Supplementary Information (SI) for Organic & Biomolecular Chemistry.

Supporting information

Synthesis of Furan-Linked Iminomethyl 2-aminochromones *via* Base-Promoted Cascade Reaction of 3-Cyanochromones with *β*-Ketodinitriles

Behnaz Farajpour,[a] Fatima Khawari,[a] Nafiseh Bakhshi Davoudi,[a] Yazdanbakhsh Lotfi Nosood,[b] Behrouz Notash,[c] Salman Taheri,[d] Fatemeh Moradkhani,[e] Morteza Shiri,[a]* Ahmed Al-Harrasi,[b]* Faiq H. S. Hussain[f]*

[a]*Department of Organic Chemistry, Faculty of Chemistry, Alzahra University, Vanak, Tehran 1993893973, Iran. (e.mail: mshiri@alzahra.ac.ir)*

[b]*Natural and Medical Sciences Research Center, University of Nizwa, P. O. Box 33, Postal Code 616, Birkat Al Mauz, Nizwa, Sultanate of Oman.*

[c]*Department of Inorganic Chemistry, Shahid Beheshti University, Tehran 1983969411, Iran.*

[d]*Chemistry & Chemical Engineering Research Center of Iran, Tehran 1993893973, Iran.*

[e]*Department of Medicinal Chemistry, Faculty of Pharmacy and Pharmaceutical Sciences Research Center, Tehran University of Medical Sciences, Tehran P94V+8MF, Iran.*

[f]*Medical Analysis Department, Applied Science Faculty, Tishk International University, Erbil 44001 Kurdistan Region, Iraq.*

Table of Contents

EXPERIMENTAL SECTION

General Information

All reactions were monitored by thin-layer chromatography (TLC) on Merck silica gel 60 F254 plates. The temperatures were monitored using a mercury laboratory thermometer. Column chromatography purification was carried out on silica gel (63−200 mesh ASTM). Melting points were measured on an Electrothermal 9100 apparatus. ^{1}H NMR (500 MHz), ^{19}F{^{1}H} NMR (470 MHz), and ^{13}C{^{1}H} NMR (125 MHz) spectra were obtained using a Bruker spectrometer. NMR spectra were recorded at r.t. in DMSO-d_6. Chemical shifts are reported in parts per million (δ) downfield from an internal TMS reference. Standard abbreviations were used to indicate spin multiplicities (s = singlet, d = doublet, t = triplet, br = broad, m = multiplet, dd = doublet of doublets). Coupling constants (J values) are reported in hertz (Hz). High-resolution mass spectra (HRMS) were obtained on an Agilent HRMS-ESI/QTOF instrument. FTIR-ATR spectra were recorded on a Bruker Equinox 55 spectrometer and frequencies were expressed in cm^{-1}. Purchased from Merck or Aldrich, all chemicals and solvents were used without further purification. 3-cyanochromone[1] and β-ketodinitrile[2] derivatives were synthesized according to the procedures reported in the literature. Single crystals of compound **3e** were formed in the mixture of DMF, $CHCl_3$ and *n*-hexane.

General procedure for the synthesis of 4a.

To a solution of β-ketodinitrile **2a** (1 mmol, 184 mg) in MeCN (2.0 mL) was added Et_3N (1 mmol, 101 mg, 140 μL). After stirring at 80 °C for 3 h, the reaction mixture was cooled to room temperature and then concentrated under vacuum. In continue, 10 mL of ethanol (96%) was added to the residue, resulting in the formation of a pale yellow solid. The obtained solid was recrystallized from ethanol to deliver the pure product **4a** as colorless crystals.

General procedure for the synthesis of 3.

A mixture of 3-cyanochromone **1** (1.0 mmol), β-ketodinitrile **2** (1.0 mmol), and Et_3N (2.0 mmol, 202 mg, 280 μL) in MeCN (3.0 mL) was magnetically stirred at 80 °C in an oil bath. The reaction progress was monitored by TLC. After 6 h of stirring, the reaction was completed and cooled to room temperature. Subsequently, a solid yellow product was filtered, washed with ethanol (96%), then with diethyl ether, and dried in vacuo to yield **3**.

Characterization data for all compounds.

2-Amino-5-phenylfuran-3-carbonitrile (4a).

Following the general procedure, compound **4a** was isolated as a pale yellow solid, (147 mg, 80% yield), mp 182-184 °C (dec.). IR (ATR): 3404, 3319, 2204, 1637, 1607, 1563, 1456, 1430, 1169, 1065, 749, 698 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.60 (brs, 2H, NH_2), 7.47 (dd, 2H, *J* = 8.4, 1.2 Hz), 7.34 (t, 2H, *J* = 7.5 Hz), 7.18 (t, 1H, *J* = 8.5 Hz), 6.96 (s, 1H). $^{13}C\{^{1}H\}$ NMR (125 MHz, DMSO-d_6) δ 163.9, 141.7, 129.3, 128.7, 126.4, 122.0, 115.9, 106.6, 66.2.

(*E*)-2-(((2-Amino-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-phenylfuran-3-carbonitrile (3a).

Following the general procedure, compound **3a** was isolated as a yellow solid, (255 mg, 72% yield), mp 275-277 °C. IR (ATR): 3261, 3127, 2227, 1650, 1616, 1564, 1499, 1457, 1317, 1184, 755, 691 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.14 (brs, 1H, NH), 10.03 (brs, 1H, NH), 9.23 (s, 1H), 8.02 (dd, 1H, *J* = 8.2, 1.8 Hz), 7.73-7.69 (m, 3H), 7.48-7.41 (m, 4H), 7.38 (s, 1H), 7.35 (t, 1H, *J* = 7.2). $^{13}C\{^{1}H\}$ NMR (125 MHz, DMSO-d_6) δ 174.6, 165.0, 161.1, 156.0, 153.3, 149.4, 134.5, 129.4, 128.9, 125.8, 125.8, 124.1, 121.8, 117.3, 114.5, 108.0, 96.5, 87.2.

(*E*)-2-(((2-Amino-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-(*p*-tolyl)furan-3-carbonitrile (3b).

Following the general procedure, compound **3b** was isolated as a yellow solid, (273 mg, 74% yield), mp 278-280 °C. IR (ATR): 3220, 2223, 1657, 1613, 1563, 1493, 1453, 1319, 1237, 755, 676 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.11 (brs, 1H, NH), 9.99 (brs, 1H, NH), 9.18 (s, 1H), 8.01 (dd, 1H, *J* = 8.0, 1.7 Hz), 7.70 (t, 1H, *J* = 8.2 Hz), 7.59 (d, 2H, *J* = 8.1 Hz), 7.43-7.40 (m, 2H), 7.27 (s, 1H), 7.25 (d, 2H, *J* = 8.0 Hz), 2.30 (s, 3H). $^{13}C\{^{1}H\}$ NMR (125 MHz, DMSO-d_6) δ 174.1, 164.5, 160.3, 155.3, 152.8, 149.2, 138.2, 134.0, 129.5, 125.8, 125.3, 125.3, 123.6, 121.3, 116.8, 114.1, 106.7, 95.9, 86.8, 20.8.

(*E*)-2-(((2-Amino-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-(4-methoxyphenyl)furan-3-carbonitrile (3c).

Following the general procedure, compound **3c** was isolated as a yellow solid, (296 mg, 77% yield), mp 273-275 °C. IR (ATR): 3239, 2222, 1654, 1610, 1558, 1495, 1456, 1318, 1247, 758, 676 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.08 (brs, 1H, NH), 9.93 (brs, 1H, NH), 9.12 (s, 1H), 7.98 (dd, 1H, *J* = 7.9, 1.4 Hz), 7.67 (t, 1H, *J* = 8.4 Hz), 7.61 (d, 2H, *J* = 8.7 Hz), 7.40-7.37 (m, 2H), 7.13 (s, 1H), 6.98 (d, 2H, *J* = 9.1 Hz), 3.76 (s, 3H). $^{13}C\{^1H\}$ NMR (125 MHz, DMSO-d_6) δ 173.9, 164.4, 159.9, 159.4, 154.7, 152.7, 149.2, 133.8, 125.2, 125.2, 121.3, 121.1, 116.7, 114.4, 114.1, 105.5, 95.9, 86.9, 55.1.

(*E*)-2-(((2-Amino-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-(4-fluorophenyl)furan-3-carbonitrile (3d).

Following the general procedure, compound **3d** was isolated as a yellow solid, (313 mg, 84% yield), mp 280-282 °C (dec.). IR (ATR): 3267, 3131, 2226, 1648, 1612, 1561, 1497, 1454, 1217, 1159, 755, 658 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6) δ 10.12 (brs, 1H, NH), 10.01 (brs, 1H, NH), 9.21 (s, 1H), 8.01 (dd, 1H, J = 8.0, 1.7 Hz), 7.77-7.74 (m, 2H), 7.71 (t, 1H, J = 8.7 Hz), 7.43-7.40 (m, 2H), 7.34 (s, 1H), 7.31-7.28 (m, 2H). $^{19}F\{^1H\}$ NMR (470 MHz, DMSO-d_6): δ -112.3. $^{13}C\{^1H\}$ NMR (125 MHz, DMSO-d_6) δ 174.1, 164.6, 162.0 (d, J = 245.1 Hz), 160.6, 155.6, 152.8, 148.1, 134.0, 125.9 (d, J = 8.2 Hz), 125.3, 125.3, 125.1 (d, J = 3.0 Hz), 121.3, 116.8, 116.1 (d, J = 22.0 Hz), 114.0, 107.3, 96.0, 86.7.

(*E*)-2-(((2-Amino-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-(4-chlorophenyl)furan-3-carbonitrile (3e).

Following the general procedure, compound **3e** was isolated as a yellow solid, (318 mg, 82% yield), mp 248-250 °C (dec.). IR (ATR): 3237, 3101, 2224, 1658, 1612, 1559, 1491, 1455, 1317, 1217, 732, 677 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6) δ 10.15 (brs, 1H, NH), 10.07 (brs, 1H, NH), 9.26 (s, 1H), 8.04 (d, 1H, J = 7.7 Hz), 7.76-7.73 (m, 3H), 7.54 (d, 2H, J = 8.5 Hz), 7.47-7.43 (m, 3H). $^{13}C\{^1H\}$ NMR (125 MHz, DMSO-d_6) δ 174.1, 164.6, 160.8, 156.0, 152.8, 147.8, 134.1, 132.8, 129.0, 127.3, 125.4, 125.3, 121.2, 116.8, 113.9, 108.3, 96.0, 86.7.

(*E*)-2-(((2-Amino-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-(4-bromophenyl)furan-3-carbonitrile (3f).

Following the general procedure, compound **3f** was isolated as a yellow solid, (346 mg, 80% yield), mp 280-282 °C (dec.). IR (ATR): 3240, 2224, 1657, 1611, 1558, 1492, 1455, 1381, 1316, 1071, 756, 797, 676 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6) δ 10.16 (brs, 1H, NH), 10.06 (brs, 1H, NH), 9.27 (s, 1H), 8.04 (dd, 1H, J = 7.6, 1.0 Hz), 7.74 (t, 1H, J = 7.6 Hz), 7.70-7.66 (m, 4H), 7.47 (s, 1H), 7.47-7.44 (m, 2H). $^{13}C\{^1H\}$ NMR

(125 MHz, DMSO-d_6) δ 174.1, 164.6, 160.9, 156.0, 152.8, 147.9, 134.1, 132.0, 127.7, 125.5, 125.4, 125.3, 121.5, 121.3, 116.8, 113.9, 108.4, 96.1, 86.7.

(*E*)-2-(((2-Amino-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-(4-iodophenyl)furan-3-carbonitrile (3g).

Following the general procedure, compound **3g** was isolated as a yellow solid, (437 mg, 91% yield), mp 278-280 °C (dec.). IR (ATR): 3253, 2223, 1652, 1608, 1554, 1489, 1451, 1315, 1215, 753, 675 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.12 (brs, 1H, NH), 10.02 (brs, 1H, NH), 9.22 (s, 1H), 8.02 (d, 1H, *J* = 7.8 Hz), 7.81 (d, 2H, *J* = 8.1 Hz), 7.72 (t, 1H, *J* = 7.8 Hz), 7.50 (d, 2H, *J* = 8.1 Hz), 7.44-7.41 (m, 3H). ^{13}C{^{1}H} NMR (125 MHz, DMSO-d_6) δ 174.1, 164.6, 160.8, 155.8, 152.8, 148.0, 137.7, 134.1, 127.9, 125.4, 125.4, 125.3, 121.2, 116.8, 113.9, 108.4, 96.1, 94.6, 86.8.

