Supporting Information for

Electrooxidative synthesis of unknown 1,2,3-triazolone 1-amines

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S1. General Information

All reactions were carried out in well-cleaned oven-dried glassware with magnetic stirring. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AM-300 (300.13 and 75.47 MHz, respectively) spectrometer and referenced to residual solvent peak. The chemical shifts are reported in ppm (δ); multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet) and br (broad). Coupling constants, J, are reported in Hertz. Elemental analyses were performed by the CHN Analyzer Perkin-Elmer 2400. High resolution mass spectra were recorded on a Bruker microTOF spectrometer with electrospray ionization (ESI). All measurements were performed in a positive (+MS) ion mode (interface capillary voltage: 4500 V) with scan range *m/z*: 50-3000. External calibration of the mass spectrometer was performed with Electrospray Calibrant Solution (Fluka). A direct syringe injection was used for all analyzed solutions in MeCN (flow rate: 3 μL min^{-1}). Nitrogen was used as nebulizer gas (0.4 bar) and dry gas (4.0 L min^{-1}); interface temperature was set at 180 °C. All spectra were processed by using Bruker DataAnalysis 4.0 software package. The melting points were determined on Stuart SMP20 apparatus and are uncorrected. Analytical thin-layer chromatography (TLC) was carried out on Merck 25 TLC silica gel 60 F_{254} aluminum sheets. The visualization of the TLC plates was accomplished with a UV light. Column chromatography was performed on silica gel 60 A (0.060 – 0.200 mm, Acros Organics). All solvents were purified and dried using standard methods prior to use. All standard reagents were purchased from Aldrich or Acros Organics and used without further purification. For all electrochemical reactions multi-channel potentiostat-galvanostat “P-20X8” together with a glass 30 mL cell were employed.

S2. Cyclic Voltammetry analysis

Voltammetric studies were carried out using potentiostat P-20X8 with a scan rate of 0.1 V s^{-1} in a temperature-controlled (25 °C) glass cell (V = 10 mL) under an argon atmosphere using 0.1 M solution of $LiClO_4$ in MeCN/MeOH (5/1) as supporting electrolyte. A Pt disk (d = 2 mm) was used as the working electrode (carefully polished by chromium paste and washed before each measurement). A Ag/Ag+ (0.01 M AgCl, MeCN) electrode separated from the solution being studied by a salt bridge filled with the supporting electrolyte was used as the reference electrode. A platinum plate (S = 2 cm^2) was used as the counter electrode. All experiments were performed with the concentration of studied compounds of 1 mM.

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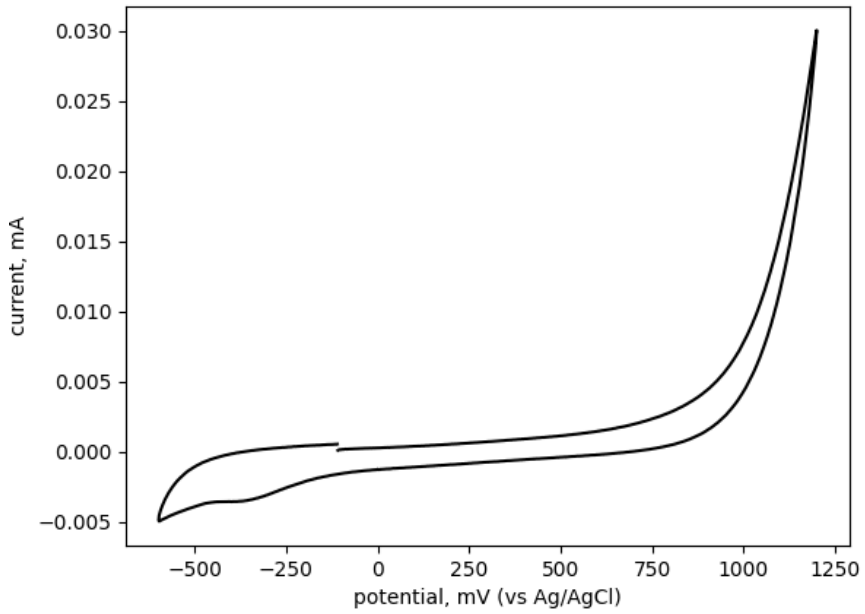
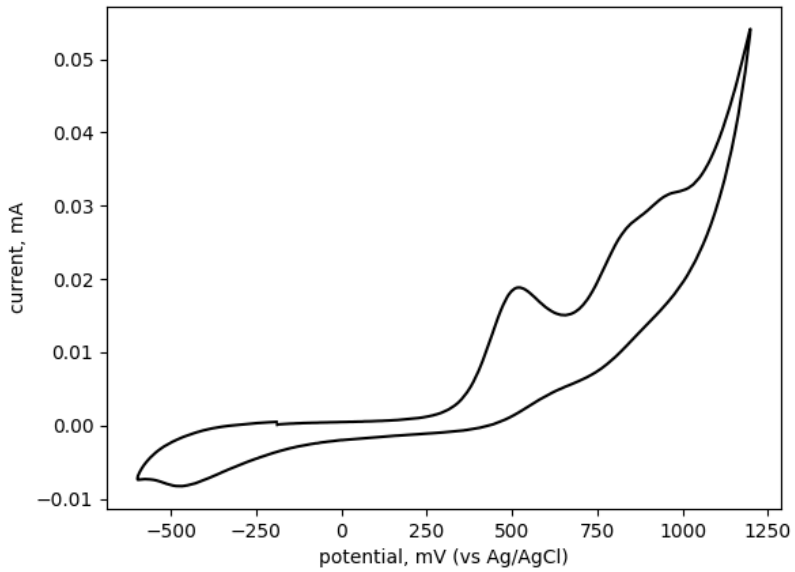


Compound 1a:



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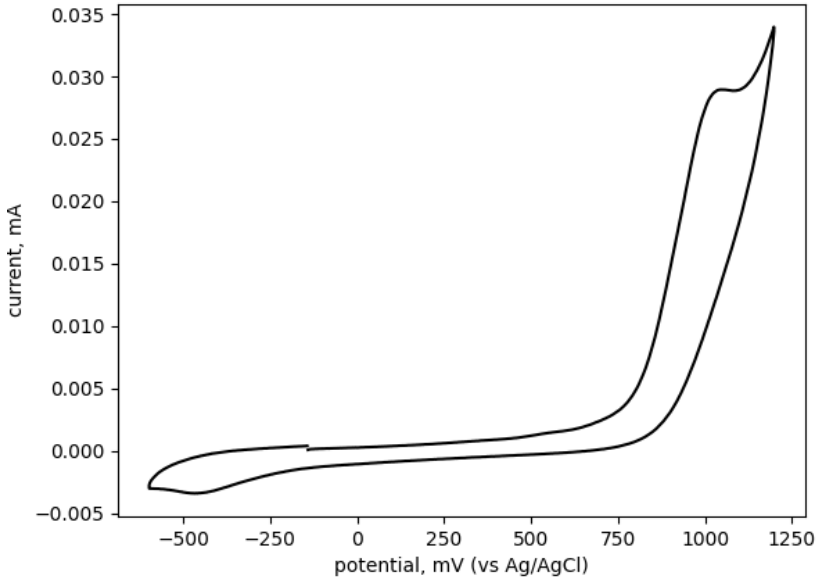
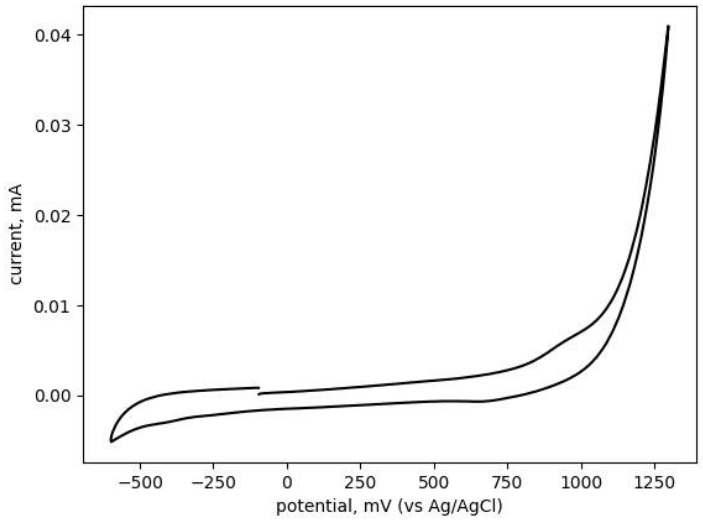
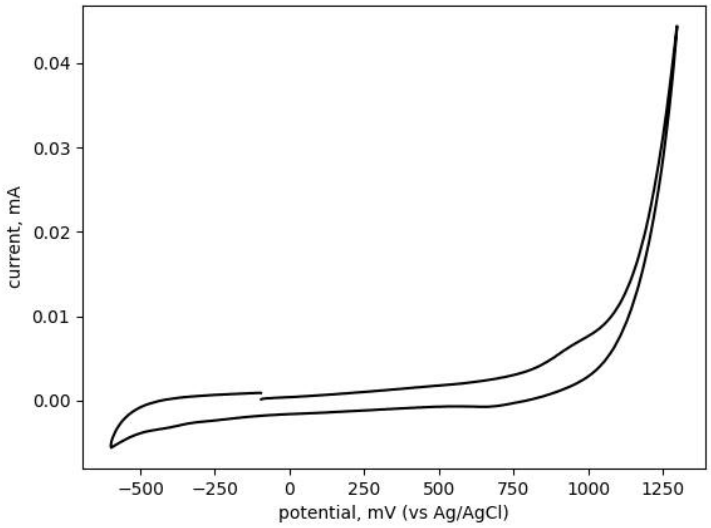


Compound 1p:



Compound 1q:

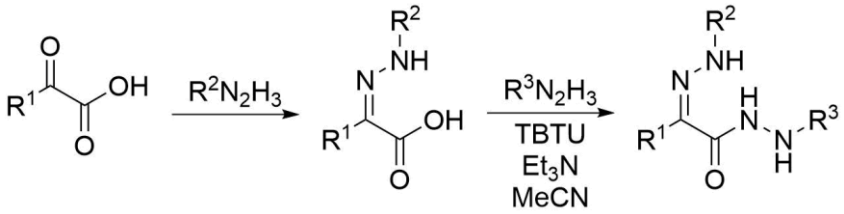


S3. Synthetic procedures and characterization of new compounds

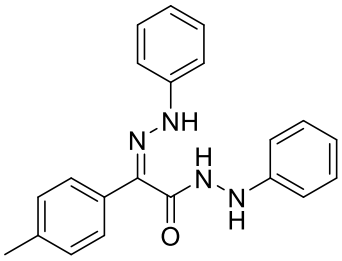
S3.1. General procedure for the synthesis of starting α-aminocarbonyl hydrazones 1

Synthesis of starting α-aminocarbonyl hydrazones **1** was accomplished according to the following scheme:

A corresponding ketoacid (2 mmol) was added to an appropriate hydrazine hydrochloride (2.1 mmol) in water (6 mL) at 20 °C and left stirred overnight. After the reaction was completed (TLC monitoring), the precipitate was filtered off and dried in air. The obtained crude hydrazone was used without further purification for the second step. Appropriate hydrazine (1.1 mmol), TBTU (2-(1*H*-benzotriazol-1-yl)-1,1,3,3-tetramethylaminium tetrafluoroborate) (1.1 mmol, 0.35 g) and Et_3N (1.1 mmol, 150 μL) were added to a magnetically stirred suspension of the corresponding crude hydrazone (1 mmol) in MeCN at 20 °C and left stirred overnight. After the reaction was completed (TLC monitoring, EtOAc/hex 1/1), the reaction mixture was evaporated, the obtained oil was diluted with 0.01M HCl solution and the formed precipitate was filtered off. The crude residue was purified via recrystallization from EtOH to afford pure hydrazones **1a-o.**



N'-phenyl-2-(2-phenylhydrazinylidene)-2-(p-tolyl)acetohydrazide (1a)



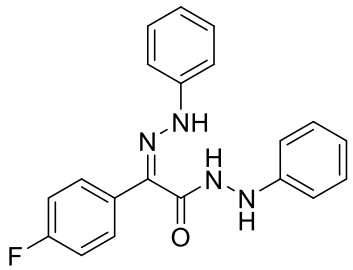
Yield 259 mg (75%). Yellow powder. Mp: 165-167°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.43 (s, 1H), 10.09 (s, 1H), 7.67 – 7.49 (m, 4H), 7.34 – 7.15 (m, 10H), 6.84 (dt, *J* = 13.5, 6.9 Hz, 5H), 2.36 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 163.9, 149.1, 140.3, 139.0, 138.4, 132.5, 129.8, 129.3, 128.9, 127.6, 119.5, 117.6, 114.4, 112.9, 21.4.

HRMS (ESI): m/z calcd for $C_{21}H_{21}N_4O$: 345.1709; found: 345.1704 $[M+H]^+$.

2-(4-fluorophenyl)-N'-phenyl-2-(2-phenylhydrazinylidene)acetohydrazide (1b)



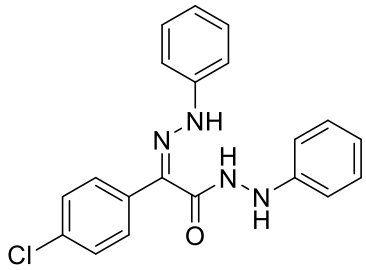
Yield 248 mg (71%). Yellow powder. Mp: 163-165°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.42 (s, 1H), 10.40 (s, 1H), 7.75 – 7.63 (m, 4H), 7.37 – 7.16 (m, 10H), 6.92 – 6.75 (m, 5H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 164.4, 164.2, 160.9, 145.6, 144.8, 129.7, 129.4, 129.3, 129.0, 128.5, 128.4, 120.9, 119.6, 116.0, 113.5, 113.0.

HRMS (ESI): m/z calcd for $C_{20}H_{18}FN_4O$: 349.1459; found: 349.1456 $[M+H]^+$.

2-(4-chlorophenyl)-N'-phenyl-2-(2-phenylhydrazinylidene)acetohydrazide (1c)



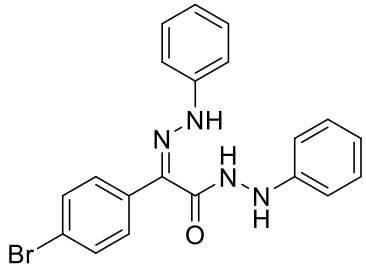
Yield 287 mg (79%). Yellow powder. Mp: 168-171°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.44 (s, 2H), 7.67 (t, *J* = 8.0 Hz, 3H), 7.54 (d, *J* = 8.6 Hz, 2H), 7.33 – 7.16 (m, 7H), 6.83 (dt, *J* = 19.2, 8.3 Hz, 5H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 169.6, 164.2, 144.7, 135.2, 134.6, 133.2, 129.7, 129.4, 129.2, 128.0, 121.0, 119.6, 113.6, 113.1.

HRMS (ESI): m/z calcd for $C_{20}H_{18}ClN_4O$: 365.1163; found: 365.1166 $[M+H]^+$.

2-(4-bromophenyl)-N'-phenyl-2-(2-phenylhydrazinylidene)acetohydrazide (1d)



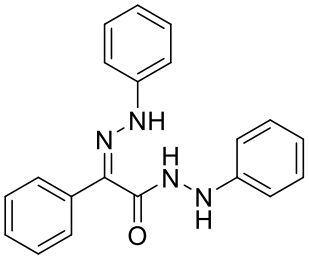
Yield 325 mg (80%). Yellow powder. Mp: 184-185°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.45 (s, 2H), 7.74 – 7.52 (m, 7H), 7.35 – 7.17 (m, 7H), 6.84 (td, *J* = 18.8, 17.5, 6.7 Hz, 5H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 164.2, 149.2, 135.3, 134.9, 132.1, 129.7, 129.4, 128.2, 121.8, 121.0, 119.6, 113.6, 113.0.

HRMS (ESI): m/z calcd for $C_{20}H_{18}BrN_4O$: 409.0658; found: 409.0655 $[M+H]^+$.

N',2-diphenyl-2-(2-phenylhydrazinylidene)acetohydrazide (1e)



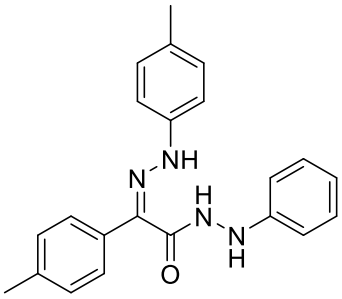
Yield 217 mg (66%). Yellow powder. Mp: 134-136°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.46 (s, 1H), 10.22 (s, 1H), 7.73 – 7.59 (m, 3H), 7.48 (t, J = 7.4 Hz, 2H), 7.40 (d, J = 7.2 Hz, 1H), 7.27 (dq, J = 15.8, 8.5 Hz, 7H), 6.88 (t, J = 7.4 Hz, 3H), 6.80 (t, J = 7.0 Hz, 1H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 164.5, 149.2, 146.8, 139.1, 138.7, 134.3, 132.4, 131.3, 129.8, 129.4, 126.1, 119.9, 119.7, 113.1, 112.7, 112.1, 21.4.

HRMS (ESI): m/z calcd for $C_{20}H_{19}N_4O$: 331.1553; found: 331.1552 $[M+H]^+$.

N'-phenyl-2-(p-tolyl)-2-(2-(p-tolyl)hydrazinylidene)acetohydrazide (1f)



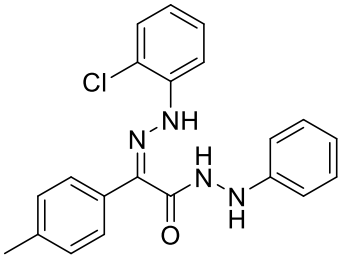
Yield 257 mg (72%). Yellow powder. Mp: 165-167°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.38 (s, 1H), 10.07 (s, 1H), 7.63 (s, 1H), 7.56 (d, *J* = 8.1 Hz, 2H), 7.31 – 7.17 (m, 5H), 7.11 (q, *J* = 8.8 Hz, 4H), 6.81 (dd, *J* = 19.8, 7.6 Hz, 3H), 2.36 (s, 3H), 2.24 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 164.8, 142.8, 138.0, 136.3, 132.9, 130.1, 129.7, 129.4, 129.3, 126.1, 119.6, 113.4, 113.0, 21.3, 20.8.

HRMS (ESI): m/z calcd for $C_{22}H_{23}N_4O$: 359.1866; found: 359.1861 $[M+H]^+$.

2-(2-(2-chlorophenyl)hydrazinylidene)-N'-phenyl-2-(p-tolyl)acetohydrazide (1g)



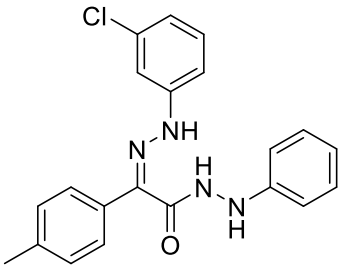
Yield 274 mg (73%). Yellow powder. Mp: 177-178°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.88 (s, 1H), 10.37 (s, 1H), 7.64 (d, *J* = 8.0 Hz, 4H), 7.46 – 7.14 (m, 7H), 6.93 (t, *J* = 8.0 Hz, 1H), 6.79 (dd, *J* = 18.5, 7.5 Hz, 3H), 2.38 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 163.9, 149.1, 140.3, 140.2, 138.9, 138.2, 129.8, 129.3, 128.9, 127.6, 121.8, 119.5, 117.5, 114.4, 112.9.

HRMS (ESI): m/z calcd for $C_{21}H_{20}ClN_4O$: 379.1320; found: 379.1323 $[M+H]^+$.

(Z)-2-(2-(3-chlorophenyl)hydrazinylidene)-N'-phenyl-2-(p-tolyl)acetohydrazide (1h)



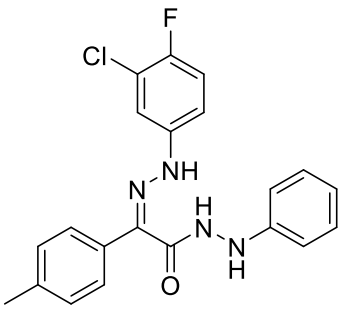
Yield 243 mg (64%). Yellow powder. Mp: 165-167°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.50 (s, 1H), 10.17 (s, 1H), 7.59 (d, *J* = 8.1 Hz, 4H), 7.36 – 7.14 (m, 9H), 6.93 – 6.74 (m, 5H), 2.36 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 164.5, 149.2, 146.8, 139.1, 138.7, 134.3, 132.4, 131.3, 129.4, 119.9, 119.7, 113.7, 113.1, 112.1, 21.4.

HRMS (ESI): m/z calcd for $C_{21}H_{20}ClN_4O$: 379.1320; found: 379.1321 $[M+H]^+$.

2-(2-(3-chloro-4-fluorophenyl)hydrazinylidene)-N'-phenyl-2-(p-tolyl)acetohydrazide (1i)



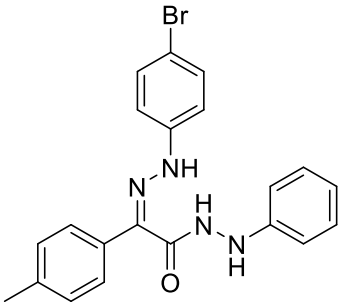
Yield 303 mg (76%). Pink powder. Mp: 190-191°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.49 (s, 1H), 10.17 (s, 1H), 7.59 (d, *J* = 5.4 Hz, 3H), 7.45 – 7.11 (m, 8H), 6.98 – 6.73 (m, 3H), 2.36 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 164.5, 150.3, 149.2, 143.3, 142.7, 139.0, 138.6, 132.4, 129.8, 129.4, 129.0, 119.6, 117.9, 117.6, 114.2, 113.3, 113.1, 21.4.

HRMS (ESI): m/z calcd for $C_{21}H_{18}ClFN_4O$: 395.1069; found: 395.1056 $[M+H]^+$.

2-(2-(4-bromophenyl)hydrazinylidene)-N'-phenyl-2-(p-tolyl)acetohydrazide (1j)



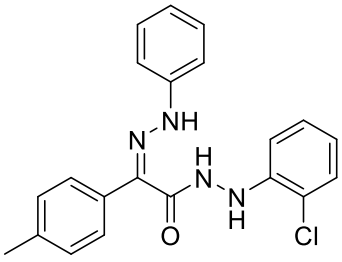
Yield 342 mg (81%). Yellow powder. Mp: 192-193°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.49 (s, 1H), 10.16 (s, 1H), 7.61 – 7.55 (m, 3H), 7.44 (d, *J* = 8.9 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 7.22 (dt, *J* = 7.2, 3.3 Hz, 4H), 6.83 (dd, *J* = 13.6, 7.5 Hz, 3H), 2.36 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 164.6, 149.2, 144.6, 138.4, 129.8, 119.6, 115.3, 113.1, 111.5, 21.4.

Calcd for $C_{21}H_{19}BrN_4O$ (%): C, 59.58; H, 4.52; N, 13.24. Found (%): C, 59.71; H, 4.47; N, 13.12.

N'-(2-chlorophenyl)-2-(2-phenylhydrazinylidene)-2-(p-tolyl)acetohydrazide (1k)



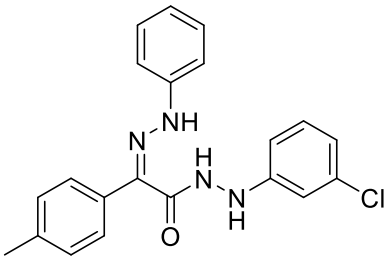
Yield 226 mg (60%). Yellow powder. Mp: 156-157°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.84 (s, 1H), 10.35 (s, 1H), 7.64 (d, *J* = 8.2 Hz, 3H), 7.42 (d, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 8.2 Hz, 2H), 7.20 (t, *J* = 7.9 Hz, 2H), 6.93 (t, *J* = 7.7 Hz, 1H), 6.84 – 6.72 (m, 3H), 2.38 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 164.5, 149.2, 146.7, 139.1, 138.7, 134.3, 132.4, 131.3, 129.8, 129.4, 119.9, 119.7, 113.7, 113.3, 113.1, 112.8, 112.1, 21.4.

HRMS (ESI): m/z calcd for $C_{21}H_{20}ClN_4O$: 379.1320; found: 379.1324 $[M+H]^+$.

N'-(3-chlorophenyl)-2-(2-phenylhydrazinylidene)-2-(p-tolyl)acetohydrazide (1l)



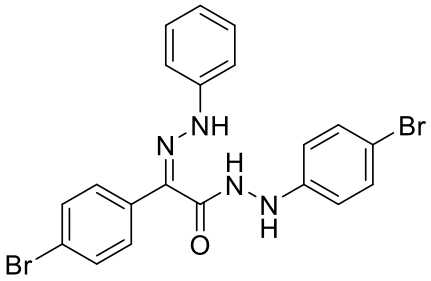
Yield 210 mg (56%). Yellow powder. Mp: 160-163°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.51 (s, 1H), 10.17 (s, 1H), 7.59 (d, *J* = 8.3 Hz, 3H), 7.31 – 7.19 (m, 7H), 6.86 (t, *J* = 7.8 Hz, 3H), 2.36 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 163.9, 149.1, 140.3, 139.0, 138.4, 132.5, 129.8, 129.3, 128.9, 127.6, 119.5, 117.6, 114.4, 112.9, 21.4.

HRMS (ESI): m/z calcd for $C_{21}H_{20}ClN_4O$: 379.1320; found: 379.1322 $[M+H]^+$.

N',2-bis(4-bromophenyl)-2-(2-phenylhydrazinylidene)acetohydrazide (1m)



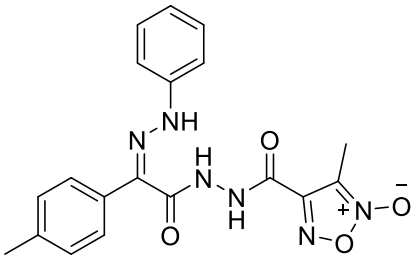
Yield 276 mg (57%). Yellow powder. Mp: 176-177°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.54 (s, 1H), 10.45 (s, 1H), 7.70 – 7.57 (m, 4H), 7.37 (d, *J* = 8.8 Hz, 2H), 7.27 (q, *J* = 6.3 Hz, 5H), 6.80 (d, *J* = 8.8 Hz, 2H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 164.1, 148.6, 144.7, 134.9, 132.0, 129.7, 121.8, 121.1, 115.1, 113.7, 110.4.

Calcd for $C_{20}H_{16}Br_2N_4O$ (%): C, 49.21; H, 3.30; N, 11.48. Found (%): C, 49.33; H, 3.19; N, 11.58.

3-methyl-4-(2-(2-(2-phenylhydrazineylidene)-2-(p-tolyl)acetyl)hydrazine-1-carbonyl)-1,2,5-oxadiazole 2-oxide (1n)



Yield 306 mg (78%). Yellow powder. Mp: 202-203°C (EtOH)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 11.44 (s, 1H), 11.10 (s, 1H), 9.99 (s, 1H), 7.61 (d, *J* = 7.8 Hz, 2H), 7.28 (dq, *J* = 14.9, 8.0 Hz, 6H), 6.88 (t, *J* = 6.8 Hz, 1H), 2.36 (s, 6H).

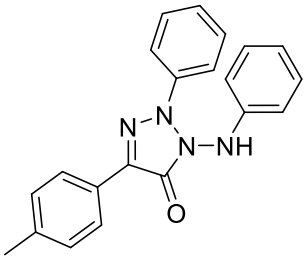
^{13}C NMR (76 MHz, DMSO-d_6): δ = 163.6, 157.7, 144.8, 138.5, 137.3, 131.8, 129.8, 113.5, 113.0, 21.4, 8.9.

HRMS (ESI): m/z calcd for $C_{19}H_{19}N_6O_4$: 395.1462; found: 395.1456 $[M+H]^+$.

S3.2. General procedure for the synthesis of 1,2,3-triazone-1-amines 2

The electrolysis was performed in an undivided cell (20 mL glass vial) with graphite plate employed as anode and nickel plate employed as cathode. Corresponding hydrazone (0.3 mmol) was dissolved in a solution of $LiClO_4$ (0.1 M) in the mixture of MeCN/MeOH (15 mL, 5/1) and oxidized under constant current conditions (j = 2.08 mA cm^{-2}) for 2.1 F/mol (TLC monitoring). After that, the reaction mixture was concentrated under reduced pressure, the crude residue was diluted with water and the formed precipitate was filtered off. The obtained product was recrystallized from EtOAc/hexanes to afford pure 1,2,3-triazolone 1-amines.

2-phenyl-3-(phenylamino)-5-(p-tolyl)-2,3-dihydro-4H-1,2,3-triazol-4-one (2a)



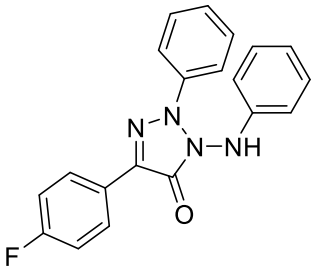
Yield 87 mg (85%). White powder. Mp: 123-124°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 9.62 (s, 1H), 8.13 (d, *J* = 7.3 Hz, 2H), 7.65 – 7.13 (m, 9H), 6.86 (t, *J* = 7.3 Hz, 1H), 6.58 (d, *J* = 7.9 Hz, 2H), 2.38 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 157.8, 140.2, 138.8, 138.5, 131.6, 130.3, 130.0, 129.9, 129.4, 129.3, 128.6, 126.0, 125.0, 123.6, 21.1.

HRMS (ESI): m/z calcd for $C_{21}H_{19}N_4O$: 343.1553; found: 343.1544 $[M+H]^+$.

5-(4-fluorophenyl)-2-phenyl-3-(phenylamino)-2,3-dihydro-4H-1,2,3-triazol-4-one (2b)



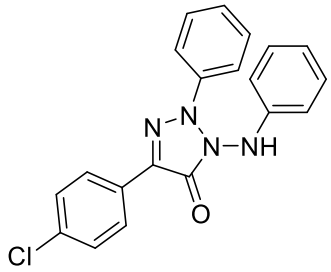
Yield 91 mg (87%). White powder. Mp: 134-135°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 9.66 (s, 1H), 8.47 – 8.23 (m, 2H), 7.99 – 7.71 (m, 2H), 7.63 – 7.36 (m, 5H), 7.25 – 7.18 (m, 1H), 6.95 – 6.79 (m, 1H), 6.64 – 6.56 (m, 1H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 155.8, 145.3, 137.7, 136.3, 131.5, 131.2, 129.9, 129.8, 129.0, 127.7, 126.7, 126.6, 123.9, 121.2, 116.5, 116.2, 112.7.

HRMS (ESI): m/z calcd for $C_{20}H_{16}FN_4O$: 347.1302; found: 347.1295 $[M+H]^+$.

5-(4-chlorophenyl)-2-phenyl-3-(phenylamino)-2,3-dihydro-4H-1,2,3-triazol-4-one (2c)



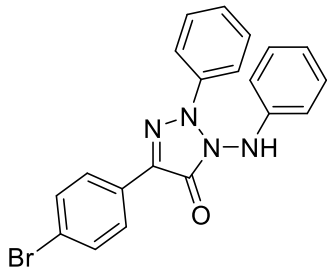
Yield 97 mg (89%). White powder. Mp: 121-122°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 9.67 (s, 1H), 8.24 (d, *J* = 8.4 Hz, 2H), 7.56 (dd, *J* = 20.9, 6.0 Hz, 7H), 7.23 (d, *J* = 7.7 Hz, 2H), 6.86 (t, *J* = 6.5 Hz, 1H), 6.60 (d, *J* = 7.9 Hz, 2H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 157.8, 139.9, 134.4, 134.0, 130.0, 129.9, 129.5, 129.0, 128.8, 128.3, 125.2, 123.7.

HRMS (ESI): m/z calcd for $C_{20}H_{16}ClN_4O$: 363.1007; found: 363.1003 $[M+H]^+$.

5-(4-bromophenyl)-2-phenyl-3-(phenylamino)-2,3-dihydro-4H-1,2,3-triazol-4-one (2d)



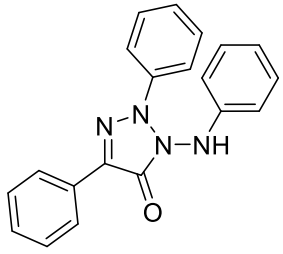
Yield 110 mg (90%). White powder. Mp: 120-121°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 9.83 (s, 1H), 8.43 (d, *J* = 5.5 Hz, 1H), 8.16 (d, *J* = 8.6 Hz, 2H), 8.04 (d, *J* = 8.9 Hz, 1H), 7.92 (t, *J* = 8.1 Hz, 2H), 7.75 (t, *J* = 8.6 Hz, 3H), 7.64 (d, *J* = 8.6 Hz, 1H), 7.52 (d, *J* = 2.9 Hz, 4H), 7.38 (d, *J* = 8.8 Hz, 2H), 6.60 (d, *J* = 8.8 Hz, 2H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 157.6, 138.3, 135.2, 132.8, 132.4, 132.0, 131.8, 131.1, 131.1, 130.0, 129.4, 129.0, 128.8, 127.5, 124.1, 122.8.

