Supporting information

Novel One-Pot Synthesis of Methyl 4-Hydroxy-2-thioxo-1,2-dihydroquinoline-3-carboxylate: Synthetic and Crystallographic Studies

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SUPPLEMENTARY MATERIAL

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Methyl 4-hydroxy-2-thioxo-1,2-dihydroquinoline-3-carboxylate **13**.

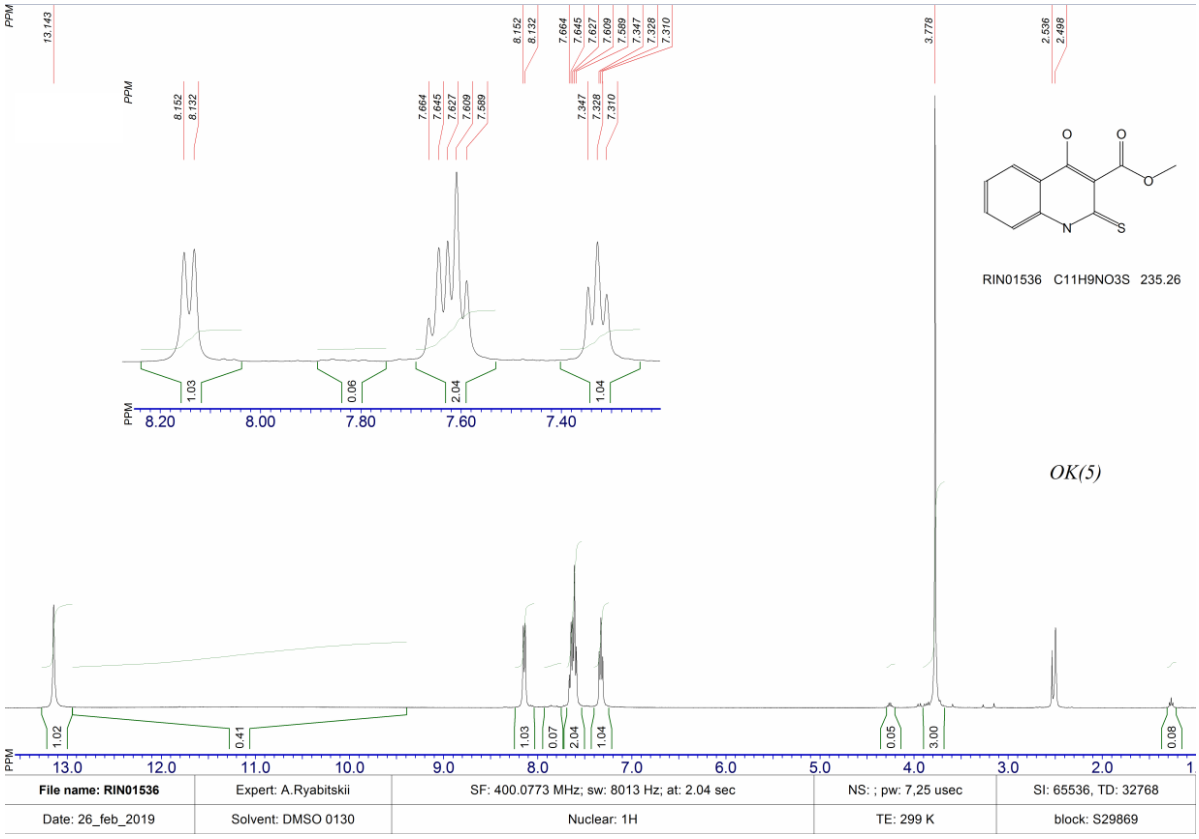


Figure S1. ^{1}H NMR spectrum (400 MHz, DMSO-d_6) of methyl 4-hydroxy-2-thioxo-1,2-dihydroquinoline-3-carboxylate **13**.

