Supplementary Information

Synthesis of 1,2,4-oxadiazoles by tandem reaction of nitroalkenes with arenes and nitriles in the superacidTfOH

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1. Copies of ^{1}H, ^{13}C, ^{19}F NMR, and IR spectra of compounds2

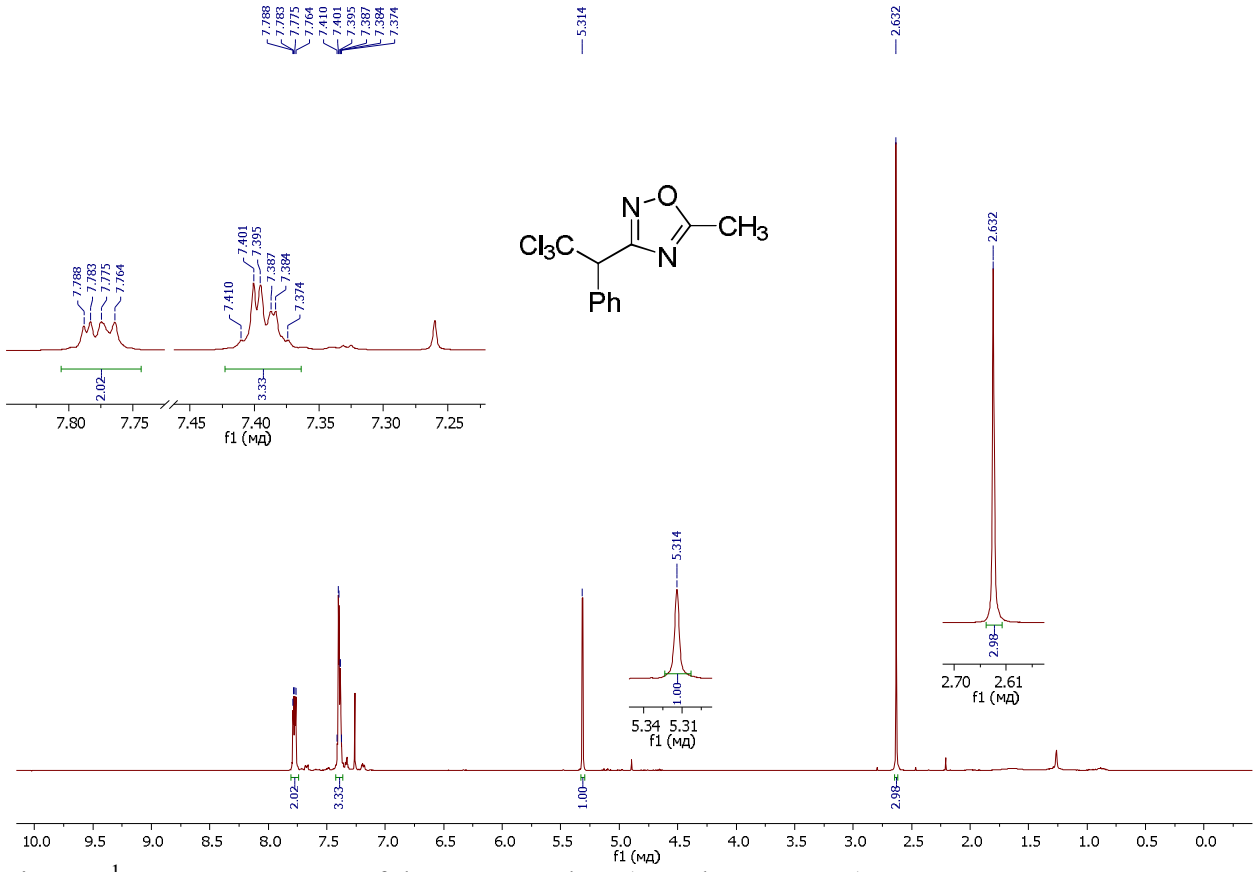

Fig. S1. ^{1}H NMR spectrum of the compound **2a** ($CDCl_3$, 400 MHz).

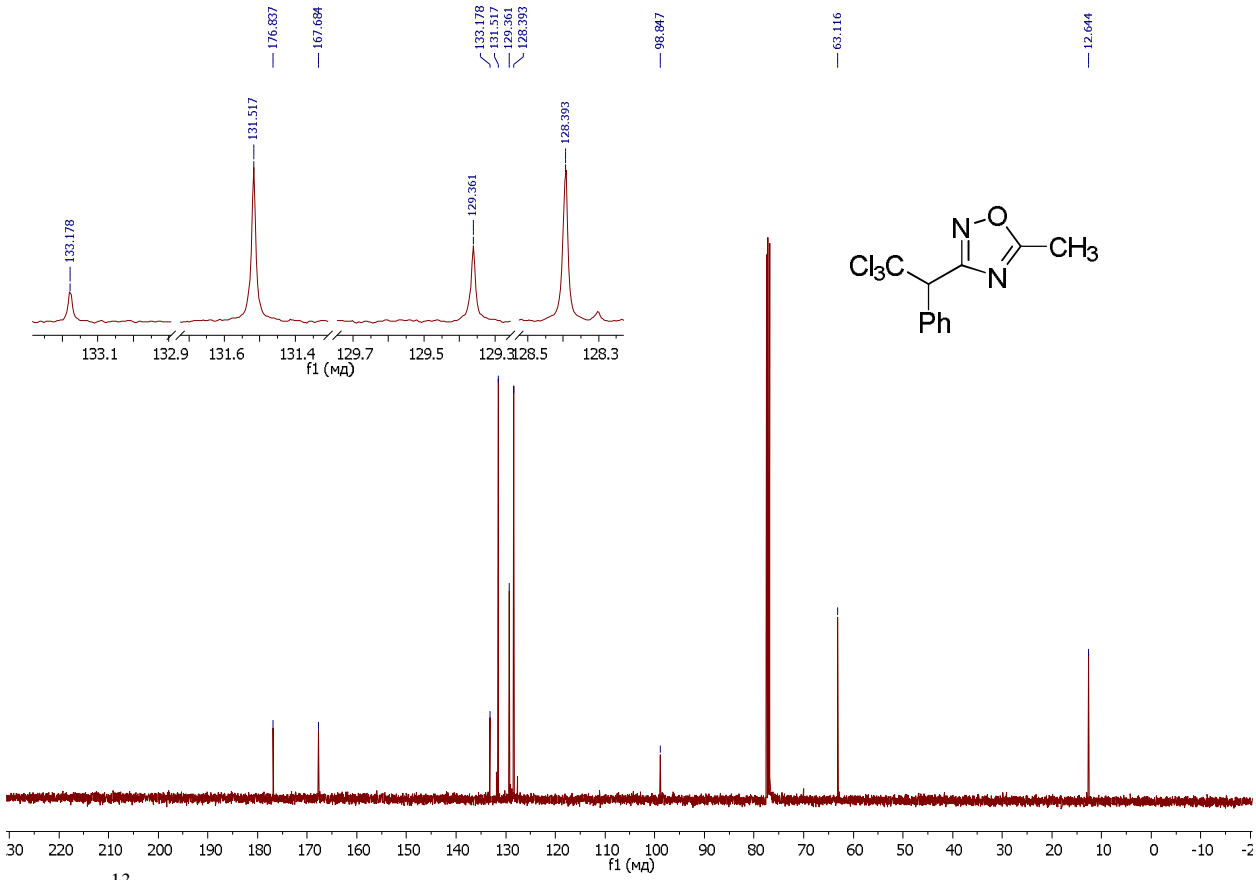

Fig. S2. $^{13}C\{H\}$ NMR spectrum of the compound **2a** ($CDCl_3$, 100 MHz).

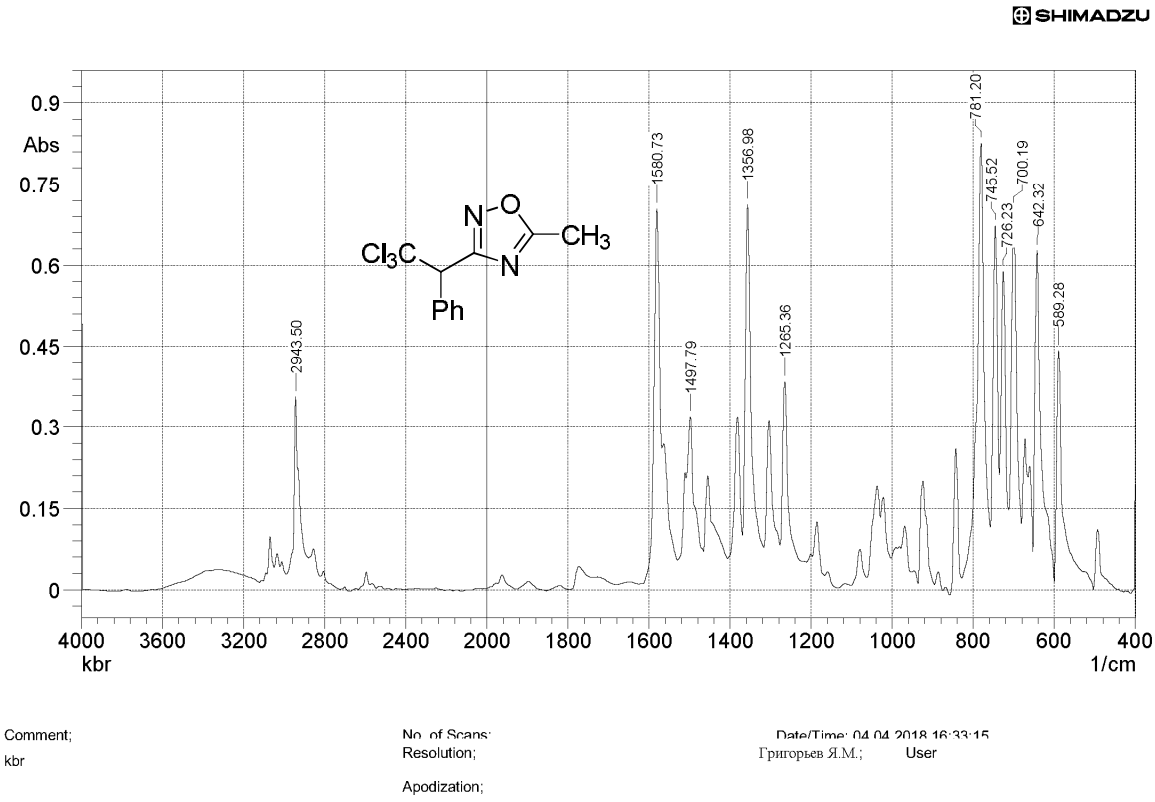

Fig. S3. ^{1}H NMR spectrum of the compound **2a** ($CDCl_3$, 400 MHz).

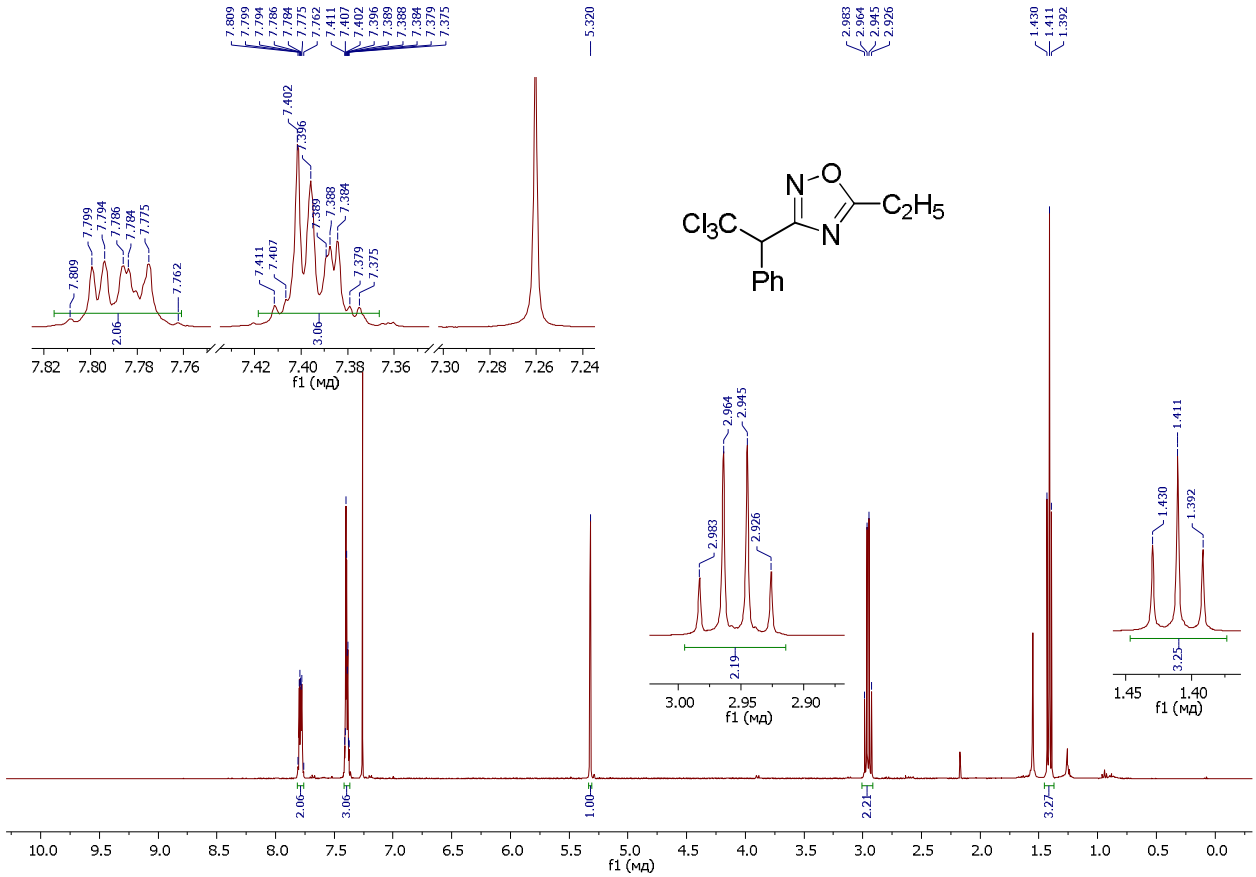

Fig. S4. ^{1}H NMR spectrum of the compound **2b** ($CDCl_3$, 400 MHz).

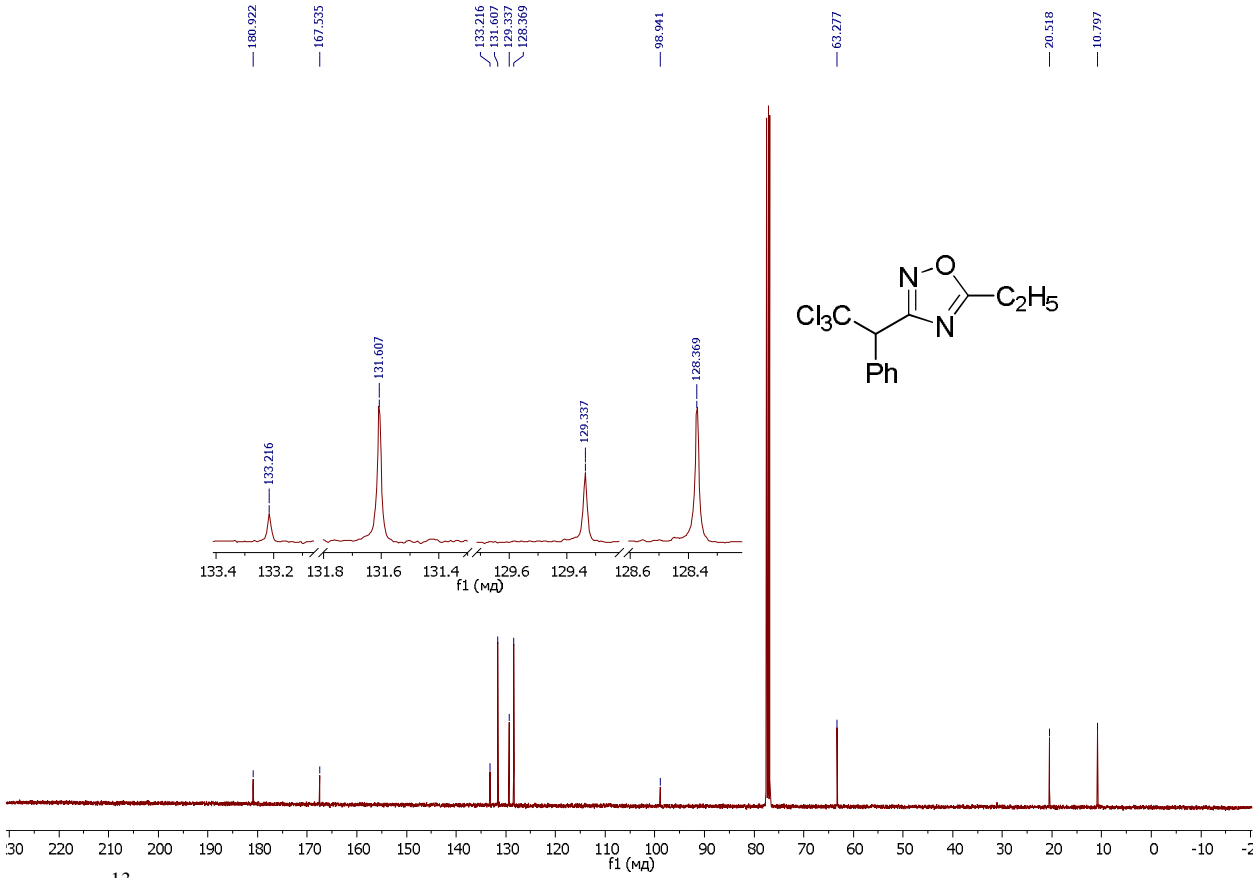

Fig. S5. $^{13}C\{H\}$ NMR spectrum of the compound **2b** ($CDCl_3$, 101 MHz).

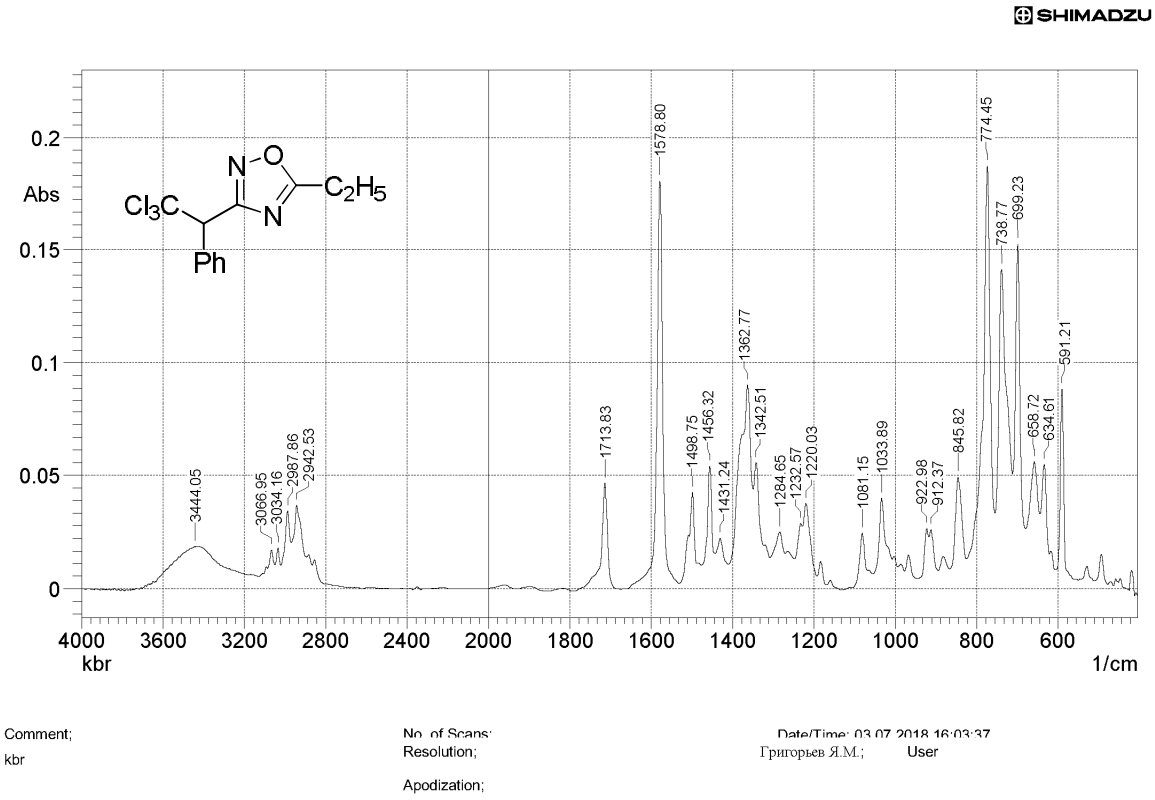

Fig. S6. IR spectrum of the compound **2b** (KBr).

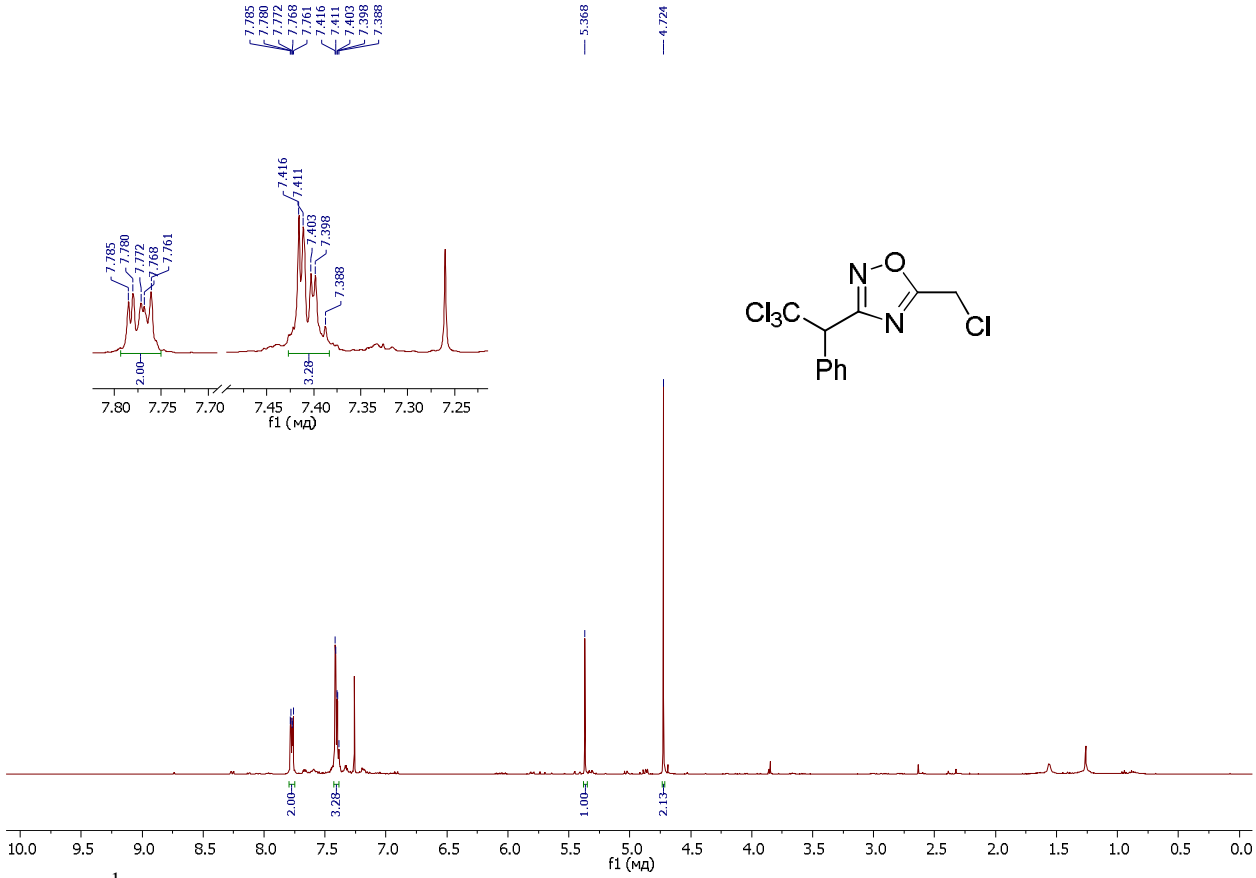

Fig. S7. ^{1}H NMR spectrum of the compound **2c** ($CDCl_3$, 400 MHz).

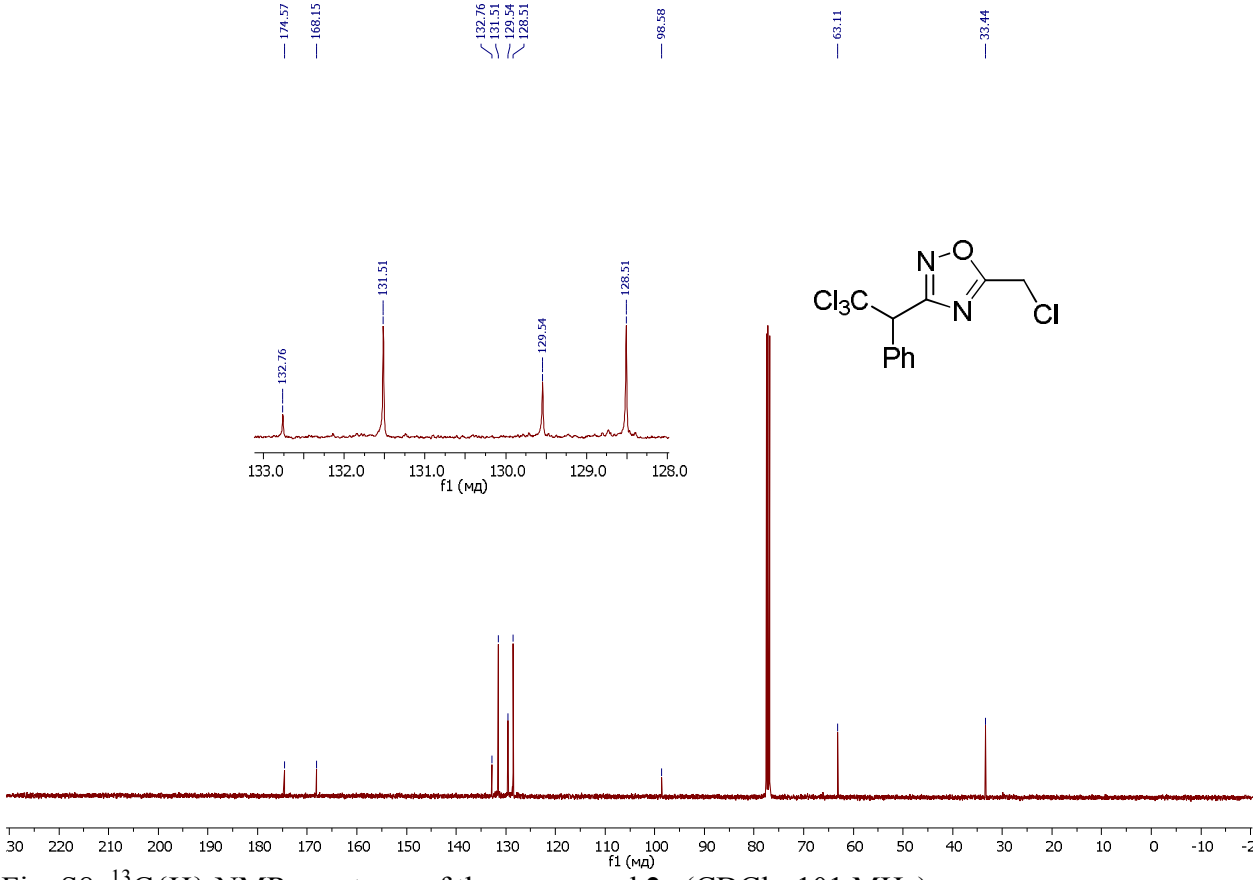

Fig. S8. ^{13}C{H} NMR spectrum of the compound **2c** ($CDCl_3$, 101 MHz).

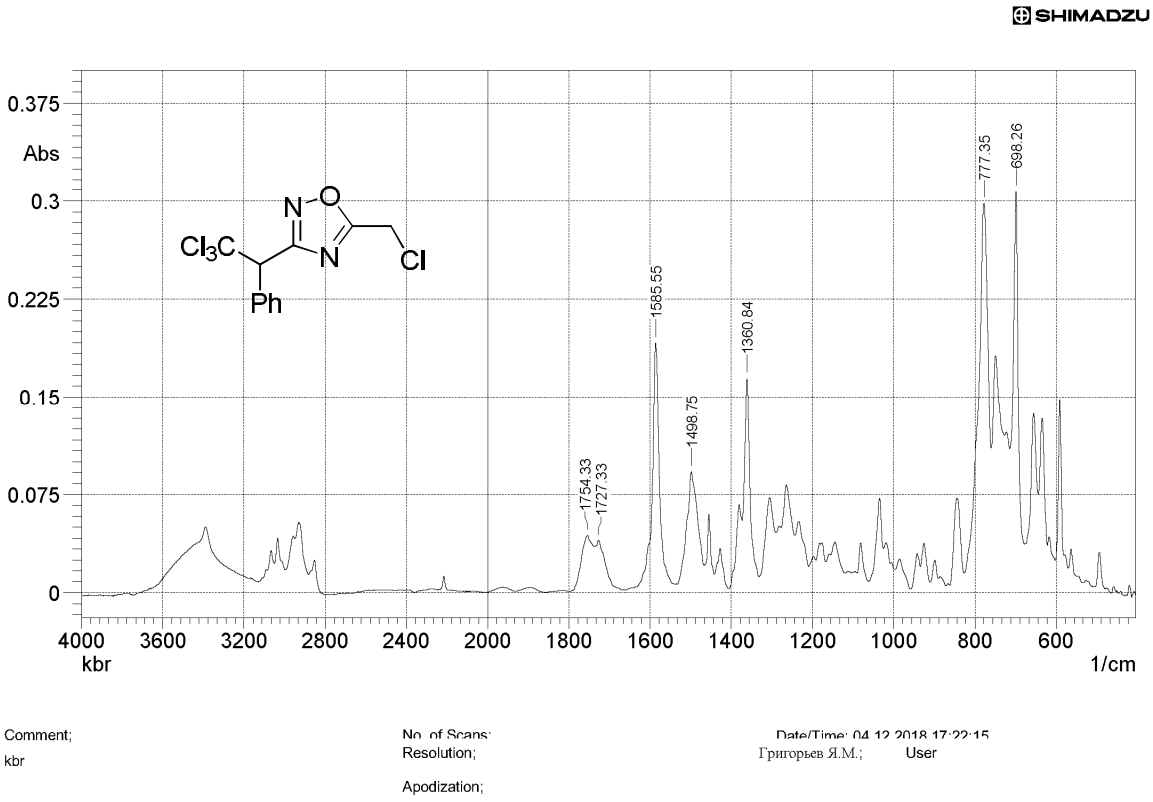

Fig. S9. IR spectrum of the compound **2c** (KBr).

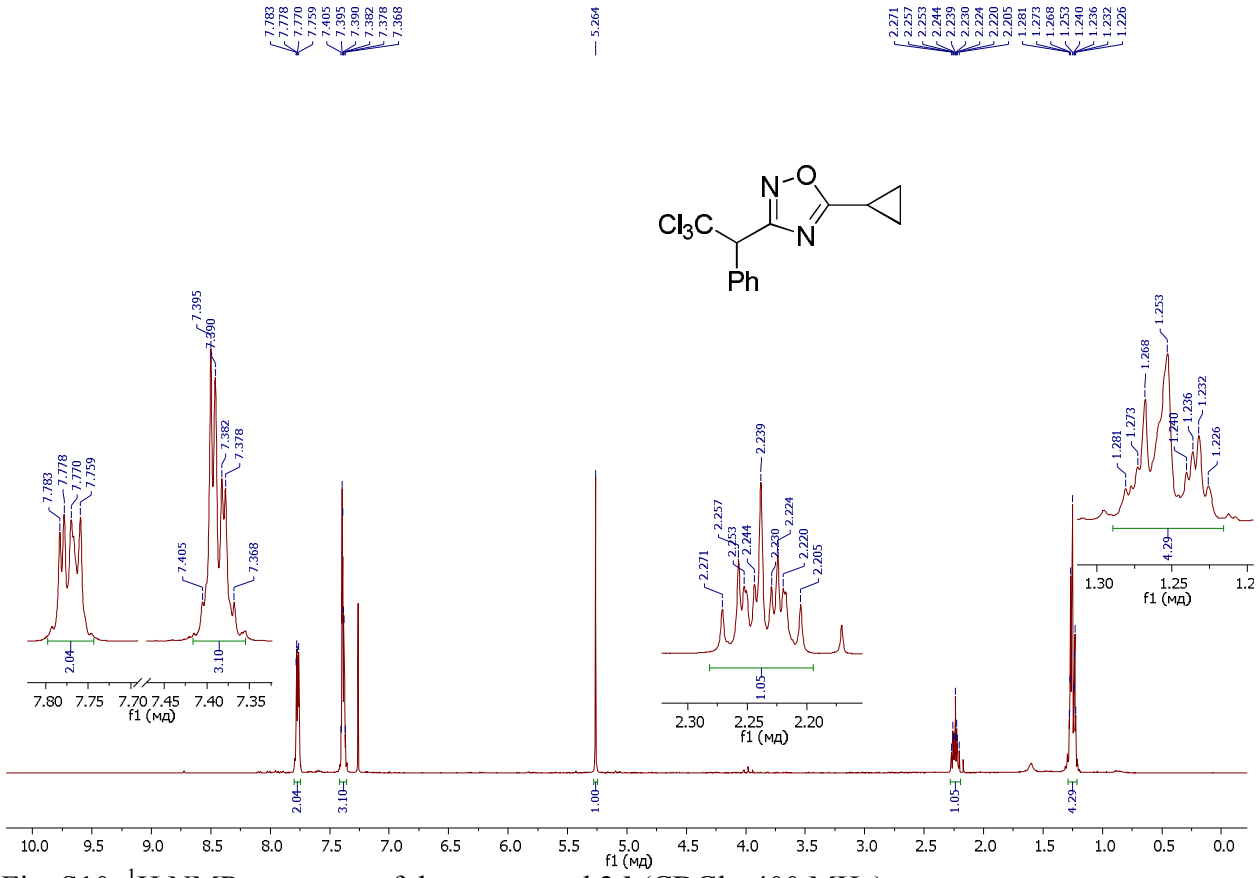

Fig. S10. ^{1}H NMR spectrum of the compound **2d** ($CDCl_3$, 400 MHz).

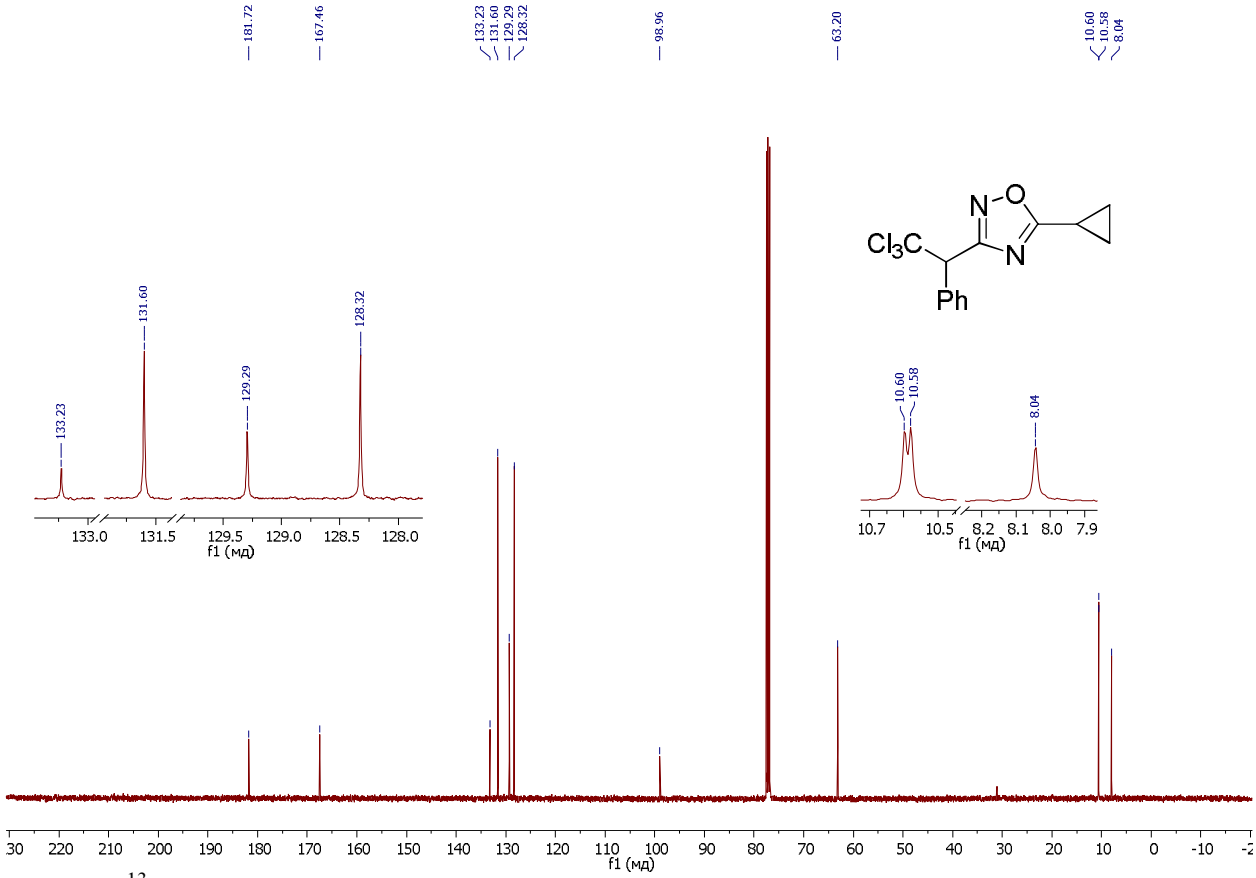

Fig. S11. $^{13}C\{H\}$ NMR spectrum of the compound **2d** ($CDCl_3$, 101 MHz).

