Short communication

Efficient Synthesis of Benzopyrans and Dihydropyranochromenes Catalyzed by Poly(4-vinylpyridine) as a Green and Commercially Available Basic Catalyst

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Received: 15-05-2013

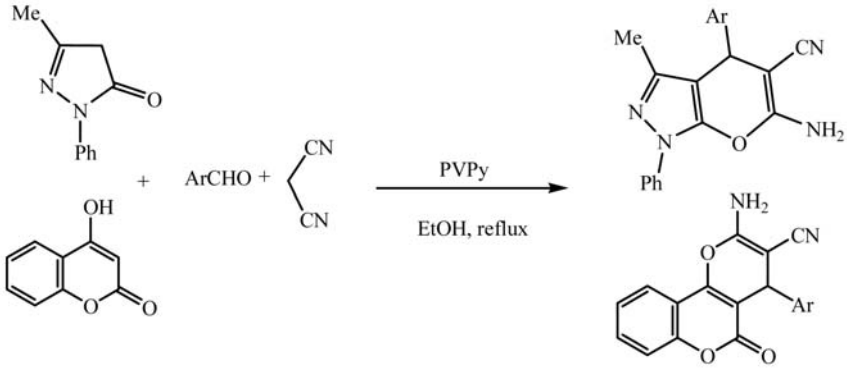
Abstract

Poly(4-vinylpyridine) is used as a green, commercially available and recyclable basic catalyst for the multicomponent synthesis of benzopyrans and dihydropyranochromenes by one-pot condensation of aromatic aldehydes, 3-methyl-1-phenyl-2-pyrazolin-5-one, and malononitrile or 4-hydroxycoumarin in ethanol at reflux temperature. This procedure provides several advantages such as mild reaction conditions, short reaction times, simple work-up and high yields.

Keywords: Poly(4-vinylpyridine), benzopyran, dihydropyranochromene, malononitrile, aldehyde, 3-methyl-1-phenyl-2-pyrazolin-5-one, 4-hydroxycoumarin.

1. Introduction

Benzopyrans and dihydropyranochromenes are considered as interesting heterocyclic compounds that have already received significant attentions because of their biological and pharmaceutical properties such as antisterility and anticancer activity.[1–2] The pyran pharmacophore is an important core structure of many natural products showing antibacterial, antitumor, antiallergic, antibiotic, hypolipidemic and immunomodulating activities.[3] Due to the great importance of dihydropyranochromenes and benzopyrans in recent years, various synthetic procedures have been developed for the preparation of these compounds.[4–27] However, some of these procedures suffer from one or more of the following disadvantages such as use of toxic solvents, tedious work-up procedure, long reaction times, low yields, use of corrosive reagents, effluent pollution, and non-recyclable catalysts. Therefore, there is a need to develop an alternative method for the synthesis of these compounds. In the condensation reaction, pyridine was used as a basic catalyst. It was especially suitable for the dehalogenation, where it acted as the base for the and bonds the resulting hydrogen halide to form a pyridinium salt. Nevertheless, pyridine is a highly flammable and toxic compound, and can be absorbed through the skin mucous membranes. Recenty, poly(4-vinylpyridine) (PVPy) has been used as a support for the numerous reagents and catalysts in many organic reaction transformations. It has been reported that PVPy as a basic catalyst can catalyze the synthesis of chromene derivatives.[28] Also, in the previous research, the application of PVPy for the protection of different types of functional groups has been reported.[29]As part of our research program to develop efficient and green methods, and catalysts in organic synthesis,[30–32] we wish to report the applicability of (PVPy) as a green, commercially available and recyclable basic catalyst for the synthesis of benzopyrans and dihydropyranochromenes in ethanol at reflux temeprature (Scheme 1).



Scheme 1. Synthesis of benzopyrans and dihydropyranochromenes catalyzed by PVPy.

2. Results and Discussion

PVPy is cheap and commercially available reagent, and its structure convinced us that this reagent could be used as an efficient, green and basic catalyst in the synthesis of dihydropyranochromenes and benzopyrans. At first the catalytic role of PVPy in the reaction of dihydropyranochromenes *via* three-component condensation of aldehydes, malononitrile and 4-hydroxycoumarin was examined. The best result was achieved by running the reaction of benzaldehyde, malononitrile and 4-hydroxycoumarin (with 1: 1: 1 mol ratio) in the presence of 0.1 g of PVPy in ethanol at reflux temperature (Table 1, entry 1).

Using the optimized conditions, the reaction of various aromatic aldehydes was explored without additional purification (Table 1). Similarly, benzopyrans were obtained by the condensation of aromatic aldehydes, malononitrile and 3-methyl-1-phenyl-2-pyrazolin-5-one in ethanol at reflux temperature (Table 2). According to the results of Table 1 and 2, different aromatic aldehydes with either electron-donating or electron-withdrawing groups, efficiently reacted to afford the desired products in good to high yields. It was also observed that aliphatic aldehydes remain intact under the same reaction conditions. All products were isolated with simple filtration and evaporation of the solvent. Solid products were easily recrystallized from hot ethanol in good to high yields during the short reaction times. All products have been identified by comparison of their melting points and analytical data (IR, NMR) with those reported for authentic samples. A distinct advantage of this method is the formation of corresponding products without by-products. The experimental procedure using PVPy as a catalyst is very simple and the catalyst can be recovered easily by filtration. Moreover, the applied procedure is environmentally friendly as it did not use any toxic auxiliary or solvent.

In order to exhibit the recyclability of the PVPy in the synthesis of dihydropyranochromenes, the reaction of benzaldehyde, 4-hydroxycoumarin and malononitrile was selected as a model. After completion of the reaction, the PVPy was washed with ethylacetate, dried and stored for another consecutive reaction run. This process was repeated for five runs and no significant decreasing in yield was

Table 1. Synthesis of dihydropyranochromene derivatives catalyzed by PVPy.

Entry	Substrate	Time (min)	Yield (%)[a]	Mp (°C)	
				Found	Reported
1	C_6H_5CHO	20	90	260–262	263–265[7]
2	2-ClC_6H_4CHO	18	90	242–244	240[7]
3	4-ClC_6H_4CHO	15	89	259–261	252–255[8]
4	2,4-$Cl_2C_6H_3CHO$	12	90	257–259	250–254[7]
5	3-$NO_2C_6H_4CHO$	12	90	251–252	248–250[5]
6	4-$NO_2C_6H_4CHO$	10	92	257–259	250–252[8]
7	4-MeC_6H_4CHO	20	89	255–257	259–260[8]
8	4-$MeOC_6H_4CHO$	32	87	228–229	226–230[8]
9	3,4,5-$(MeO)_3C_6H_2CHO$	60	86	274–276	276–278[7]
10	4-HOC_6H_4CHO	60	85	265–267	260–263[8]
11	4-BrC_6H_4CHO	20	89	250–252	244–246[7]
12	4-FC_6H_4CHO	20	90	262–263	258–261[8]

[a] Yields of pure isolated products.

Table 2. Synthesis of benzopyrans catalyzed by PVPy.

Entry	Substrate	Time (min)	Yield (%)[a]	Mp (°C)	
				Found	Reported
1	C_6H_5CHO	9	96	168–170	169–171[23]
2	4-$O_2NC_6H_4CHO$	7	92	193–195	196–198[23]
3	3-$O_2NC_6H_4CHO$	10	94	190–192	190–191[23]
4	2,4-$Cl_2C_6H_4CHO$	10	93	183–185	185–187[23]
5	4-ClC_6H_4CHO	10	93	176–178	175–177[23]
6	2-ClC_6H_4CHO	15	92	145–147	144–146[23]
7	3-ClC_6H_4CHO	12	92	157–159	158–159[23]
8	4-$MeOC_6H_4CHO$	35	87	172–174	174–176[23]
9	4-MeC_6H_4CHO	20	93	177–179	176–178[24]
10	4-FC_6H_4CHO	18	92	168–169	167–168[24]
11	4-HOC_6H_4CHO	25	89	208–210	211–212[24]
12	4-CNC_6H_4CHO	8	86	217–219	216–218[18]

[a] Yields of pure isolated products.

Table 3. The recyclability of PVPy in the synthesis of dihydropyranochromenes.[a]

Run	1	2	3	4	5
Time (min)	20	20	25	25	30
Yield (%)[b]	90	90	90	89	89

[a] Reaction conditions: benzaldehyde (1 mmol), 4-hydroxycoumarin (1 mmol), malononitrile (1 mmol), and catalyst (0.1 g) in ethanol at reflux temperature. [b] Yields of pure isolated products.

Table 4. The recyclability of PVPy in the synthesis of benzopyrans.[a]

Run	1	2	3	4	5	6
Tim (min)	9	9	10	12	12	15
Yield (%)[b]	96	95	95	92	92	90

[a] Reaction conditions: benzaldehyde (1 mmol), 3-methyl-1-phenyl-2-pyrazolin-5-one (1 mmol), malononitrile (1 mmol), and catalyst (0.1 g) in ethanol at reflux temperature. [b] Yields of pure isolated products.

Table 5. Comparison of the efficiency of various catalysts in the synthesis of dihydropyranochromenes from benzaldehyde.

Entry	Catalyst	Conditions	Time (min)	Yield (%)[a]	Reference
1	Nano ZnO	H_2O/70 °C	180	87	4
2	Trisodium citrate	H_2O-EtOH/reflux	40	65	5
3	Morpholine	H_2O/reflux	180	90	8
4	magnetic nano-organocatalyst	H_2O/reflux	10	78	9
5	PVPy	EtOH/reflux	20	90	This work

[a] Yields of pure isolated products.

observed. Almost consistent activity was observed over five runs and the desired products were obtained in high yields (Table 3).

The activity of the recovered catalyst was also examined in the synthesis of benzopyrans using benzaldehyde, 3-methyl-1-phenyl-2-pyrazolin-5-one and malononitrile under the optimized conditions. As shown in the Table 4, PVPy can be recycled up to 6 consecutive runs without any lost of its efficiency and the desired product was obtained in high yields.

In order to show the efficiency of method, Table 5 compares the results of the synthesis of dihydropyranochromenes with various catalysts. In comparison with previously reported methods, low amounts of PVPy efficiently promoted the reaction and gave the desired product in very short times and high yields. Moreover, the PVPy is cheap, easy to handle and commercially available. It can be simply recovered by filtration and reused in the next runs without significant decrease of catalytic activity. Finally, our method does not use any toxic auxiliary or solvent.

3. Experimental

All products were characterized by comparison of their spectroscopic data (NMR, IR) and physical properties with those reported in the literature. Chemicals were

purchased from Fluka and Merck chemical companies and used as received. IR spectra were recorded on a Perkin Elmer 781 spectrophotometer. All NMR spectra were recorded on a Bruker Avance 500 MHz spectrometer using tetramethylsilane (TMS) as an internal standard. Melting points were recorded on Bransted Electrothermal 9100BZ melting point apparatus.

3. 1. General Procedure for Synthesis of Dihydropyranochromenes and Benzopyrans

A mixture of aldehyde (1 mmol), malononitrile (1 mmol), 4-hydroxycoumarin or 3-methyl-1-phenyl-2-pyrazolin-5-one (1 mmol), and PVPy (0.1 g) in ethanol (10 mL) was stirred at reflux temperature. After completion of the reaction (moitored by TLC; n-hexane/ethyl acetate, 3:1), the catalyst was recovered by filtration to be reused subsequently, and the reaction mixture allowed to cool at room temperature. Evaporation of the solvent from the filtrate and recrystallization of the solid residue from hot ethanol afforded pure products in high yields. The structure of the products was identified by meltig points, IR, ^{1}H and ^{13}C NMR spectroscopy (see Supplementary Material), and compared with authentic samples prepared by reported methods.

The analytical and spectroscopic data for the known compounds are as follows:

Table 1, entry 1: IR (KBr): ν 3395, 3320, 3190, 2930, 2200, 1650, 1405, 1305, 1100 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6): δ 4.46 (s, 1H), 7.23–7.28 (m, 3H), 7.33 (t, 2H, J = 7.6 Hz), 7.41 (s, 2H), 7.45–7.52 (m, 2H), 7.72 (t, 1H, J = 7.6 Hz), 7.90 (d, 1H, J = 8 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6,): δ 37.5, 58.5, 104.5, 113.5, 117.1, 119.7, 123.0, 125.1, 127.6, 128.1, 129.0, 133.4, 143.8, 152.6, 153.9, 158.5, 160.0 ppm.

Table 1, entry 2: IR (KBr): ν 3415, 3300, 3195, 2910, 2205, 1655, 1405, 1315, 1105 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6): δ 4.74 (s, 1H, CH), 7.48 (t, 1H, J = 8.4 Hz), 7.52 (d, 1H, J = 7.6 Hz), 7.56 (s, 2H), 764 (t, 1H, J = 8 Hz), 7.72–7.76 (m, 1H), 7.81 (d, 1H, J = 7.6 Hz), 8.12 (s, 1H), 8.14 (s, 1H) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 37.1, 57.4, 103.4, 113.4, 117.1, 119.4, 122.8, 122.9, 123.1, 125.2, 130.6, 133.6, 135.3, 146.0, 148.3, 152.8, 154.4, 158.6, 160.1 ppm.

Table 1, entry 3: IR (KBr): ν 3420, 3320, 3195, 2920, 2110, 1675, 1470, 1345, 1150 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6) δ 4.49 (s, 1H,), 7.31 (d, 2H, J = 8.4 Hz), 7.36 (d, 2H, J = 8.4 Hz), 7.44–7.50 (m, 4H), 7.70 (t, 1H, J = 7.6 Hz), 7.89 (d, 1H, J = 7.6 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 36.9, 58.0, 104.0, 113.4, 117.0, 119.6, 123.0, 125.1, 128.9, 130.1, 132.2, 133.5, 142.8, 152.7, 154.0, 158.5, 160.0 ppm.

Table 1, entry 4: IR (KBr): ν 3320, 3315, 3190, 2930, 2100, 1680, 1465, 1345, 1150 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6): δ 4.99 (s, 1H), 7.35–7.41 (m, 2H), 7.48–7.54 (m, 4H), 7.59 (m, 1H), 7.72–7.76 (m, 1H), 7.90 (m, 2H, J = 8 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 34.4, 56.5, 103.0, 113.3, 117.2, 119.2, 123.0, 125.2, 128.4, 129.3, 132.6, 132.9, 133.6, 133.8, 139.9, 152.7, 154.6, 158.6, 159.9 ppm.

Table 1, entry 5: IR (KBr): ν 3315, 3310, 3195, 2920, 2200, 1655, 1475, 1325, 1100 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6): δ 4.98 (s, 1H), 7.25–7.29 (m, 2H), 7.31–7.34 (m, 1H), 7.41–7.52 (m, 5H), 7.70–7.74 (m, 1H), 7.91–7.93 (m, 1H) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 34.8, 57.0, 103.4, 113.3, 117.1, 119.3, 123.0, 125.2, 128.2, 129.3, 130.1, 131.1, 132.9, 133.5, 140.7, 152.7, 154.5, 158.6, 159.9 ppm.

Table 1, entry 6: IR (KBr): ν 3410, 3310, 3195, 2930, 2210, 1655, 1475, 1335, 1050 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6): δ 4.69 (s, 1H), 7.47–7.62 (m, 6H), 7.74 (t, 1H, J = 8 Hz), 7.92 (d, 1H, J = 8 Hz), 8.18 (d, 2H, J = 8.8 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 37.3, 57.3, 103.3, 113.4, 117.1, 119.4, 123.1, 124.2, 125.2, 129.7, 133.7, 147.1, 151.2, 152.8, 154.4, 158.5, 160.1 ppm.

Table 1, entry 7: IR (KBr): ν 3415, 3315, 3205, 2910, 2215, 1650, 1405, 1305, 1015 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6): δ 1.33 (3H), 4.50 (s, 1H), 7.16 (d, 2H, J = 8.4 Hz), 7.38 (d, 2H, J = 8.8 Hz), 7.43 (s, 2H), 7.46–7.52 (m, 2H), 7.68–7.74 (m, 1H), 7.92 (d, 1H, 2H, J = 7.6 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 32.1, 35.6, 58.6, 103.7, 110.2, 112.3, 115.1, 117.7, 120.8, 124.0, 126.1, 131.3, 133.7, 150.4, 150.5, 155.3, 155.7, 160.1 ppm.

Table 1, entry 8: IR (KBr): ν 3415, 3310, 3200, 2920, 2210, 1650, 1405, 1305, 1010 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6): δ 3.73 (s, 3H), 4.73 (s, 1H), 6.87 (d, 2H, J = 7.8 Hz), 7.18 (d, 2H, J = 7.8 Hz), 7.37 (s, 2H), 7.48 (m 2H), 7.71 (m, 1H), 7.91 (m, 1H) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 36.7, 55.5, 58.7, 104.8, 113.5, 114.3, 117.0, 119.8, 122.9, 125.1, 129.2, 133.3, 135.9, 152.6, 153.6, 158.4, 158.8, 160.0 ppm.

Table 1, entry 9: IR (KBr): ν 3410, 3320, 3205, 2980, 1996, 1670, 1415, 1305, 1010 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6): δ 3.64 (s, 3H, OCH_3), 3.73 (s, 6H), 4.45 (s, 1H), 6.54 (s, 2H), 7.38 (s, 2H), 747–7.52 (m, 2H), 7.70–7.74 (m, 1H), 7.90 (d, 1H, J = 7.2 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 37.7, 56.4, 58.4, 60.4, 104.2, 105.5, 113.6, 117.1, 119.7, 123.0, 125.1, 133.4, 137.1, 139.5, 152.7, 153.3, 154.0, 158.5, 160.1 ppm.

Table 1, entry 10: IR (KBr): ν 3410, 3320, 3205, 2980, 1996, 1670, 1415, 1305, 1010 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6): δ 4.34 (s, 1H), 6.69 (d, 2H, J = 8.4 Hz), 7.04 (d, 2H, J = 8.4 Hz), 7.34 (s, 2H), 744–7.50 (m, 2H), 7.70 (t, 1H, J = 7.6 Hz), 7.88 (d, 1H, J = 7.6 Hz), 9.36 (s, 1H) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 36.6, 58.9, 105.0, 113.5, 115.7, 117.0, 119.9, 122.9, 125.1, 129.2, 133.3, 134.2, 152.5, 153.5, 157.0, 158.4, 160.0 ppm.

Table 1, entry 11: IR (KBr): ν 3410, 3320, 3210, 2920, 2210, 1655, 1405, 1305, 1000 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ 4.48 (s, 1H), 7.26 (d, 2H, J = 7.8 Hz), 7.49 (m, 6H), 7.71 (m, 1H), 7.90 (d, 1H, J = 7.8 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 37.0, 57.9, 103.9, 113.4, 117.1, 119.6, 120.7, 123.0, 125.2, 130.5, 131.8, 133.5, 143.0, 152.7, 154.0, 158.4, 160.0 ppm.

Table 1, entry 12: IR (KBr): ν 3335, 3310, 3190, 2920, 2115, 1675, 1475, 1345, 1150 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ 4.48 (s, 1H), 7.35 (d, 2H, J = 8.4 Hz), 7.39 (d, 2H, J = 8.4 Hz), 7.45–7.51 (m, 4H), 7.70 (t, 1H, J = 7.6 Hz), 7.88 (d, 1H, J = 7.6 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 36.9, 59.1, 103.8, 113.4, 116.8, 119.4, 122.8, 125.2, 128.9, 130.2, 132.3, 132.9, 142.7, 152.6, 154.1, 158.4, 160.1 ppm.

Table 2, entry 1: IR (KBr): ν 3390, 3305, 3200, 29005, 2190, 1655, 1405, 1300, 1010 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 1.79 (s, 3H), 4.78 (s, 2H), 5.29 (s, 1H), 7.22–7.27 (m, 3H), 7.30 (t, 2H, J = 7.8 Hz), 7.32 (s, 2H), 7.35 (m, 1H), 7.46 (m, 2H), 7.75 (d, 2H, J = 7.8 Hz) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 12.8, 32.4, 60.8, 96.7, 117.4, 120.3, 125.9, 127.1, 128.4, 129.3, 130.4, 132.8, 136.3, 136.8, 142.9, 145.3, 158.7 ppm.

Table 2, entry 2: IR (KBr): ν 3320, 3080, 2200, 1675, 1590, 1455, 1335, 1255, 1125, 1070, 1020 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ 1.75 (s, 3H), 4.72 (s, 1H), 7.45–7.56 (m, 3H), 7.61–7.70 (m, 4H), 7.82 (d, 2H, J = 8.6 Hz), 7.88 (d, 2H, J = 8.0 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.5, 37.5, 58.1, 98.5, 110.8, 119.6, 120.0, 120.9, 126.9, 129.8, 130.1, 133.8, 138.6, 145.0, 145.9, 150.1, 160.5 ppm.

Table 2, entry 3: IR (KBr): ν 3325, 3085, 2200, 1675, 1590, 1475, 1335, 1250, 1120, 1100, 1020 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ 1.79 (s, 3H), 4.80 (s, 1H), 7.30–7.55 (m, 5H), 7.40–7.8 (m, 5H), 7.92 (m, 1H, Ar), 7.98 ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.4, 37.5, 57.9, 99.5, 111.2, 119.6, 121.6, 121.9, 127.2, 129.5, 130.2, 132.9, 137.8, 144.5, 146.9, 150.2, 160.4 ppm.

Table 2, entry 4: IR (KBr): ν 3400, 3300, 3200, 2900, 2200, 1645, 1400, 1300, 1000 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 1.90 (s, 3H), 4.80 (s, 2H), 5.30 (s, 1H), 7.17 (d, 1H, J = 8.4 Hz), 7.26 (s, 1H), 7.35 (t, 1H, J = 7.2 Hz), 7.46–7.51 (m, 3H), 7.66 (d, 2H, J = 8 Hz) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 12.8, 33.5, 61.9, 97.6, 118.6, 121.3, 127.0, 128.0, 129.4, 129.7, 131.5, 133.9, 137.4, 137.9, 144.0, 146.1, 158.9 ppm.

Table 2, entry 5: IR (KBr): ν 3289, 3082, 2200, 1675, 1585, 1510, 1390, 1230, 1120, 1070, 1030 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ 1.75 (s, 3H), 4.82 (s, 1H), 7.25–7.30 (m, 3H), 7.45–7.51 (m, 4H), 7.75 (d, 2H, J = 8.6 Hz), 7.80 (d, 2H, J = 8.4 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.4, 37.5, 57.9, 97.9, 110.8, 118.9, 120.6, 120.9, 127.1, 129.8, 130.1, 133.5, 138.0, 144.9, 146.1, 150.1, 160.3 ppm.

Table 2, entry 6: IR (KBr): ν 3305, 3300, 3200, 2900, 2200, 1645, 1400, 1300, 1000 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 1.89 (s, 3H), 4.82 (s, 2H), 5.32 (s, 1H), 7.17–7.39 (m, 5H), 7.42–7.45 (m, 2H), 7.56 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 12.8, 32.8, 61.0, 97.5, 117.2, 120.3, 125.7, 126.9, 128.4, 128.9, 130.5, 132.8, 136.4, 136.4, 141.9, 145.6, 158.6 ppm.

Table 2, entry 7: IR (KBr): ν 3250, 3100, 2205, 1680, 1570, 1220, 1120, 1100, 1020 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ 1.78 (s, 3H), 4.82 (s, 1H), 7.15–7.21 (m, 5H), 7.30–7.38 (m, 3H), 7.42 (s, 2H), 7.78 (m, 1H) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.4, 37.5, 57.9, 99.5, 111.2, 119.6, 121.6, 121.9, 127.2, 129.5, 130.2, 132.9, 137.8, 144.5, 146.9, 150.2, 160.4 ppm.

Table 2, entry 8: IR (KBr): ν 3410, 3310, 3205, 2910, 2215, 1675, 1405, 1315, 1015 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ 1.79 (s, 3H), 3.75 (3H), 4.50 (s, 1H), 7.20 (s, 2H), 7.31 (t, 1H, J = 7.4 Hz), 7.30 (d, 2H, J = 8 Hz), 7.42 (d, 2H, J = 8 Hz), 7.46 (t, 2H, J = 7.6 Hz), 7.70 (d, 2H, J = 7.6 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.4, 31.8, 37.5, 57.9, 98.5, 110.8, 119.0, 120.6, 120.9, 127.1, 129.8, 131.1, 134.2, 138.3, 145.3, 145.9, 150.2, 160.2 ppm.

Table 2, entry 9: IR (KBr): ν 3380, 3312, 3200, 2910, 2215, 1670, 1415, 1315, 1010 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ 1.78 (s, 3H), 1.85 (3H), 4.55 (s, 1H), 7.12 (s, 2H), 7.18 (t, 1H, J = 7.4 Hz), 7.21 (d, 2H, J = 8 Hz), 7.28 (d, 2H, J = 8 Hz), 7.36 (t, 2H, J = 7.6 Hz), 7.60 (d, 2H, J = 7.6 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.4, 36.7, 37.5, 56.9, 97.4, 110.1, 116.8, 119.5, 120.4, 125.2, 128.7, 130.1, 133.1, 137.3, 144.3, 145.6, 150.2, 160.2 ppm.

Table 2, entry 10: IR (KBr): ν 3315, 3010, 2210, 1670, 1580, 1510, 1235, 1120, 1075, 1030 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ 1.74 (s, 3H), 4.82 (s, 1H), 7.32–7.40 (m, 3H), 7.48–7.55 (m, 4H), 7.87 (d, 2H, J = 8.6 Hz), 7.92 (d, 2H, J = 8.6 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.4, 37.6, 57.9, 97.9, 110.8, 118.9, 120.7, 121.0, 128.1, 129.8, 130.1, 133.5, 138.6, 145.7, 146.2, 151.2, 160.4 ppm.

Table 2, entry 11: IR (KBr): ν 3400, 3300, 3100, 2200, 1645, 1400, 1300, 1000 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ 1.79 (s, 3H), 4.55 (s, 1H), 6.71 (d, 2H, J = 7.8 Hz), 7.03 (d, 2H, J = 7.81 Hz), 7.2 (s, 2H), 7.31 (t, 1H, J = 7.03 Hz), 7.48 (t, 2H, J = 7.31 Hz), 7.77 (d, 2H, J = 7.74 Hz), 9.32 (s, 1H) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.4, 37.5, 57.9, 98.5, 110.8, 119.6, 120.6, 120.9, 127.1, 129.8, 130.1, 133.5, 138.3, 144.9, 145.9, 150.0, 160.0 ppm.

Table 2, entry 12: IR (KBr): ν 3089, 3082, 2200, 2215, 1673, 1590, 1515, 1390, 1250, 1120, 1070, 1030 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ 1.77 (s, 3H), 4.83 (s, 1H), 7.30–7.33 (m, 3H), 7.47–7.50 (m, 4H), 7.78 (d, 2H, J = 8.5 Hz), 7.82 (d, 2H, J = 8.0 Hz) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.4, 37.5, 57.9, 98.5, 110.8, 119.6, 120.7, 120.9, 127.1, 129.8, 130.1, 133.6, 138.3, 144.9, 145.9, 150.1, 160.5 ppm.

4. Conclusions

We have developed a mild, simple and green procedure for the one-pot synthesis of benzopyrans and dihydropyranochromenes in the presence of PVPy as a commercially available and recyclable basic catalyst at reflux temperature. Moreover, short reaction times, ease of work-up, high yields and clean procedure are the most important advantages of this method, making the procedure a useful addition to the available methods.

5. Acknowledgement

We are thankful to research council of Behbahan Khatam Alanbia University of Technology, for the support of this work.

6. References

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Povzetek

V prispevku je opisana uporaba poli(4-vinilpiridina) kot zelenega, komercialno dosegljivega in obnovljivega bazičnega katalizatorja za multikomponentno sintezo benzopiranov in dihidropiranokromenov z enostopenjsko kondenzacijo aromatskih aldehidov, 3-metil-1-fenil-2-pirazolin-5-ona in malononitrila, oziroma 4-hidroksikumarina, v etanolu pri temperaturi refluksa. Prednosti tega postopka so mili reakcijski pogoji, kratki reakcijski časi, enostavna izolacija produktov in visoki izkoristki.