(*E*)-2-(((2-Amino-6-methyl-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-phenylfuran-3-carbonitrile (3h).

Following the general procedure, compound **3h** was isolated as a yellow solid, (250 mg, 68% yield), mp 279-281 °C (dec.). IR (ATR): 3198, 3064, 2222, 1663, 1614, 1564, 1476, 1305, 1234, 749, 696 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.10 (brs, 1H, NH), 9.95 (brs, 1H, NH), 9.18 (s, 1H), 7.76-7.70 (m, 3H), 7.49-7.29 (m, 6H), 2.35 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz, DMSO-d_6) δ 174.1, 164.5, 160.6, 155.6, 151.0, 148.9, 134.8, 134.7, 129.0, 128.4, 124.9, 123.6, 120.9, 116.6, 114.1, 107.6, 96.0, 86.7, 20.3.

(*E*)-2-(((2-Amino-6-methyl-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-(p-tolyl)furan-3-carbonitrile (3i).

Following the general procedure, compound **3i** was isolated as a yellow solid, (268 mg, 70% yield), mp 268-270 °C (dec.). IR (ATR): 3232, 2225, 1652, 1612, 1560, 1466, 1302, 1231, 805, 694 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.11 (brs, 1H, NH), 9.95 (brs, 1H, NH), 9.19 (s, 1H), 7.79 (d, 1H, *J* = 1.4 Hz), 7.61 (d, 2H, *J* = 8.1 Hz), 7.51 (dd, 1H, *J* = 8.5, 2.3 Hz), 7.32 (d, 1H, *J* = 8.4 Hz), 7.29 (s, 1H), 7.27 (d, 2H, *J* = 7.9 Hz), 2.37 (s, 3H), 2.31 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz, DMSO-d_6) δ 174.1, 164.5, 160.3, 155.3, 151.0, 149.2, 138.1, 134.8, 134.7, 129.5, 125.8, 124.8, 123.6, 120.9, 116.6, 114.1, 106.7, 95.9, 86.7, 20.8, 20.3.

(*E*)-2-(((2-Amino-6-methyl-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-(4-methoxyphenyl)furan-3-carbonitrile (3j).

Following the general procedure, compound **3j** was isolated as a yellow solid, (299 mg, 75% yield), mp 263-266 °C (dec.). IR (ATR): 3200, 3063, 2222, 1663, 1612, 1562, 1496, 1386, 1305, 1251, 804, 698 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-*d$_6$*) *δ* 10.11 (brs, 1H, NH), 9.92 (brs, 1H, NH), 9.19 (s, 1H), 7.80 (d, 1H, *J* = 1.6 Hz), 7.66 (d, 2H, *J* = 8.7 Hz), 7.52 (dd, 1H, *J* = 8.4, 1.7 Hz), 7.33 (d, 1H, *J* = 8.4 Hz), 7.20 (s, 1H), 7.03 (d, 2H, *J* = 8.7 Hz), 3.79 (s, 3H), 2.38 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz, DMSO-*d$_6$*) *δ* 174.1, 164.5, 160.1, 159.5, 155.0, 151.0, 149.3, 134.8, 134.7, 125.3, 124.8, 121.2, 120.9, 116.6, 114.5, 114.1, 105.6, 95.9, 86.7, 55.2, 20.3.

(*E*)-2-(((2-Amino-6-methyl-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-(4-chlorophenyl)furan-3-carbonitrile (3k).

Following the general procedure, compound **3k** was isolated as a yellow solid, (314 mg, 78% yield), mp 265-268 °C (dec.). IR (ATR): 3203, 2222, 1660, 1614, 1566, 1476, 1388, 1302, 1234, 826, 797, 695, 672 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-*d$_6$*) *δ* 10.09 (brs, 1H, NH), 9.96 (brs, 1H, NH), 9.17 (s, 1H), 7.75 (d, 1H, *J* = 1.2 Hz), 7.70 (d, 2H, *J* = 8.5 Hz), 7.51-7.48 (m, 3H), 7.39 (s, 1H), 7.30 (d, 1H, *J* = 8.4 Hz), 2.36 (s,3H). ^{13}C{^{1}H} NMR (125 MHz, DMSO-*d$_6$*) *δ* 174.1, 164.5, 160.7, 155.7, 150.9, 147.8, 134.8, 134.7, 132.8, 129.0, 127.3, 125.2, 124.8, 120.8, 116.5, 113.9, 108.3, 96.0, 86.8, 20.3.

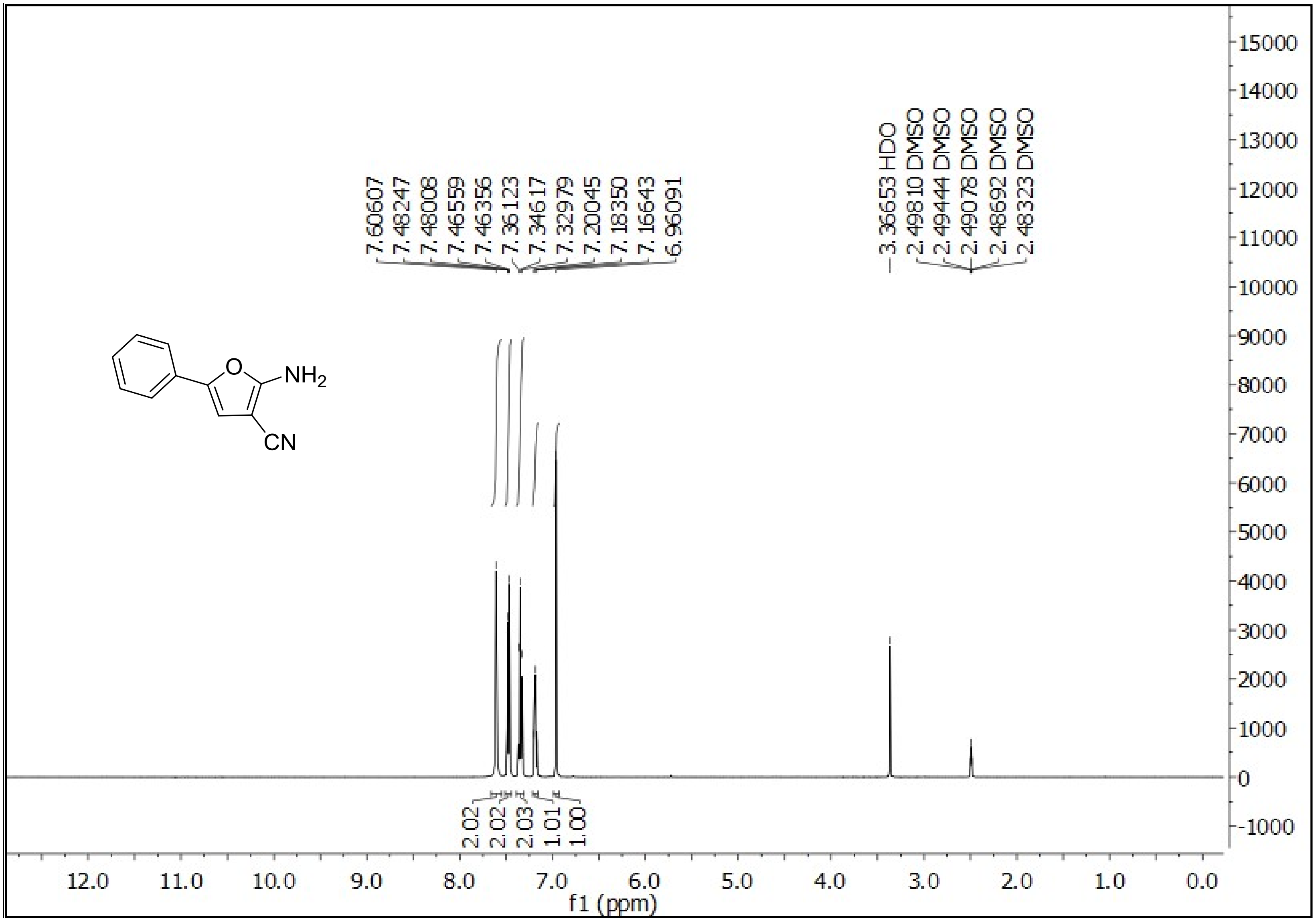
(*E*)-2-(((2-Amino-6-methyl-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-(4-bromophenyl)furan-3-carbonitrile (3l).

Following the general procedure, compound **3l** was isolated as a yellow solid, (339 mg, 76% yield), mp 250-253 °C (dec.). IR (ATR): 3204, 2222, 1660, 1614, 1566, 1475, 1301, 1234, 796, 700 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-*d$_6$*) *δ* 10.04 (brs, 1H, NH), 9.90 (brs, 1H, NH), 9.10 (s, 1H), 7.70 (s, 1H), 7.59 (s, 4H), 7.44 (d, 1H, *J* = 8.4 Hz), 7.34 (s, 1H), 7.24 (d, 1H, *J* = 8.4 Hz), 2.33 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz, DMSO-*d$_6$*) *δ* 174.1, 164.5, 160.7, 155.7, 150.9, 147.8, 134.8, 134.7, 131.9, 127.6, 125.4, 124.9, 121.4, 120.8, 116.5, 113.9, 108.4, 96.0, 86.8, 20.3.

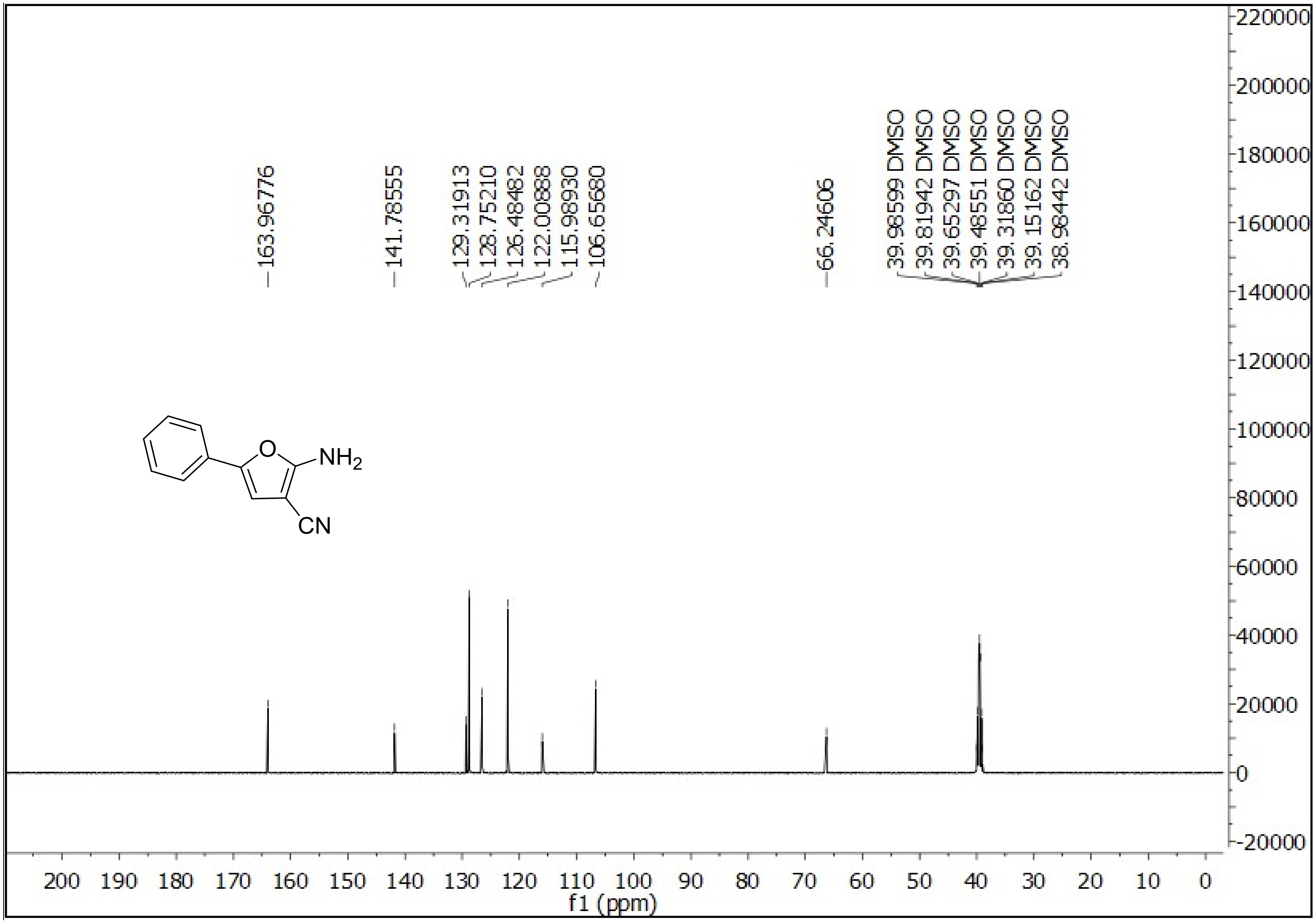
(*E*)-2-(((2-Amino-6-methyl-4-oxo-4*H*-chromen-3-yl)methylene)amino)-5-(4-iodophenyl)furan-3-carbonitrile (3m).