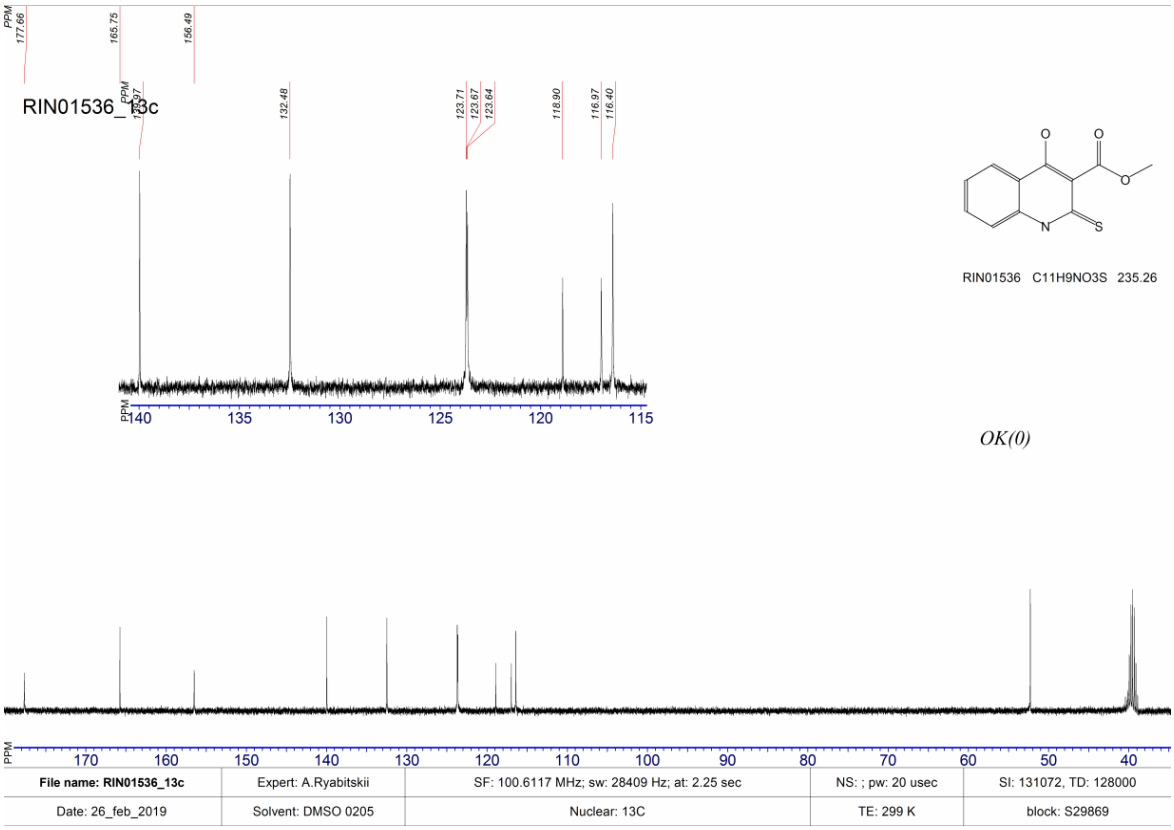
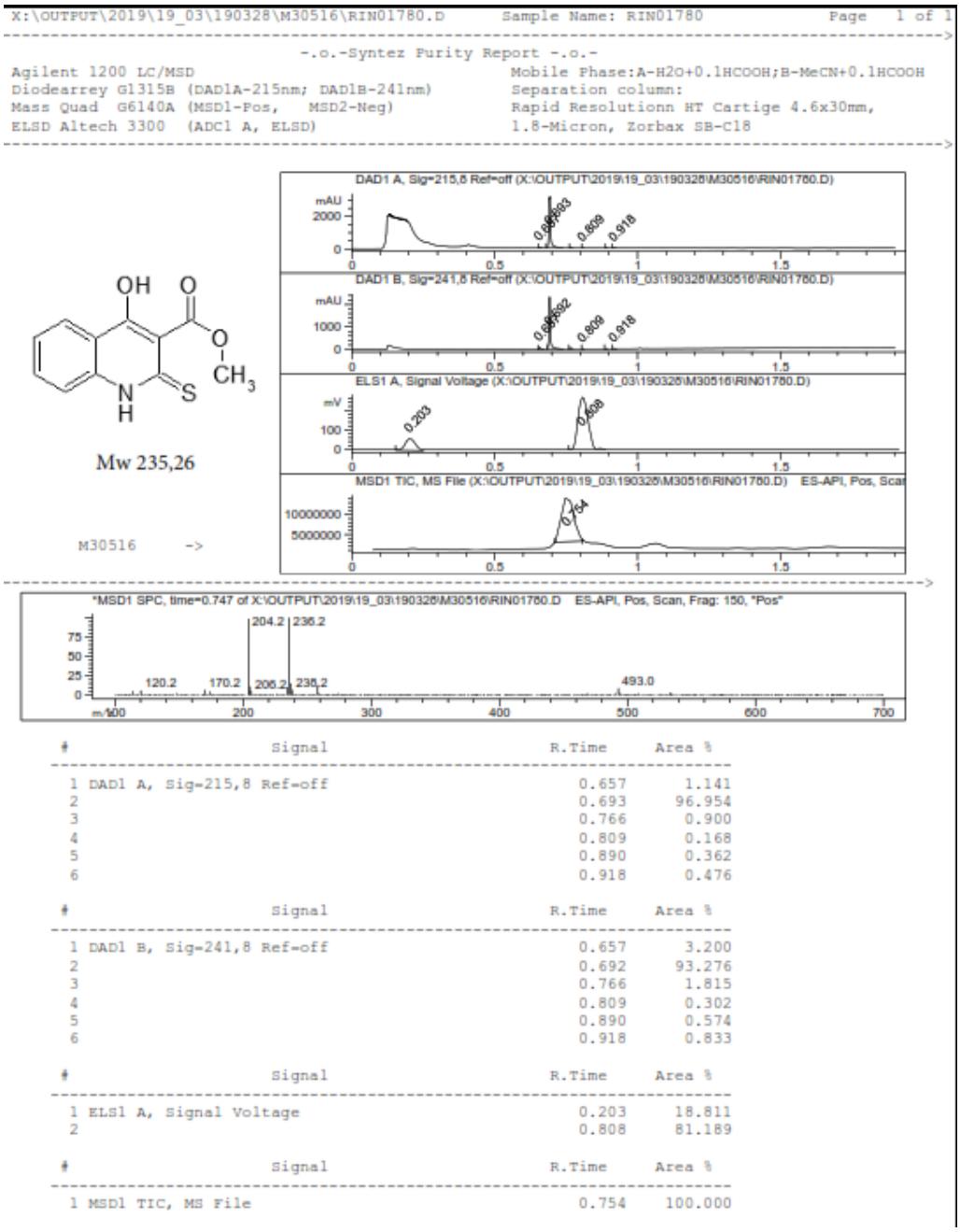


Figure S2. ^{13}C NMR spectrum (100 MHz, DMSO-d_6) of methyl 4-hydroxy-2-thioxo-1,2-dihydroquinoline-3-carboxylate **13**.