Supporting Materials

Efficient Synthesis of Benzopyrans and Dihydropyranochromenes Catalyzed by Poly(4-vinylpyridine) as a Green and Commercially Available Basic Catalyst

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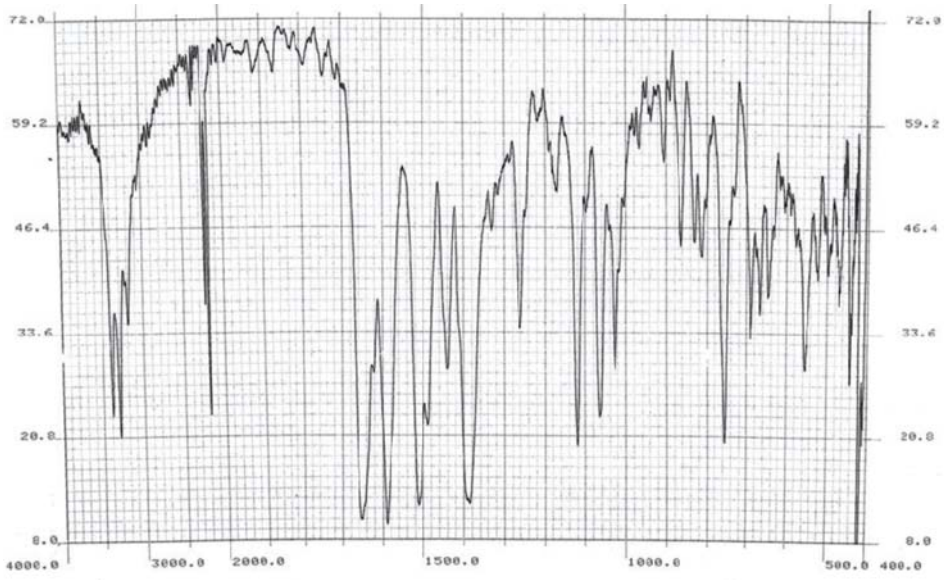
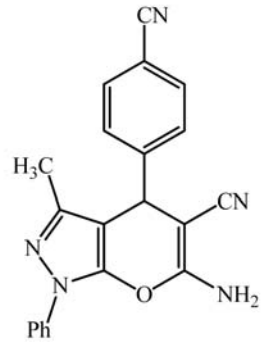
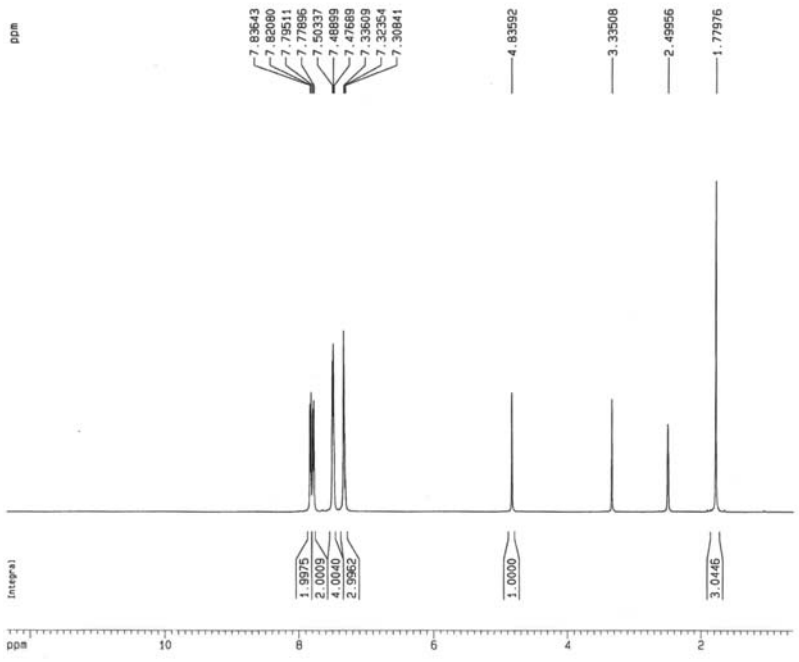
Received: 15-05-2013





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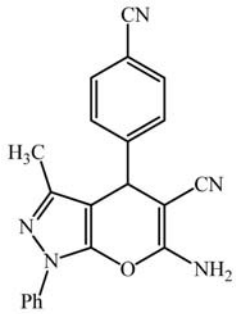

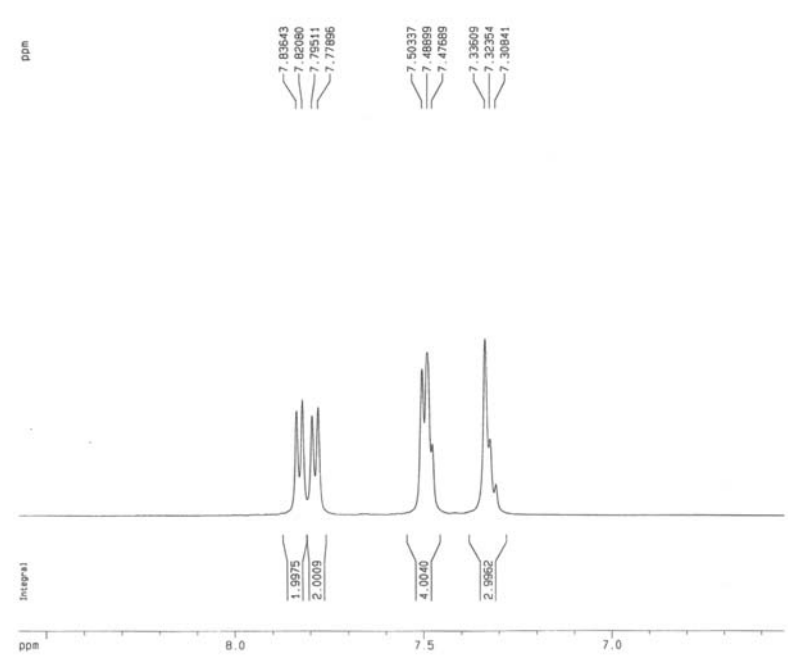


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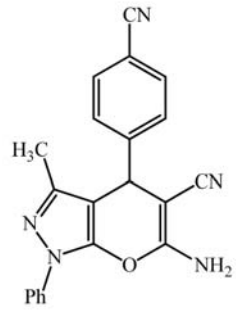

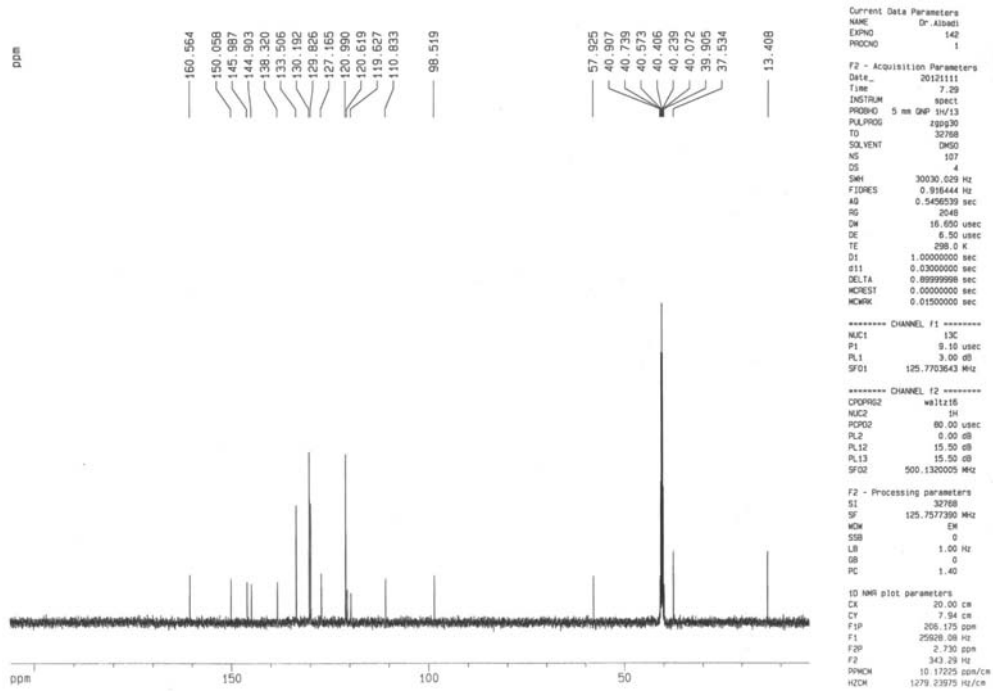
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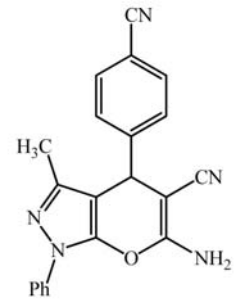
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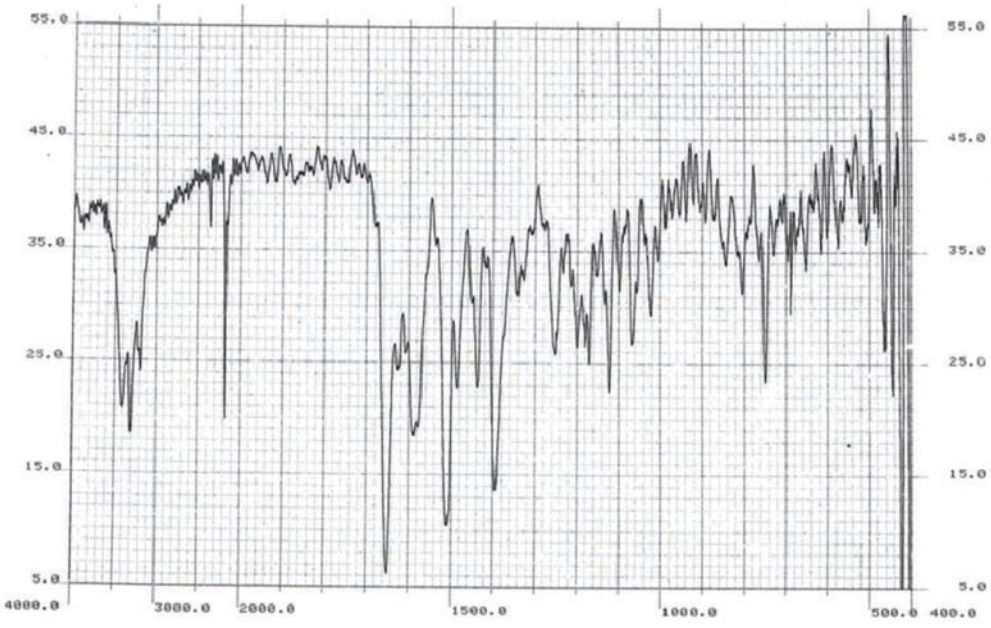
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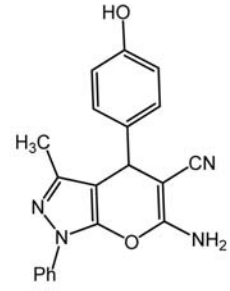
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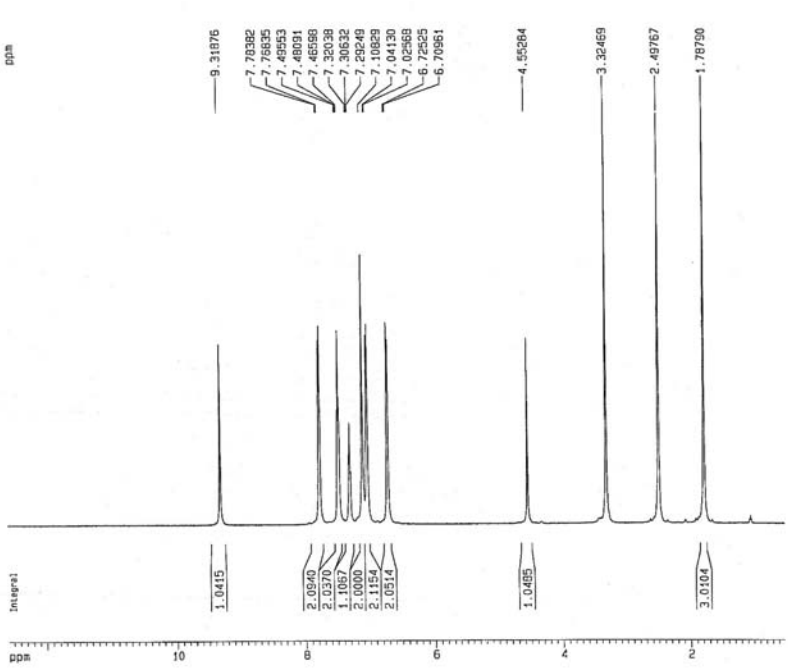


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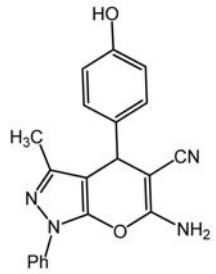

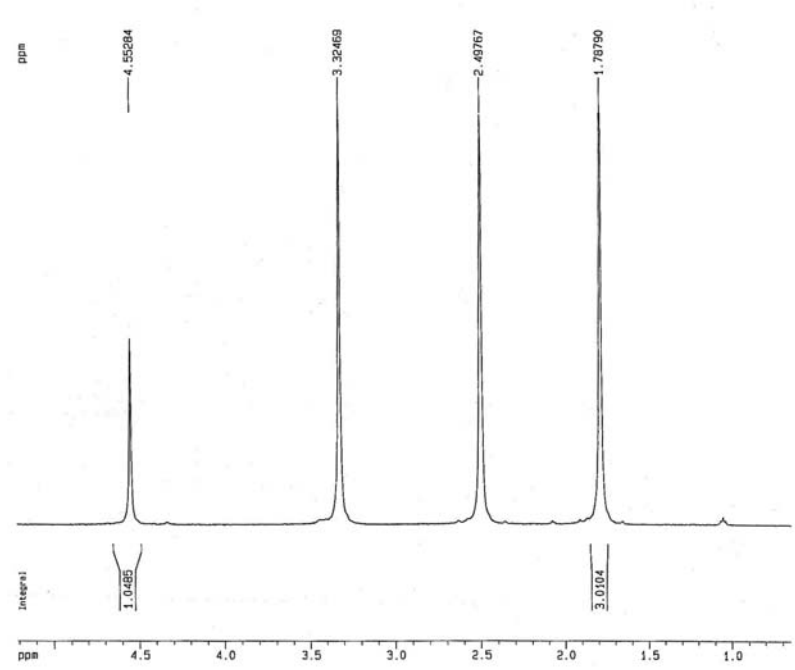


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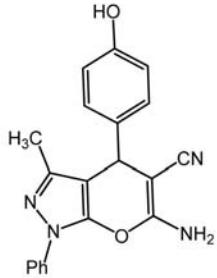

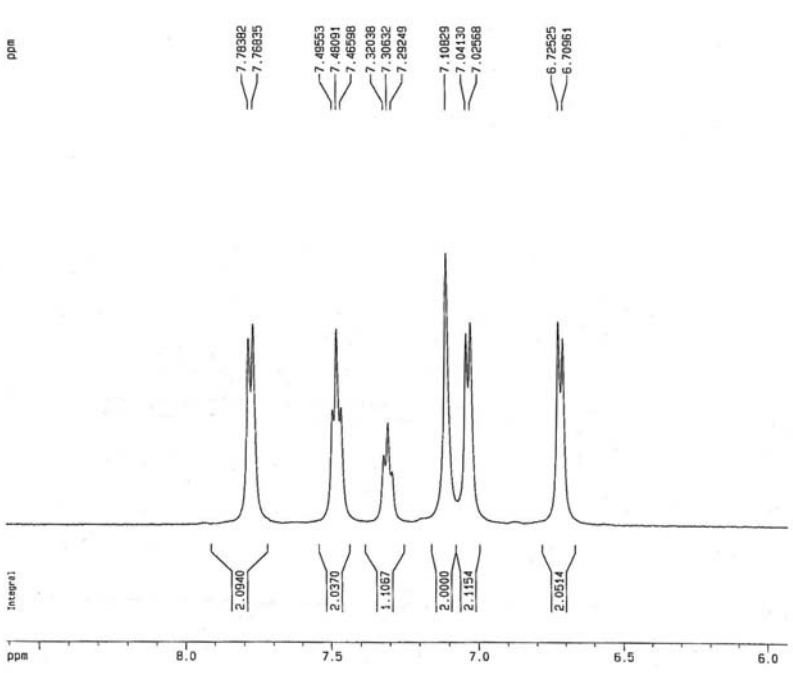


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TE              298.0 K
D1           5.00000000 sec
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MCWRK        0.01500000 sec

======== CHANNEL f1 ========
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PL1             -3.00 dB
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HZCM          66.89282 Hz/cm
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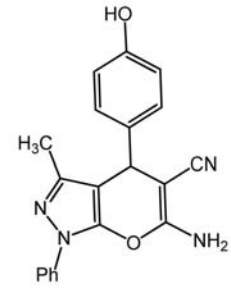
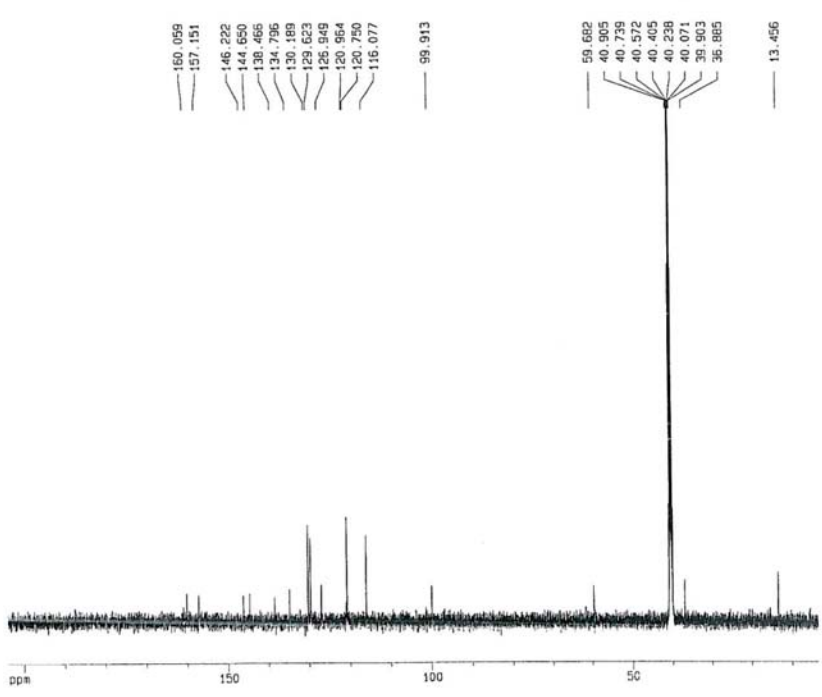
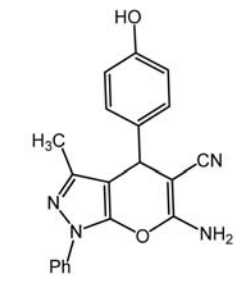
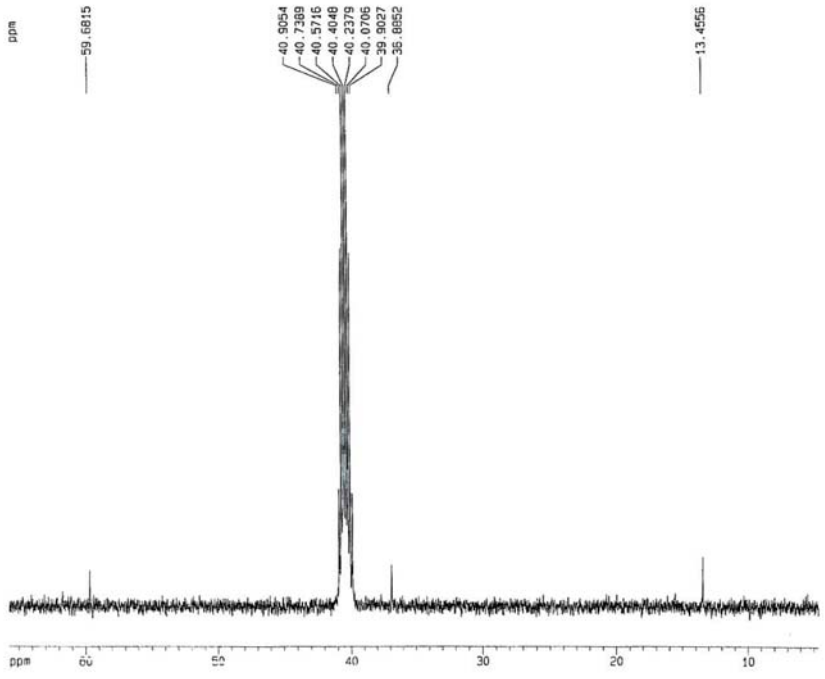


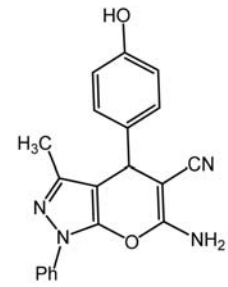


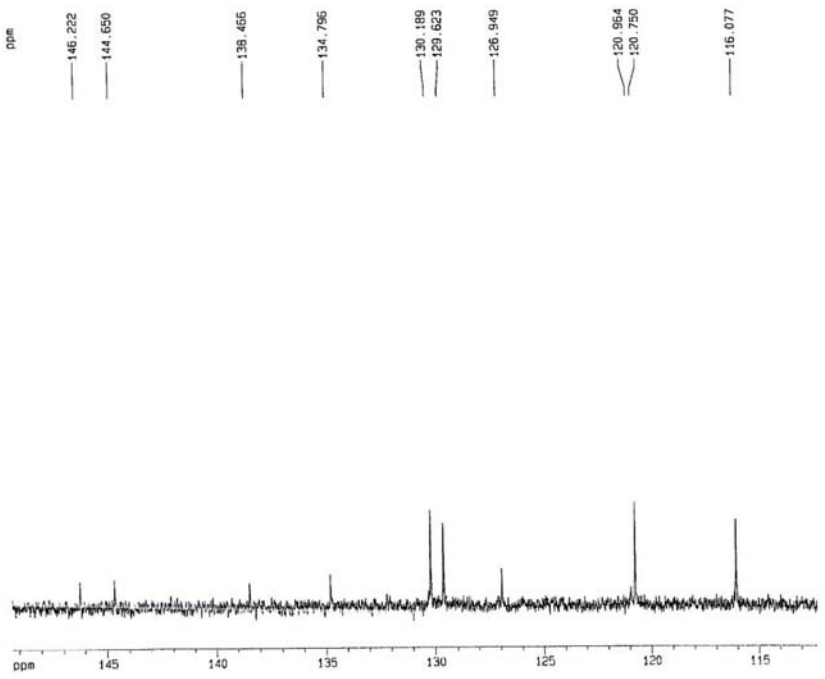

```
Current Data Parameters
NAME        Mansournejad
EXPNO              105
PROCNO               1

F2 - Acquisition Parameters
Date_          20121101
Time              13.39
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD               32768
SOLVENT          DMSO
NS                 404
DS                   4
SWH          30030.029 Hz
FIDRES        0.916444 Hz
AQ            0.5456539 sec
RG                4597.6
DW              16.650 usec
DE               6.50 usec
TE              298.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1              9.10 usec
PL1             3.00 dB
SFO1       125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2          80.00 usec
PL2             0.00 dB
PL12           15.50 dB
PL13           15.50 dB
SFO2       500.1320005 MHz

F2 - Processing parameters
SI               32768
SF         125.7577390 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40

1D NMR plot parameters
CX              20.00 cm
CY              19.90 cm
F1P            203.924 ppm
F1           25642.20 Hz
F2P             3.586 ppm
F2            450.97 Hz
PPMCM         10.01652 ppm/cm
HZCM        1259.70557 Hz/cm
```



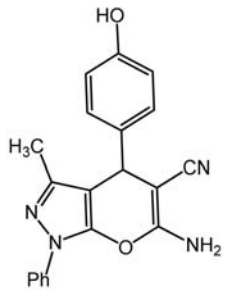


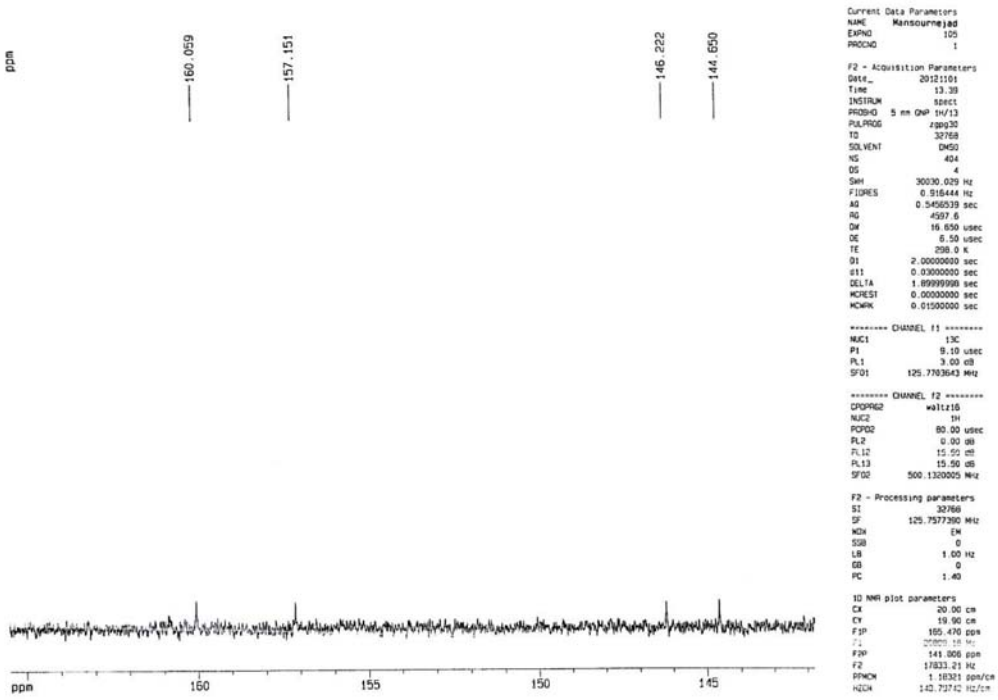

```
Current Data Parameters
NAME        Mansournejad
EXPNO              105
PROCNO               1

F2 - Acquisition Parameters
Date_           20121101
Time               13.39
INSTRUM            spect
PROBHD  5 mm QNP 1H/13
PULPROG          zgpg30
TD                32768
SOLVENT            DMSO
NS                  404
DS                    4
SWH           30030.029 Hz
FIDRES        0.916444 Hz
AQ            0.5456539 sec
RG                4597.6
DW               16.650 usec
DE                6.50 usec
TE                298.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MOREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                9.10 usec
PL1               3.00 dB
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            80.00 usec
PL2               0.00 dB
PL12             15.50 dB
PL13             15.50 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577390 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                20.00 cm
CY                19.90 cm
F1P               65.704 ppm
F1              8262.83 Hz
F2P               4.602 ppm
F2               586.24 Hz
PPMCM          3.05214 ppm/cm
HZCM          383.82974 Hz/cm
```

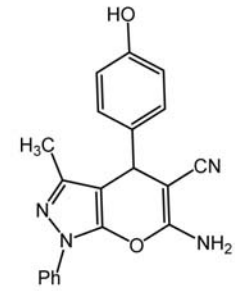


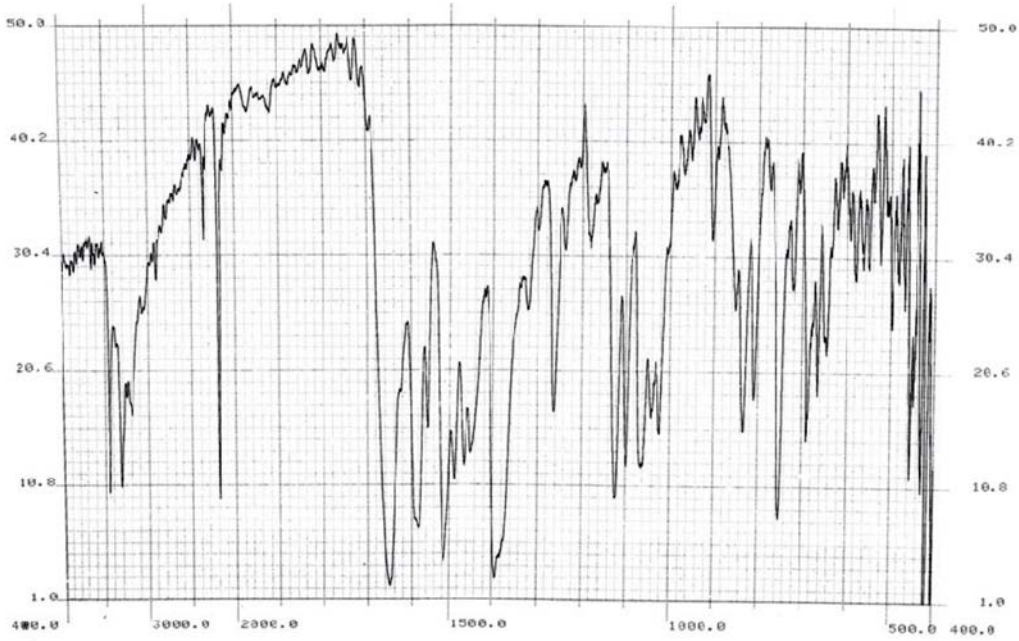


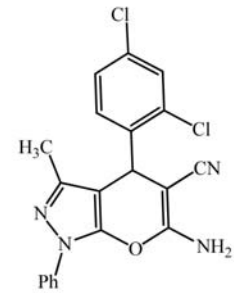

```
Current Data Parameters
NAME        Mansournejad
EXPNO              105
PROCNO               1

F2 - Acquisition Parameters
Date_           20121101
Time               13.39
INSTRUM            spect
PROBHD  5 mm QNP 1H/13
PULPROG          zgpg30
TD                32768
SOLVENT            DMSO
NS                  404
DS                    4
SWH           30030.029 Hz
FIDRES        0.916444 Hz
AQ            0.5456539 sec
RG                4597.6
DW               16.650 usec
DE                6.50 usec
TE                298.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MOREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                9.10 usec
PL1               3.00 dB
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            80.00 usec
PL2               0.00 dB
PL12             15.50 dB
PL13             15.50 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577390 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                20.00 cm
CY                19.90 cm
F1P               149.319 ppm
F2P               112.205 ppm
F2              14102.67 Hz
PPMCM          1.85171 ppm/cm
```

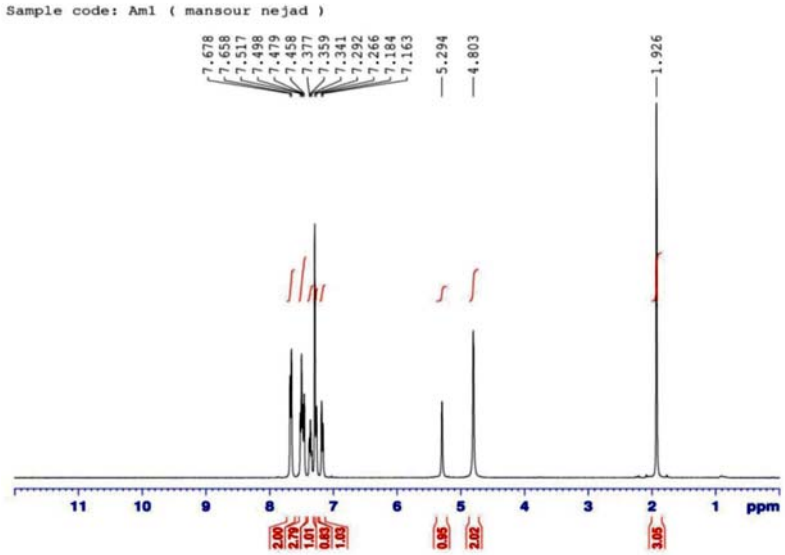



ppm
160.059
157.151
146.222
144.650
ppm
160
155
150
145
Current Data Parameters
NAME Mansournejad
EXPNO 105
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121101
Time 13.39
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 404
DS 4
SWH 30030.029 Hz
FIDRES 0.916444 Hz
AQ 0.5456539 sec
RG 4597.6
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 3.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 15.50 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 19.90 cm
F1P 165.470 ppm
F2P 141.006 ppm
F2 17833.21 Hz
PPMCM 1.18301 ppm/cm
HZCM 140.73742 Hz/cm