Following the general procedure, compound **3m** was isolated as a yellow solid, (440 mg, 89% yield), mp 280-283 °C (dec.). IR (ATR): 3448, 3216, 2226, 1660, 1608, 1558, 1471, 1381, 1308, 1243, 816, 787, 691, 671 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.11 (brs, 1H, NH), 9.99 (brs, 1H, NH), 9.21 (s, 1H), 7.82-7.79 (m, 3H), 7.53-7.50 (m, 3H), 7.43 (s, 1H), 7.33 (d, 1H, J = 8.3 Hz), 2.38 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz, DMSO-d_6) δ 174.2, 164.6, 160.9, 156.0, 151.0, 148.0, 137.8, 134.9, 134.8, 127.9, 125.4, 124.9, 120.9, 116.6, 113.9, 108.4, 96.1, 94.5, 86.7, 20.3.

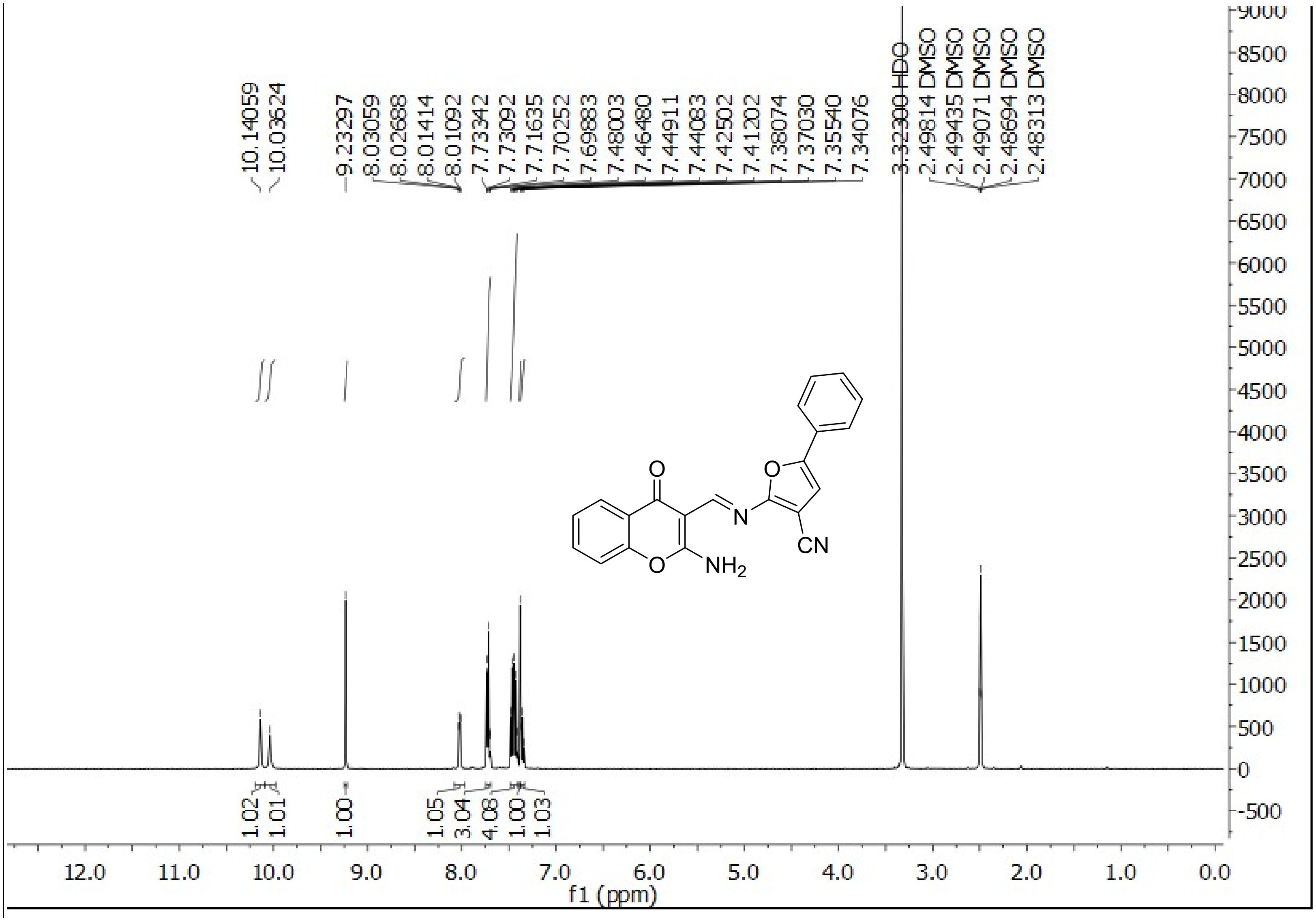
Copies of NMR spectra



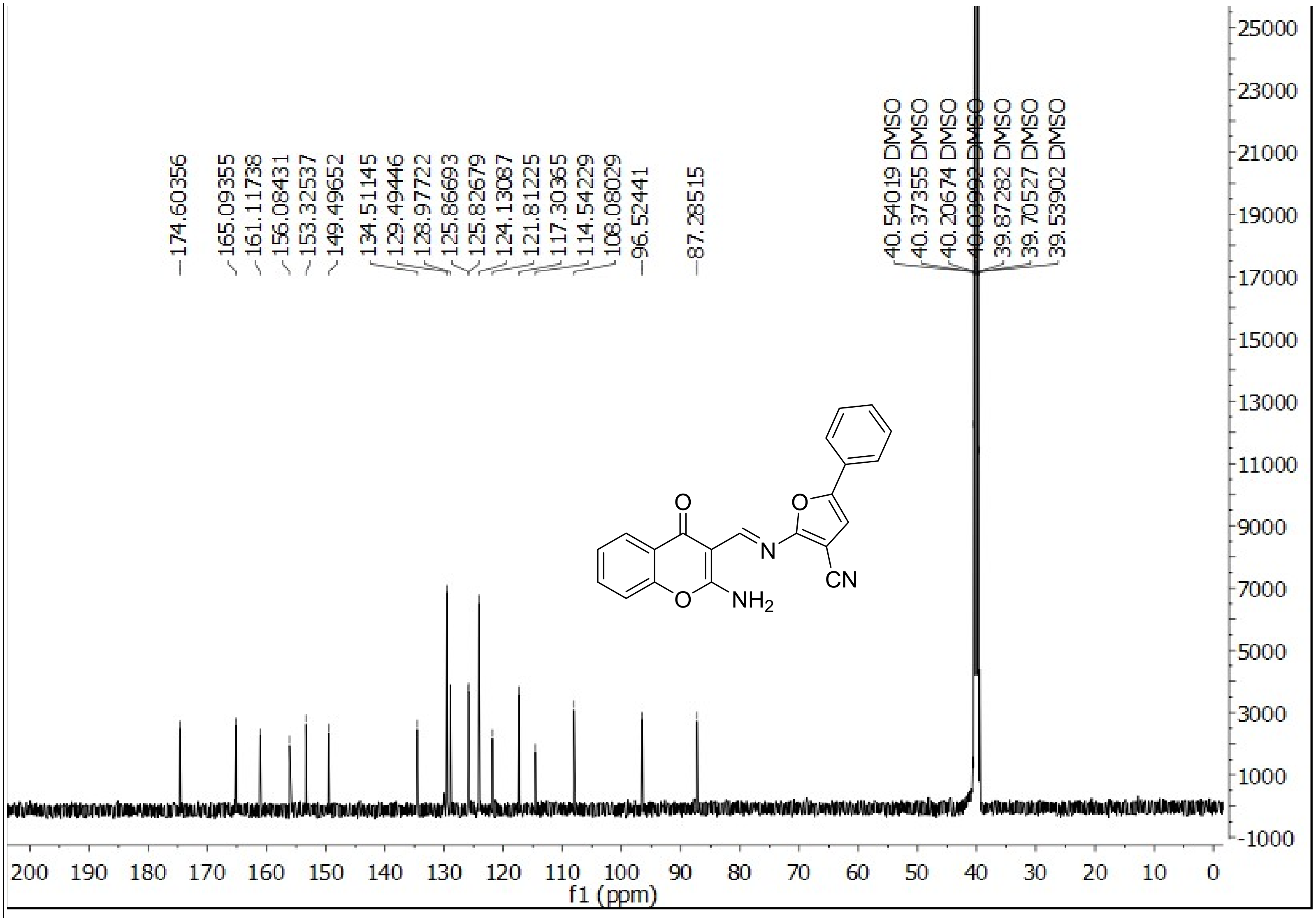
^{1}H NMR of **4a** (500 MHz, DMSO-d_6)



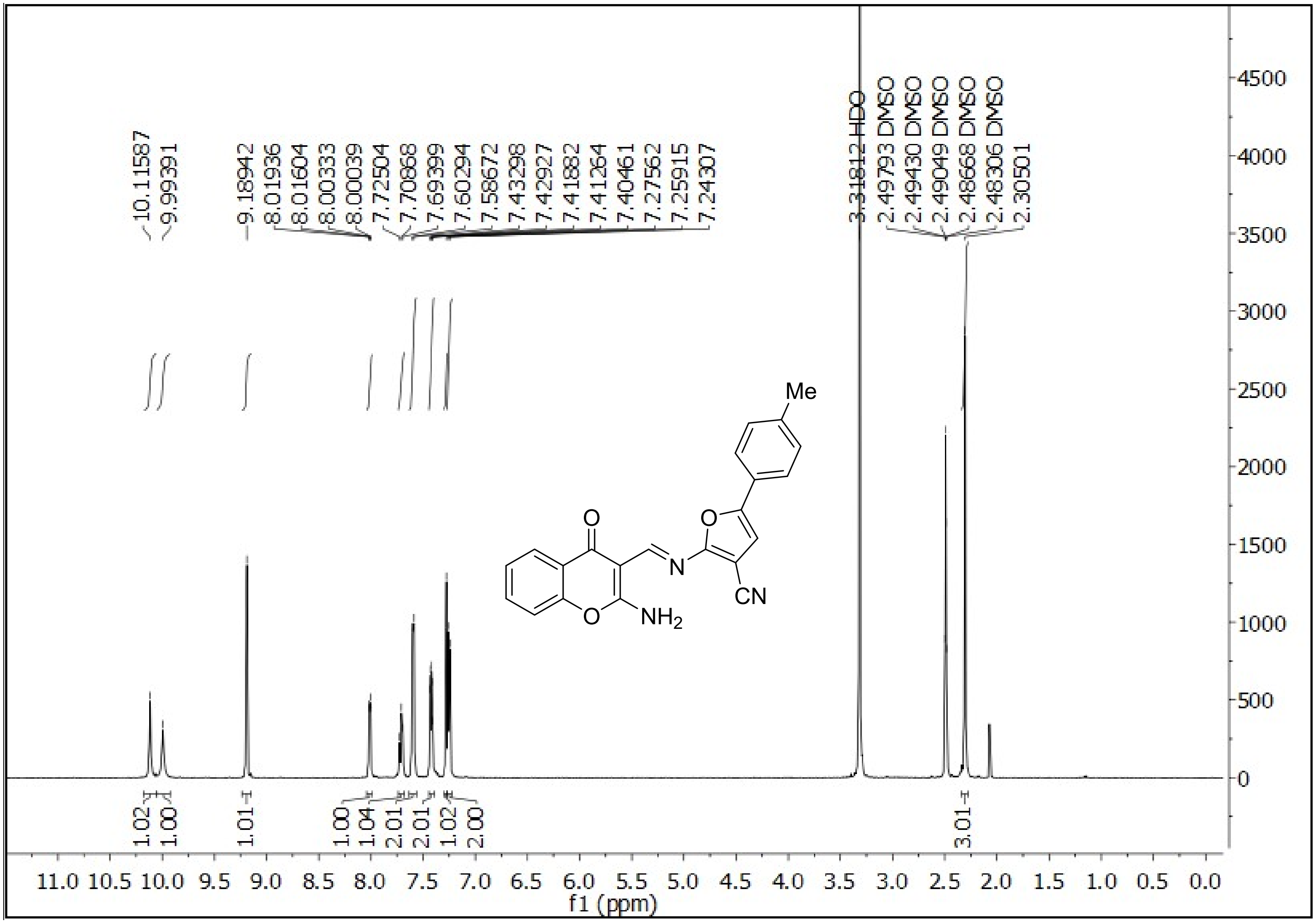
^{13}C{^{1}H} NMR of **4a** (125 MHz, DMSO-d_6)