HRMS (ESI): m/z calcd for $C_{20}H_{16}BrN_4O$: 407.0502; found: 407.0495 $[M+H]^+$.

2,5-diphenyl-3-(phenylamino)-2,3-dihydro-4H-1,2,3-triazol-4-one (2e)



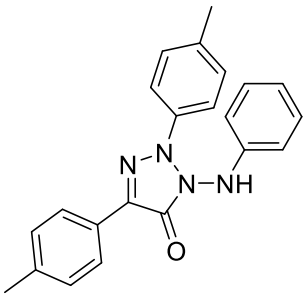
Yield 72 mg (73%). Beige powder. Mp: 110-111°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 9.65 (s, 1H), 8.42 (d, *J* = 7.6 Hz, 1H), 8.23 (d, *J* = 7.3 Hz, 2H), 7.60 – 7.38 (m, 12H), 7.21 (t, *J* = 7.4 Hz, 2H), 7.06 (t, *J* = 7.1 Hz, 1H), 6.86 (t, *J* = 7.4 Hz, 1H), 6.59 (d, *J* = 7.6 Hz, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 157.5, 139.9, 137.5, 133.9, 132.4, 130.0, 128.9, 128.8, 128.6, 127.8, 125.2, 123.7, 123.2.

HRMS (ESI): m/z calcd for $C_{20}H_{17}N_4O$: 329.1396; found: 329.1391 $[M+H]^+$.

3-(phenylamino)-2,5-di-p-tolyl-2,3-dihydro-4H-1,2,3-triazol-4-one (2f)



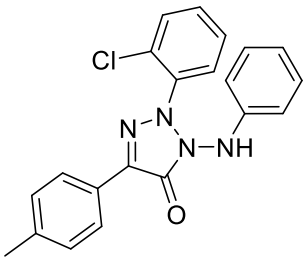
Yield 90 mg (84%). Beige powder. Mp: 118-119°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 9.57 (s, 1H), 8.31 (d, *J* = 8.3 Hz, 1H), 8.11 (d, *J* = 8.1 Hz, 2H), 7.86 – 7.77 (m, 2H), 7.72 – 7.41 (m, 3H), 7.35 (dd, *J* = 12.0, 8.1 Hz, 7H), 7.21 (t, *J* = 7.8 Hz, 3H), 6.85 (t, *J* = 7.9 Hz, 1H), 6.57 (d, *J* = 7.7 Hz, 2H), 2.37 (s, 3H), 2.33 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 158.9, 155.7, 145.4, 145.0, 139.0, 138.8, 137.2, 135.5, 132.8, 132.5, 131.8, 131.1, 130.7, 130.3, 129.9, 129.8, 127.4, 126.7, 125.3, 124.1, 121.6, 117.1, 112.6, 21.5, 21.1.

Calcd for $C_{22}H_{20}N_4O$ (%): C, 74.14; H, 5.66; N, 15.72. Found (%): C, 74.21; H, 5.72; N, 15.79.

2-(2-chlorophenyl)-3-(phenylamino)-5-(p-tolyl)-2,3-dihydro-4H-1,2,3-triazol-4-one (2g)



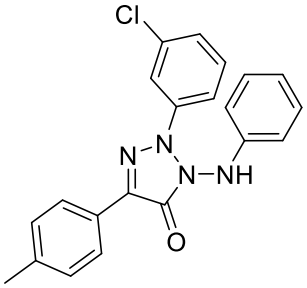
Yield 98 mg (87%). Beige powder. Mp: 140-142°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 9.47 (s, 1H), 8.11 (d, *J* = 7.7 Hz, 2H), 7.68 (d, *J* = 8.4 Hz, 1H), 7.60 – 7.46 (m, 3H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.23 – 7.14 (m, 2H), 6.89 – 6.79 (m, 1H), 6.61 (d, *J* = 6.8 Hz, 2H), 2.38 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 145.5, 139.8, 139.6, 135.1, 132.6, 132.4, 131.1, 130.3, 129.8, 129.6, 128.7, 127.1, 125.5, 121.2, 112.9, 21.1.

HRMS (ESI): m/z calcd for $C_{21}H_{18}ClN_4O$: 377.1163; found: 377.1158 $[M+H]^+$.

2-(3-chlorophenyl)-3-(phenylamino)-5-(p-tolyl)-2,3-dihydro-4H-1,2,3-triazol-4-one (2h)



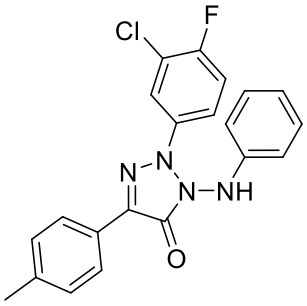
Yield 93 mg (82%). White powder. Mp: 120-121°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 9.65 (s, 1H), 8.13 (d, J = 8.1 Hz, 2H), 7.61 (s, 1H), 7.51 (d, *J* = 9.0 Hz, 4H), 7.35 (d, *J* = 8.2 Hz, 2H), 7.22 (t, *J* = 7.8 Hz, 2H), 6.87 (t, *J* = 7.3 Hz, 1H), 6.60 (d, *J* = 7.7 Hz, 2H), 2.38 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 157.5, 139.8, 138.7, 137.2, 134.3, 131.4, 130.3, 130.0, 129.4, 128.7, 128.3, 127.6, 125.3, 123.7, 21.1.

HRMS (ESI): m/z calcd for $C_{21}H_{18}ClN_4O$: 377.1163; found: 377.1161 $[M+H]^+$.

2-(3-chloro-4-fluorophenyl)-3-(phenylamino)-5-(p-tolyl)-2,3-dihydro-4H-1,2,3-triazol-4-one (2i)



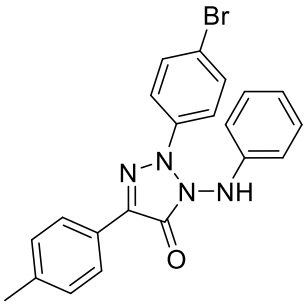
Yield 99 mg (84%). Beige powder. Mp: 127-130°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 9.58 (s, 1H), 8.13 (d, J = 7.8 Hz, 2H), 7.86 – 7.74 (m, 3H), 7.63 – 7.45 (m, 4H), 7.35 (d, *J* = 7.9 Hz, 2H), 7.22 (t, *J* = 7.3 Hz, 2H), 6.92 – 6.82 (m, 1H), 6.61 (d, *J* = 8.7 Hz, 2H), 2.38 (s, 3H)

^{13}C NMR (76 MHz, DMSO-d_6): δ = 156.1, 145.2, 139.7, 139.4, 135.3, 131.2, 130.7, 130.0, 129.8, 126.8, 126.6, 126.3, 125.7, 125.0, 124.9, 118.3, 118.0, 112.8, 21.5.

Calcd for $C_{21}H_{16}ClFN_4O$ (%): C, 63.88; H, 4.08; N, 14.19. Found (%): C, 63.73; H, 4.01; N, 14.26.

2-(4-bromophenyl)-3-(phenylamino)-5-(p-tolyl)-2,3-dihydro-4H-1,2,3-triazol-4-one (2j)



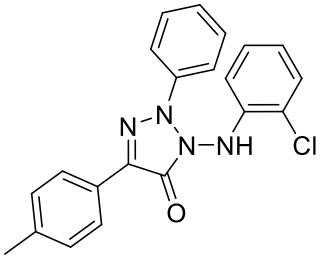
Yield 100 mg (79%). Beige powder. Mp: 123-125°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 8.37 (d, *J* = 7.5 Hz, 2H), 8.11 (d, *J* = 9.0 Hz, 2H), 7.81 (s, 5H), 7.57 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 8.1 Hz, 2H), 2.41 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 157.5, 138.8, 137.3, 134.3, 130.3, 130.0, 129.4, 128.8, 128.3, 125.3, 123.8, 21.1.

Calcd for $C_{21}H_{17}BrN_4O$ (%): C, 59.87; H, 4.07; N, 13.30. Found (%): C, 59.95; H, 4.13; N, 13.41.

3-((2-chlorophenyl)amino)-2-phenyl-5-(p-tolyl)-2,3-dihydro-4H-1,2,3-triazol-4-one (2k)



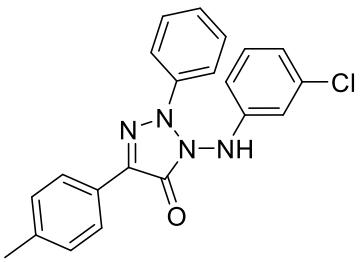
Yield 86 mg (76%). Beige powder. Mp: 131-132°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 9.48 (s, 1H), 8.11 (d, *J* = 8.1 Hz, 2H), 7.67 (d, *J* = 8.8 Hz, 1H), 7.59 – 7.46 (m, 3H), 7.34 (d, *J* = 8.1 Hz, 2H), 7.19 (t, *J* = 7.9 Hz, 2H), 6.84 (t, *J* = 7.3 Hz, 1H), 6.61 (d, *J* = 7.7 Hz, 2H), 2.37 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 155.3, 145.5, 139.8, 139.6, 135.1, 132.6, 132.4, 130.7, 130.3, 129.8, 129.6, 128.7, 125.5, 121.2, 112.9, 21.5.

HRMS (ESI): m/z calcd for $C_{21}H_{18}ClN_4O$: 377.1163; found: 377.1160 $[M+H]^+$.

3-((3-chlorophenyl)amino)-2-phenyl-5-(p-tolyl)-2,3-dihydro-4H-1,2,3-triazol-4-one (2l)



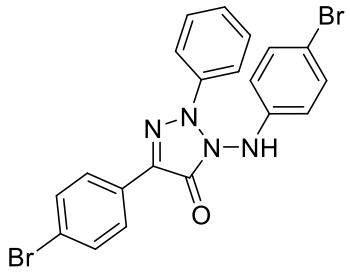
Yield 85 mg (75%). Beige powder. Mp: 122-123°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = δ 9.65 (s, 1H), 8.13 (d, *J* = 7.8 Hz, 2H), 7.82 (s, 1H), 7.61 (s, 1H), 7.56 – 7.46 (m, 3H), 7.35 (d, *J* = 7.6 Hz, 2H), 7.22 (t, *J* = 7.6 Hz, 2H), 6.87 (t, J = 5.5 Hz, 1H), 6.60 (d, J = 7.8 Hz, 2H), 2.38 (s, 3H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 189.3, 158.1, 145.2, 140.7, 139.7, 134.9, 134.5, 130.2, 129.8, 129.3, 126.8, 126.2, 122.7, 121.8, 119.9, 119.3, 116.5, 114.9, 114.3, 89.2, 21.4.

HRMS (ESI): m/z calcd for $C_{21}H_{18}ClN_4O$: 377.1163; found: 377.1161 $[M+H]^+$.

5-(4-bromophenyl)-3-((4-bromophenyl)amino)-2-phenyl-2,3-dihydro-4H-1,2,3-triazol-4-one (2m)



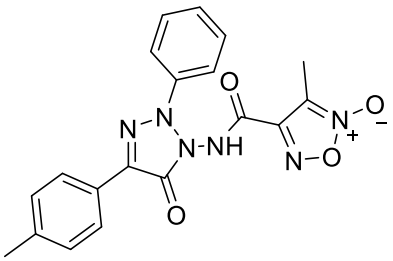
Yield 277 mg (68%). Beige powder. Mp: 132-133°C (EtOAc)

^{1}H NMR (300 MHz, DMSO-d_6): δ = 9.83 (s, 1H), 8.43 (d, *J* = 8.6 Hz, 1H), 8.16 (d, *J* = 8.6 Hz, 2H), 8.04 (d, *J* = 8.9 Hz, 1H), 7.92 (t, *J* = 8.0 Hz, 2H), 7.75 (t, *J* = 8.7 Hz, 3H), 7.64 (d, *J* = 8.6 Hz, 1H), 7.55 – 7.49 (m, 4H), 7.38 (d, *J* = 8.8 Hz, 2H), 6.60 (d, *J* = 8.8 Hz, 2H).

^{13}C NMR (76 MHz, DMSO-d_6): δ = 158.4, 155.5, 144.6, 137.4, 135.7, 134.4, 133.5, 132.5, 132.3, 132.1, 131.3, 130.7, 130.0, 129.3, 128.7, 128.5, 127.4, 127.2, 124.1, 123.1, 122.7, 121.9, 114.9, 112.6.

Calcd for $C_{21}H_{16}BrN_3O$ (%): C, 49.41; H, 2.90; N, 11.52. Found (%): C, 49.53; H, 2.98; N, 11.64.

3-methyl-4-((5-oxo-2-phenyl-4-(p-tolyl)-2,5-dihydro-1H-1,2,3-triazol-1-yl)carbamoyl)-1,2,5-oxadiazole 2-oxide (2n)



Yield 104 mg (88%). Beige powder. Mp: 100-102°C (EtOAc)

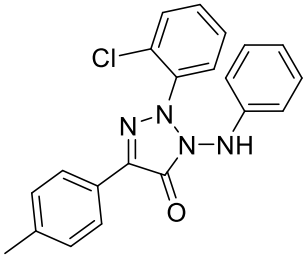
^{1}H NMR (300 MHz, DMSO-d_6): δ = 10.53 (s, 1H), 7.93 – 7.82 (m, 2H), 7.64 – 7.56 (m, 3H), 7.52 (d, *J* = 8.1 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 2.32 (s, 3H), 2.26 (s, 3H).

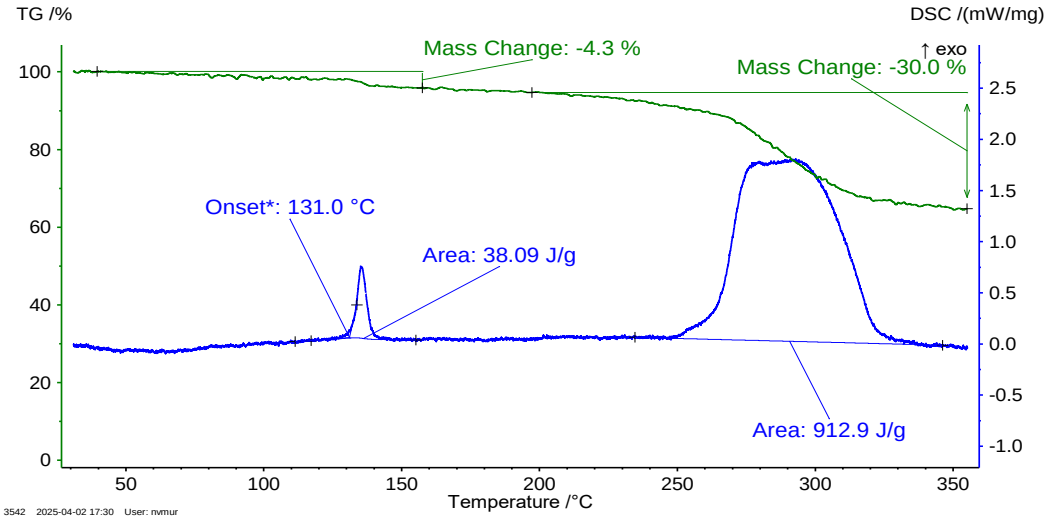
^{13}C NMR (76 MHz, DMSO-d_6): δ = 167.4, 156.6, 151.5, 138.5, 135.0, 132.4, 129.8, 129.2, 127.6, 123.3, 113.2, 101.3, 53.7, 23.5, 21.2, 19.7, 14.0, 8.7.

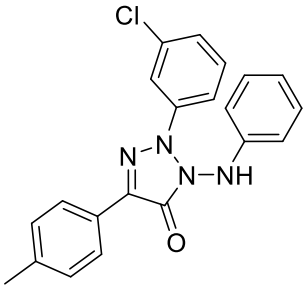
HRMS (ESI): m/z calcd for $C_{19}H_{17}N_6O_4$: 393.1305; found: 393.1301 $[M+H]^+$.

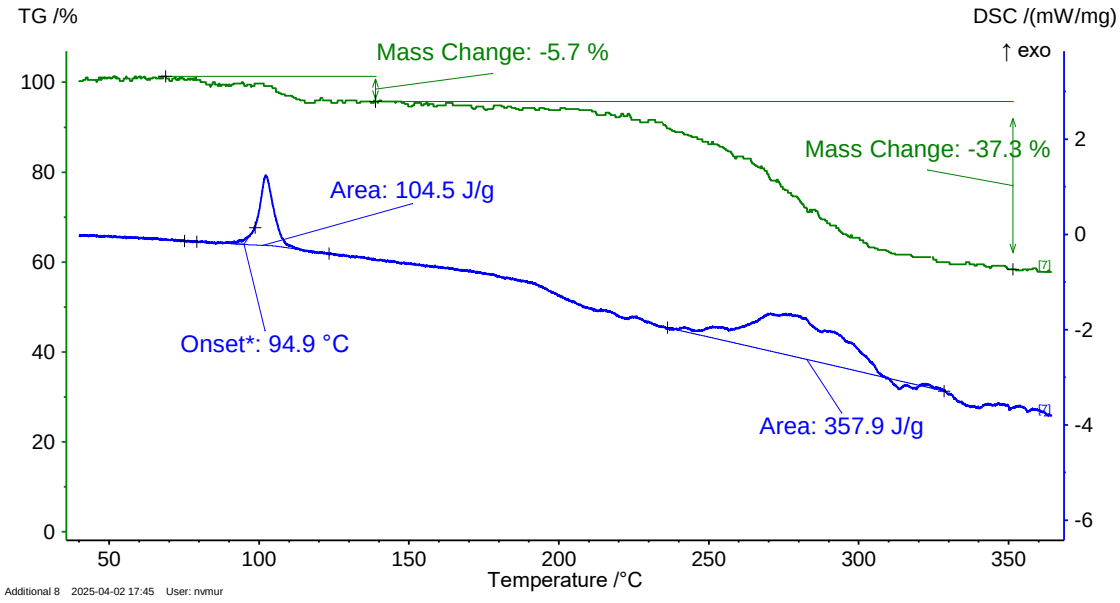
Scale-up synthesis (1 mmol) of 2a: The electrolysis was performed in an undivided cell (50 mL glass vial) equipped with a magnetic stirrer, graphite plate as anode and nickel plate as cathode. N'-phenyl-2-(2-phenylhydrazinylidene)-2-(*p*-tolyl)acetohydrazide **1a** (1 mmol, 344 mg) was dissolved in a solution of $LiClO_4$ (0.1 M) in the mixture of MeCN/MeOH (40 mL, 5/1) and oxidized under constant current conditions (j = 2.08 mA cm^{-2}) for 2.1 F/mol (TLC monitoring). After that, the reaction mixture was concentrated under reduced pressure, the crude residue was diluted with water and the formed precipitate was filtered off. The obtained product was recrystallized from EtOAc/hexanes to afford pure product **2a** (280 mg, 82%).

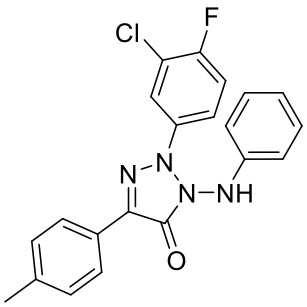
S4. DSC data

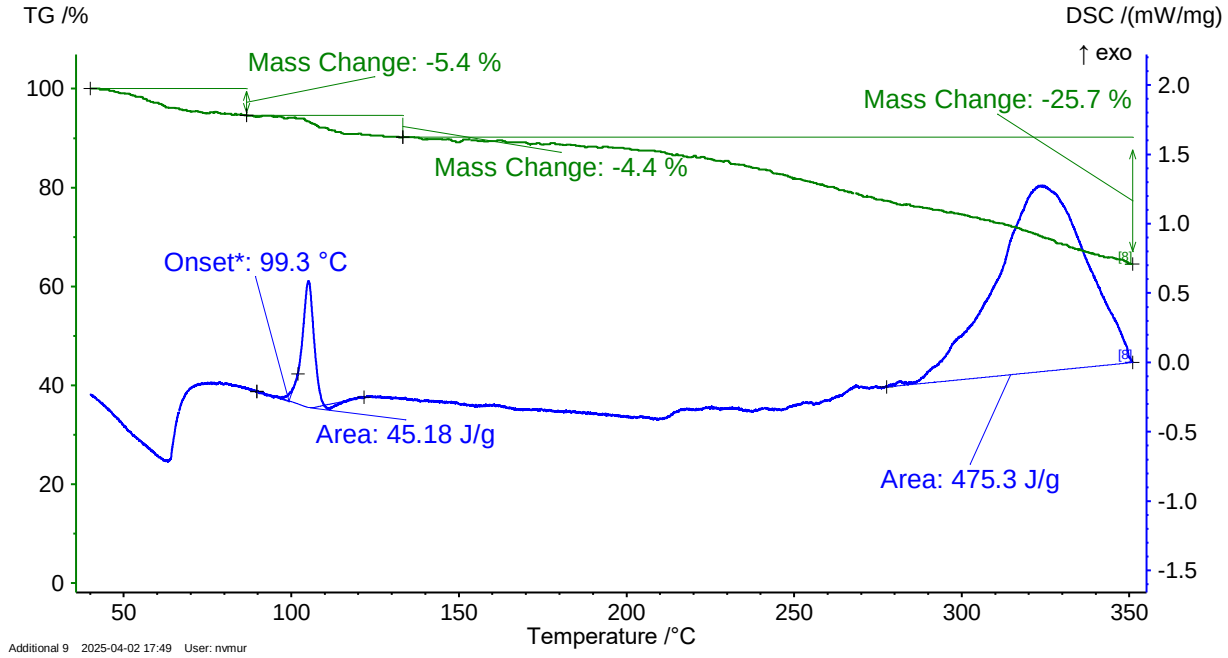

Cl
N
N
N
NH
O


TG /%
DSC /(mW/mg)
↑ exo
Mass Change: -4.3 %
Mass Change: -30.0 %
Onset*: 131.0 °C
Area: 38.09 J/g
Area: 912.9 J/g
100
80
60
40
20
0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
50
100
150
200
250
300
350
Temperature /°C
3542 2025-04-02 17:30 User: nvmur

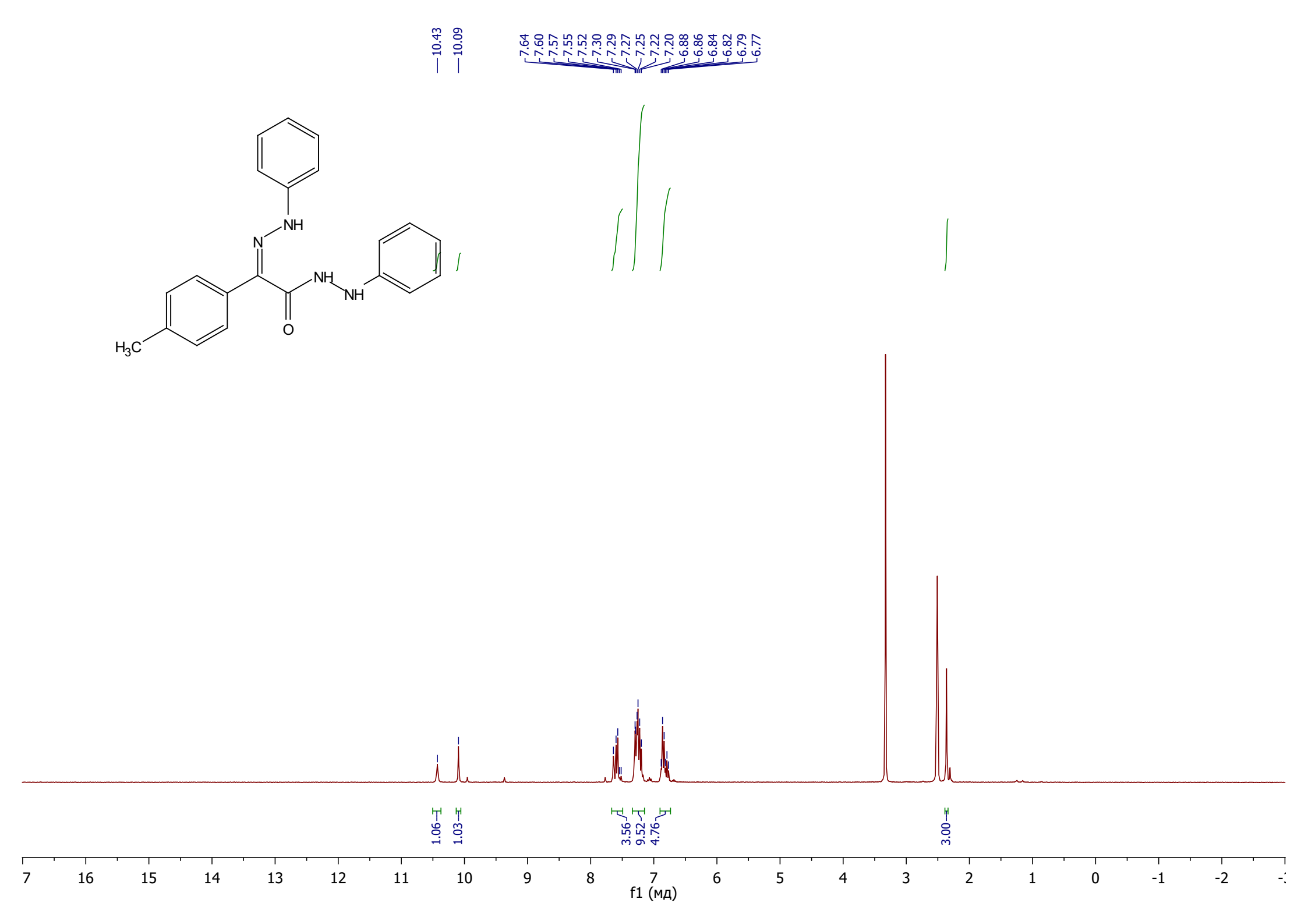

Cl
N
N
N
NH
O


TG /%
DSC /(mW/mg)
↑ exo
Mass Change: -5.7 %
Mass Change: -37.3 %
Area: 104.5 J/g
Onset*: 94.9 °C
Area: 357.9 J/g
100
80
60
40
20
0
2
0
-2
-4
-6
50
100
150
200
250
300
350
Temperature /°C
Additional 8 2025-04-02 17:45 User: nvmur





