber (cm-1)
3137.62
2994.91
2979.48
2927.41
2254.38
2190.74
2171.45
2132.88
1592.91
1473.35
1434.78
1388.5
1371.14
1274.72
1245.79
1159.01
1114.65
1081.87
981.59
937.23
896.74
796.46
777.17
707.75
667.25
Br

1-(4-bromophenyl)-3,3-bis(4,5,6,7,8,9-hexahydrocycloocta[d][1,2,3]triazol-1-yl)prop-2-en-1-one **6a**

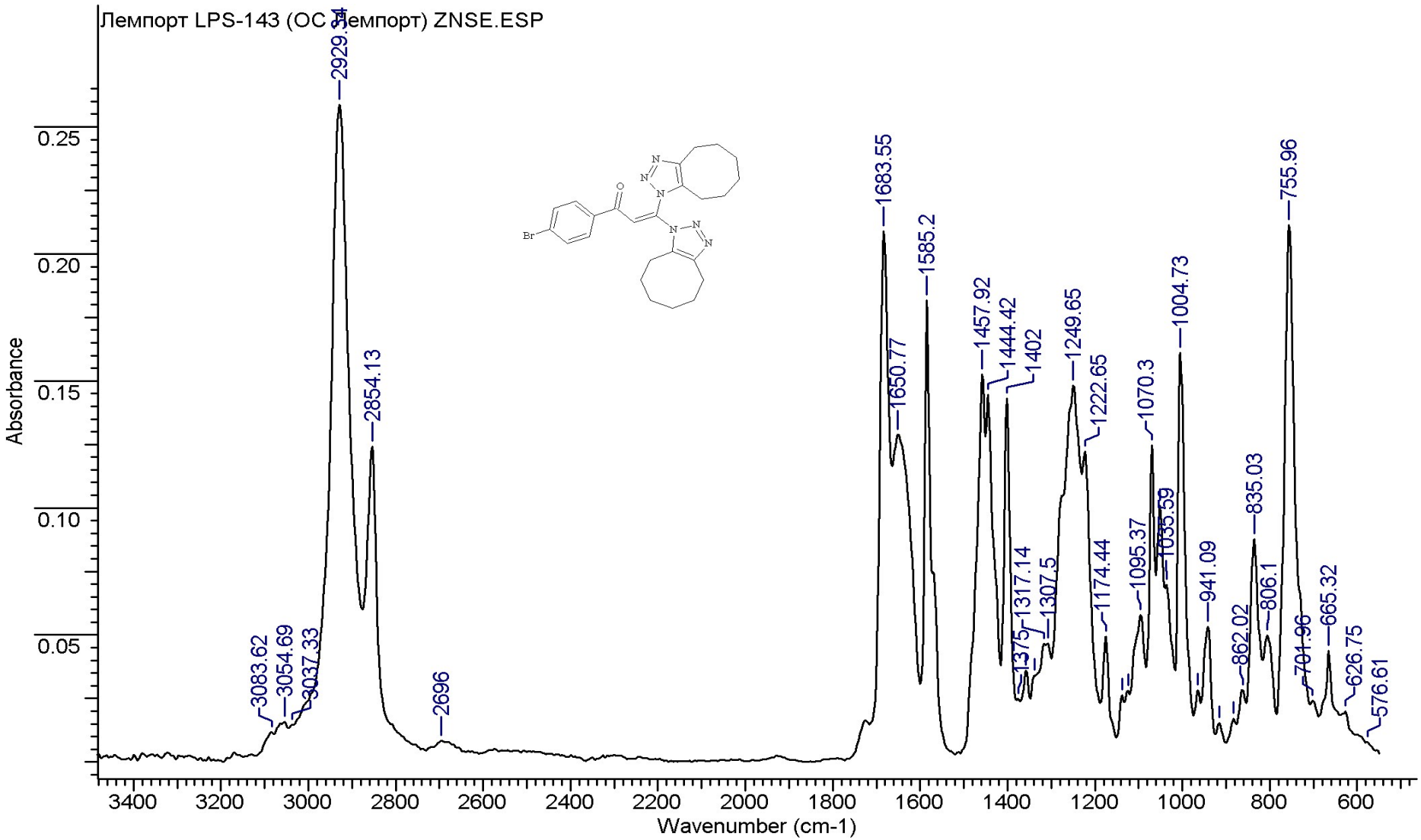
^{1}H NMR ($CDCl_3$)



^{13}C NMR ($CDCl_3$)

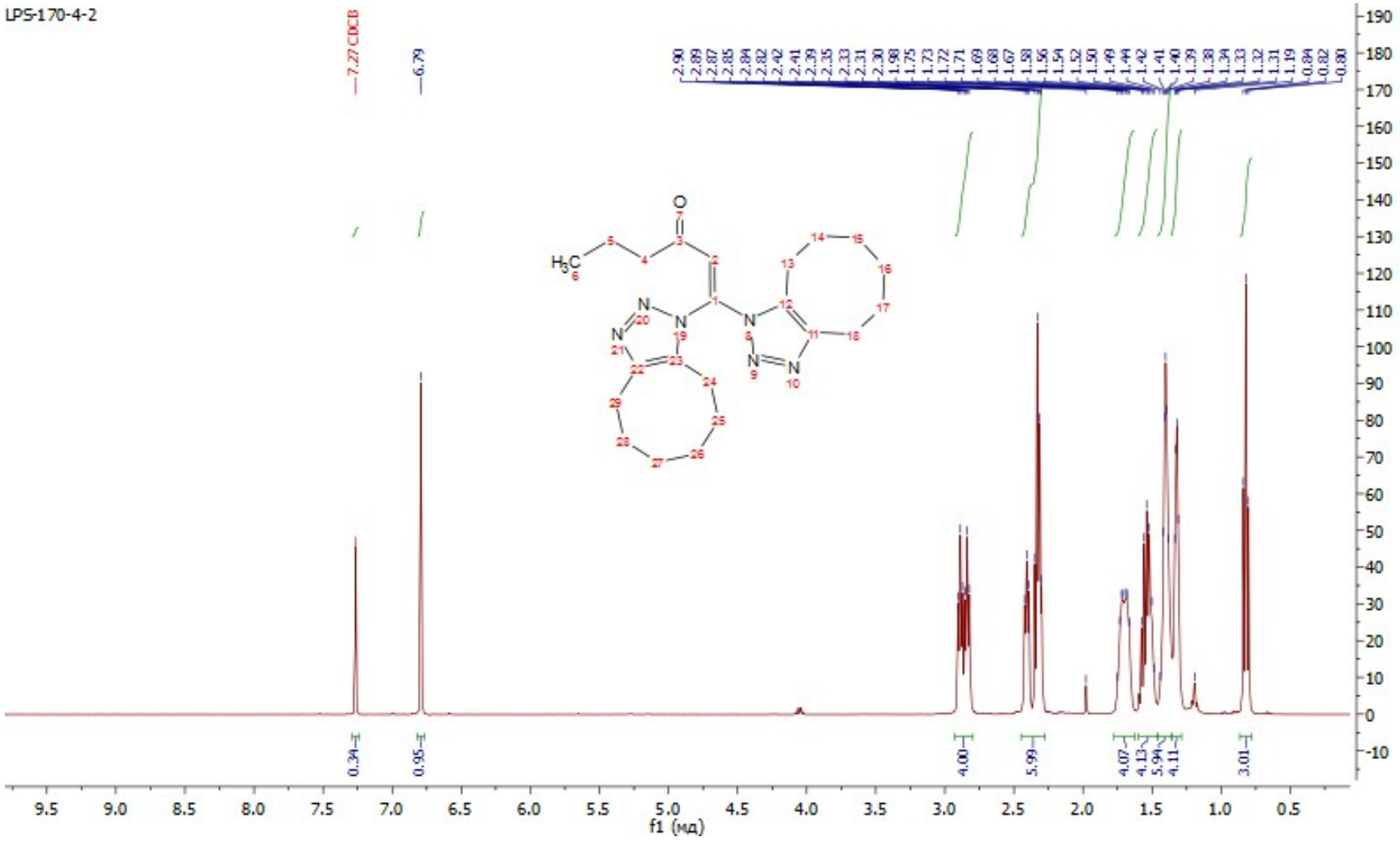


IR

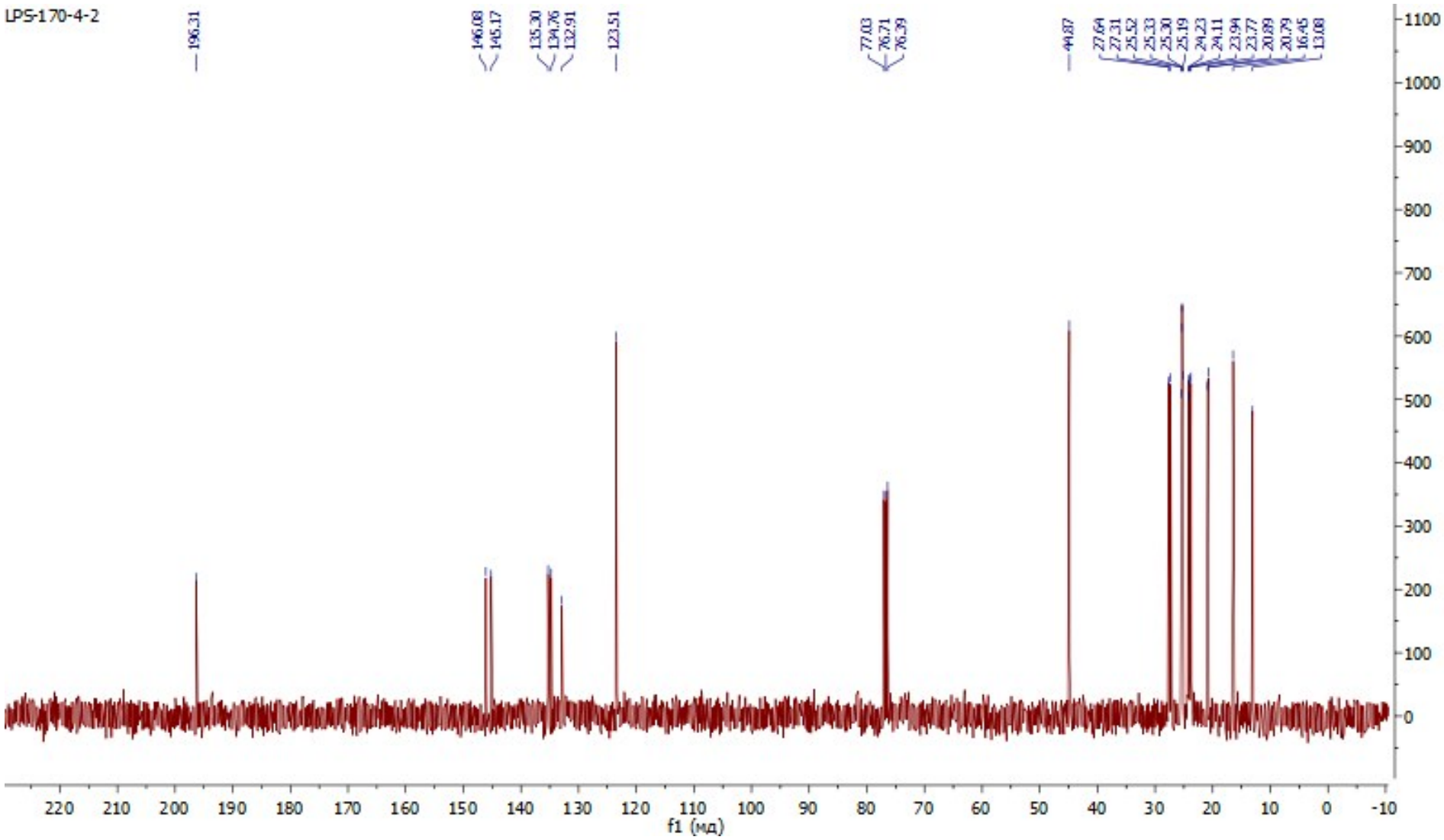

Лемпорт LPS-143 (ОС Лемпорт) ZNSE.ESP
Absorbance
0.25
0.20
0.15
0.10
0.05
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
Wavenumber (cm-1)
2929.34
2854.13
3083.62
3054.69
3037.33
2696
1683.55
1650.77
1585.2
1457.92
1444.42
1402
1375
1317.14
1307.5
1249.65
1222.65
1174.44
1095.37
1070.3
1035.59
1004.73
941.09
862.02
835.03
806.1
755.96
701.96
665.32
626.75
576.61
Br
O
N

1,1-bis(4,5,6,7,8,9-hexahydrocycloocta[d]-[1,2,3]triazol-1-yl)hex-1-en-3-one **6e**

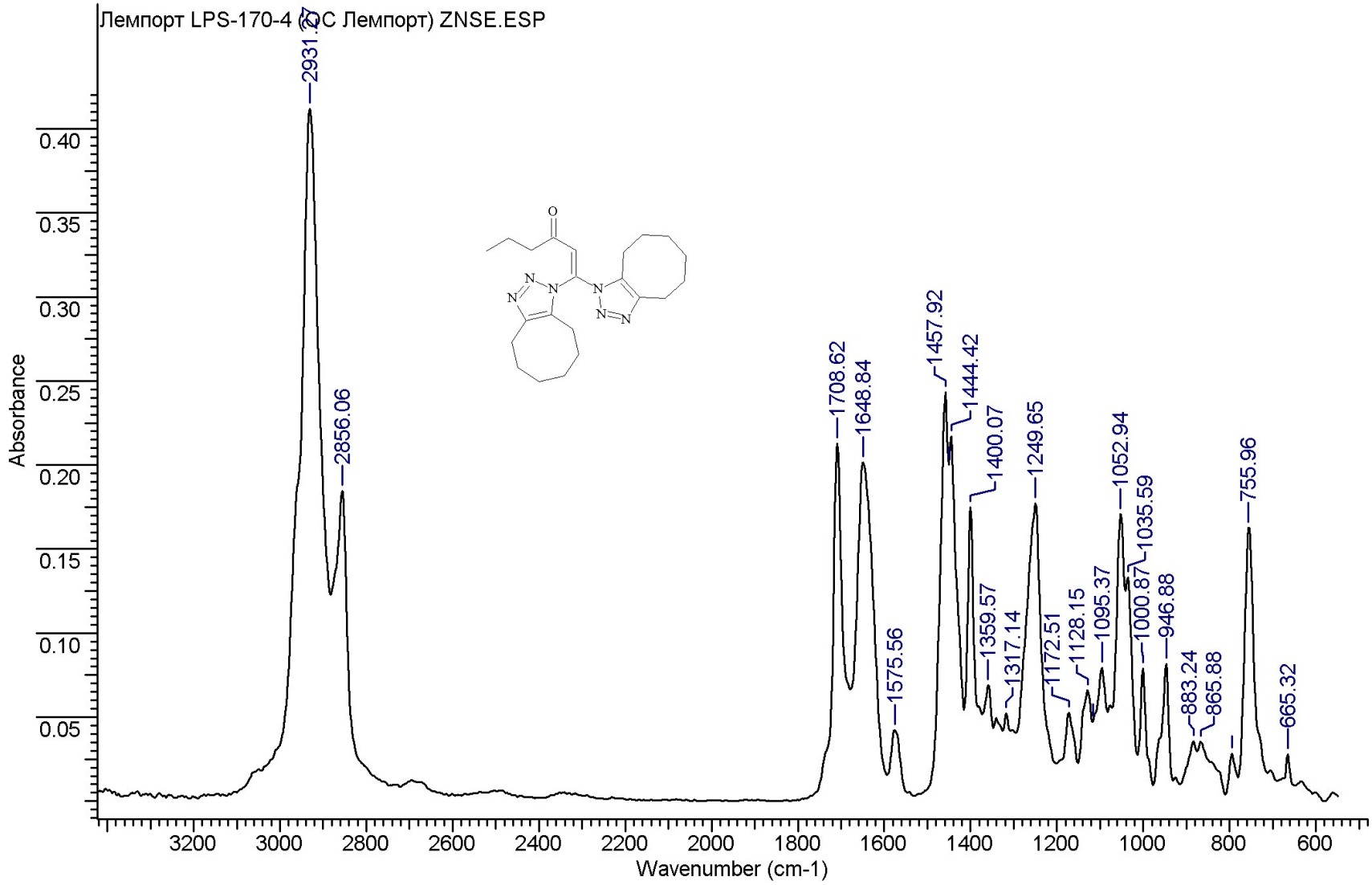
^{1}H NMR ($CDCl_3$)



^{13}C NMR ($CDCl_3$)

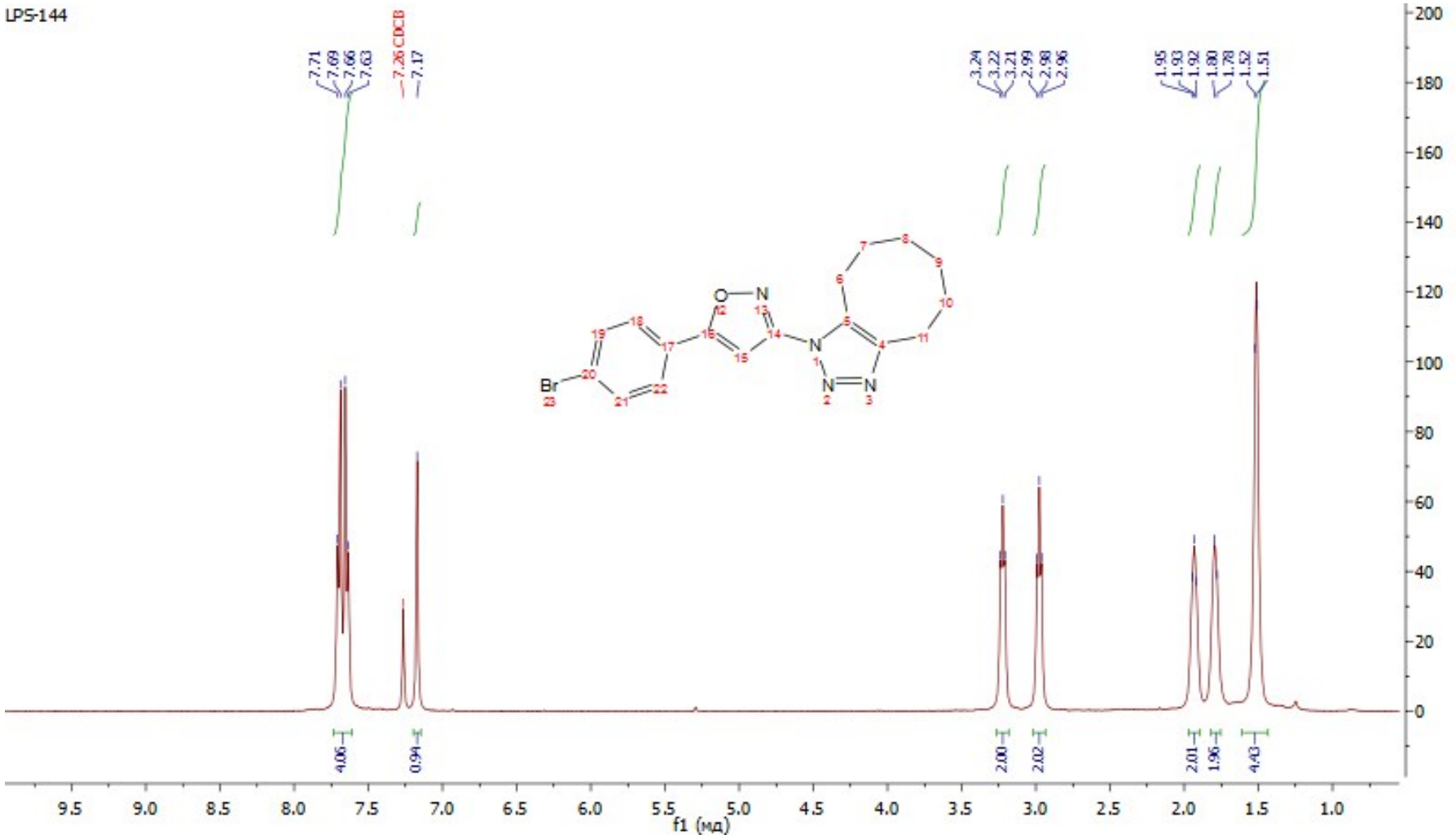


IR

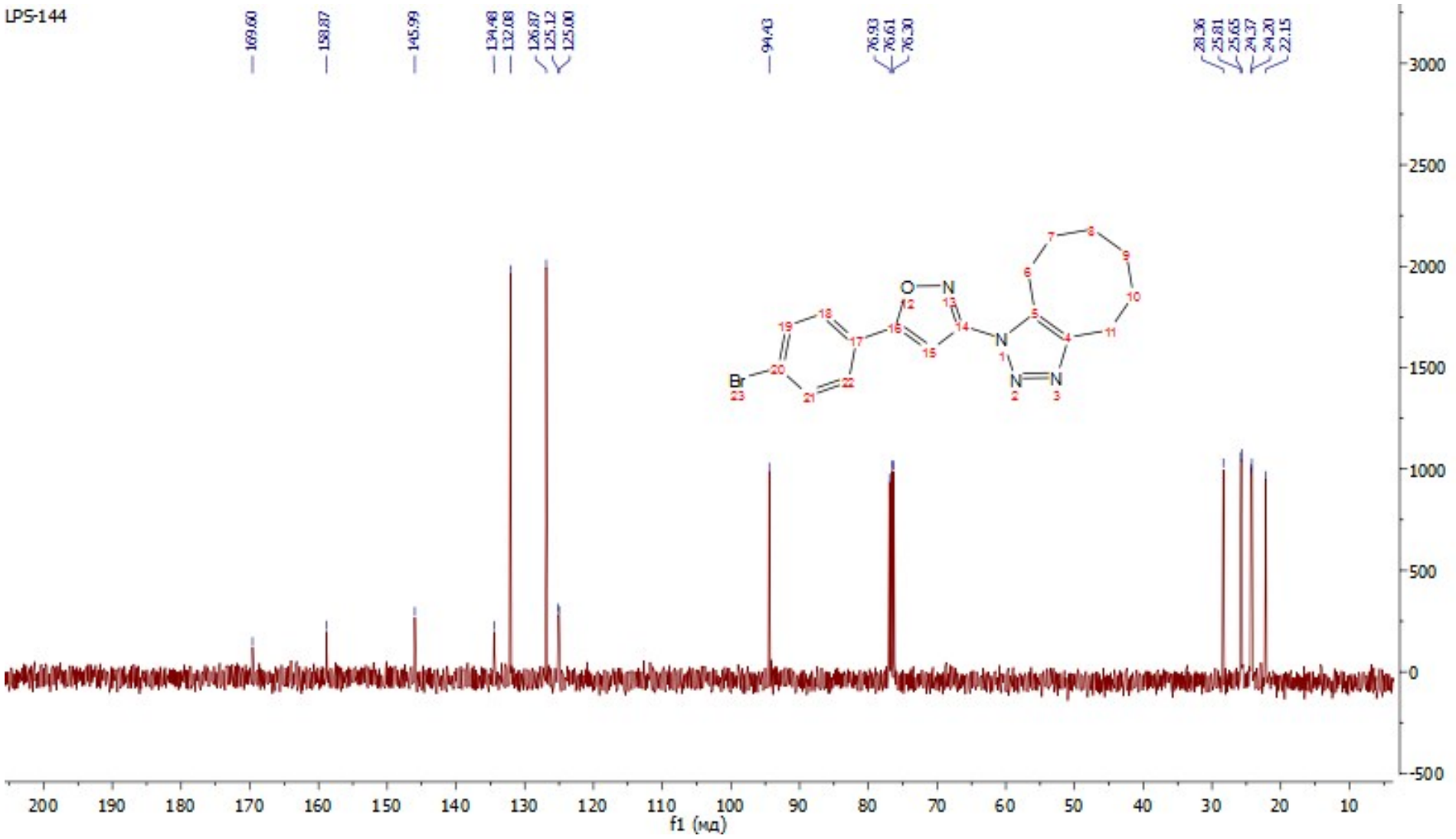

Лемпорт LPS-170-4 (ЭС Лемпорт) ZNSE.ESP
2931.27
2856.06
1708.62
1648.84
1575.56
1457.92
1444.42
1400.07
1359.57
1317.14
1249.65
1172.51
1128.15
1095.37
1052.94
1035.59
1000.87
946.88
883.24
865.88
755.96
665.32
Absorbance
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
Wavenumber (cm-1)

1-(5-(4-bromophenyl)isoxazol-3-yl)-4,5,6,7,8,9-hexahydro-1H-cycloocta[d][1,2,3]triazole **7a**

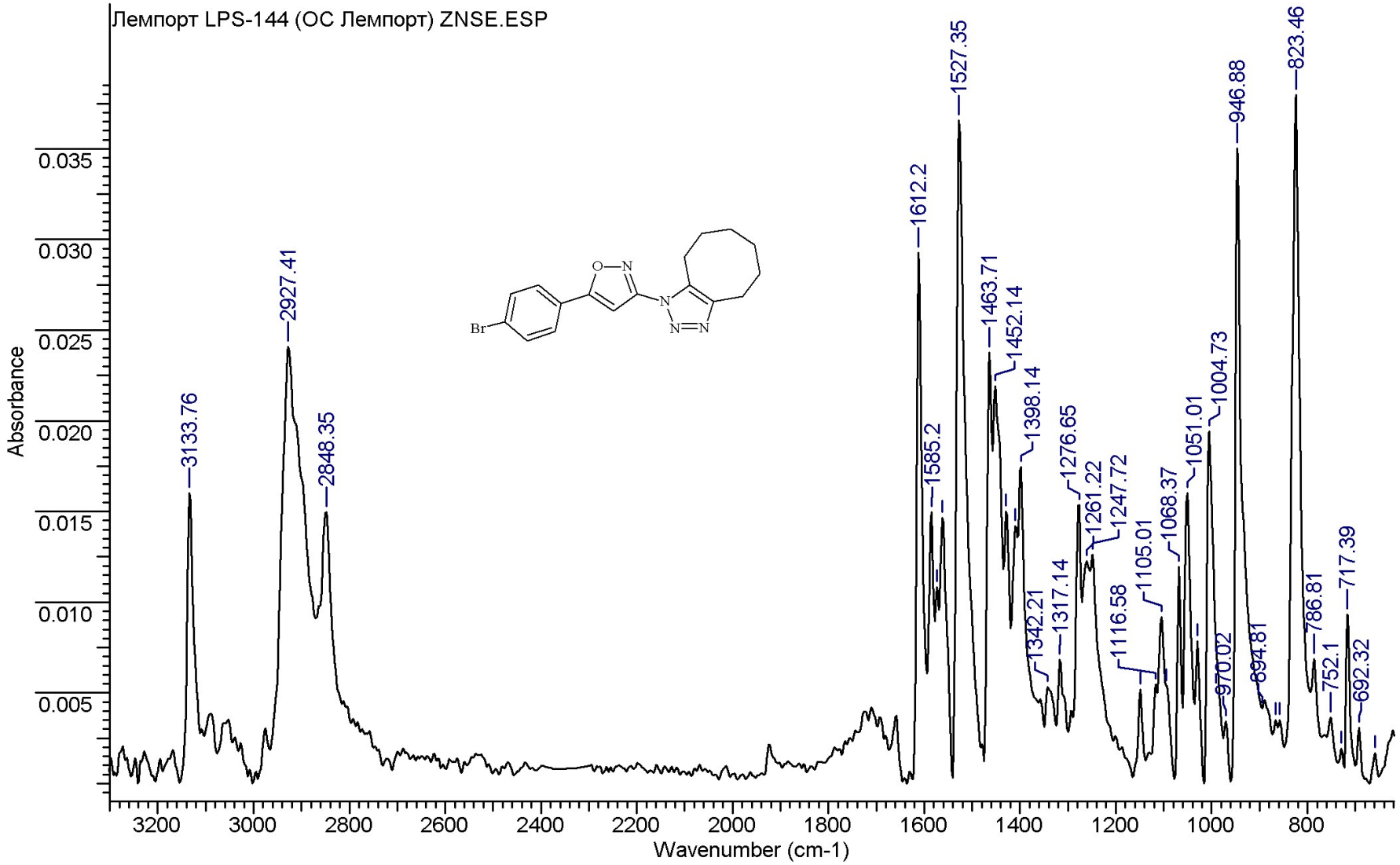
^{1}H NMR ($CDCl_3$)



^{13}C NMR ($CDCl_3$)

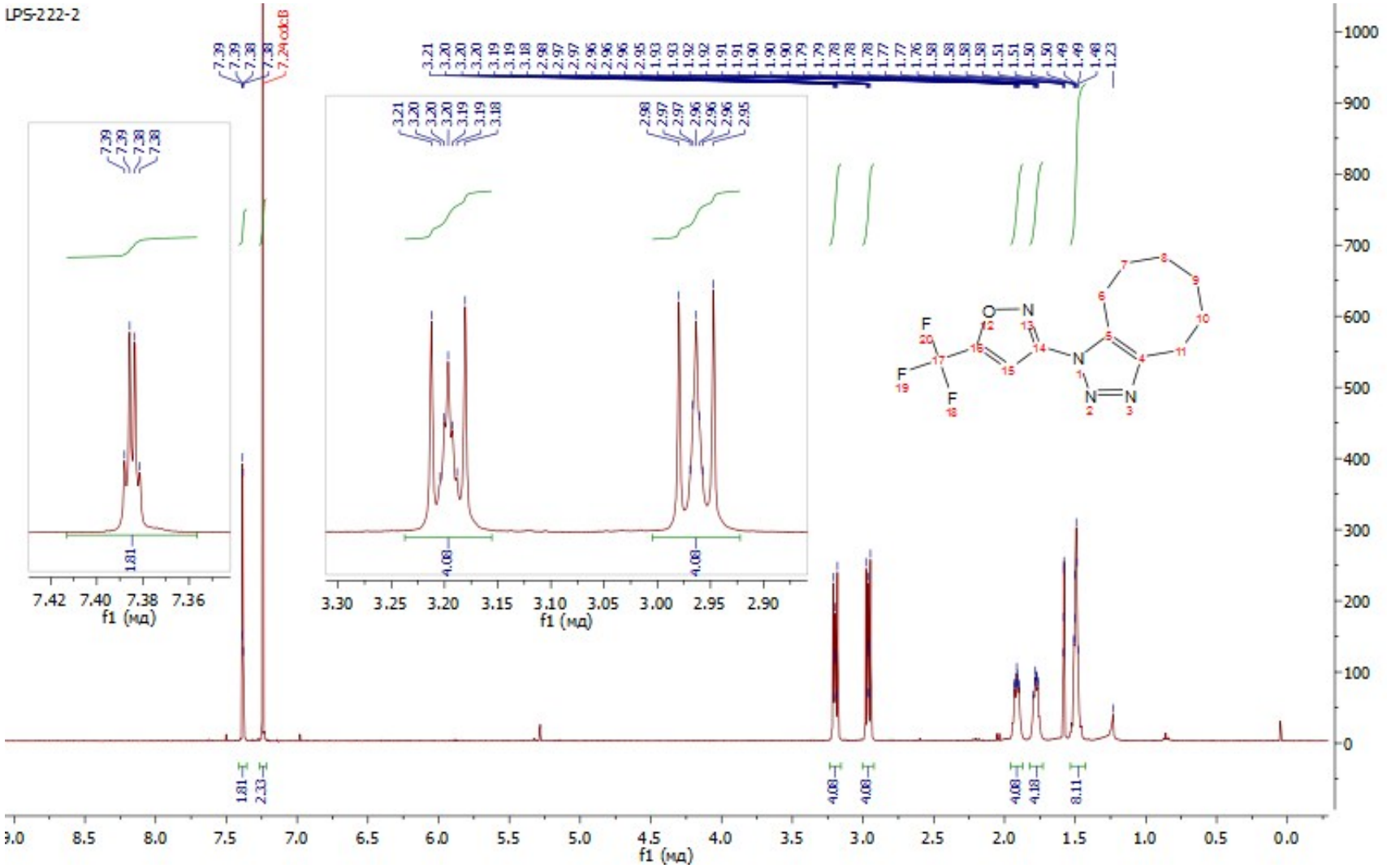


IR

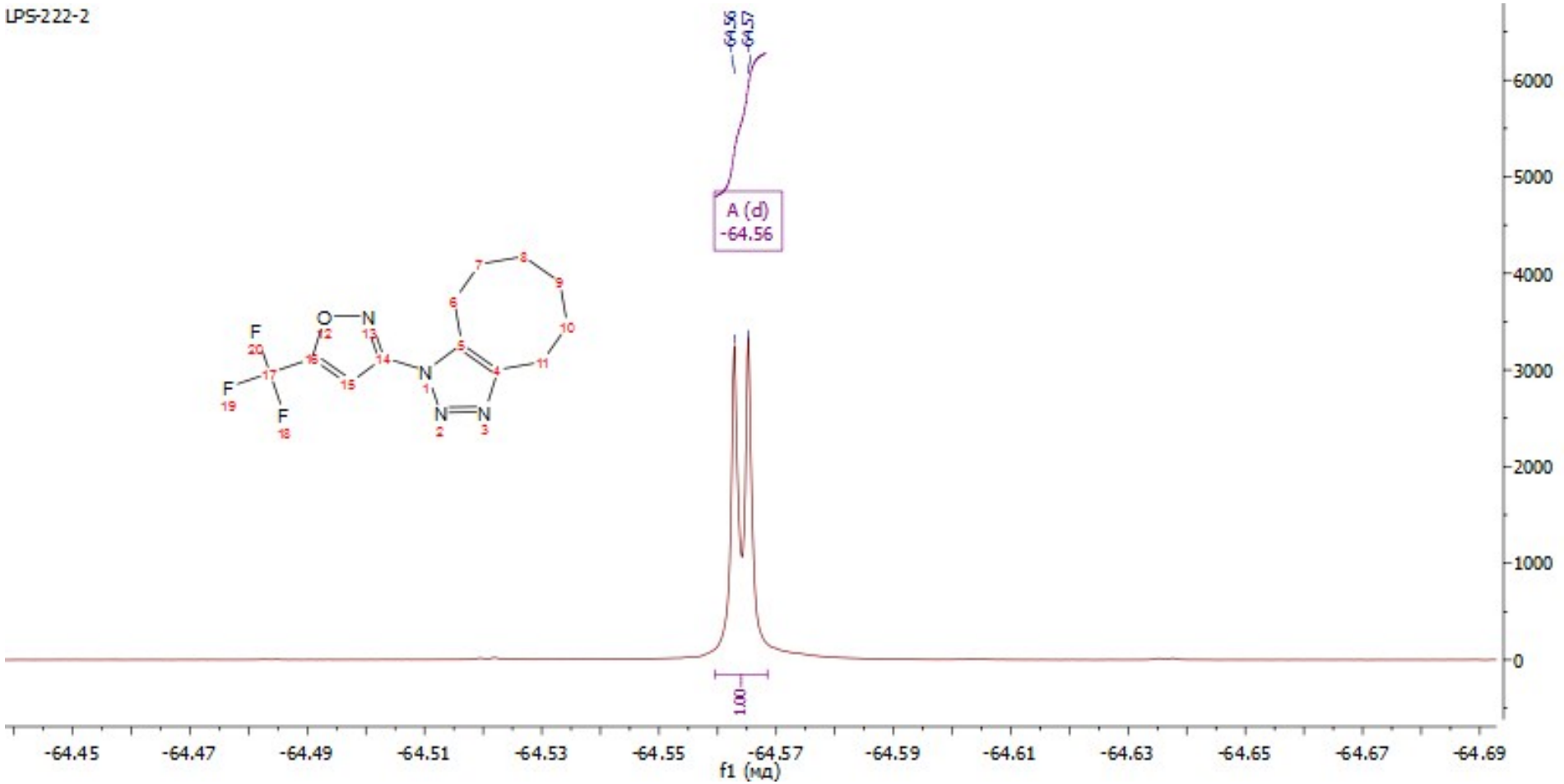

Лемпорт LPS-144 (ОС Лемпорт) ZNSE.ESP
Absorbance
0.035
0.030
0.025
0.020
0.015
0.010
0.005
3133.76
2927.41
2848.35
1612.2
1585.2
1527.35
1463.71
1452.14
1398.14
1342.21
1317.14
1276.65
1261.22
1247.72
1116.58
1105.01
1068.37
1051.01
1004.73
970.02
946.88
894.81
823.46
786.81
752.1
717.39
692.32
Br
O
N
N
N
N
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
Wavenumber (cm-1)

1-(5-(trifluoromethyl)isoxazol-3-yl)-4,5,6,7,8,9-hexahydro-1H-cycloocta[d][1,2,3]triazole **7h**

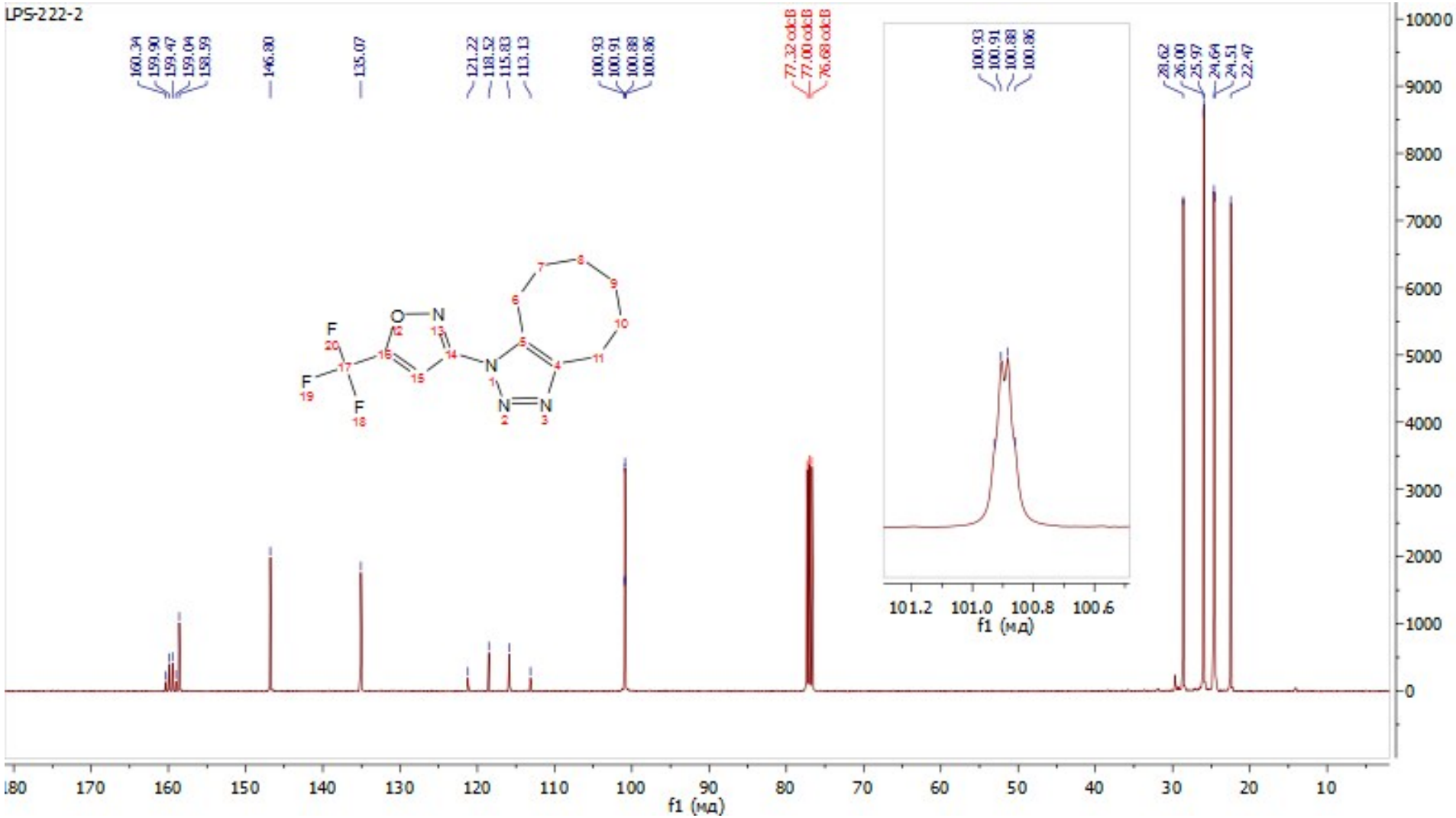
^{1}H NMR ($CDCl_3$)



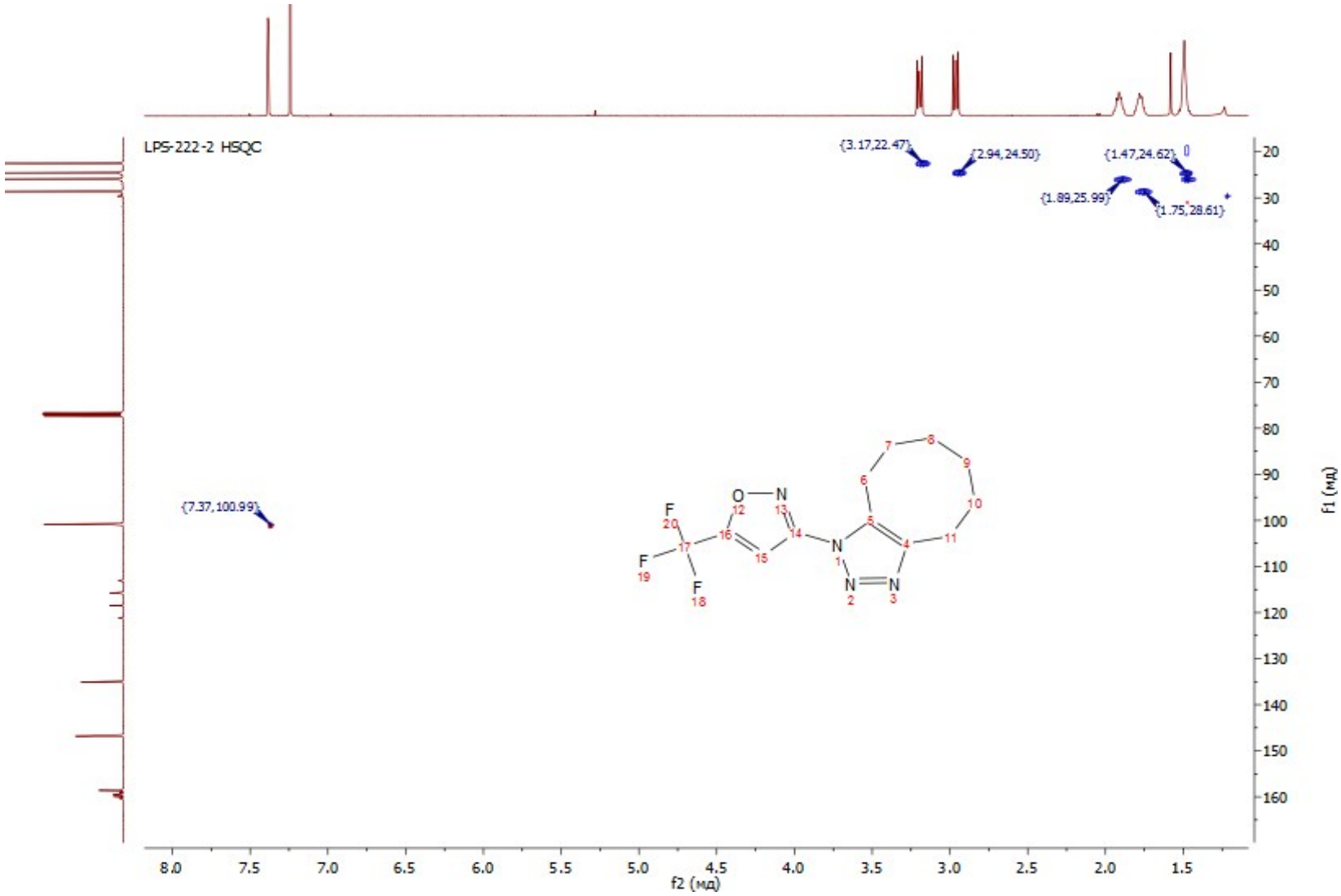
^{19}F NMR ($CDCl_3$)



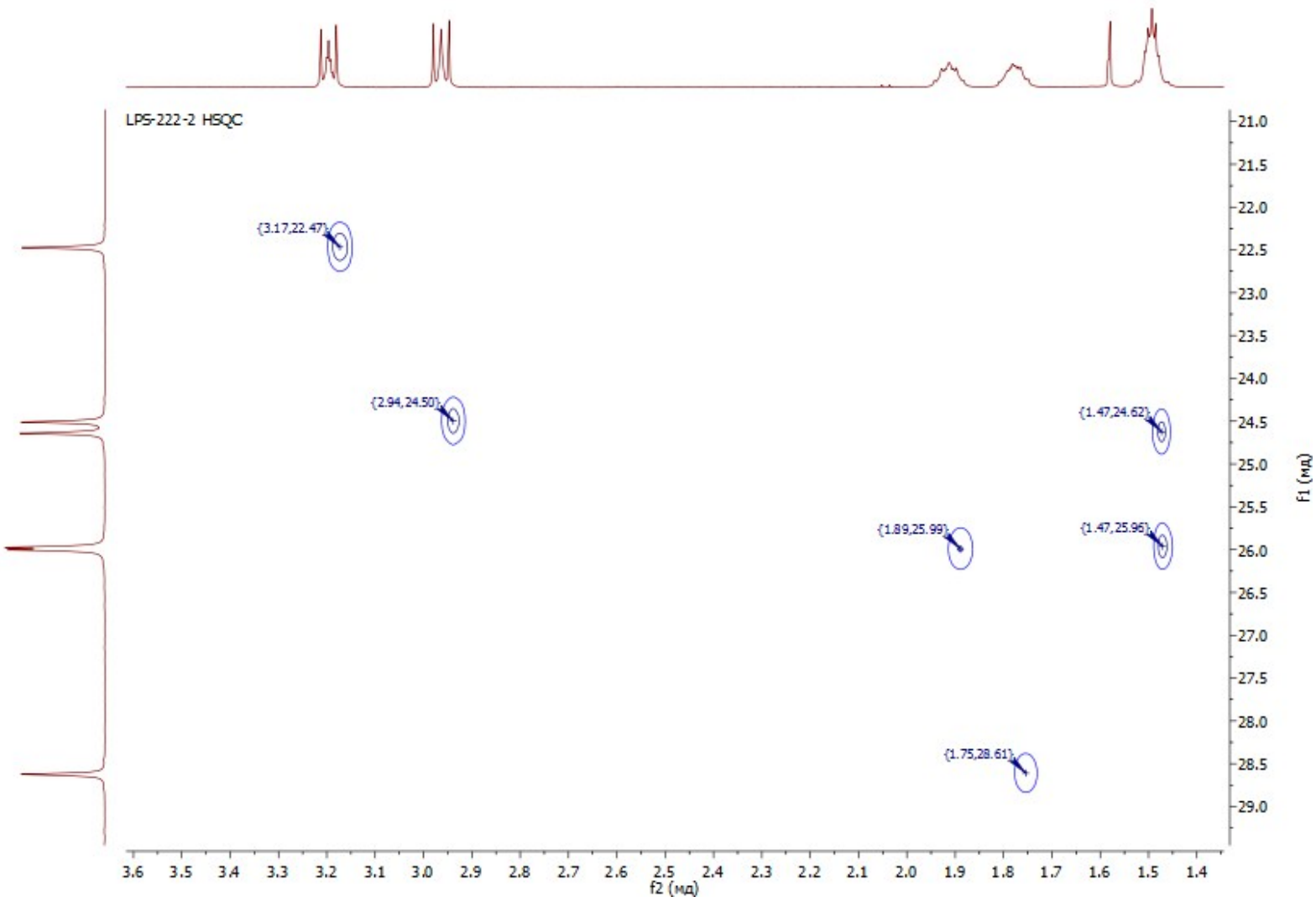
^{13}C NMR ($CDCl_3$)



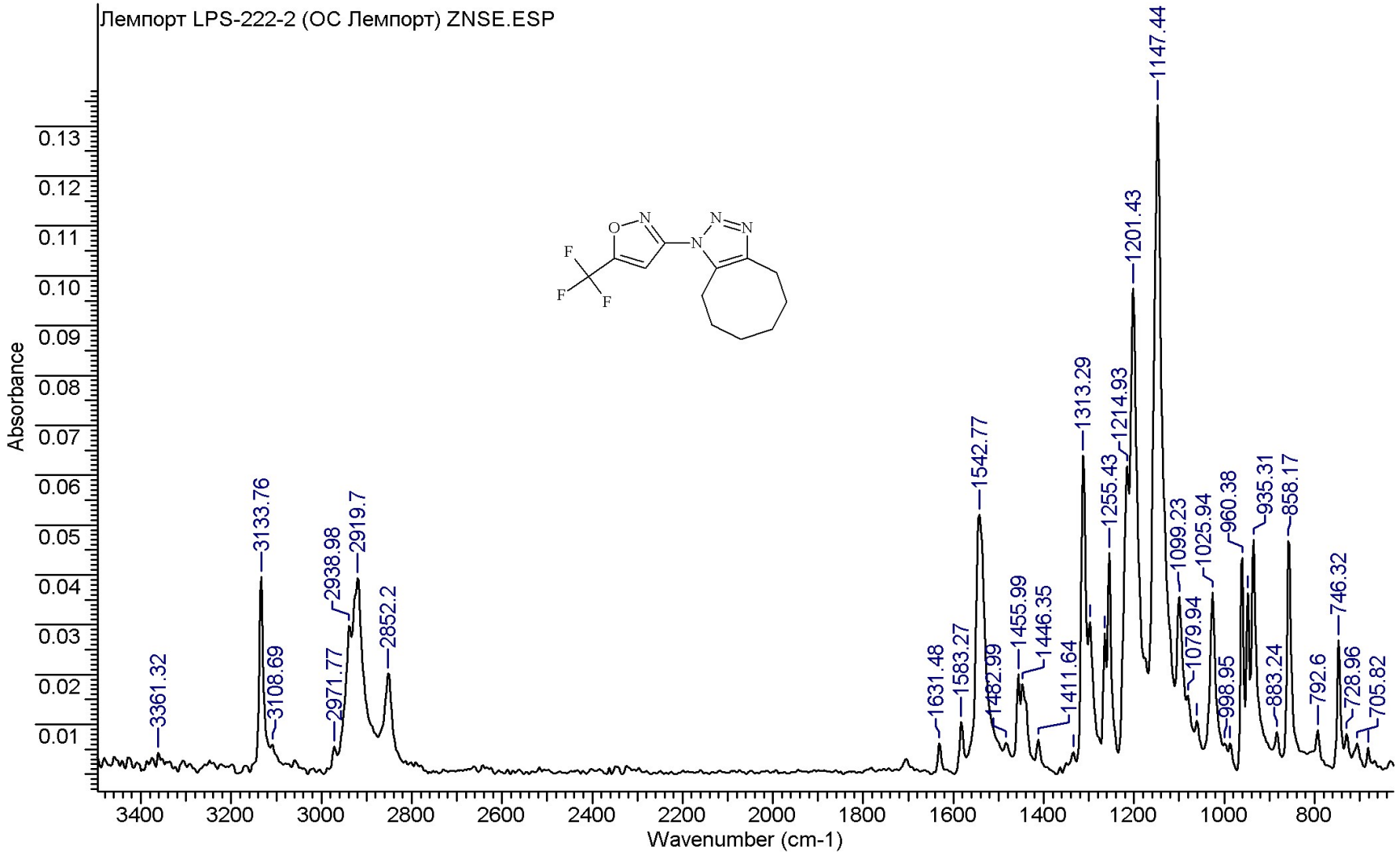
$^{1}H/^{13}C$ HSQC NMR ($CDCl_3$)



