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Microwave irradiated synthesis of Schiff bases of 4-(arylideneamino)-5-alkyl-2,4-dihydro-1,2,4-triazole-3-thione containing 1,2,4-triazole segment

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Abstract: Novel compounds based on the 1,2,4-triazole skeleton were synthesized. A class of 4-amino-5-alkyl-4H-1,2,4-triazole-3-thione created by reaction of thiocarbohydrazide with long-chain aliphatic carboxylic acids, and then the Schiff bases were obtained in the media of heat and microwave waves, in the presence and the absence of a catalyst. Their chemical structures were assayed by elemental analysis, also device spectroscopic methods.

Key words: 4-Amino-5-alkyl-4H-1,2,4-triazole-3-thione, thiocarbohydrazide, long-chain aliphatic carboxylic acids, Schiff bases

1. Introduction

Over the past decades, heterocyclic compounds and their various derivatives have attracted chemists due to their diverse applications in chemical and pharmaceutical fields. Review references indicate that triazole compounds are of particular importance to other heterocyclic compounds due to their biological properties. 1,2,4-triazoles exhibit a variety of biological properties, such as antimicrobial [1–5], antiinflammatory [6–8], anticonvulsant [9,10], anticancer [11,12,14,15], antitubercular [13], antibacterial [1,7,16-19], antifungal [1,20–22], antitubulin [23], insecticides [24], herbicidal [25] and anticorrosion [26] activities. Due to potential properties of triazoles and the fact that one of the tasks of our research team is to investigate industrial emulsifiers, we expect such compounds to have emulsifying properties because they have a polar head and a long nonpolar tail. So, we decided to make compounds that have such characteristics in addition to being new.

We succeeded to synthesize the structures of 4a-f and 5a-l using thiocarbohydrazide.

2. Materials and methods

Solvents and analytical chemicals used were of analytical grade or dry distilled. The qualitative analysis of compounds was evaluated by TLC, and the Rf values were assessed using prefabricated aluminum-silicon plates and Kieselgel 60 F254 (obtained from Merck) by using ethyl acetate as a molecule and the TLC which then visualized by means of a UV lamp. Determining melting points was performed using Electrothermal melting furnace (B1 4300 BAMSETEP B1). Bruker Tensor 27 FT-IR spectrophotometer was used to record IR spectra. Recording the NMR spectrum was handled in a Bruker Avance DRX-300 spectrometer with TMS as standard.

Mass spectra recorded on Finnigan-Matt 5973. Elemental analysis for C, H, N, and S determined using a Heracus CHN-O-Rapid analyzer. Microwave irradiations were carried in a MicroSynth, Milestone microwave oven with 2500 W power.

2.1. Synthesis of 4-Amino-5-alkyl-2,4-dihydro-[1,2,4]triazole-3-thione (4a-f)

A mixture consisting of carboxylic acid (0.01 mol) and thiocarbohydrazide (0.015 mol) was made in a round-bottomed flask heated on a mantle until content melted. The resulting mixture was washed several times with warm water to remove unreacted thiocarbohydrazide and carboxylic acid and then collected by filtration. To produce prementioned compounds, the product was recrystallized using ethanol.

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2.1.1. 4-amino-5-heptyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (4a)
Yield 80%; white solid; m.p. 115–117 °C; IR υ (cm^{-1}): 3320, 3200, 3152 (NH strength vibration of NH and NH_2 groups), 2949-2851 (strength vibration of SP_3 CH), 1486 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.84 (t, 3H, CH_3), 1.23-1.26 (m, 8H, CH_2), 1.56-1.63 (m, 2H, CH_2), 2.59 (t, 2H, CH_2), 5.49 (s, 2H, NH_2), 13.40 (s, 1H, NH). Anal. Calcd. for $C_9H_{18}N_4S$: C 50.44, H 8.47, N 26.14, S 14.95. found C 50.41, H 8.49, N 26.04, S 15.06.

2.1.2. 4-amino-5-nonyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (4b)
Yield 79%; white solid; m.p. 114–115 °C; IR υ (cm^{-1}): 3320, 3200, 3152 (NH strength vibration of NH and NH_2 groups), 2941-2851 (strength vibration of SP_3 CH), 1486 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.84 (t, 3H, CH_3), 1.23-1.25 (m, 12H, CH_2), 1.58–1.64 (m, 2H, CH_2), 2.59 (t, 2H, CH_2), 5.50 (s, 2H, NH_2), 13.40 (s, 1H, NH). Anal. Calcd. for $C_{11}H_{22}N_4S$: C 54.51, H 9.15, N 23.12, S 13.22. found C 54.50, H 9.11, N 23.16, S 13.23.

2.1.3. 4-amino-5-undecyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (4c)
Yield 78%; white solid; m.p. 112–113.5 °C; IR υ (cm^{-1}): 3320, 3248, 3138 (NH strength vibration of NH and NH_2 groups), 2933-2851 (strength vibration of SP_3 CH), 1486 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.83 (t, 3H, CH_3), 1.23-1.26 (m, 16H, CH_2), 1.58–1.63 (m, 2H, CH_2), 2.59 (t, 2H, CH_2), 5.49 (s, 2H, NH_2), 13.40 (s, 1H, NH). Anal. Calcd. for $C_{13}H_{26}N_4S$: C 57.74, H 9.69, N 20.72, S 11.85. found C 57.80, H 9.65, N 20.75, S 11.80.

2.1.4. 4-amino-5-tridecyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (4d)
Yield 75%; white solid; m.p. 110–112.5 °C; IR υ (cm^{-1}): 3320, 3252, 3142 (NH strength vibration of NH and NH_2 groups), 2927-2851 (strength vibration of SP_3 CH), 1486 (bending vibration of CH2); 1H NMR (300 MHz DMSO-d6) δ 0.84 (t, 3H, CH3), 1.22-1.26 (m, 20H, CH2), 1.58–1.60 (m, 2H, CH2), 2.59 (t, 2H, CH2), 5.49 (s, 2H, NH2), 13.40 (s, 1H, NH). Anal. Calcd. for C15H30N4S: C 60.36, H 10.13, N 18.77, S 10.74. found C 60.26, H 10.20, N 18.80, S 10.74.

2.1.5. 4-amino-5-pentadecyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (4e)
Yield 71%; white solid; m.p. 108–110 °C; IR υ (cm^{-1}): 3320, 3153, 3043 (NH strength vibration of NH and NH_2 groups), 2920-2851 (strength vibration of SP_3 CH), 1486 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.83 (t, 3H, CH_3), 1.22-1.26 (m, 24H, CH_2), 1.58–1.60 (m, 2H, CH_2), 2.59 (t, 2H, CH_2), 5.48 (s, 2H, NH_2), 13.40 (s, 1H, NH). Anal. Calcd. for $C_{17}H_{34}N_4S$: C 62.53, H 10.50, N 17.16, S 9.82. found C 62.49, H 10.41, N 17.18, S 9.92.

2.1.6. 4-amino-5-heptadecyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (4f)
Yield 70%; white solid; m.p. 106–108 °C; IR υ (cm^{-1}): 3282, 3200, 3138 (NH strength vibration of NH and NH_2 groups), 2922-2850 (strength vibration of SP_3 CH), 1488 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.83 (t, 3H, CH_3), 1.21-1.25 (m, 28H, CH_2), 1.55–1.62 (m, 2H, CH_2), 2.58 (t, 2H, CH_2), 5.48 (s, 2H, NH_2), 13.39 (s, 1H, NH). Anal. Calcd. for $C_{19}H_{38}N_4S$: C 64.36, H 10.80, N 15.80, S 9.04. found C 64.25, H 10.70, N 15.90, S 9.15.

2.2. Synthesis of 4-(arylideneamino)-5-substituted-2,4-dihydro-1,2,4-triazole-3-thione (5a-l)

2.2.1. Conventional procedure
An equimolar amount of corresponding substituted benzaldehyde with 3 to 4 drops of glacial acetic acid was added to a suspension of substituted amino triazole (1.2 mol) in methanol. The reaction mixture vessel was refluxed at temperature of 80–90°C for duration of 2 to 3 hours. Obtained precipitate was then washed with water, subsequently filtered and finally dried.

2.2.2. Microwave procedure
Substituted amino triazole (1.2 mol) with an equimolar amount of the substituted benzaldehyde were mixed with 4–5 drops of DMSO and exposed to microwave irradiation at 80 °C (400 W) (see Table 1) using a Micro Synth lab station reactor. The reaction was carried within high-pressure Teflon reactor equipped with a magnetic stir bar and an optical fiber (to control resulting temperature).

Then, the mixture vessel was allowed to cool down while adding 20 mL water, and the obtained material was then filtered and washed using 10 mL of hot water and finally recrystallized in methanol.

2.2.3. 4-(benzylideneamino)-5-heptyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (5a)
Yield(%): 93(60); white solid; m.p. 119–121 °C; IR υ (cm^{-1}): 3114 (NH strength vibration of NH group), 2950-2850 (strength vibration of SP_3 CH), 1581, 1500 (strength vibration of C = C aromatic), 1469 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.82 (t, 3H, CH_3), 1.26–1.42 (m, 8H, CH_2), 1.74–1.80 (m, 2H, CH_2), 2.84 (t, 2H, CH_2), 7.46-7.87 (m, 5H, Ar), 10.35 (s, 1H, HC = N), 13.40 (s, 1H, NH); ^{13}C NMR (75 MHz DMSO-d6) δ 14.2-31.8 (aliphatic, 7 carbons), 128.6-133.4 (aromatic, 6 carbons) 153.9, 157.2 (imine groups, 2 carbons), 181.3 (thione group, 1 carbon); M.S, m/z 302 (M^+, 5%), 287 (M-NH, 3%), 258 (M-CS, 10%), 243 (M-NH-CS, 12%). Anal. Calcd. for $C_{16}H_{22}N_4S$: C 63.54, H 7.33, N 18.53, S 10.60. found C 63.60, H 7.30, N 18.55, S 10.55.

Table 1. Chemical structures of 1,2,4-triazole 4a-f and Schiff base 5a-l.

Entry	Compounds	R1	R2	Yield (%)	
				Conventional heating	Microwave heating (12 min.)
1	4a	n-C_7H_{15}	---	80	---
2	4b	n-C_9H_{19}	---	79	---
3	4c	n-$C_{11}H_{23}$	---	78	---
4	4d	n-$C_{13}H_{27}$	---	75	---
5	4e	n-$C_{15}H_{31}$	---	71	---
6	4f	n-$C_{17}H_{35}$	---	70	---
7	5a	n-C_7H_{15}	H	60	93
8	5b	n-C_9H_{19}	H	60	93
9	5c	n-$C_{11}H_{23}$	H	59	92
10	5d	n-$C_{13}H_{27}$	H	58	92
11	5e	n-$C_{15}H_{31}$	H	57	91
12	5f	n-$C_{17}H_{35}$	H	57	90
13	5g	n-C_7H_{15}	OH	62	94
14	5h	n-C_9H_{19}	OH	61	93
15	5i	n-$C_{11}H_{23}$	OH	61	93
16	5j	n-$C_{13}H_{27}$	OH	59	92
17	5k	n-$C_{15}H_{31}$	OH	59	91
18	5l	n-$C_{17}H_{35}$	OH	58	91

2.2.4. 4-(benzylideneamino)-5-nonyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (5b)

Yield(%): 93(60); white solid; m.p. 116–118 °C; IR υ (cm^{-1}): 3114 (strength vibration of NH group), 2950-2850 (strength vibration of SP_3 CH), 1581, 1500 (strength vibration of C = C aromatic), 1468 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.82 (t, 3H, CH_3), 1.21–1.40 (m, 12H, CH_2), 1.70-1.89 (m, 2H, CH_2), 2.80 (t, 2H, CH_2), 7.46-7.87 (m, 5H, Ar), 10.31 (s, 1H, HC=N), 13.40 (s, 1H, NH); ^{13}C NMR (75 MHz DMSO-d6) δ14.4-34.10 (aliphatic, 9 carbons), 128.6–133.4 (aromatic, 6 carbons), 153.9, 157.2 (imine, 2 carbons), 181.3 (thione, 1 carbon); M.S, m/z 330 (M^+, 6%), 315 (M-NH, 4%), 286 (N-C-S, 11%), 271 (M-NH-CS, 13%). Anal. Calcd. for $C_{18}H_{26}N_4S$: C 65.42, H 7.93, N 16.95, S 9.70. found C 65.55, H 7.86, N 16.84, S 9.75.

2.2.5. 4-(benzylideneamino)-5-undecyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (5c)

Yield(%): 92(59); white solid; m.p. 112–115 °C; IR υ (cm^{-1}): 3112 (strength vibration of NH group), 2950-2850 (strength vibration of SP_3 CH), 1581, 1500 (strength vibration of C = C aromatic), 1468 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.80 (t, 3H, CH_3), 1.24–1.41 (m, 16H, CH_2), 1.67–1.83 (m, 2H, CH_2), 2.78 (t, 2H, CH_2), 7.46–7.87 (m, 5H, Ar), 10.28 (s, 1H, HC = N), 13.39 (s, 1H, NH); ^{13}C NMR (75 MHz DMSO-d6)δ14.3-34.0 (aliphatic, 11 carbons), 128.8–133.2 (aromatic, 6 carbons), 154.9, 157.1 (imine, 2 carbons), 181.6 (thione, 1 carbon); M.S, m/z 358 (M^+, 4%), 343 (M-NH, 4%), 314 (N-C-S, 11%), 299 (M-NH-CS, 12%). Anal. Calcd. for $C_{20}H_{30}N_4S$: C 67.00, H 8.43, N 15.63, S 8.94. found C 66.91, H 8.50, N 15.60, S 8.99.

2.2.6. 4-(benzylideneamino)-5-tridecyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (5d)

Yield(%): 92(58); white solid; m.p. 99–111.5 °C; IR υ (cm^{-1}): 3112 (strength vibration of NH group), 2950-2850 (strength vibration of SP_3 CH), 1581, 1500 (strength vibration of C = C aromatic), 1468 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.80 (t, 3H, CH_3), 1.16–1.38 (m, 20H, CH_2), 1.60-1.62 (m, 2H, CH_2), 2.73 (t, 2H, CH_2), 7.47–7.86 (m, 5H, Ar), 10.23 (s, 1H, HC=N), 13.40 (s, 1H, NH); ^{13}C NMR (75 MHz DMSO-d6)δ14.1-35.1 (aliphatic, 13 carbons), 127.9–133.1 (aromatic, 6 carbons), 154.1, 158.1 (imine, 2 carbons), 181.2 (thione, 1 carbon); M.S, m/z 386 (M^+, 5%), 371 (M-NH, 4%), 342 (N-C-S, 12%), 327 (M-NH-CS, 11%). Anal. Calcd. for $C_{22}H_{34}N_4S$: C 68.36, H 8.86, N 14.49, S 8.29. found C 68.33, H 8.85, N 14.47, S 8.35.

2.2.7. 4-(benzylideneamino)-5-pentadecyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (5e)
Yield(%): 91(57); white solid; m.p. 96–98 °C; IR υ (cm^{-1}): 3112 (NH strength vibration of NH group), 2950-2850 (strength vibration of SP_3 CH), 1581, 1500 (strength vibration of C=C aromatic), 1468 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.80 (t, 3H, CH_3), 1.15–1.23 (m, 24H, CH_2), 1.57–1.64 (m, 2H, CH_2), 2.68 (t, 2H, CH_2), 7.47–7.86 (m, 5H, Ar), 10.23 (s, 1H, HC=N), 13.41 (s, 1H, NH); ^{13}C NMR (75 MHz DMSO-d6)δ14.1-29.7 (aliphatic, 15 carbons), 127.9-133.1 (aromatic, 6 carbons), 154.1, 158.1 (imine, 2 carbons), 181.2 (thione, 1 carbon); M.S, m/z 414 (M^+, 6%), 399 (M-NH, 6%), 370 (N-C-S, 13%), 355 (M-NH-CS, 14%). Anal. Calcd. for $C_{24}H_{38}N_4S$: C 69.52, H 9.24, N 13.51, S 7.73. found C 69.38, H 9.17, N 13.63, S 7.82.

2.2.8. 4-(benzylideneamino)-5-heptadecyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (5f)
Yield(%): 90(57); white solid; m.p. 90–92 °C; IR υ (cm^{-1}): 3115 (NH strength vibration of NH group), 2921-2851 (strength vibration of SP_3 CH), 1583, 1499 (strength vibration of C=C aromatic), 1417 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.81 (t, 3H, CH_3), 1.08–1.26 (m, 28H, CH_2), 1.60–1.71 (m, 2H, CH_2), 2.69 (t, 2H, CH_2), 7.52-7.88 (m, 5H, Ar), 10.23 (s, 1H, HC = N), 13.40 (s, 1H, NH); ^{13}C NMR (75 MHz DMSO-d6) δ13.9-34.2 (aliphatic, 17 carbons), 128.7-132.9 (aromatic, 6 carbons), 153.7, 157.2 (imine, 2 carbons), 182.1 (thione, 1 carbon); M.S, m/z 442 (M^+, 7%), 427 (M-NH, 5%), 398 (N-C-S, 9%), 383 (M-NH-CS, 13%). Anal. Calcd. for $C_{26}H_{42}N_4S$: C 70.54, H 9.56, N 12.66, S 7.24. found C 70.61, H 9.51 N 12.61, S 7.27.

2.2.9. 5-heptyl-4-((2-hydroxybenzylidene)amino)-2,4-dihydro-3H-1,2,4-triazole-3-thione (5g)
Yield(%): 94(62); white solid; m.p. 125–127 °C; IR υ (cm^{-1}): 3450-3200 (strength vibration of OH group), 3105 (strength vibration of NH group), 2917-2850 (strength vibration of SP_3 CH), 1585, 1488 (strength vibration of C=C aromatic), 1465 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.82 (t, 3H, CH_3), 1.26–1.42 (m, 8H, CH_2), 1.56–1.71 (m, 2H, CH_2), 2.73 (t, 2H, CH_2), 6.80–7.33 (m, 4H, Ar), 10.32 (s, 1H, HC=N), 10.89 (s, 1H, OH), 13.40 (s, 1H, NH); ^{13}C NMR (75 MHz DMSO-d6) δ13.8-31.9 (aliphatic, 7 carbons), 117.7–132.3, 156.7 (aromatic, 6 carbons), 143.4, 156.0 (imine, 2 carbons), 181.2 (thione, 1 carbon); M.S, m/z 318 (M^+, 6%), 303 (M-NH, 5%), 274 (N-C-S, 10%), 259 (M-NH-CS, 11%). Anal. Calcd. for $C_{16}H_{22}N_4OS$: C 60.35, H 6.96, N 17.59, O 5.03, S 10.07. found C 60.50, H 6.99, N 17.51, O 4.99 S 10.0.1

2.2.10. 4-((2-hydroxybenzylidene)amino)-5-nonyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (5h)
Yield(%): 93(61); white solid; m.p. 121–124 °C; IR υ (cm^{-1}): 3430-3190 (strength vibration of OH group), 3150 (strength vibration of NH group), 2930-2860 (strength vibration of SP_3 CH), 1590, 1495 (strength vibration of C = C aromatic), 1475 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.83 (t, 3H, CH_3), 1.20–1.25 (m, 12H, CH_2), 1.60–1.65 (m, 2H, CH_2), 2.65 (t, 2H, CH_2), 6.83-7.45 (m, 4H, Ar), 10.51 (s, 1H, HC = N), 10.89 (s, 1H, OH), 13.40 (s, 1H, NH); ^{13}C NMR (75 MHz DMSO-d6) δ14.3-34.2 (aliphatic, 9 carbons), 117.5–132.1, 157.2 (aromatic, 6 carbons), 143.1, 156.3 (imine, 2 carbons), 181.5 (thione, 1 carbon); M.S, m/z 346 (M^+, 5%), 331 (M-NH, 4%), 302 (N-C-S, 12%), 287 (M-NH-CS, 14%). Anal. Calcd. for $C_{18}H_{26}N_4OS$: C 62.40, H 7.56, N 16.17, O 4.62, S 9.25. found C 62.30, H 7.53, N 16.21, O 4.66, S 9.30

2.2.11. 4-((2-hydroxybenzylidene)amino)-5-undecyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (5i)
Yield(%): 93(61); white solid; m.p. 116-118 °C; IR υ (cm^{-1}): 3500-3250 (strength vibration of OH group), 3180 (strength vibration of NH group), 2950-2840 (strength vibration of SP_3 CH), 1595, 1500 (strength vibration of C=C aromatic), 1470 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.82 (t, 3H, CH_3), 1.26–1.42 (m, 16H, CH_2), 1.47–1.80 (m, 2H, CH_2), 2.49 (t, 2H, CH_2), 6.79–7.45 (m, 4H, Ar), 10.50 (s, 1H, HC = N), 10.89 (s, 1H, OH), 13.41 (s, 1H, NH); ^{13}C NMR (75 MHz DMSO-d6) δ14.4-34.1 (aliphatic, 11 carbons), 117.5-133.1, 157.4 (aromatic, 6 carbons), 144.8, 156.9 (imine, 2 carbons), 181.4 (thione, 1 carbon); M.S, m/z 374 (M^+, 5%), 359 (M-NH, 5%), 330 (N-C-S, 12%), 315 (M-NH-CS, 11%). Anal. Calcd. for $C_{20}H_{30}N_4OS$: C 64.14, H 8.07, N 14.96, O 4.27, S 8.56. found C 64.25, H 8.05, N 14.86, O 4.31, S 8.53

2.2.12. 4-((2-hydroxybenzylidene)amino)-5-tridecyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (5j)
Yield(%): 92(59); white solid; m.p. 112-115 °C; IR υ (cm^{-1}): 3480-3210 (strength vibration of OH group)), 3145 (strength vibration of NH group), 2945-2833 (strength vibration of SP_3 CH), 1600, 1490 (strength vibration of C = C aromatic), 1466 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.81 (t, 3H, CH_3), 1.06–1.18 (m, 20H, CH_2), 1.56–1.63 (m, 2H, CH_2), 2.67 (t, 2H, CH_2), 6.82-7.44 (m, 4H, Ar), 10.49 (s, 1H, HC = N), 10.89 (s, 1H, OH), 13.39 (s, 1H, NH); ^{13}C NMR (75 MHz DMSO-d6) δ14.5-34.9 (aliphatic, 13 carbons), 117.3–131.9, 157.7 (aromatic, 6 carbons), 143.6, 157.1 (imine, 2 carbons), 181.3 (thione, 1 carbon); M.S, m/z 402 (M^+, 6%), 387 (M-NH, 4%), 358 (N-C-S, 12%), 343 (M-NH-CS, 12%). Anal. Calcd. for $C_{22}H_{34}N_4OS$: C 65.63, H 8.51, N 13.92, O 3.97 S 7.97. found C 65.75, H 8.47, N 13.85, O 3.95, S 7.98.

2.2.13. 4-((2-hydroxybenzylidene)amino)-5-pentadecyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (5k)
Yield(%): 91(59); white solid; m.p. 108–110 °C; IR υ (cm^{-1}): 3500-3200 (strength vibration of OH group)), 3105 (strength vibration of NH group), 2917-2850 (strength vibration of SP_3 CH), 1597, 1509 (strength vibration of C = C aromatic), 1467 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.81 (t, 3H, CH_3), 1.26-1.32 (m, 24H, CH_2), 1.74–1.80 (m,

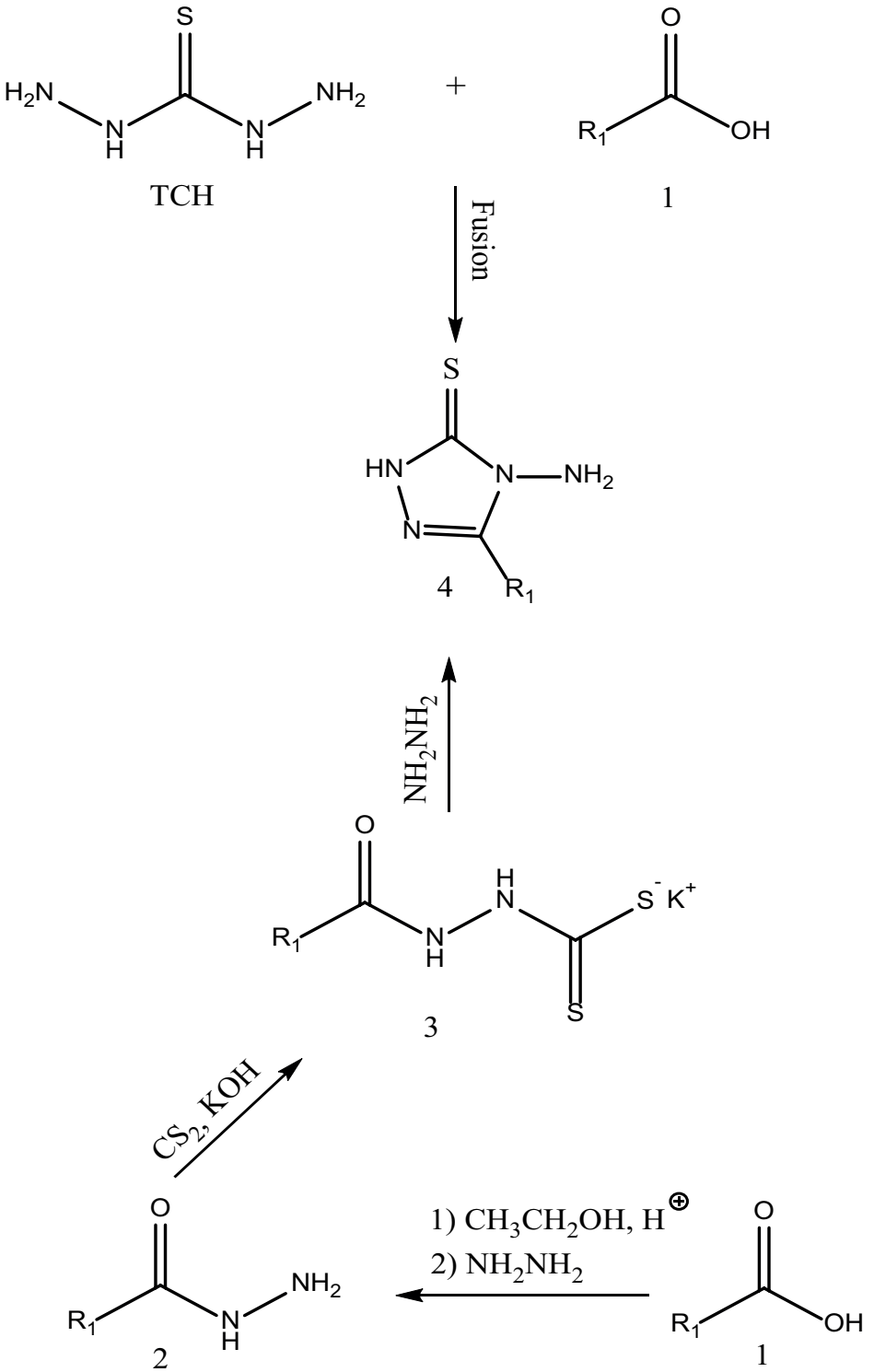


Figure 1. Synthesis of 1,2,4-triazole amines 4a-f.

2H, CH_2), 2.65 (t, 2H, CH_2), 6.80–7.46 (m, 4H, Ar), 10.43 (s, 1H, HC=N), 10.84 (s, 1H, OH), 13.40 (s, 1H, NH); ^{13}C NMR (75 MHz DMSO-d6) δ14.6-34.0 (aliphatic, 15 carbons), 117.8–132.3, 157.8 (aromatic, 6 carbons), 143.3, 157.1 (imine, 2 carbons), 182.1 (thione, 1 carbon); M.S, m/z 430 (M^+, 7%), 415 (M-NH, 3%), 386 (N-C-S, 11%), 371 (M-NH-CS, 14%). Anal. Calcd. for $C_{24}H_{38}N_4OS$: C 66.94, H 8.89, N 13.01, O 3.72 S 7.44. found C 67.01, H 8.94, N 12.96, O 3.69, S 7.40.

2.2.14. 5-heptadecyl-4-((2-hydroxybenzylidene)amino)-2,4-dihydro-3H-1,2,4-triazole-3-thione (5l)
Yield(%): 91(58); white solid; m.p. 97–99 °C; IR υ (cm^{-1}): 3480-3330 (strength vibration of OH group)), 3105 (strength vibration of NH group), 2918-2840 (strength vibration of SP_3 CH), 1593, 1505 (strength vibration of C = C aromatic), 1466 (bending vibration of CH_2); ^{1}H NMR (300 MHz DMSO-d6) δ 0.80 (t, 3H, CH_3), 1.16–1.22 (m, 28H, CH_2), 1.57–1.64 (m, 2H, CH_2), 2.68 (t, 2H, CH_2), 6.85–7.46 (m, 4H, Ar), 10.44 (s, 1H, HC = N), 10.88 (s, 1H, OH), 13.41 (s, 1H, NH); ^{13}C NMR (75 MHz DMSO-d6) δ14.9-34.2 (aliphatic, 17 carbons), 118.1–131.9, 157.1 (aromatic, 6 carbons), 143.4, 156.9 (imine, 2 carbons), 182.4 (thione, 1 carbon); M.S, m/z 458 (M^+, 6%), 443 (M-NH, 5%), 414 (N-C-S, 11%), 399 (M-NH-CS, 11%). Anal. Calcd. for $C_{26}H_{42}N_4OS$: C 68.08, H 9.22, N 12.21, O 3.50, S 6.99. found C 68.07, H 9.25, N 12.18, O 3.52, S 6.98.

3. Results and discussion
In a continuous research works of this group [27–34], 18 triazole amines and Schiff bases were synthesized. The difference among these compounds is in the R_1 acid group and the R_2 aldehyde group. Our research team performed the reaction of thiocarbohydrazide (TCH) with long-chain aliphatic carboxylic acids (1a-f) in different conditions and analyzed the reaction of the resulting products with benzaldehyde and its derivatives under various conditions.

The reaction of TCH with octanoic acid 1a in the fusion method yielded product 4a (Figure 1), and it was confirmed as 4-amino-5-heptyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (4a). The structure of 4a was assessed and approved as compared to its spectral data (^{1}H NMR, IR). Also, triazole 4a was synthesized in three steps reaction as well. Hydrazide (2) was obtained by the ethanolysis and then hydrazinolysis of carboxylic acid (1). The required dithiocarbazinate (3) was synthesized by reacting hydrazide with carbon disulfide and ethanolic solution of potassium hydroxide. And 1,2,4-triazole(4) was produced by the reaction of intramolecular cyclization from the yielded salt with hydrazine. The advantage of the first method is the lower steps and higher efficiency. The triazole synthesized in this way was then condensed with benzaldehyde. As a catalyst, the reaction was performed in the presence of concentrated sulfuric acid or glacial acetic acid (few drops) to yield Schiff base 5 (Figure 2). The Schiff base formation reaction was carried out by the use of bronsted acid. The reaction was also accomplished in the presence of heterogeneous inorganic solid acidic catalysts such as beta-zeolite and montmorillonite-KSF under heat conditions. And also, the reaction was conducted in the absence of catalyst in microwave environment. The efficiency obtained using zeolite and montmorillonite was approximately similar. Besides, when the reaction was carried out under microwave conditions, the reaction time was reduced and the efficiency increased. The obtained yield using catalyst and in conventional method was moderate. Reactions were evaluated at different times. The best results were obtained with acetic acid in the thermal method for 90 min and in the microwave method for 12 min (see Table 2).

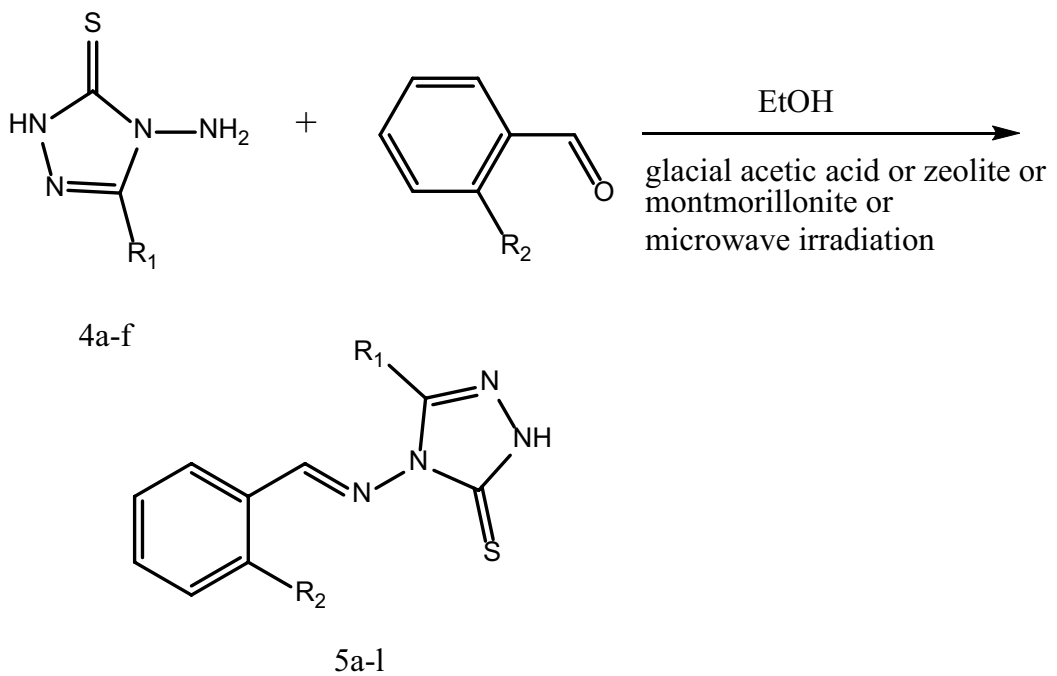


Figure 2. Synthesis of Schiff base 5a-l.

Table 2. The effect of catalyst on the reaction efficiency of Schiff base 5a*.

Time (min.)	Yield (%)			
	Conventional heating			**Microwave heating**
	Acetic acid	β-Zeolite	Montmorillonite KSF	
5	---	---	---	90
8	---	---	---	91
10	---	---	---	92
12	---	---	---	93
30	53	51	54	---
45	55	51	55	---
60	56	52	56	---
90	60	52	57	---

* The effect of the catalyst was investigated only on 5a. The reaction efficiency using microwaves did not change significantly with increasing time. The start of the reaction in the presence of the catalyst was after 30 min.

The path for synthesizing proposed compounds 4a-f and 5a-l is outlined in Figures 1 and 2. 4a-f was approved by IR and ^{1}H NMR spectroscopic methods. 5a-f was also fully characterized using various spectroscopic methods. The progress for reactions was assessed using TLC (thin layer chromatography). Elemental analysis was used to check the purity of all compounds.

The position of IR bands suggests enough evidence regarding the formation of 4a and 5a. The bands due to υ (C = N) and υ (C = S) stretch at 1616 cm^{-1} and 1223 cm^{-1}, which approves the formation of triazole 4a. The absence of υ (NH_2) band in the IR spectrum of 4a shows the formation of 5a.

Other signs for the formation of 4a and 5a were attained by ^{1}H NMR and ^{13}C NMR spectroscopies. In the ^{1}H NMR of triazole 4a in d_6-DMSO, signals for the NH proton of triazole ring and for the NH_2 protons were observed at 13.40 ppm and 5.49 ppm, respectively. In the ^{1}H NMR of Schiff base 5a in d_6-DMSO, signal for the CH proton of imine bond was observed at 10.35 ppm, beside the ^{13}C NMR spectrum signal at 154.1 or 157.0 ppm due to -CH=N- carbon atom. Also, in the mass spectrum of 5a, fragment 302 obtained. These results support for the proposed structures of 4a and 5a.

4. Conclusion

In this work, Schiff base 5 was prepared in two steps. In the first stage, amine triazole 4 was prepared from the reaction of thiocarbohydrazide and carboxylic acid, and in the second stage, compound 4 reacted with benzaldehyde and its derivatives and the final product 5 was obtained.The second step reaction was performed using sulfuric or acetic acid, zeolite, montmorillonite, and microwave irradiations. The best efficiency was achieved in the microwave.