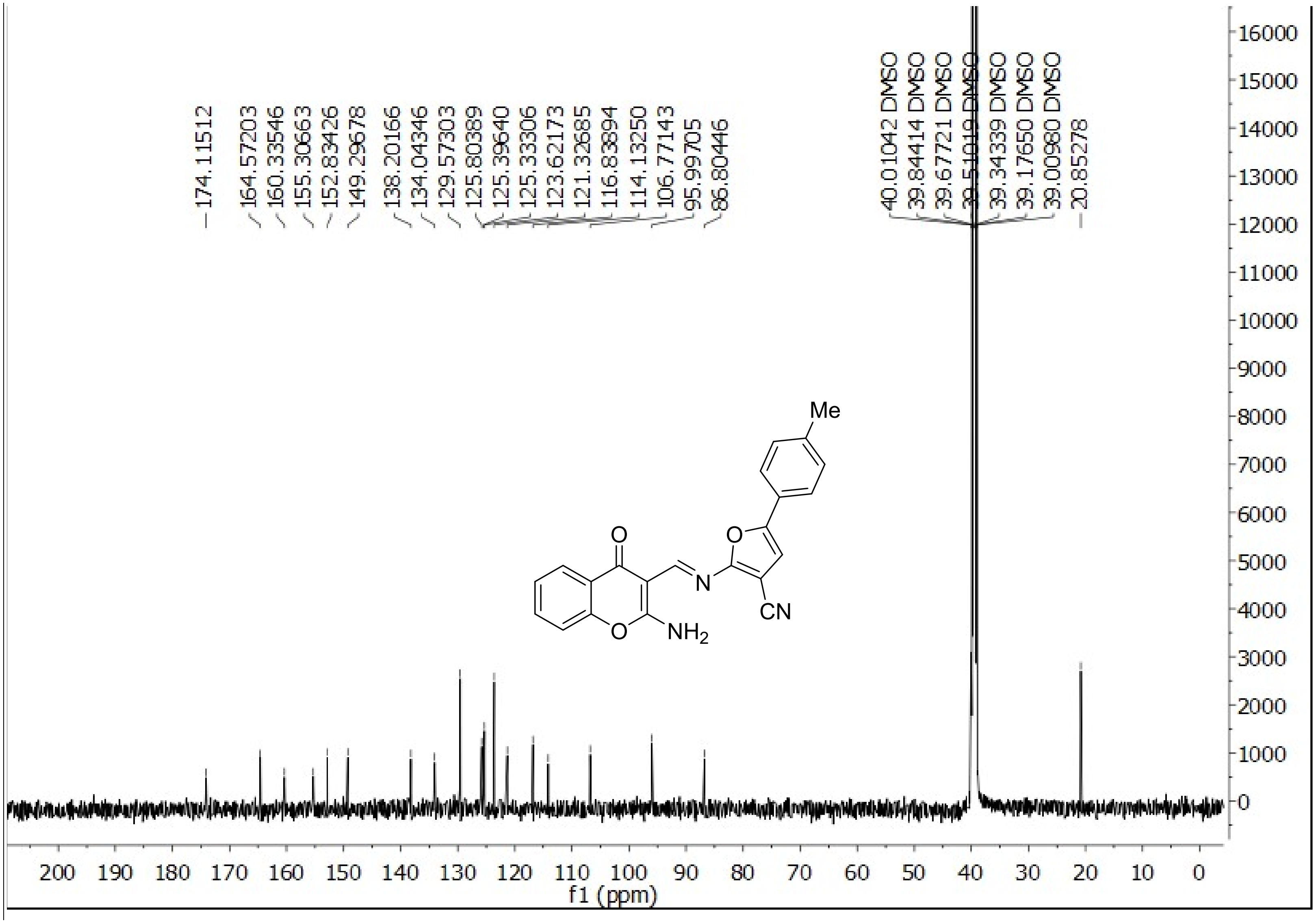
^{1}H NMR of **3a** (500 MHz, DMSO-d_6)



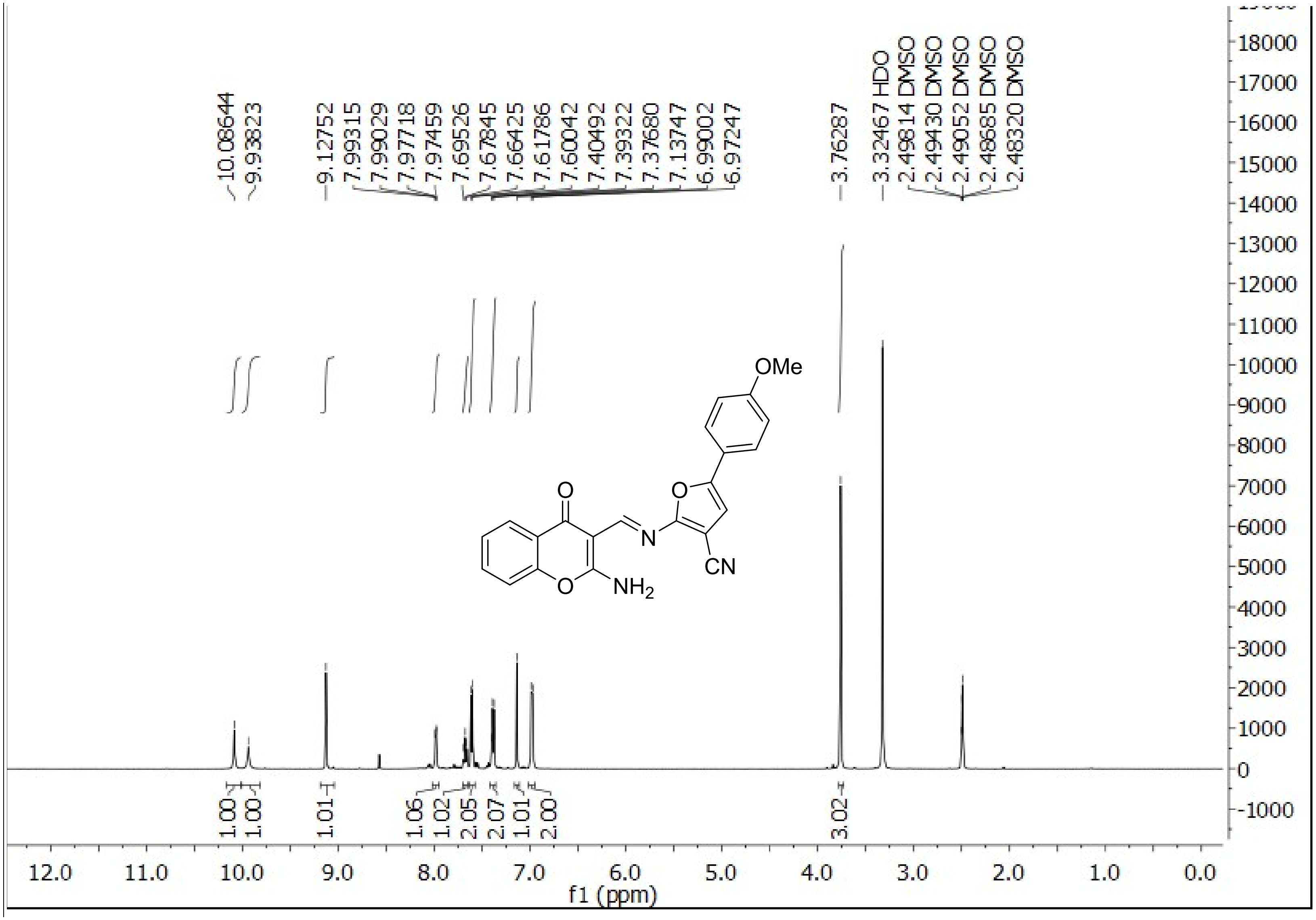
$^{13}C\{^1H\}$ NMR of **3a** (125 MHz, DMSO-d_6)



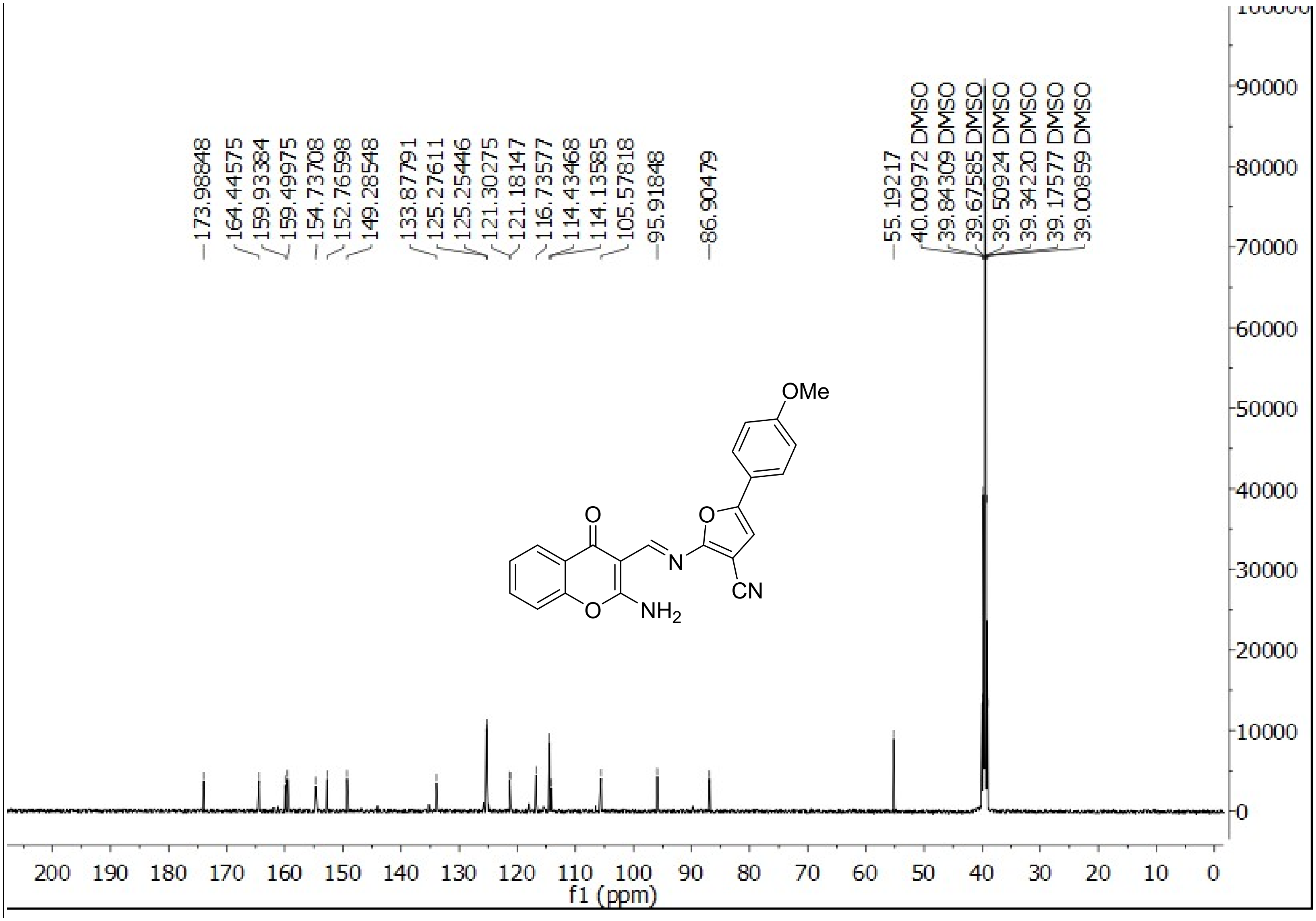
^{1}H NMR of **3b** (500 MHz, DMSO-d_6)