S5. Copies of NMR spectra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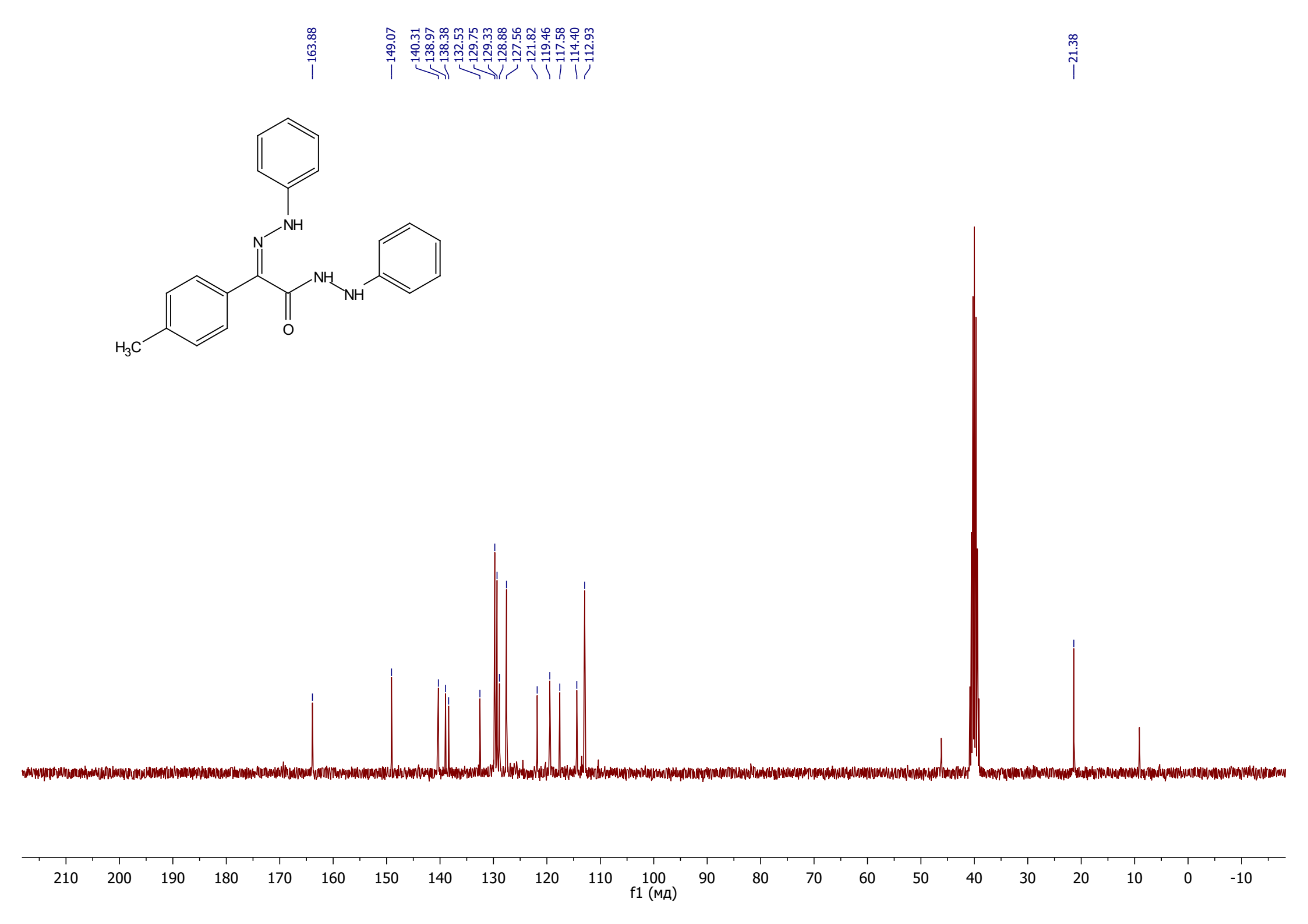
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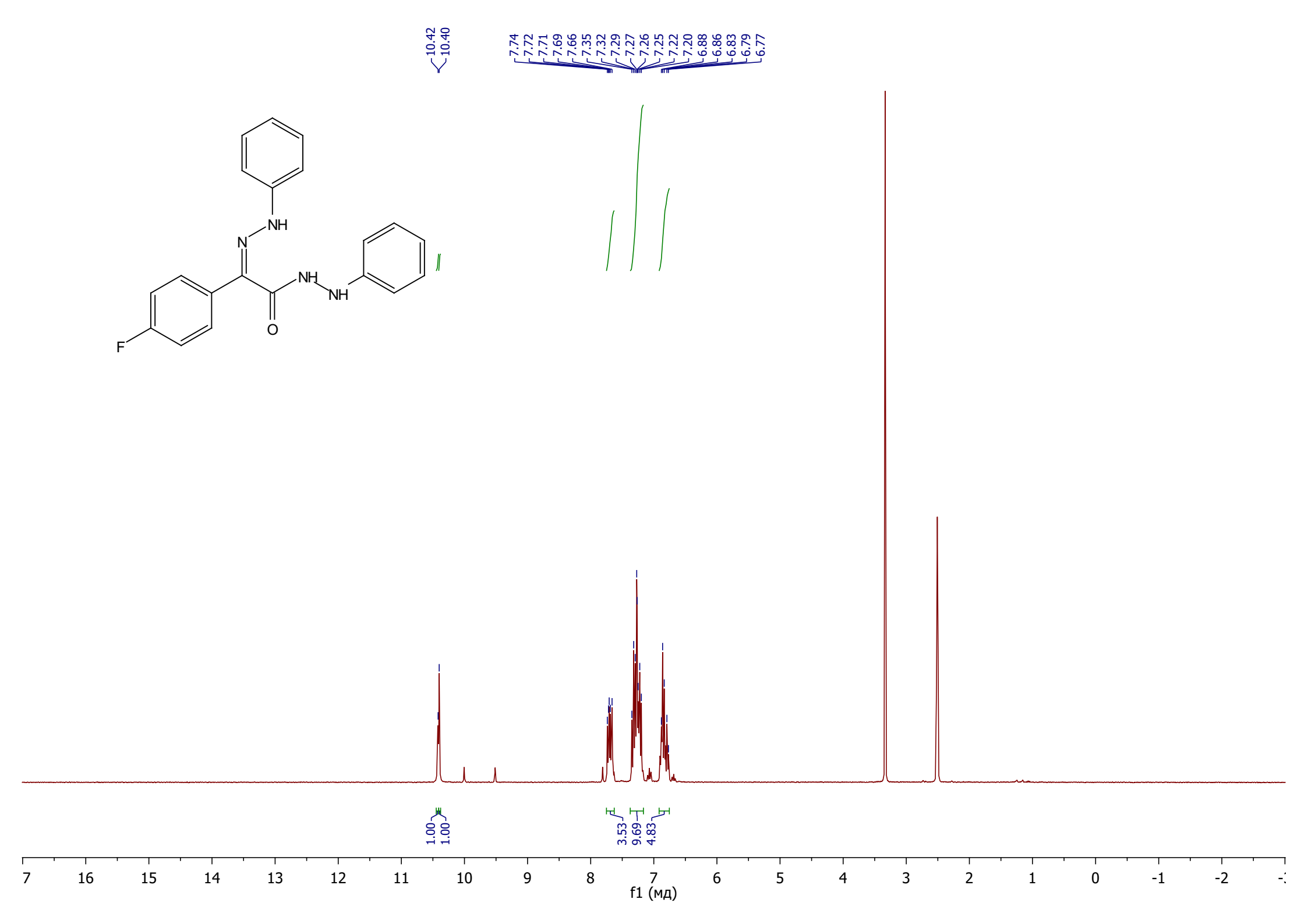
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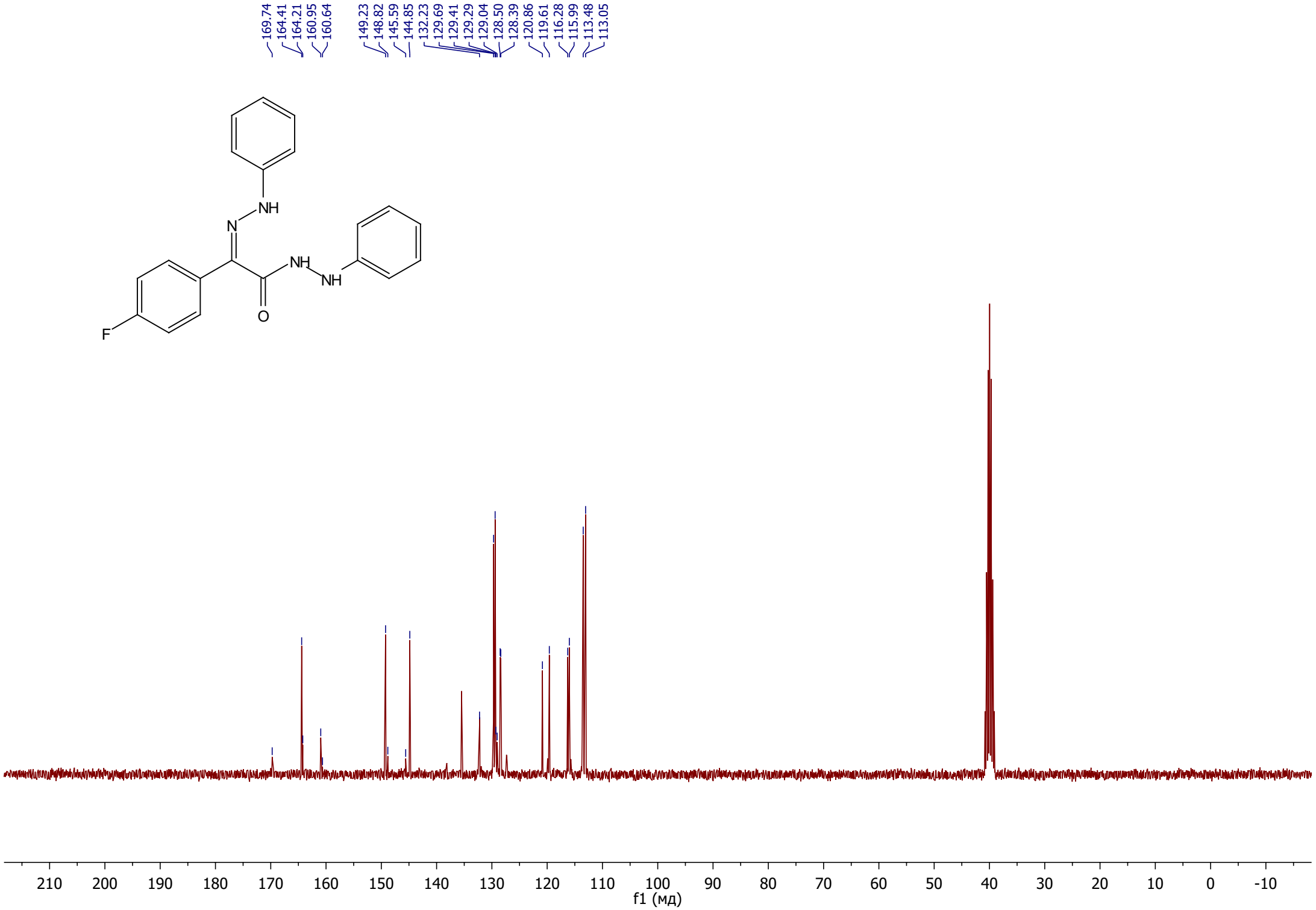
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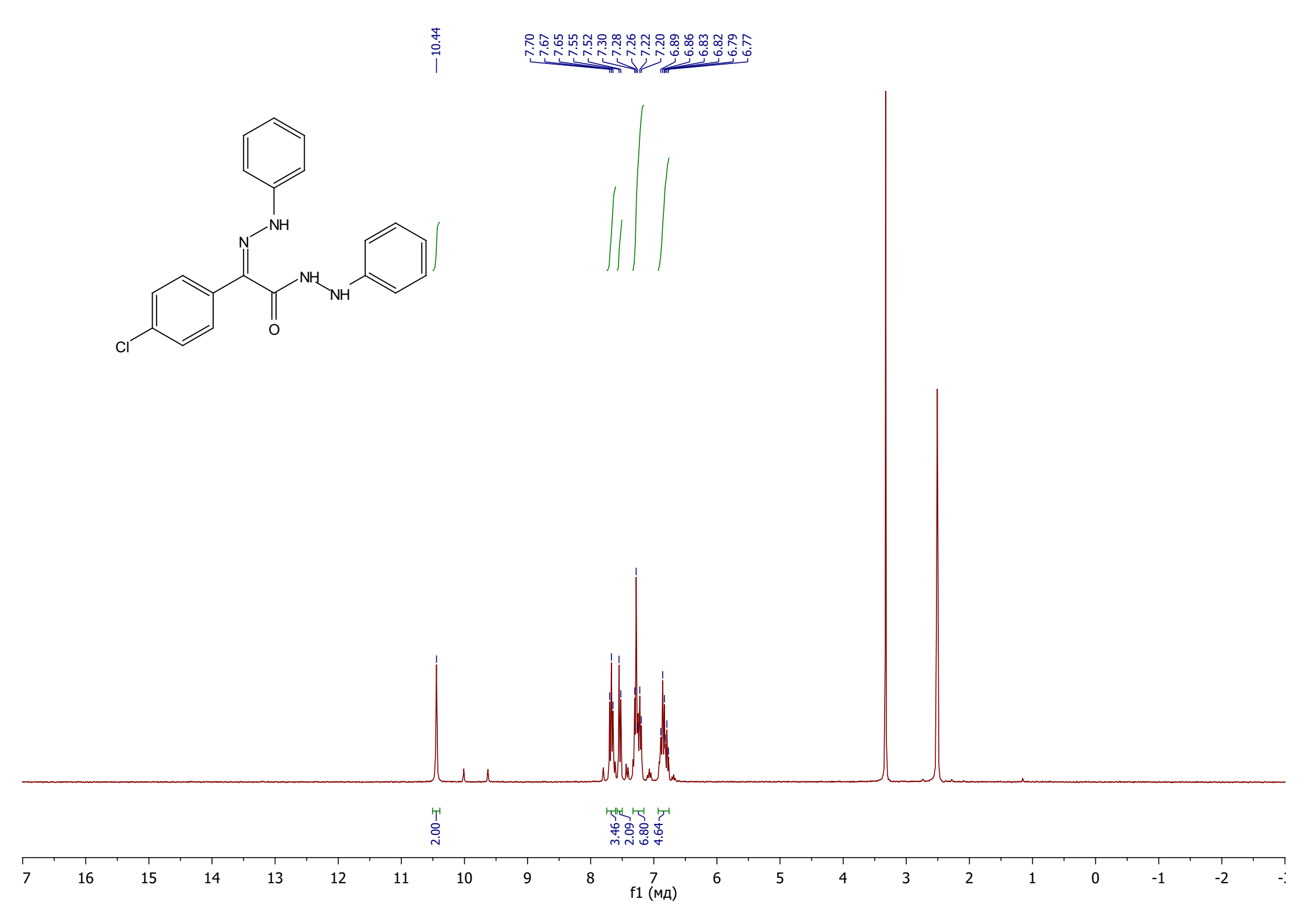
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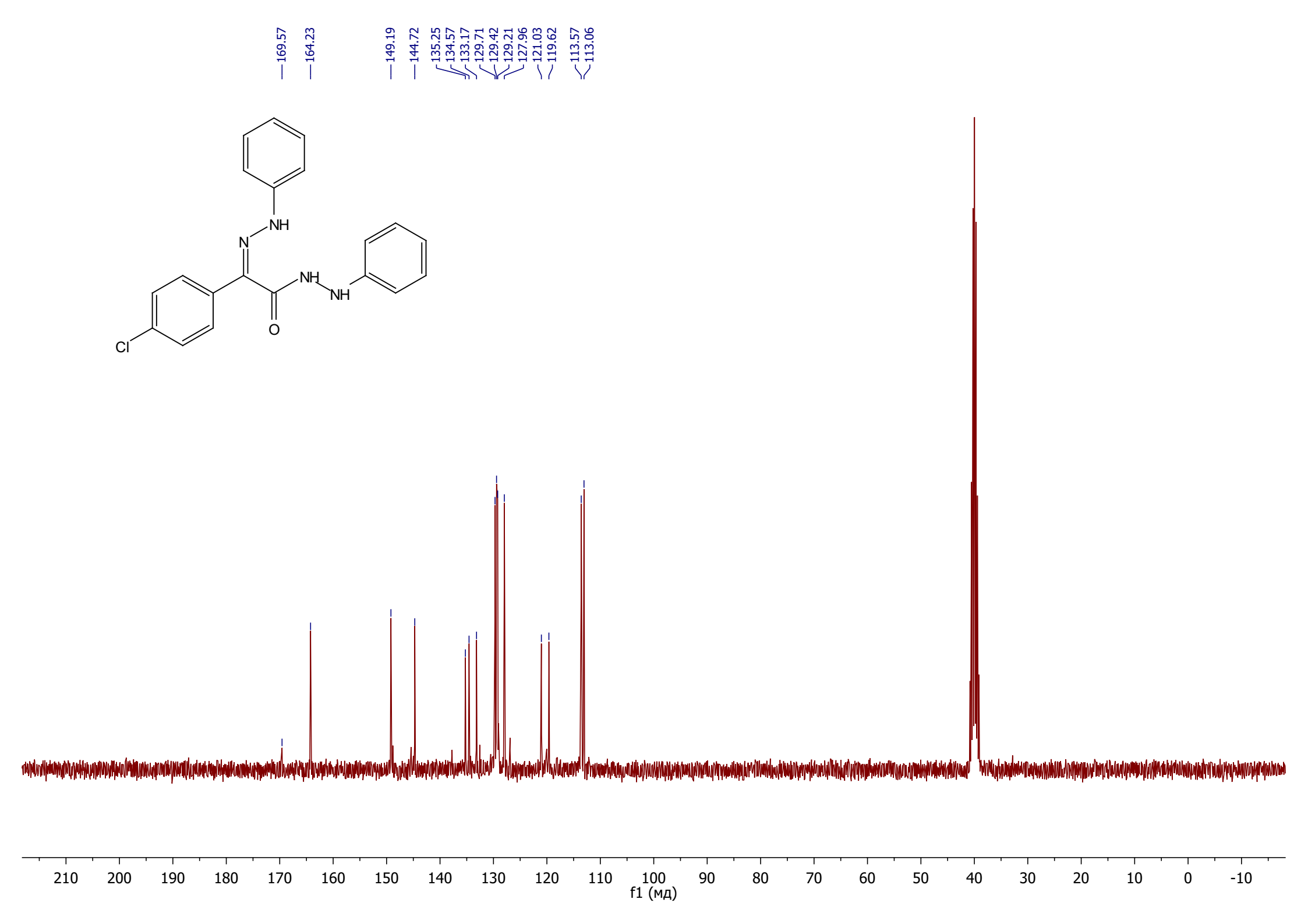
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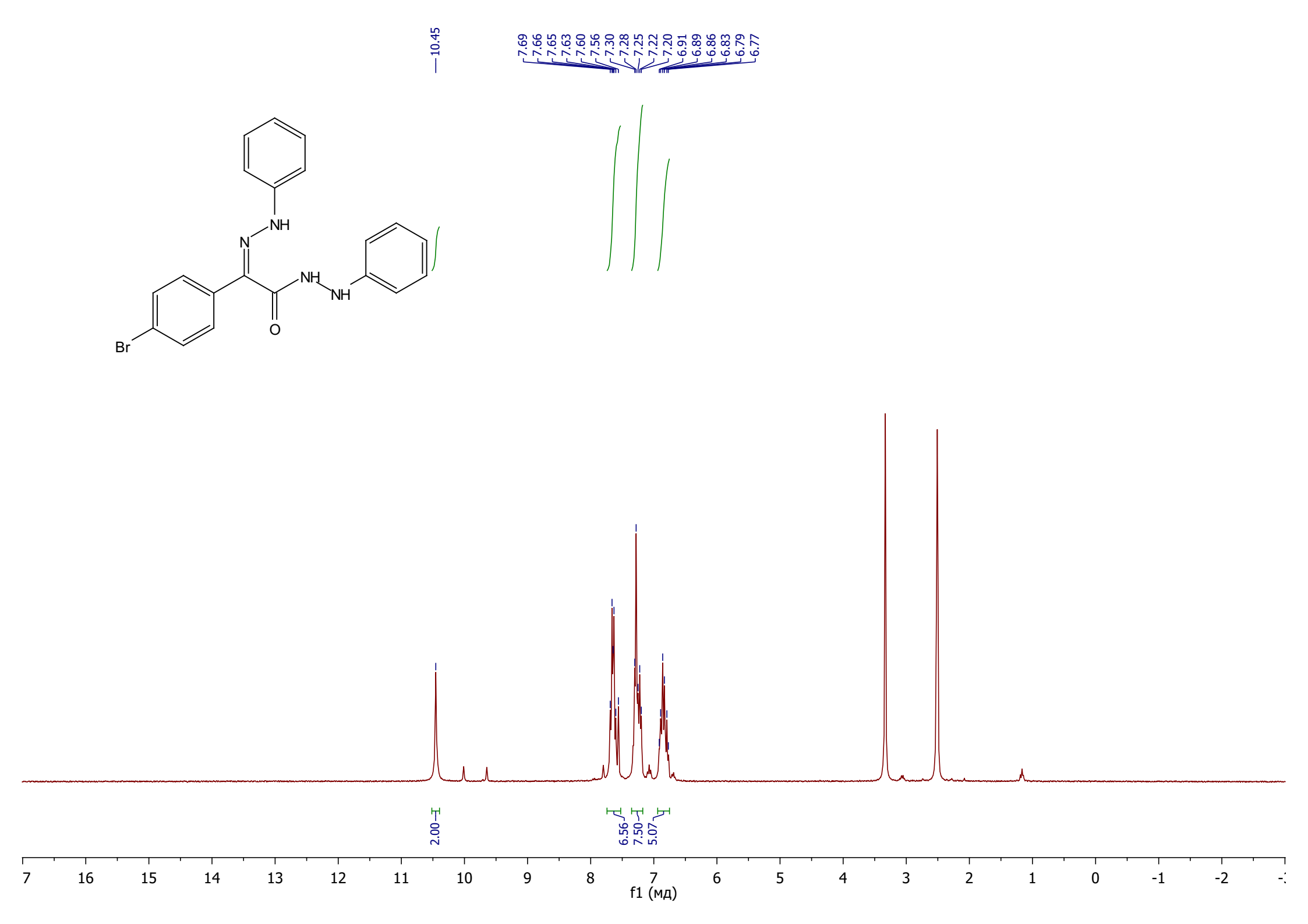
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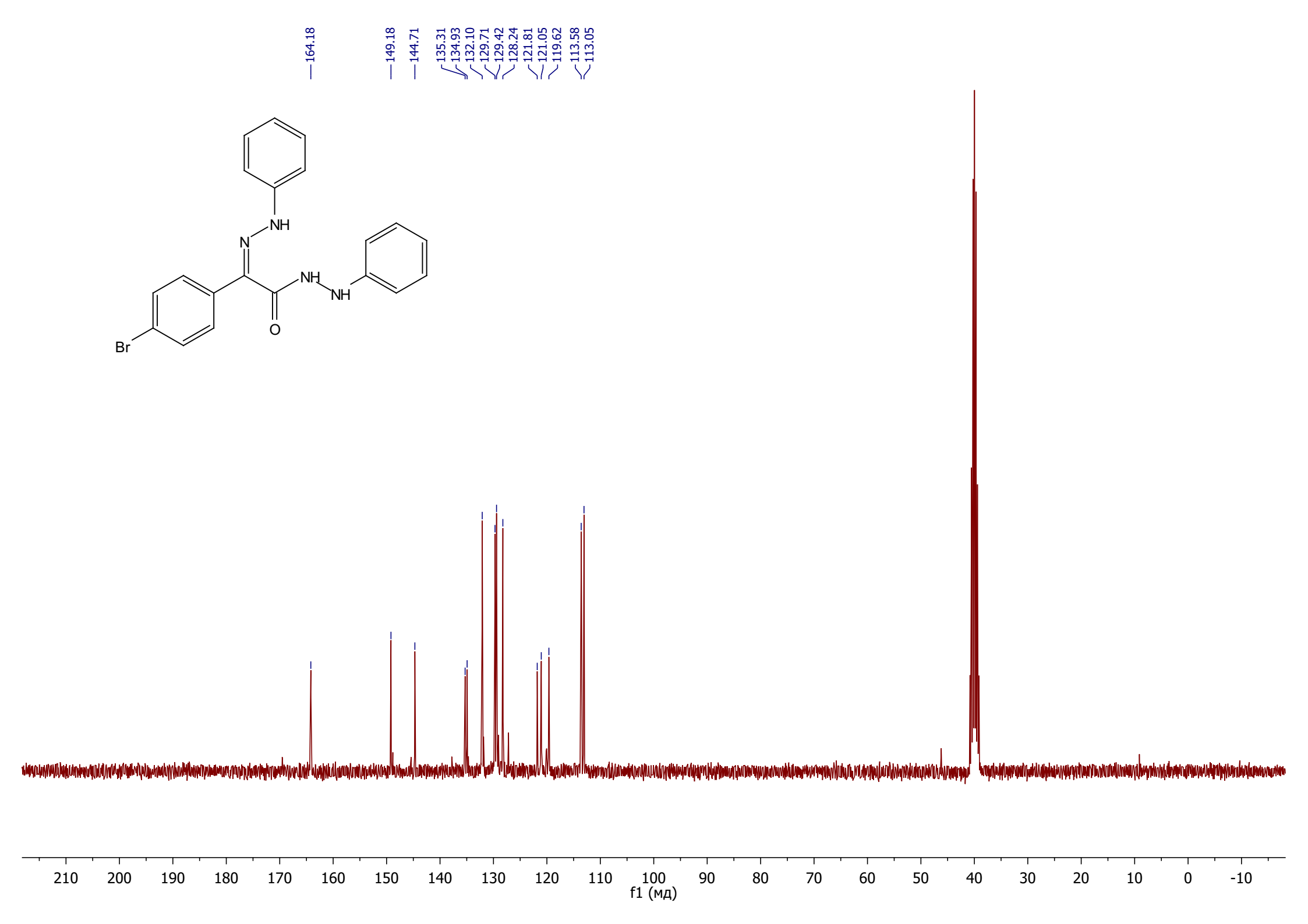
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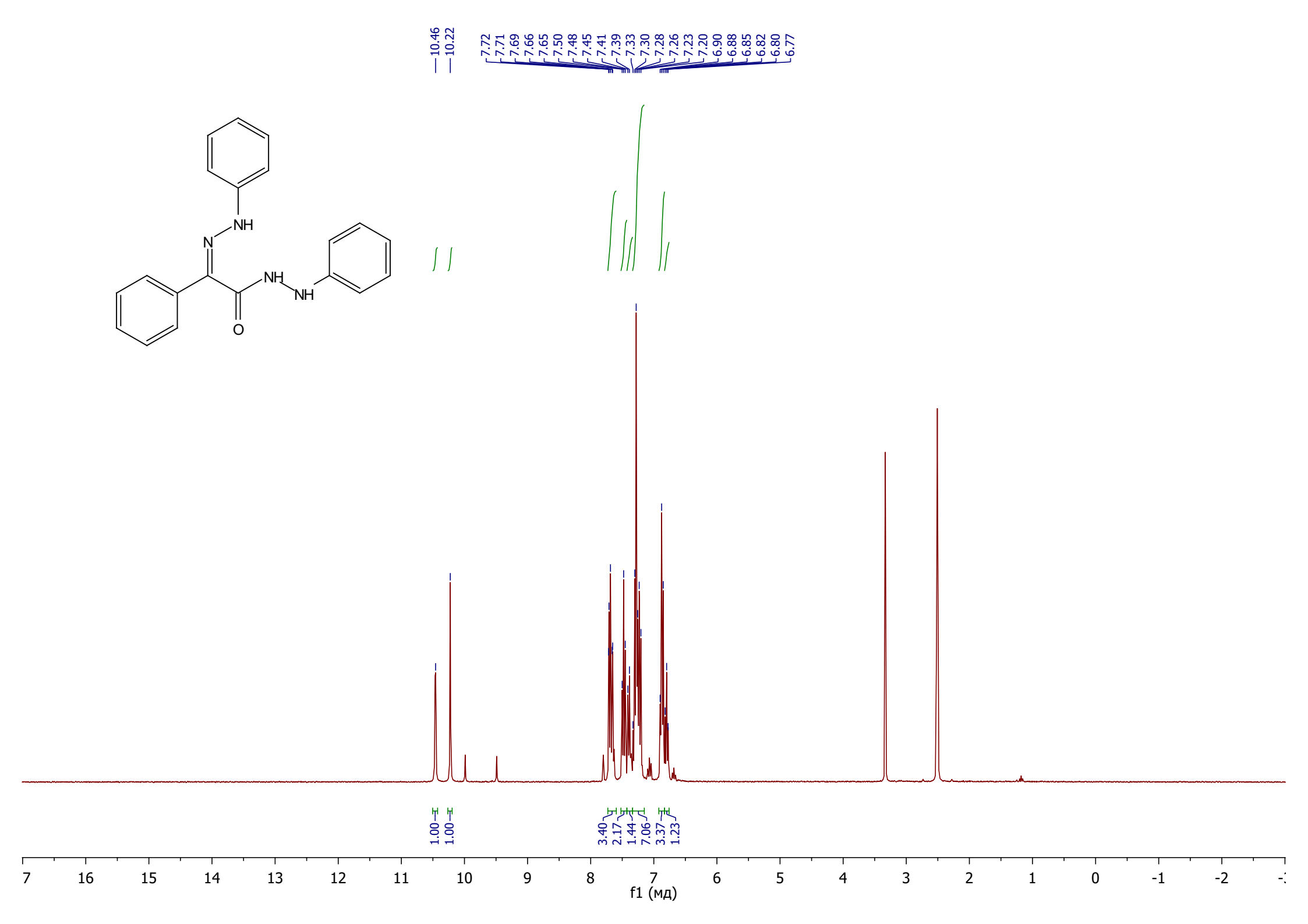
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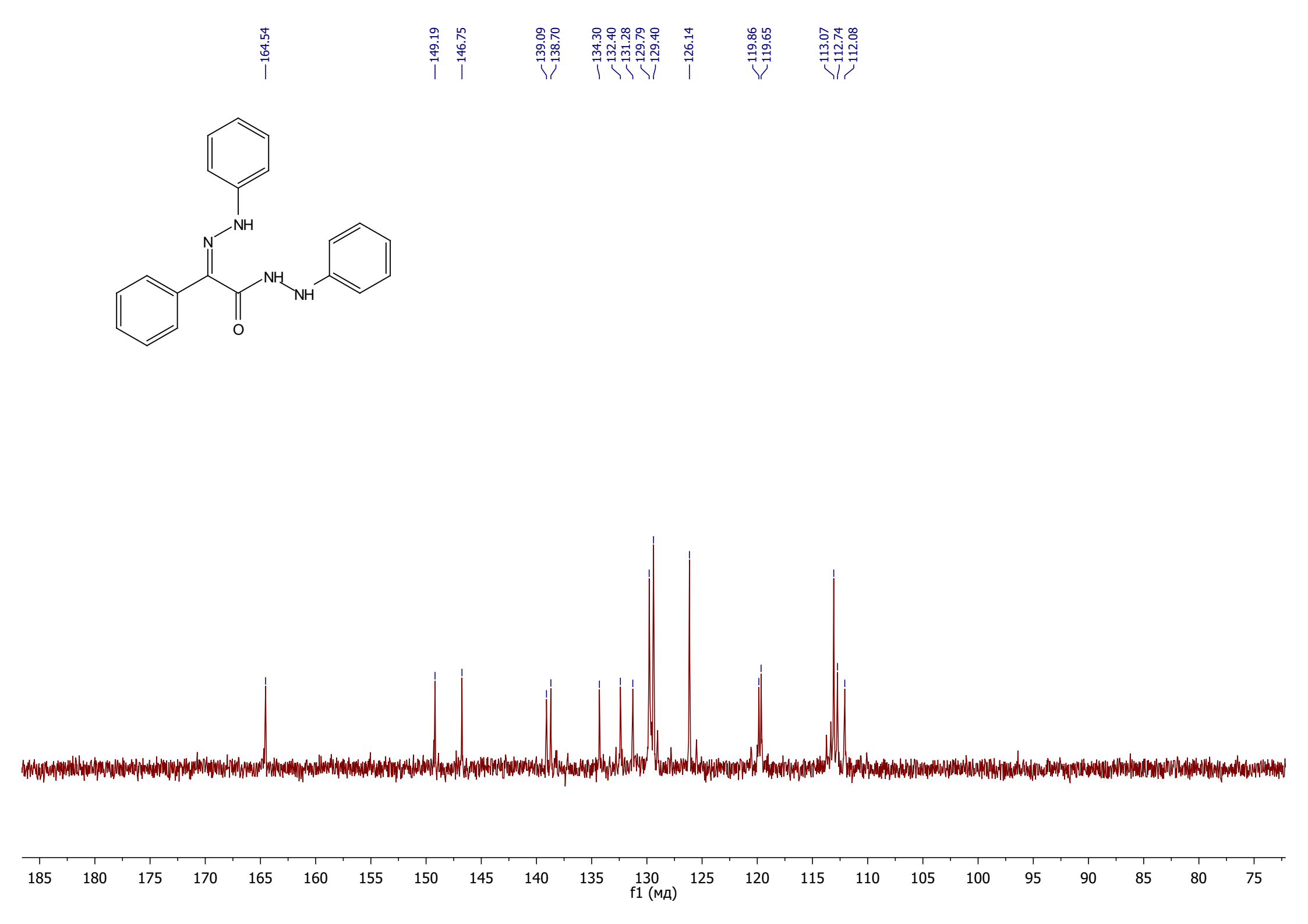