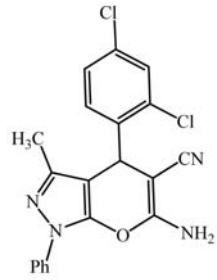

HO
H3C
CN
N
N
O
NH2
Ph

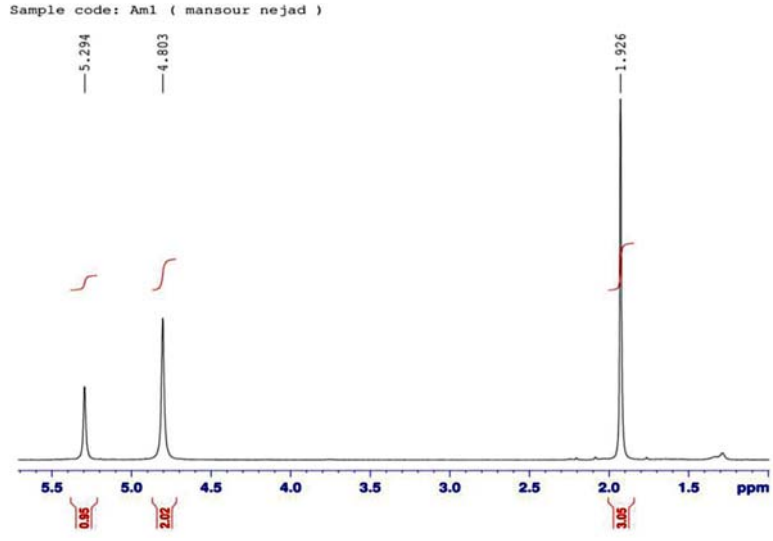

50.0
40.2
30.4
20.6
10.8
1.0
4000.0
3000.0
2000.0
1500.0
1000.0
500.0
400.0

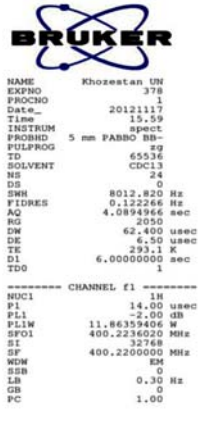

Cl
Cl
H3C
CN
N
N
O
NH2
Ph

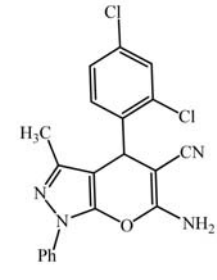

Sample code: Am1 (mansour nejad)
7.678
7.658
7.517
7.498
7.479
7.458
7.377
7.359
7.341
7.292
7.266
7.184
7.163
5.294
4.803
1.926
11 10 9 8 7 6 5 4 3 2 1 ppm
2.00
2.79
1.01
0.83
1.03
0.95
2.02
3.05

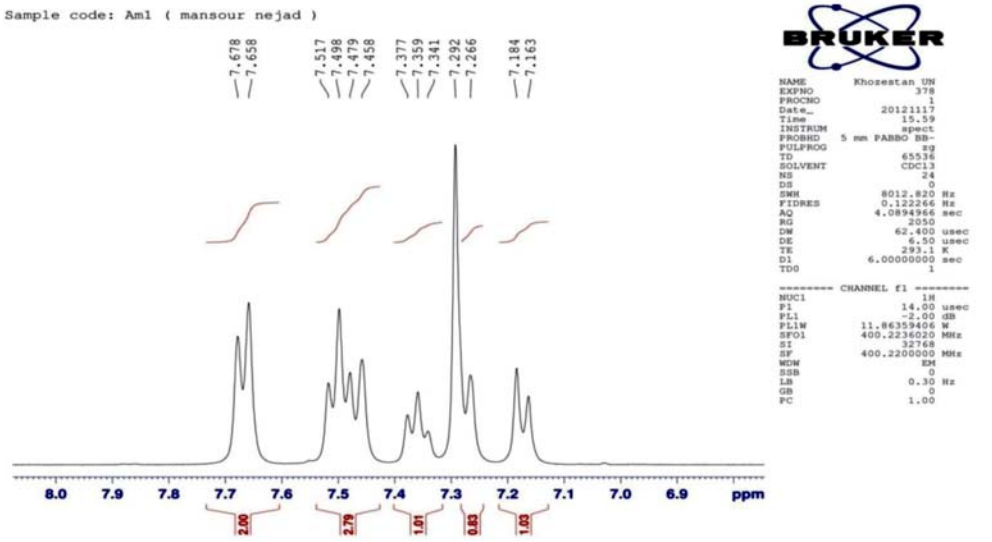

BRUKER
NAME Khozestan UN
EXPNO 378
PROCNO 1
Date_ 20121117
Time 15.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 2050
DW 62.400 usec
DE 6.50 usec
TE 293.1 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

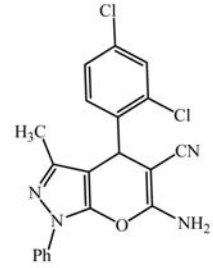

Cl
Cl
H3C
CN
N
N
O
NH2
Ph

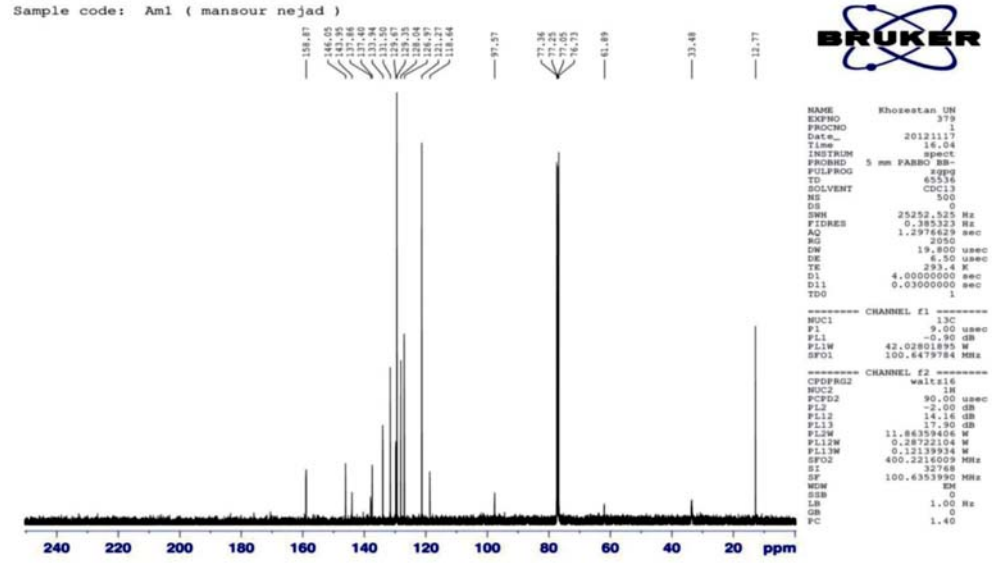

Sample code: Am1 (mansour nejad)
5.294
4.803
1.926
5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm
0.95
2.02
3.05

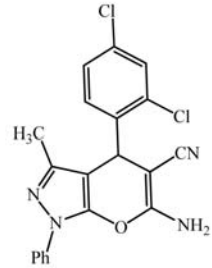

BRUKER
NAME Khozestan UN
EXPNO 378
PROCNO 1
Date_ 20121117
Time 15.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 2050
DW 62.400 usec
DE 6.50 usec
TE 293.1 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

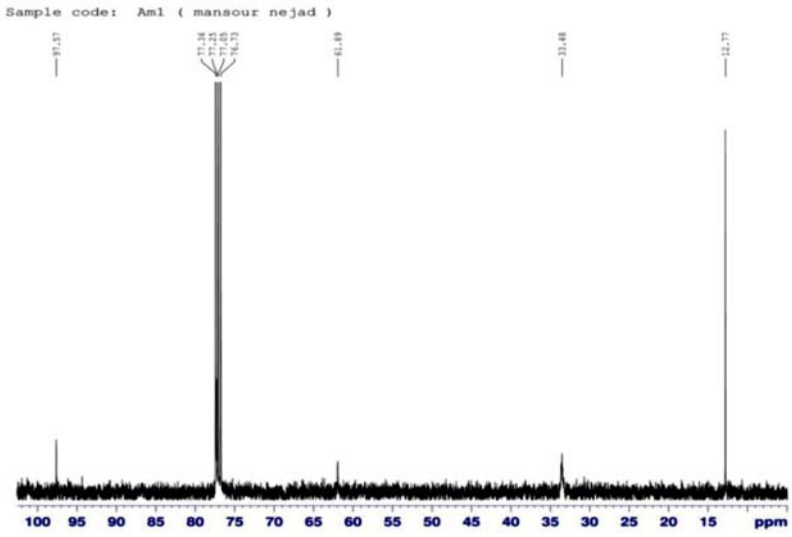

Cl
Cl
H3C
CN
N
N
O
NH2
Ph

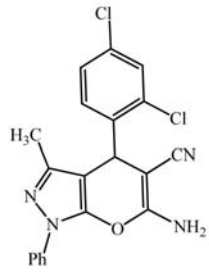

Sample code: Am1 (mansour nejad)
7.678
7.658
7.517
7.498
7.479
7.458
7.377
7.359
7.341
7.292
7.266
7.184
7.163
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 ppm
2.00
2.79
1.01
0.83
1.03
BRUKER
NAME Khozestan UN
EXPNO 378
PROCNO 1
Date_ 20121117
Time 15.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 2050
DW 62.400 usec
DE 6.50 usec
TE 293.1 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

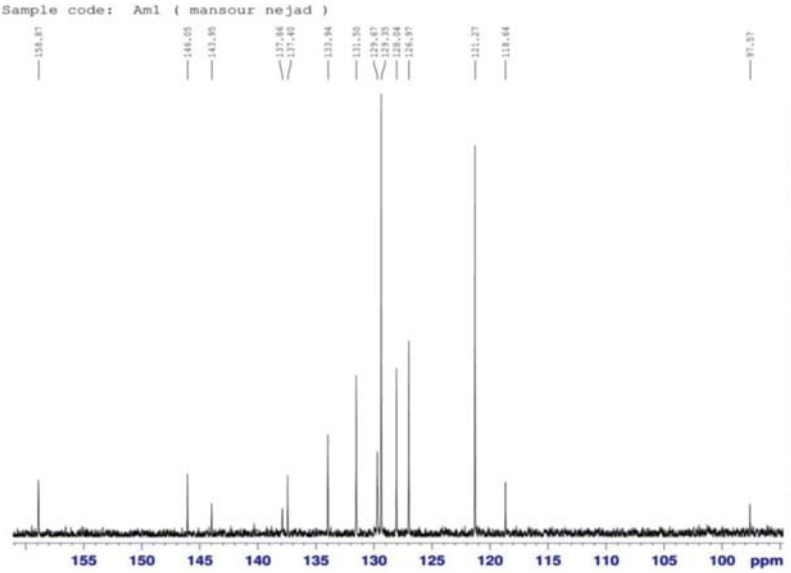

Cl
Cl
H3C
CN
N
N
O
NH2
Ph

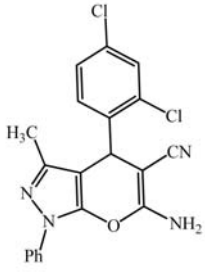

Sample code: Am1 (mansour nejad)
158.87
146.05
143.45
137.86
137.40
133.94
131.50
129.67
129.35
128.04
126.97
121.27
118.64
97.57
77.36
77.25
77.05
76.73
61.89
33.48
12.77
240 220 200 180 160 140 120 100 80 60 40 20 ppm
BRUKER
NAME Khozestan UN
EXPNO 379
PROCNO 1
Date_ 20121117
Time 16.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 500
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 293.4 K
D1 4.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

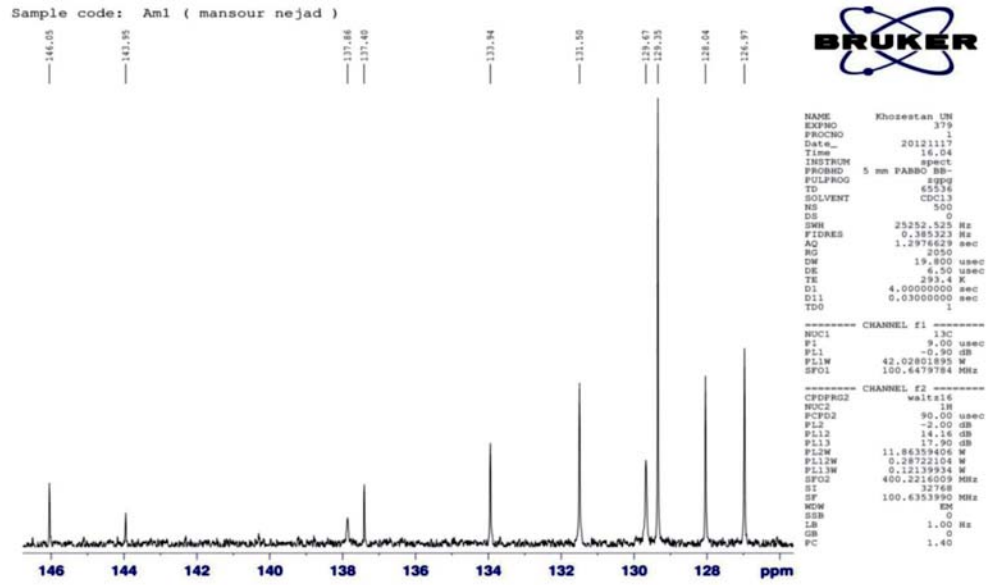

Cl
Cl
H3C
CN
N
N
O
NH2
Ph

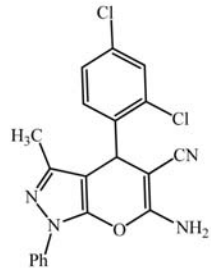

Sample code: Am1 (mansour nejad)
BRUKER
ppm

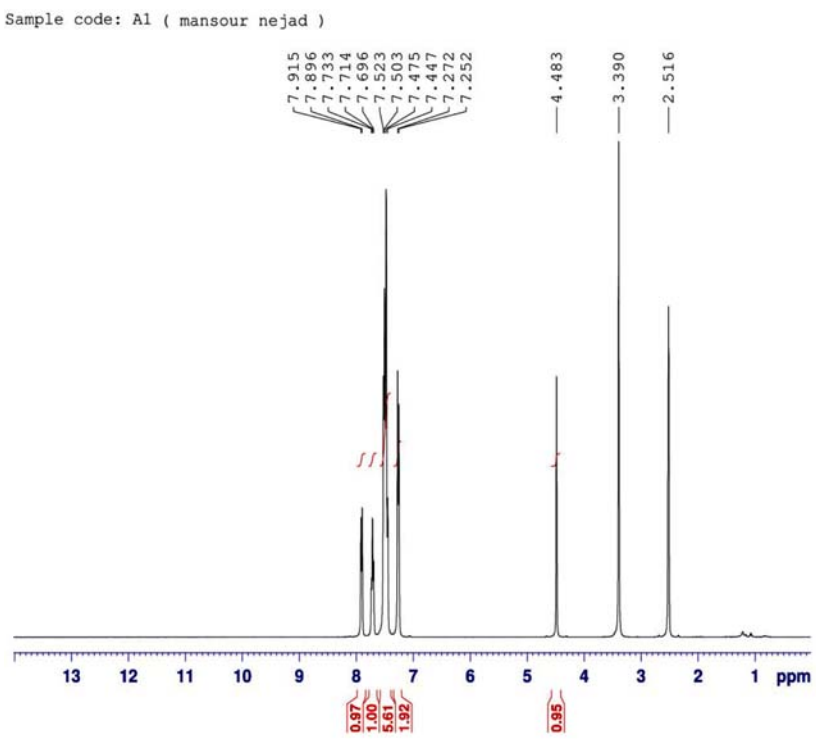

Cl
Cl
H3C
CN
N
N
O
NH2
Ph

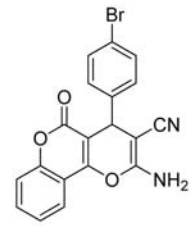

Sample code: Am1 (mansour nejad)
BRUKER
ppm

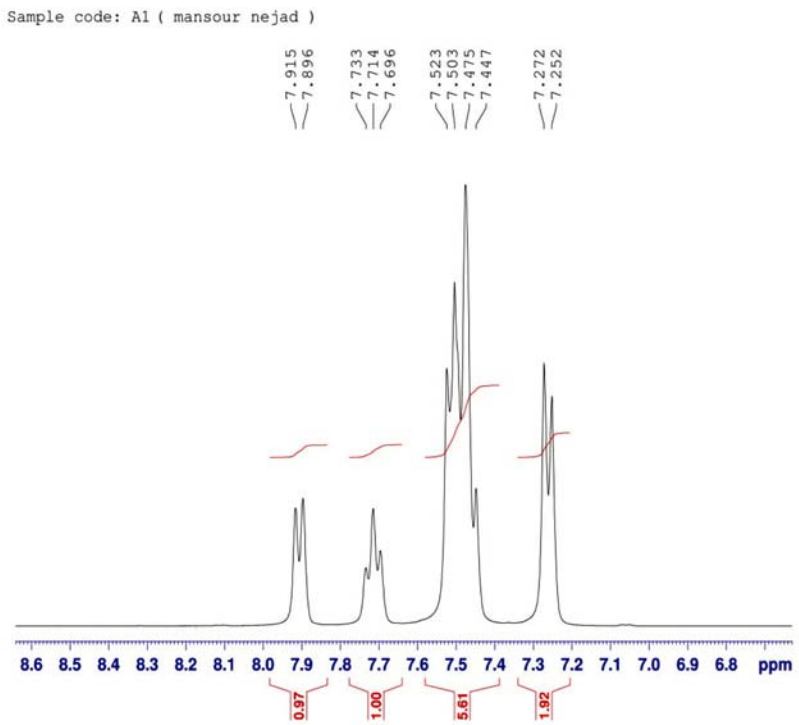

Cl
Cl
H3C
CN
N
N
O
NH2
Ph


Sample code: Am1 (mansour nejad)
146.05
143.95
137.86
137.40
133.94
131.50
129.67
129.35
128.04
126.97
NAME Khozestan UN
EXPNO 379
PROCNO 1
Date_ 20121117
Time 16.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 500
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 293.4 K
D1 4.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
146
144
142
140
138
136
134
132
130
128
ppm


Cl
Cl
H3C
CN
N
N
O
NH2
Ph

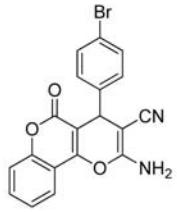

Sample code: A1 (mansour nejad)
7.915
7.896
7.733
7.714
7.696
7.523
7.503
7.475
7.447
7.272
7.252
4.483
3.390
2.516
NAME Azad-Gachsaran UN
EXPNO 341
PROCNO 1
Date_ 20120318
Time 15.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 1290
DW 62.400 usec
DE 6.50 usec
TE 295.8 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.97
1.00
5.61
1.92
0.95

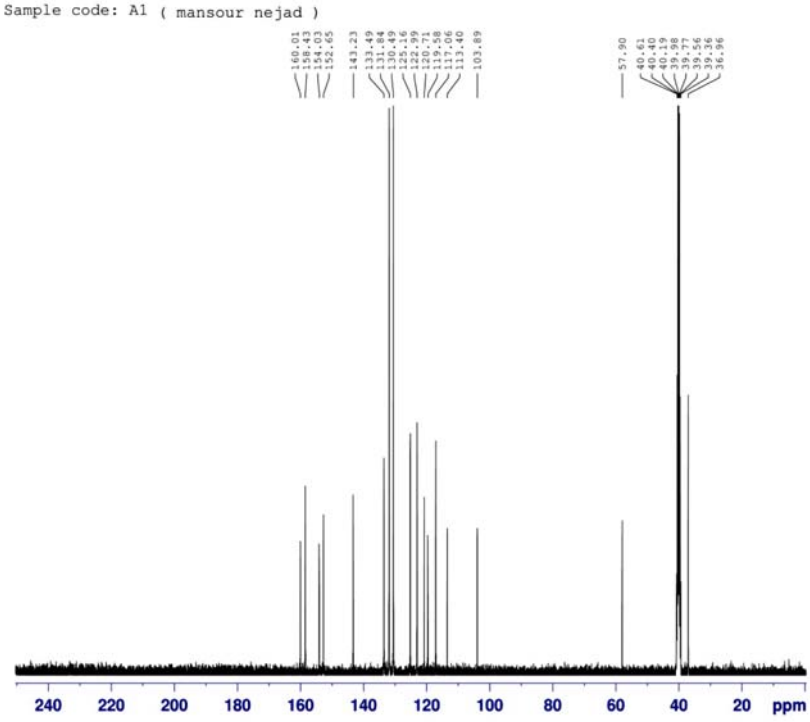

Br
O
O
CN
O
NH2



BRUKER

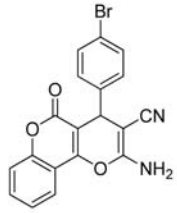
```
NAME          Azad-Gachsaran UN
EXPNO                     341
PROCNO                      1
Date_              20120318
Time                   15.02
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                DMSO
NS                       24
DS                        0
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                      1290
DW                   62.400 usec
DE                    6.50 usec
TE                   295.8 K
D1                6.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      14.00 usec
PL1                     -2.00 dB
PL1W              11.86359406 W
SFO1              400.2236020 MHz
SI                       32768
SF                400.2200000 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                      1.00
```

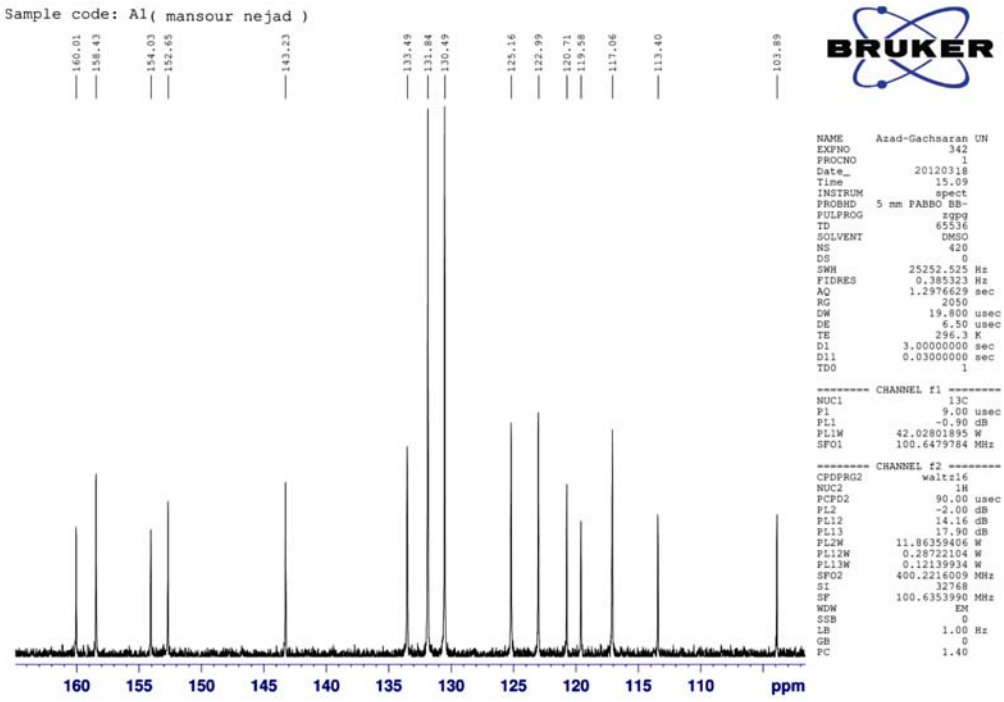


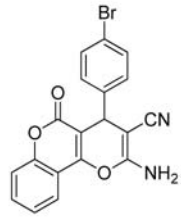


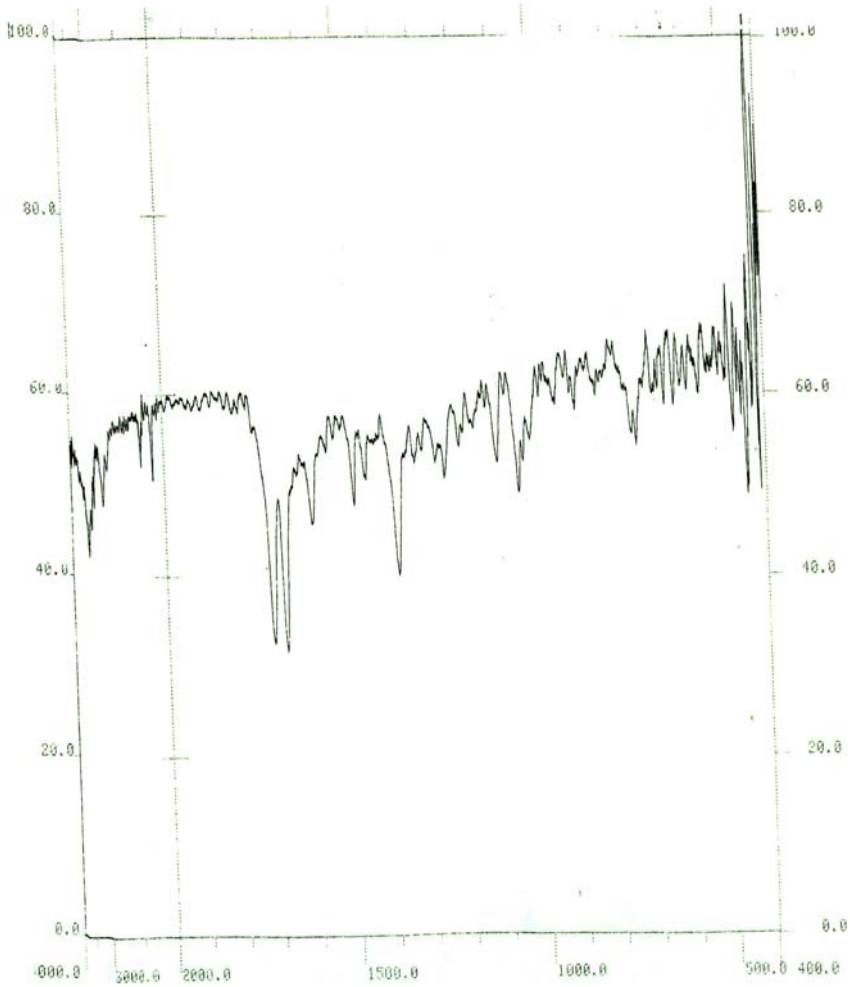
BRUKER

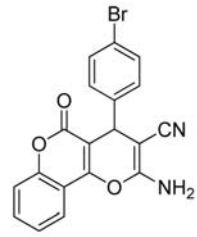
```
NAME          Azad-Gachsaran UN
EXPNO                     342
PROCNO                      1
Date_              20120318
Time                   15.09
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zgpg
TD                     65536
SOLVENT                DMSO
NS                       420
DS                        0
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                      2050
DW                   19.800 usec
DE                    6.50 usec
TE                   296.3 K
D1                3.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                      9.00 usec
PL1                     -0.90 dB
PL1W              42.02801895 W
SFO1              100.6479784 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  90.00 usec
PL2                     -2.00 dB
PL12                    14.16 dB
PL13                    17.90 dB
PL2W              11.86359406 W
PL12W              0.28722104 W
PL13W              0.12139934 W
SFO2              400.2216009 MHz
SI                       32768
SF                100.6353990 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                      1.40
```

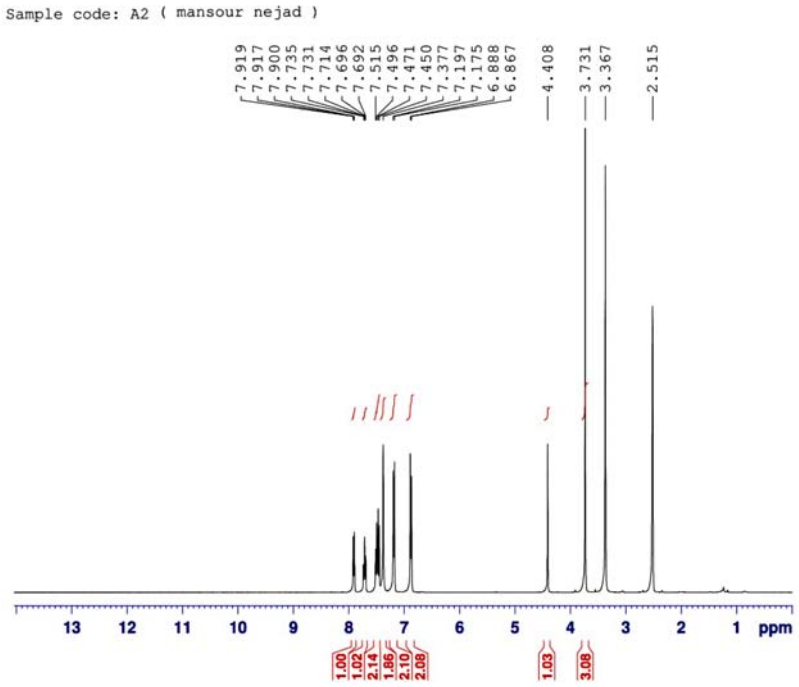



Sample code: A1(mansour nejad)
160.01
158.43
154.03
152.65
143.23
133.49
131.84
130.49
125.16
122.99
120.71
119.58
117.06
113.40
103.89
BRUKER
NAME Azad-Gachsaran UN
EXPNO 342
PROCNO 1
Date_ 20120318
Time 15.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 420
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 296.3 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
155
150
145
140
135
130
125
120
115
110
ppm