#	Signal	R.Time	Area %
1	DADl A, Sig=215,8 Ref=off	0.657	1.141
2		0.693	96.954
3		0.766	0.900
4		0.809	0.168
5		0.890	0.362
6		0.918	0.476

#	Signal	R.Time	Area %
1	DADl B, Sig=241,8 Ref=off	0.657	3.200
2		0.692	93.276
3		0.766	1.815
4		0.809	0.302
5		0.890	0.574
6		0.918	0.833

#	Signal	R.Time	Area %
1	ELSl A, Signal Voltage	0.203	18.811
2		0.808	81.189

#	Signal	R.Time	Area %
1	MSDl TIC, MS File	0.754	100.000

Figure S3. LC/MS data for methyl 4-hydroxy-2-thioxo-1,2-dihydroquinoline-3-carboxylate **13**.

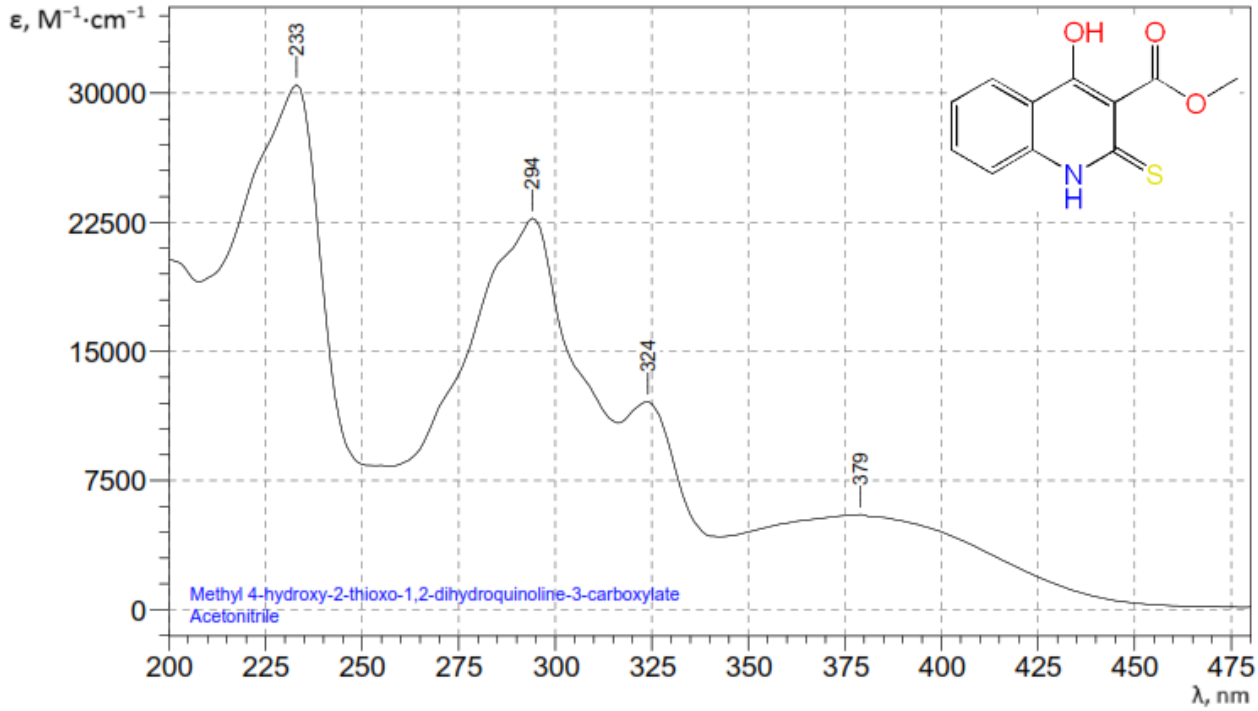


Figure S4. UV/Vis spectrum (CH_3CN) of methyl 4-hydroxy-2-thioxo-1,2-dihydroquinoline-3-carboxylate **13**.