References

1. Behalo MS, Gad El-karim IA, Issac YA, Farag MA. Synthesis of novel pyridazine derivatives as potential antimicrobial agents. Journal of Sulfur Chemistry 2014; 35 (3): 661-673. doi: 10.1080/17415993.2014.950661

2. Mishra R, Kumar R, Kumar S, Majeed J, Rashid M et al. Synthesis and in vitro antimicrobial activity of some triazole derivatives. Journal of the Chilean Chemical Society 2010; 55 (3): 359-362. doi: 10.4067/S0717-97072010000300019

3. Karabasanagouda T, Adhikari AV, Shetty NS. Synthesis and antimicrobial activities of some novel 1,2,4-triazolo[3,4-b]-1,3,4-thiadiazoles and 1,2,4-triazolo[3,4-b]-1,3,4-thiadiazines carrying thioalkyl and sulphonyl phenoxy moieties. European Journal of Medicinal Chemistry 2007; 42 (4): 521-529. doi: 10.1016/j.ejmech.2006.10.010

4. Patil BS, Krishnamurthy G, Shashikumar ND, Lokesh MR, Naik HSB. Synthesis and antimicrobial activity of some [1,2,4]-triazole derivatives. Journal of Chemistry 2013; 1-7. doi: 10.1155/2013/462594

5. Li C, Liu JC, Li YR, Gou C, Zhang ML et al. Synthesis and antimicrobial evaluation of 5-aryl-1, 2, 4-triazole-3-thione derivatives containing a rhodanine moiety. Bioorganic & Medicinal Chemistry Letters 2015; 25 (15): 3052-3056. doi: 10.1016/j.bmcl.2015.04.081

6. Popiołek L, Biernasiuk A, Paruch K, Paruch K, Patrejko P, Wujec M. Synthesis and evaluation of antimicrobial properties of new Mannich bases of 4,5-disubstituted-1,2,4-triazole-3-thiones. Phosphorus, Sulfur, and Silicon and the Related Elements 2017; 192 (7): 880–885. doi: 10.1080/10426507.2017.1290629

7. El-Serwy WS, Mohamed NA, Abbas EM, Abdel-Rahman RF. Synthesis and anti-inflammatory properties of novel 1,2,4-triazole derivatives. Research on Chemical Intermediates 2013; 39 (6): 2543–2554. doi: 10.1007/s11164-012-0781-9

8. Hussein MA, Shaker RM, Ameen MA, Mohammad MF. Synthesis, anti-inflammatory, analgesic, and antibacterial activities of some triazole, triazolothiadiazole, and triazolothiadiazine derivatives. Archives of Pharmacal Research 2011; 34 (8): 1239-1250. doi: 10.1007/s12272-011-0802-z

9. El-Shehry MF, Abu-Hashem AA, El-Telbani EM. Synthesis of 3-((2,4-dichlorophenoxy)methyl)-1,2,4-triazolo (thiadiazoles and thiadiazines) as anti-inflammatory and molluscicidal agents. European Journal of Medicinal Chemistry 2010; 45 (5): 1906–1911. doi: 10.1016/j.ejmech.2010.01.030

10. Romagnoli R, Baraldi PG, Salvador MK, Prencipe F, Bertolasi V et al. Synthesis, antimitotic and antivascular activity of 1-(3′,4′,5′-trimethoxybenzoyl)-3-arylamino-5-amino-1,2,4-triazoles. Journal of Medicinal Chemistry 2014; 57 (15): 6795–6808. doi: 10.1021/jm5008193

11. Husain A, Naseer MA, Sarafroz M. Synthesis and anticonvulsant activity of some novel fused heterocyclic 1,2,4-triazolo-[3,4-b]-1,3,4-thiadiazole derivatives. Acta Poloniae Pharmaceutica2009; 66 (2): 135-140. pmid: 19719046

12. Kamal A, Khan MNA, Reddy KS, Srikanth YVV, Sridhar B. Synthesis, structural characterization and biological evaluation of novel [1,2,4] triazolo[1,5-b][1,2,4]benzothiadiazine-benzothiazole conjugates as potential anticancer agents. Chemical Biology & Drug Design 2008; 71 (1): 78–86. doi: 10.1111/j.1747-0285.2007.00609.x

13. Joshi SD, Vagdevi HM, Vaidya VP, Gadaginamath GS. Synthesis of new 4-pyrrol-1-yl benzoic acid hydrazide analogs and some derived oxadiazole, triazole and pyrrole ring systems: A novel class of potential antibacterial and antitubercular agents. European Journal of Medicinal Chemistry 2008; 43 (9): 1989-1996. doi: 10.1016/j.ejmech.2007.11.016

14. Ghammamy S, Sedaghat S. Synthesis, Characterization, and antitumor activity of azomethine derivative of triazole and its complexes with copper(I) and zinc(II) salts. Russian Journal of General Chemistry 2013; 83 (4): 722–725. doi: 10.1134/S1070363213040191

15. Ibrahim DA. Synthesis and biological evaluation of 3,6-disubstituted [1,2,4]triazolo[3,4-b][1,3,4]thiadiazole derivatives as a novel class of potential anti-tumor agents. European Journal of Medicinal Chemistry 2009; 44 (7): 2776–2781. doi: 10.1016/j.ejmech.2009.01.003

16. Maqsood MR, Hanif M, Rafiq M, Saleem M, Ziba S et al. Some pyridyl- and thiophenyl- substituted 1,2,4-triazolo[3,4-b]1,3,4-thiadiazole derivatives as potent antibacterial. Bulletin of the Korean Chemical Society 2012; 33 (12): 4180-4184. doi: 10.5012/bkcs.2012.33.12.4180

17. Plech T, Wujec M, Kosikowska U, Malm A, Kapron B. Studies on the synthesis and antibacterial activity of 3,6-disubstituted 1,2,4-triazolo[3,4-b]1,3,4-thiadiazoles. European Journal of Medicinal Chemistry 2012; 47: 580-584. doi: 10.1016/j.ejmech.2011.10.055

18. Tee EHL, Karoli T, Ramu S, Huang JX, Butler MS et al. Synthesis of essramycin and comparison of its antibacterial activity. Journal of Natural Products 2010; 73 (11): 1940–1942. doi: 10.1021/np100648q

19. Yang J, Zhao Z, Li H. Synthesis using microwave irradiation, characterisation and antibacterial activity of novel deoxycholic acid-triazole conjugates. Journal of Chemical Research 2012; 36 (7): 383–386. doi: 10.3184/174751912X13364636691591

20. Joshi R, Pandey N, Yadav SK, Tilak R, Mishra H et al. Synthesis, spectroscopic characterization, DFT studies and antifungalactivity of (E)-4-amino-5-[N'-(2-nitro-benzylidene)-hydrazino]-2,4-dihydro-[1,2,4]triazole-3-thione. Journal of Molecular Structure 2018; 1164: 386-403. doi: 10.1016/j.molstruc.2018.03.081

21. Mares D, Romagnoli C, Andreotti E, Manfrini M, Vicentini CB. Synthesis and antifungal action of new tricyclazole analogues. Journal of Agricultural and Food Chemistry 2004; 52 (7): 2003-2009. doi: 10.1021/jf030695y

22. Wang BL, Zhan YZ, Zhang LY, Zhang Y, Zhang X et al. Synthesis and fungicidal activities of novel 1,2,4-triazole thione derivatives containing 1,2,3-triazole and substituted piperazine moieties. Phosphorus, Sulfur, and Silicon and the Related Elements 2015; 191 (1): 1–7. doi: 10.1080/10426507.2015.1085040

23. Xu Q, Sun M, Bai Z, Wang Y, Wu Y et al. Design, synthesis and bioevaluation of antitubulin agents carrying diaryl-5,5-fused-heterocycle scaffold. European Journal of Medicinal Chemistry 2017; 139: 242–249. doi: 10.1016/j.ejmech.2017.05.065

24. Hull JW, Romer DR, Adaway TJ, Podhorez DE. Development of manufacturing processes for a new family of 2,6-dihaloaryl1,2,4-triazole insecticides. Organic Process Research & Development 2009; 13 (6): 1125–1129. doi: 10.1021/op9001577

25. Ma Y, Liu R, Gong X, Li Z, Huang Q et al. Synthesis and herbicidal activity of N,N-Diethyl-3-(arylselenonyl)-1H-1,2,4-triazole-1-carboxamide. Journal of Agricultural and Food Chemistry 2006; 54 (20): 7724-7728. doi: 10.1021/jf0609328

26. Kumar MS, Kumar SLA, Sreekanth A. Anticorrosion potential of 4-Amino-3-methyl-1,2,4-triazole-5-thione derivatives (SAMTT and DBAMTT) on mild steel in hydrochloric acid solution. Industrial & Engineering Chemistry Research 2012; 51 (15): 5408–5418. doi: 10.1021/ie203022g

27. Azizian J, Soozangarzadeh S, Jadidi K. Microwave-induced one-pot systhesis of some new spiro[3H-indol-3,5′(4′H)-[1,2,4]- triazoline]-2-ones. Synthetic Communications 2001; 31 (7): 1069-1073. doi: 10.1081/SCC-100103539

28. Azizian J, Varasteh-Morady A, Soozangarzadeh S, Asadi A. Synthesis of novel spiro-[3H-indole-3,3′-[1,2,4]triazolidine]-2-ones via azomethine imines. Tetrahedron Letters 2002; 43 (52): 9721-9723. doi: 10.1016/S0040-4039(02)02061-0

29. Azizian J, Madani M, Souzangarzadeh S. One-pot synthesis of novel derivatives of spiro-[6H-indolo [2,1-b]quinazoline-6,3′-[1,2,4] oxadiazoline]. Synthetic Communications 2005; 35 (6): 765. doi: 10.1081/scc-200050932

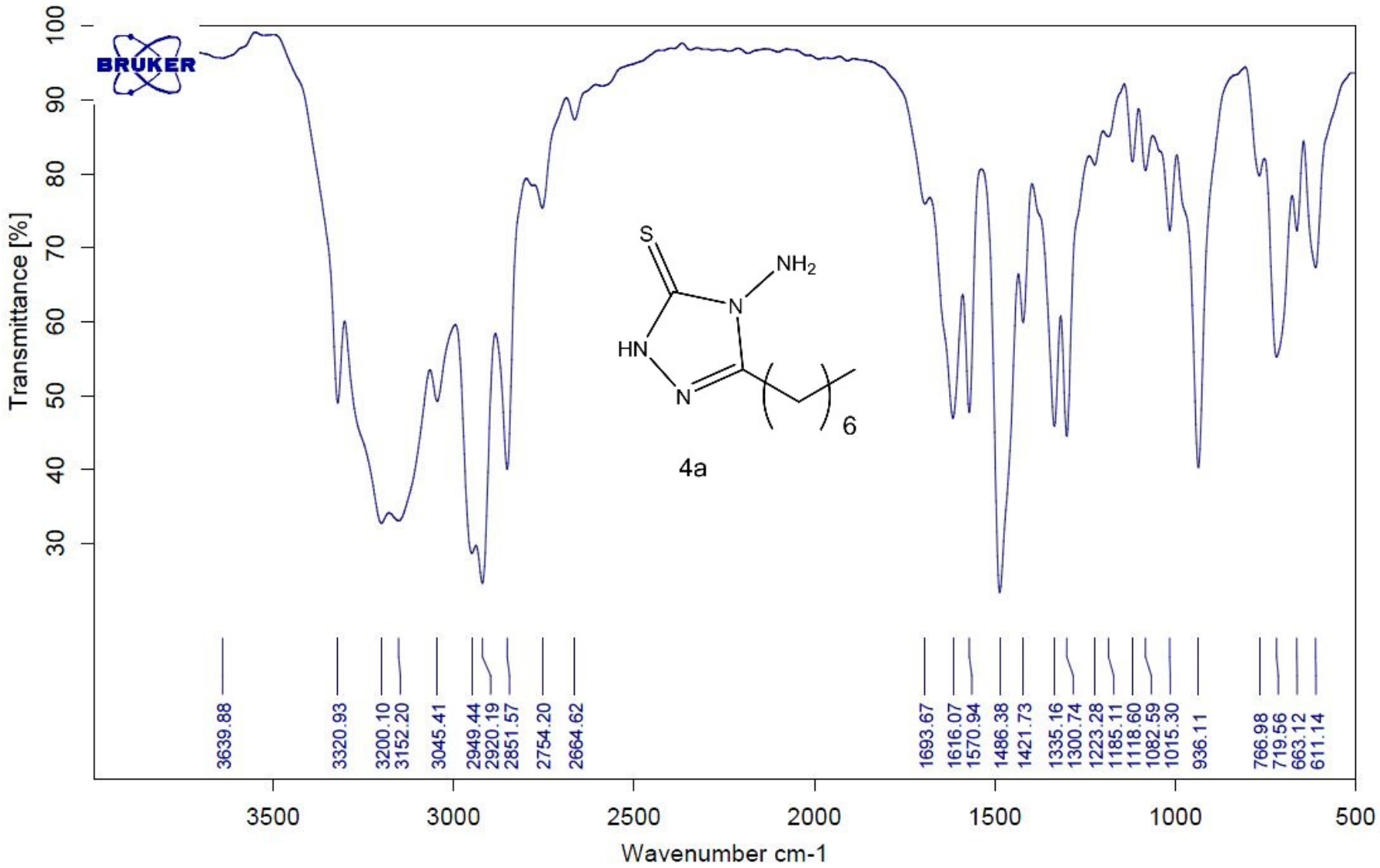
30. Souzangarzadeh S, Bazian A, Anaraki-Ardakani H. A facile synthesis of novel spiro indoline-based heterocycles through 1,3-dipolar cycloaddition reactions. Journal of Chemical Research 2012; 36 (2): 94-95. doi: 10.3184/174751912X13279492179667

31. Hajiaghababaei L, Sharafi A, Suzangarzadeh S, Faridbod F. Mercury Recognition: A Potentiometric Membrane Sensor based on 4-(Benzylidene amino)-3,4-dihydro-6- methyl-3-thioxo-1,2,4-triazin-5(2H)one. Analytical and Bioanalytical Chemistry 2013; 5 (4): 481-493.

32. Souzangarzadeh S. 1,3-Dipolar cycloaddition reaction of nitrile oxides to isatin imines. Iranian Journal of Chemistry and Chemical Engineering 2016; 35 (1): 31-35. doi: 10.30492/IJCCE.2016.18804

33. Hajiaghababaei L, Zandinejad S, Berijani S, Suzangarzadeh S. Ultrasound assisted emulsification microextraction for selective determination of trace amount of mercury(II). Indian Journal of Chemistry 2016; 55: 423-428.

34. Sharifi A, Hajiaghababaei L, Suzangarzadeh S, Jalali-Sarvestani MR. Synthesis of 3-((6-methyl-5-oxo-3-thioxo-2,5-dihydro-1,2,4-triazin-4(3H)-yl)imino)indolin-2-one as an excellent ionophore to the construction of a potentiometric membrane sensor for rapid determination at zinc. Analytical & Bioanalytical Electrochemistry 2017; 9 (7): 888-9033.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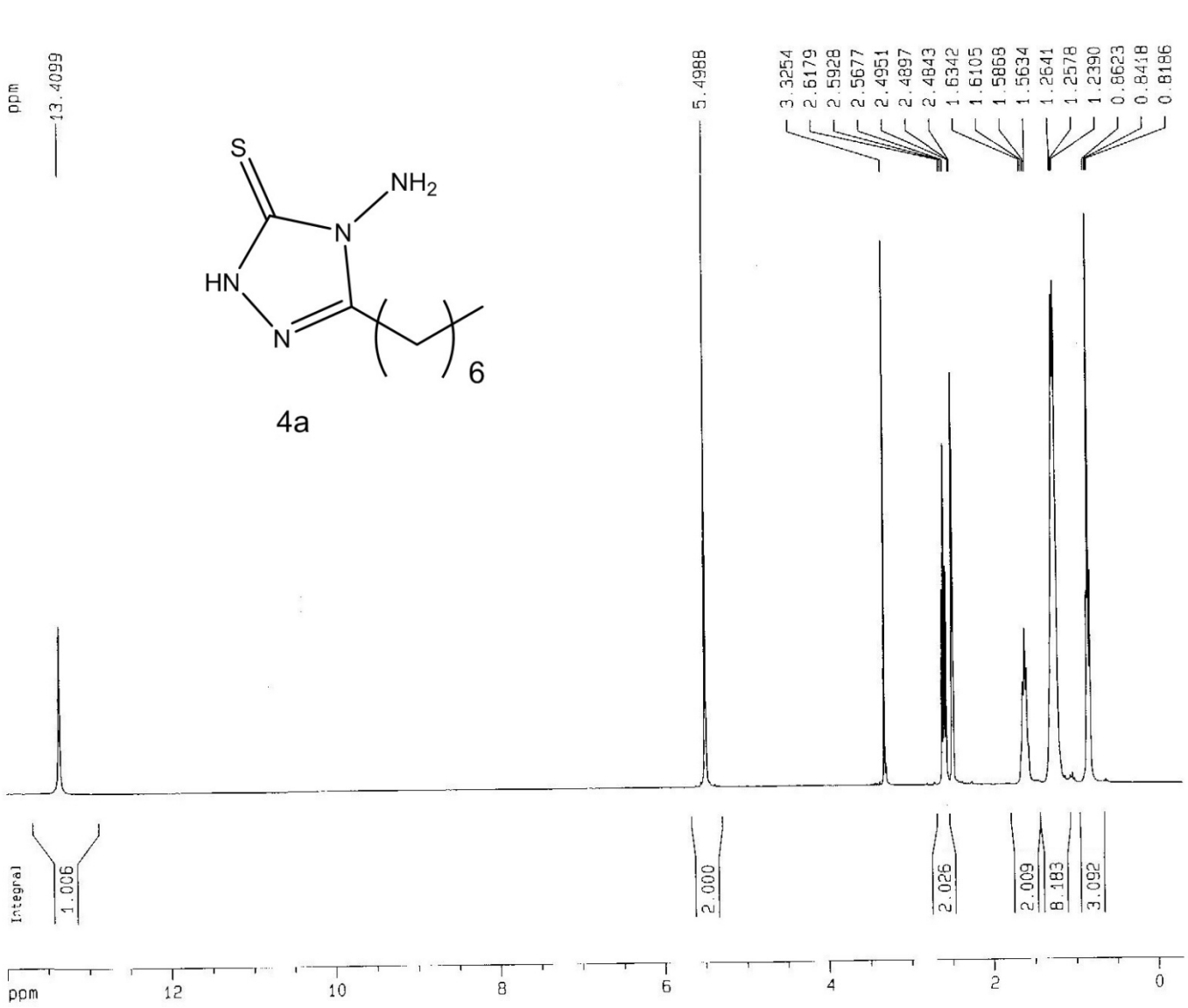

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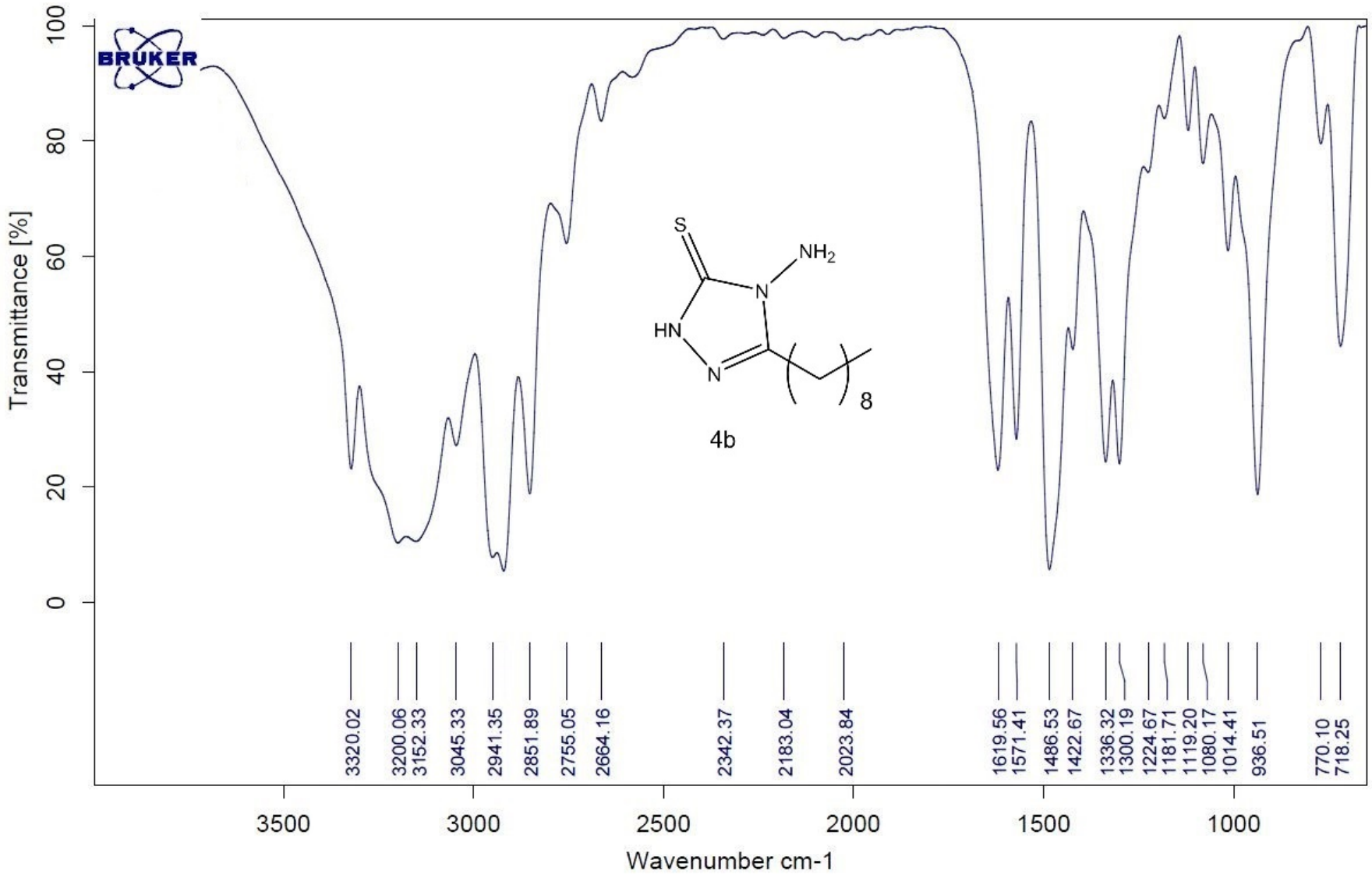
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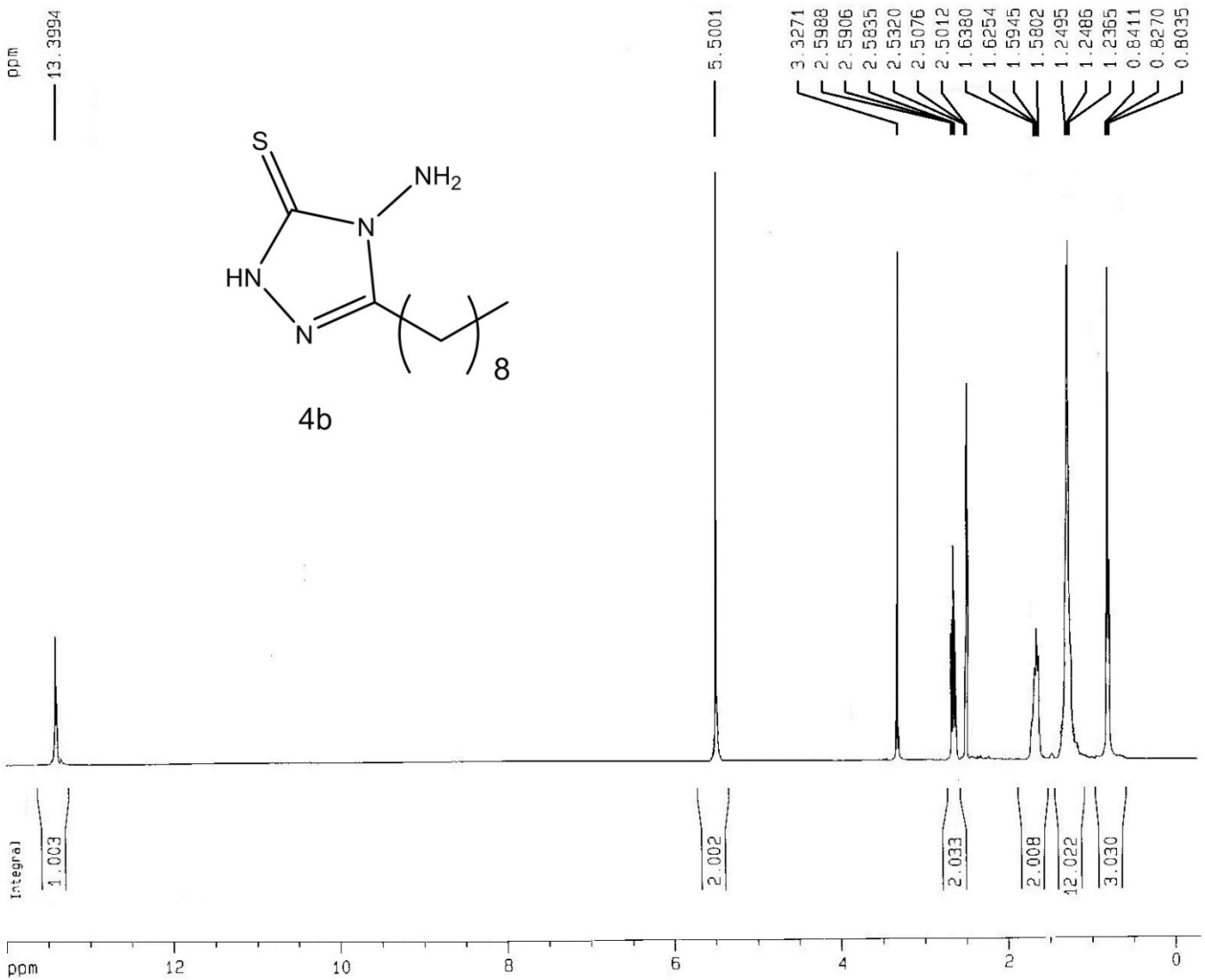

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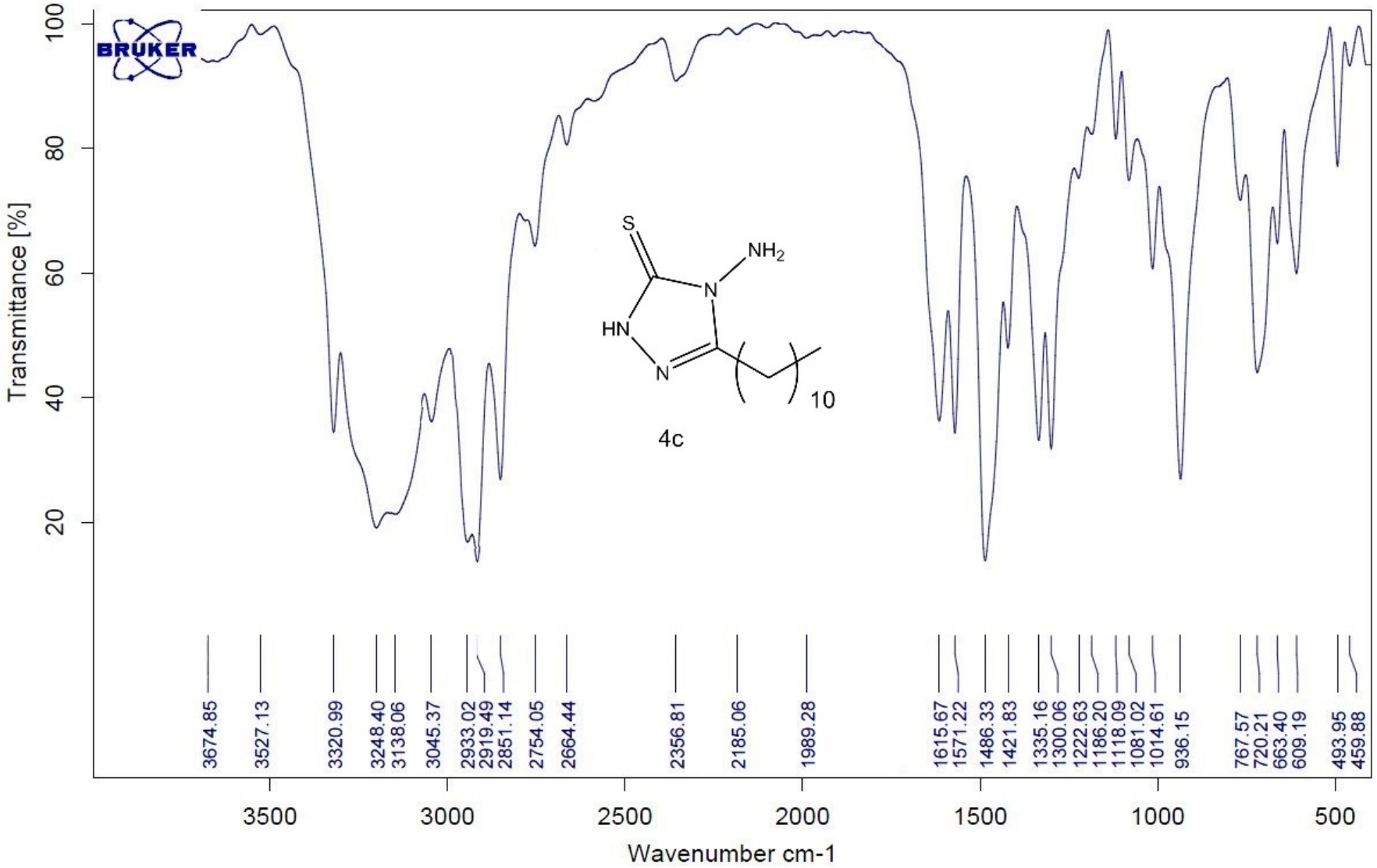
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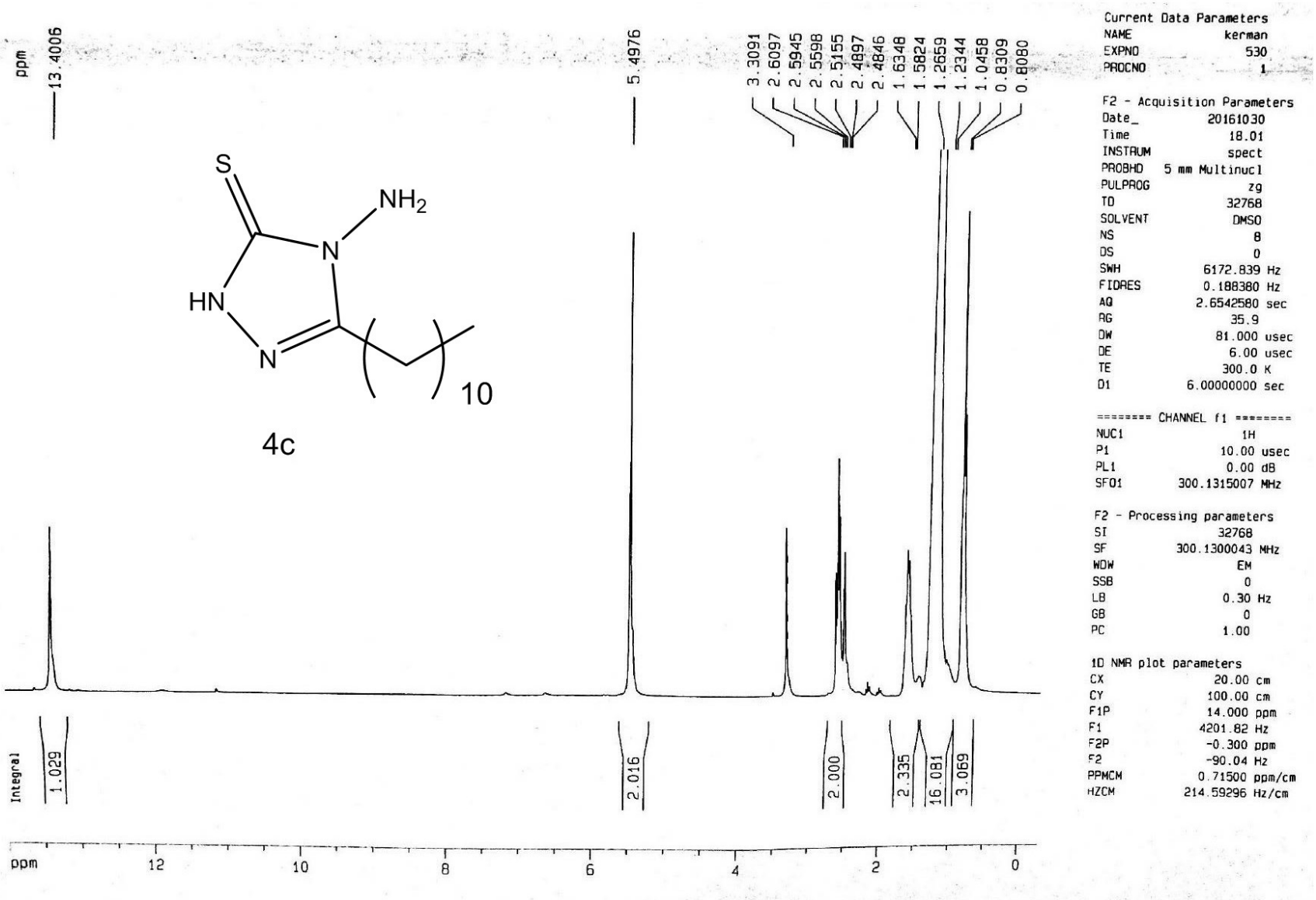

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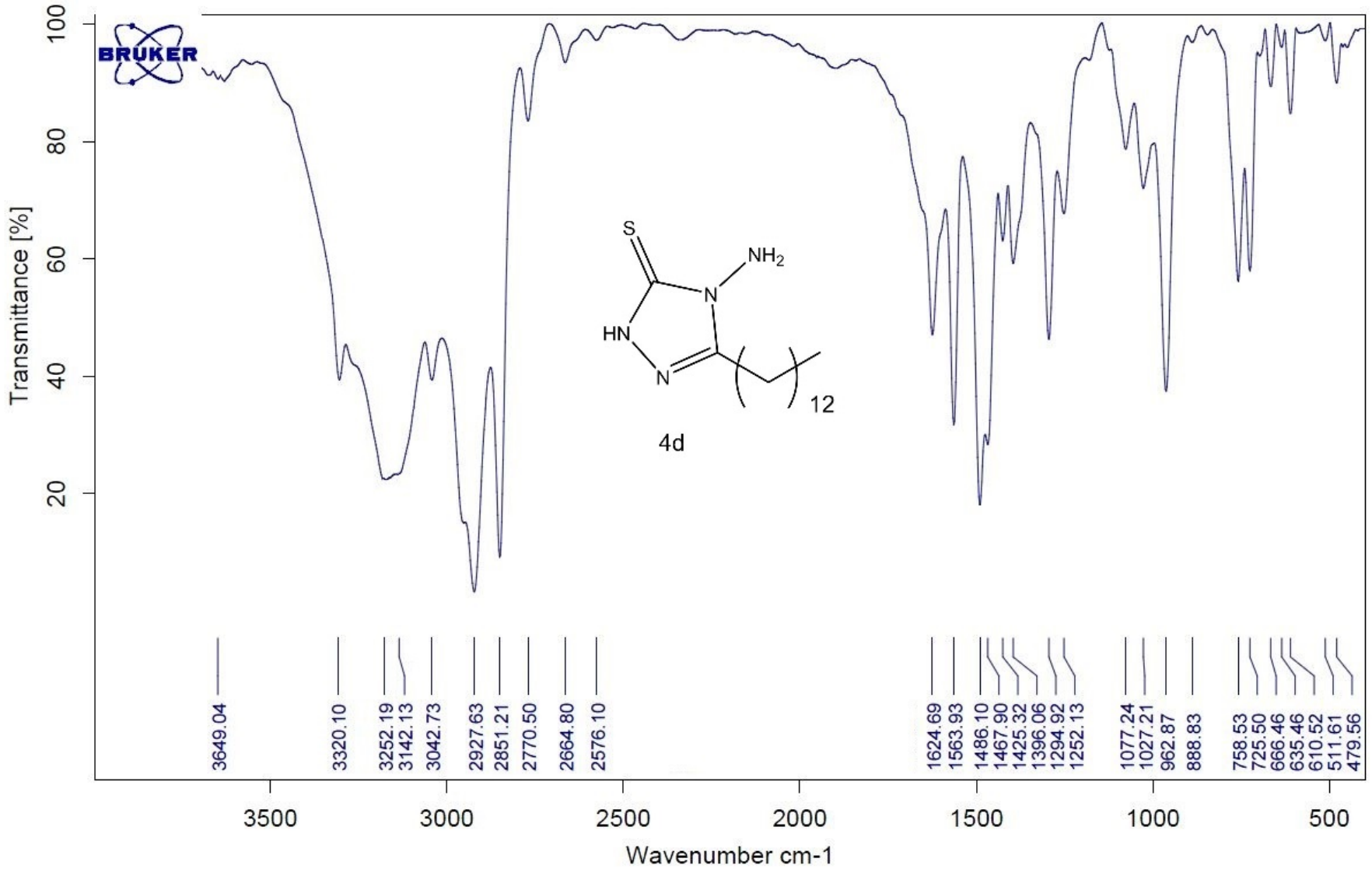
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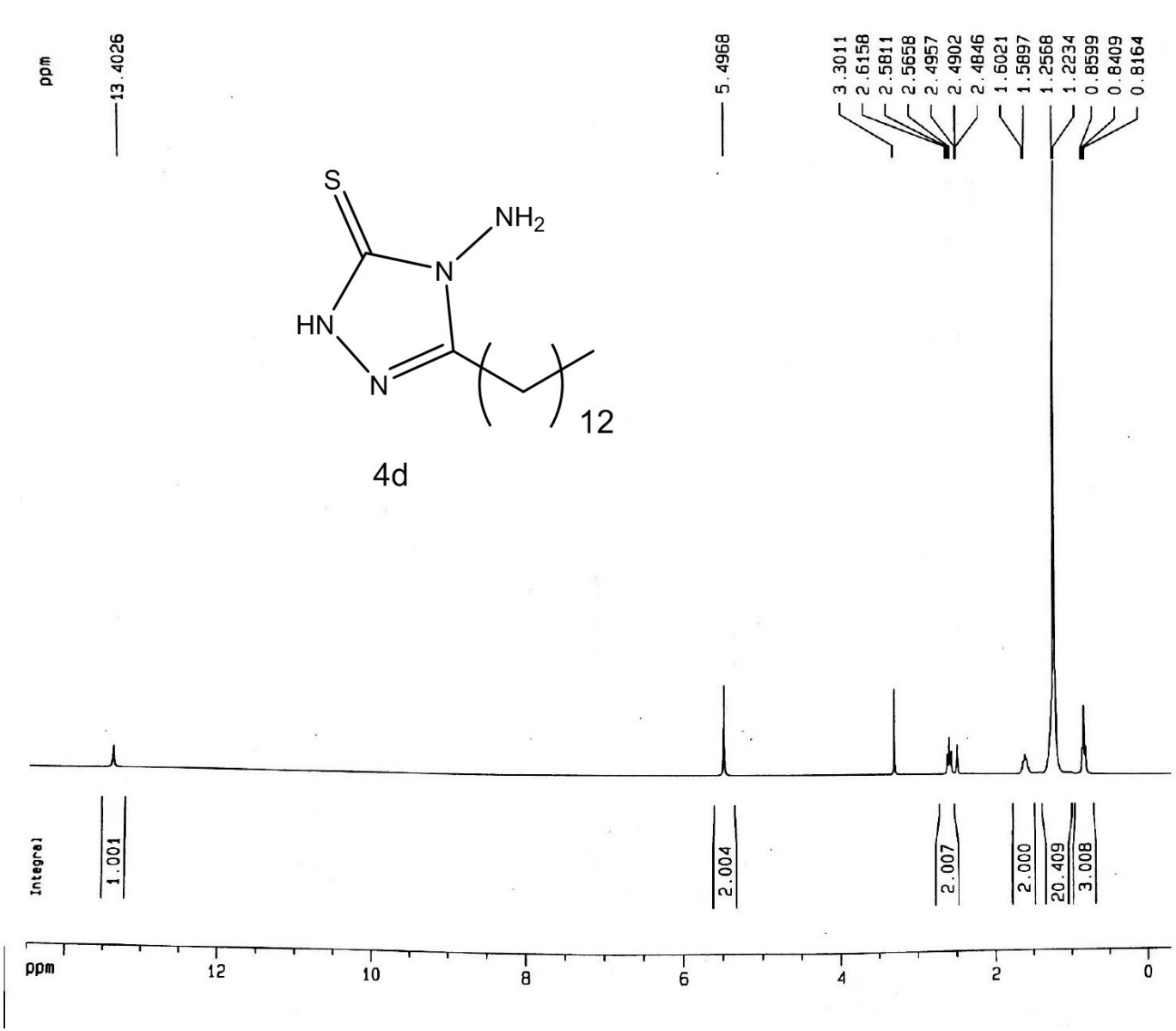
888.83
758.53
725.50
666.46
635.46
610.52
511.61
479.56
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1