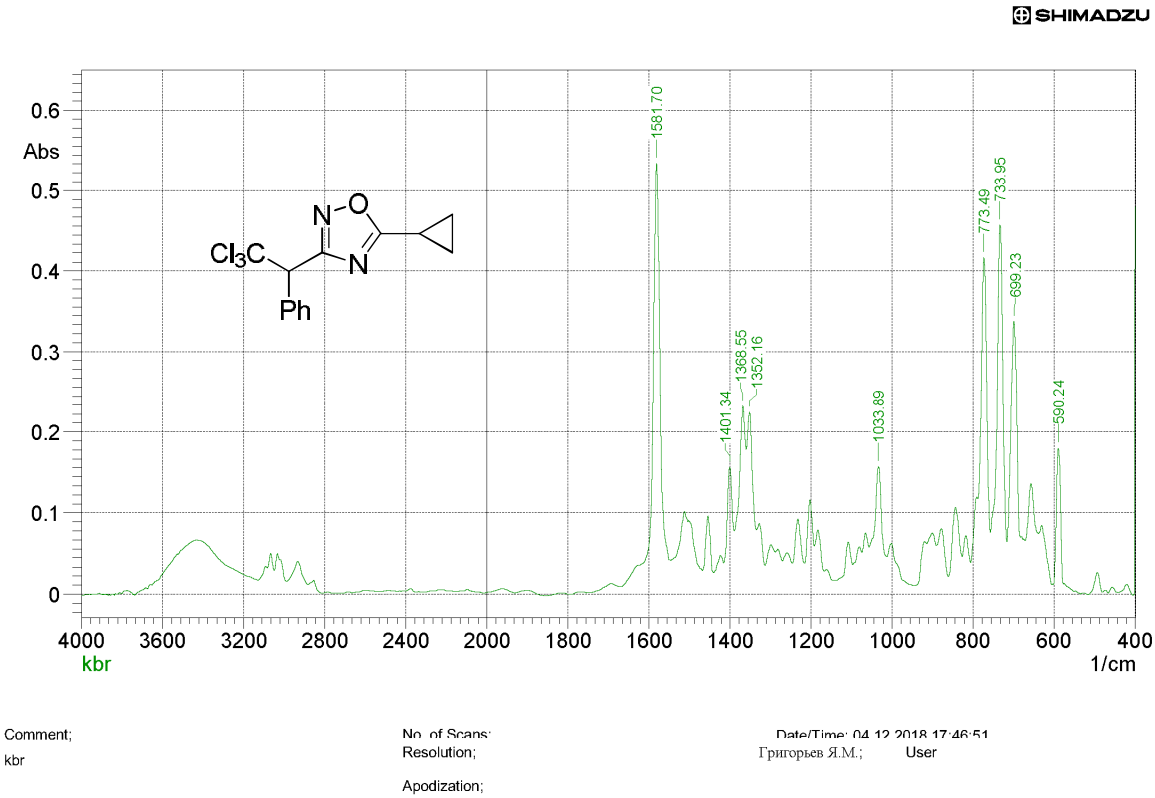

Fig. S12.IR spectrum of the compound **2d** (KBr).

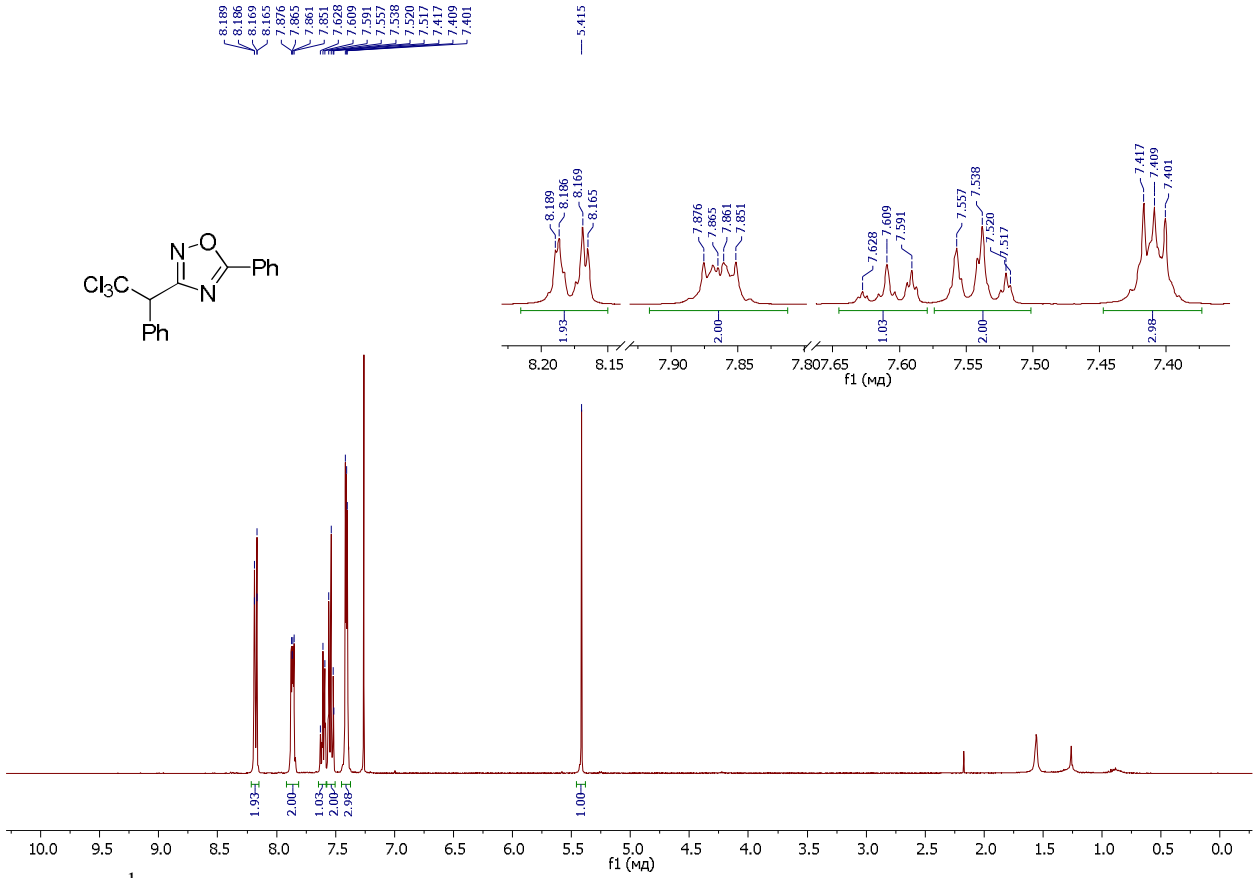

Fig. S13. ^{1}H NMR spectrum of the compound **2e** ($CDCl_3$, 400 MHz).

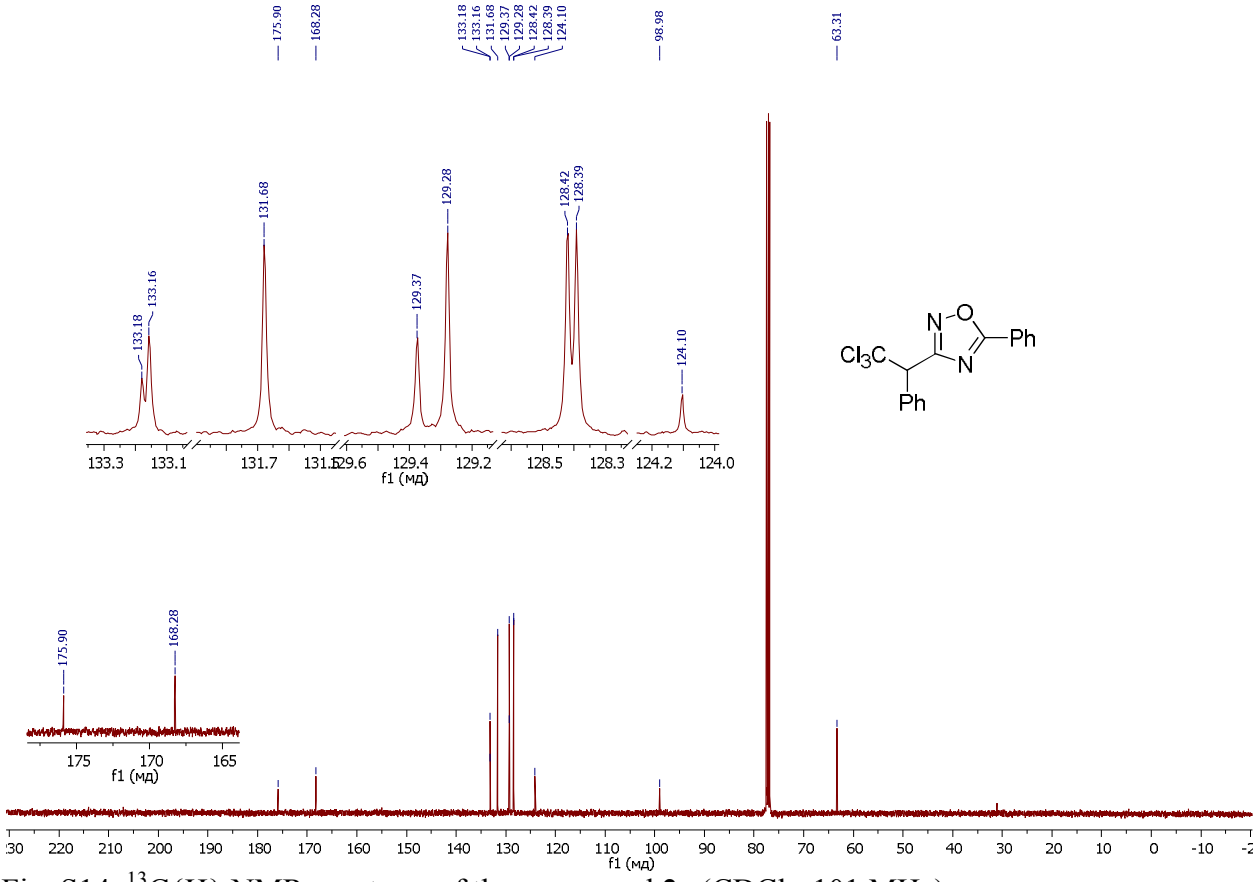

Fig. S14. ^{13}C{H} NMR spectrum of the compound **2e** ($CDCl_3$, 101 MHz).

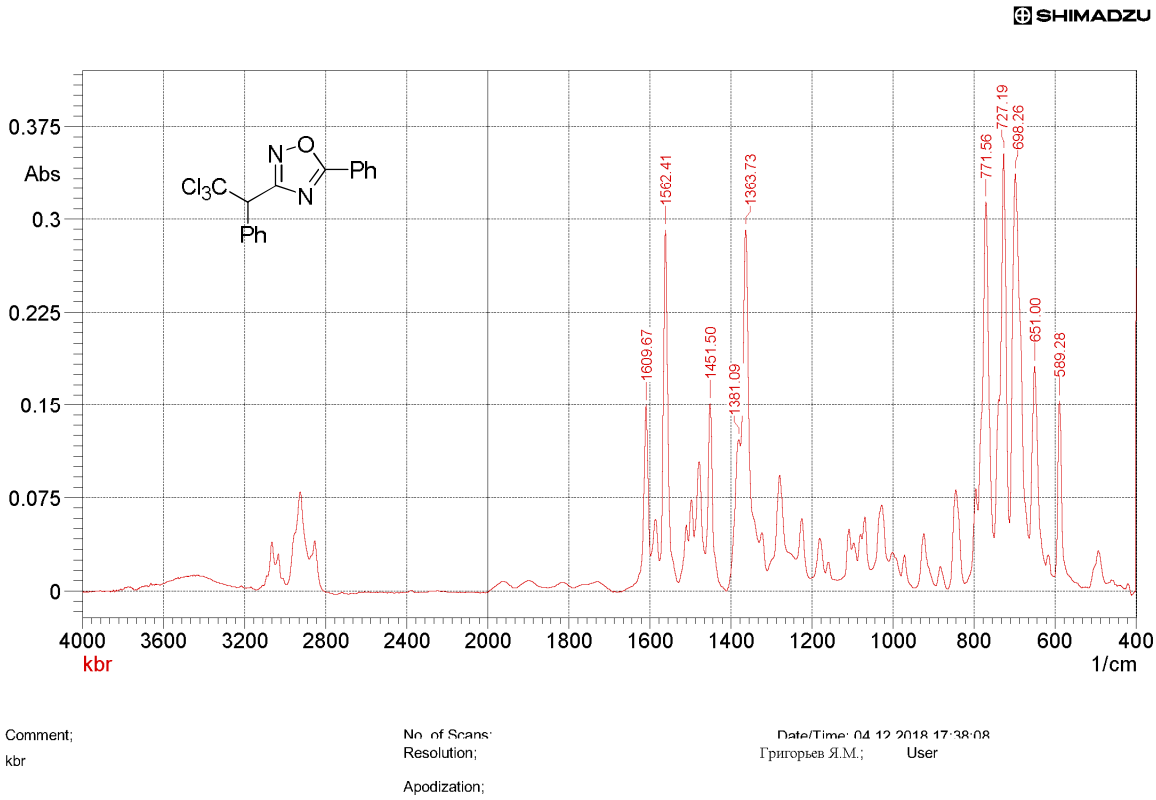

Fig. S15. IR spectrum of the compound **2e** (KBr).

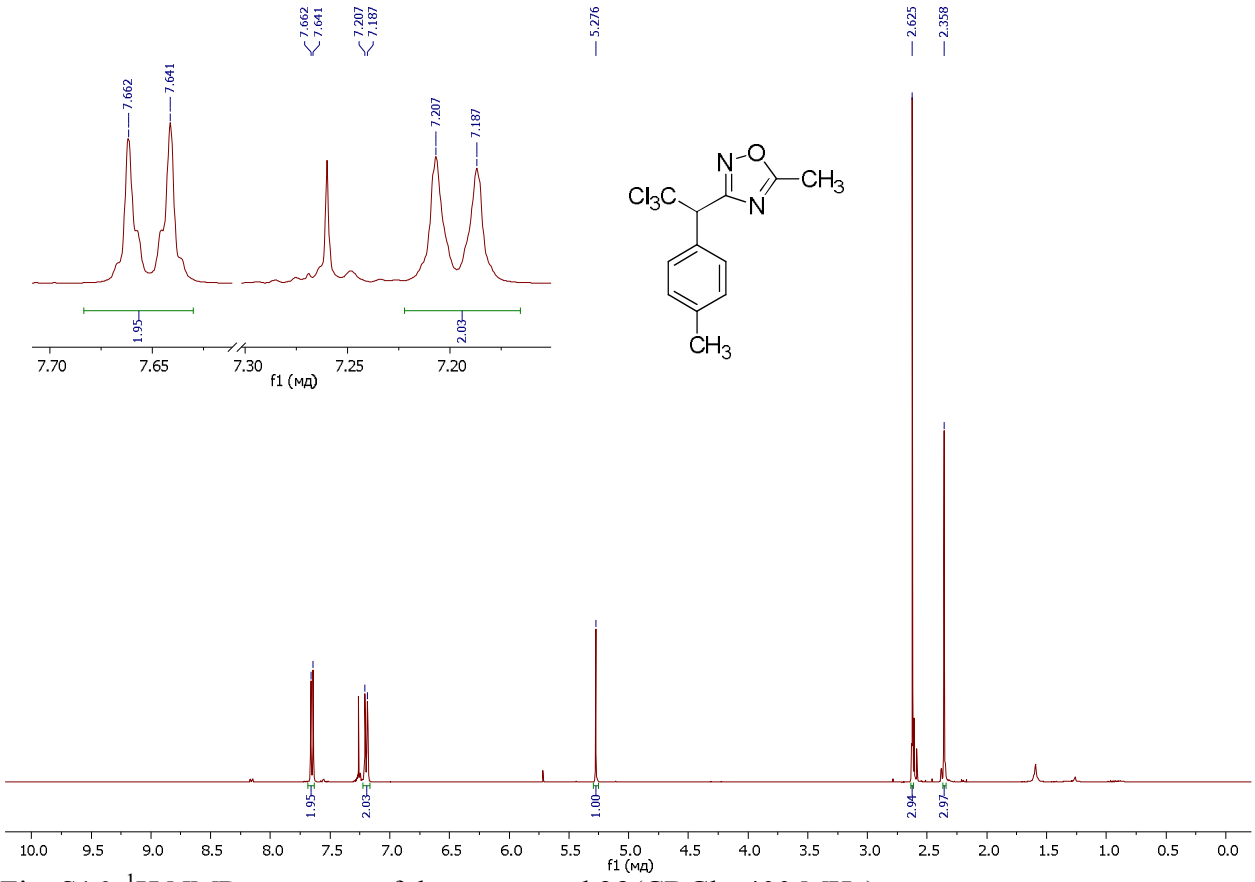

Fig. S16. ^{1}H NMR spectrum of the compound **2f** ($CDCl_3$, 400 MHz).

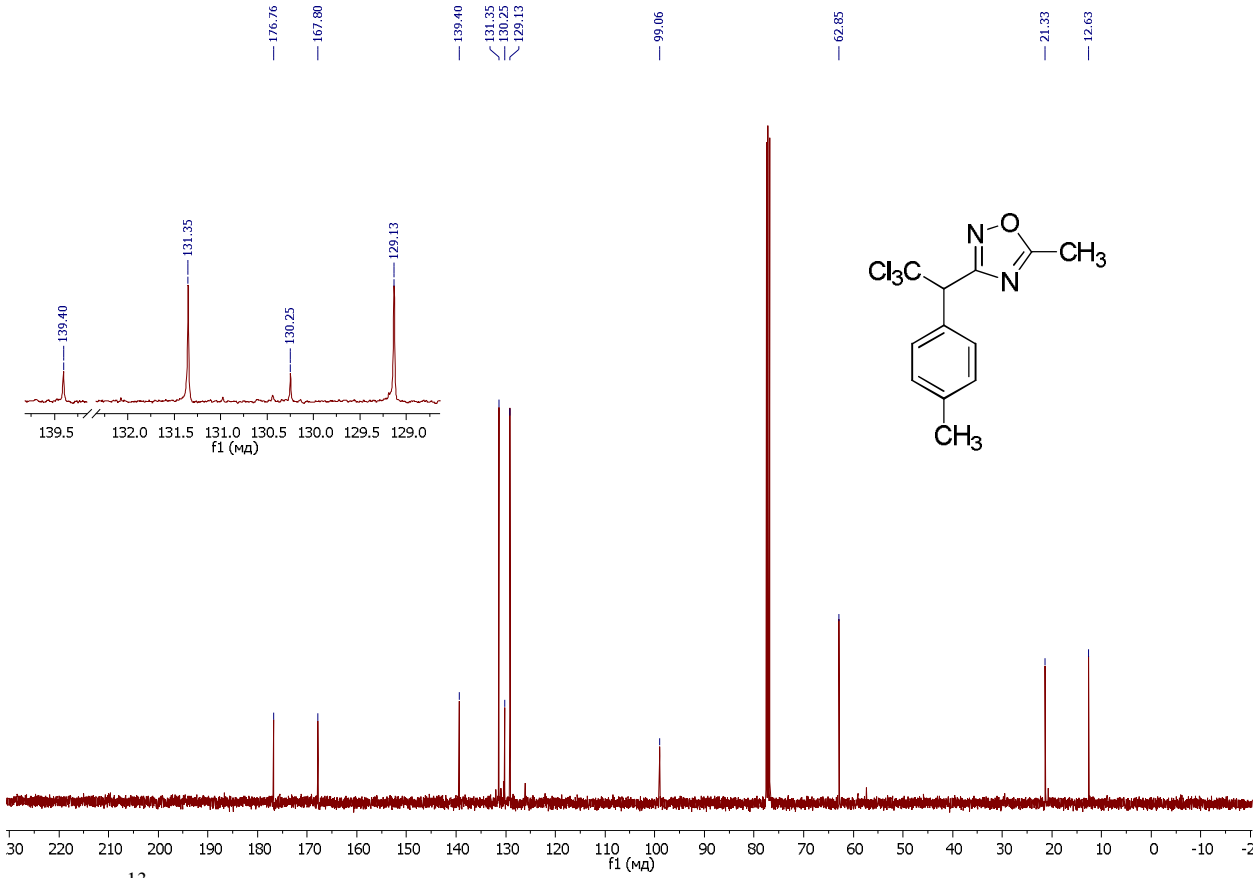

Fig. S17. $^{13}C\{H\}$ NMR spectrum of the compound **2f** ($CDCl_3$, 101 MHz).

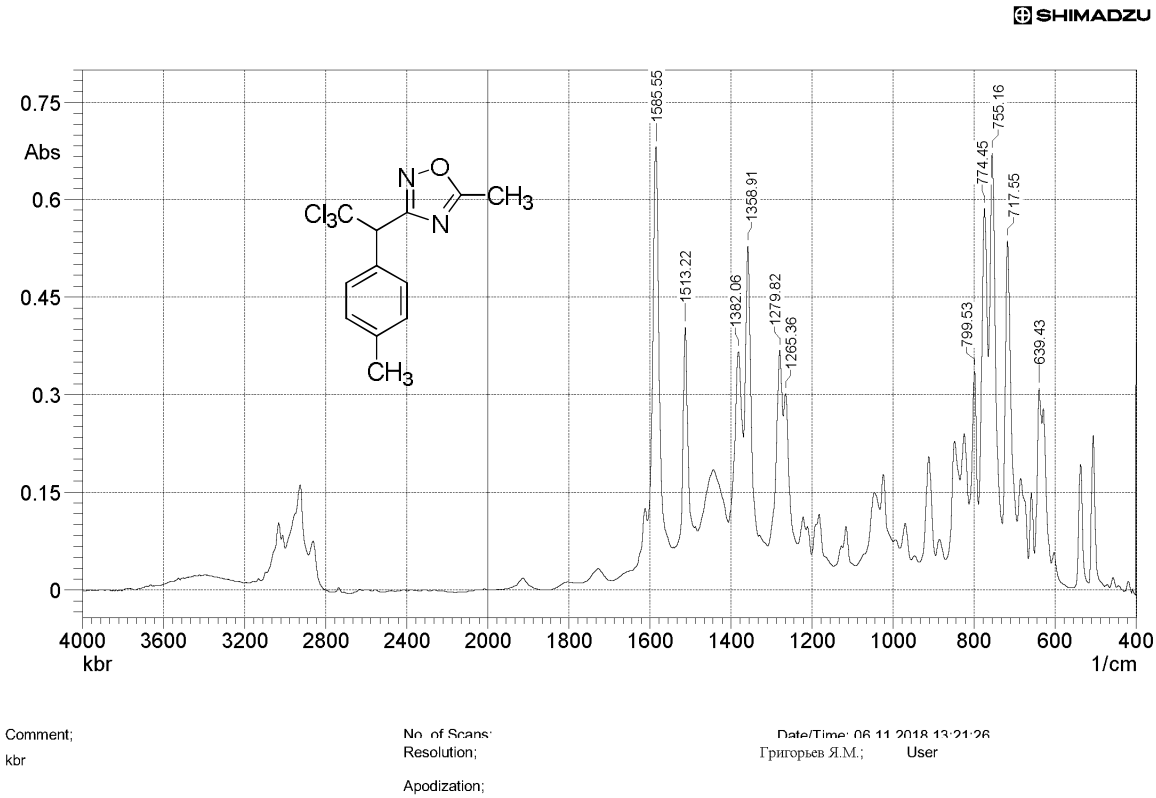

Fig. S18. IR spectrum of the compound **2f** (KBr).

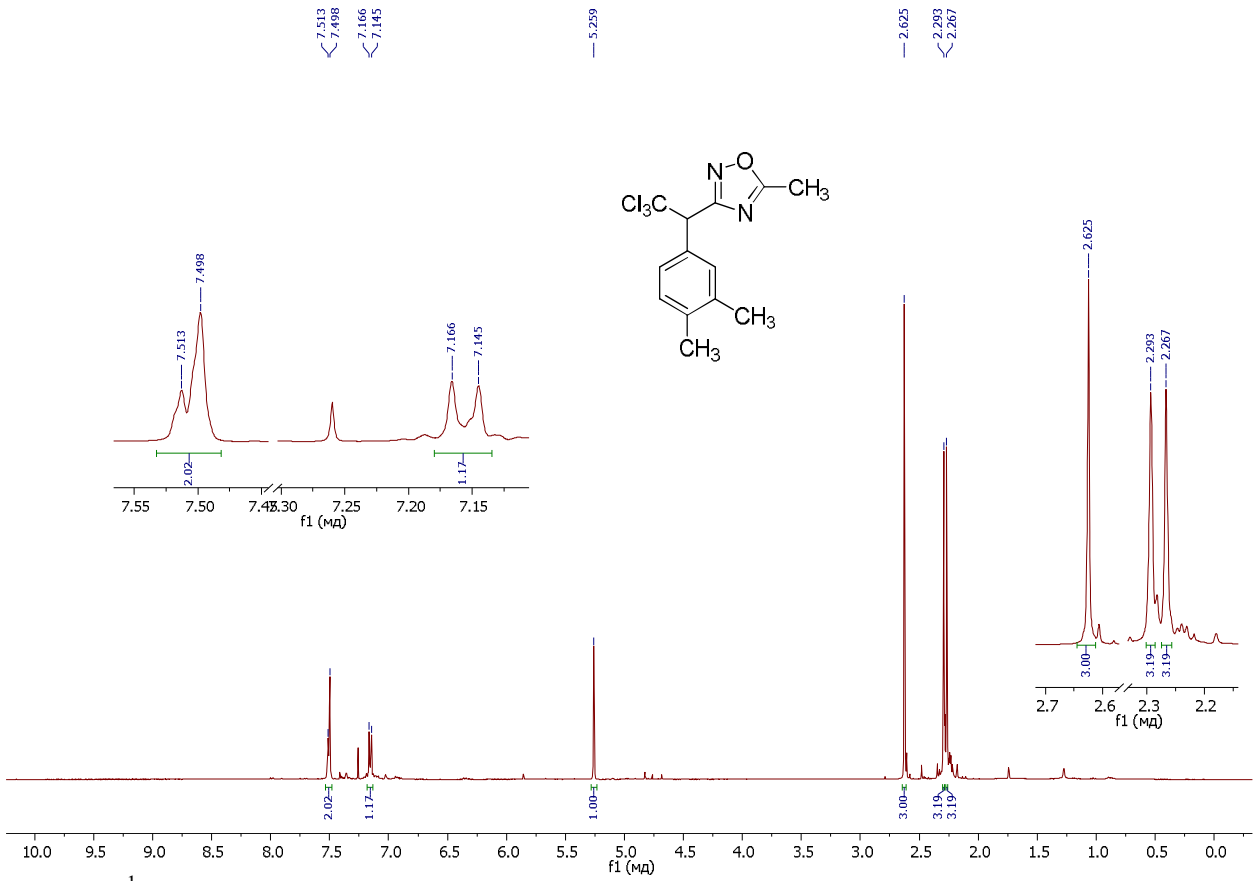

Fig. S19. ^{1}H NMR spectrum of the compound **2g** ($CDCl_3$, 400 MHz).

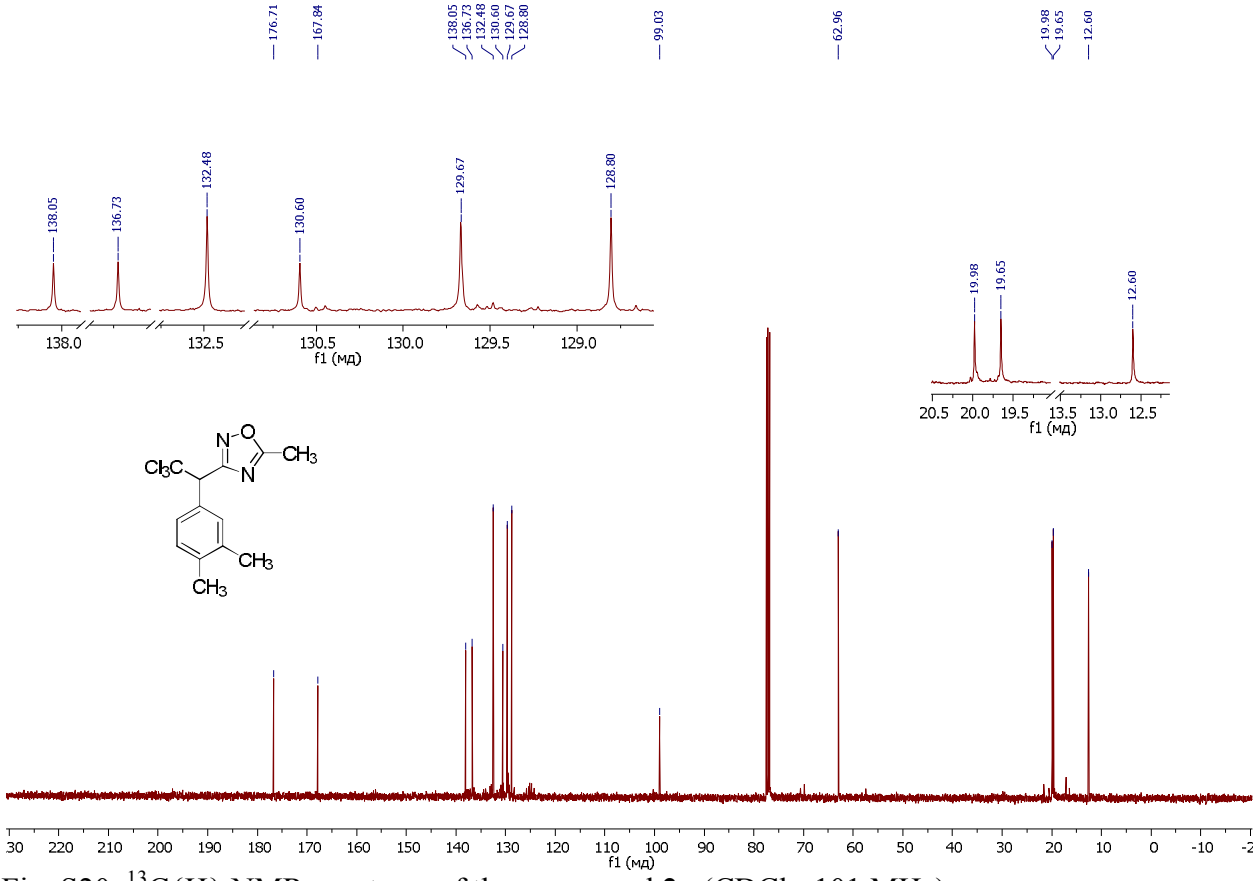

Fig. S20. ^{13}C{H} NMR spectrum of the compound **2g** ($CDCl_3$, 101 MHz).

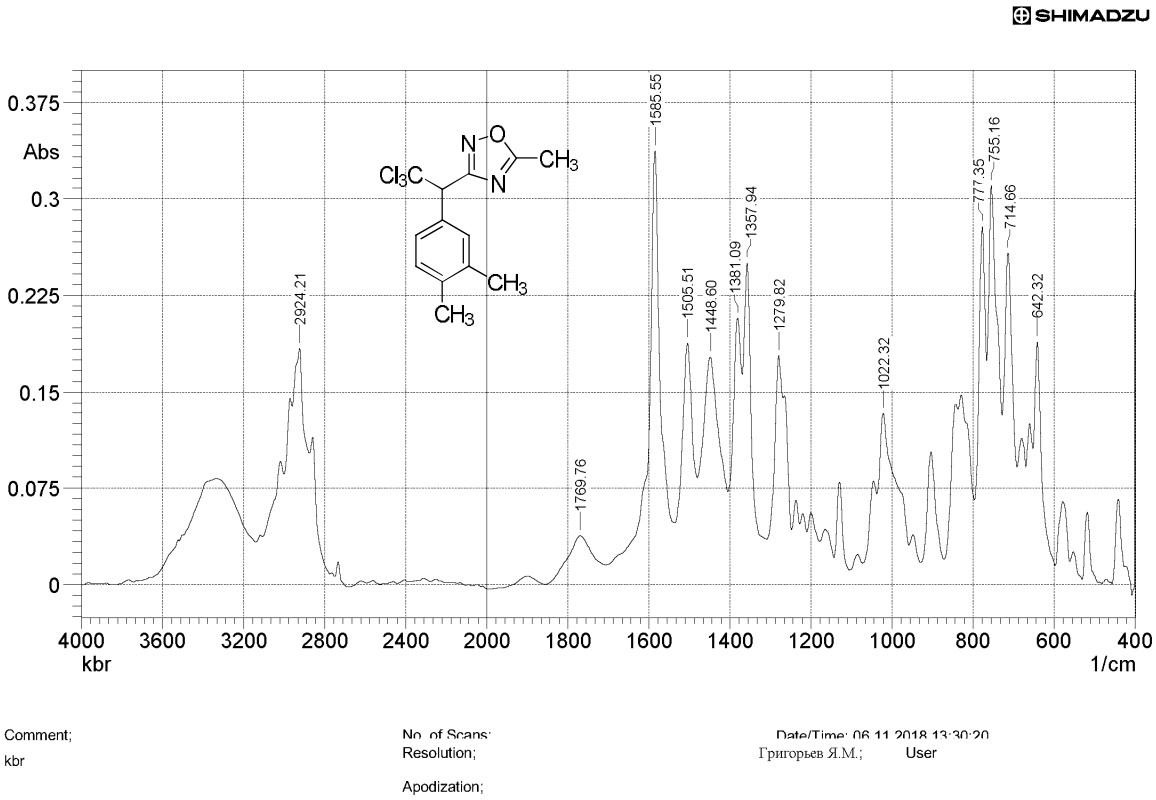

Fig. S21. IR spectrum of the compound **2g** (KBr).

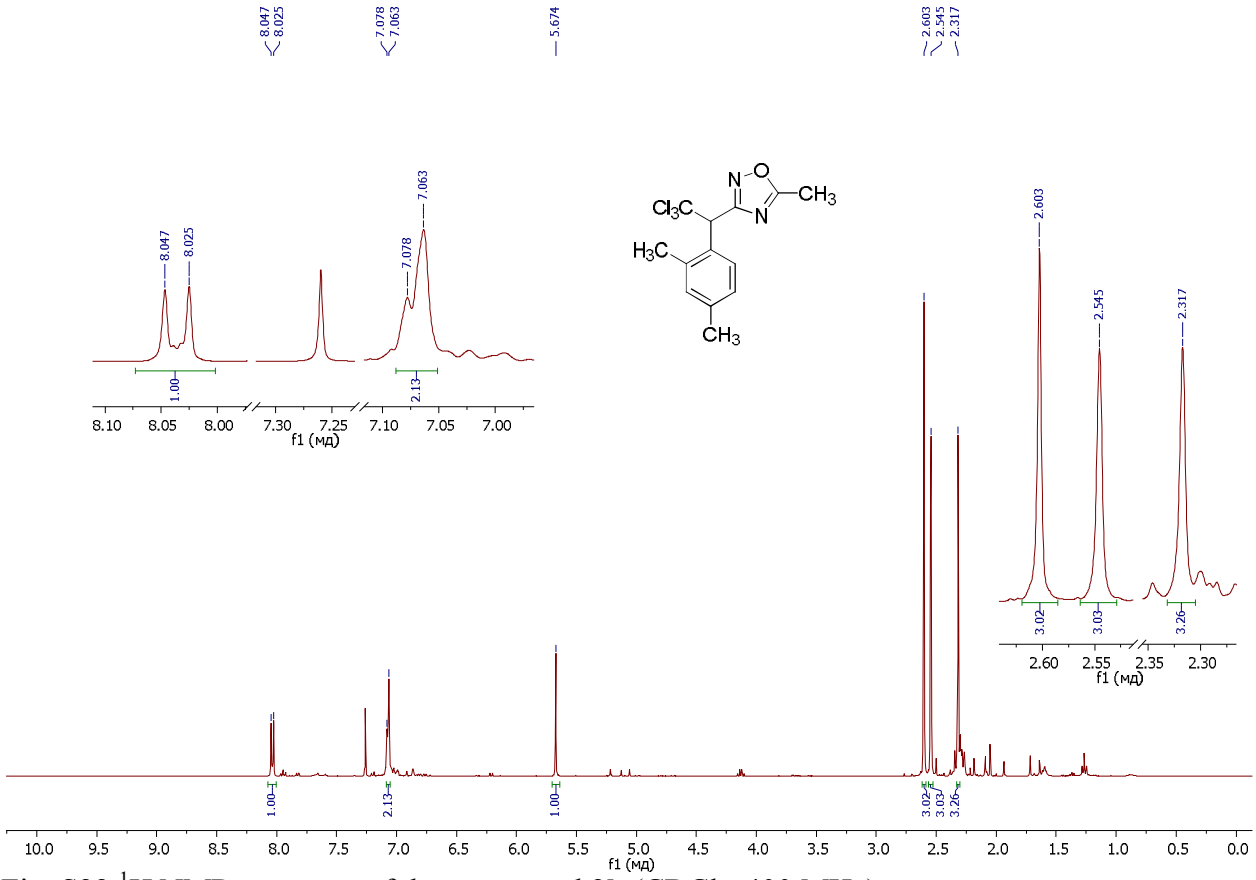

Fig. S22. ^{1}H NMR spectrum of the compound **2h** ($CDCl_3$, 400 MHz).