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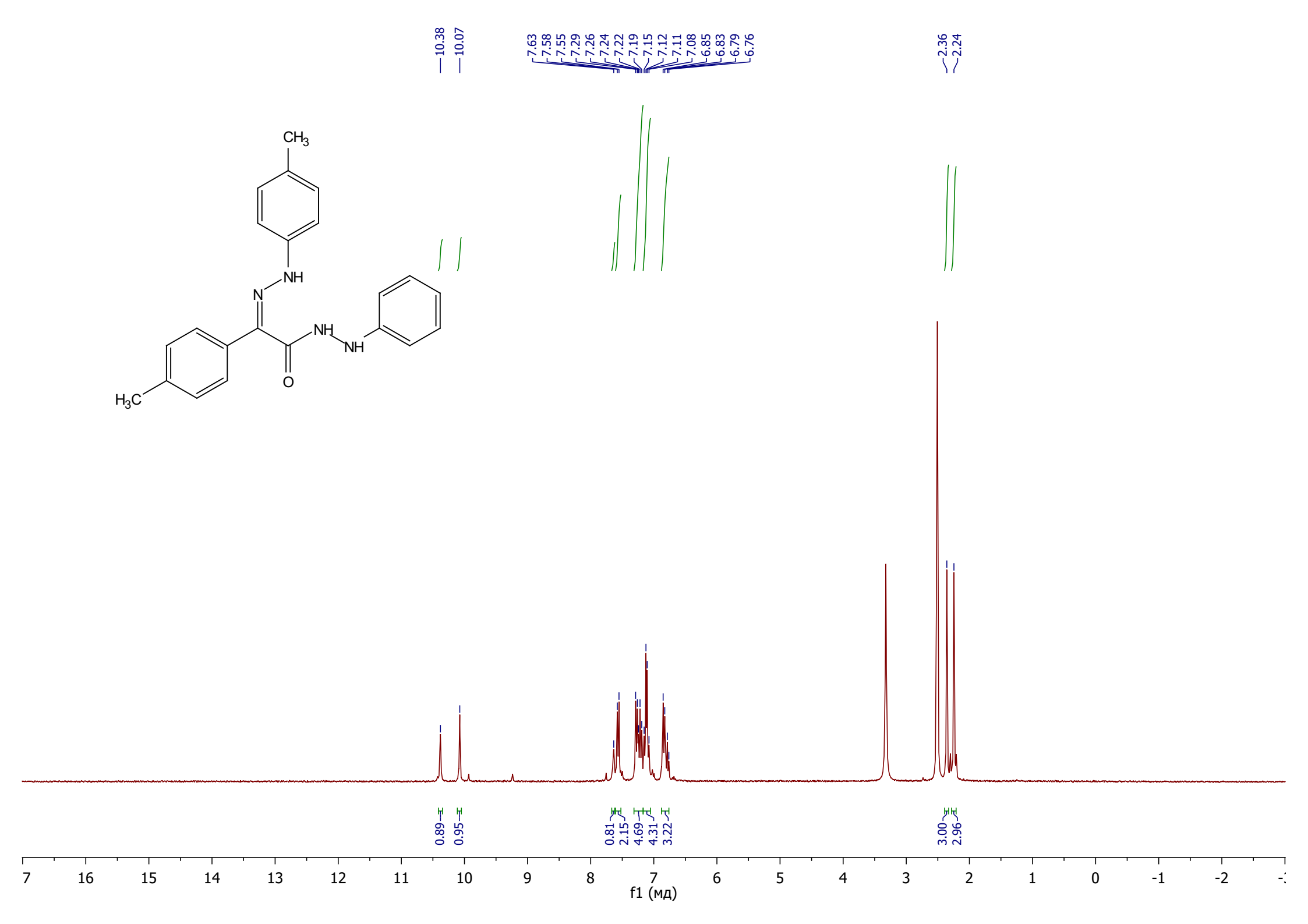
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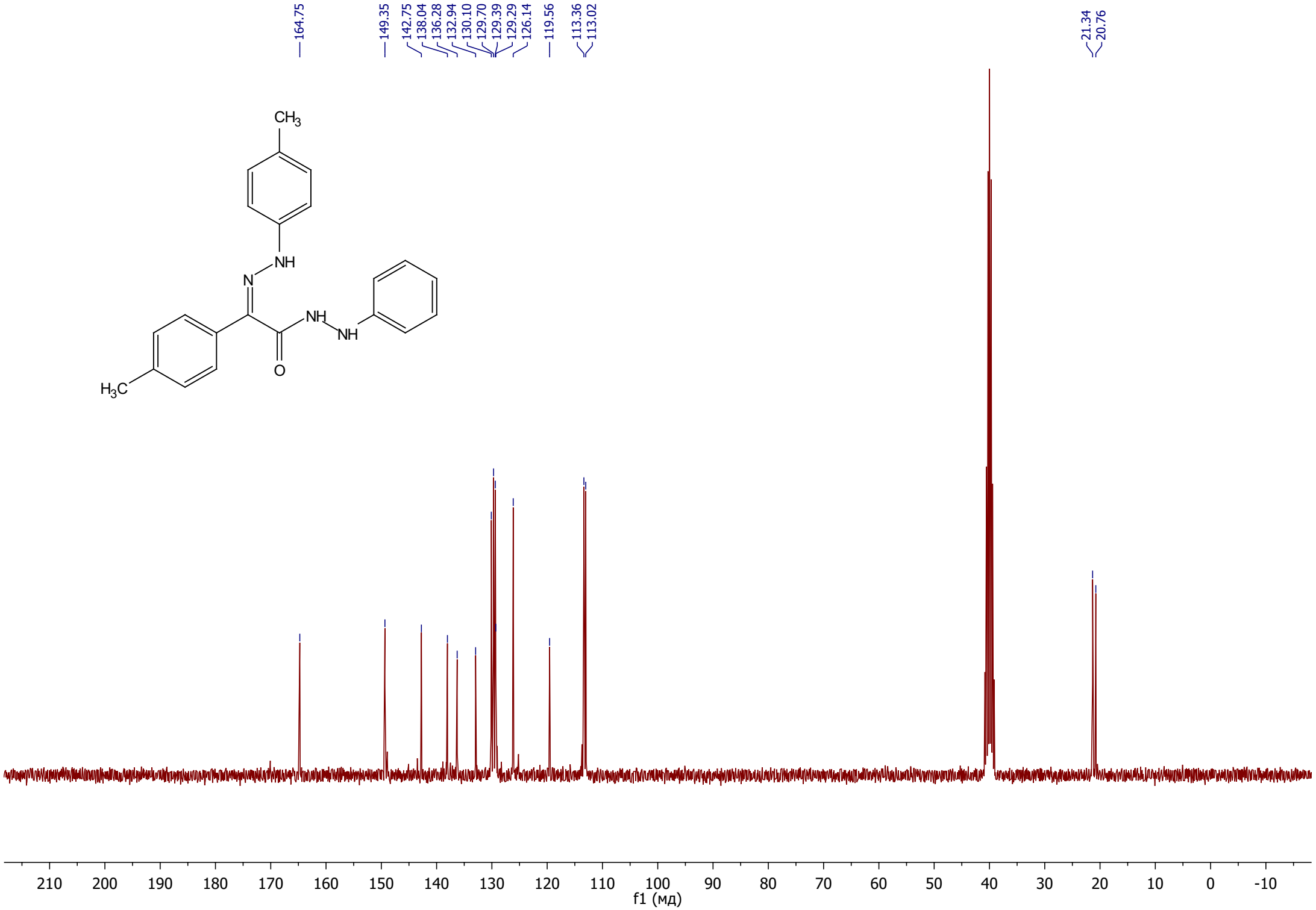
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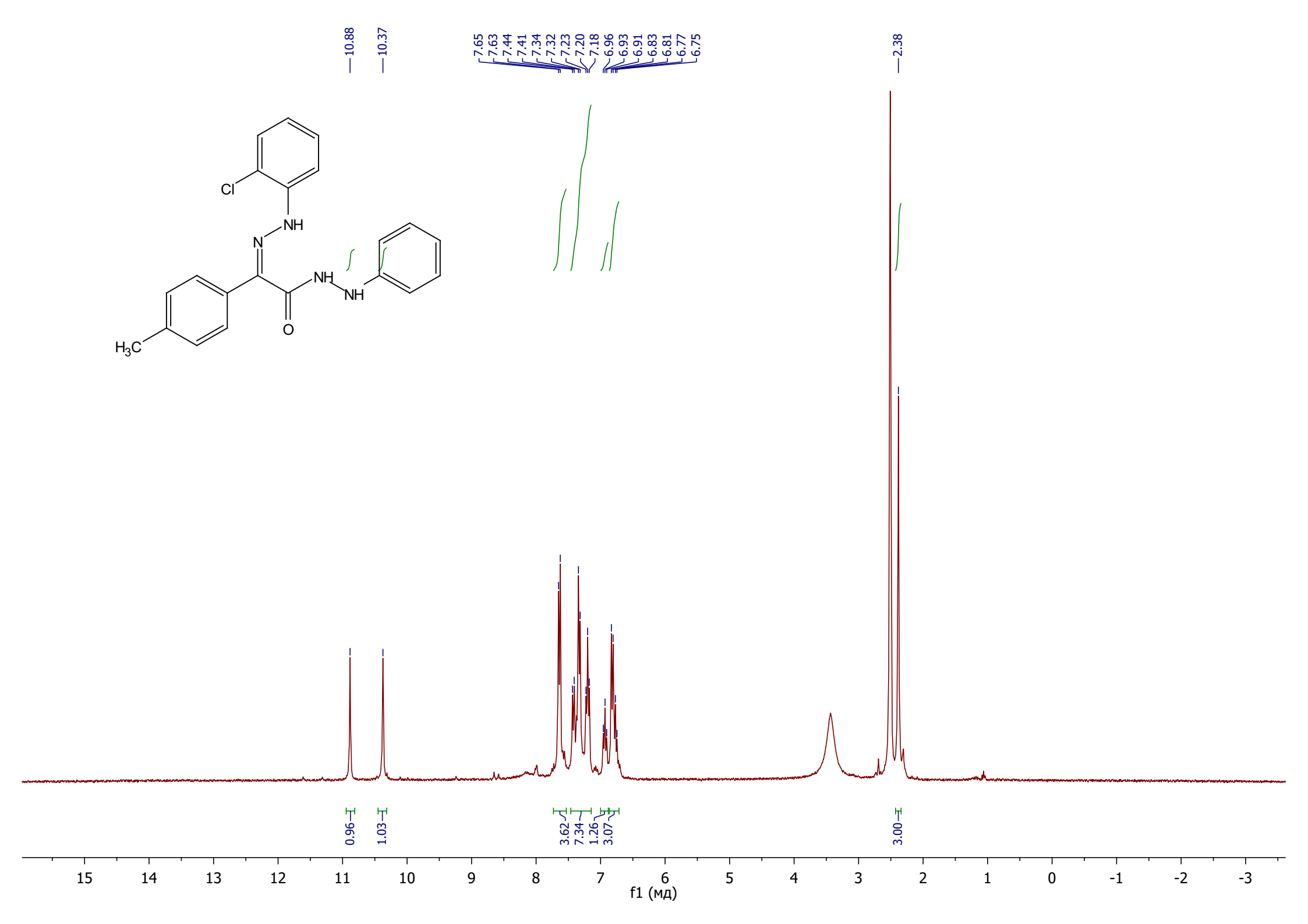
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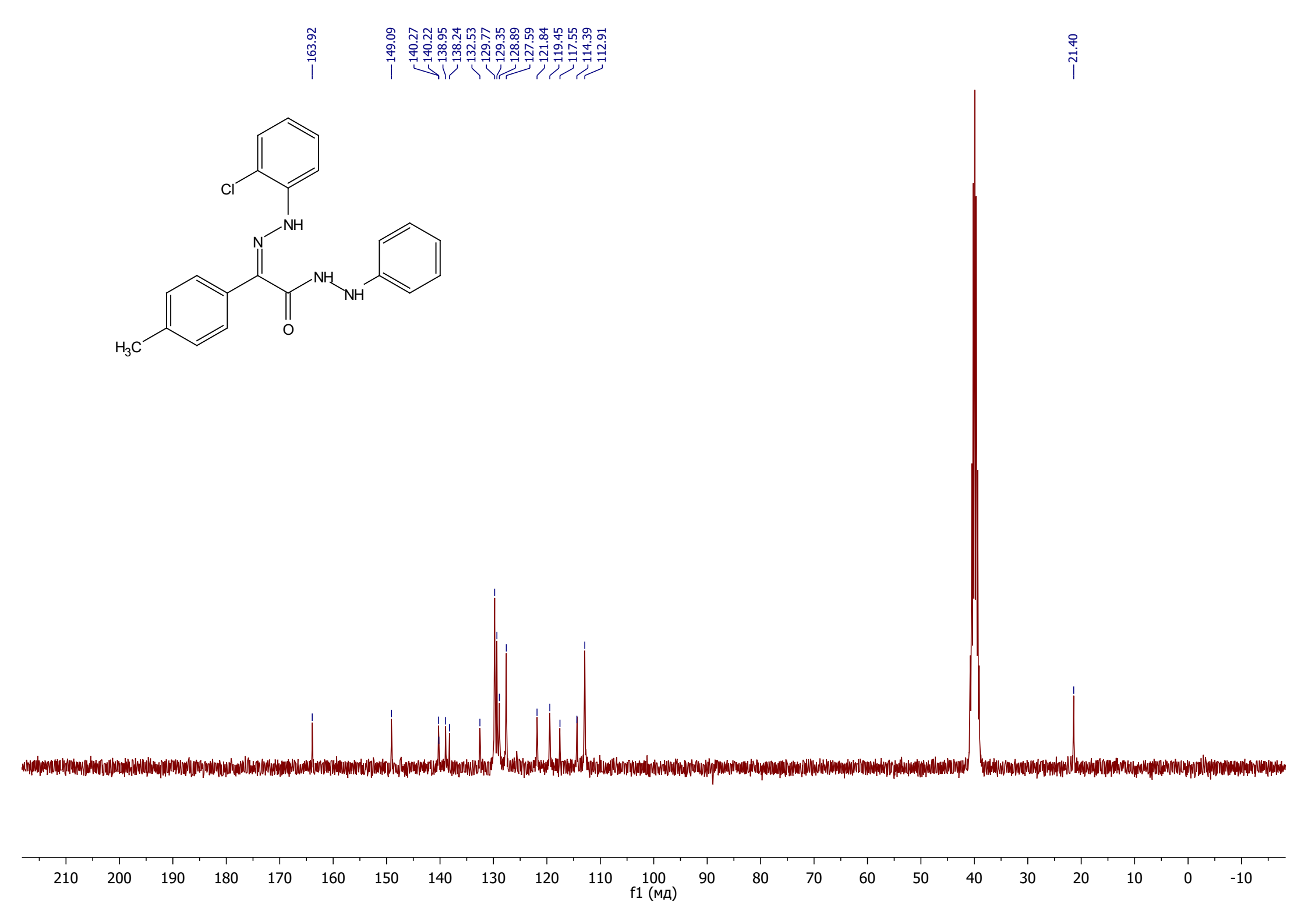
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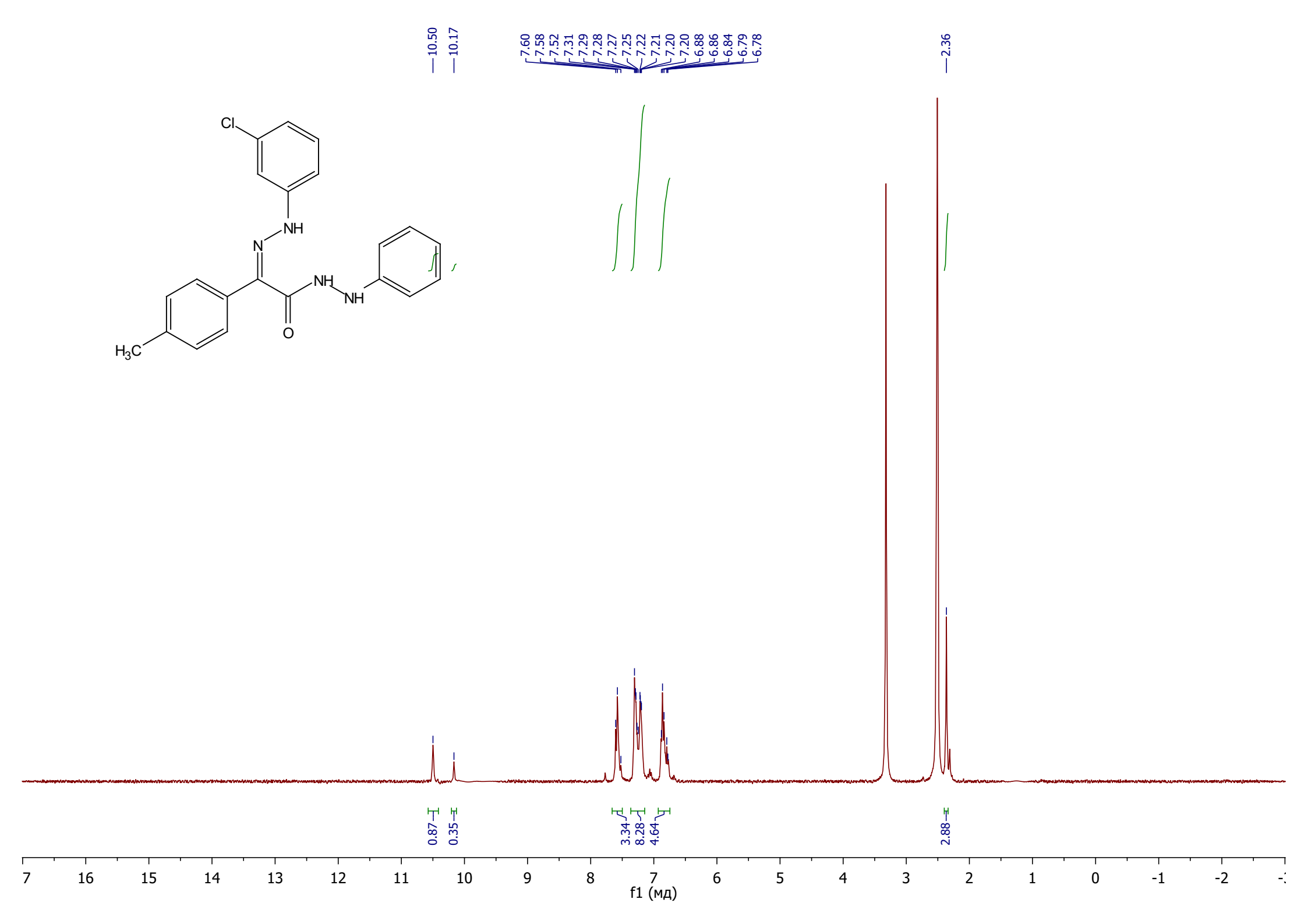
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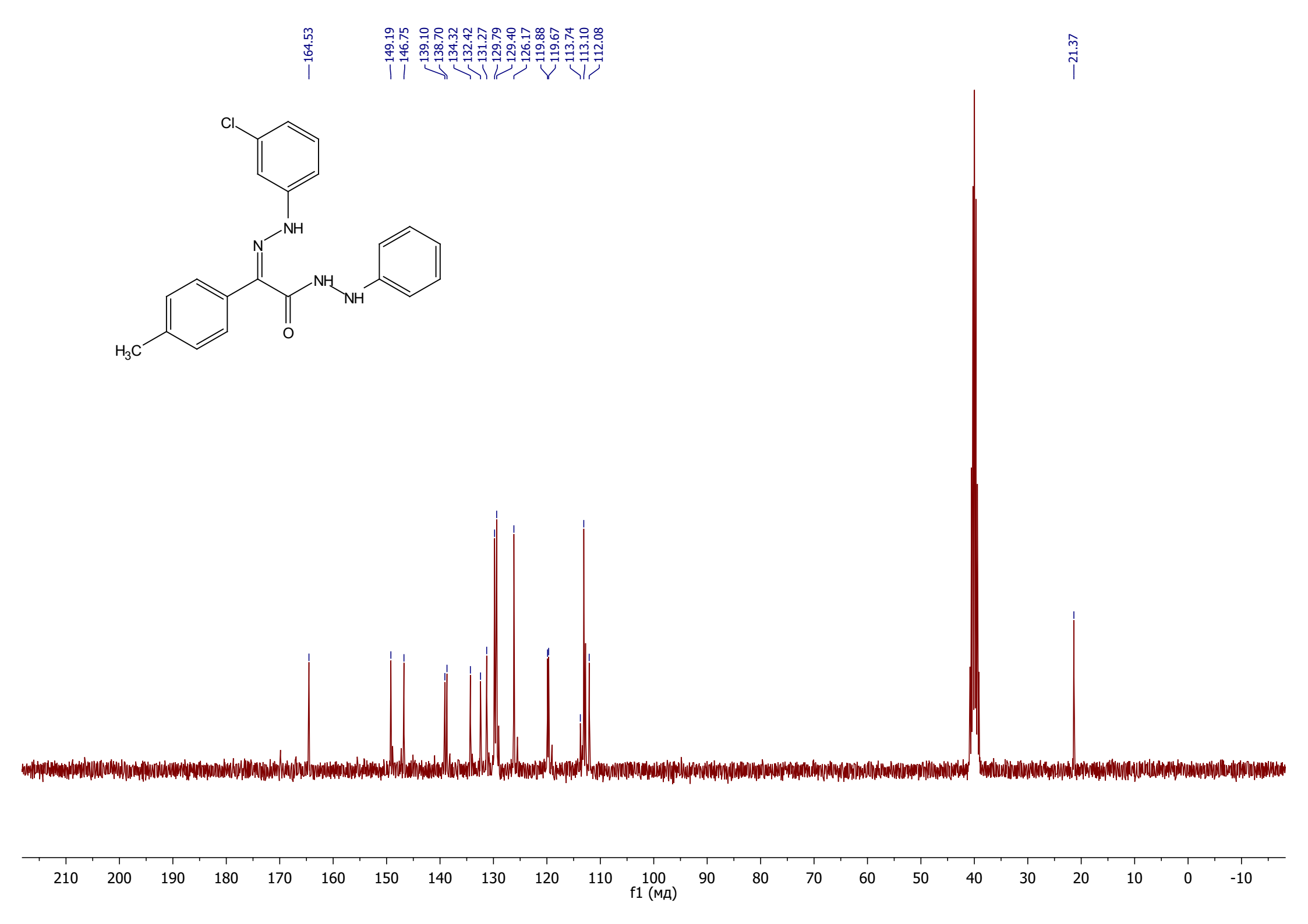
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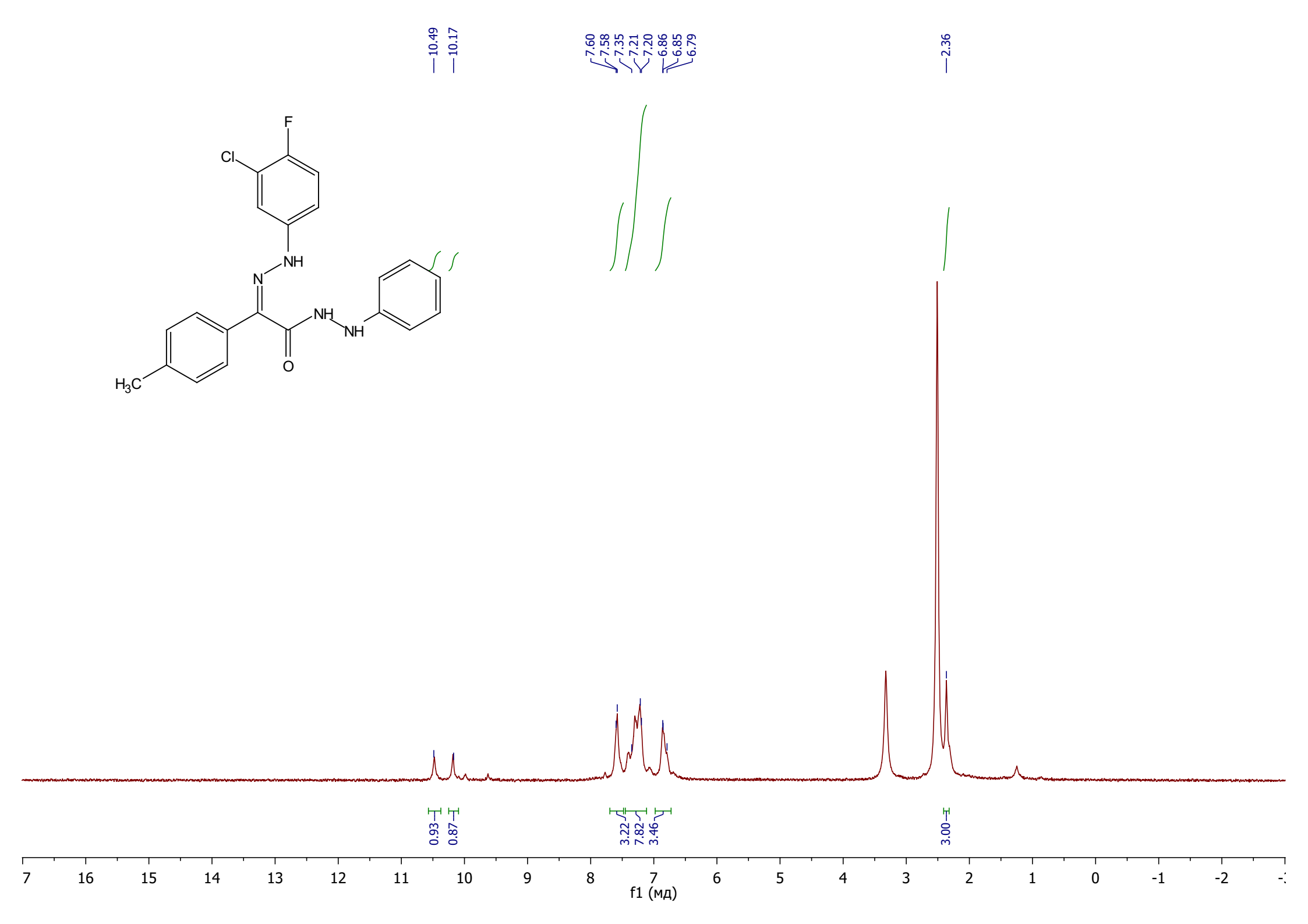
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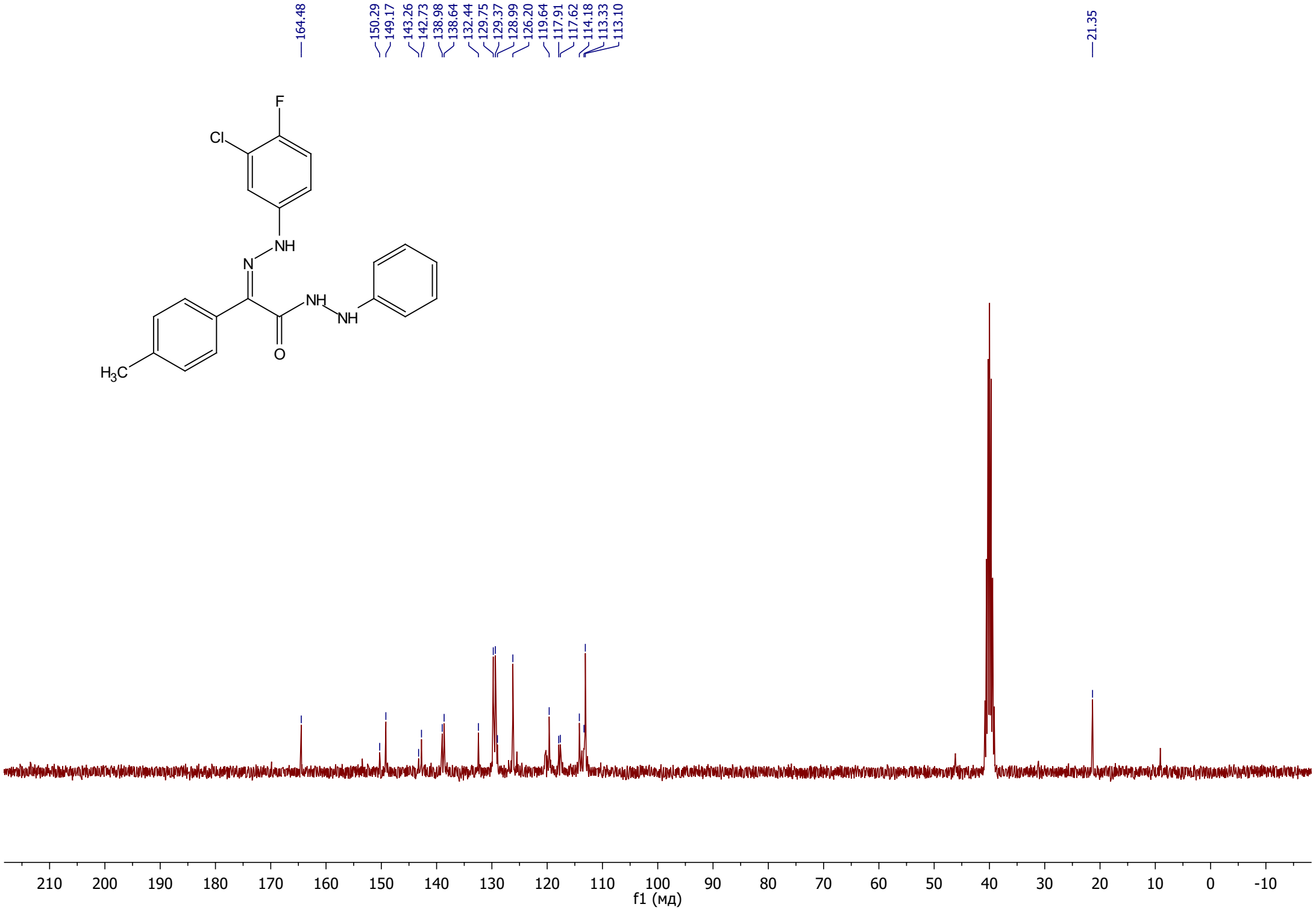
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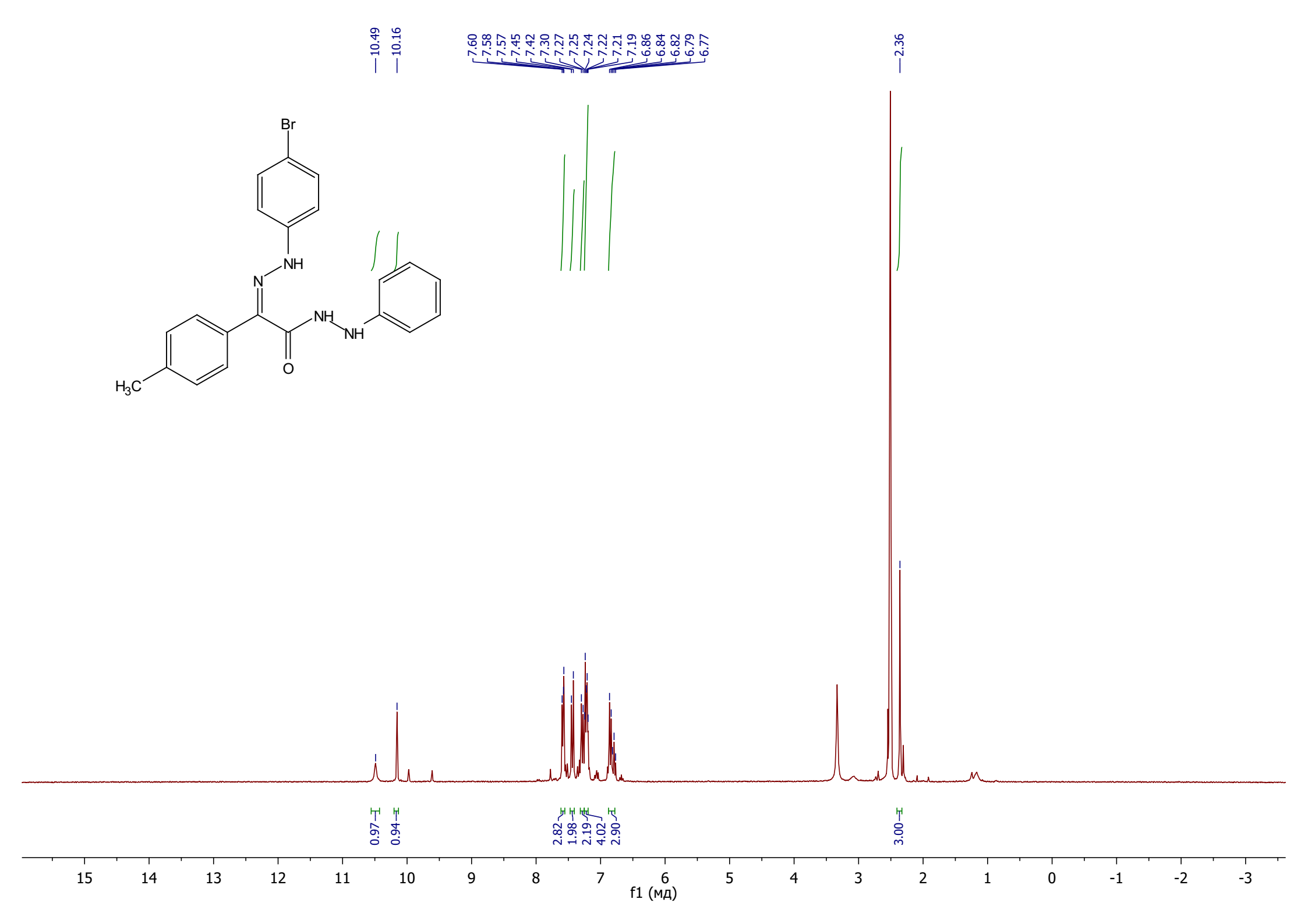
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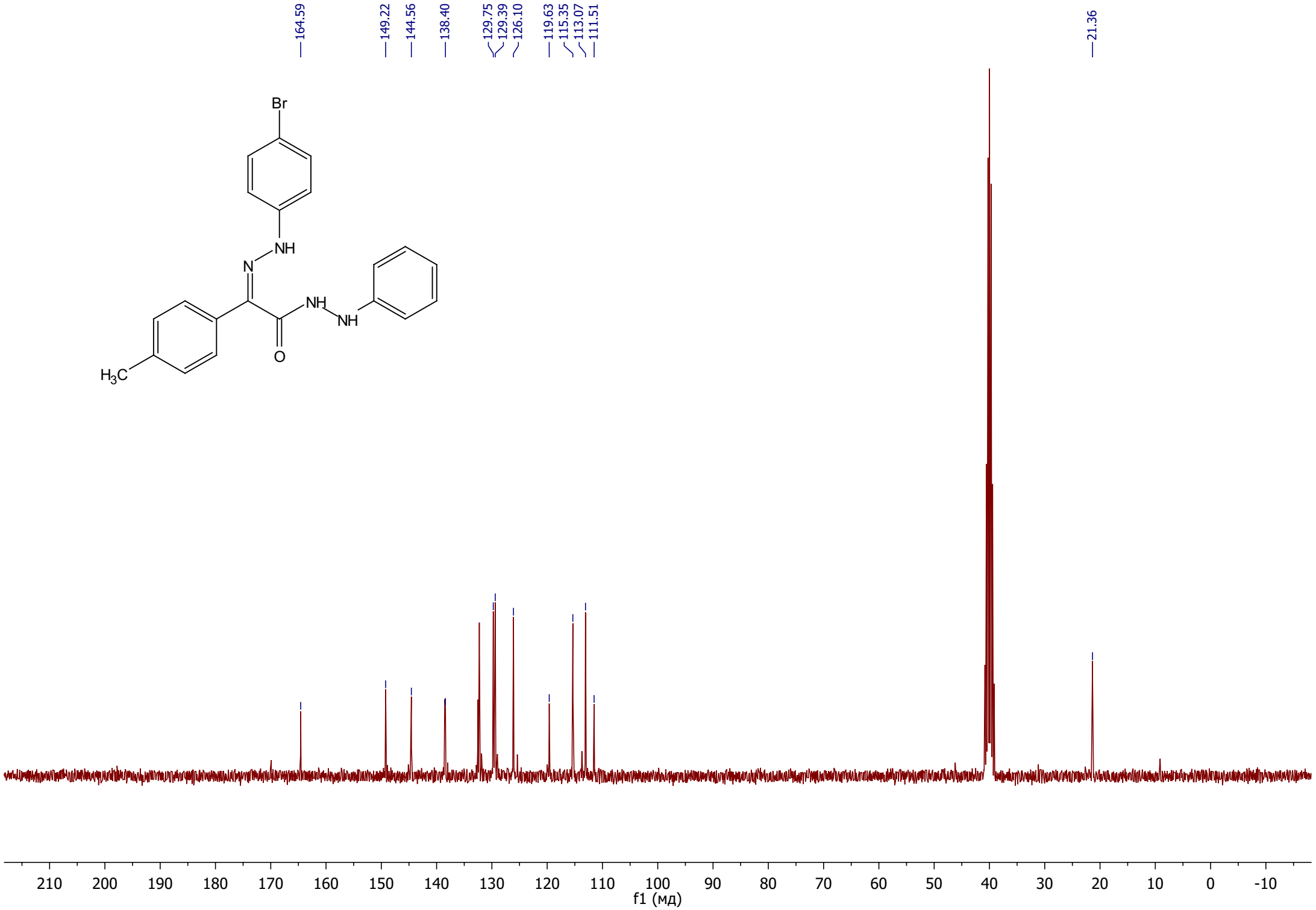