$^{1}H/^{13}C$ HSQC NMR ($CDCl_3$): fragment

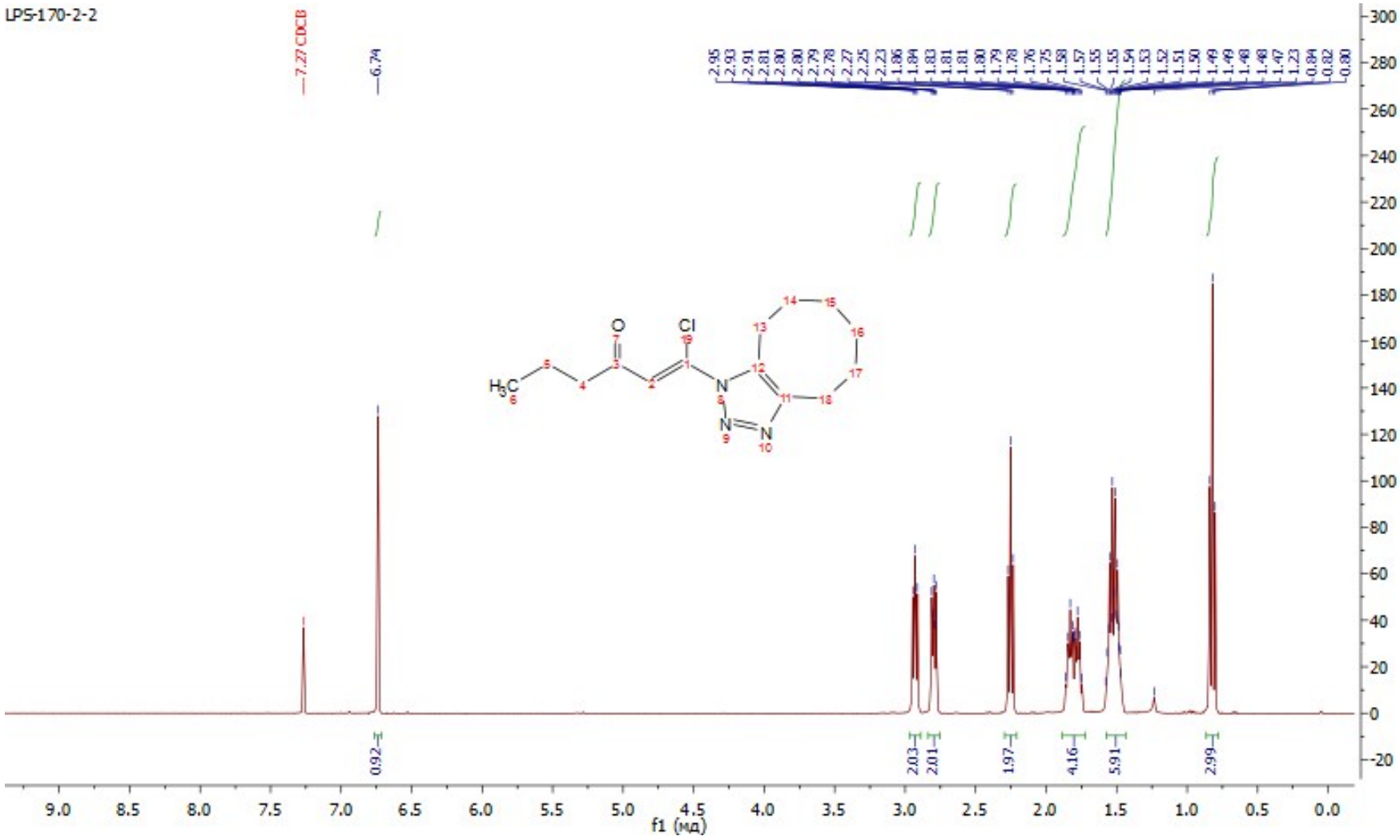


IR

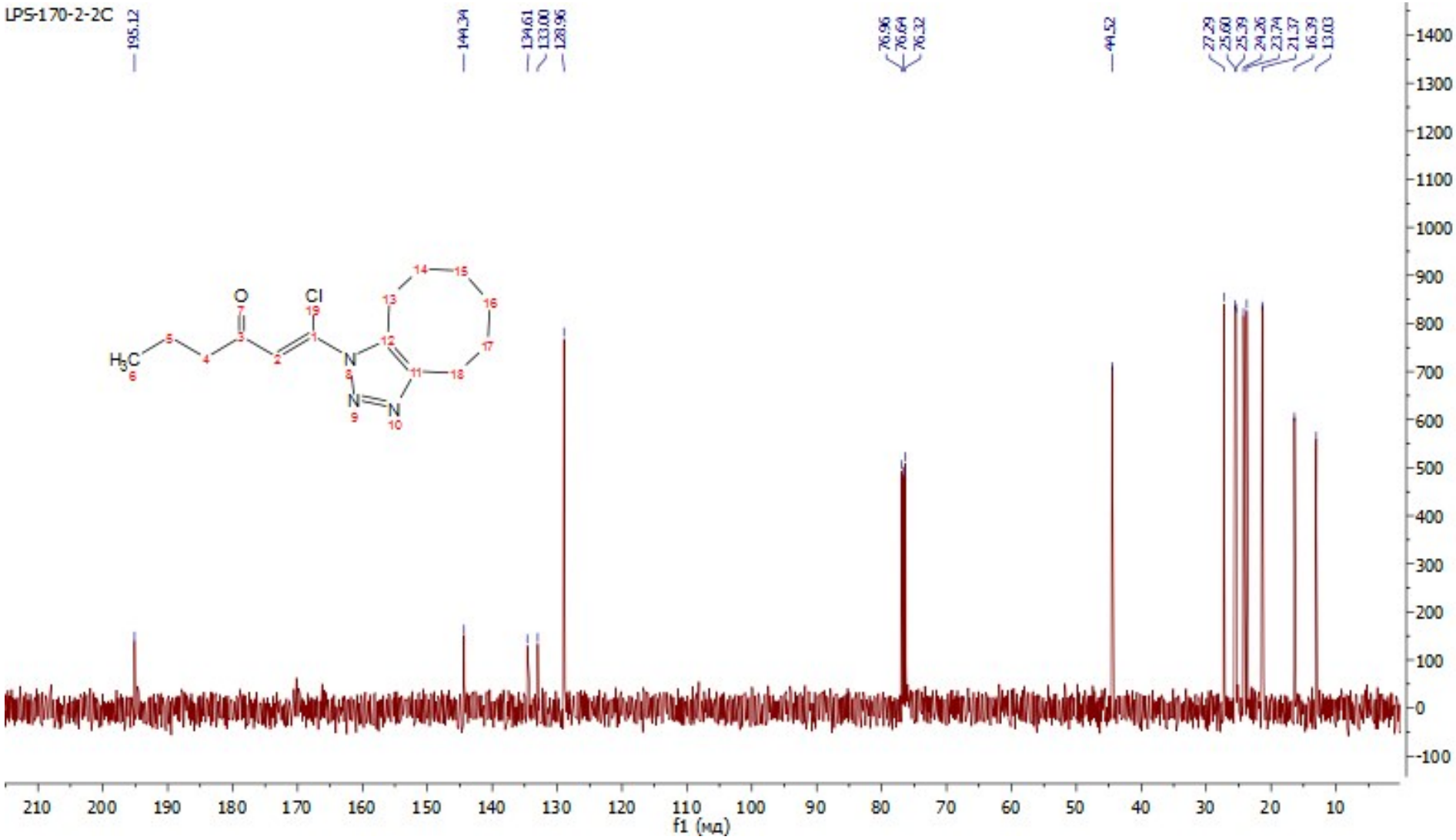

Лемпорт LPS-222-2 (ОС Лемпорт) ZNSE.ESP
Absorbance
Wavenumber (cm-1)
3361.32
3133.76
3108.69
2971.77
2938.98
2919.7
2852.2
1631.48
1583.27
1542.77
1482.99
1455.99
1446.35
1411.64
1313.29
1255.43
1214.93
1201.43
1147.44
1099.23
1079.94
1025.94
998.95
960.38
935.31
883.24
858.17
792.6
746.32
728.96
705.82

1-chloro-1-(4,5,6,7,8,9-hexahydrocycloocta[d]-[1,2,3]triazol-1-yl)hex-1-en-3-one **8e**

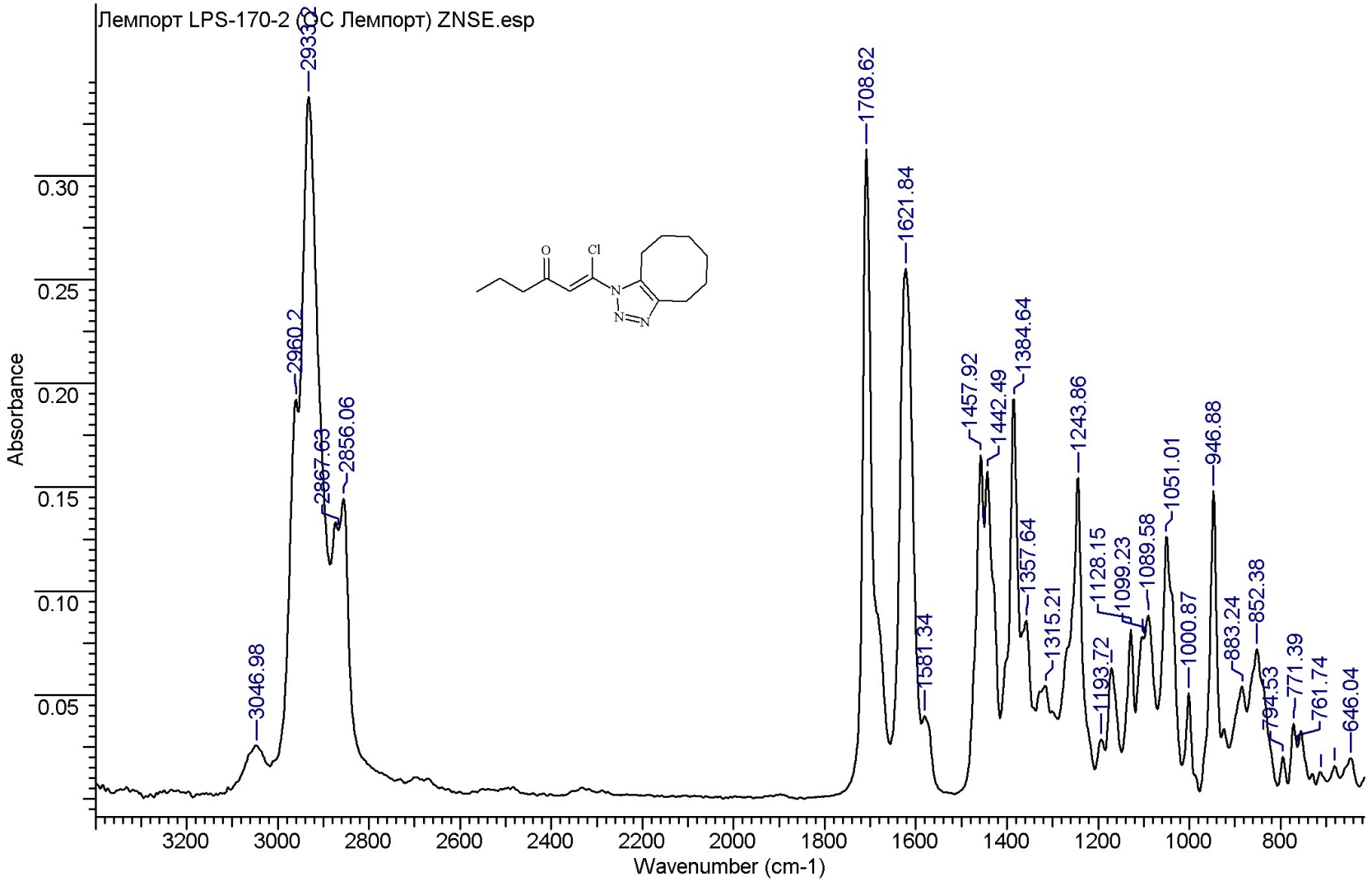
^{1}H NMR ($CDCl_3$)



^{13}C NMR ($CDCl_3$)

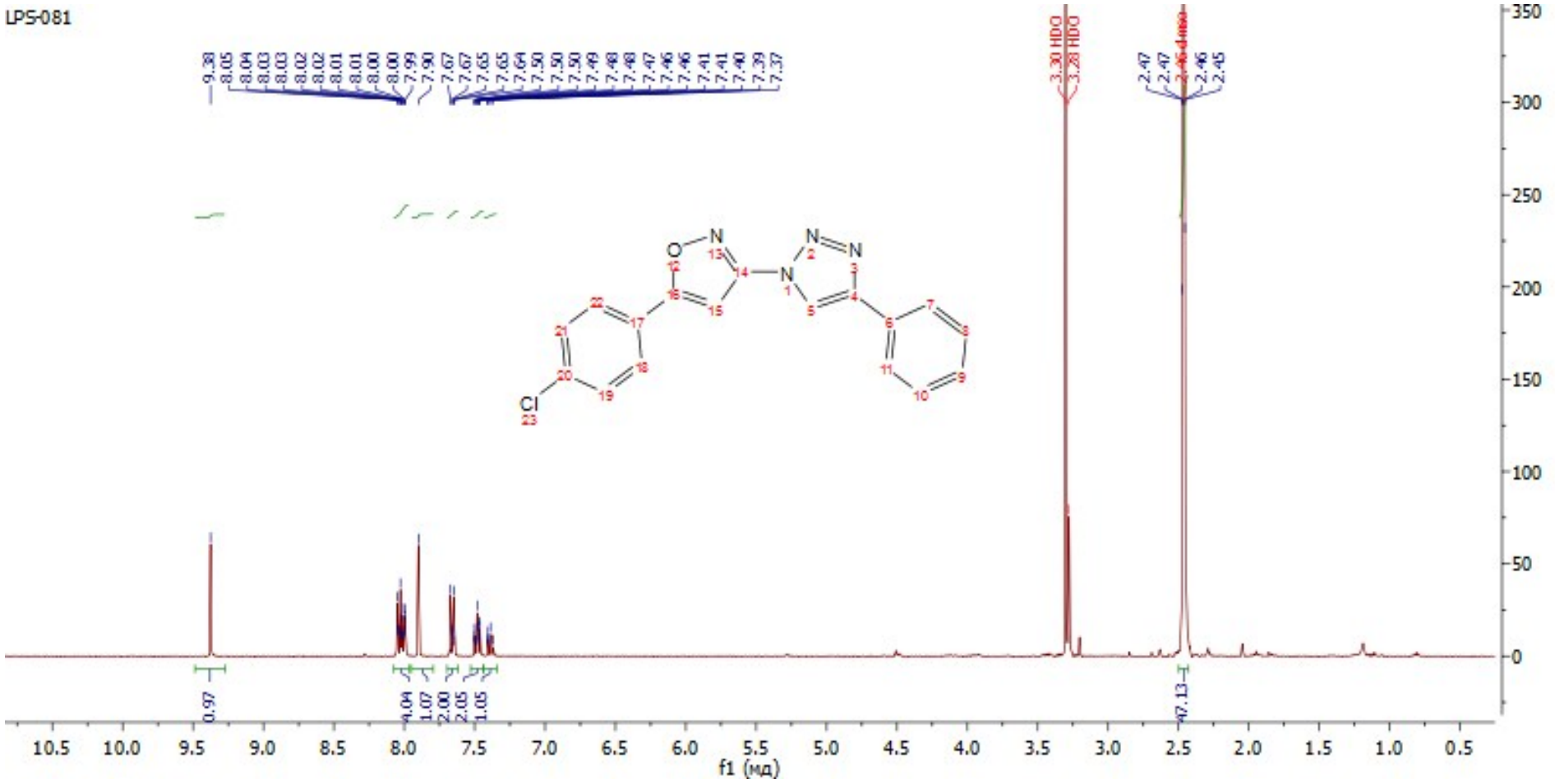


IR

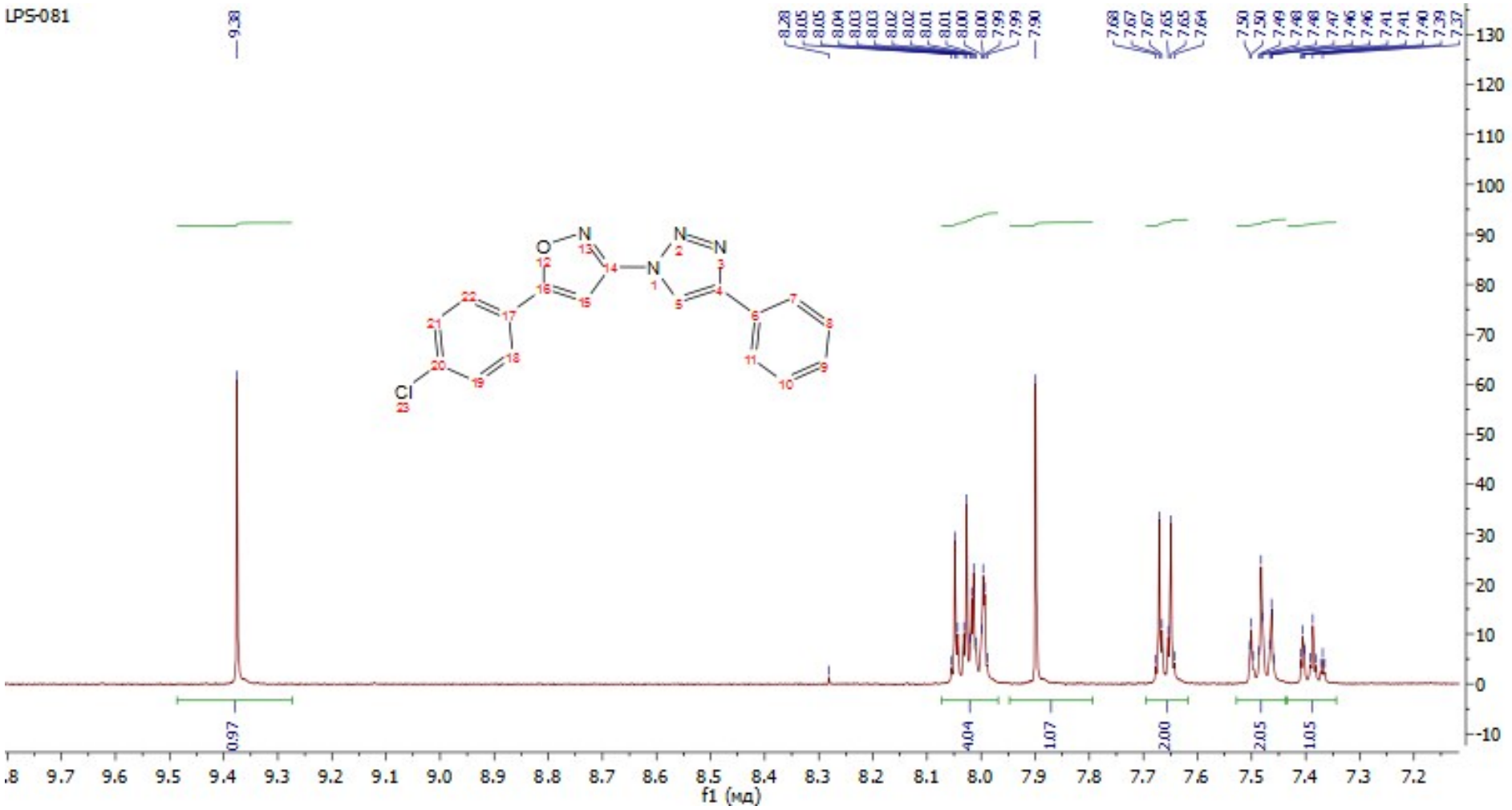

Лемпорт LPS-170-2 (ЭС Лемпорт) ZNSE.esp
Absorbance
0.30
0.25
0.20
0.15
0.10
0.05
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
Wavenumber (cm-1)
3046.98
2960.2
2933.02
2867.63
2856.06
1708.62
1621.84
1581.34
1457.92
1442.49
1384.64
1357.64
1315.21
1243.86
1193.72
1128.15
1099.23
1089.58
1051.01
1000.87
946.88
883.24
852.38
794.53
771.39
761.74
646.04
O
Cl
N
N
N

1-[5-(4-Chloro-phenyl)-isoxazol-3-yl]-4-phenyl-1H-[1,2,3]triazole **9b**

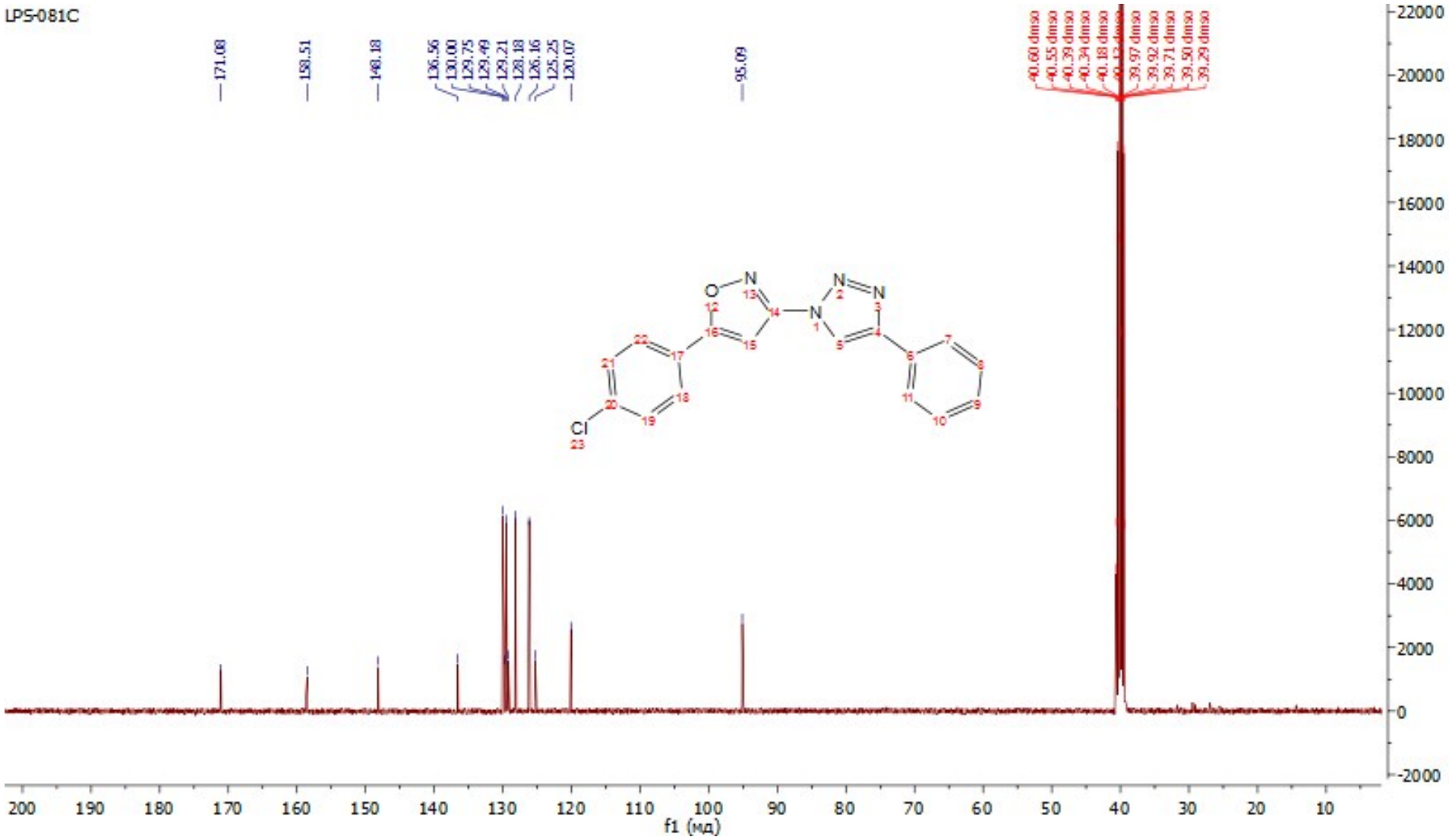
^{1}H NMR (DMSO-d6)



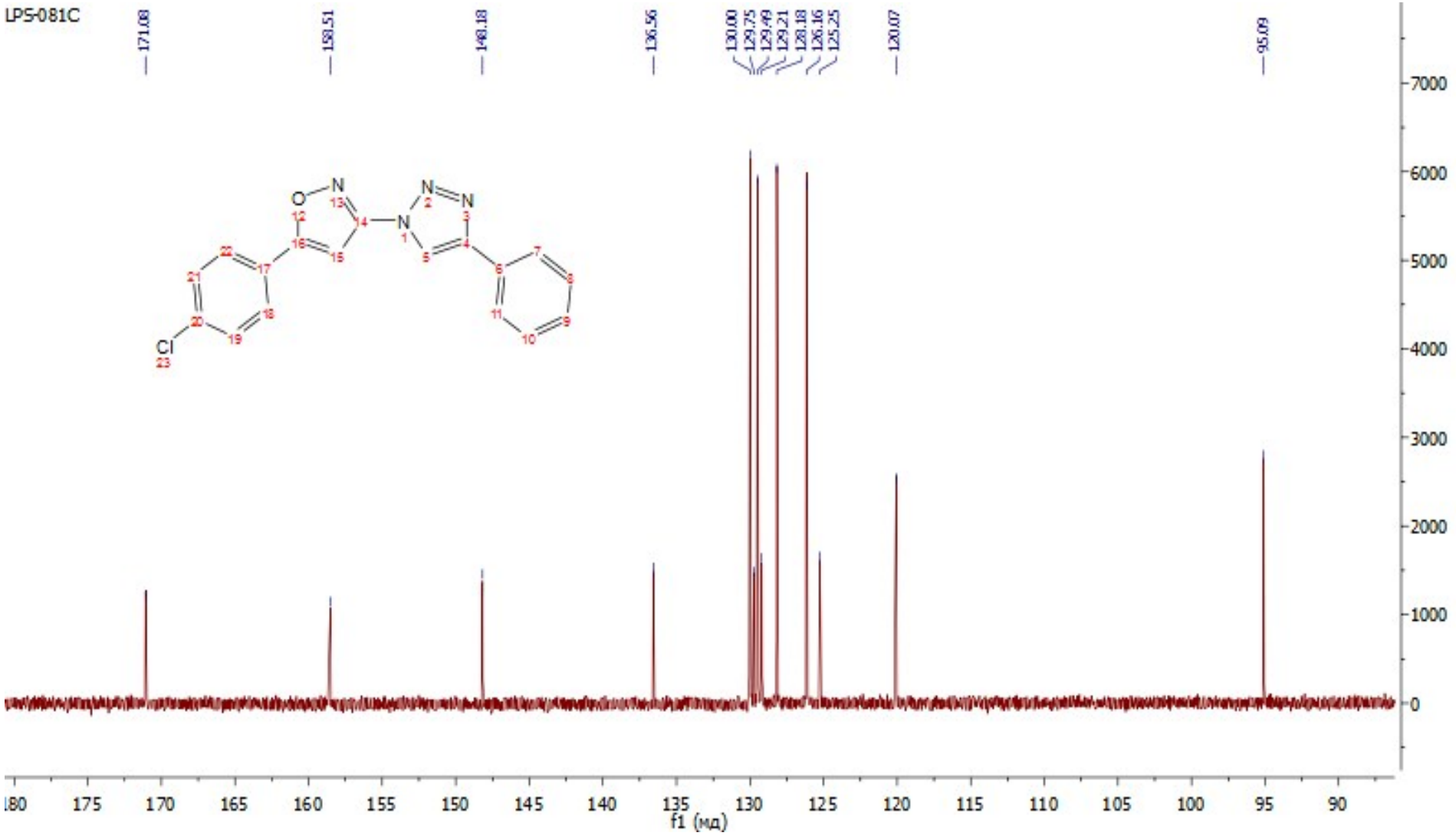
[1]H NMR (DMSO-d6): region from 9.8 to 7.2 ppm



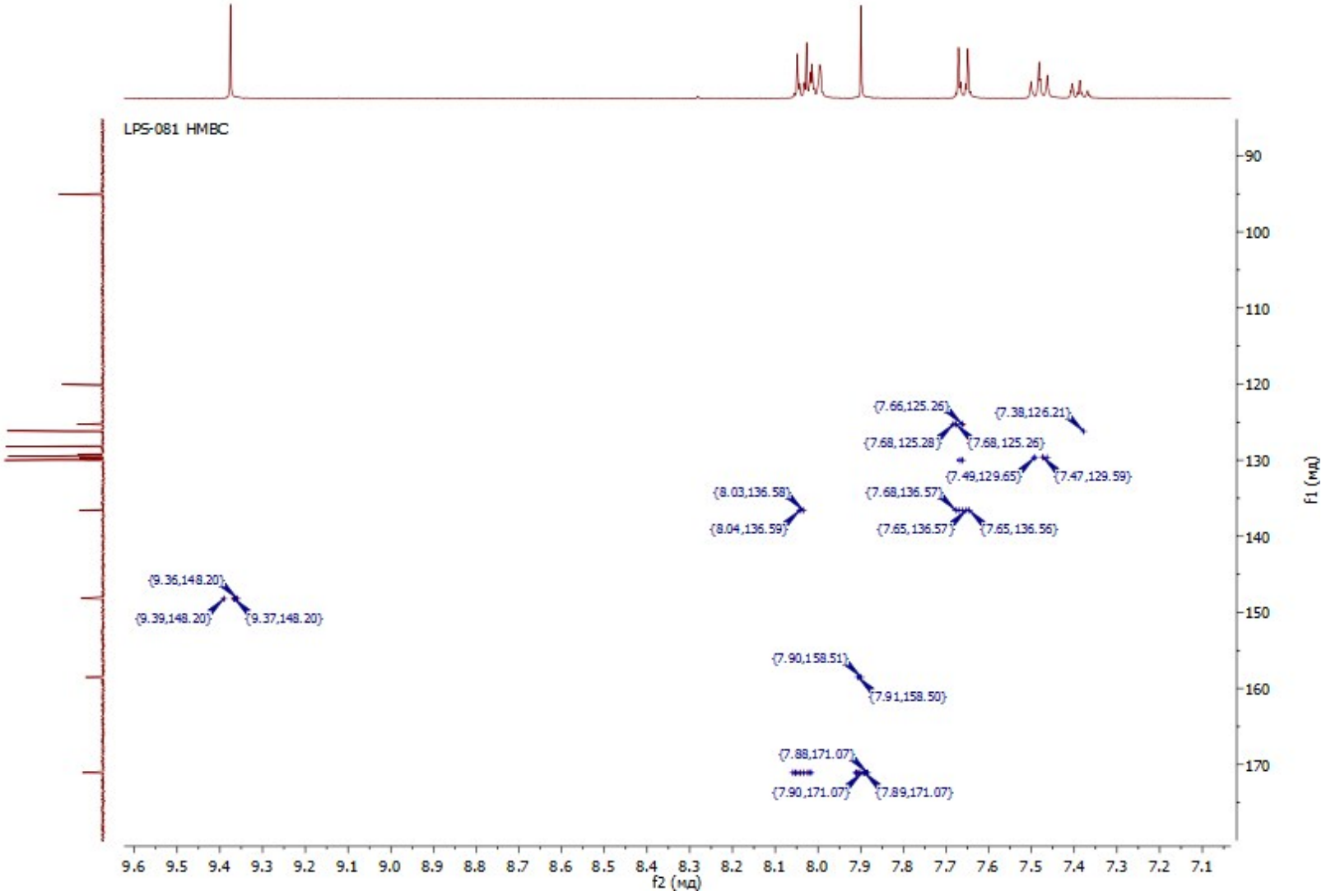
^{13}C NMR (DMSO-d_6)



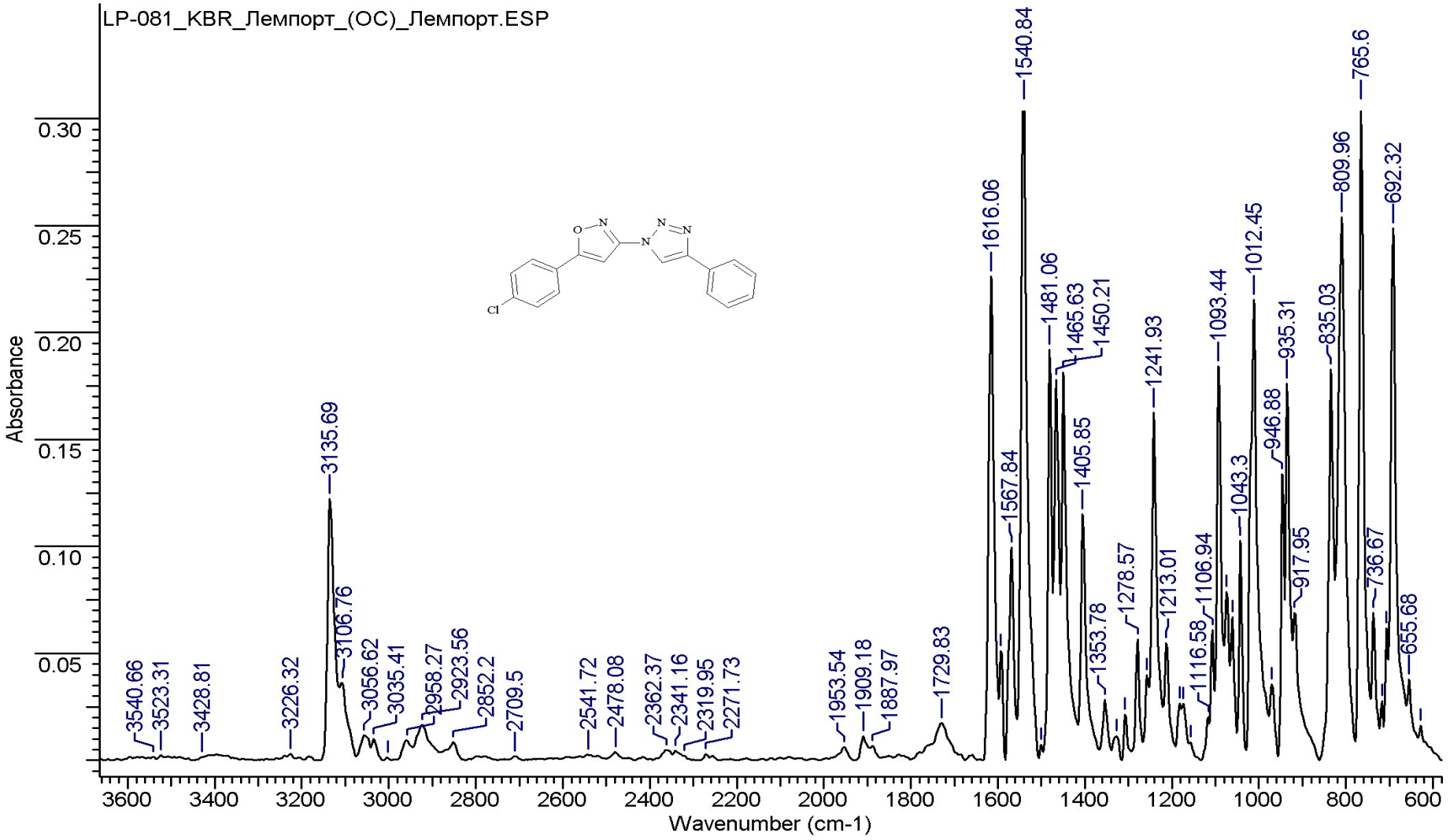
^{13}C NMR (DMSO-d_6): region from 180 to 90 ppm



$^{1}H/^{13}C$ HMBC (DMSO-d_6)

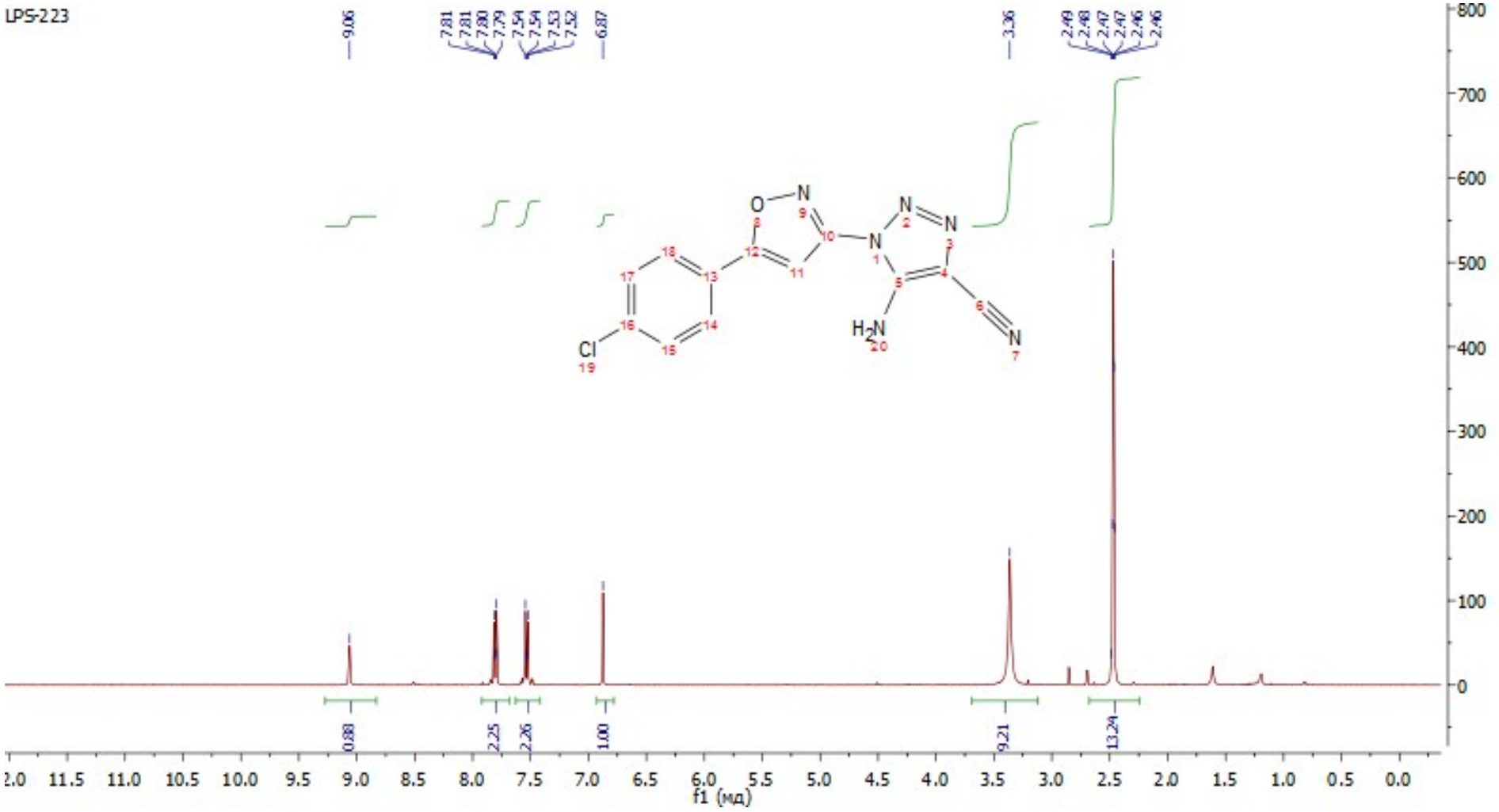


IR

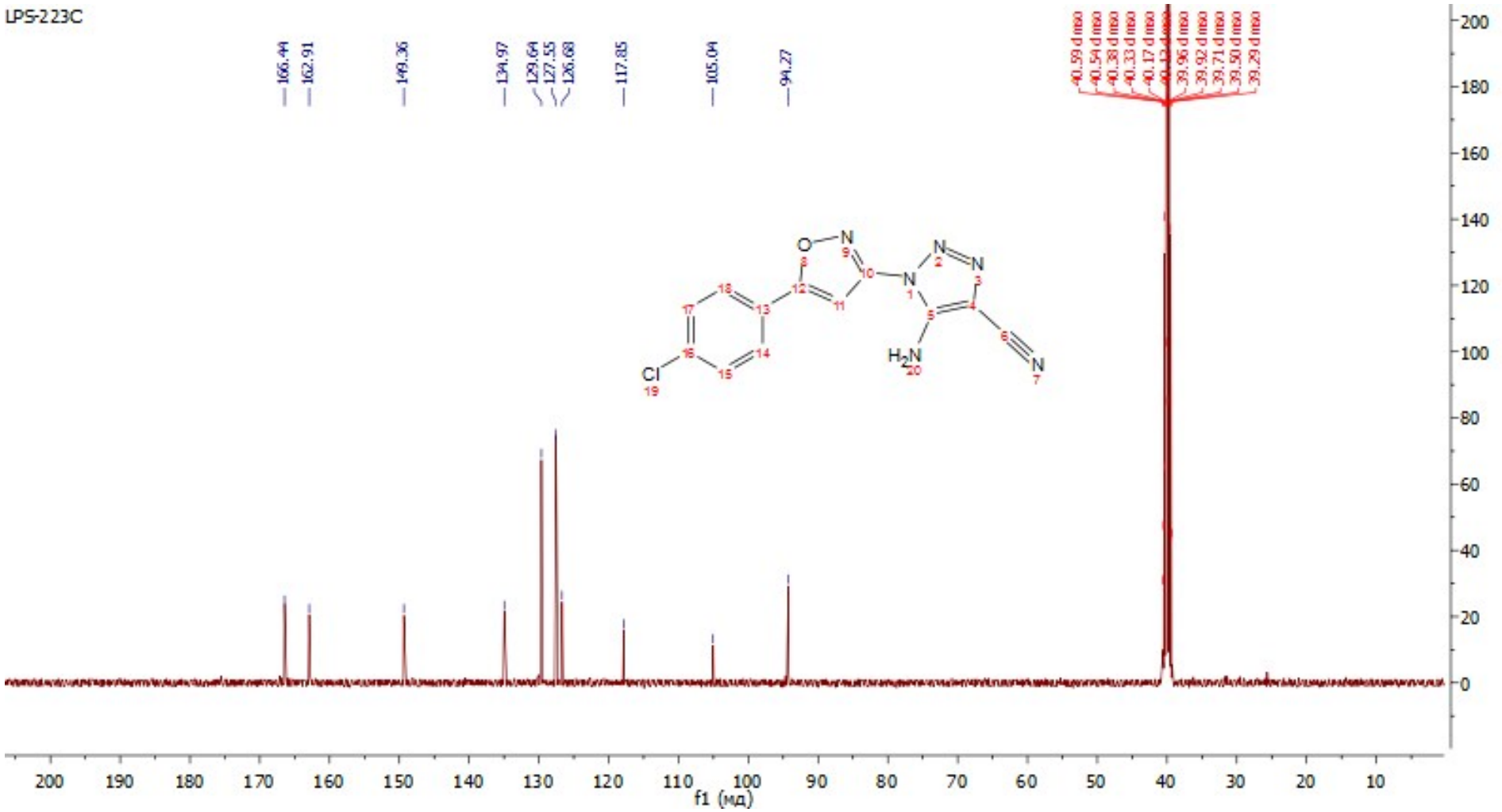

LP-081_KBR_Лемпорт_(ОС)_Лемпорт.ESP
Absorbance
Wavenumber (cm-1)
3540.66
3523.31
3428.81
3226.32
3135.69
3106.76
3056.62
3035.41
2958.27
2923.56
2852.2
2709.5
2541.72
2478.08
2362.37
2341.16
2319.95
2271.73
1953.54
1909.18
1887.97
1729.83
1616.06
1567.84
1540.84
1481.06
1465.63
1450.21
1405.85
1353.78
1278.57
1241.93
1213.01
1116.58
1106.94
1093.44
1043.3
1012.45
946.88
935.31
917.95
835.03
809.96
765.6
736.67
692.32
655.68

5-Amino-1-[5-(4-chloro-phenyl)-isoxazol-3-yl]-1H-[1,2,3]triazole-4-carbonitrile **10b**

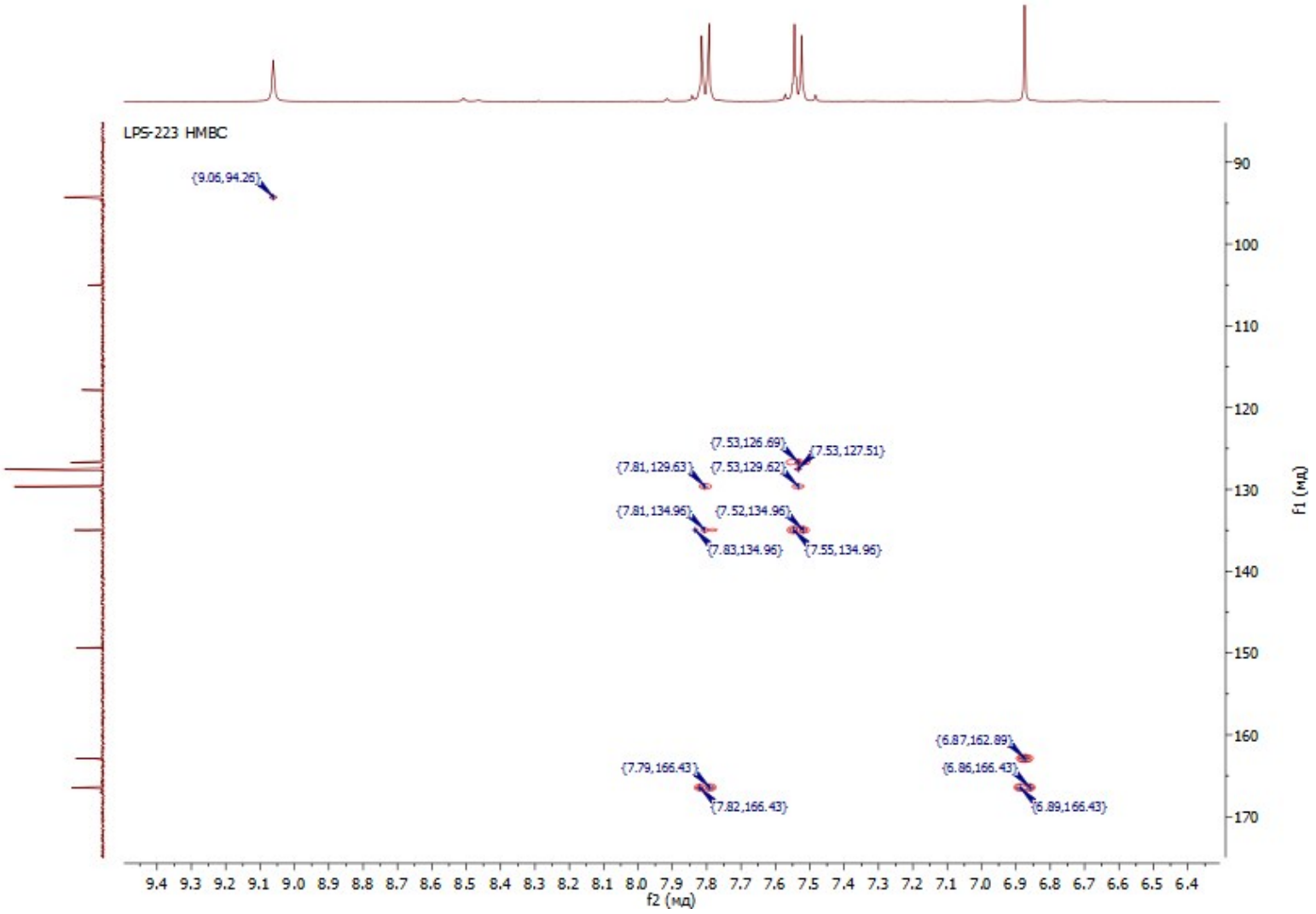
^{1}H NMR (DMSO-d_6)



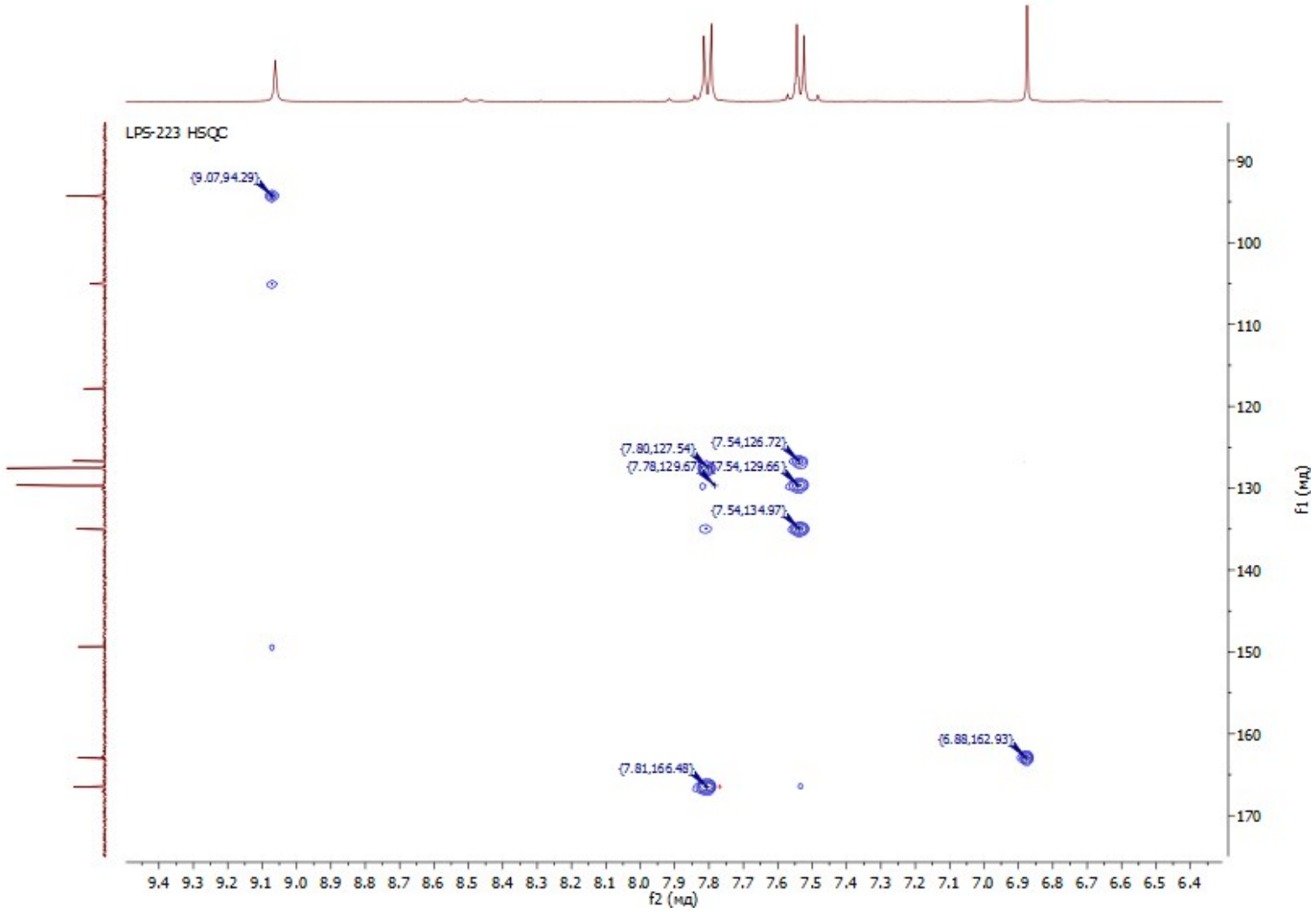
^{13}C NMR (DMSO-d_6)



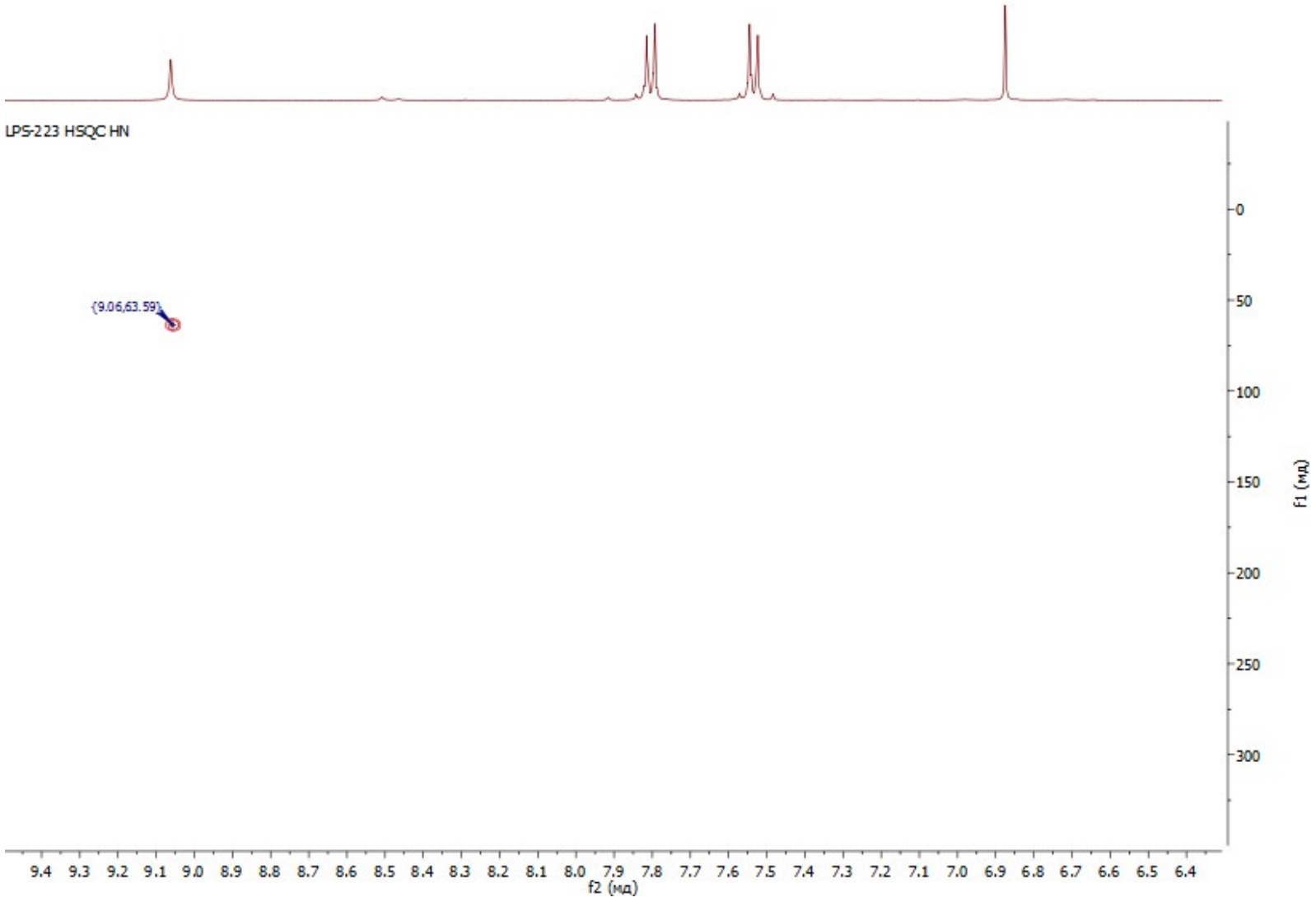
HMBC $^{1}H/^{13}C$ NMR (DMSO-d_6)