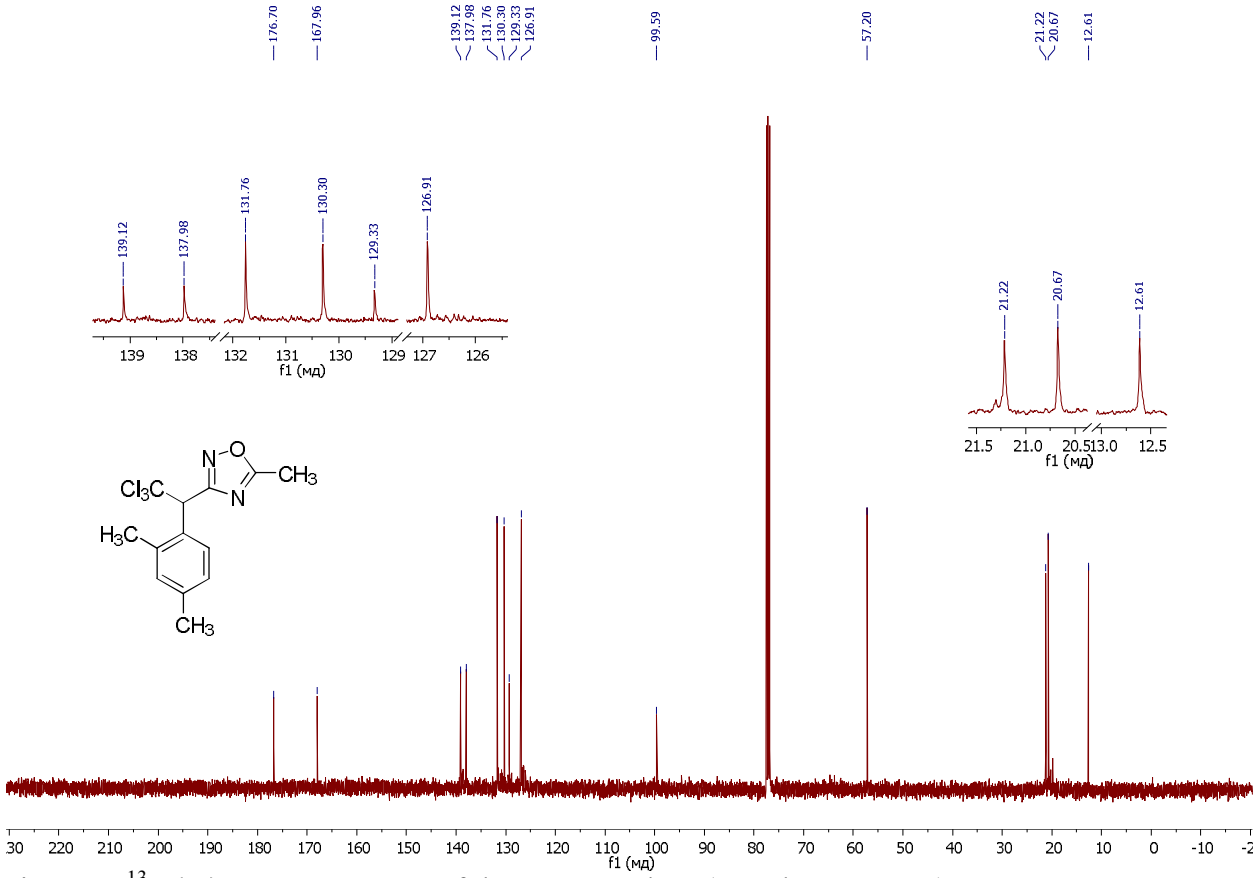

Fig. S23. $^{13}C\{H\}$ NMR spectrum of the compound **2h** ($CDCl_3$, 101 MHz).

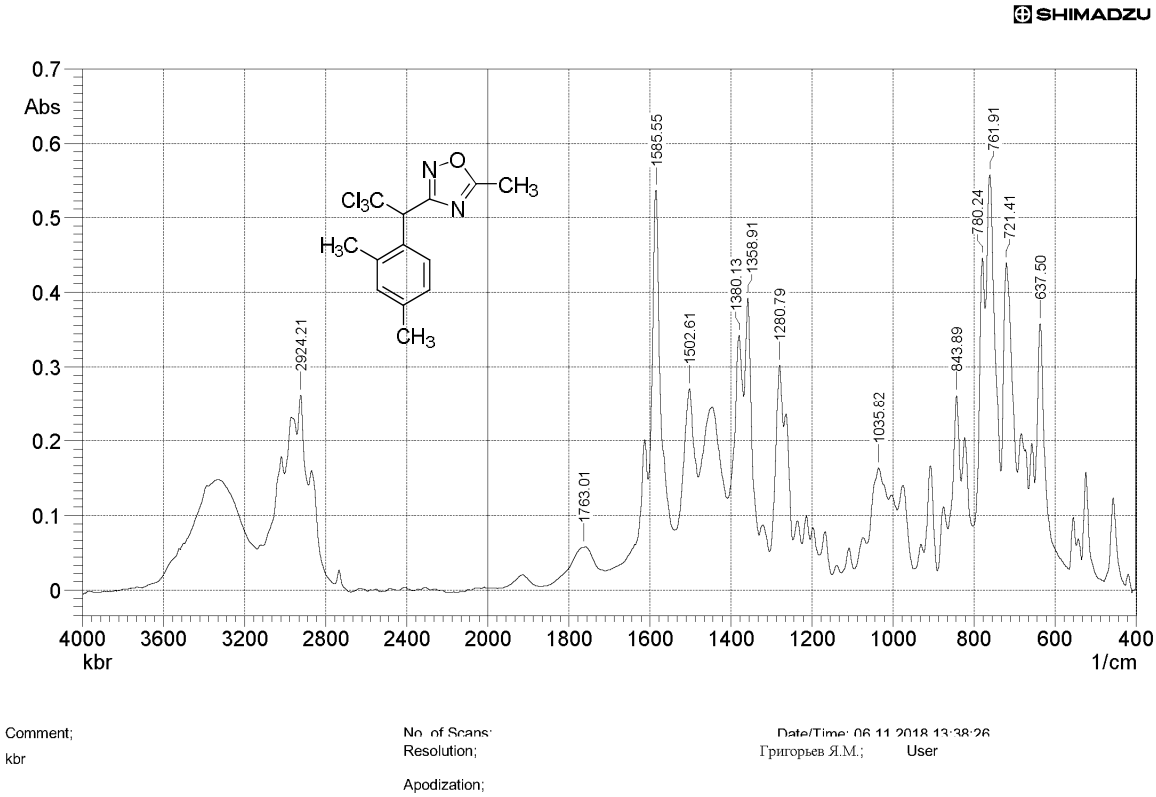

Fig. S24. IR spectrum of the compound **2h** (KBr).

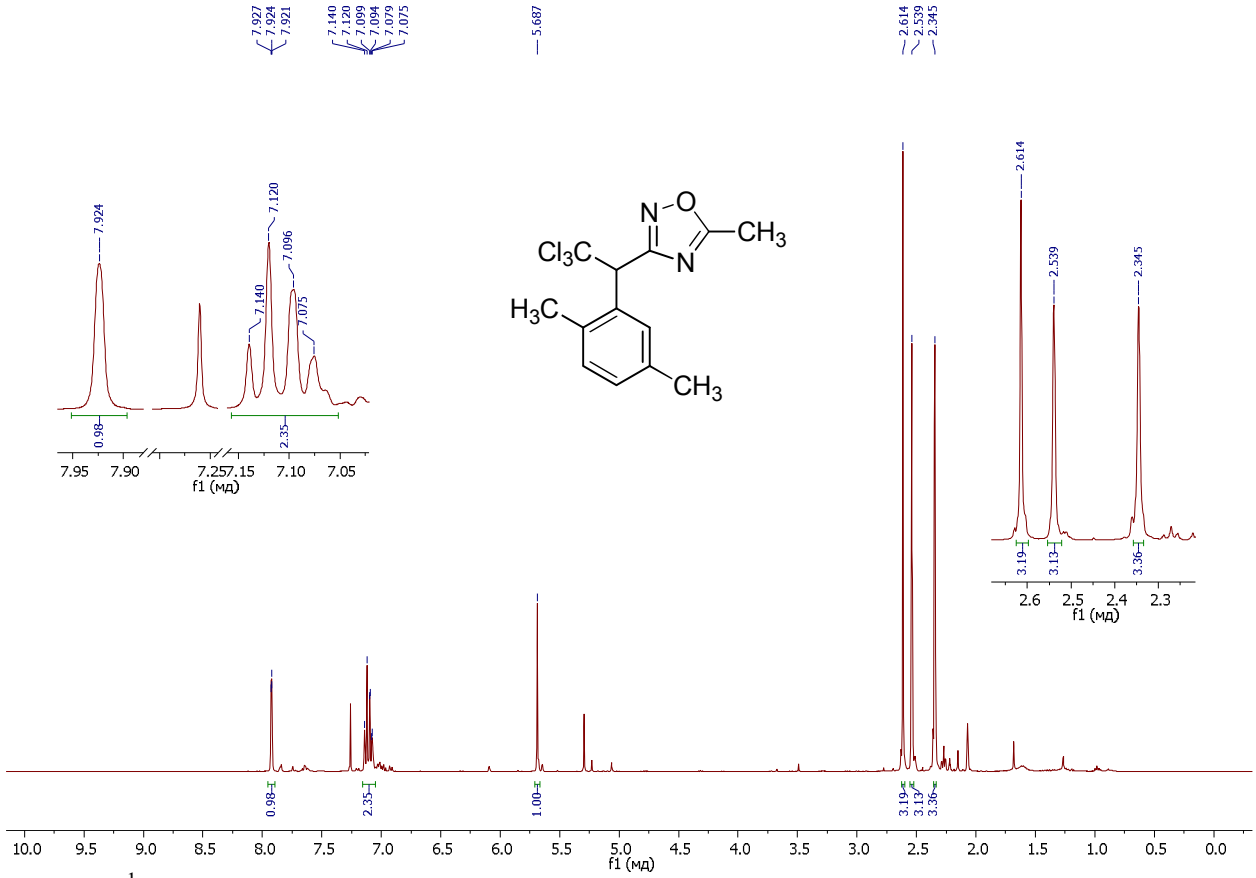

Fig. S25. ^{1}H NMR spectrum of the compound **2i** ($CDCl_3$, 400 MHz).

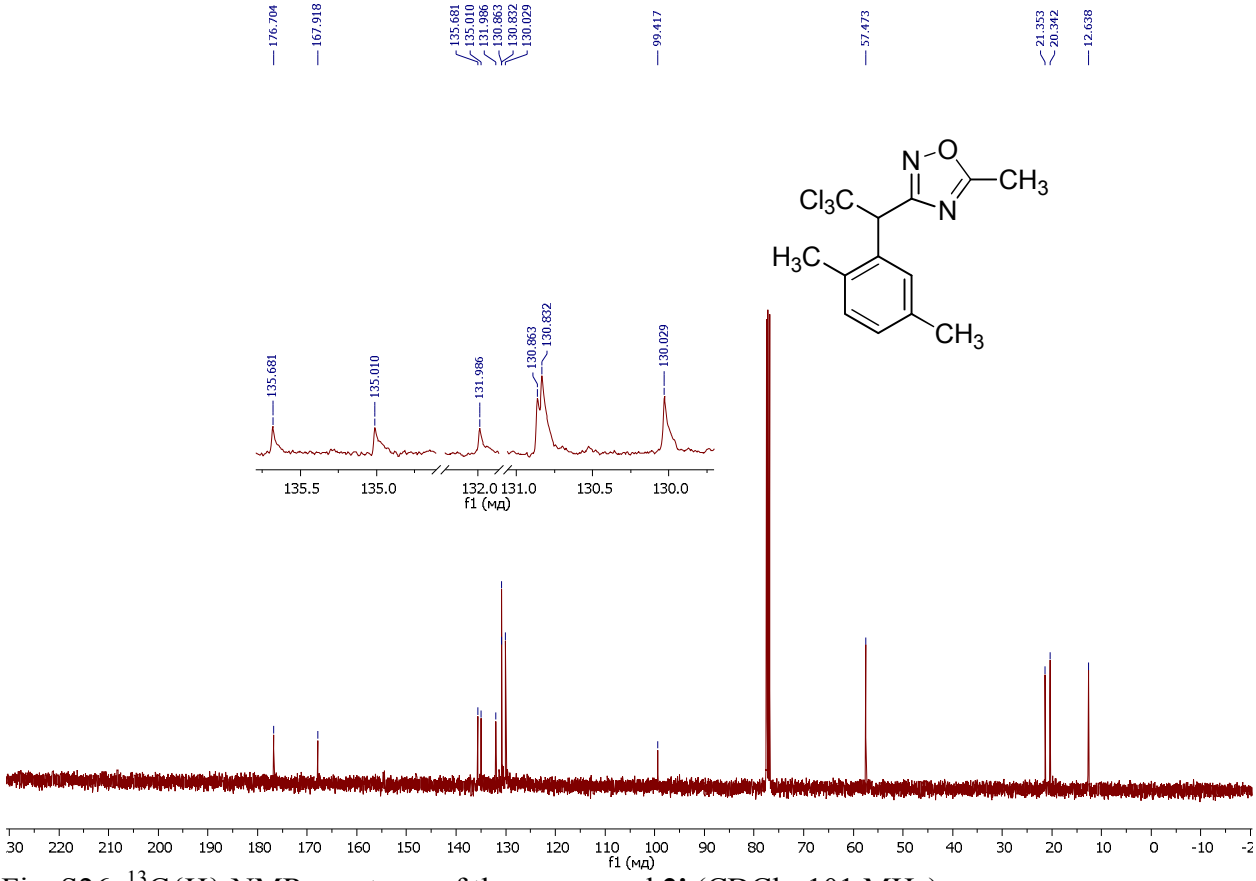

Fig. S26. ^{13}C{H} NMR spectrum of the compound **2i** ($CDCl_3$, 101 MHz).

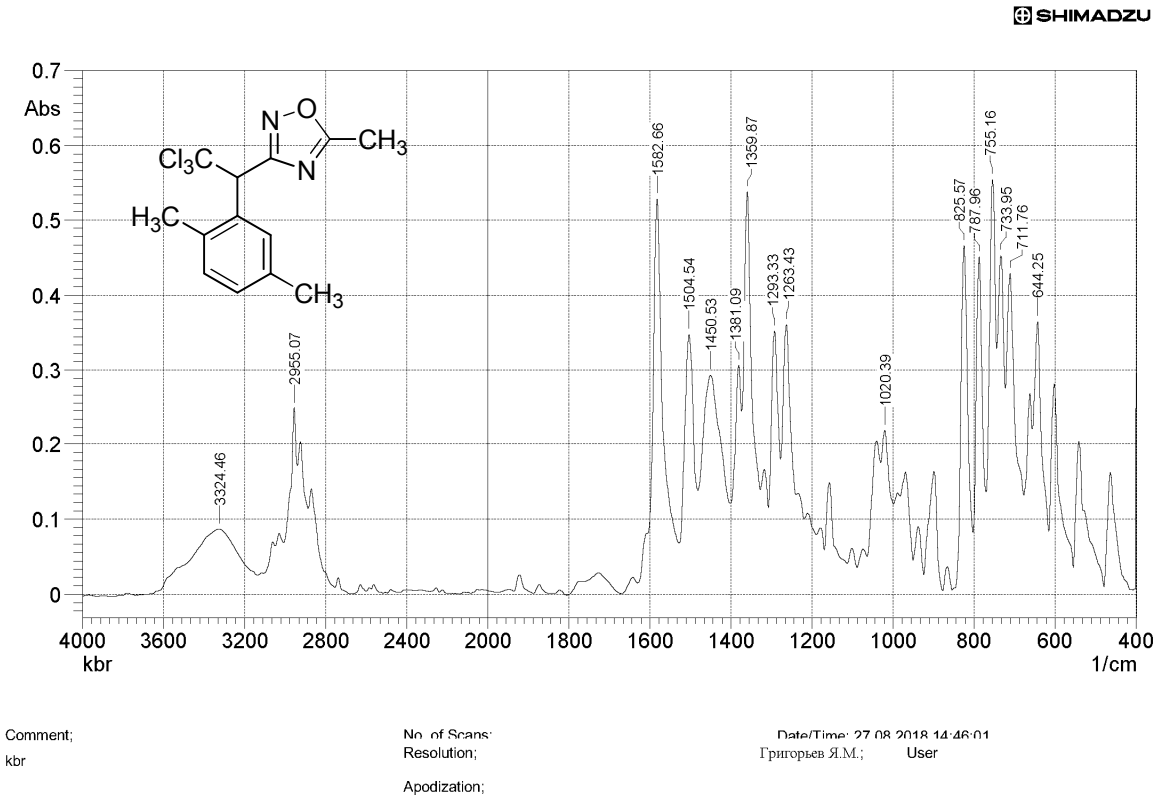

Fig. S27. IR spectrum of the compound **2i** (KBr).

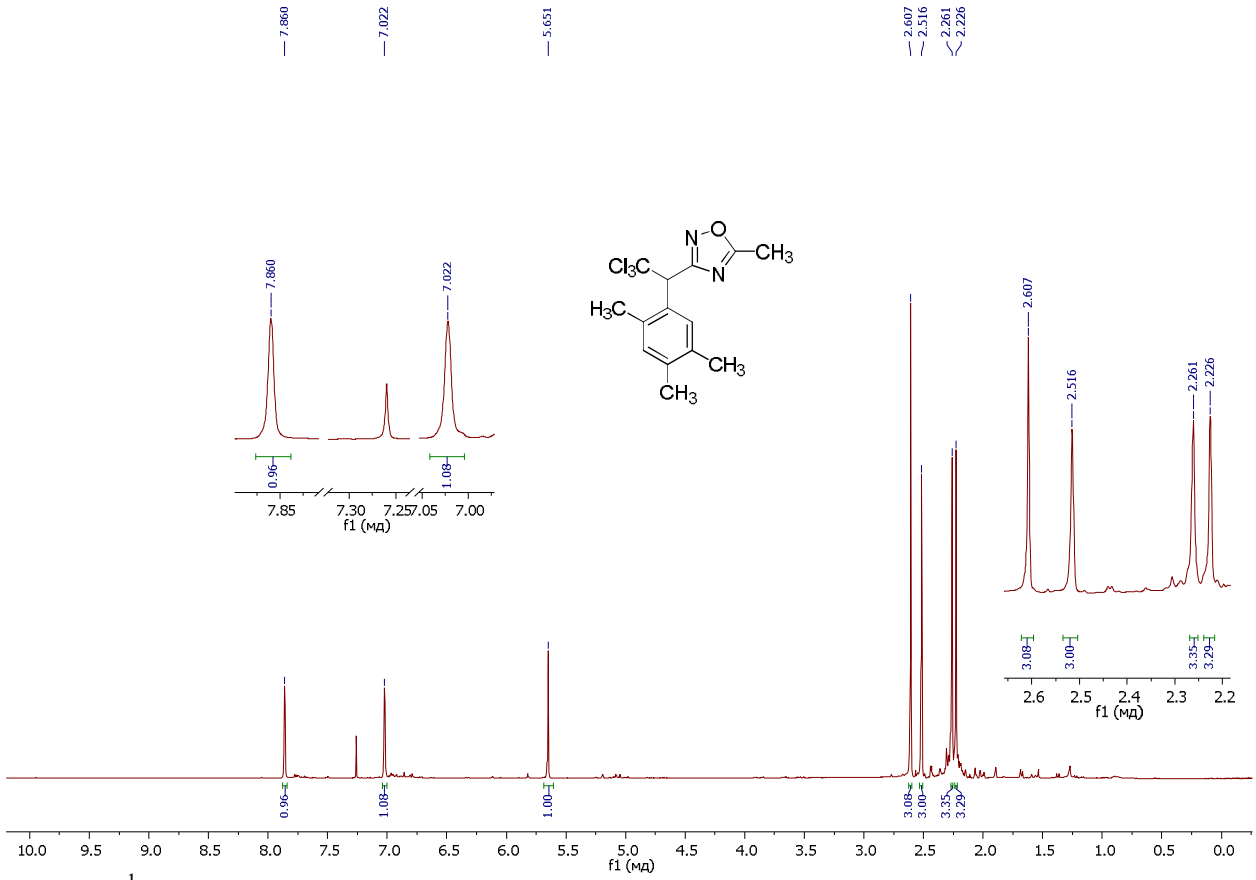

Fig. S28. ^{1}H NMR spectrum of the compound **2j** ($CDCl_3$, 400 MHz).

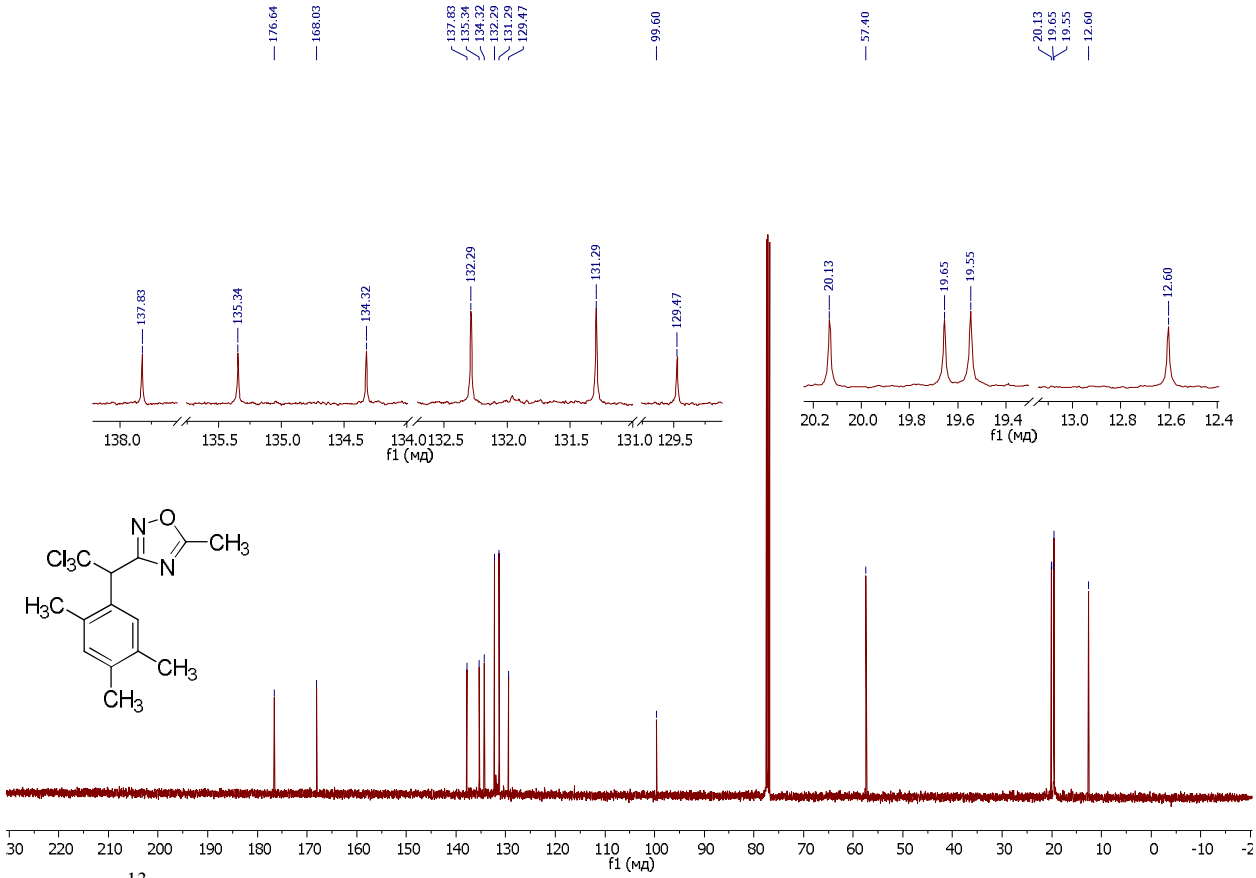

Fig. S29. $^{13}C\{H\}$ NMR spectrum of the compound **2j** ($CDCl_3$, 101 MHz).

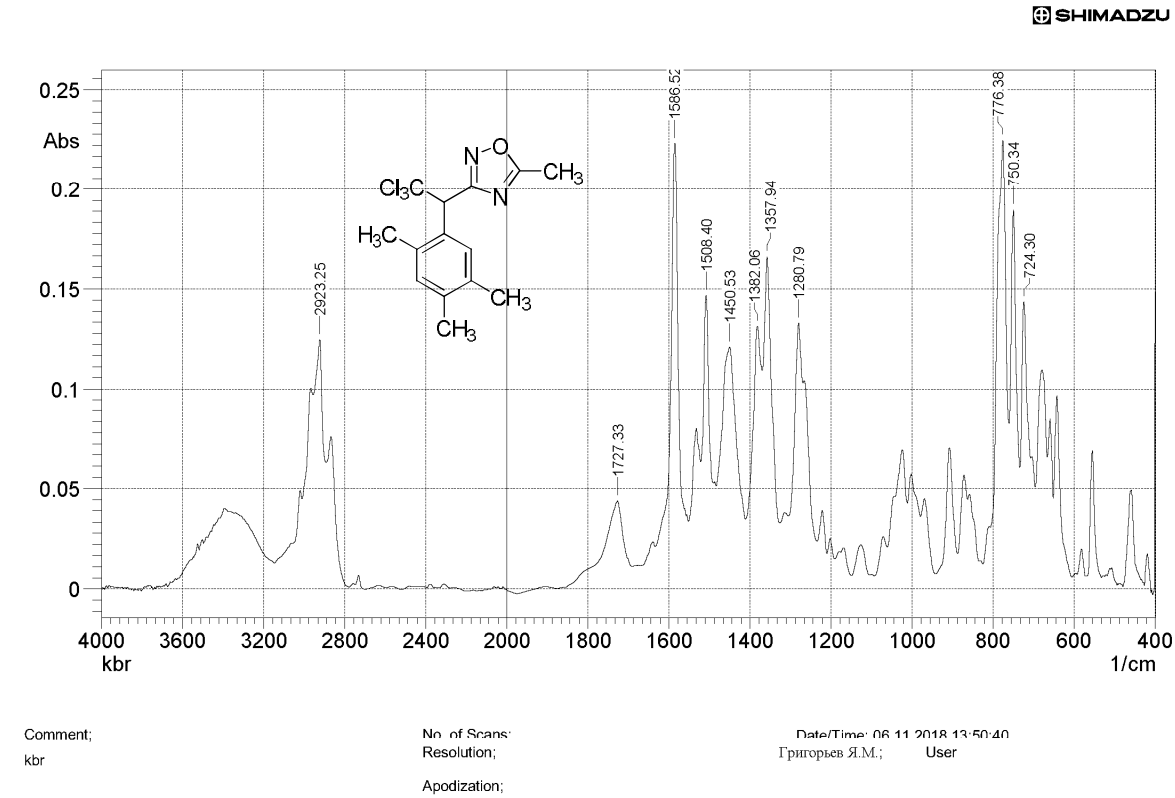

Fig. S30. IR spectrum of the compound **2j** (KBr).

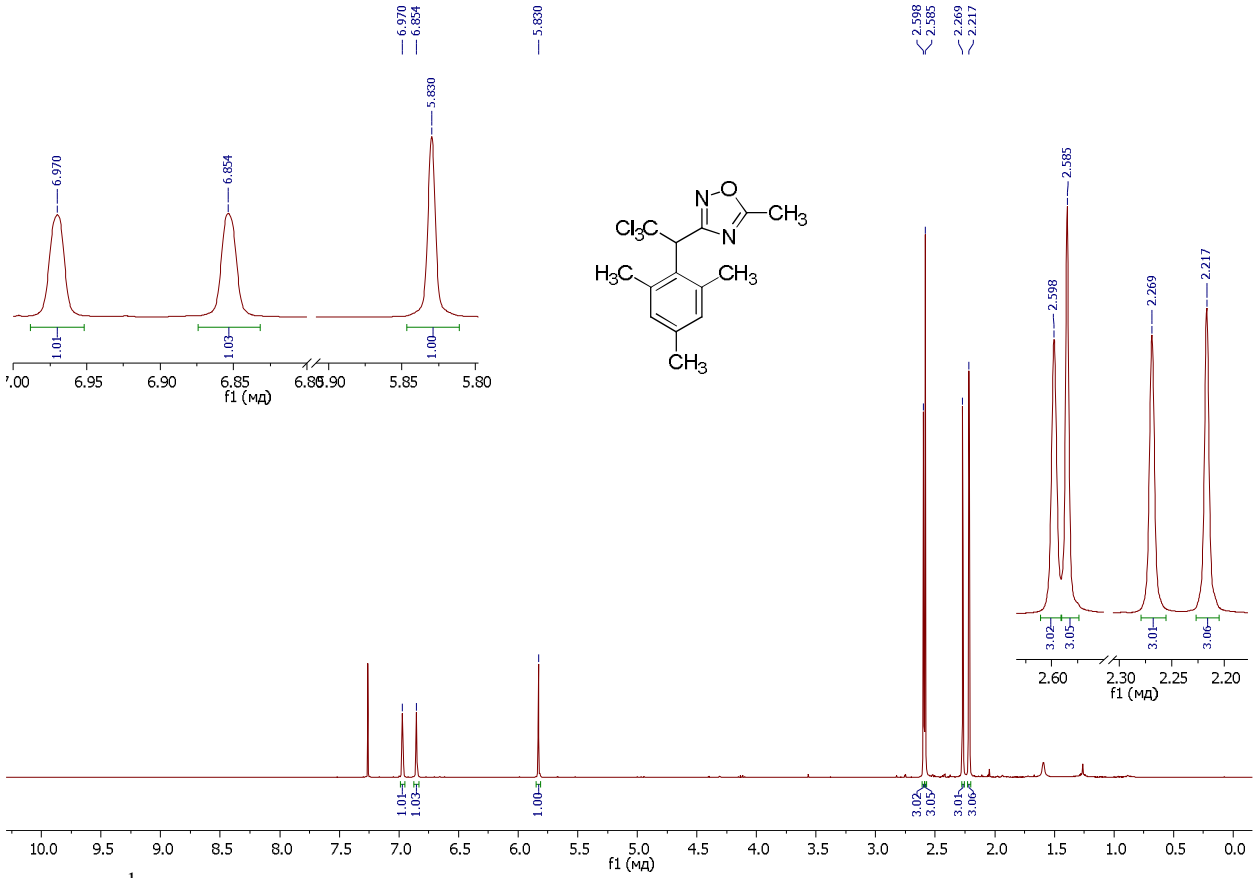

Fig. S31. ^{1}H NMR spectrum of the compound **2k** ($CDCl_3$, 400 MHz).

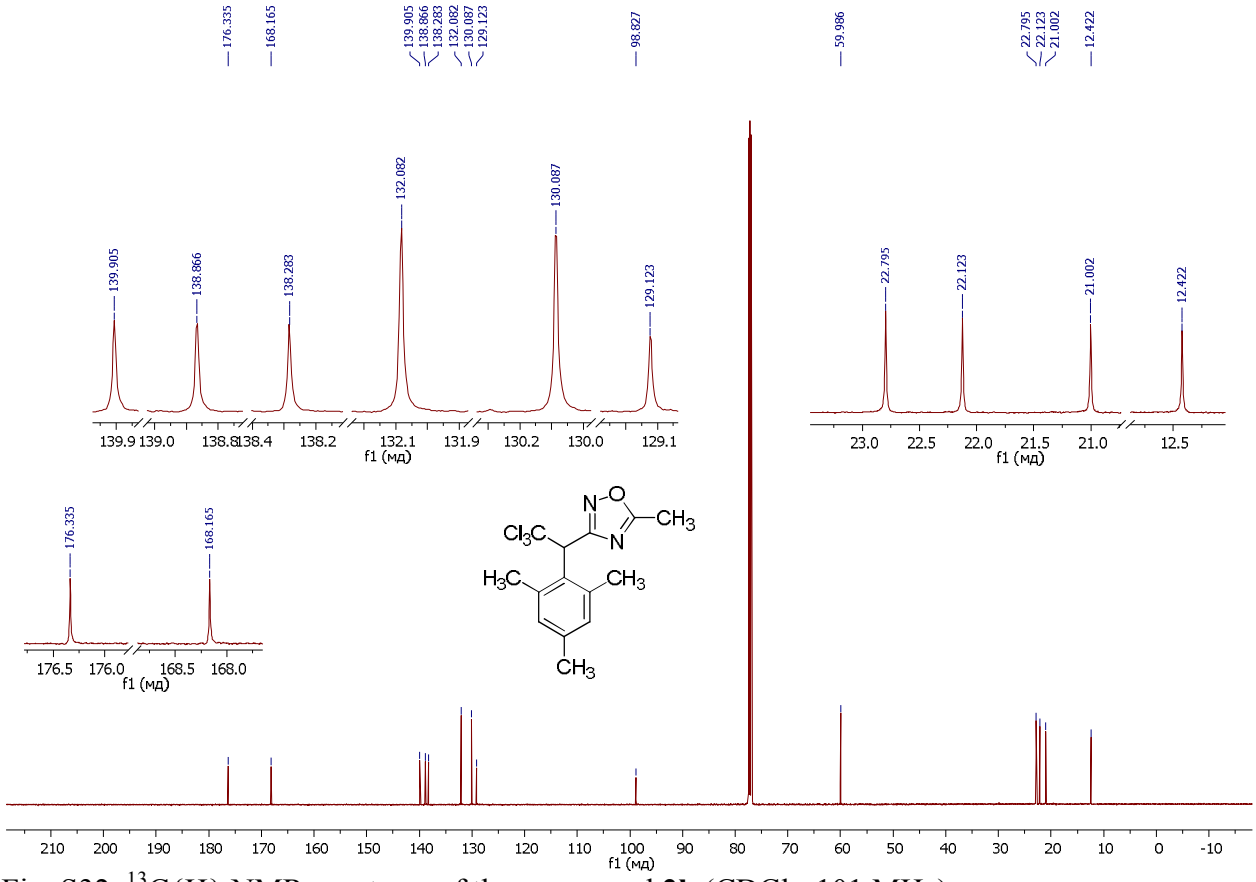

Fig. S32. ^{13}C{H} NMR spectrum of the compound **2k** ($CDCl_3$, 101 MHz).

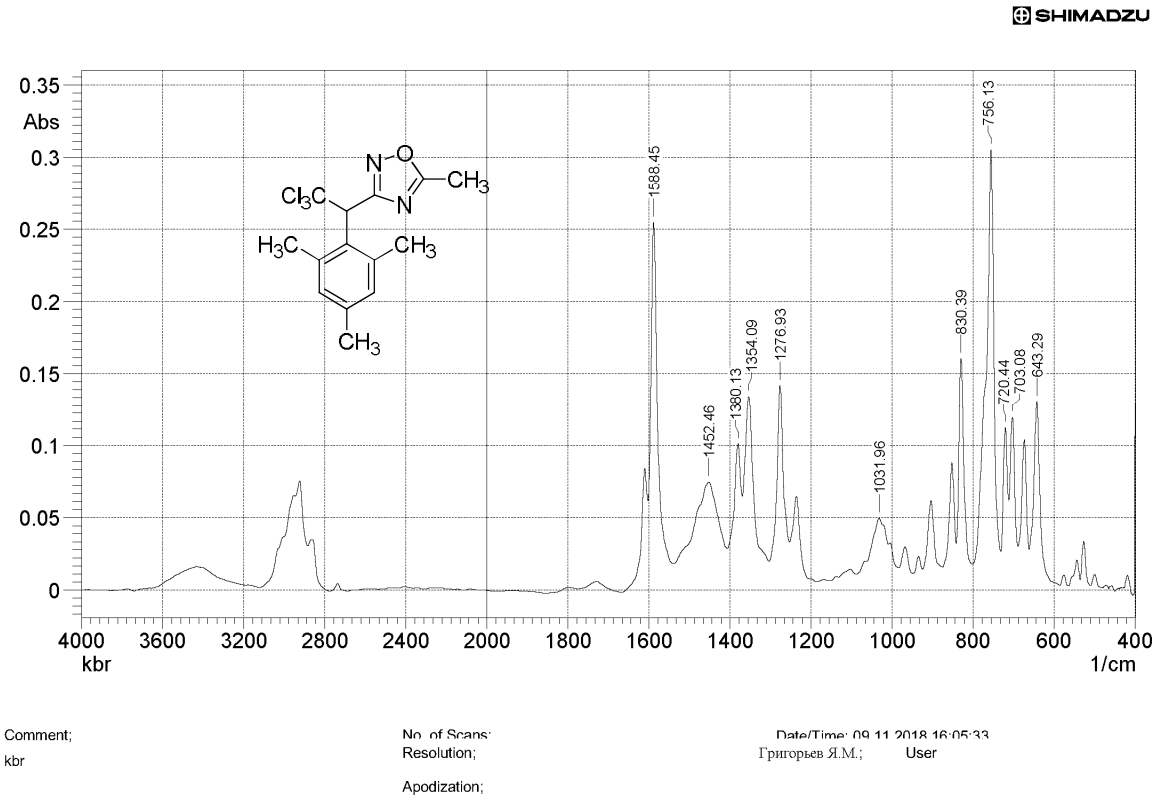

Fig. S33. IR spectrum of the compound **2k** (KBr).

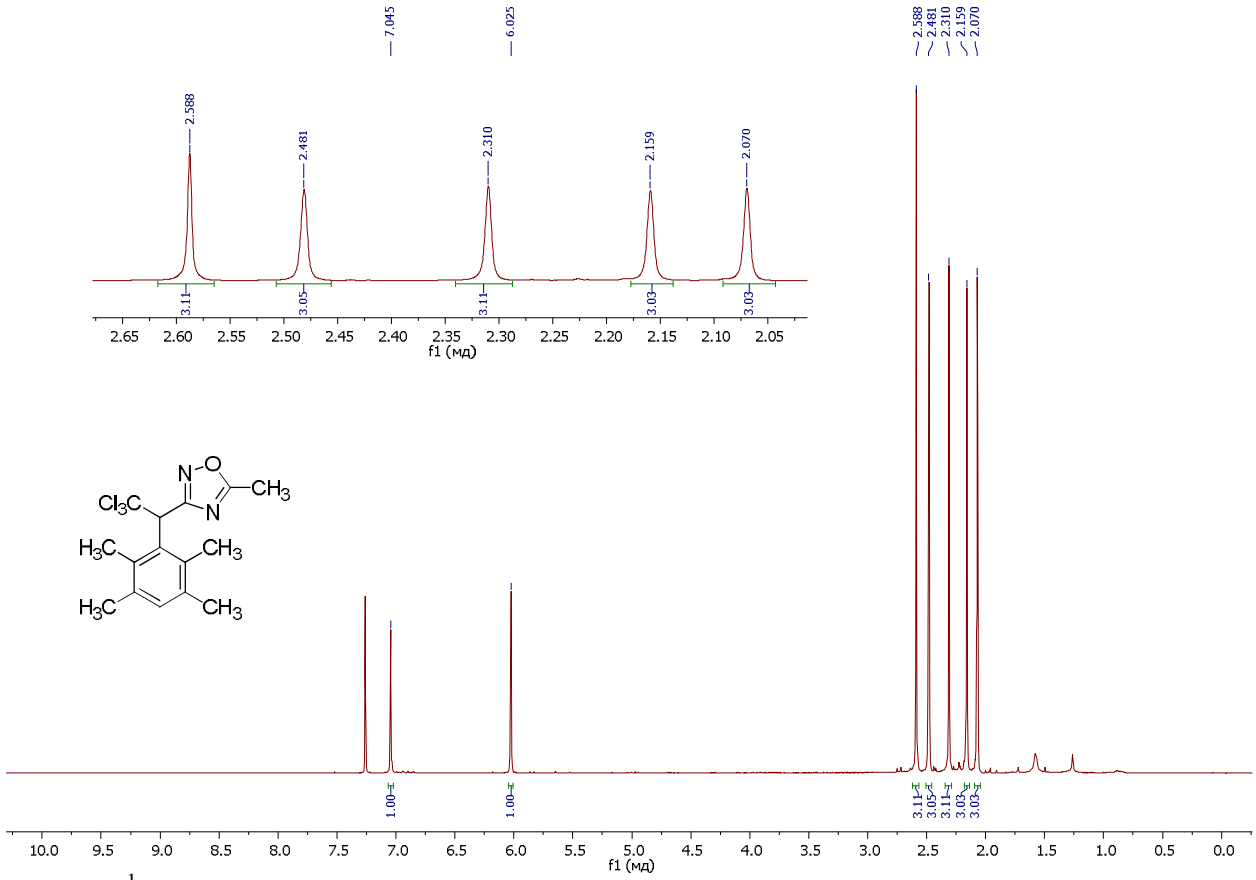

Fig. S34. ^{1}H NMR spectrum of the compound **2l** ($CDCl_3$, 400 MHz).