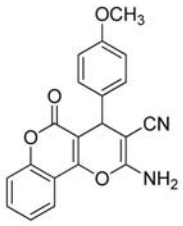

Br
O
O
CN
O
NH2

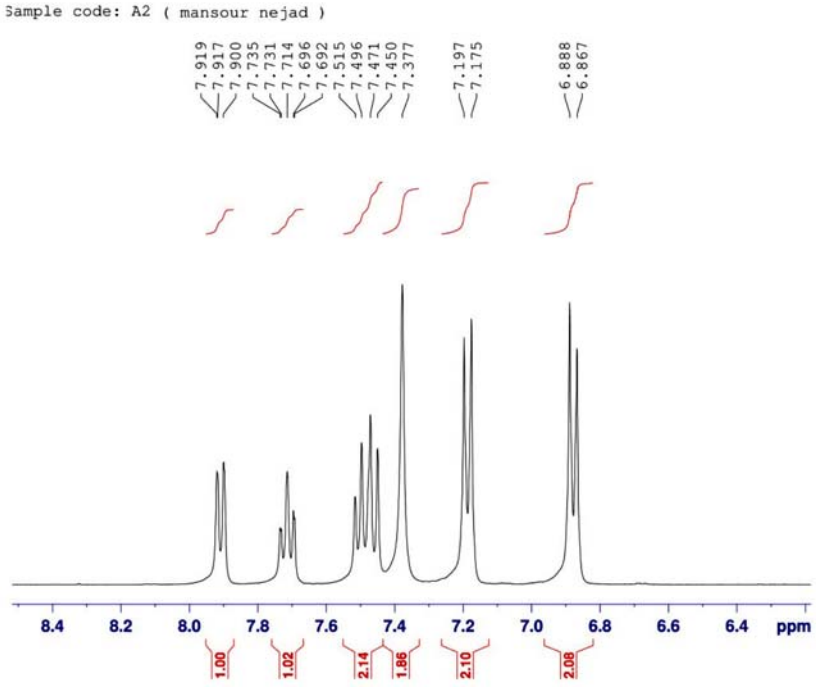

100.0
80.0
60.0
40.0
20.0
0.0
3000.0
2000.0
1500.0
1000.0
500.0
400.0


Br
O
O
CN
O
NH2

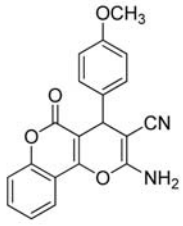


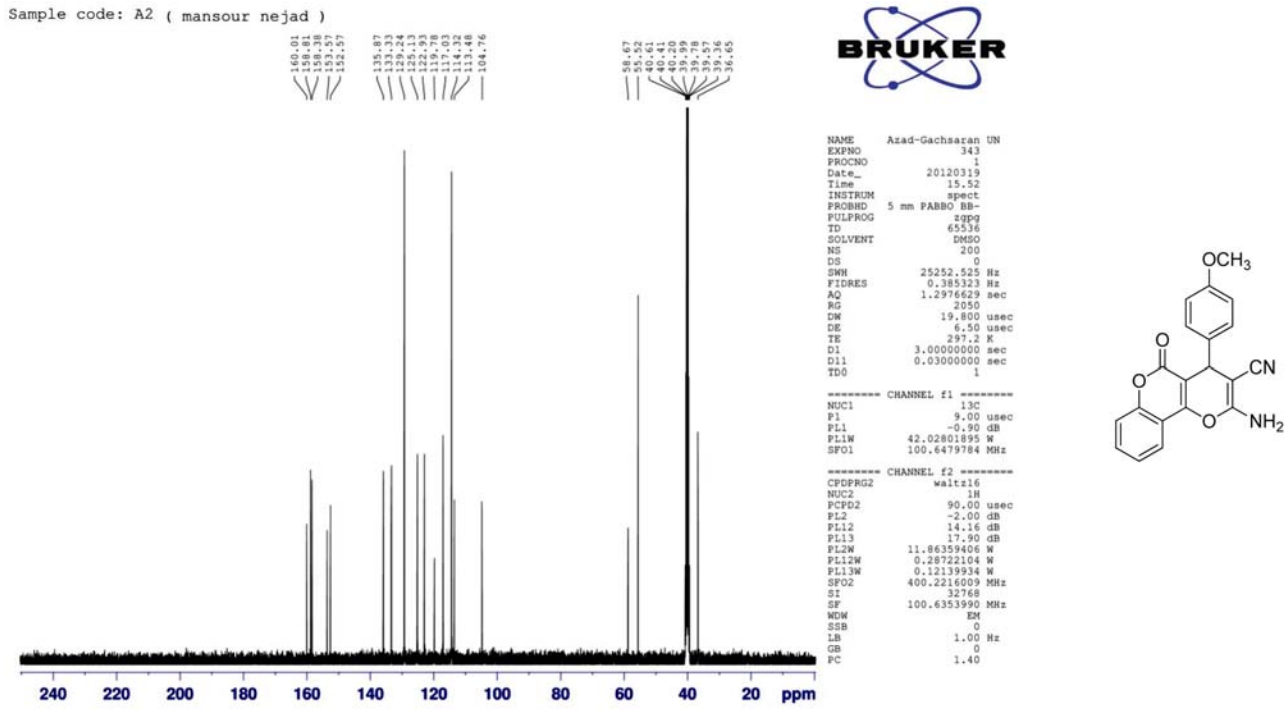

```
NAME      Azad-Gachsaran UN
EXPNO                 344
PROCNO                  1
Date_            20120319
Time                15.46
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     24
DS                      0
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                   1290
DW                 62.400 usec
DE                  6.50 usec
TE                 296.7 K
D1              6.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                -2.00 dB
PL1W         11.86359406 W
SFO1         400.2236020 MHz
SI                   32768
SF           400.2200000 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

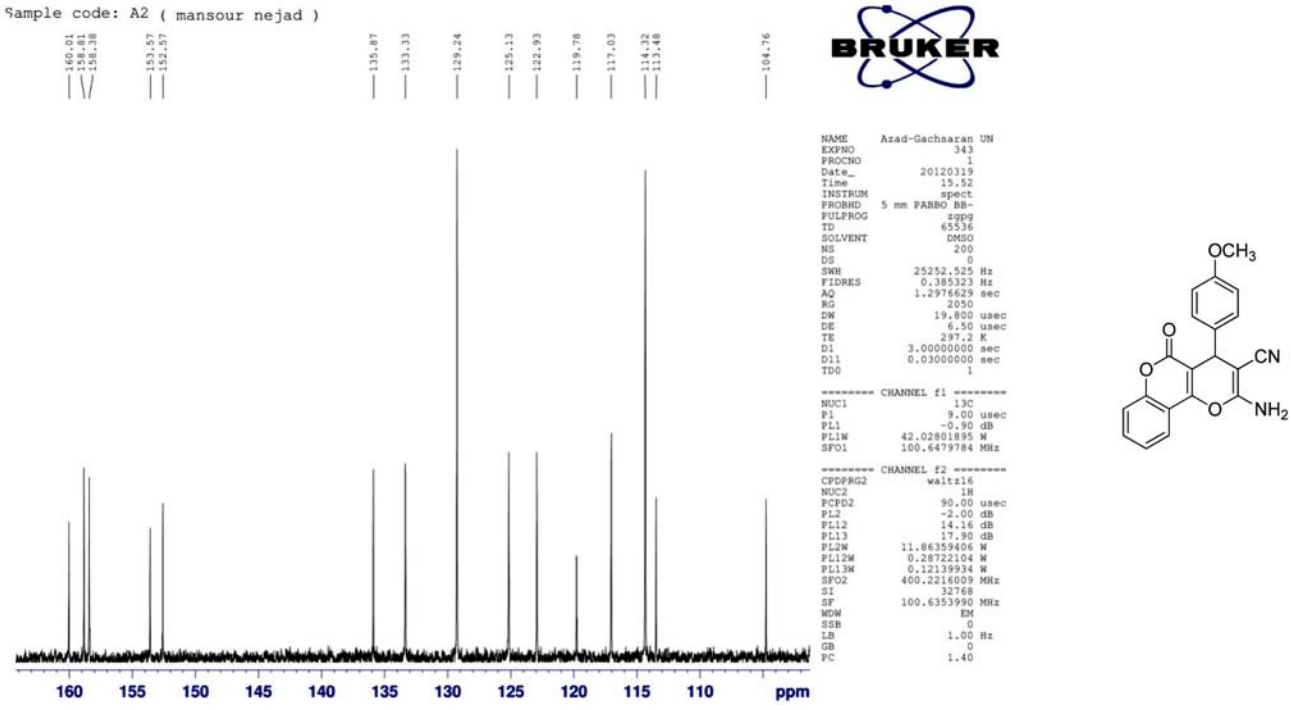


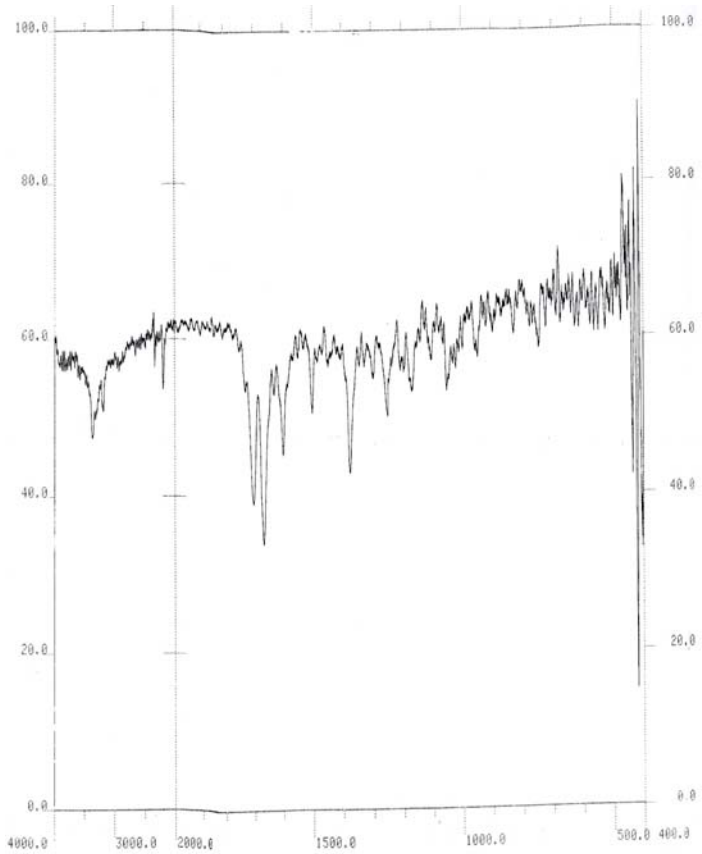


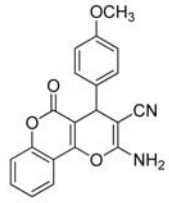

```
NAME      Azad-Gachsaran UN
EXPNO                 344
PROCNO                  1
Date_            20120319
Time                15.46
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     24
DS                      0
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                   1290
DW                 62.400 usec
DE                  6.50 usec
TE                 296.7 K
D1              6.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                -2.00 dB
PL1W         11.86359406 W
SFO1         400.2236020 MHz
SI                   32768
SF           400.2200000 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

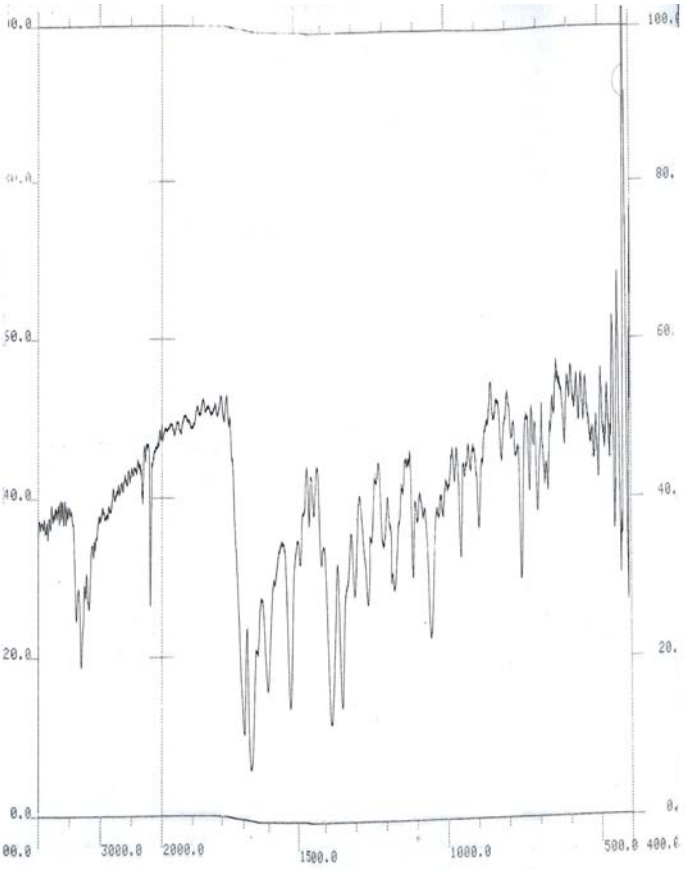


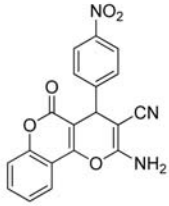

Sample code: A2 (mansour nejad)
160.01
158.81
158.38
153.57
152.57
135.87
133.33
129.24
125.13
122.93
119.78
117.03
114.32
113.48
104.76
58.67
55.52
40.61
40.41
40.20
39.99
39.78
39.57
39.36
34.65
BRUKER
NAME Azad-Gachsaran UN
EXPNO 343
PROCNO 1
Date_ 20120319
Time 15.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 200
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 297.2 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
240 220 200 180 160 140 120 100 80 60 40 20 ppm
OCH3
CN
NH2

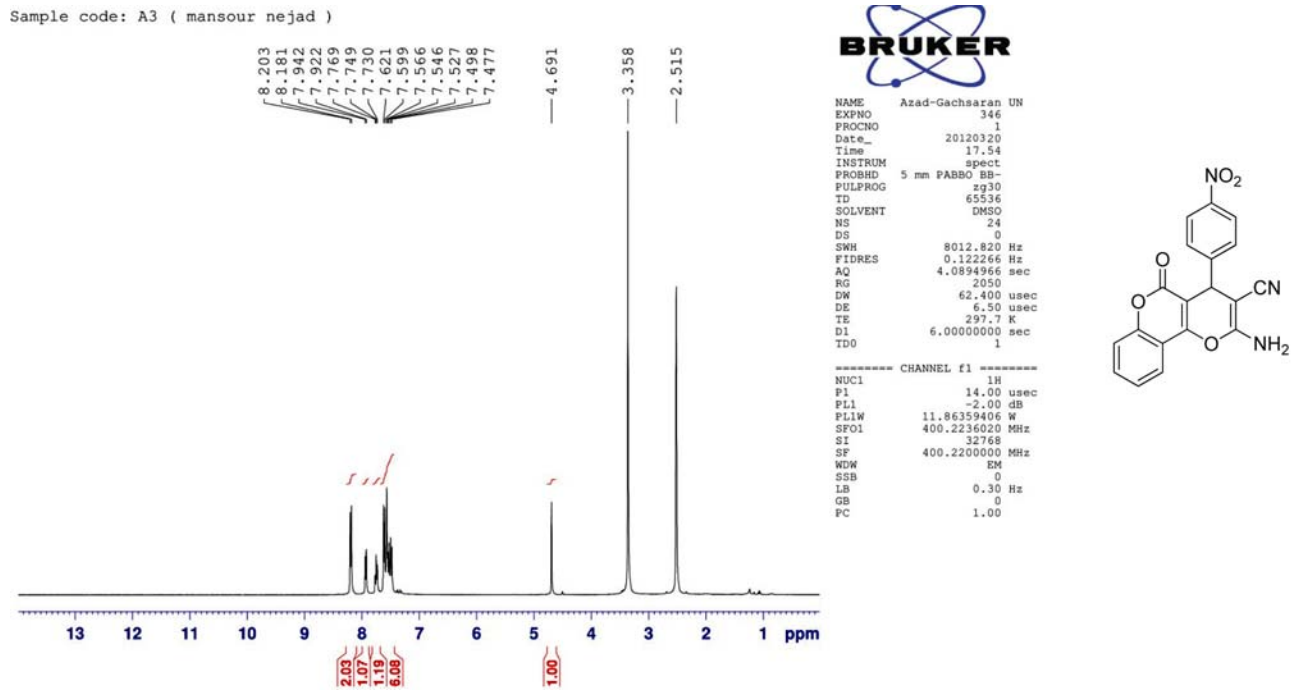

Sample code: A2 (mansour nejad)
160.01
158.81
158.38
153.57
152.57
135.87
133.33
129.24
125.13
122.93
119.78
117.03
114.32
113.48
104.76
BRUKER
NAME Azad-Gachsaran UN
EXPNO 343
PROCNO 1
Date_ 20120319
Time 15.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 200
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 297.2 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 155 150 145 140 135 130 125 120 115 110 ppm
OCH3
CN
NH2

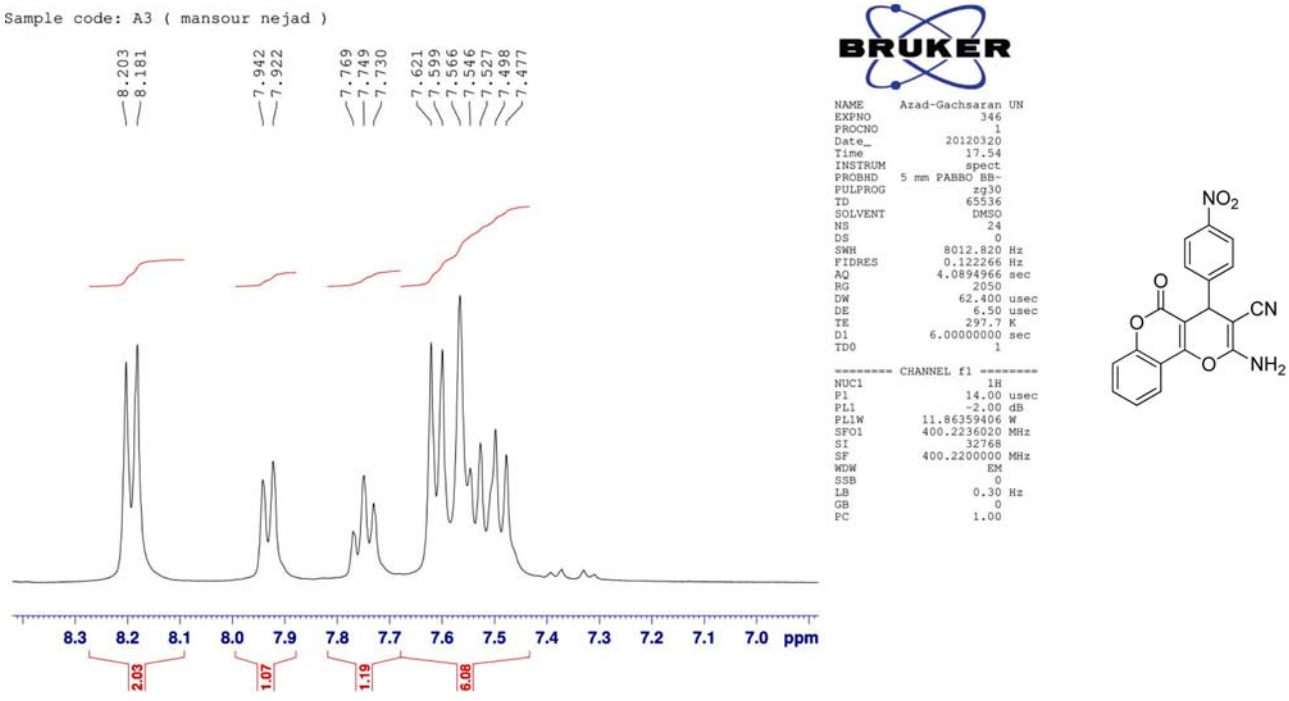

100.0
80.0
60.0
40.0
20.0
0.0
4000.0
3000.0
2000.0
1500.0
1000.0
500.0
400.0

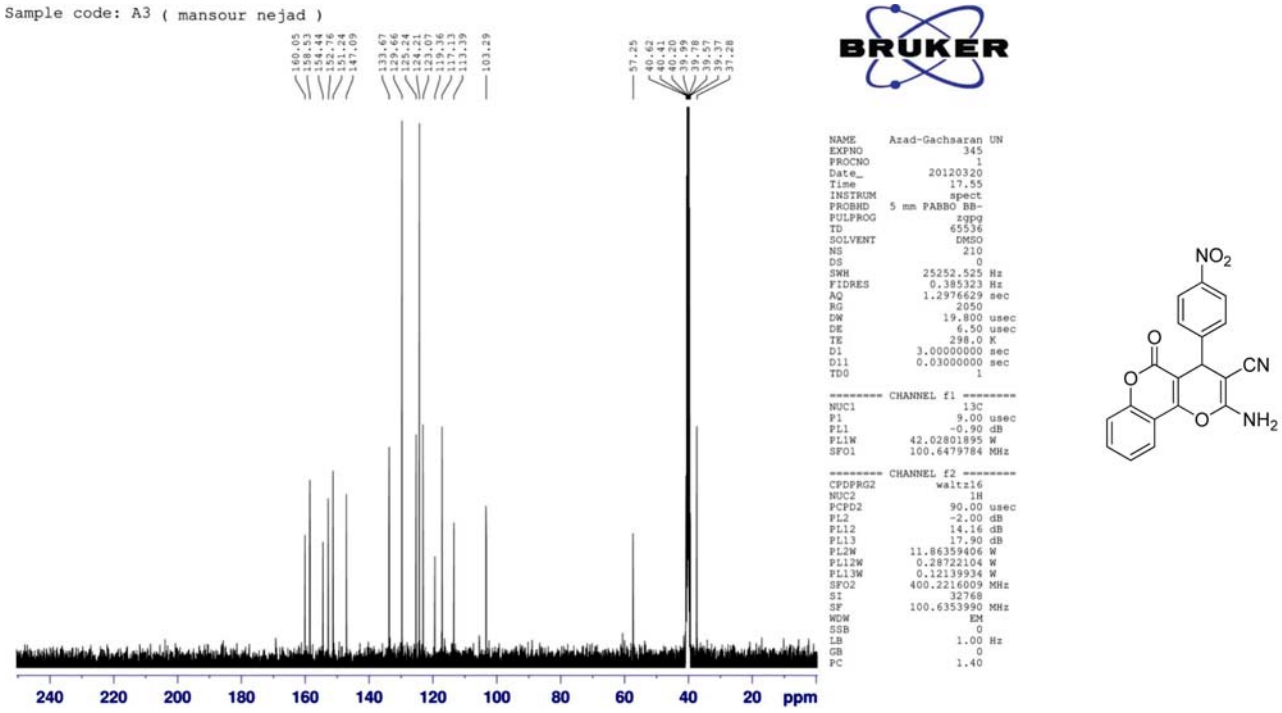

OCH3
O
O
CN
O
NH2

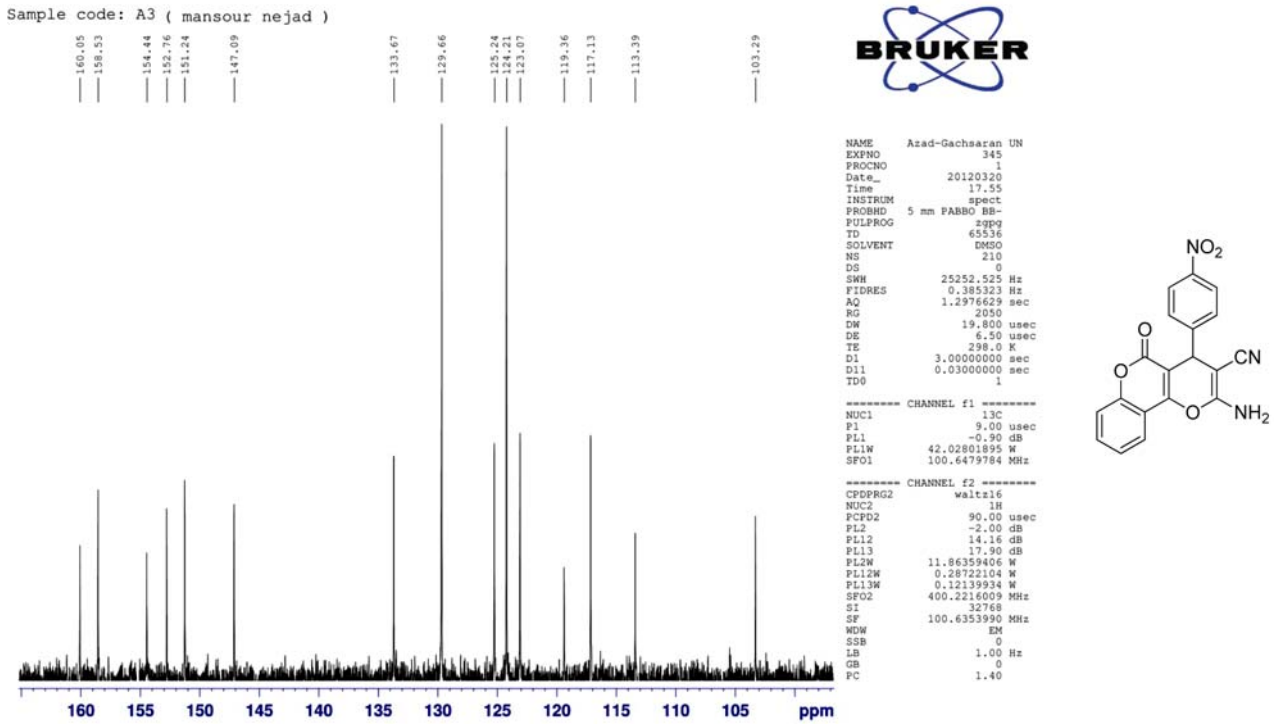

100.0
80.0
60.0
40.0
20.0
0.0
3000.0
2000.0
1500.0
1000.0
500.0

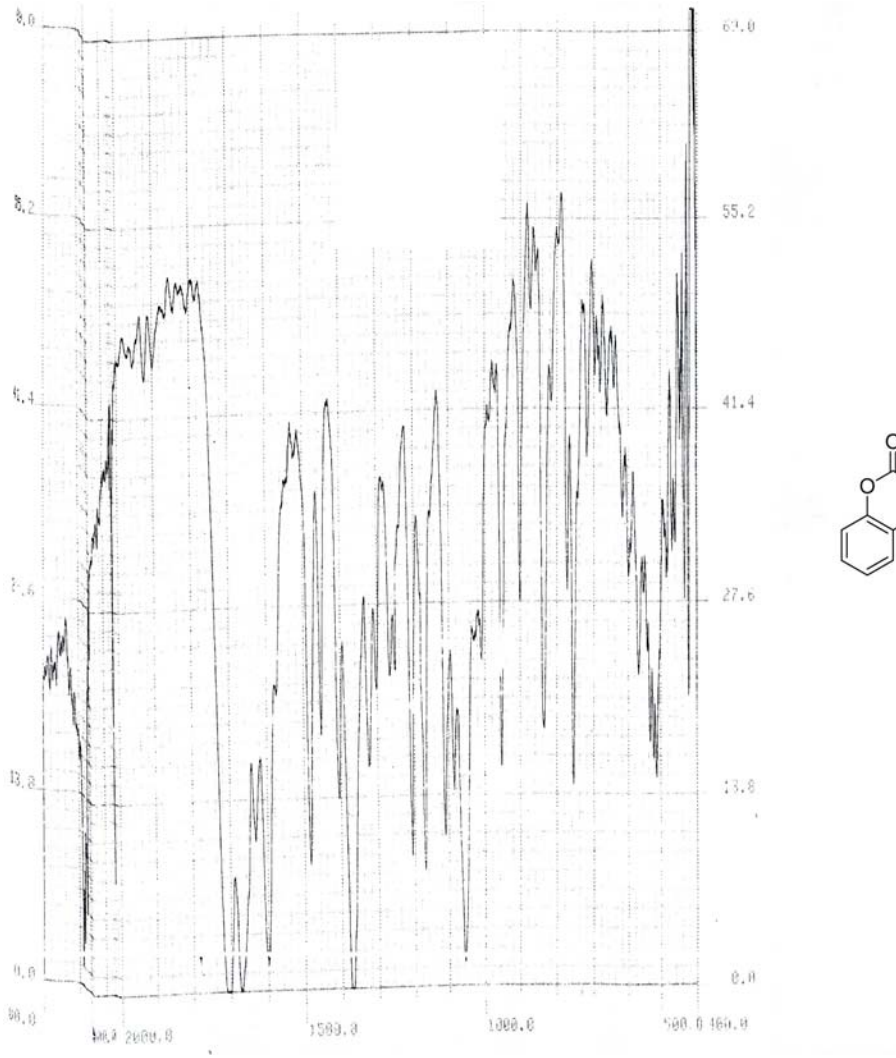

NO2
O
O
CN
O
NH2

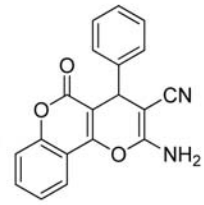

Sample code: A3 (mansour nejad)
8.203
8.181
7.942
7.922
7.769
7.749
7.730
7.621
7.599
7.566
7.546
7.527
7.498
7.477
4.691
3.358
2.515
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
2.03
1.07
1.19
6.08
1.00
BRUKER
NAME Azad-Gachsaran UN
EXPNO 346
PROCNO 1
Date_ 20120320
Time 17.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 2050
DW 62.400 usec
DE 6.50 usec
TE 297.7 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NO2
CN
NH2
O

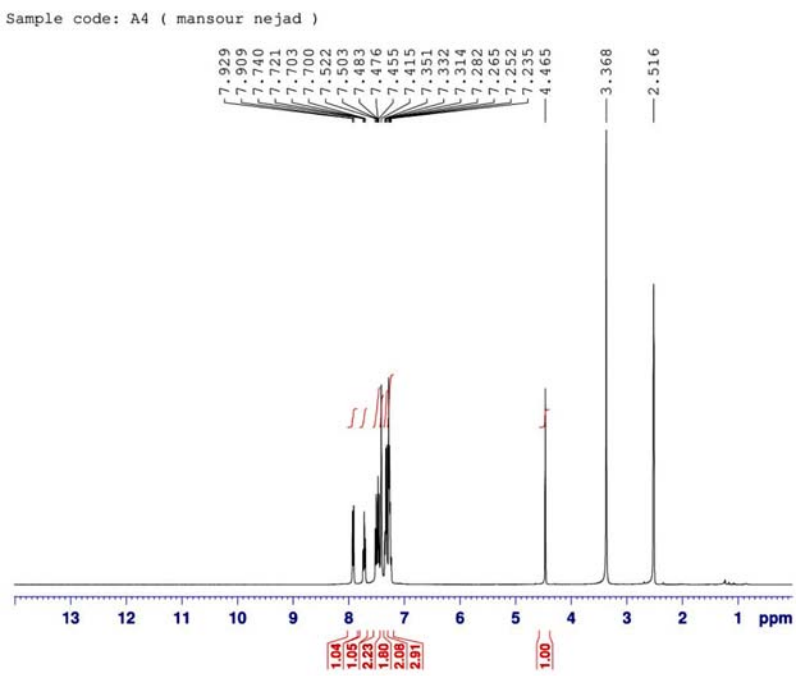

Sample code: A3 (mansour nejad)
8.203
8.181
7.942
7.922
7.769
7.749
7.730
7.621
7.599
7.566
7.546
7.527
7.498
7.477
8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 ppm
2.03
1.07
1.19
6.08
BRUKER
NAME Azad-Gachsaran UN
EXPNO 346
PROCNO 1
Date_ 20120320
Time 17.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 2050
DW 62.400 usec
DE 6.50 usec
TE 297.7 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NO2
CN
NH2
O