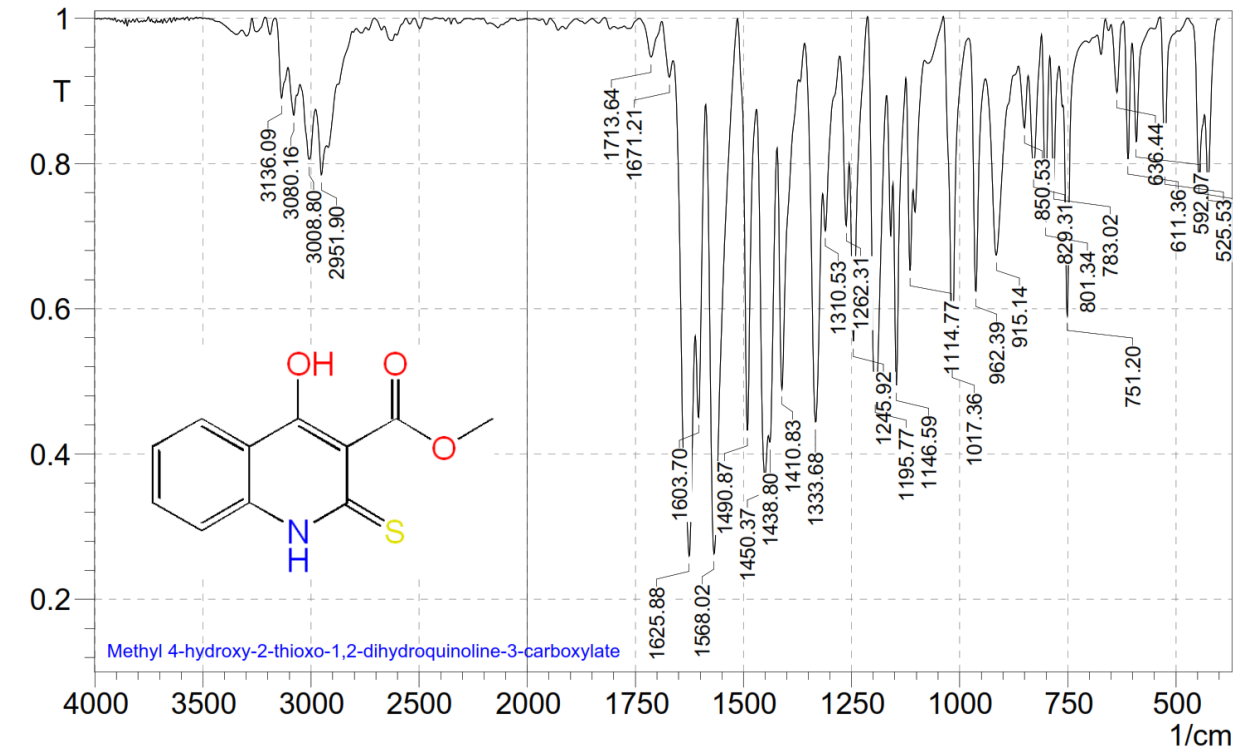


Figure S5. IR spectrum of methyl 4-hydroxy-2-thioxo-1,2-dihydroquinoline-3-carboxylate **13** (KBr pellet)

Table S1. Coordinates (×10^4) and equivalent isotropic thermal parameters (Å^2 × 10^3) of atoms in structure **13**.

Atom	x	y	z	U(eq)
S1	-1441(2)	3123(1)	5156(1)	63.8(3)
O1	6175(6)	3848(3)	8846(2)	73.0(8)
O2	2112(6)	1696(3)	7861(2)	76.3(8)
O3	-1331(6)	1331(2)	6345(2)	76.8(7)
N1	3001(6)	5222(3)	6505(3)	52.4(7)
C1	5447(7)	6066(3)	7383(3)	51.7(8)
C2	6887(8)	7368(4)	7478(3)	62.4(9)
C3	9267(8)	8184(4)	8367(3)	71.9(1)
C4	10336(8)	7743(4)	9211(3)	73.0(1)
C5	8983(8)	6473(4)	9125(3)	65.0(1)
C6	6502(7)	5602(3)	8207(3)	51.3(9)
C7	5000(7)	4266(4)	8063(3)	53.4(9)
C8	2484(7)	3450(3)	7179(3)	48.8(8)
C9	1422(7)	3938(3)	6321(3)	46.7(8)
C10	1092(8)	2094(4)	7140(3)	56.3(9)
C11	-2630(9)	-6(4)	6314(4)	89.5(1)

Table S2. Bond lengths (Å) in structure **13**.

S1-C9	1.671(3)
O1-C7	1.340(4)
O2-C10	1.233(4)
O3-C10	1.303(4)
O3-C11	1.449(4)
N1-C1	1.370(4)
N1-C9	1.364(4)
C1-C2	1.399(4)
C1-C6	1.398(4)
C2-C3	1.351(4)
C3-C4	1.403(5)
C4-C5	1.362(5)
C5-C6	1.408(4)
C6-C7	1.419(5)
C7-C8	1.389(4)
C8-C9	1.453(4)
C8-C10	1.474(4)

Table S3. Valence angles (deg.) in structure **13**.