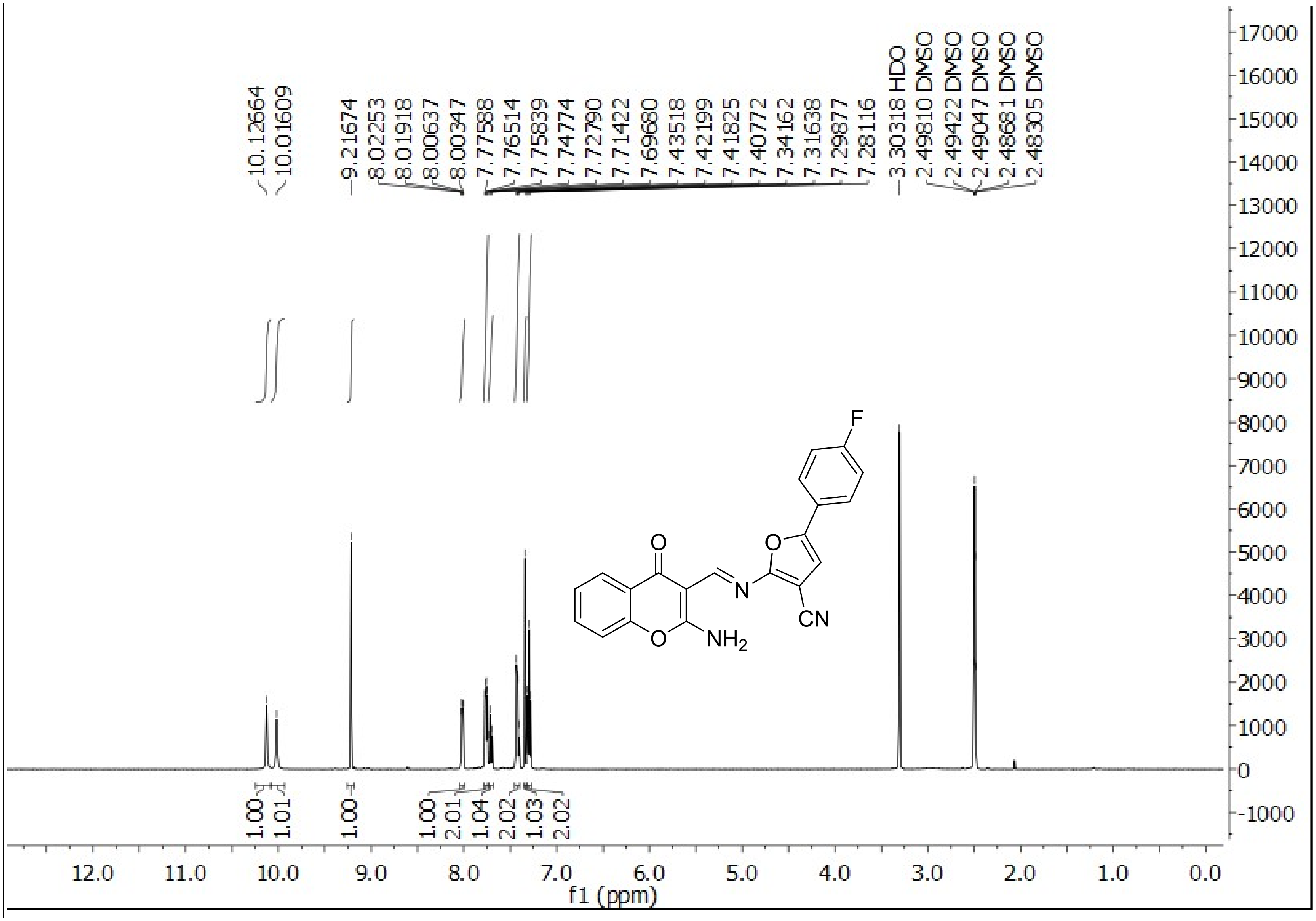
^{13}C{^{1}H} NMR of **3b** (125 MHz, DMSO-d_6)



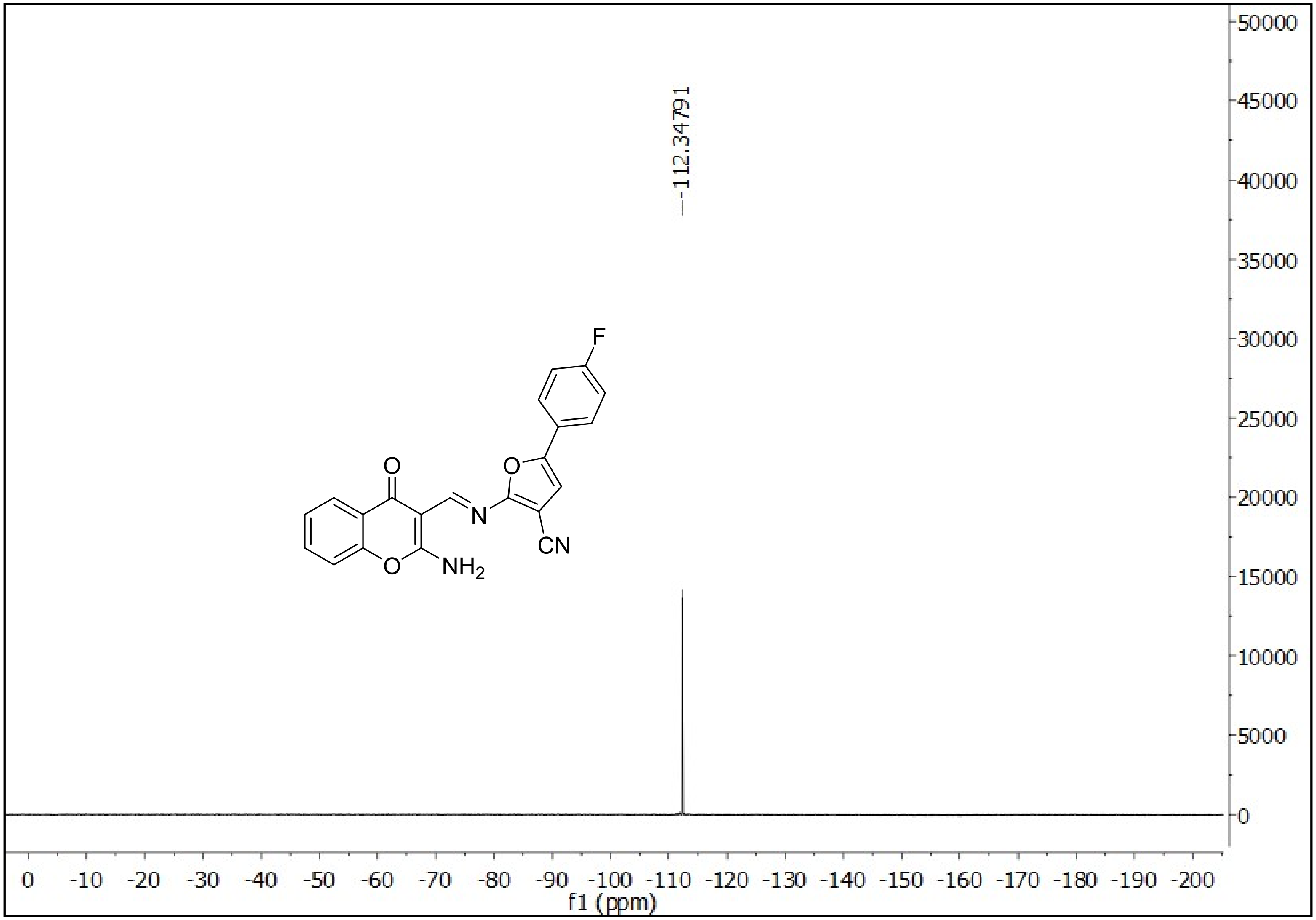
^{1}H NMR of **3c** (500 MHz, DMSO-d_6)



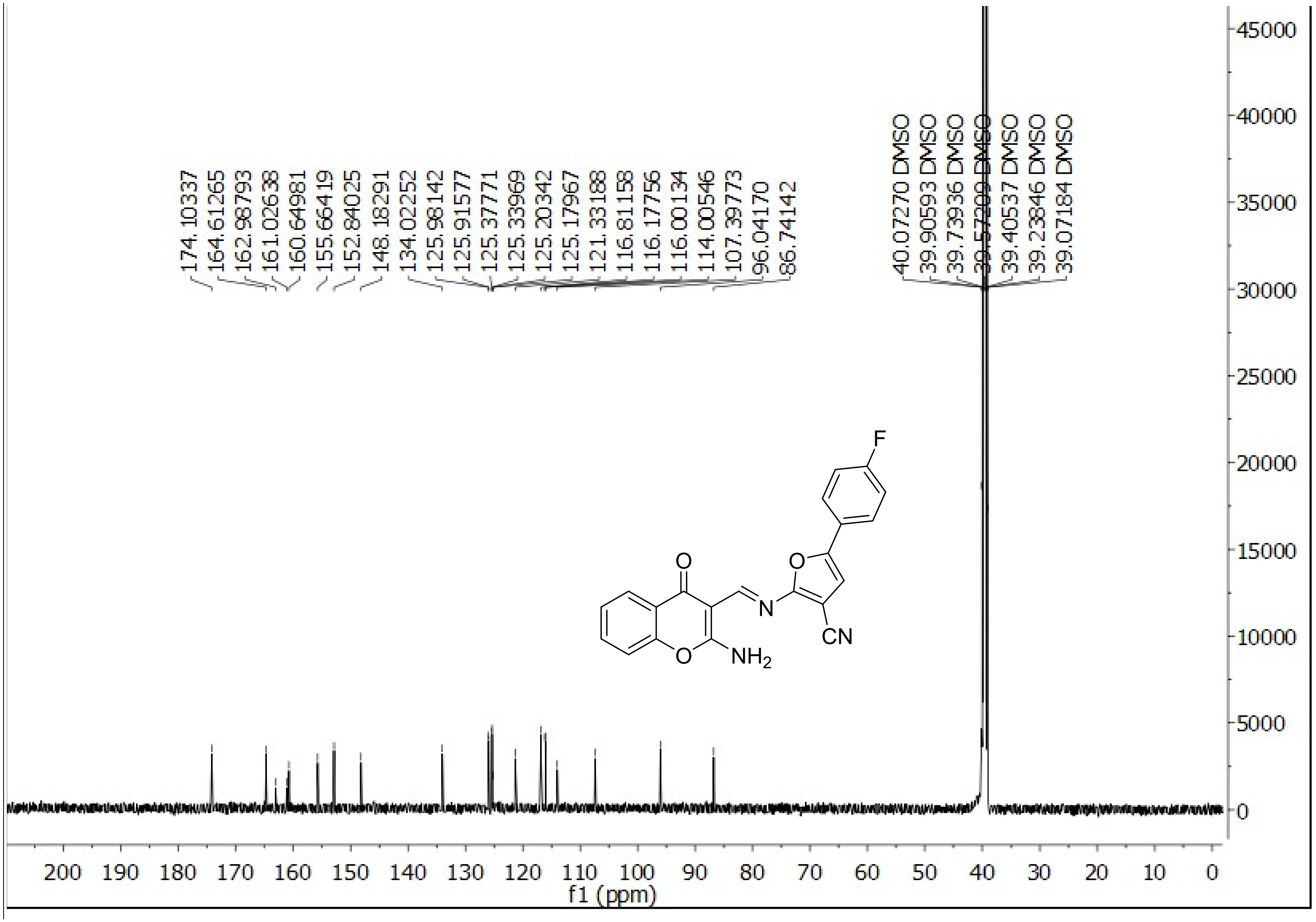
^{13}C{^{1}H} NMR of **3c** (125 MHz, DMSO-d_6)