Sample code: A3 (mansour nejad)
160.05
158.53
154.44
152.76
151.24
147.09
133.67
129.66
125.24
124.21
123.07
119.36
117.13
113.39
103.29
57.25
40.62
40.41
40.20
39.99
39.78
39.57
39.37
39.28
240 220 200 180 160 140 120 100 80 60 40 20 ppm
BRUKER
NAME Azad-Gachsaran UN
EXPNO 345
PROCNO 1
Date_ 20120320
Time 17.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 210
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.0 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
NO2
O
O
CN
O
NH2


Sample code: A3 (mansour nejad)
160.05
158.53
154.44
152.76
151.24
147.09
133.67
129.66
125.24
124.21
123.07
119.36
117.13
113.39
103.29
160 155 150 145 140 135 130 125 120 115 110 105 ppm
BRUKER
NAME Azad-Gachsaran UN
EXPNO 345
PROCNO 1
Date_ 20120320
Time 17.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 210
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.0 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
NO2
O
O
CN
O
NH2

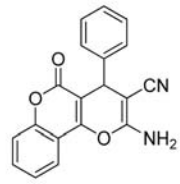


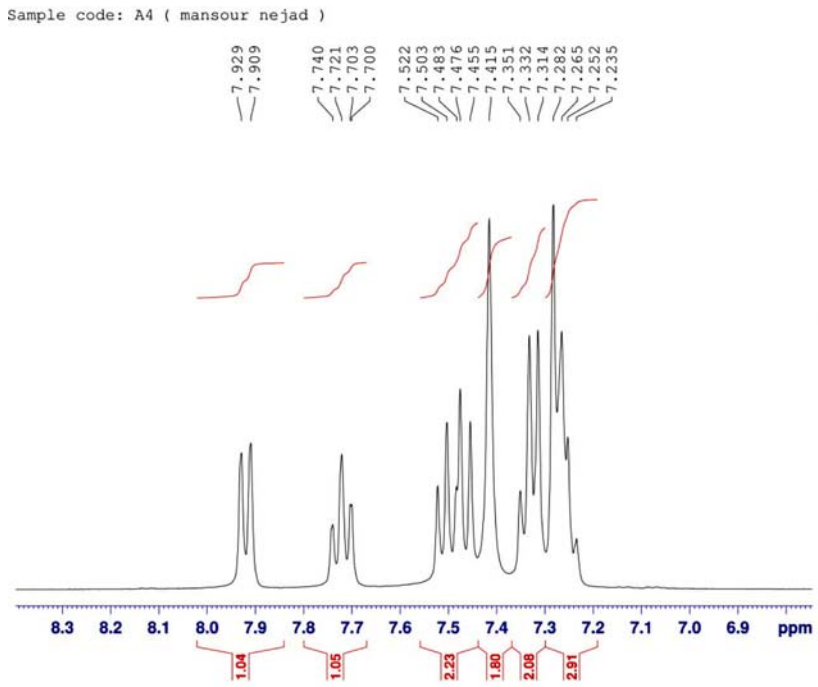




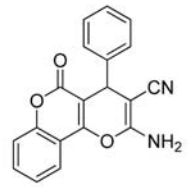
BRUKER

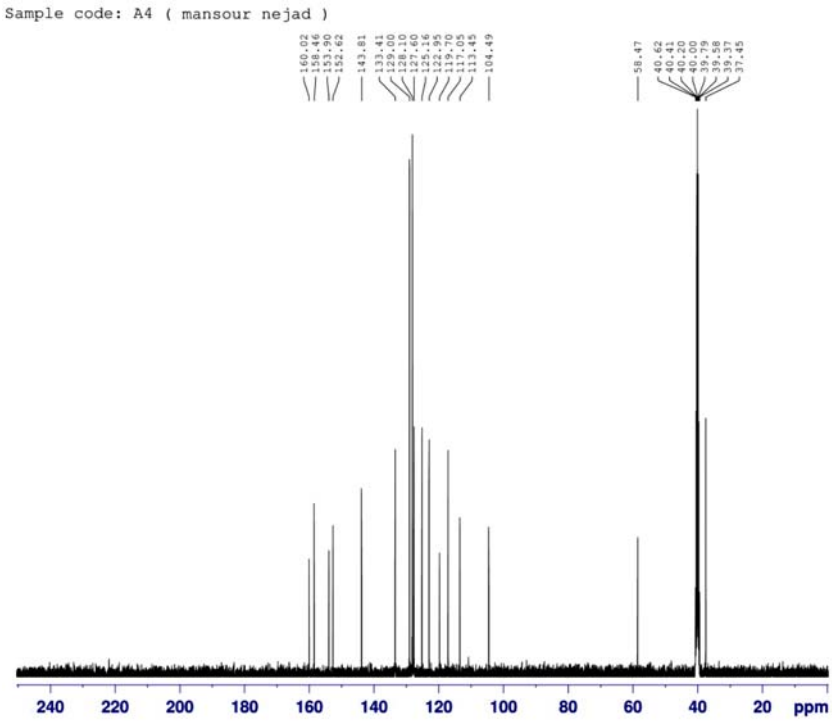
NAME	Azad-Gachsaran UN
EXPNO	348
PROCNO	1
Date_	20120320
Time	18.52
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	24
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	1620
DW	62.400 usec
DE	6.50 usec
TE	298.0 K
D1	6.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-2.00 dB
PL1W	11.86359406 W
SFO1	400.2236020 MHz
SI	32768
SF	400.2200000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



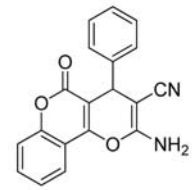


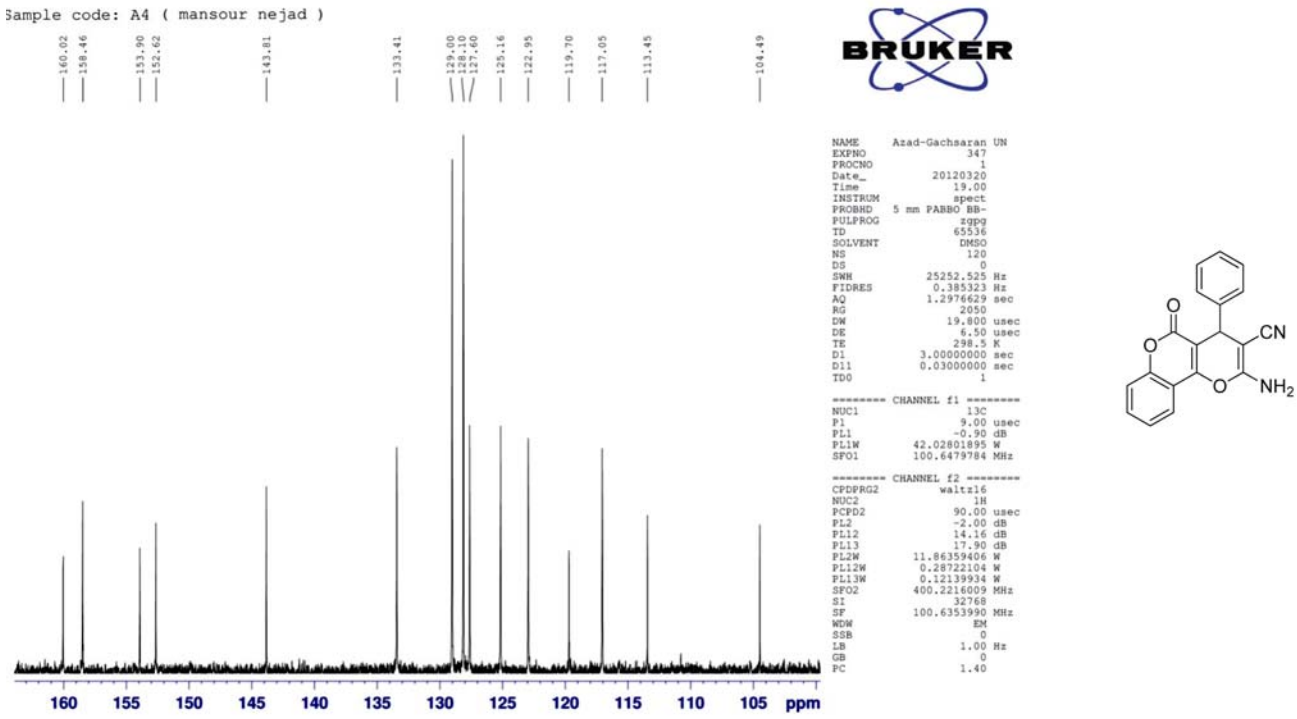

```
NAME          Azad-Gachsaran UN
EXPNO                    348
PROCNO                     1
Date_               20120320
Time                   18.52
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                        24
DS                         0
SWH                8012.820 Hz
FIDRES                0.122266 Hz
AQ                 4.0894966 sec
RG                      1620
DW                    62.400 usec
DE                     6.50 usec
TE                   298.0 K
D1                 6.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                    14.00 usec
PL1                   -2.00 dB
PL1W              11.86359406 W
SFO1              400.2236020 MHz
SI                      32768
SF                400.2200000 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                      1.00
```

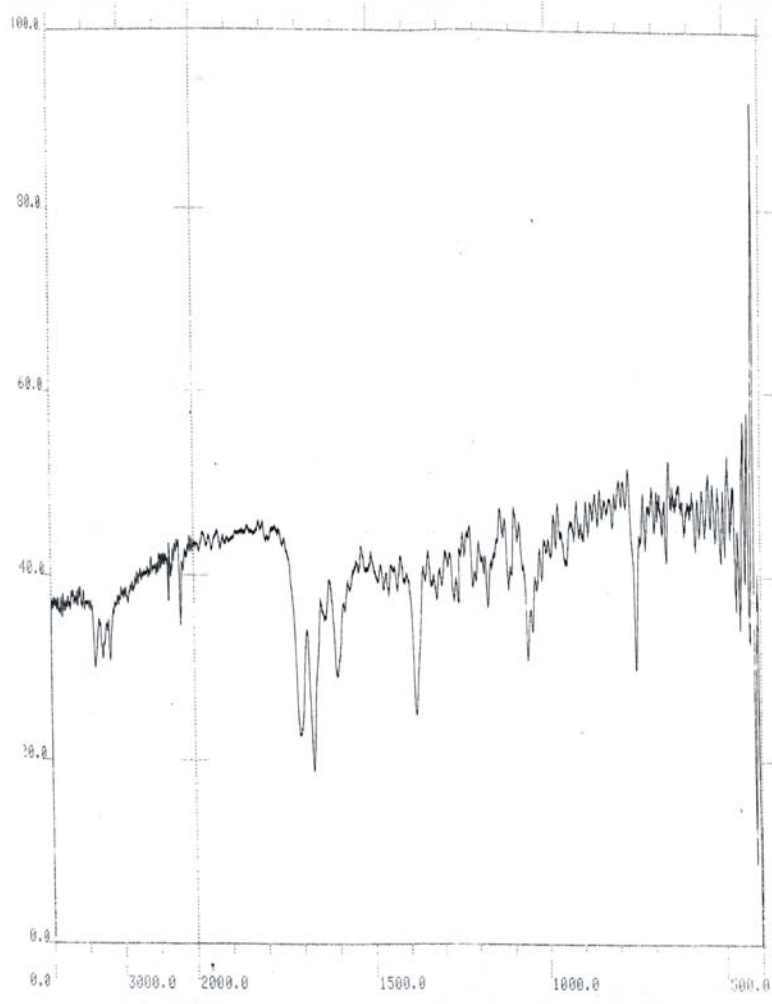


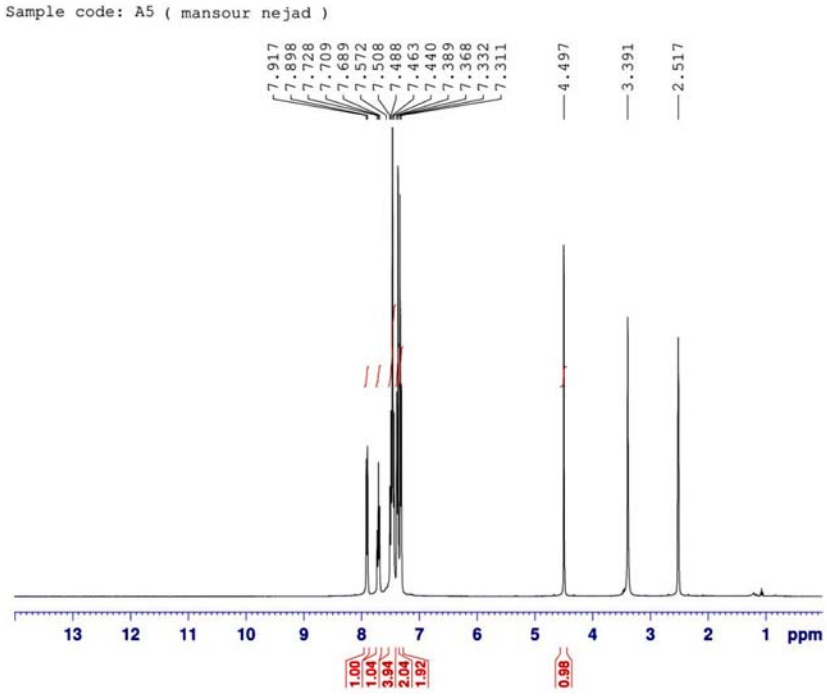



```
NAME          Azad-Gachsaran UN
EXPNO                    347
PROCNO                     1
Date_               20120320
Time                   19.00
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg
TD                    65536
SOLVENT                DMSO
NS                       120
DS                         0
SWH               25252.525 Hz
FIDRES                0.385323 Hz
AQ                 1.2976629 sec
RG                      2050
DW                    19.800 usec
DE                     6.50 usec
TE                   298.5 K
D1                 3.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                     9.00 usec
PL1                   -0.90 dB
PL1W              42.02801895 W
SFO1              100.6479784 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 90.00 usec
PL2                   -2.00 dB
PL12                  14.16 dB
PL13                  17.90 dB
PL2W              11.86359406 W
PL12W              0.28722104 W
PL13W              0.12139934 W
SFO2              400.2216009 MHz
SI                      32768
SF                100.6353990 MHz
WDW                       EM
SSB                        0
LB                     1.00 Hz
GB                         0
PC                      1.40
```

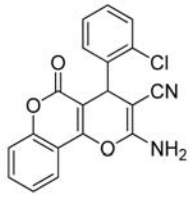


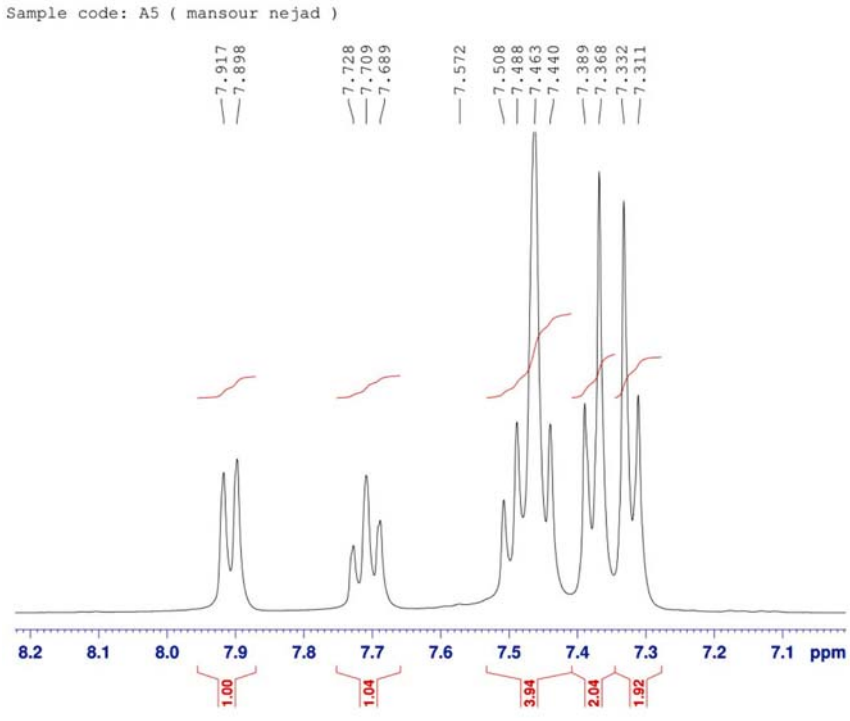

Sample code: A4 (mansour nejad)
160.02
158.46
153.90
152.62
143.81
133.41
129.00
128.10
127.60
125.16
122.95
119.70
117.05
113.45
104.49
BRUKER
NAME Azad-Gachsaran UN
EXPNO 347
PROCNO 1
Date_ 20120320
Time 19.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 120
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.5 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 155 150 145 140 135 130 125 120 115 110 105 ppm
O
O
CN
O
NH2

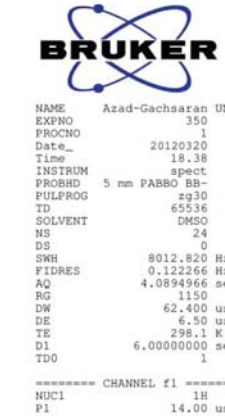

100.0
80.0
60.0
40.0
20.0
0.0
0.0
3000.0
2000.0
1500.0
1000.0
500.0 40
O
O
Cl
CN
O
NH2

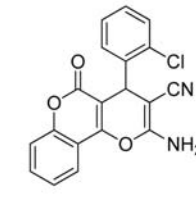

Sample code: A5 (mansour nejad)
7.917
7.898
7.728
7.709
7.689
7.572
7.508
7.488
7.463
7.440
7.389
7.368
7.332
7.311
4.497
3.391
2.517
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.00 1.04 3.94 2.04 1.92 0.98

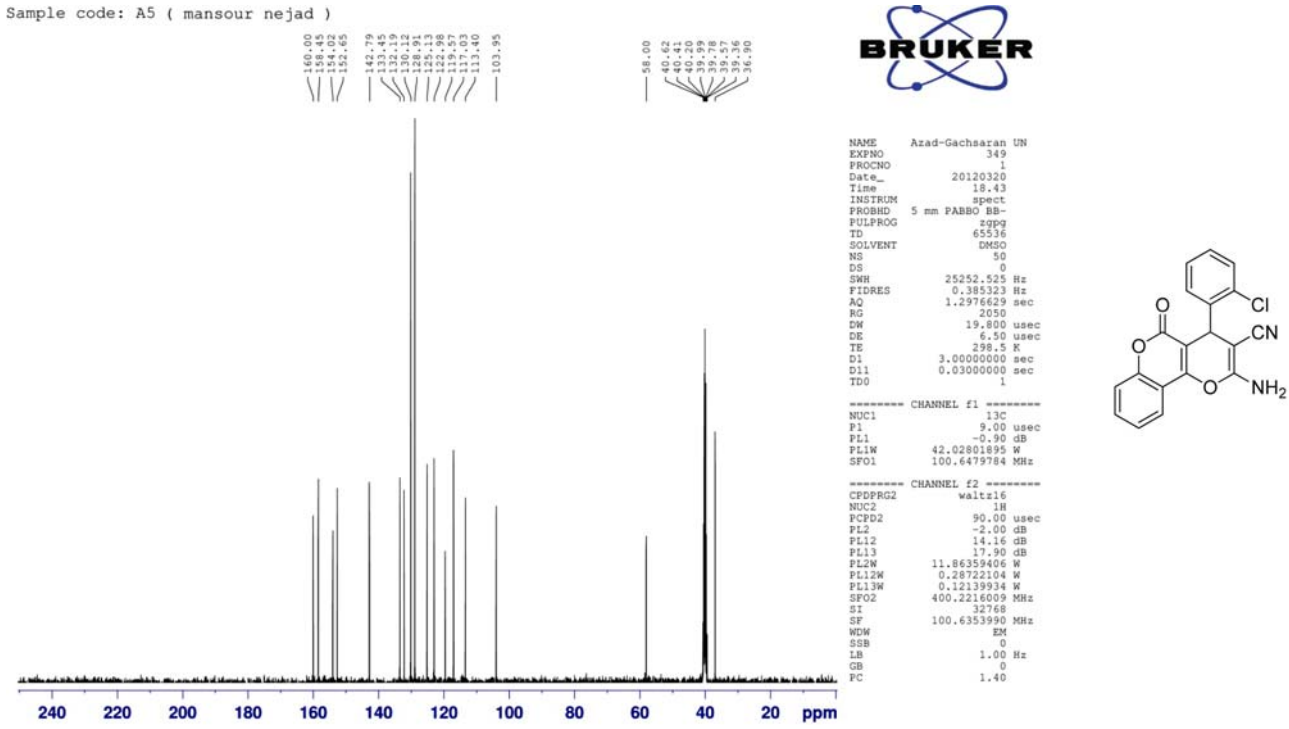

BRUKER
NAME Azad-Gachsaran UN
EXPNO 350
PROCNO 1
Date_ 20120320
Time 18.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 1150
DW 62.400 usec
DE 6.50 usec
TE 298.1 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

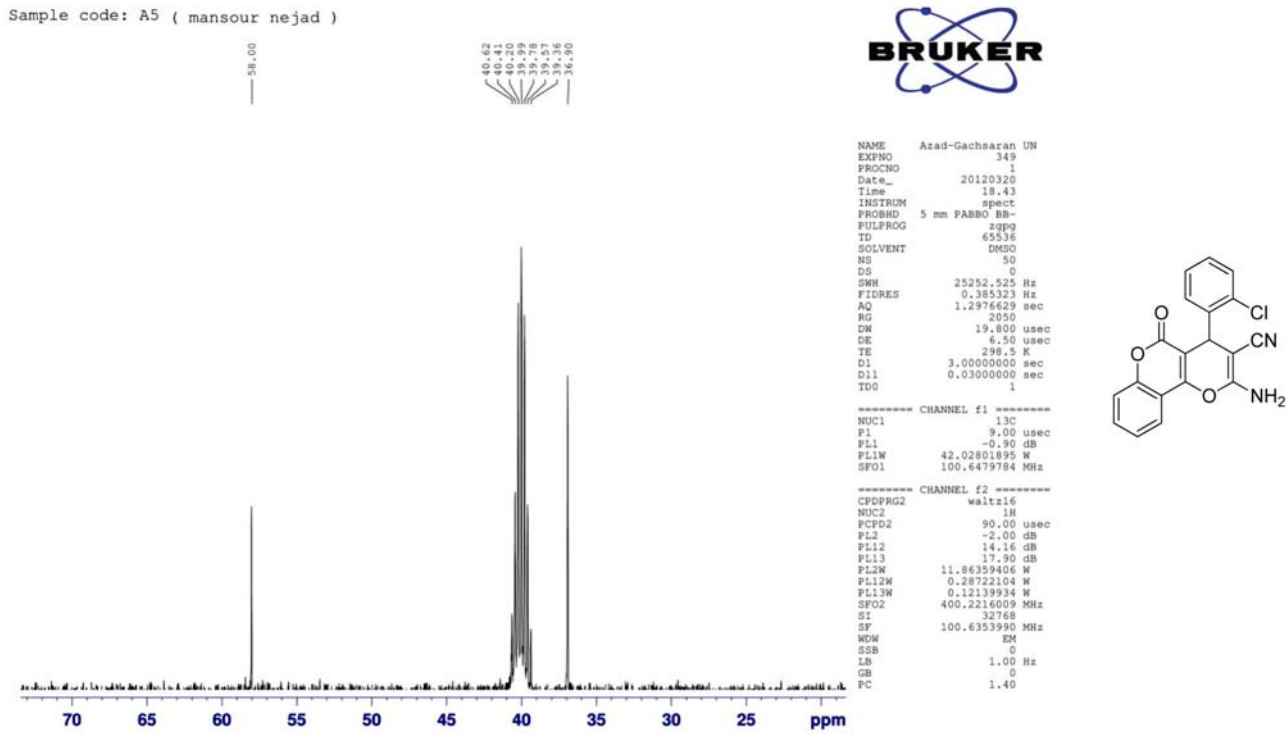

O
O
O
Cl
CN
NH2

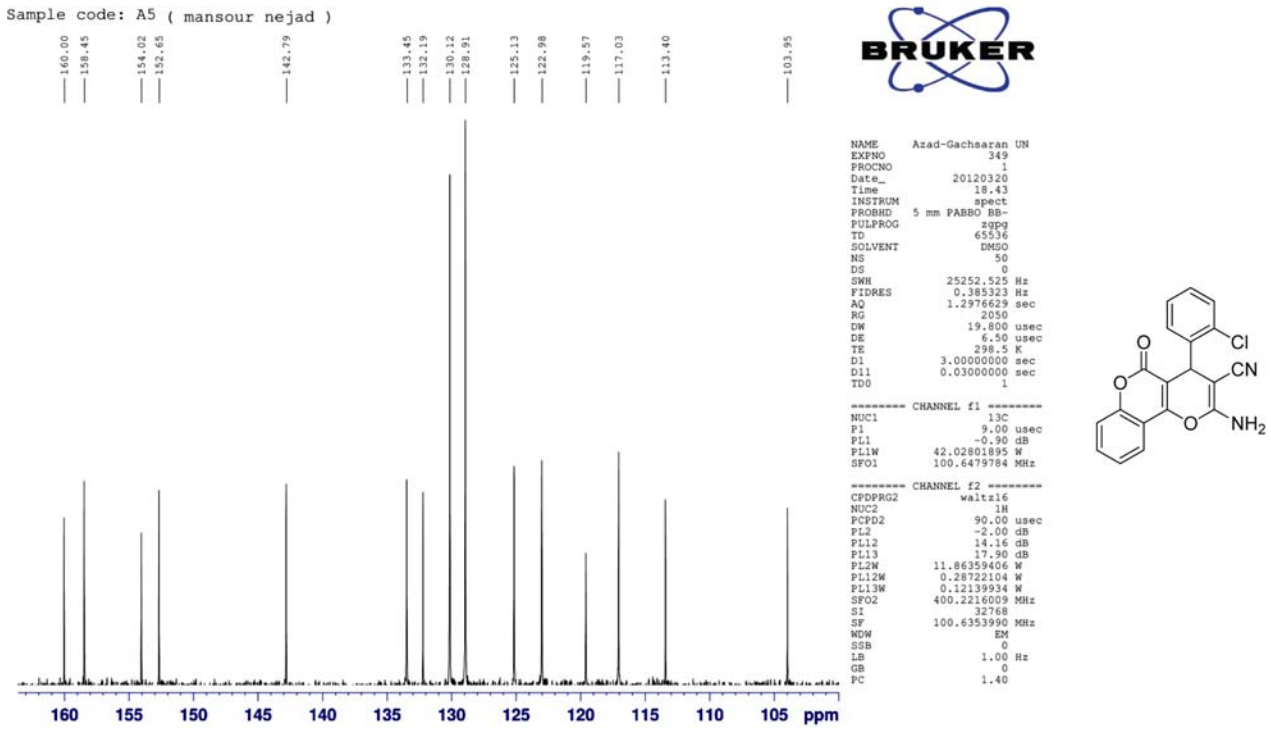

Sample code: A5 (mansour nejad)
7.917
7.898
7.728
7.709
7.689
7.572
7.508
7.488
7.463
7.440
7.389
7.368
7.332
7.311
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 ppm
1.00 1.04 3.94 2.04 1.92

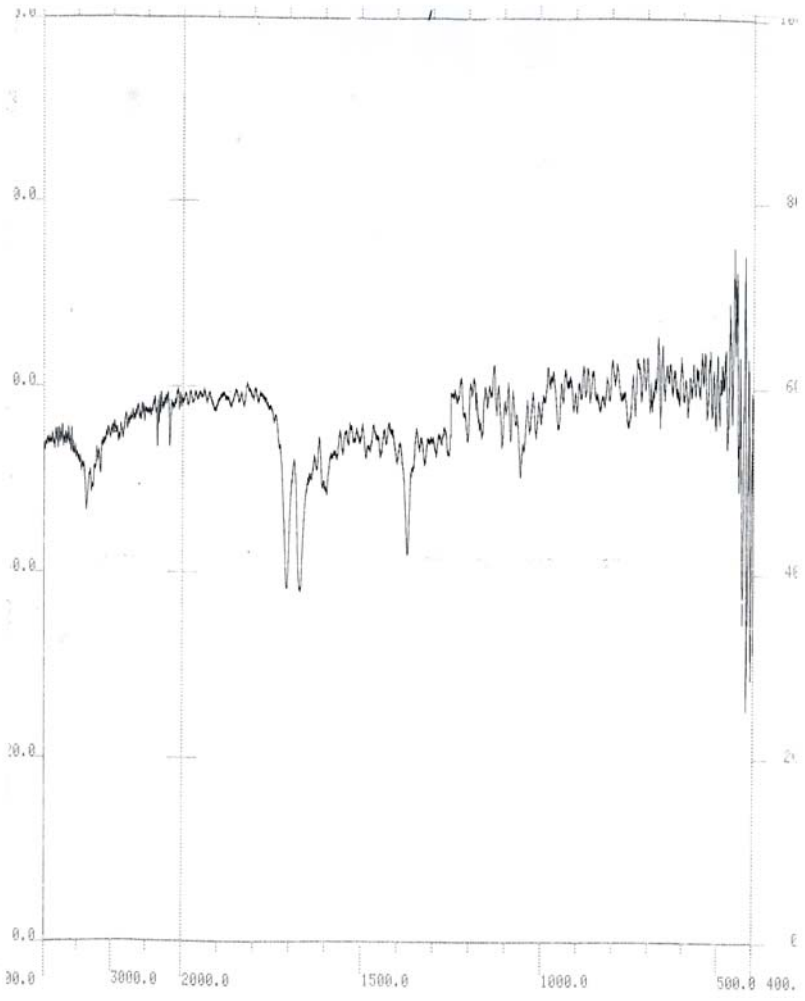

BRUKER
NAME Azad-Gachsaran UN
EXPNO 350
PROCNO 1
Date_ 20120320
Time 18.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 1150
DW 62.400 usec
DE 6.50 usec
TE 298.1 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