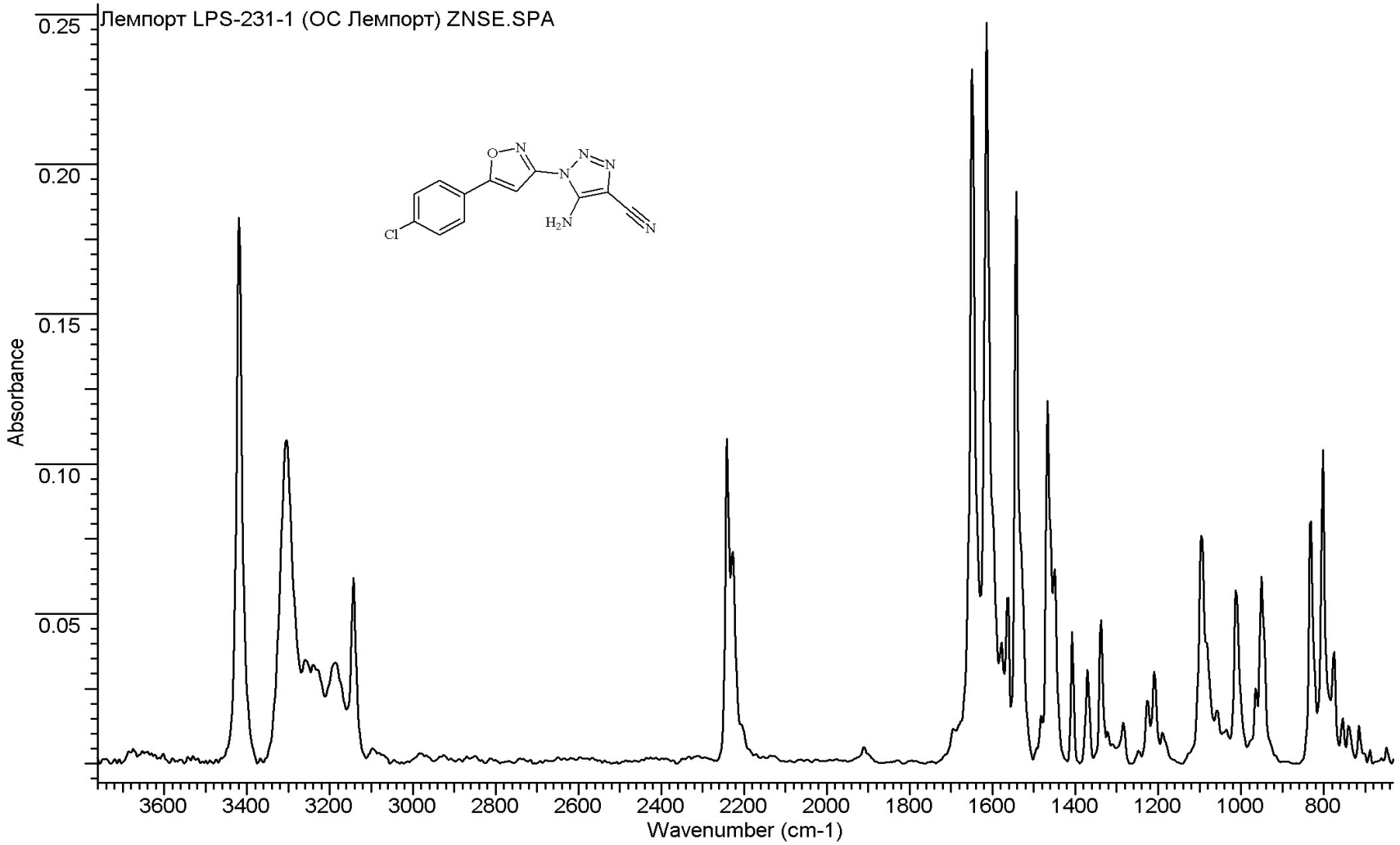
$^{1}H/^{13}C$ HSQC NMR (DMSO-d_6)



$^{1}H/^{15}N$ HSQC NMR (DMSO-d_6)

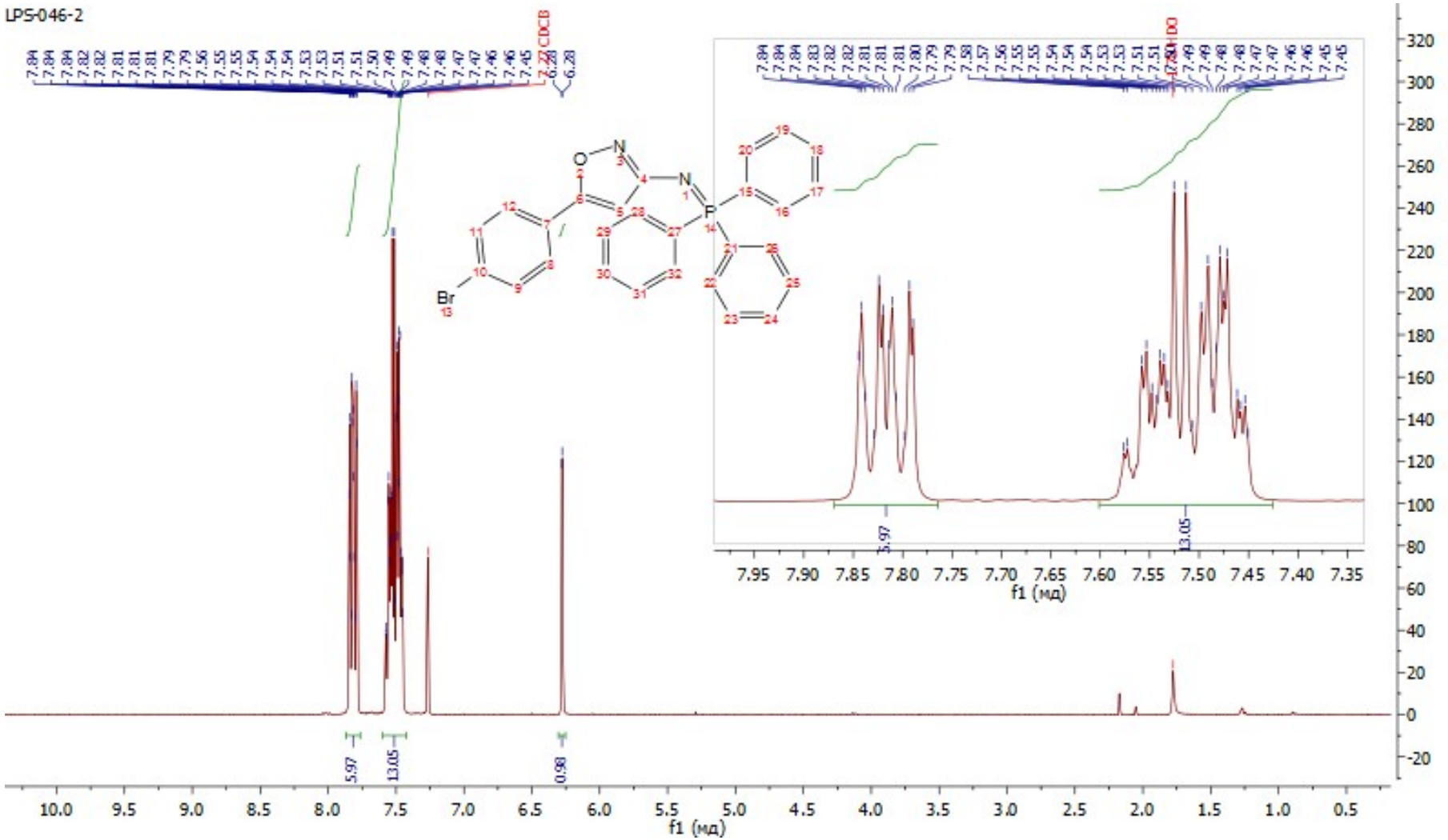


IR

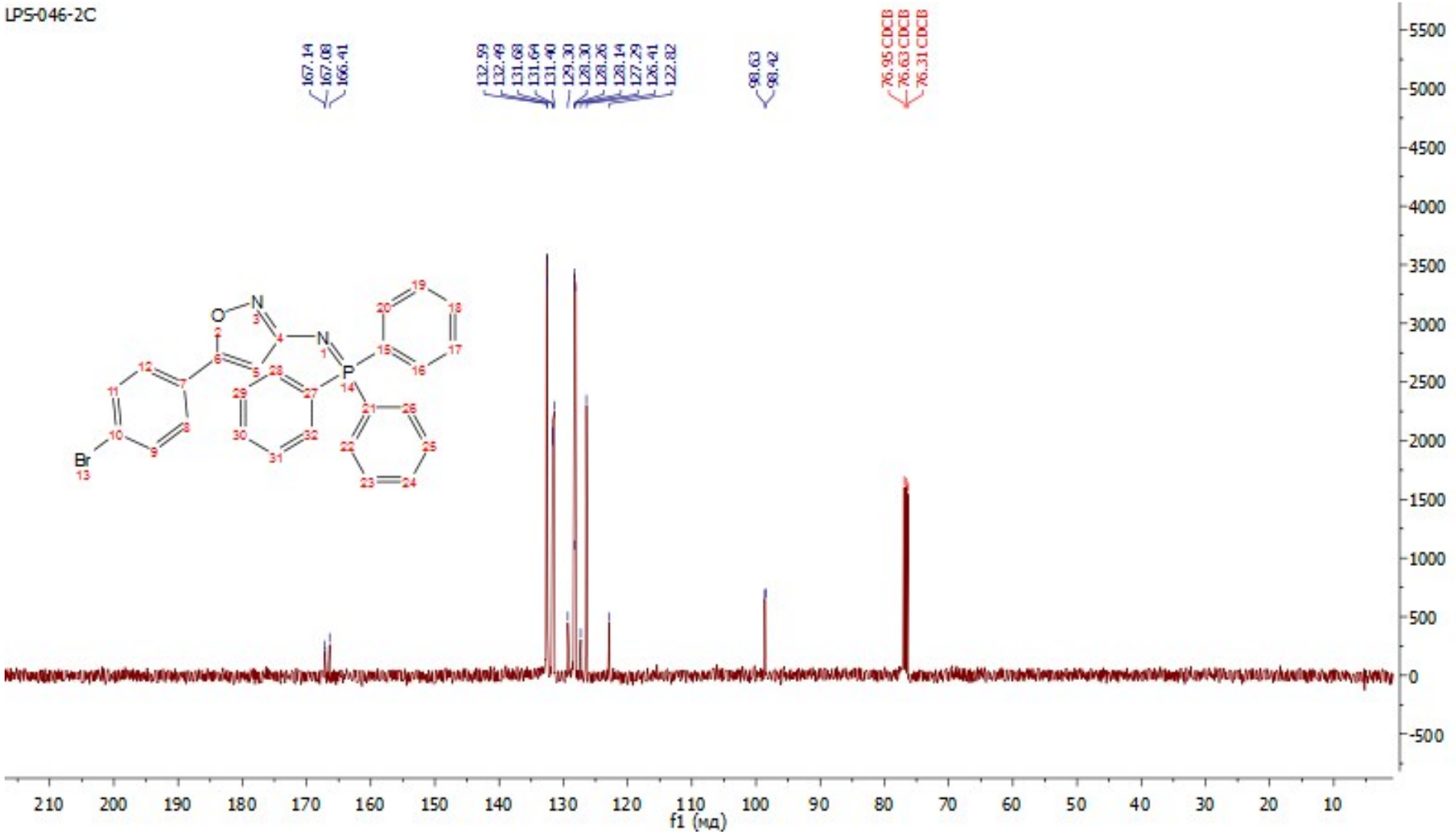

Лемпорт LPS-231-1 (ОС Лемпорт) ZNSE.SPA
Absorbance
0.25
0.20
0.15
0.10
0.05
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
Wavenumber (cm-1)
Cl
O
N
N
N
N
H2N
N

3-Imino-(triphenyl)-phosphorano-5-(4-bromophenyl)-isoxazole **11a**

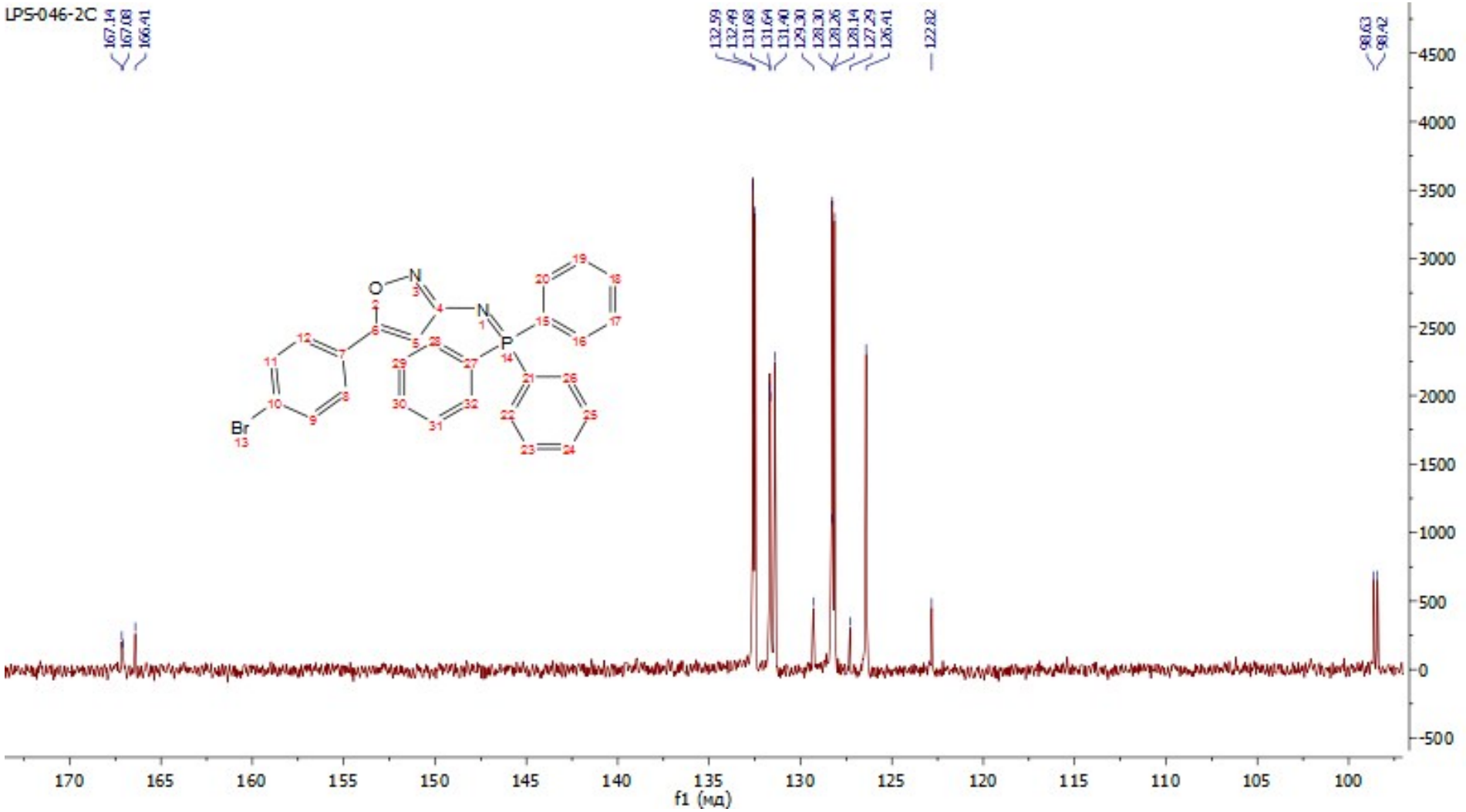
^{1}H NMR ($CDCl_3$)



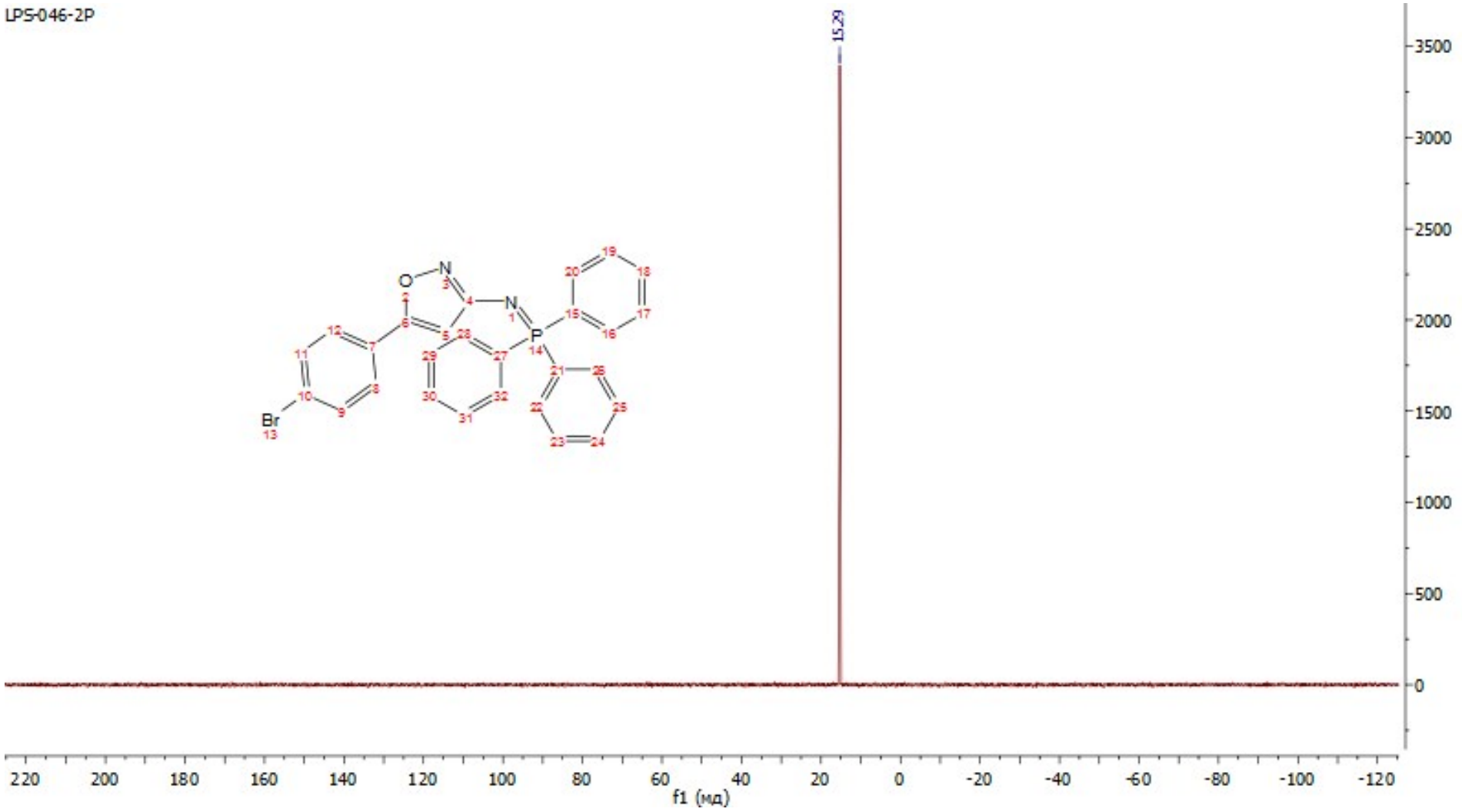
^{13}C NMR ($CDCl_3$)



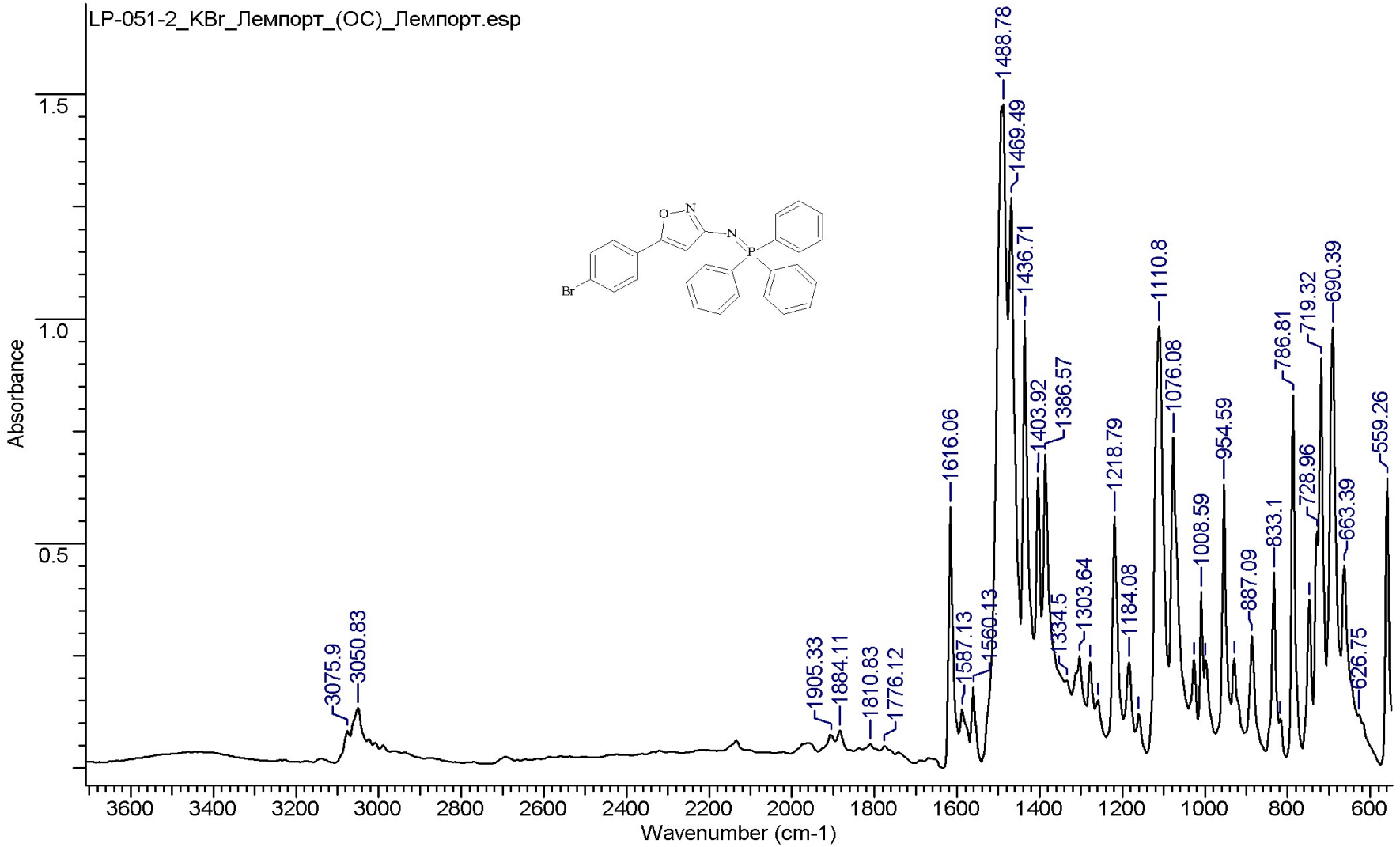
^{13}C NMR ($CDCl_3$): region from 170 to 95 ppm



^{31}P NMR ($CDCl_3$)



IR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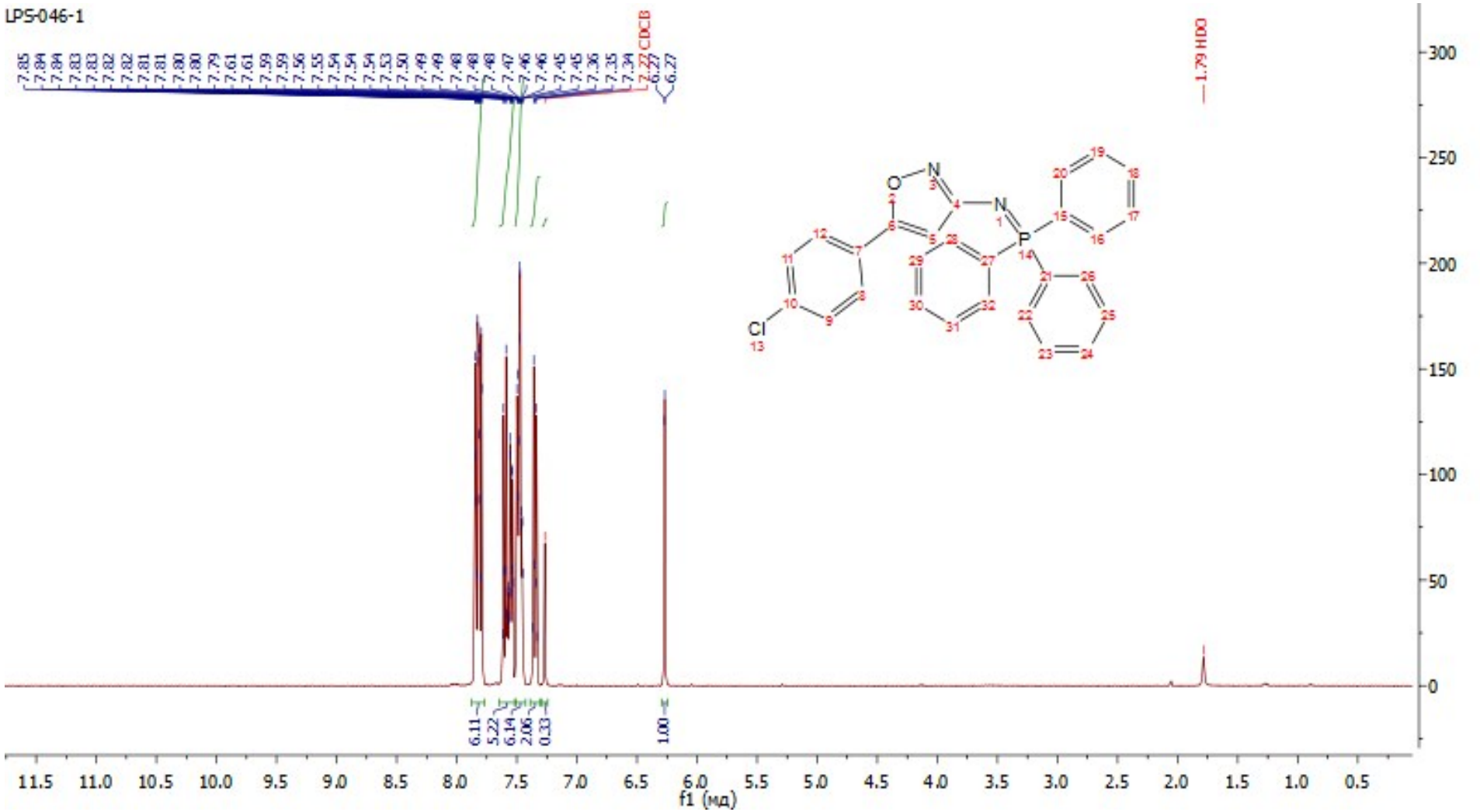
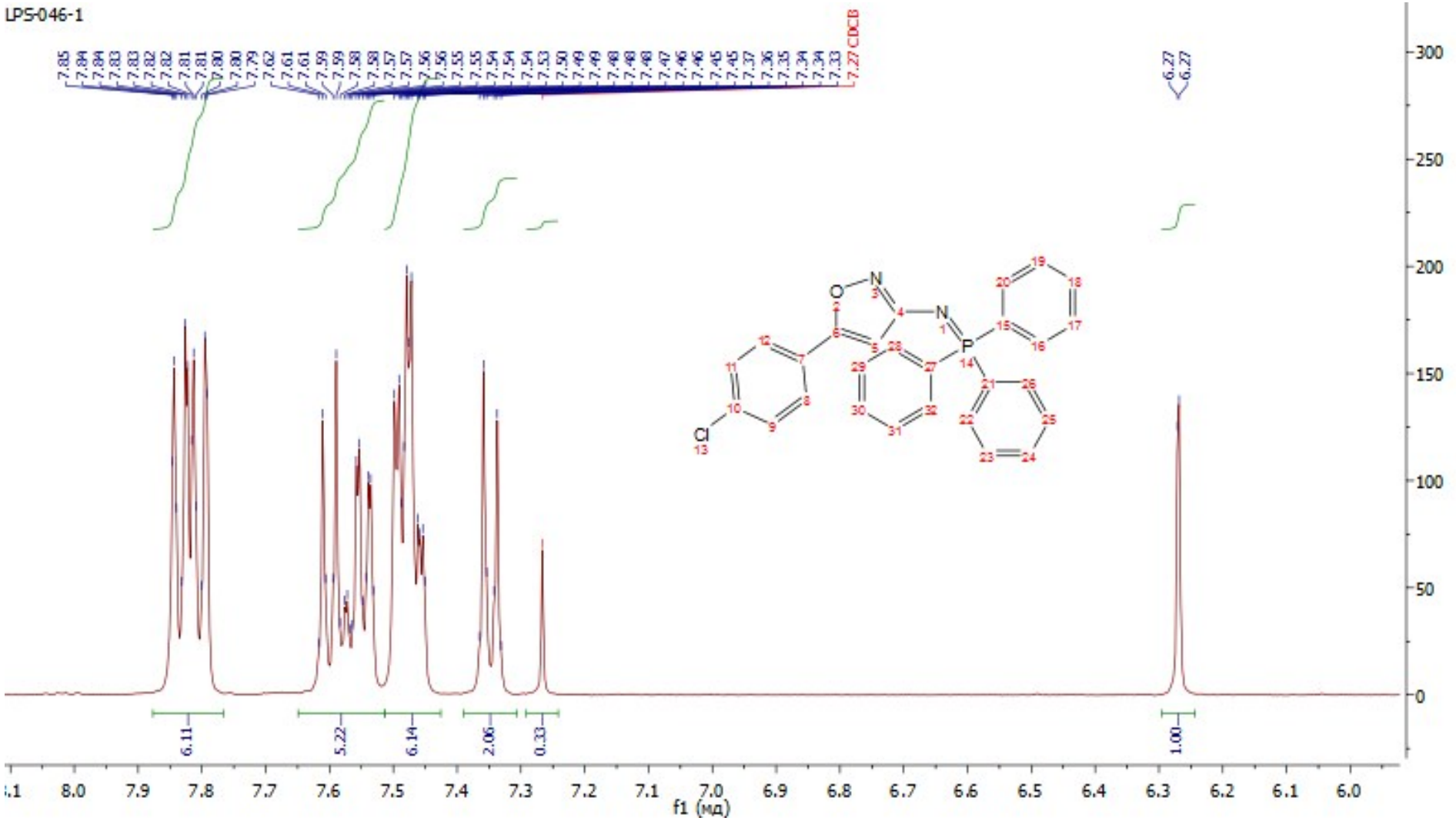
1200
1000
800
600
Wavenumber (cm-1)
3075.9
3050.83
1905.33
1884.11
1810.83
1776.12
1616.06
1587.13
1560.13
1488.78
1469.49
1436.71
1403.92
1386.57
1334.5
1303.64
1218.79
1184.08
1110.8
1076.08
1008.59
954.59
887.09
833.1
786.81
728.96
719.32
690.39
663.39
626.75
559.26
Br
O
N
N
P

3-Imino-(triphenyl)-phosphorano-5-(4-chlorophenyl)-isoxazole **11b**

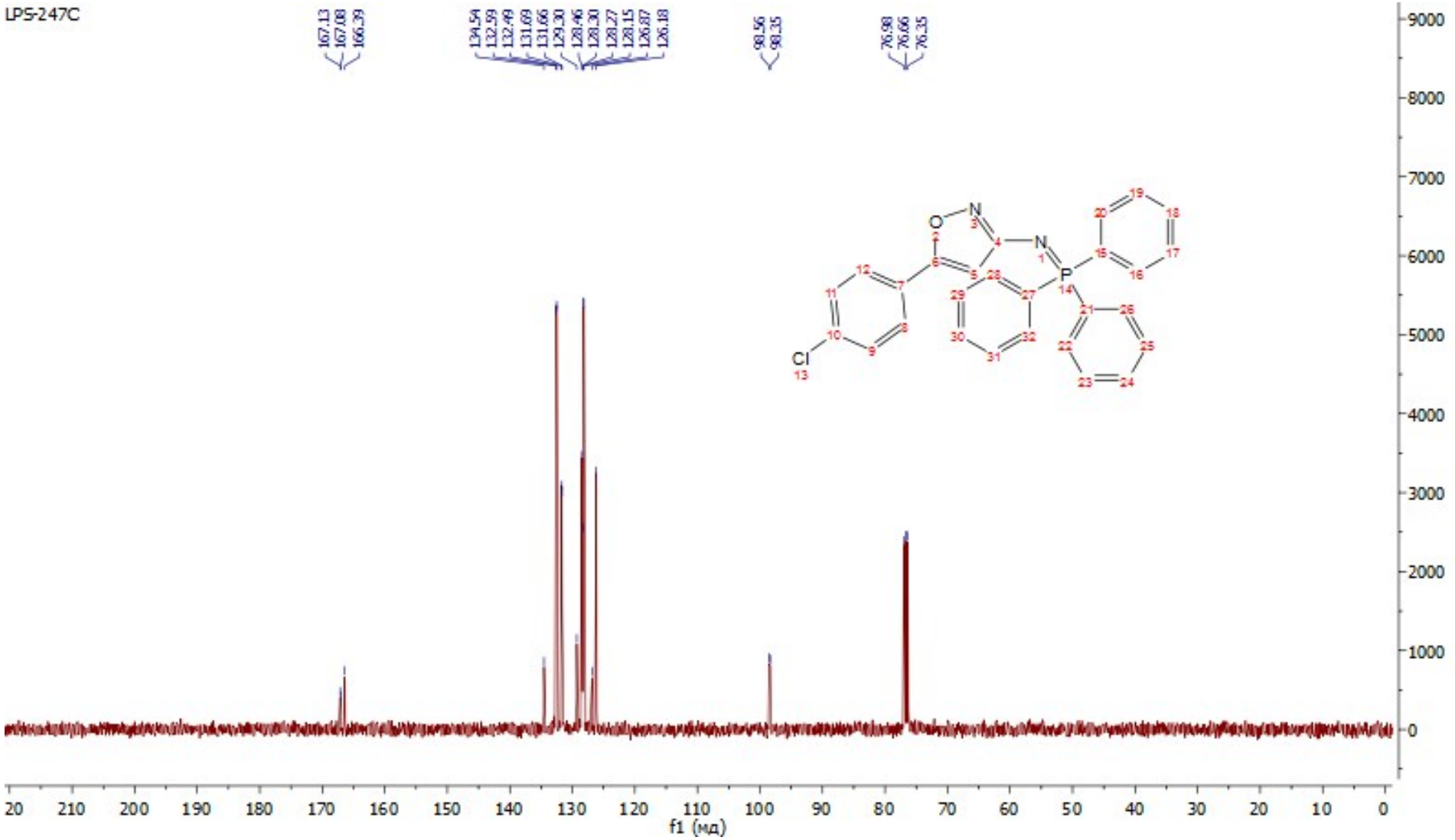
^{1}H NMR ($CDCl_3$)



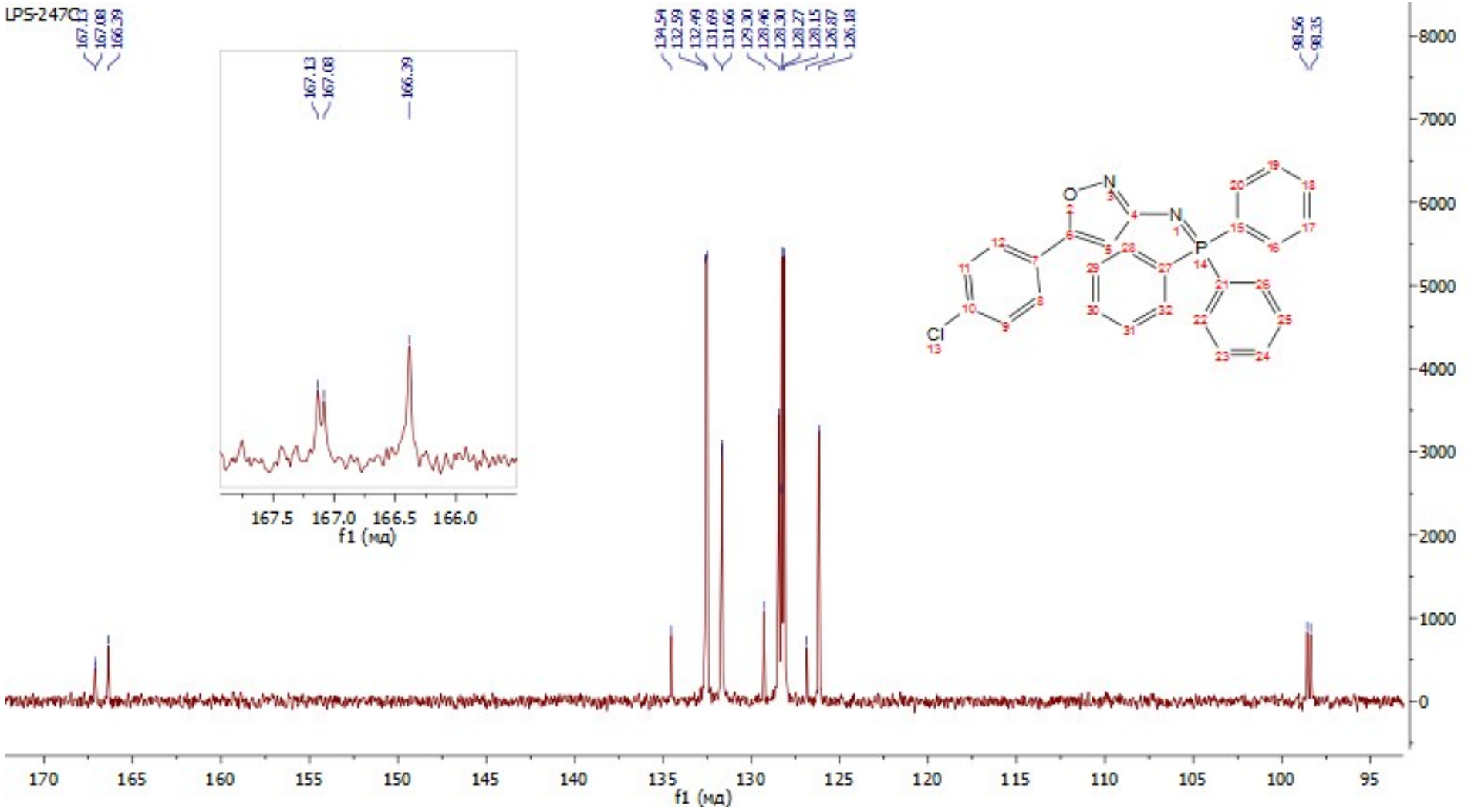
^{1}H NMR ($CDCl_3$): region from 8.0 to 6.0 ppm



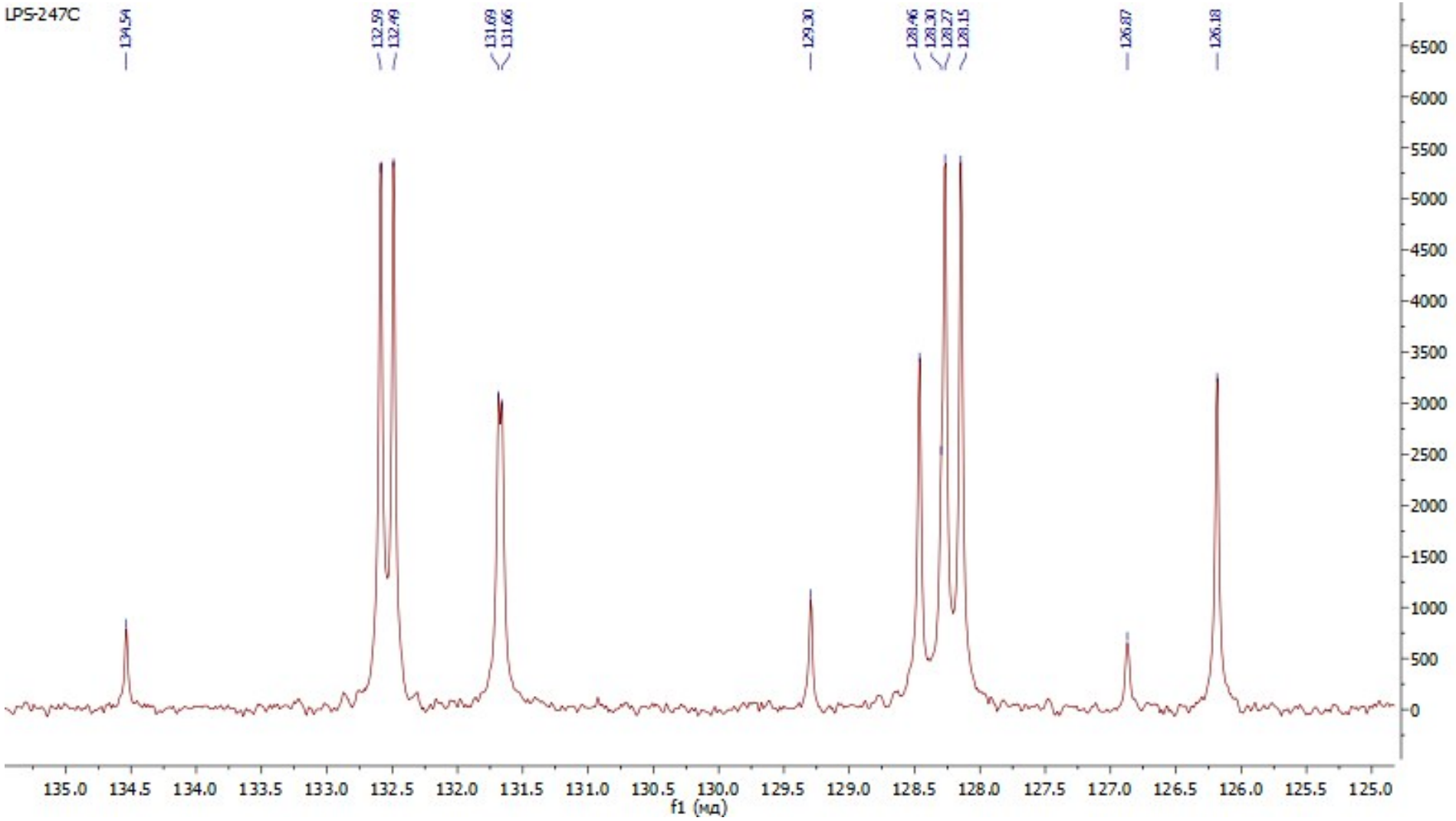
^{13}C NMR ($CDCl_3$)



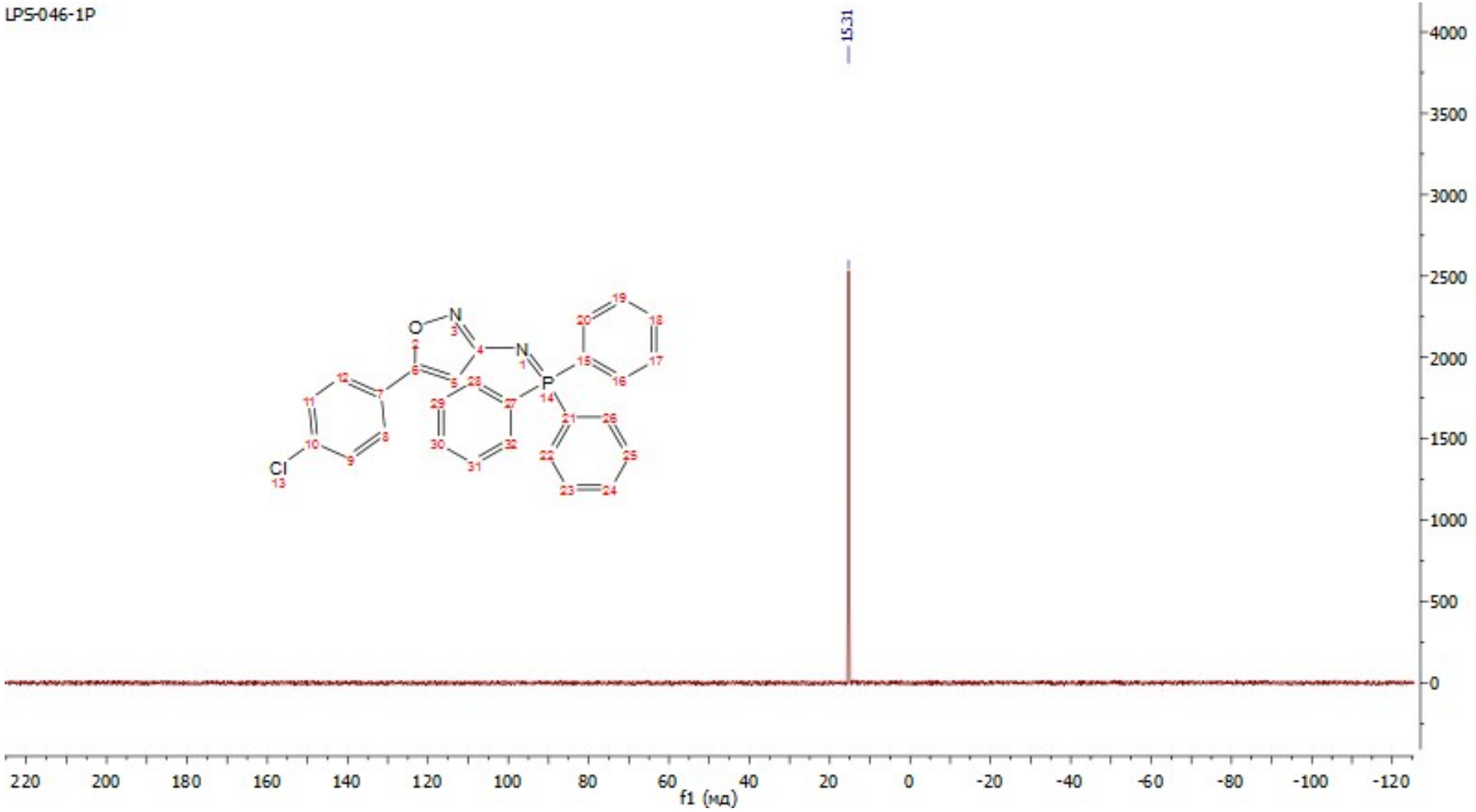
^{13}C NMR ($CDCl_3$): region from 170 to 95 ppm



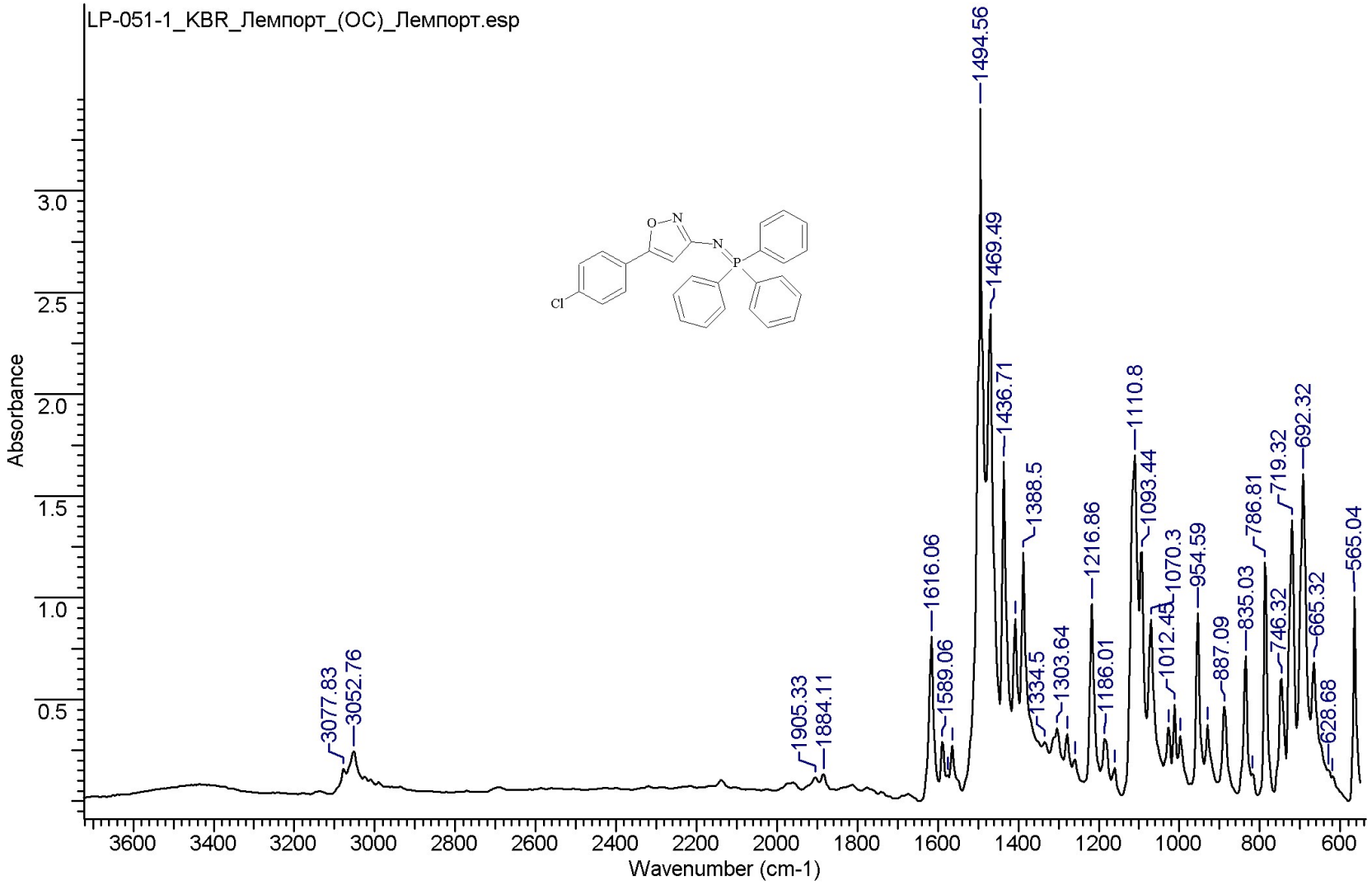
^{13}C NMR ($CDCl_3$): region from 135 to 125 ppm



^{31}P NMR ($CDCl_3$)



IR


LP-051-1_KBR_Лемпорт_(ОС)_Лемпорт.esp
Absorbance
Wavenumber (cm-1)
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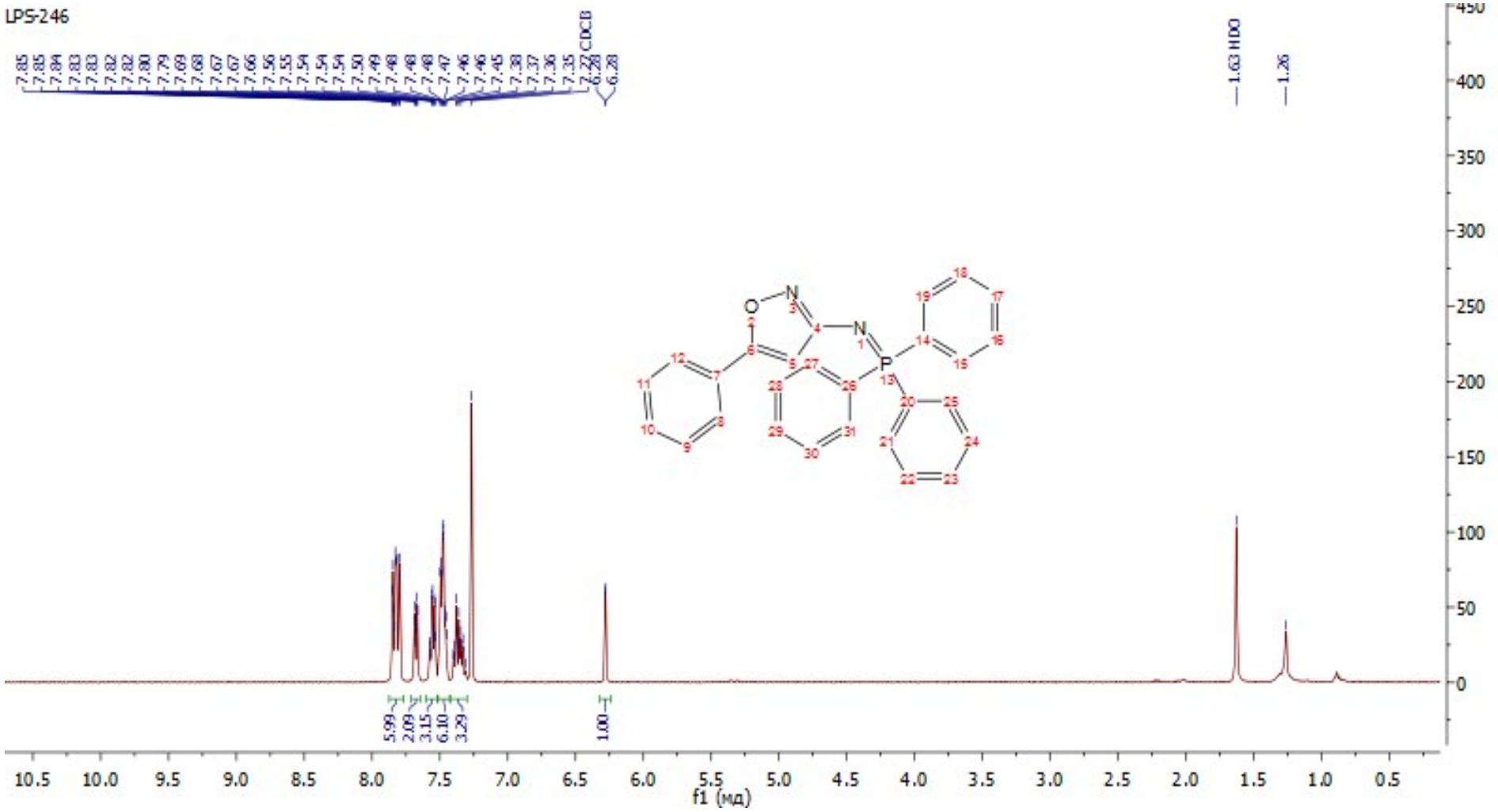
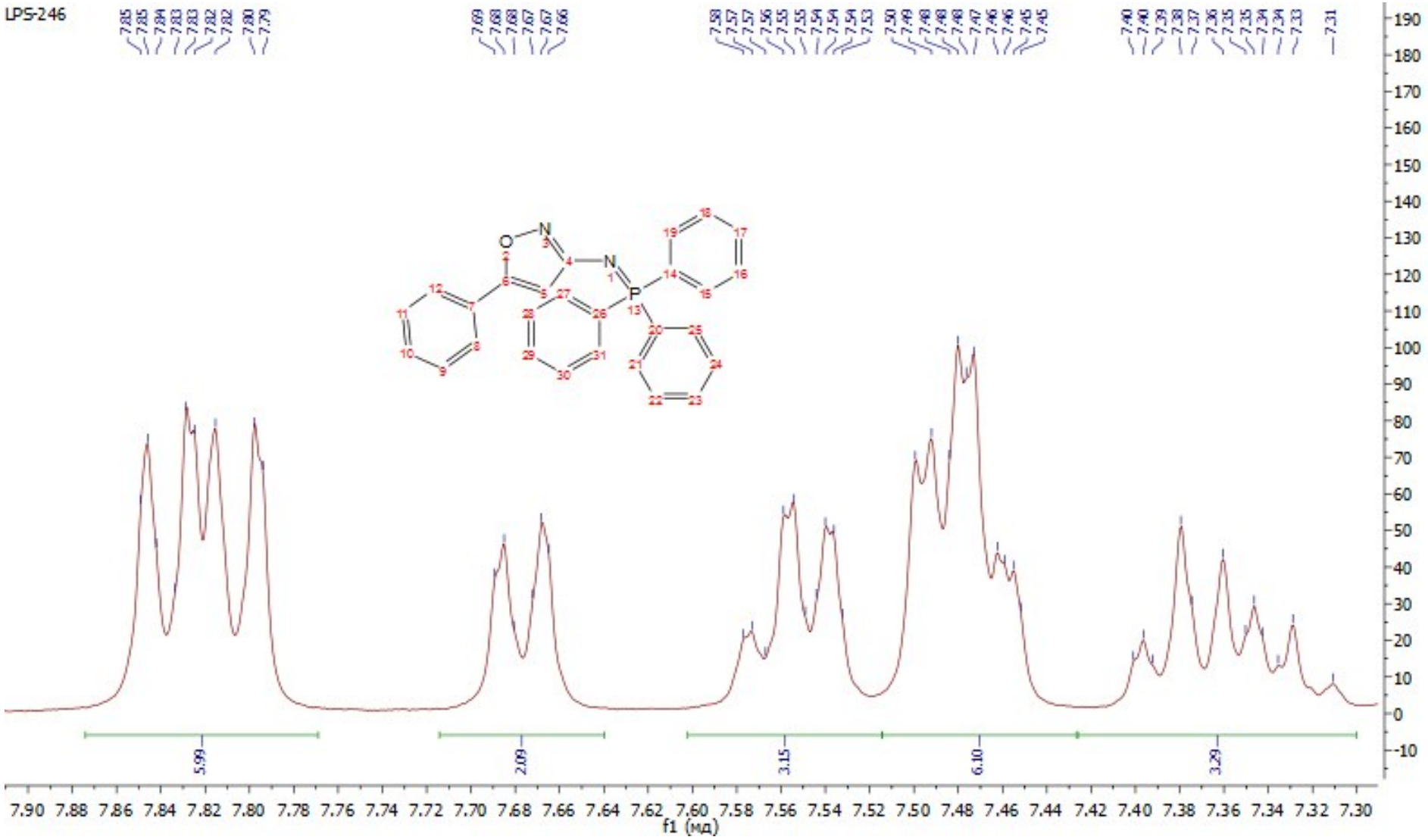
3052.76
1905.33
1884.11
1616.06
1589.06
1494.56
1469.49
1436.71
1388.5
1334.5
1303.64
1216.86
1186.01
1110.8
1093.44
1070.3
1012.45
954.59
887.09
835.03
786.81
746.32
719.32
692.32
665.32
628.68
565.04
O
N
N
P
Cl