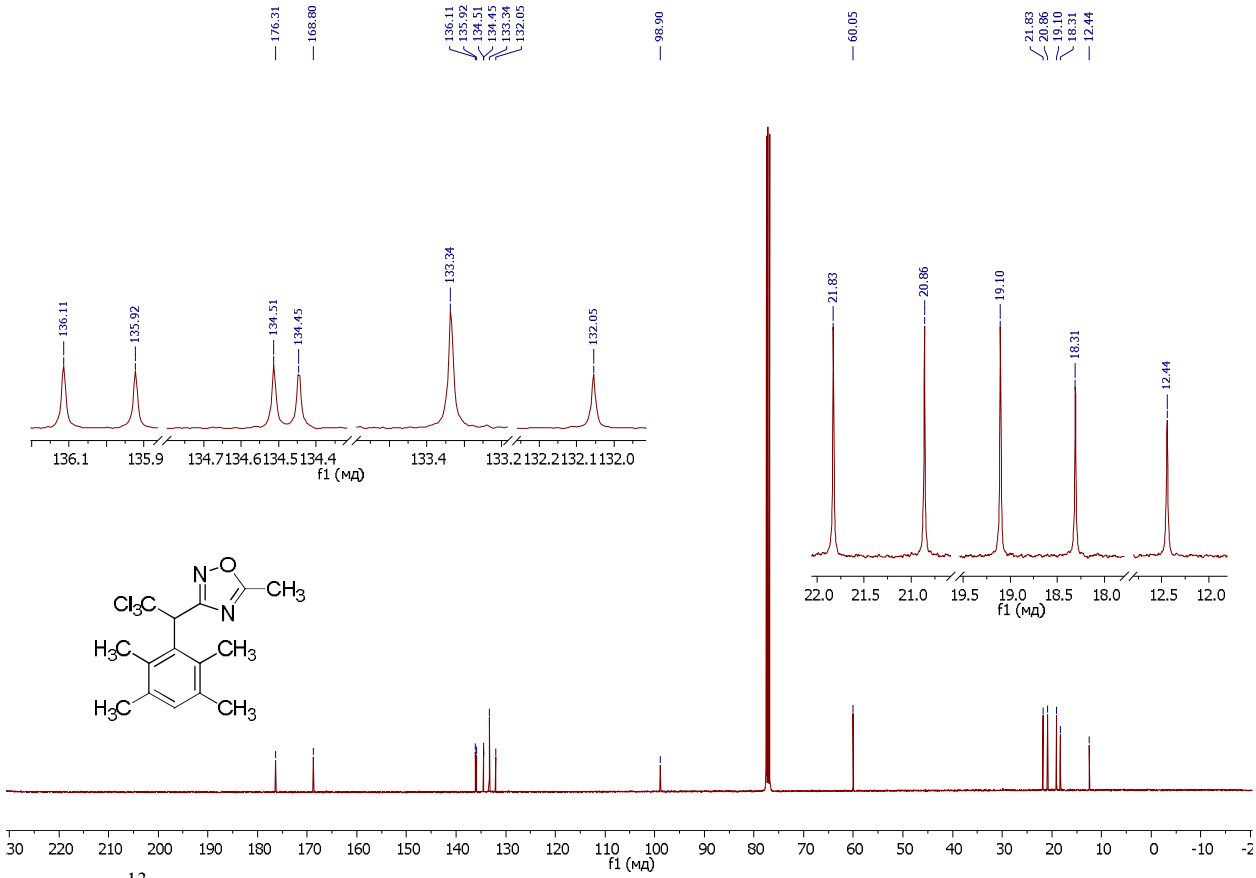

Fig. S35. $^{13}C\{H\}$ NMR spectrum of the compound **2l** ($CDCl_3$, 101 MHz).

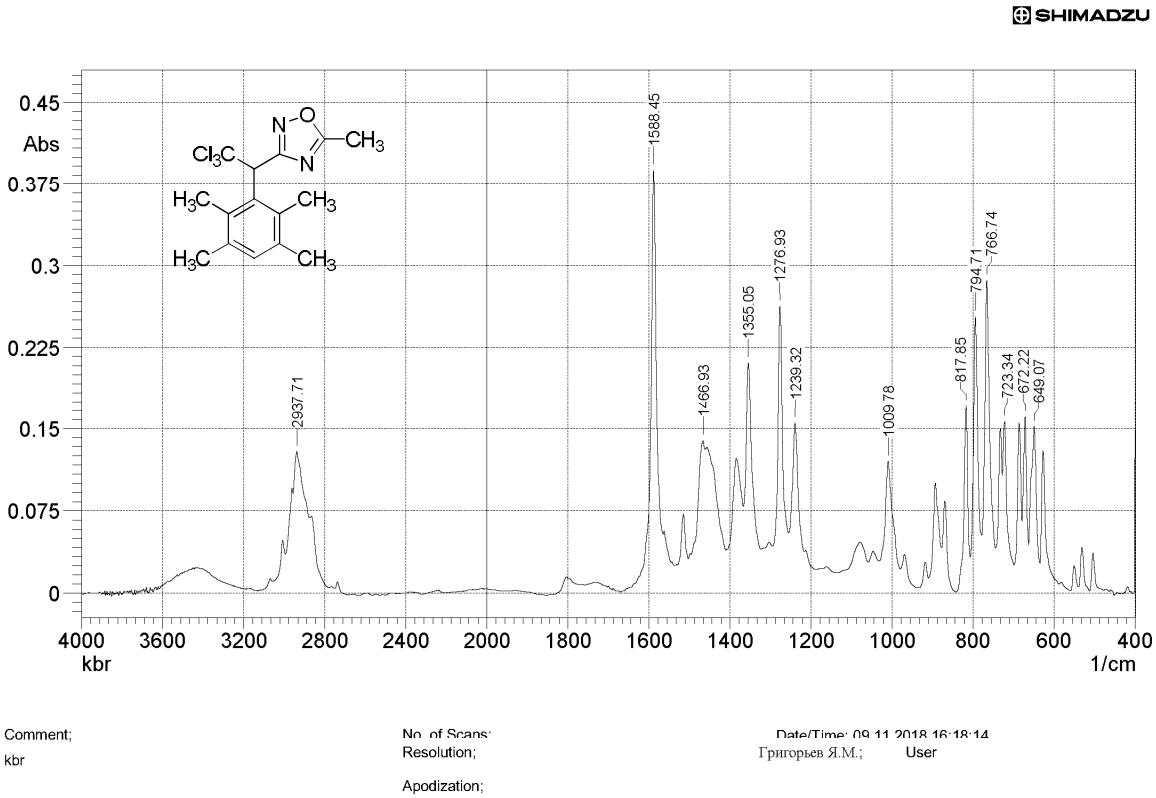

Fig. S36. IR spectrum of the compound **2l** (KBr).

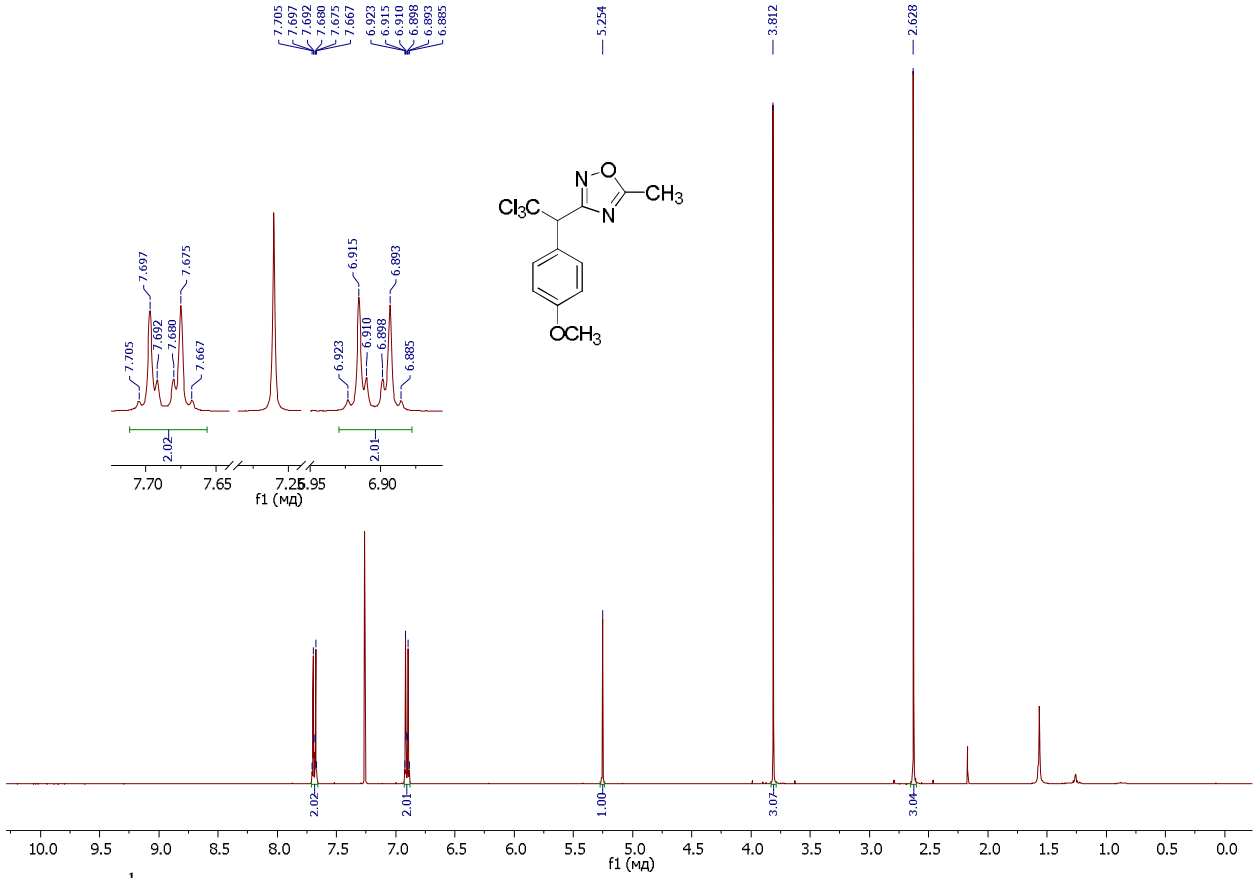

Fig. S37. ^{1}H NMR spectrum of the compound **2m** ($CDCl_3$, 400 MHz).

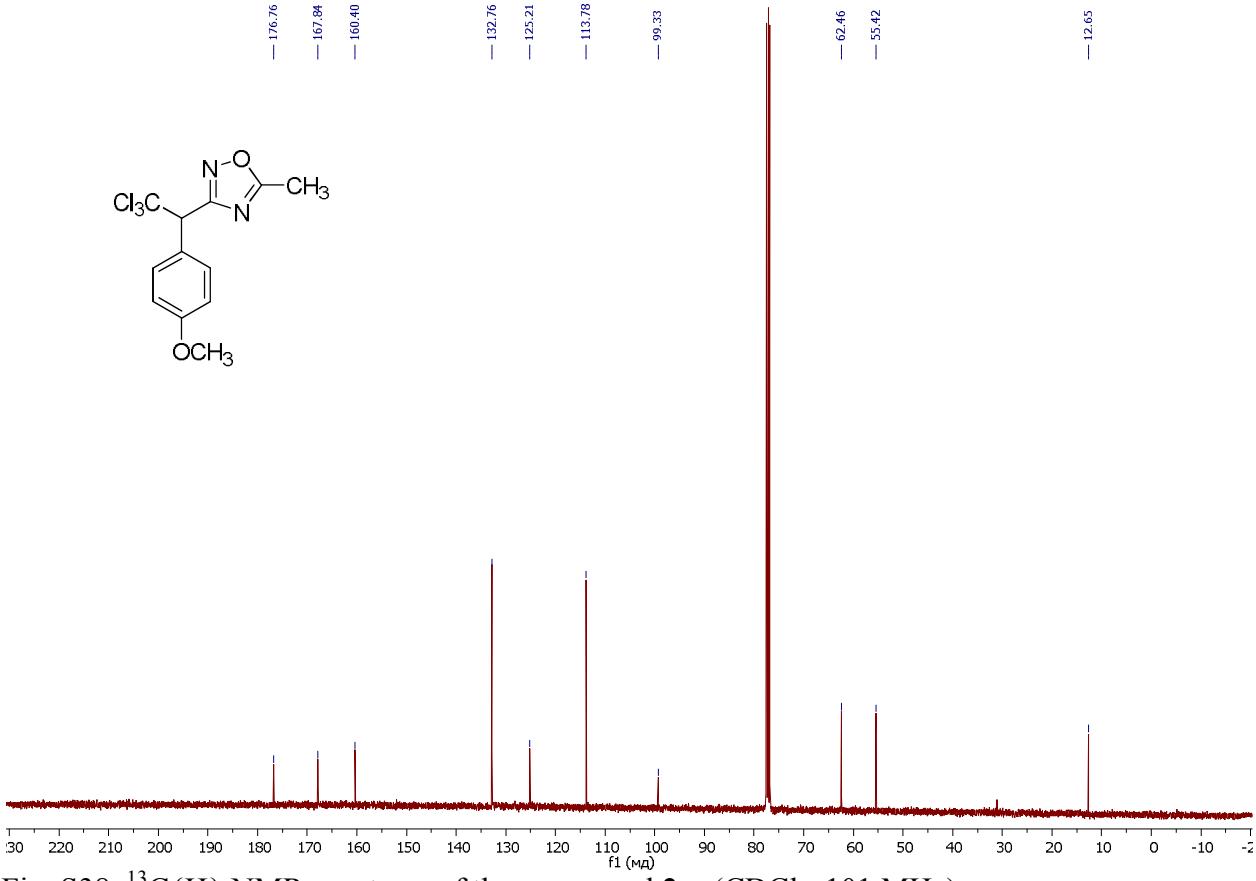

Fig. S38. ^{13}C{H} NMR spectrum of the compound **2m** ($CDCl_3$, 101 MHz).

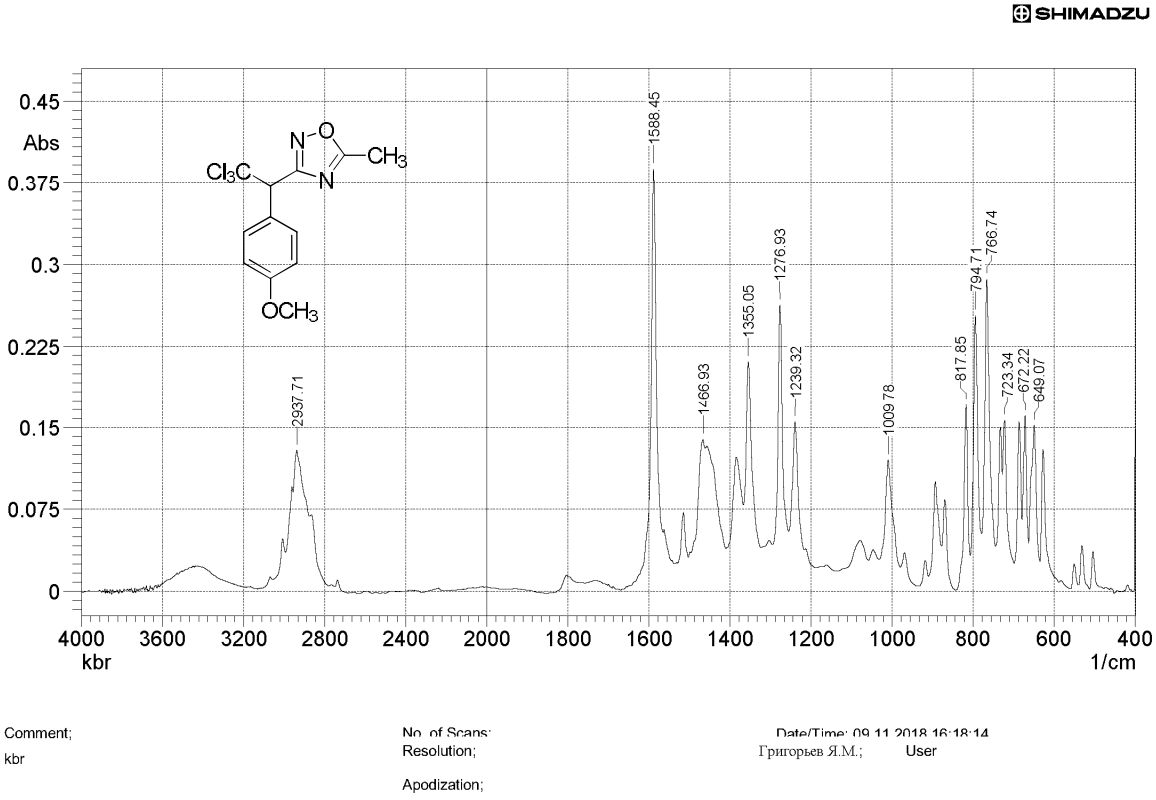

Fig. S39. IR spectrum of the compound **2m** (KBr).

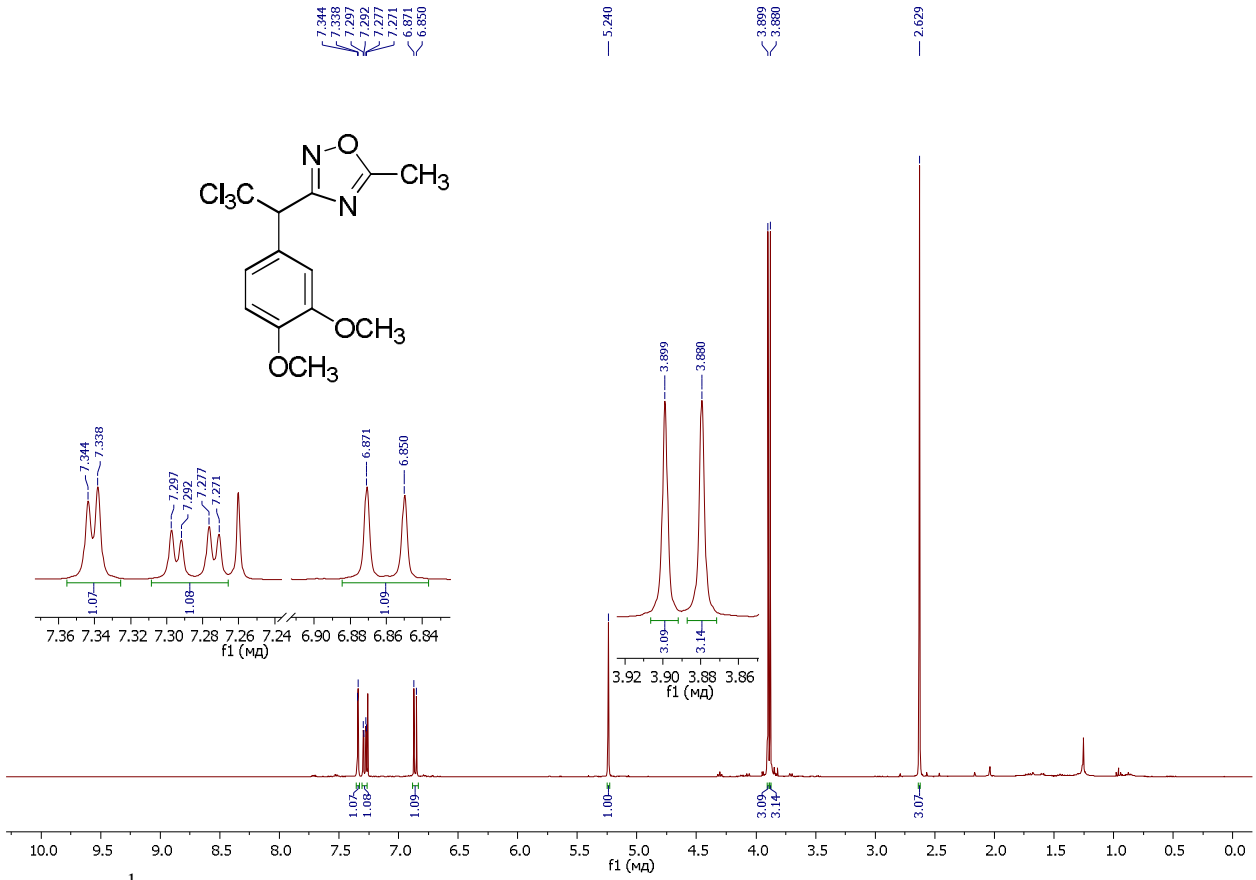

Fig. S40. ^{1}H NMR spectrum of the compound **2n** ($CDCl_3$, 400 MHz).

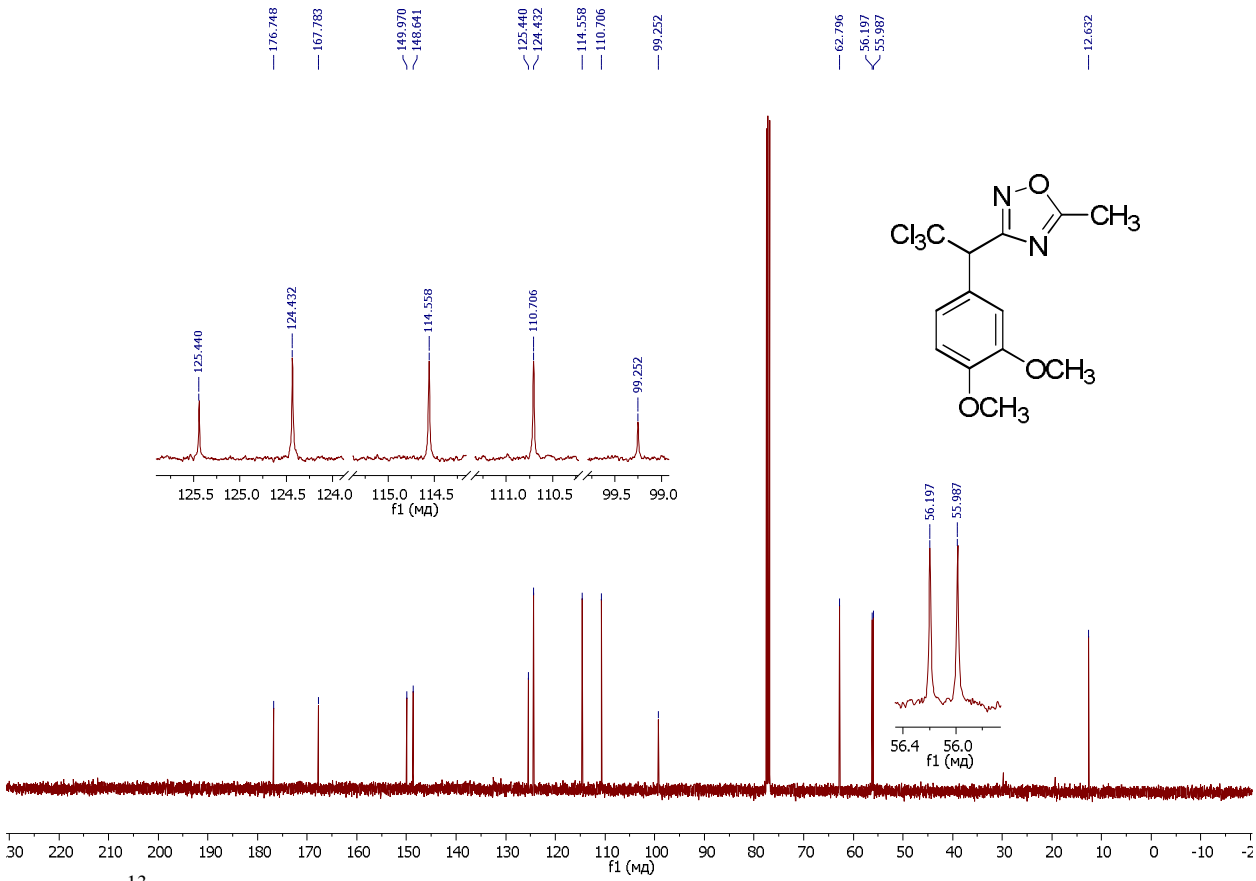

Fig. S41. $^{13}C\{H\}$ NMR spectrum of the compound **2n** ($CDCl_3$, 101 MHz).

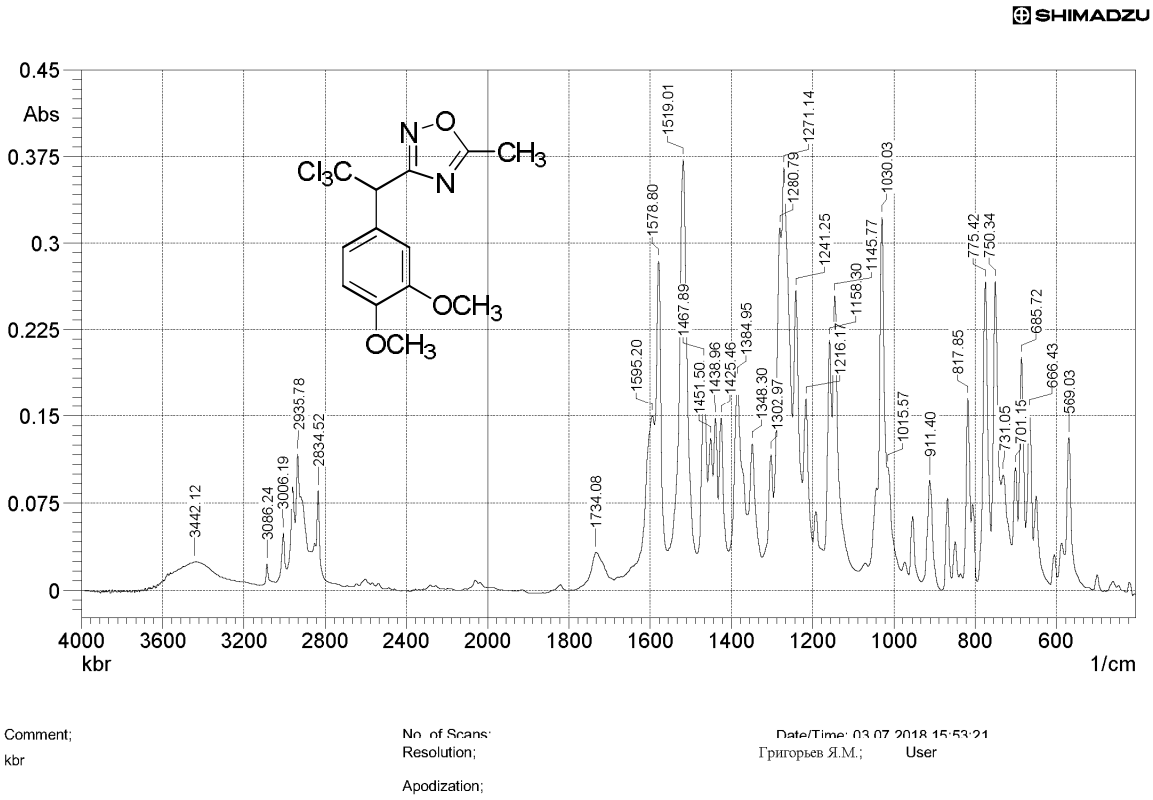

Fig. S42. IR spectrum of the compound **2n** (KBr).

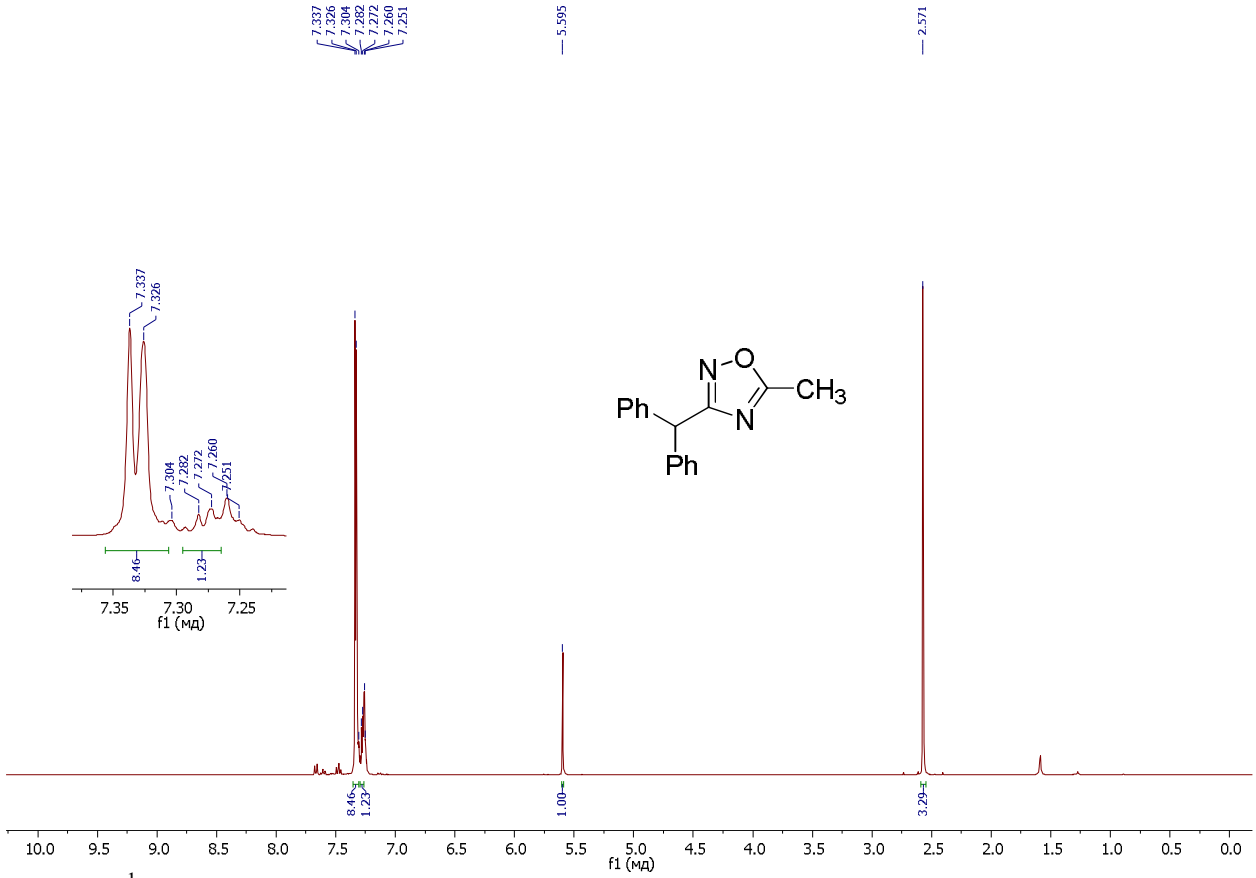

Fig. S43. ^{1}H NMR spectrum of the compound **2o** ($CDCl_3$, 400 MHz).

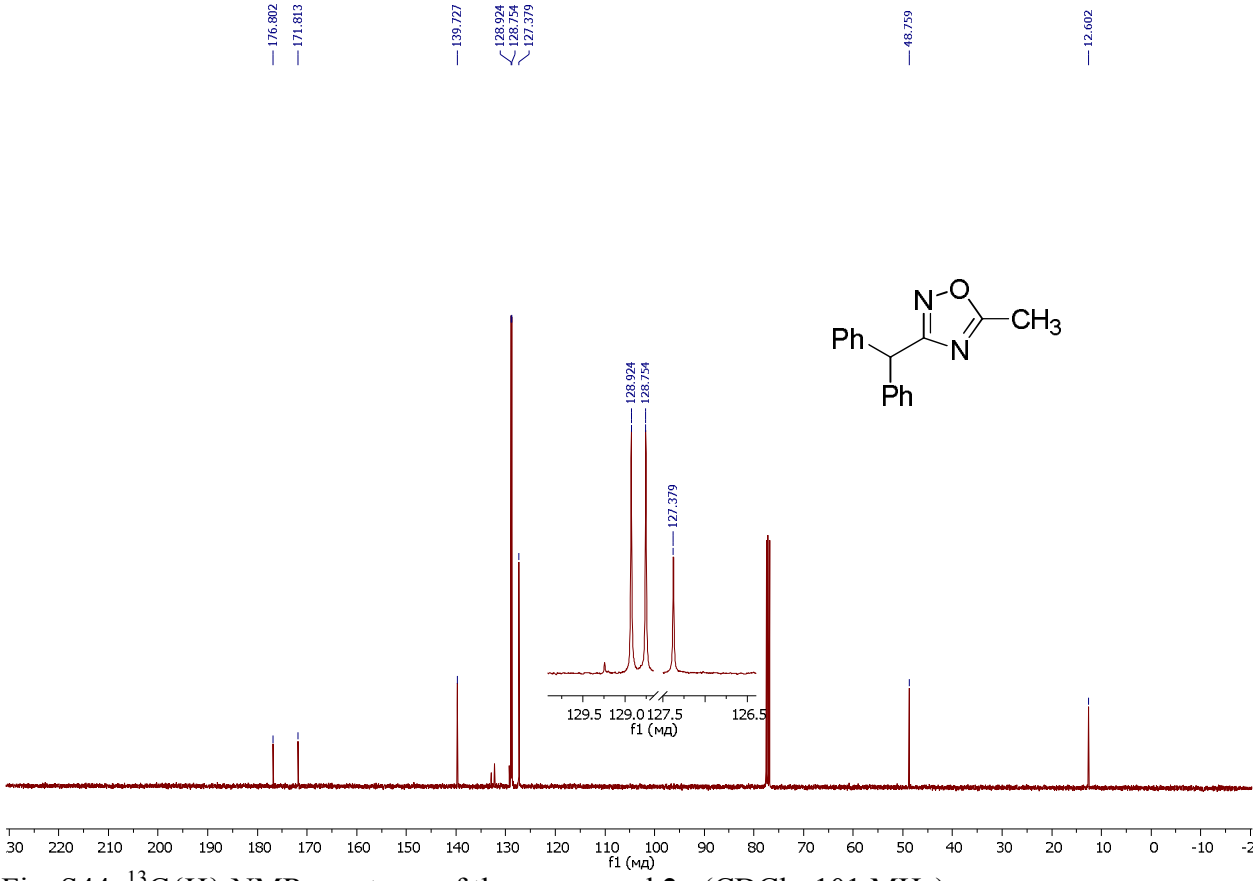

Fig. S44. ^{13}C{H} NMR spectrum of the compound **2o** ($CDCl_3$, 101 MHz).

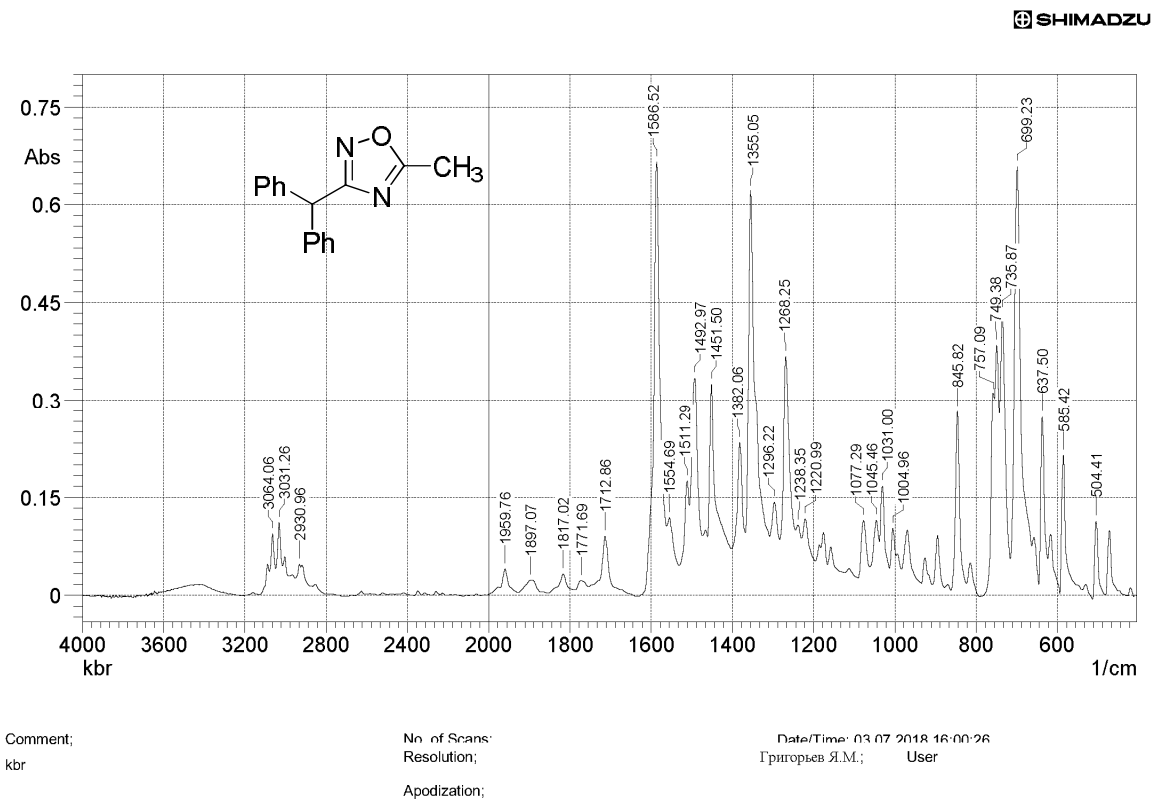

Fig. S45. IR spectrum of the compound **2o** (KBr).