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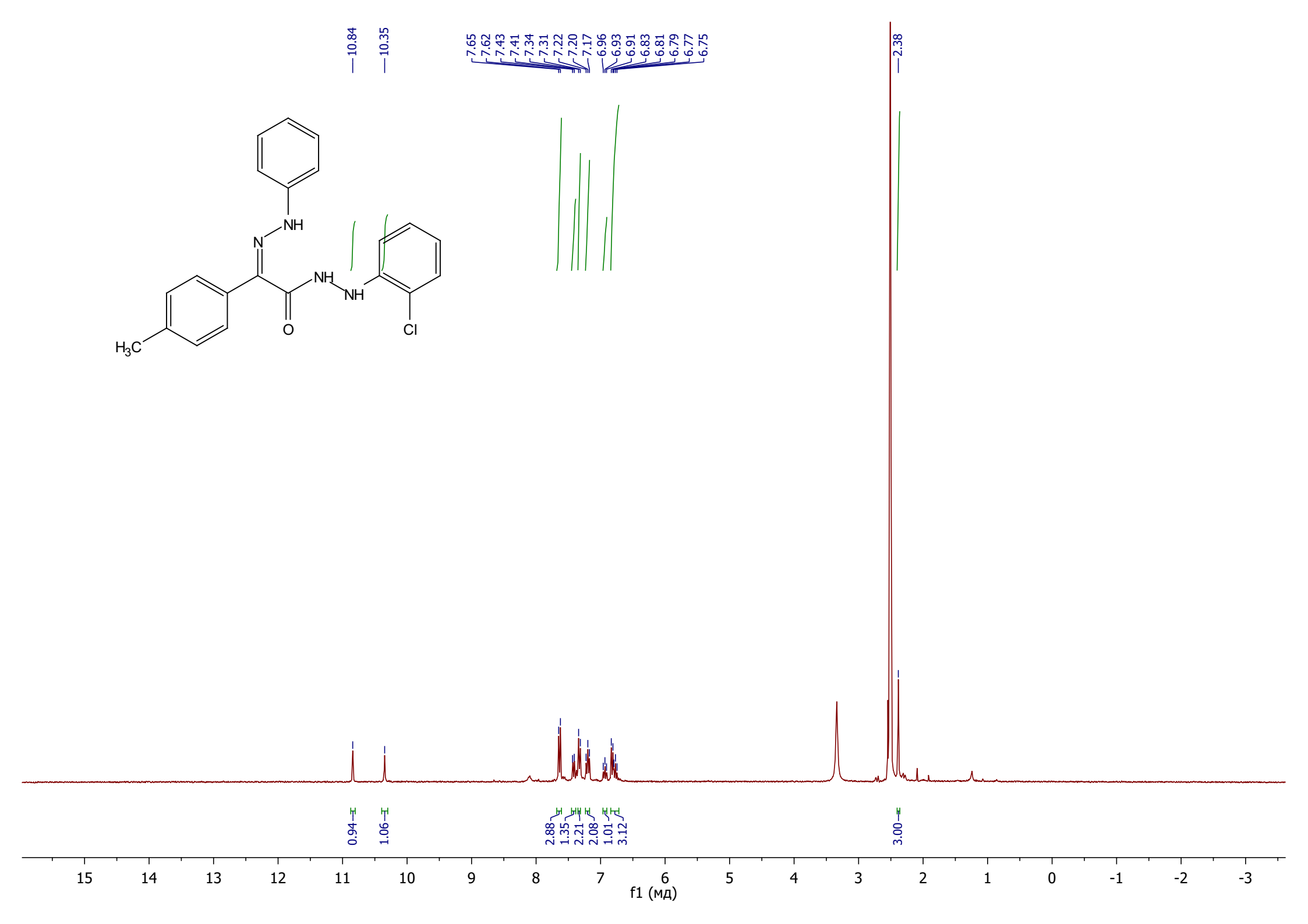
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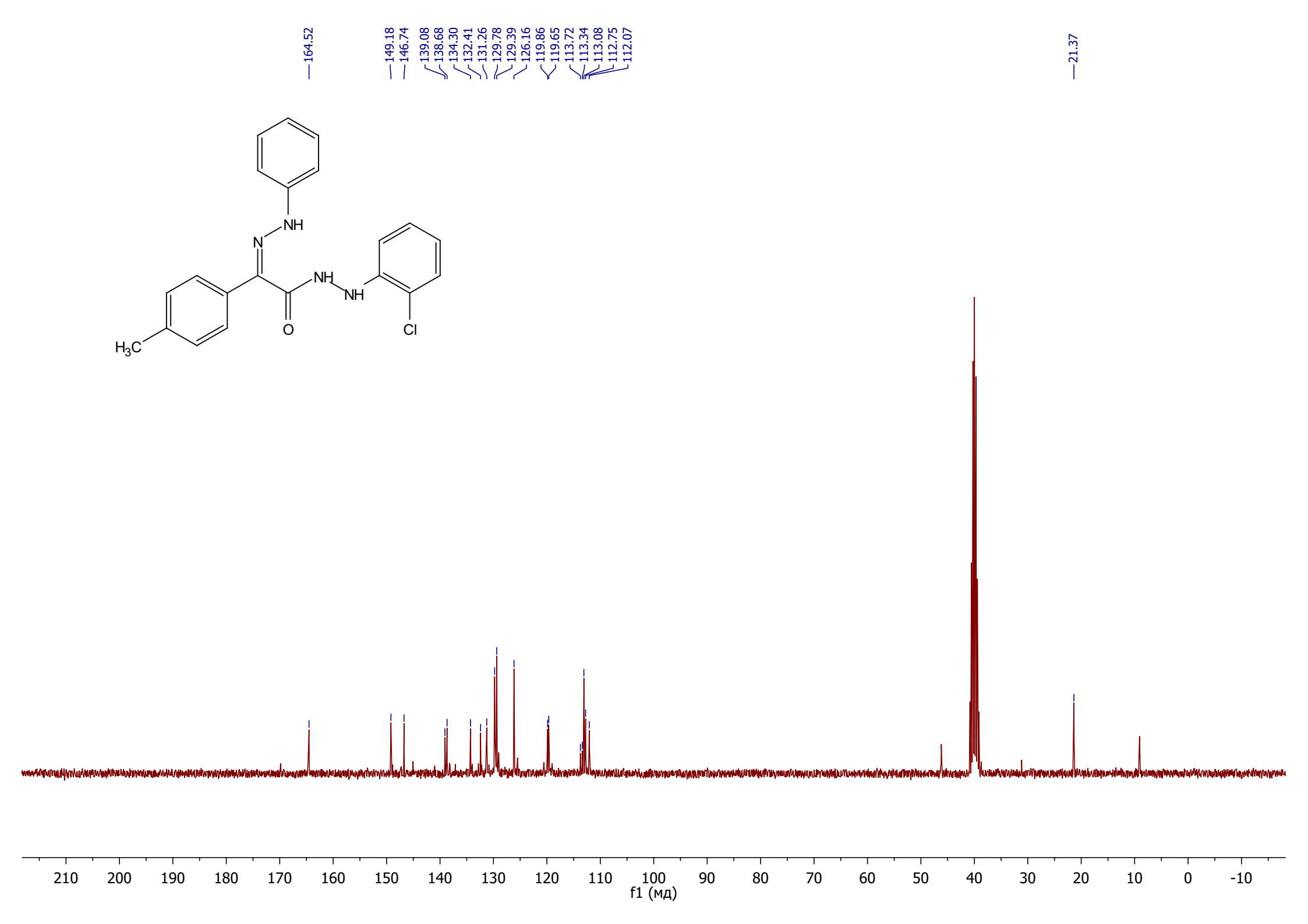
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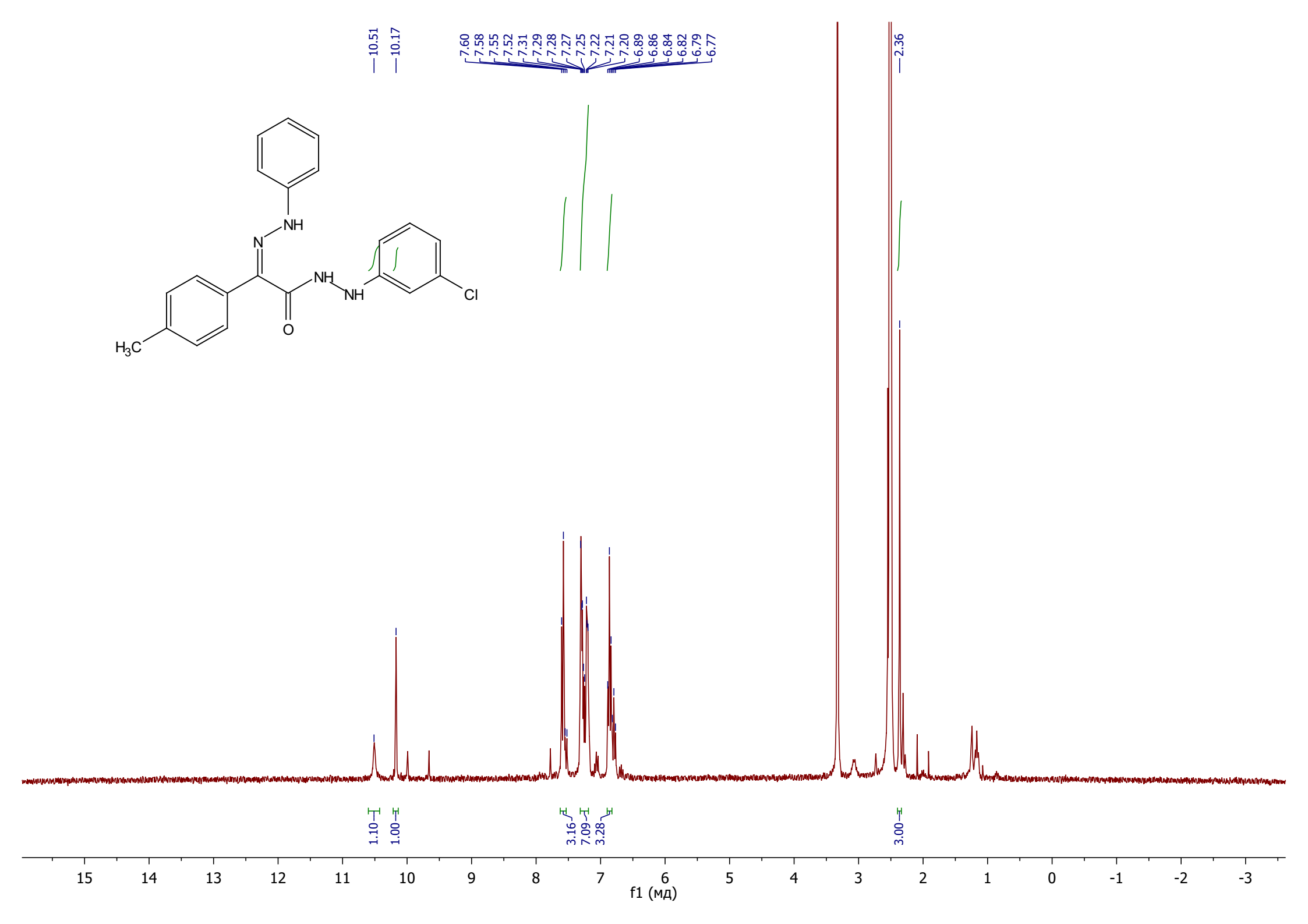
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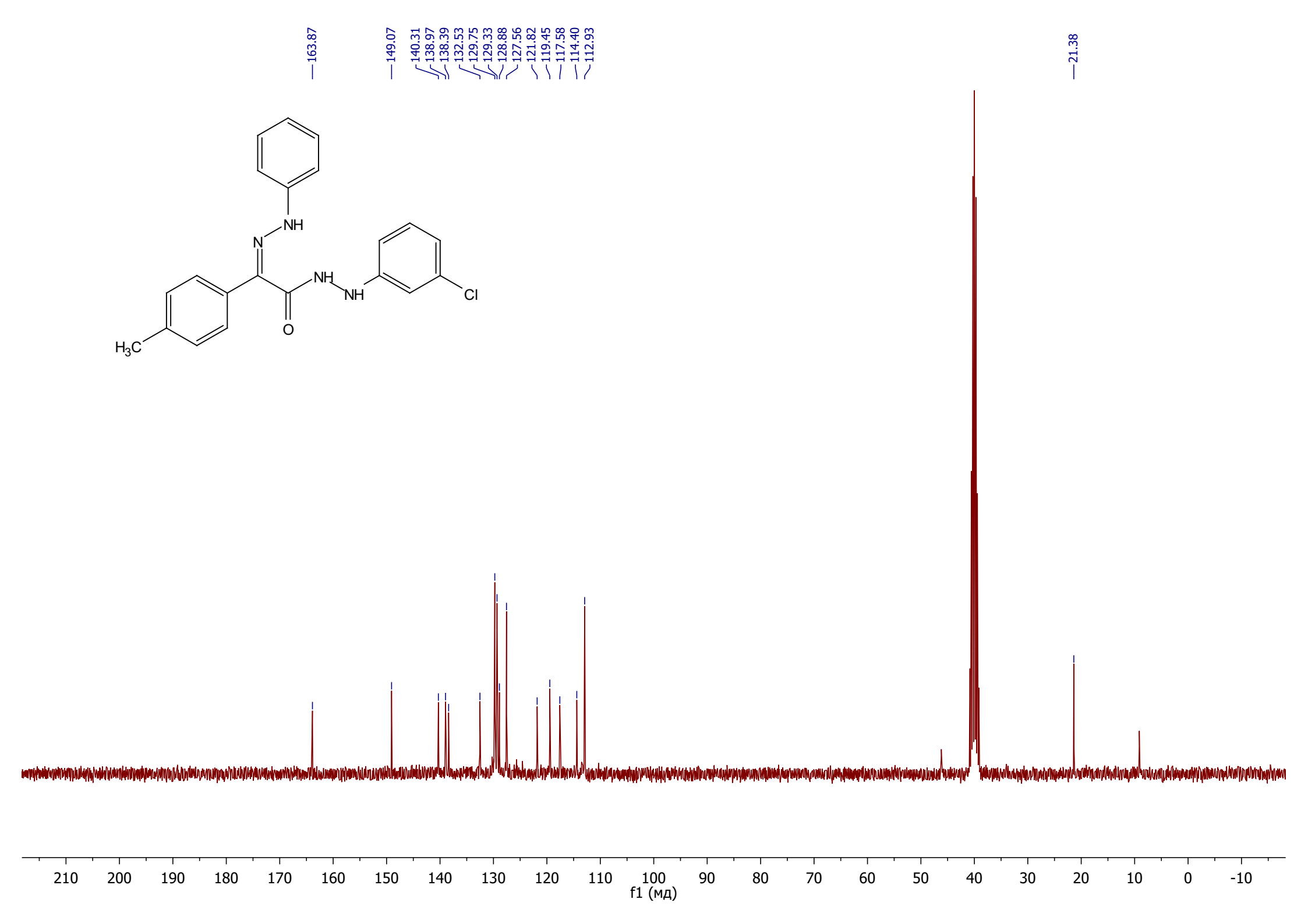
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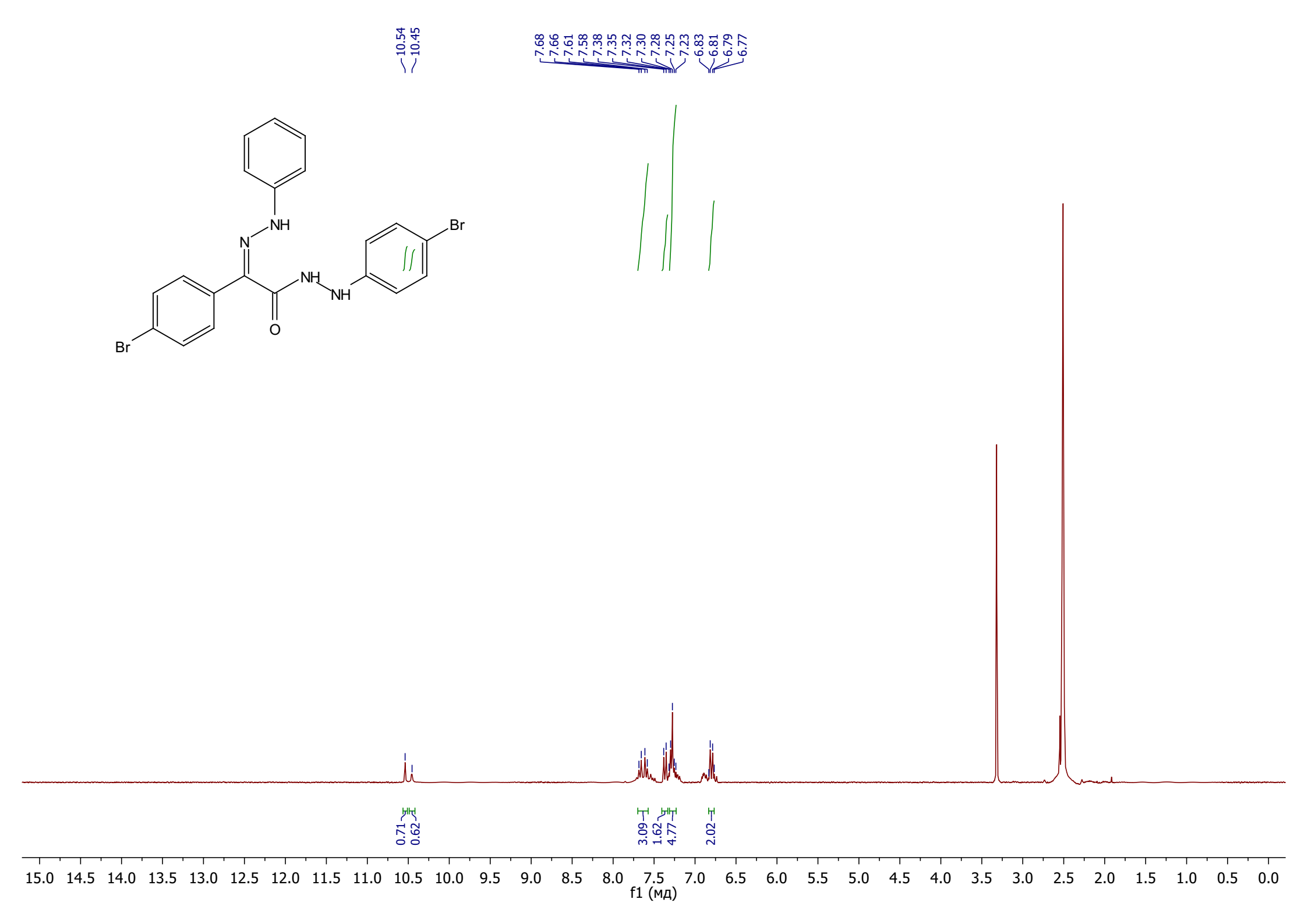
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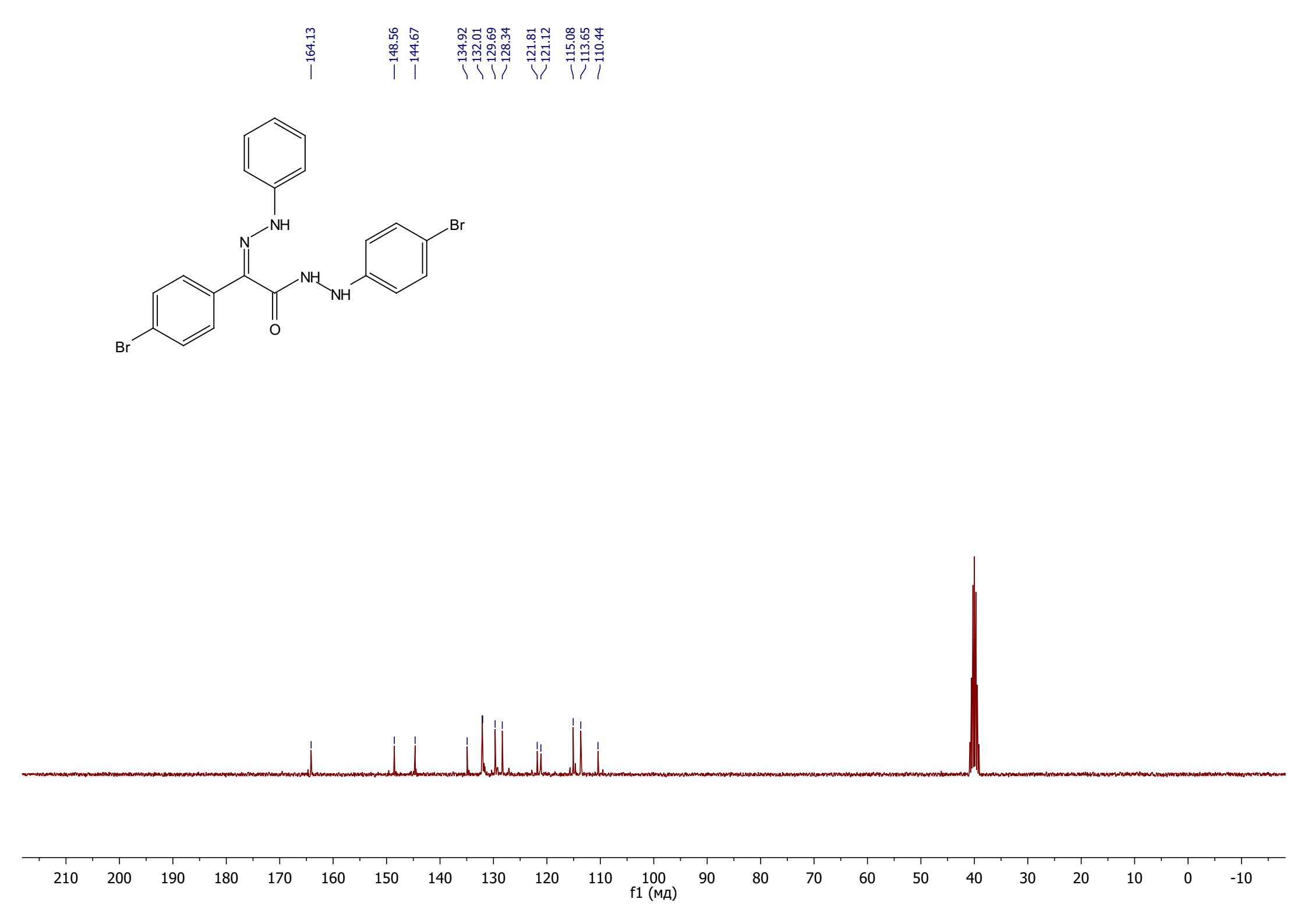
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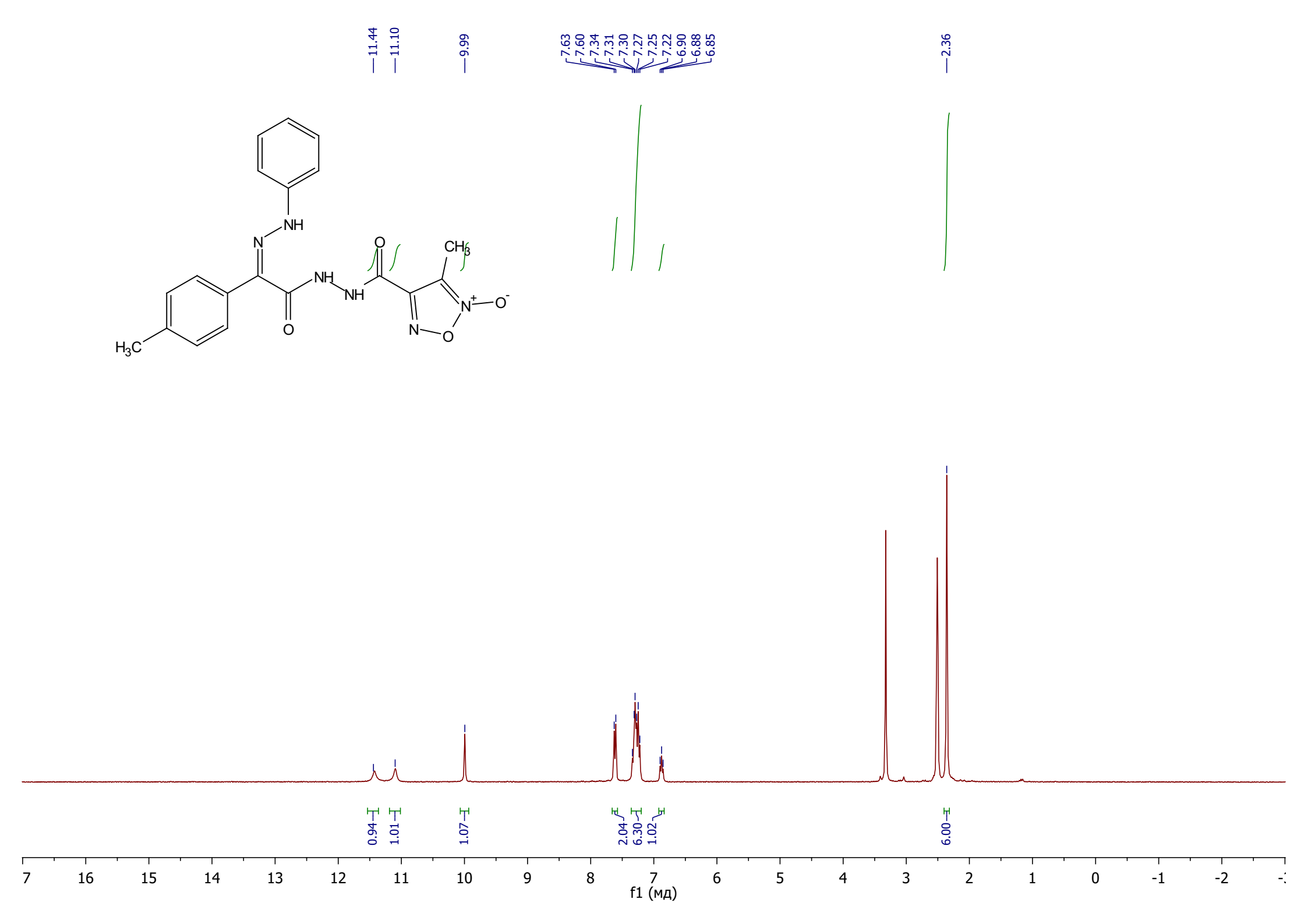
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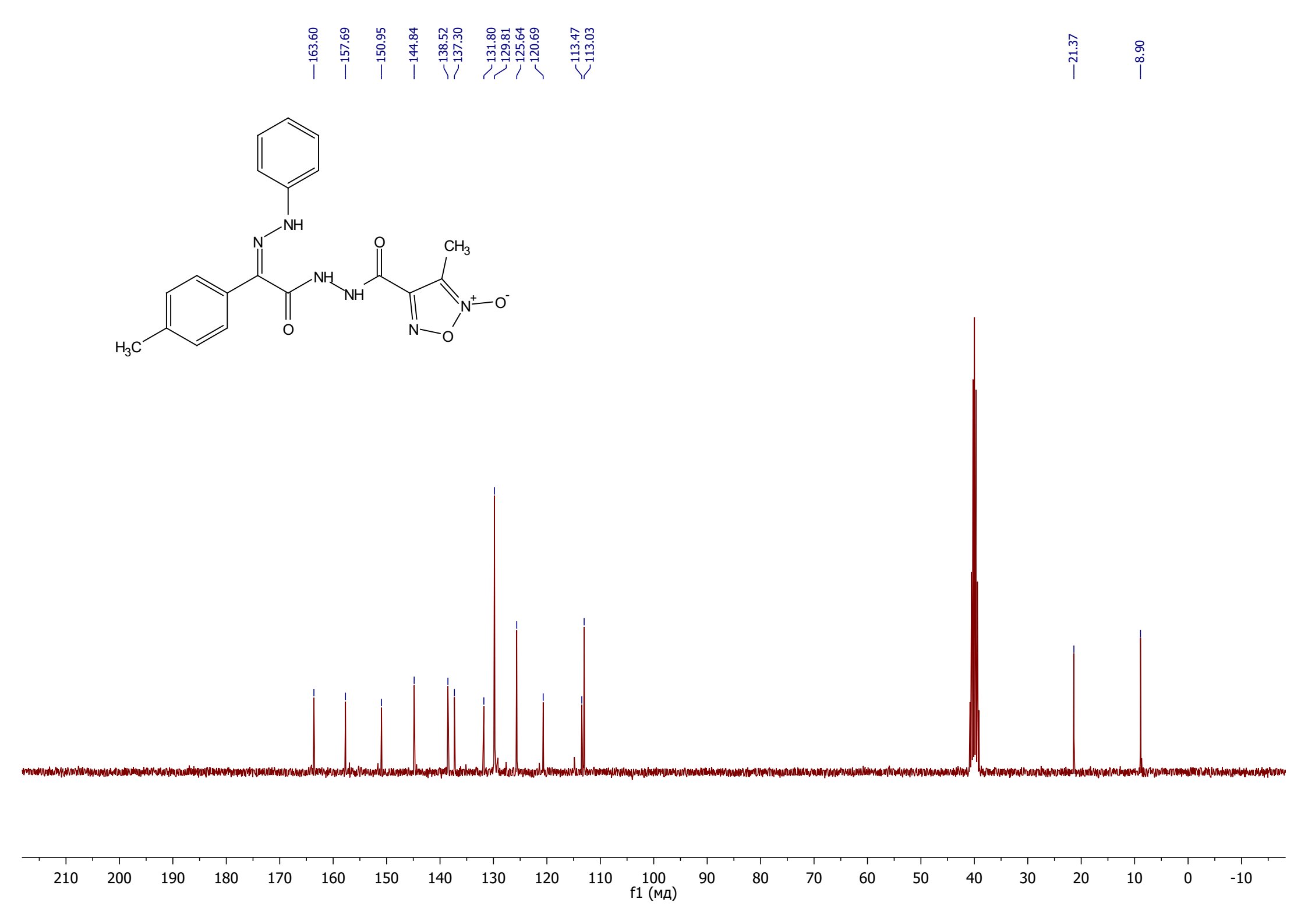
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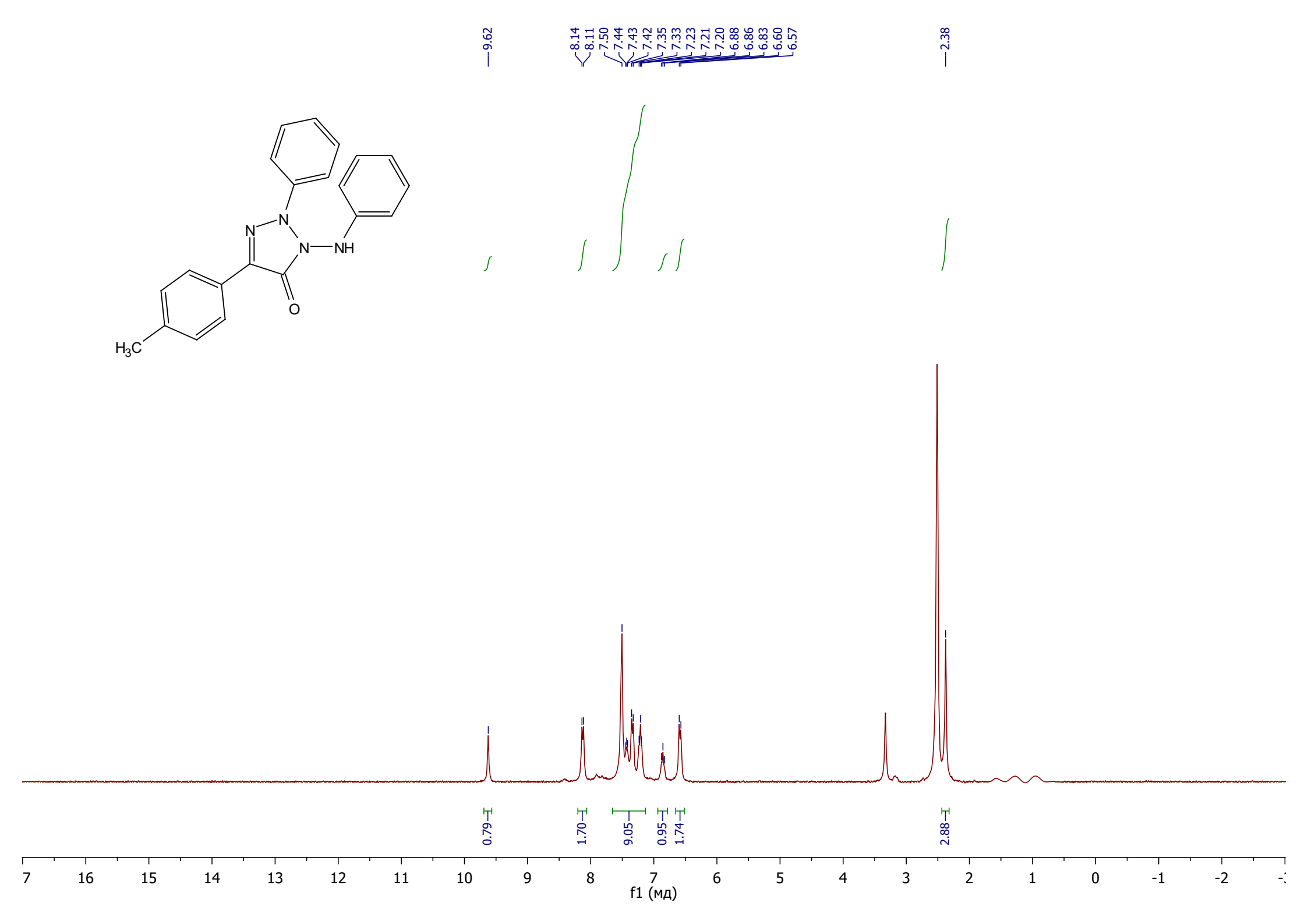