C11-O3-C10	117.2(3)
C9-N1-C1	127.4(3)
C2-C1-N1	122.1(3)
C6-C1-N1	118.3(3)

C6-C1-C2	119.6(3)
C3-C2-C1	120.5(4)
C4-C3-C2	120.7(4)
C5-C4-C3	119.6(4)
C6-C5-C4	120.8(4)
C5-C6-C1	118.8(3)
C7-C6-C1	117.6(3)
C7-C6-C5	123.6(3)
C6-C7-O1	115.2(3)
C8-C7-O1	122.3(3)
C8-C7-C6	122.5(3)
C9-C8-C7	119.3(3)
C10-C8-C7	117.2(3)
C10-C8-C9	123.4(3)
N1-C9-S1	117.1(2)
C8-C9-S1	128.1(3)
C8-C9-N1	114.8(3)
O3-C10-O2	120.1(3)
C8-C10-O2	121.6(3)
C8-C10-O3	118.3(3)

Methyl 2-(methoxycarbonothioylamino)benzoate **14**

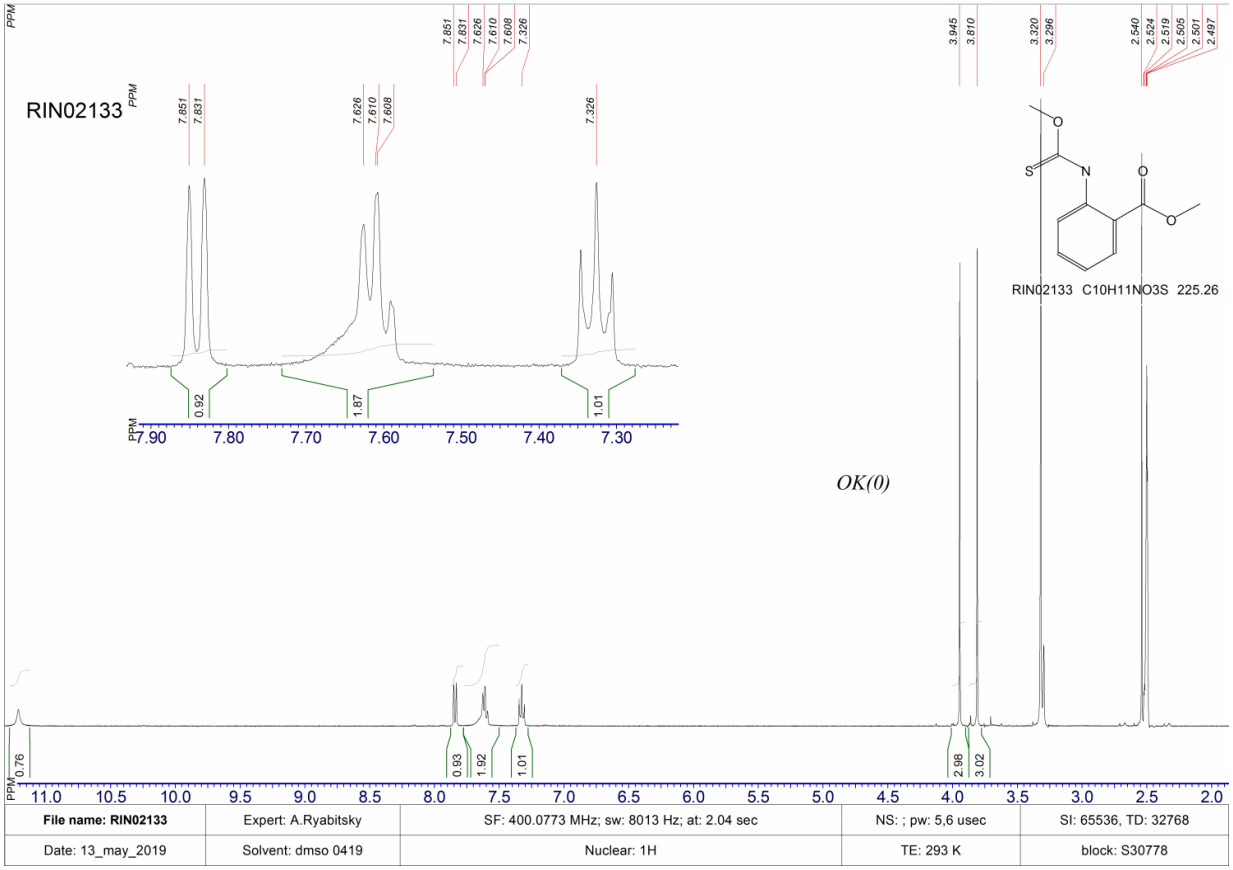


Figure S6. ^{1}H NMR spectrum (400 MHz, DMSO-d_6) of methyl 2-(methoxycarbonothioylamino)benzoate **14**.