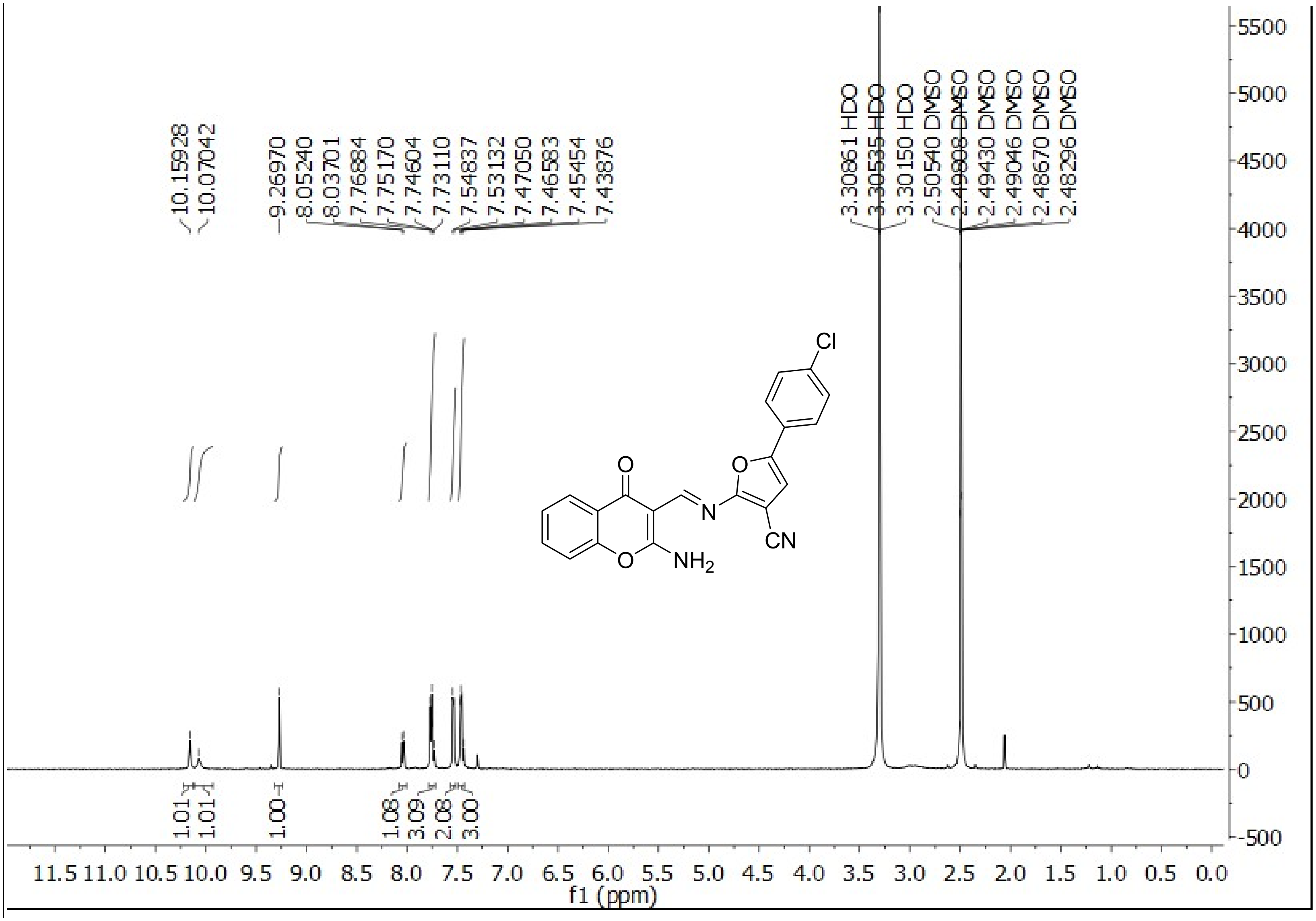
^{1}H NMR of **3d** (500 MHz, DMSO-d_6)



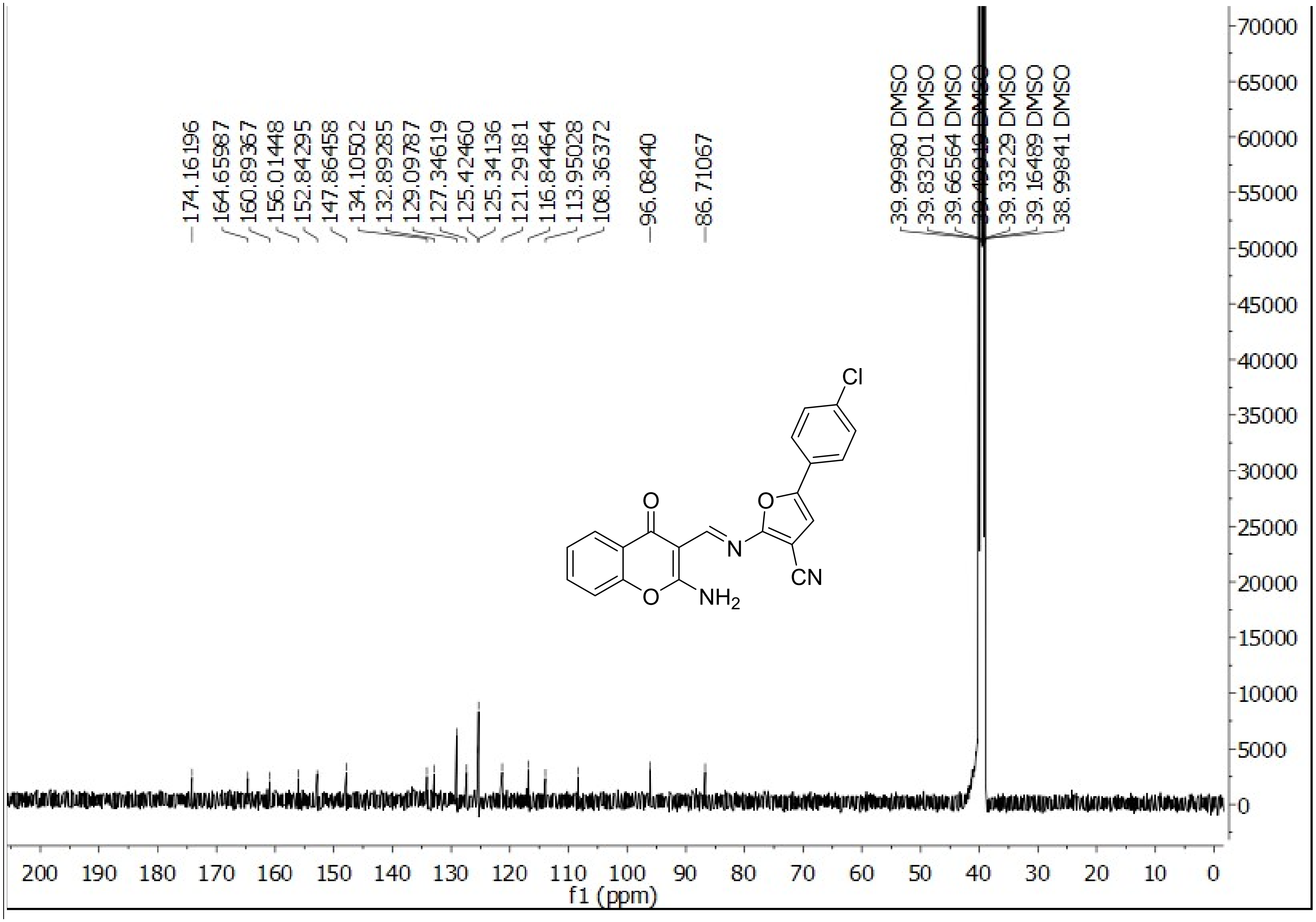
$^{19}F\{^1H\}$ NMR of **3d** (470 MHz, DMSO-d_6)



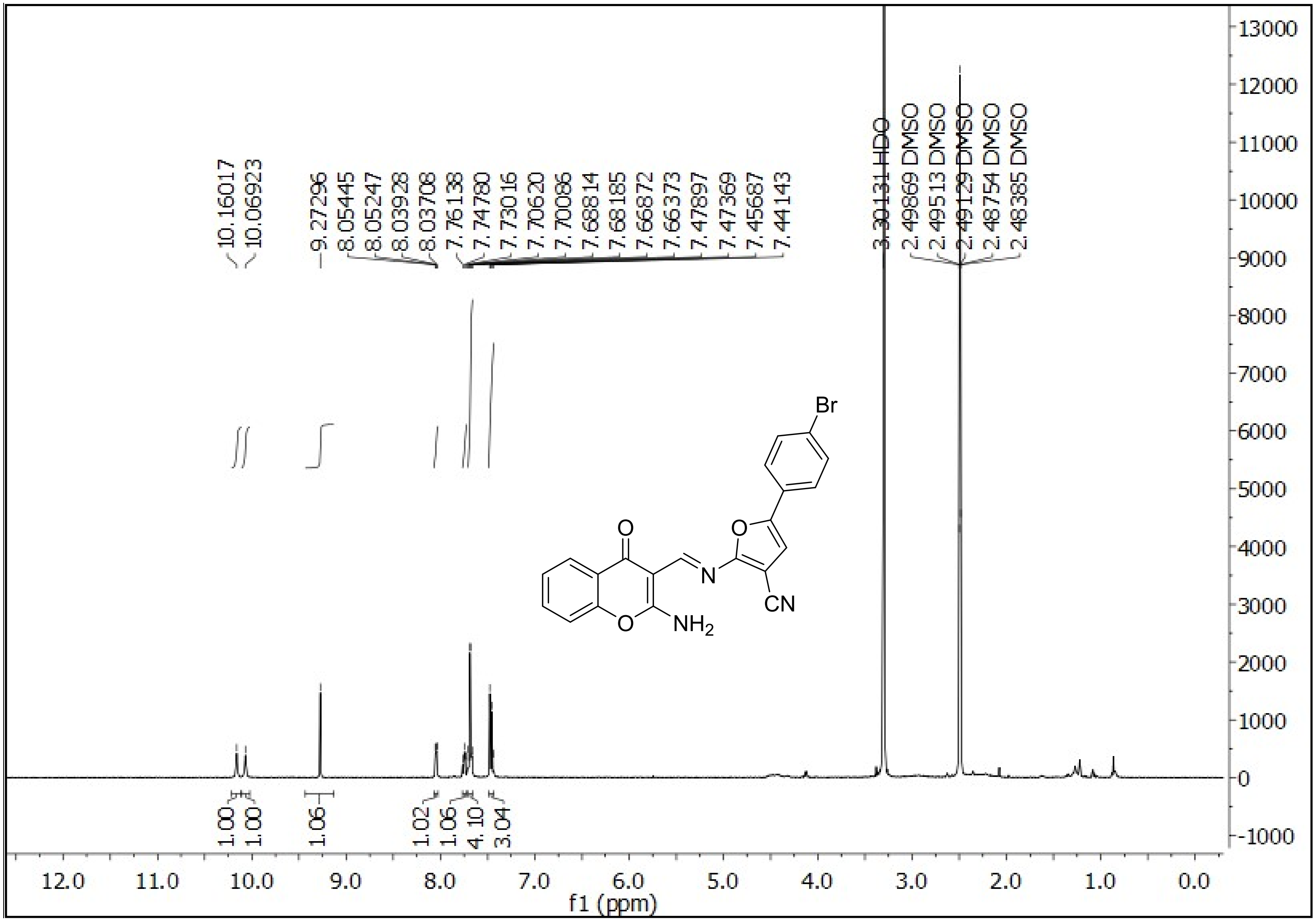
$^{13}C\{^{1}H\}$ NMR of **3d** (125 MHz, DMSO-d_6)



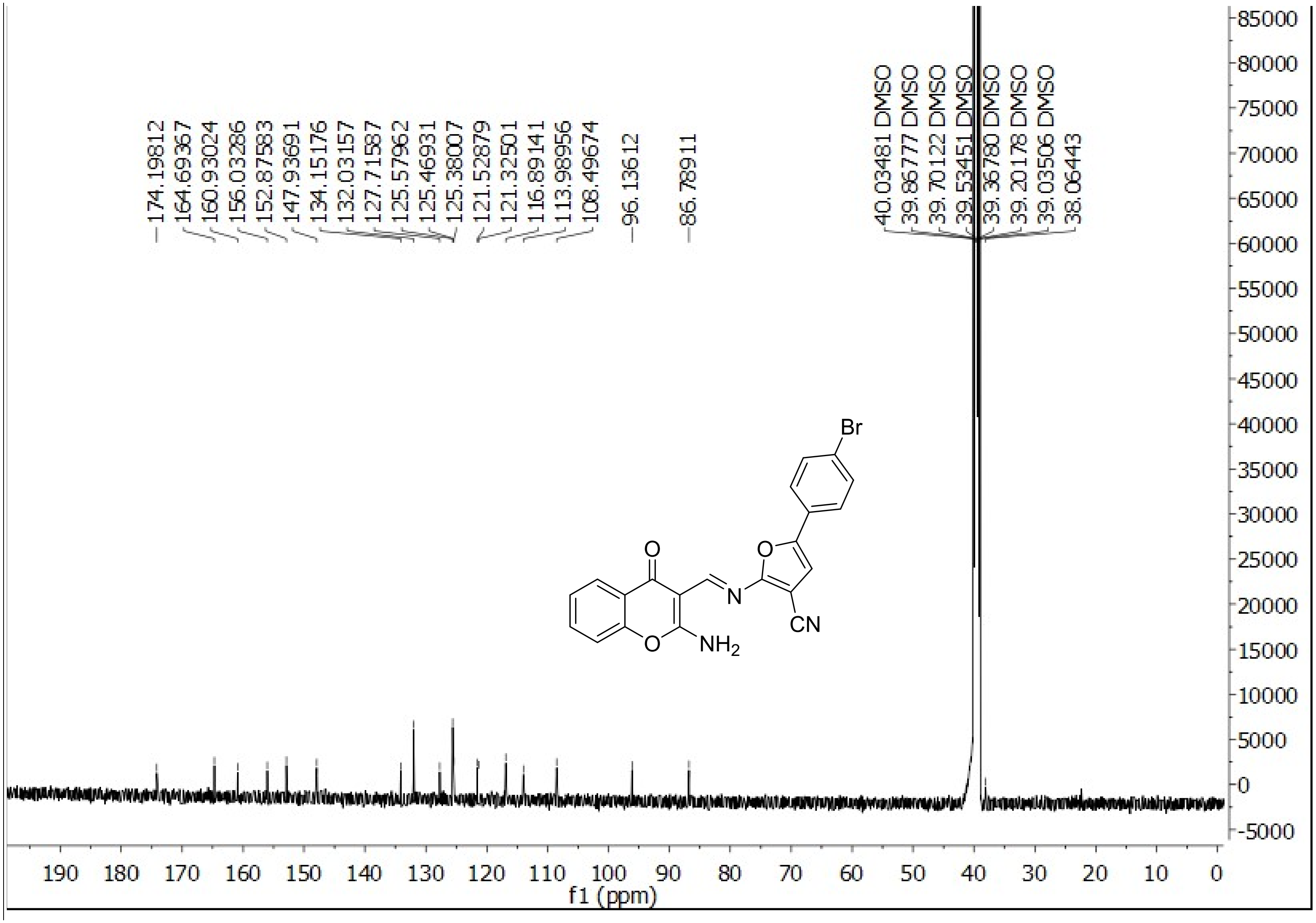
^{1}H NMR of **3e** (500 MHz, DMSO-d_6)