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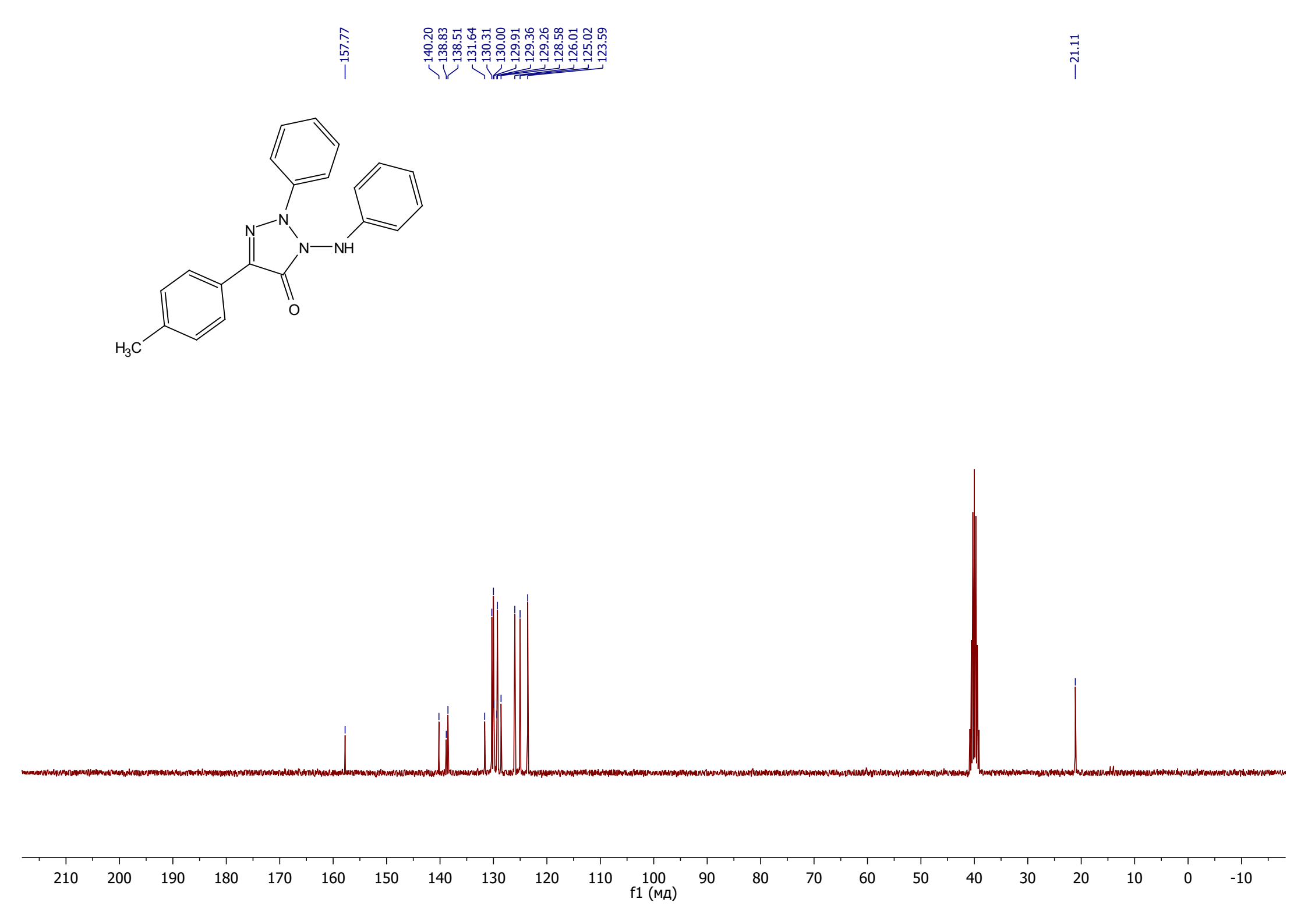
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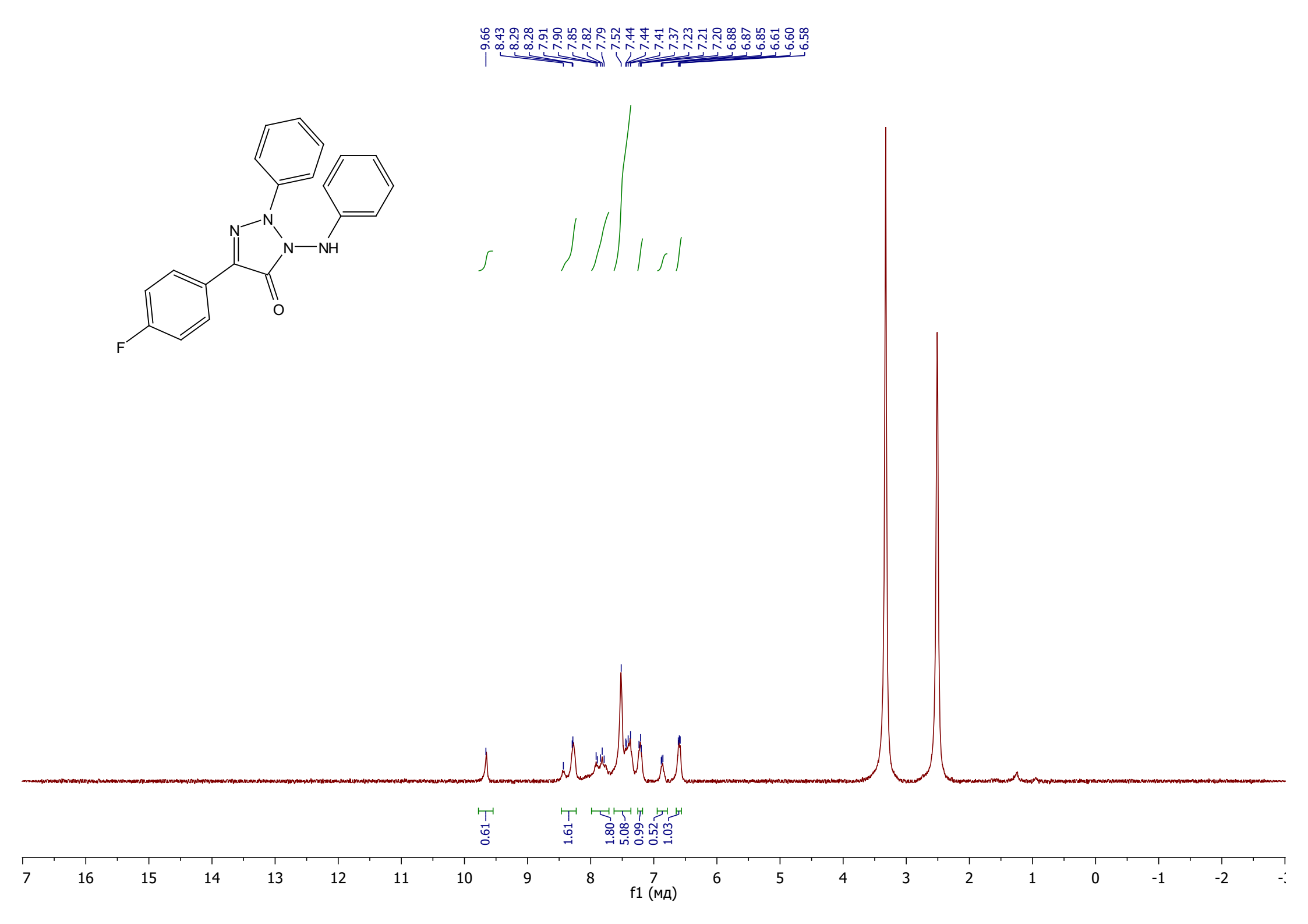
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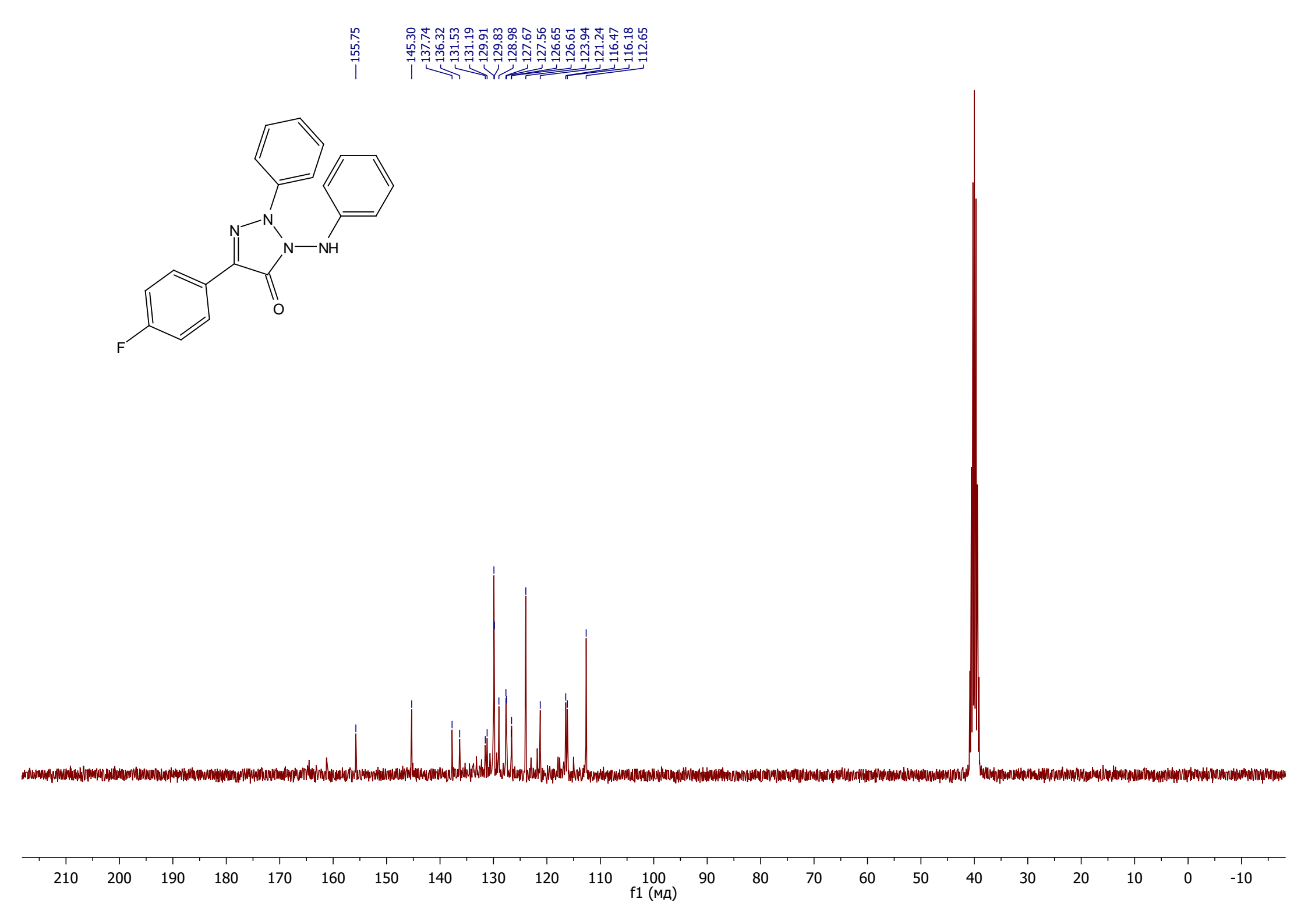
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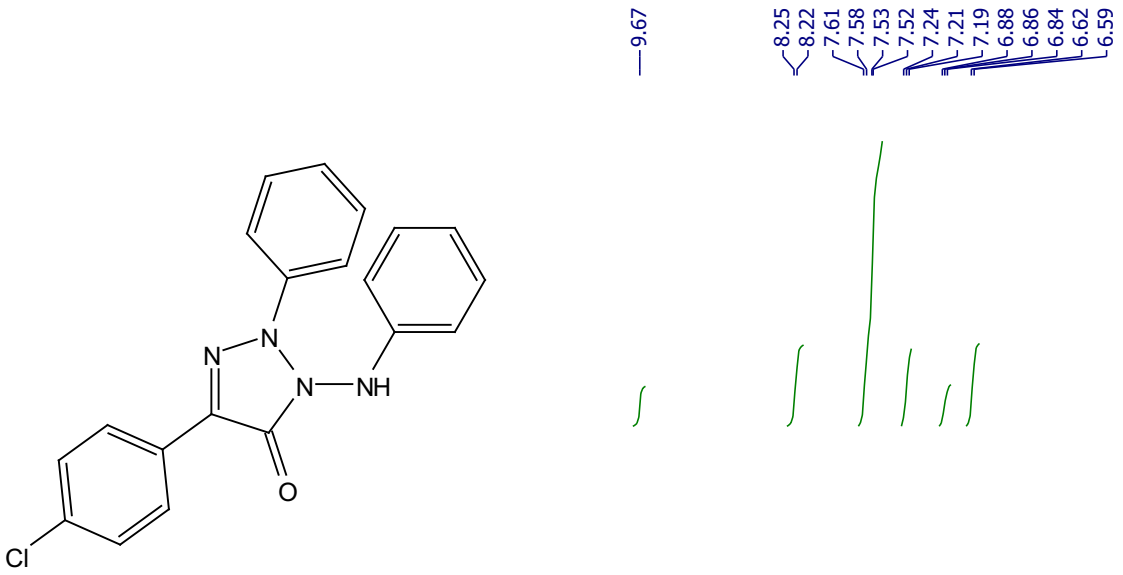
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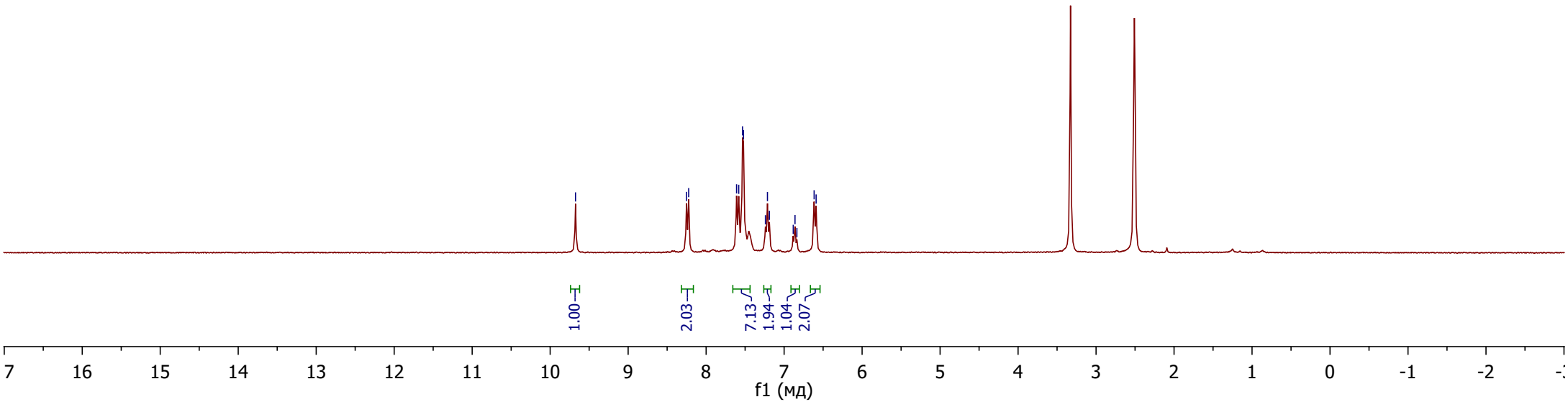
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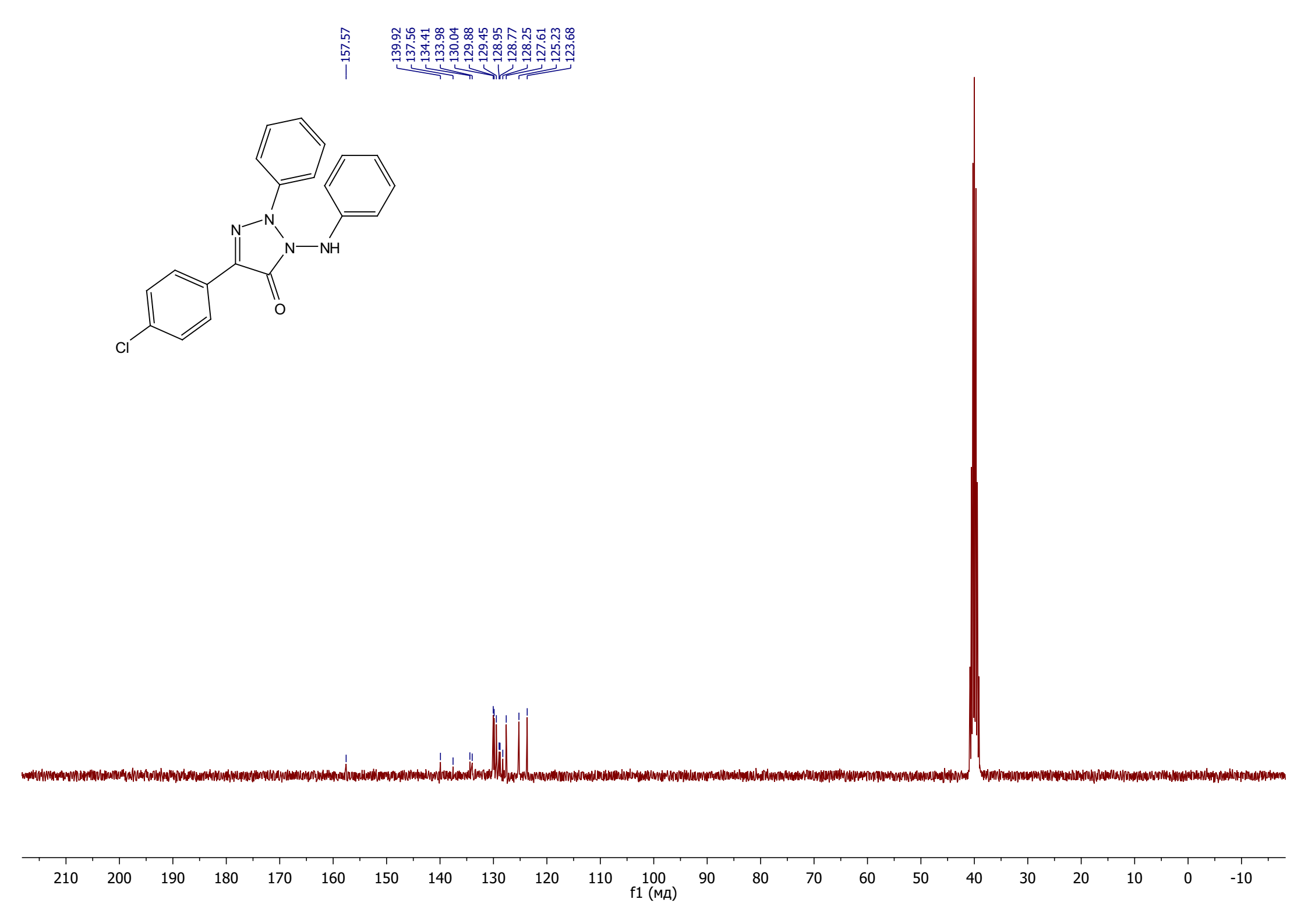
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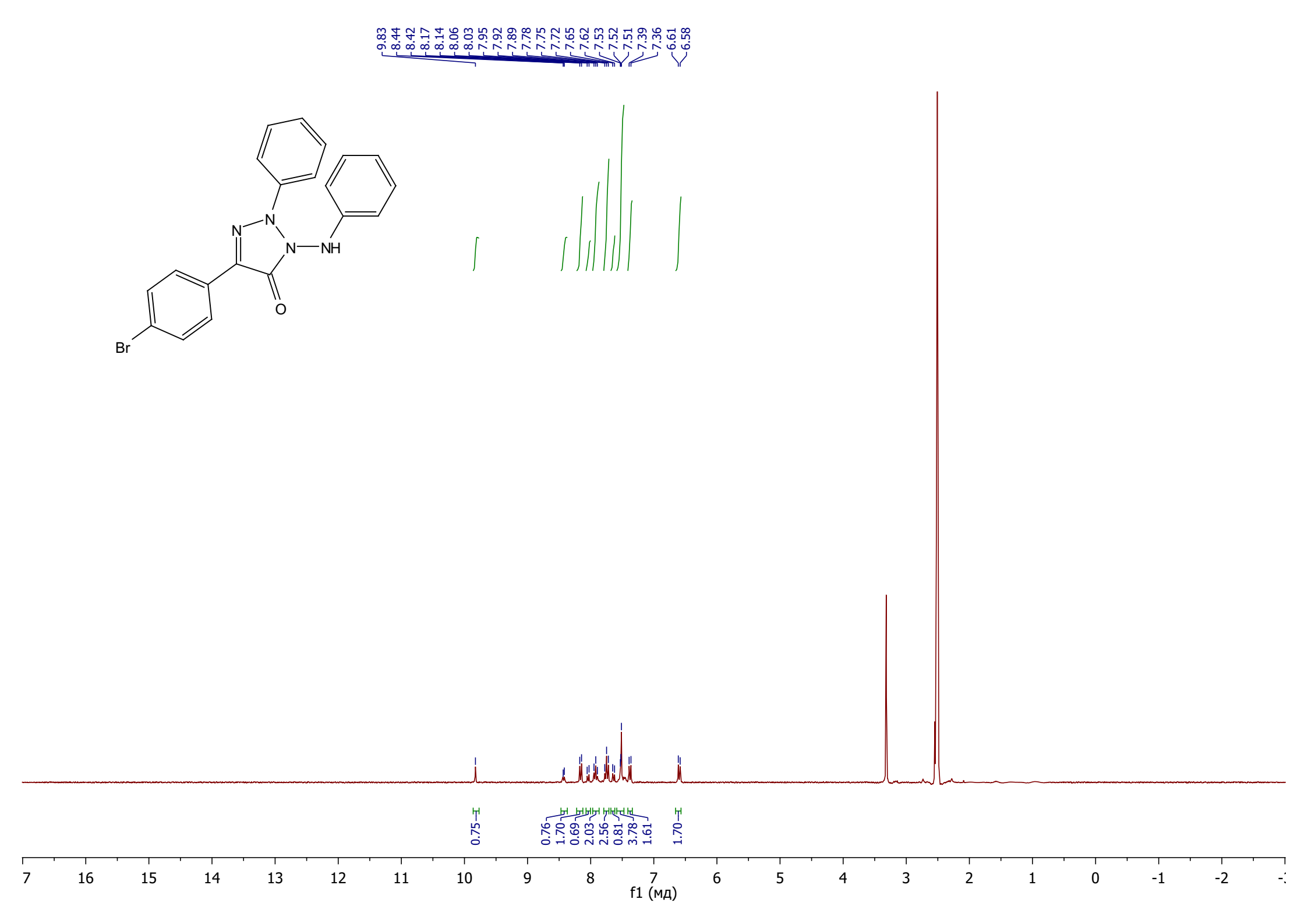
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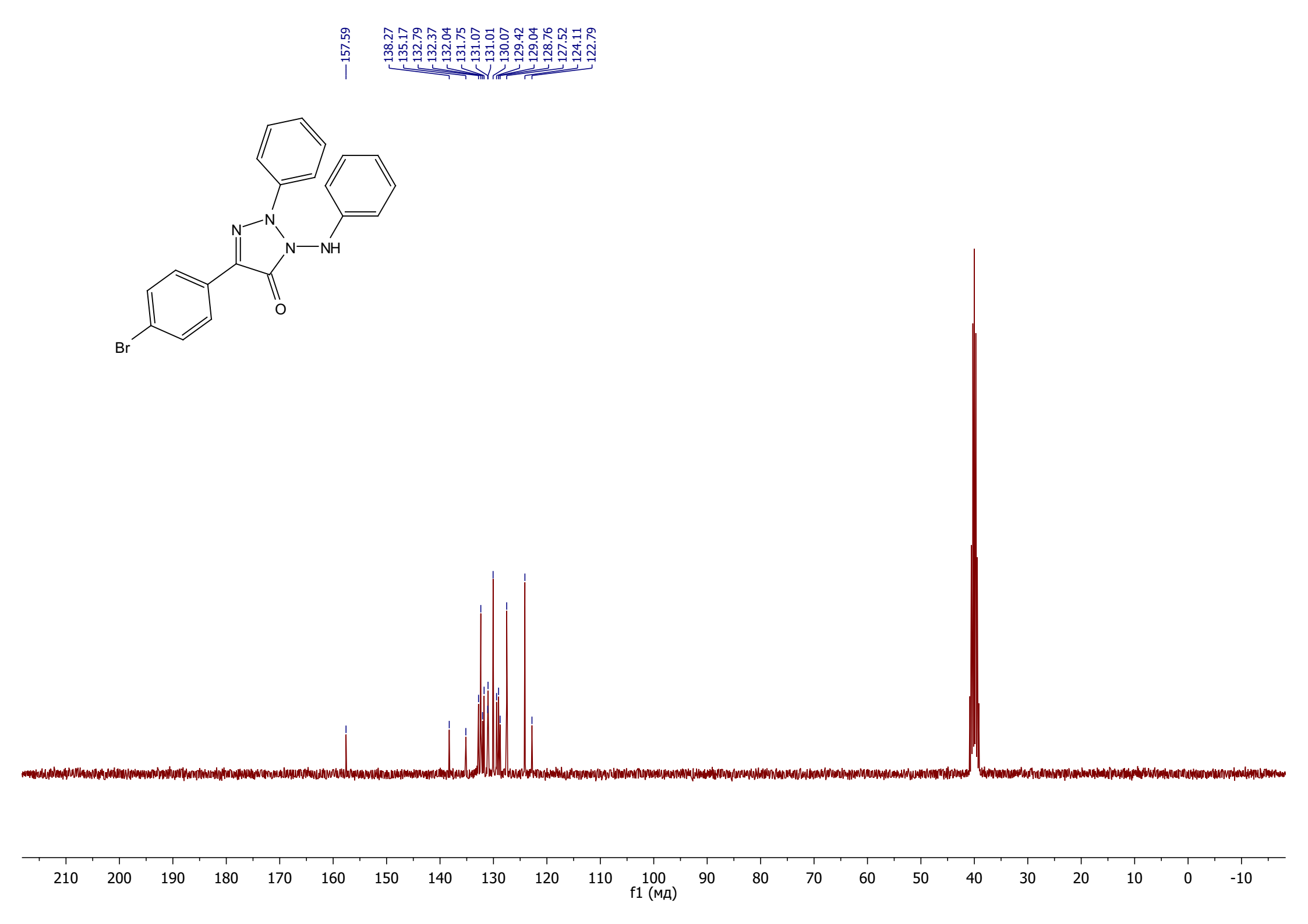
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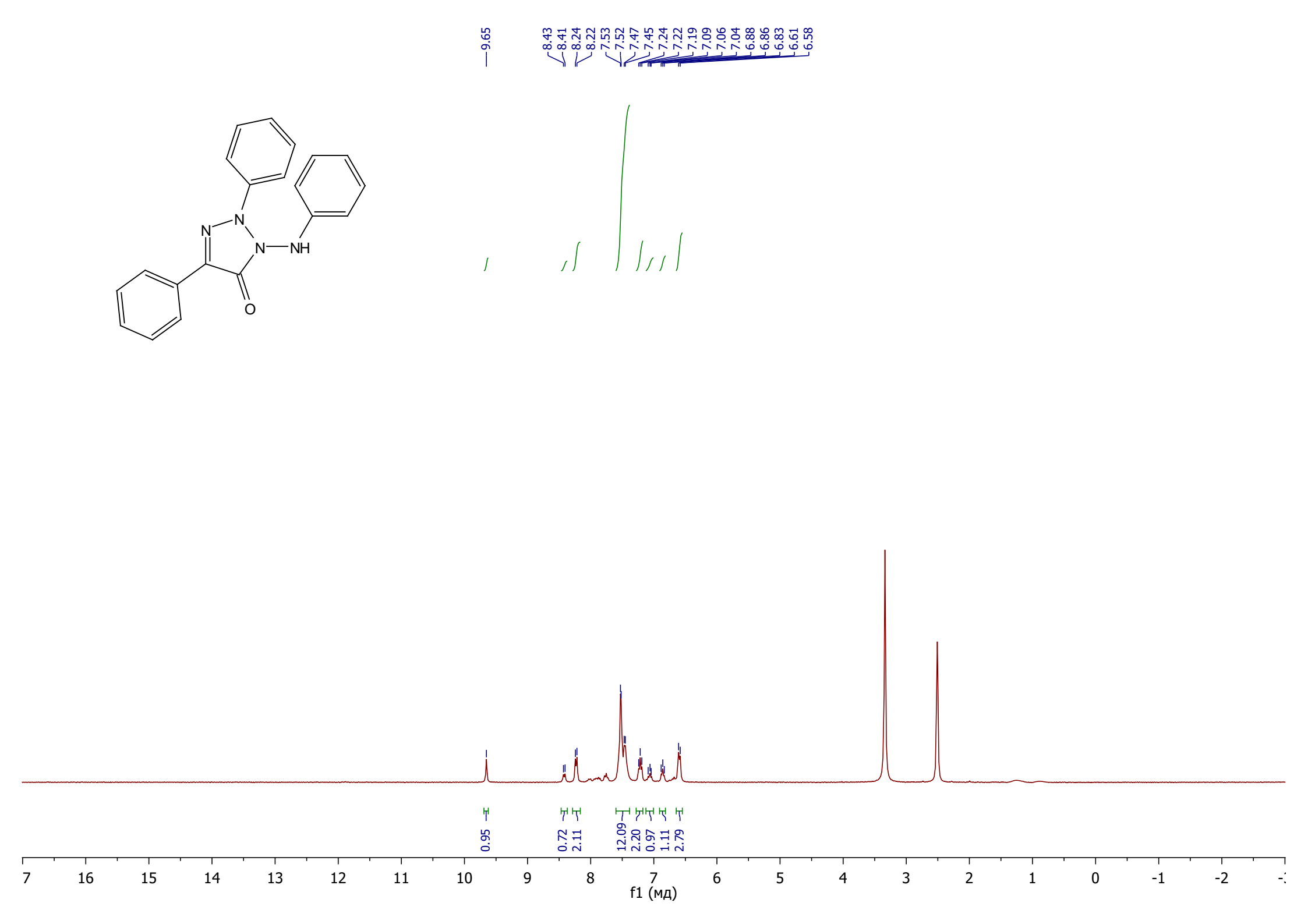
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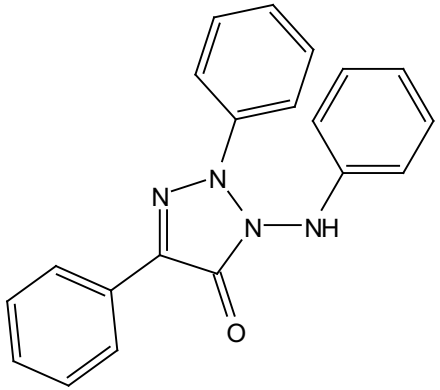