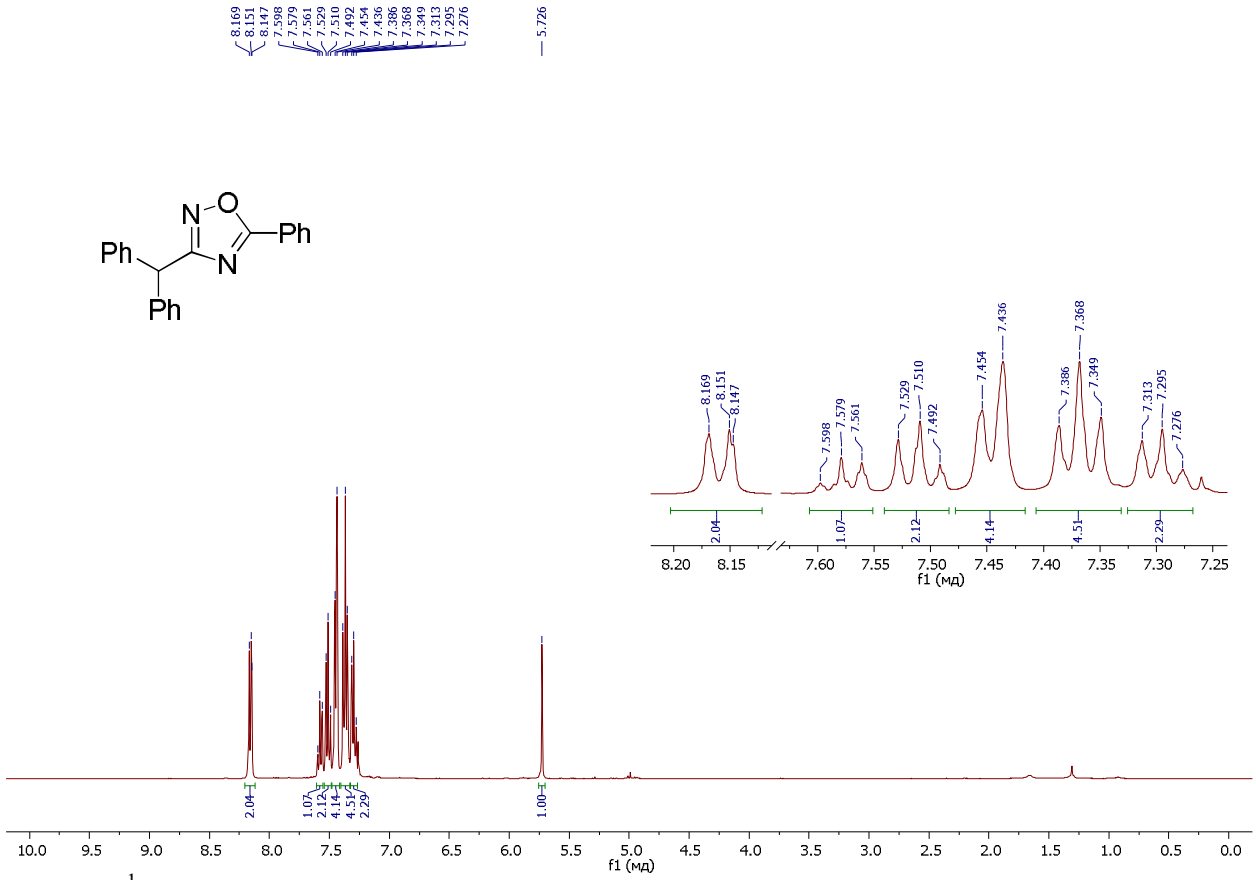

Fig. S46. ^{1}H NMR spectrum of the compound **2p** ($CDCl_3$, 400 MHz).

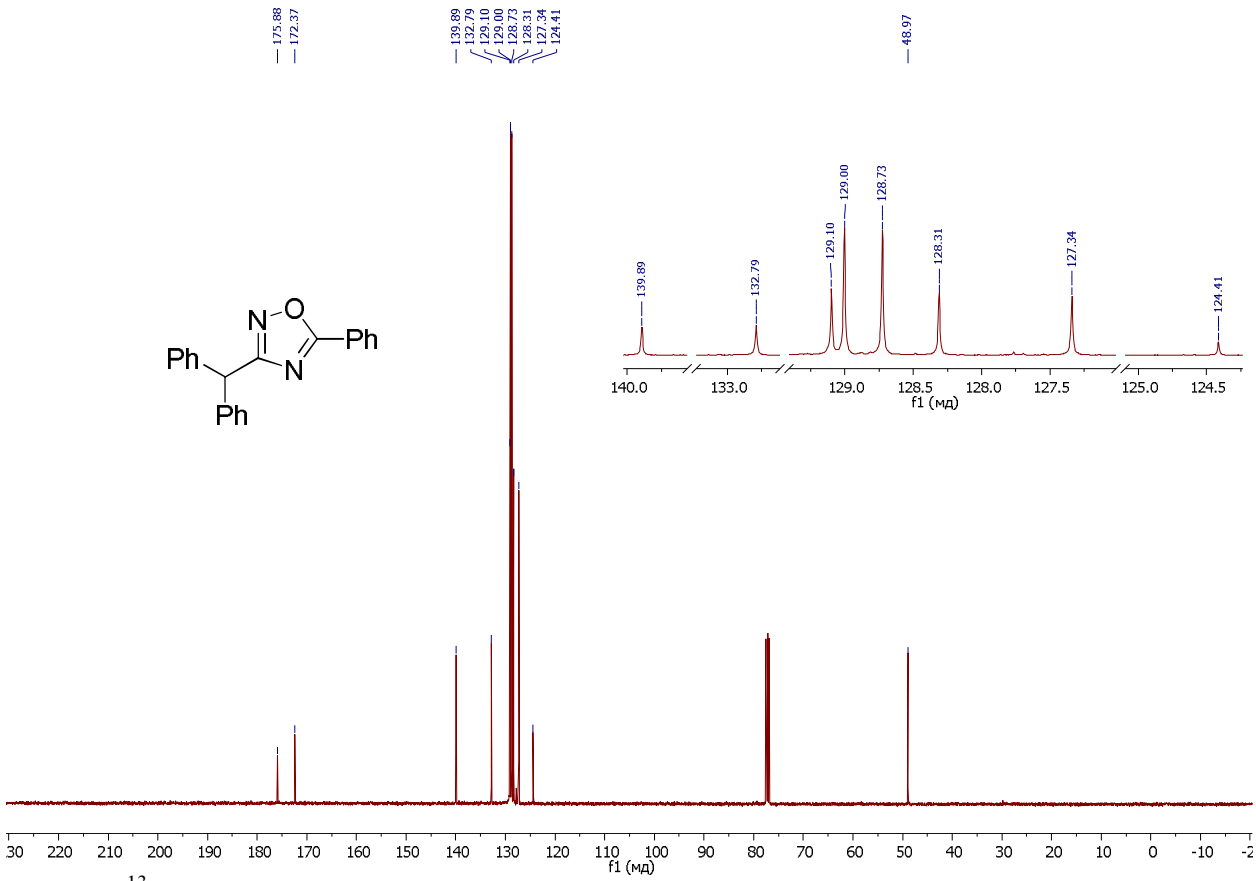

Fig. S47. $^{13}C\{H\}$ NMR spectrum of the compound **2p** ($CDCl_3$, 101 MHz).

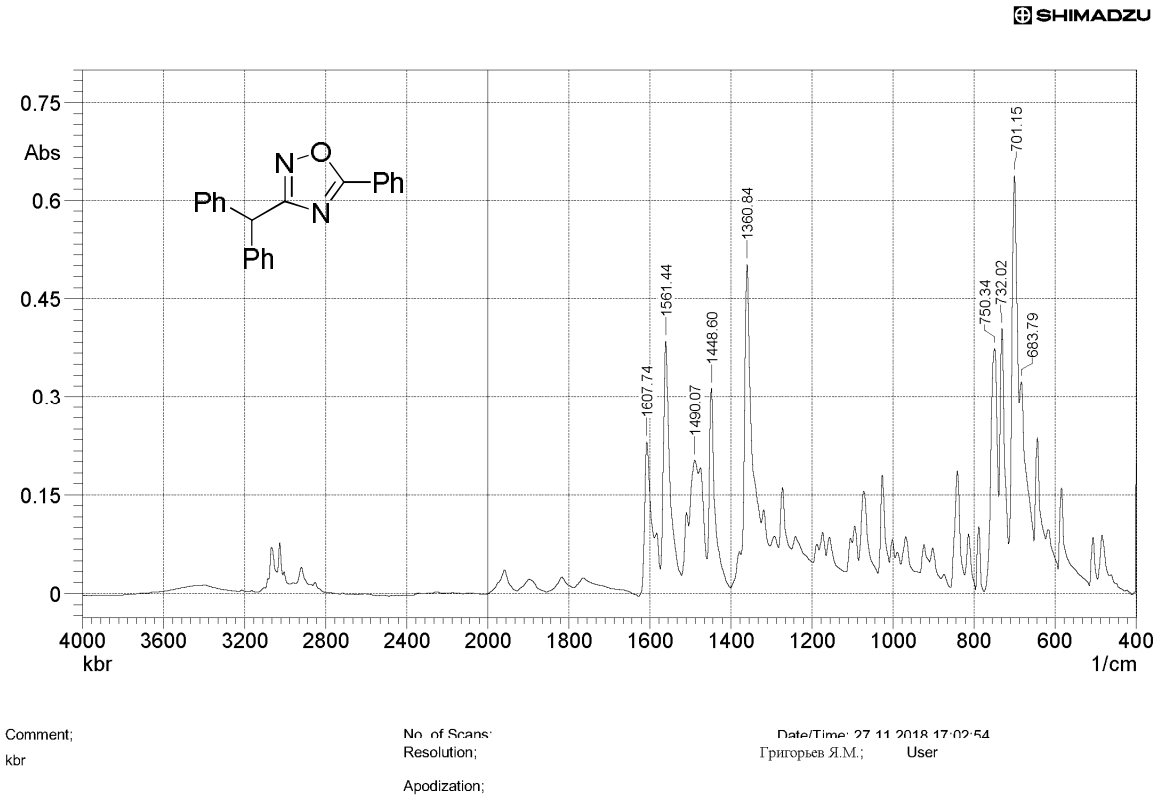

Fig. S48. IR spectrum of the compound **2p** (KBr).

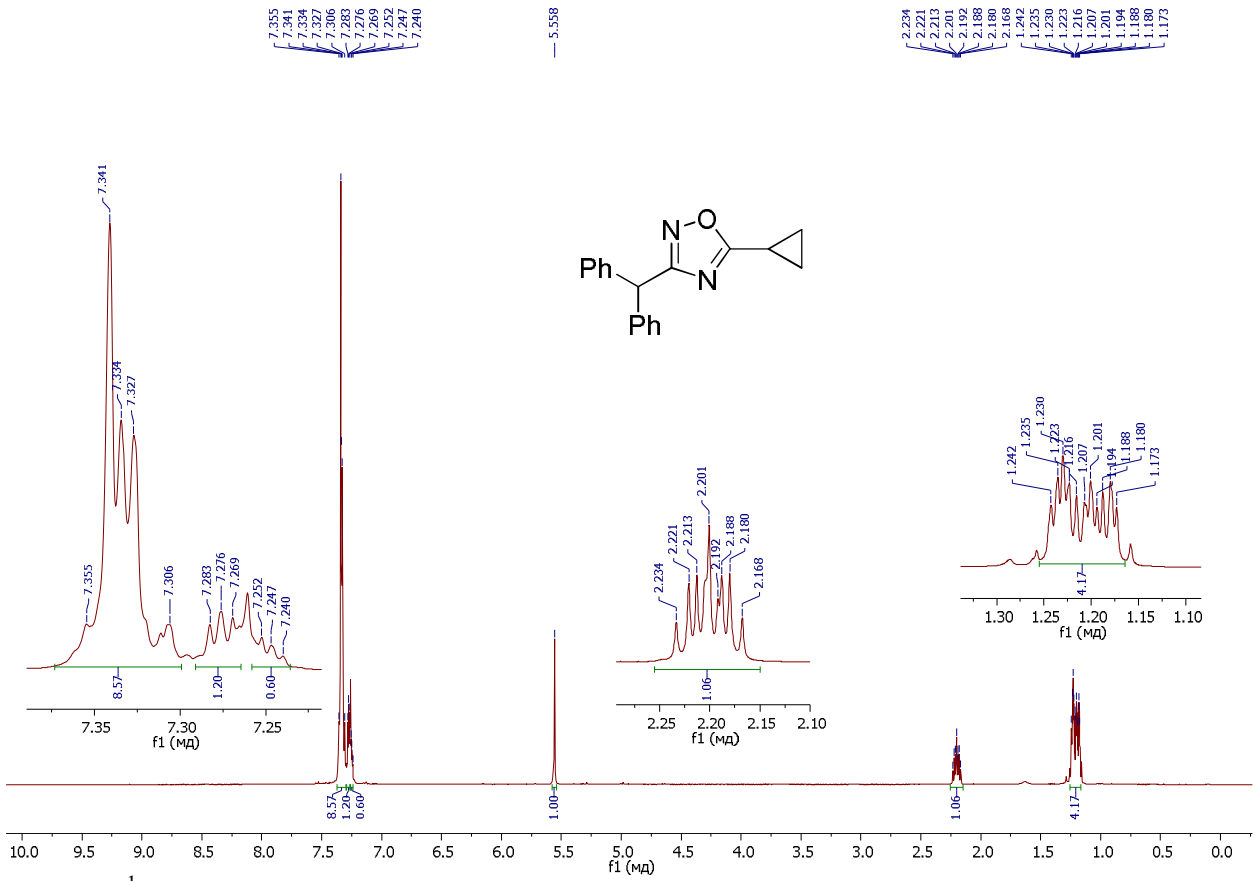

Fig. S49. ^{1}H NMR spectrum of the compound **2q** ($CDCl_3$, 400 MHz).

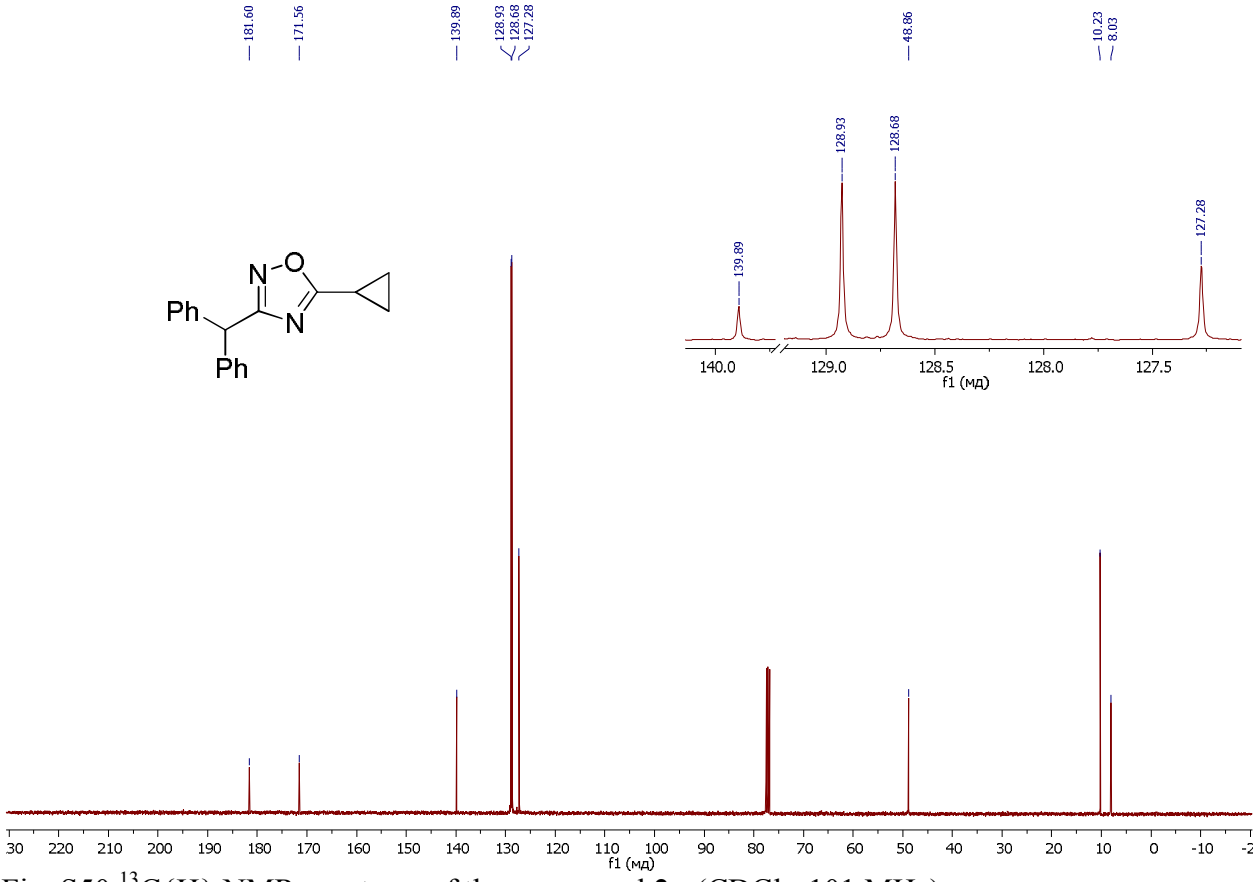

Fig. S50. ^{13}C{H} NMR spectrum of the compound **2q** ($CDCl_3$, 101 MHz).

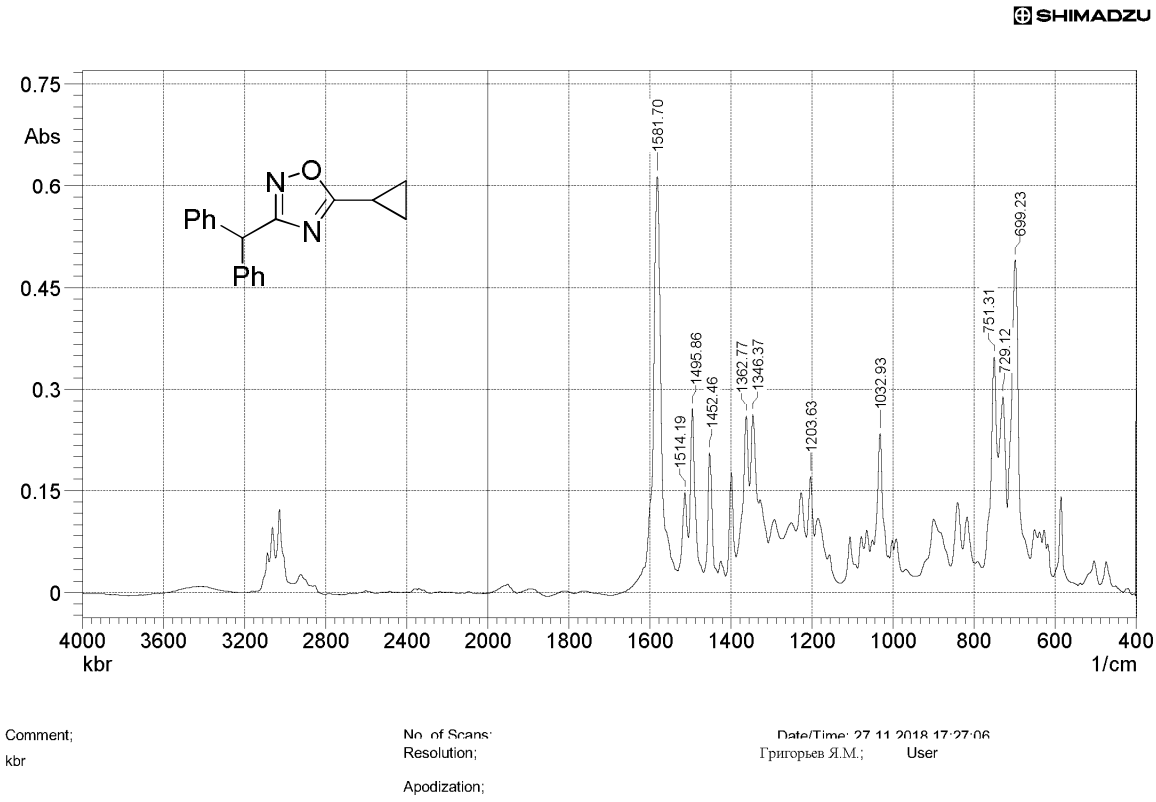

Fig. S51. IR spectrum of the compound **2q** (KBr).

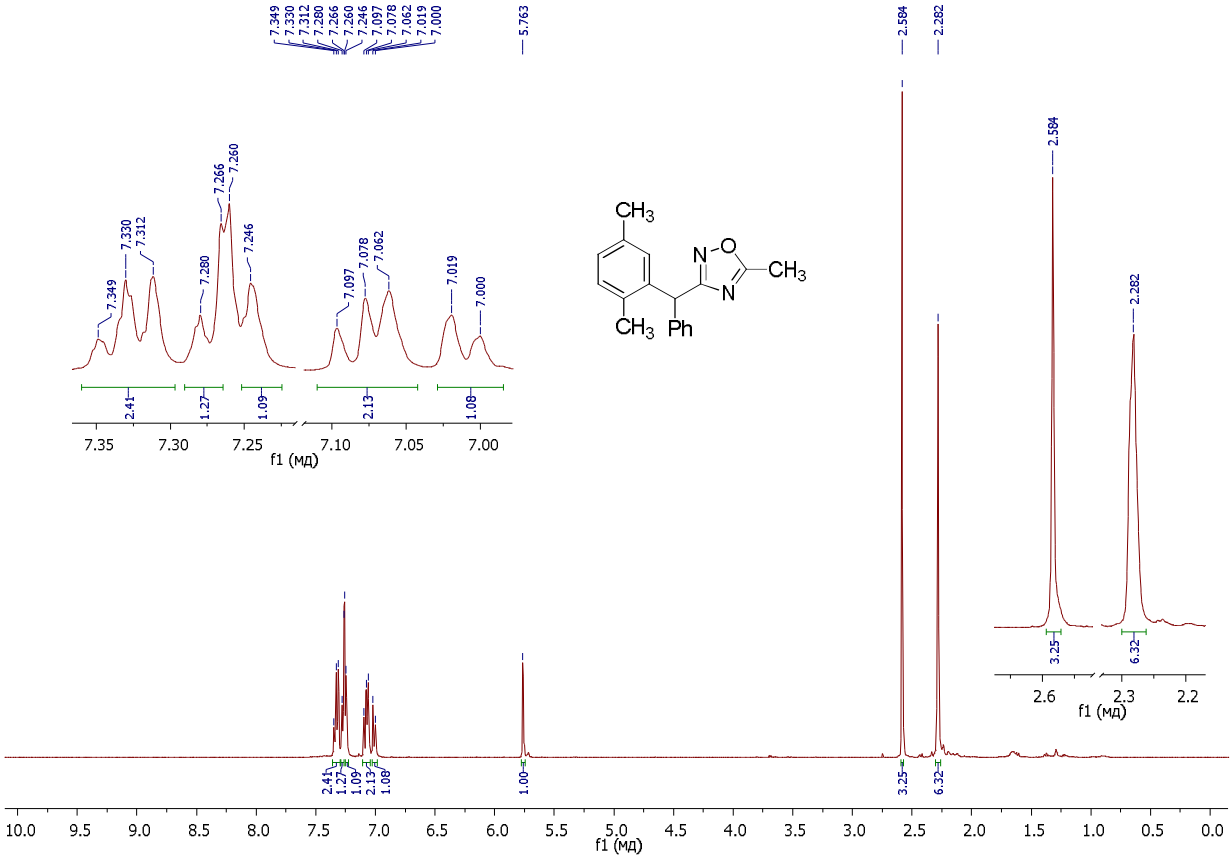

Fig. S52. ^{1}H NMR spectrum of the compound **2r** ($CDCl_3$, 400 MHz).

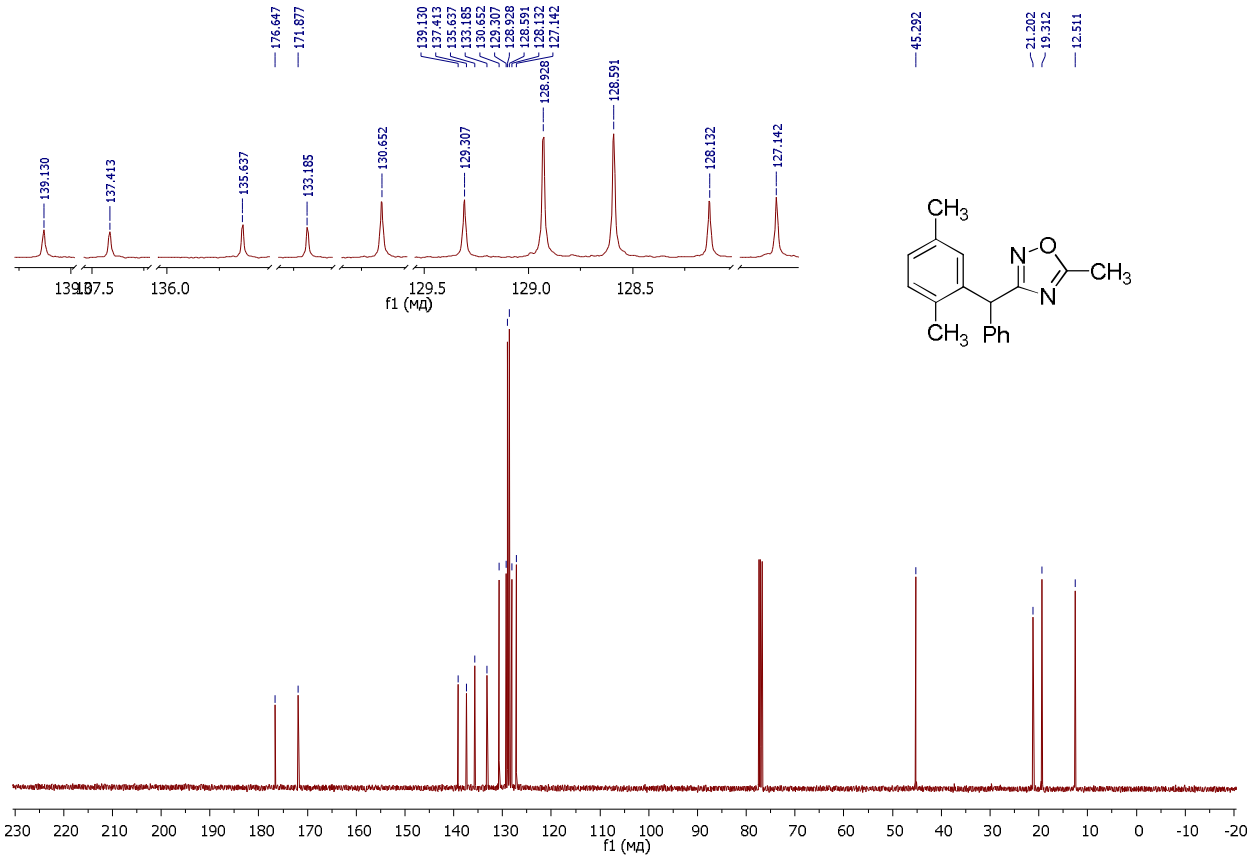

Fig. S53. $^{13}C\{H\}$ NMR spectrum of the compound **2r** ($CDCl_3$, 101 MHz).

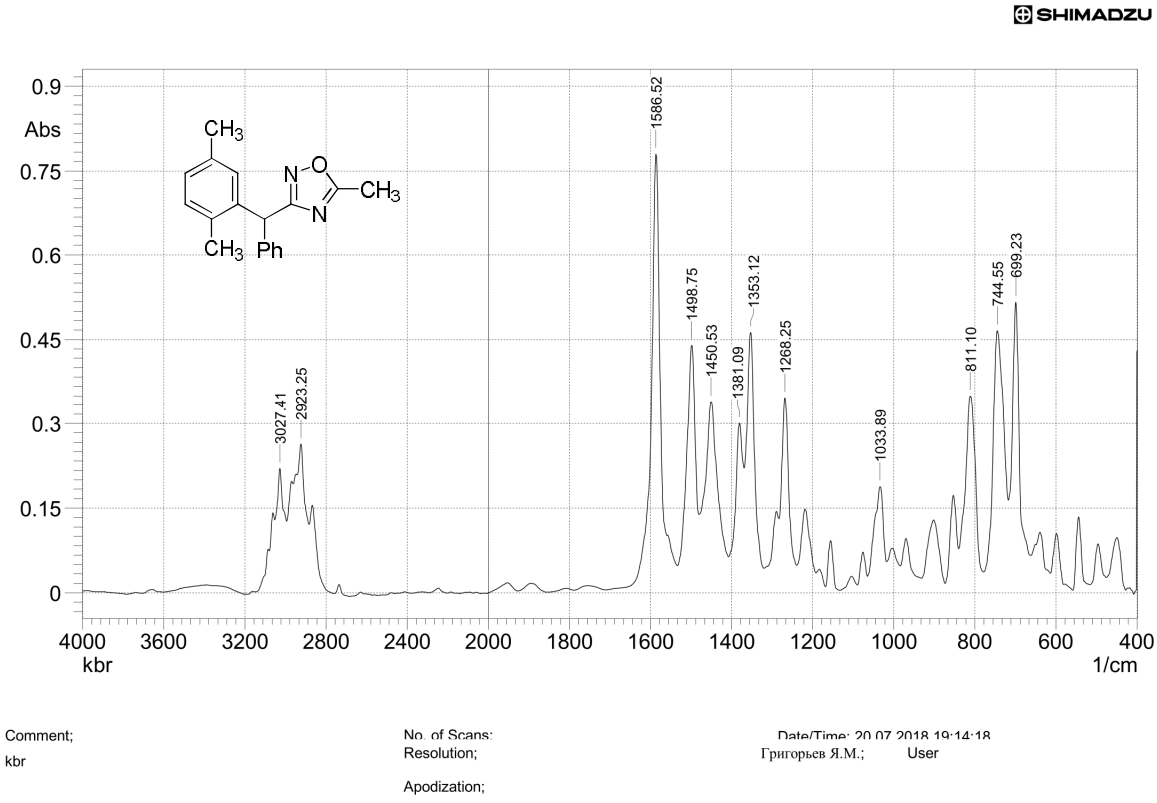

Fig. S54. IR spectrum of the compound **2r** (KBr).

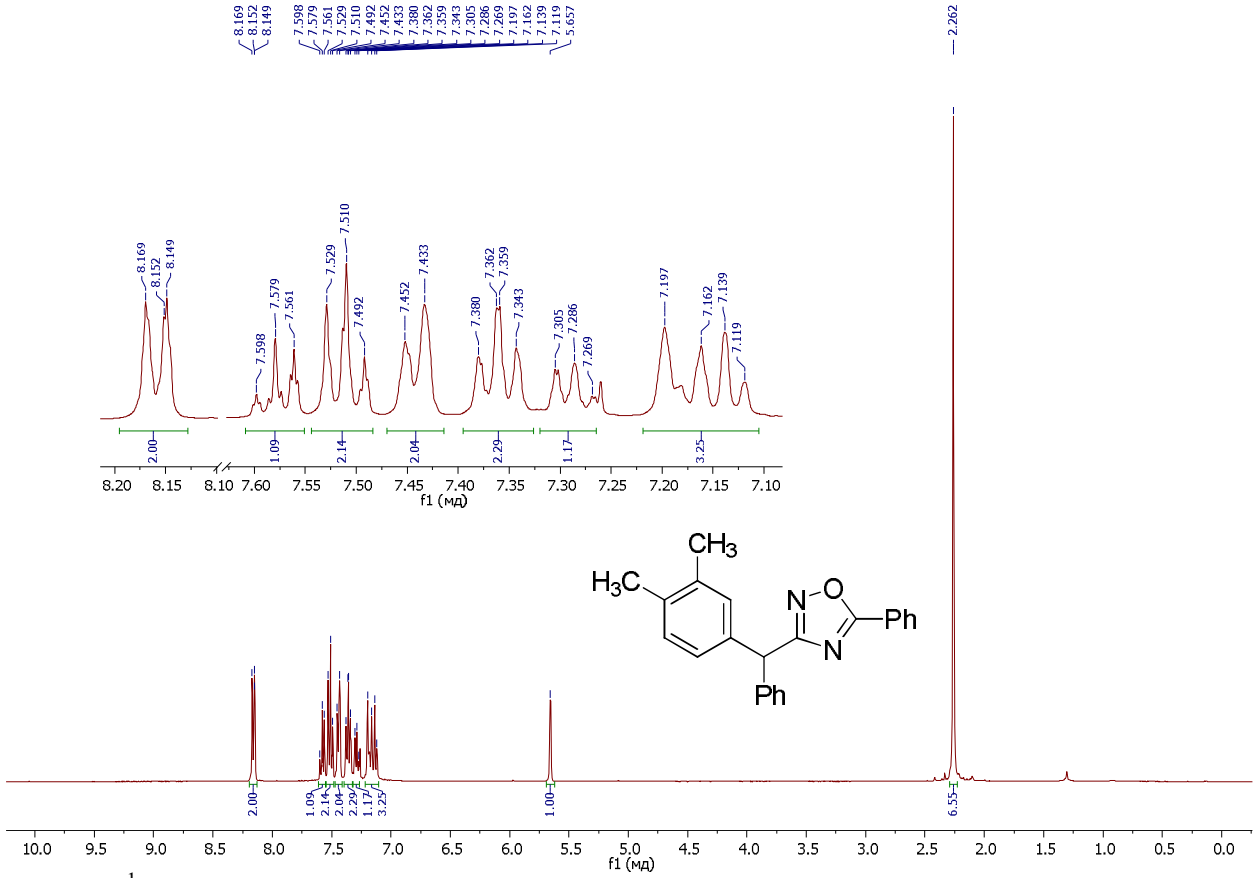

Fig. S55. ^{1}H NMR spectrum of the compound **2s** ($CDCl_3$, 400 MHz).

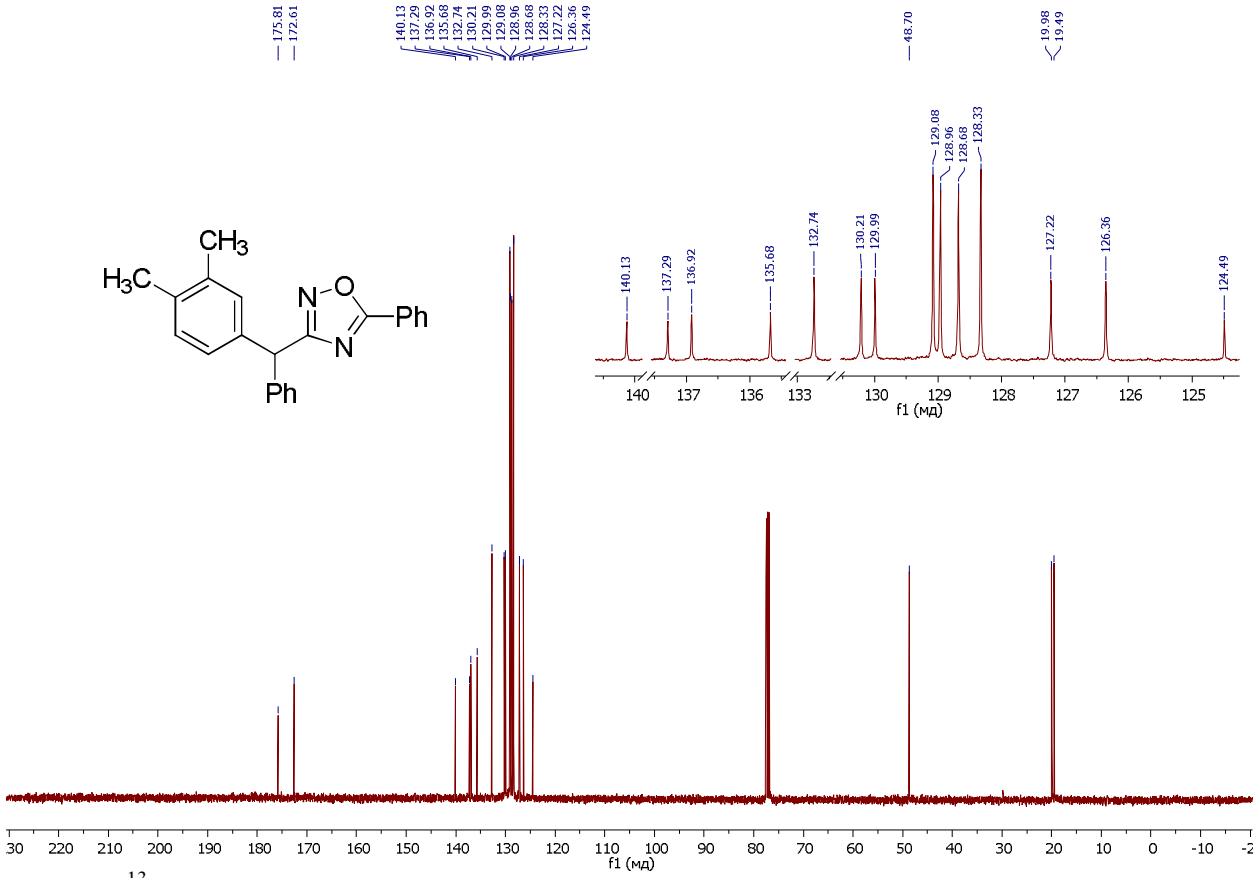

Fig. S56. ^{13}C{H} NMR spectrum of the compound **2s** ($CDCl_3$, 101 MHz).