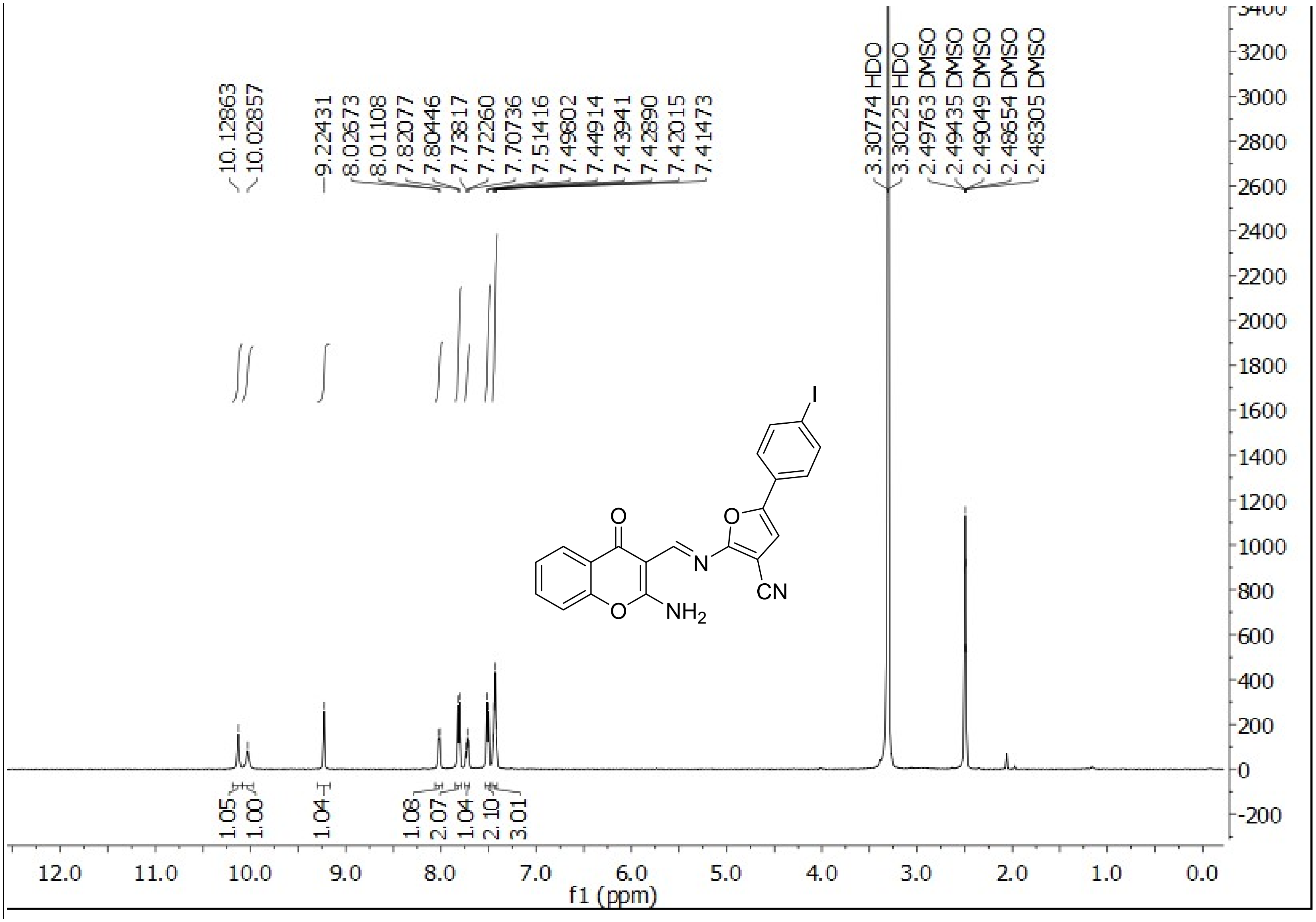
$^{13}C\{^{1}H\}$ NMR of **3e** (125 MHz, DMSO-d_6)



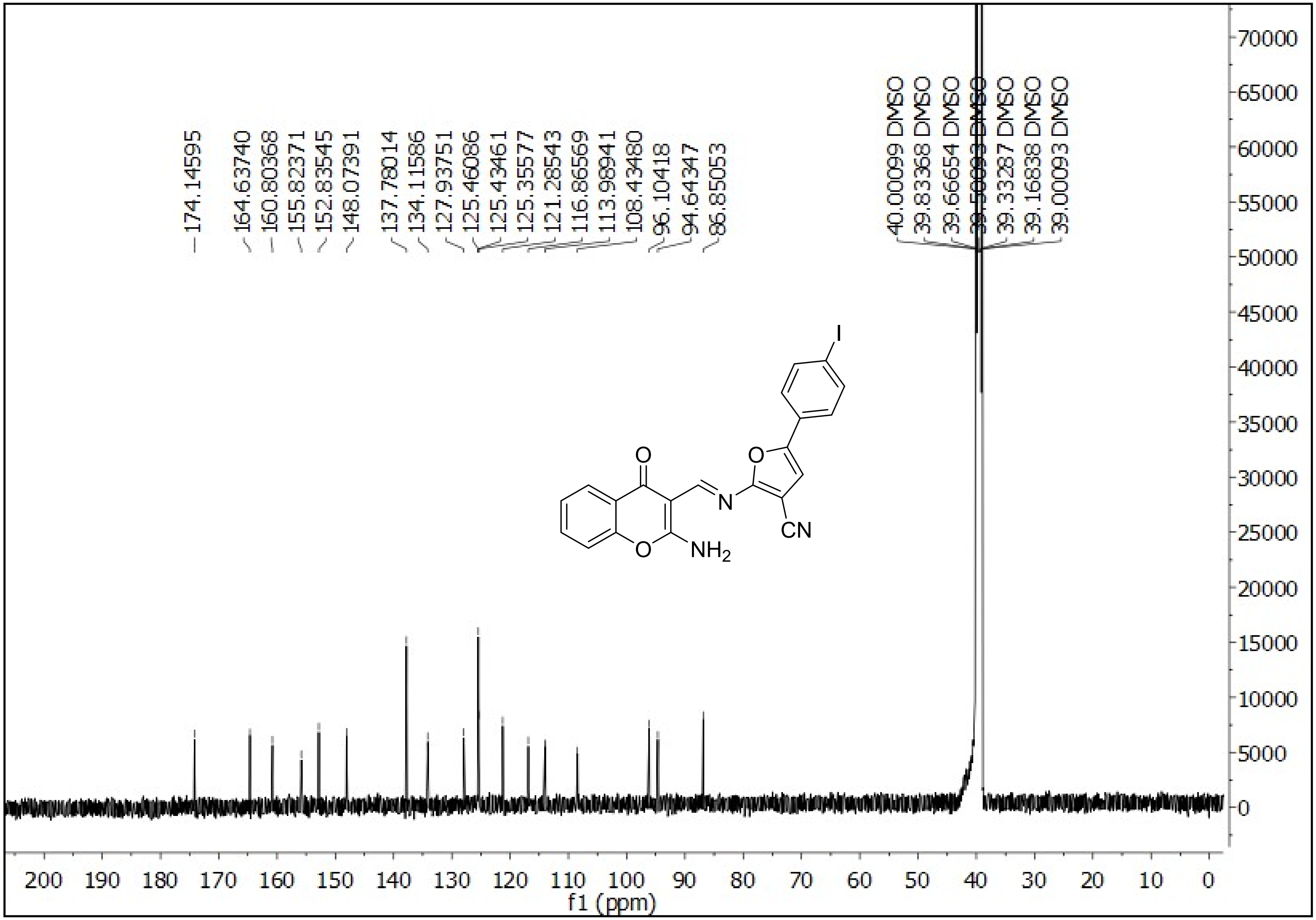
^{1}H NMR of **3f** (500 MHz, DMSO-*d_6*)



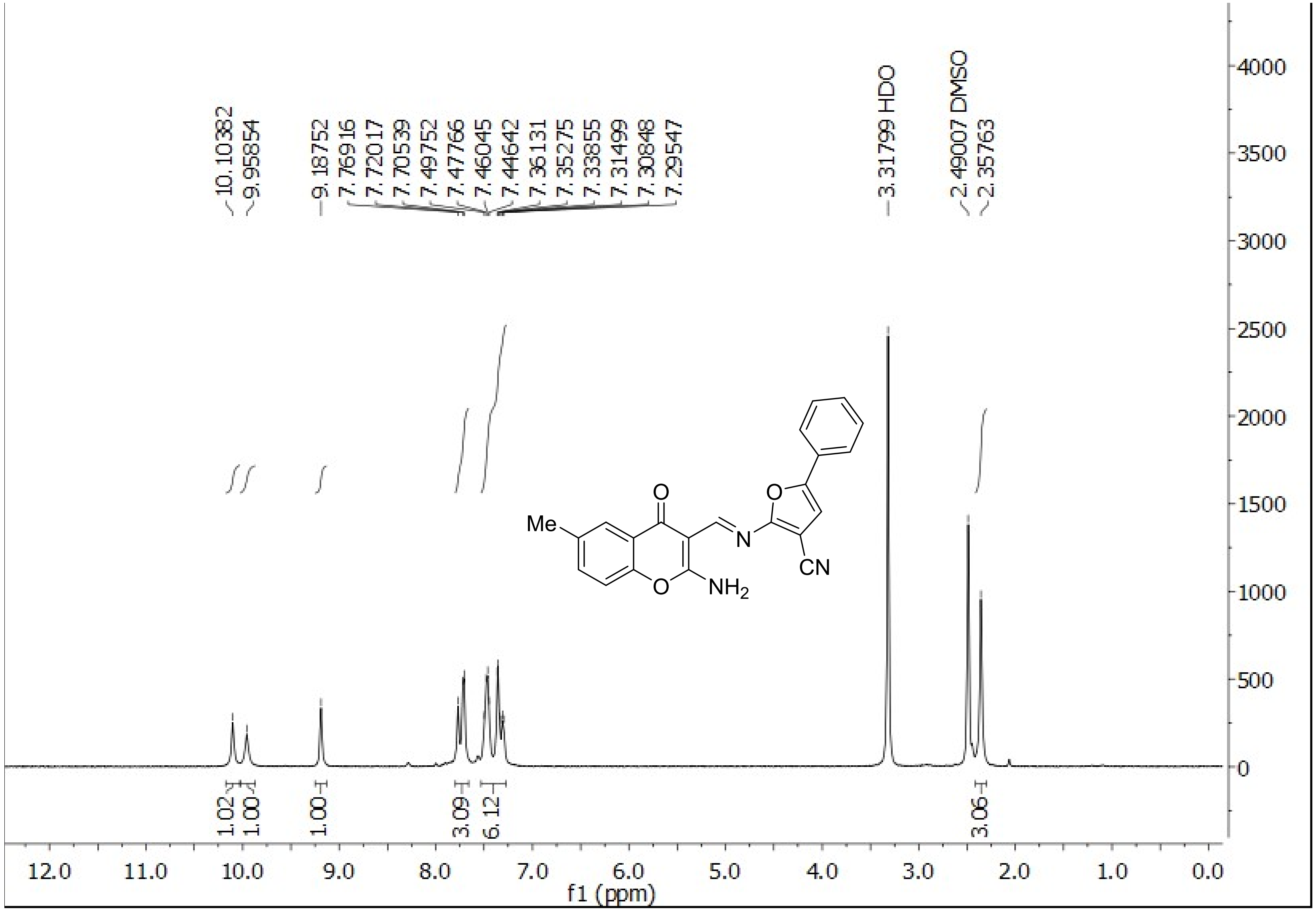
$^{13}C\{^{1}H\}$ NMR of **3f** (125 MHz, DMSO-d_6)