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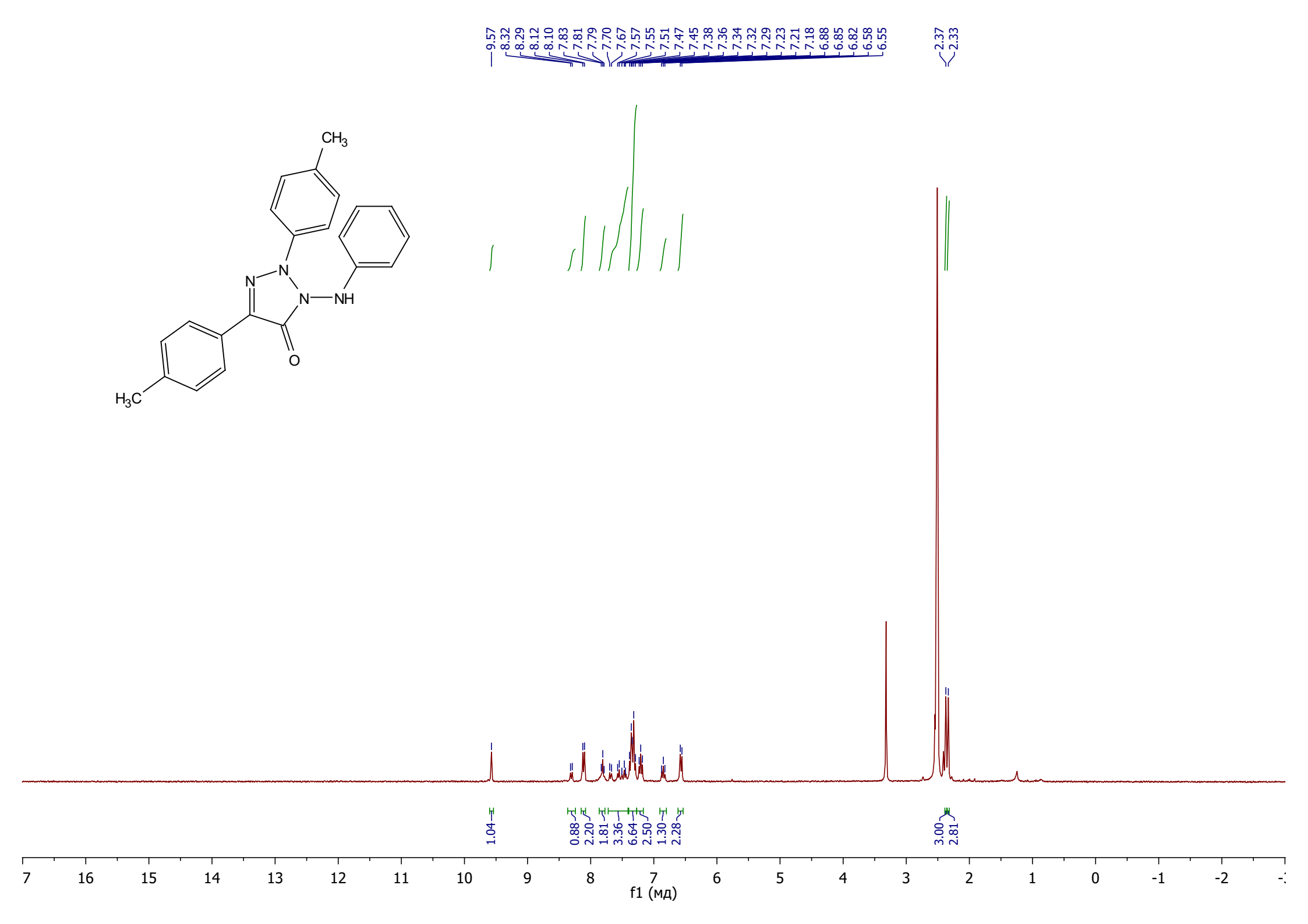
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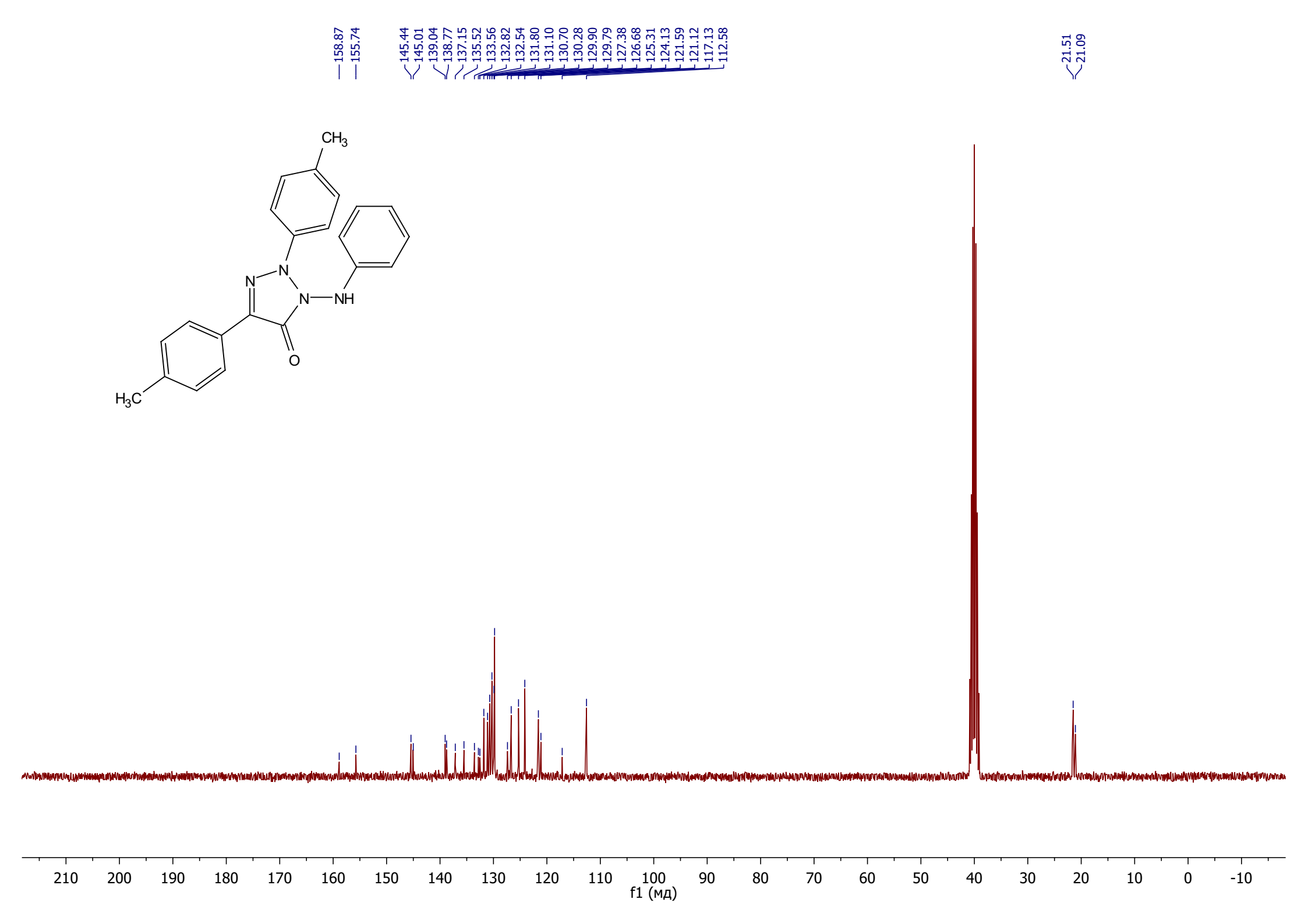
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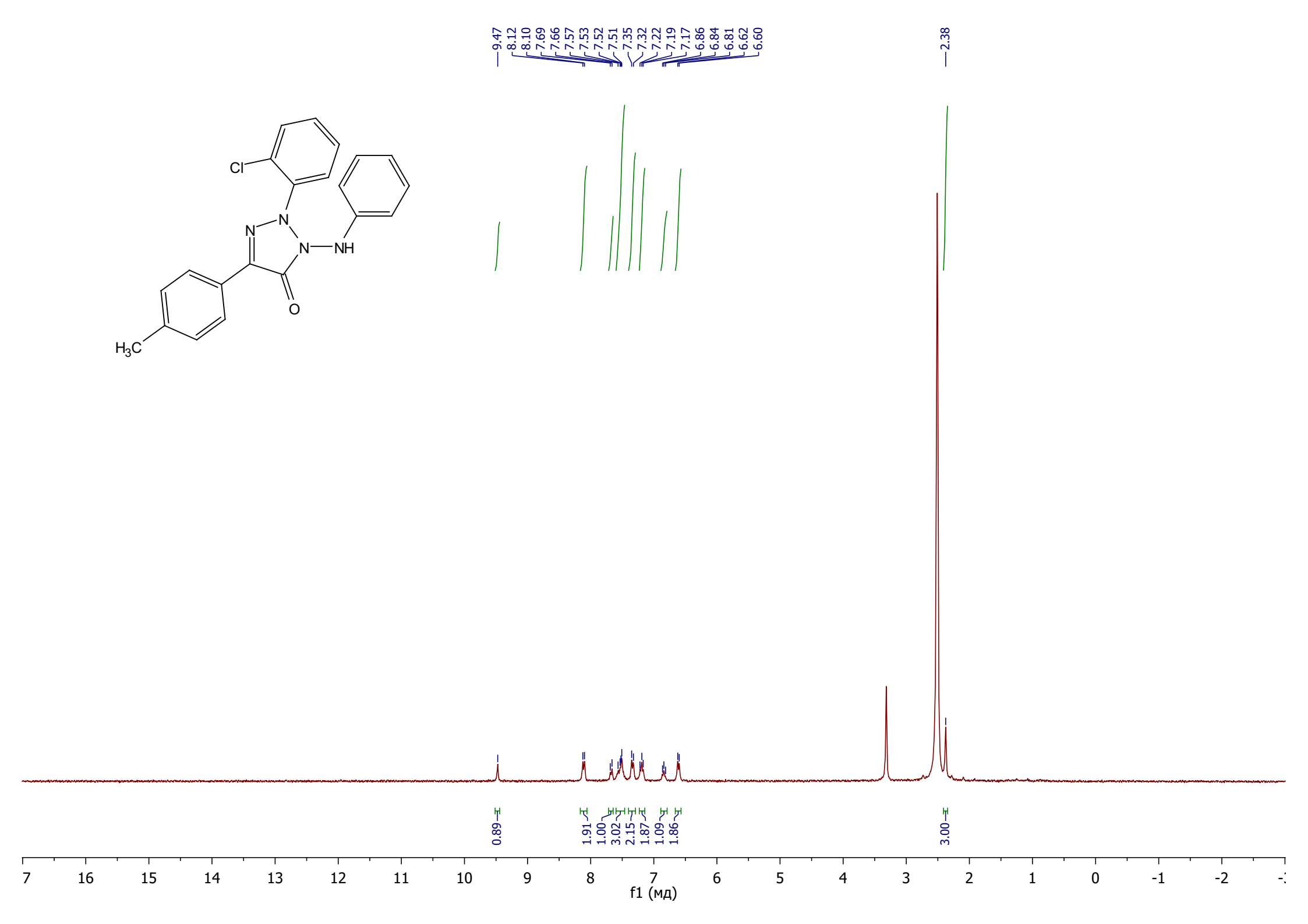
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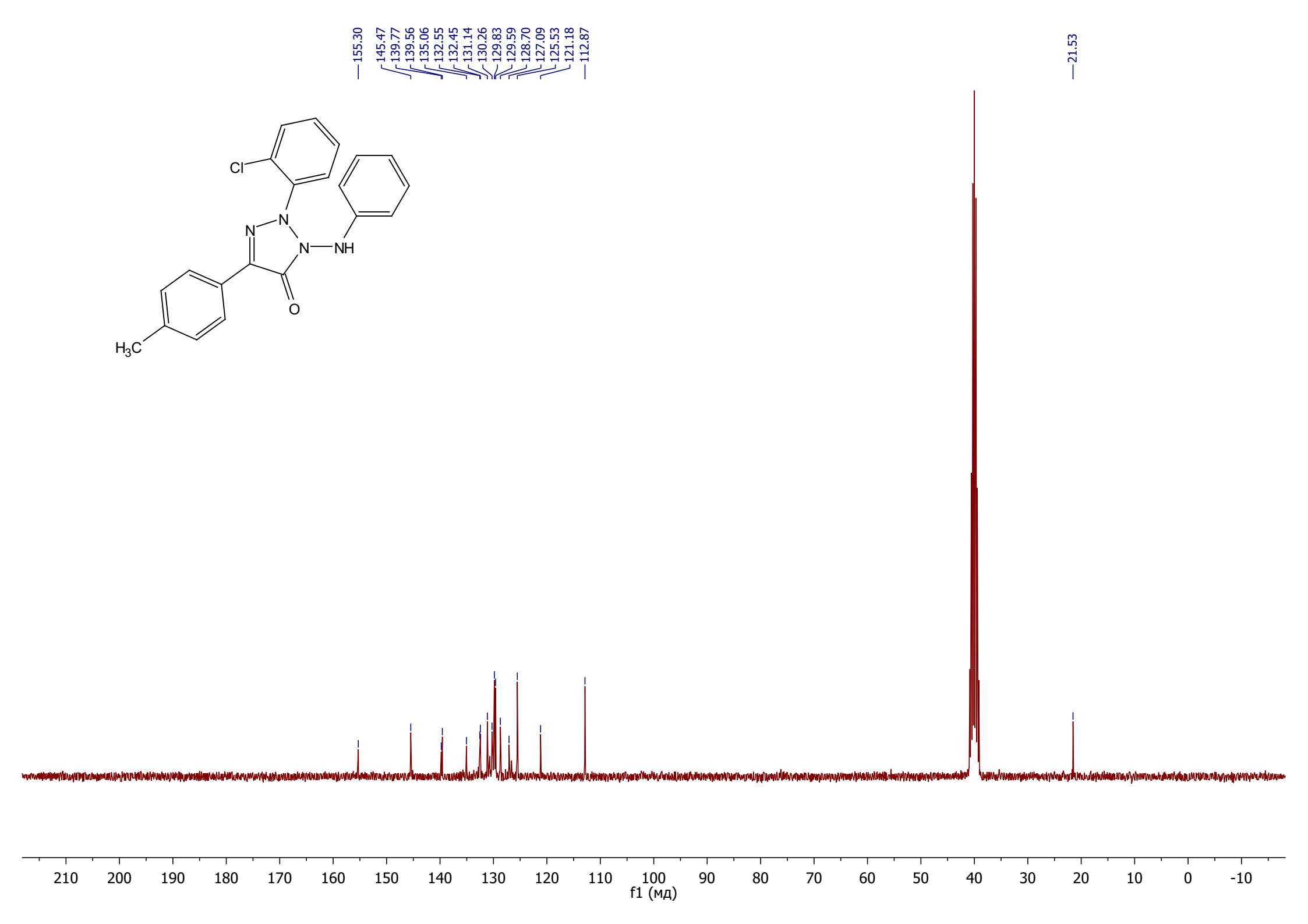
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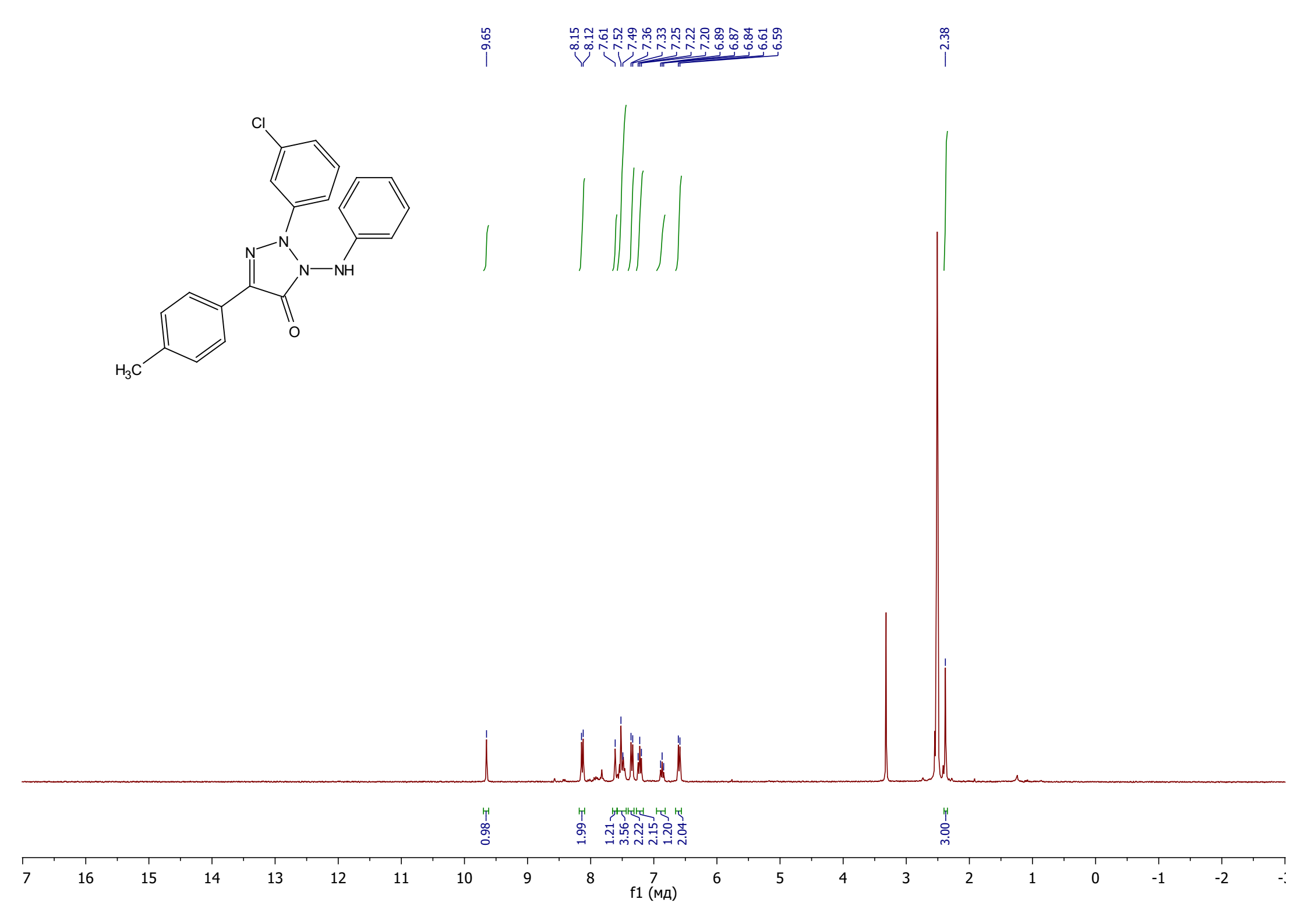
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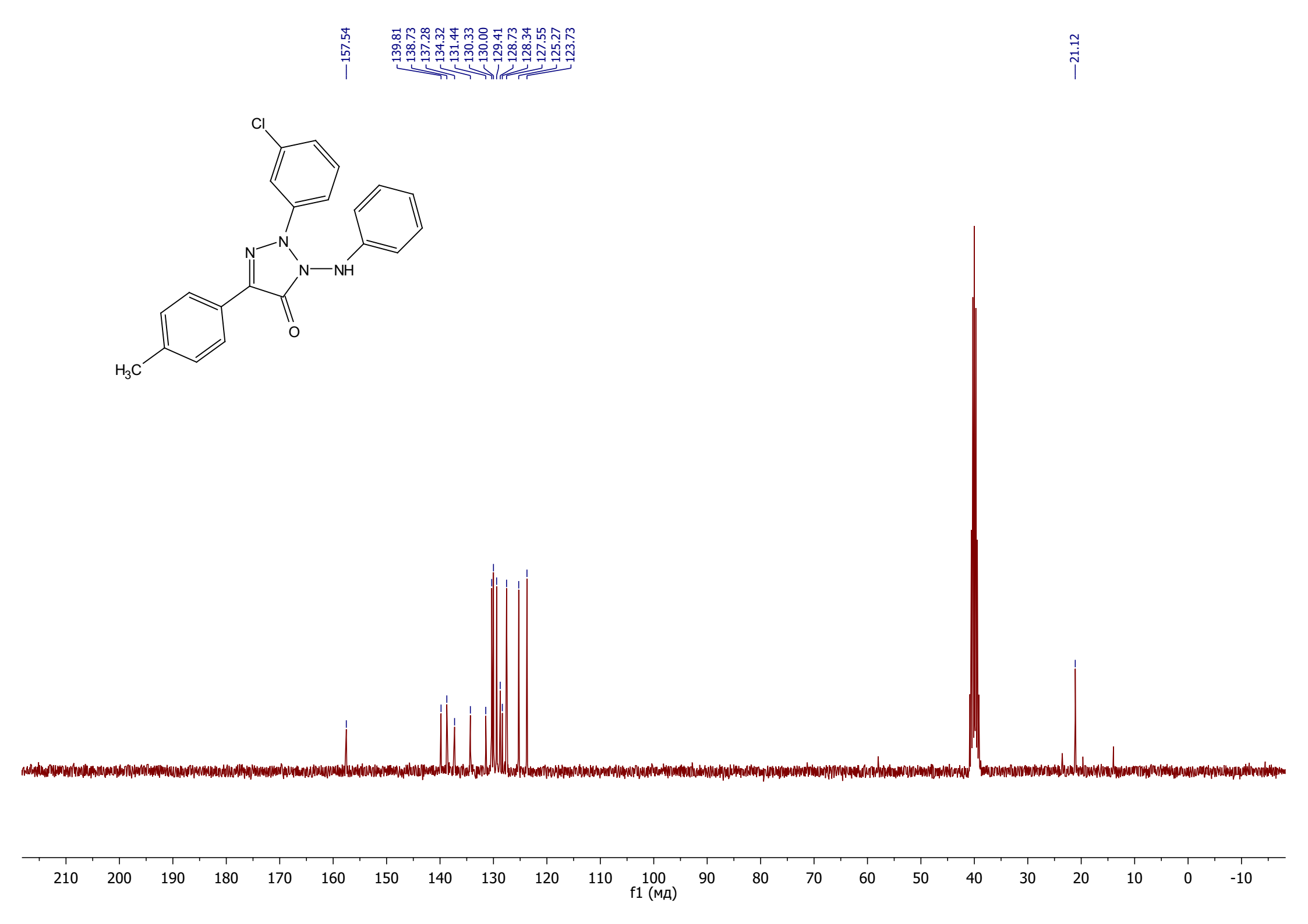
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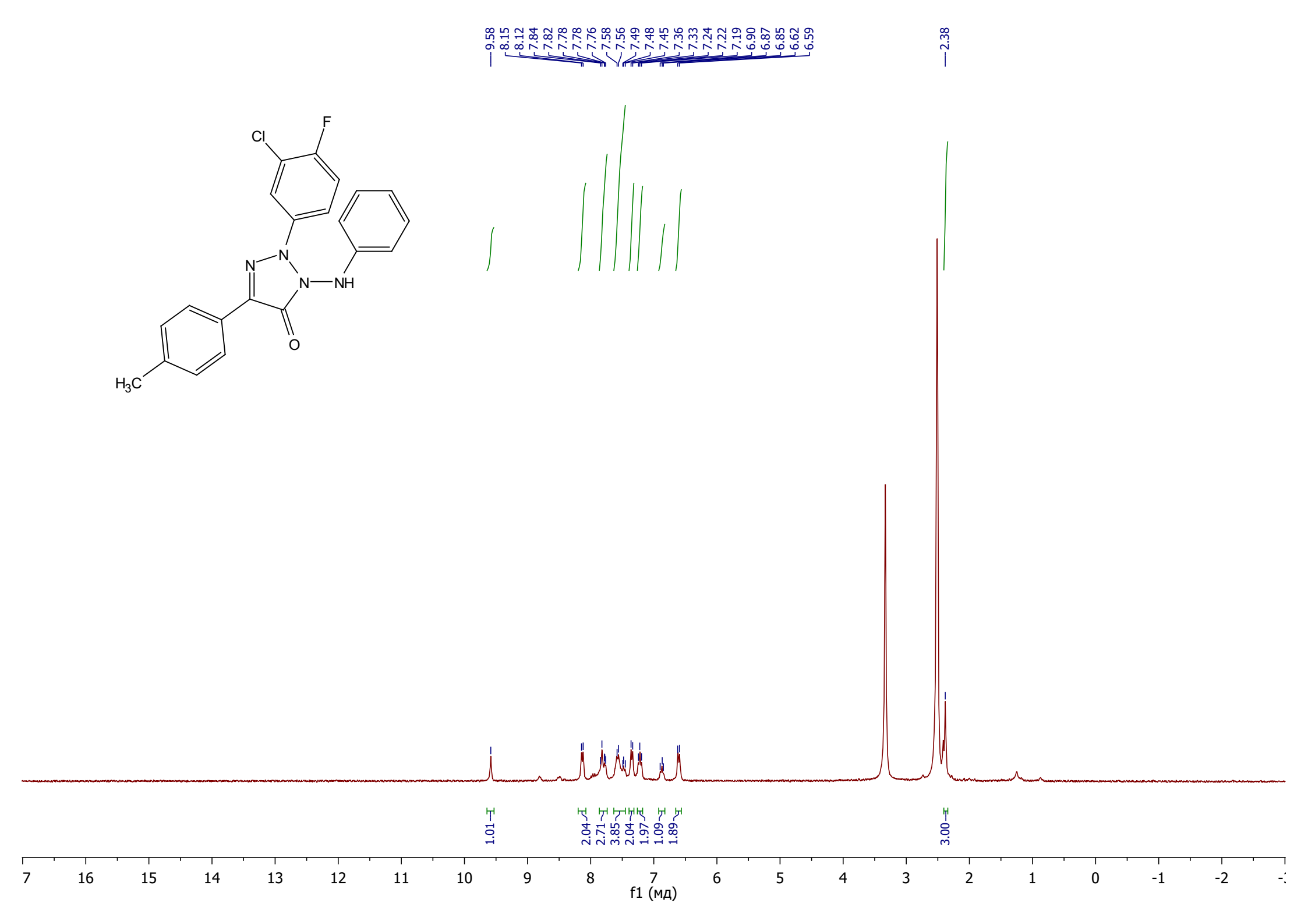
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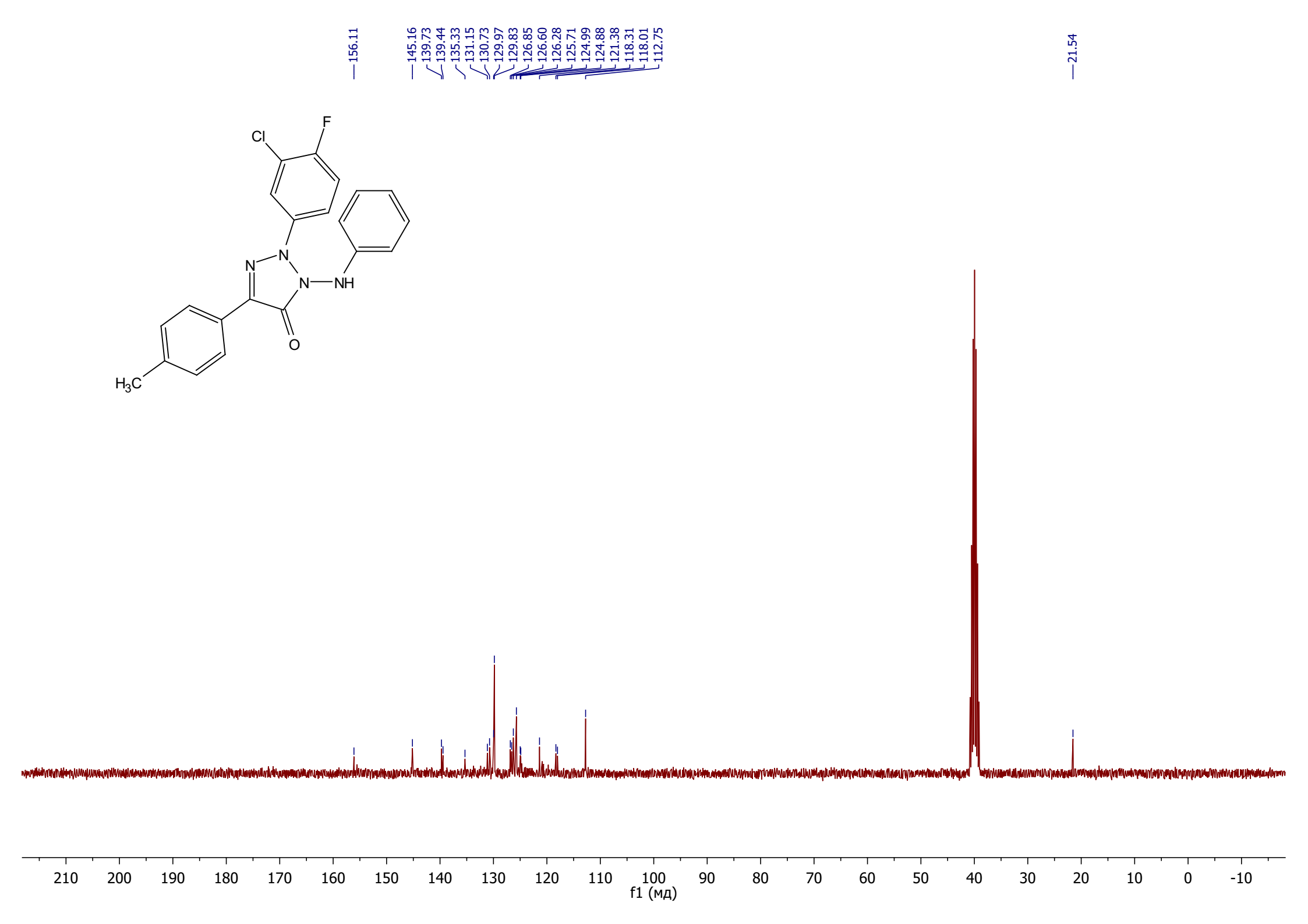
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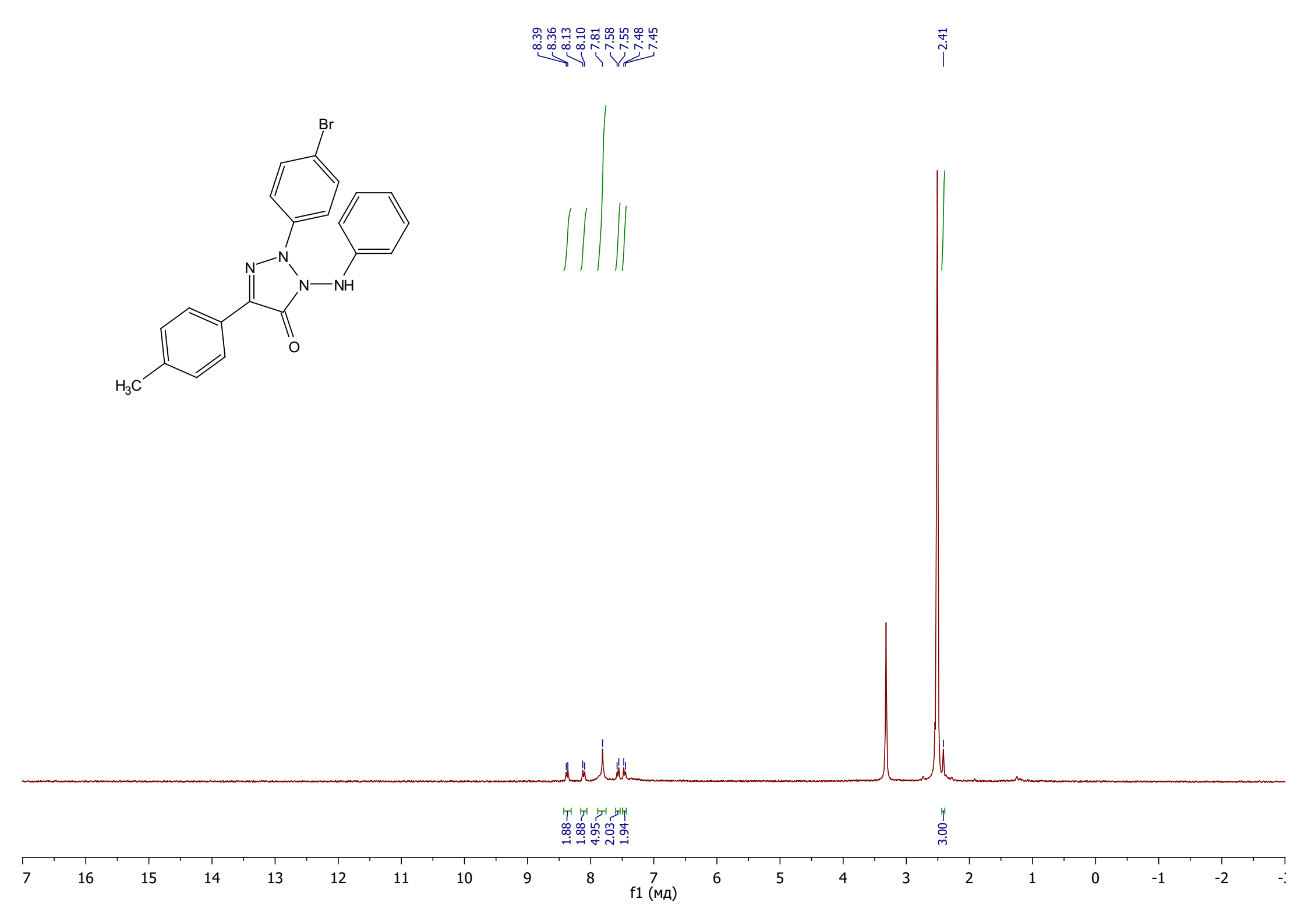
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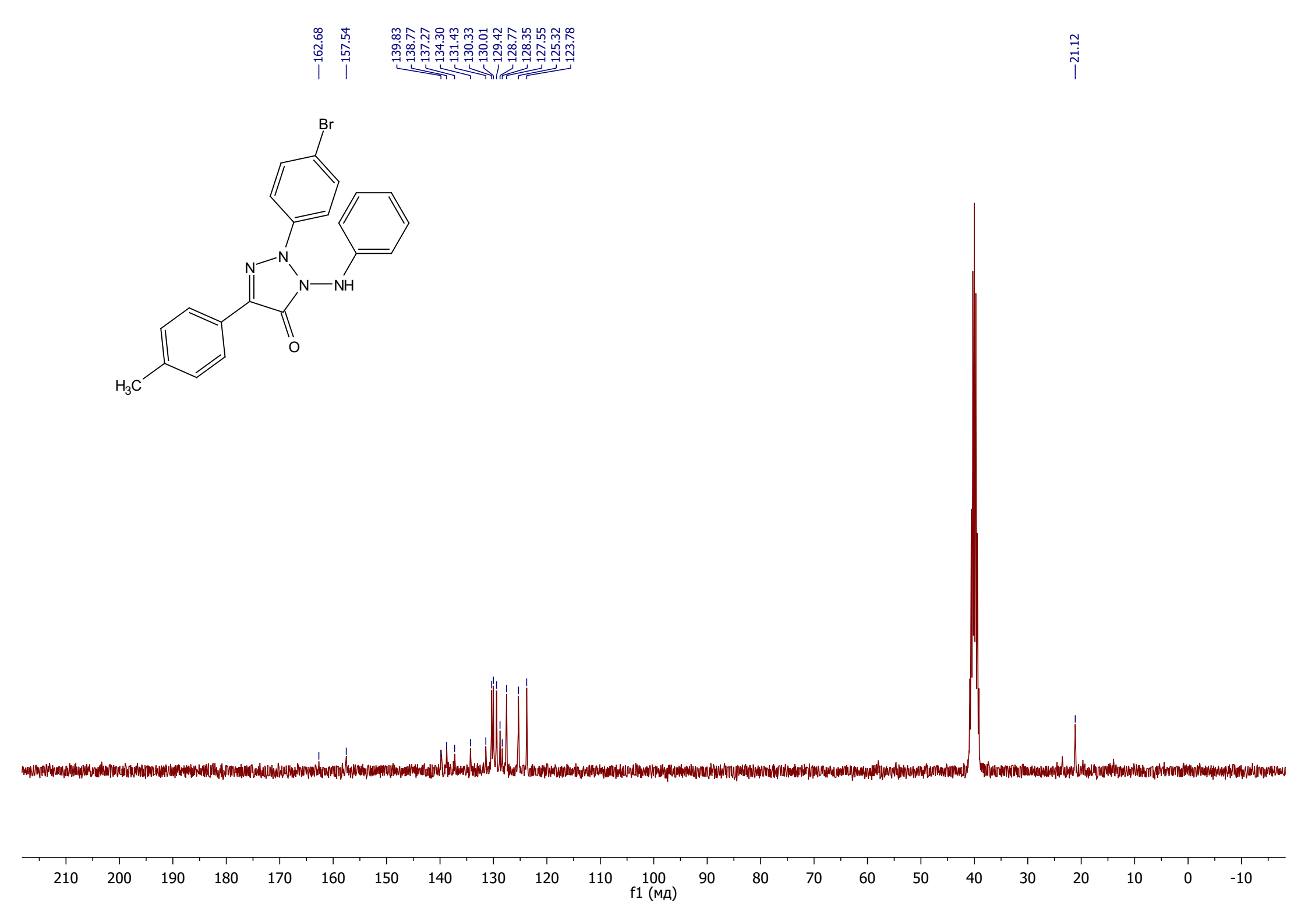
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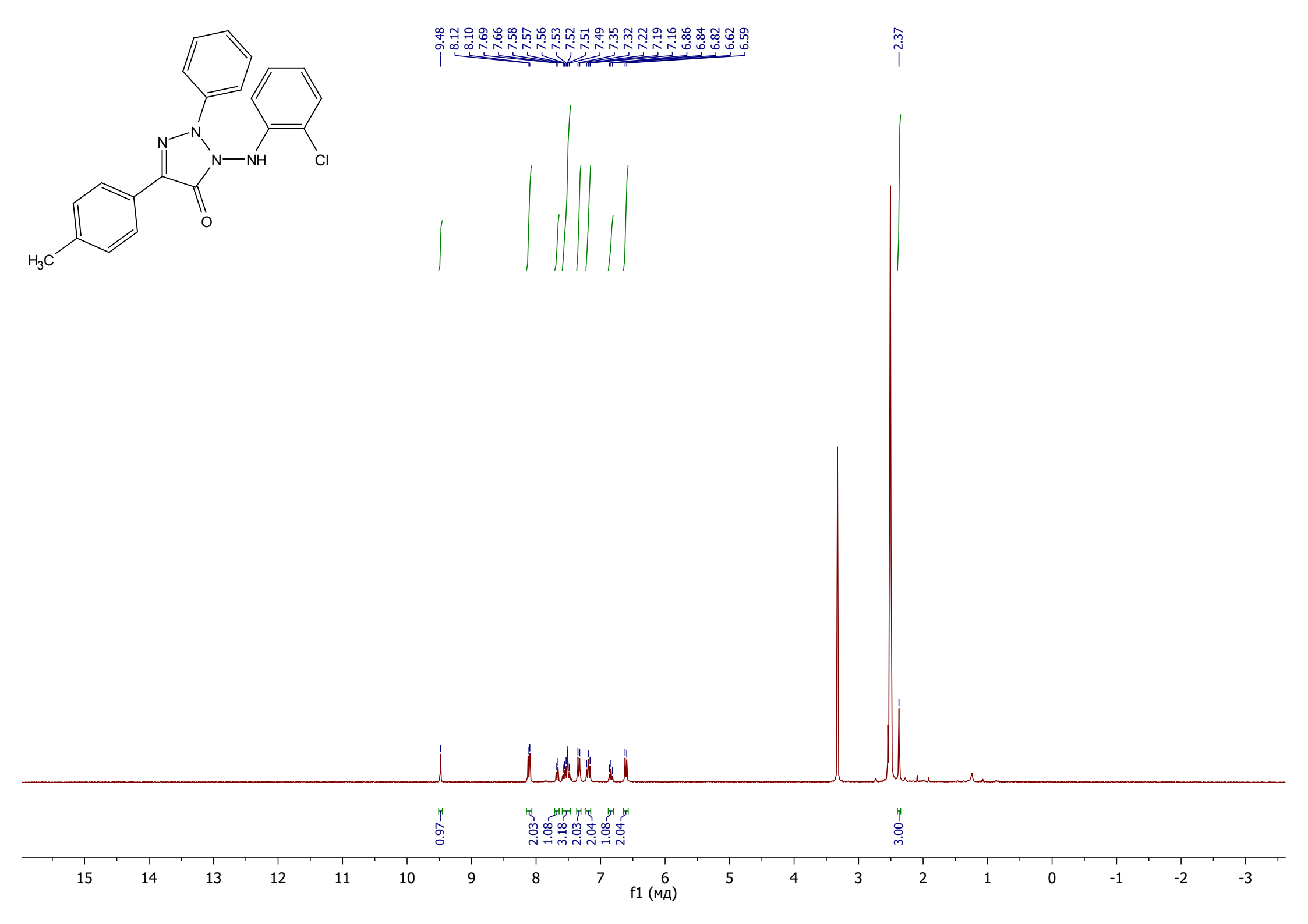
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1.08
2.04
3.00
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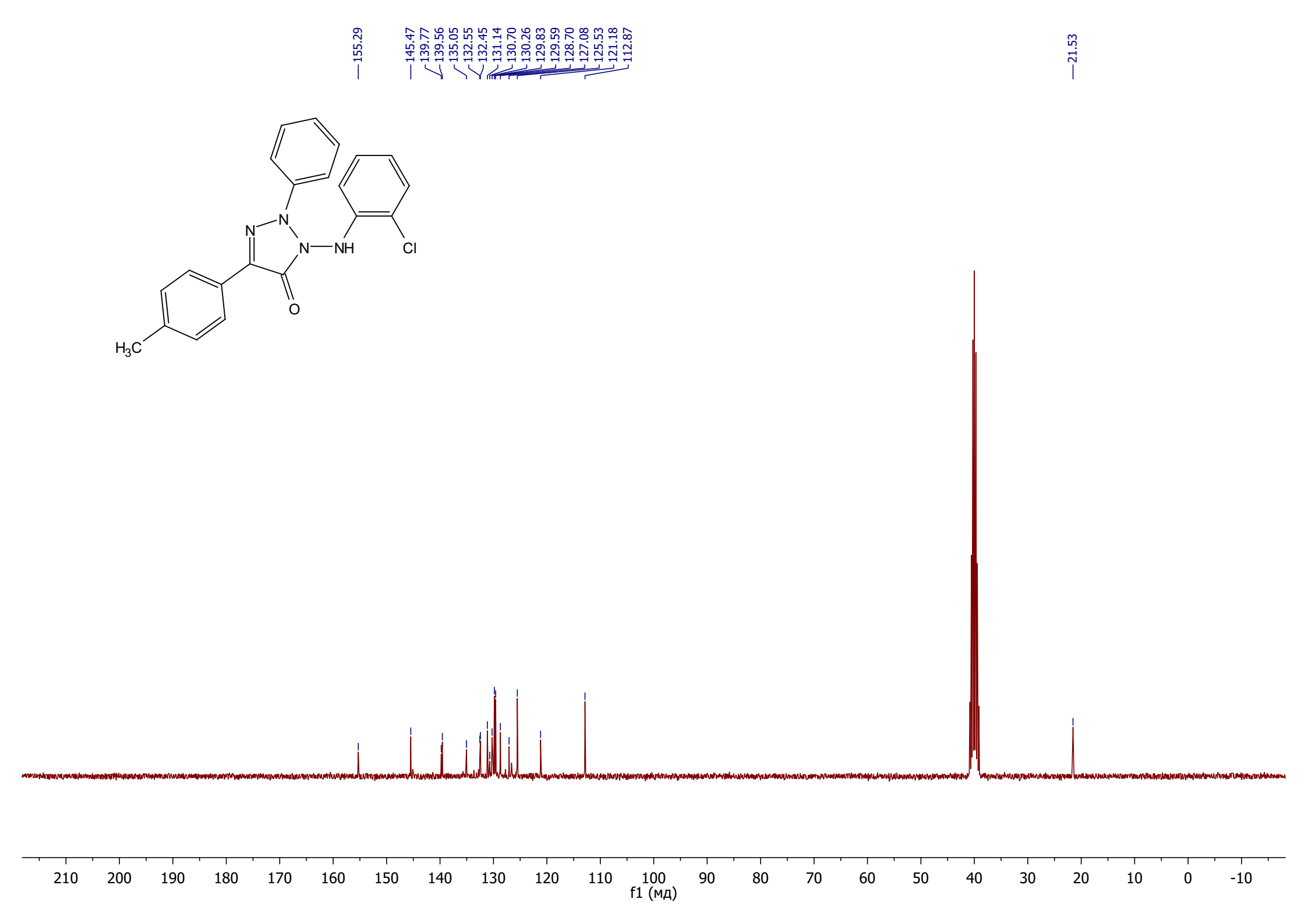
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127.08
125.53
121.18
112.87
21.53
N
N
N
NH
Cl
O
H3C
210
200
190
180
170
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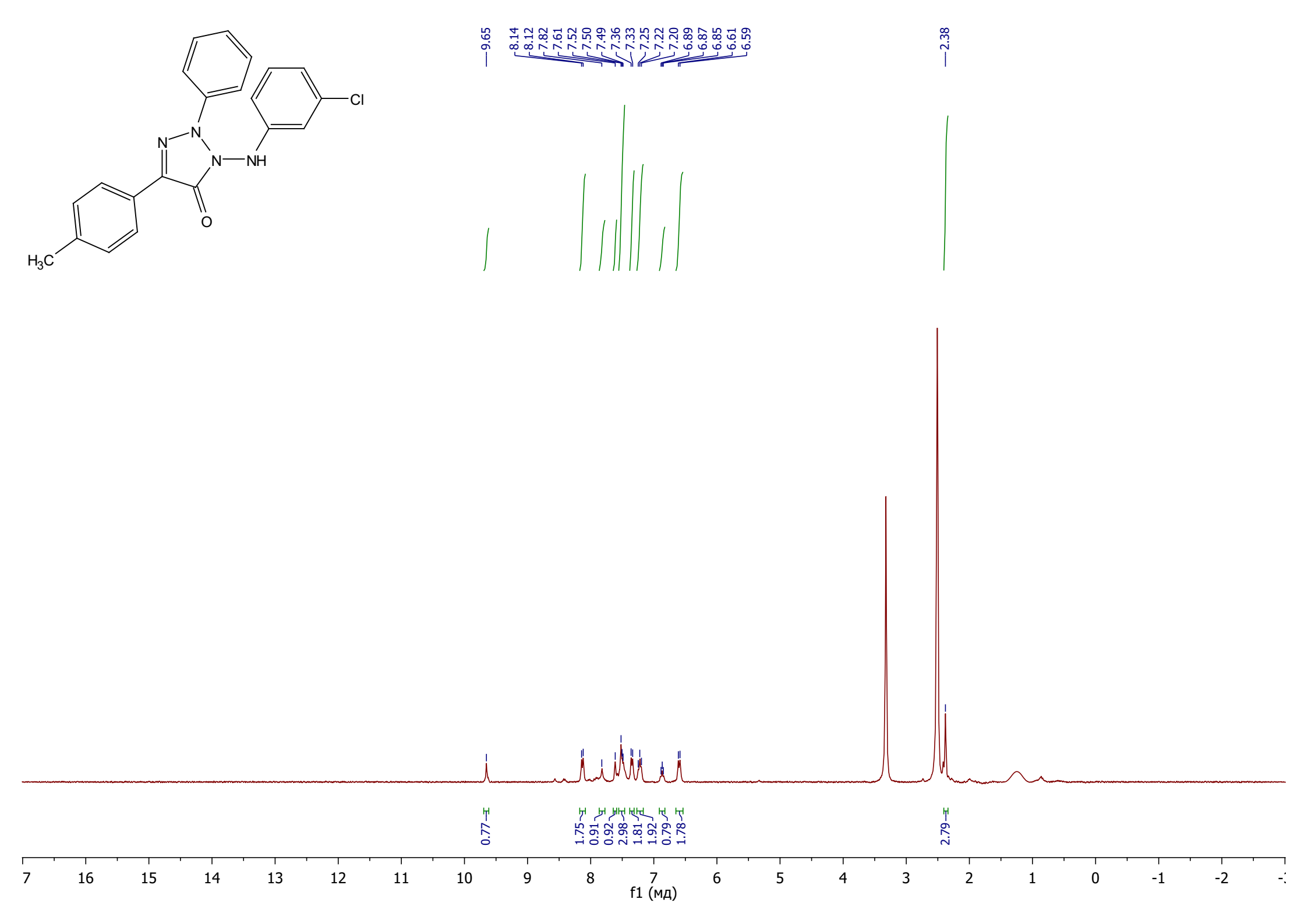
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6.61
6.59
2.38
Cl
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N
N
NH
O
H3C
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2.98
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1.92
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2.79