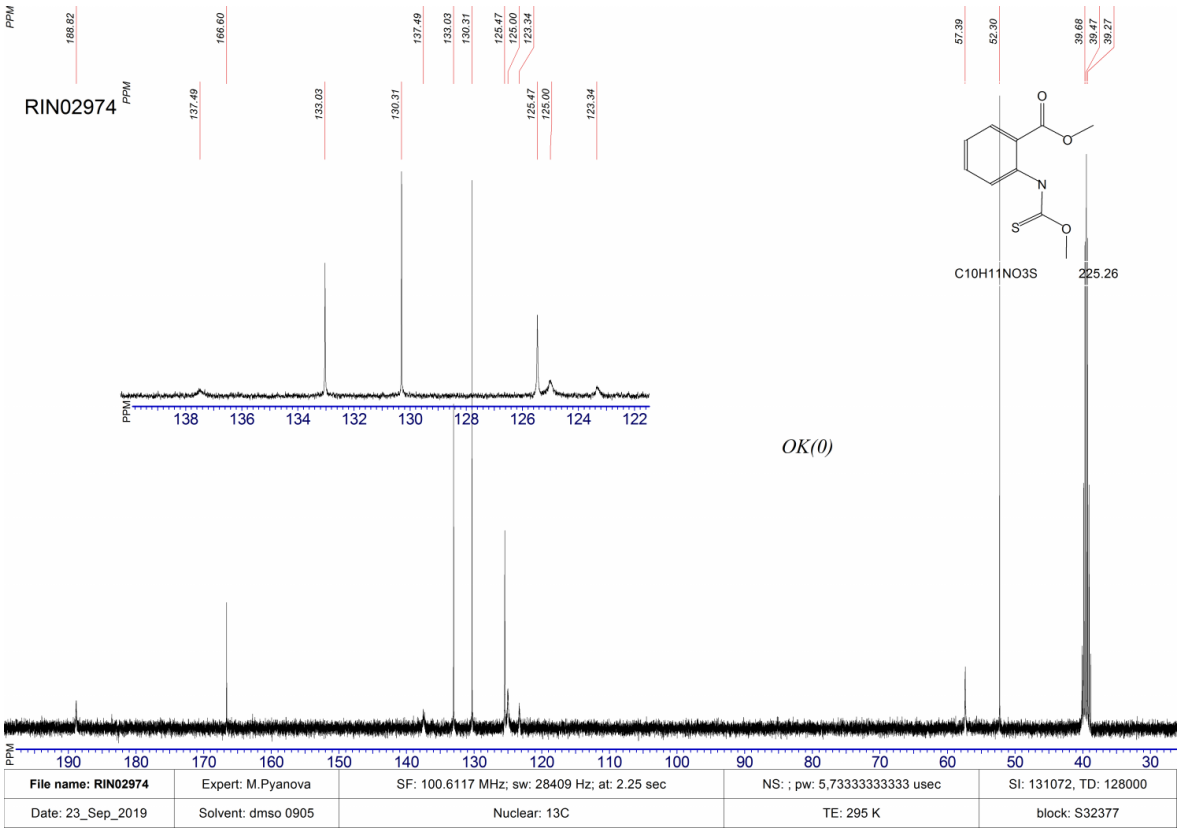


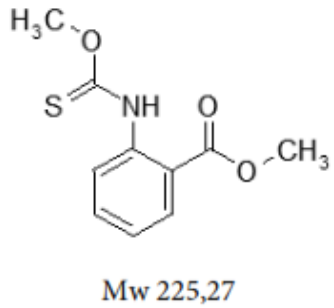
Figure S7. ^{13}C NMR spectrum (100 MHz, DMSO-d_6) of methyl 2-(methoxycarbonothioylamino)benzoate **14**.

X:\OUTPUT\2019\19_05\190515\M31062\RIN02133.D Sample Name: RIN02133 Page 1 of 1

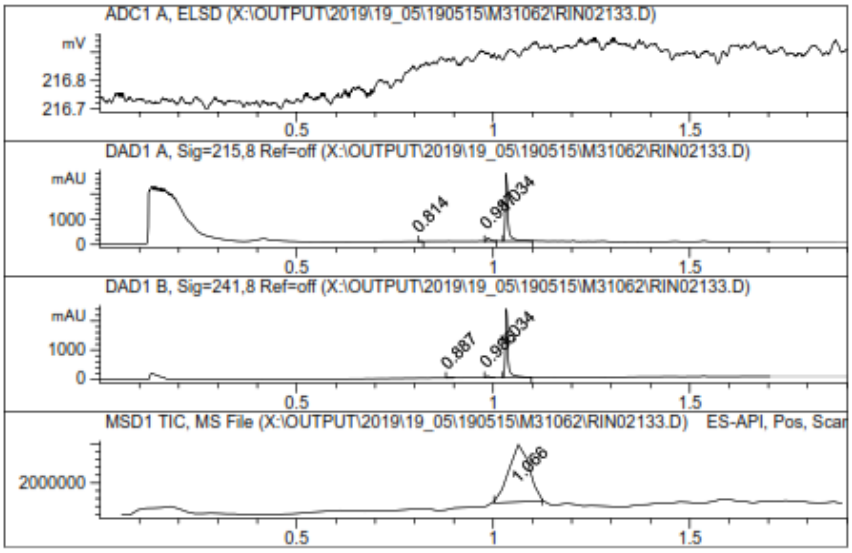
-.o.-Syntez Purity Report -.o.-

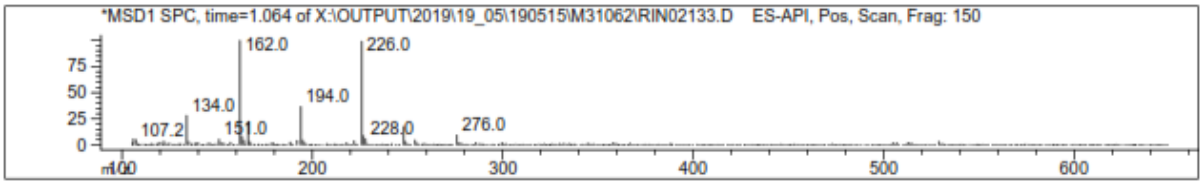
Agilent 1100 LC/MSD
Diodearrey G1315B (DAD1A-215nm; DAD1B-241nm)
Mass Quad G1956B (MSD1-Pos, MSD2-Neg)
ELSD Altech 3300 (ADC1 A, ELSD)