Current Data Parameters

NAME	kerman
EXPNO	508
PROCNO	1

F2 - Acquisition Parameters

Date_	20160726
Time	14.12
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	101.6
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	6.00000000 sec

======== CHANNEL f1 ========

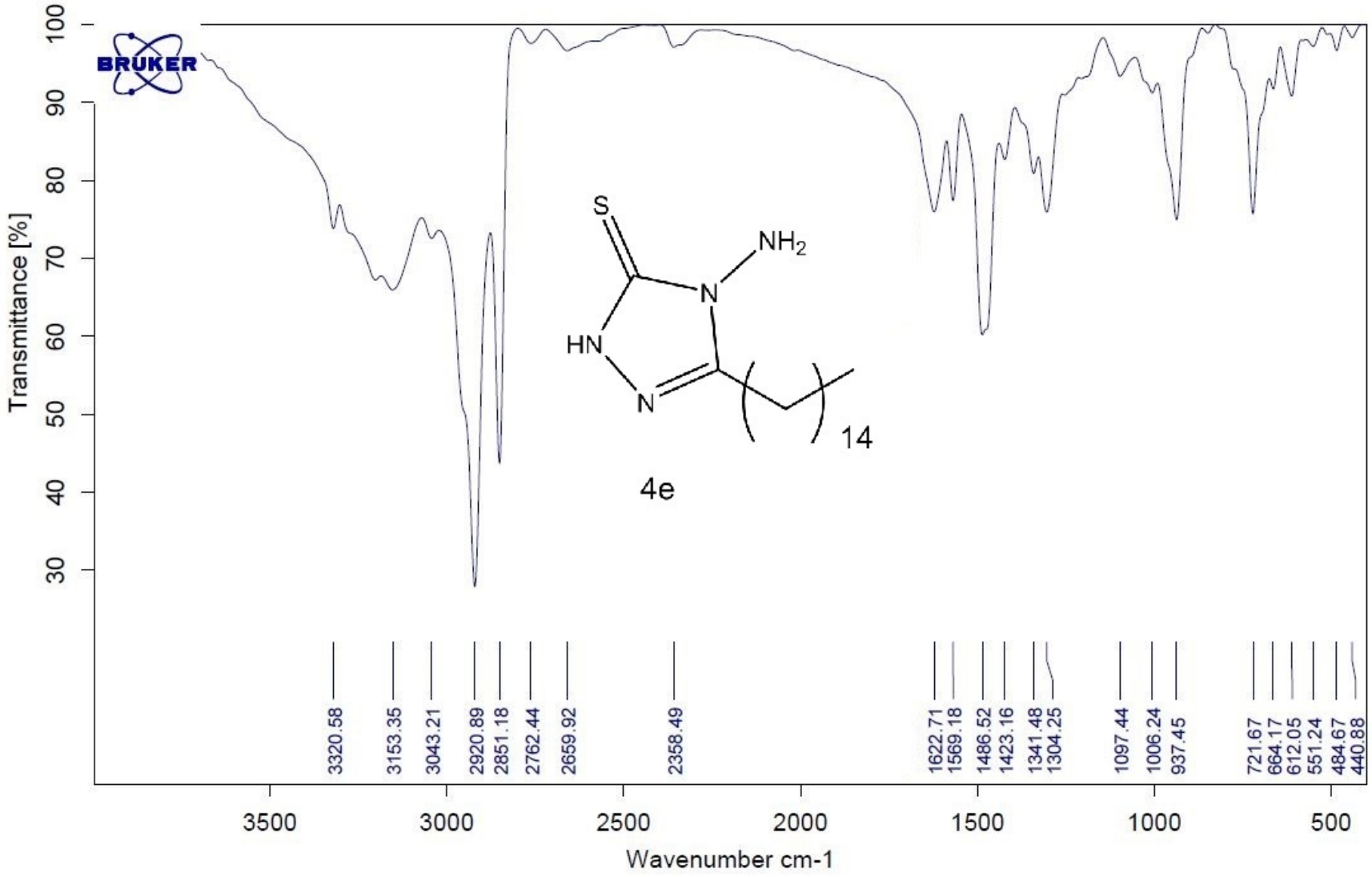
NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SFO1	300.1315007 MHz

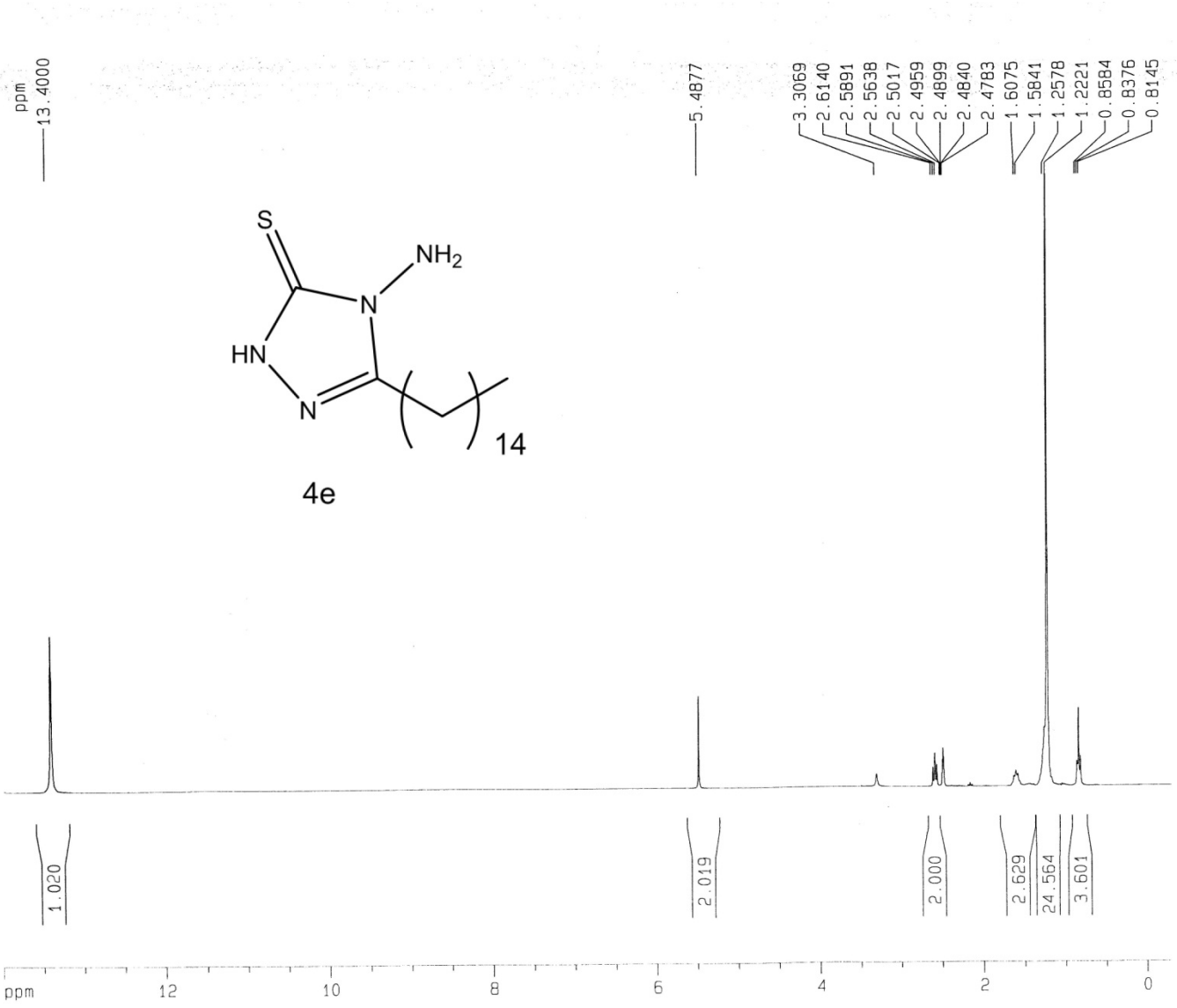
F2 - Processing parameters

SI	32768
SF	300.1300038 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	14.500 ppm
F1	4351.89 Hz
F2P	-0.300 ppm
F2	-90.04 Hz
PPMCM	0.74000 ppm/cm
HZCM	222.09619 Hz/cm


Transmittance [%]
100
90
80
70
60
50
40
30
3320.58
3153.35
3043.21
2920.89
2851.18
2762.44
2659.92
2358.49
1622.71
1569.18
1486.52
1423.16
1341.48
1304.25
1097.44
1006.24
937.45
721.67
664.17
612.05
551.24
484.67
440.88
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
S
NH2
N
HN
N
14
4e



Current Data Parameters

NAME	kerman
EXPNO	528
PROCNO	1

F2 - Acquisition Parameters

Date_	20170531
Time	17.49
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	8
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	57
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	6.00000000 sec

======== CHANNEL f1 ========

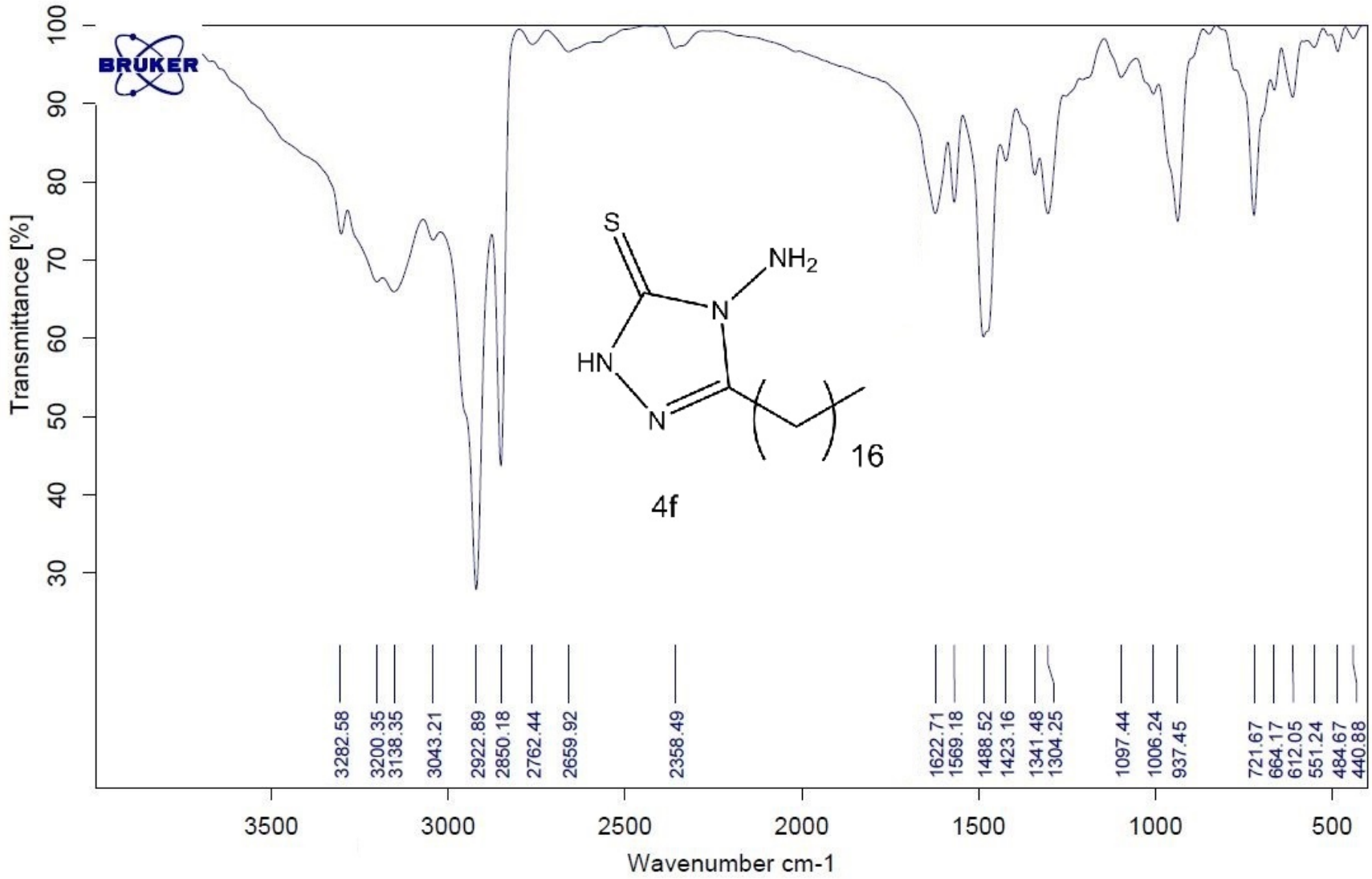
NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SFO1	300.1315007 MHz

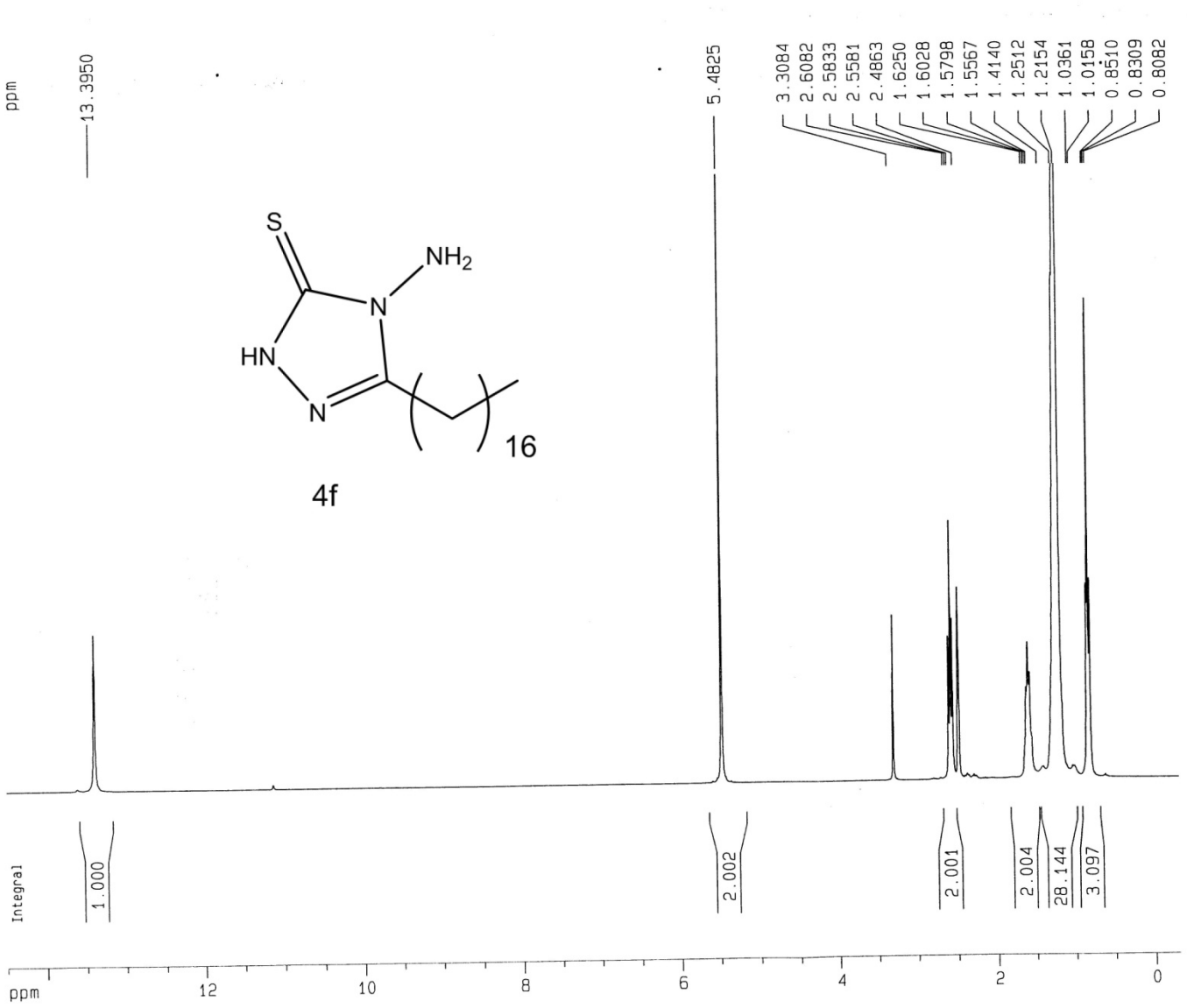
F2 - Processing parameters

SI	32768
SF	300.1300040 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	14.000 ppm
F1	4201.82 Hz
F2P	-0.300 ppm
F2	-90.04 Hz
PPMCM	0.71500 ppm/cm
HZCM	214.59296 Hz/cm


BRUKER
Transmittance [%]
100
90
80
70
60
50
40
30
S
NH2
N
HN
N
16
4f
3282.58
3200.35
3138.35
3043.21
2922.89
2850.18
2762.44
2659.92
2358.49
1622.71
1569.18
1488.52
1423.16
1341.48
1304.25
1097.44
1006.24
937.45
721.67
664.17
612.05
551.24
484.67
440.88
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1



Current Data Parameters

NAME	kerman
EXPNO	512
PROCNO	1

F2 - Acquisition Parameters

Date_	20170419
Time	15.33
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	64
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	6.00000000 sec

======== CHANNEL f1 ========

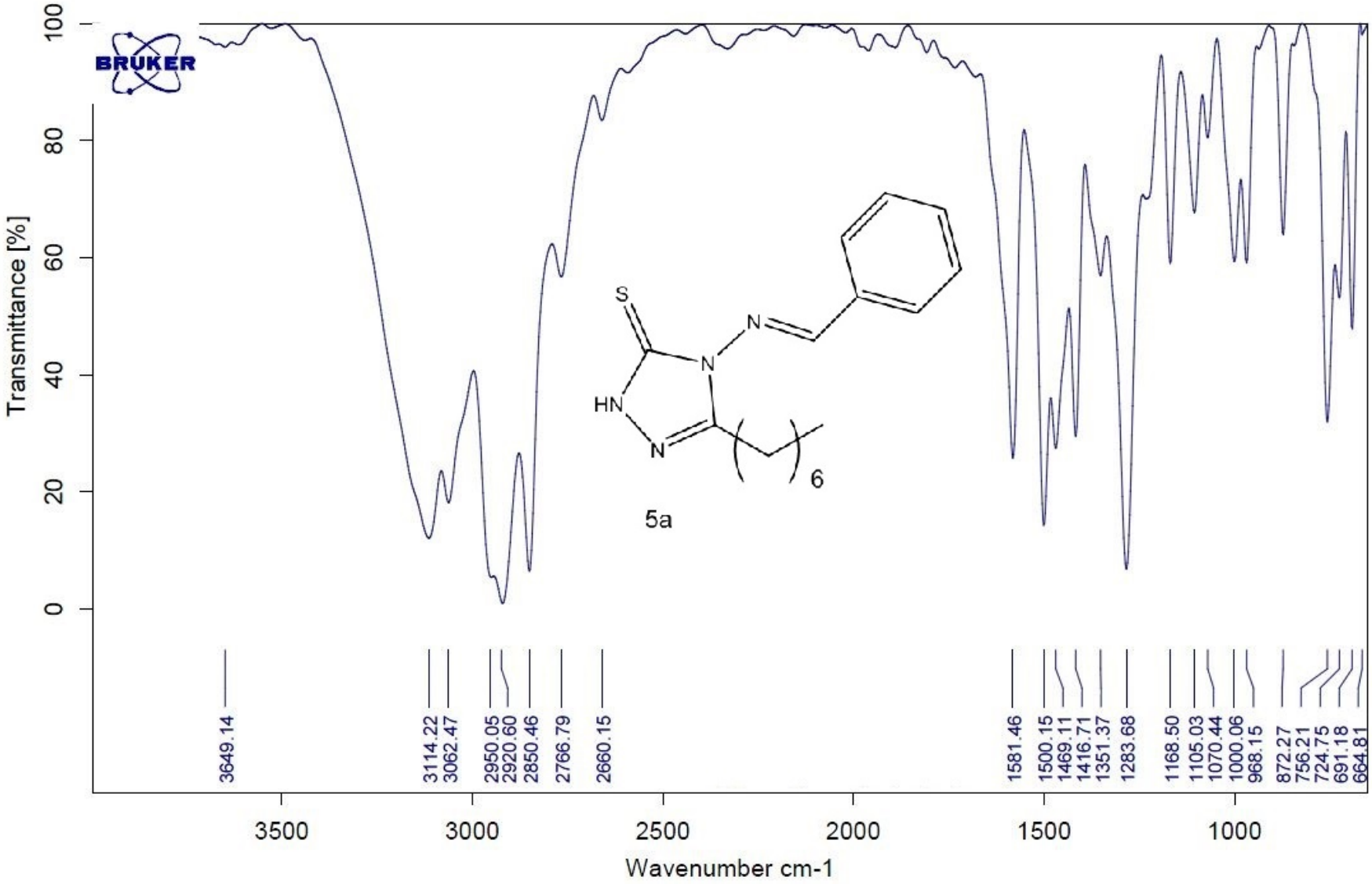
NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SF01	300.1315007 MHz

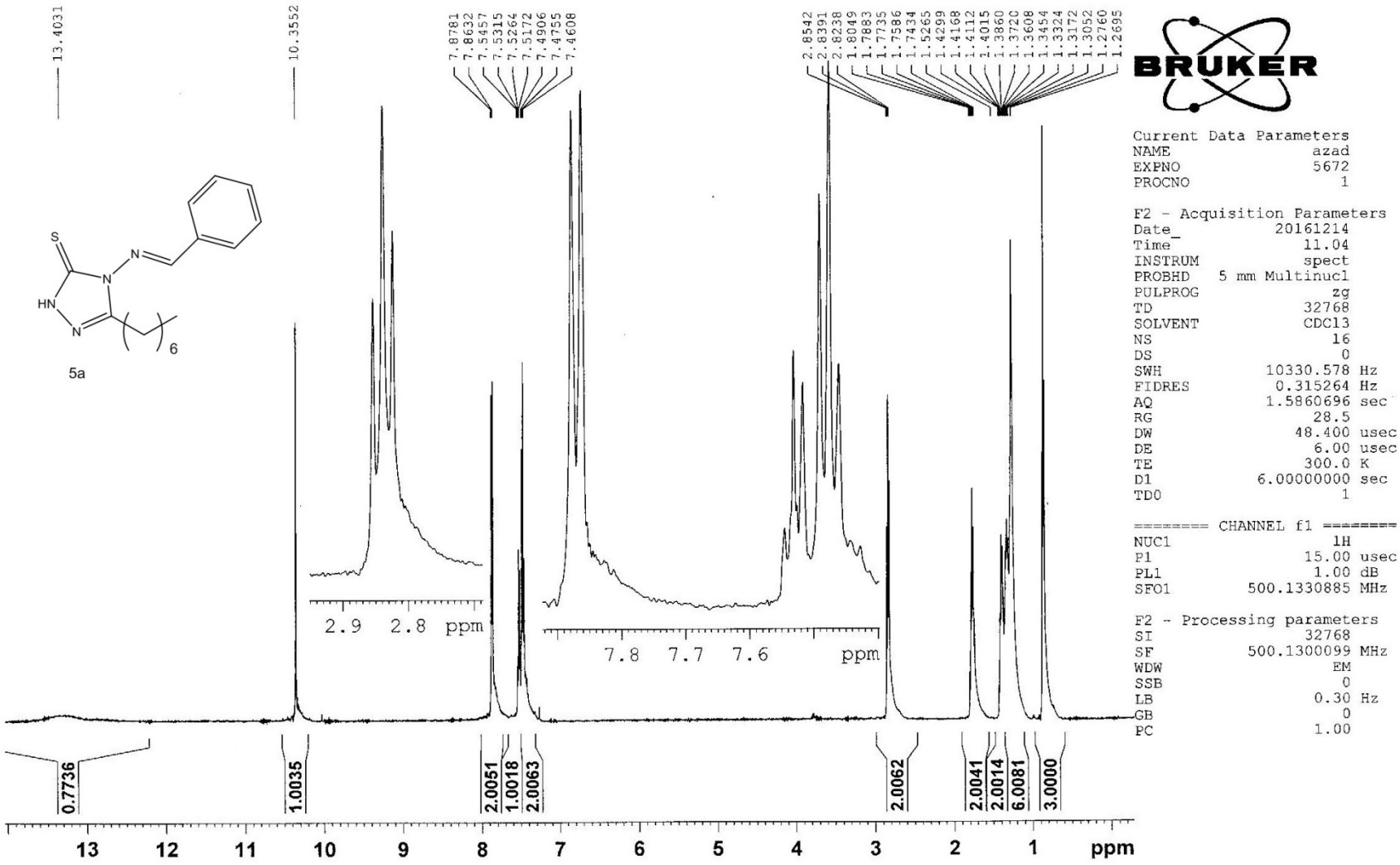
F2 - Processing parameters

SI	32768
SF	300.1300062 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	90.00 cm
F1P	14.500 ppm
F1	4351.89 Hz
F2P	-0.300 ppm
F2	-90.04 Hz
PPMCM	0.74000 ppm/cm
HZCM	222.09619 Hz/cm


Transmittance [%]
100
80
60
40
20
0
3649.14
3114.22
3062.47
2950.05
2920.60
2850.46
2766.79
2660.15
1581.46
1500.15
1469.11
1416.71
1351.37
1283.68
1168.50
1105.03
1070.44
1000.06
968.15
872.27
756.21
724.75
691.18
664.81
3500
3000
2500
2000
1500
1000
Wavenumber cm-1
S
N
N
HN
N
6
5a



BRUKER

Current Data Parameters
NAME azad
EXPNO 5672
PROCNO 1

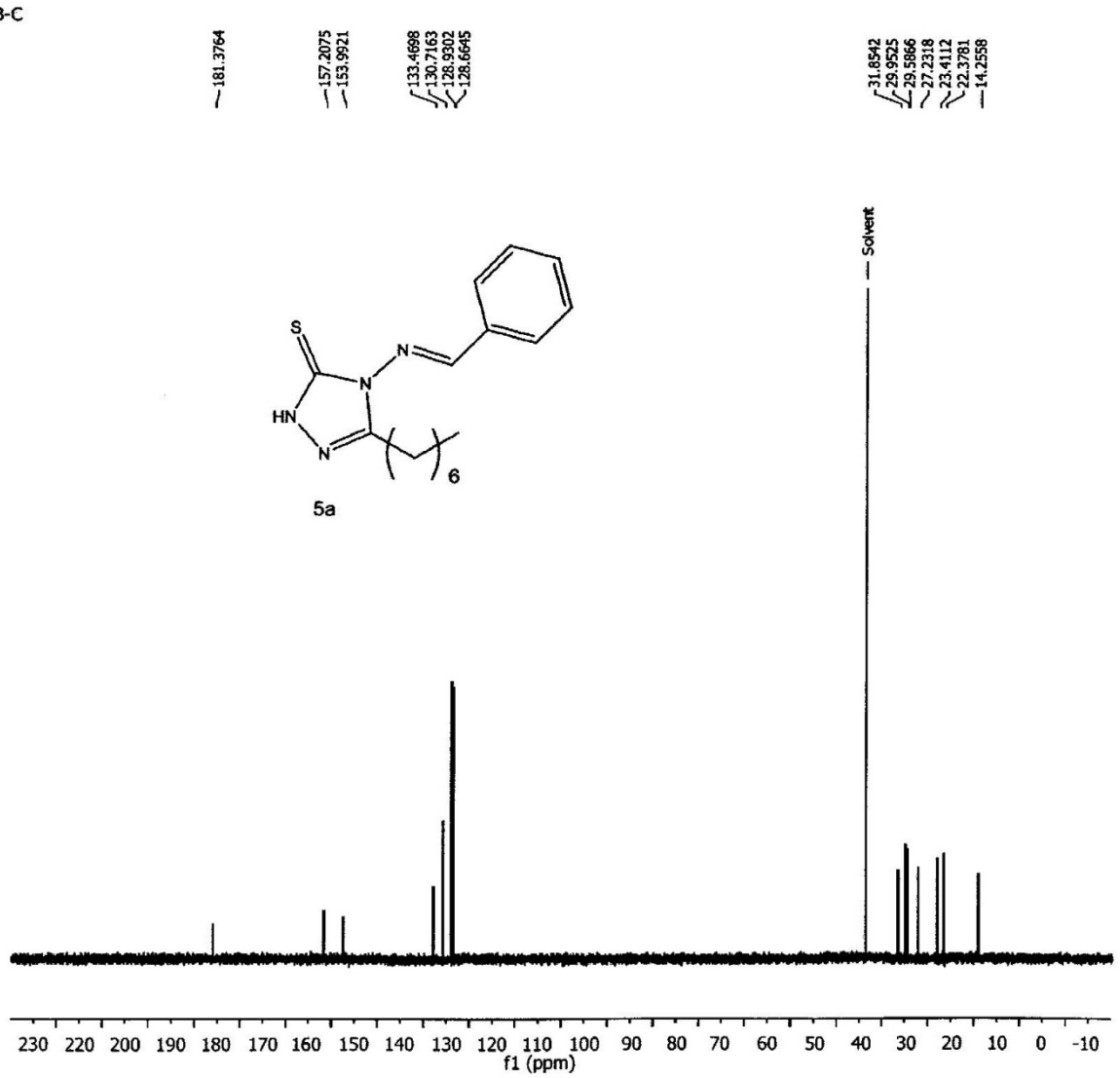
F2 - Acquisition Parameters
Date_ 20161214
Time 11.04
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 28.5
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 6.00000000 sec
TD0 1

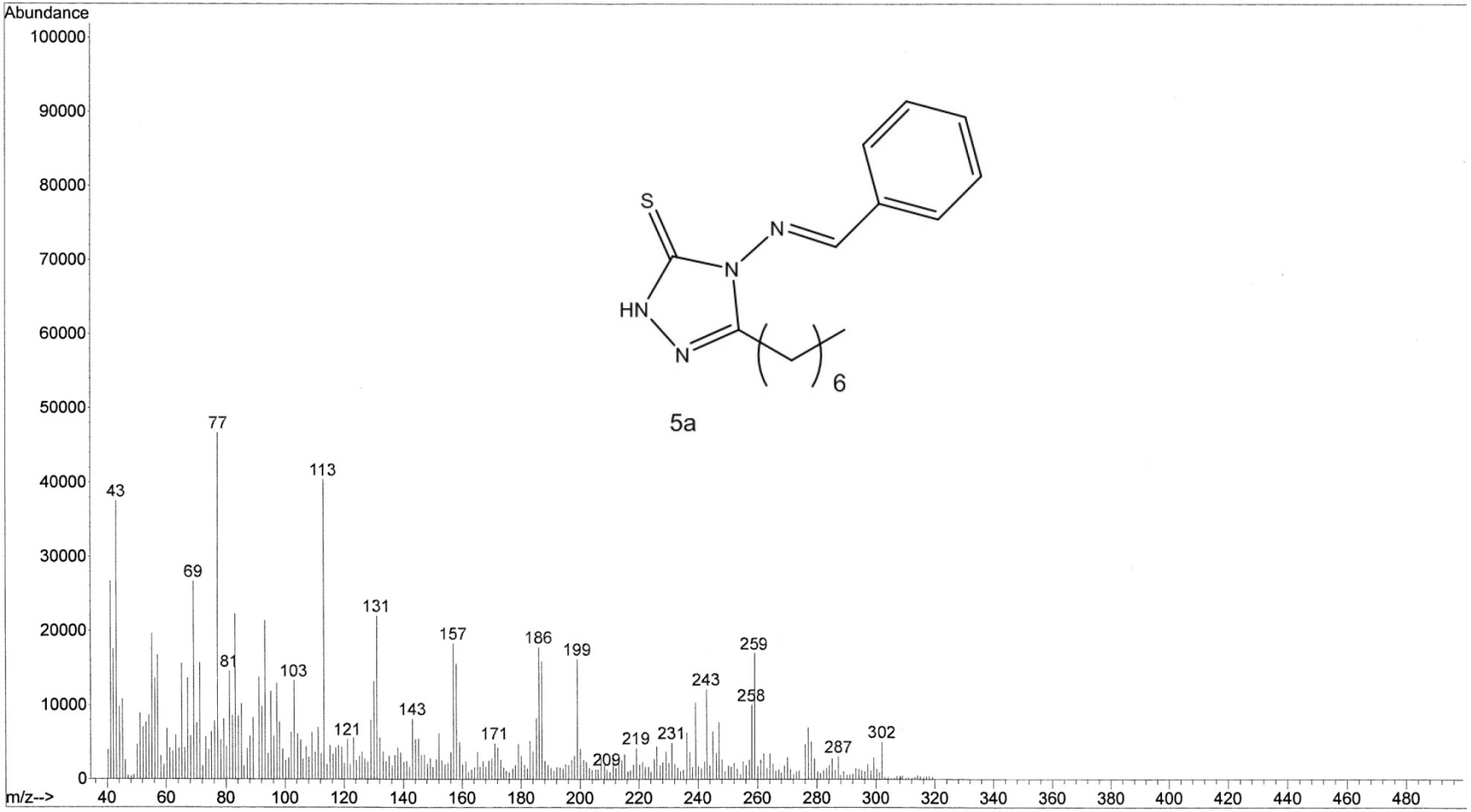
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.00 dB
SFO1 500.1330885 MHz