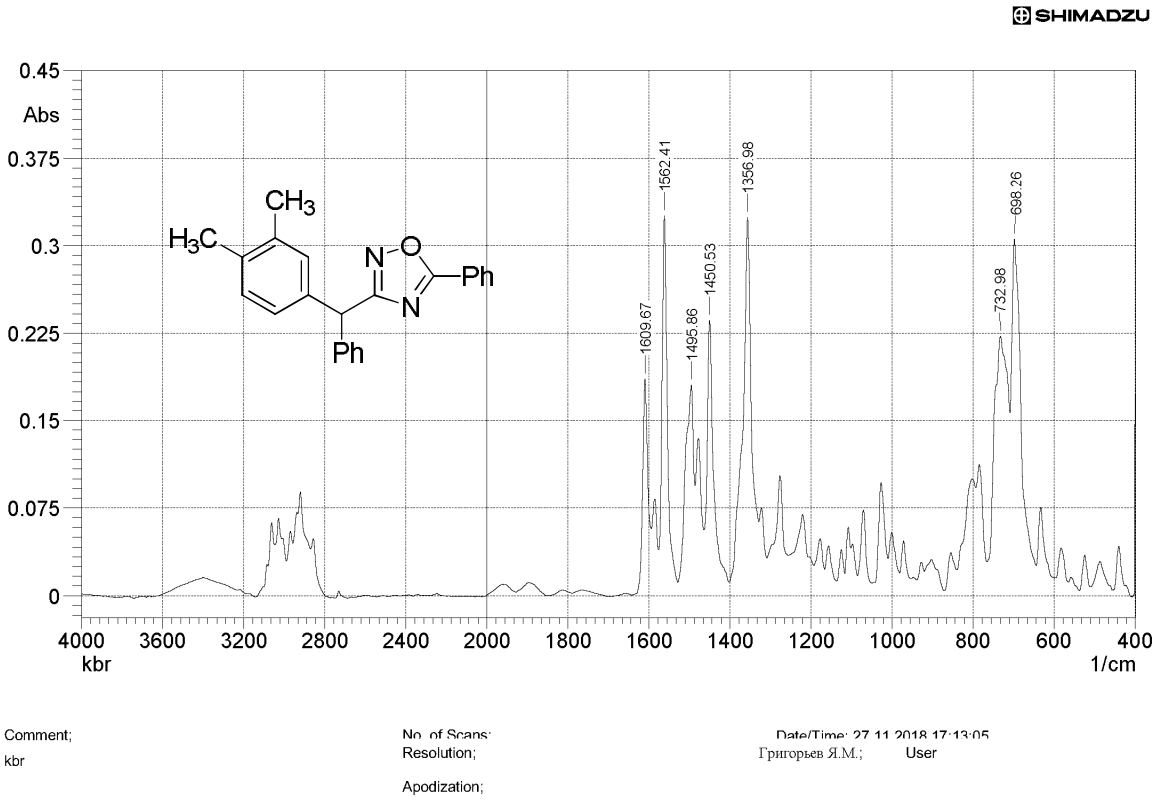

Fig. S57. IR spectrum of the compound **2s** (KBr).

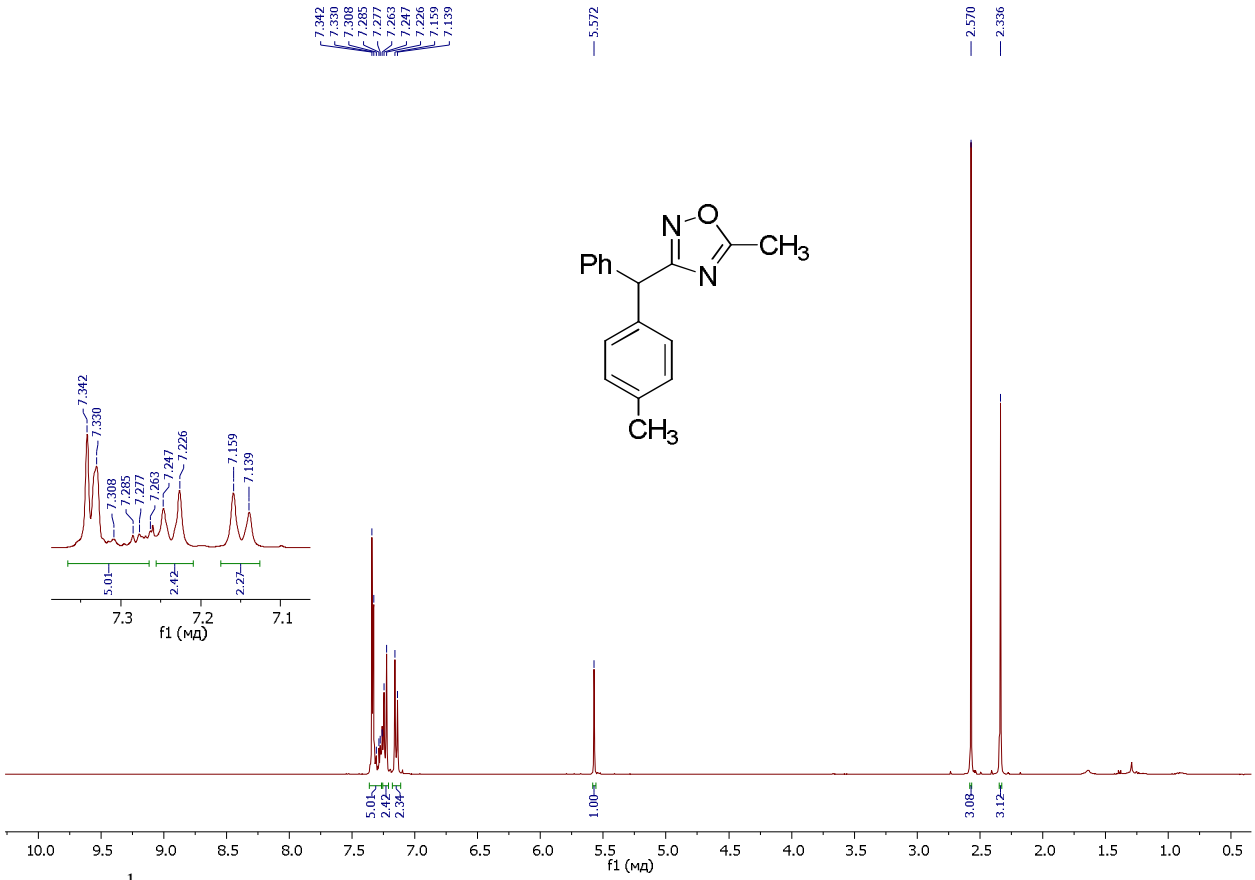

Fig. S58. ^{1}H NMR spectrum of the compound **2t** ($CDCl_3$, 400 MHz).

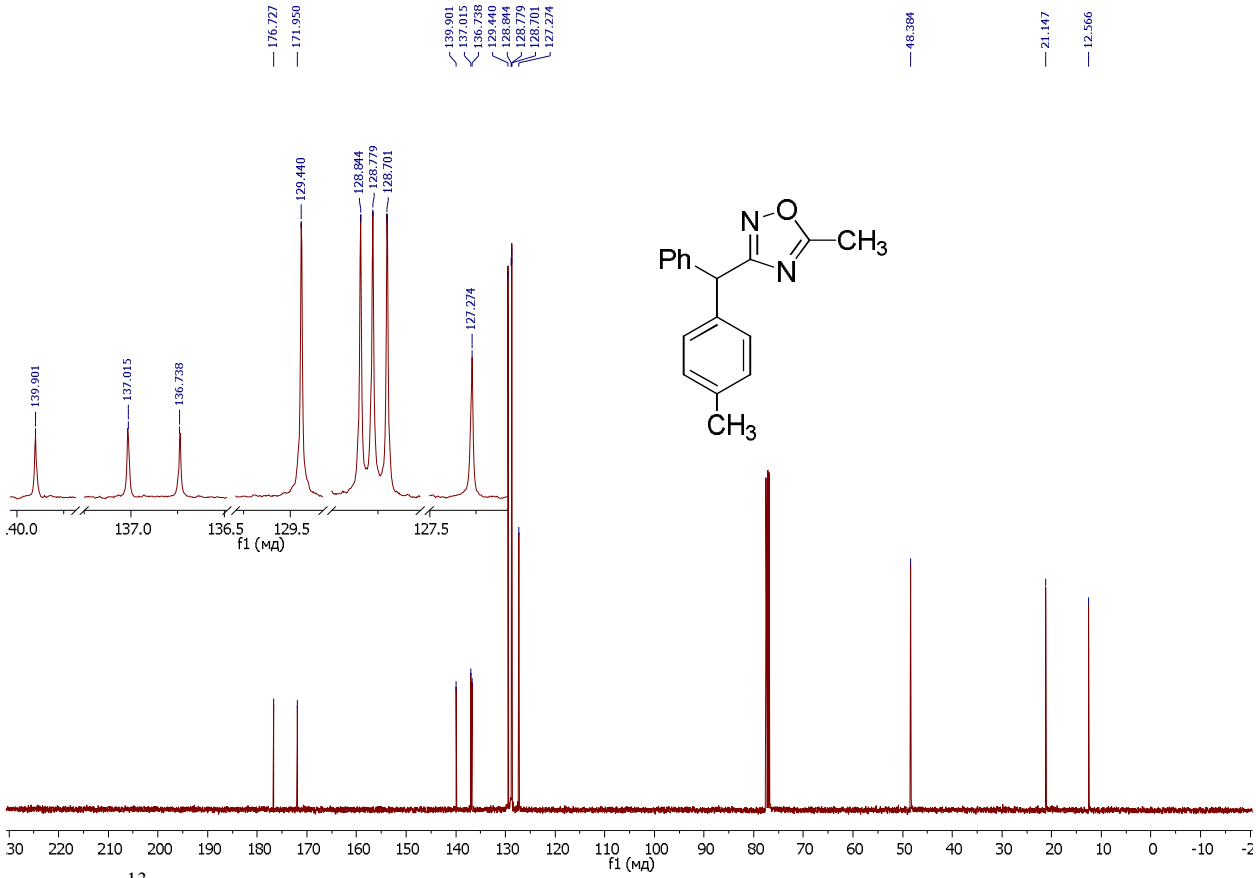

Fig. S59. $^{13}C\{H\}$ NMR spectrum of the compound **2t** ($CDCl_3$, 101 MHz).

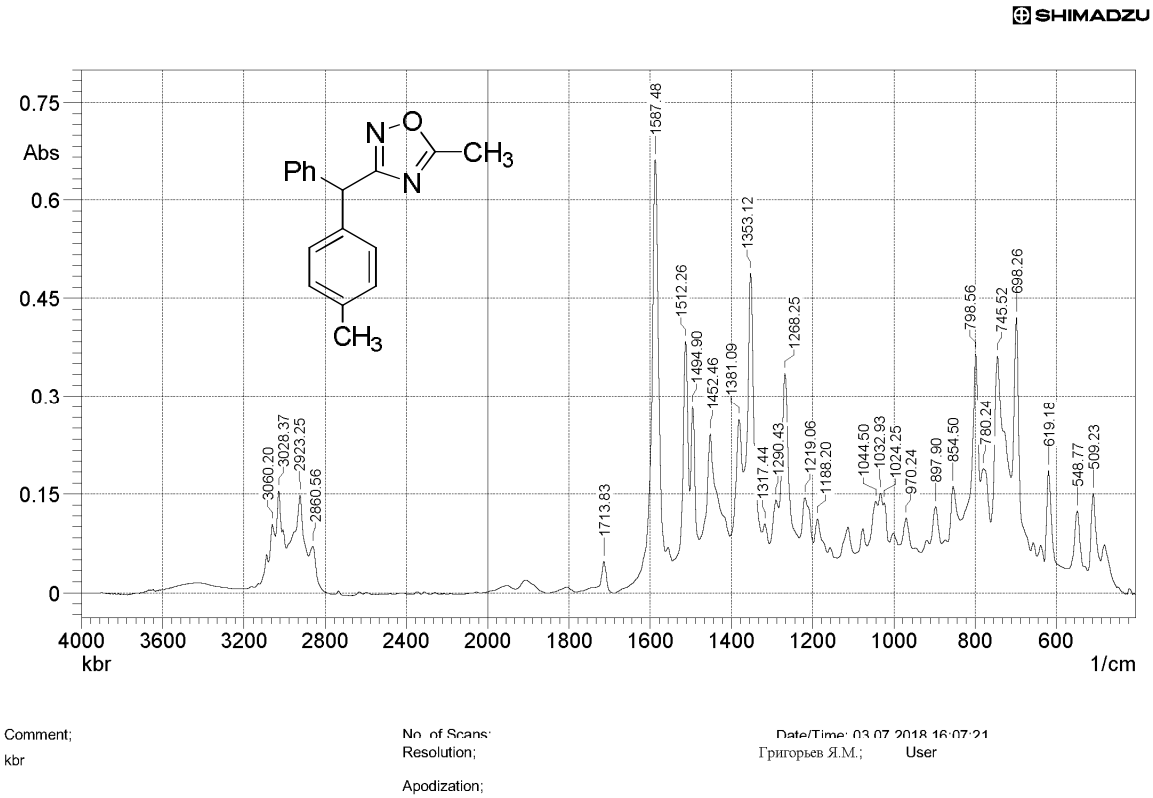

Fig. S60. IR spectrum of the compound **2t** (KBr).

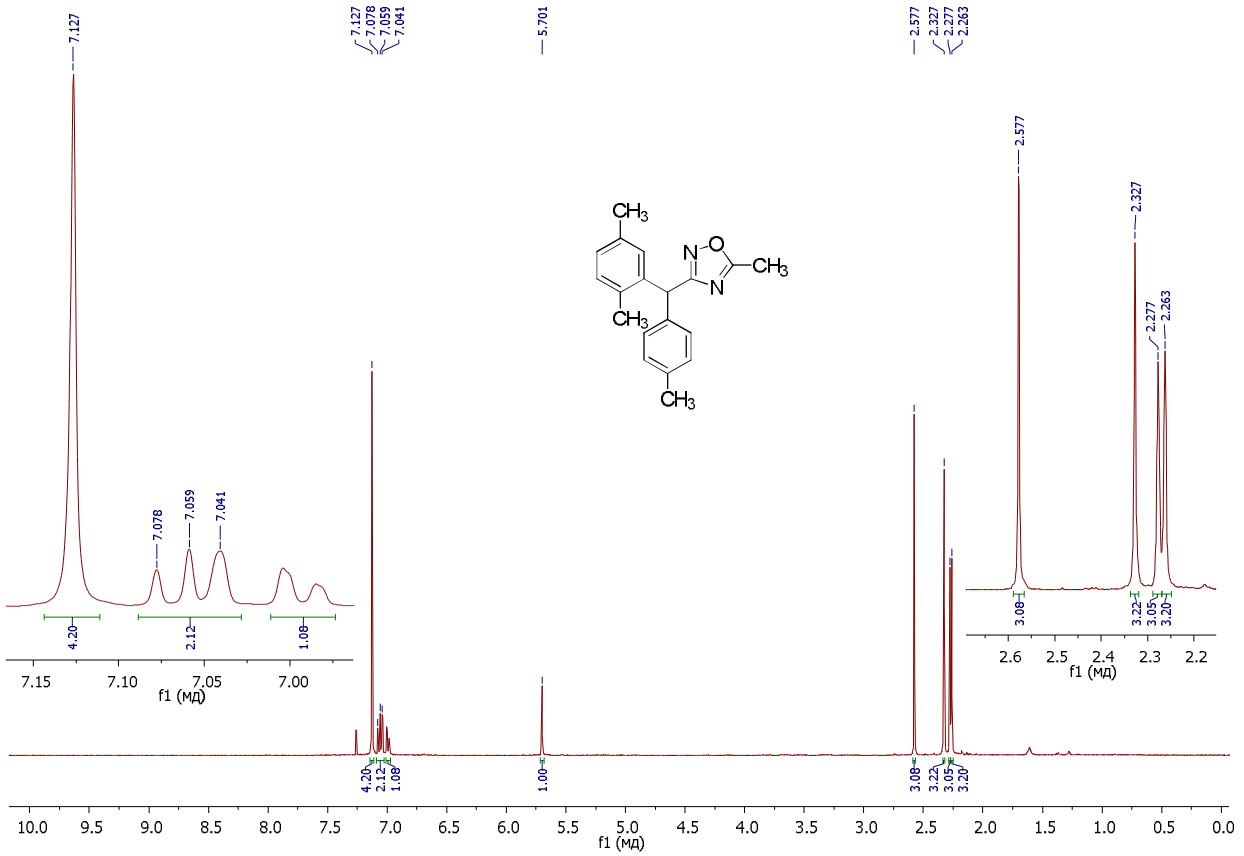

Fig. S61. ^{1}H NMR spectrum of the compound **2u** ($CDCl_3$, 400 MHz).

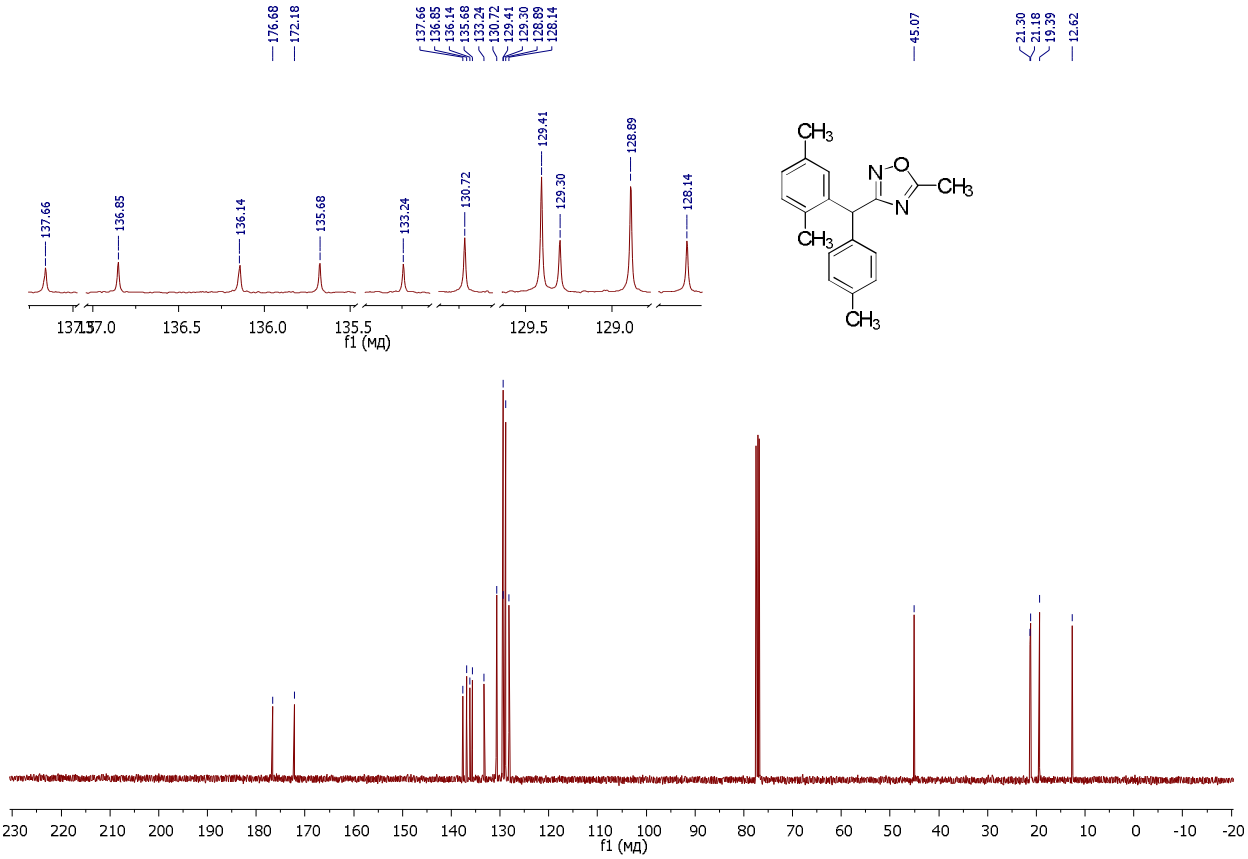

Fig. S62. ^{13}C{H} NMR spectrum of the compound **2u** ($CDCl_3$, 101 MHz).

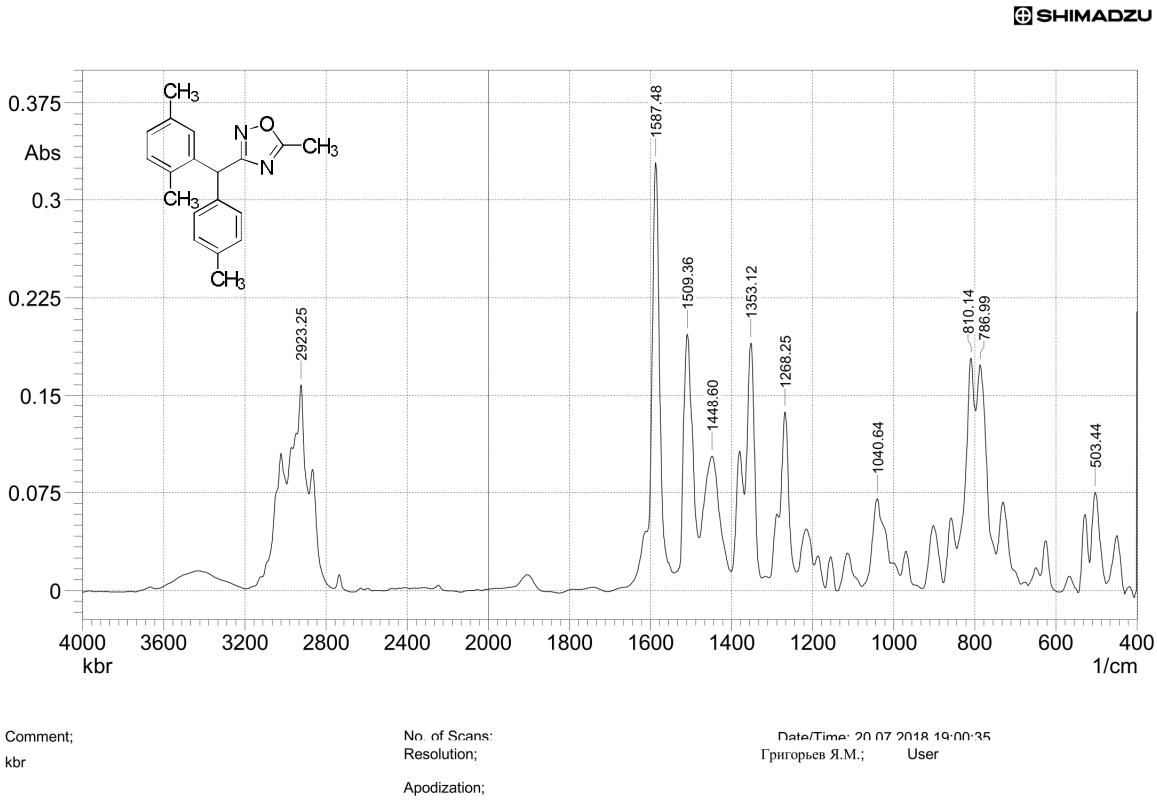

Fig. S63. IR spectrum of the compound **2u** (KBr).

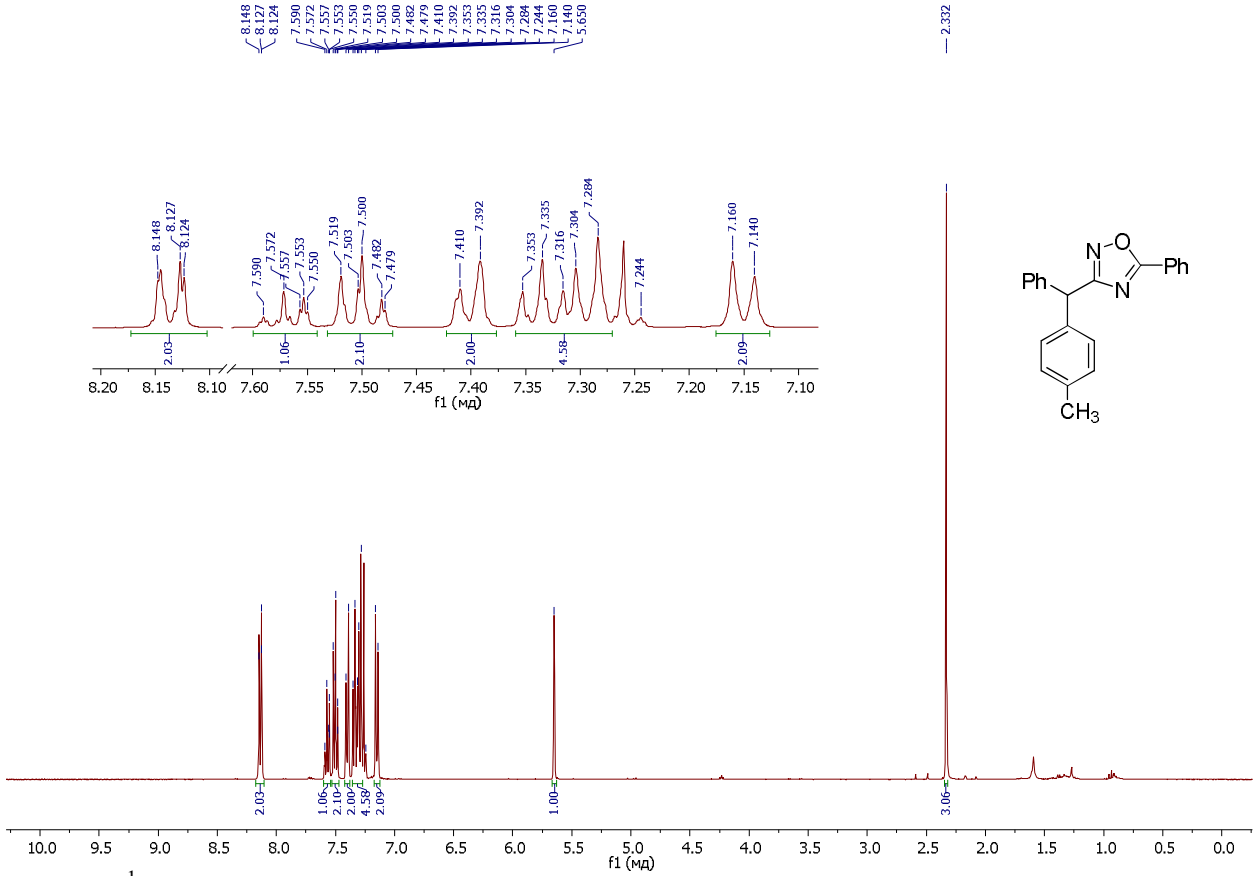

Fig. S64. ^{1}H NMR spectrum of the compound **2v** ($CDCl_3$, 400 MHz).

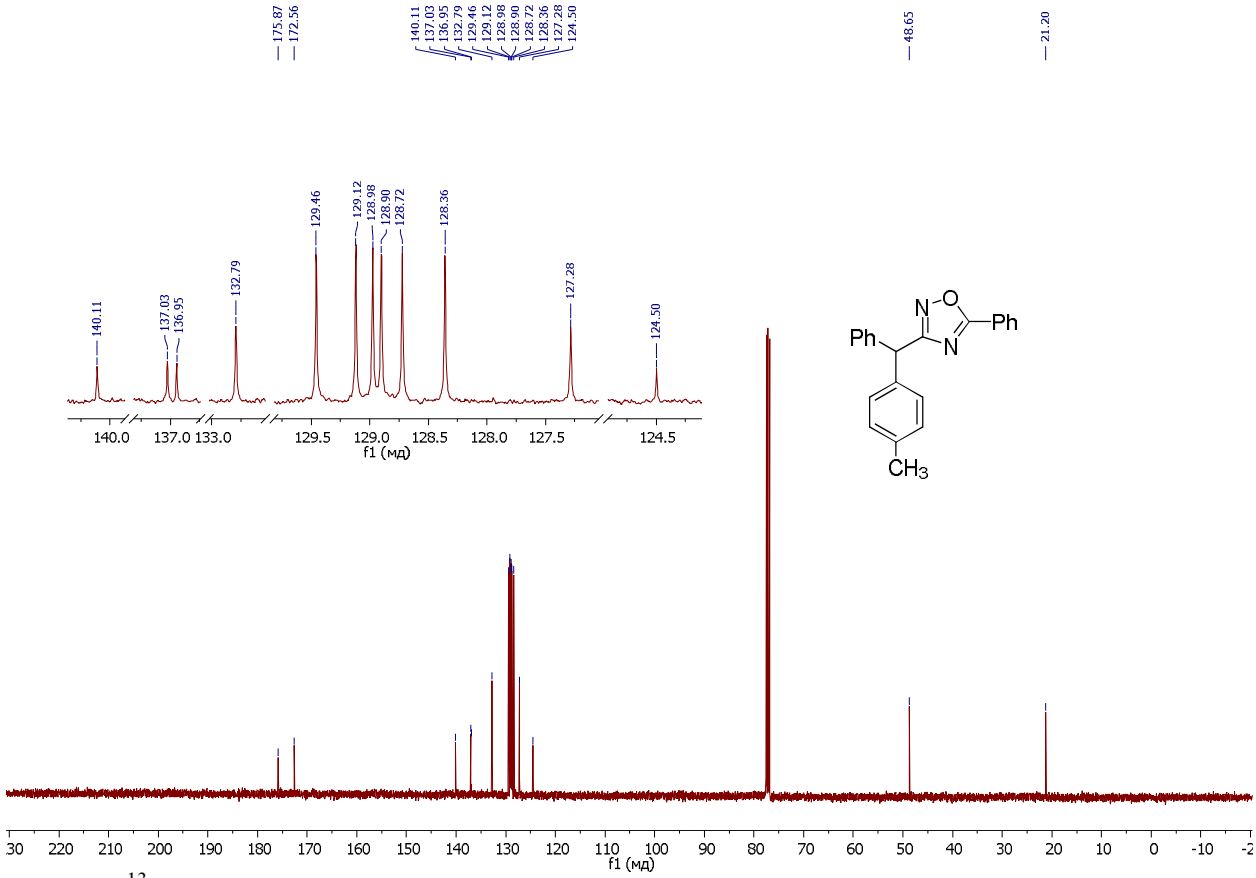

Fig. S65. $^{13}C\{H\}$ NMR spectrum of the compound **2v** ($CDCl_3$, 101 MHz).

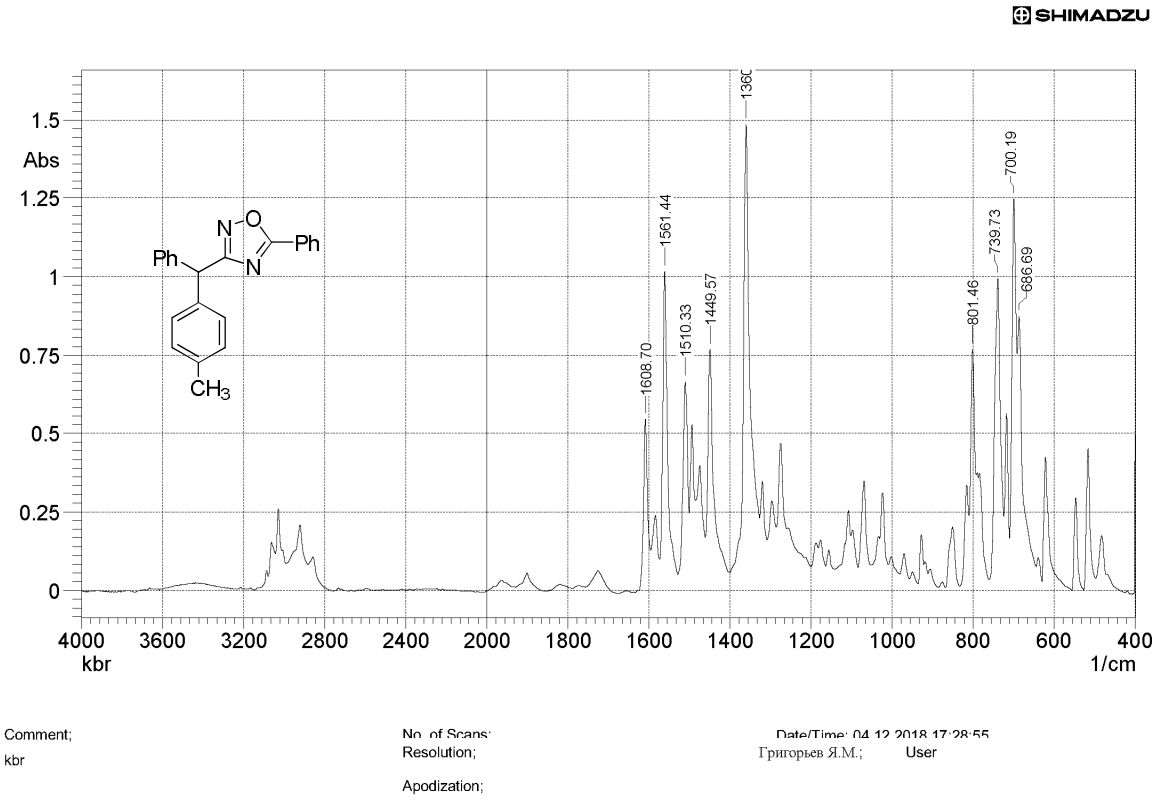

Fig. S66. IR spectrum of the compound **2v** (KBr).

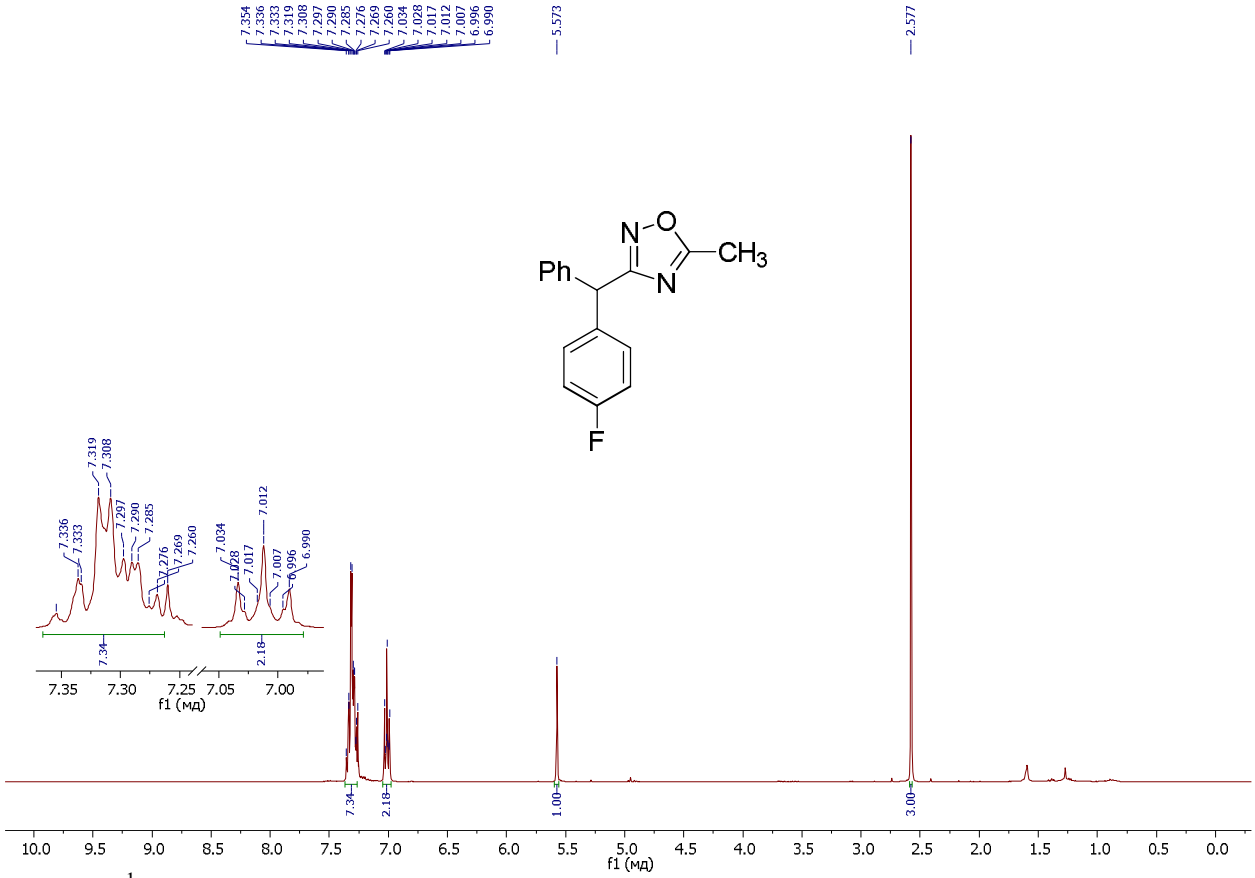

Fig. S67. ^{1}H NMR spectrum of the compound **2w** ($CDCl_3$, 400 MHz).