3-Imino-(triphenyl)-phosphorano-5-phenylisoxazole **11c**

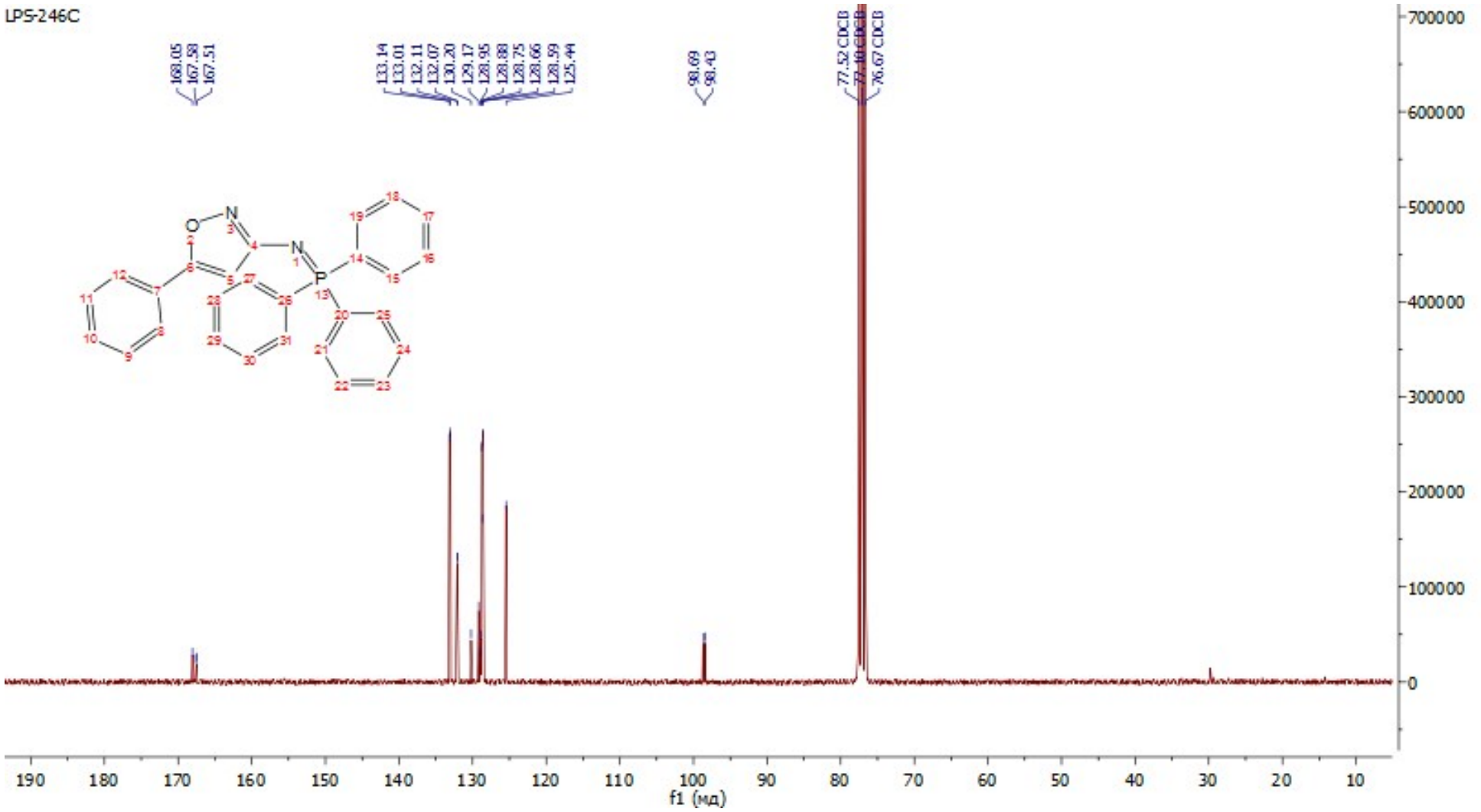
^{1}H NMR ($CDCl_3$)



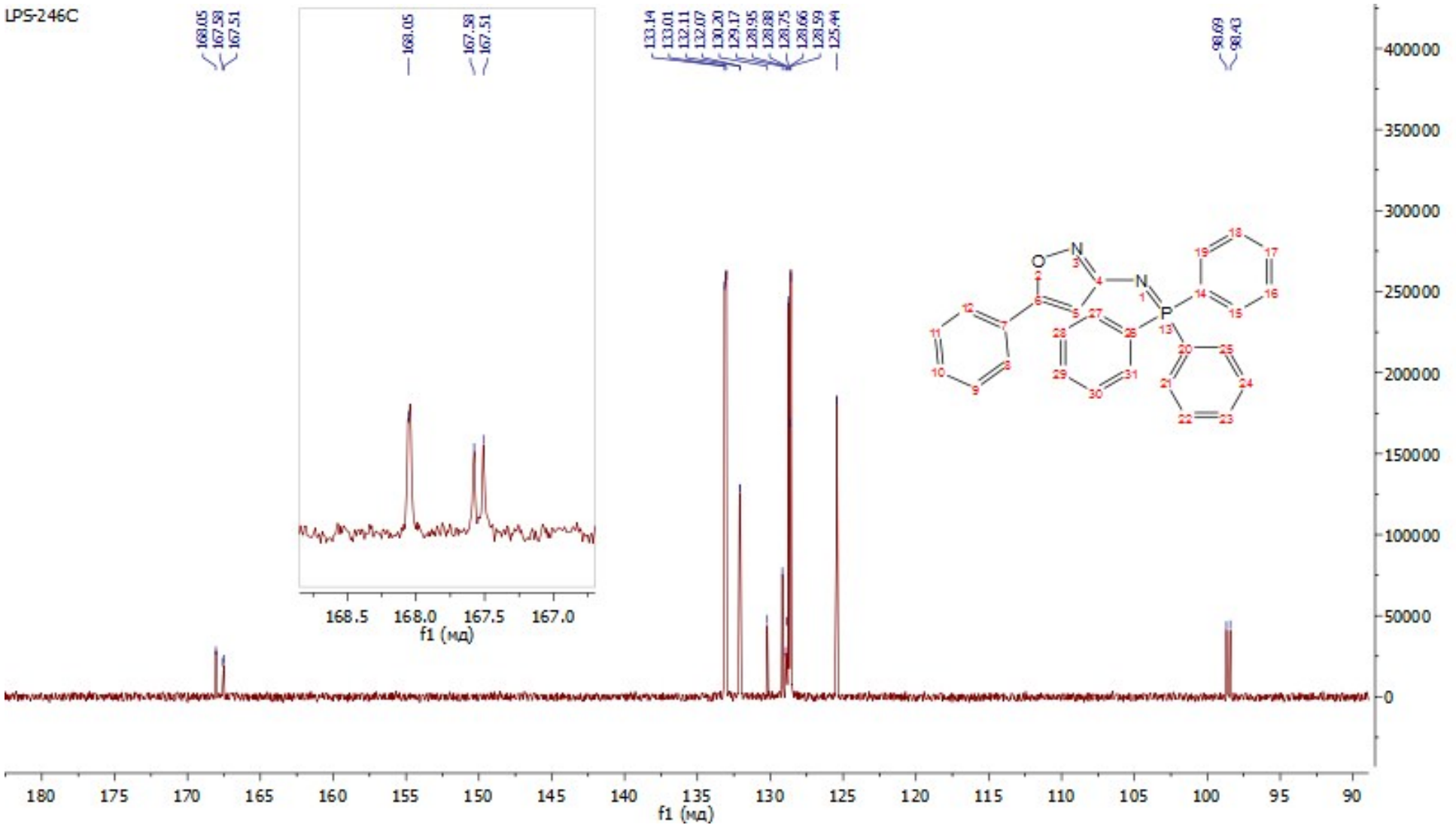
^{1}H NMR ($CDCl_3$): region from 7.90 to 7.30 ppm



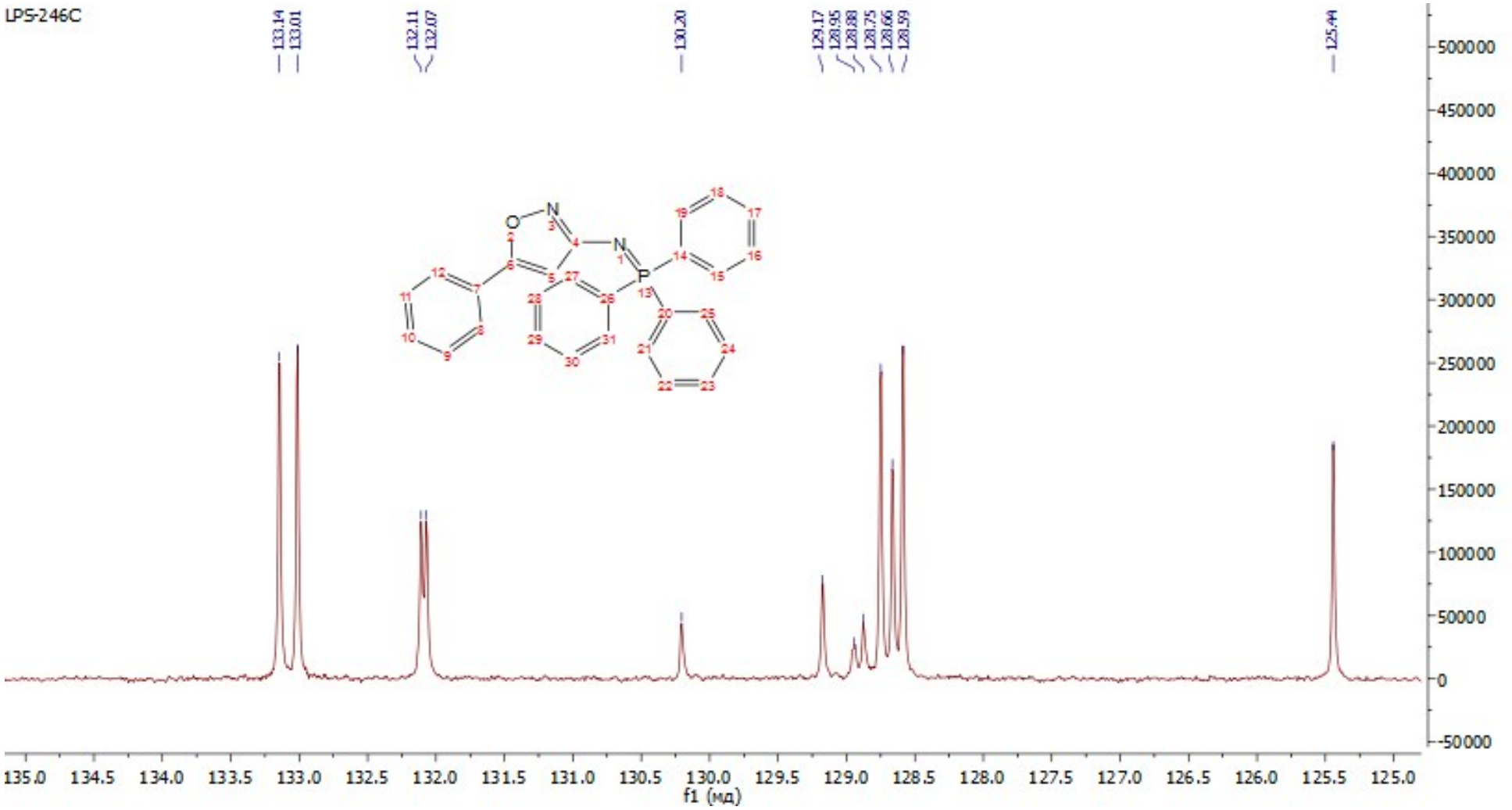
^{13}C NMR ($CDCl_3$)



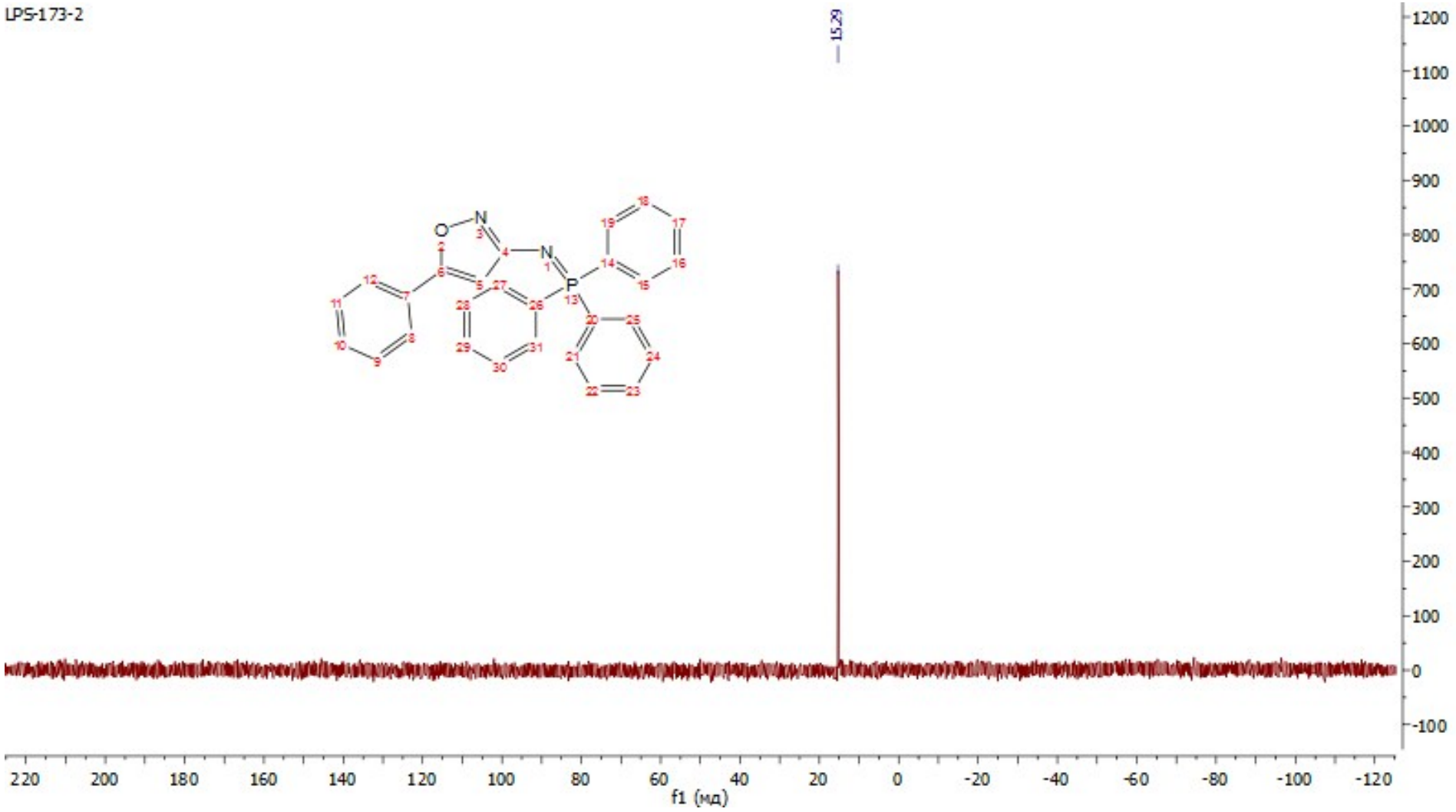
^{13}C NMR ($CDCl_3$): region from 180 to 90 ppm



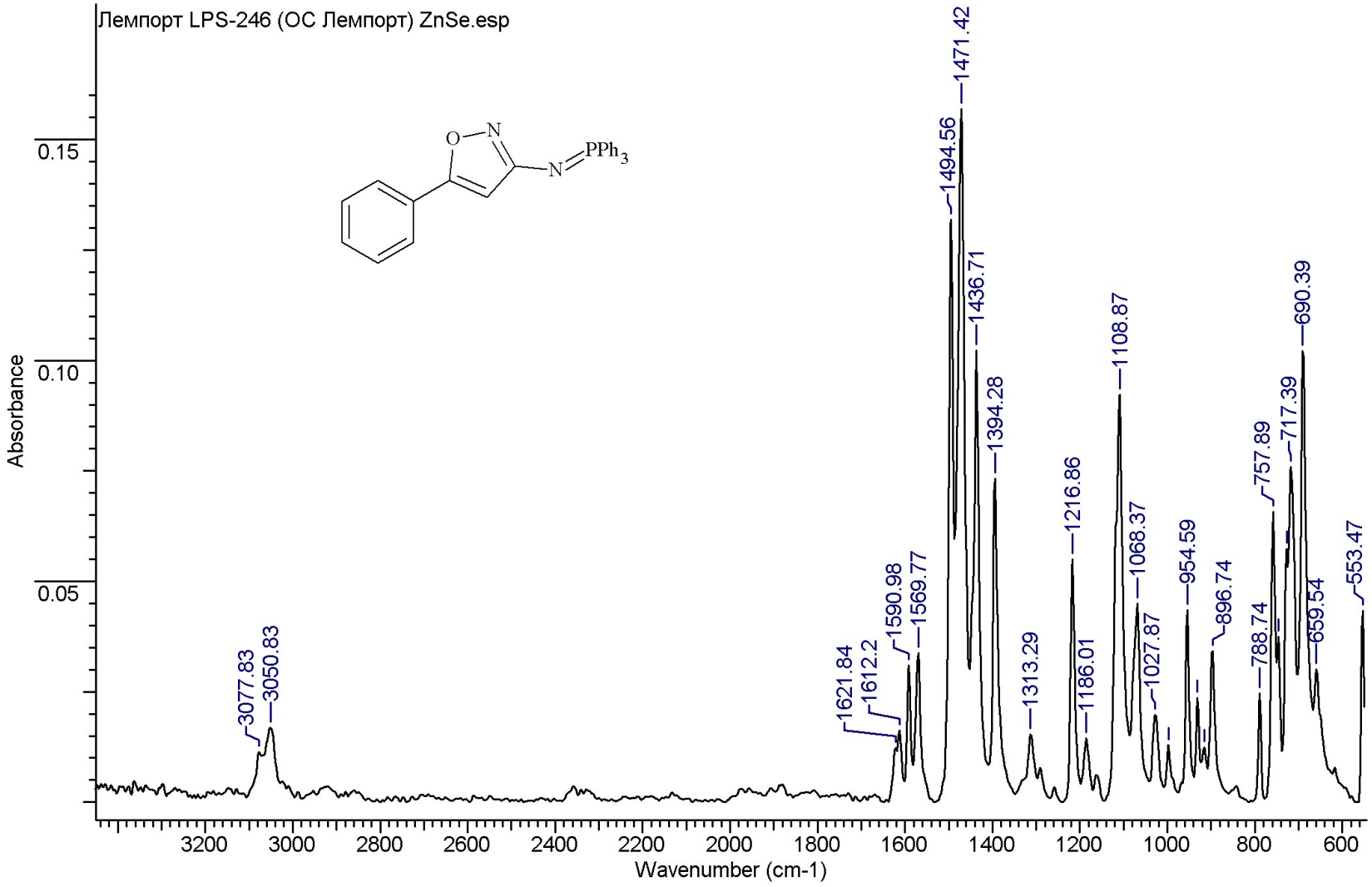
^{13}C NMR ($CDCl_3$): region from 125 to 135 ppm



^{31}P NMR ($CDCl_3$)



IR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2800
2600
2400
2200
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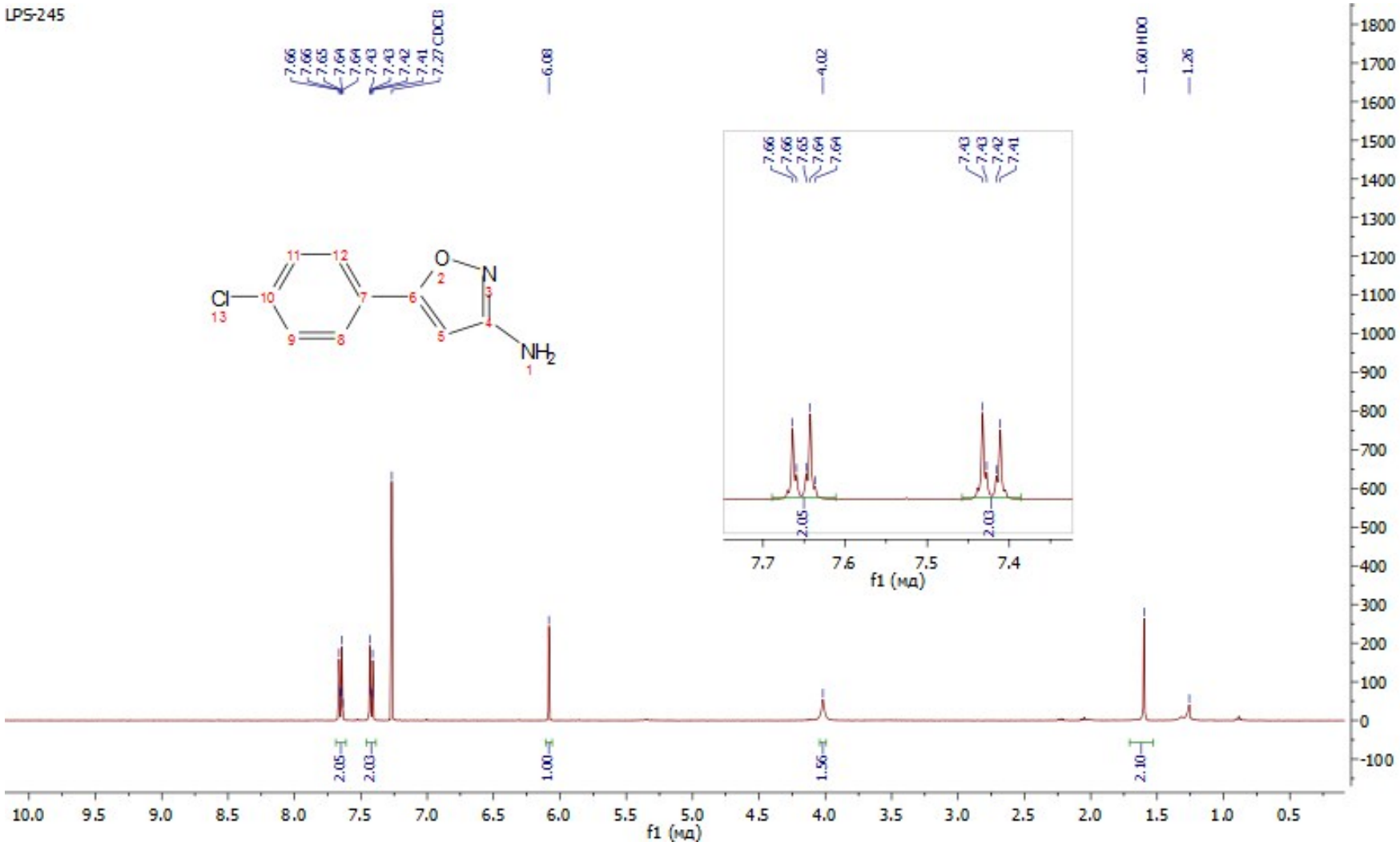
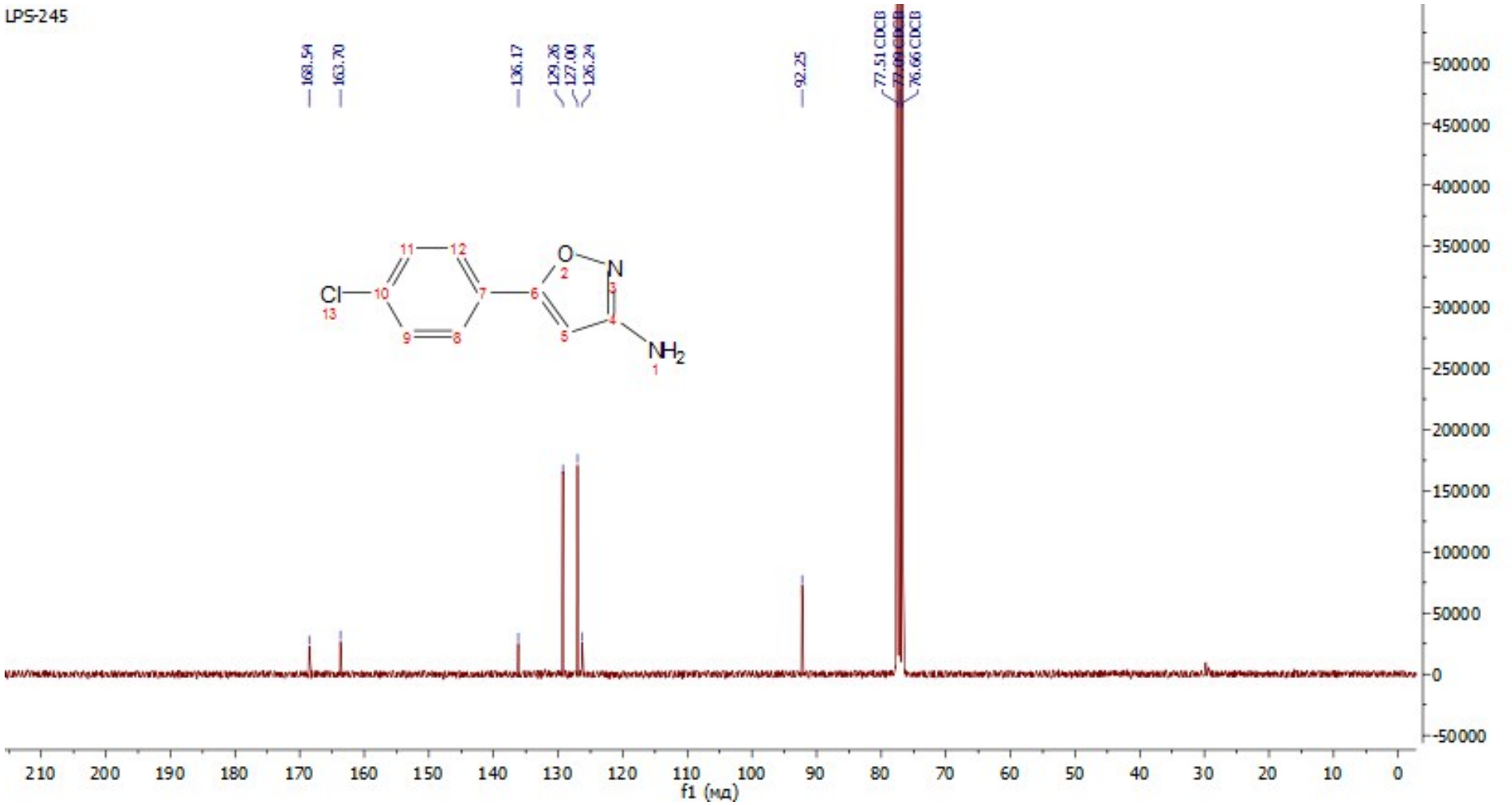
1800
1600
1400
1200
1000
800
600
Wavenumber (cm-1)
3077.83
3050.83
1621.84
1612.2
1590.98
1569.77
1494.56
1471.42
1436.71
1394.28
1313.29
1216.86
1186.01
1108.87
1068.37
1027.87
954.59
896.74
788.74
757.89
717.39
690.39
659.54
553.47
O
N
N
PPh3

5-(4-Chloro-phenyl)-isoxazol-3-ylamine **12b**

^{1}H NMR ($CDCl_3$)

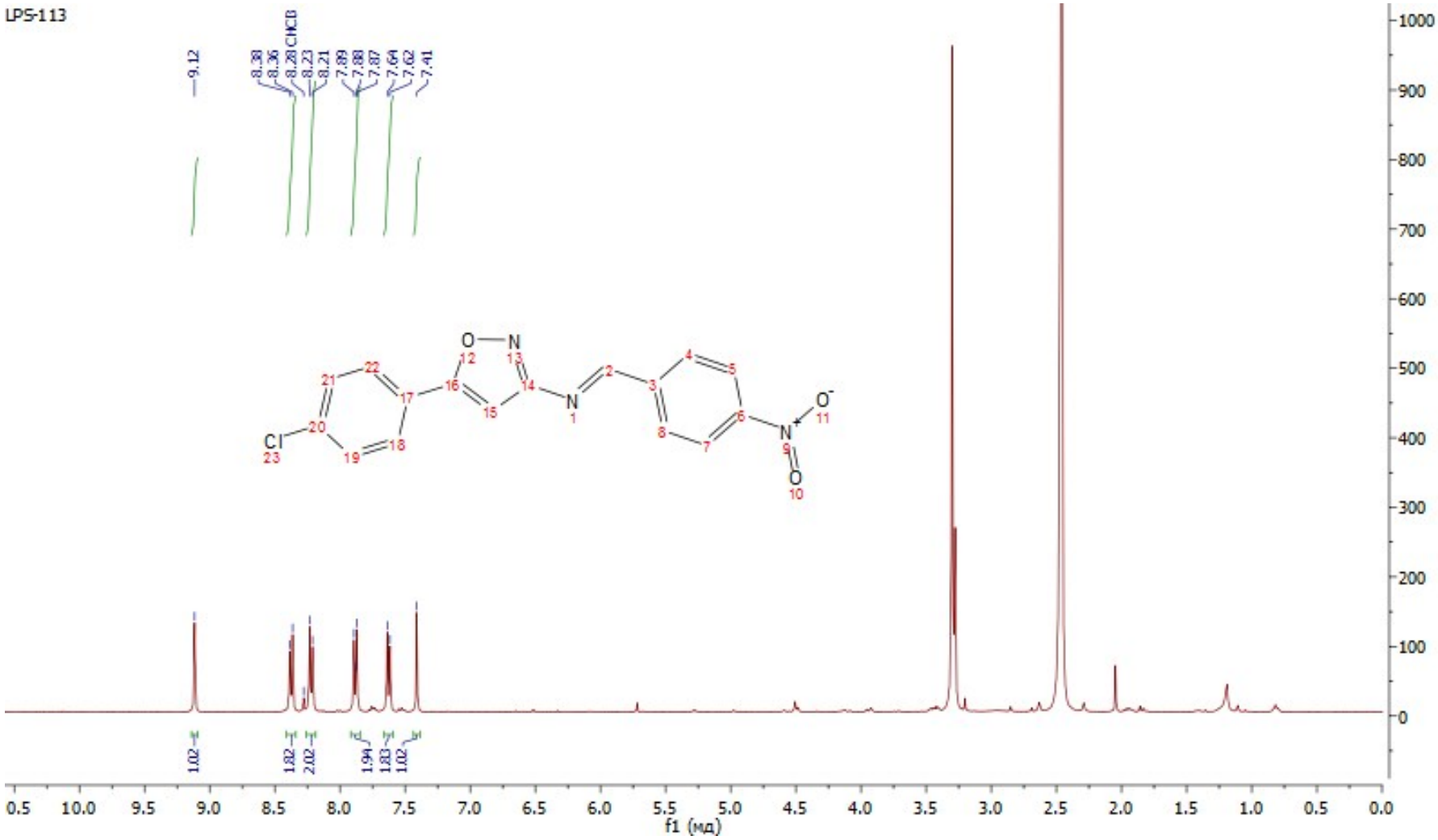


^{13}C NMR ($CDCl_3$)

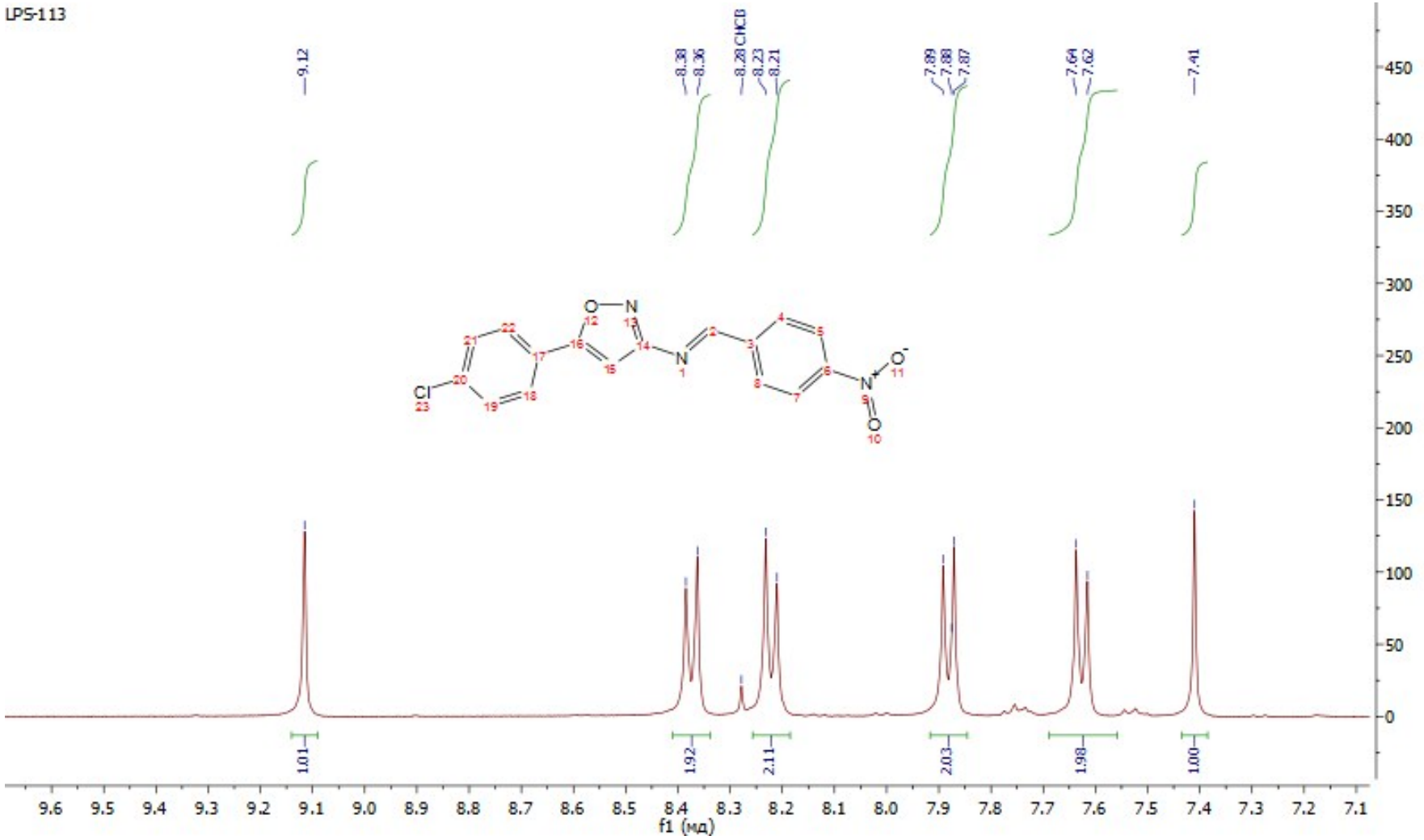


[5-(4-Chloro-phenyl)-isoxazol-3-yl]-(4-nitro-benzylidene)-amine **13b**

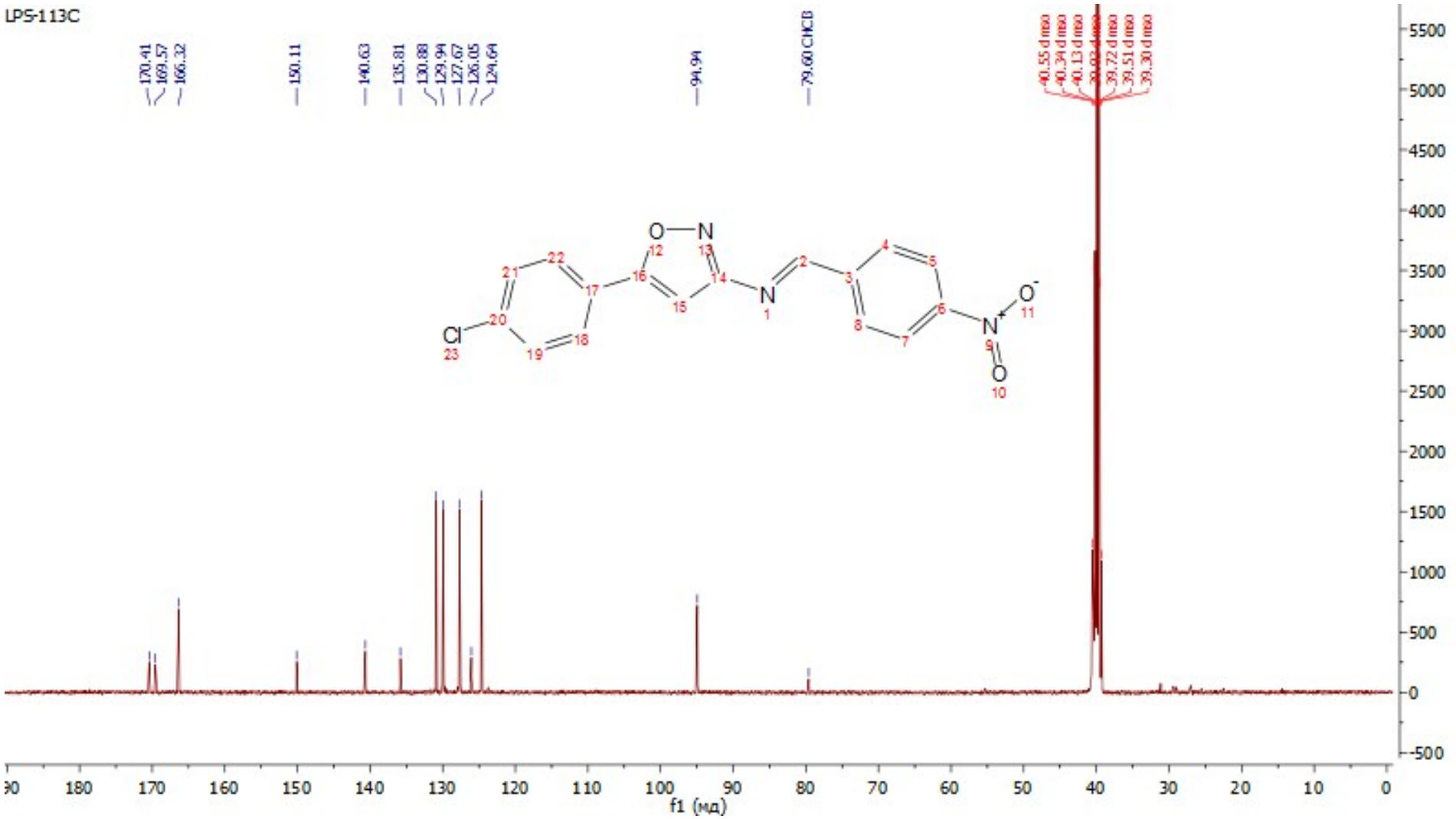
^{1}H NMR (DMSO-d_6)



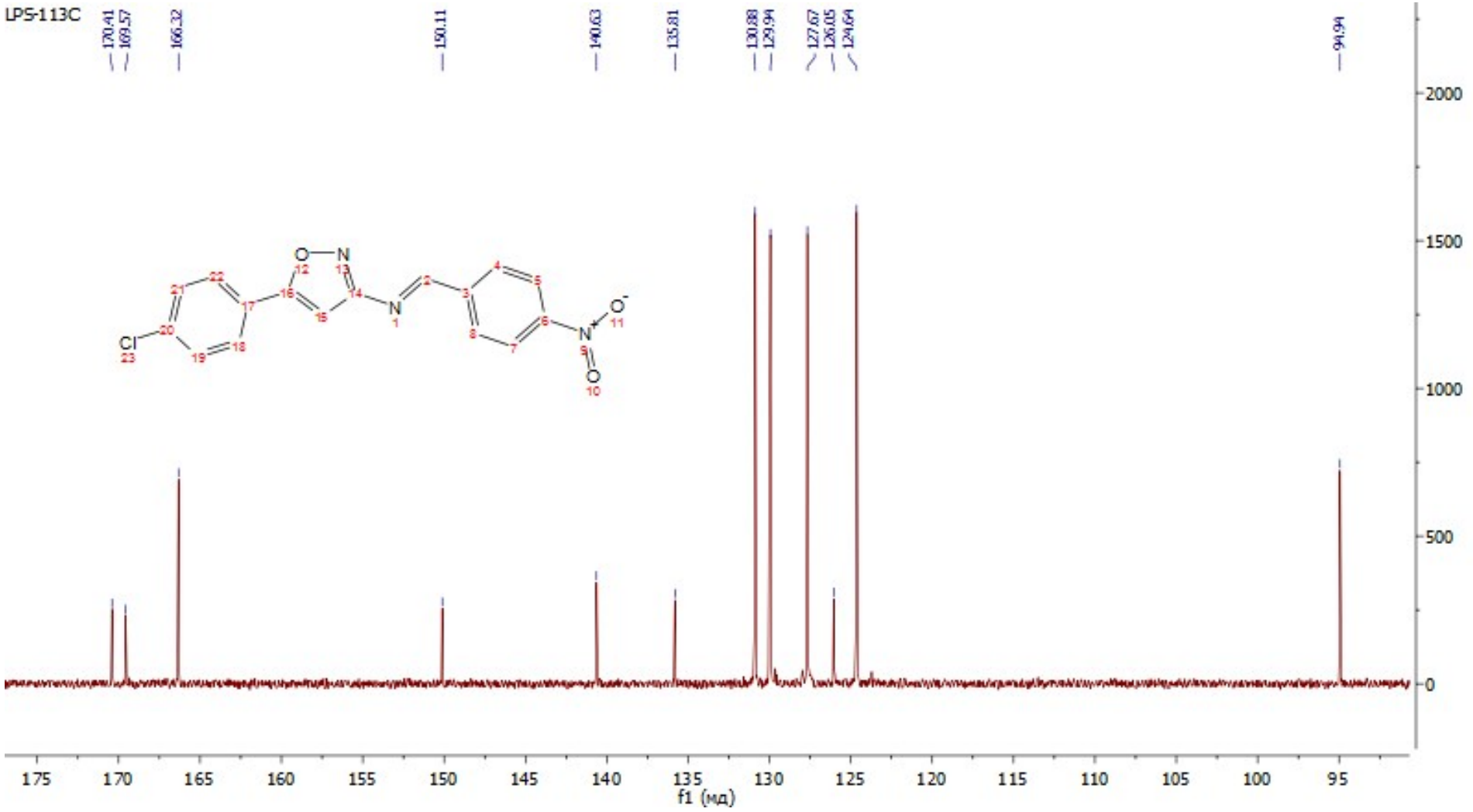
^{1}H NMR (DMSO-d6): region from 9.6 to 7.2 ppm



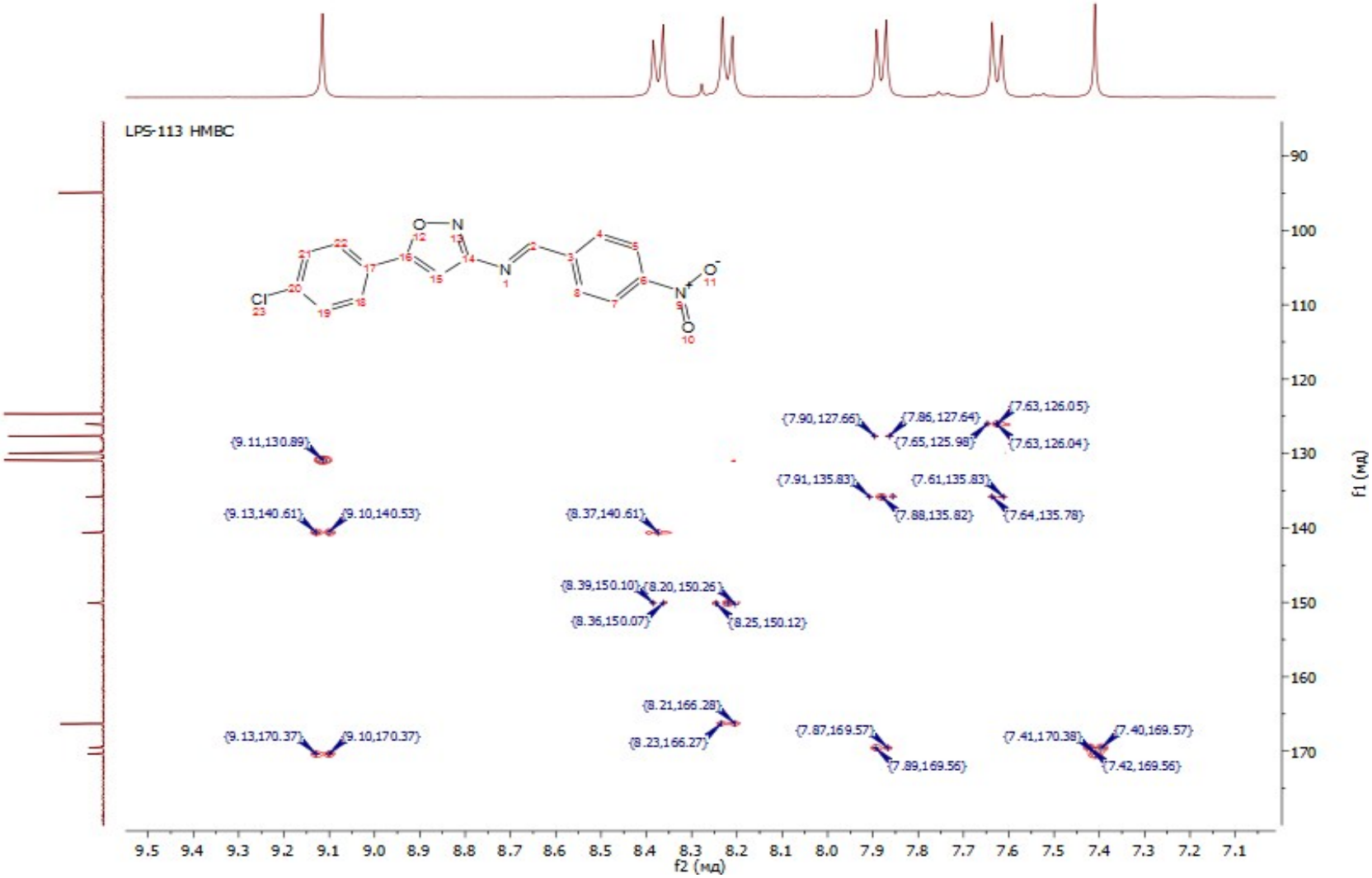
^{13}C NMR (DMSO-d_6)



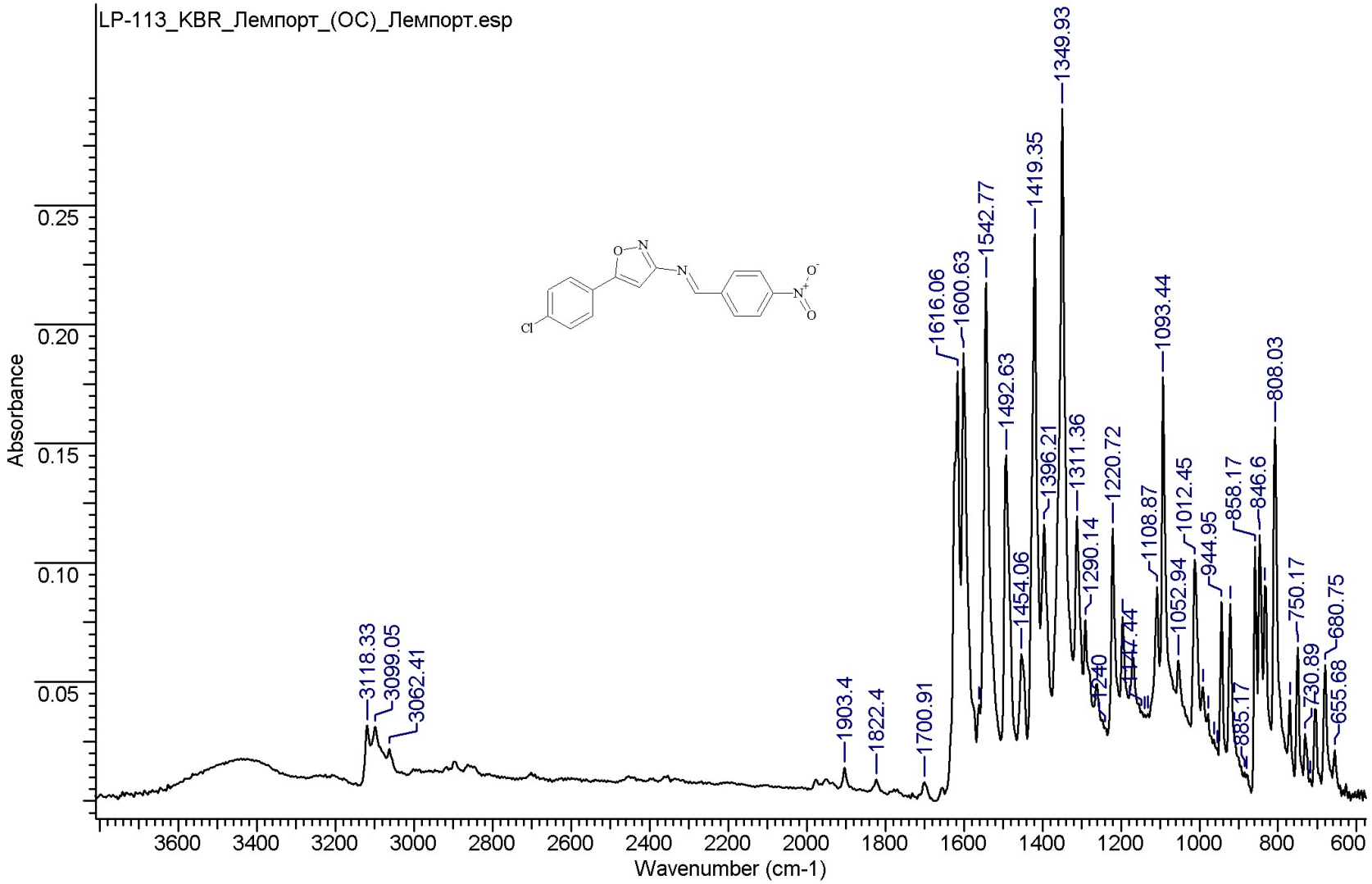
^{13}C NMR (DMSO-d_6): region from 175 to 90 ppm