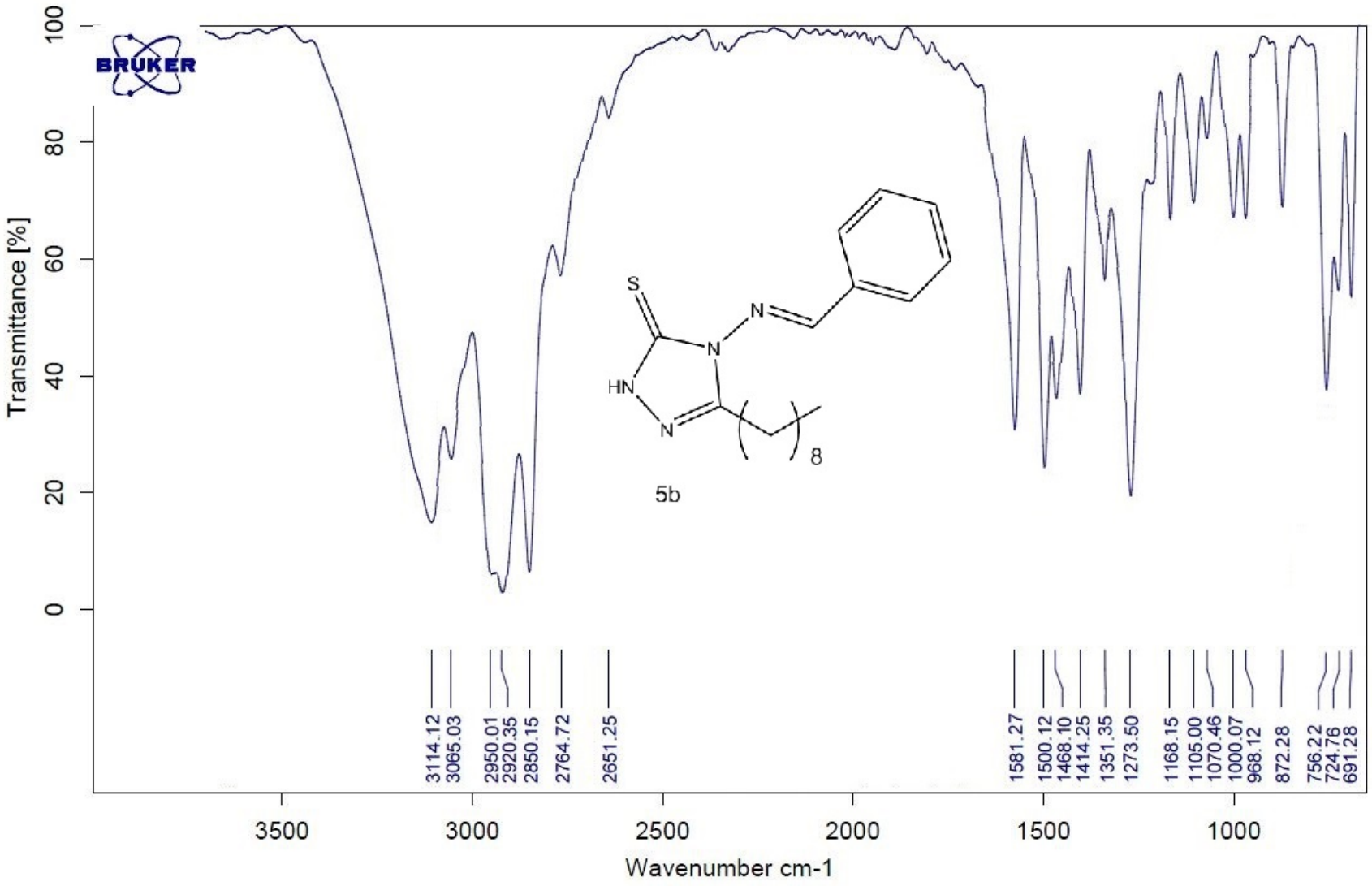
F2 - Processing parameters
SI 32768
SF 500.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

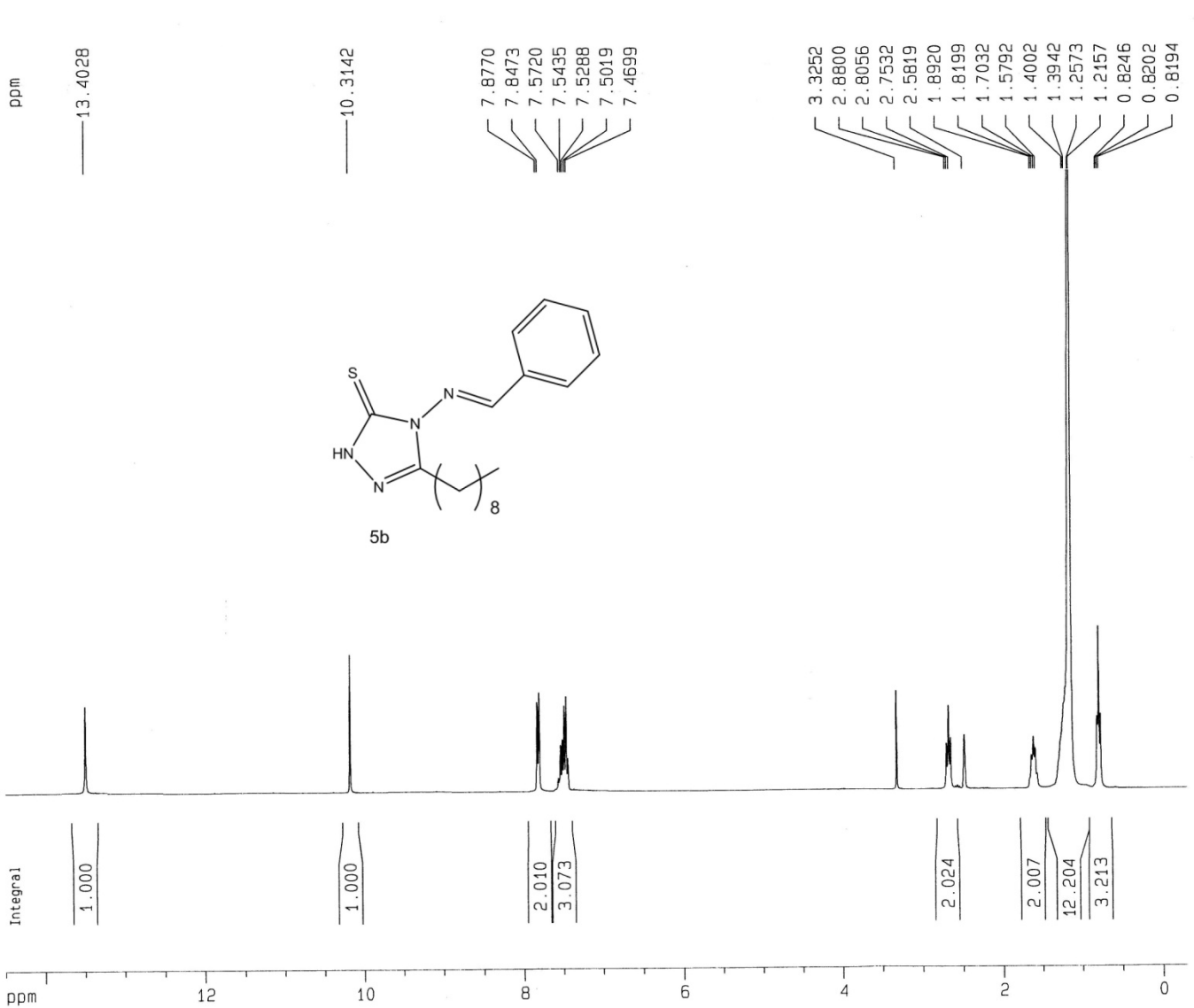
OTEB-C

	Parameter	Value
1	Data File Name	D:/ NMR/ 1397/ 97-10/ 97-10-11/ Kerman/ OTEB-C.fid/ fid
2	Title	OTEB-C
3	Comment	
4	Origin	Varian
5	Owner	
6	Site	
7	Instrument	inova
8	Author	
9	Solvent	dmso
10	Temperature	25.0
11	Pulse Sequence	s2pul
12	Experiment	1D
13	Probe	ID
14	Number of Scans	1024
15	Receiver Gain	60
16	Relaxation Delay	1.0000
17	Pulse Width	7.5000
18	Presaturation Frequency	
19	Acquisition Time	1.0433
20	Acquisition Date	2019-01-01T14:41:56
21	Modification Date	2019-01-01T14:46:10
22	Class	
23	Spectrometer Frequency	125.67
24	Spectral Width	31409.5
25	Lowest Frequency	-1833.0
26	Nucleus	13C
27	Acquired Size	32768
28	Spectral Size	65536




Abundance
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
m/z-->
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
43
77
69
81
103
113
121
131
143
157
171
186
199
209
219
231
243
258
259
287
302
S
N
N
HN
N
6
5a


BRUKER
Transmittance [%]
100
80
60
40
20
0
3500
3000
2500
2000
1500
1000
Wavenumber cm-1
3114.12
3065.03
2950.01
2920.35
2850.15
2764.72
2651.25
1581.27
1500.12
1468.10
1414.25
1351.35
1273.50
1168.15
1105.00
1070.46
1000.07
968.12
872.28
756.22
724.76
691.28
S
N
N
HN
N
8
5b



Current Data Parameters

NAME	kerman
EXPNO	515
PROCNO	1

F2 - Acquisition Parameters

Date_	20170419
Time	15.30
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	12
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	60.0
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	6.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SF01	300.1315007 MHz

F2 - Processing parameters

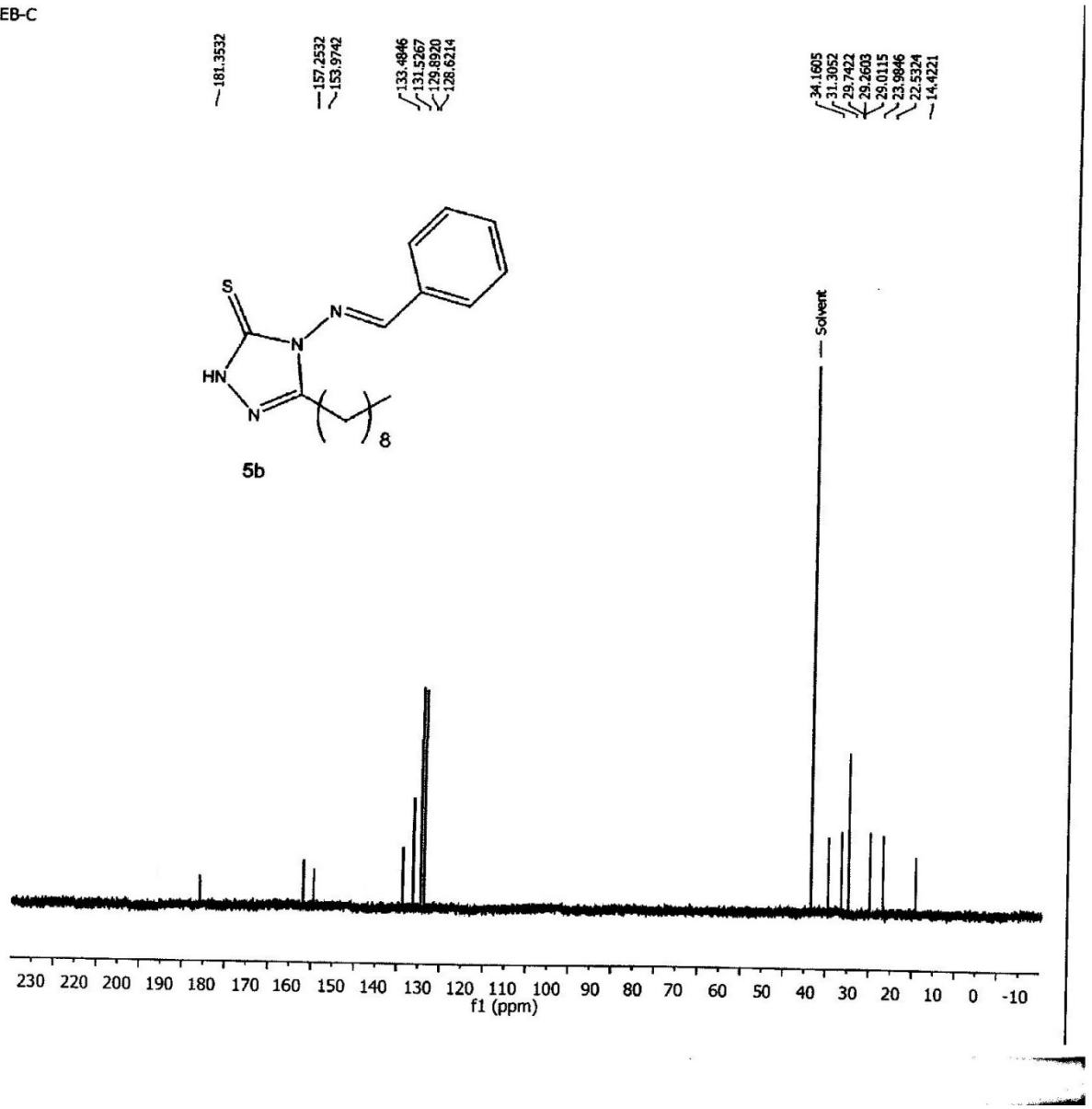
SI	32768
SF	300.1300062 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

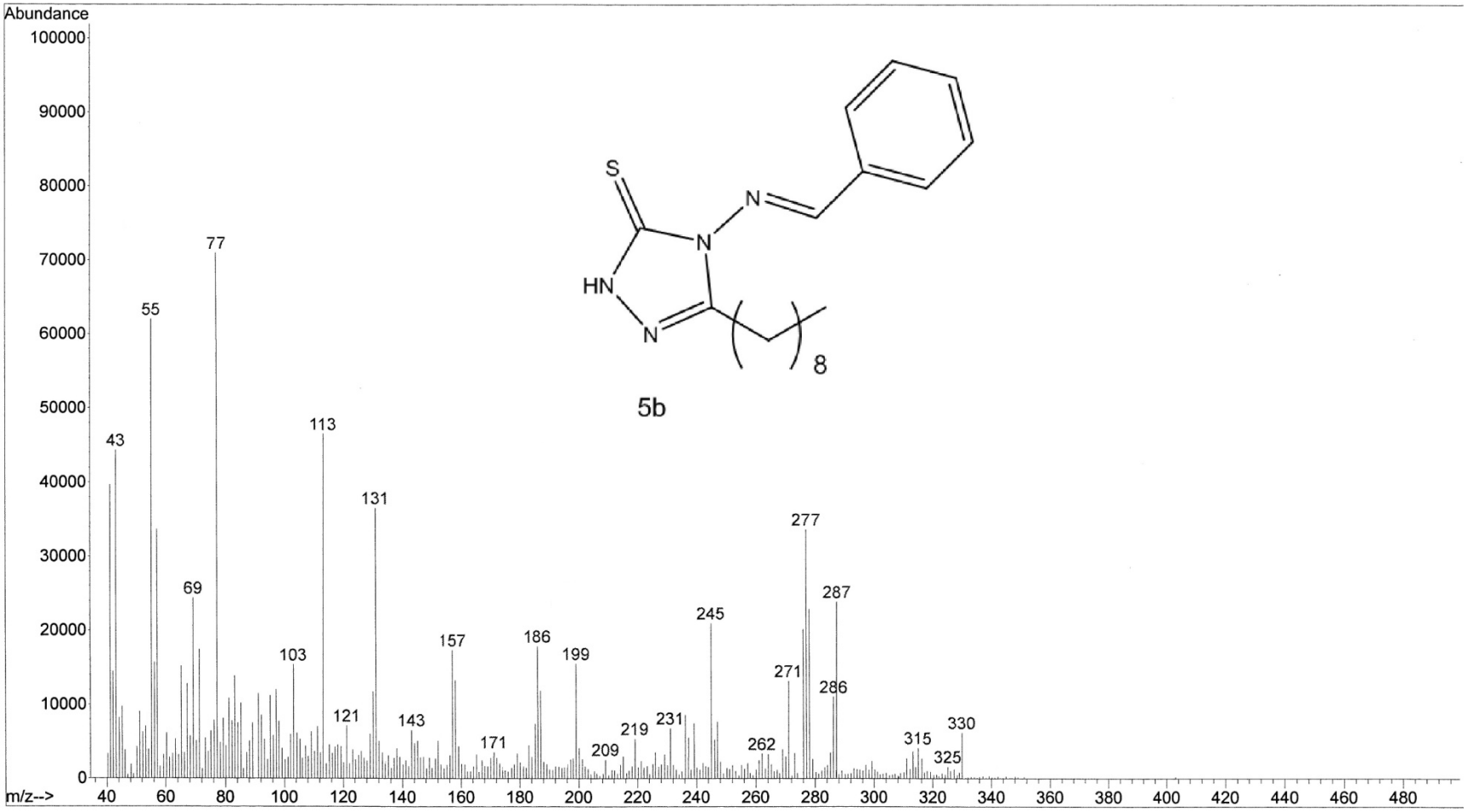
1D NMR plot parameters

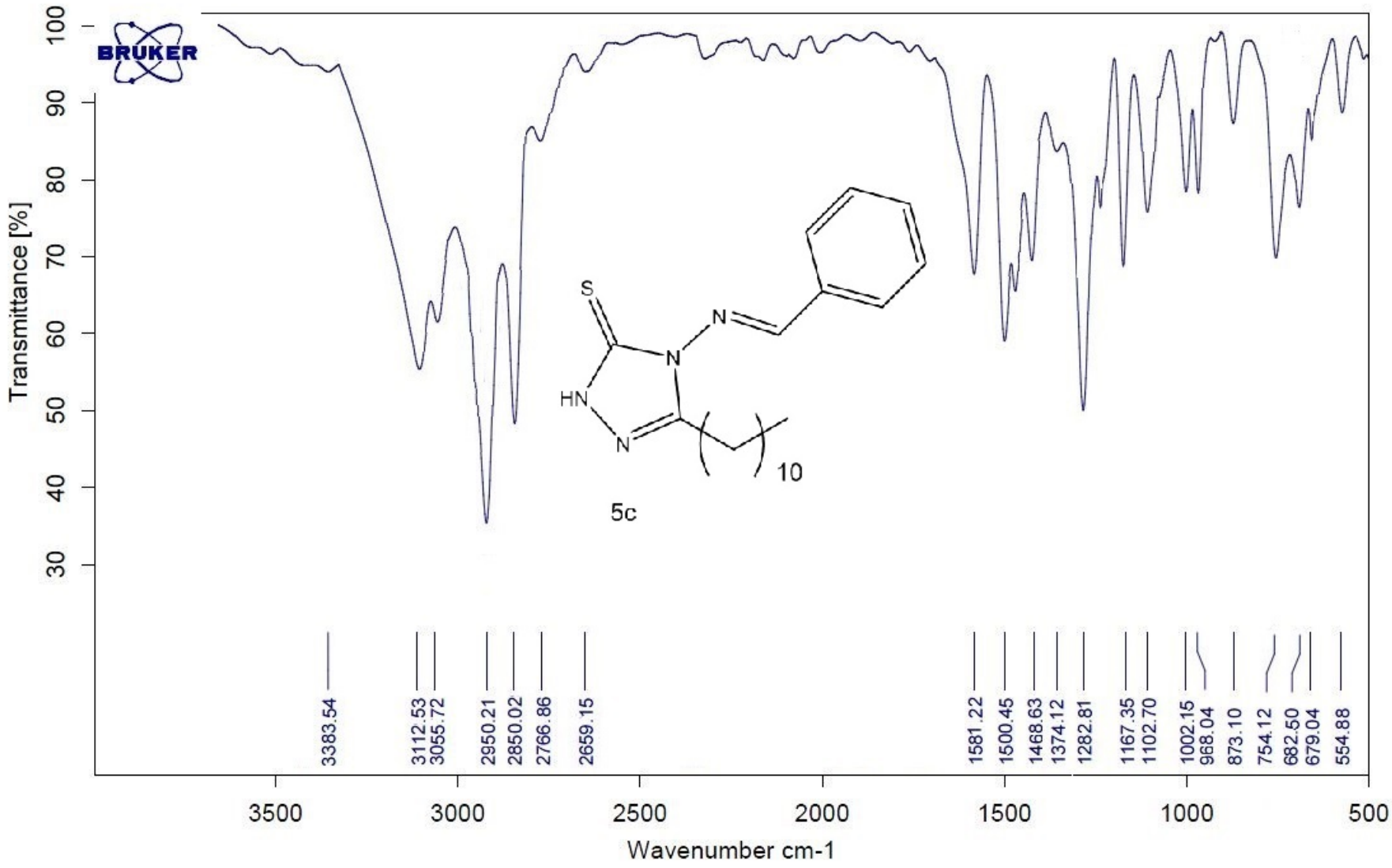
CX	20.00 cm
CY	12.50 cm
F1P	14.500 ppm
F1	4351.89 Hz
F2P	-0.300 ppm
F2	-90.04 Hz
PPMCM	0.71400 ppm/cm
HZCM	222.09619 Hz/cm

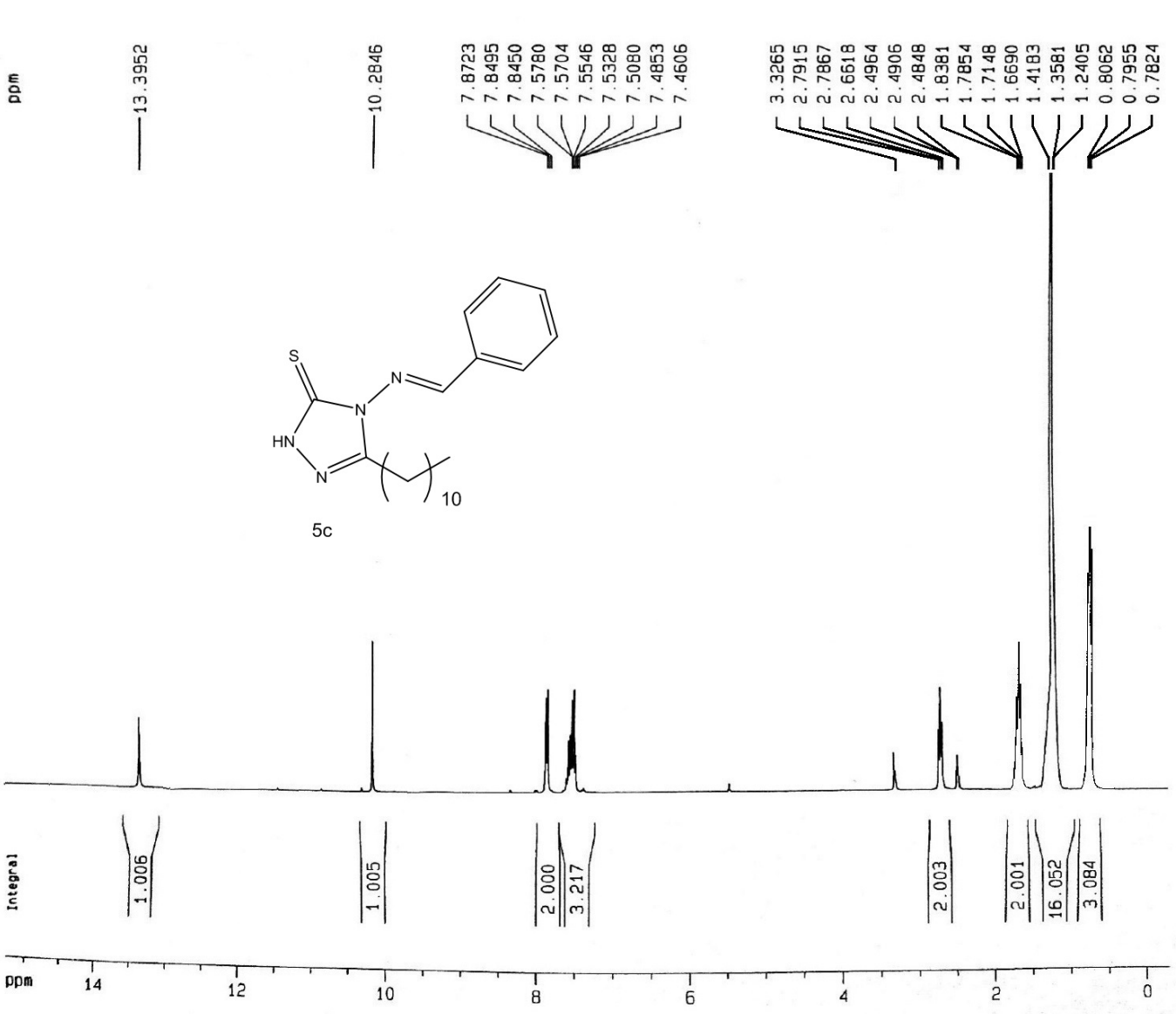
DTEB-C

	Parameter	Value
1	Data File Name	D:/ NMR/ 1397/ 97-10/ 97-10-11/ Kerman/ DTEB-C.fid/ fid
2	Title	DTEB-C
3	Comment	
4	Origin	Varian
5	Owner	
6	Site	
7	Instrument	inova
8	Author	
9	Solvent	dmso
10	Temperature	25.0
11	Pulse Sequence	s2pul
12	Experiment	1D
13	Probe	ID
14	Number of Scans	1024
15	Receiver Gain	60
16	Relaxation Delay	1.0000
17	Pulse Width	7.5000
18	Presaturation Frequency	
19	Acquisition Time	1.0548
20	Acquisition Date	2019-01-01T13:52:12
21	Modification Date	2019-01-01T13:56:25
22	Class	
23	Spectrometer Frequency	125.67
24	Spectral Width	31451.2
25	Lowest Frequency	-1833.0
26	Nucleus	13C
27	Acquired Size	32768
28	Spectral Size	65536




Abundance
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
m/z-->
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
43
55
69
77
103
113
121
131
143
157
171
186
199
209
219
231
245
262
271
277
286
287
315
325
330
S
N
N
HN
N
8
5b


BRUKER
Transmittance [%]
100
90
80
70
60
50
40
30
3383.54
3112.53
3055.72
2950.21
2850.02
2766.86
2659.15
1581.22
1500.45
1468.63
1374.12
1282.81
1167.35
1102.70
1002.15
968.04
873.10
754.12
682.50
679.04
554.88
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
S
N
N
HN
N
10
5c



Current Data Parameters

NAME	kerman
EXPNO	510
PROCNO	1

F2 - Acquisition Parameters

Date_	20170531
Time	18.11
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	50.8
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	6.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SFO1	300.1315007 MHz

F2 - Processing parameters

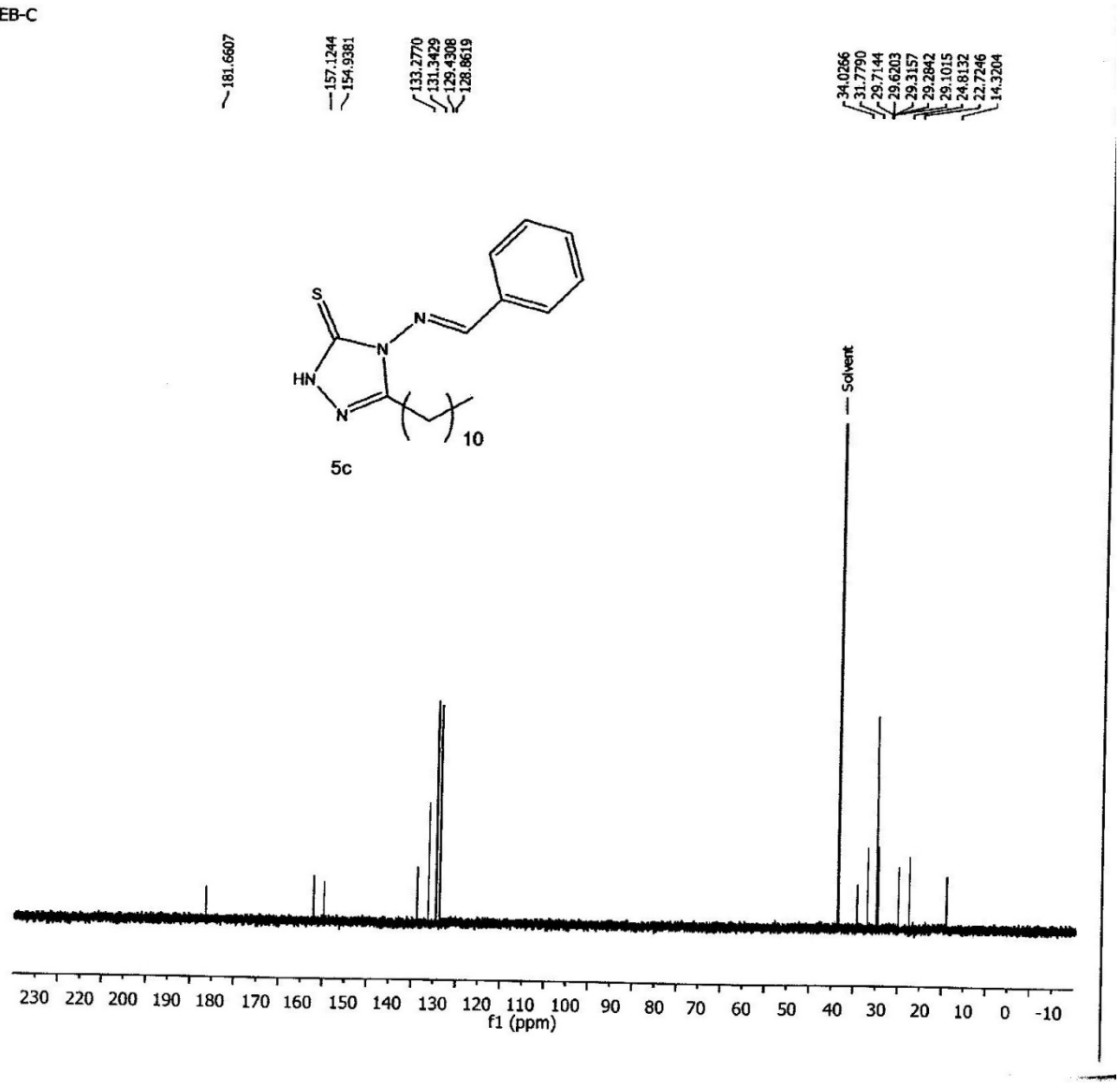
SI	32768
SF	300.1300039 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

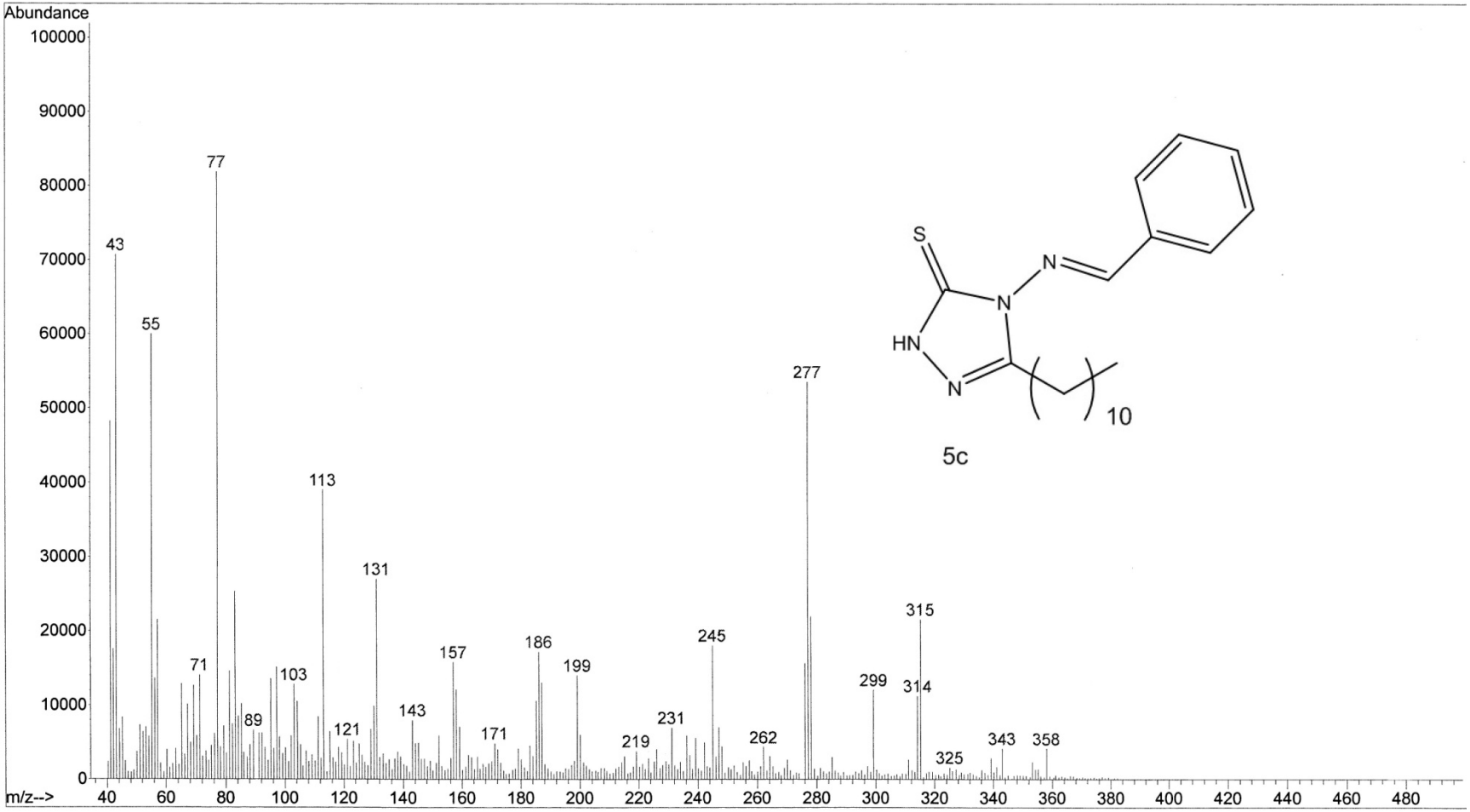
1D NMR plot parameters

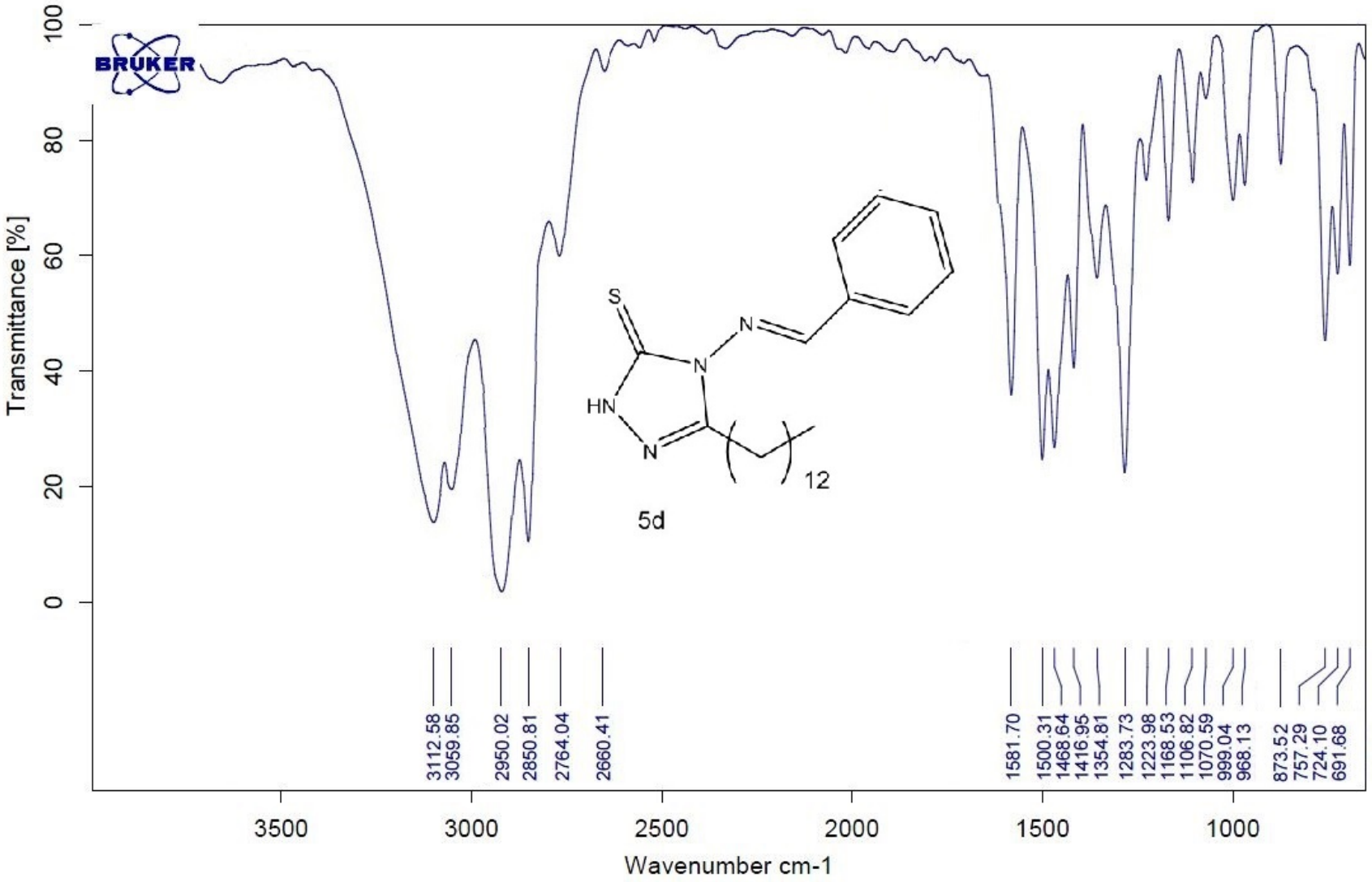
CX	20.00 cm
CY	12.50 cm
F1P	15.272 ppm
F1	4583.50 Hz
F2P	-0.300 ppm
F2	-90.04 Hz
PPMCM	0.77859 ppm/cm
HZCM	233.67674 Hz/cm