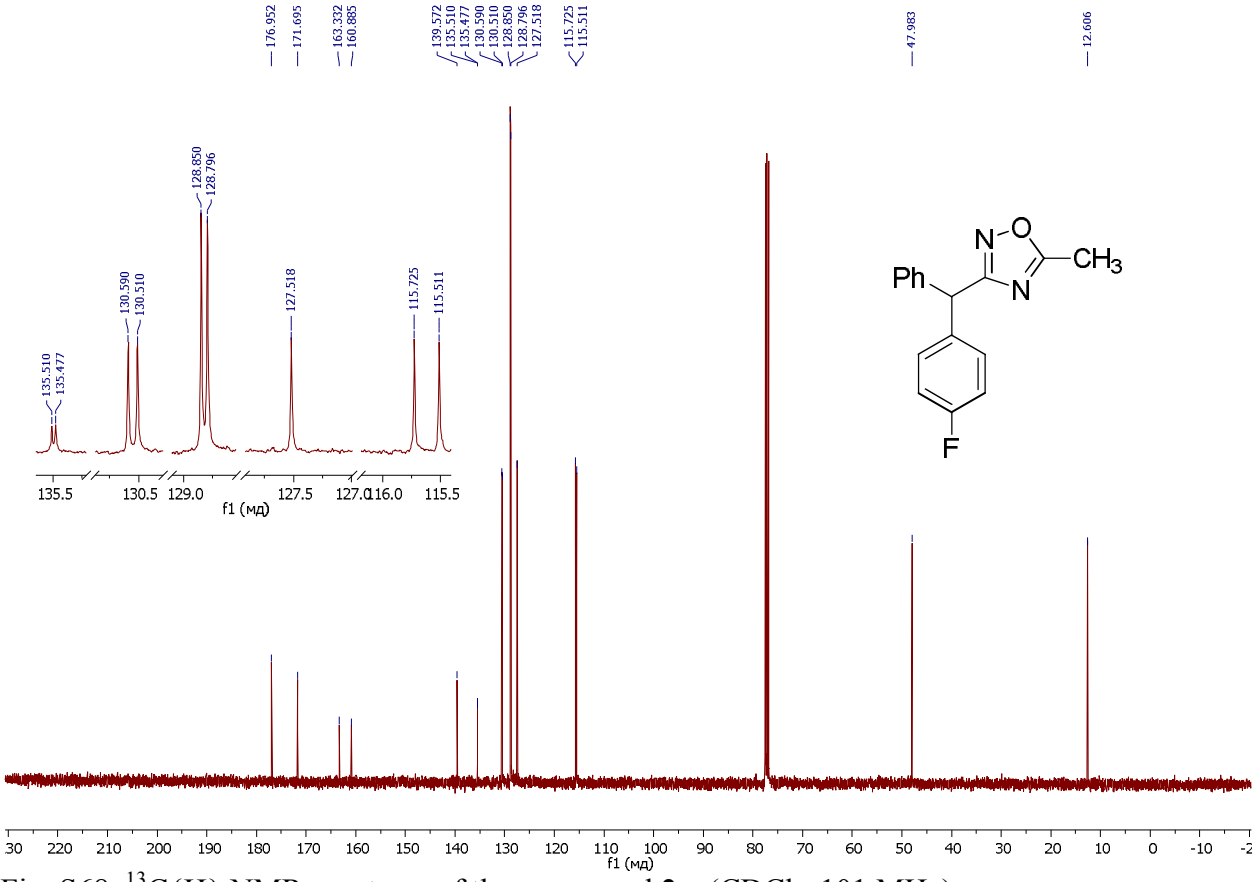

Fig. S68. ^{13}C{H} NMR spectrum of the compound **2w** ($CDCl_3$, 101 MHz)

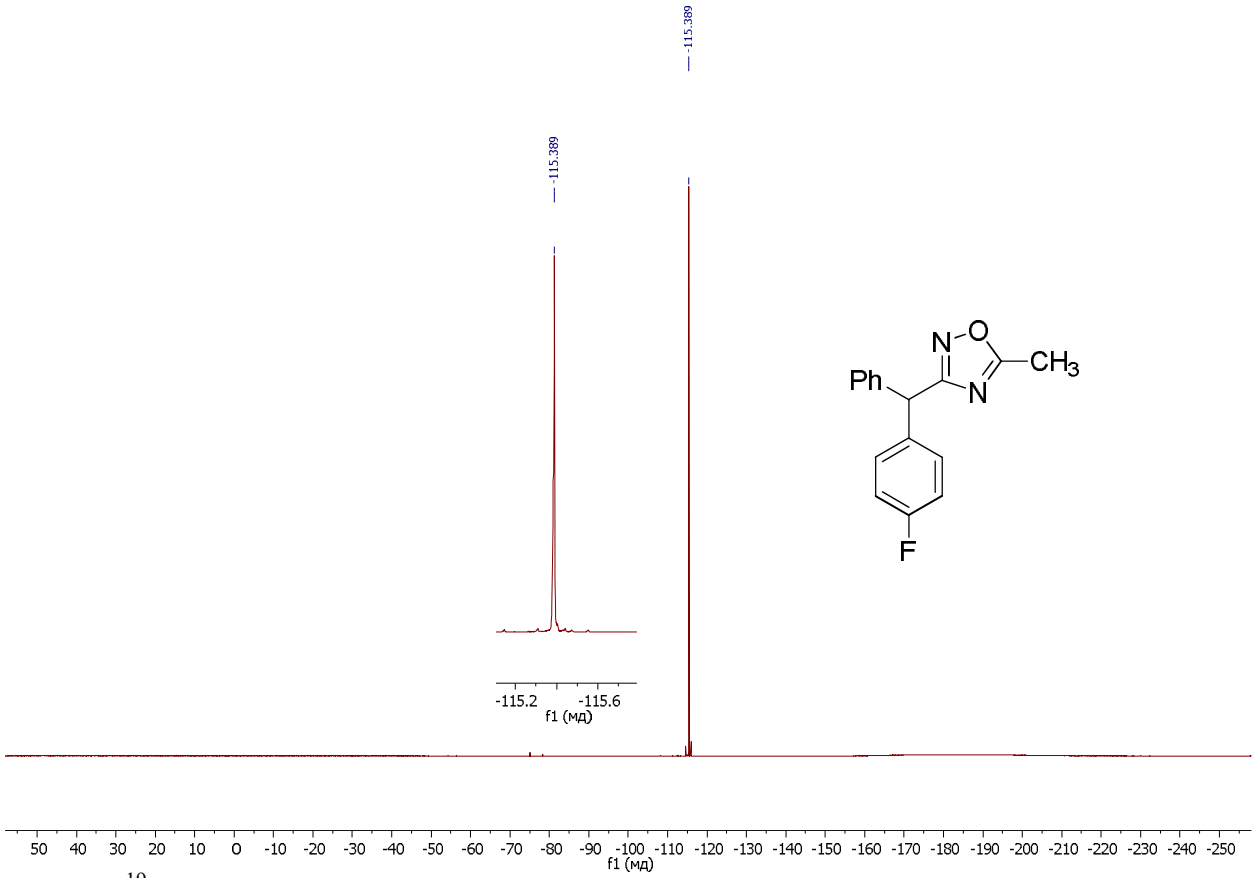

Fig. S69. $^{19}F\{H\}$ NMR spectrum of the compound **2w** ($CDCl_3$, 376 MHz).

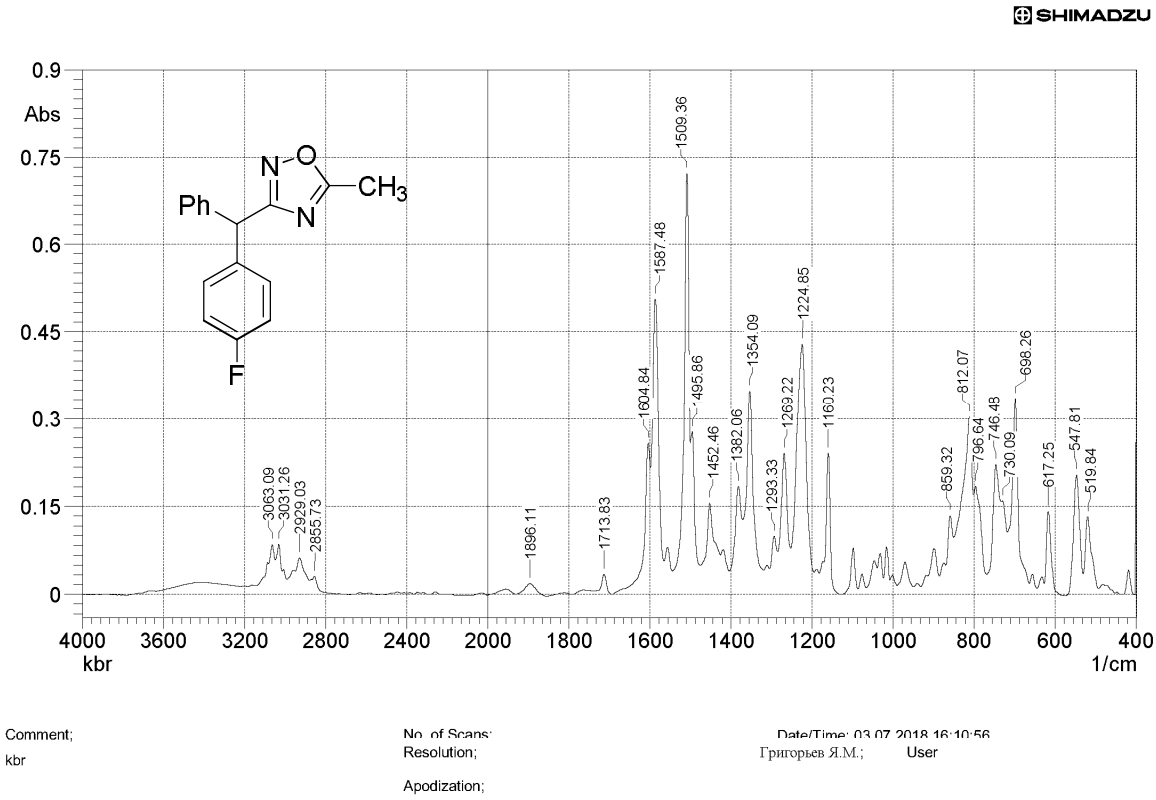

Fig. S70. IR spectrum of the compound **2w** (KBr).

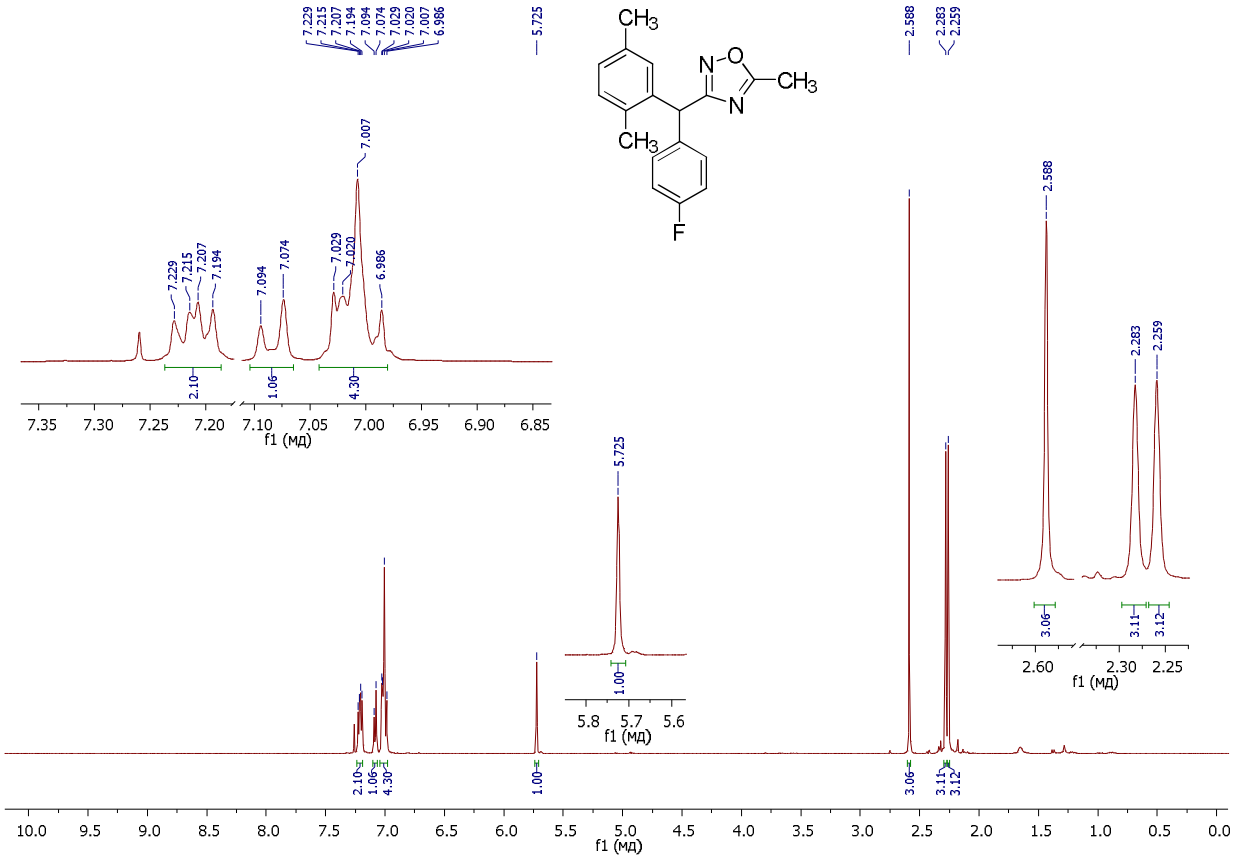

Fig. S71. ^{1}H NMR spectrum of the compound **2x** ($CDCl_3$, 400 MHz).

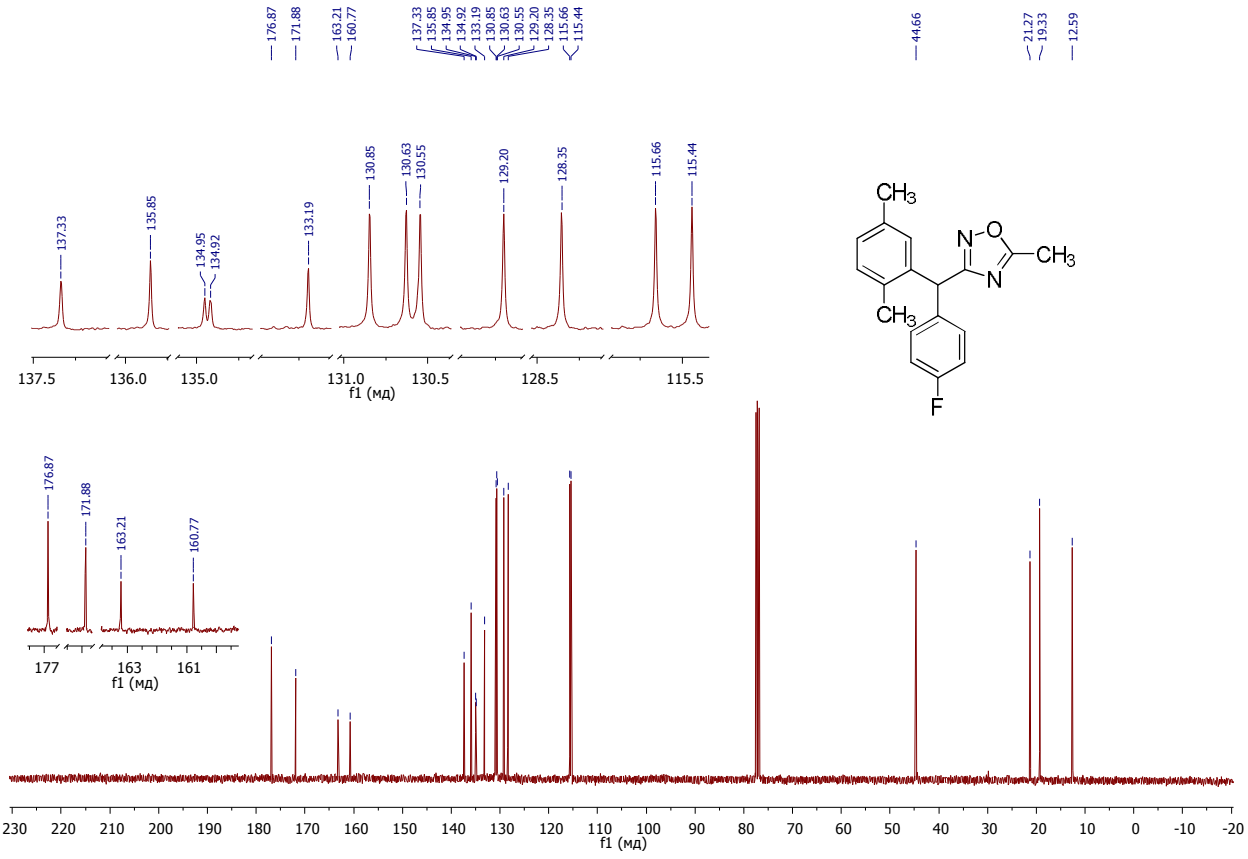

Fig. S72. ^{13}C{H} NMR spectrum of the compound **2x** ($CDCl_3$, 101 MHz).

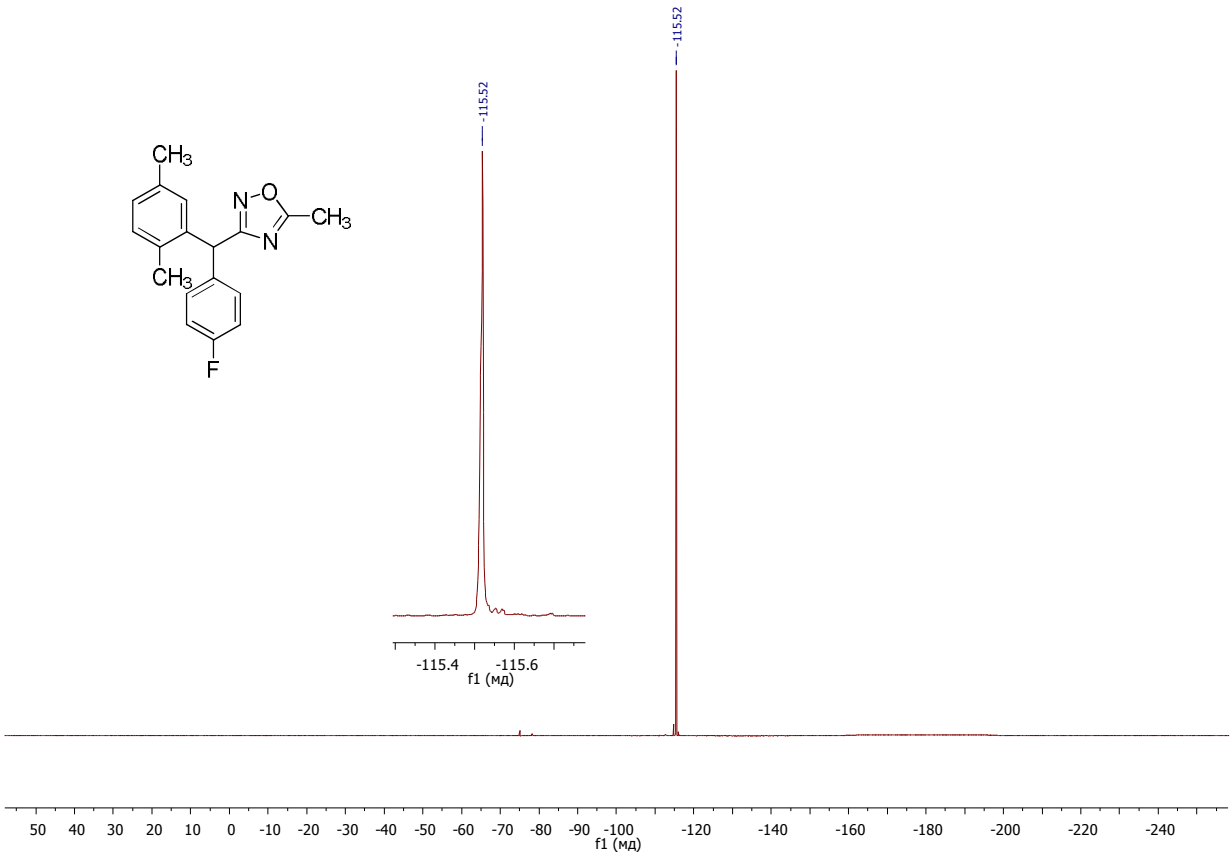

Fig. S73. $^{19}F\{H\}$ NMR spectrum of the compound **2x** ($CDCl_3$, 376 MHz).

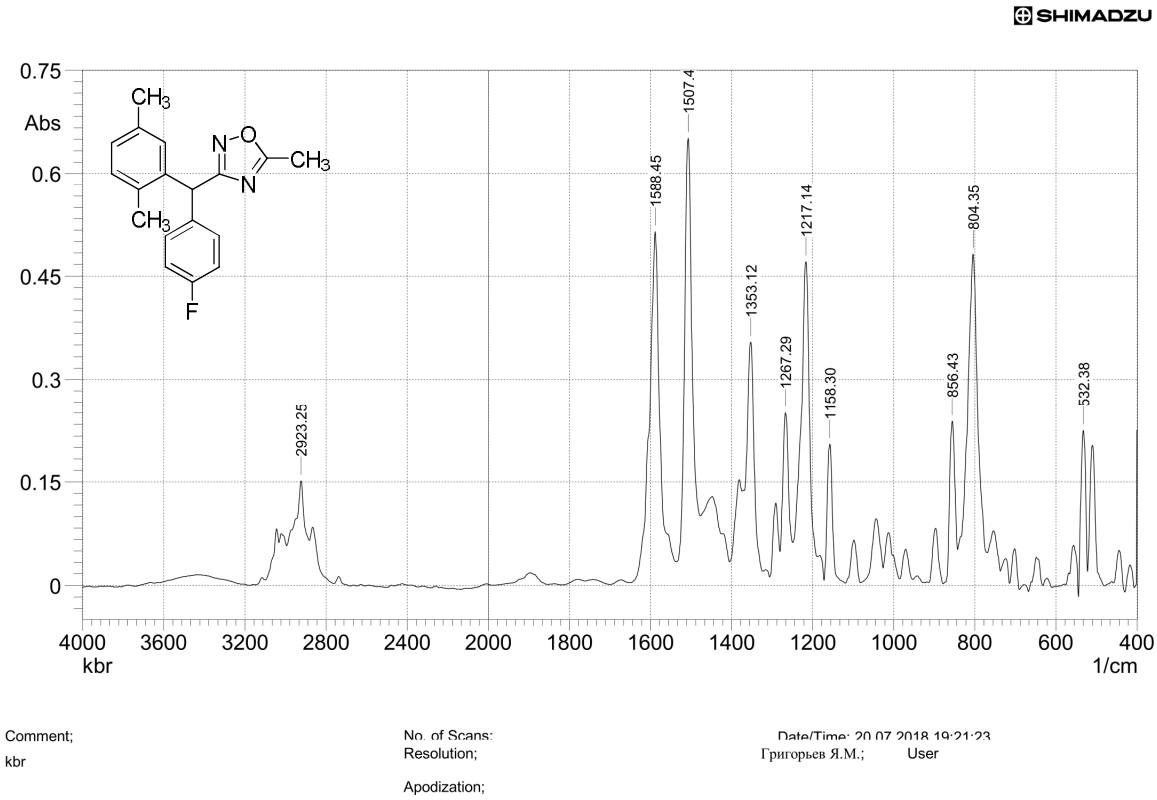

Fig. S74. IR spectrum of the compound **2x** (KBr).

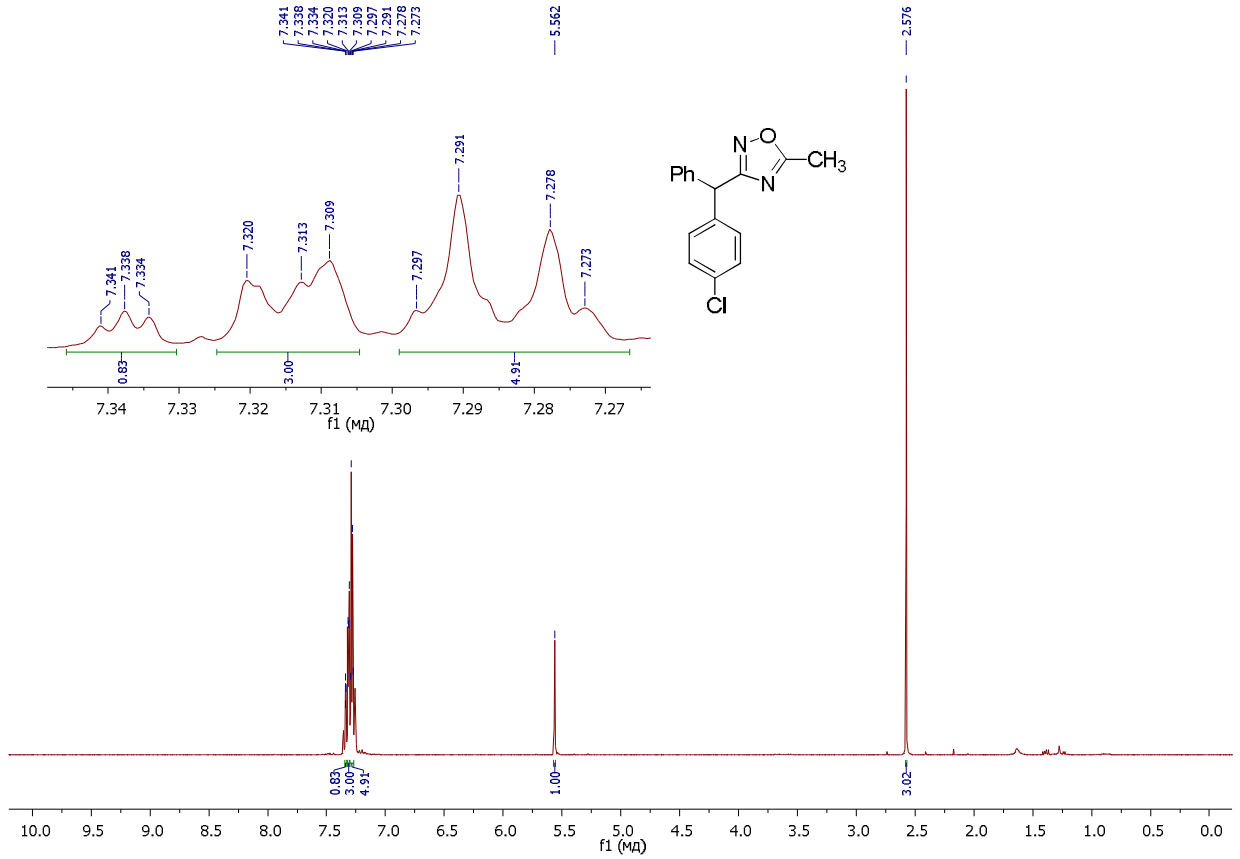

Fig. S75. ^{1}H NMR spectrum of the compound **2y** ($CDCl_3$, 400 MHz).

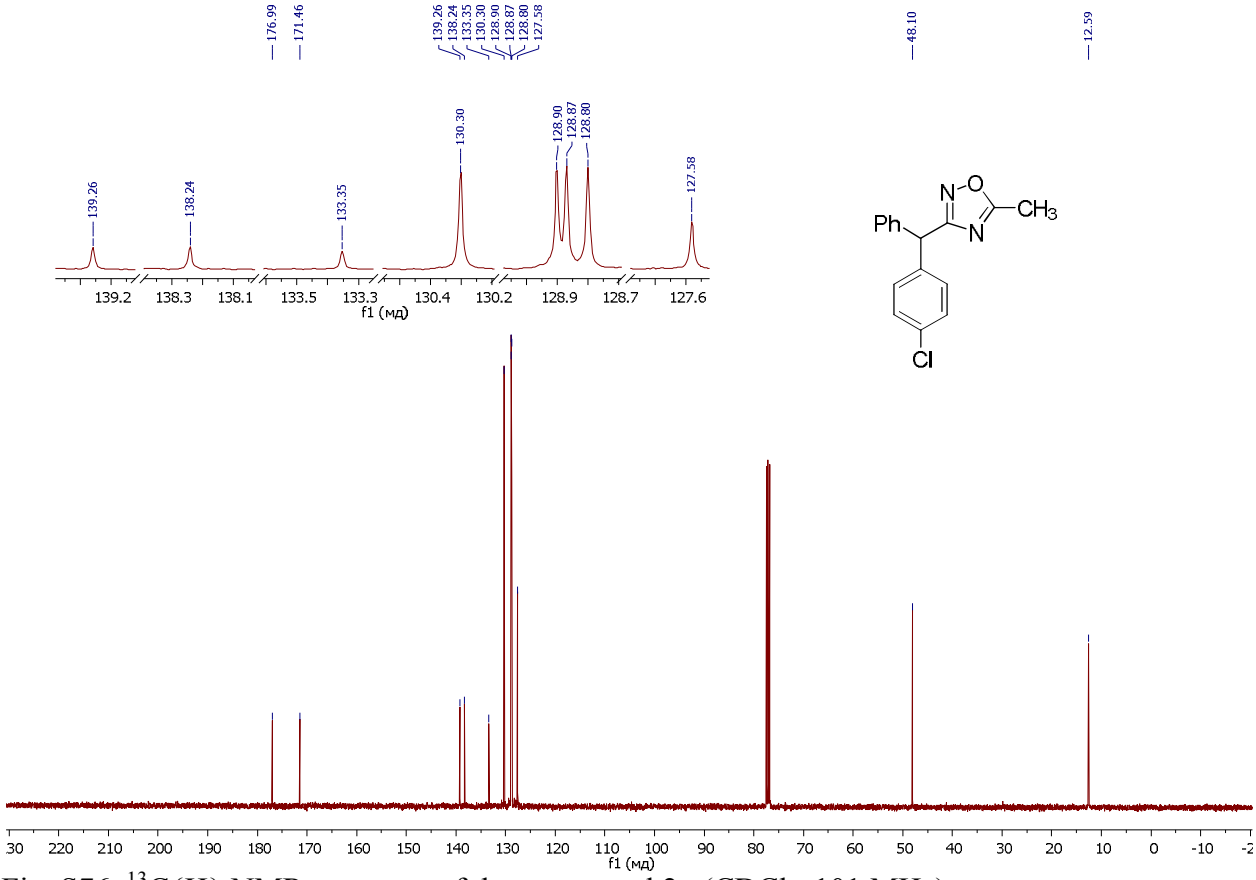

Fig. S76. ^{13}C{H} NMR spectrum of the compound **2y** ($CDCl_3$, 101 MHz).

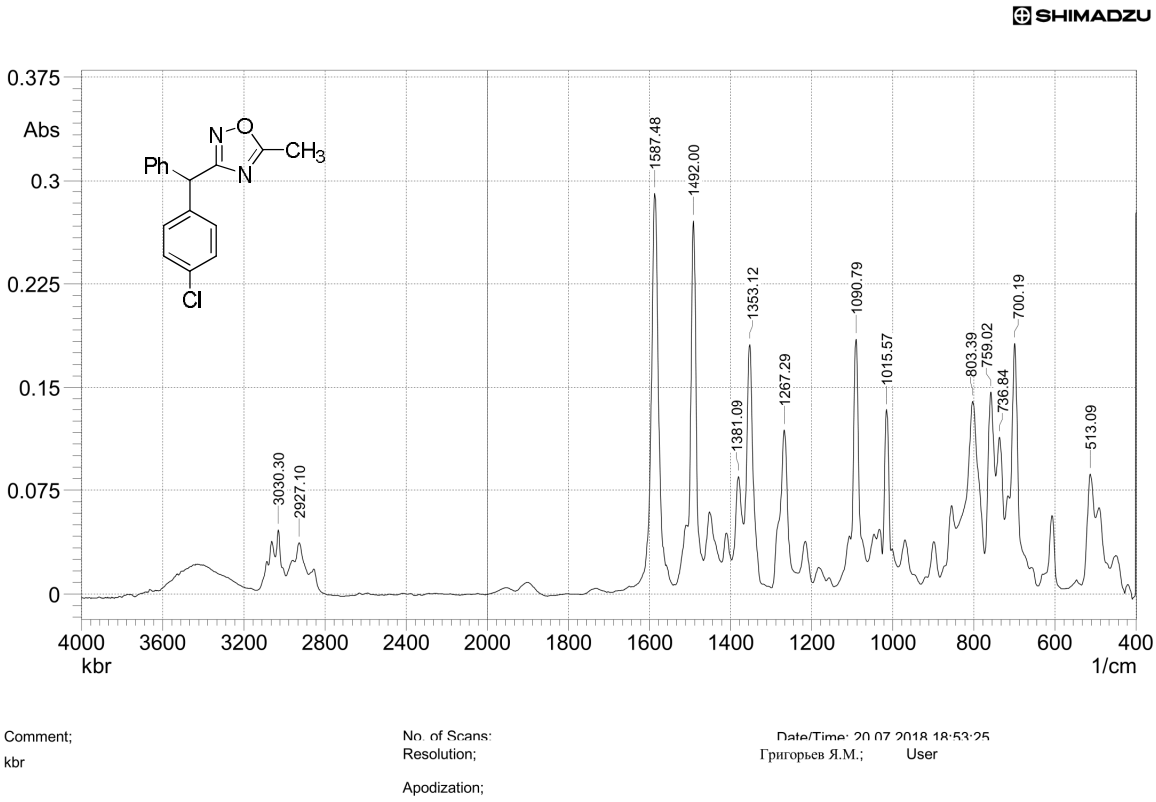

Fig. S77. IR spectrum of the compound **2y** (KBr).

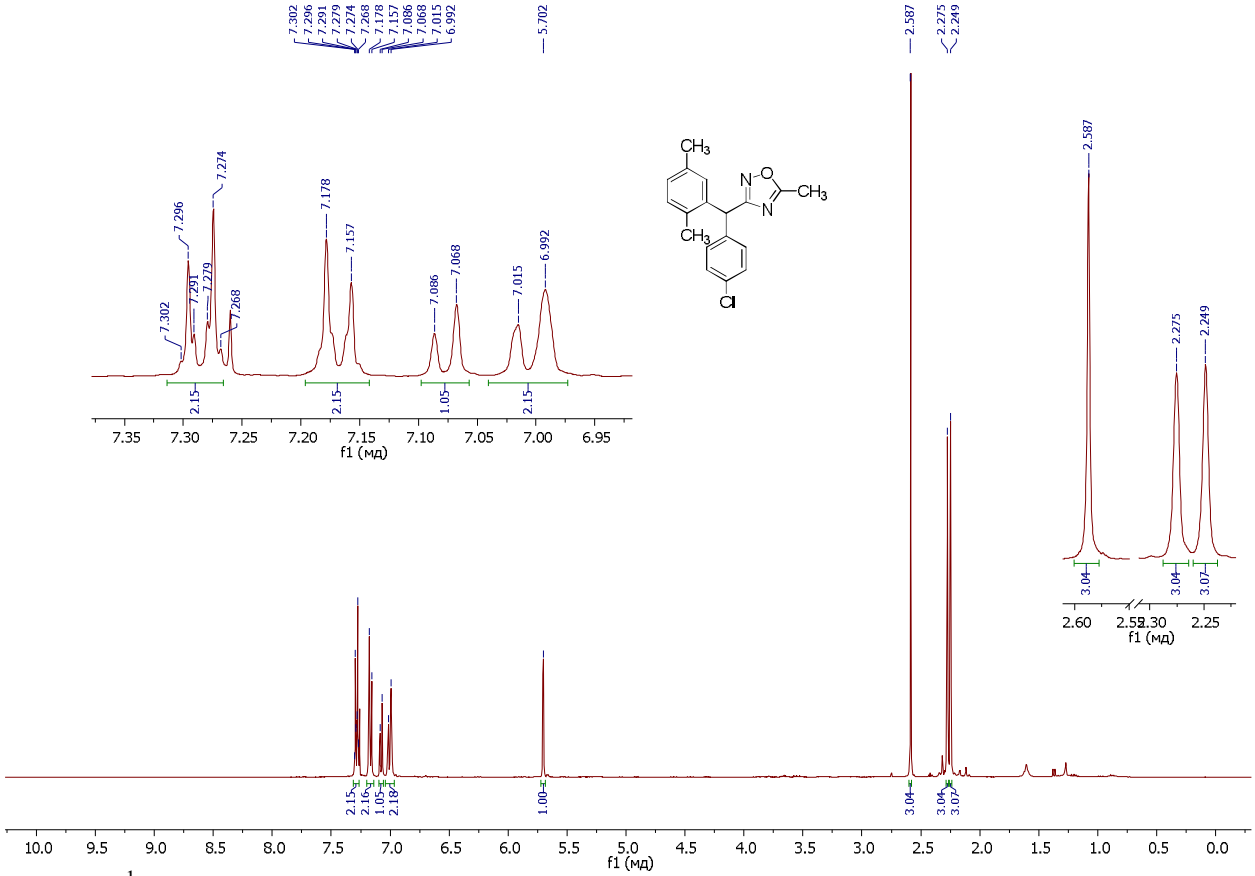

Fig. S78. ^{1}H NMR spectrum of the compound **2z** ($CDCl_3$, 400 MHz).

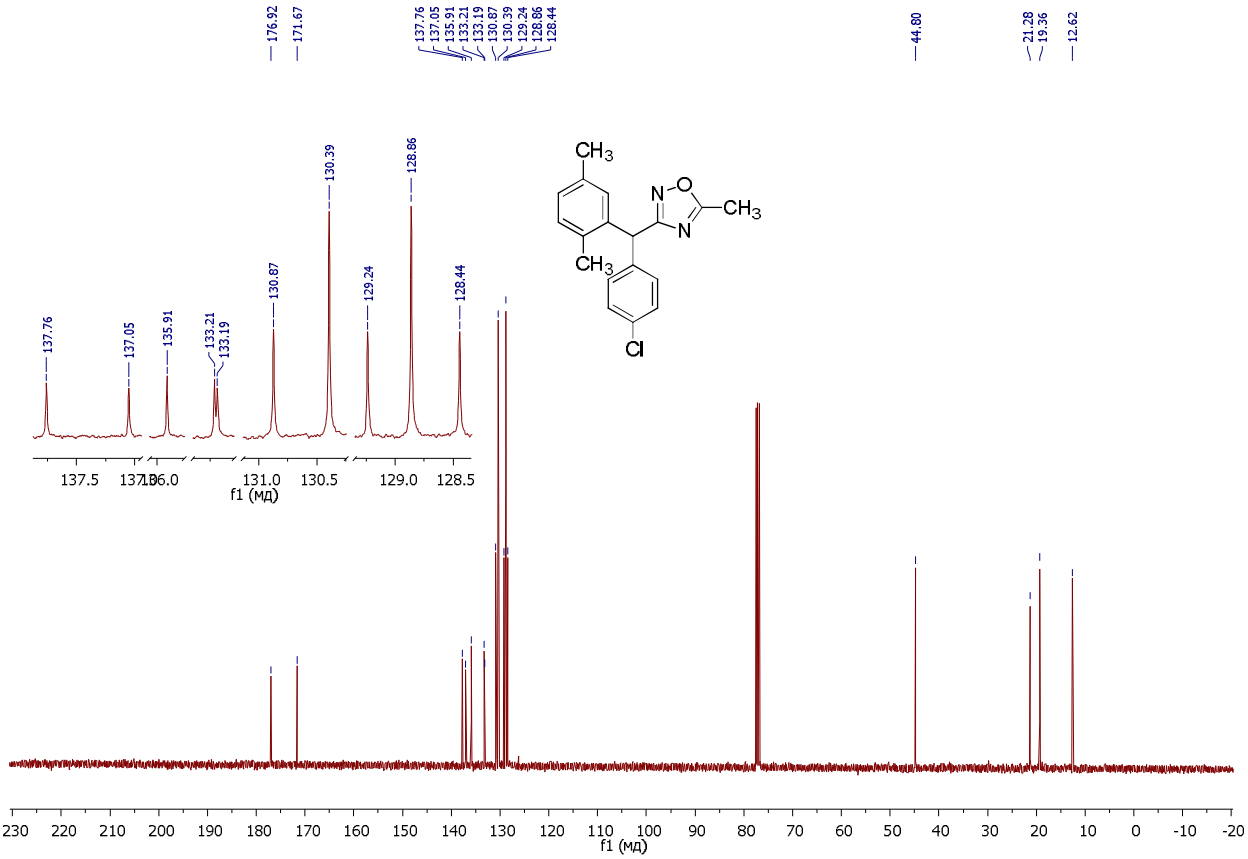

Fig. S79. $^{13}C\{H\}$ NMR spectrum of the compound **2z** ($CDCl_3$, 101 MHz).