Mobile Phase:A-H2O+0.1HCOOH;B-MeCN+0.1HCOOH
Separation column: **96%**
Rapid Resolutionn HT Cartige 4.6x30mm,
1.8-Micron, Zorbax SB-C18



Mw 225,27





#	Signal	R.Time	Area %

#	Signal	R.Time	Area %
1	DAD1 A, Sig=215,8 Ref=off	0.814	0.431
2		0.987	6.275
3		1.034	93.294

#	Signal	R.Time	Area %
1	DAD1 B, Sig=241,8 Ref=off	0.887	0.461
2		0.986	2.556
3		1.034	96.983

#	Signal	R.Time	Area %
1	MSD1 TIC, MS File	1.066	100.000

Figure S8. LC/MS data for methyl 2-(methoxycarbonothioylamino)benzoate **14**

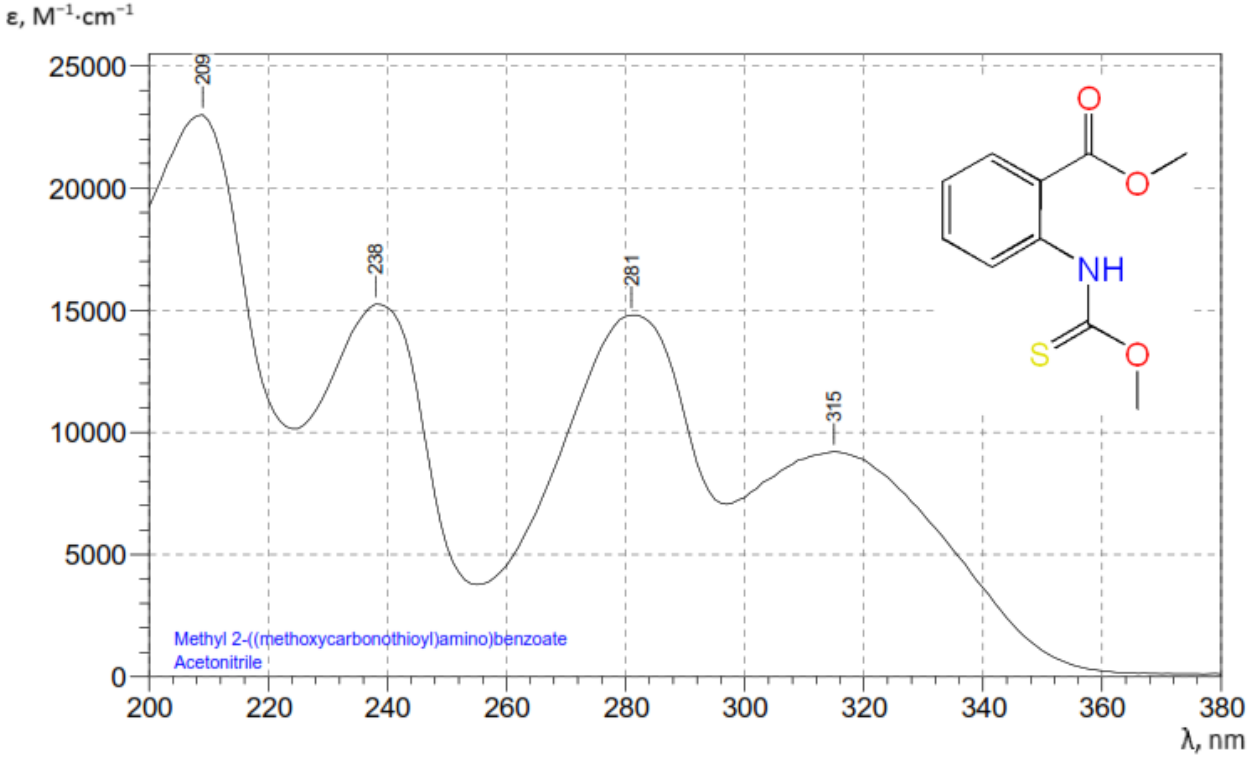


Figure S9. UV/Vis spectrum (CH_3CN) of methyl 2-(methoxycarbonothioylamino)benzoate **14**.