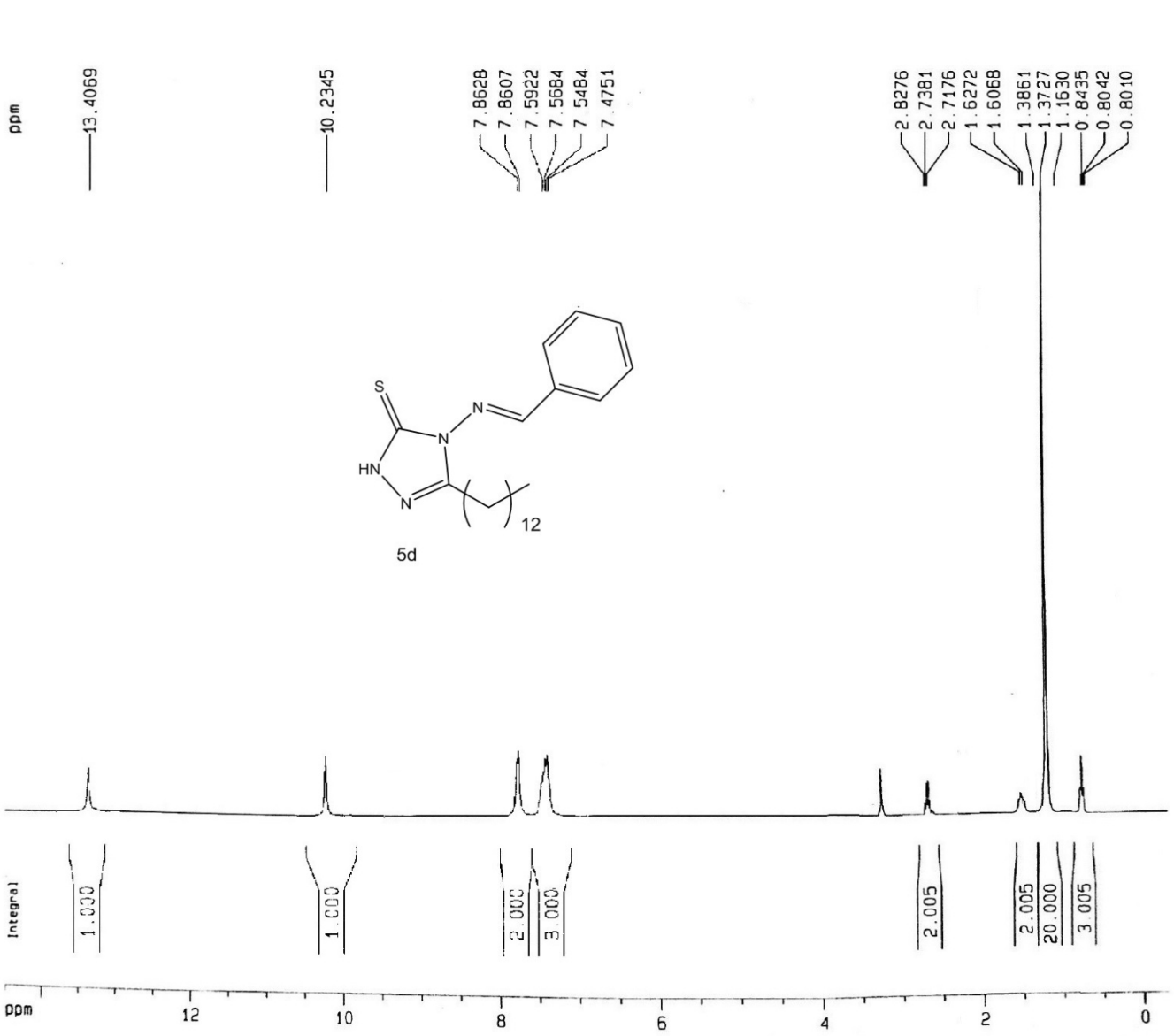
LTEB-C

Parameter	Value
1 Data File Name	D:/ NMR/ 1397/ 97-10/ 97-10-11/ Kerman/ LTEB-C.fid/ fid
2 Title	LTEB-C
3 Comment	
4 Origin	Varian
5 Owner	
6 Site	
7 Instrument	inova
8 Author	
9 Solvent	dmso
10 Temperature	25.0
11 Pulse Sequence	s2pul
12 Experiment	1D
13 Probe	ID
14 Number of Scans	1024
15 Receiver Gain	60
16 Relaxation Delay	1.0000
17 Pulse Width	7.5000
18 Presaturation Frequency	
19 Acquisition Time	1.0374
20 Acquisition Date	2019-01-01T15:14:11
21 Modification Date	2019-01-01T15:18:25
22 Class	
23 Spectrometer Frequency	125.67
24 Spectral Width	31395.1
25 Lowest Frequency	-1833.0
26 Nucleus	13C
27 Acquired Size	32768
28 Spectral Size	65536




Abundance
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
m/z-->
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
43
55
77
71
89
103
113
121
131
143
157
171
186
199
219
231
245
262
277
299
314
315
325
343
358
S
N
N
HN
N
10
5c


BRUKER
Transmittance [%]
100
80
60
40
20
0
3500
3000
2500
2000
1500
1000
Wavenumber cm-1
3112.58
3059.85
2950.02
2850.81
2764.04
2660.41
1581.70
1500.31
1468.64
1416.95
1354.81
1283.73
1223.98
1168.53
1106.82
1070.59
999.04
968.13
873.52
757.29
724.10
691.68
S
N
N
HN
N
12
5d



Current Data Parameters

NAME	kerman
EXPNO	525
PROCNO	1

F2 - Acquisition Parameters

Date_	20160818
Time	15.25
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	8
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	71.8
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	6.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SFO1	300.1315007 MHz

F2 - Processing parameters

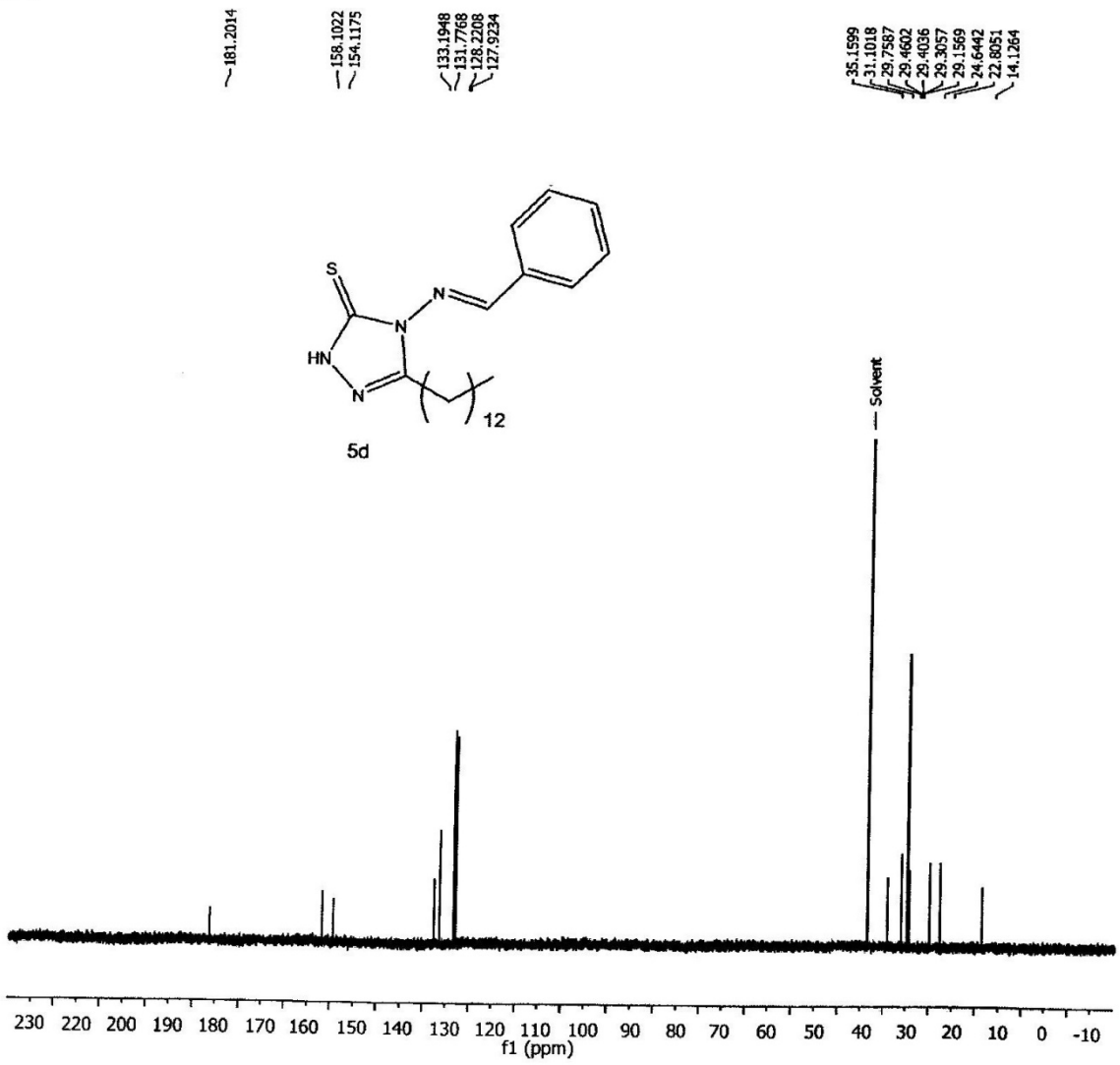
SI	32768
SF	300.1300045 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

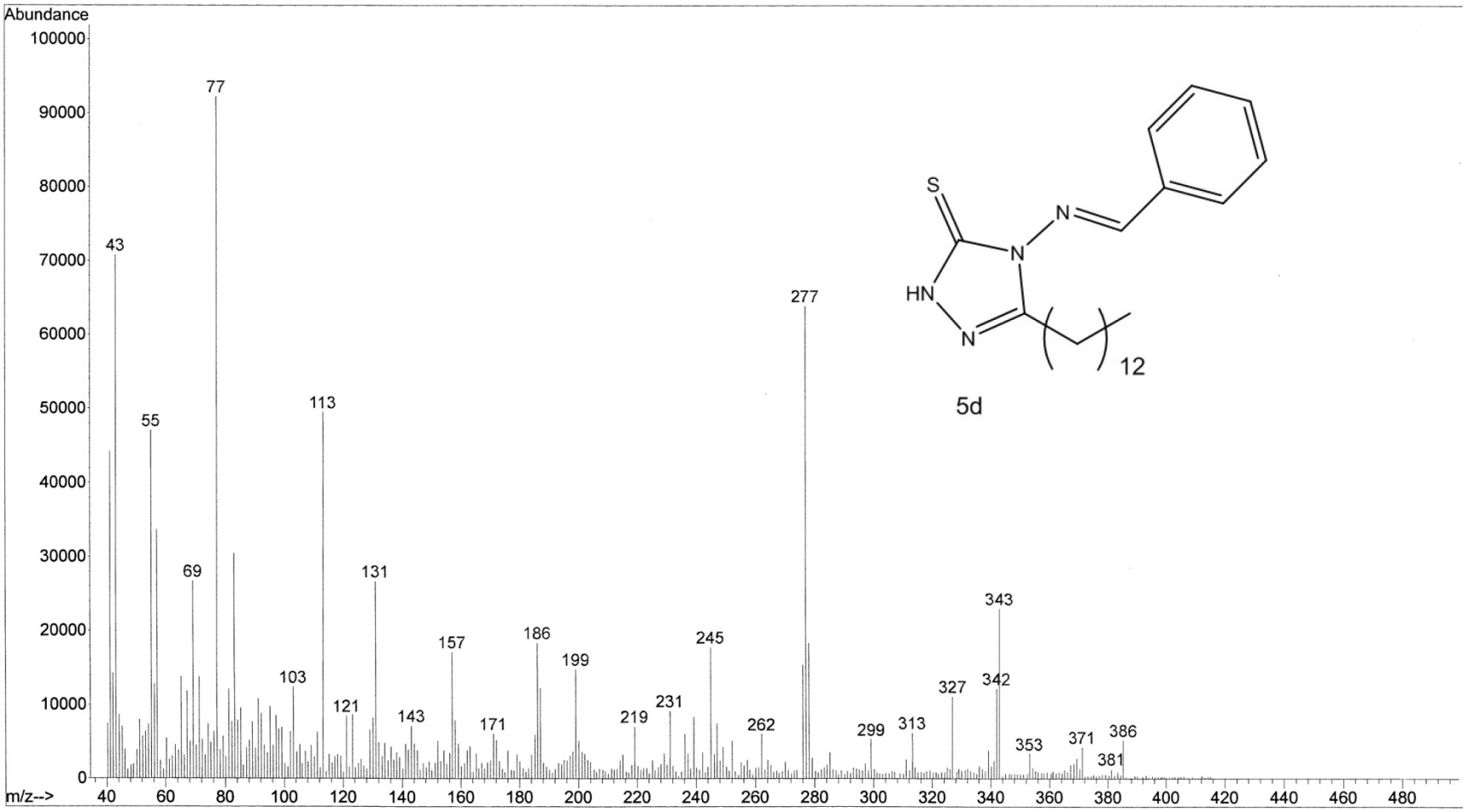
1D NMR plot parameters

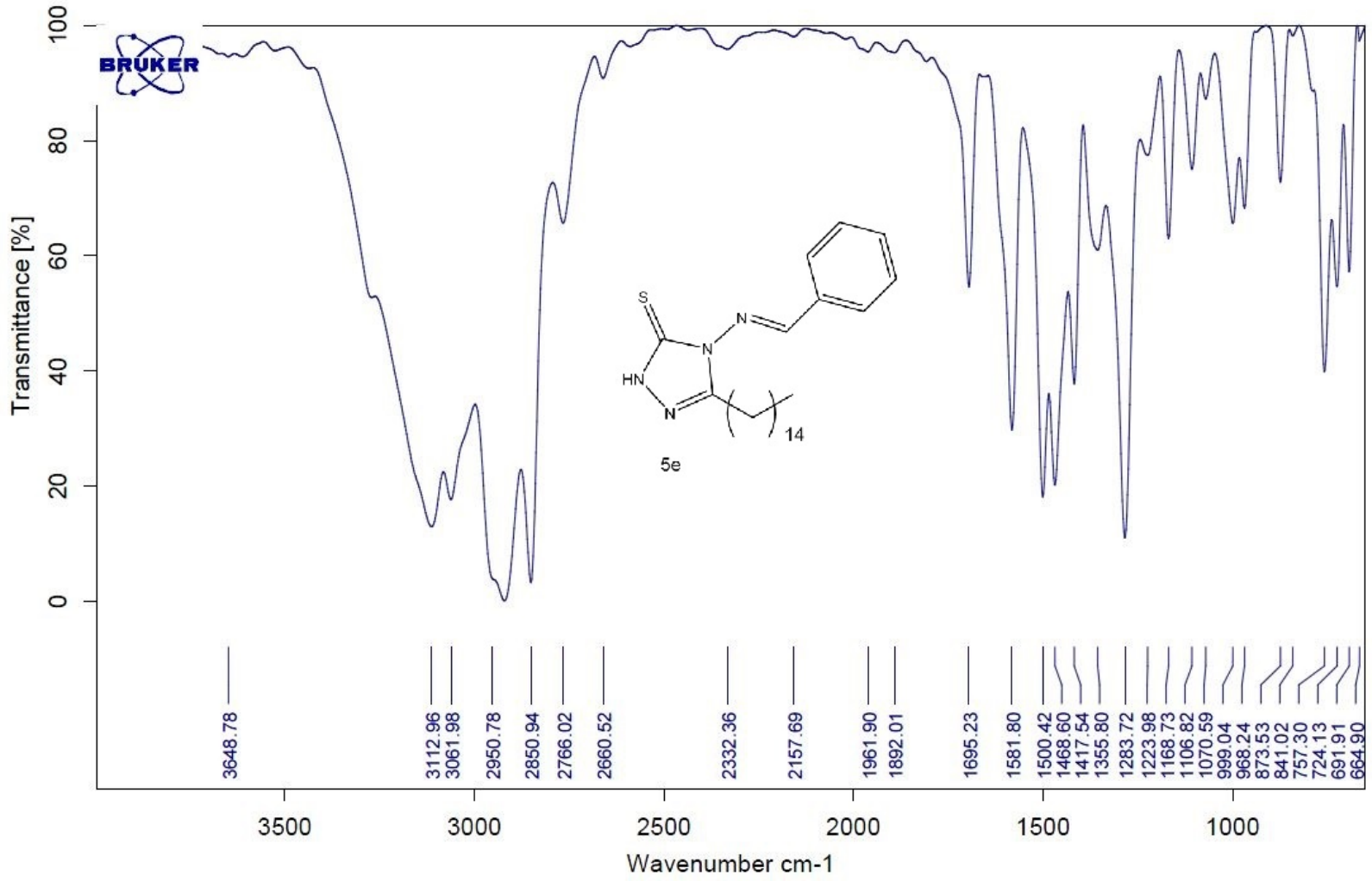
CX	20.00 cm
CY	12.50 cm
F1P	14.500 ppm
F1	4351.89 Hz
F2P	-0.300 ppm
F2	-90.04 Hz
PPMCM	0.74000 ppm/cm
HZCM	222.09618 Hz/cm

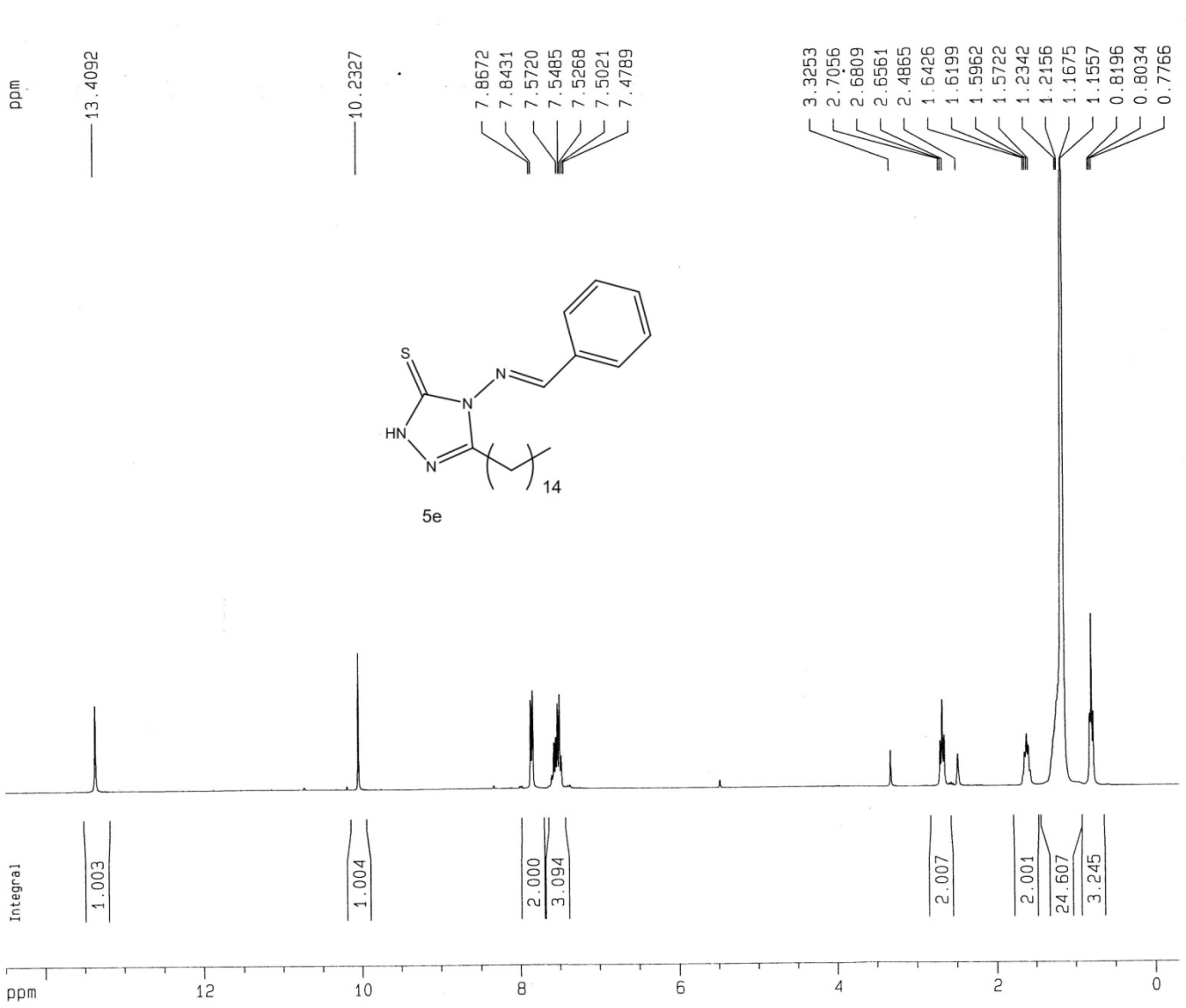
MTEB-C

	Parameter	Value
1	Data File Name	D:/ NMR/ 1397/ 97-10/ 97-10-22/ Kerman/ MTEB-C.fid/ fid
2	Title	MTEB-C
3	Comment	
4	Origin	Varian
5	Owner	
6	Site	
7	Instrument	inova
8	Author	
9	Solvent	dmso
10	Temperature	25.0
11	Pulse Sequence	s2pul
12	Experiment	1D
13	Probe	ID
14	Number of Scans	1024
15	Receiver Gain	60
16	Relaxation Delay	1.0000
17	Pulse Width	7.5000
18	Presaturation Frequency	
19	Acquisition Time	1.0681
20	Acquisition Date	2019-01-12T12:35:04
21	Modification Date	2019-01-12T12:39:17
22	Class	
23	Spectrometer Frequency	125.67
24	Spectral Width	31512.4
25	Lowest Frequency	-1833.0
26	Nucleus	13C
27	Acquired Size	32768
28	Spectral Size	65536




Abundance
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
m/z-->
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
43
77
55
69
113
103
121
131
143
157
171
186
199
219
231
245
262
277
299
313
327
342
343
353
371
381
386
S
N
N
HN
N
12
5d


BRUKER
Transmittance [%]
100
80
60
40
20
0
3500
3000
2500
2000
1500
1000
Wavenumber cm-1
3648.78
3112.96
3061.98
2950.78
2850.94
2766.02
2660.52
2332.36
2157.69
1961.90
1892.01
1695.23
1581.80
1500.42
1468.60
1417.54
1355.80
1283.72
1223.98
1168.73
1106.82
1070.59
999.04
968.24
873.53
841.02
757.30
724.13
691.91
664.90
S
N
N
HN
N
14
5e



Current Data Parameters

NAME	kerman
EXPNO	514
PROCNO	1

F2 - Acquisition Parameters

Date_	20170619
Time	15.13
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	40.3
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	6.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SF01	300.1315007 MHz

F2 - Processing parameters

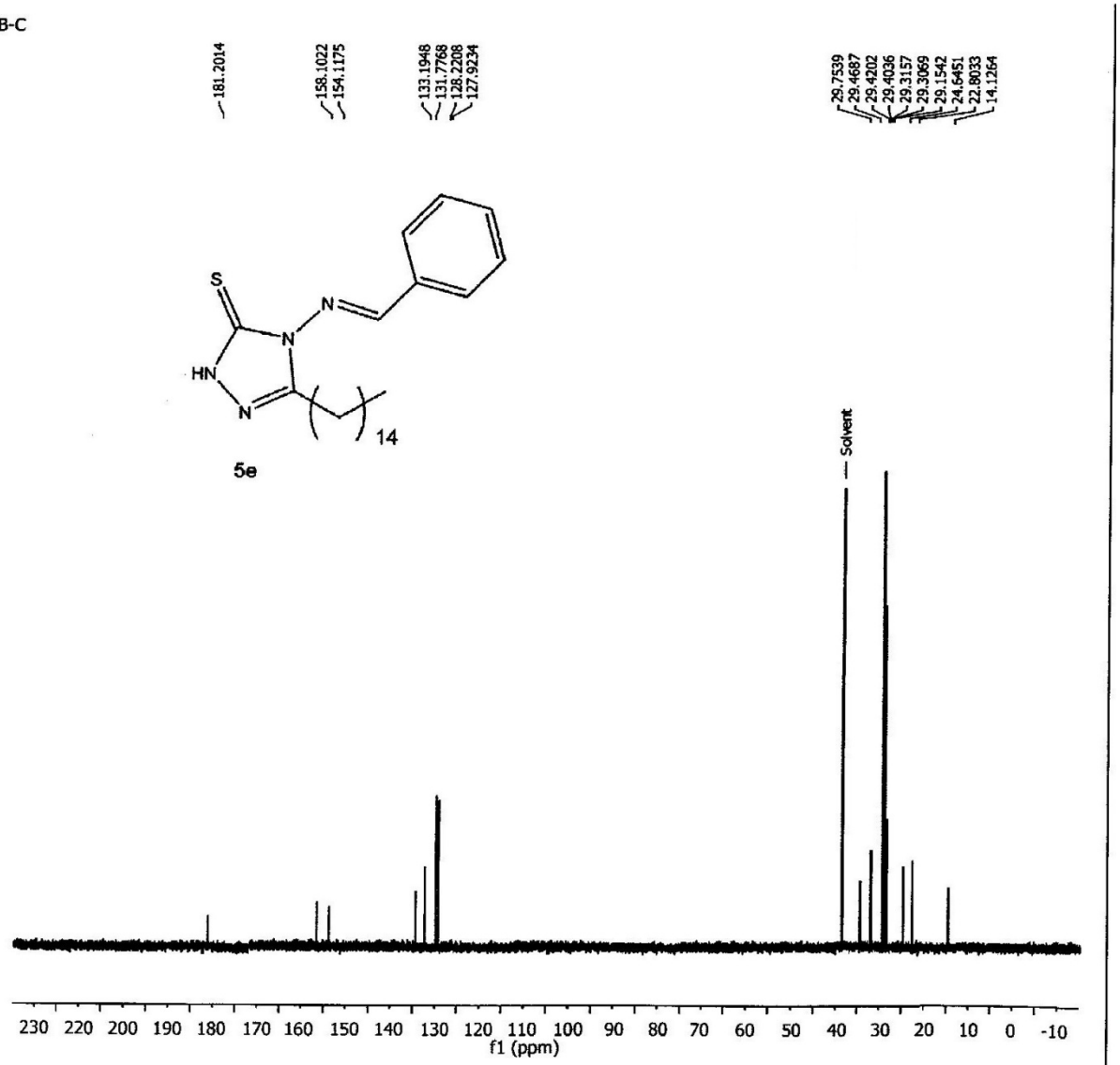
SI	32768
SF	300.1300062 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

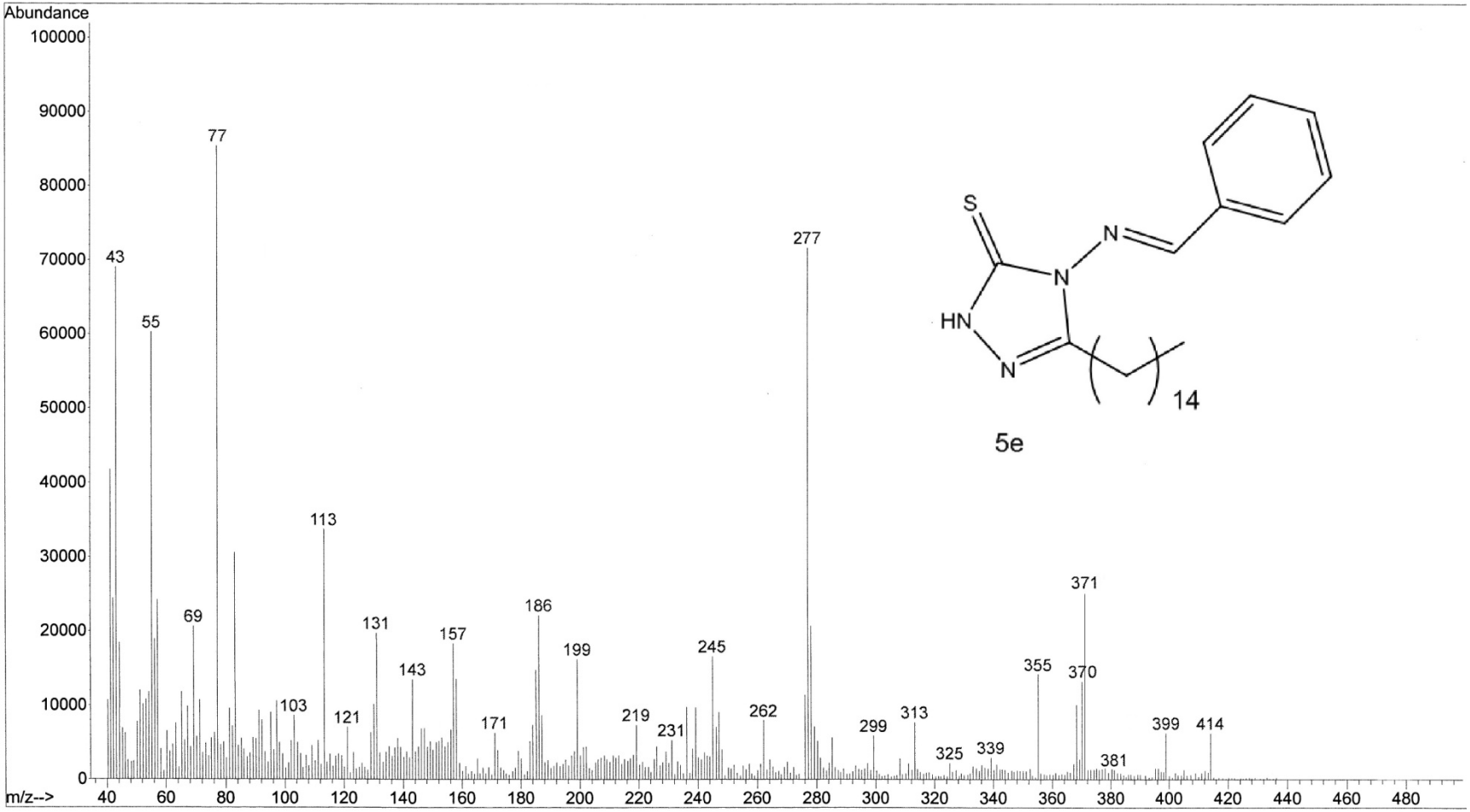
1D NMR plot parameters

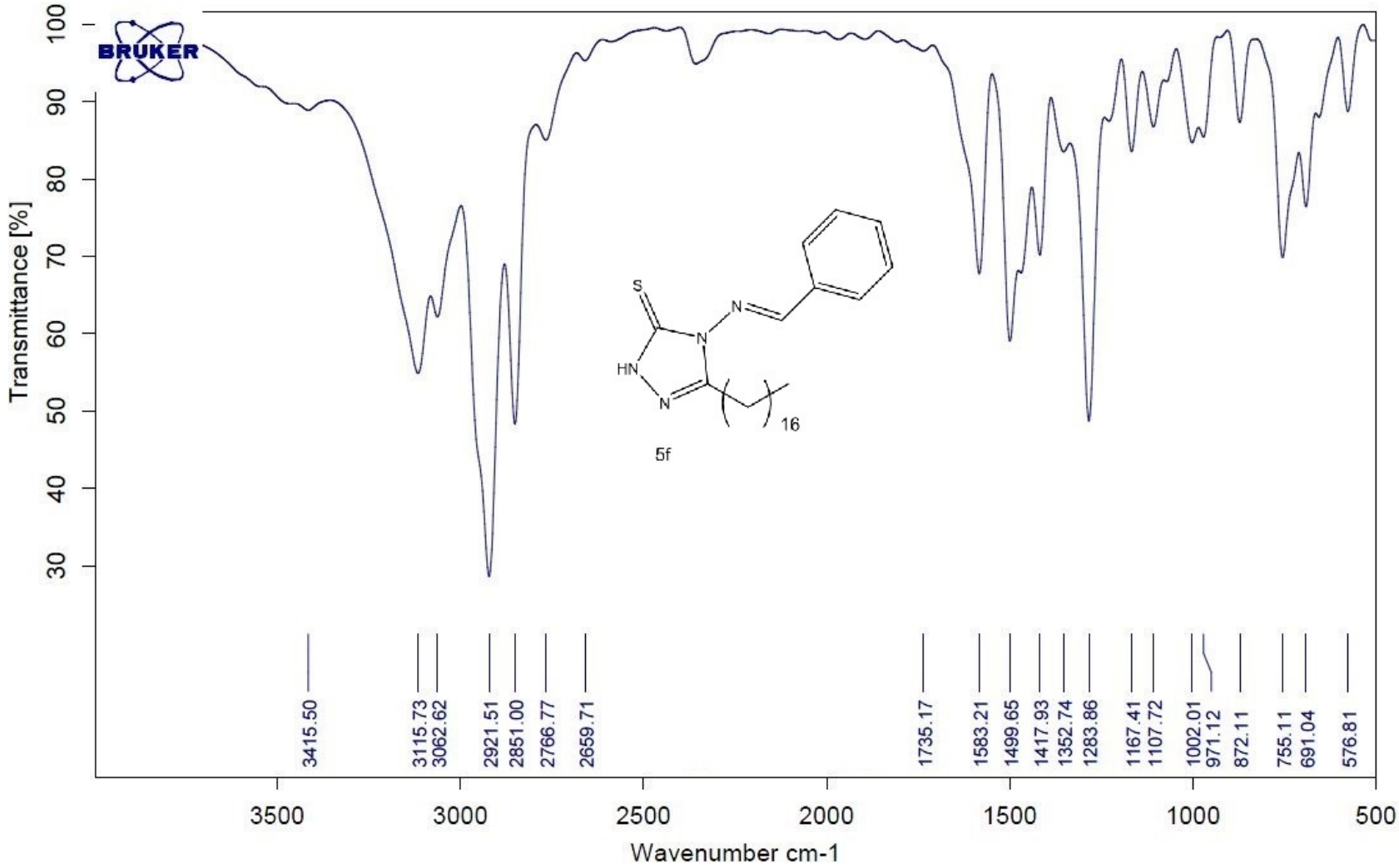
CX	20.00 cm
CY	12.50 cm
F1P	14.500 ppm
F1	4351.89 Hz
F2P	-0.300 ppm
F2	-90.04 Hz
PPMCM	0.74000 ppm/cm
HZCM	222.09619 Hz/cm