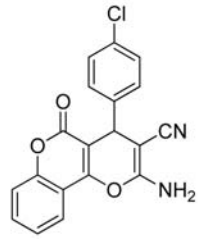

O
O
O
Cl
CN
NH2

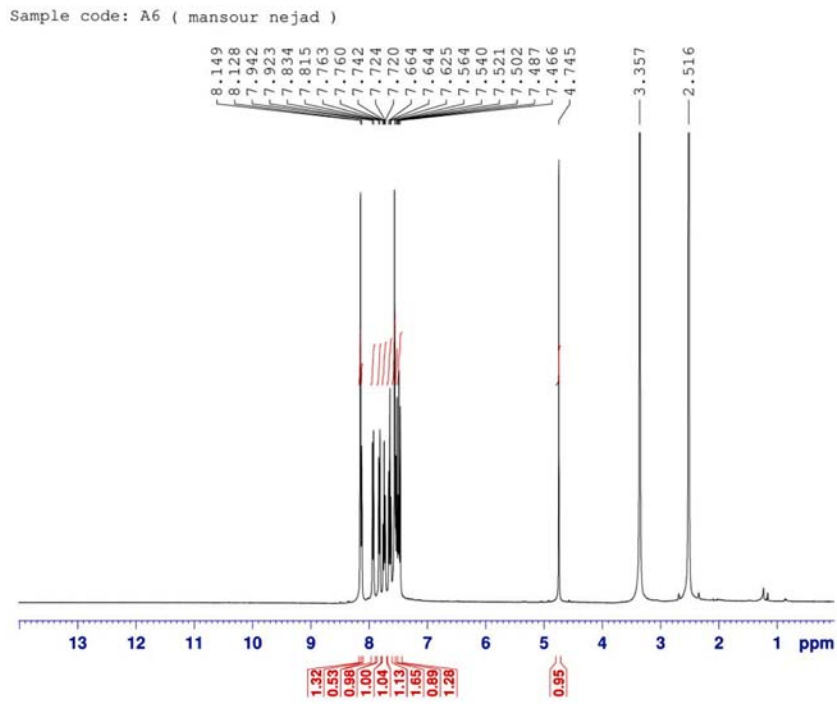

Sample code: A5 (mansour nejad)
BRUKER
160.00
158.45
154.02
152.65
142.79
133.45
132.19
130.12
128.91
125.13
124.98
124.57
117.03
113.40
103.95
58.00
40.62
40.41
40.20
39.99
39.78
39.57
39.36
36.90
NAME Azad-Gachsaran UN
EXPNO 349
PROCNO 1
Date_ 20120320
Time 18.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 50
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.5 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
240 220 200 180 160 140 120 100 80 60 40 20 ppm
O
O
Cl
CN
O
NH2


Sample code: A5 (mansour nejad)
BRUKER
58.00
40.62
40.41
40.20
39.99
39.78
39.57
39.36
36.90
NAME Azad-Gachsaran UN
EXPNO 349
PROCNO 1
Date_ 20120320
Time 18.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 50
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.5 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
70 65 60 55 50 45 40 35 30 25 ppm
O
O
Cl
CN
O
NH2

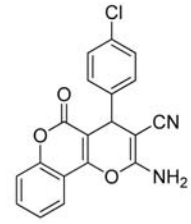

Sample code: A5 (mansour nejad)
BRUKER
160.00
158.45
154.02
152.65
142.79
133.45
132.19
130.12
128.91
125.13
122.98
119.57
117.03
113.40
103.95
NAME Azad-Gachsaran UN
EXPNO 349
PROCNO 1
Date_ 20120320
Time 18.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 50
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.5 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 155 150 145 140 135 130 125 120 115 110 105 ppm
Cl
CN
NH2
O

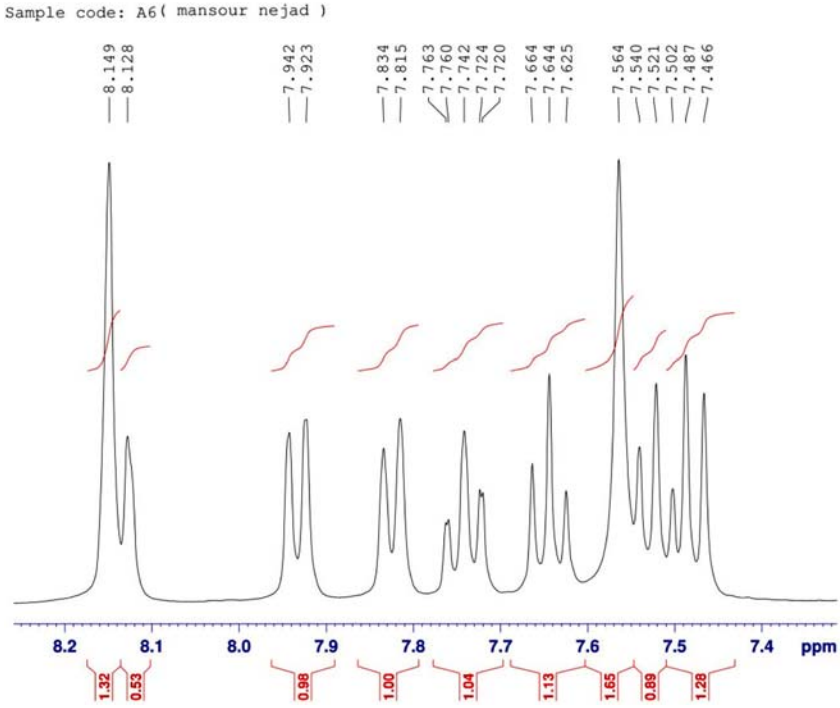

3000.0
2000.0
1500.0
1000.0
500.0
400.


Cl
CN
NH2
O

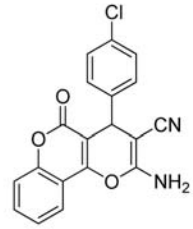


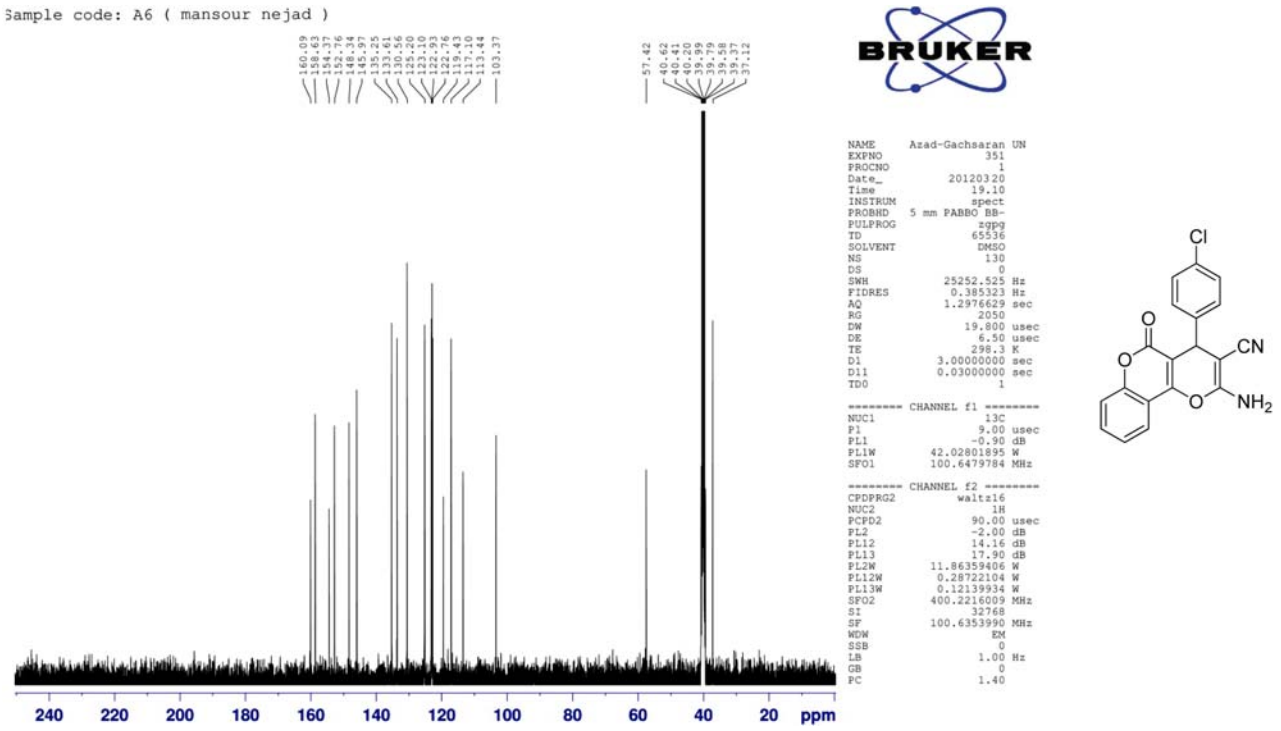

```
NAME          Azad-Gachsaran UN
EXPNO                    352
PROCNO                     1
Date_               20120320
Time                   19.05
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       24
DS                        0
SWH                8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                     2050
DW                   62.400 usec
DE                    6.50 usec
TE                   298.0 K
D1                6.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     14.00 usec
PL1                    -2.00 dB
PL1W           11.86359406 W
SFO1           400.2236020 MHz
SI                      32768
SF             400.2200000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

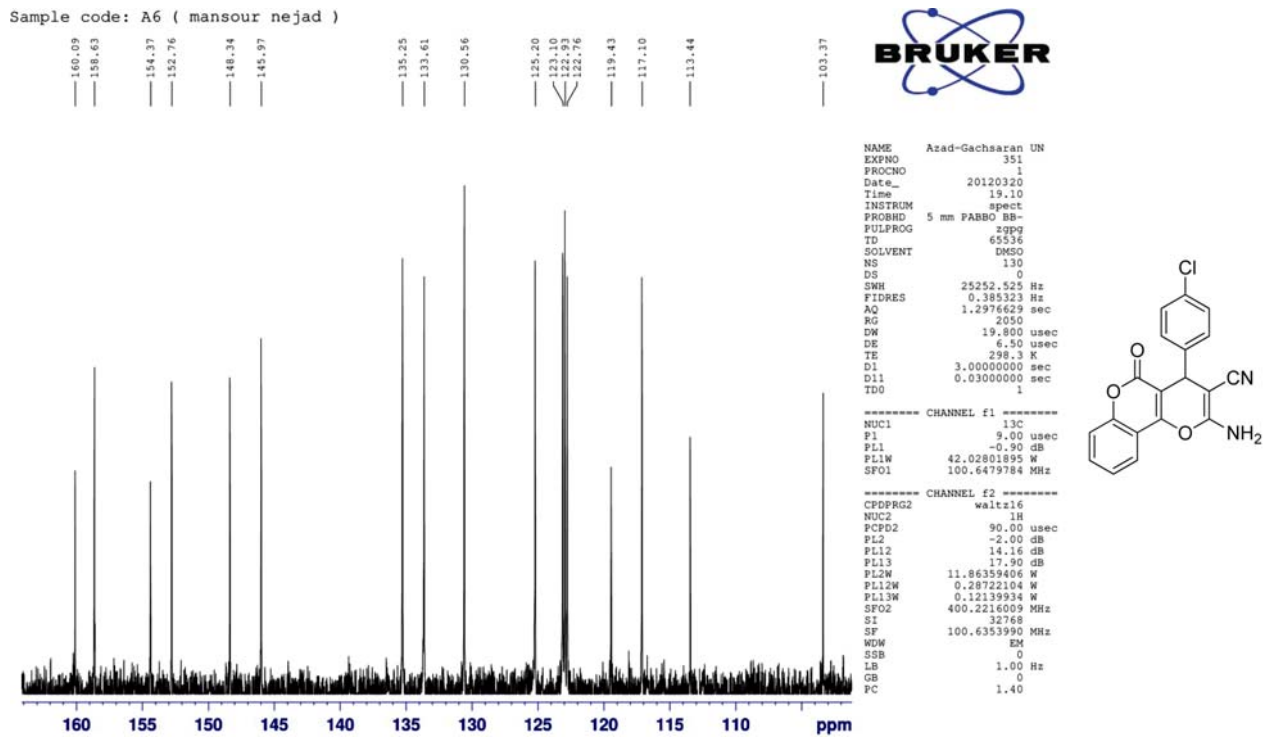


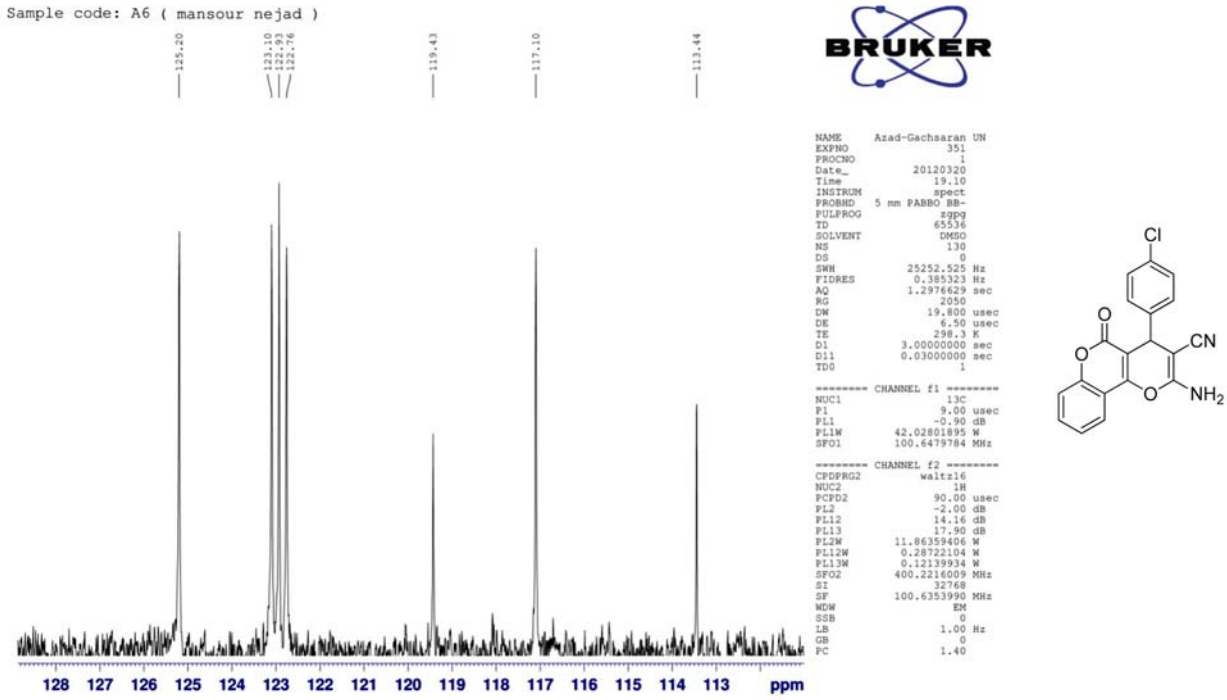


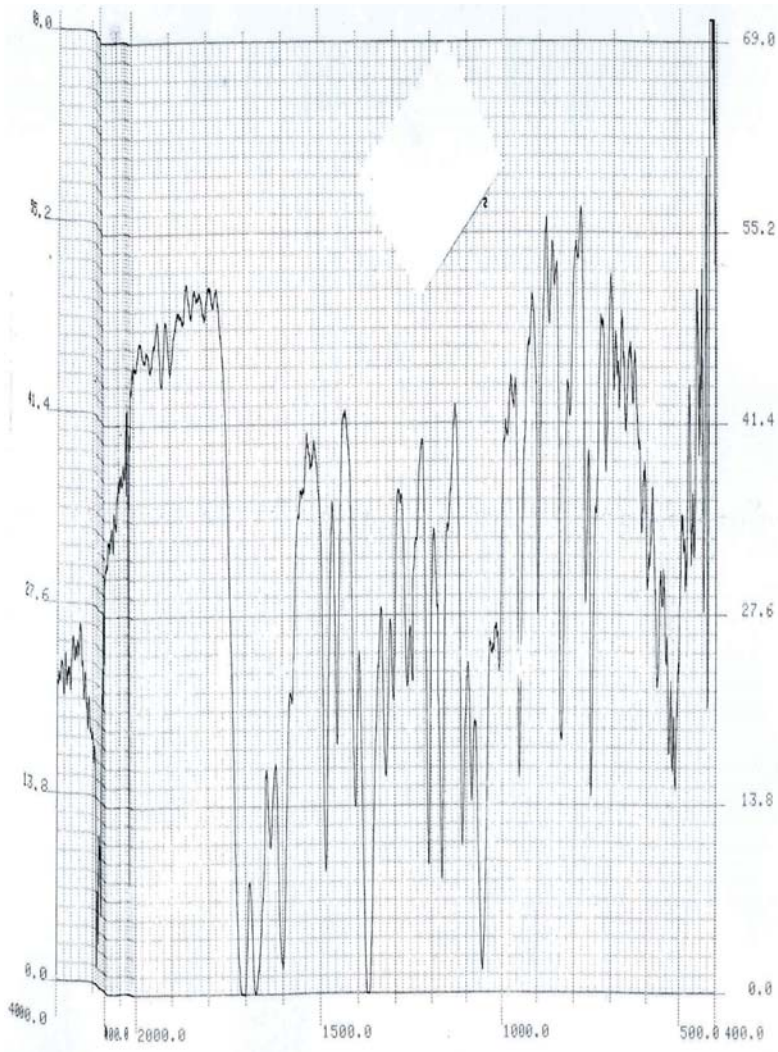

```
NAME          Azad-Gachsaran UN
EXPNO                    352
PROCNO                     1
Date_               20120320
Time                   19.05
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       24
DS                        0
SWH                8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                     2050
DW                   62.400 usec
DE                    6.50 usec
TE                   298.0 K
D1                6.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     14.00 usec
PL1                    -2.00 dB
PL1W           11.86359406 W
SFO1           400.2236020 MHz
SI                      32768
SF             400.2200000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

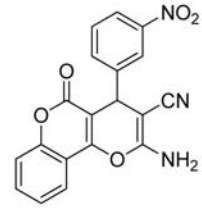


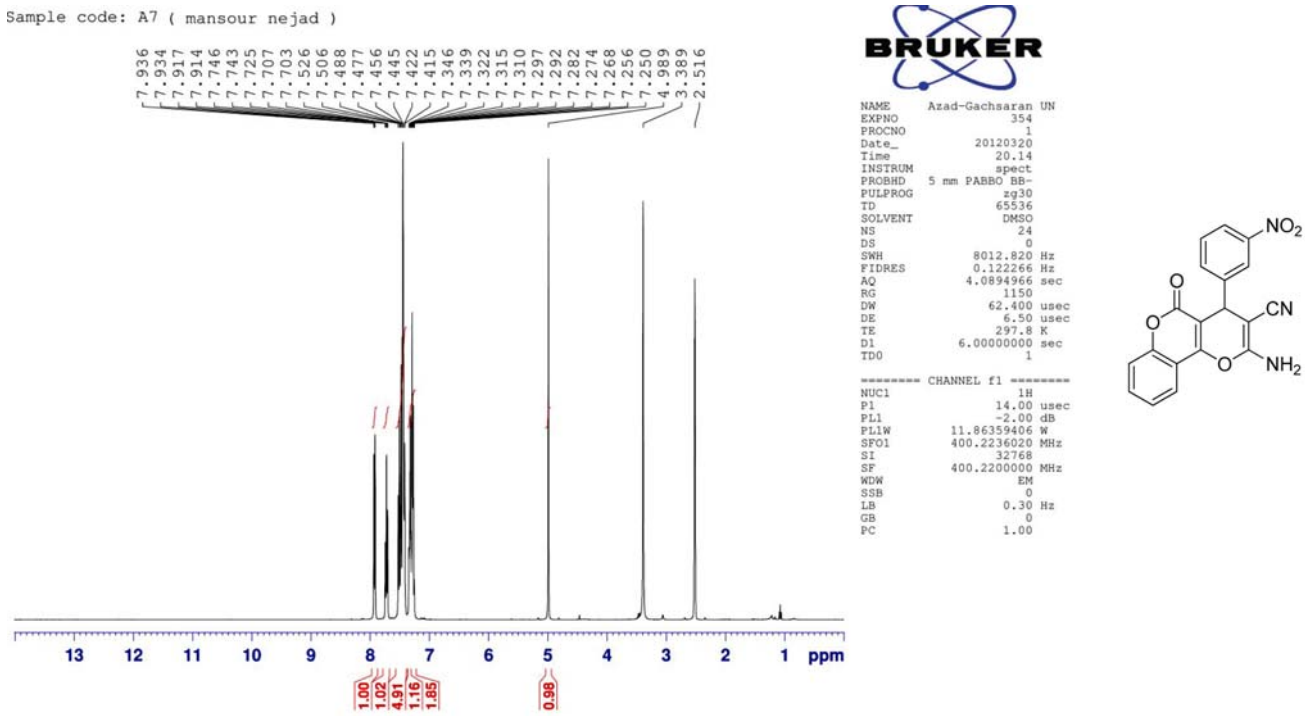

Sample code: A6 (mansour nejad)
BRUKER
160.09
158.63
154.37
152.76
148.34
145.97
135.25
133.61
130.56
125.20
123.10
122.93
122.76
119.43
117.10
113.44
103.37
57.42
40.62
40.41
40.20
39.99
39.79
39.58
39.37
37.12
NAME Azad-Gachsaran UN
EXPNO 351
PROCNO 1
Date_ 20120320
Time 19.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 130
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.3 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
240 220 200 180 160 140 120 100 80 60 40 20 ppm
Cl
O
O
CN
O
NH2

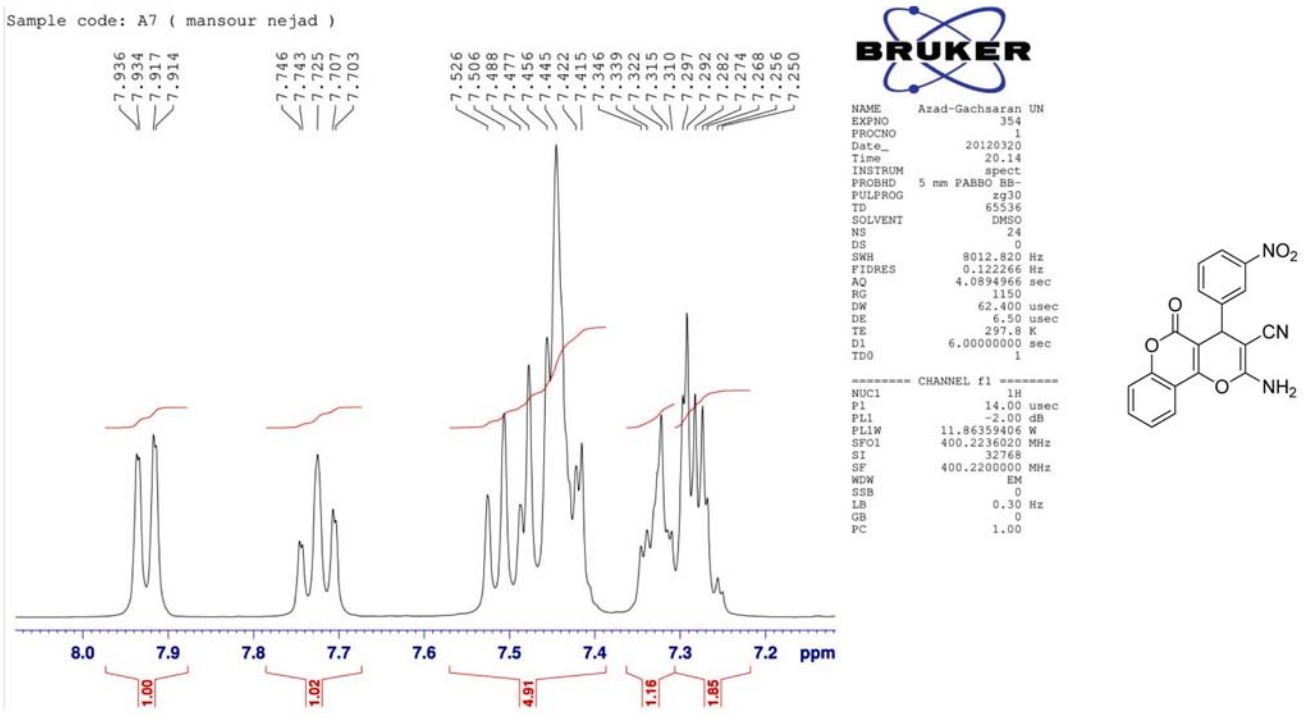

Sample code: A6 (mansour nejad)
BRUKER
160.09
158.63
154.37
152.76
148.34
145.97
135.25
133.61
130.56
125.20
123.10
122.93
122.76
119.43
117.10
113.44
103.37
NAME Azad-Gachsaran UN
EXPNO 351
PROCNO 1
Date_ 20120320
Time 19.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 130
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.3 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 155 150 145 140 135 130 125 120 115 110 ppm
Cl
O
O
CN
O
NH2

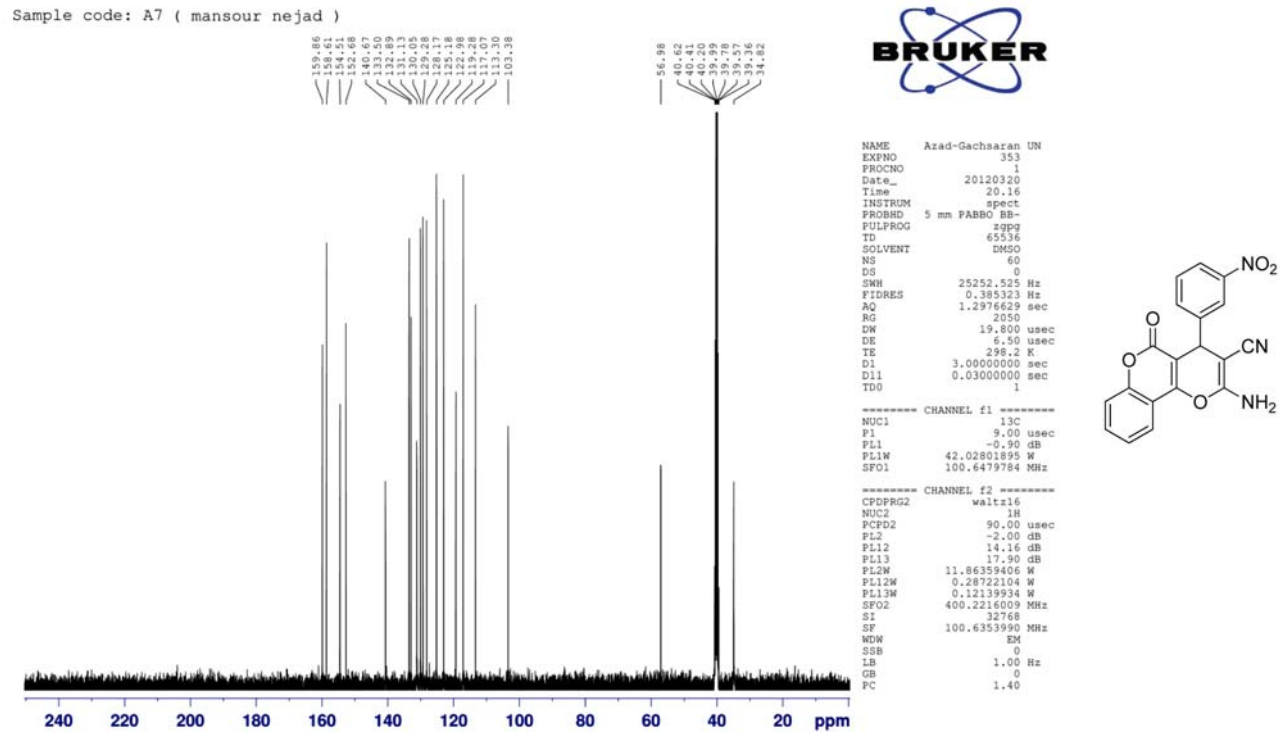

Sample code: A6 (mansour nejad)
BRUKER
125.20
123.10
122.81
122.76
119.43
117.10
113.44
NAME Azad-Gachsaran UN
EXPNO 351
PROCNO 1
Date_ 20120320
Time 19.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 130
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.3 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
128 127 126 125 124 123 122 121 120 119 118 117 116 115 114 113 ppm
Cl
O
O
CN
O
NH2