$^{1}H/^{13}C$ HMBC NMR (DMSO-d_6)



IR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1616.06
1600.63
1542.77
1492.63
1454.06
1419.35
1396.21
1349.93
1311.36
1290.14
1240
1220.72
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1108.87
1093.44
1052.94
1012.45
944.95
885.17
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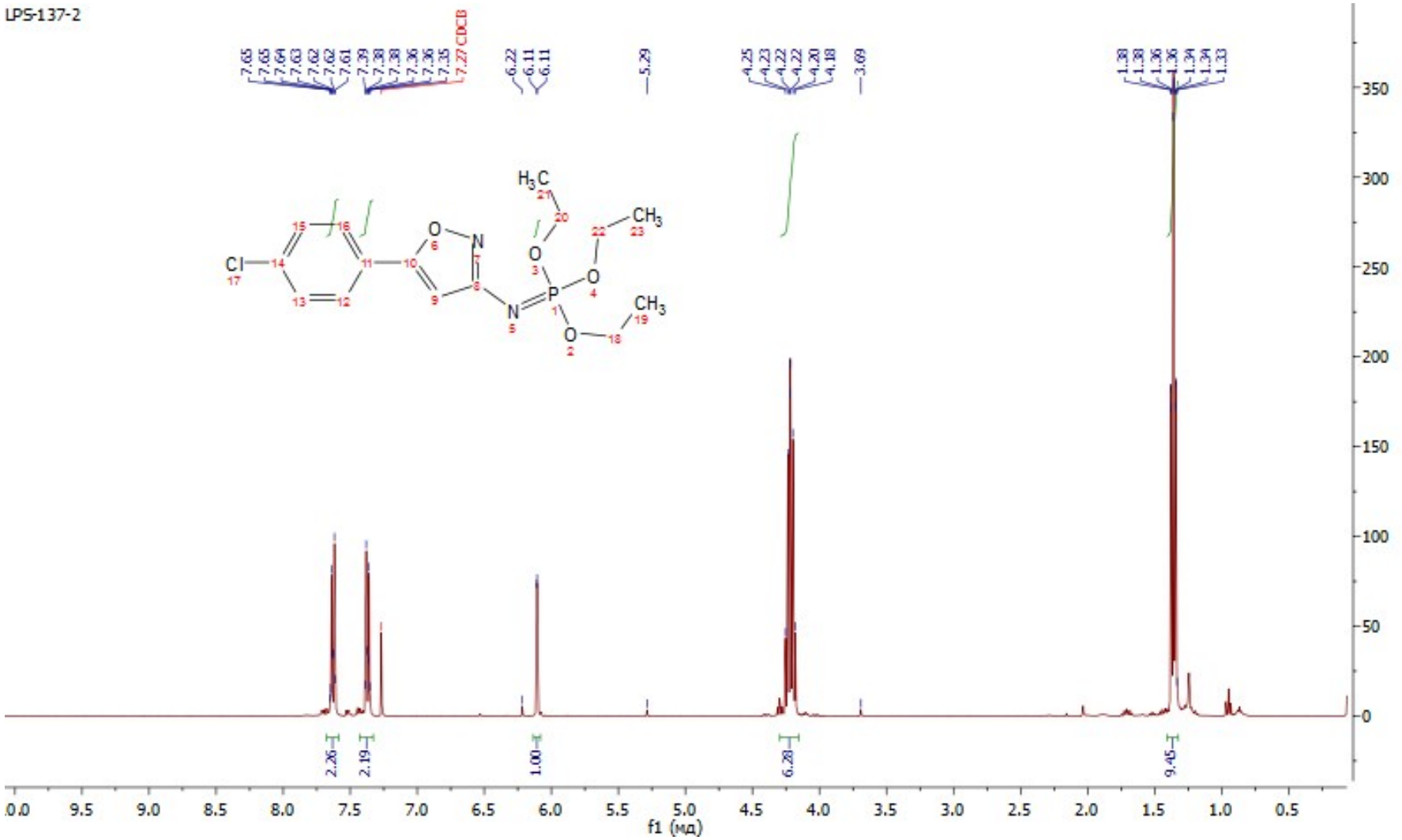
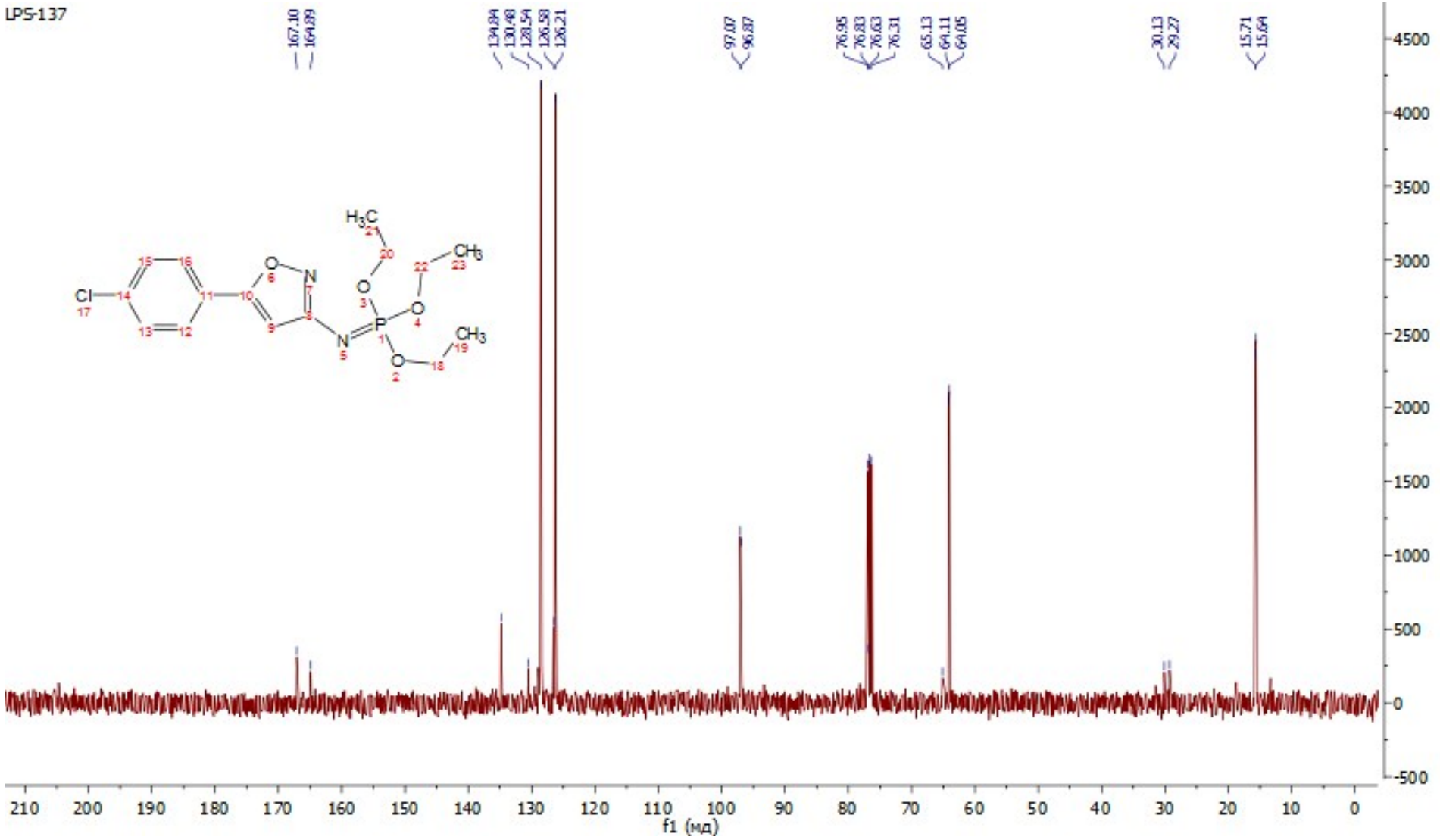
808.03
750.17
730.89
680.75
655.68
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
Wavenumber (cm-1)

5-(4-Chloro-phenyl)-isoxazol-3-yl-phosphorimidic acid triethyl ester 14b

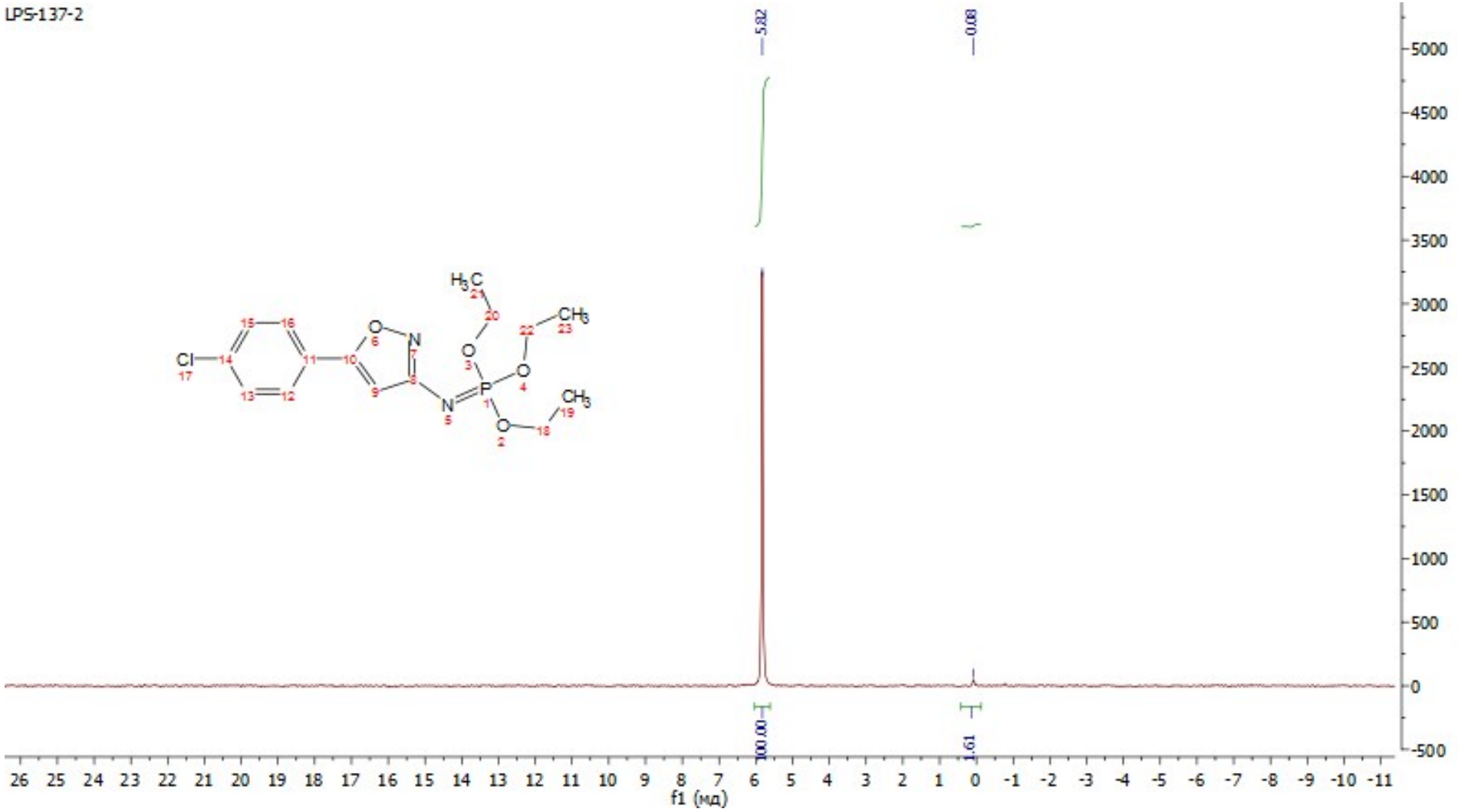
^{1}H NMR ($CDCl_3$)



^{13}C NMR ($CDCl_3$)

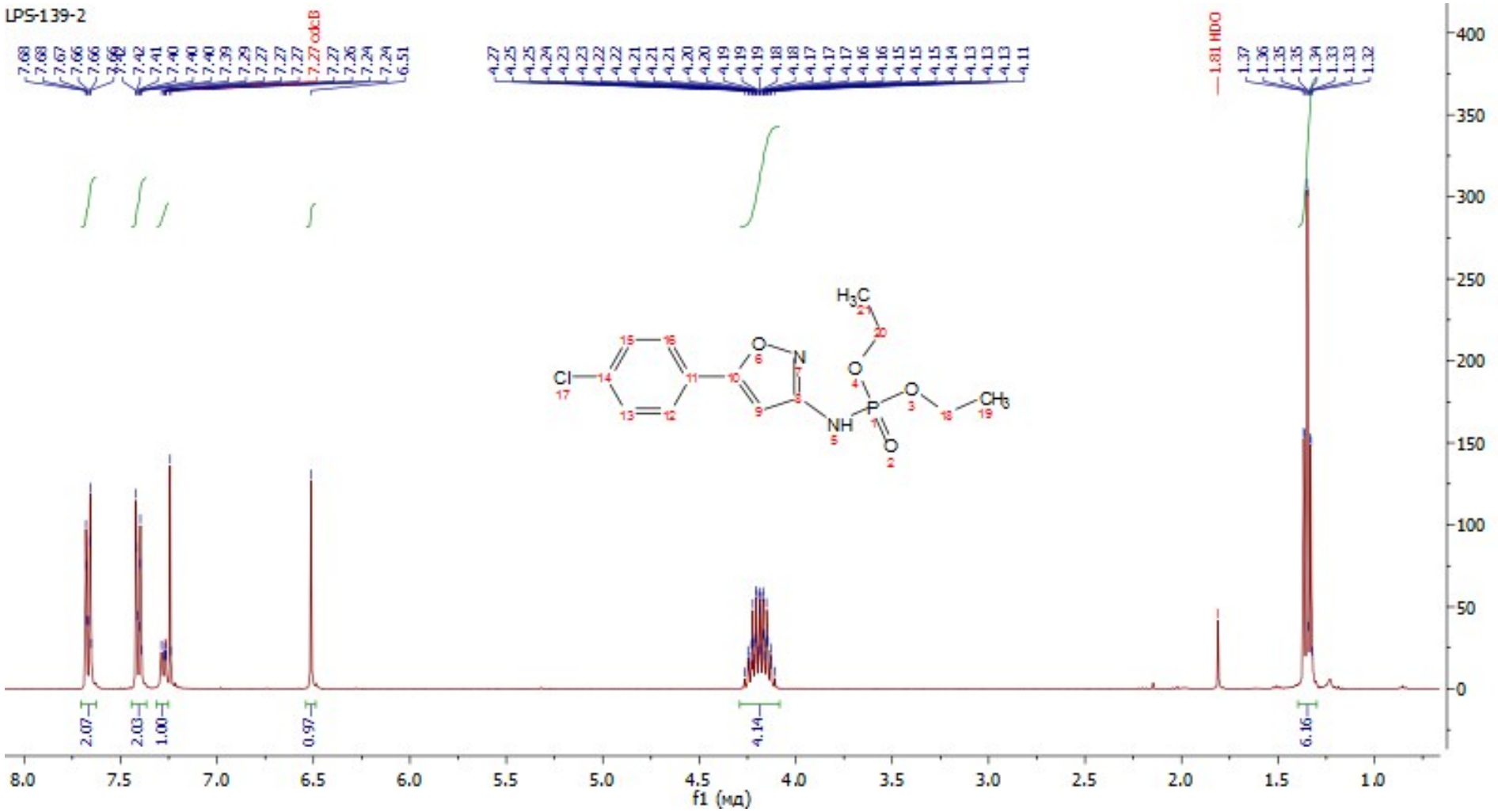


^{31}P NMR ($CDCl_3$)

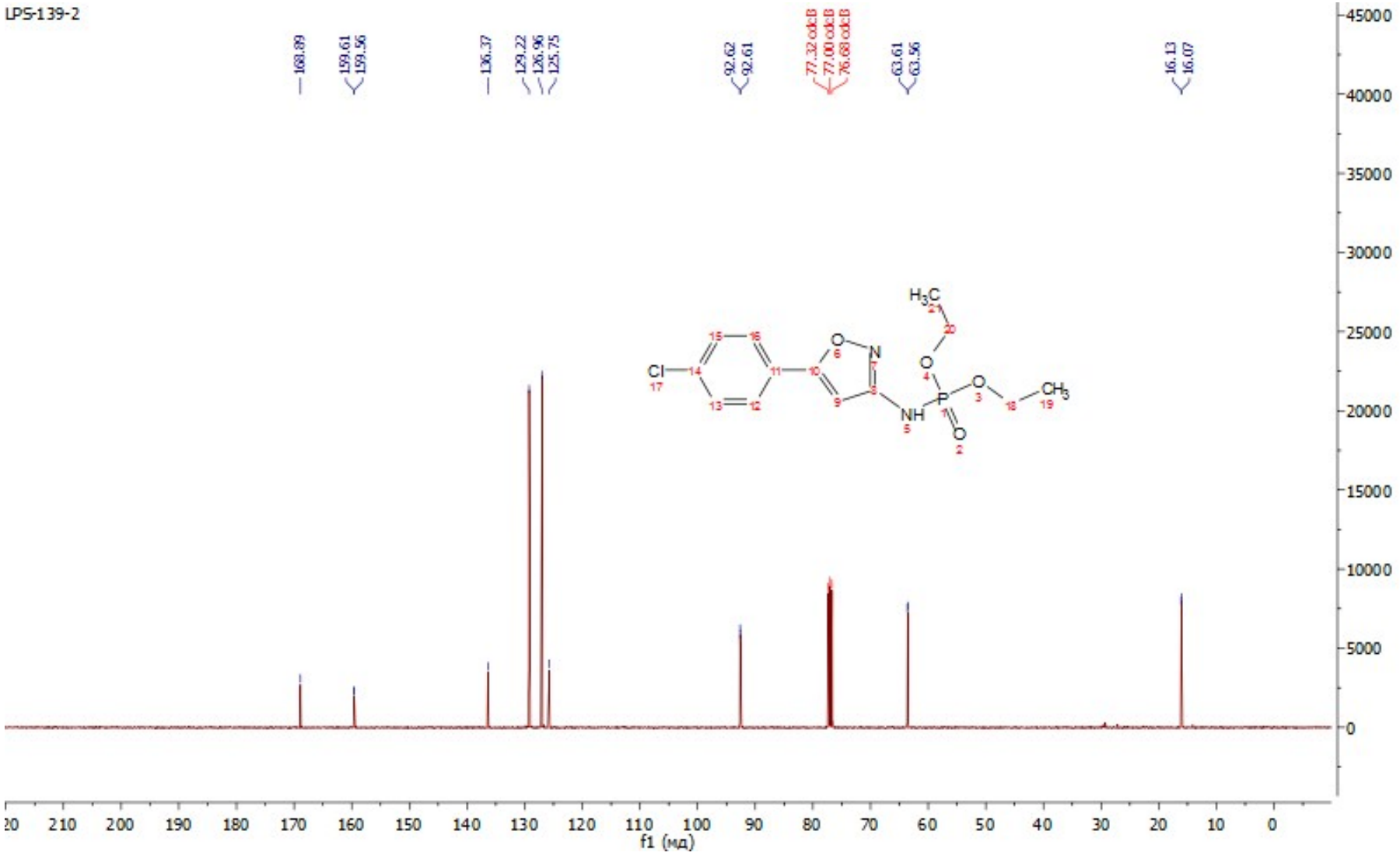


[5-(4-Chloro-phenyl)-isoxazol-3-yl]-phosphoramidic acid diethyl ester **15b**

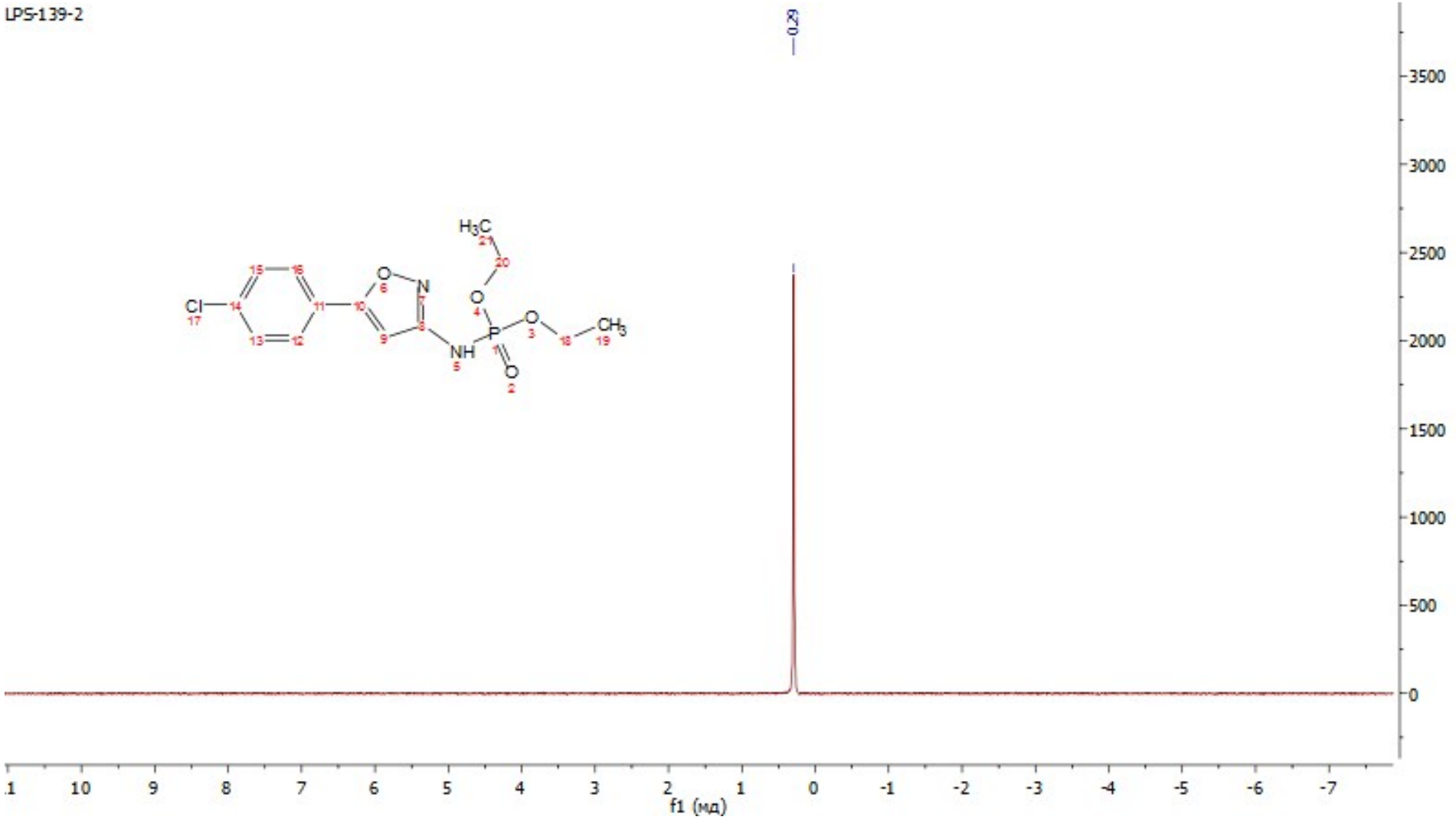
^{1}H NMR ($CDCl_3$)



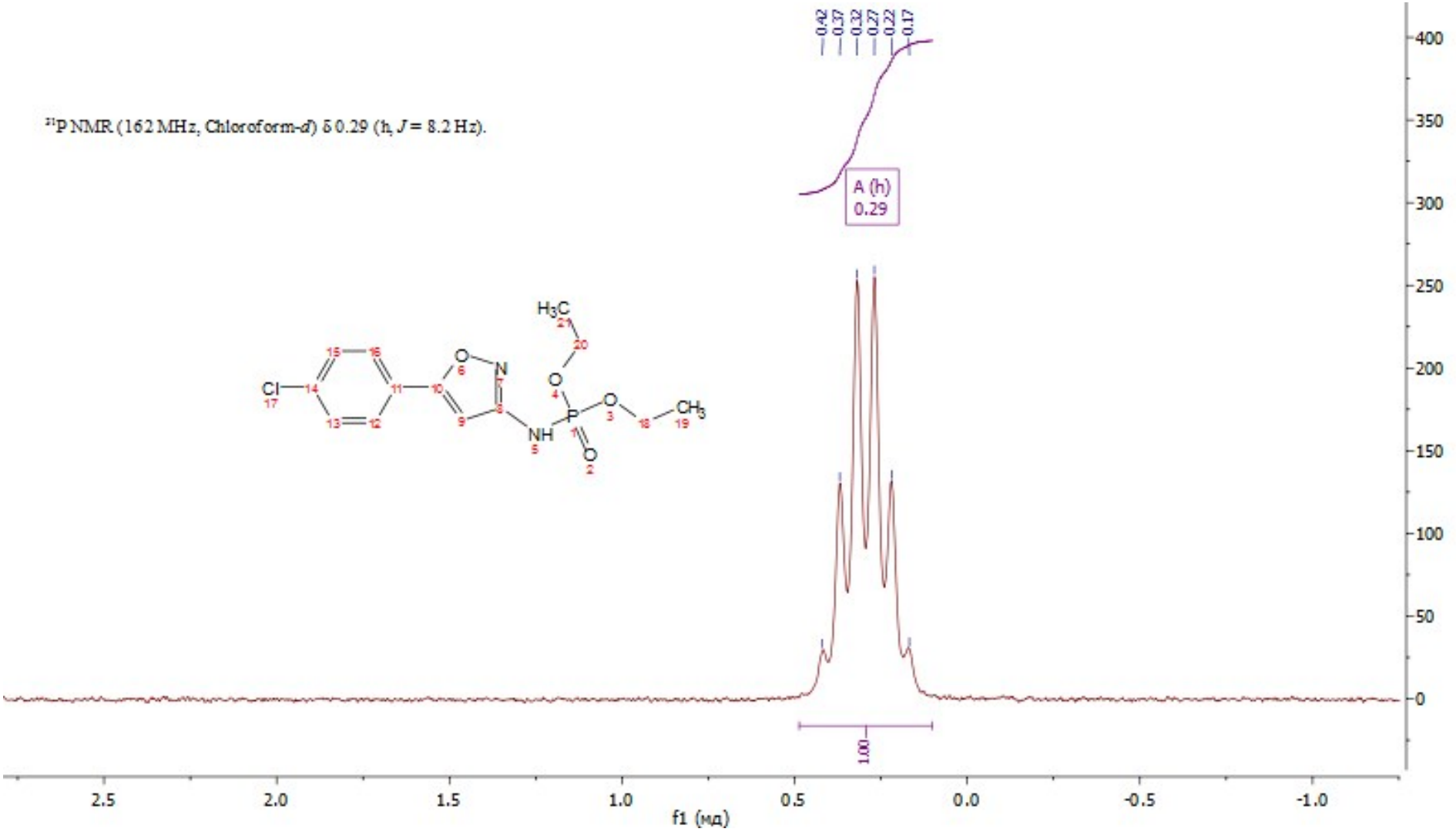
^{13}C NMR ($CDCl_3$)



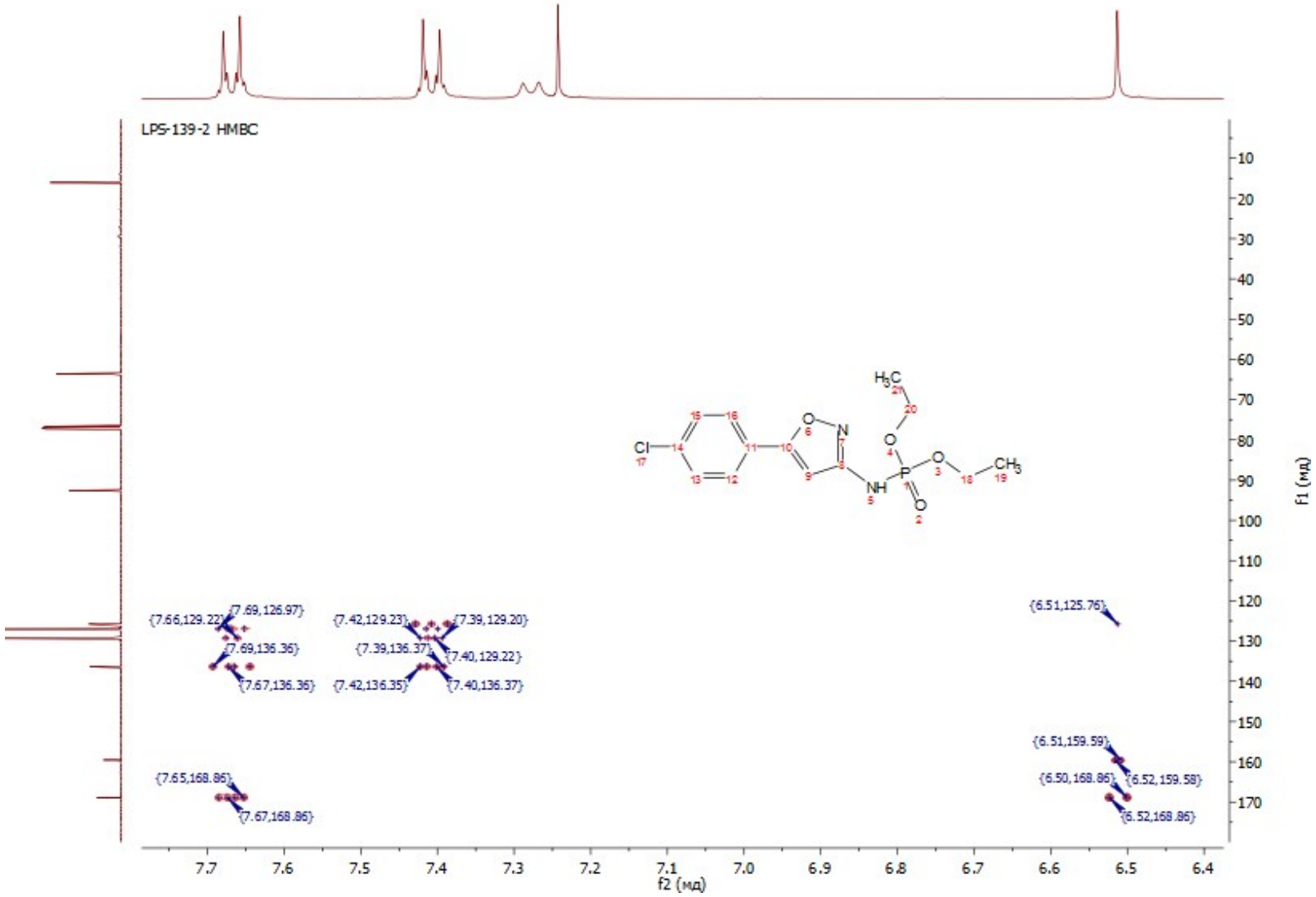
^{31}P NMR ($CDCl_3$)



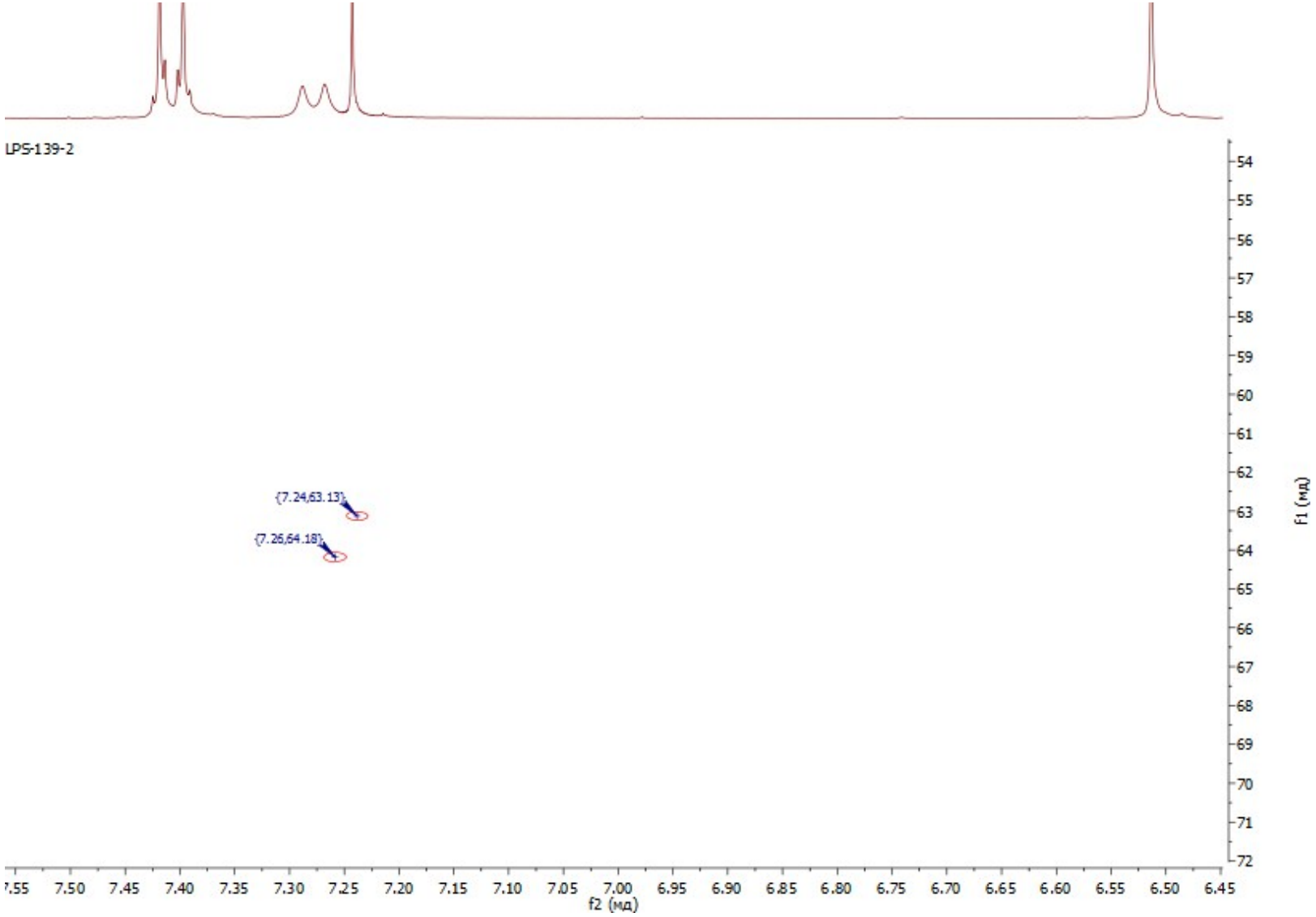
^{31}P NMR ($CDCl_3$) without decoupling from ^{1}H



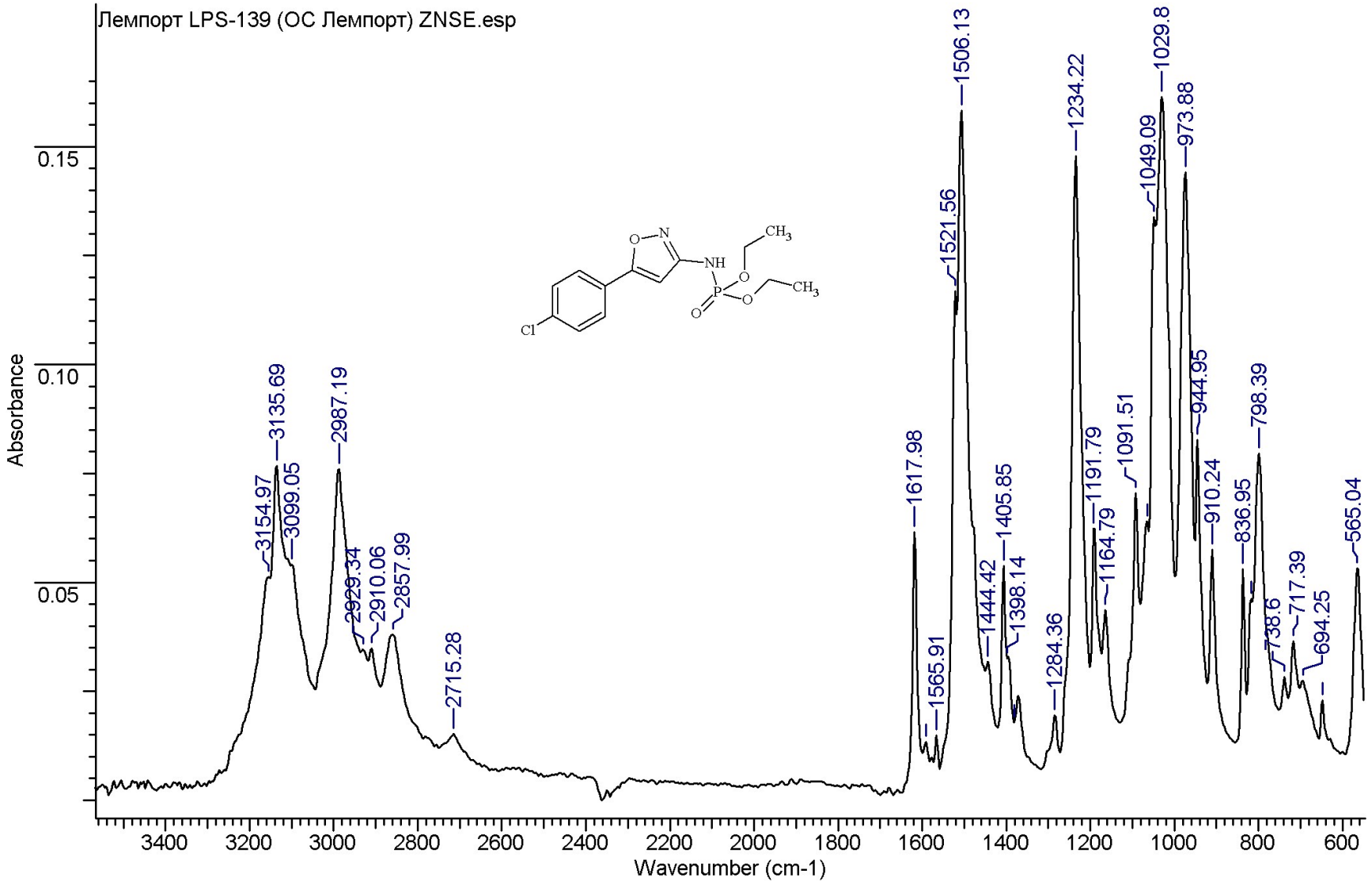
$^{1}H/^{13}C$ HMBC NMR ($CDCl_3$)



$^{1}H/^{15}N$ HSQC NMR ($CDCl_3$)



IR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2715.28
1617.98
1565.91
1521.56
1506.13
1444.42
1405.85
1398.14
1284.36
1234.22
1191.79
1164.79
1091.51
1049.09
1029.8
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717.39
694.25
565.04
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
Wavenumber (cm-1)

X-ray structure analysis data

1-(5-(4-Bromophenyl)isoxazol-3-yl)-4,5,6,7,8,9-hexahydro-1H-cycloocta[d][1,2,3]triazole **7a**

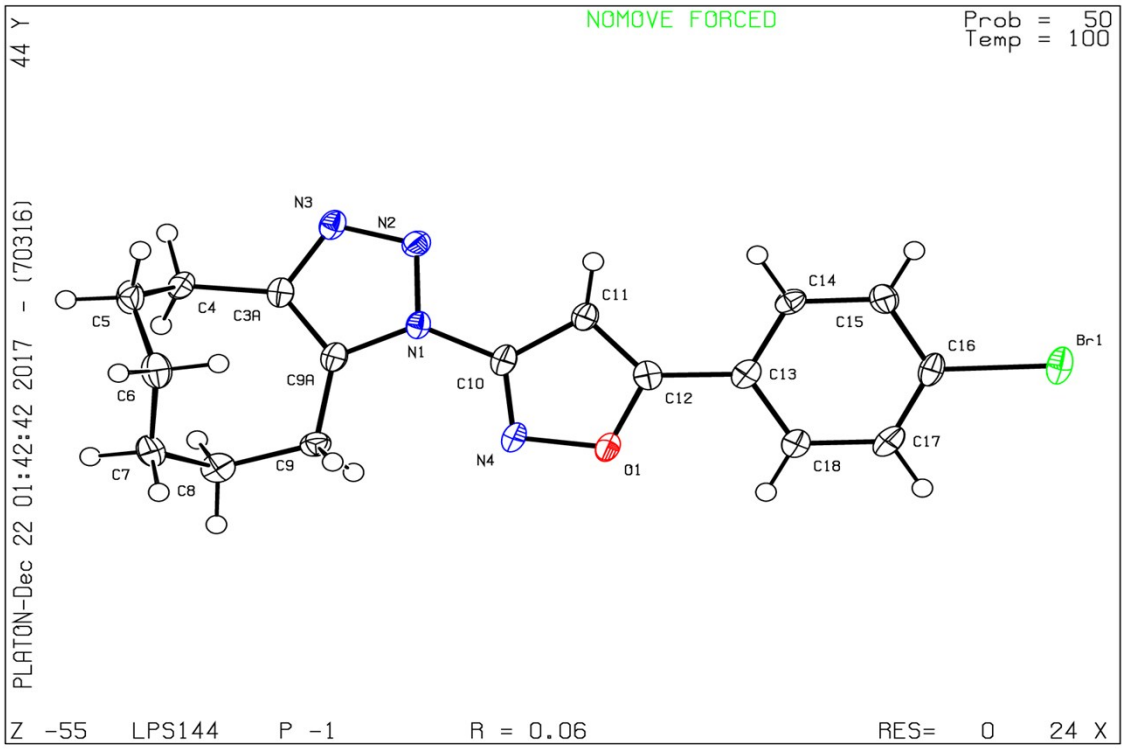


Table 1. Crystal data and structure refinement for **7a**.

Identification code	**LPS144**	
Empirical formula	C17 H17 Br N4 O	
Formula weight	373.25	
Temperature	100(2) K	
Wavelength	0.96260 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 5.2572(10) Å	α = 69.94(3)°.
	b = 12.458(3) Å	β = 83.57(3)°.
	c = 13.205(3) Å	γ = 89.74(3)°.
Volume	806.7(3) $Å^3$	
Z	2	
Density (calculated)	1.537 Mg/m^3	
Absorption coefficient	0.947 mm^{-1}	
F(000)	380	
Crystal size	0.35 x 0.20 x 0.12 mm^3	
Theta range for data collection	2.239 to 38.307°.	
Index ranges	-6<=h<=6, -14<=k<=15, -16<=l<=16	
Reflections collected	13207	

Independent reflections	3237 [R(int) = 0.1002]
Completeness to theta = 35.279°	96.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.910 and 0.780
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3237 / 0 / 209
Goodness-of-fit on F^2	1.057
Final R indices [for 2399 rflns with $I>2\sigma(I)$]	R1 = 0.0615, wR2 = 0.1321
R indices (all data)	R1 = 0.0871, wR2 = 0.1567
Extinction coefficient	0.009(2)
Largest diff. peak and hole	0.809 and -1.391 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for ***LPS144***. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U(eq)
Br(1)	14535(1)	357(1)	1196(1)	29(1)
O(1)	6157(5)	1317(2)	5233(2)	22(1)
N(1)	5602(6)	3637(3)	6210(2)	18(1)
N(2)	7089(6)	4646(3)	5846(2)	20(1)
N(3)	6009(6)	5305(3)	6359(2)	21(1)
N(4)	4863(6)	1869(3)	5919(2)	22(1)
C(3A)	3842(7)	4740(3)	7043(3)	18(1)
C(4)	2345(7)	5304(3)	7756(3)	21(1)
C(5)	2931(8)	4803(3)	8954(3)	27(1)
C(6)	2745(8)	3491(3)	9451(3)	27(1)
C(7)	210(8)	2875(3)	9432(3)	25(1)
C(8)	-514(8)	2902(3)	8303(3)	25(1)
C(9)	1655(7)	2674(3)	7524(3)	20(1)
C(9A)	3556(7)	3671(3)	6960(3)	19(1)
C(10)	6315(7)	2806(3)	5714(3)	18(1)
C(11)	8530(7)	2922(3)	4937(3)	18(1)
C(12)	8305(7)	1956(3)	4665(3)	19(1)
C(13)	9818(7)	1545(3)	3862(3)	19(1)
C(14)	12125(7)	2138(3)	3296(3)	20(1)
C(15)	13540(7)	1786(3)	2503(3)	22(1)
C(16)	12603(8)	833(3)	2292(3)	21(1)
C(17)	10345(8)	235(3)	2845(3)	25(1)
C(18)	8950(7)	592(3)	3634(3)	21(1)

Table 3. Bond lengths [Å] and angles [°] for ***LPS144***.

Br(1)-C(16)	1.931(4)	C(7)-H(7B)	0.9900
O(1)-C(12)	1.371(5)	C(8)-C(9)	1.541(5)
O(1)-N(4)	1.429(4)	C(8)-H(8A)	0.9900
N(1)-N(2)	1.389(4)	C(8)-H(8B)	0.9900
N(1)-C(9A)	1.390(5)	C(9)-C(9A)	1.518(6)
N(1)-C(10)	1.430(4)	C(9)-H(9A)	0.9900
N(2)-N(3)	1.322(4)	C(9)-H(9B)	0.9900
N(3)-C(3A)	1.396(5)	C(10)-C(11)	1.436(5)
N(4)-C(10)	1.328(5)	C(11)-C(12)	1.378(5)
C(3A)-C(9A)	1.383(5)	C(11)-H(11)	0.9500
C(3A)-C(4)	1.511(5)	C(12)-C(13)	1.483(5)
C(4)-C(5)	1.556(5)	C(13)-C(18)	1.410(5)
C(4)-H(4A)	0.9900	C(13)-C(14)	1.415(6)
C(4)-H(4B)	0.9900	C(14)-C(15)	1.407(5)
C(5)-C(6)	1.537(6)	C(14)-H(14)	0.9500
C(5)-H(5A)	0.9900	C(15)-C(16)	1.412(5)
C(5)-H(5B)	0.9900	C(15)-H(15)	0.9500
C(6)-C(7)	1.548(6)	C(16)-C(17)	1.392(6)
C(6)-H(6A)	0.9900	C(17)-C(18)	1.401(5)
C(6)-H(6B)	0.9900	C(17)-H(17)	0.9500
C(7)-C(8)	1.568(5)	C(18)-H(18)	0.9500
C(7)-H(7A)	0.9900		

C(12)-O(1)-N(4)	109.6(3)
N(2)-N(1)-C(9A)	111.4(3)
N(2)-N(1)-C(10)	116.4(3)
C(9A)-N(1)-C(10)	132.0(3)
N(3)-N(2)-N(1)	106.1(3)
N(2)-N(3)-C(3A)	109.8(3)
C(10)-N(4)-O(1)	103.2(3)
C(9A)-C(3A)-N(3)	109.0(3)
C(9A)-C(3A)-C(4)	131.3(4)
N(3)-C(3A)-C(4)	119.6(3)
C(3A)-C(4)-C(5)	112.1(3)
C(3A)-C(4)-H(4A)	109.2
C(5)-C(4)-H(4A)	109.2
C(3A)-C(4)-H(4B)	109.2
C(5)-C(4)-H(4B)	109.2
H(4A)-C(4)-H(4B)	107.9
C(6)-C(5)-C(4)	114.8(3)
C(6)-C(5)-H(5A)	108.6
C(4)-C(5)-H(5A)	108.6
C(6)-C(5)-H(5B)	108.6
C(4)-C(5)-H(5B)	108.6
H(5A)-C(5)-H(5B)	107.5
C(5)-C(6)-C(7)	118.2(3)
C(5)-C(6)-H(6A)	107.8
C(7)-C(6)-H(6A)	107.8
C(5)-C(6)-H(6B)	107.8
C(7)-C(6)-H(6B)	107.8
H(6A)-C(6)-H(6B)	107.1
C(6)-C(7)-C(8)	117.9(3)
C(6)-C(7)-H(7A)	107.8
C(8)-C(7)-H(7A)	107.8
C(6)-C(7)-H(7B)	107.8
C(8)-C(7)-H(7B)	107.8
H(7A)-C(7)-H(7B)	107.2
C(9)-C(8)-C(7)	116.5(3)
C(9)-C(8)-H(8A)	108.2
C(7)-C(8)-H(8A)	108.2
C(9)-C(8)-H(8B)	108.2
C(7)-C(8)-H(8B)	108.2
H(8A)-C(8)-H(8B)	107.3

C(9A)-C(9)-C(8)	114.8(3)
C(9A)-C(9)-H(9A)	108.6
C(8)-C(9)-H(9A)	108.6
C(9A)-C(9)-H(9B)	108.6
C(8)-C(9)-H(9B)	108.6
H(9A)-C(9)-H(9B)	107.5
C(3A)-C(9A)-N(1)	103.7(3)
C(3A)-C(9A)-C(9)	133.1(3)
N(1)-C(9A)-C(9)	123.2(3)
N(4)-C(10)-N(1)	121.1(3)
N(4)-C(10)-C(11)	114.6(3)
N(1)-C(10)-C(11)	124.3(3)
C(12)-C(11)-C(10)	102.4(3)
C(12)-C(11)-H(11)	128.8
C(10)-C(11)-H(11)	128.8
O(1)-C(12)-C(11)	110.2(3)
O(1)-C(12)-C(13)	117.2(3)
C(11)-C(12)-C(13)	132.6(4)
C(18)-C(13)-C(14)	119.7(3)
C(18)-C(13)-C(12)	120.9(4)
C(14)-C(13)-C(12)	119.4(3)
C(15)-C(14)-C(13)	120.2(3)
C(15)-C(14)-H(14)	119.9
C(13)-C(14)-H(14)	119.9
C(14)-C(15)-C(16)	118.5(4)
C(14)-C(15)-H(15)	120.8
C(16)-C(15)-H(15)	120.8
C(17)-C(16)-C(15)	122.0(3)
C(17)-C(16)-Br(1)	119.6(3)
C(15)-C(16)-Br(1)	118.4(3)
C(16)-C(17)-C(18)	119.1(3)
C(16)-C(17)-H(17)	120.5
C(18)-C(17)-H(17)	120.5
C(17)-C(18)-C(13)	120.5(4)
C(17)-C(18)-H(18)	119.8
C(13)-C(18)-H(18)	119.8

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for ***LPS144***. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	39(1)	31(1)	22(1)	-15(1)	-4(1)	11(1)
O(1)	26(2)	21(1)	23(1)	-13(1)	-1(1)	3(1)
N(1)	22(2)	19(2)	15(2)	-8(1)	-3(1)	4(1)
N(2)	25(2)	17(2)	20(2)	-7(1)	-2(1)	1(2)
N(3)	28(2)	20(2)	16(2)	-8(1)	-4(1)	4(2)
N(4)	27(2)	22(2)	23(2)	-14(1)	-2(1)	4(2)
C(3A)	20(2)	22(2)	15(2)	-7(2)	-6(2)	6(2)
C(4)	22(2)	21(2)	24(2)	-13(2)	-4(2)	3(2)
C(5)	30(2)	36(3)	19(2)	-15(2)	-2(2)	1(2)
C(6)	30(2)	35(3)	17(2)	-9(2)	-5(2)	6(2)
C(7)	26(2)	29(2)	18(2)	-7(2)	1(2)	2(2)
C(8)	28(2)	23(2)	23(2)	-6(2)	-7(2)	2(2)
C(9)	25(2)	17(2)	19(2)	-5(2)	-3(2)	-2(2)
C(9A)	22(2)	22(2)	15(2)	-9(2)	-7(2)	5(2)
C(10)	25(2)	20(2)	14(2)	-7(2)	-9(2)	8(2)
C(11)	22(2)	21(2)	15(2)	-8(2)	-5(2)	3(2)
C(12)	17(2)	23(2)	17(2)	-6(2)	-6(2)	6(2)
C(13)	25(2)	17(2)	17(2)	-6(2)	-9(2)	6(2)
C(14)	24(2)	19(2)	20(2)	-9(2)	-7(2)	-1(2)
C(15)	21(2)	25(2)	18(2)	-7(2)	-3(2)	3(2)
C(16)	28(2)	23(2)	17(2)	-11(2)	-9(2)	10(2)
C(17)	33(3)	18(2)	26(2)	-11(2)	-10(2)	6(2)
C(18)	23(2)	21(2)	21(2)	-9(2)	-4(2)	3(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for ***LPS144***.

Atom	x	y	z	U(iso)
H(4A)	490	5199	7731	25
H(4B)	2772	6136	7469	25
H(5A)	4682	5064	8993	32
H(5B)	1723	5118	9402	32
H(6A)	3083	3272	10217	32
H(6B)	4142	3189	9069	32
H(7A)	-1203	3212	9770	30
H(7B)	283	2064	9899	30
H(8A)	-1918	2327	8434	30
H(8B)	-1192	3662	7933	30
H(9A)	2598	2008	7938	24
H(9B)	880	2467	6963	24
H(11)	9822	3515	4676	22
H(14)	12721	2776	3452	24
H(15)	15090	2180	2120	26
H(17)	9757	-406	2690	29
H(18)	7409	189	4019	25

Table 6. Torsion angles [°] for ***LPS144***.