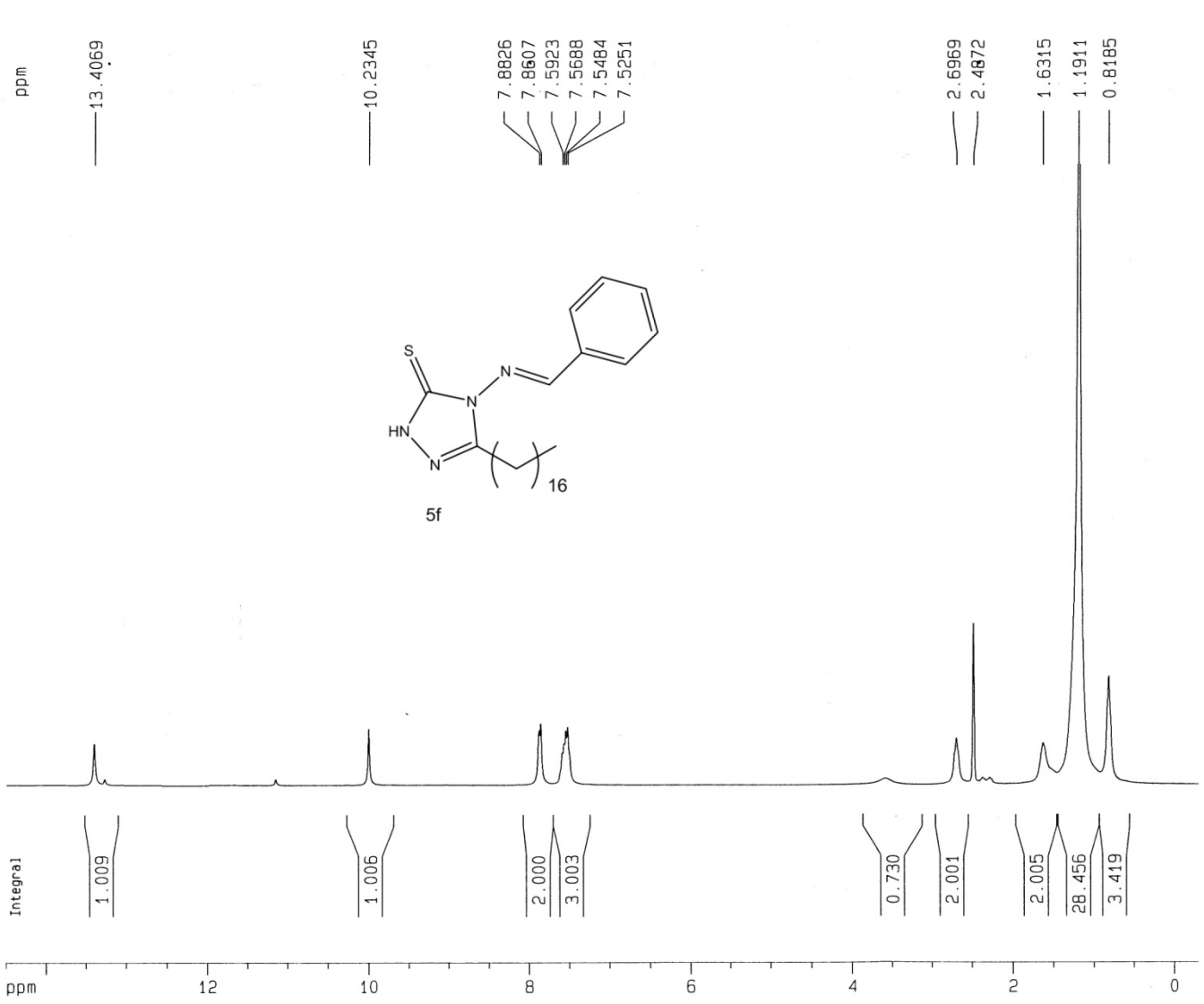
PTEB-C

Parameter	Value
1 Data File Name	D:/ NMR/ 1397/ 97-10/ 97-10-11/ Kerman/ PTEB-C.fid/ fid
2 Title	PTEB-C
3 Comment	
4 Origin	Varian
5 Owner	
6 Site	
7 Instrument	inova
8 Author	
9 Solvent	dmso
10 Temperature	25.0
11 Pulse Sequence	s2pul
12 Experiment	1D
13 Probe	ID
14 Number of Scans	1024
15 Receiver Gain	60
16 Relaxation Delay	1.0000
17 Pulse Width	7.5000
18 Presaturation Frequency	
19 Acquisition Time	1.0322
20 Acquisition Date	2019-01-01T15:22:19
21 Modification Date	2019-01-01T15:26:35
22 Class	
23 Spectrometer Frequency	125.67
24 Spectral Width	31425.7
25 Lowest Frequency	-1833.0
26 Nucleus	13C
27 Acquired Size	32768
28 Spectral Size	65536




Abundance
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
m/z-->
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
43
55
77
69
103
113
121
131
143
157
171
186
199
219
231
245
262
277
299
313
325
339
355
370
371
381
399
414
S
N
N
HN
N
14
5e


BRUKER
Transmittance [%]
100
90
80
70
60
50
40
30
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
3415.50
3115.73
3062.62
2921.51
2851.00
2766.77
2659.71
1735.17
1583.21
1499.65
1417.93
1352.74
1283.86
1167.41
1107.72
1002.01
971.12
872.11
755.11
691.04
576.81
S
N
N
HN
N
16
5f



Current Data Parameters

NAME	kerman
EXPNO	517
PROCNO	1

F2 - Acquisition Parameters

Date_	20170419
Time	16.46
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	80.6
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	6.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SFO1	300.1315007 MHz

F2 - Processing parameters

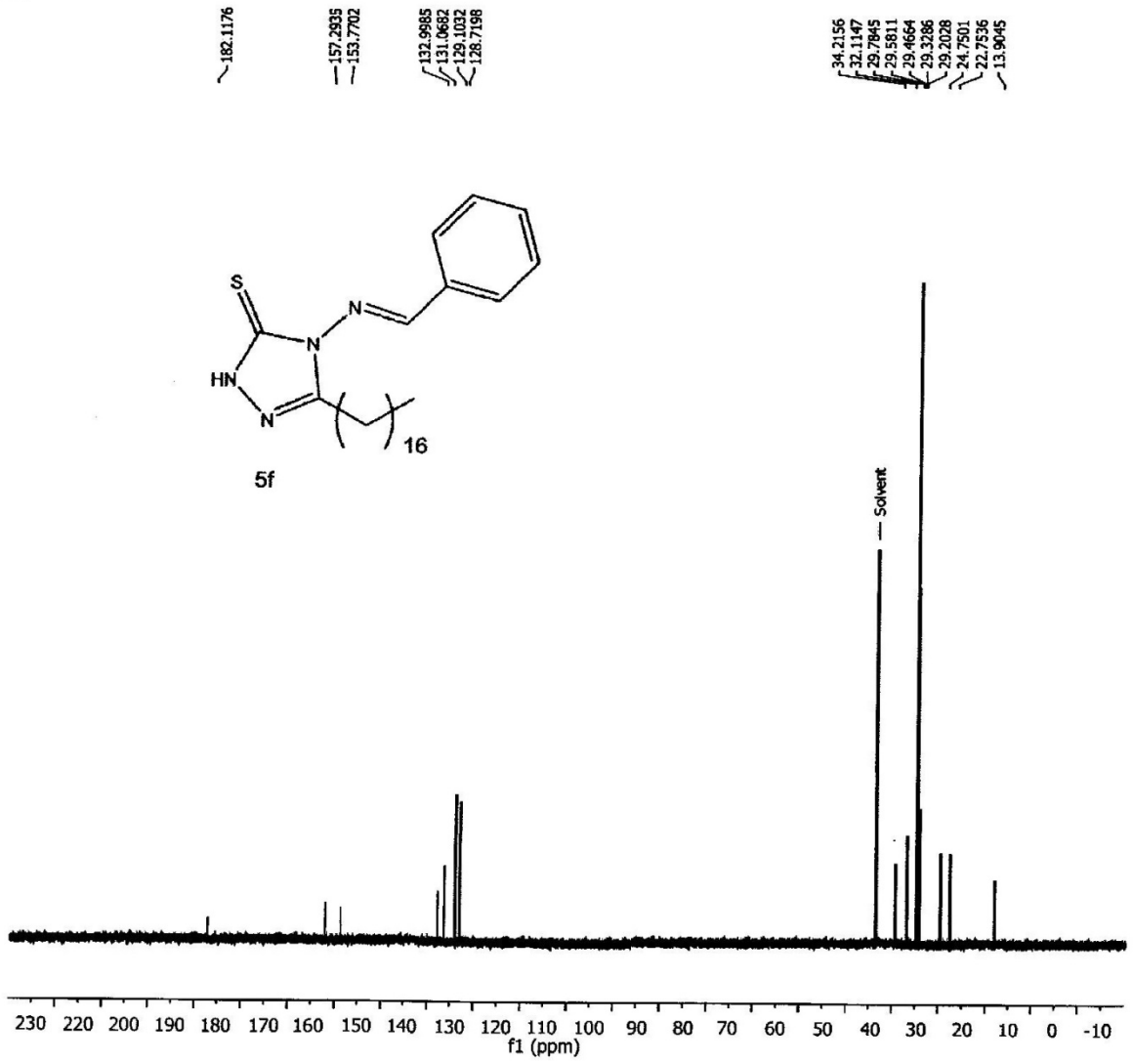
SI	32768
SF	300.1300062 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

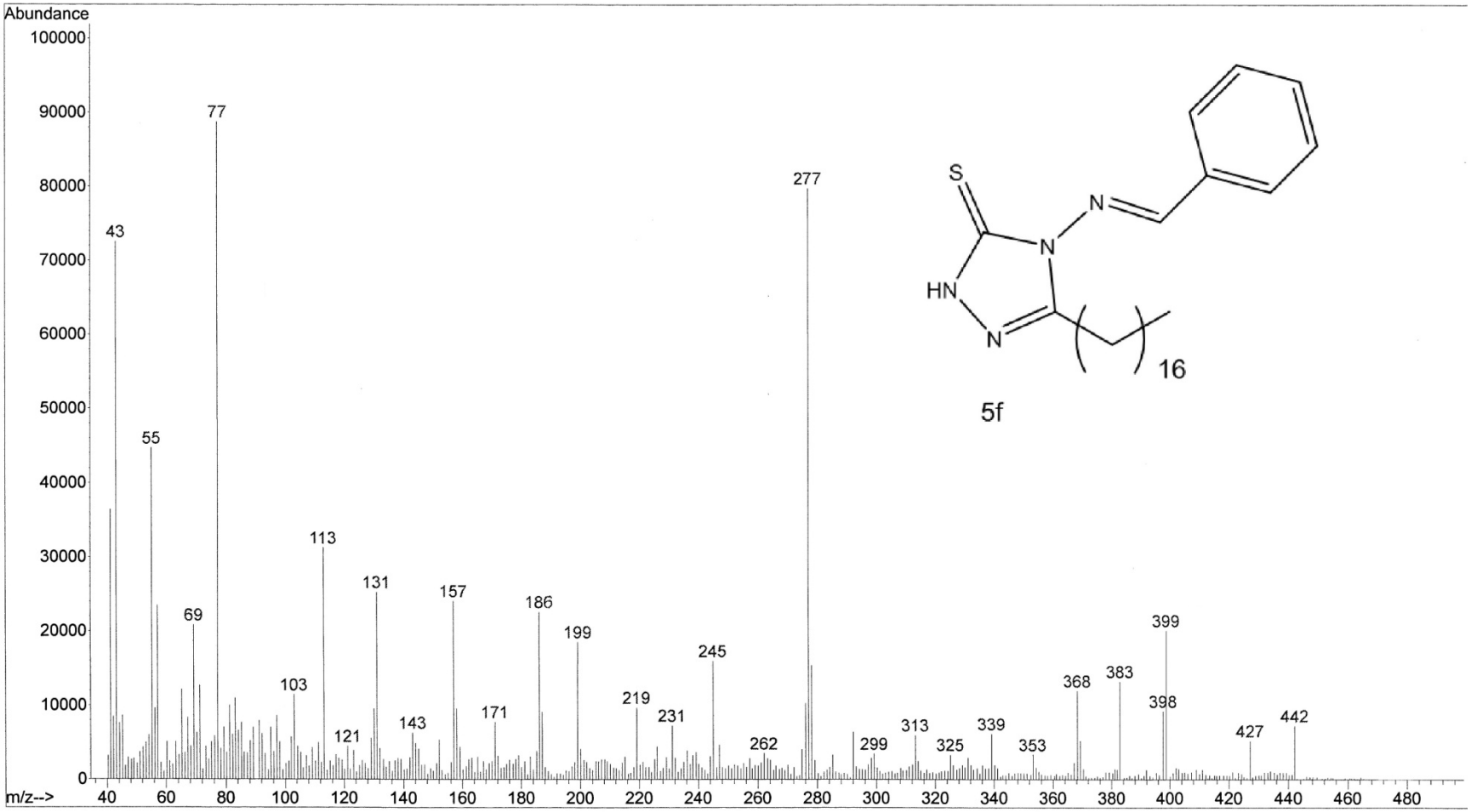
1D NMR plot parameters

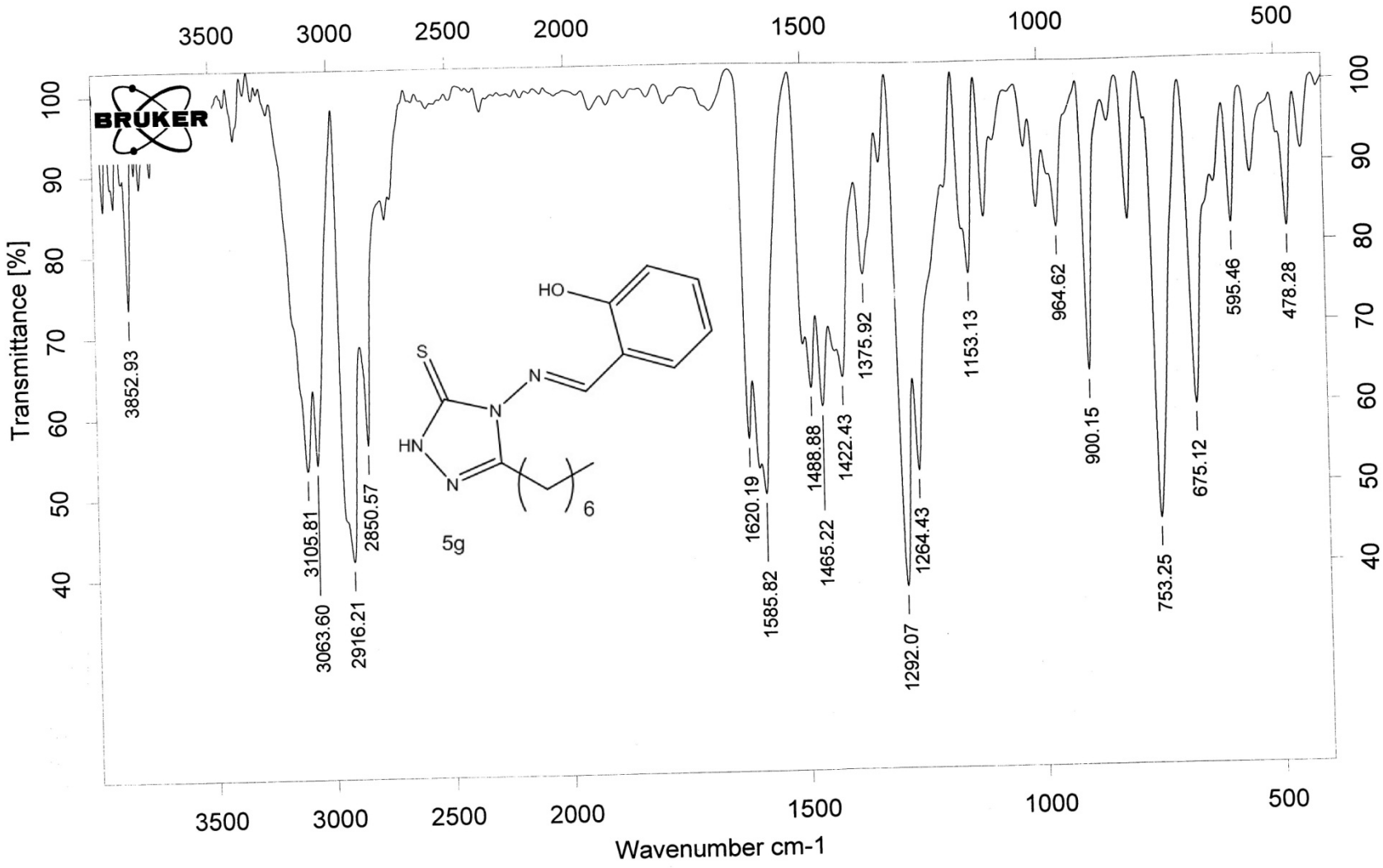
CX	20.00 cm
CY	12.50 cm
F1P	14.500 ppm
F1	4351.89 Hz
F2P	-0.300 ppm
F2	-90.04 Hz
PPMCM	0.74000 ppm/cm
HZCM	222.09619 Hz/cm

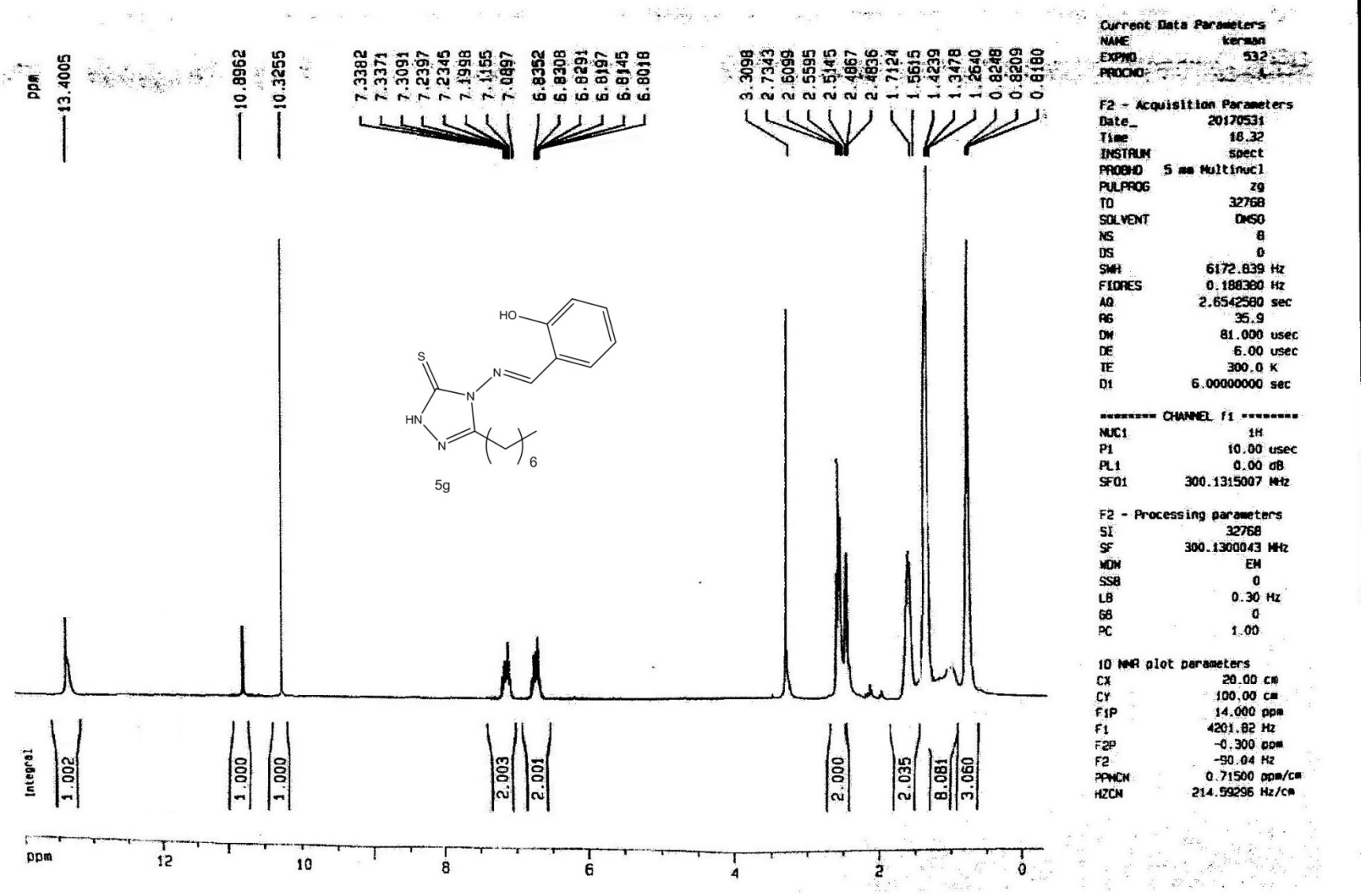
STEB-C

	Parameter	Value
1	Data File Name	D:/ NMR/ 1397/ 97-10/ 97-10-11/ Kerman/ STEB-C.fid/ fid
2	Title	STEB-C
3	Comment	
4	Origin	Varian
5	Owner	
6	Site	
7	Instrument	inova
8	Author	
9	Solvent	dmso
10	Temperature	25.0
11	Pulse Sequence	s2pul
12	Experiment	1D
13	Probe	ID
14	Number of Scans	1024
15	Receiver Gain	60
16	Relaxation Delay	1.0000
17	Pulse Width	7.5000
18	Presaturation Frequency	
19	Acquisition Time	1.0568
20	Acquisition Date	2019-01-01T15:45:42
21	Modification Date	2019-01-01T15:48:54
22	Class	
23	Spectrometer Frequency	125.67
24	Spectral Width	31409.5
25	Lowest Frequency	-1833.0
26	Nucleus	13C
27	Acquired Size	32768
28	Spectral Size	65536




Abundance
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
m/z-->
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
43
77
55
69
103
113
121
131
143
157
171
186
199
219
231
245
262
277
299
313
325
339
353
368
383
398
399
427
442
S
N
N
HN
N
16
5f


BRUKER
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
100
90
80
70
60
50
40
3852.93
3105.81
3063.60
2916.21
2850.57
1620.19
1585.82
1488.88
1465.22
1422.43
1375.92
1292.07
1264.43
1153.13
964.62
900.15
753.25
675.12
595.46
478.28
HO
S
N
HN
N
6
5g



Current Data Parameters
NAME kerman
EXPNO 532
PROCNO

F2 - Acquisition Parameters
Date_ 20170531
Time 18.32
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 35.9
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 6.00000000 sec

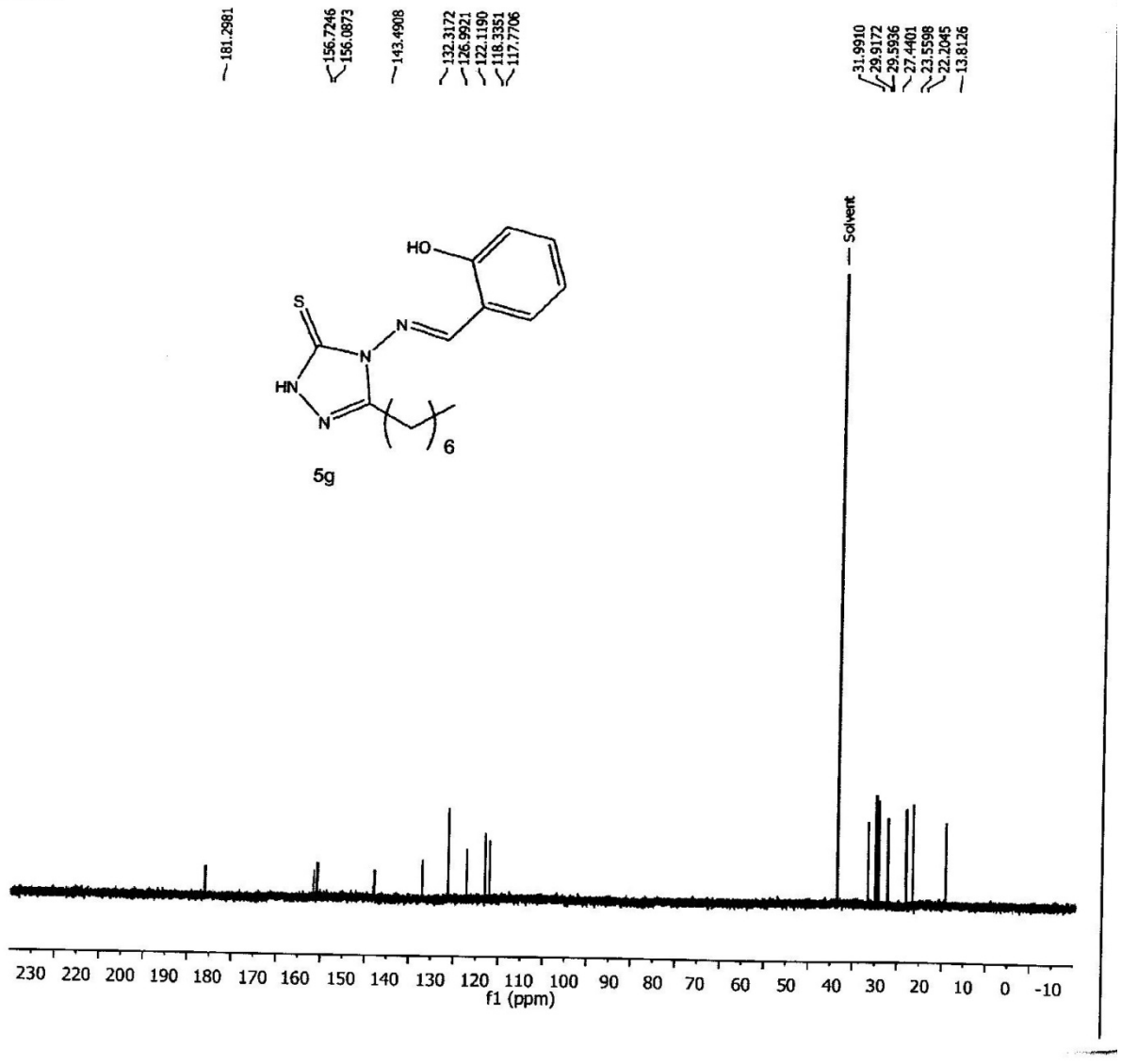
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SFO1 300.1315007 MHz

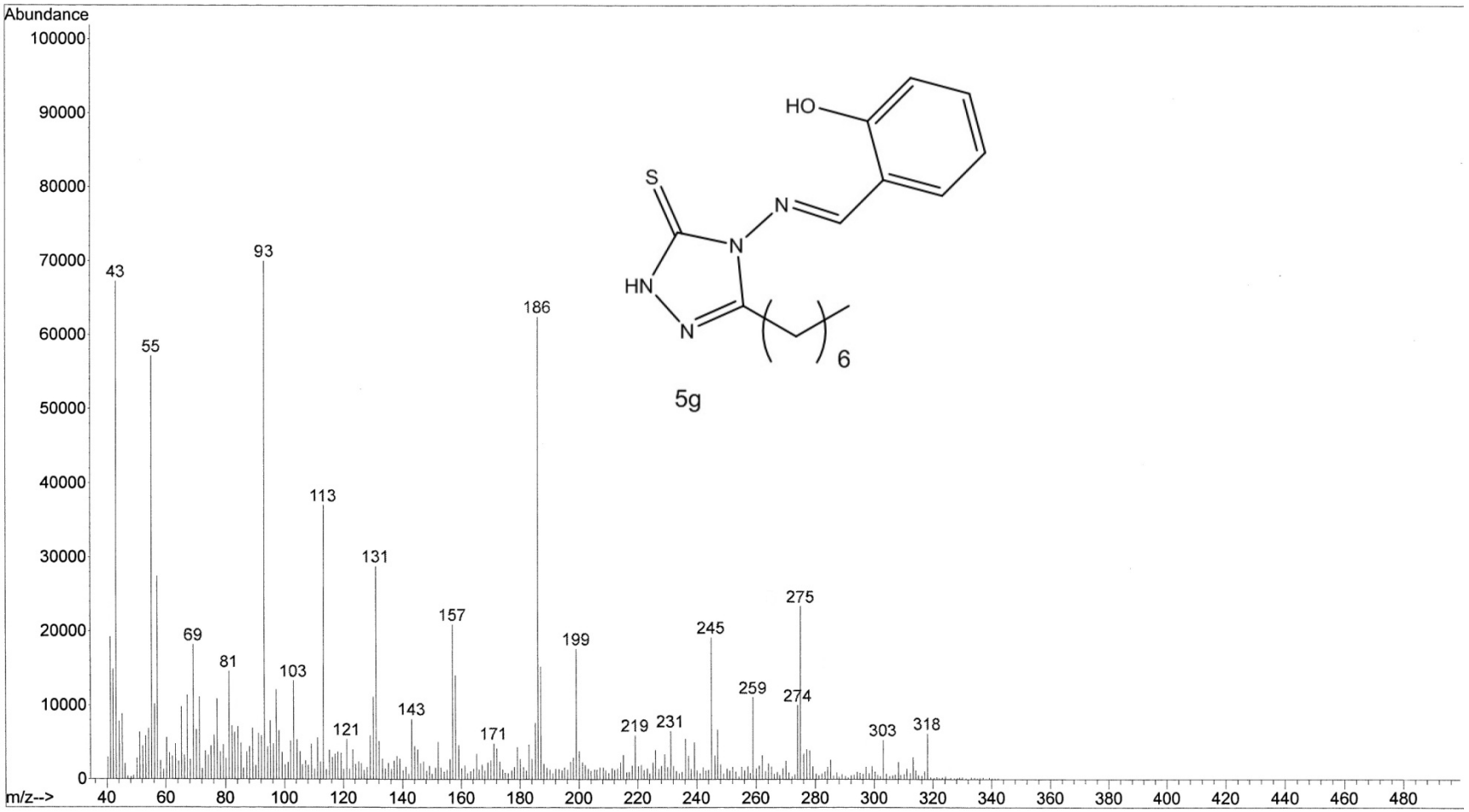
F2 - Processing parameters
SI 32768
SF 300.1300043 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

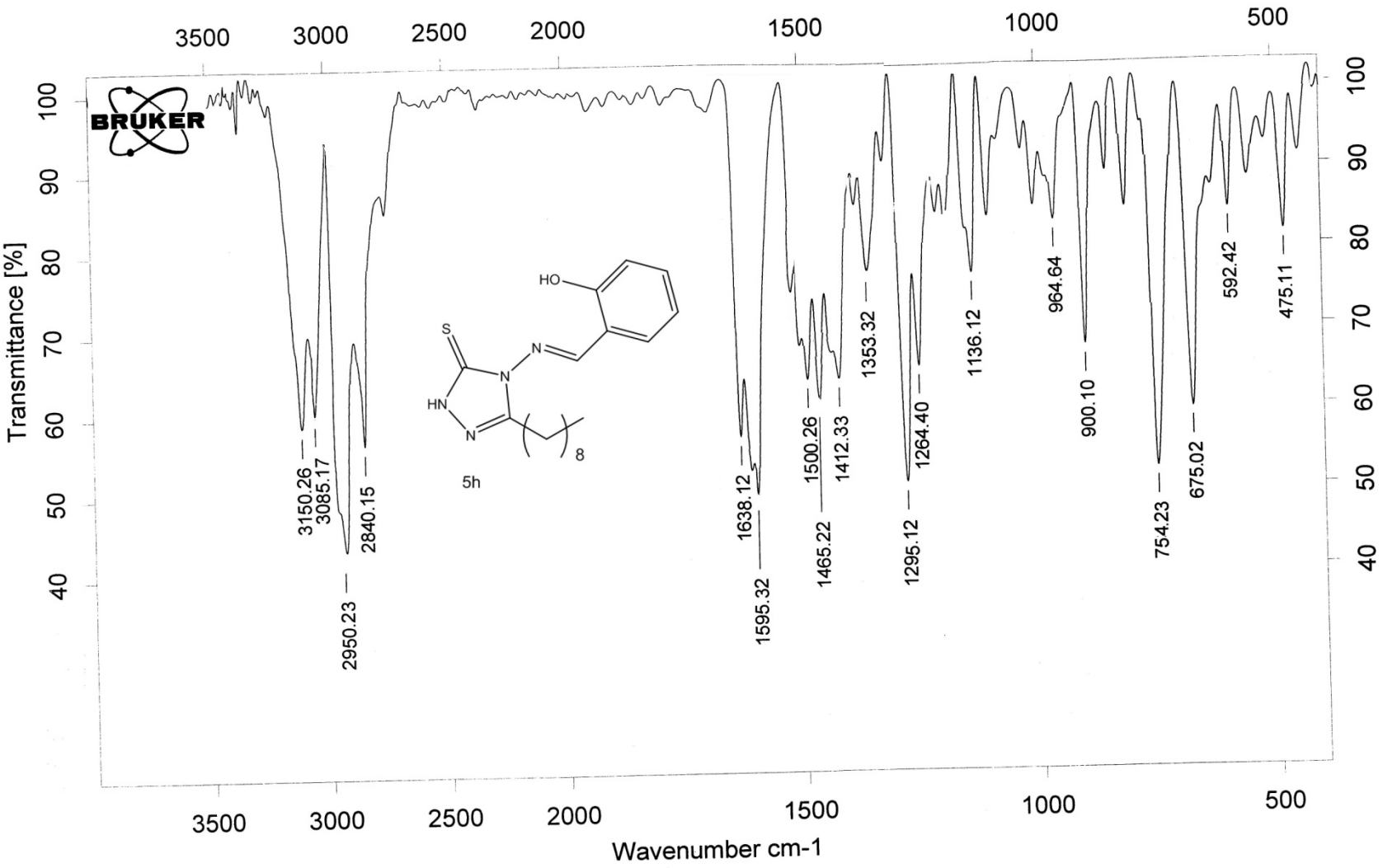
1D NMR plot parameters
CX 20.00 cm
CY 100.00 cm
F1P 14.000 ppm
F1 4201.82 Hz
F2P -0.300 ppm
F2 -90.04 Hz
PPMCM 0.71500 ppm/cm
HZCM 214.59296 Hz/cm

OTE2OH-C

	Parameter	Value
1	Data File Name	D:/ NMR/ 1397/ 97-10/ 97-10-26/ Kerman/ OTE2OH-C.fid/ fid
2	Title	OTE2OH-C
3	Comment	
4	Origin	Varian
5	Owner	
6	Site	
7	Instrument	inova
8	Author	
9	Solvent	dmso
10	Temperature	25.0
11	Pulse Sequence	s2pul
12	Experiment	1D
13	Probe	ID
14	Number of Scans	1024
15	Receiver Gain	60
16	Relaxation Delay	1.0000
17	Pulse Width	7.5000
18	Presaturation Frequency	
19	Acquisition Time	1.0489
20	Acquisition Date	2019-01-16T16:52:07
21	Modification Date	2019-01-16T16:56:19
22	Class	
23	Spectrometer Frequency	125.67
24	Spectral Width	31455.1
25	Lowest Frequency	-1833.0
26	Nucleus	13C
27	Acquired Size	32768
28	Spectral Size	65536




Abundance
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
m/z-->
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
43
55
69
81
93
103
113
121
131
143
157
171
186
199
219
231
245
259
274
275
303
318
HO
S
N
N
HN
N
6
5g


BRUKER
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
100
90
80
70
60
50
40
3150.26
3085.17
2950.23
2840.15
1638.12
1595.32
1500.26
1465.22
1412.33
1353.32
1295.12
1264.40
1136.12
964.64
900.10
754.23
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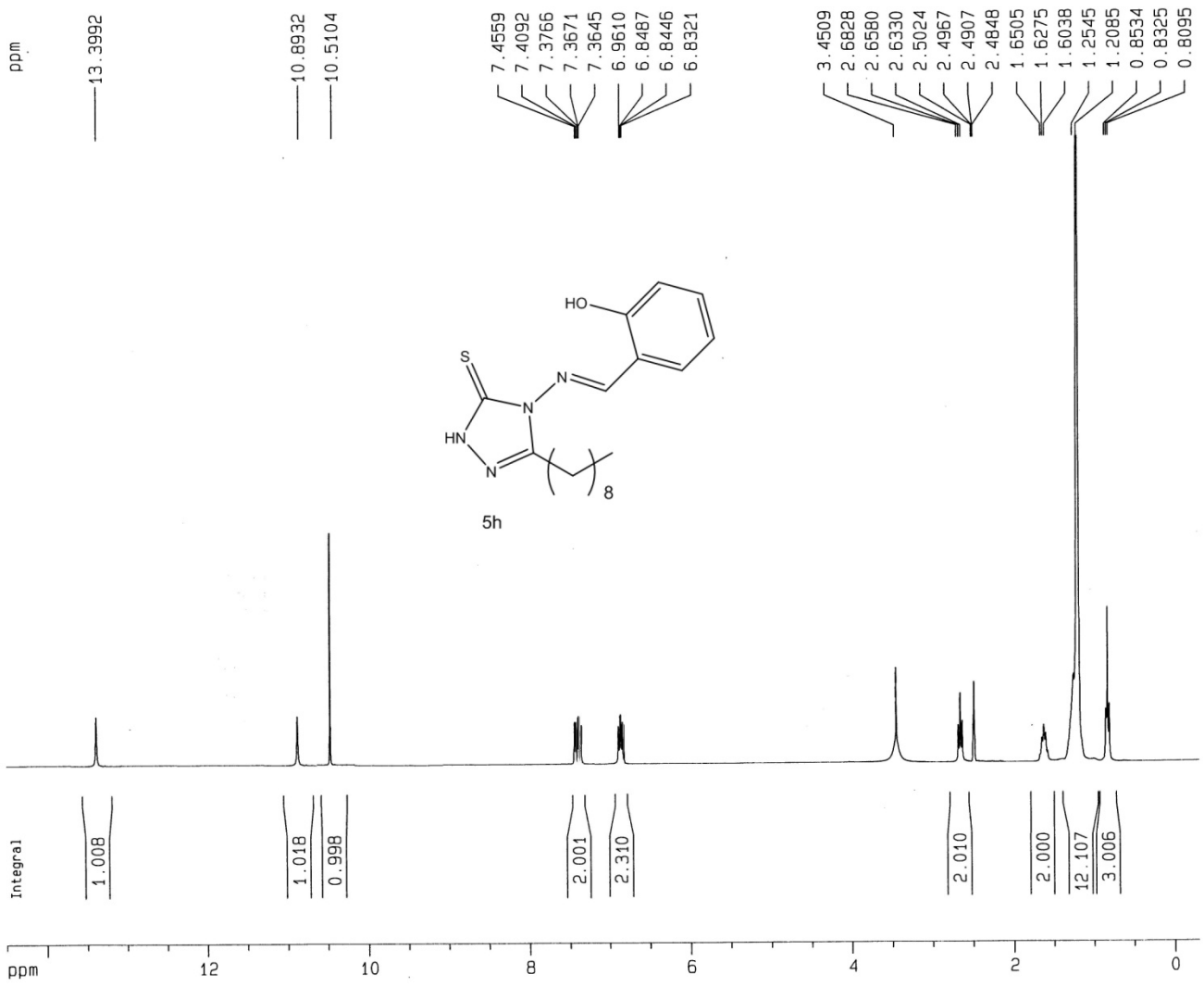
475.11
HO
S
N
HN
N
8
5h