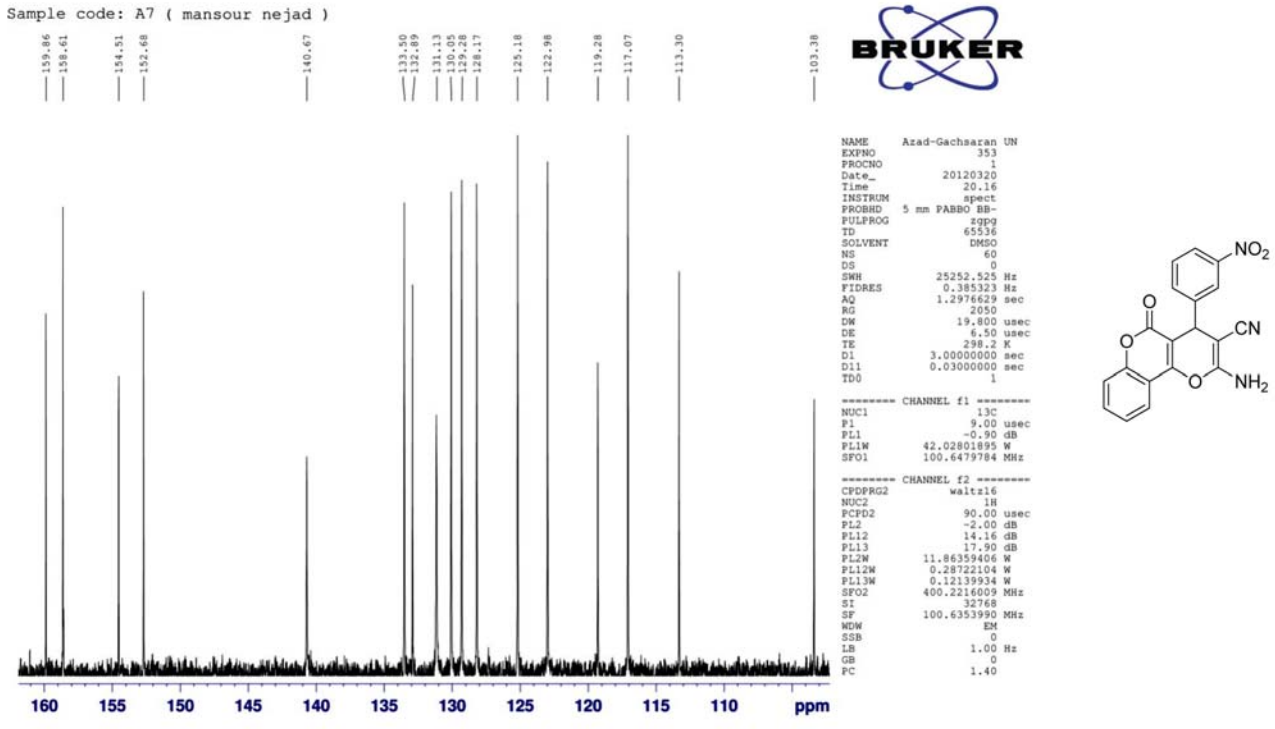

69.0
55.2
41.4
27.6
13.8
0.0
2000.0
1500.0
1000.0
500.0
400.0

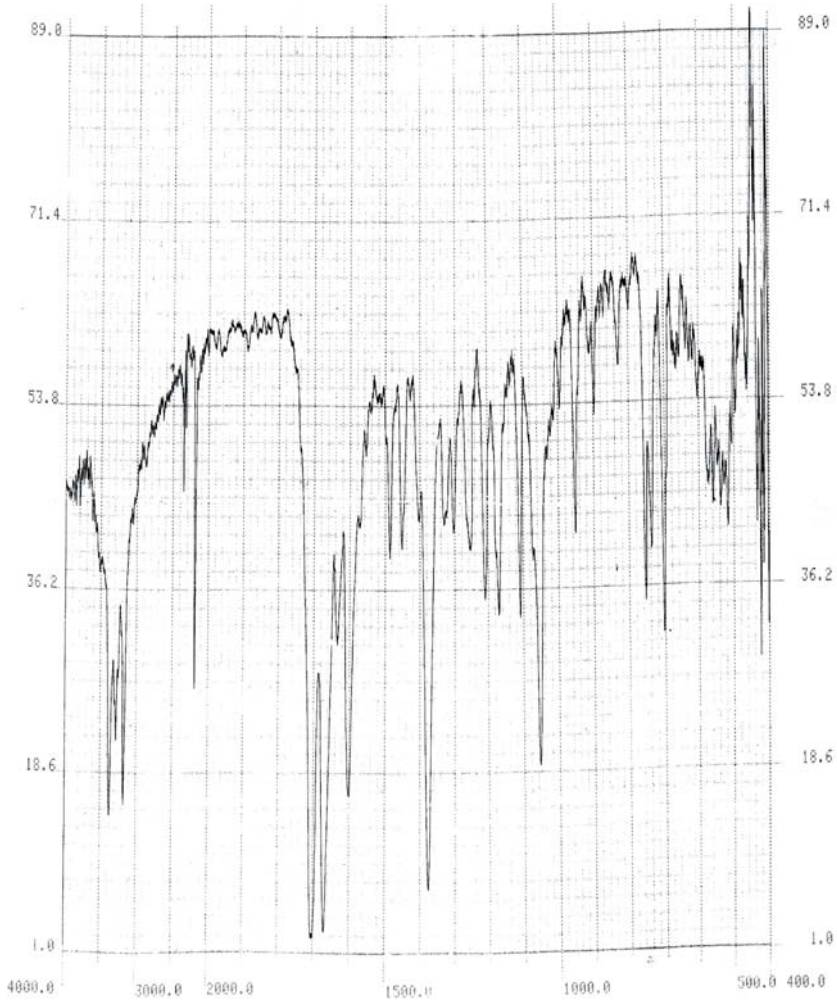

NO2
O
O
CN
O
NH2

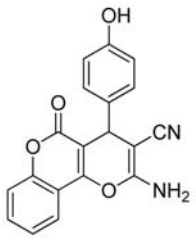

Sample code: A7 (mansour nejad)
7.936
7.934
7.917
7.914
7.746
7.743
7.725
7.707
7.703
7.526
7.506
7.488
7.477
7.456
7.445
7.422
7.415
7.346
7.339
7.322
7.315
7.310
7.297
7.292
7.282
7.274
7.268
7.256
7.250
4.989
3.389
2.516
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.00 1.02 4.91 1.16 1.85 0.98
BRUKER
NAME Azad-Gachsaran UN
EXPNO 354
PROCNO 1
Date_ 20120320
Time 20.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 1150
DW 62.400 usec
DE 6.50 usec
TE 297.8 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NO2
CN
NH2
O

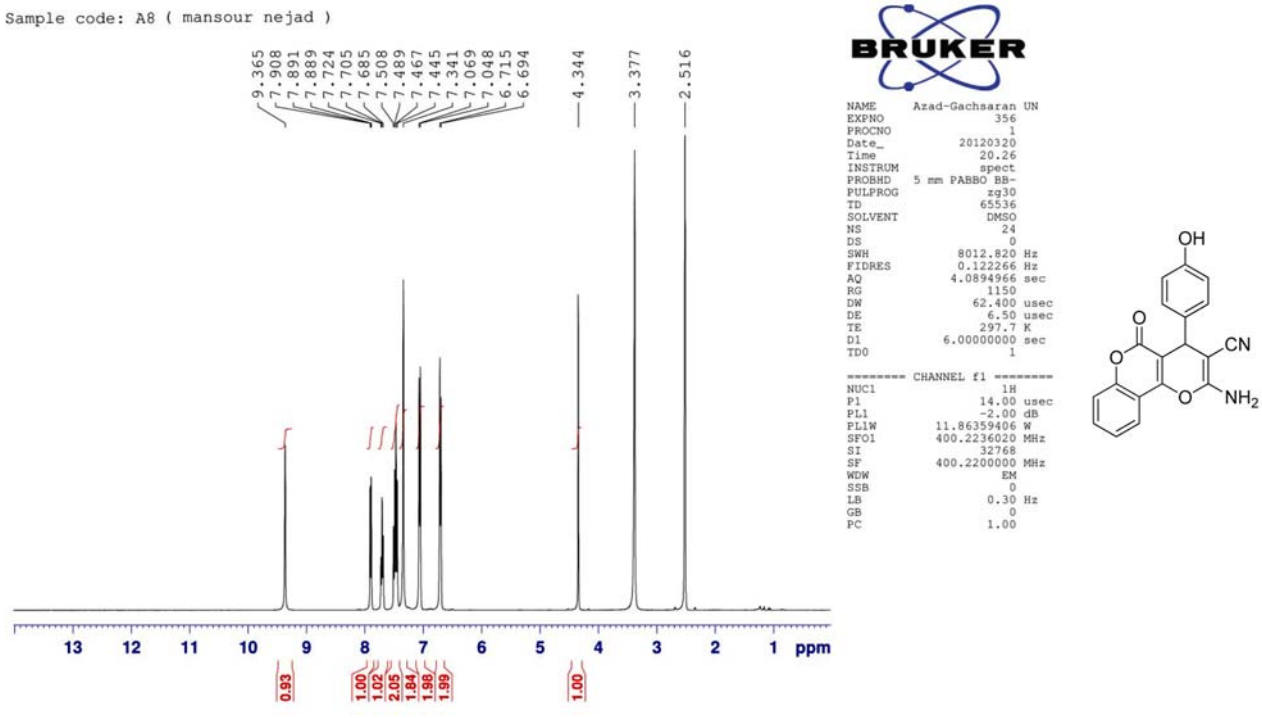

Sample code: A7 (mansour nejad)
7.936
7.934
7.917
7.914
7.746
7.743
7.725
7.707
7.703
7.526
7.506
7.488
7.477
7.456
7.445
7.422
7.415
7.346
7.339
7.322
7.315
7.310
7.297
7.292
7.282
7.274
7.268
7.256
7.250
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 ppm
1.00 1.02 4.91 1.16 1.85
BRUKER
NAME Azad-Gachsaran UN
EXPNO 354
PROCNO 1
Date_ 20120320
Time 20.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 1150
DW 62.400 usec
DE 6.50 usec
TE 297.8 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NO2
CN
NH2
O


Sample code: A7 (mansour nejad)
BRUKER
159.86
158.61
154.51
152.68
140.67
133.50
132.89
131.13
130.05
129.28
128.17
125.18
122.98
119.28
117.07
113.30
103.38
56.98
40.62
40.41
40.20
39.99
39.78
39.57
39.36
34.82
NAME Azad-Gachsaran UN
EXPNO 353
PROCNO 1
Date_ 20120320
Time 20.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 60
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.2 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
240 220 200 180 160 140 120 100 80 60 40 20 ppm
NO2
CN
NH2
O


Sample code: A7 (mansour nejad)
BRUKER
159.86
158.61
154.51
152.68
140.67
133.50
132.89
131.13
130.05
129.28
128.17
125.18
122.98
119.28
117.07
113.30
103.38
NAME Azad-Gachsaran UN
EXPNO 353
PROCNO 1
Date_ 20120320
Time 20.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 60
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.2 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 155 150 145 140 135 130 125 120 115 110 ppm
NO2
CN
NH2
O

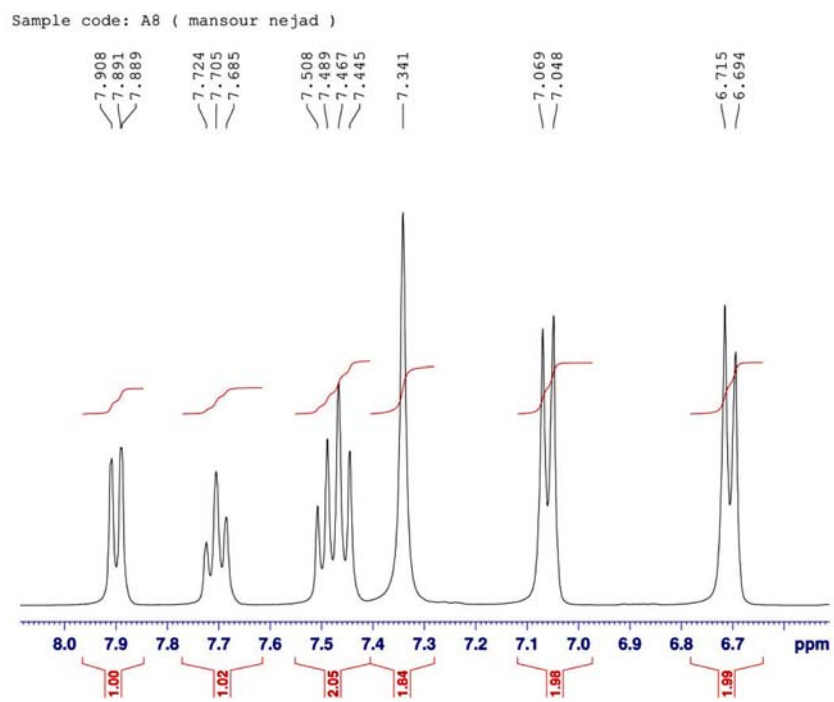




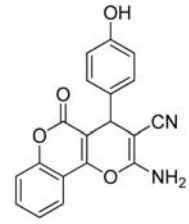


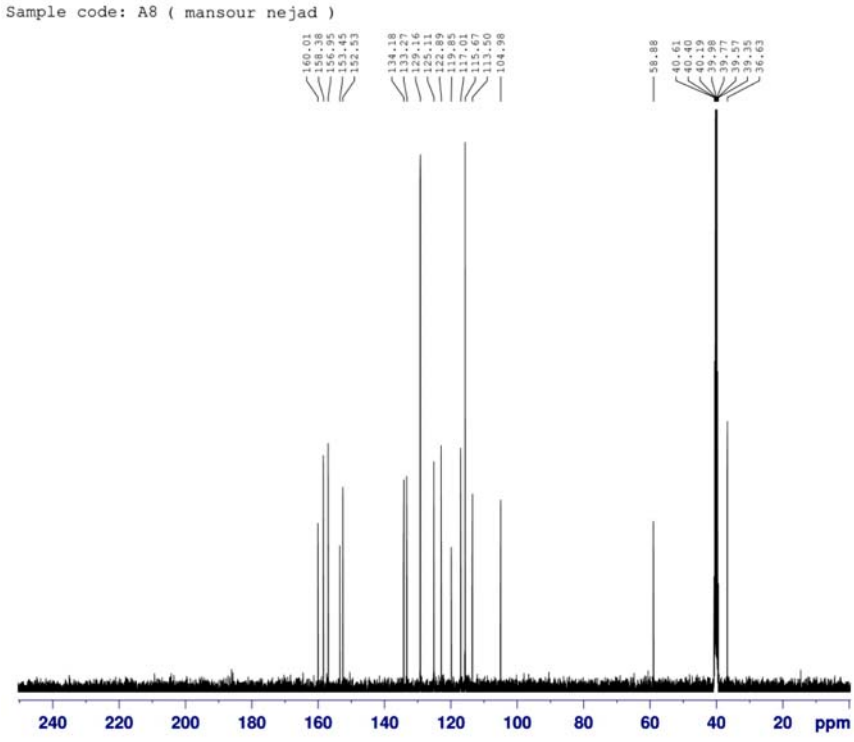
BRUKER

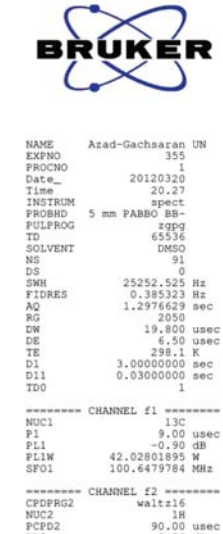
NAME	Azad-Gachsaran UN
EXPNO	356
PROCNO	1
Date_	20120320
Time	20.26
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	24
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	1150
DW	62.400 usec
DE	6.50 usec
TE	297.7 K
D1	6.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-2.00 dB
PL1W	11.86359406 W
SFO1	400.2236020 MHz
SI	32768
SF	400.2200000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

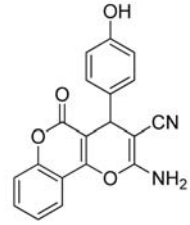

Sample code: A8 (mansour nejad)
7.908
7.891
7.889
7.724
7.705
7.685
7.508
7.489
7.467
7.445
7.341
7.069
7.048
6.715
6.694
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 ppm
1.00
1.02
2.05
1.84
1.98
1.89

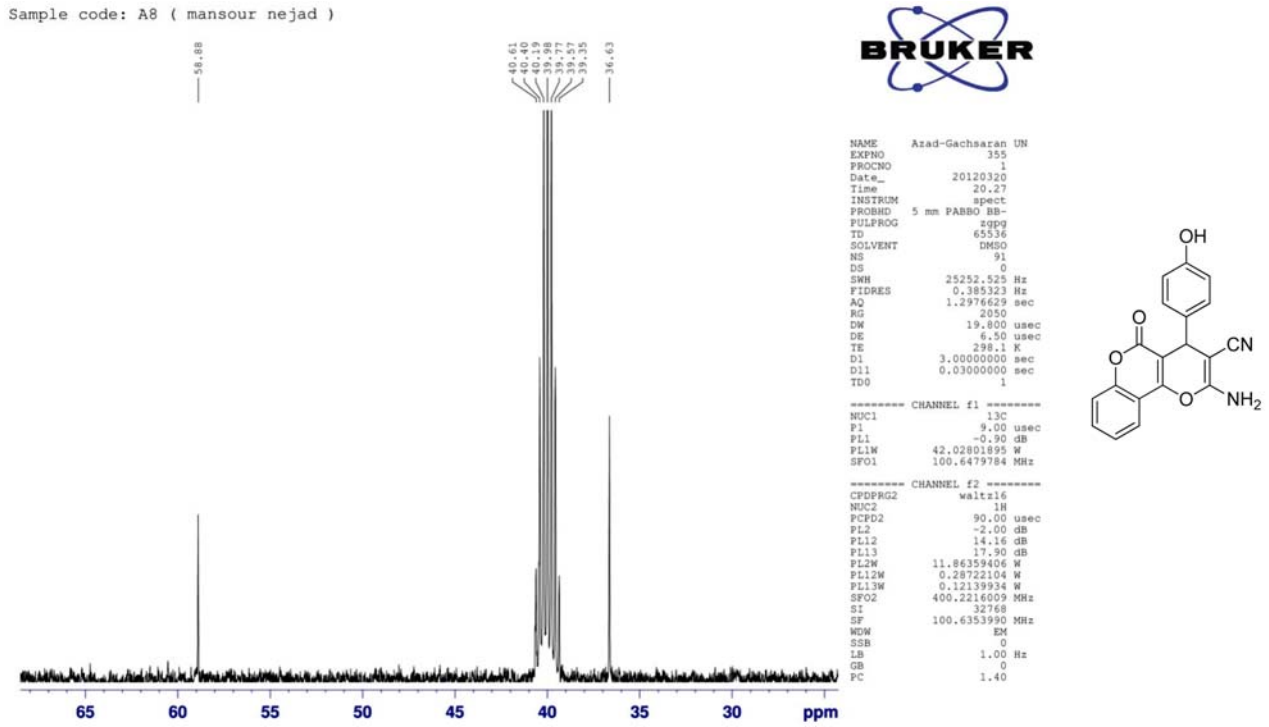

BRUKER
NAME Azad-Gachsaran UN
EXPNO 356
PROCNO 1
Date_ 20120320
Time 20.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 1150
DW 62.400 usec
DE 6.50 usec
TE 297.7 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


OH
O
O
CN
O
NH2

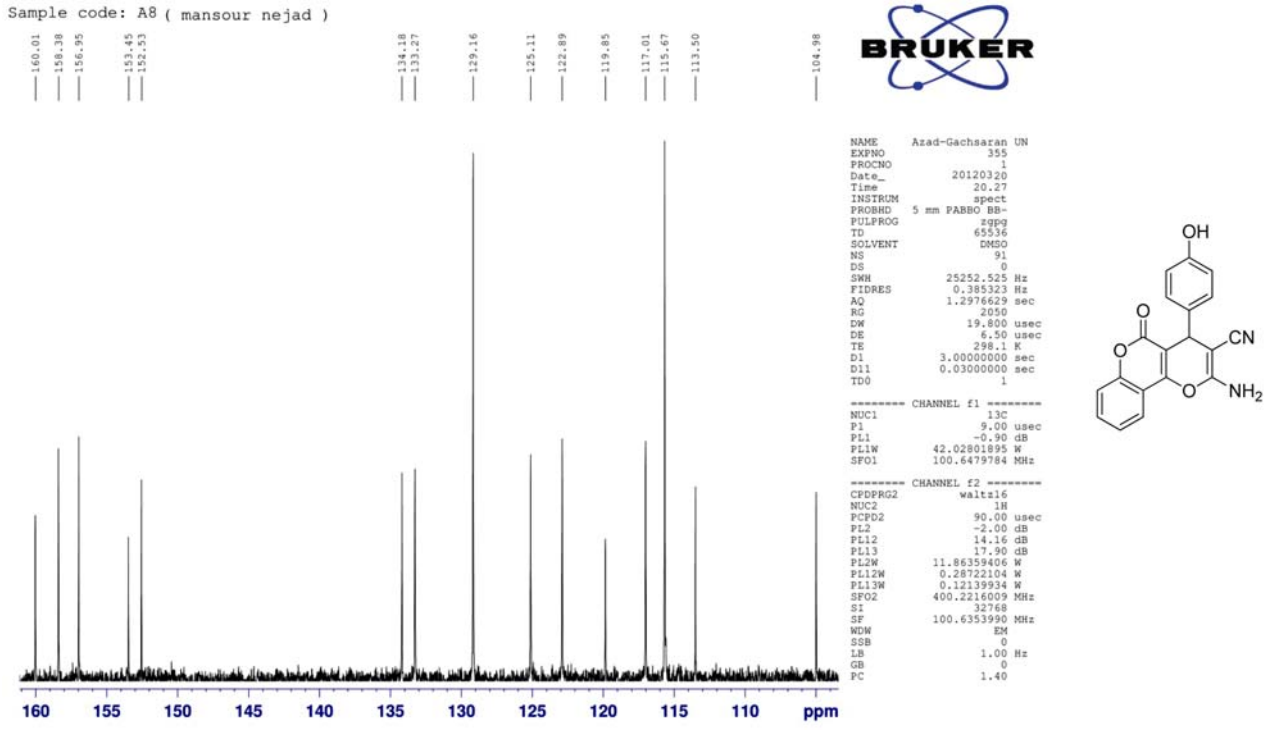

Sample code: A8 (mansour nejad)
160.01
158.38
156.95
153.45
152.53
134.18
133.27
129.16
125.11
122.89
119.85
117.01
115.67
113.50
104.98
58.88
40.61
40.40
40.19
39.98
39.77
39.57
39.35
36.63
240 220 200 180 160 140 120 100 80 60 40 20 ppm


BRUKER
NAME Azad-Gachsaran UN
EXPNO 355
PROCNO 1
Date_ 20120320
Time 20.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 91
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.1 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

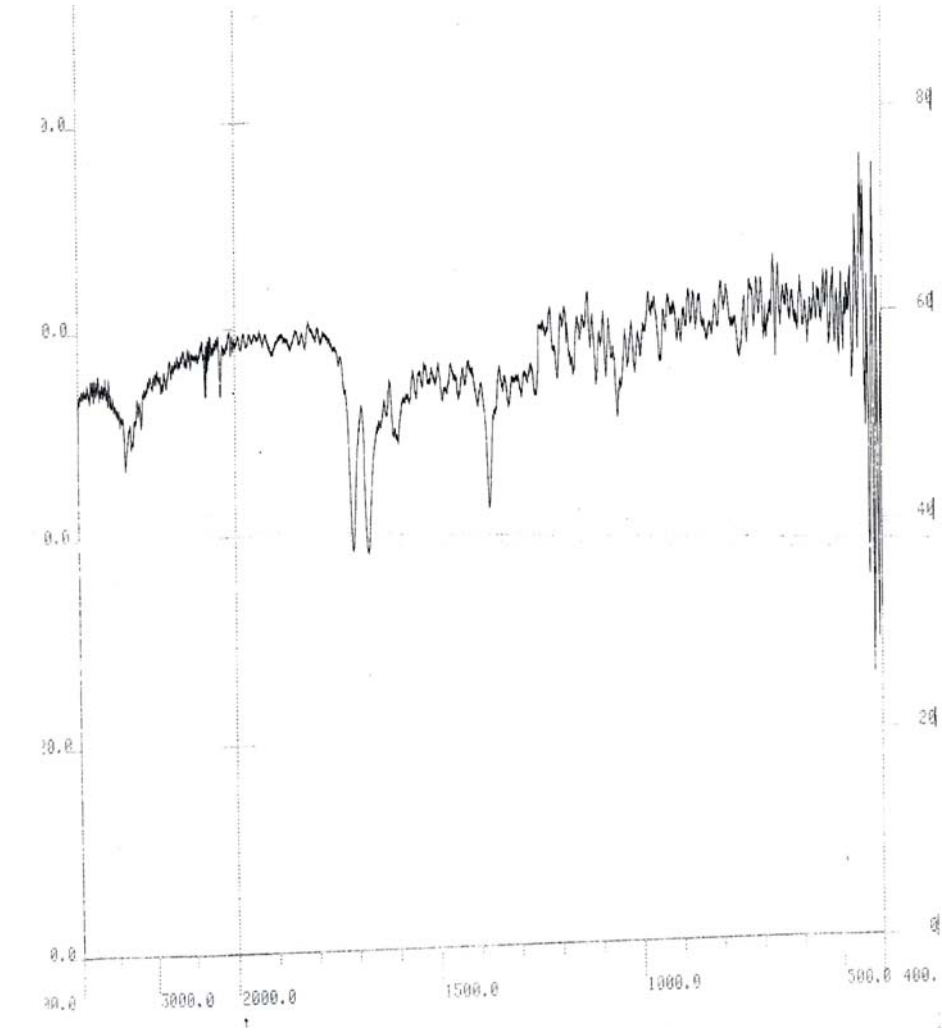

OH
O
O
CN
O
NH2

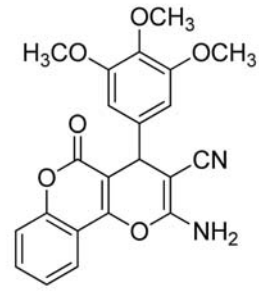


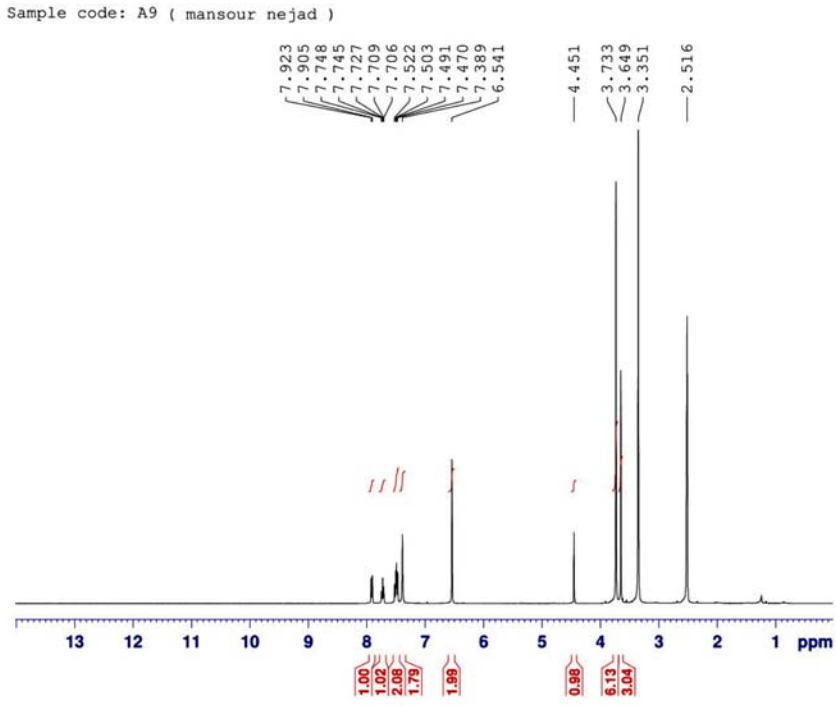

```
NAME        Azad-Gachsaran UN
EXPNO                  355
PROCNO                   1
Date_             20120320
Time                 20.27
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg
TD                  65536
SOLVENT              DMSO
NS                     91
DS                      0
SWH           25252.525 Hz
FIDRES        0.385323 Hz
AQ            1.2976629 sec
RG                   2050
DW               19.800 usec
DE                6.50 usec
TE                298.1 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.00 usec
PL1               -0.90 dB
PL1W        42.02801895 W
SFO1       100.6479784 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               -2.00 dB
PL12              14.16 dB
PL13              17.90 dB
PL2W        11.86359406 W
PL12W        0.28722104 W
PL13W        0.12139934 W
SFO2       400.2216009 MHz
SI                 32768
SF         100.6353990 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```


```
NAME        Azad-Gachsaran UN
EXPNO                  355
PROCNO                   1
Date_             20120320
Time                 20.27
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg
TD                  65536
SOLVENT              DMSO
NS                     91
DS                      0
SWH           25252.525 Hz
FIDRES        0.385323 Hz
AQ            1.2976629 sec
RG                   2050
DW               19.800 usec
DE                6.50 usec
TE                298.1 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.00 usec
PL1               -0.90 dB
PL1W        42.02801895 W
SFO1       100.6479784 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               -2.00 dB
PL12              14.16 dB
PL13              17.90 dB
PL2W        11.86359406 W
PL12W        0.28722104 W
PL13W        0.12139934 W
SFO2       400.2216009 MHz
SI                 32768
SF         100.6353990 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

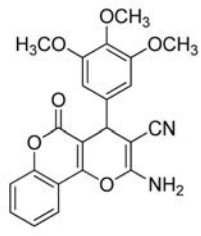


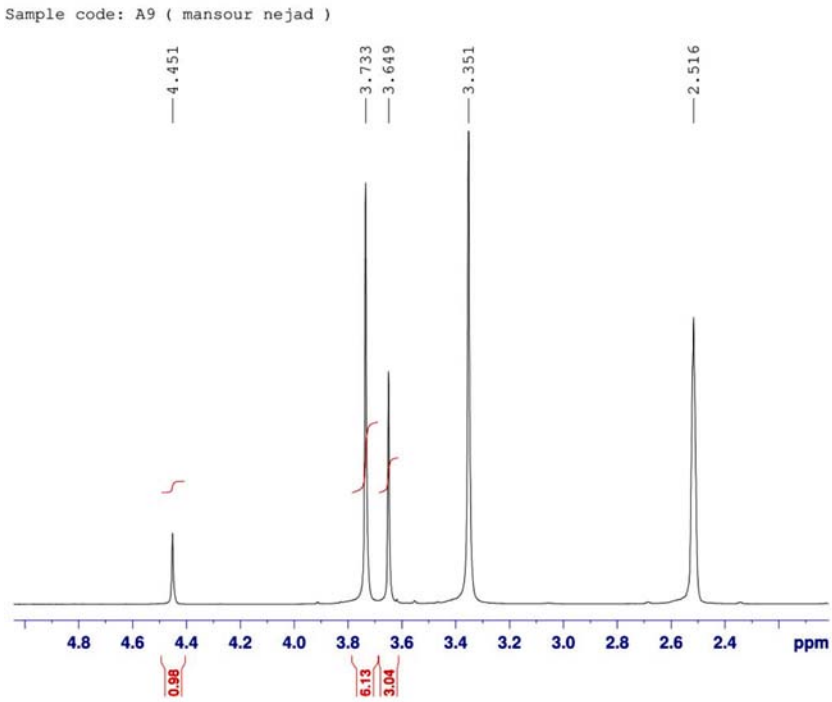




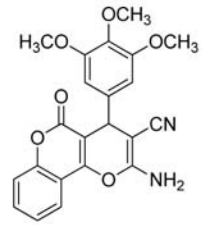
BRUKER

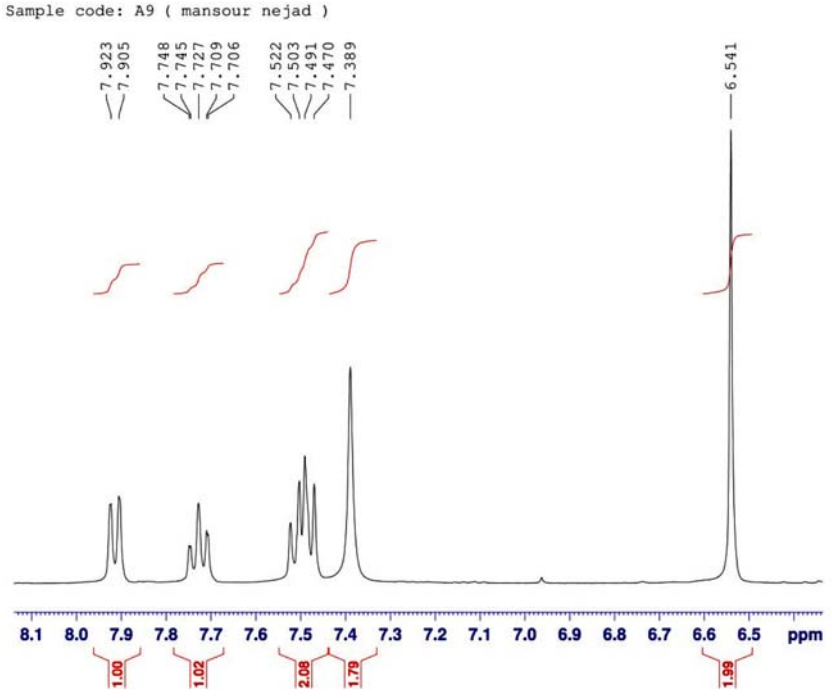
NAME	Azad-Gachsaran UN
EXPNO	358
PROCNO	1
Date_	20120320
Time	20.37
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	24
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	2050
DW	62.400 usec
DE	6.50 usec
TE	297.9 K
D1	6.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-2.00 dB
PL1W	11.86359406 W
SFO1	400.2236020 MHz
SI	32768
SF	400.2200000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