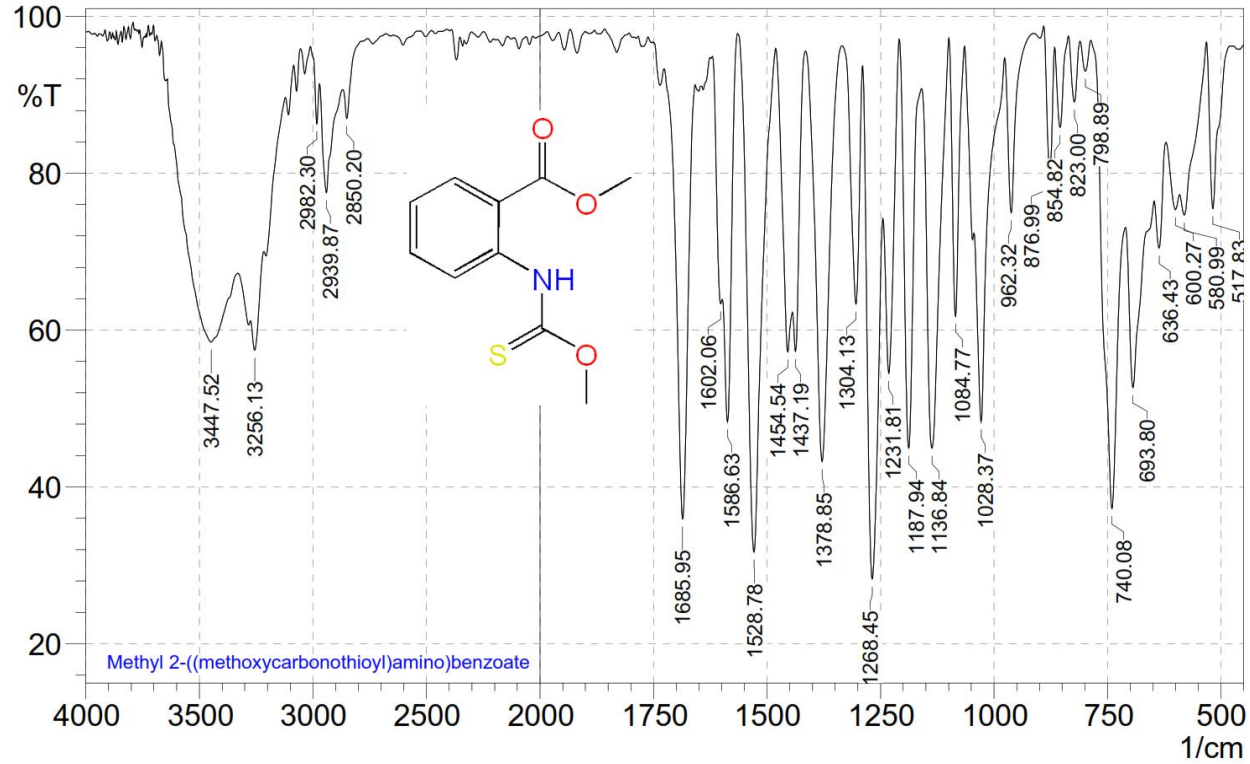


Figure S10. IR spectrum of methyl 2-(methoxycarbonothioylamino)benzoate **14** (KBr pellet).

Table S4. Coordinates (×10^4) and equivalent isotropic thermal parameters (Å^2 × 10^3) of atoms in structure **14**.

Atom	x	y	z	U(eq)
S1	1208(1)	205(3)	3774(1)	96(1)
O1	4293(3)	4132(6)	5905(3)	75(1)
O2	4110(2)	7873(5)	6427(2)	64(1)
O3	3040(3)	-1130(5)	4297(3)	71(1)
N1	2643(3)	1735(6)	5193(3)	61(1)
C1	2181(4)	3468(7)	5662(3)	52(1)
C2	1204(4)	3257(8)	5711(4)	60(1)
C3	769(4)	4949(9)	6187(4)	65(2)
C4	1295(4)	6887(8)	6641(4)	60(1)
C5	2267(4)	7115(8)	6617(3)	56(1)
C6	2719(3)	5422(7)	6138(3)	51(1)
C7	2301(4)	294(8)	4438(4)	58(1)
C8	2871(5)	-2909(9)	3538(4)	87(2)
C9	3774(4)	5681(9)	6130(3)	56(1)
C10	5124(4)	8358(8)	6423(4)	76(2)

Table S5. Bond lengths (Å) in structure **14**.

S1-C7	1.632(5)
O1-C9	1.207(5)
O2-C9	1.333(5)
O2-C10	1.449(6)
O3-C7	1.348(5)
O3-C8	1.442(5)
N1-C1	1.396(6)
N1-C7	1.344(5)
C1-C2	1.392(7)
C1-C6	1.407(6)
C2-C3	1.366(7)
C3-C4	1.381(6)
C4-C5	1.379(7)
C5-C6	1.382(6)
C6-C9	1.490(7)

Table S6. Valence angles (deg.) in structure **14**.

C9-O2-C10	116.8(4)
C7-O3-C8	119.4(4)
C7-N1-C1	131.2(4)
N1-C1-C6	119.7(5)
C2-C1-N1	121.6(4)
C2-C1-C6	118.6(4)
C3-C2-C1	120.8(5)

C2-C3-C4	120.6(5)
C5-C4-C3	119.8(5)
C4-C5-C6	120.4(5)
C1-C6-C9	120.5(4)
C5-C6-C1	119.9(5)
C5-C6-C9	119.6(4)
O3-C7-S1	124.0(4)
N1-C7-S1	128.5(4)
N1-C7-O3	107.4(4)
O1-C9-O2	121.9(5)
O1-C9-C6	126.1(5)
O2-C9-C6	111.9(4)