Current Data Parameters

NAME	kerman
EXPNO	507
PROCNO	1

F2 - Acquisition Parameters

Date_	20170726
Time	16.19
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	128
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	6.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SFO1	300.1315007 MHz

F2 - Processing parameters

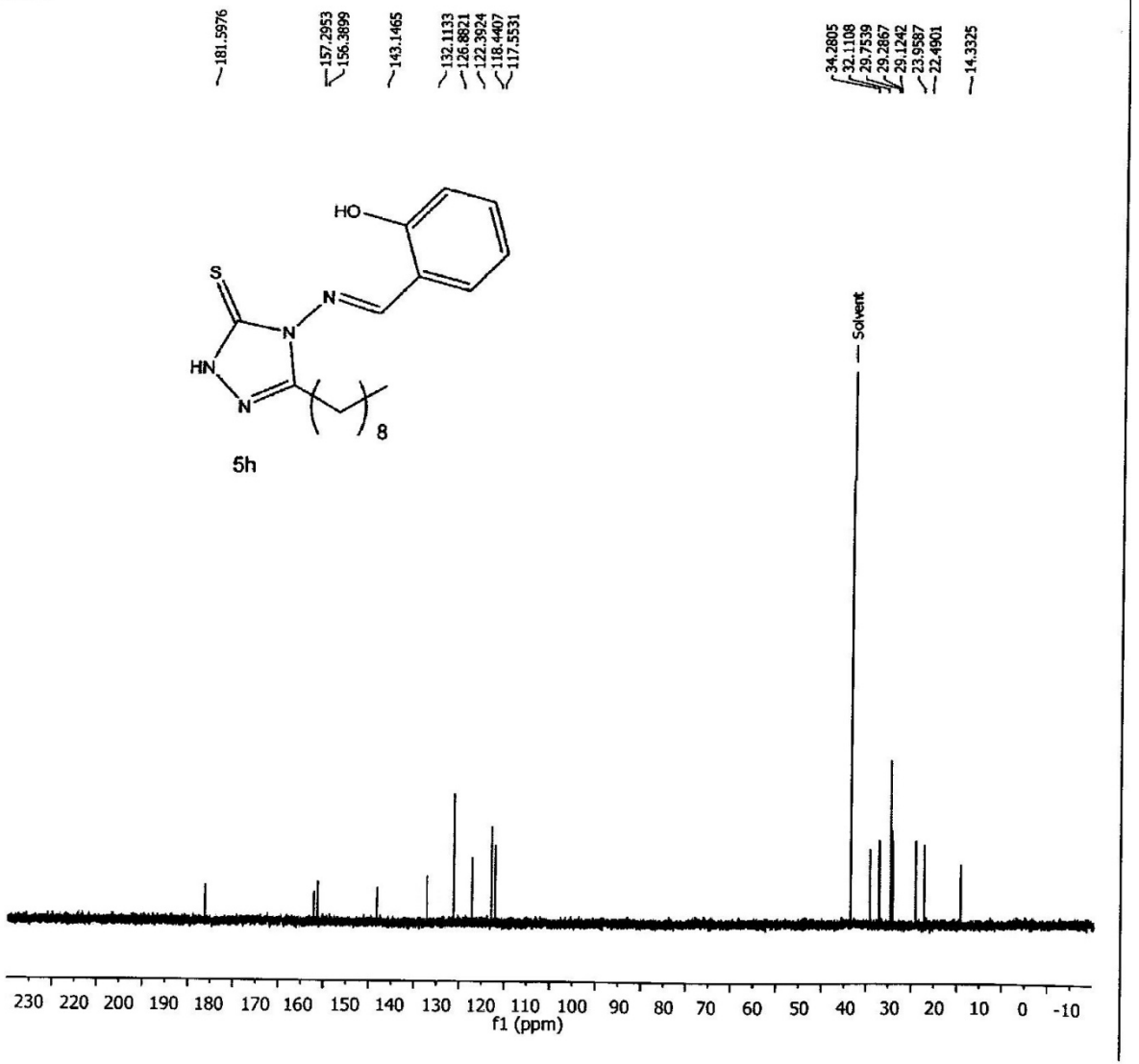
SI	32768
SF	300.1300038 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

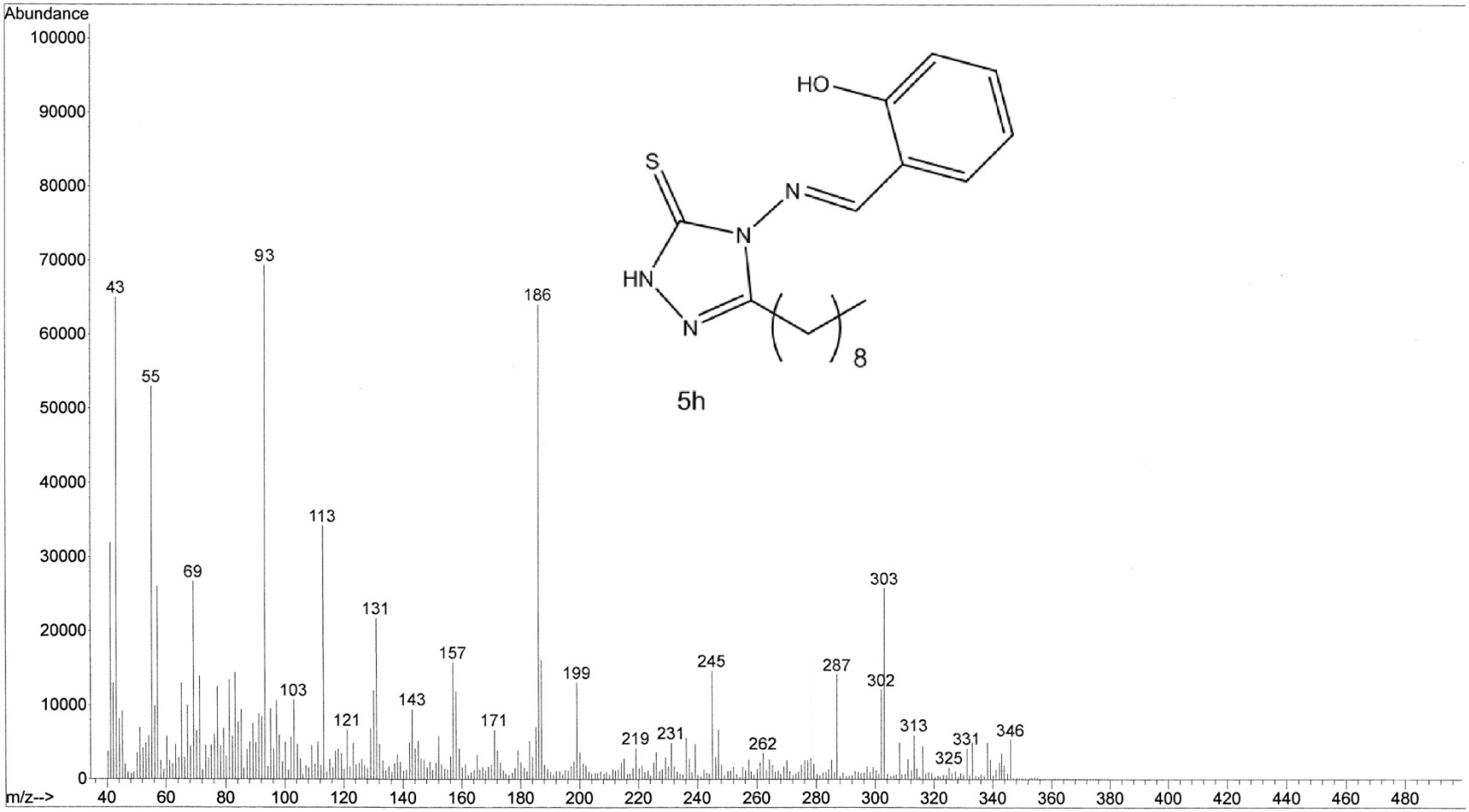
1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	14.500 ppm
F1	4351.89 Hz
F2P	-0.300 ppm
F2	-90.04 Hz
PPMCM	0.74000 ppm/cm
HZCM	222.09619 Hz/cm

DTE2OH-C

	Parameter	Value
1	Data File Name	D:/ NMR/ 1397/ 97-10/ 97-10-26/ Kerman/ DTE2O-C.fid/ fid
2	Title	DTE2OH-C
3	Comment	
4	Origin	Varian
5	Owner	
6	Site	
7	Instrument	inova
8	Author	
9	Solvent	dmso
10	Temperature	25.0
11	Pulse Sequence	s2pul
12	Experiment	1D
13	Probe	ID
14	Number of Scans	1024
15	Receiver Gain	60
16	Relaxation Delay	1.0000
17	Pulse Width	7.5000
18	Presaturation Frequency	
19	Acquisition Time	1.0357
20	Acquisition Date	2019-01-16T16:27:06
21	Modification Date	2019-01-16T16:31:15
22	Class	
23	Spectrometer Frequency	125.67
24	Spectral Width	31986.2
25	Lowest Frequency	-1833.0
26	Nucleus	13C
27	Acquired Size	32768
28	Spectral Size	65536




Abundance
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
m/z-->
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
43
55
69
93
103
113
121
131
143
157
171
186
199
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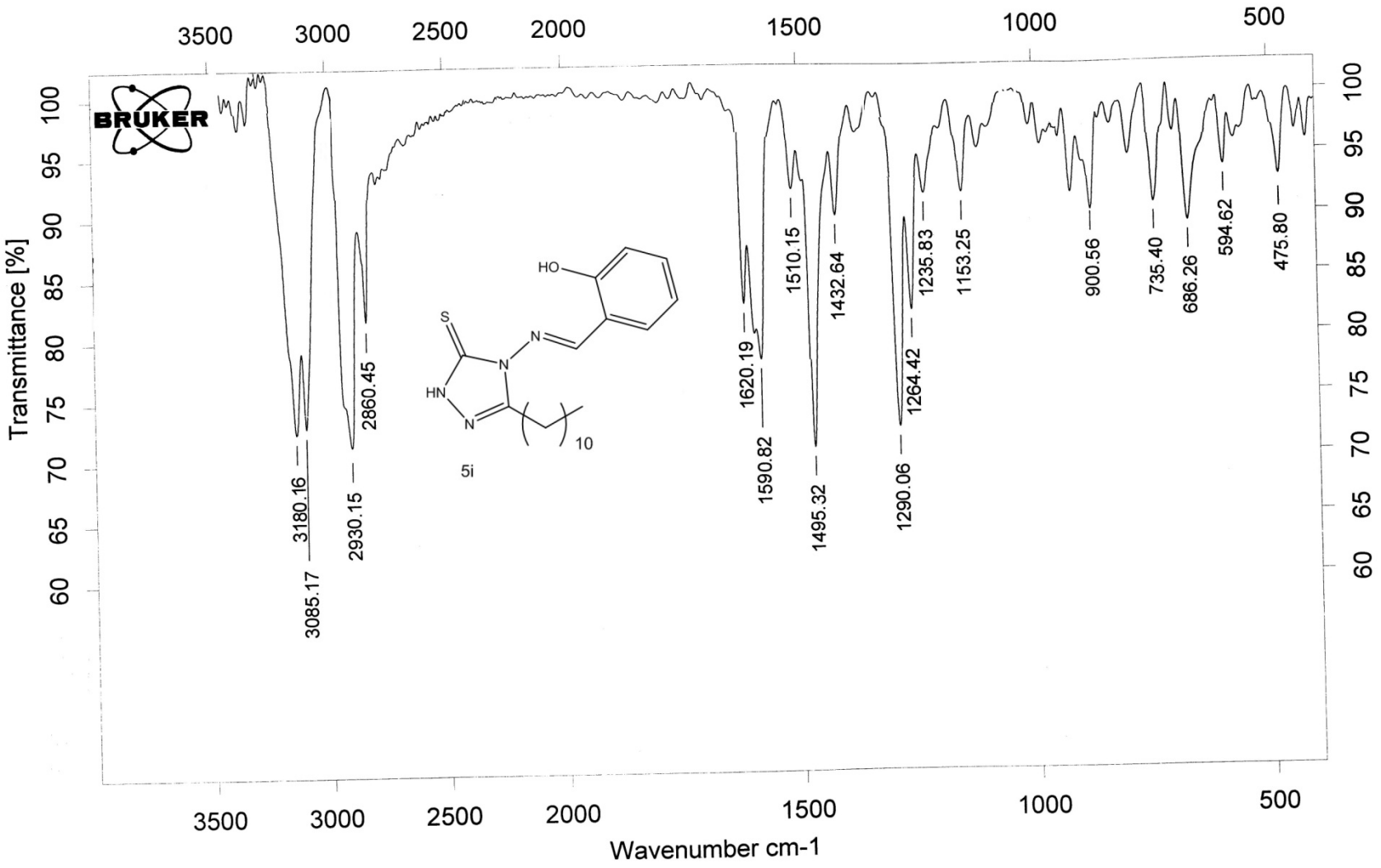
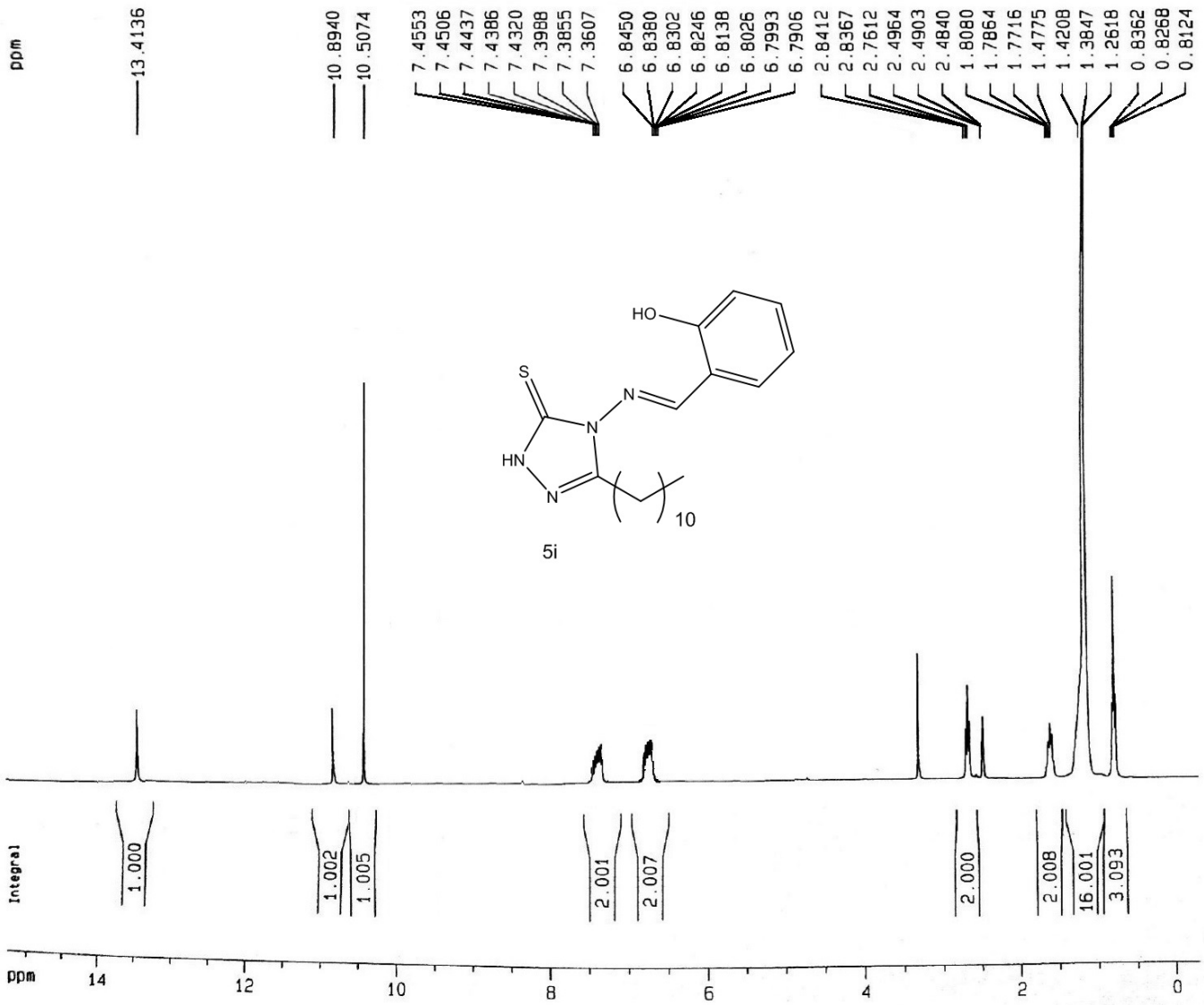
262
287
302
303
313
325
331
346
HO
S
N
N
HN
N
8
5h


BRUKER
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
100
95
90
85
80
75
70
65
60
3180.16
3085.17
2930.15
2860.45
1620.19
1590.82
1510.15
1495.32
1432.64
1290.06
1264.42
1235.83
1153.25
900.56
735.40
686.26
594.62
475.80
HO
S
N
HN
10
5i



Current Data Parameters

NAME	kerman
EXPNO	505
PROCNO	1

F2 - Acquisition Parameters

Date_	20170725
Time	17.32
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	12
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	50.0
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	6.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SFO1	300.1315007 MHz

F2 - Processing parameters

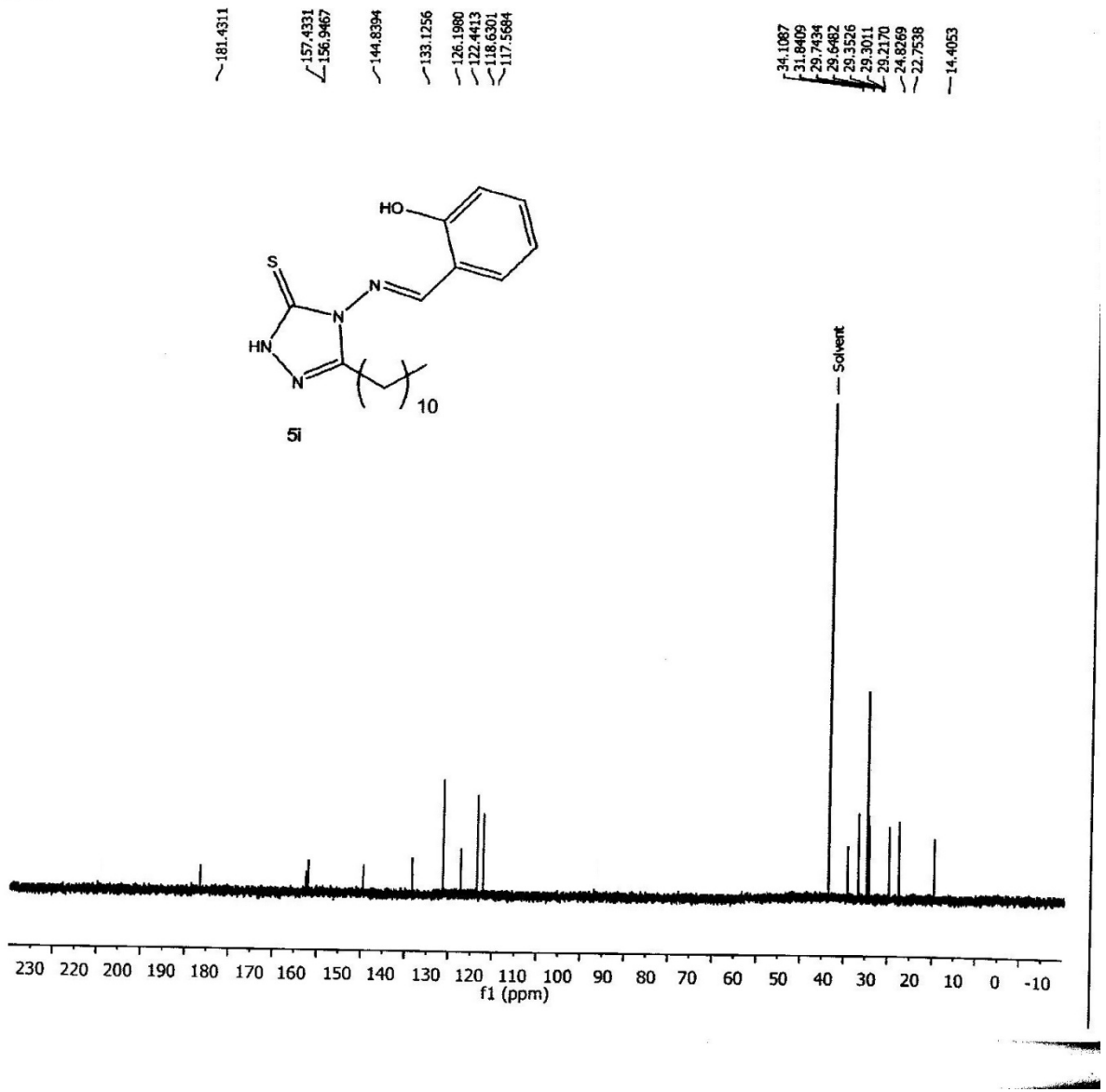
SI	32768
SF	300.1300032 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

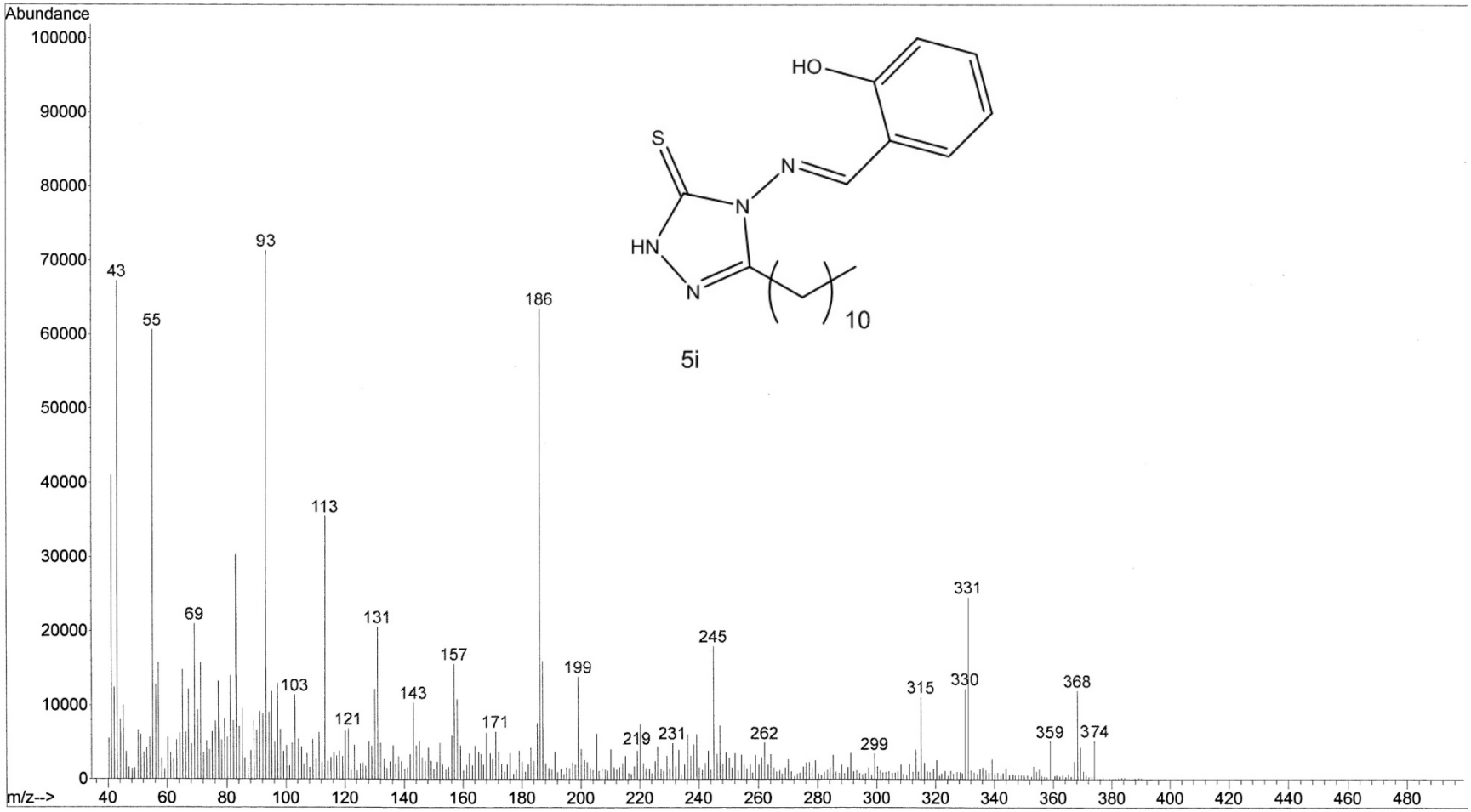
1D NMR plot parameters

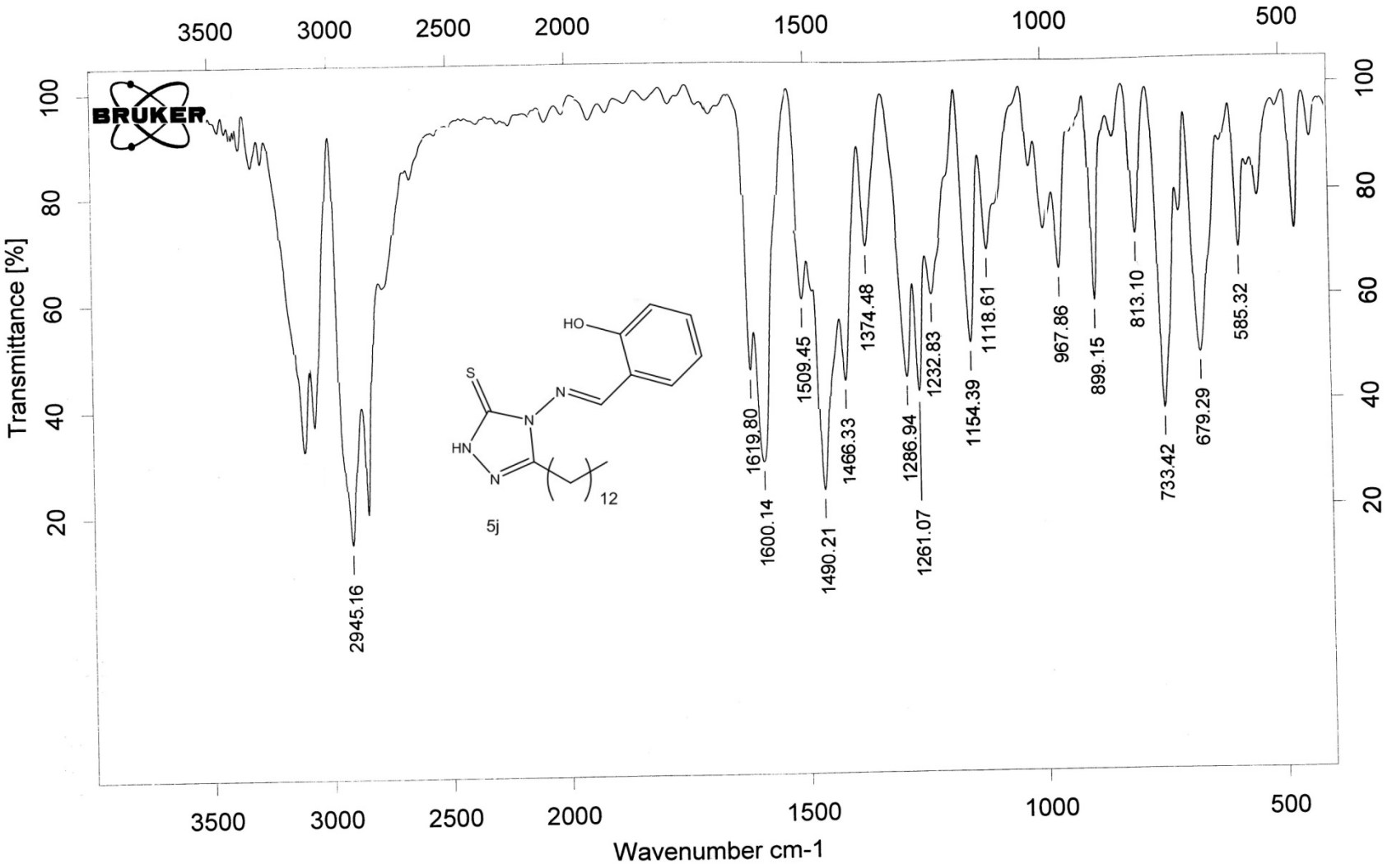
CX	20.00 cm
CY	12.50 cm
F1P	15.200 ppm
F1	4503.20 Hz
F2P	-0.300 ppm
F2	-90.04 Hz
PPMCM	0.77899 ppm/cm
HZCM	232.67693 Hz/cm

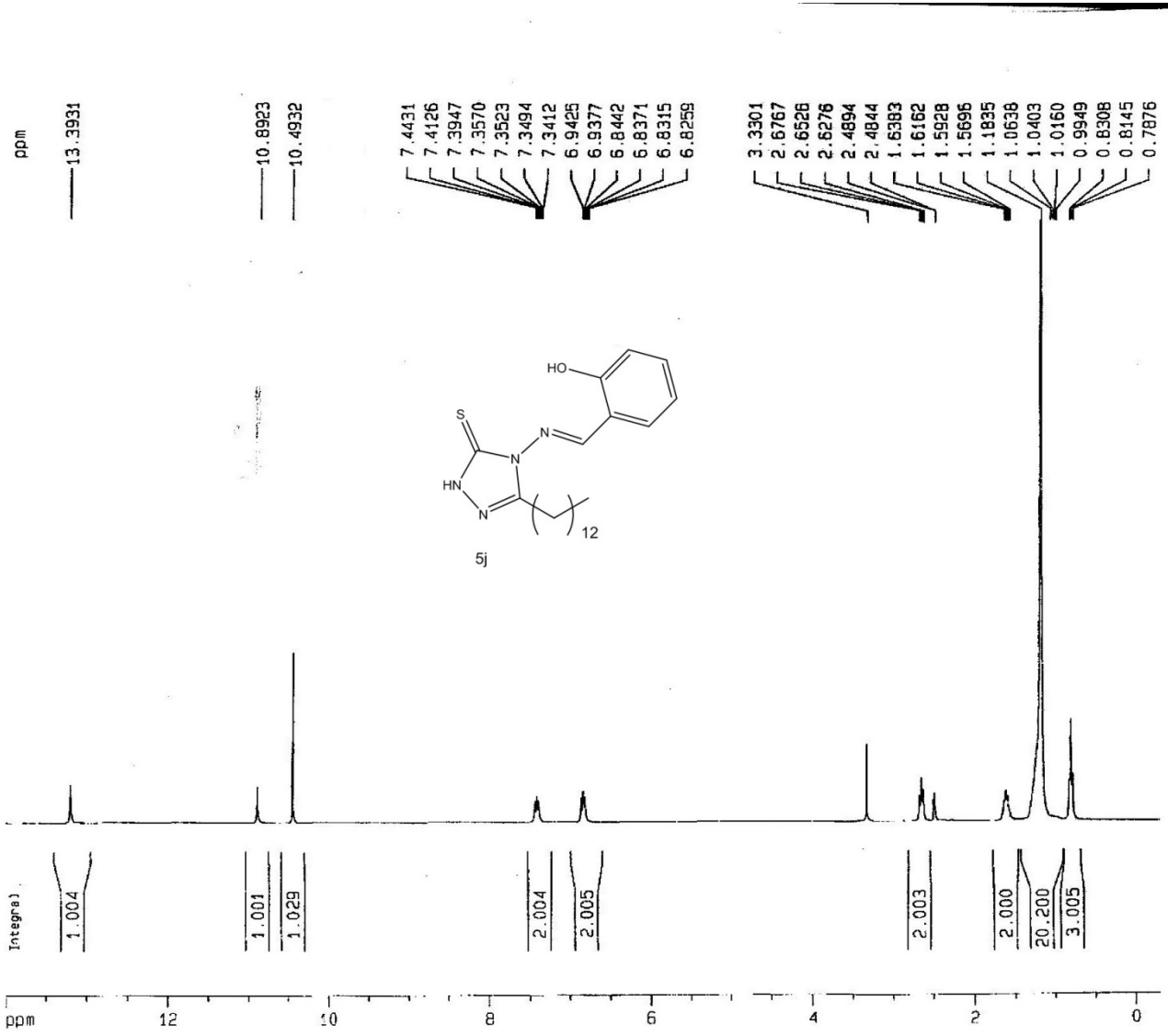
LTE2OH-C

	Parameter	Value
1	Data File Name	D:/ NMR/ 1397/ 97-10/ 97-10-26/ Kerman/ LTE2OH-C.fid/ fid
2	Title	LTE2OH-C
3	Comment	
4	Origin	Varian
5	Owner	
6	Site	
7	Instrument	inova
8	Author	
9	Solvent	dmso
10	Temperature	25.0
11	Pulse Sequence	s2pul
12	Experiment	1D
13	Probe	ID
14	Number of Scans	1024
15	Receiver Gain	60
16	Relaxation Delay	1.0000
17	Pulse Width	7.5000
18	Presaturation Frequency	
19	Acquisition Time	1.0381
20	Acquisition Date	2019-01-16T14:35:33
21	Modification Date	2019-01-16T14:39:47
22	Class	
23	Spectrometer Frequency	125.67
24	Spectral Width	31467.4
25	Lowest Frequency	-1833.0
26	Nucleus	13C
27	Acquired Size	32768
28	Spectral Size	65536




Abundance
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
m/z-->
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
43
55
69
93
103
113
121
131
143
157
171
186
199
219
231
245
262
299
315
330
331
359
368
374
HO
S
N
N
HN
N
10
5i


BRUKER
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
20
40
60
80
100
2945.16
1619.80
1600.14
1509.45
1490.21
1466.33
1374.48
1286.94
1261.07
1232.83
1154.39
1118.61
967.86
899.15
813.10
733.42
679.29
585.32
HO
S
N
HN
N
12
5j



Current Data Parameters

NAME	kerman
EXPNO	518
PROCNO	1

F2 - Acquisition Parameters

Date_	20161026
Time	16.09
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	35.9
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	6.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SFO1	300.1315007 MHz

F2 - Processing parameters

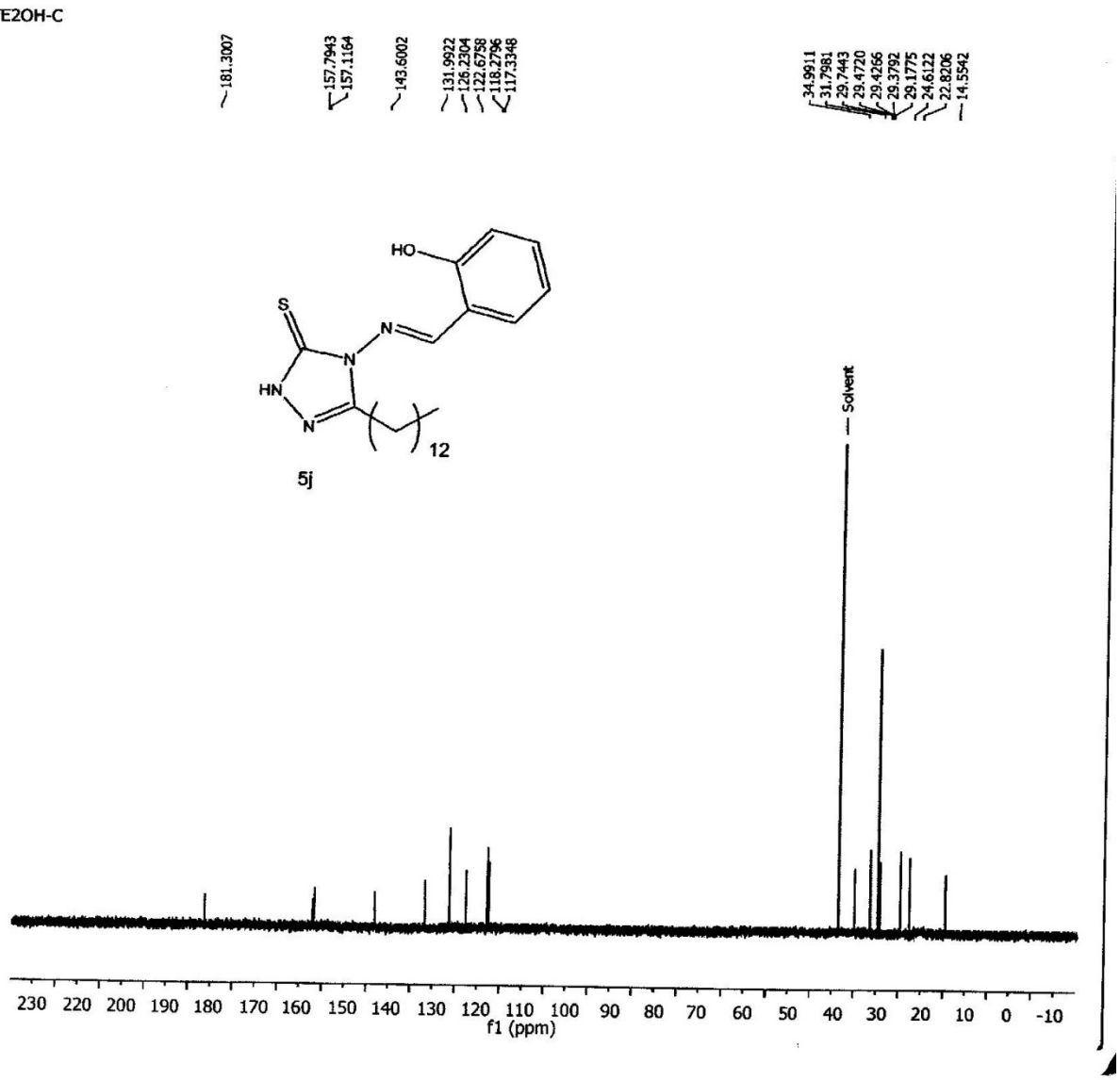
SI	32768
SF	300.1300043 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