C(9A)-N(1)-N(2)-N(3)	0.2(3)
C(10)-N(1)-N(2)-N(3)	176.4(3)
N(1)-N(2)-N(3)-C(3A)	-0.3(3)
C(12)-O(1)-N(4)-C(10)	0.2(3)
N(2)-N(3)-C(3A)-C(9A)	0.4(4)
N(2)-N(3)-C(3A)-C(4)	177.3(3)
C(9A)-C(3A)-C(4)-C(5)	74.0(5)
N(3)-C(3A)-C(4)-C(5)	-102.2(4)
C(3A)-C(4)-C(5)-C(6)	-50.1(5)
C(4)-C(5)-C(6)-C(7)	-55.4(5)
C(5)-C(6)-C(7)-C(8)	68.3(5)
C(6)-C(7)-C(8)-C(9)	44.3(5)
C(7)-C(8)-C(9)-C(9A)	-76.4(4)
N(3)-C(3A)-C(9A)-N(1)	-0.2(3)
C(4)-C(3A)-C(9A)-N(1)	-176.7(3)
N(3)-C(3A)-C(9A)-C(9)	177.5(3)
C(4)-C(3A)-C(9A)-C(9)	1.0(6)
N(2)-N(1)-C(9A)-C(3A)	0.0(3)
C(10)-N(1)-C(9A)-C(3A)	-175.4(3)
N(2)-N(1)-C(9A)-C(9)	-178.0(3)
C(10)-N(1)-C(9A)-C(9)	6.6(5)
C(8)-C(9)-C(9A)-C(3A)	4.3(5)
C(8)-C(9)-C(9A)-N(1)	-178.4(3)
O(1)-N(4)-C(10)-N(1)	177.0(3)
O(1)-N(4)-C(10)-C(11)	-0.4(4)
N(2)-N(1)-C(10)-N(4)	-172.7(3)
C(9A)-N(1)-C(10)-N(4)	2.5(5)
N(2)-N(1)-C(10)-C(11)	4.4(4)
C(9A)-N(1)-C(10)-C(11)	179.6(3)
N(4)-C(10)-C(11)-C(12)	0.5(4)
N(1)-C(10)-C(11)-C(12)	-176.8(3)
N(4)-O(1)-C(12)-C(11)	0.1(4)
N(4)-O(1)-C(12)-C(13)	-176.7(3)
C(10)-C(11)-C(12)-O(1)	-0.3(4)
C(10)-C(11)-C(12)-C(13)	175.8(3)
O(1)-C(12)-C(13)-C(18)	5.2(5)
C(11)-C(12)-C(13)-C(18)	-170.7(3)
O(1)-C(12)-C(13)-C(14)	-176.6(3)

C(11)-C(12)-C(13)-C(14)	7.5(5)
C(18)-C(13)-C(14)-C(15)	0.5(5)
C(12)-C(13)-C(14)-C(15)	-177.7(3)
C(13)-C(14)-C(15)-C(16)	-0.1(5)
C(14)-C(15)-C(16)-C(17)	-0.3(5)
C(14)-C(15)-C(16)-Br(1)	179.6(2)
C(15)-C(16)-C(17)-C(18)	0.3(5)
Br(1)-C(16)-C(17)-C(18)	-179.6(2)
C(16)-C(17)-C(18)-C(13)	0.2(5)
C(14)-C(13)-C(18)-C(17)	-0.6(5)
C(12)-C(13)-C(18)-C(17)	177.6(3)

1-(5-(trifluoromethyl)isoxazol-3-yl)-4,5,6,7,8,9-hexahydro-1H-cycloocta[d][1,2,3]triazole **7h**

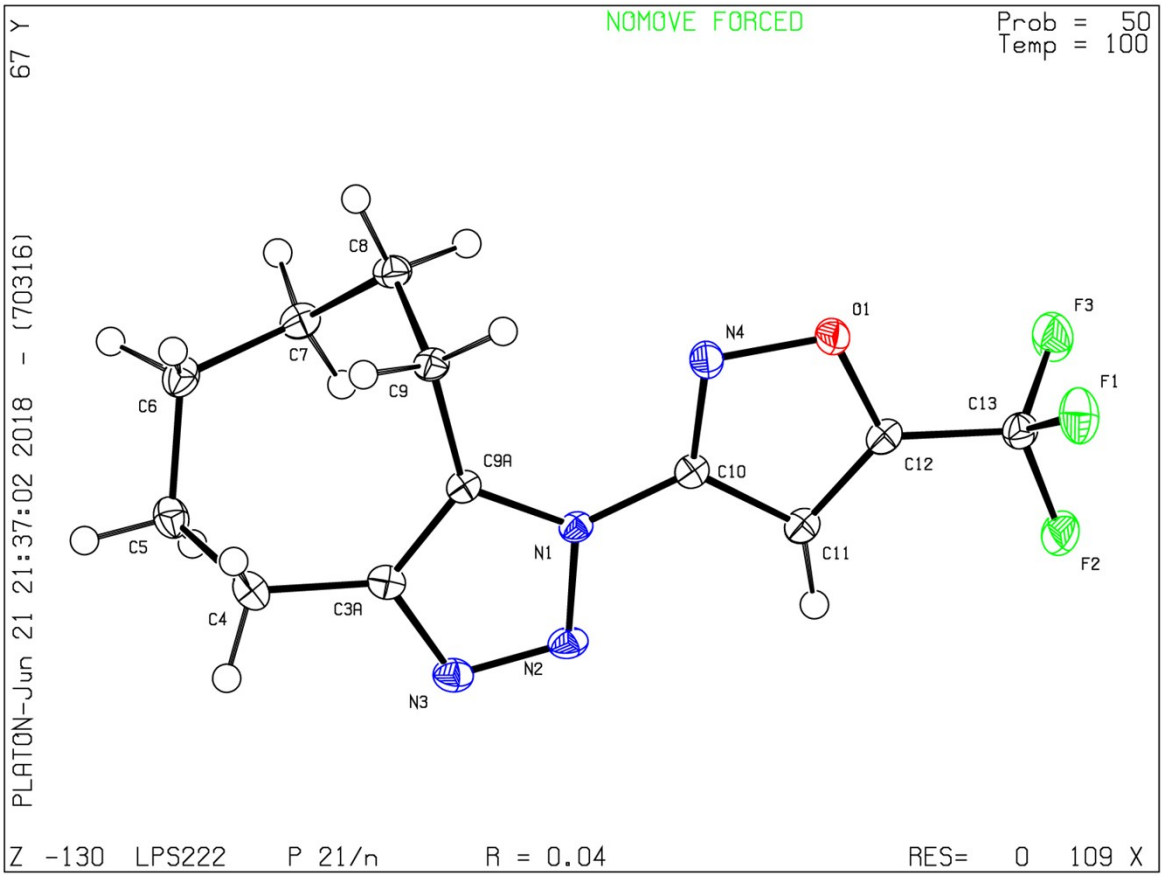


Table 1. Crystal data and structure refinement for **7h**.

Identification code	**LPS222**	
Empirical formula	C12 H13 F3 N4 O	
Formula weight	286.26	
Temperature	100(2) K	
Wavelength	0.80246 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 4.7655(10) Å	α = 90°.
	b = 21.615(4) Å	β = 90.58(3)°.
	c = 11.859(2) Å	γ = 90°.
Volume	1221.5(4) Å^3	
Z	4	
Density (calculated)	1.557 Mg/m^3	
Absorption coefficient	0.179 mm^{-1}	
F(000)	592	
Crystal size	0.20 x 0.15 x 0.10 mm^3	
Theta range for data collection	3.736 to 30.804°.	
Index ranges	-6<=h<=6, -27<=k<=26, -15<=l<=15	

Reflections collected	20034
Independent reflections	2604 [R(int) = 0.0535]
Completeness to theta = 28.782°	98.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.975 and 0.955
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2604 / 0 / 182
Goodness-of-fit on F^2	1.051
Final R indices [for 2512 rflns with $I>2\sigma(I)$]	R1 = 0.0398, wR2 = 0.1048
R indices (all data)	R1 = 0.0407, wR2 = 0.1055
Extinction coefficient	0.054(5)
Largest diff. peak and hole	0.437 and -0.243 e. $Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for ***LPS222***. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U(eq)
F(1)	3523(2)	5484(1)	481(1)	27(1)
F(2)	-472(2)	5281(1)	1226(1)	27(1)
F(3)	452(2)	6199(1)	641(1)	28(1)
O(1)	4736(2)	6282(1)	2333(1)	18(1)
N(1)	4331(2)	5891(1)	5186(1)	13(1)
N(2)	2598(2)	5478(1)	5706(1)	16(1)
N(3)	3211(2)	5499(1)	6778(1)	17(1)
N(4)	5607(2)	6349(1)	3460(1)	18(1)
C(3A)	5318(2)	5918(1)	6962(1)	14(1)
C(4)	6511(3)	6051(1)	8108(1)	17(1)
C(5)	5567(3)	6664(1)	8637(1)	18(1)
C(6)	6564(3)	7261(1)	8066(1)	18(1)
C(7)	5136(2)	7419(1)	6928(1)	17(1)
C(8)	6911(3)	7323(1)	5871(1)	16(1)
C(9)	8158(2)	6671(1)	5745(1)	14(1)
C(9A)	6068(2)	6173(1)	5950(1)	12(1)
C(10)	4044(2)	5956(1)	4016(1)	13(1)
C(11)	2117(2)	5624(1)	3323(1)	14(1)
C(12)	2685(2)	5852(1)	2299(1)	14(1)
C(13)	1528(3)	5703(1)	1155(1)	16(1)

Table 3. Bond lengths [E] and angles [°] for ***LPS222***.

F(1)-C(13)	1.3343(14)	C(5)-H(5B)	0.9900
F(2)-C(13)	1.3224(15)	C(6)-C(7)	1.5440(17)
F(3)-C(13)	1.3337(15)	C(6)-H(6A)	0.9900
O(1)-C(12)	1.3500(15)	C(6)-H(6B)	0.9900
O(1)-N(4)	1.4027(13)	C(7)-C(8)	1.5327(17)
N(1)-C(9A)	1.3652(15)	C(7)-H(7A)	0.9900
N(1)-N(2)	1.3685(14)	C(7)-H(7B)	0.9900
N(1)-C(10)	1.3995(15)	C(8)-C(9)	1.5393(17)
N(2)-N(3)	1.3022(15)	C(8)-H(8A)	0.9900
N(3)-C(3A)	1.3690(16)	C(8)-H(8B)	0.9900
N(4)-C(10)	1.3118(16)	C(9)-C(9A)	1.4873(15)
C(3A)-C(9A)	1.3704(16)	C(9)-H(9A)	0.9900
C(3A)-C(4)	1.4955(16)	C(9)-H(9B)	0.9900
C(4)-C(5)	1.5347(17)	C(10)-C(11)	1.4211(16)
C(4)-H(4A)	0.9900	C(11)-C(12)	1.3413(17)
C(4)-H(4B)	0.9900	C(11)-H(11)	0.9500
C(5)-C(6)	1.5356(18)	C(12)-C(13)	1.4946(17)
C(5)-H(5A)	0.9900		

C(12)-O(1)-N(4)	107.84(9)	C(4)-C(5)-H(5B)	108.1
C(9A)-N(1)-N(2)	110.90(9)	C(6)-C(5)-H(5B)	108.1
C(9A)-N(1)-C(10)	131.80(10)	H(5A)-C(5)-H(5B)	107.3
N(2)-N(1)-C(10)	117.30(10)	C(5)-C(6)-C(7)	115.86(10)
N(3)-N(2)-N(1)	106.66(9)	C(5)-C(6)-H(6A)	108.3
N(2)-N(3)-C(3A)	109.62(10)	C(7)-C(6)-H(6A)	108.3
C(10)-N(4)-O(1)	104.39(9)	C(5)-C(6)-H(6B)	108.3
N(3)-C(3A)-C(9A)	108.92(10)	C(7)-C(6)-H(6B)	108.3
N(3)-C(3A)-C(4)	122.91(11)	H(6A)-C(6)-H(6B)	107.4
C(9A)-C(3A)-C(4)	128.17(11)	C(8)-C(7)-C(6)	116.28(10)
C(3A)-C(4)-C(5)	115.24(10)	C(8)-C(7)-H(7A)	108.2
C(3A)-C(4)-H(4A)	108.5	C(6)-C(7)-H(7A)	108.2
C(5)-C(4)-H(4A)	108.5	C(8)-C(7)-H(7B)	108.2
C(3A)-C(4)-H(4B)	108.5	C(6)-C(7)-H(7B)	108.2
C(5)-C(4)-H(4B)	108.5	H(7A)-C(7)-H(7B)	107.4
H(4A)-C(4)-H(4B)	107.5	C(7)-C(8)-C(9)	114.85(10)
C(4)-C(5)-C(6)	116.84(10)	C(7)-C(8)-H(8A)	108.6
C(4)-C(5)-H(5A)	108.1	C(9)-C(8)-H(8A)	108.6
C(6)-C(5)-H(5A)	108.1	C(7)-C(8)-H(8B)	108.6

C(9)-C(8)-H(8B)	108.6	N(1)-C(10)-C(11)	125.35(11)
H(8A)-C(8)-H(8B)	107.5	C(12)-C(11)-C(10)	101.67(10)
C(9A)-C(9)-C(8)	112.75(9)	C(12)-C(11)-H(11)	129.2
C(9A)-C(9)-H(9A)	109.0	C(10)-C(11)-H(11)	129.2
C(8)-C(9)-H(9A)	109.0	C(11)-C(12)-O(1)	112.28(11)
C(9A)-C(9)-H(9B)	109.0	C(11)-C(12)-C(13)	131.83(11)
C(8)-C(9)-H(9B)	109.0	O(1)-C(12)-C(13)	115.86(10)
H(9A)-C(9)-H(9B)	107.8	F(2)-C(13)-F(3)	108.05(10)
N(1)-C(9A)-C(3A)	103.91(10)	F(2)-C(13)-F(1)	108.17(10)
N(1)-C(9A)-C(9)	128.03(10)	F(3)-C(13)-F(1)	106.55(10)
C(3A)-C(9A)-C(9)	127.96(11)	F(2)-C(13)-C(12)	110.50(10)
N(4)-C(10)-N(1)	120.83(10)	F(3)-C(13)-C(12)	112.21(10)
N(4)-C(10)-C(11)	113.81(10)	F(1)-C(13)-C(12)	111.17(10)

Table 4. Anisotropic displacement parameters ($Å^2 \times 10^3$) for ***LPS222.*** The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
F(1)	26(1)	36(1)	19(1)	-9(1)	6(1)	4(1)
F(2)	29(1)	33(1)	20(1)	-2(1)	-2(1)	-16(1)
F(3)	39(1)	23(1)	23(1)	0(1)	-10(1)	7(1)
O(1)	19(1)	21(1)	14(1)	1(1)	0(1)	-6(1)
N(1)	11(1)	13(1)	14(1)	-1(1)	3(1)	-2(1)
N(2)	14(1)	15(1)	19(1)	1(1)	4(1)	-4(1)
N(3)	16(1)	16(1)	18(1)	1(1)	4(1)	-1(1)
N(4)	18(1)	21(1)	14(1)	0(1)	0(1)	-5(1)
C(3A)	12(1)	15(1)	17(1)	1(1)	2(1)	2(1)
C(4)	18(1)	20(1)	14(1)	2(1)	1(1)	2(1)
C(5)	17(1)	23(1)	14(1)	-1(1)	2(1)	0(1)
C(6)	16(1)	20(1)	19(1)	-3(1)	2(1)	-2(1)
C(7)	14(1)	15(1)	21(1)	-1(1)	2(1)	0(1)
C(8)	16(1)	15(1)	18(1)	2(1)	1(1)	-2(1)
C(9)	10(1)	16(1)	16(1)	0(1)	2(1)	-3(1)
C(9A)	9(1)	13(1)	15(1)	-1(1)	2(1)	1(1)
C(10)	10(1)	13(1)	15(1)	-1(1)	2(1)	1(1)
C(11)	12(1)	15(1)	17(1)	-2(1)	2(1)	-1(1)
C(12)	12(1)	14(1)	18(1)	-3(1)	2(1)	0(1)
C(13)	17(1)	16(1)	16(1)	-2(1)	2(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å ^{2} x 10^3) for ***LPS222***.

Atom	x	y	z	U(iso)
H(4A)	8584	6054	8057	21
H(4B)	5985	5709	8619	21
H(5A)	6225	6670	9431	22
H(5B)	3490	6668	8646	22
H(6A)	8611	7228	7944	22
H(6B)	6256	7610	8591	22
H(7A)	3419	7164	6852	20
H(7B)	4539	7858	6950	20
H(8A)	8468	7627	5883	20
H(8B)	5731	7413	5199	20
H(9A)	8907	6624	4974	17
H(9B)	9743	6622	6284	17
H(11)	778	5320	3531	17

Table 6. Torsion angles [°] for ***LPS222***.

C(9A)-N(1)-N(2)-N(3)	0.31(13)
C(10)-N(1)-N(2)-N(3)	-179.44(9)
N(1)-N(2)-N(3)-C(3A)	-0.11(13)
C(12)-O(1)-N(4)-C(10)	0.29(12)
N(2)-N(3)-C(3A)-C(9A)	-0.12(14)
N(2)-N(3)-C(3A)-C(4)	-179.87(10)
N(3)-C(3A)-C(4)-C(5)	-104.30(14)
C(9A)-C(3A)-C(4)-C(5)	76.00(16)
C(3A)-C(4)-C(5)-C(6)	-66.98(14)
C(4)-C(5)-C(6)-C(7)	73.01(14)
C(5)-C(6)-C(7)-C(8)	-106.54(12)
C(6)-C(7)-C(8)-C(9)	54.59(14)
C(7)-C(8)-C(9)-C(9A)	46.60(14)
N(2)-N(1)-C(9A)-C(3A)	-0.36(12)
C(10)-N(1)-C(9A)-C(3A)	179.34(11)
N(2)-N(1)-C(9A)-C(9)	-176.80(11)
C(10)-N(1)-C(9A)-C(9)	2.9(2)
N(3)-C(3A)-C(9A)-N(1)	0.29(12)
C(4)-C(3A)-C(9A)-N(1)	-179.98(11)
N(3)-C(3A)-C(9A)-C(9)	176.72(11)
C(4)-C(3A)-C(9A)-C(9)	-3.5(2)
C(8)-C(9)-C(9A)-N(1)	90.27(14)
C(8)-C(9)-C(9A)-C(3A)	-85.33(15)
O(1)-N(4)-C(10)-N(1)	179.87(10)
O(1)-N(4)-C(10)-C(11)	-0.40(13)
C(9A)-N(1)-C(10)-N(4)	-0.53(19)
N(2)-N(1)-C(10)-N(4)	179.15(10)
C(9A)-N(1)-C(10)-C(11)	179.77(11)
N(2)-N(1)-C(10)-C(11)	-0.54(16)
N(4)-C(10)-C(11)-C(12)	0.36(13)
N(1)-C(10)-C(11)-C(12)	-179.93(11)
C(10)-C(11)-C(12)-O(1)	-0.15(13)
C(10)-C(11)-C(12)-C(13)	177.87(12)
N(4)-O(1)-C(12)-C(11)	-0.07(13)
N(4)-O(1)-C(12)-C(13)	-178.44(9)
C(11)-C(12)-C(13)-F(2)	2.49(19)
O(1)-C(12)-C(13)-F(2)	-179.54(10)
C(11)-C(12)-C(13)-F(3)	123.16(14)
O(1)-C(12)-C(13)-F(3)	-58.88(14)
C(11)-C(12)-C(13)-F(1)	-117.63(14)
O(1)-C(12)-C(13)-F(1)	60.34(14)

1-[5-(4-Chloro-phenyl)-isoxazol-3-yl]-4-phenyl-1H-[1,2,3]triazole **9b**

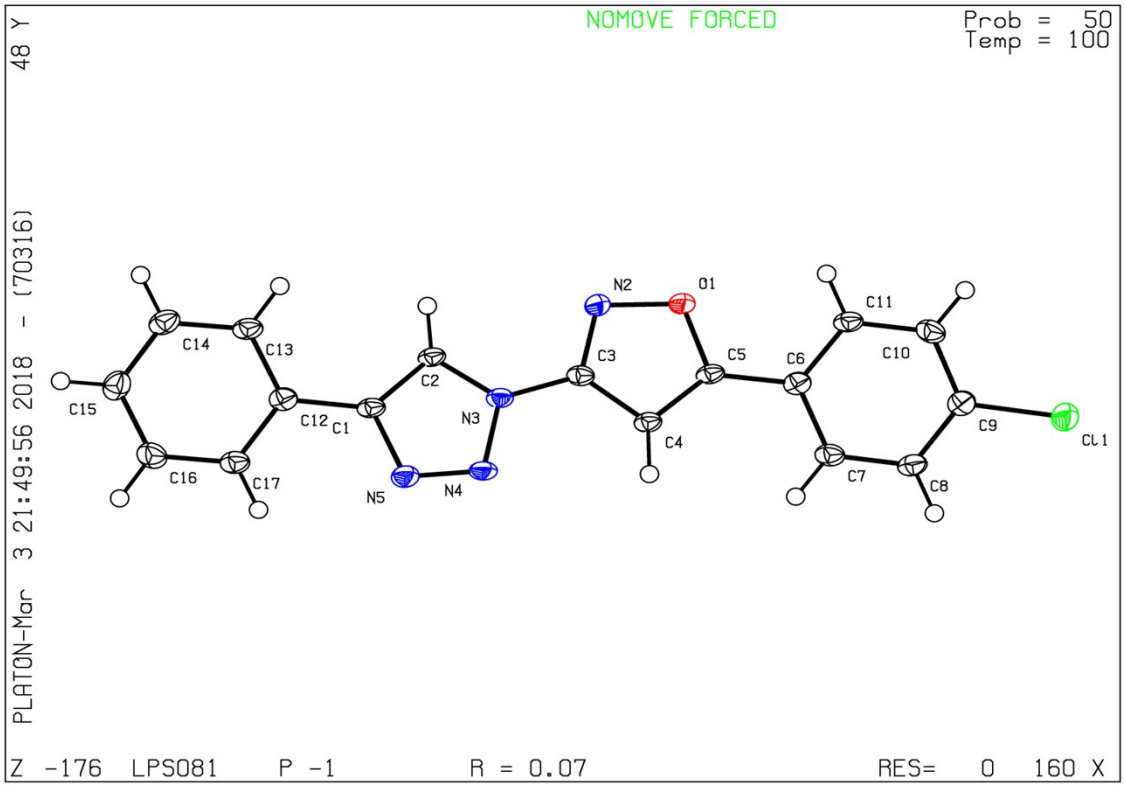


Table 1. Crystal data and structure refinement for **9b**

Identification code	**LPS081**	
Empirical formula	C17 H11 Cl N4 O	
Formula weight	322.75	
Temperature	100(2) K	
Wavelength	0.81182 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 4.6842(9) Å	α = 110.88(3)°.
	b = 11.706(2) Å	β = 93.00(3)°.
	c = 14.673(3) Å	γ = 95.97(3)°.
Volume	744.2(3) $Å^3$	
Z	2	
Density (calculated)	1.440 Mg/m^3	
Absorption coefficient	0.376 mm^{-1}	
F(000)	332	
Crystal size	0.30 x 0.15 x 0.03 mm^3	
Theta range for data collection	3.302 to 31.002°.	
Index ranges	-5<=h<=5, -14<=k<=14, -18<=l<=18	
Reflections collected	14734	
Independent reflections	3134 [R(int) = 0.0856]	

Completeness to theta = 29.150°	99.3 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.980 and 0.890
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3134 / 0 / 208
Goodness-of-fit on F^2	1.012
Final R indices [for 2717 rflns with $I>2\sigma(I)$]	R1 = 0.0672, wR2 = 0.1615
R indices (all data)	R1 = 0.0742, wR2 = 0.1678
Extinction coefficient	n/a
Largest diff. peak and hole	0.987 and -0.760 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for ***LPS081***. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U(eq)
Cl(1)	-9411(1)	3520(1)	434(1)	27(1)
O(1)	1087(3)	7774(1)	3533(1)	22(1)
N(2)	3431(4)	8325(2)	4275(1)	22(1)
N(3)	6091(4)	7668(2)	5376(1)	18(1)
N(4)	6569(4)	6731(2)	5700(1)	21(1)
N(5)	8727(4)	7178(2)	6396(1)	22(1)
C(1)	9644(4)	8402(2)	6532(2)	18(1)
C(2)	7964(4)	8719(2)	5873(2)	18(1)
C(3)	3854(4)	7443(2)	4615(1)	17(1)
C(4)	1951(4)	6327(2)	4156(2)	18(1)
C(5)	265(4)	6583(2)	3476(2)	18(1)
C(6)	-2118(4)	5852(2)	2729(2)	18(1)
C(7)	-3411(4)	4719(2)	2755(2)	21(1)
C(8)	-5673(4)	4009(2)	2053(2)	21(1)
C(9)	-6627(4)	4440(2)	1328(2)	21(1)
C(10)	-5393(5)	5568(2)	1292(2)	23(1)
C(11)	-3136(4)	6271(2)	1996(2)	20(1)
C(12)	12037(4)	9135(2)	7289(2)	19(1)
C(13)	13415(4)	10268(2)	7294(2)	22(1)
C(14)	15700(5)	10933(2)	8012(2)	24(1)
C(15)	16622(5)	10468(2)	8722(2)	26(1)
C(16)	15251(5)	9343(2)	8722(2)	26(1)
C(17)	12956(5)	8680(2)	8012(2)	22(1)

Table 3. Bond lengths [Å] and angles [°] for ***LPS081***.

Cl(1)-C(9)	1.761(2)
O(1)-C(5)	1.378(3)
O(1)-N(2)	1.426(2)
N(2)-C(3)	1.325(3)
N(3)-C(2)	1.373(3)
N(3)-N(4)	1.374(3)
N(3)-C(3)	1.422(3)
N(4)-N(5)	1.319(3)
N(5)-C(1)	1.392(3)
C(1)-C(2)	1.388(3)
C(1)-C(12)	1.489(3)
C(2)-H(2)	0.9500
C(3)-C(4)	1.422(3)
C(4)-C(5)	1.374(3)
C(4)-H(4)	0.9500
C(5)-C(6)	1.478(3)
C(6)-C(11)	1.413(3)
C(6)-C(7)	1.414(3)
C(7)-C(8)	1.403(3)
C(7)-H(7)	0.9500
C(8)-C(9)	1.402(3)
C(8)-H(8)	0.9500
C(9)-C(10)	1.405(3)
C(10)-C(11)	1.400(3)
C(10)-H(10)	0.9500
C(11)-H(11)	0.9500
C(12)-C(13)	1.411(3)
C(12)-C(17)	1.414(3)
C(13)-C(14)	1.409(3)
C(13)-H(13)	0.9500
C(14)-C(15)	1.406(3)
C(14)-H(14)	0.9500
C(15)-C(16)	1.404(4)
C(15)-H(15)	0.9500
C(16)-C(17)	1.405(3)
C(16)-H(16)	0.9500
C(17)-H(17)	0.9500

C(5)-O(1)-N(2)	109.07(15)
C(3)-N(2)-O(1)	103.64(16)
C(2)-N(3)-N(4)	111.39(16)
C(2)-N(3)-C(3)	129.80(19)
N(4)-N(3)-C(3)	118.81(18)
N(5)-N(4)-N(3)	106.52(17)
N(4)-N(5)-C(1)	109.74(17)
C(2)-C(1)-N(5)	108.05(18)
C(2)-C(1)-C(12)	131.2(2)
N(5)-C(1)-C(12)	120.79(18)
N(3)-C(2)-C(1)	104.30(19)
N(3)-C(2)-H(2)	127.8
C(1)-C(2)-H(2)	127.8
N(2)-C(3)-N(3)	119.4(2)
N(2)-C(3)-C(4)	114.47(18)
N(3)-C(3)-C(4)	126.1(2)
C(5)-C(4)-C(3)	103.01(19)
C(5)-C(4)-H(4)	128.5
C(3)-C(4)-H(4)	128.5
C(4)-C(5)-O(1)	109.80(18)
C(4)-C(5)-C(6)	133.3(2)
O(1)-C(5)-C(6)	116.92(17)
C(11)-C(6)-C(7)	119.58(19)
C(11)-C(6)-C(5)	121.1(2)
C(7)-C(6)-C(5)	119.30(19)
C(8)-C(7)-C(6)	120.3(2)
C(8)-C(7)-H(7)	119.9
C(6)-C(7)-H(7)	119.9
C(9)-C(8)-C(7)	119.0(2)
C(9)-C(8)-H(8)	120.5
C(7)-C(8)-H(8)	120.5
C(8)-C(9)-C(10)	121.7(2)
C(8)-C(9)-Cl(1)	118.46(18)
C(10)-C(9)-Cl(1)	119.82(16)
C(11)-C(10)-C(9)	118.9(2)
C(11)-C(10)-H(10)	120.6
C(9)-C(10)-H(10)	120.6
C(10)-C(11)-C(6)	120.5(2)
C(10)-C(11)-H(11)	119.7

C(6)-C(11)-H(11)	119.7
C(13)-C(12)-C(17)	119.5(2)
C(13)-C(12)-C(1)	121.28(19)
C(17)-C(12)-C(1)	119.3(2)
C(14)-C(13)-C(12)	120.0(2)
C(14)-C(13)-H(13)	120.0
C(12)-C(13)-H(13)	120.0
C(15)-C(14)-C(13)	120.2(2)
C(15)-C(14)-H(14)	119.9
C(13)-C(14)-H(14)	119.9
C(16)-C(15)-C(14)	120.0(2)
C(16)-C(15)-H(15)	120.0
C(14)-C(15)-H(15)	120.0
C(15)-C(16)-C(17)	120.1(2)
C(15)-C(16)-H(16)	119.9
C(17)-C(16)-H(16)	119.9
C(16)-C(17)-C(12)	120.3(2)
C(16)-C(17)-H(17)	119.9
C(12)-C(17)-H(17)	119.9

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for ***LPS081***. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	23(1)	25(1)	34(1)	11(1)	-4(1)	1(1)
O(1)	23(1)	16(1)	29(1)	12(1)	-4(1)	1(1)
N(2)	21(1)	18(1)	29(1)	11(1)	-4(1)	0(1)
N(3)	18(1)	15(1)	24(1)	10(1)	3(1)	4(1)
N(4)	22(1)	16(1)	30(1)	14(1)	0(1)	3(1)
N(5)	21(1)	17(1)	30(1)	12(1)	0(1)	3(1)
C(1)	16(1)	15(1)	27(1)	11(1)	5(1)	5(1)
C(2)	18(1)	12(1)	25(1)	9(1)	3(1)	3(1)
C(3)	16(1)	16(1)	23(1)	10(1)	5(1)	5(1)
C(4)	17(1)	14(1)	25(1)	9(1)	4(1)	4(1)
C(5)	17(1)	12(1)	26(1)	9(1)	5(1)	5(1)
C(6)	15(1)	15(1)	26(1)	8(1)	5(1)	6(1)
C(7)	21(1)	18(1)	27(1)	11(1)	4(1)	7(1)
C(8)	18(1)	17(1)	31(1)	12(1)	4(1)	3(1)
C(9)	16(1)	18(1)	28(1)	8(1)	3(1)	6(1)
C(10)	23(1)	22(1)	27(1)	13(1)	2(1)	8(1)
C(11)	21(1)	15(1)	29(1)	12(1)	4(1)	5(1)
C(12)	16(1)	17(1)	27(1)	9(1)	5(1)	6(1)
C(13)	21(1)	16(1)	31(1)	12(1)	4(1)	6(1)
C(14)	21(1)	17(1)	36(1)	10(1)	4(1)	4(1)
C(15)	23(1)	19(1)	33(1)	4(1)	-2(1)	4(1)
C(16)	29(1)	22(1)	28(1)	10(1)	0(1)	8(1)
C(17)	22(1)	18(1)	27(1)	10(1)	4(1)	5(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for ***LPS081***.

Atom	x	y	z	U(iso)
H(2)	8083	9488	5786	21
H(4)	1866	5587	4287	22
H(7)	-2746	4438	3251	25
H(8)	-6545	3247	2068	25
H(10)	-6078	5849	799	27
H(11)	-2283	7036	1981	24
H(13)	12804	10583	6812	26
H(14)	16619	11698	8016	29
H(15)	18172	10915	9203	31
H(16)	15875	9028	9202	31
H(17)	12019	7924	8019	26

Table 6. Torsion angles [°] for ***LPS081***.

C(5)-O(1)-N(2)-C(3)	-0.3(2)
C(2)-N(3)-N(4)-N(5)	-0.1(2)
C(3)-N(3)-N(4)-N(5)	-179.97(17)
N(3)-N(4)-N(5)-C(1)	0.4(2)
N(4)-N(5)-C(1)-C(2)	-0.5(2)
N(4)-N(5)-C(1)-C(12)	179.04(17)
N(4)-N(3)-C(2)-C(1)	-0.3(2)
C(3)-N(3)-C(2)-C(1)	179.63(19)
N(5)-C(1)-C(2)-N(3)	0.5(2)
C(12)-C(1)-C(2)-N(3)	-179.0(2)
O(1)-N(2)-C(3)-N(3)	179.54(16)
O(1)-N(2)-C(3)-C(4)	-0.1(2)
C(2)-N(3)-C(3)-N(2)	2.2(3)
N(4)-N(3)-C(3)-N(2)	-177.87(17)
C(2)-N(3)-C(3)-C(4)	-178.20(18)
N(4)-N(3)-C(3)-C(4)	1.7(3)
N(2)-C(3)-C(4)-C(5)	0.4(2)
N(3)-C(3)-C(4)-C(5)	-179.15(19)
C(3)-C(4)-C(5)-O(1)	-0.6(2)
C(3)-C(4)-C(5)-C(6)	178.8(2)
N(2)-O(1)-C(5)-C(4)	0.6(2)
N(2)-O(1)-C(5)-C(6)	-178.89(16)
C(4)-C(5)-C(6)-C(11)	-168.1(2)
O(1)-C(5)-C(6)-C(11)	11.3(3)
C(4)-C(5)-C(6)-C(7)	12.4(3)
O(1)-C(5)-C(6)-C(7)	-168.20(17)
C(11)-C(6)-C(7)-C(8)	0.6(3)
C(5)-C(6)-C(7)-C(8)	-179.87(17)
C(6)-C(7)-C(8)-C(9)	-0.1(3)
C(7)-C(8)-C(9)-C(10)	-0.5(3)
C(7)-C(8)-C(9)-Cl(1)	178.49(14)
C(8)-C(9)-C(10)-C(11)	0.5(3)
Cl(1)-C(9)-C(10)-C(11)	-178.46(15)
C(9)-C(10)-C(11)-C(6)	0.0(3)
C(7)-C(6)-C(11)-C(10)	-0.6(3)
C(5)-C(6)-C(11)-C(10)	179.90(17)
C(2)-C(1)-C(12)-C(13)	-14.8(3)
N(5)-C(1)-C(12)-C(13)	165.73(18)
C(2)-C(1)-C(12)-C(17)	165.7(2)
N(5)-C(1)-C(12)-C(17)	-13.8(3)
C(17)-C(12)-C(13)-C(14)	0.4(3)
C(1)-C(12)-C(13)-C(14)	-179.14(17)
C(12)-C(13)-C(14)-C(15)	0.3(3)
C(13)-C(14)-C(15)-C(16)	-0.5(3)
C(14)-C(15)-C(16)-C(17)	0.0(3)
C(15)-C(16)-C(17)-C(12)	0.7(3)
C(13)-C(12)-C(17)-C(16)	-0.9(3)
C(1)-C(12)-C(17)-C(16)	178.62(17)

3-Imino-(triphenyl)-phosphorano-5-(4-bromophenyl)-isoxazole **11a**

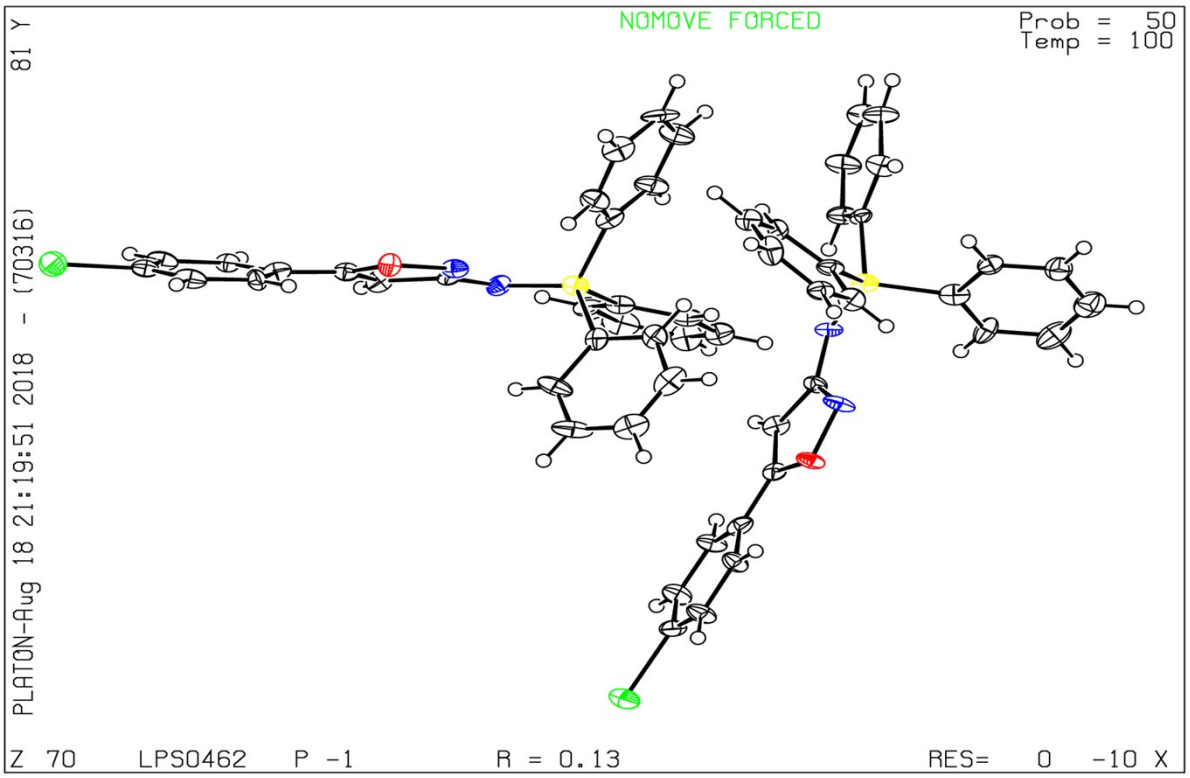


Table 1. Crystal data and structure refinement for **11a.**

Identification code	**LPS0462**	
Empirical formula	C27 H20 Br N2 O P	
Formula weight	499.32	
Temperature	100(2) K	
Wavelength	0.96990 Å	
Crystal system	Triclinic	
Space group	*P*-1	
Unit cell dimensions	a = 10.480(2) Å	α = 85.69(3)°.
	b = 11.525(2) Å	β = 82.79(3)°.
	c = 21.545(4) Å	γ = 66.42(3)°.
Volume	2365.2(9) $Å^3$	
Z	4	
Density (calculated)	1.402 Mg/m^3	
Absorption coefficient	0.854 mm^{-1}	
F(000)	1016	
Crystal size	0.30 x 0.02 x 0.01 mm^3	
Theta range for data collection	3.310 to 35.999°.	
Index ranges	-12<=h<=12, -13<=k<=12, -26<=l<=19	
Reflections collected	20295	
Independent reflections	8256 [R(int) = 0.1129]	
Completeness to theta = 35.587°	99.6 %	

Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.980 and 0.800
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8256 / 0 / 579
Goodness-of-fit on F^2	0.932
Final R indices [for 4845 rflns with $I>2\sigma(I)$]	R1 = 0.1279, wR2 = 0.2490
R indices (all data)	R1 = 0.1728, wR2 = 0.2775
Extinction coefficient	0.0179(18)
Largest diff. peak and hole	1.553 and -0.682 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for ***LPS0462***. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U(eq)
Br(1)	9886(1)	342(1)	-910(1)	40(1)
P(1)	4840(2)	8798(2)	1943(1)	21(1)
O(1)	7782(4)	6031(4)	582(2)	23(1)
N(1)	4701(5)	7668(4)	1603(2)	21(1)
N(2)	6950(5)	7107(5)	978(3)	27(1)
C(3)	5795(6)	6879(5)	1184(3)	19(2)
C(4)	5817(6)	5763(6)	927(3)	20(2)
C(5)	7062(6)	5276(6)	562(3)	19(2)
C(6)	3319(6)	9390(6)	2522(3)	19(2)
C(7)	2186(6)	9057(6)	2520(3)	27(2)
C(8)	989(6)	9594(7)	2945(3)	32(2)
C(9)	918(6)	10461(7)	3358(3)	31(2)
C(10)	2066(7)	10789(7)	3381(3)	36(2)
C(11)	3270(6)	10283(6)	2953(3)	26(2)
C(12)	4722(5)	10199(6)	1433(3)	24(2)
C(13)	4081(6)	10340(7)	879(3)	31(2)
C(14)	3803(7)	11451(8)	517(4)	41(2)
C(15)	4116(7)	12406(8)	708(4)	40(2)
C(16)	4754(7)	12267(7)	1259(4)	34(2)
C(17)	5068(6)	11145(6)	1617(3)	24(2)
C(18)	6384(6)	8397(6)	2373(3)	23(2)
C(19)	7625(6)	8455(6)	2054(3)	27(2)
C(20)	8819(6)	8045(6)	2374(3)	24(2)
C(21)	8791(6)	7610(6)	2993(4)	32(2)
C(22)	7549(6)	7534(6)	3302(3)	26(2)

C(23)	6364(6)	7899(5)	2984(3)	22(2)
C(24)	7738(6)	4120(6)	205(3)	24(2)
C(25)	9165(6)	3685(6)	-32(3)	24(2)
C(26)	9823(6)	2570(6)	-366(3)	30(2)
C(27)	9004(7)	1904(6)	-463(3)	28(2)
C(28)	7613(6)	2301(6)	-245(3)	29(2)
C(29)	6965(6)	3432(6)	83(3)	24(2)
Br(2)	10202(1)	-4833(1)	6015(1)	36(1)
P(2)	5133(2)	3793(2)	3165(1)	21(1)
O(2)	7992(4)	890(4)	4397(2)	24(1)
N(3)	5144(5)	2477(4)	3494(2)	23(1)
N(4)	7204(5)	1954(5)	4019(2)	22(1)
C(30)	6129(5)	1717(6)	3864(3)	16(1)
C(31)	6212(6)	503(6)	4132(3)	24(2)
C(32)	7362(6)	59(6)	4458(3)	21(2)
C(33)	3731(5)	4317(6)	2669(3)	22(2)
C(34)	2796(6)	3716(6)	2711(3)	25(2)
C(35)	1736(6)	4094(7)	2316(4)	36(2)
C(36)	1600(6)	5068(7)	1881(4)	32(2)
C(37)	2474(6)	5690(7)	1844(3)	30(2)
C(38)	3520(6)	5339(6)	2232(3)	26(2)
C(39)	4827(6)	5092(6)	3684(3)	26(2)
C(40)	3531(6)	6127(6)	3744(3)	33(2)
C(41)	3301(7)	7111(7)	4148(4)	37(2)
C(42)	4392(7)	7041(7)	4494(3)	38(2)
C(43)	5655(7)	6000(7)	4432(3)	34(2)
C(44)	5897(6)	5053(6)	4021(3)	30(2)
C(45)	6714(6)	3624(6)	2653(3)	22(2)
C(46)	7069(6)	4660(6)	2460(3)	26(2)
C(47)	8251(7)	4474(8)	2033(3)	37(2)
C(48)	9058(7)	3342(8)	1795(4)	38(2)
C(49)	8745(7)	2291(8)	1992(4)	42(2)
C(50)	7565(7)	2425(6)	2410(3)	34(2)
C(51)	8051(6)	-1127(6)	4808(3)	24(2)
C(52)	9459(6)	-1519(6)	4926(3)	24(2)
C(53)	10097(6)	-2631(6)	5271(3)	28(2)
C(54)	9317(6)	-3320(6)	5505(3)	26(2)
C(55)	7930(6)	-2972(6)	5400(3)	27(2)
C(56)	7306(6)	-1889(6)	5049(3)	21(2)

Table 3. Bond lengths [Å] and angles [°] for ***LPS0462***.

Br(1)-C(27)	1.931(6)	C(19)-H(19)	0.9500
P(1)-N(1)	1.606(5)	C(20)-C(21)	1.389(9)
P(1)-C(6)	1.822(6)	C(20)-H(20)	0.9500
P(1)-C(18)	1.845(7)	C(21)-C(22)	1.418(9)
P(1)-C(12)	1.853(7)	C(21)-H(21)	0.9500
O(1)-C(5)	1.366(7)	C(22)-C(23)	1.394(8)
O(1)-N(2)	1.461(6)	C(22)-H(22)	0.9500
N(1)-C(3)	1.409(7)	C(23)-H(23)	0.9500
N(2)-C(3)	1.354(8)	C(24)-C(29)	1.398(9)
C(3)-C(4)	1.429(8)	C(24)-C(25)	1.412(8)
C(4)-C(5)	1.364(8)	C(25)-C(26)	1.397(8)
C(4)-H(4)	0.9500	C(25)-H(25)	0.9500
C(5)-C(24)	1.460(8)	C(26)-C(27)	1.402(9)
C(6)-C(7)	1.386(8)	C(26)-H(26)	0.9500
C(6)-C(11)	1.420(8)	C(27)-C(28)	1.372(8)
C(7)-C(8)	1.404(8)	C(28)-C(29)	1.403(8)
C(7)-H(7)	0.9500	C(28)-H(28)	0.9500
C(8)-C(9)	1.362(9)	C(29)-H(29)	0.9500
C(8)-H(8)	0.9500	Br(2)-C(54)	1.945(7)
C(9)-C(10)	1.404(9)	P(2)-N(3)	1.621(5)
C(9)-H(9)	0.9500	P(2)-C(33)	1.803(6)
C(10)-C(11)	1.408(8)	P(2)-C(45)	1.821(6)
C(10)-H(10)	0.9500	P(2)-C(39)	1.836(7)
C(11)-H(11)	0.9500	O(2)-C(32)	1.355(7)
C(12)-C(17)	1.377(8)	O(2)-N(4)	1.434(7)
C(12)-C(13)	1.410(9)	N(3)-C(30)	1.360(7)
C(13)-C(14)	1.393(10)	N(4)-C(30)	1.346(7)
C(13)-H(13)	0.9500	C(30)-C(31)	1.446(9)
C(14)-C(15)	1.370(10)	C(31)-C(32)	1.369(8)
C(14)-H(14)	0.9500	C(31)-H(31)	0.9500
C(15)-C(16)	1.403(11)	C(32)-C(51)	1.466(9)
C(15)-H(15)	0.9500	C(33)-C(34)	1.399(8)
C(16)-C(17)	1.395(9)	C(33)-C(38)	1.414(9)
C(16)-H(16)	0.9500	C(34)-C(35)	1.393(9)
C(17)-H(17)	0.9500	C(34)-H(34)	0.9500
C(18)-C(23)	1.396(9)	C(35)-C(36)	1.381(10)
C(18)-C(19)	1.417(8)	C(35)-H(35)	0.9500
C(19)-C(20)	1.399(9)	C(36)-C(37)	1.363(9)

C(36)-H(36)	0.9500
C(37)-C(38)	1.375(9)
C(37)-H(37)	0.9500
C(38)-H(38)	0.9500
C(39)-C(44)	1.395(9)
C(39)-C(40)	1.403(8)
C(40)-C(41)	1.407(9)
C(40)-H(40)	0.9500
C(41)-C(42)	1.415(10)
C(41)-H(41)	0.9500
C(42)-C(43)	1.385(9)
C(42)-H(42)	0.9500
C(43)-C(44)	1.382(9)
C(43)-H(43)	0.9500
C(44)-H(44)	0.9500
C(45)-C(50)	1.409(8)
C(45)-C(46)	1.408(9)
C(46)-C(47)	1.398(9)
C(46)-H(46)	0.9500
C(47)-C(48)	1.337(10)
C(47)-H(47)	0.9500
C(48)-C(49)	1.400(10)
C(48)-H(48)	0.9500
C(49)-C(50)	1.398(9)
C(49)-H(49)	0.9500
C(50)-H(50)	0.9500
C(51)-C(52)	1.410(8)
C(51)-C(56)	1.425(9)
C(52)-C(53)	1.393(9)
C(52)-H(52)	0.9500
C(53)-C(54)	1.382(9)
C(53)-H(53)	0.9500
C(54)-C(55)	1.388(8)
C(55)-C(56)	1.374(9)
C(55)-H(55)	0.9500
C(56)-H(56)	0.9500

N(1)-P(1)-C(6)	106.2(3)
N(1)-P(1)-C(18)	117.4(3)
C(6)-P(1)-C(18)	106.1(3)
N(1)-P(1)-C(12)	115.0(3)
C(6)-P(1)-C(12)	104.4(3)
C(18)-P(1)-C(12)	106.7(3)
C(5)-O(1)-N(2)	109.5(4)
C(3)-N(1)-P(1)	121.5(4)
C(3)-N(2)-O(1)	103.0(4)
N(2)-C(3)-N(1)	123.1(5)
N(2)-C(3)-C(4)	112.5(5)
N(1)-C(3)-C(4)	124.4(5)
C(5)-C(4)-C(3)	105.3(5)
C(5)-C(4)-H(4)	127.3
C(3)-C(4)-H(4)	127.3
C(4)-C(5)-O(1)	109.7(5)
C(4)-C(5)-C(24)	132.2(6)
O(1)-C(5)-C(24)	118.0(5)
C(7)-C(6)-C(11)	120.0(5)
C(7)-C(6)-P(1)	121.7(5)
C(11)-C(6)-P(1)	118.2(4)
C(6)-C(7)-C(8)	120.4(6)
C(6)-C(7)-H(7)	119.8
C(8)-C(7)-H(7)	119.8
C(9)-C(8)-C(7)	120.4(6)
C(9)-C(8)-H(8)	119.8
C(7)-C(8)-H(8)	119.8
C(8)-C(9)-C(10)	120.3(6)
C(8)-C(9)-H(9)	119.8
C(10)-C(9)-H(9)	119.8
C(9)-C(10)-C(11)	120.4(6)
C(9)-C(10)-H(10)	119.8
C(11)-C(10)-H(10)	119.8
C(10)-C(11)-C(6)	118.3(5)
C(10)-C(11)-H(11)	120.8
C(6)-C(11)-H(11)	120.8
C(17)-C(12)-C(13)	120.6(7)
C(17)-C(12)-P(1)	122.3(5)
C(13)-C(12)-P(1)	116.6(5)
C(14)-C(13)-C(12)	119.2(7)

C(14)-C(13)-H(13)	120.4
C(12)-C(13)-H(13)	120.4
C(15)-C(14)-C(13)	120.2(7)
C(15)-C(14)-H(14)	119.9
C(13)-C(14)-H(14)	119.9
C(14)-C(15)-C(16)	120.7(7)
C(14)-C(15)-H(15)	119.6
C(16)-C(15)-H(15)	119.6
C(17)-C(16)-C(15)	119.5(7)
C(17)-C(16)-H(16)	120.3
C(15)-C(16)-H(16)	120.3
C(12)-C(17)-C(16)	119.8(6)
C(12)-C(17)-H(17)	120.1
C(16)-C(17)-H(17)	120.1
C(23)-C(18)-C(19)	120.9(6)
C(23)-C(18)-P(1)	119.4(4)
C(19)-C(18)-P(1)	119.3(5)
C(20)-C(19)-C(18)	118.4(7)
C(20)-C(19)-H(19)	120.8
C(18)-C(19)-H(19)	120.8
C(21)-C(20)-C(19)	121.2(6)
C(21)-C(20)-H(20)	119.4
C(19)-C(20)-H(20)	119.4
C(20)-C(21)-C(22)	119.7(6)
C(20)-C(21)-H(21)	120.1
C(22)-C(21)-H(21)	120.1
C(23)-C(22)-C(21)	119.7(7)
C(23)-C(22)-H(22)	120.1
C(21)-C(22)-H(22)	120.1
C(22)-C(23)-C(18)	119.9(6)
C(22)-C(23)-H(23)	120.1
C(18)-C(23)-H(23)	120.1
C(29)-C(24)-C(25)	118.7(5)
C(29)-C(24)-C(5)	120.0(5)
C(25)-C(24)-C(5)	121.3(6)
C(26)-C(25)-C(24)	121.6(6)
C(26)-C(25)-H(25)	119.2
C(24)-C(25)-H(25)	119.2
C(25)-C(26)-C(27)	117.2(6)
C(25)-C(26)-H(26)	121.4

C(27)-C(26)-H(26)	121.4
C(28)-C(27)-C(26)	123.0(6)
C(28)-C(27)-Br(1)	118.6(5)
C(26)-C(27)-Br(1)	118.4(5)
C(27)-C(28)-C(29)	119.0(6)
C(27)-C(28)-H(28)	120.5
C(29)-C(28)-H(28)	120.5
C(24)-C(29)-C(28)	120.4(5)
C(24)-C(29)-H(29)	119.8
C(28)-C(29)-H(29)	119.8
N(3)-P(2)-C(33)	105.3(3)
N(3)-P(2)-C(45)	114.2(3)
C(33)-P(2)-C(45)	106.1(3)
N(3)-P(2)-C(39)	117.1(3)
C(33)-P(2)-C(39)	107.7(3)
C(45)-P(2)-C(39)	105.7(3)
C(32)-O(2)-N(4)	108.6(4)
C(30)-N(3)-P(2)	123.9(4)
C(30)-N(4)-O(2)	106.2(5)
N(4)-C(30)-N(3)	125.4(6)
N(4)-C(30)-C(31)	109.6(5)
N(3)-C(30)-C(31)	125.0(5)
C(32)-C(31)-C(30)	105.6(5)
C(32)-C(31)-H(31)	127.2
C(30)-C(31)-H(31)	127.2
O(2)-C(32)-C(31)	110.0(6)
O(2)-C(32)-C(51)	116.8(5)
C(31)-C(32)-C(51)	133.0(6)
C(34)-C(33)-C(38)	117.5(6)
C(34)-C(33)-P(2)	120.2(5)
C(38)-C(33)-P(2)	122.3(4)
C(35)-C(34)-C(33)	120.4(7)
C(35)-C(34)-H(34)	119.8
C(33)-C(34)-H(34)	119.8
C(36)-C(35)-C(34)	120.1(6)
C(36)-C(35)-H(35)	120.0
C(34)-C(35)-H(35)	120.0
C(37)-C(36)-C(35)	120.5(6)
C(37)-C(36)-H(36)	119.8
C(35)-C(36)-H(36)	119.8