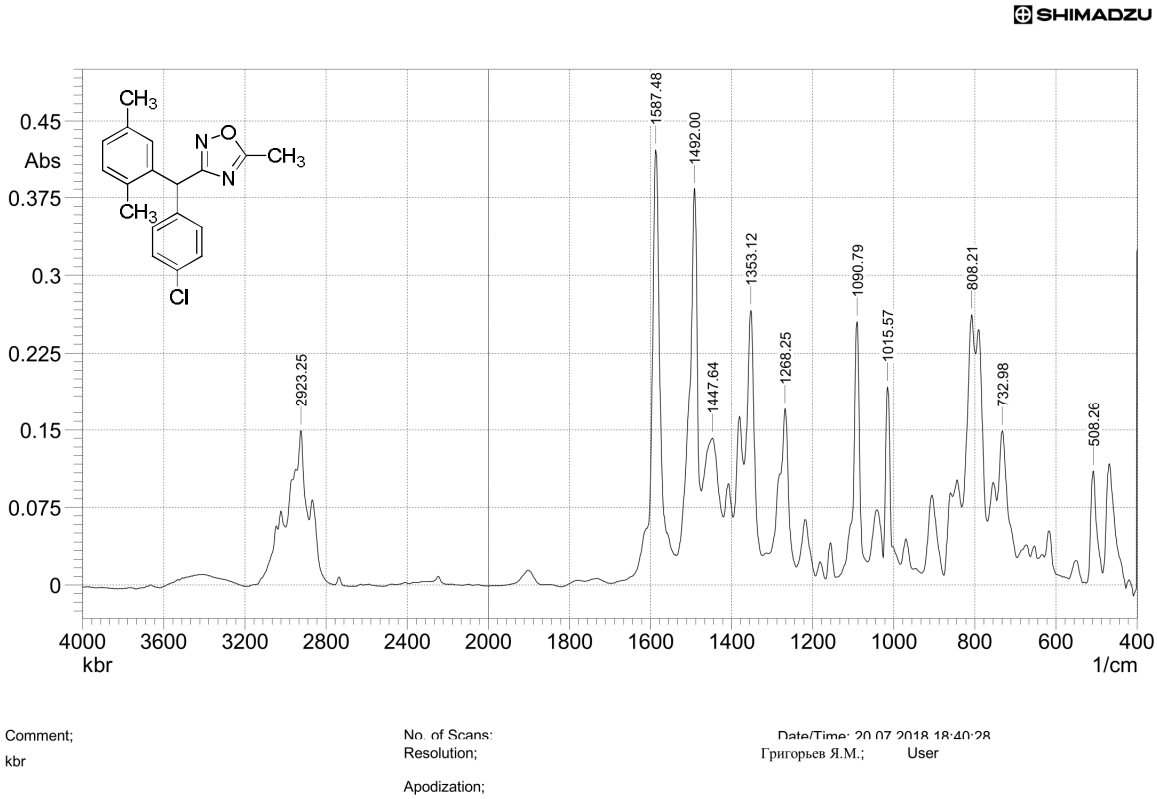

Fig. S80. IR spectrum of the compound **2z** (KBr).

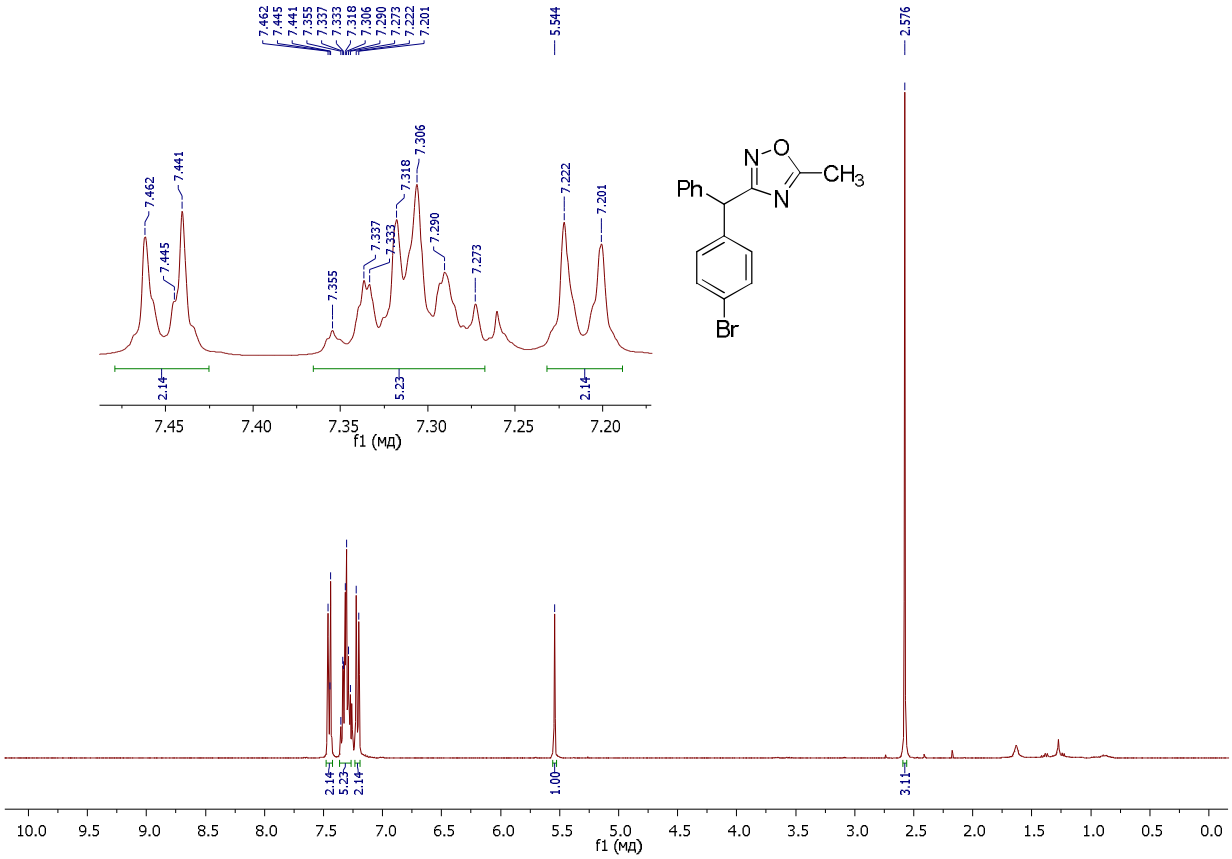

Fig. S81. ^{1}H NMR spectrum of the compound **2za** ($CDCl_3$, 400 MHz).

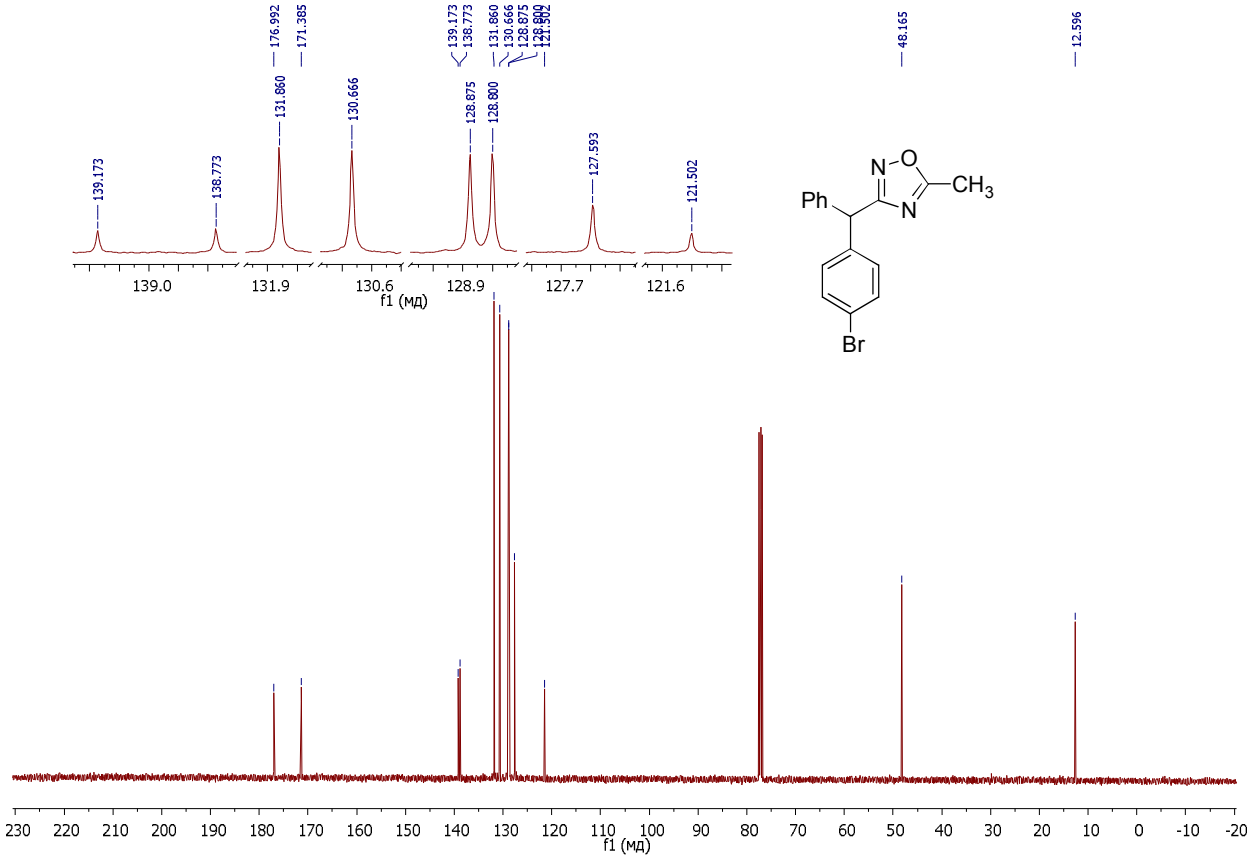

Fig. S82. ^{13}C{H} NMR spectrum of the compound **2za** ($CDCl_3$, 101 MHz).

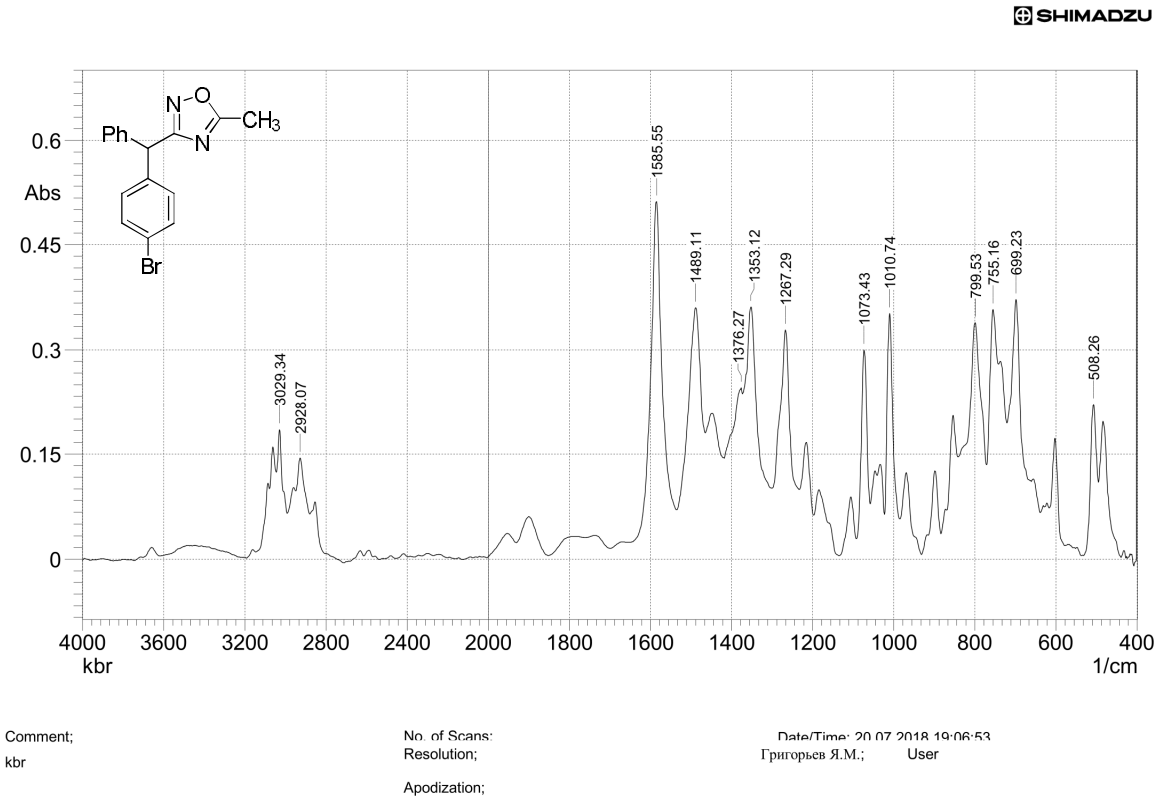

Fig. S83. IR spectrum of the compound **2za** (KBr).

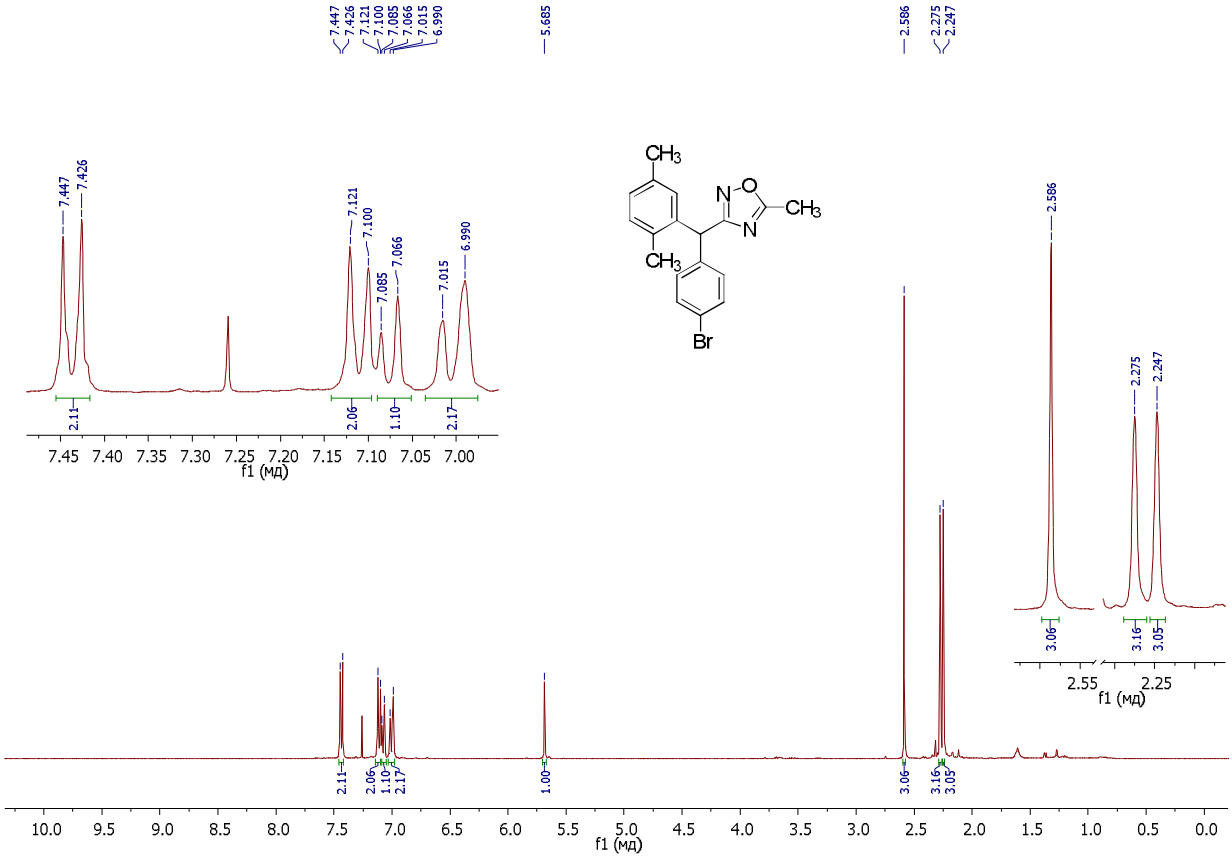

Fig. S84. ^{1}H NMR spectrum of the compound **2zb** ($CDCl_3$, 400 MHz).

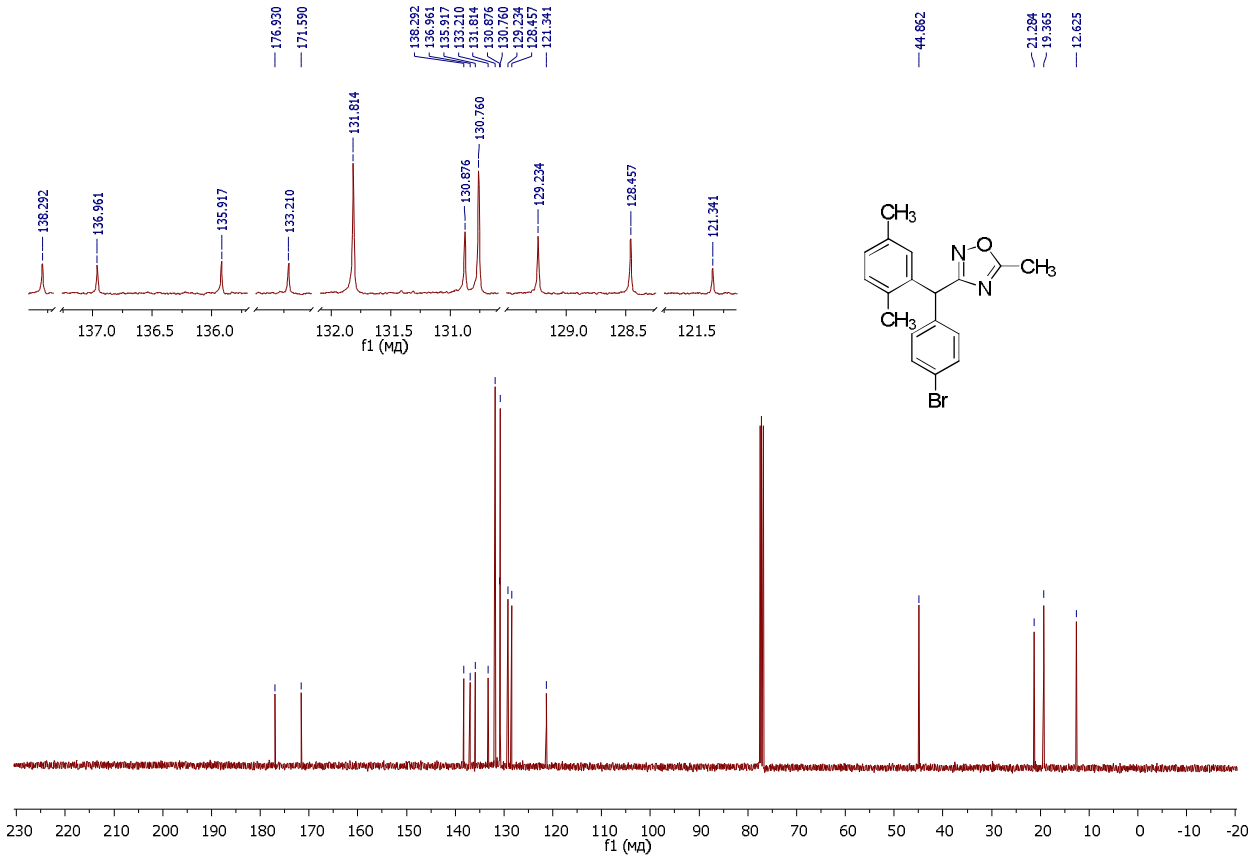

Fig. S85. $^{13}C\{H\}$ NMR spectrum of the compound **2zb** ($CDCl_3$, 101 MHz).

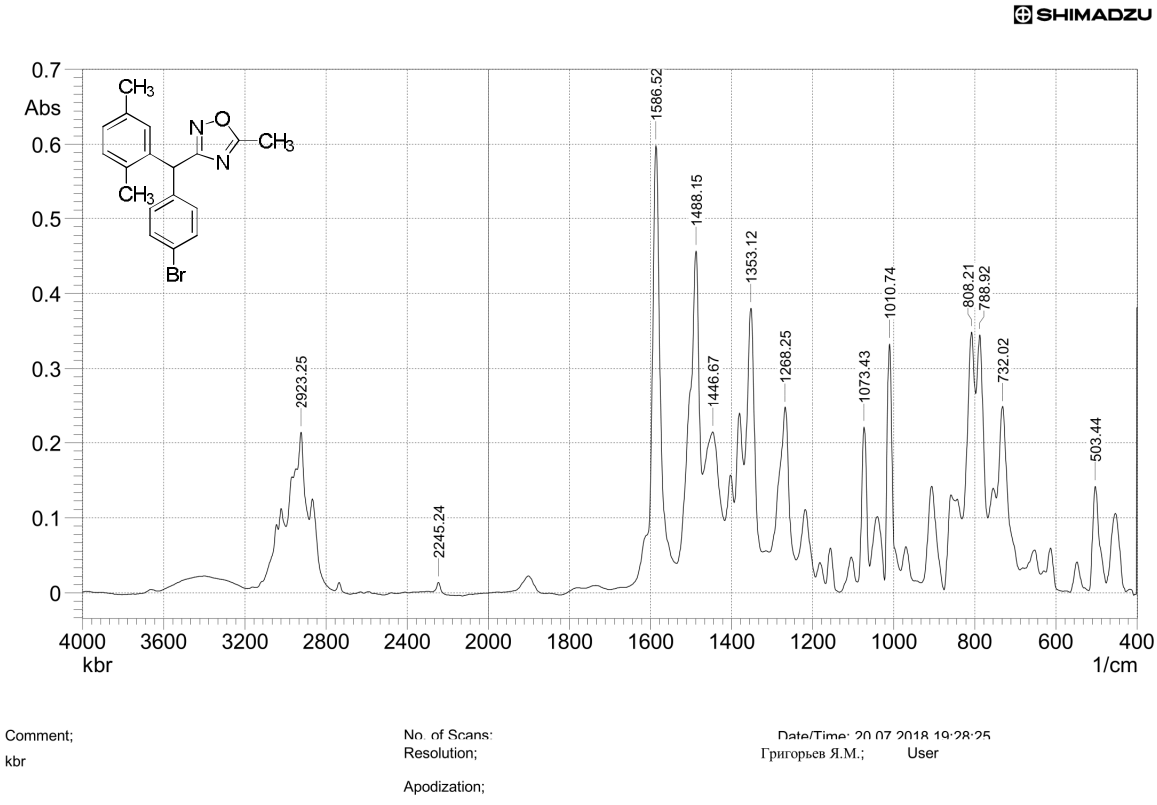

Fig. S86. IR spectrum of the compound **2zb** (KBr).

2. X-ray data for compounds 2i,n,o,x

2i

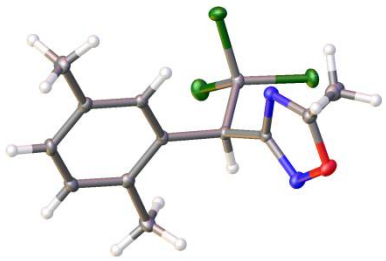

Fig. S86. Molecular structure of **2i**, CCDC 1888351 (ellipsoid contour of probability levels is 50 %).

Table S1 Crystal data and structure refinement for 2i.

Identificationcode	2i
Empiricalformula	$C_{13}H_{13}Cl_3N_2O$
Formulaweight	319.60
Temperature/K	100(2)
Crystalsystem	monoclinic
Spacegroup	$P2_1/n$
a/Å	11.1472(3)
b/Å	8.8578(2)
c/Å	14.7757(4)
α/°	90
β/°	101.894(3)
γ/°	90
Volume/Å^3	1427.63(7)
Z	4
ρ_{calc}g/cm^3	1.487
μ/mm^{-1}	0.634
F(000)	656.0
Crystalsize/mm^3	0.52 × 0.5 × 0.42
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.12 to 61.632
Indexranges	$-8 \le h \le 15$, $-11 \le k \le 12$, $-21 \le l \le 18$
Reflectionscollected	7311
Independentreflections	3993 [R_{int} = 0.0238, R_{sigma} = 0.0397]
Data/restraints/parameters	3993/0/175
Goodness-of-fit on F^2	1.052
Final R indexes [I>=2σ (I)]	R_1 = 0.0325, wR_2 = 0.0686
Final R indexes [all data]	R_1 = 0.0426, wR_2 = 0.0733
Largest diff. peak/hole / e Å^{-3}	0.39/-0.30

Experimental

Single crystals of $C_{13}H_{13}Cl_3N_2O$ **2i** were obtained at slow evaporation of solution of **2i** in diethyl ether. A suitable crystal was selected and studied on a Xcalibur, Eos diffractometer. The crystal was kept at 100(2) K during data collection. Using Olex2 [1], the structure was solved with the Superflip [2] structure solution program using Charge Flipping and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. &Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Palatinus, L. &Chapuis, G. (2007). J. Appl. Cryst., 40, 786-790; Palatinus, L. & van der Lee, A. (2008). J. Appl. Cryst. 41, 975-984; Palatinus, L., Prathapa, S. J. & van Smaalen, S. (2012). J. Appl. Cryst. 45, 575-580.
3. Sheldrick, G.M. (2015). ActaCryst. C71, 3-8.

Crystal structure determination of 2i

Crystal Data for $C_{13}H_{13}Cl_3N_2O$ (M =319.60 g/mol): monoclinic, space group $P2_1/n$ (no. 14), a = 11.1472(3) Å, b = 8.8578(2) Å, c = 14.7757(4) Å, β = 101.894(3)°, V = 1427.63(7) Å^3, Z = 4, T = 100(2) K, μ(MoKα) = 0.634 mm^{-1}, *Dcalc* = 1.487 g/cm^3, 7311 reflections measured (5.12° ≤ 2Θ ≤ 61.632°), 3993 unique (R_{int} = 0.0238, R_{sigma} = 0.0397) which were used in all calculations. The final R_1 was 0.0325 (I >2σ(I)) and wR_2 was 0.0733 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Ternary CH refined with riding coordinates:
 C4(H4)
2.b Aromatic/amide H refined with riding coordinates:
 C8(H8), C11(H11), C9(H9)
2.c Idealised Me refined as rotating group:
 C3(H3A,H3B,H3C), C13(H13A,H13B,H13C), C12(H12A,H12B,H12C)
```

2n

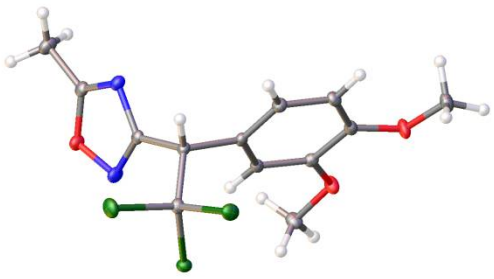

Fig. S87. Molecular structure of **2n**, CCDC 1894770 (ellipsoid contour of probability levels is 50 %).

Table S2 Crystal data and structure refinement for 2n.

Identificationcode	2n
Empiricalformula	$C_{13}H_{13}Cl_3N_2O_3$
Formulaweight	351.60
Temperature/K	100.01(10)
Crystalsystem	triclinic
Spacegroup	P-1
a/Å	8.5663(4)
b/Å	9.2482(4)

c/Å	10.8111(5)
α/°	105.441(4)
β/°	113.046(4)
γ/°	97.005(4)
Volume/Å^3	734.58(6)
Z	2
ρ_{calc}g/cm^3	1.590
μ/mm^{-1}	0.634
F(000)	360.0
Crystalsize/mm^3	0.5 × 0.34 × 0.26
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.214 to 55
Indexranges	-11 ≤ h ≤ 11, -12 ≤ k ≤ 12, -14 ≤ l ≤ 14
Reflectionscollected	12653
Independentreflections	3353 [R_{int} = 0.0179, R_{sigma} = 0.0161]
Data/restraints/parameters	3353/0/193
Goodness-of-fit on F^2	1.070
Final R indexes [I>=2σ (I)]	R_1 = 0.0232, wR_2 = 0.0540
Final R indexes [all data]	R_1 = 0.0255, wR_2 = 0.0553
Largest diff. peak/hole / e Å^{-3}	0.41/-0.20

Experimental

Single crystals of $C_{13}H_{13}Cl_3N_2O_3$ **2n** were obtained at slow evaporation of solution of **2n** in diethyl ether. A suitable crystal was selected and **studied** on a **Xcalibur, Eos** diffractometer. The crystal was kept at 100.01(10) K during data collection. Using Olex2 [1], the structure was solved with the Superflip [2] structure solution program using Charge Flipping and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. &Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Palatinus, L. &Chapuis, G. (2007). J. Appl. Cryst., 40, 786-790; Palatinus, L. & van der Lee, A. (2008). J. Appl. Cryst. 41, 975-984; Palatinus, L., Prathapa, S. J. & van Smaalen, S. (2012). J. Appl. Cryst. 45, 575-580.
3. Sheldrick, G.M. (2015). ActaCryst. C71, 3-8.

Crystal structure determination of 2n

Crystal Data for $C_{13}H_{13}Cl_3N_2O_3$ (M =351.60 g/mol): triclinic, space group P-1 (no. 2), a = 8.5663(4) Å, b = 9.2482(4) Å, c = 10.8111(5) Å, α = 105.441(4)°, β = 113.046(4)°, γ = 97.005(4)°, V = 734.58(6) Å^3, Z = 2, T = 100.01(10) K, μ(MoKα) = 0.634 mm^{-1},*Dcalc* = 1.590 g/cm^3, 12653 reflections measured (5.214° ≤ 2Θ ≤ 55°), 3353 unique (R_{int} = 0.0179, R_{sigma} = 0.0161) which were used in all calculations. The final R_1 was 0.0232 (I >2σ(I)) and wR_2 was 0.0553 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Ternary CH refined with riding coordinates:
 C10(H10)
2.b Aromatic/amide H refined with riding coordinates:
 C4(H4), C7(H7), C8(H8)

2.c Idealised Me refined as rotating group:
C11(H11A,H11B,H11C), C12(H12A,H12B,H12C), C13(H13A,H13B,H13C)

2o

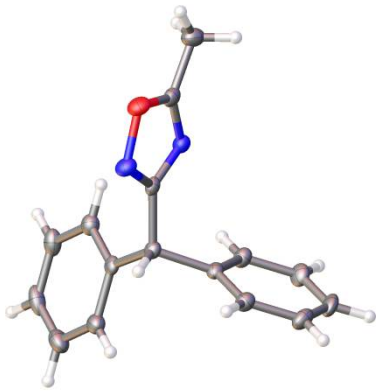

Fig. S88. Molecular structure of **2o**, CCDC 1894769 (ellipsoid contour of probability levels is 50 %).

Table S3 Crystal data and structure refinement for 2o.

Identificationcode	2o
Empiricalformula	$C_{16}H_{14}N_2O$
Formulaweight	250.29
Temperature/K	100(2)
Crystalsystem	monoclinic
Spacegroup	$P2_1/c$
a/Å	12.8072(7)
b/Å	6.0525(4)
c/Å	16.8346(9)
α/°	90
β/°	91.053(5)
γ/°	90
Volume/Å^3	1304.72(13)
Z	4
ρ_{calc}g/cm^3	1.274
μ/mm^{-1}	0.643
F(000)	528.0
Crystalsize/mm^3	0.59 × 0.5 × 0.49
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	6.904 to 152.282
Indexranges	$-15 \leq h \leq 15$, $-7 \leq k \leq 6$, $-20 \leq l \leq 19$
Reflectionscollected	4425
Independentreflections	2516 [R_{int} = 0.0230, R_{sigma} = 0.0242]
Data/restraints/parameters	2516/0/173
Goodness-of-fit on F^2	1.032
Final R indexes [I>=2σ (I)]	R_1 = 0.0428, wR_2 = 0.1110
Final R indexes [all data]	R_1 = 0.0466, wR_2 = 0.1147
Largest diff. peak/hole / e Å^{-3}	0.23/-0.25

Experimental

Single crystals of $C_{16}H_{14}N_2O$ **2o** were obtained at slow evaporation of solution of **2o** in diethyl ether. A suitable crystal was selected and studied on a Xcalibur, Eos diffractometer.The crystal was kept at 100(2) K during data collection. Using Olex2 [1], the structure was solved with the Unknown [2] structure solution program using Unknown and refined with the Unknown [3] refinement package using Unknown minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. &Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Palatinus, L. &Chapuis, G. (2007). J. Appl. Cryst., 40, 786-790; Palatinus, L. & van der Lee, A. (2008). J. Appl. Cryst. 41, 975-984; Palatinus, L., Prathapa, S. J. & van Smaalen, S. (2012). J. Appl. Cryst. 45, 575-580.
3. Sheldrick, G.M. (2015). ActaCryst. C71, 3-8.

Crystal structure determination of 2o

Crystal Data for $C_{16}H_{14}N_2O$ (M =250.29 g/mol): monoclinic, space group $P2_1/c$ (no. 14), a = 12.8072(7) Å, b = 6.0525(4) Å, c = 16.8346(9) Å, β = 91.053(5)°, V = 1304.72(13) Å^3, Z = 4, T = 100(2) K, μ(CuKα) = 0.643 mm^{-1}, *Dcalc* = 1.274 g/cm^3, 4425 reflections measured (6.904° ≤ 2Θ ≤ 152.282°), 2516 unique (R_{int} = 0.0230, R_{sigma} = 0.0242) which were used in all calculations. The final R_1 was 0.0428 (I >2σ(I)) and wR_2 was 0.1147 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Ternary CH refined with riding coordinates:
 C3(H3)
2.b Aromatic/amide H refined with riding coordinates:
 C6(H6), C16(H16), C10(H10), C12(H12), C7(H7), C8(H8), C15(H15), C9(H9),
 C13(H13), C14(H14)
2.c Idealised Me refined as rotating group:
 C4(H4A,H4B,H4C)

A suitable crystal was selected and studied on a Xcalibur, Eos diffractometer.

2x

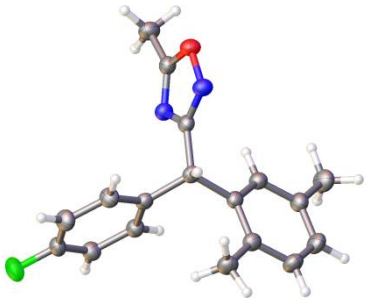

Fig. S90. Molecular structure of **2x**, CCDC 1894768 (ellipsoid contour of probability levels is 50 %).

Table S4 Crystal data and structure refinement for 2x.

Identificationcode	2x
Empiricalformula	$C_{18}H_{17}FN_2O$

Formulaweight	296.33
Temperature/K	114(2)
Crystalsystem	triclinic
Spacegroup	P-1
a/Å	7.5047(3)
b/Å	9.2211(4)
c/Å	11.4499(6)
α/°	90.884(4)
β/°	103.956(4)
γ/°	95.264(4)
Volume/Å^3	765.14(6)
Z	2
ρ_{calc}g/cm^3	1.286
μ/mm^{-1}	0.726
F(000)	312.0
Crystalsize/mm^3	0.17 × 0.12 × 0.1
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.962 to 145.04
Indexranges	-9 ≤ h ≤ 9, -10 ≤ k ≤ 11, -14 ≤ l ≤ 14
Reflectionscollected	9305
Independentreflections	2990 [R_{int} = 0.0292, R_{sigma} = 0.0312]
Data/restraints/parameters	2990/0/202
Goodness-of-fit on F^2	1.071
Final R indexes [I>=2σ (I)]	R_1 = 0.0411, wR_2 = 0.1097
Final R indexes [all data]	R_1 = 0.0497, wR_2 = 0.1156
Largest diff. peak/hole / e Å^{-3}	0.23/-0.26

Experimental

Single crystals of $C_{18}H_{17}FN_2O$ **2x** were obtained at slow evaporation of solution of 2x in diethyl ether. A suitable crystal was selected and **studied** on a **SuperNova, Single source at offset/far, HyPix3000** diffractometer. The crystal was kept at 114(2) K during data collection. Using Olex2 [1], the structure was solved with the ShelXT [2] structure solution program using Intrinsic Phasing and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. &Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). ActaCryst. A71, 3-8.
3. Sheldrick, G.M. (2015). ActaCryst. C71, 3-8.

Crystal structure determination of 2x.

Crystal Data for $C_{18}H_{17}FN_2O$ (M =296.33 g/mol): triclinic, space group P-1 (no. 2), a = 7.5047(3) Å, b = 9.2211(4) Å, c = 11.4499(6) Å, α = 90.884(4)°, β = 103.956(4)°, γ = 95.264(4)°, V = 765.14(6) Å^3, Z = 2, T = 114(2) K, μ(CuKα) = 0.726 mm^{-1}, *Dcalc* = 1.286 g/cm^3, 9305 reflections measured (7.962° ≤ 2Θ ≤ 145.04°), 2990 unique (R_{int} = 0.0292, R_{sigma} = 0.0312) which were used in all calculations. The final R_1 was 0.0411 (I >2σ(I)) and wR_2 was 0.1156 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Ternary CH refined with riding coordinates:
 C4(H4)
2.b Aromatic/amide H refined with riding coordinates:
 C7(H7), C6(H6), C12(H12), C10(H10), C9(H9), C15(H15), C14(H14)
2.c Idealised Me refined as rotating group:
 C18(H18A,H18B,H18C), C3(H3A,H3B,H3C), C17(H17A,H17B,H17C)