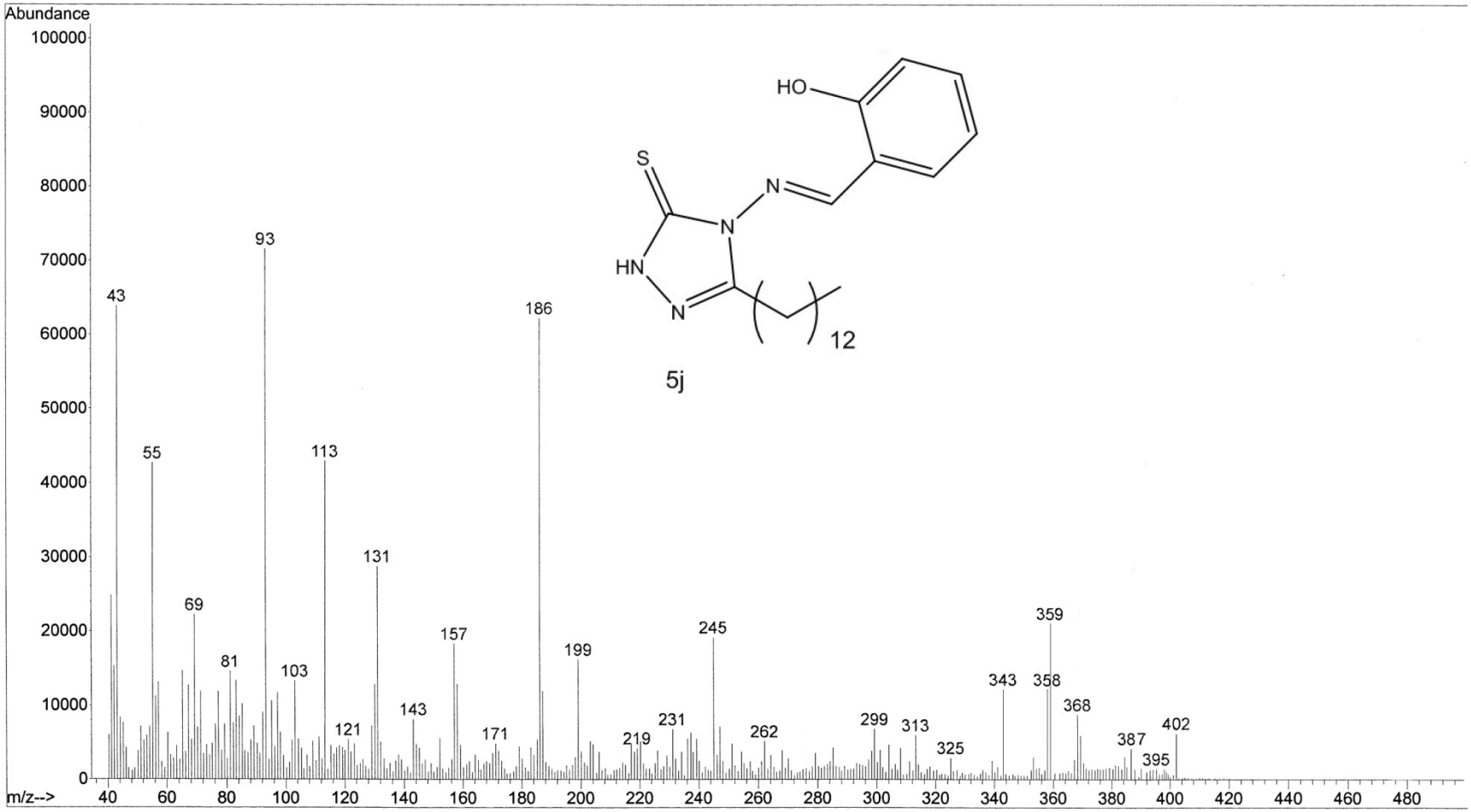
1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	14.000 ppm
F1	4201.82 Hz
F2P	-0.300 ppm
F2	-90.04 Hz
PPMCM	0.71500 ppm/cm
HZCM	214.59296 Hz/cm

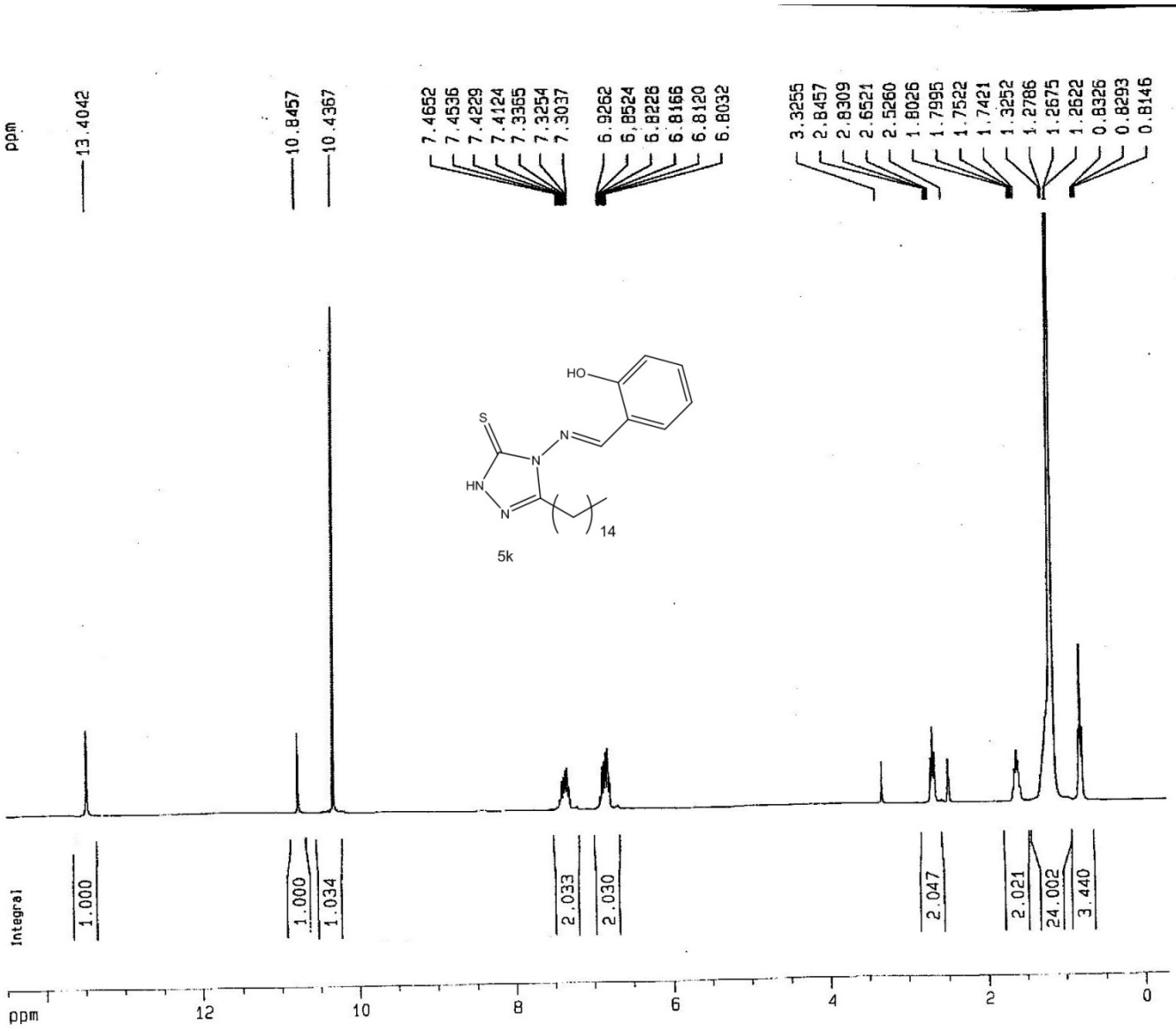
MTE2OH-C

	Parameter	Value
1	Data File Name	D:/ NMR/ 1397/ 97-10/ 97-10-26/ Kerman/ MTE2OH-C.fid/ fid
2	Title	MTE2OH-C
3	Comment	
4	Origin	Varian
5	Owner	
6	Site	
7	Instrument	inova
8	Author	
9	Solvent	dmso
10	Temperature	25.0
11	Pulse Sequence	s2pul
12	Experiment	1D
13	Probe	ID
14	Number of Scans	1024
15	Receiver Gain	60
16	Relaxation Delay	1.0000
17	Pulse Width	7.5000
18	Presaturation Frequency	
19	Acquisition Time	1.0420
20	Acquisition Date	2019-01-16T16:13:21
21	Modification Date	2019-01-16T16:17:34
22	Class	
23	Spectrometer Frequency	125.67
24	Spectral Width	31501.2
25	Lowest Frequency	-1833.0
26	Nucleus	13C
27	Acquired Size	32768
28	Spectral Size	65536




Abundance
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
m/z-->
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
43
55
69
81
93
103
113
121
131
143
157
171
186
199
219
231
245
262
299
313
325
343
358
359
368
387
395
402
HO
S
N
N
HN
N
12
5j

BRUKER
Transmittance [%]
Wavenumber cm-1
3974.38
3875.13
3703.42
3672.11
3105.21
3065.60
2917.56
2850.56
1620.14
1597.09
1509.45
1467.52
1416.67
1286.94
1261.07
1232.83
1153.25
900.63
759.40
686.95
594.72
477.82
HO
S
N
HN
N
14
5k



Current Data Parameters

NAME	kerman
EXPNO	515
PROCNO	1

F2 - Acquisition Parameters

Date_	20170715
Time	12.11
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	42.2
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	6.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SFO1	300.1315007 MHz

F2 - Processing parameters

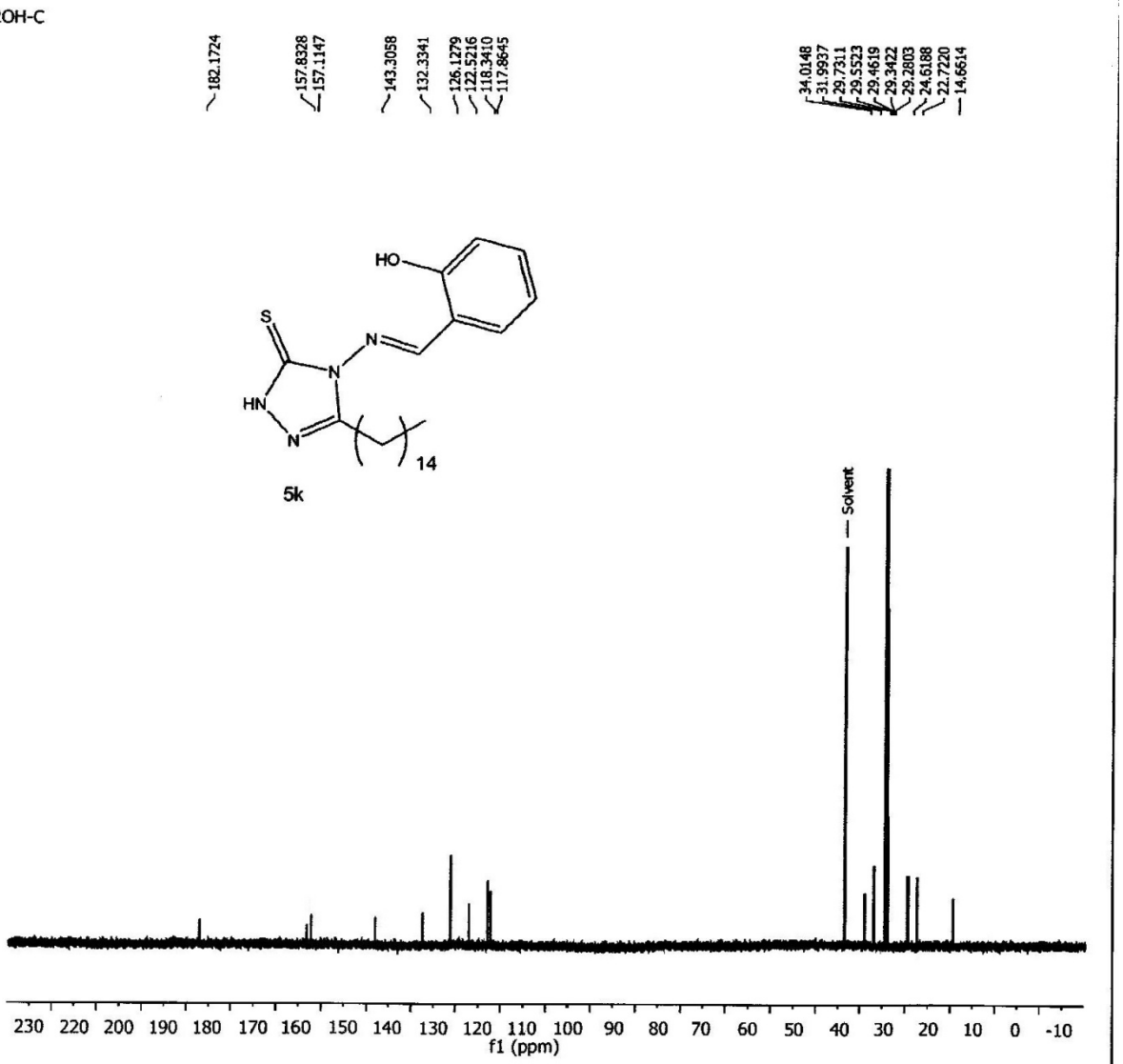
SI	32768
SF	300.1300067 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

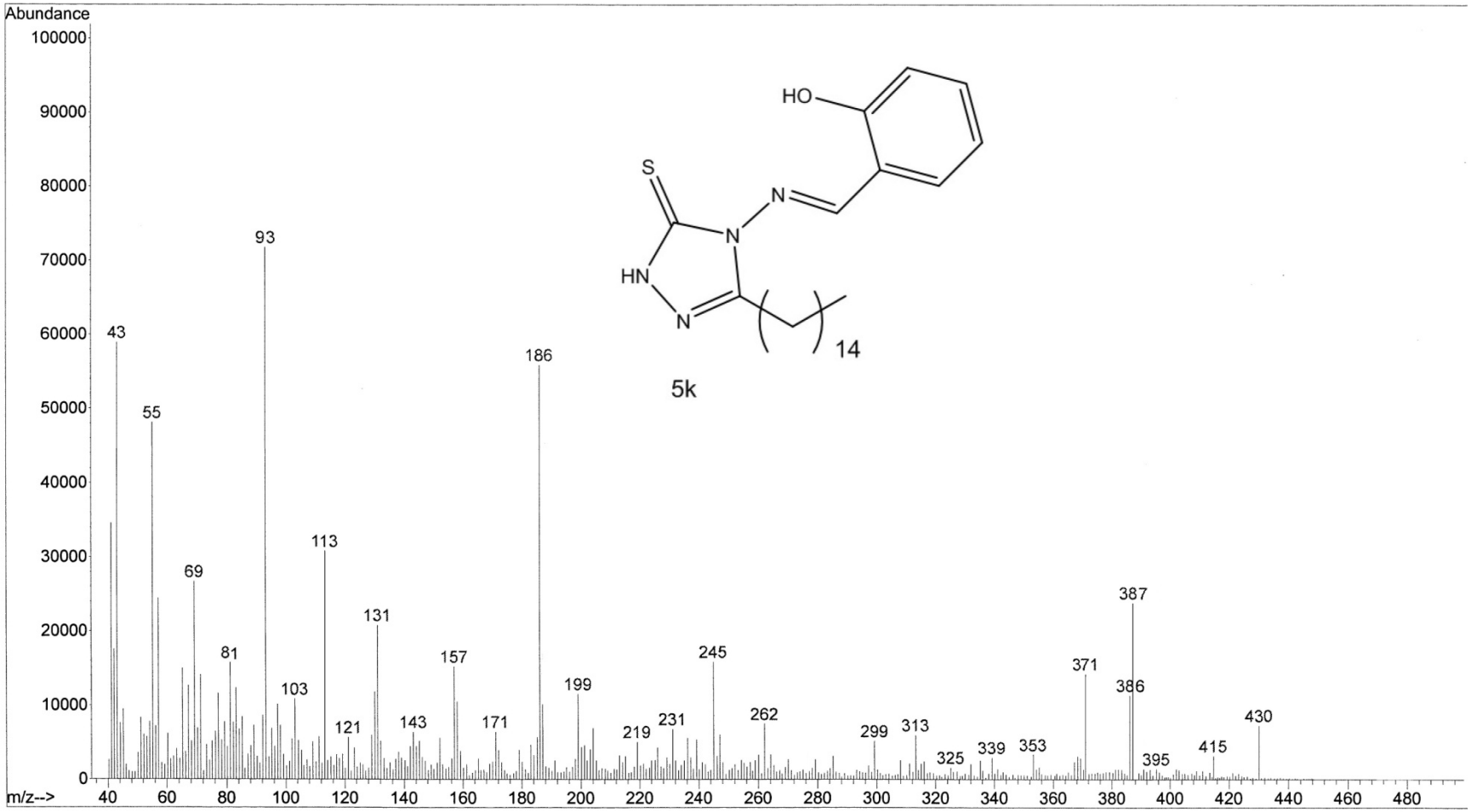
1D NMR plot parameters

CX	20.00 cm
CY	12.55 cm
F1P	14.530 ppm
F1	4352.20 Hz
F2P	-0.300 ppm
F2	-90.04 Hz
PPMCM	0.74003 ppm/
HZCM	223.09614 Hz/

PTE2OH-C

	Parameter	Value
1	Data File Name	D:/ NMR/ 1397/ 97-10/ 97-10-26/ Kerman/ PTE2OH-C.fid/ fid
2	Title	PTE2OH-C
3	Comment	
4	Origin	Varian
5	Owner	
6	Site	
7	Instrument	inova
8	Author	
9	Solvent	dmso
10	Temperature	25.0
11	Pulse Sequence	s2pul
12	Experiment	1D
13	Probe	ID
14	Number of Scans	1024
15	Receiver Gain	60
16	Relaxation Delay	1.0000
17	Pulse Width	7.5000
18	Presaturation Frequency	
19	Acquisition Time	1.0398
20	Acquisition Date	2019-01-16T17:01:08
21	Modification Date	2019-01-16T17:05:11
22	Class	
23	Spectrometer Frequency	125.67
24	Spectral Width	31410.6
25	Lowest Frequency	-1833.0
26	Nucleus	13C
27	Acquired Size	32768
28	Spectral Size	65536




Abundance
100000
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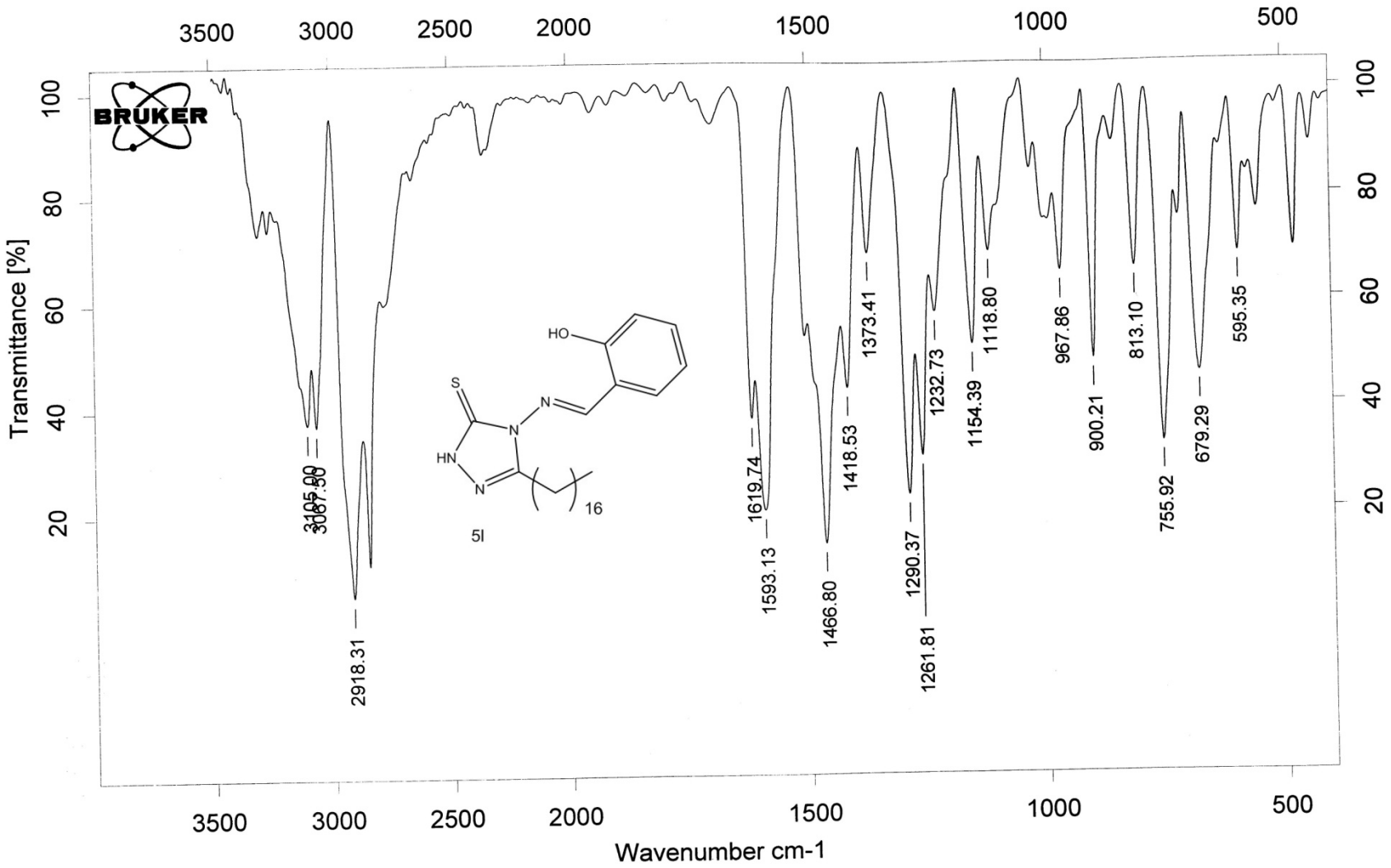
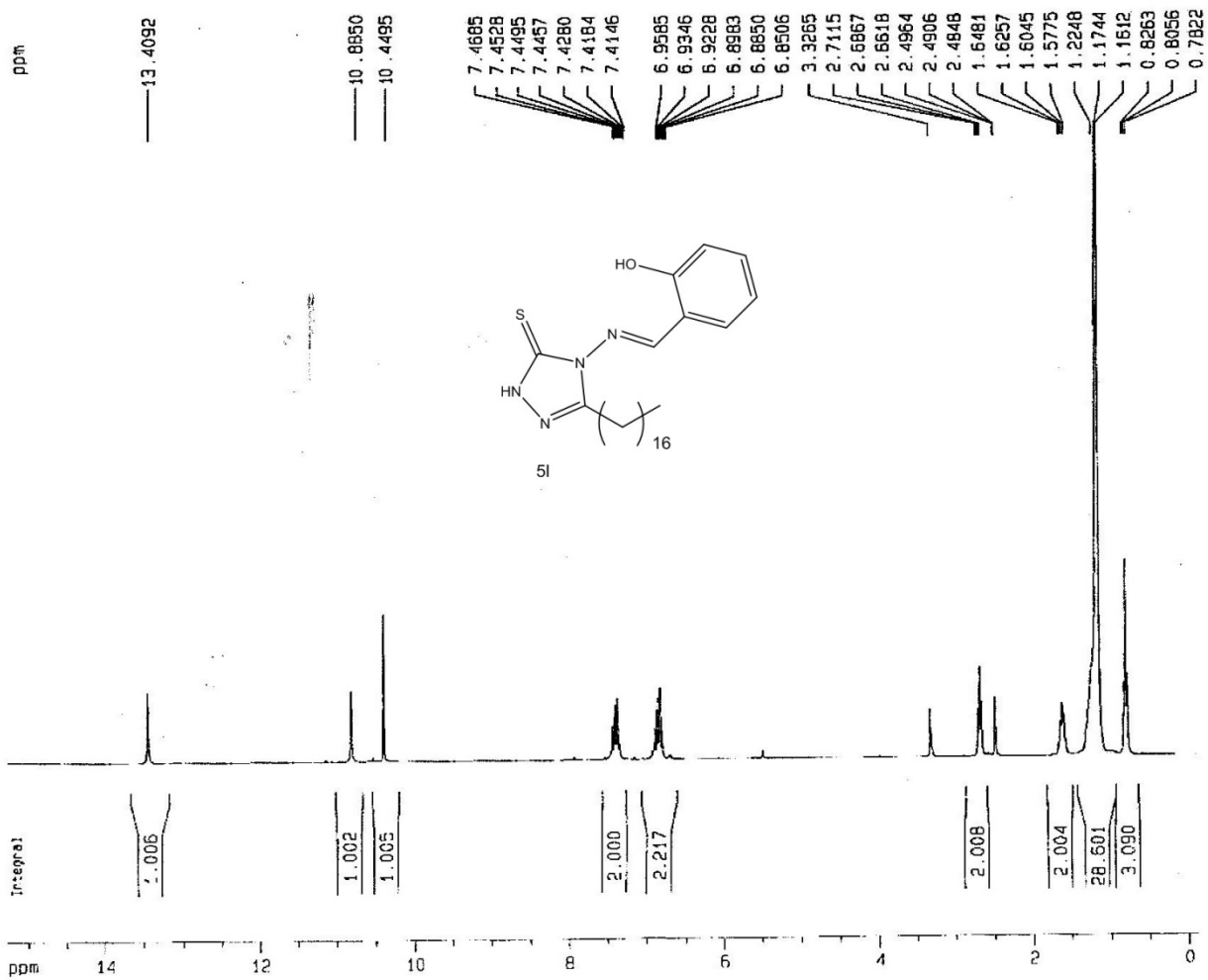
80000
70000
60000
50000
40000
30000
20000
10000
0
m/z-->
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
43
55
69
81
93
103
113
121
131
143
157
171
186
199
219
231
245
262
299
313
325
339
353
371
386
387
395
415
430
HO
S
N
N
HN
N
14
5k

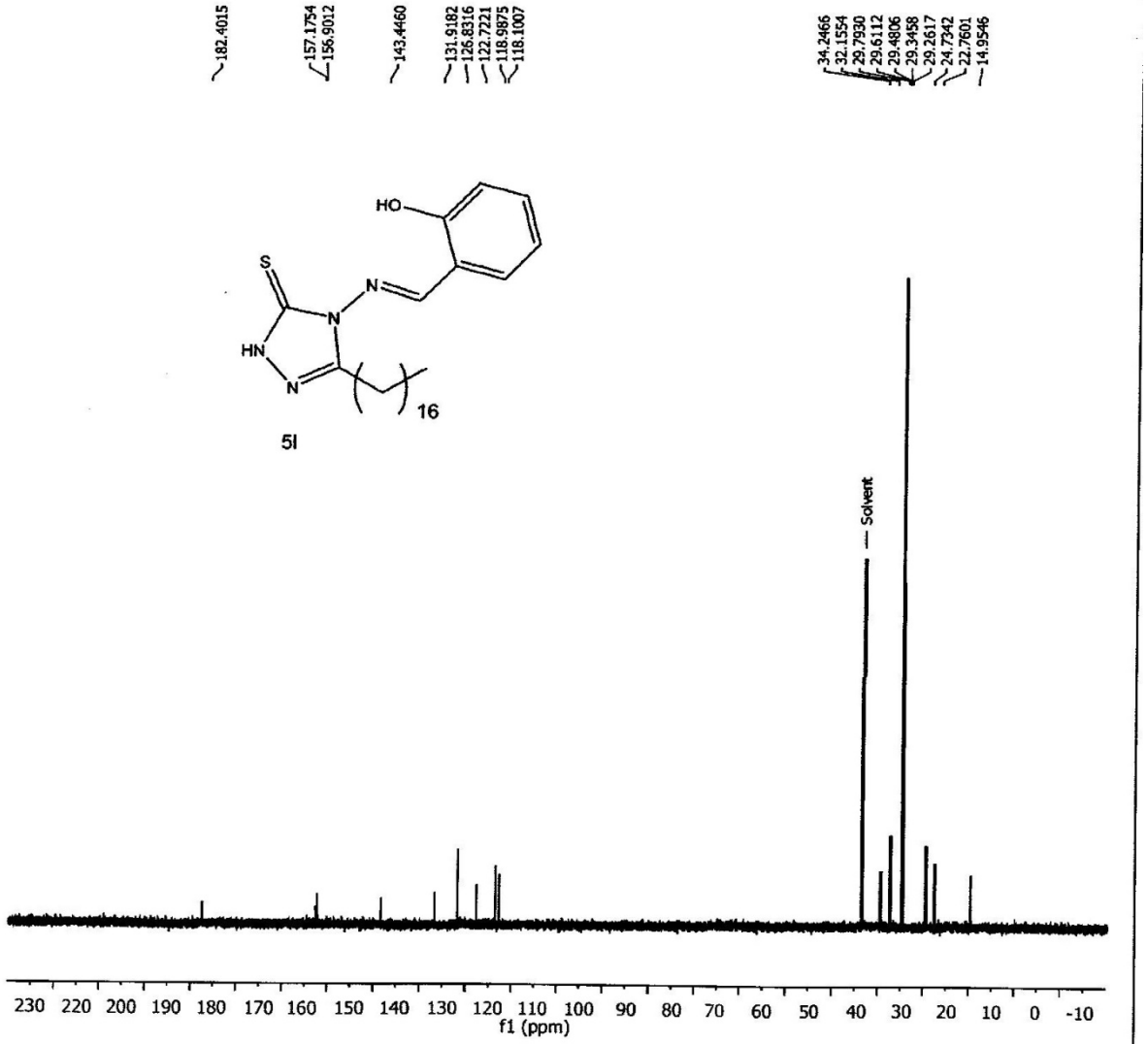

BRUKER
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
100
80
60
40
20
3105.00
3087.30
2918.31
1619.74
1593.13
1466.80
1418.53
1373.41
1290.37
1261.81
1232.73
1154.39
1118.80
967.86
900.21
813.10
755.92
679.29
595.35
HO
S
N
HN
N
16
5l

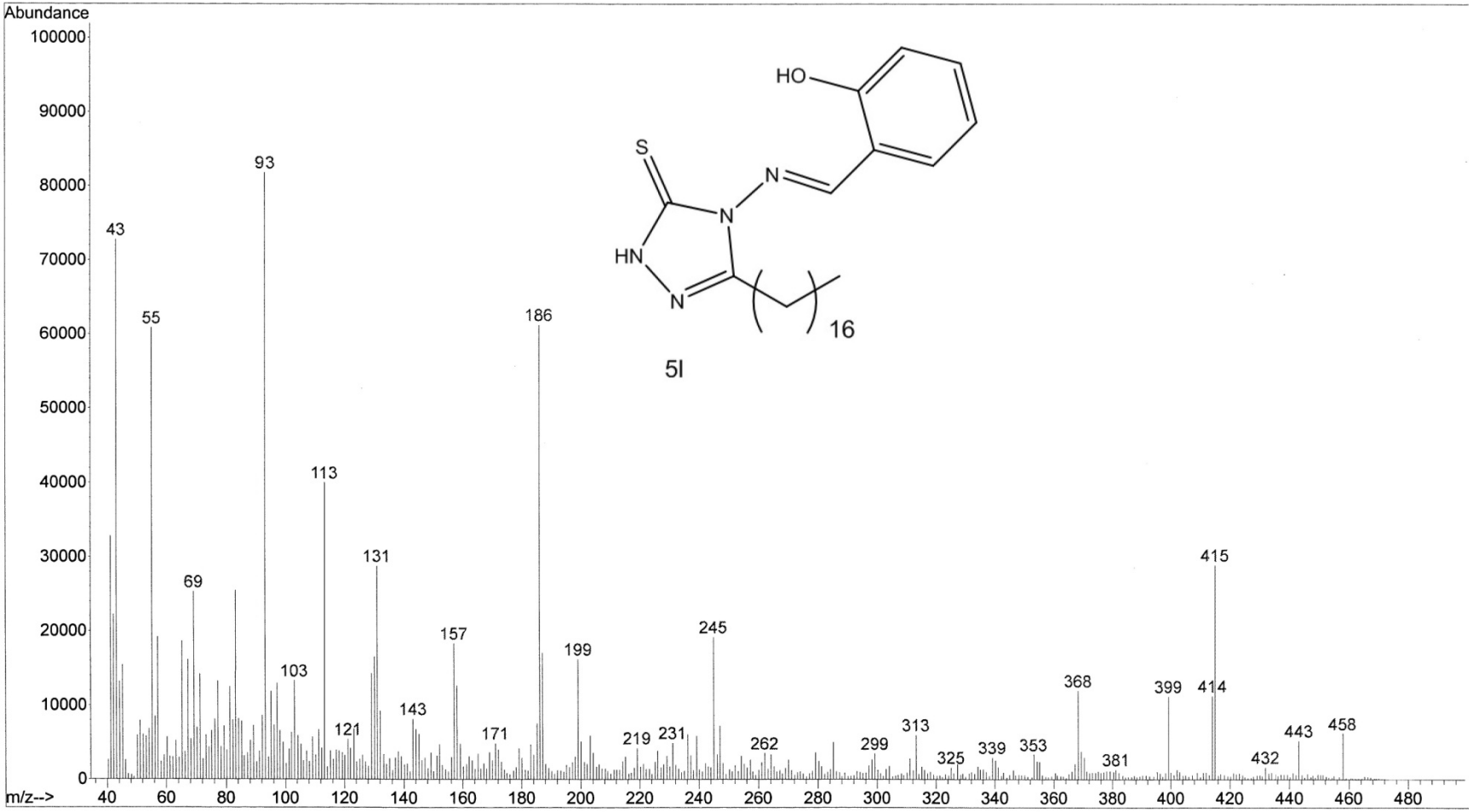



```
Current Data Parameters
NAME                  kerman
EXPNO                    520
PROCNO                     1

F2 - Acquisition Parameters
Date_              20170530
Time                  18.11
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG                  zg
TD                    32768
SOLVENT                DMSO
NS                       16
DS                        0
SWH                6172.839 Hz
FIDRES             0.188380 Hz
AQ                 2.6542580 sec
RG                      50.8
DW                 81.000 usec
DE                  6.00 usec
TE                  300.0 K
D1                 6.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                  10.00 usec
PL1                  0.00 dB
SFO1           300.1315007 MHz

F2 - Processing parameters
SI                    32768
SF                300.1300039 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   12.50 cm
F1P                 15.272 ppm
F1                 4583.50 Hz
F2P                -0.300 ppm
F2                 -90.04 Hz
PPMCM               0.77859 ppm/cm
HZCM              233.67674 Hz/cm
```

STEB-C

	Parameter	Value
1	Data File Name	D:/ NMR/ 1397/ 97-10/ 97-10-26/ Kerman/ STEB-C.fid/ fid
2	Title	STEB-C
3	Comment	
4	Origin	Varian
5	Owner	
6	Site	
7	Instrument	inova
8	Author	
9	Solvent	dmso
10	Temperature	25.0
11	Pulse Sequence	s2pul
12	Experiment	1D
13	Probe	ID
14	Number of Scans	1024
15	Receiver Gain	60
16	Relaxation Delay	1.0000
17	Pulse Width	7.5000
18	Presaturation Frequency	
19	Acquisition Time	1.0472
20	Acquisition Date	2019-01-16T17:21:14
21	Modification Date	2019-01-16T17:25:26
22	Class	
23	Spectrometer Frequency	125.67
24	Spectral Width	31428.5
25	Lowest Frequency	-1833.0
26	Nucleus	13C
27	Acquired Size	32768
28	Spectral Size	65536




Abundance
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60000
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30000
20000
10000
0
m/z-->
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
43
55
69
93
103
113
121
131
143
157
171
186
199
219
231
245
262
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325
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353
368
381
399
414
415
432
443
458
HO
S
N
N
HN
N
16

5l