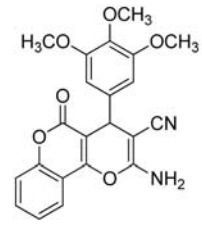


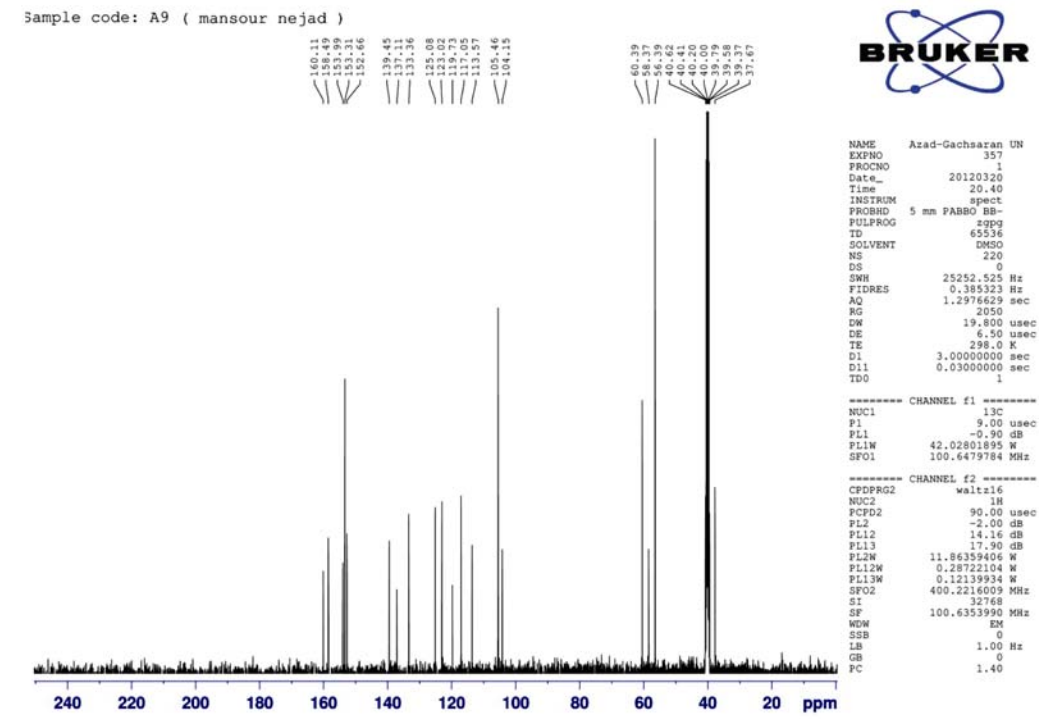

```
NAME          Azad-Gachsaran UN
EXPNO                    358
PROCNO                     1
Date_               20120320
Time                   20.37
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       24
DS                        0
SWH                8012.820 Hz
FIDRES                0.122266 Hz
AQ                 4.0894966 sec
RG                     2050
DW                   62.400 usec
DE                    6.50 usec
TE                   297.9 K
D1                6.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     14.00 usec
PL1                    -2.00 dB
PL1W              11.86359406 W
SFO1              400.2236020 MHz
SI                       32768
SF                400.2200000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

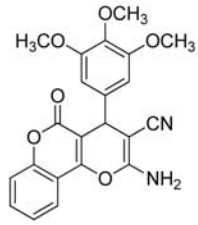


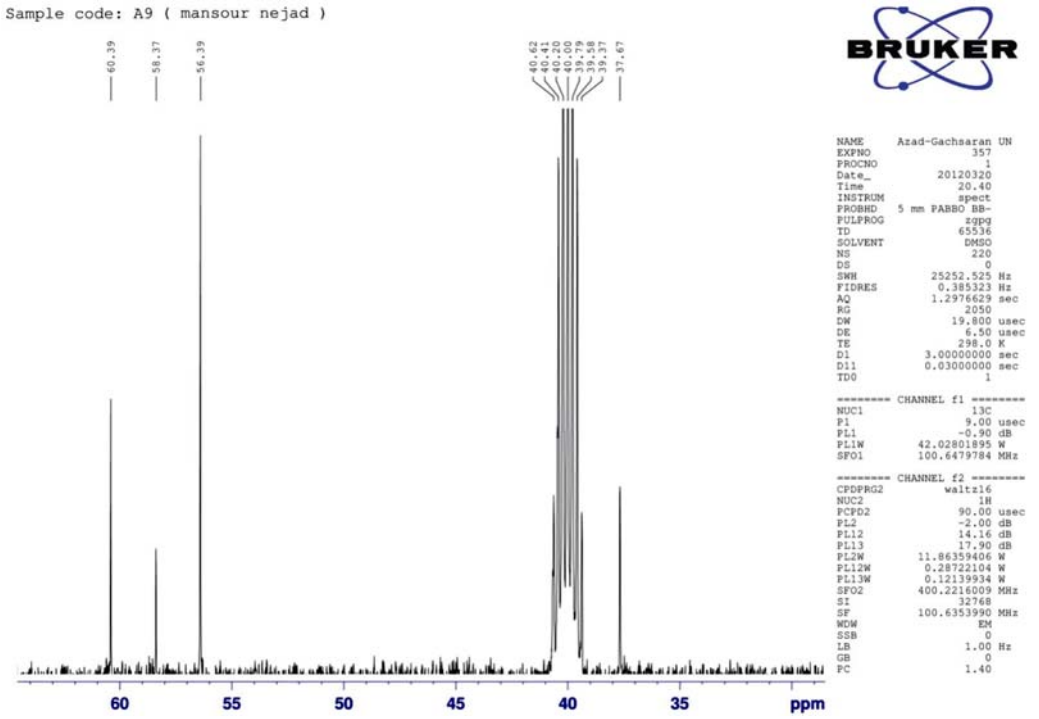


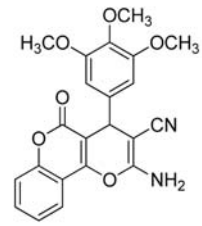

```
NAME          Azad-Gachsaran UN
EXPNO                    358
PROCNO                     1
Date_               20120320
Time                   20.37
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       24
DS                        0
SWH                8012.820 Hz
FIDRES                0.122266 Hz
AQ                 4.0894966 sec
RG                     2050
DW                   62.400 usec
DE                    6.50 usec
TE                   297.9 K
D1                6.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     14.00 usec
PL1                    -2.00 dB
PL1W              11.86359406 W
SFO1              400.2236020 MHz
SI                       32768
SF                400.2200000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

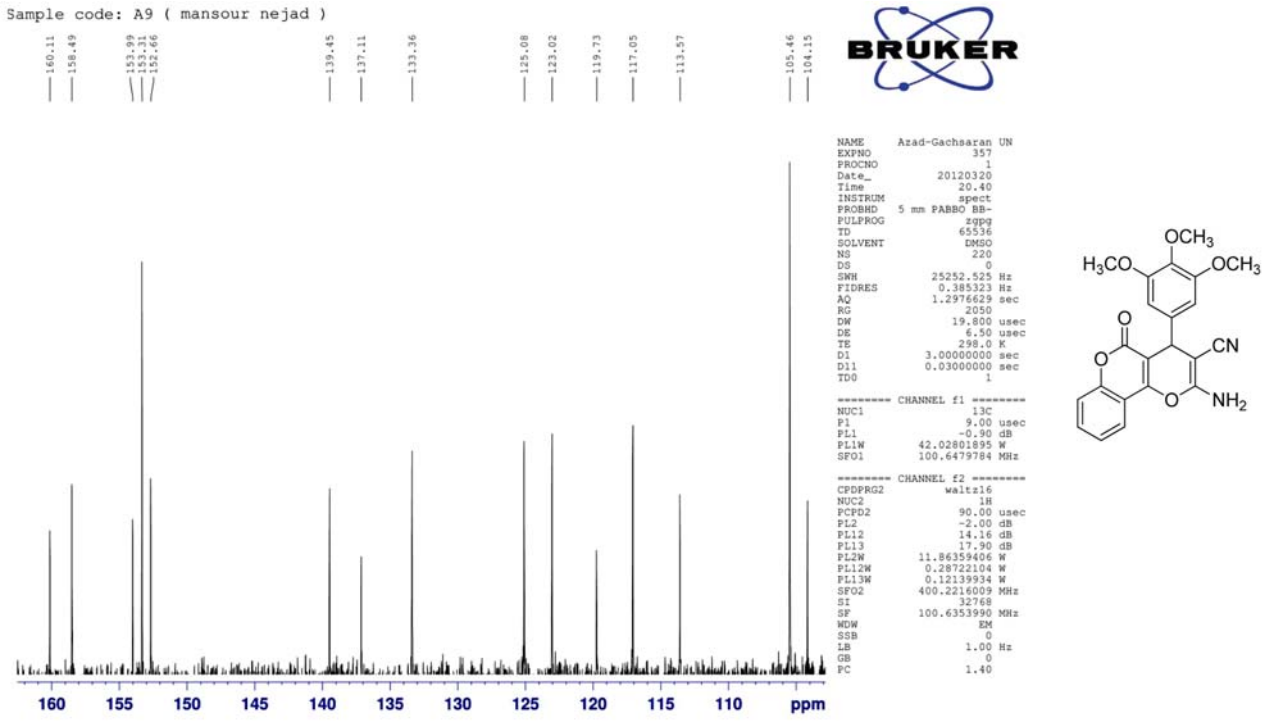


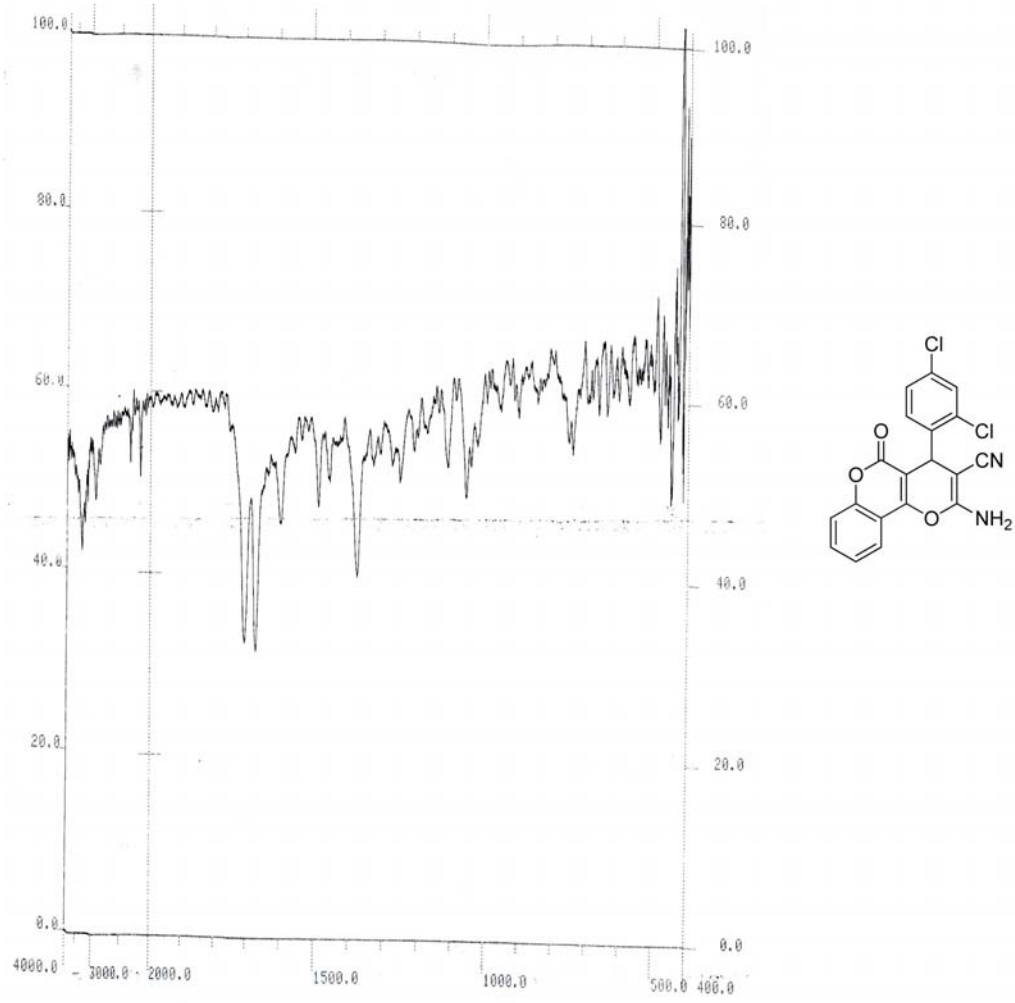

Sample code: A9 (mansour nejad)
BRUKER
NAME Azad-Gachsaran UN
EXPNO 357
PROCNO 1
Date_ 20120320
Time 20.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 220
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.0 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
240 220 200 180 160 140 120 100 80 60 40 20 ppm

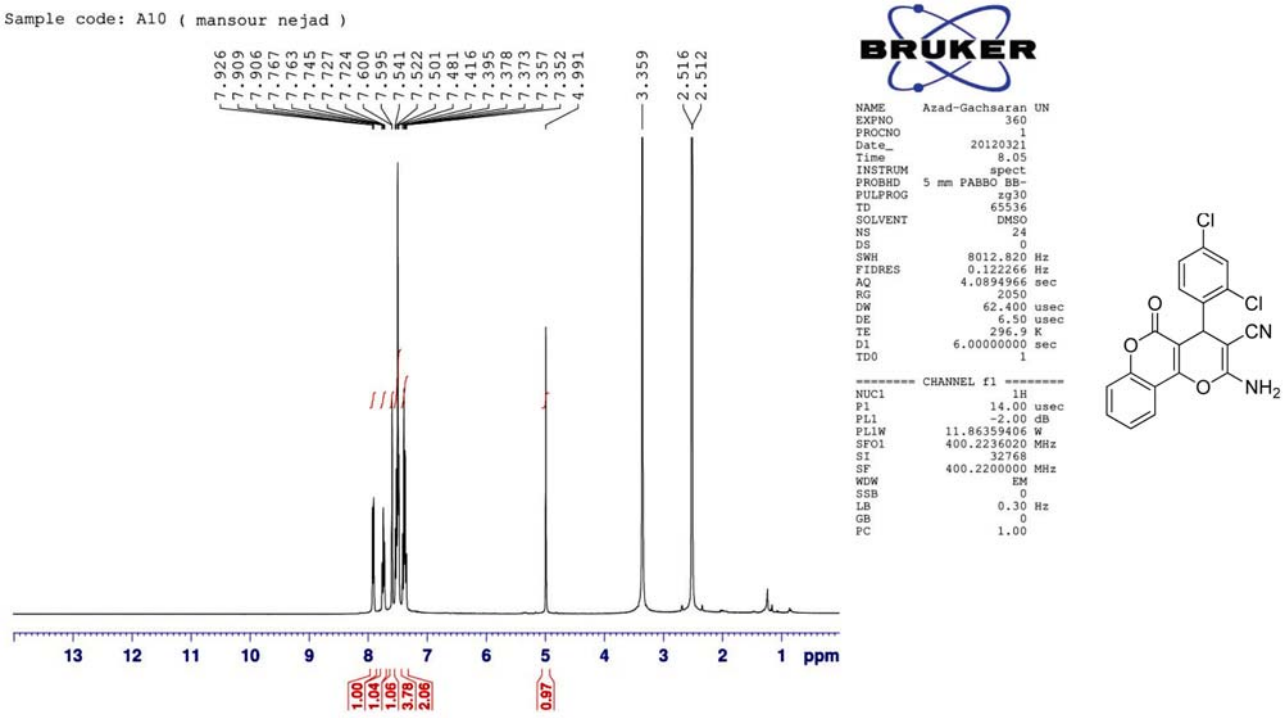

OCH3
H3CO
OCH3
O
CN
O
O
NH2

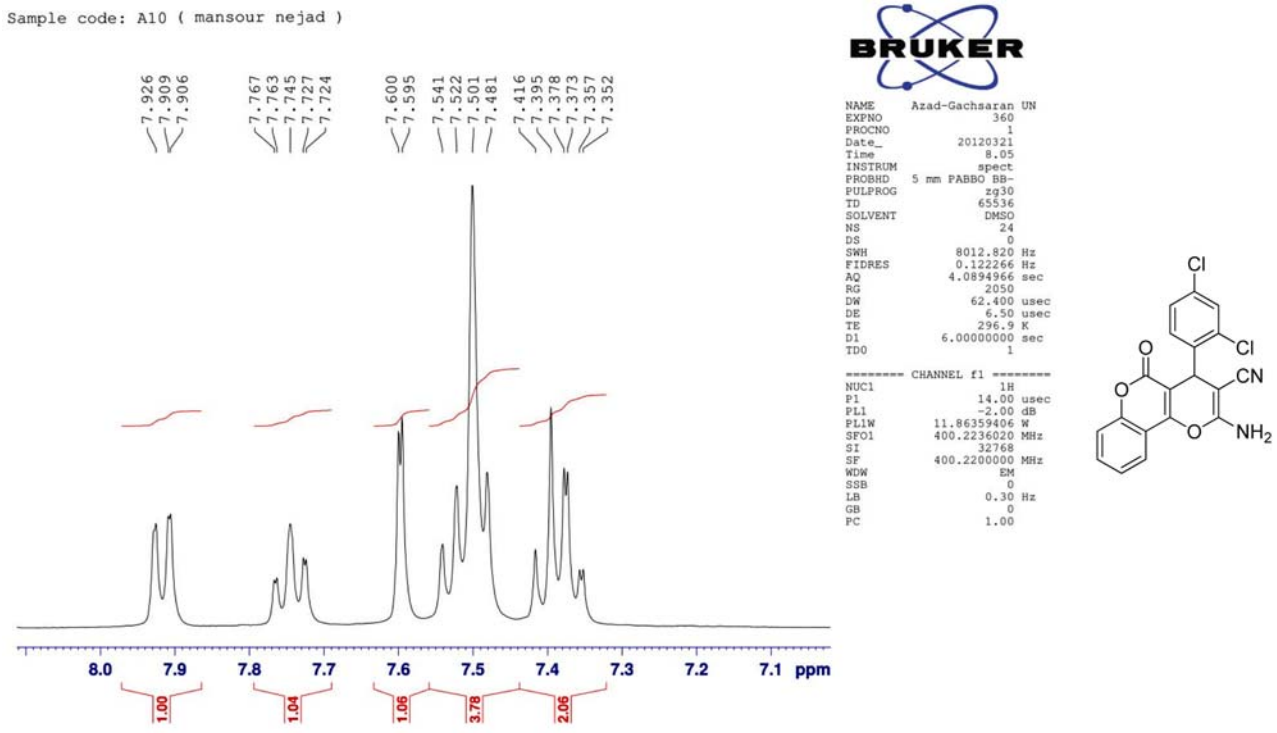

Sample code: A9 (mansour nejad)
BRUKER
NAME Azad-Gachsaran UN
EXPNO 357
PROCNO 1
Date_ 20120320
Time 20.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 220
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.0 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
60 55 50 45 40 35 ppm

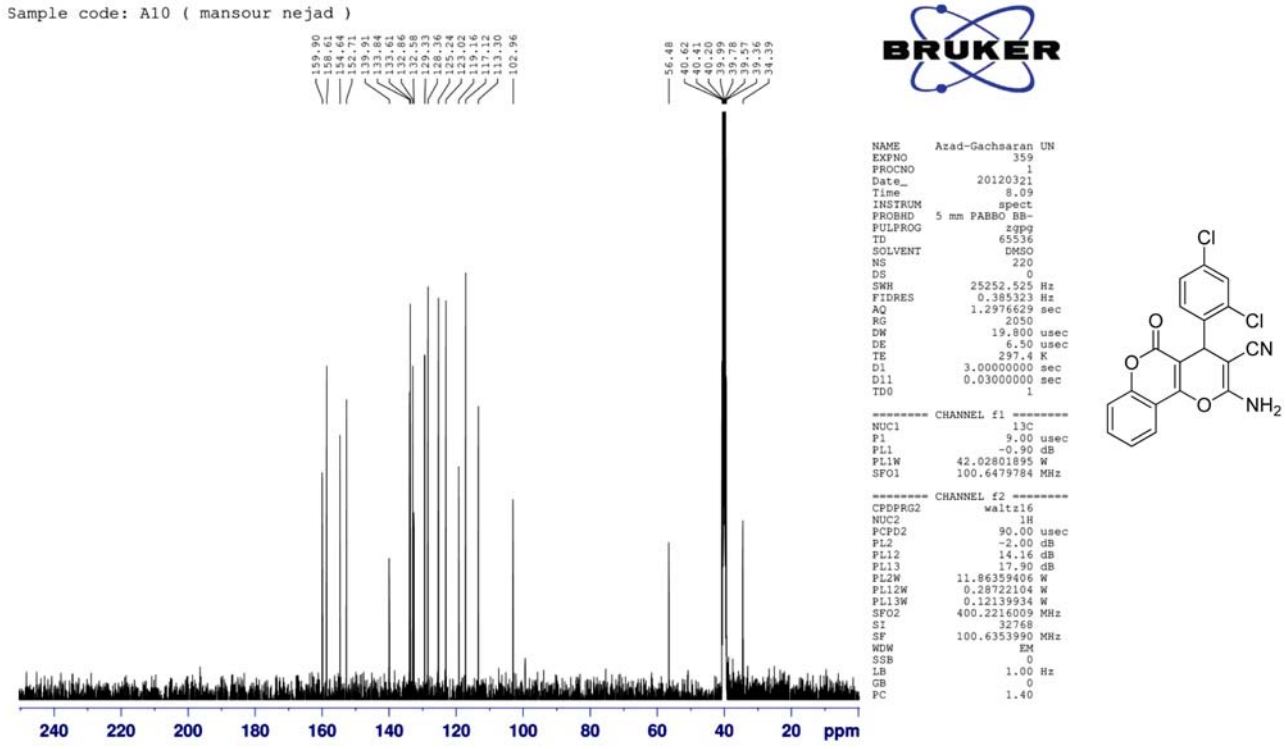

OCH3
H3CO
OCH3
O
CN
O
O
NH2

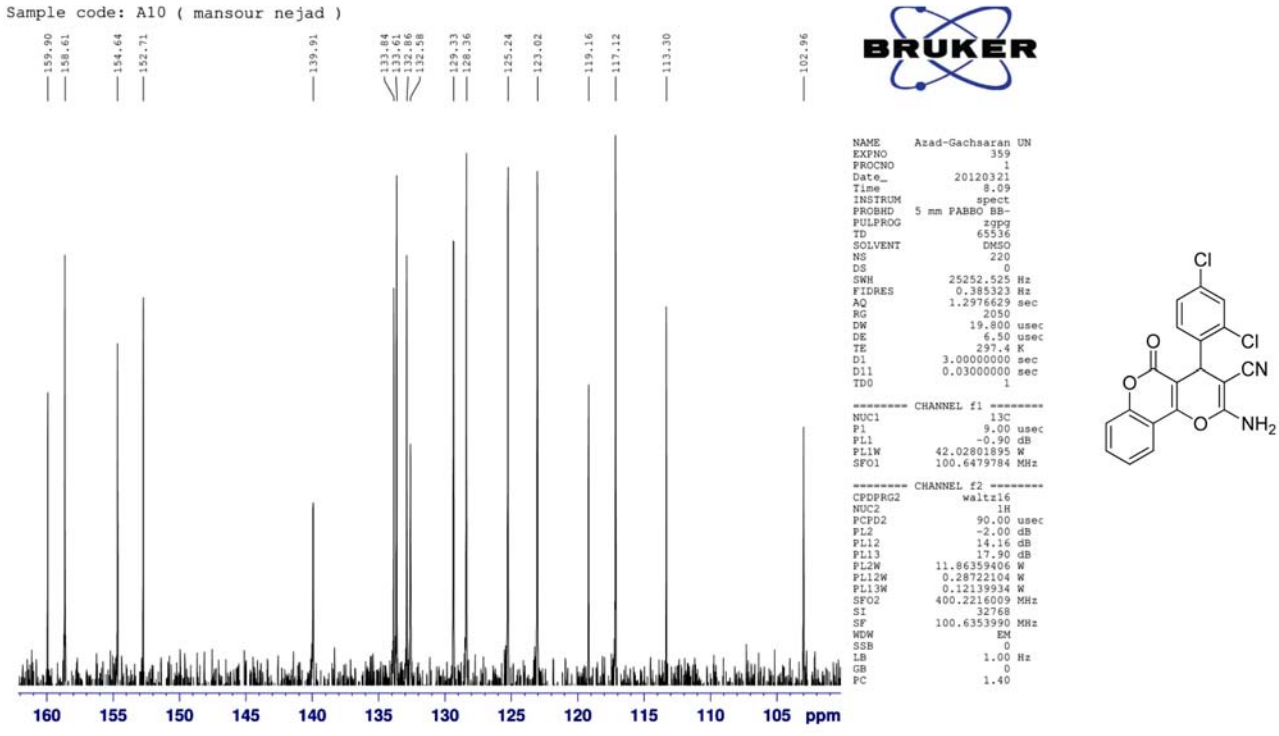

Sample code: A9 (mansour nejad)
BRUKER
160.11
158.49
153.99
153.31
152.66
139.45
137.11
133.36
125.08
123.02
119.73
117.05
113.57
105.46
104.15
NAME Azad-Gachsaran UN
EXPNO 357
PROCNO 1
Date_ 20120320
Time 20.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 220
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.0 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 155 150 145 140 135 130 125 120 115 110 ppm
OCH3
H3CO
OCH3
O
CN
O
O
NH2


100.0
80.0
60.0
40.0
20.0
0.0
4000.0 3000.0 2000.0 1500.0 1000.0 500.0 400.0
Cl
Cl
O
O
CN
O
NH2


Sample code: A10 (mansour nejad)
7.926
7.909
7.906
7.767
7.763
7.745
7.727
7.724
7.600
7.595
7.541
7.522
7.501
7.481
7.416
7.395
7.378
7.373
7.357
7.352
4.991
3.359
2.516
2.512
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.00 1.04 1.06 3.78 2.06
0.97
BRUKER
NAME Azad-Gachsaran UN
EXPNO 360
PROCNO 1
Date_ 20120321
Time 8.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 2050
DW 62.400 usec
DE 6.50 usec
TE 296.9 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cl
Cl
CN
O
O
O
NH2


Sample code: A10 (mansour nejad)
7.926
7.909
7.906
7.767
7.763
7.745
7.727
7.724
7.600
7.595
7.541
7.522
7.501
7.481
7.416
7.395
7.378
7.373
7.357
7.352
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 ppm
1.00 1.04 1.06 3.78 2.06
BRUKER
NAME Azad-Gachsaran UN
EXPNO 360
PROCNO 1
Date_ 20120321
Time 8.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 2050
DW 62.400 usec
DE 6.50 usec
TE 296.9 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cl
Cl
CN
O
O
O
NH2


Sample code: A10 (mansour nejad)
159.90
158.61
154.64
152.71
139.91
133.84
133.61
132.86
132.58
129.33
128.36
125.24
123.02
119.16
117.12
113.30
102.96
56.48
40.62
40.41
40.20
39.99
39.78
39.57
39.36
34.39
240 220 200 180 160 140 120 100 80 60 40 20 ppm
BRUKER
NAME Azad-Gachsaran UN
EXPNO 359
PROCNO 1
Date_ 20120321
Time 8.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 220
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 297.4 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cl
Cl
CN
O
O
O
NH2


Sample code: A10 (mansour nejad)
159.90
158.61
154.64
152.71
139.91
133.84
133.61
132.86
132.58
129.33
128.36
125.24
123.02
119.16
117.12
113.30
102.96
160 155 150 145 140 135 130 125 120 115 110 105 ppm
BRUKER
NAME Azad-Gachsaran UN
EXPNO 359
PROCNO 1
Date_ 20120321
Time 8.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 220
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 297.4 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -0.90 dB
PL1W 42.02801895 W
SFO1 100.6479784 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.16 dB
PL13 17.90 dB
PL2W 11.86359406 W
PL12W 0.28722104 W
PL13W 0.12139934 W
SFO2 400.2216009 MHz
SI 32768
SF 100.6353990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cl
Cl
CN
O
O
O
NH2