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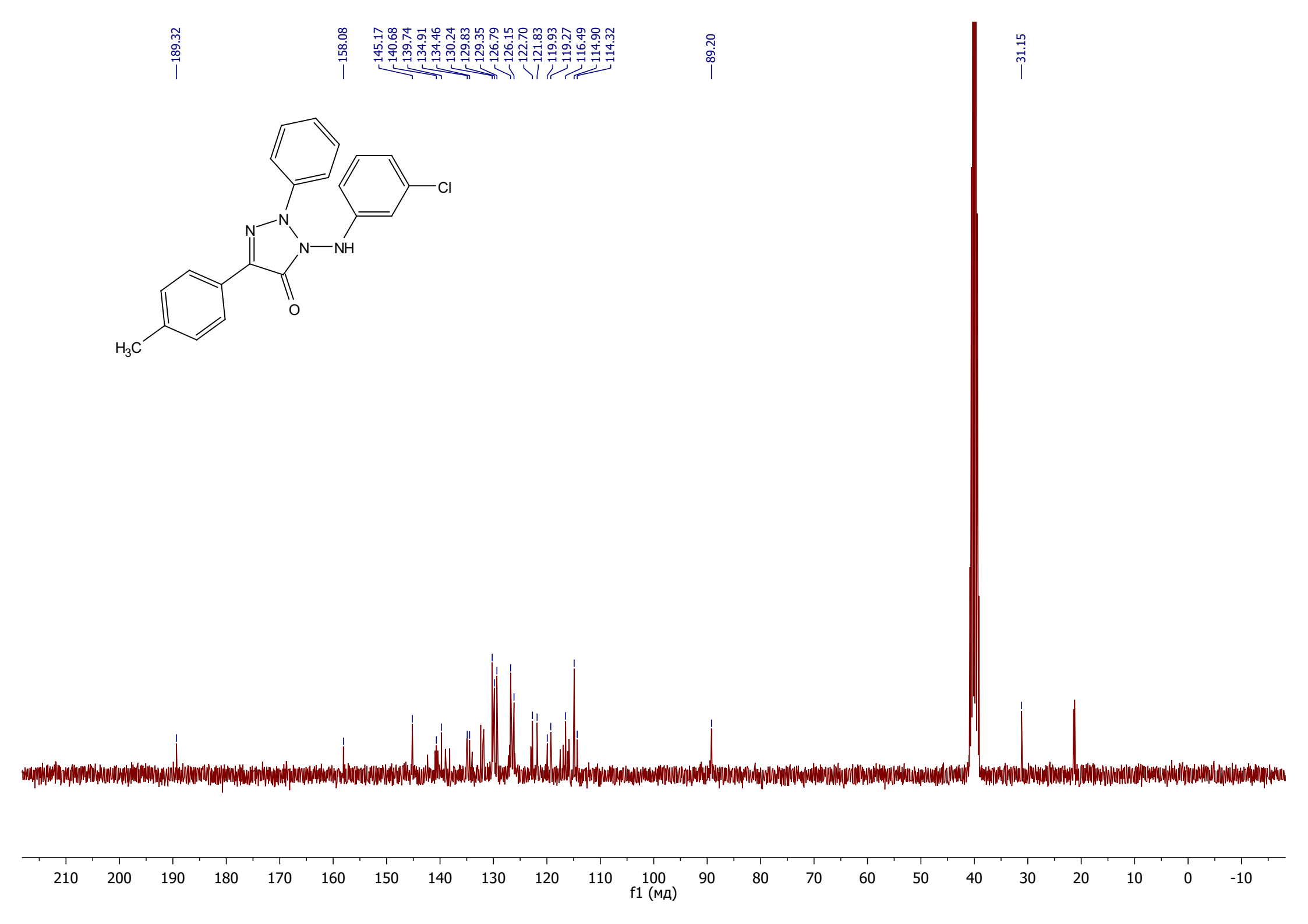
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119.93
119.27
116.49
114.90
114.32
89.20
31.15
Cl
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N
N
NH
O
H3C
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200
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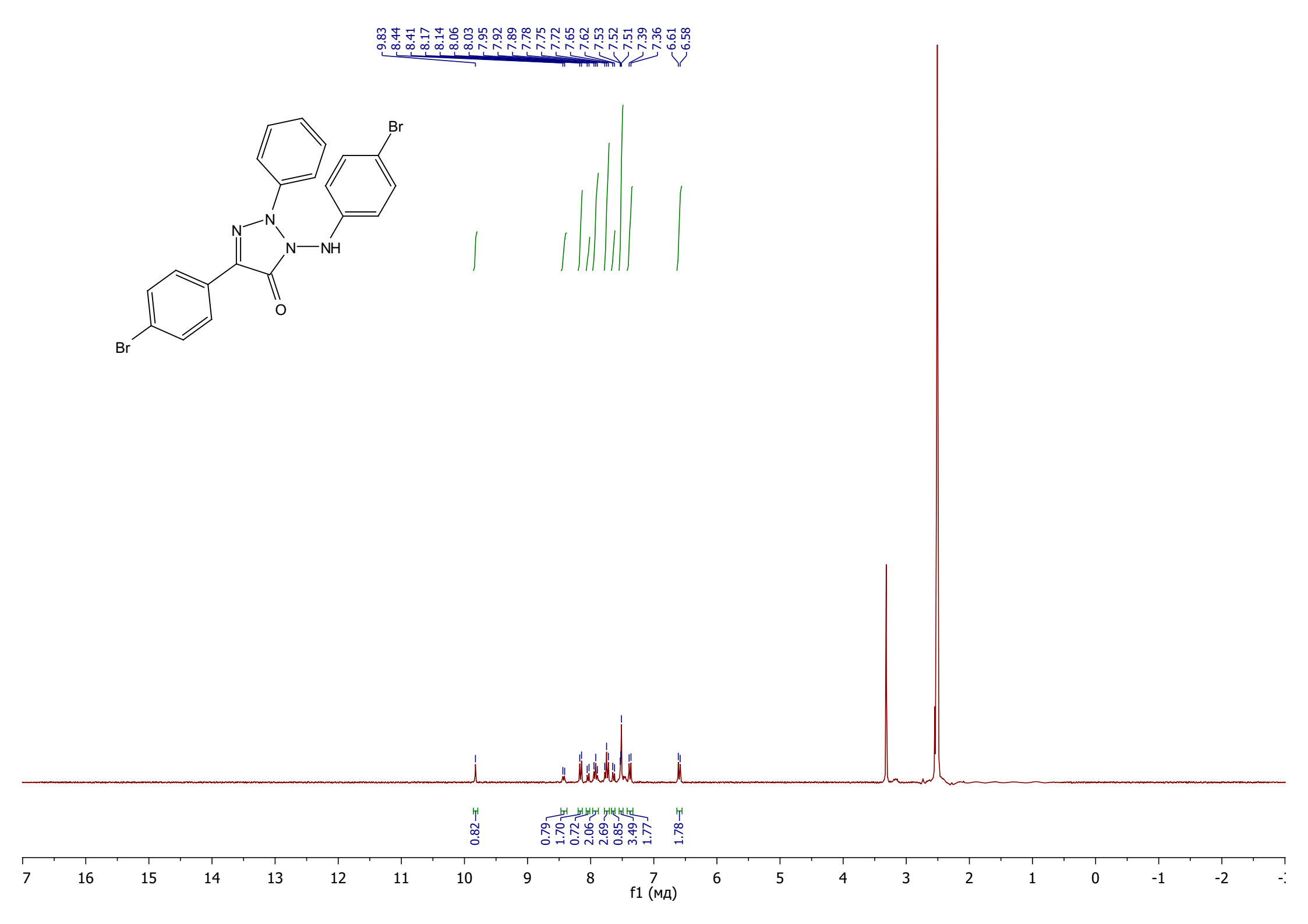
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7.51
7.39
7.36
6.61
6.58
Br
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N
N
NH
O
Br
0.82
0.79
1.70
0.72
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3.49
1.77
1.78
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16
15
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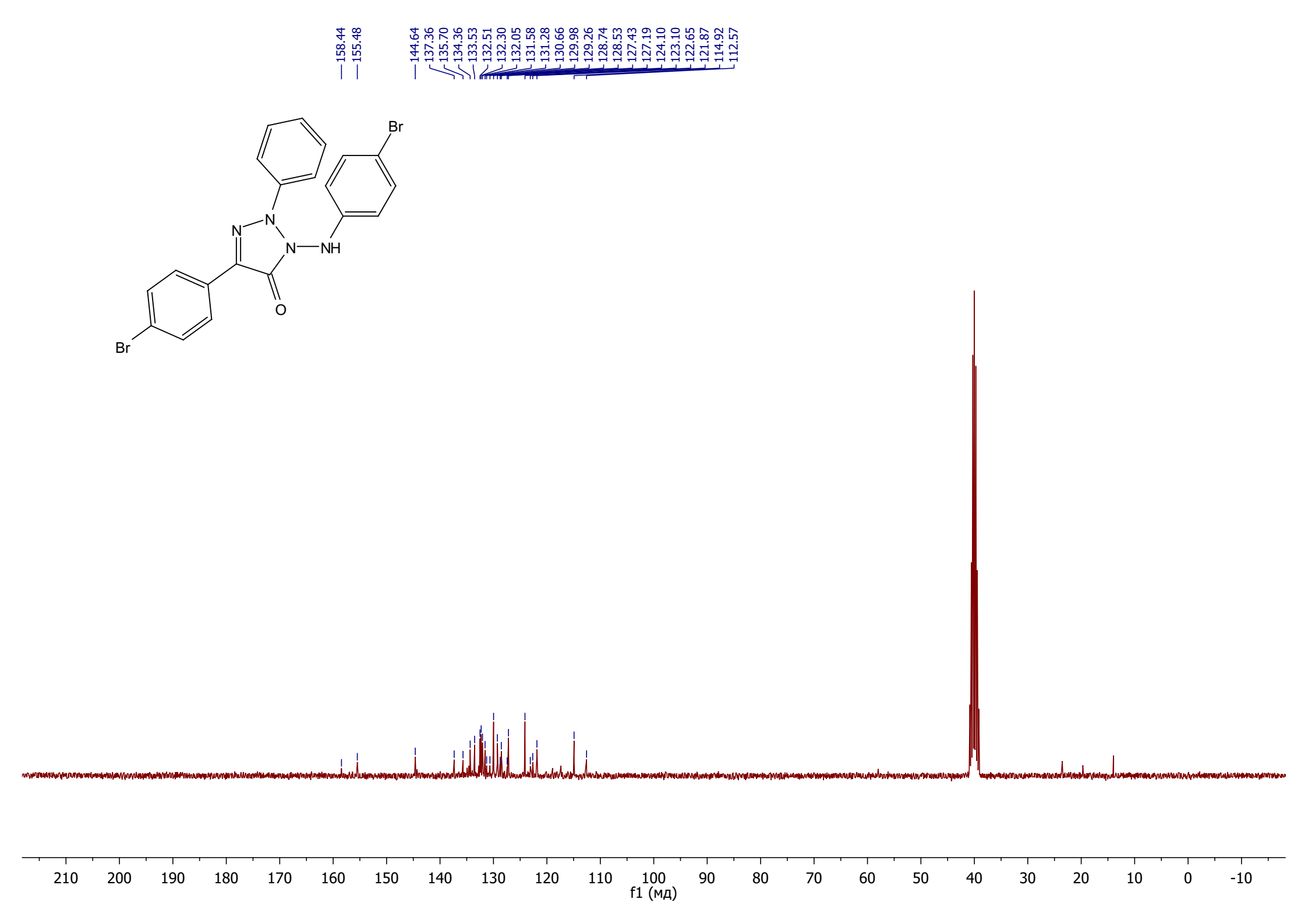
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127.19
124.10
123.10
122.65
121.87
114.92
112.57
Br
N
N
N
NH
O
Br
210
200
190
180
170
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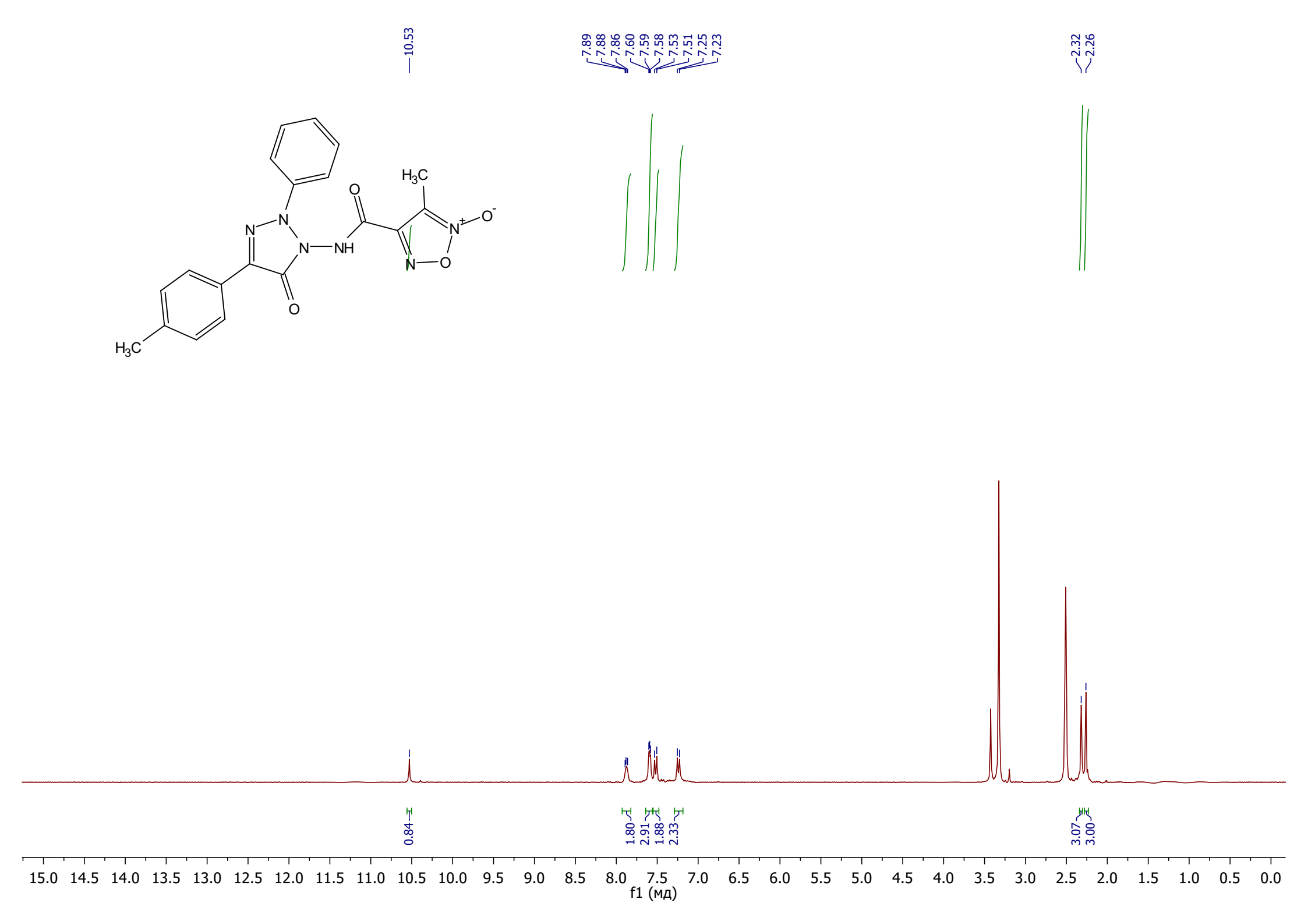
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2.26
H3C
O
N
N
N
NH
N+
O-
N
O
O
H3C
0.84
1.80
2.91
1.88
2.33
3.07
3.00
15.0 14.5 14.0 13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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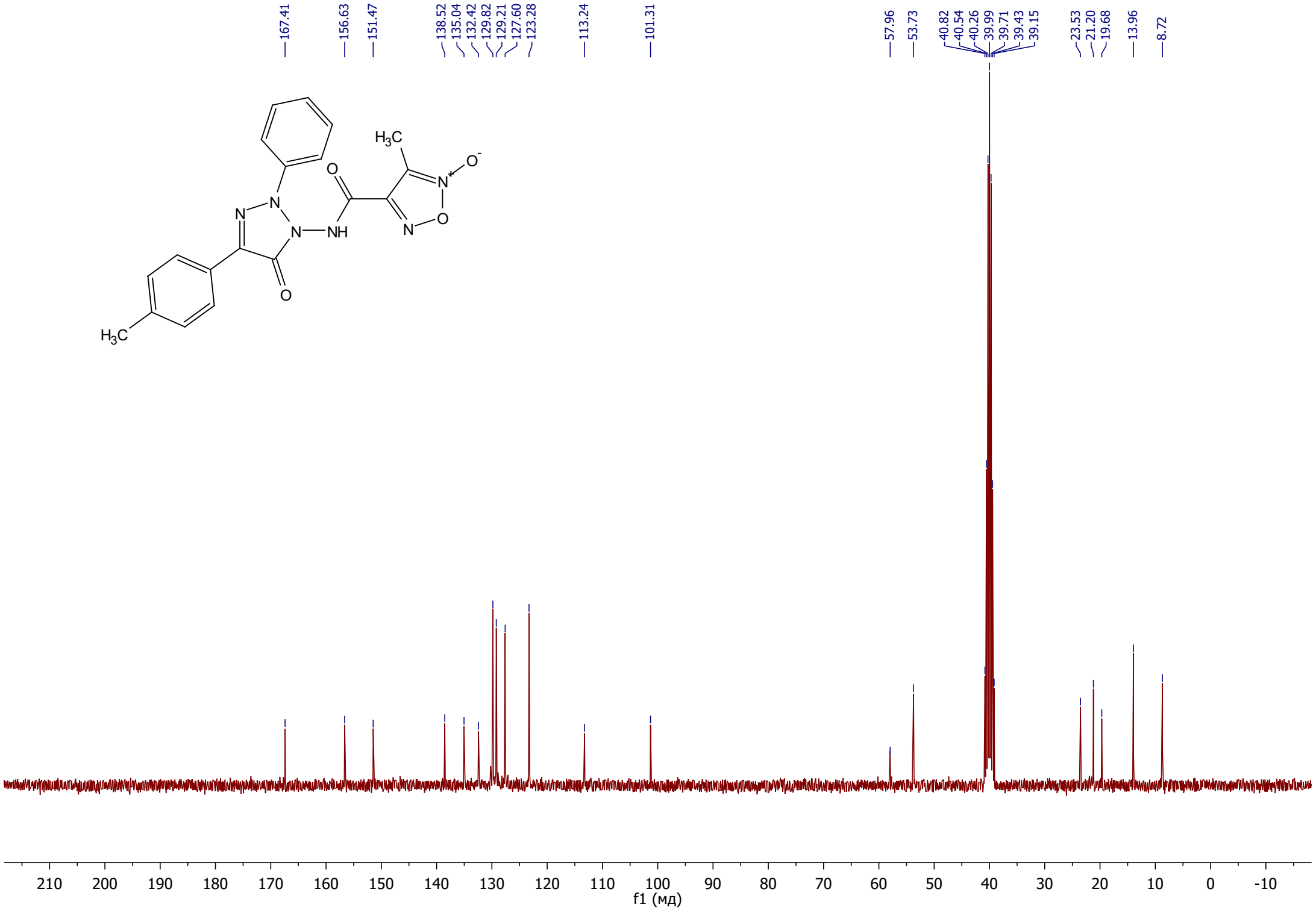
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40.54
40.26
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39.71
39.43
39.15
23.53
21.20
19.68
13.96
8.72
H3C
O
N
N
N
NH
O
N+
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H3C
210
200
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160
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-10
f1 (мд)