C(36)-C(37)-C(38)	120.3(7)
C(36)-C(37)-H(37)	119.8
C(38)-C(37)-H(37)	119.8
C(37)-C(38)-C(33)	121.1(6)
C(37)-C(38)-H(38)	119.5
C(33)-C(38)-H(38)	119.5
C(44)-C(39)-C(40)	119.5(6)
C(44)-C(39)-P(2)	119.7(5)
C(40)-C(39)-P(2)	120.8(5)
C(39)-C(40)-C(41)	120.4(6)
C(39)-C(40)-H(40)	119.8
C(41)-C(40)-H(40)	119.8
C(40)-C(41)-C(42)	119.2(6)
C(40)-C(41)-H(41)	120.4
C(42)-C(41)-H(41)	120.4
C(43)-C(42)-C(41)	119.2(6)
C(43)-C(42)-H(42)	120.4
C(41)-C(42)-H(42)	120.4
C(44)-C(43)-C(42)	121.7(6)
C(44)-C(43)-H(43)	119.2
C(42)-C(43)-H(43)	119.2
C(43)-C(44)-C(39)	119.9(6)
C(43)-C(44)-H(44)	120.0
C(39)-C(44)-H(44)	120.0
C(50)-C(45)-C(46)	118.8(6)
C(50)-C(45)-P(2)	118.6(5)
C(46)-C(45)-P(2)	122.5(4)
C(47)-C(46)-C(45)	119.3(6)
C(47)-C(46)-H(46)	120.3
C(45)-C(46)-H(46)	120.3
C(48)-C(47)-C(46)	122.3(7)
C(48)-C(47)-H(47)	118.8
C(46)-C(47)-H(47)	118.8
C(47)-C(48)-C(49)	119.4(7)
C(47)-C(48)-H(48)	120.3
C(49)-C(48)-H(48)	120.3
C(50)-C(49)-C(48)	120.7(6)
C(50)-C(49)-H(49)	119.7
C(48)-C(49)-H(49)	119.7
C(49)-C(50)-C(45)	119.4(7)

C(49)-C(50)-H(50)	120.3
C(45)-C(50)-H(50)	120.3
C(52)-C(51)-C(56)	118.7(6)
C(52)-C(51)-C(32)	120.5(6)
C(56)-C(51)-C(32)	120.8(5)
C(53)-C(52)-C(51)	120.2(6)
C(53)-C(52)-H(52)	119.9
C(51)-C(52)-H(52)	119.9
C(54)-C(53)-C(52)	118.7(6)
C(54)-C(53)-H(53)	120.6
C(52)-C(53)-H(53)	120.6
C(53)-C(54)-C(55)	123.1(6)
C(53)-C(54)-Br(2)	118.2(5)
C(55)-C(54)-Br(2)	118.7(5)
C(56)-C(55)-C(54)	118.3(6)
C(56)-C(55)-H(55)	120.8
C(54)-C(55)-H(55)	120.8
C(55)-C(56)-C(51)	121.0(6)
C(55)-C(56)-H(56)	119.5
C(51)-C(56)-H(56)	119.5

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for ***LPS0462***. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	49(1)	30(1)	35(1)	-13(1)	6(1)	-12(1)
P(1)	14(1)	26(1)	22(1)	-7(1)	-1(1)	-7(1)
O(1)	16(2)	24(2)	30(3)	-14(2)	11(2)	-11(2)
N(1)	17(3)	28(3)	16(3)	-10(3)	2(2)	-7(2)
N(2)	20(3)	27(3)	34(4)	-20(3)	9(3)	-8(2)
C(3)	14(3)	19(3)	21(4)	-5(3)	-1(3)	-1(3)
C(4)	14(3)	32(4)	22(4)	-3(3)	-3(3)	-15(3)
C(5)	19(3)	25(3)	14(4)	-2(3)	-8(3)	-9(3)
C(6)	14(3)	23(3)	12(4)	0(3)	0(3)	-1(3)
C(7)	25(4)	36(4)	26(5)	-12(3)	7(3)	-19(3)
C(8)	11(3)	44(4)	36(5)	-17(4)	3(3)	-4(3)
C(9)	8(3)	42(4)	38(5)	-8(4)	12(3)	-7(3)
C(10)	27(4)	48(5)	28(5)	-18(4)	11(3)	-12(3)
C(11)	13(3)	33(4)	34(5)	-10(3)	-5(3)	-10(3)
C(12)	7(3)	38(4)	28(5)	-7(3)	-5(3)	-6(3)
C(13)	35(4)	40(4)	21(5)	11(4)	-14(3)	-17(3)
C(14)	39(4)	66(6)	27(5)	5(4)	-8(4)	-31(4)
C(15)	31(4)	49(5)	37(6)	1(4)	6(4)	-16(4)
C(16)	41(4)	33(4)	31(5)	-2(4)	1(4)	-19(4)
C(17)	25(3)	30(4)	19(4)	-2(3)	-4(3)	-13(3)
C(18)	22(3)	27(4)	23(5)	-10(3)	9(3)	-14(3)
C(19)	12(3)	31(4)	33(5)	-1(3)	0(3)	-3(3)
C(20)	28(4)	28(4)	24(5)	-8(3)	1(3)	-18(3)
C(21)	27(4)	30(4)	44(6)	6(4)	-18(4)	-12(3)
C(22)	19(3)	31(4)	23(4)	-1(3)	-10(3)	-2(3)
C(23)	18(3)	18(3)	28(5)	1(3)	5(3)	-7(3)
C(24)	17(3)	35(4)	15(4)	2(3)	-2(3)	-5(3)
C(25)	16(3)	21(3)	33(5)	-7(3)	4(3)	-4(3)
C(26)	21(3)	26(4)	37(5)	-13(3)	8(3)	-4(3)
C(27)	32(4)	30(4)	17(4)	-7(3)	1(3)	-7(3)
C(28)	22(4)	28(4)	35(5)	-10(3)	1(3)	-7(3)
C(29)	16(3)	29(4)	28(5)	-14(3)	-1(3)	-10(3)
Br(2)	31(1)	34(1)	36(1)	2(1)	-1(1)	-4(1)
P(2)	12(1)	26(1)	26(1)	-4(1)	-2(1)	-6(1)
O(2)	22(2)	25(2)	27(3)	3(2)	-5(2)	-12(2)

N(3)	23(3)	29(3)	20(4)	1(3)	0(2)	-16(3)
N(4)	13(3)	28(3)	22(4)	-5(3)	-2(2)	-3(2)
C(30)	13(3)	22(3)	10(4)	-3(3)	-6(3)	-3(3)
C(31)	15(3)	36(4)	27(4)	1(3)	-1(3)	-17(3)
C(32)	18(3)	26(4)	20(4)	-5(3)	-4(3)	-7(3)
C(33)	9(3)	33(4)	24(4)	-9(3)	0(3)	-9(3)
C(34)	25(3)	36(4)	17(4)	-3(3)	-5(3)	-11(3)
C(35)	18(3)	49(5)	47(6)	-17(4)	-6(3)	-17(3)
C(36)	17(3)	44(5)	40(5)	0(4)	-14(3)	-13(3)
C(37)	21(3)	37(4)	30(5)	-3(3)	-1(3)	-10(3)
C(38)	12(3)	36(4)	26(5)	-10(3)	9(3)	-7(3)
C(39)	16(3)	39(4)	19(4)	1(3)	-2(3)	-9(3)
C(40)	23(4)	40(4)	36(5)	-16(4)	11(3)	-13(3)
C(41)	22(4)	39(4)	43(6)	-17(4)	4(3)	-6(3)
C(42)	43(4)	57(5)	21(5)	-23(4)	18(4)	-29(4)
C(43)	29(4)	48(5)	33(5)	-3(4)	-5(3)	-24(4)
C(44)	20(3)	32(4)	36(5)	-6(4)	2(3)	-9(3)
C(45)	17(3)	26(4)	22(4)	4(3)	-12(3)	-4(3)
C(46)	16(3)	29(4)	31(5)	3(3)	-7(3)	-4(3)
C(47)	20(4)	60(5)	33(5)	10(4)	-8(3)	-19(4)
C(48)	17(3)	58(5)	33(5)	-2(4)	-6(3)	-8(4)
C(49)	31(4)	55(5)	39(6)	-30(4)	13(4)	-17(4)
C(50)	30(4)	33(4)	42(5)	-23(4)	-5(3)	-10(3)
C(51)	17(3)	38(4)	20(4)	-4(3)	-4(3)	-11(3)
C(52)	26(3)	16(3)	25(5)	1(3)	-6(3)	-3(3)
C(53)	17(3)	36(4)	32(5)	-14(3)	0(3)	-9(3)
C(54)	22(3)	27(4)	24(5)	-8(3)	5(3)	-6(3)
C(55)	21(3)	31(4)	31(5)	-14(3)	1(3)	-11(3)
C(56)	14(3)	25(4)	19(4)	-3(3)	-8(3)	-1(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for ***LPS0462***.

Atom	x	y	z	U(iso)
H(4)	5113	5431	996	24
H(7)	2220	8462	2229	33
H(8)	224	9351	2944	38
H(9)	89	10846	3631	37
H(10)	2029	11356	3687	43
H(11)	4030	10533	2953	31
H(13)	3842	9686	754	37
H(14)	3397	11547	136	49
H(15)	3898	13170	465	48
H(16)	4971	12931	1388	41
H(17)	5519	11034	1985	28
H(19)	7646	8763	1634	32
H(20)	9664	8064	2165	29
H(21)	9601	7365	3208	39
H(22)	7525	7235	3725	31
H(23)	5545	7810	3182	27
H(25)	9689	4163	38	29
H(26)	10785	2276	-522	36
H(28)	7096	1817	-315	35
H(29)	5994	3731	223	28
H(31)	5597	98	4090	28
H(34)	2886	3046	3009	31
H(35)	1105	3682	2347	43
H(36)	892	5306	1605	39
H(37)	2360	6369	1548	36
H(38)	4111	5791	2207	31
H(40)	2804	6165	3510	40
H(41)	2424	7813	4188	44
H(42)	4260	7700	4767	46
H(43)	6373	5937	4678	40
H(44)	6792	4375	3967	36
H(46)	6512	5478	2617	32
H(47)	8490	5176	1908	44
H(48)	9835	3252	1495	45
H(49)	9341	1478	1839	50
H(50)	7339	1714	2530	41
H(52)	9973	-1023	4769	28
H(53)	11051	-2909	5345	34
H(55)	7426	-3470	5567	32
H(56)	6363	-1644	4966	25

Table 6. Torsion angles [°] for ***LPS0462***.

C(6)-P(1)-N(1)-C(3)	-171.4(5)
C(18)-P(1)-N(1)-C(3)	-53.1(6)
C(12)-P(1)-N(1)-C(3)	73.7(5)
C(5)-O(1)-N(2)-C(3)	2.0(6)
O(1)-N(2)-C(3)-N(1)	178.1(5)
O(1)-N(2)-C(3)-C(4)	-2.2(7)
P(1)-N(1)-C(3)-N(2)	-8.7(9)
P(1)-N(1)-C(3)-C(4)	171.7(5)
N(2)-C(3)-C(4)-C(5)	1.7(8)
N(1)-C(3)-C(4)-C(5)	-178.7(6)
C(3)-C(4)-C(5)-O(1)	-0.3(7)
C(3)-C(4)-C(5)-C(24)	176.0(6)
N(2)-O(1)-C(5)-C(4)	-1.1(7)
N(2)-O(1)-C(5)-C(24)	-178.0(5)
N(1)-P(1)-C(6)-C(7)	-13.1(6)
C(18)-P(1)-C(6)-C(7)	-138.6(6)
C(12)-P(1)-C(6)-C(7)	108.9(6)
N(1)-P(1)-C(6)-C(11)	171.5(5)
C(18)-P(1)-C(6)-C(11)	45.9(6)
C(12)-P(1)-C(6)-C(11)	-66.6(6)
C(11)-C(6)-C(7)-C(8)	-0.3(10)
P(1)-C(6)-C(7)-C(8)	-175.7(5)
C(6)-C(7)-C(8)-C(9)	0.9(11)
C(7)-C(8)-C(9)-C(10)	-2.7(12)
C(8)-C(9)-C(10)-C(11)	3.8(11)
C(9)-C(10)-C(11)-C(6)	-3.1(10)
C(7)-C(6)-C(11)-C(10)	1.4(10)
P(1)-C(6)-C(11)-C(10)	176.9(5)
N(1)-P(1)-C(12)-C(17)	-168.0(4)
C(6)-P(1)-C(12)-C(17)	76.1(5)
C(18)-P(1)-C(12)-C(17)	-36.0(5)
N(1)-P(1)-C(12)-C(13)	20.2(6)
C(6)-P(1)-C(12)-C(13)	-95.7(5)
C(18)-P(1)-C(12)-C(13)	152.3(5)
C(17)-C(12)-C(13)-C(14)	0.1(10)
P(1)-C(12)-C(13)-C(14)	172.0(5)
C(12)-C(13)-C(14)-C(15)	-1.8(10)
C(13)-C(14)-C(15)-C(16)	1.8(10)

C(14)-C(15)-C(16)-C(17)	-0.2(10)
C(13)-C(12)-C(17)-C(16)	1.5(9)
P(1)-C(12)-C(17)-C(16)	-170.0(5)
C(15)-C(16)-C(17)-C(12)	-1.4(9)
N(1)-P(1)-C(18)-C(23)	-82.8(5)
C(6)-P(1)-C(18)-C(23)	35.5(6)
C(12)-P(1)-C(18)-C(23)	146.4(5)
N(1)-P(1)-C(18)-C(19)	89.5(5)
C(6)-P(1)-C(18)-C(19)	-152.1(5)
C(12)-P(1)-C(18)-C(19)	-41.2(6)
C(23)-C(18)-C(19)-C(20)	-2.4(9)
P(1)-C(18)-C(19)-C(20)	-174.7(5)
C(18)-C(19)-C(20)-C(21)	-1.0(9)
C(19)-C(20)-C(21)-C(22)	2.3(10)
C(20)-C(21)-C(22)-C(23)	-0.2(9)
C(21)-C(22)-C(23)-C(18)	-3.2(9)
C(19)-C(18)-C(23)-C(22)	4.6(9)
P(1)-C(18)-C(23)-C(22)	176.8(4)
C(4)-C(5)-C(24)-C(29)	14.7(12)
O(1)-C(5)-C(24)-C(29)	-169.2(6)
C(4)-C(5)-C(24)-C(25)	-166.4(7)
O(1)-C(5)-C(24)-C(25)	9.7(10)
C(29)-C(24)-C(25)-C(26)	-2.0(11)
C(5)-C(24)-C(25)-C(26)	179.1(6)
C(24)-C(25)-C(26)-C(27)	0.6(11)
C(25)-C(26)-C(27)-C(28)	-0.1(11)
C(25)-C(26)-C(27)-Br(1)	-178.8(5)
C(26)-C(27)-C(28)-C(29)	0.9(11)
Br(1)-C(27)-C(28)-C(29)	179.7(5)
C(25)-C(24)-C(29)-C(28)	2.8(11)
C(5)-C(24)-C(29)-C(28)	-178.3(6)
C(27)-C(28)-C(29)-C(24)	-2.3(11)
C(33)-P(2)-N(3)-C(30)	174.1(5)
C(45)-P(2)-N(3)-C(30)	58.1(6)
C(39)-P(2)-N(3)-C(30)	-66.2(5)
C(32)-O(2)-N(4)-C(30)	-0.4(6)
O(2)-N(4)-C(30)-N(3)	-178.2(5)
O(2)-N(4)-C(30)-C(31)	-0.5(6)
P(2)-N(3)-C(30)-N(4)	1.4(8)
P(2)-N(3)-C(30)-C(31)	-176.1(4)

N(4)-C(30)-C(31)-C(32)	1.2(7)
N(3)-C(30)-C(31)-C(32)	179.0(6)
N(4)-O(2)-C(32)-C(31)	1.2(6)
N(4)-O(2)-C(32)-C(51)	177.4(5)
C(30)-C(31)-C(32)-O(2)	-1.4(7)
C(30)-C(31)-C(32)-C(51)	-176.8(7)
N(3)-P(2)-C(33)-C(34)	10.0(6)
C(45)-P(2)-C(33)-C(34)	131.5(5)
C(39)-P(2)-C(33)-C(34)	-115.7(5)
N(3)-P(2)-C(33)-C(38)	-170.4(5)
C(45)-P(2)-C(33)-C(38)	-48.9(6)
C(39)-P(2)-C(33)-C(38)	63.9(6)
C(38)-C(33)-C(34)-C(35)	2.3(9)
P(2)-C(33)-C(34)-C(35)	-178.2(5)
C(33)-C(34)-C(35)-C(36)	0.0(10)
C(34)-C(35)-C(36)-C(37)	-1.8(10)
C(35)-C(36)-C(37)-C(38)	1.2(10)
C(36)-C(37)-C(38)-C(33)	1.2(10)
C(34)-C(33)-C(38)-C(37)	-2.9(9)
P(2)-C(33)-C(38)-C(37)	177.6(5)
N(3)-P(2)-C(39)-C(44)	75.4(6)
C(33)-P(2)-C(39)-C(44)	-166.2(6)
C(45)-P(2)-C(39)-C(44)	-53.1(6)
N(3)-P(2)-C(39)-C(40)	-104.8(6)
C(33)-P(2)-C(39)-C(40)	13.6(7)
C(45)-P(2)-C(39)-C(40)	126.7(6)
C(44)-C(39)-C(40)-C(41)	-1.0(11)
P(2)-C(39)-C(40)-C(41)	179.2(6)
C(39)-C(40)-C(41)-C(42)	-0.2(11)
C(40)-C(41)-C(42)-C(43)	-0.6(11)
C(41)-C(42)-C(43)-C(44)	2.7(11)
C(42)-C(43)-C(44)-C(39)	-3.9(11)
C(40)-C(39)-C(44)-C(43)	3.0(11)
P(2)-C(39)-C(44)-C(43)	-177.2(5)
N(3)-P(2)-C(45)-C(50)	21.4(6)
C(33)-P(2)-C(45)-C(50)	-94.2(5)
C(39)-P(2)-C(45)-C(50)	151.6(5)
N(3)-P(2)-C(45)-C(46)	-162.7(5)
C(33)-P(2)-C(45)-C(46)	81.7(6)
C(39)-P(2)-C(45)-C(46)	-32.5(6)

C(50)-C(45)-C(46)-C(47)	0.1(10)
P(2)-C(45)-C(46)-C(47)	-175.8(5)
C(45)-C(46)-C(47)-C(48)	0.6(10)
C(46)-C(47)-C(48)-C(49)	-2.1(11)
C(47)-C(48)-C(49)-C(50)	3.0(11)
C(48)-C(49)-C(50)-C(45)	-2.4(11)
C(46)-C(45)-C(50)-C(49)	0.8(10)
P(2)-C(45)-C(50)-C(49)	176.8(5)
O(2)-C(32)-C(51)-C(52)	-16.2(9)
C(31)-C(32)-C(51)-C(52)	158.9(7)
O(2)-C(32)-C(51)-C(56)	162.2(6)
C(31)-C(32)-C(51)-C(56)	-22.6(11)
C(56)-C(51)-C(52)-C(53)	0.0(9)
C(32)-C(51)-C(52)-C(53)	178.5(6)
C(51)-C(52)-C(53)-C(54)	-1.3(9)
C(52)-C(53)-C(54)-C(55)	1.4(10)
C(52)-C(53)-C(54)-Br(2)	-177.5(5)
C(53)-C(54)-C(55)-C(56)	-0.2(9)
Br(2)-C(54)-C(55)-C(56)	178.8(4)
C(54)-C(55)-C(56)-C(51)	-1.2(9)
C(52)-C(51)-C(56)-C(55)	1.3(9)
C(32)-C(51)-C(56)-C(55)	-177.2(6)

3-Imino-(triphenyl)-phosphorano-5-(4-chlorophenyl)-isoxazole **11b**

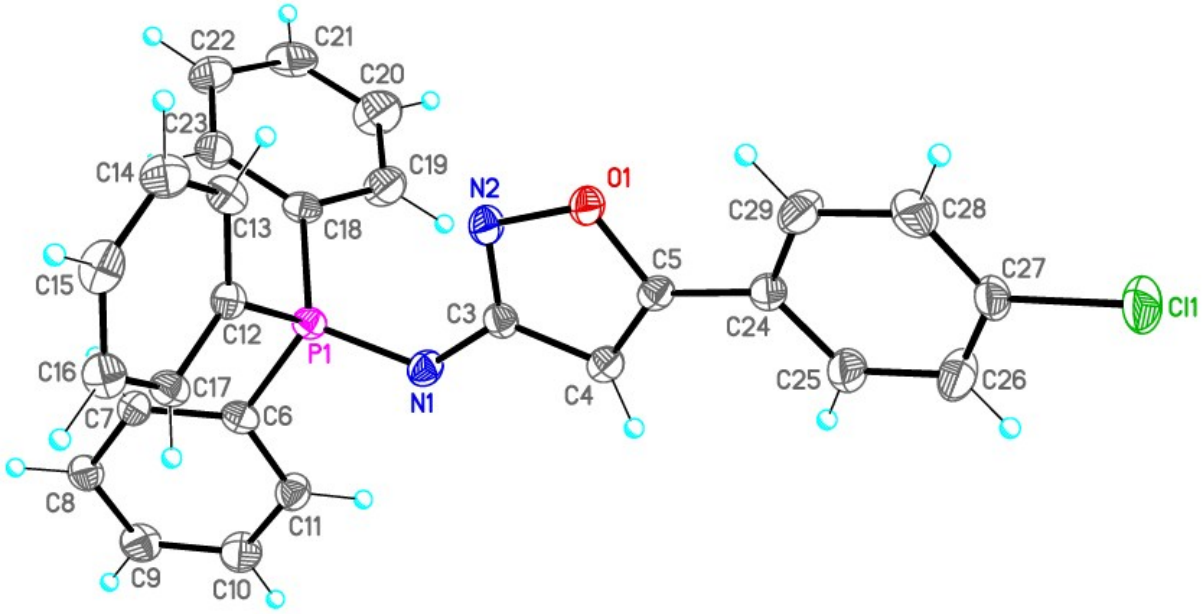


Table 1. Crystal data and structure refinement for **11b**

Identification code	**LSP0461**	
Empirical formula	C27 H20 Cl N2 O P	
Formula weight	454.87	
Temperature	100(2) K	
Wavelength	0.96330 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.490(2) Å	α = 85.32(3)°.
	b = 11.419(2) Å	β = 82.36(3)°.
	c = 21.515(4) Å	γ = 66.54(3)°.
Volume	2341.8(10) $Å^3$	
Z	4	
Density (calculated)	1.290 Mg/m^3	
Absorption coefficient	0.578 mm^{-1}	
F(000)	944	
Crystal size	0.15 x 0.10 x 0.10 mm^3	
Theta range for data collection	3.292 to 38.393°.	
Index ranges	-13<=h<=13, -14<=k<=14, -26<=l<=27	
Reflections collected	24646	
Independent reflections	9589 [R(int) = 0.1121]	
Completeness to theta = 35.309°	97.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.930 and 0.900	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9589 / 0 / 578	

Goodness-of-fit on F^2	0.840
Final R indices [for 6798 rflns with $I>2\sigma(I)$]	R1 = 0.0861, wR2 = 0.1944
R indices (all data)	R1 = 0.1039, wR2 = 0.2212
Extinction coefficient	0.016(3)
Largest diff. peak and hole	0.747 and -0.590 $eÅ^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for ***LSP0461***. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U(eq)
Cl(1)	4886(1)	392(1)	-883(1)	41(1)
P(1)	-158(1)	8780(1)	1949(1)	18(1)
O(1)	2789(2)	6009(2)	579(1)	22(1)
N(1)	-280(2)	7633(2)	1612(1)	22(1)
N(2)	1948(2)	7070(2)	978(1)	22(1)
C(3)	785(2)	6864(2)	1187(1)	20(1)
C(4)	816(2)	5731(2)	925(1)	21(1)
C(5)	2063(2)	5244(2)	564(1)	19(1)
C(6)	-1650(2)	9352(2)	2544(1)	20(1)
C(7)	-1702(2)	10211(2)	2990(1)	22(1)
C(8)	-2889(3)	10730(2)	3422(1)	25(1)
C(9)	-4043(3)	10417(3)	3402(1)	29(1)
C(10)	-3991(3)	9566(3)	2967(1)	32(1)
C(11)	-2809(2)	9024(2)	2536(1)	24(1)
C(12)	1391(2)	8370(2)	2356(1)	20(1)
C(13)	2612(2)	8495(2)	2045(1)	23(1)
C(14)	3819(3)	8087(3)	2349(1)	28(1)
C(15)	3829(3)	7566(2)	2959(1)	28(1)
C(16)	2623(3)	7450(2)	3272(1)	25(1)
C(17)	1418(3)	7838(2)	2968(1)	22(1)
C(18)	-312(2)	10184(2)	1445(1)	21(1)
C(19)	-926(3)	10330(3)	885(1)	35(1)
C(20)	-1230(3)	11454(3)	520(1)	43(1)
C(21)	-934(3)	12448(3)	713(1)	35(1)
C(22)	-302(3)	12303(3)	1257(1)	34(1)
C(23)	-5(3)	11181(2)	1627(1)	27(1)
C(24)	2766(2)	4079(2)	192(1)	20(1)
C(25)	2012(3)	3355(3)	83(1)	28(1)

C(26)	2657(3)	2233(3)	-253(1)	34(1)
C(27)	4069(3)	1825(2)	-479(1)	27(1)
C(28)	4829(3)	2536(3)	-388(1)	32(1)
C(29)	4185(3)	3656(3)	-52(1)	31(1)
Cl(2)	4786(1)	14843(1)	4015(1)	34(1)
P(2)	9868(1)	6278(1)	6829(1)	18(1)
O(2)	7013(2)	9177(2)	5598(1)	21(1)
N(3)	7810(2)	8097(2)	5978(1)	22(1)
N(4)	9885(2)	7582(2)	6490(1)	23(1)
C(30)	8872(2)	8360(2)	6123(1)	19(1)
C(31)	8797(2)	9581(2)	5848(1)	22(1)
C(32)	7633(2)	10041(2)	5540(1)	18(1)
C(33)	8286(2)	6473(2)	7355(1)	21(1)
C(34)	7925(2)	5429(3)	7555(1)	25(1)
C(35)	6757(3)	5597(3)	7995(1)	32(1)
C(36)	5931(3)	6799(3)	8226(1)	37(1)
C(37)	6269(3)	7841(3)	8020(1)	38(1)
C(38)	7443(3)	7690(3)	7585(1)	30(1)
C(39)	10151(2)	4965(2)	6326(1)	22(1)
C(40)	11451(3)	3921(3)	6234(1)	28(1)
C(41)	11648(3)	2951(3)	5834(1)	35(1)
C(42)	10570(3)	3006(3)	5504(1)	31(1)
C(43)	9273(3)	4046(3)	5583(1)	28(1)
C(44)	9050(3)	5012(2)	5992(1)	25(1)
C(45)	11309(2)	5716(2)	7318(1)	19(1)
C(46)	12256(2)	6324(2)	7258(1)	24(1)
C(47)	13329(3)	5930(3)	7642(1)	28(1)
C(48)	13472(3)	4934(3)	8087(1)	28(1)
C(49)	12545(2)	4316(3)	8141(1)	26(1)
C(50)	11471(2)	4696(2)	7755(1)	22(1)
C(51)	6923(2)	11232(2)	5175(1)	19(1)
C(52)	5523(2)	11605(2)	5064(1)	24(1)
C(53)	4853(2)	12717(2)	4709(1)	25(1)
C(54)	5600(3)	13466(2)	4476(1)	24(1)
C(55)	6985(3)	13128(2)	4585(1)	23(1)
C(56)	7644(2)	12012(2)	4939(1)	21(1)

Table 3. Bond lengths [Å] and angles [°] for ***LSP0461***.

Cl(1)-C(27)	1.757(3)	C(19)-H(19)	0.9500
P(1)-N(1)	1.603(2)	C(20)-C(21)	1.397(4)
P(1)-C(6)	1.818(3)	C(20)-H(20)	0.9500
P(1)-C(18)	1.823(3)	C(21)-C(22)	1.387(4)
P(1)-C(12)	1.825(3)	C(21)-H(21)	0.9500
O(1)-C(5)	1.373(3)	C(22)-C(23)	1.398(4)
O(1)-N(2)	1.449(3)	C(22)-H(22)	0.9500
N(1)-C(3)	1.390(3)	C(23)-H(23)	0.9500
N(2)-C(3)	1.345(3)	C(24)-C(25)	1.403(3)
C(3)-C(4)	1.440(3)	C(24)-C(29)	1.409(3)
C(4)-C(5)	1.359(3)	C(25)-C(26)	1.397(4)
C(4)-H(4)	0.9500	C(25)-H(25)	0.9500
C(5)-C(24)	1.479(3)	C(26)-C(27)	1.394(4)
C(6)-C(7)	1.409(3)	C(26)-H(26)	0.9500
C(6)-C(11)	1.410(3)	C(27)-C(28)	1.384(4)
C(7)-C(8)	1.400(3)	C(28)-C(29)	1.396(4)
C(7)-H(7)	0.9500	C(28)-H(28)	0.9500
C(8)-C(9)	1.398(4)	C(29)-H(29)	0.9500
C(8)-H(8)	0.9500	Cl(2)-C(54)	1.757(3)
C(9)-C(10)	1.384(4)	P(2)-N(4)	1.610(2)
C(9)-H(9)	0.9500	P(2)-C(33)	1.825(3)
C(10)-C(11)	1.399(4)	P(2)-C(39)	1.826(3)
C(10)-H(10)	0.9500	P(2)-C(45)	1.828(2)
C(11)-H(11)	0.9500	O(2)-C(32)	1.372(3)
C(12)-C(17)	1.404(3)	O(2)-N(3)	1.443(2)
C(12)-C(13)	1.418(3)	N(3)-C(30)	1.343(3)
C(13)-C(14)	1.395(3)	N(4)-C(30)	1.379(3)
C(13)-H(13)	0.9500	C(30)-C(31)	1.445(3)
C(14)-C(15)	1.395(4)	C(31)-C(32)	1.362(3)
C(14)-H(14)	0.9500	C(31)-H(31)	0.9500
C(15)-C(16)	1.402(4)	C(32)-C(51)	1.480(3)
C(15)-H(15)	0.9500	C(33)-C(38)	1.406(4)
C(16)-C(17)	1.395(3)	C(33)-C(34)	1.407(3)
C(16)-H(16)	0.9500	C(34)-C(35)	1.403(4)
C(17)-H(17)	0.9500	C(34)-H(34)	0.9500
C(18)-C(23)	1.397(3)	C(35)-C(36)	1.390(4)
C(18)-C(19)	1.409(4)	C(35)-H(35)	0.9500
C(19)-C(20)	1.393(4)	C(36)-C(37)	1.394(4)

C(36)-H(36)	0.9500	C(46)-H(46)	0.9500
C(37)-C(38)	1.404(4)	C(47)-C(48)	1.399(4)
C(37)-H(37)	0.9500	C(47)-H(47)	0.9500
C(38)-H(38)	0.9500	C(48)-C(49)	1.400(3)
C(39)-C(40)	1.412(3)	C(48)-H(48)	0.9500
C(39)-C(44)	1.419(3)	C(49)-C(50)	1.396(3)
C(40)-C(41)	1.392(4)	C(49)-H(49)	0.9500
C(40)-H(40)	0.9500	C(50)-H(50)	0.9500
C(41)-C(42)	1.392(4)	C(51)-C(52)	1.406(3)
C(41)-H(41)	0.9500	C(51)-C(56)	1.408(3)
C(42)-C(43)	1.406(4)	C(52)-C(53)	1.401(3)
C(42)-H(42)	0.9500	C(52)-H(52)	0.9500
C(43)-C(44)	1.394(4)	C(53)-C(54)	1.398(3)
C(43)-H(43)	0.9500	C(53)-H(53)	0.9500
C(44)-H(44)	0.9500	C(54)-C(55)	1.395(3)
C(45)-C(50)	1.405(3)	C(55)-C(56)	1.400(3)
C(45)-C(46)	1.409(3)	C(55)-H(55)	0.9500
C(46)-C(47)	1.391(3)	C(56)-H(56)	0.9500

N(1)-P(1)-C(6)	106.88(11)	C(8)-C(7)-C(6)	120.4(2)
N(1)-P(1)-C(18)	114.99(11)	C(8)-C(7)-H(7)	119.8
C(6)-P(1)-C(18)	104.36(11)	C(6)-C(7)-H(7)	119.8
N(1)-P(1)-C(12)	116.15(11)	C(9)-C(8)-C(7)	119.9(2)
C(6)-P(1)-C(12)	106.13(11)	C(9)-C(8)-H(8)	120.1
C(18)-P(1)-C(12)	107.32(11)	C(7)-C(8)-H(8)	120.1
C(5)-O(1)-N(2)	108.25(17)	C(10)-C(9)-C(8)	119.8(2)
C(3)-N(1)-P(1)	122.24(16)	C(10)-C(9)-H(9)	120.1
C(3)-N(2)-O(1)	105.14(18)	C(8)-C(9)-H(9)	120.1
N(2)-C(3)-N(1)	124.0(2)	C(9)-C(10)-C(11)	121.3(2)
N(2)-C(3)-C(4)	110.9(2)	C(9)-C(10)-H(10)	119.4
N(1)-C(3)-C(4)	125.1(2)	C(11)-C(10)-H(10)	119.4
C(5)-C(4)-C(3)	105.6(2)	C(10)-C(11)-C(6)	119.4(2)
C(5)-C(4)-H(4)	127.2	C(10)-C(11)-H(11)	120.3
C(3)-C(4)-H(4)	127.2	C(6)-C(11)-H(11)	120.3
C(4)-C(5)-O(1)	110.0(2)	C(17)-C(12)-C(13)	119.1(2)
C(4)-C(5)-C(24)	133.2(2)	C(17)-C(12)-P(1)	119.95(18)
O(1)-C(5)-C(24)	116.7(2)	C(13)-C(12)-P(1)	120.79(18)
C(7)-C(6)-C(11)	119.2(2)	C(14)-C(13)-C(12)	119.8(2)
C(7)-C(6)-P(1)	119.86(17)	C(14)-C(13)-H(13)	120.1
C(11)-C(6)-P(1)	120.74(19)	C(12)-C(13)-H(13)	120.1

C(15)-C(14)-C(13)	120.5(2)	C(28)-C(27)-Cl(1)	119.8(2)
C(15)-C(14)-H(14)	119.8	C(26)-C(27)-Cl(1)	119.4(2)
C(13)-C(14)-H(14)	119.8	C(27)-C(28)-C(29)	119.7(2)
C(14)-C(15)-C(16)	120.1(2)	C(27)-C(28)-H(28)	120.2
C(14)-C(15)-H(15)	119.9	C(29)-C(28)-H(28)	120.2
C(16)-C(15)-H(15)	119.9	C(28)-C(29)-C(24)	120.8(2)
C(17)-C(16)-C(15)	119.8(2)	C(28)-C(29)-H(29)	119.6
C(17)-C(16)-H(16)	120.1	C(24)-C(29)-H(29)	119.6
C(15)-C(16)-H(16)	120.1	N(4)-P(2)-C(33)	114.48(12)
C(16)-C(17)-C(12)	120.7(2)	N(4)-P(2)-C(39)	117.20(11)
C(16)-C(17)-H(17)	119.7	C(33)-P(2)-C(39)	105.85(12)
C(12)-C(17)-H(17)	119.7	N(4)-P(2)-C(45)	105.43(11)
C(23)-C(18)-C(19)	118.9(2)	C(33)-P(2)-C(45)	106.54(11)
C(23)-C(18)-P(1)	122.73(19)	C(39)-P(2)-C(45)	106.63(11)
C(19)-C(18)-P(1)	117.93(19)	C(32)-O(2)-N(3)	108.56(16)
C(20)-C(19)-C(18)	120.6(3)	C(30)-N(3)-O(2)	105.38(18)
C(20)-C(19)-H(19)	119.7	C(30)-N(4)-P(2)	123.08(17)
C(18)-C(19)-H(19)	119.7	N(3)-C(30)-N(4)	124.7(2)
C(19)-C(20)-C(21)	119.8(3)	N(3)-C(30)-C(31)	110.7(2)
C(19)-C(20)-H(20)	120.1	N(4)-C(30)-C(31)	124.6(2)
C(21)-C(20)-H(20)	120.1	C(32)-C(31)-C(30)	105.6(2)
C(22)-C(21)-C(20)	120.1(3)	C(32)-C(31)-H(31)	127.2
C(22)-C(21)-H(21)	120.0	C(30)-C(31)-H(31)	127.2
C(20)-C(21)-H(21)	120.0	C(31)-C(32)-O(2)	109.8(2)
C(21)-C(22)-C(23)	120.3(3)	C(31)-C(32)-C(51)	134.5(2)
C(21)-C(22)-H(22)	119.9	O(2)-C(32)-C(51)	115.8(2)
C(23)-C(22)-H(22)	119.9	C(38)-C(33)-C(34)	119.5(2)
C(18)-C(23)-C(22)	120.4(2)	C(38)-C(33)-P(2)	118.79(19)
C(18)-C(23)-H(23)	119.8	C(34)-C(33)-P(2)	121.7(2)
C(22)-C(23)-H(23)	119.8	C(35)-C(34)-C(33)	120.1(3)
C(25)-C(24)-C(29)	118.4(2)	C(35)-C(34)-H(34)	119.9
C(25)-C(24)-C(5)	119.5(2)	C(33)-C(34)-H(34)	119.9
C(29)-C(24)-C(5)	122.1(2)	C(36)-C(35)-C(34)	120.3(3)
C(26)-C(25)-C(24)	120.7(2)	C(36)-C(35)-H(35)	119.8
C(26)-C(25)-H(25)	119.6	C(34)-C(35)-H(35)	119.8
C(24)-C(25)-H(25)	119.6	C(35)-C(36)-C(37)	119.7(2)
C(27)-C(26)-C(25)	119.6(2)	C(35)-C(36)-H(36)	120.1
C(27)-C(26)-H(26)	120.2	C(37)-C(36)-H(36)	120.1
C(25)-C(26)-H(26)	120.2	C(36)-C(37)-C(38)	120.9(3)
C(28)-C(27)-C(26)	120.7(3)	C(36)-C(37)-H(37)	119.6

C(38)-C(37)-H(37)	119.6	C(46)-C(47)-H(47)	119.8
C(37)-C(38)-C(33)	119.5(3)	C(48)-C(47)-H(47)	119.8
C(37)-C(38)-H(38)	120.3	C(47)-C(48)-C(49)	119.9(2)
C(33)-C(38)-H(38)	120.3	C(47)-C(48)-H(48)	120.0
C(40)-C(39)-C(44)	118.4(2)	C(49)-C(48)-H(48)	120.0
C(40)-C(39)-P(2)	122.2(2)	C(50)-C(49)-C(48)	120.2(2)
C(44)-C(39)-P(2)	119.38(19)	C(50)-C(49)-H(49)	119.9
C(41)-C(40)-C(39)	121.0(2)	C(48)-C(49)-H(49)	119.9
C(41)-C(40)-H(40)	119.5	C(49)-C(50)-C(45)	119.8(2)
C(39)-C(40)-H(40)	119.5	C(49)-C(50)-H(50)	120.1
C(40)-C(41)-C(42)	120.5(3)	C(45)-C(50)-H(50)	120.1
C(40)-C(41)-H(41)	119.7	C(52)-C(51)-C(56)	119.1(2)
C(42)-C(41)-H(41)	119.7	C(52)-C(51)-C(32)	120.7(2)
C(41)-C(42)-C(43)	119.3(3)	C(56)-C(51)-C(32)	120.2(2)
C(41)-C(42)-H(42)	120.4	C(53)-C(52)-C(51)	121.0(2)
C(43)-C(42)-H(42)	120.4	C(53)-C(52)-H(52)	119.5
C(44)-C(43)-C(42)	120.9(3)	C(51)-C(52)-H(52)	119.5
C(44)-C(43)-H(43)	119.6	C(54)-C(53)-C(52)	118.6(2)
C(42)-C(43)-H(43)	119.6	C(54)-C(53)-H(53)	120.7
C(43)-C(44)-C(39)	120.0(2)	C(52)-C(53)-H(53)	120.7
C(43)-C(44)-H(44)	120.0	C(55)-C(54)-C(53)	121.7(2)
C(39)-C(44)-H(44)	120.0	C(55)-C(54)-Cl(2)	119.27(19)
C(50)-C(45)-C(46)	119.9(2)	C(53)-C(54)-Cl(2)	119.05(18)
C(50)-C(45)-P(2)	120.70(18)	C(54)-C(55)-C(56)	119.2(2)
C(46)-C(45)-P(2)	119.41(18)	C(54)-C(55)-H(55)	120.4
C(47)-C(46)-C(45)	119.8(2)	C(56)-C(55)-H(55)	120.4
C(47)-C(46)-H(46)	120.1	C(55)-C(56)-C(51)	120.5(2)
C(45)-C(46)-H(46)	120.1	C(55)-C(56)-H(56)	119.8
C(46)-C(47)-C(48)	120.4(2)	C(51)-C(56)-H(56)	119.8

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for ***LSP0461***. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	60(1)	24(1)	35(1)	-11(1)	6(1)	-13(1)
P(1)	18(1)	16(1)	18(1)	1(1)	-2(1)	-5(1)
O(1)	23(1)	18(1)	25(1)	-6(1)	2(1)	-7(1)
N(1)	22(1)	17(1)	25(1)	-2(1)	-2(1)	-7(1)
N(2)	22(1)	17(1)	25(1)	-6(1)	1(1)	-5(1)
C(3)	23(1)	17(1)	17(1)	1(1)	-3(1)	-6(1)
C(4)	22(1)	20(1)	22(1)	2(1)	-6(1)	-9(1)
C(5)	22(1)	17(1)	19(1)	4(1)	-6(1)	-9(1)
C(6)	20(1)	18(1)	19(1)	5(1)	-4(1)	-5(1)
C(7)	24(1)	20(1)	22(1)	-2(1)	-2(1)	-9(1)
C(8)	27(1)	22(1)	24(1)	-6(1)	5(1)	-9(1)
C(9)	22(1)	28(2)	32(2)	-7(1)	8(1)	-7(1)
C(10)	22(1)	42(2)	36(2)	-9(1)	5(1)	-16(1)
C(11)	22(1)	25(1)	27(1)	-4(1)	-1(1)	-10(1)
C(12)	20(1)	14(1)	22(1)	-3(1)	1(1)	-3(1)
C(13)	22(1)	22(1)	21(1)	1(1)	-1(1)	-5(1)
C(14)	19(1)	30(2)	34(2)	0(1)	-1(1)	-8(1)
C(15)	25(1)	22(1)	35(2)	2(1)	-15(1)	-4(1)
C(16)	28(1)	18(1)	25(1)	4(1)	-8(1)	-4(1)
C(17)	24(1)	17(1)	21(1)	1(1)	1(1)	-6(1)
C(18)	18(1)	22(1)	20(1)	5(1)	-2(1)	-6(1)
C(19)	45(2)	38(2)	31(2)	10(1)	-15(1)	-25(1)
C(20)	58(2)	50(2)	34(2)	20(2)	-26(2)	-31(2)
C(21)	40(2)	31(2)	33(2)	18(1)	-7(1)	-16(1)
C(22)	45(2)	25(2)	34(2)	7(1)	-4(1)	-18(1)
C(23)	35(2)	23(1)	24(1)	3(1)	-7(1)	-13(1)
C(24)	22(1)	17(1)	19(1)	2(1)	-4(1)	-7(1)
C(25)	28(1)	29(2)	30(2)	-7(1)	2(1)	-12(1)
C(26)	38(2)	29(2)	40(2)	-10(1)	-4(1)	-16(1)
C(27)	39(2)	19(1)	20(1)	-3(1)	1(1)	-9(1)
C(28)	28(1)	31(2)	32(2)	-7(1)	7(1)	-8(1)
C(29)	25(1)	24(2)	44(2)	-8(1)	4(1)	-13(1)
Cl(2)	34(1)	23(1)	31(1)	10(1)	-5(1)	1(1)
P(2)	18(1)	17(1)	20(1)	3(1)	-5(1)	-5(1)
O(2)	19(1)	17(1)	25(1)	5(1)	-7(1)	-6(1)

N(3)	22(1)	18(1)	22(1)	7(1)	-6(1)	-3(1)
N(4)	22(1)	19(1)	28(1)	9(1)	-9(1)	-8(1)
C(30)	18(1)	15(1)	21(1)	4(1)	-4(1)	-5(1)
C(31)	22(1)	19(1)	26(1)	2(1)	-4(1)	-9(1)
C(32)	22(1)	16(1)	18(1)	0(1)	-1(1)	-7(1)
C(33)	19(1)	25(1)	18(1)	3(1)	-9(1)	-8(1)
C(34)	22(1)	24(1)	29(2)	4(1)	-9(1)	-8(1)
C(35)	21(1)	48(2)	28(2)	12(1)	-10(1)	-16(1)
C(36)	23(1)	66(2)	22(2)	-2(2)	-2(1)	-16(2)
C(37)	29(2)	47(2)	30(2)	-18(2)	-1(1)	-4(1)
C(38)	26(1)	32(2)	30(2)	-8(1)	-3(1)	-9(1)
C(39)	22(1)	22(1)	17(1)	2(1)	1(1)	-7(1)
C(40)	23(1)	30(2)	29(2)	-5(1)	-1(1)	-10(1)
C(41)	28(2)	33(2)	38(2)	-10(1)	4(1)	-7(1)
C(42)	38(2)	30(2)	24(2)	-6(1)	4(1)	-16(1)
C(43)	35(2)	32(2)	22(1)	3(1)	-8(1)	-17(1)
C(44)	29(1)	22(1)	24(1)	3(1)	-9(1)	-9(1)
C(45)	19(1)	19(1)	18(1)	-1(1)	-2(1)	-4(1)
C(46)	24(1)	24(1)	23(1)	2(1)	-3(1)	-10(1)
C(47)	24(1)	32(2)	32(2)	2(1)	-5(1)	-14(1)
C(48)	24(1)	33(2)	29(2)	4(1)	-10(1)	-10(1)
C(49)	21(1)	24(1)	27(1)	6(1)	-5(1)	-4(1)
C(50)	19(1)	21(1)	24(1)	4(1)	-2(1)	-7(1)
C(51)	18(1)	18(1)	17(1)	-3(1)	-2(1)	-2(1)
C(52)	20(1)	22(1)	24(1)	3(1)	1(1)	-5(1)
C(53)	17(1)	24(1)	26(1)	2(1)	-3(1)	0(1)
C(54)	25(1)	19(1)	20(1)	1(1)	-3(1)	-1(1)
C(55)	31(1)	18(1)	19(1)	-3(1)	1(1)	-8(1)
C(56)	20(1)	22(1)	21(1)	-1(1)	-6(1)	-6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for ***LSP0461***.

Atom	x	y	z	U(iso)
H(4)	112	5395	991	25
H(7)	-927	10439	2998	26
H(8)	-2910	11293	3729	30
H(9)	-4861	10788	3687	35
H(10)	-4772	9346	2961	39
H(11)	-2788	8441	2240	29
H(13)	2609	8857	1631	28
H(14)	4639	8164	2139	34
H(15)	4655	7291	3162	34
H(16)	2626	7107	3689	30
H(17)	608	7742	3178	26
H(19)	-1134	9657	754	42
H(20)	-1638	11544	141	52
H(21)	-1165	13225	471	42
H(22)	-70	12969	1378	40
H(23)	408	11096	2004	32
H(25)	1051	3630	240	34
H(26)	2137	1750	-326	41
H(28)	5784	2262	-553	39
H(29)	4710	4139	13	37
H(31)	9428	9978	5876	26
H(34)	8472	4609	7391	30
H(35)	6529	4886	8135	38
H(36)	5140	6911	8524	45
H(37)	5698	8663	8176	45
H(38)	7665	8405	7448	36
H(40)	12203	3880	6449	33
H(41)	12524	2246	5787	42
H(42)	10710	2348	5227	37
H(43)	8538	4092	5355	34
H(44)	8163	5701	6048	30
H(46)	12161	7002	6957	28
H(47)	13968	6339	7602	34
H(48)	14197	4678	8353	34
H(49)	12647	3636	8441	31
H(50)	10852	4267	7787	26
H(52)	5023	11095	5231	28
H(53)	3911	12958	4628	30
H(55)	7475	13648	4422	28
H(56)	8583	11780	5021	25

Table 6. Torsion angles [°] for ***LSP0461***.

C(6)-P(1)-N(1)-C(3)	-171.43(18)	C(14)-C(15)-C(16)-C(17)	0.9(4)
C(18)-P(1)-N(1)-C(3)	73.3(2)	C(15)-C(16)-C(17)-C(12)	-1.3(4)
C(12)-P(1)-N(1)-C(3)	-53.2(2)	C(13)-C(12)-C(17)-C(16)	0.7(4)
C(5)-O(1)-N(2)-C(3)	1.2(2)	P(1)-C(12)-C(17)-C(16)	176.19(18)
O(1)-N(2)-C(3)-N(1)	176.8(2)	N(1)-P(1)-C(18)-C(23)	-169.18(19)
O(1)-N(2)-C(3)-C(4)	-1.8(3)	C(6)-P(1)-C(18)-C(23)	74.1(2)
P(1)-N(1)-C(3)-N(2)	-6.1(3)	C(12)-P(1)-C(18)-C(23)	-38.3(2)
P(1)-N(1)-C(3)-C(4)	172.31(18)	N(1)-P(1)-C(18)-C(19)	18.7(3)
N(2)-C(3)-C(4)-C(5)	1.7(3)	C(6)-P(1)-C(18)-C(19)	-98.0(2)
N(1)-C(3)-C(4)-C(5)	-176.9(2)	C(12)-P(1)-C(18)-C(19)	149.6(2)
C(3)-C(4)-C(5)-O(1)	-0.9(3)	C(23)-C(18)-C(19)-C(20)	-0.4(4)
C(3)-C(4)-C(5)-C(24)	176.9(2)	P(1)-C(18)-C(19)-C(20)	172.0(2)
N(2)-O(1)-C(5)-C(4)	-0.2(2)	C(18)-C(19)-C(20)-C(21)	-0.4(5)
N(2)-O(1)-C(5)-C(24)	-178.39(18)	C(19)-C(20)-C(21)-C(22)	1.8(5)
N(1)-P(1)-C(6)-C(7)	169.09(19)	C(20)-C(21)-C(22)-C(23)	-2.4(4)
C(18)-P(1)-C(6)-C(7)	-68.7(2)	C(19)-C(18)-C(23)-C(22)	-0.1(4)
C(12)-P(1)-C(6)-C(7)	44.5(2)	P(1)-C(18)-C(23)-C(22)	-172.1(2)
N(1)-P(1)-C(6)-C(11)	-15.9(2)	C(21)-C(22)-C(23)-C(18)	1.5(4)
C(18)-P(1)-C(6)-C(11)	106.4(2)	C(4)-C(5)-C(24)-C(25)	11.3(4)
C(12)-P(1)-C(6)-C(11)	-140.4(2)	O(1)-C(5)-C(24)-C(25)	-171.0(2)
C(11)-C(6)-C(7)-C(8)	0.2(4)	C(4)-C(5)-C(24)-C(29)	-167.8(3)
P(1)-C(6)-C(7)-C(8)	175.36(19)	O(1)-C(5)-C(24)-C(29)	9.9(3)
C(6)-C(7)-C(8)-C(9)	-1.5(4)	C(29)-C(24)-C(25)-C(26)	0.7(4)
C(7)-C(8)-C(9)-C(10)	1.9(4)	C(5)-C(24)-C(25)-C(26)	-178.4(2)
C(8)-C(9)-C(10)-C(11)	-1.2(4)	C(24)-C(25)-C(26)-C(27)	0.4(4)
C(9)-C(10)-C(11)-C(6)	-0.1(4)	C(25)-C(26)-C(27)-C(28)	-1.6(4)
C(7)-C(6)-C(11)-C(10)	0.5(4)	C(25)-C(26)-C(27)-Cl(1)	178.4(2)
P(1)-C(6)-C(11)-C(10)	-174.6(2)	C(26)-C(27)-C(28)-C(29)	1.7(4)
N(1)-P(1)-C(12)-C(17)	-82.8(2)	Cl(1)-C(27)-C(28)-C(29)	-178.3(2)
C(6)-P(1)-C(12)-C(17)	35.8(2)	C(27)-C(28)-C(29)-C(24)	-0.5(4)
C(18)-P(1)-C(12)-C(17)	146.9(2)	C(25)-C(24)-C(29)-C(28)	-0.7(4)
N(1)-P(1)-C(12)-C(13)	92.6(2)	C(5)-C(24)-C(29)-C(28)	178.4(2)
C(6)-P(1)-C(12)-C(13)	-148.8(2)	C(32)-O(2)-N(3)-C(30)	-0.7(2)
C(18)-P(1)-C(12)-C(13)	-37.7(2)	C(33)-P(2)-N(4)-C(30)	-57.3(2)
C(17)-C(12)-C(13)-C(14)	0.2(4)	C(39)-P(2)-N(4)-C(30)	67.5(2)
P(1)-C(12)-C(13)-C(14)	-175.20(19)	C(45)-P(2)-N(4)-C(30)	-174.07(19)
C(12)-C(13)-C(14)-C(15)	-0.6(4)	O(2)-N(3)-C(30)-N(4)	179.1(2)
C(13)-C(14)-C(15)-C(16)	0.0(4)	O(2)-N(3)-C(30)-C(31)	-0.1(2)

P(2)-N(4)-C(30)-N(3)	-3.7(3)
P(2)-N(4)-C(30)-C(31)	175.38(19)
N(3)-C(30)-C(31)-C(32)	0.8(3)
N(4)-C(30)-C(31)-C(32)	-178.4(2)
C(30)-C(31)-C(32)-O(2)	-1.2(3)
C(30)-C(31)-C(32)-C(51)	178.3(3)
N(3)-O(2)-C(32)-C(31)	1.2(3)
N(3)-O(2)-C(32)-C(51)	-178.42(18)
N(4)-P(2)-C(33)-C(38)	-20.1(2)
C(39)-P(2)-C(33)-C(38)	-150.8(2)
C(45)-P(2)-C(33)-C(38)	96.0(2)
N(4)-P(2)-C(33)-C(34)	162.51(19)
C(39)-P(2)-C(33)-C(34)	31.9(2)
C(45)-P(2)-C(33)-C(34)	-81.4(2)
C(38)-C(33)-C(34)-C(35)	-2.0(4)
P(2)-C(33)-C(34)-C(35)	175.32(18)
C(33)-C(34)-C(35)-C(36)	1.5(4)
C(34)-C(35)-C(36)-C(37)	-0.1(4)
C(35)-C(36)-C(37)-C(38)	-0.6(4)
C(36)-C(37)-C(38)-C(33)	0.0(4)
C(34)-C(33)-C(38)-C(37)	1.3(4)
P(2)-C(33)-C(38)-C(37)	-176.1(2)
N(4)-P(2)-C(39)-C(40)	101.5(2)
C(33)-P(2)-C(39)-C(40)	-129.5(2)
C(45)-P(2)-C(39)-C(40)	-16.3(2)
N(4)-P(2)-C(39)-C(44)	-75.9(2)
C(33)-P(2)-C(39)-C(44)	53.2(2)
C(45)-P(2)-C(39)-C(44)	166.35(18)
C(44)-C(39)-C(40)-C(41)	-0.9(4)
P(2)-C(39)-C(40)-C(41)	-178.3(2)
C(39)-C(40)-C(41)-C(42)	1.5(4)
C(40)-C(41)-C(42)-C(43)	-0.8(4)
C(41)-C(42)-C(43)-C(44)	-0.6(4)
C(42)-C(43)-C(44)-C(39)	1.3(4)
C(40)-C(39)-C(44)-C(43)	-0.5(3)
P(2)-C(39)-C(44)-C(43)	176.97(19)
N(4)-P(2)-C(45)-C(50)	170.30(19)
C(33)-P(2)-C(45)-C(50)	48.3(2)
C(39)-P(2)-C(45)-C(50)	-64.4(2)
N(4)-P(2)-C(45)-C(46)	-9.0(2)
C(33)-P(2)-C(45)-C(46)	-131.0(2)
C(39)-P(2)-C(45)-C(46)	116.3(2)
C(50)-C(45)-C(46)-C(47)	-1.4(4)
P(2)-C(45)-C(46)-C(47)	177.9(2)
C(45)-C(46)-C(47)-C(48)	-0.1(4)
C(46)-C(47)-C(48)-C(49)	1.0(4)
C(47)-C(48)-C(49)-C(50)	-0.5(4)
C(48)-C(49)-C(50)-C(45)	-1.0(4)
C(46)-C(45)-C(50)-C(49)	1.9(4)
P(2)-C(45)-C(50)-C(49)	-177.37(19)
C(31)-C(32)-C(51)-C(52)	-161.4(3)
O(2)-C(32)-C(51)-C(52)	18.1(3)
C(31)-C(32)-C(51)-C(56)	18.1(4)
O(2)-C(32)-C(51)-C(56)	-162.4(2)
C(56)-C(51)-C(52)-C(53)	1.7(4)
C(32)-C(51)-C(52)-C(53)	-178.8(2)
C(51)-C(52)-C(53)-C(54)	-1.1(4)
C(52)-C(53)-C(54)-C(55)	0.3(4)
C(52)-C(53)-C(54)-Cl(2)	178.52(19)
C(53)-C(54)-C(55)-C(56)	-0.1(4)
Cl(2)-C(54)-C(55)-C(56)	-178.36(18)
C(54)-C(55)-C(56)-C(51)	0.7(4)
C(52)-C(51)-C(56)-C(55)	-1.5(3)
C(32)-C(51)-C(56)-C(55)	178.9(2)

3-Imino-(triphenyl)-phosphorano-5-phenylisoxazole **11c**

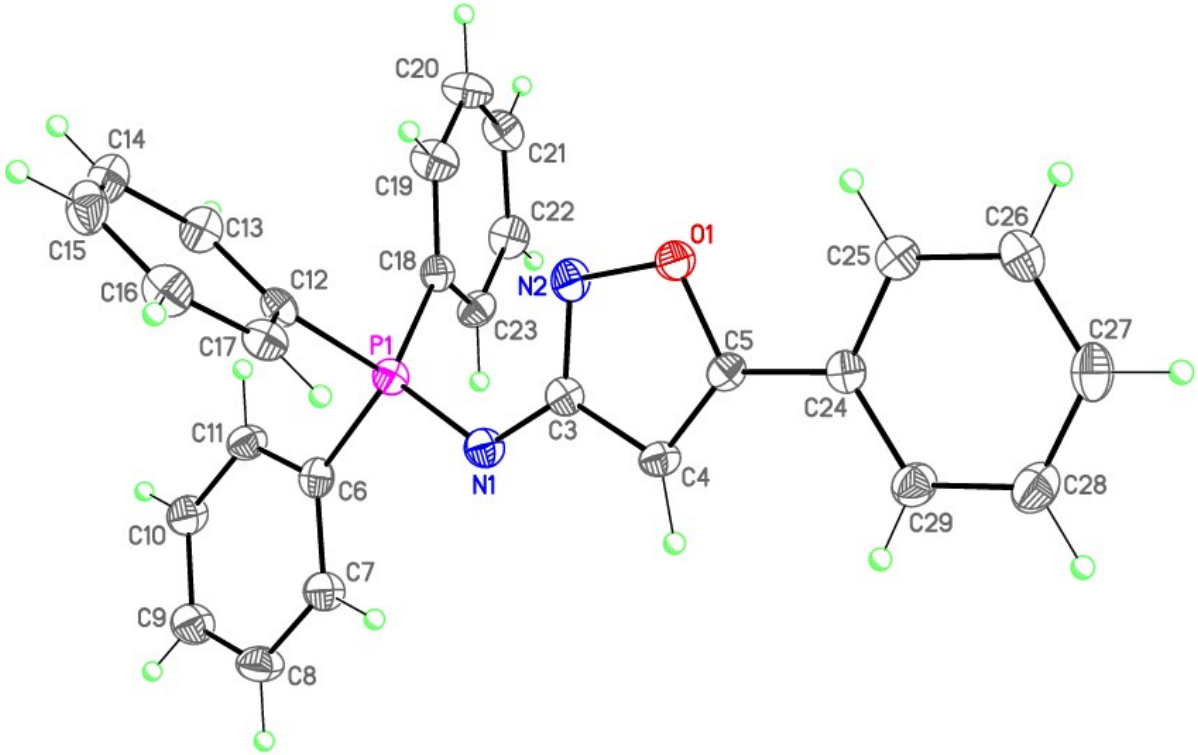


Table 1. Crystal data and structure refinement for **11c**.

Identification code	**LPS048_sq**	
Empirical formula	C27 H21 N2 O P	
Formula weight	420.43	
Temperature	100(2) K	
Wavelength	0.96260 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 12.429(3) Å	α = 90°.
	b = 10.415(2) Å	β = 90.13(3)°.
	c = 37.841(8) Å	γ = 90°.
Volume	4898.4(18) Å^3	
Z	8	
Density (calculated)	1.140 Mg/m^3	
Absorption coefficient	0.289 mm^{-1}	
F(000)	1760	
Crystal size	0.40 x 0.35 x 0.30 mm^3	
Theta range for data collection	3.436 to 38.468°.	
Index ranges	-15<=h<=14, -13<=k<=13, -32<=l<=48	
Reflections collected	46720	
Independent reflections	10579 [R(int) = 0.1035]	
Completeness to theta = 35.279°	98.4 %	
Absorption correction	Semi-empirical from equivalents	

Max. and min. transmission	0.910 and 0.880
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	10579 / 0 / 560
Goodness-of-fit on F^2	1.057
Final R indices [for 8072 rflns with $I>2\sigma(I)$]	R1 = 0.0524, wR2 = 0.1369
R indices (all data)	R1 = 0.0717, wR2 = 0.1567
Extinction coefficient	0.0062(5)
Largest diff. peak and hole	0.395 and -0.540 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for ***LPS048***. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U(eq)
P(1)	7652(1)	7318(1)	1085(1)	18(1)
O(1)	5142(1)	8601(1)	334(1)	21(1)
N(1)	6757(1)	6541(2)	867(1)	21(1)
N(2)	6028(1)	8400(2)	576(1)	22(1)
C(3)	6049(1)	7138(2)	637(1)	18(1)
C(4)	5214(1)	6488(2)	438(1)	19(1)
C(5)	4687(1)	7433(2)	261(1)	18(1)
C(6)	8362(1)	6118(2)	1343(1)	21(1)
C(7)	8256(2)	4809(2)	1258(1)	24(1)
C(8)	8888(2)	3895(2)	1435(1)	29(1)
C(9)	9616(2)	4279(2)	1697(1)	29(1)
C(10)	9712(2)	5580(2)	1784(1)	26(1)
C(11)	9092(2)	6497(2)	1609(1)	23(1)
C(12)	8693(1)	8058(2)	809(1)	19(1)
C(13)	9458(2)	8923(2)	946(1)	25(1)
C(14)	10272(2)	9410(2)	728(1)	28(1)
C(15)	10342(2)	9018(2)	376(1)	29(1)
C(16)	9588(2)	8150(2)	239(1)	28(1)
C(17)	8767(2)	7673(2)	452(1)	23(1)
C(18)	7214(1)	8558(2)	1396(1)	19(1)
C(19)	7270(2)	9867(2)	1312(1)	28(1)
C(20)	6948(2)	10794(2)	1561(1)	30(1)
C(21)	6552(2)	10417(2)	1888(1)	26(1)
C(22)	6458(2)	9113(2)	1970(1)	26(1)
C(23)	6798(1)	8178(2)	1727(1)	22(1)
C(24)	3733(1)	7452(2)	25(1)	18(1)
C(25)	3383(1)	8607(2)	-134(1)	20(1)
C(26)	2462(1)	8618(2)	-347(1)	23(1)
C(27)	1899(2)	7489(2)	-413(1)	26(1)
C(28)	2251(2)	6335(2)	-262(1)	28(1)
C(29)	3156(2)	6316(2)	-42(1)	24(1)
P(2)	6132(1)	-458(1)	3238(1)	18(1)
O(2)	8524(1)	909(1)	2453(1)	21(1)
N(3)	7070(1)	-1172(2)	3025(1)	21(1)
N(4)	7688(1)	695(2)	2711(1)	22(1)

C(30)	7731(1)	-561(2)	2786(1)	19(1)
C(31)	8571(1)	-1194(2)	2585(1)	18(1)
C(32)	9026(1)	-243(2)	2387(1)	18(1)
C(33)	5621(1)	-1630(2)	3550(1)	20(1)
C(34)	5990(2)	-2906(2)	3532(1)	28(1)
C(35)	5609(2)	-3817(2)	3772(1)	36(1)
C(36)	4873(2)	-3463(2)	4035(1)	33(1)
C(37)	4506(2)	-2200(2)	4054(1)	28(1)
C(38)	4868(2)	-1278(2)	3813(1)	24(1)
C(39)	4985(1)	60(2)	2969(1)	20(1)
C(40)	4040(2)	-693(2)	2954(1)	30(1)
C(41)	3212(2)	-371(2)	2719(1)	37(1)
C(42)	3324(2)	686(2)	2494(1)	32(1)
C(43)	4248(2)	1429(2)	2508(1)	27(1)
C(44)	5082(2)	1137(2)	2745(1)	23(1)
C(45)	6560(1)	898(2)	3508(1)	22(1)
C(46)	7631(2)	914(2)	3627(1)	34(1)
C(47)	7973(2)	1849(2)	3865(1)	42(1)
C(48)	7243(2)	2748(2)	3998(1)	38(1)
C(49)	6178(2)	2732(2)	3882(1)	31(1)
C(50)	5836(2)	1822(2)	3634(1)	26(1)
C(51)	9926(1)	-206(2)	2132(1)	19(1)
C(52)	10016(2)	810(2)	1889(1)	24(1)
C(53)	10876(2)	846(2)	1649(1)	28(1)
C(54)	11638(2)	-136(2)	1649(1)	29(1)
C(55)	11555(2)	-1158(2)	1889(1)	29(1)
C(56)	10706(1)	-1191(2)	2132(1)	23(1)

Table 3. Bond lengths [Å] and angles [°] for ***LPS048***.

P(1)-N(1)	1.6034(15)	C(20)-C(21)	1.389(3)
P(1)-C(6)	1.8132(19)	C(20)-H(20)	0.9500
P(1)-C(18)	1.8299(18)	C(21)-C(22)	1.398(3)
P(1)-C(12)	1.8338(19)	C(21)-H(21)	0.9500
O(1)-C(5)	1.370(2)	C(22)-C(23)	1.403(3)
O(1)-N(2)	1.4448(18)	C(22)-H(22)	0.9500
N(1)-C(3)	1.382(2)	C(23)-H(23)	0.9500
N(2)-C(3)	1.336(2)	C(24)-C(29)	1.407(3)
C(3)-C(4)	1.450(2)	C(24)-C(25)	1.412(3)
C(4)-C(5)	1.358(2)	C(25)-C(26)	1.400(2)
C(4)-H(4)	0.9500	C(25)-H(25)	0.9500
C(5)-C(24)	1.482(2)	C(26)-C(27)	1.390(3)
C(6)-C(7)	1.407(3)	C(26)-H(26)	0.9500
C(6)-C(11)	1.412(2)	C(27)-C(28)	1.401(3)
C(7)-C(8)	1.403(3)	C(27)-H(27)	0.9500
C(7)-H(7)	0.9500	C(28)-C(29)	1.398(3)
C(8)-C(9)	1.399(3)	C(28)-H(28)	0.9500
C(8)-H(8)	0.9500	C(29)-H(29)	0.9500
C(9)-C(10)	1.400(3)	P(2)-N(3)	1.6012(16)
C(9)-H(9)	0.9500	P(2)-C(33)	1.8163(18)
C(10)-C(11)	1.392(3)	P(2)-C(45)	1.821(2)
C(10)-H(10)	0.9500	P(2)-C(39)	1.8305(19)
C(11)-H(11)	0.9500	O(2)-C(32)	1.375(2)
C(12)-C(13)	1.407(3)	O(2)-N(4)	1.4450(18)
C(12)-C(17)	1.412(2)	N(3)-C(30)	1.379(2)
C(13)-C(14)	1.400(3)	N(4)-C(30)	1.340(2)
C(13)-H(13)	0.9500	C(30)-C(31)	1.450(2)
C(14)-C(15)	1.397(3)	C(31)-C(32)	1.366(3)
C(14)-H(14)	0.9500	C(31)-H(31)	0.9500
C(15)-C(16)	1.401(3)	C(32)-C(51)	1.478(2)
C(15)-H(15)	0.9500	C(33)-C(34)	1.407(3)
C(16)-C(17)	1.394(3)	C(33)-C(38)	1.415(2)
C(16)-H(16)	0.9500	C(34)-C(35)	1.397(3)
C(17)-H(17)	0.9500	C(34)-H(34)	0.9500
C(18)-C(19)	1.401(3)	C(35)-C(36)	1.403(3)
C(18)-C(23)	1.415(2)	C(35)-H(35)	0.9500
C(19)-C(20)	1.407(3)	C(36)-C(37)	1.394(3)
C(19)-H(19)	0.9500	C(36)-H(36)	0.9500

C(37)-C(38)	1.401(3)	C(47)-C(48)	1.397(3)
C(37)-H(37)	0.9500	C(47)-H(47)	0.9500
C(38)-H(38)	0.9500	C(48)-C(49)	1.394(3)
C(39)-C(44)	1.411(3)	C(48)-H(48)	0.9500
C(39)-C(40)	1.413(3)	C(49)-C(50)	1.401(3)
C(40)-C(41)	1.400(3)	C(49)-H(49)	0.9500
C(40)-H(40)	0.9500	C(50)-H(50)	0.9500
C(41)-C(42)	1.398(3)	C(51)-C(52)	1.408(3)
C(41)-H(41)	0.9500	C(51)-C(56)	1.412(3)
C(42)-C(43)	1.386(3)	C(52)-C(53)	1.403(3)
C(42)-H(42)	0.9500	C(52)-H(52)	0.9500
C(43)-C(44)	1.405(3)	C(53)-C(54)	1.393(3)
C(43)-H(43)	0.9500	C(53)-H(53)	0.9500
C(44)-H(44)	0.9500	C(54)-C(55)	1.405(3)
C(45)-C(50)	1.401(3)	C(54)-H(54)	0.9500
C(45)-C(46)	1.405(3)	C(55)-C(56)	1.401(3)
C(46)-C(47)	1.393(3)	C(55)-H(55)	0.9500
C(46)-H(46)	0.9500	C(56)-H(56)	0.9500

N(1)-P(1)-C(6)	105.44(8)	C(8)-C(7)-C(6)	119.87(17)
N(1)-P(1)-C(18)	118.70(8)	C(8)-C(7)-H(7)	120.1
C(6)-P(1)-C(18)	106.61(8)	C(6)-C(7)-H(7)	120.1
N(1)-P(1)-C(12)	114.12(8)	C(9)-C(8)-C(7)	120.23(19)
C(6)-P(1)-C(12)	104.60(8)	C(9)-C(8)-H(8)	119.9
C(18)-P(1)-C(12)	106.25(9)	C(7)-C(8)-H(8)	119.9
C(5)-O(1)-N(2)	108.26(13)	C(8)-C(9)-C(10)	119.93(18)
C(3)-N(1)-P(1)	122.45(13)	C(8)-C(9)-H(9)	120.0
C(3)-N(2)-O(1)	105.52(13)	C(10)-C(9)-H(9)	120.0
N(2)-C(3)-N(1)	124.37(16)	C(11)-C(10)-C(9)	120.31(17)
N(2)-C(3)-C(4)	110.80(15)	C(11)-C(10)-H(10)	119.8
N(1)-C(3)-C(4)	124.82(17)	C(9)-C(10)-H(10)	119.8
C(5)-C(4)-C(3)	105.21(16)	C(10)-C(11)-C(6)	120.13(18)
C(5)-C(4)-H(4)	127.4	C(10)-C(11)-H(11)	119.9
C(3)-C(4)-H(4)	127.4	C(6)-C(11)-H(11)	119.9
C(4)-C(5)-O(1)	110.20(15)	C(13)-C(12)-C(17)	119.17(16)
C(4)-C(5)-C(24)	133.71(17)	C(13)-C(12)-P(1)	122.52(13)
O(1)-C(5)-C(24)	116.05(15)	C(17)-C(12)-P(1)	118.17(14)
C(7)-C(6)-C(11)	119.53(17)	C(14)-C(13)-C(12)	120.33(17)
C(7)-C(6)-P(1)	120.02(13)	C(14)-C(13)-H(13)	119.8
C(11)-C(6)-P(1)	120.25(15)	C(12)-C(13)-H(13)	119.8

C(15)-C(14)-C(13)	120.07(19)	C(26)-C(27)-H(27)	120.1
C(15)-C(14)-H(14)	120.0	C(28)-C(27)-H(27)	120.1
C(13)-C(14)-H(14)	120.0	C(29)-C(28)-C(27)	120.37(19)
C(14)-C(15)-C(16)	119.91(18)	C(29)-C(28)-H(28)	119.8
C(14)-C(15)-H(15)	120.0	C(27)-C(28)-H(28)	119.8
C(16)-C(15)-H(15)	120.0	C(28)-C(29)-C(24)	120.28(18)
C(17)-C(16)-C(15)	120.37(17)	C(28)-C(29)-H(29)	119.9
C(17)-C(16)-H(16)	119.8	C(24)-C(29)-H(29)	119.9
C(15)-C(16)-H(16)	119.8	N(3)-P(2)-C(33)	105.73(8)
C(16)-C(17)-C(12)	120.14(18)	N(3)-P(2)-C(45)	115.50(8)
C(16)-C(17)-H(17)	119.9	C(33)-P(2)-C(45)	104.95(8)
C(12)-C(17)-H(17)	119.9	N(3)-P(2)-C(39)	115.13(8)
C(19)-C(18)-C(23)	119.32(17)	C(33)-P(2)-C(39)	106.64(8)
C(19)-C(18)-P(1)	121.84(13)	C(45)-P(2)-C(39)	108.01(9)
C(23)-C(18)-P(1)	118.84(14)	C(32)-O(2)-N(4)	108.36(13)
C(18)-C(19)-C(20)	120.22(17)	C(30)-N(3)-P(2)	123.44(14)
C(18)-C(19)-H(19)	119.9	C(30)-N(4)-O(2)	105.37(13)
C(20)-C(19)-H(19)	119.9	N(4)-C(30)-N(3)	124.44(16)
C(21)-C(20)-C(19)	120.24(19)	N(4)-C(30)-C(31)	111.18(15)
C(21)-C(20)-H(20)	119.9	N(3)-C(30)-C(31)	124.37(17)
C(19)-C(20)-H(20)	119.9	C(32)-C(31)-C(30)	104.94(16)
C(20)-C(21)-C(22)	120.06(18)	C(32)-C(31)-H(31)	127.5
C(20)-C(21)-H(21)	120.0	C(30)-C(31)-H(31)	127.5
C(22)-C(21)-H(21)	120.0	C(31)-C(32)-O(2)	110.15(15)
C(21)-C(22)-C(23)	120.33(17)	C(31)-C(32)-C(51)	133.78(17)
C(21)-C(22)-H(22)	119.8	O(2)-C(32)-C(51)	116.06(15)
C(23)-C(22)-H(22)	119.8	C(34)-C(33)-C(38)	119.79(17)
C(22)-C(23)-C(18)	119.77(18)	C(34)-C(33)-P(2)	119.16(13)
C(22)-C(23)-H(23)	120.1	C(38)-C(33)-P(2)	121.05(15)
C(18)-C(23)-H(23)	120.1	C(35)-C(34)-C(33)	119.79(18)
C(29)-C(24)-C(25)	118.93(16)	C(35)-C(34)-H(34)	120.1
C(29)-C(24)-C(5)	120.20(16)	C(33)-C(34)-H(34)	120.1
C(25)-C(24)-C(5)	120.87(16)	C(34)-C(35)-C(36)	120.4(2)
C(26)-C(25)-C(24)	120.23(18)	C(34)-C(35)-H(35)	119.8
C(26)-C(25)-H(25)	119.9	C(36)-C(35)-H(35)	119.8
C(24)-C(25)-H(25)	119.9	C(37)-C(36)-C(35)	120.00(18)
C(27)-C(26)-C(25)	120.42(18)	C(37)-C(36)-H(36)	120.0
C(27)-C(26)-H(26)	119.8	C(35)-C(36)-H(36)	120.0
C(25)-C(26)-H(26)	119.8	C(36)-C(37)-C(38)	120.40(18)
C(26)-C(27)-C(28)	119.74(17)	C(36)-C(37)-H(37)	119.8

C(38)-C(37)-H(37)	119.8	C(46)-C(47)-H(47)	119.9
C(37)-C(38)-C(33)	119.62(18)	C(48)-C(47)-H(47)	119.9
C(37)-C(38)-H(38)	120.2	C(49)-C(48)-C(47)	119.76(19)
C(33)-C(38)-H(38)	120.2	C(49)-C(48)-H(48)	120.1
C(44)-C(39)-C(40)	119.26(17)	C(47)-C(48)-H(48)	120.1
C(44)-C(39)-P(2)	119.99(14)	C(48)-C(49)-C(50)	120.4(2)
C(40)-C(39)-P(2)	120.37(14)	C(48)-C(49)-H(49)	119.8
C(41)-C(40)-C(39)	120.18(19)	C(50)-C(49)-H(49)	119.8
C(41)-C(40)-H(40)	119.9	C(49)-C(50)-C(45)	119.95(19)
C(39)-C(40)-H(40)	119.9	C(49)-C(50)-H(50)	120.0
C(42)-C(41)-C(40)	120.1(2)	C(45)-C(50)-H(50)	120.0
C(42)-C(41)-H(41)	119.9	C(52)-C(51)-C(56)	119.35(16)
C(40)-C(41)-H(41)	119.9	C(52)-C(51)-C(32)	120.52(16)
C(43)-C(42)-C(41)	120.01(18)	C(56)-C(51)-C(32)	120.13(16)
C(43)-C(42)-H(42)	120.0	C(53)-C(52)-C(51)	120.30(18)
C(41)-C(42)-H(42)	120.0	C(53)-C(52)-H(52)	119.9
C(42)-C(43)-C(44)	120.90(19)	C(51)-C(52)-H(52)	119.9
C(42)-C(43)-H(43)	119.5	C(54)-C(53)-C(52)	120.00(19)
C(44)-C(43)-H(43)	119.5	C(54)-C(53)-H(53)	120.0
C(43)-C(44)-C(39)	119.51(18)	C(52)-C(53)-H(53)	120.0
C(43)-C(44)-H(44)	120.2	C(53)-C(54)-C(55)	120.29(18)
C(39)-C(44)-H(44)	120.2	C(53)-C(54)-H(54)	119.9
C(50)-C(45)-C(46)	119.40(18)	C(55)-C(54)-H(54)	119.9
C(50)-C(45)-P(2)	122.45(14)	C(56)-C(55)-C(54)	120.02(19)
C(46)-C(45)-P(2)	117.68(15)	C(56)-C(55)-H(55)	120.0
C(47)-C(46)-C(45)	120.3(2)	C(54)-C(55)-H(55)	120.0
C(47)-C(46)-H(46)	119.9	C(55)-C(56)-C(51)	120.03(18)
C(45)-C(46)-H(46)	119.9	C(55)-C(56)-H(56)	120.0
C(46)-C(47)-C(48)	120.2(2)	C(51)-C(56)-H(56)	120.0

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for ***LPS048***. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
P(1)	19(1)	19(1)	15(1)	1(1)	-2(1)	1(1)
O(1)	22(1)	20(1)	23(1)	3(1)	-7(1)	-1(1)
N(1)	23(1)	21(1)	19(1)	2(1)	-4(1)	1(1)
N(2)	21(1)	25(1)	20(1)	3(1)	-8(1)	0(1)
C(3)	20(1)	20(1)	15(1)	1(1)	-1(1)	1(1)
C(4)	23(1)	16(1)	18(1)	-1(1)	-2(1)	-2(1)
C(5)	19(1)	18(1)	16(1)	-1(1)	0(1)	-2(1)
C(6)	19(1)	25(1)	18(1)	1(1)	1(1)	-1(1)
C(7)	26(1)	23(1)	22(1)	1(1)	-6(1)	0(1)
C(8)	36(1)	19(1)	33(1)	2(1)	-9(1)	2(1)
C(9)	31(1)	26(1)	30(1)	6(1)	-9(1)	2(1)
C(10)	27(1)	26(1)	27(1)	3(1)	-11(1)	-2(1)
C(11)	26(1)	20(1)	25(1)	1(1)	-6(1)	-3(1)
C(12)	19(1)	20(1)	18(1)	1(1)	-2(1)	5(1)
C(13)	26(1)	30(1)	18(1)	0(1)	-2(1)	-2(1)
C(14)	24(1)	34(1)	27(1)	4(1)	-2(1)	-5(1)
C(15)	23(1)	38(1)	27(1)	10(1)	6(1)	2(1)
C(16)	33(1)	32(1)	19(1)	0(1)	4(1)	4(1)
C(17)	27(1)	22(1)	19(1)	0(1)	0(1)	3(1)
C(18)	18(1)	21(1)	17(1)	0(1)	-2(1)	0(1)
C(19)	36(1)	27(1)	19(1)	3(1)	5(1)	3(1)
C(20)	43(1)	20(1)	28(1)	1(1)	3(1)	4(1)
C(21)	29(1)	29(1)	21(1)	-5(1)	-2(1)	5(1)
C(22)	28(1)	33(1)	16(1)	-1(1)	1(1)	-1(1)
C(23)	24(1)	23(1)	20(1)	2(1)	-2(1)	-5(1)
C(24)	18(1)	22(1)	15(1)	-1(1)	1(1)	1(1)
C(25)	22(1)	22(1)	17(1)	0(1)	1(1)	-1(1)
C(26)	23(1)	29(1)	18(1)	1(1)	0(1)	5(1)
C(27)	20(1)	37(1)	20(1)	0(1)	-2(1)	1(1)
C(28)	27(1)	29(1)	28(1)	0(1)	-5(1)	-7(1)
C(29)	25(1)	22(1)	23(1)	2(1)	-4(1)	-3(1)
P(2)	19(1)	20(1)	15(1)	2(1)	1(1)	0(1)
O(2)	22(1)	20(1)	19(1)	1(1)	4(1)	0(1)
N(3)	24(1)	21(1)	18(1)	1(1)	3(1)	-1(1)
N(4)	22(1)	25(1)	18(1)	0(1)	6(1)	-1(1)

C(30)	20(1)	21(1)	16(1)	0(1)	-3(1)	-2(1)
C(31)	20(1)	19(1)	16(1)	-2(1)	0(1)	-1(1)
C(32)	18(1)	20(1)	18(1)	-2(1)	-3(1)	1(1)
C(33)	20(1)	22(1)	18(1)	3(1)	0(1)	1(1)
C(34)	28(1)	28(1)	29(1)	8(1)	9(1)	7(1)
C(35)	38(1)	30(1)	41(1)	14(1)	11(1)	11(1)
C(36)	32(1)	34(1)	32(1)	17(1)	7(1)	2(1)
C(37)	27(1)	34(1)	22(1)	4(1)	6(1)	0(1)
C(38)	25(1)	25(1)	20(1)	2(1)	2(1)	1(1)
C(39)	22(1)	21(1)	17(1)	-1(1)	1(1)	2(1)
C(40)	31(1)	30(1)	29(1)	6(1)	-6(1)	-4(1)
C(41)	30(1)	44(2)	38(1)	2(1)	-12(1)	-7(1)
C(42)	32(1)	41(1)	23(1)	-1(1)	-8(1)	9(1)
C(43)	34(1)	28(1)	18(1)	4(1)	1(1)	10(1)
C(44)	26(1)	25(1)	18(1)	0(1)	2(1)	1(1)
C(45)	23(1)	25(1)	17(1)	2(1)	2(1)	-2(1)
C(46)	28(1)	44(1)	29(1)	-10(1)	-1(1)	0(1)
C(47)	35(1)	58(2)	34(1)	-13(1)	-7(1)	-7(1)
C(48)	54(1)	39(1)	23(1)	-8(1)	4(1)	-17(1)
C(49)	45(1)	21(1)	28(1)	-1(1)	15(1)	-5(1)
C(50)	30(1)	22(1)	26(1)	5(1)	6(1)	-1(1)
C(51)	20(1)	19(1)	17(1)	-3(1)	-2(1)	-2(1)
C(52)	27(1)	23(1)	22(1)	0(1)	2(1)	1(1)
C(53)	34(1)	26(1)	23(1)	0(1)	6(1)	-6(1)
C(54)	24(1)	35(1)	29(1)	-7(1)	7(1)	-7(1)
C(55)	22(1)	32(1)	33(1)	-5(1)	2(1)	1(1)
C(56)	21(1)	24(1)	24(1)	-1(1)	-2(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for ***LPS048***.

Atom	x	y	z	U(iso)
H(4)	5068	5593	432	23
H(7)	7757	4545	1082	28
H(8)	8823	3011	1376	35
H(9)	10043	3658	1816	35
H(10)	10203	5840	1963	32
H(11)	9161	7378	1670	28
H(13)	9422	9178	1186	30
H(14)	10777	10007	820	34
H(15)	10900	9340	229	35
H(16)	9636	7884	-1	34
H(17)	8256	7087	358	27
H(19)	7526	10129	1087	33
H(20)	7001	11681	1505	36
H(21)	6344	11046	2056	32
H(22)	6163	8858	2190	31
H(23)	6748	7293	1786	27
H(25)	3773	9378	-95	24
H(26)	2220	9402	-448	28
H(27)	1277	7500	-559	31
H(28)	1872	5561	-309	34
H(29)	3383	5532	63	28
H(31)	8762	-2076	2591	22
H(34)	6497	-3147	3356	34
H(35)	5850	-4681	3757	44
H(36)	4625	-4084	4200	39
H(37)	4006	-1964	4233	33
H(38)	4611	-420	3826	28
H(40)	3967	-1420	3104	36
H(41)	2574	-872	2711	45
H(42)	2766	895	2332	39
H(43)	4318	2147	2354	32
H(44)	5707	1661	2755	28
H(46)	8124	285	3545	40
H(47)	8705	1875	3938	51
H(48)	7473	3368	4166	46
H(49)	5681	3343	3972	38
H(50)	5114	1830	3551	31
H(52)	9493	1475	1887	29
H(53)	10939	1539	1488	33
H(54)	12215	-114	1485	35
H(55)	12074	-1827	1887	35
H(56)	10656	-1876	2297	28

Table 6. Torsion angles [°] for ***LPS048***.

C(6)-P(1)-N(1)-C(3)	-179.94(13)	C(13)-C(14)-C(15)-C(16)	0.8(3)
C(18)-P(1)-N(1)-C(3)	60.76(16)	C(14)-C(15)-C(16)-C(17)	0.0(3)
C(12)-P(1)-N(1)-C(3)	-65.73(16)	C(15)-C(16)-C(17)-C(12)	-0.3(3)
C(5)-O(1)-N(2)-C(3)	-0.69(17)	C(13)-C(12)-C(17)-C(16)	-0.2(3)
O(1)-N(2)-C(3)-N(1)	-177.54(15)	P(1)-C(12)-C(17)-C(16)	-175.98(14)
O(1)-N(2)-C(3)-C(4)	1.13(18)	N(1)-P(1)-C(18)-C(19)	-102.12(16)
P(1)-N(1)-C(3)-N(2)	-0.5(2)	C(6)-P(1)-C(18)-C(19)	139.20(15)
P(1)-N(1)-C(3)-C(4)	-178.99(13)	C(12)-P(1)-C(18)-C(19)	28.04(17)
N(2)-C(3)-C(4)-C(5)	-1.17(19)	N(1)-P(1)-C(18)-C(23)	77.20(16)
N(1)-C(3)-C(4)-C(5)	177.49(16)	C(6)-P(1)-C(18)-C(23)	-41.49(16)
C(3)-C(4)-C(5)-O(1)	0.70(18)	C(12)-P(1)-C(18)-C(23)	-152.65(14)
C(3)-C(4)-C(5)-C(24)	-176.79(17)	C(23)-C(18)-C(19)-C(20)	2.1(3)
N(2)-O(1)-C(5)-C(4)	-0.04(18)	P(1)-C(18)-C(19)-C(20)	-178.61(15)
N(2)-O(1)-C(5)-C(24)	177.94(13)	C(18)-C(19)-C(20)-C(21)	-1.3(3)
N(1)-P(1)-C(6)-C(7)	15.54(16)	C(19)-C(20)-C(21)-C(22)	-0.9(3)
C(18)-P(1)-C(6)-C(7)	142.58(14)	C(20)-C(21)-C(22)-C(23)	2.2(3)
C(12)-P(1)-C(6)-C(7)	-105.12(15)	C(21)-C(22)-C(23)-C(18)	-1.4(3)
N(1)-P(1)-C(6)-C(11)	-169.67(14)	C(19)-C(18)-C(23)-C(22)	-0.7(3)
C(18)-P(1)-C(6)-C(11)	-42.64(16)	P(1)-C(18)-C(23)-C(22)	179.96(13)
C(12)-P(1)-C(6)-C(11)	69.66(16)	C(4)-C(5)-C(24)-C(29)	2.8(3)
C(11)-C(6)-C(7)-C(8)	-1.2(3)	O(1)-C(5)-C(24)-C(29)	-174.62(15)
P(1)-C(6)-C(7)-C(8)	173.60(15)	C(4)-C(5)-C(24)-C(25)	-177.93(18)
C(6)-C(7)-C(8)-C(9)	0.8(3)	O(1)-C(5)-C(24)-C(25)	4.7(2)
C(7)-C(8)-C(9)-C(10)	0.0(3)	C(29)-C(24)-C(25)-C(26)	1.3(2)
C(8)-C(9)-C(10)-C(11)	-0.4(3)	C(5)-C(24)-C(25)-C(26)	-178.05(15)
C(9)-C(10)-C(11)-C(6)	-0.1(3)	C(24)-C(25)-C(26)-C(27)	-1.5(3)
C(7)-C(6)-C(11)-C(10)	0.9(3)	C(25)-C(26)-C(27)-C(28)	0.5(3)
P(1)-C(6)-C(11)-C(10)	-173.93(14)	C(26)-C(27)-C(28)-C(29)	0.8(3)
N(1)-P(1)-C(12)-C(13)	170.92(15)	C(27)-C(28)-C(29)-C(24)	-1.1(3)
C(6)-P(1)-C(12)-C(13)	-74.37(17)	C(25)-C(24)-C(29)-C(28)	0.0(3)
C(18)-P(1)-C(12)-C(13)	38.18(17)	C(5)-C(24)-C(29)-C(28)	179.37(16)
N(1)-P(1)-C(12)-C(17)	-13.41(17)	C(33)-P(2)-N(3)-C(30)	-173.54(13)
C(6)-P(1)-C(12)-C(17)	101.29(15)	C(45)-P(2)-N(3)-C(30)	-57.99(16)
C(18)-P(1)-C(12)-C(17)	-146.15(14)	C(39)-P(2)-N(3)-C(30)	69.03(16)
C(17)-C(12)-C(13)-C(14)	1.0(3)	C(32)-O(2)-N(4)-C(30)	-0.04(16)
P(1)-C(12)-C(13)-C(14)	176.61(15)	O(2)-N(4)-C(30)-N(3)	179.35(15)
C(12)-C(13)-C(14)-C(15)	-1.3(3)	O(2)-N(4)-C(30)-C(31)	-0.02(18)

P(2)-N(3)-C(30)-N(4)	2.0(2)
P(2)-N(3)-C(30)-C(31)	-178.75(13)
N(4)-C(30)-C(31)-C(32)	0.06(19)
N(3)-C(30)-C(31)-C(32)	-179.30(15)
C(30)-C(31)-C(32)-O(2)	-0.08(18)
C(30)-C(31)-C(32)-C(51)	178.57(17)
N(4)-O(2)-C(32)-C(31)	0.08(17)
N(4)-O(2)-C(32)-C(51)	-178.84(13)
N(3)-P(2)-C(33)-C(34)	-7.22(17)
C(45)-P(2)-C(33)-C(34)	-129.78(16)
C(39)-P(2)-C(33)-C(34)	115.78(16)
N(3)-P(2)-C(33)-C(38)	172.20(14)
C(45)-P(2)-C(33)-C(38)	49.64(16)
C(39)-P(2)-C(33)-C(38)	-64.80(16)
C(38)-C(33)-C(34)-C(35)	0.1(3)
P(2)-C(33)-C(34)-C(35)	179.57(16)
C(33)-C(34)-C(35)-C(36)	-0.9(3)
C(34)-C(35)-C(36)-C(37)	0.9(3)
C(35)-C(36)-C(37)-C(38)	-0.1(3)
C(36)-C(37)-C(38)-C(33)	-0.7(3)
C(34)-C(33)-C(38)-C(37)	0.7(3)
P(2)-C(33)-C(38)-C(37)	-178.76(14)
N(3)-P(2)-C(39)-C(44)	-74.62(17)
C(33)-P(2)-C(39)-C(44)	168.46(14)
C(45)-P(2)-C(39)-C(44)	56.11(16)
N(3)-P(2)-C(39)-C(40)	98.29(16)
C(33)-P(2)-C(39)-C(40)	-18.63(18)
C(45)-P(2)-C(39)-C(40)	-130.98(16)
C(44)-C(39)-C(40)-C(41)	0.1(3)
P(2)-C(39)-C(40)-C(41)	-172.88(16)
C(39)-C(40)-C(41)-C(42)	0.9(3)
C(40)-C(41)-C(42)-C(43)	-1.0(3)
C(41)-C(42)-C(43)-C(44)	0.0(3)
C(42)-C(43)-C(44)-C(39)	1.0(3)
C(40)-C(39)-C(44)-C(43)	-1.0(3)
P(2)-C(39)-C(44)-C(43)	171.96(14)
N(3)-P(2)-C(45)-C(50)	162.75(14)
C(33)-P(2)-C(45)-C(50)	-81.25(17)
C(39)-P(2)-C(45)-C(50)	32.23(17)
N(3)-P(2)-C(45)-C(46)	-25.19(18)
C(33)-P(2)-C(45)-C(46)	90.81(16)
C(39)-P(2)-C(45)-C(46)	-155.71(15)
C(50)-C(45)-C(46)-C(47)	-0.7(3)
P(2)-C(45)-C(46)-C(47)	-173.00(17)
C(45)-C(46)-C(47)-C(48)	2.4(3)
C(46)-C(47)-C(48)-C(49)	-2.0(3)
C(47)-C(48)-C(49)-C(50)	-0.1(3)
C(48)-C(49)-C(50)-C(45)	1.8(3)
C(46)-C(45)-C(50)-C(49)	-1.4(3)
P(2)-C(45)-C(50)-C(49)	170.54(14)
C(31)-C(32)-C(51)-C(52)	160.70(19)
O(2)-C(32)-C(51)-C(52)	-20.7(2)
C(31)-C(32)-C(51)-C(56)	-19.6(3)
O(2)-C(32)-C(51)-C(56)	159.01(16)
C(56)-C(51)-C(52)-C(53)	-0.2(3)
C(32)-C(51)-C(52)-C(53)	179.52(16)
C(51)-C(52)-C(53)-C(54)	0.8(3)
C(52)-C(53)-C(54)-C(55)	-0.6(3)
C(53)-C(54)-C(55)-C(56)	-0.2(3)
C(54)-C(55)-C(56)-C(51)	0.9(3)
C(52)-C(51)-C(56)-C(55)	-0.6(3)
C(32)-C(51)-C(56)-C(55)	179.64(16)

DFT calculations

Geometry optimizations, electronic population and vibrational analyzes were performed using Gaussian 09 program package[ix]. Density functional theory (DFT), B3LYP hybrid functional[x] with 6-311+G(d,p) basis set were used. All stationary points on the potential energy surface (PES) were confirmed by the analysis of the harmonic vibrational frequencies using analytical second derivatives. The intrinsic reaction coordinate (IRC) procedure was used to connect the reagents and the products with the corresponding transition state (TS). To model the solute-solvent interactions, polarizable continuum model (PCM)[xi] was used. All units are a.u. unless stated otherwise.

B3LYP/6-311+G//PCM(Acetonitrile)**

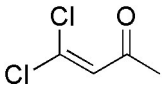
Molecule	ε_0	$\varepsilon_0 + \varepsilon_{ZPE}$	$\varepsilon_0 + \varepsilon_{tot}$	ε_0 + *H corr*	ε_0 + G *corr*
1	-1150.5505320	-1150.479937	-1150.472037	-1150.471093	-1150.514316
2	-854.5649537	-854.481767	-854.472533	-854.471589	-854.517546
3	-854.5670246	-854.484201	-854.474683	-854.473739	-854.520730
4	-558.5734929	-558.478357	-558.467319	-558.466375	-558.517029
5	-745.0638222	-744.988167	-744.981798	-744.980854	-745.019195
6	-449.0769581	-448.988763	-448.980900	-448.979956	-449.021953
TS1-2/1-3	-1314.9104287	-1314.829511	-1314.818491	-1314.817547	-1314.868419
TS2-4	-1018.9275635	-1018.833964	-1018.821671	-1018.820727	-1018.873963
TS3-4	-1018.9301691	-1018.836587	-1018.824163	-1018.823219	-1018.877438
TS3-5	-854.5233145	-854.443431	-854.434071	-854.433127	-854.479614
TS4-6	-558.5319348	-558.439722	-558.428914	-558.427970	-558.477611
TS5-6	-909.4027230	-909.317077	-909.307404	-909.306460	-909.354049
N_3^-	-164.3833660	-164.372771	-164.370111	-164.369167	-164.393977
Cl^-	-460.4130615	-460.413062	-460.411645	-460.410701	-460.428084
N_2	-109.5601813	-109.554608	-109.552247	-109.551303	-109.573040

Computed single imaginary frequencies of transition states

Transition state	Frequency, cm^{-1}
TS1-2	-360.644
TS2-4	-354.507
TS3-4	-331.105
TS3-5	-675.071
TS4-6	-682.085
TS5-6	-429.962

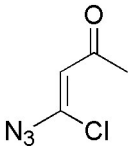
Cartesian coordinates

1



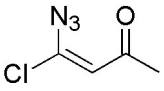
Atom	X	Y	Z
6	1.827668	-0.044553	-0.000037
6	0.501842	-0.718370	-0.000119
6	-0.705871	-0.140704	-0.000052
8	1.968804	1.167449	0.000056
6	3.007808	-0.987985	0.000008
1	3.938260	-0.422851	-0.000147
1	0.515480	-1.801583	-0.000164
1	2.969030	-1.638546	-0.879265
1	2.969210	-1.637963	0.879758
17	-2.153092	-1.114265	0.000044
17	-1.019325	1.556089	-0.000010

2



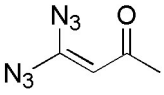
Atom	X	Y	Z
6	0.358619	-0.299829	0.000208
6	-0.843264	-0.913409	0.000260
7	1.499370	-1.099096	0.000234
17	0.612184	1.431206	0.000371
7	2.624450	-0.582468	-0.000231
7	3.705717	-0.264602	-0.000558
1	-0.792226	-1.997077	0.000242
6	-2.221965	-0.398692	-0.000056
8	-3.129873	-1.227962	0.000072
6	-2.544666	1.075852	-0.000591
1	-2.123584	1.568279	-0.880197
1	-2.124019	1.568893	0.878881
1	-3.627425	1.192730	-0.000851

3



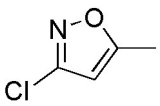
Atom	X	Y	Z
6	0.305295	0.355132	0.000200
6	-1.010860	0.652656	0.000124
7	0.796227	-0.933893	0.000626
17	1.479239	1.669252	-0.000076
6	-2.119144	-0.321832	-0.000045
8	-1.959294	-1.535787	-0.000139
6	-3.501940	0.294413	-0.000124
1	-1.276963	1.700384	0.000045
1	-4.259671	-0.487522	-0.000240
1	-3.630445	0.932615	-0.879805
1	-3.630573	0.932417	0.879686
7	2.017276	-1.141504	-0.000026
7	3.084330	-1.503330	-0.000346

4



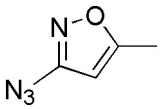
Atom	X	Y	Z
6	-0.087245	-0.347716	-0.086421
6	1.204752	-0.751868	-0.147417
7	-0.437848	0.945555	0.313035
7	-1.072527	-1.293405	-0.443103
7	-2.194592	-1.248584	0.082341
7	-3.251076	-1.354417	0.464758
7	-1.487956	1.462516	-0.092331
7	-2.384855	2.089723	-0.369220
6	2.399302	0.078538	0.058330
8	2.366174	1.276272	0.320904
6	3.716407	-0.657422	-0.078632
1	1.360393	-1.795997	-0.385458
1	3.767220	-1.478719	0.642843
1	4.546569	0.027568	0.087166
1	3.799110	-1.101933	-1.075303

5



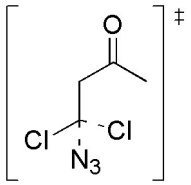
Atom	X	Y	Z
6	0.710517	0.034806	0.000036
6	-0.321473	-0.937151	0.000059
7	0.268557	1.265412	0.000023
17	2.412642	-0.262152	-0.000029
6	-1.457294	-0.185871	0.000044
8	-1.125778	1.127345	-0.000005
6	-2.904384	-0.514929	-0.000037
1	-0.225779	-2.008812	0.000073
1	-3.393290	-0.098656	-0.884525
1	-3.393754	-0.098006	0.883873
1	-3.039967	-1.595729	0.000325

6



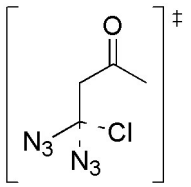
Atom	X	Y	Z
6	-0.354762	0.308372	-0.000008
6	0.857948	1.048433	-0.000052
7	-0.156880	-0.991511	0.000050
7	-1.625265	0.893047	-0.000001
7	-2.600808	0.126971	0.000029
7	-3.576627	-0.437001	-0.000044
6	1.820889	0.087655	0.000053
8	1.238224	-1.134791	-0.000012
6	3.304898	0.125452	-0.000017
1	0.979966	2.117860	0.000201
1	3.703437	-0.379132	-0.883953
1	3.703633	-0.379009	0.883881
1	3.650387	1.158606	-0.000138

TS1-2/1-3



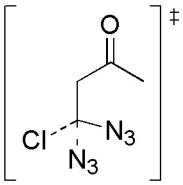
Atom	X	Y	Z
6	-2.289702	-0.220563	-0.205138
6	-1.176463	0.674059	-0.237112
6	0.135706	0.382622	0.129678
8	-2.261533	-1.407262	0.174671
6	-3.620891	0.349068	-0.686899
1	-4.374525	0.203232	0.091954
1	-1.321383	1.635613	-0.710666
1	-3.951091	-0.210213	-1.567632
1	-3.571759	1.408923	-0.940912
17	1.217556	1.817349	0.212110
17	0.448291	-0.706173	1.519789
7	0.987664	-0.617294	-1.273707
7	2.144359	-0.852175	-1.033739
7	3.253652	-1.070612	-0.794778

TS2-4



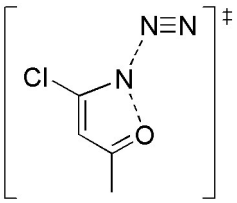
Atom	X	Y	Z
6	0.383021	-0.233894	0.304329
6	-0.837348	-0.197273	0.999765
7	1.502807	-0.098650	1.176821
17	0.662747	-1.625845	-0.915720
7	2.624107	0.004921	0.672810
7	3.704596	0.090112	0.353894
1	-0.744484	0.072949	2.045701
6	-2.164938	-0.400164	0.527949
8	-3.138831	-0.372875	1.316839
6	-2.459056	-0.660102	-0.943534
1	-2.493345	-1.739366	-1.122949
1	-1.716621	-0.236285	-1.619290
1	-3.443602	-0.250146	-1.174673
7	0.664839	1.117052	-0.912222
7	-0.098456	2.017841	-0.669752
7	-0.867618	2.829270	-0.396882

TS3-4



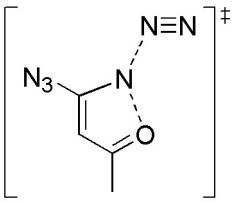
Atom	X	Y	Z
6	-0.357112	-0.061450	0.373152
6	1.010313	-0.058076	0.734520
7	-0.915388	-1.289625	-0.066789
17	-1.413126	0.718026	1.697068
7	-2.037374	-1.272471	-0.581256
7	-3.055889	-1.424013	-1.046043
1	1.301940	0.628246	1.515054
6	2.031364	-0.775307	0.054015
8	1.877567	-1.499796	-0.955421
6	3.441418	-0.646971	0.627644
1	3.505363	0.046074	1.468142
1	4.123796	-0.317349	-0.160814
1	3.780310	-1.633396	0.959301
7	-0.792209	1.023602	-1.002195
7	0.074201	1.846025	-1.156129
7	0.945985	2.590649	-1.251081

TS3-5



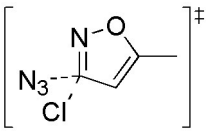
Atom	X	Y	Z
6	-0.212915	0.429675	-0.000001
6	1.113888	0.830727	-0.000007
7	-0.538427	-0.888354	0.000003
17	-1.419057	1.688418	0.000002
6	2.029529	-0.244242	-0.000007
8	1.551449	-1.406815	-0.000001
6	3.521858	-0.038618	0.000003
1	1.386674	1.874258	-0.000013
1	3.953739	-0.518971	-0.881681
1	3.953670	-0.518638	0.881904
1	3.786449	1.018390	-0.000177
7	-2.174603	-0.991579	0.000001
7	-3.013016	-1.715622	-0.000001

TS4-6



Atom	X	Y	Z
6	0.003742	-0.423175	0.111891
6	1.297213	-0.939831	0.157100
7	-0.155904	0.911277	-0.138627
7	-1.004271	-1.366791	0.366745
7	-2.148911	-1.232550	-0.085858
7	-3.222954	-1.289190	-0.425262
7	-1.696279	1.322786	0.150420
7	-2.348359	2.218617	0.229877
6	2.316385	0.005017	-0.061689
8	1.967954	1.184350	-0.333667
6	3.780266	-0.342197	0.036237
1	1.454821	-1.991805	0.339371
1	3.934822	-1.409697	0.191597
1	4.223713	0.207251	0.871345
1	4.294126	-0.028484	-0.875272

TS5-6



Atom	X	Y	Z
6	-0.354133	-0.382204	0.039031
6	0.763646	-0.464515	-0.879172
7	0.045652	-0.024038	1.253561
17	-1.553348	-1.944045	0.027905
6	1.839570	-0.098085	-0.149615
8	1.480025	0.183272	1.123390
6	3.283810	0.042250	-0.481562
1	0.724368	-0.739169	-1.919513
1	3.890106	-0.626358	0.136198
1	3.626721	1.064875	-0.301139
1	3.454600	-0.202402	-1.529966
7	-1.642548	0.693067	-0.681950
7	-1.466562	1.815399	-0.280566
7	-1.268890	2.872854	0.134787

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