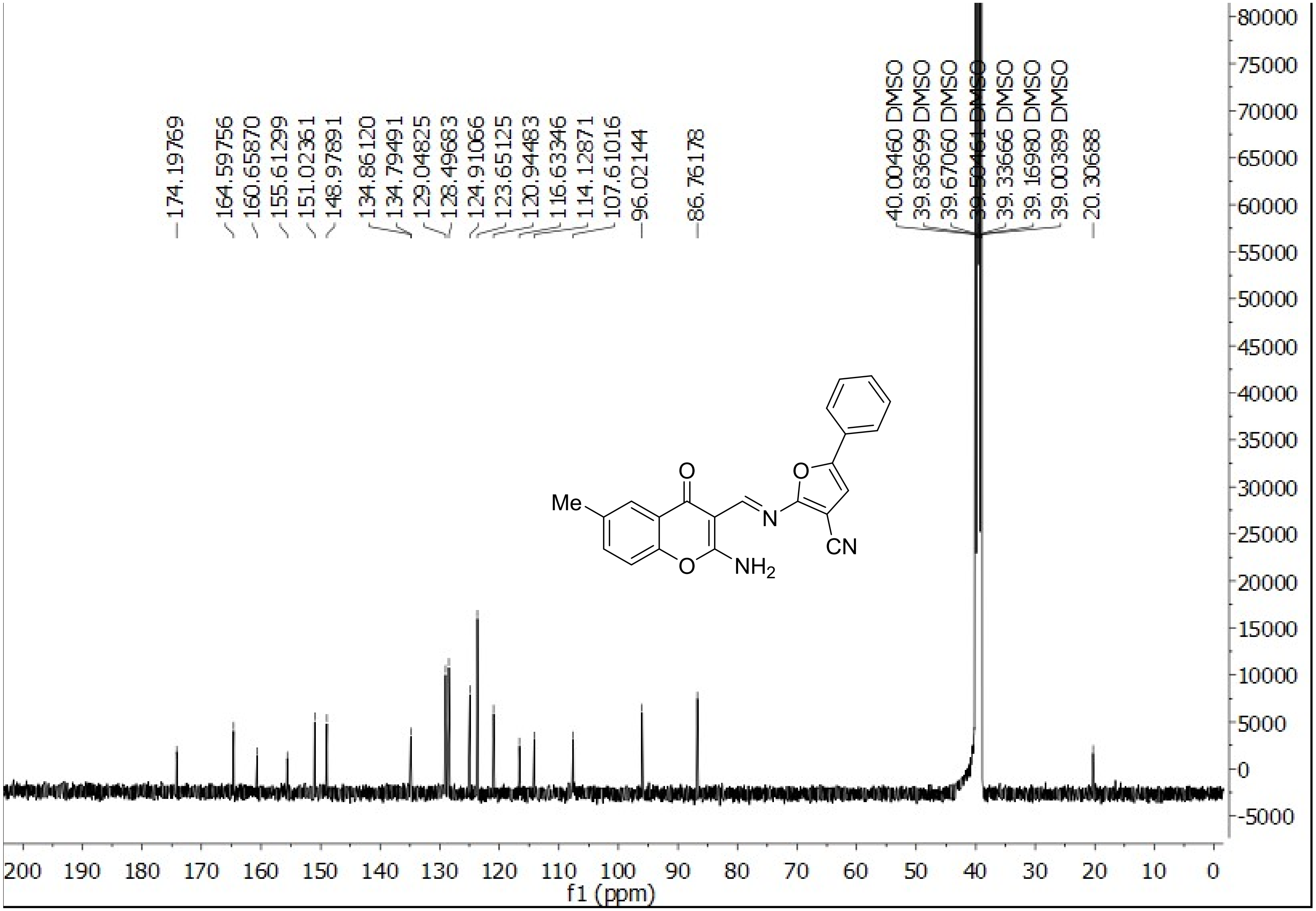
^{1}H NMR of **3g** (500 MHz, DMSO-d_6)



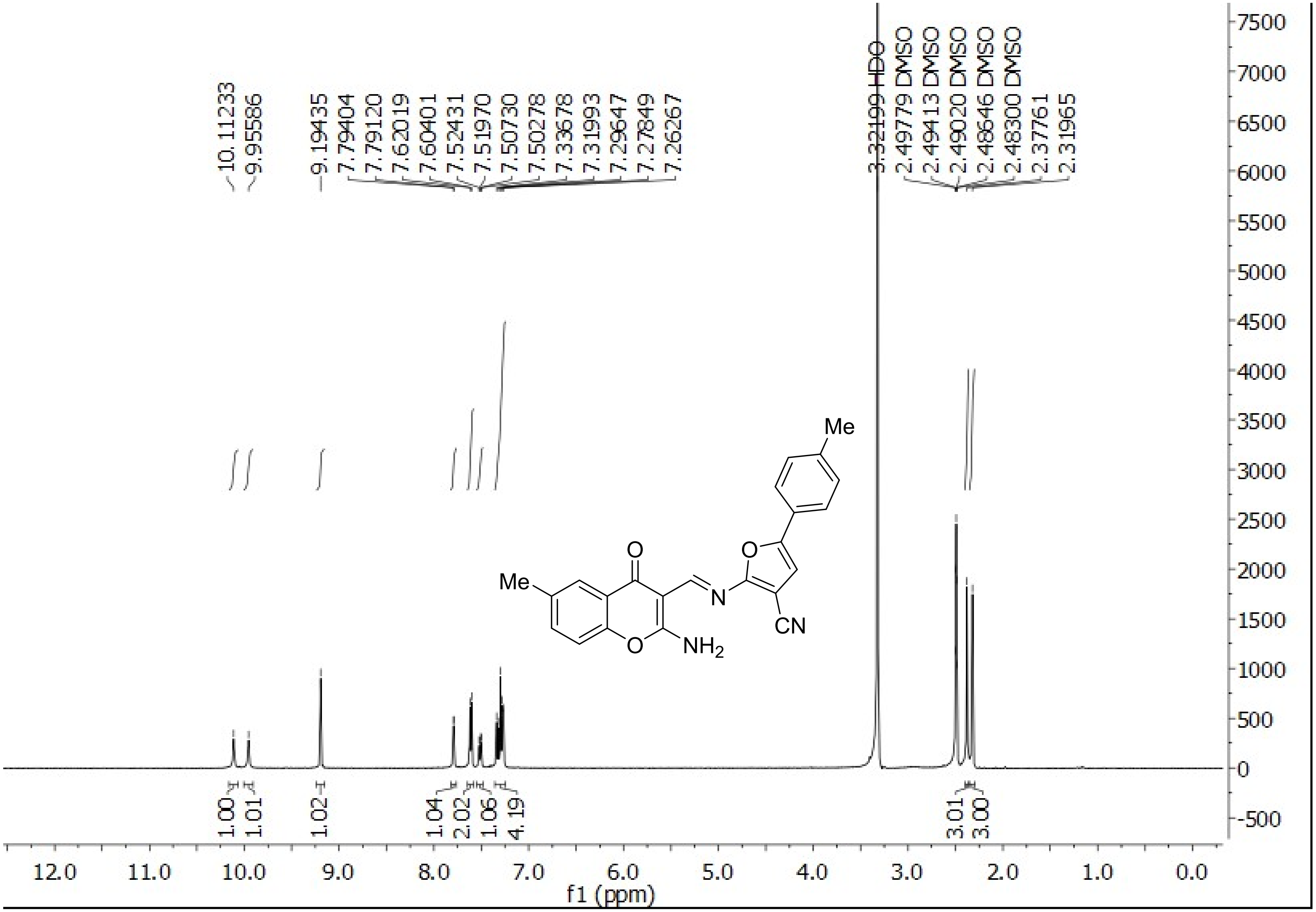
^{13}C{^{1}H} NMR of **3g** (125 MHz, DMSO-*d*$_6$)



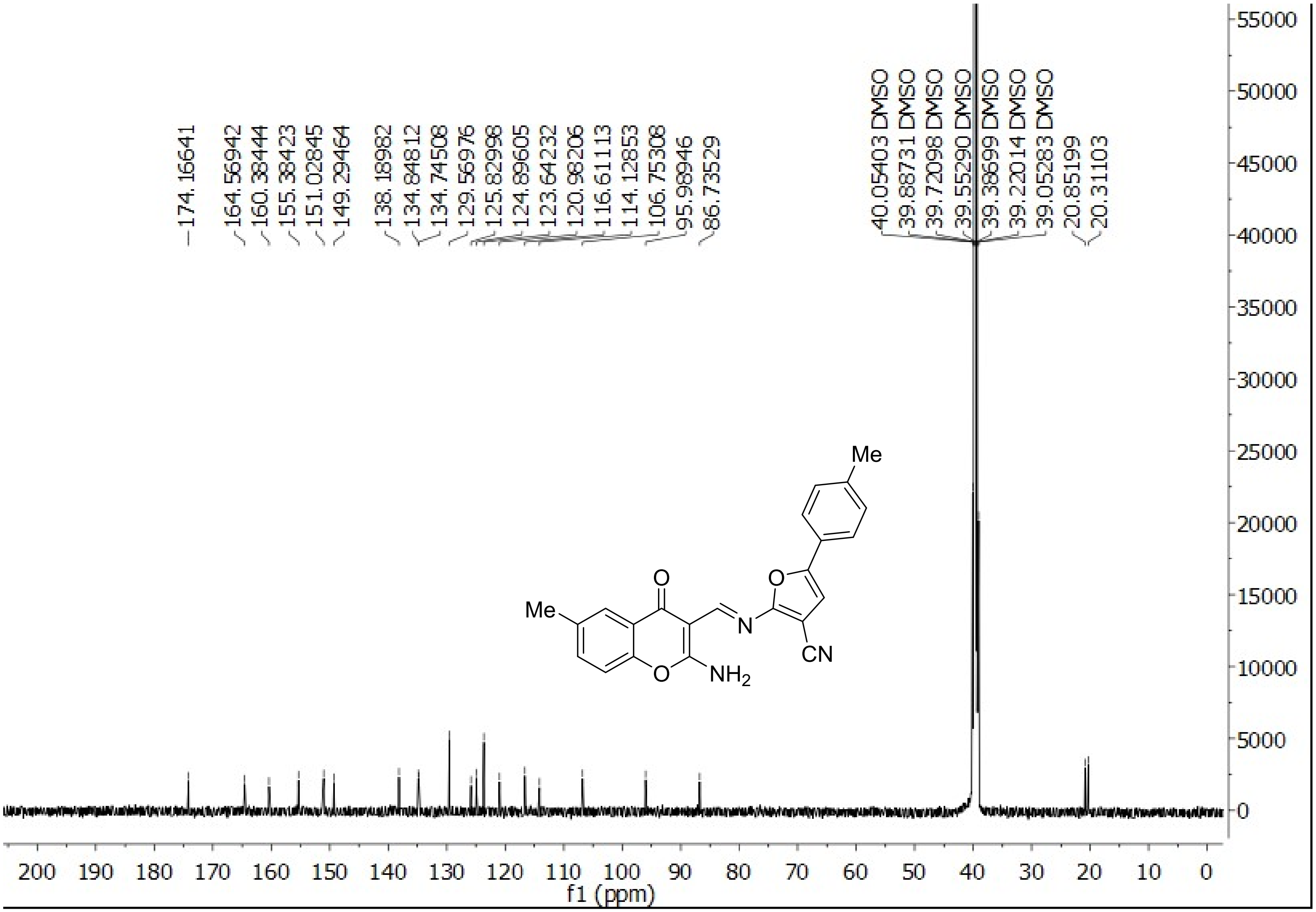
^{1}H NMR of **3h** (500 MHz, DMSO-d_6)



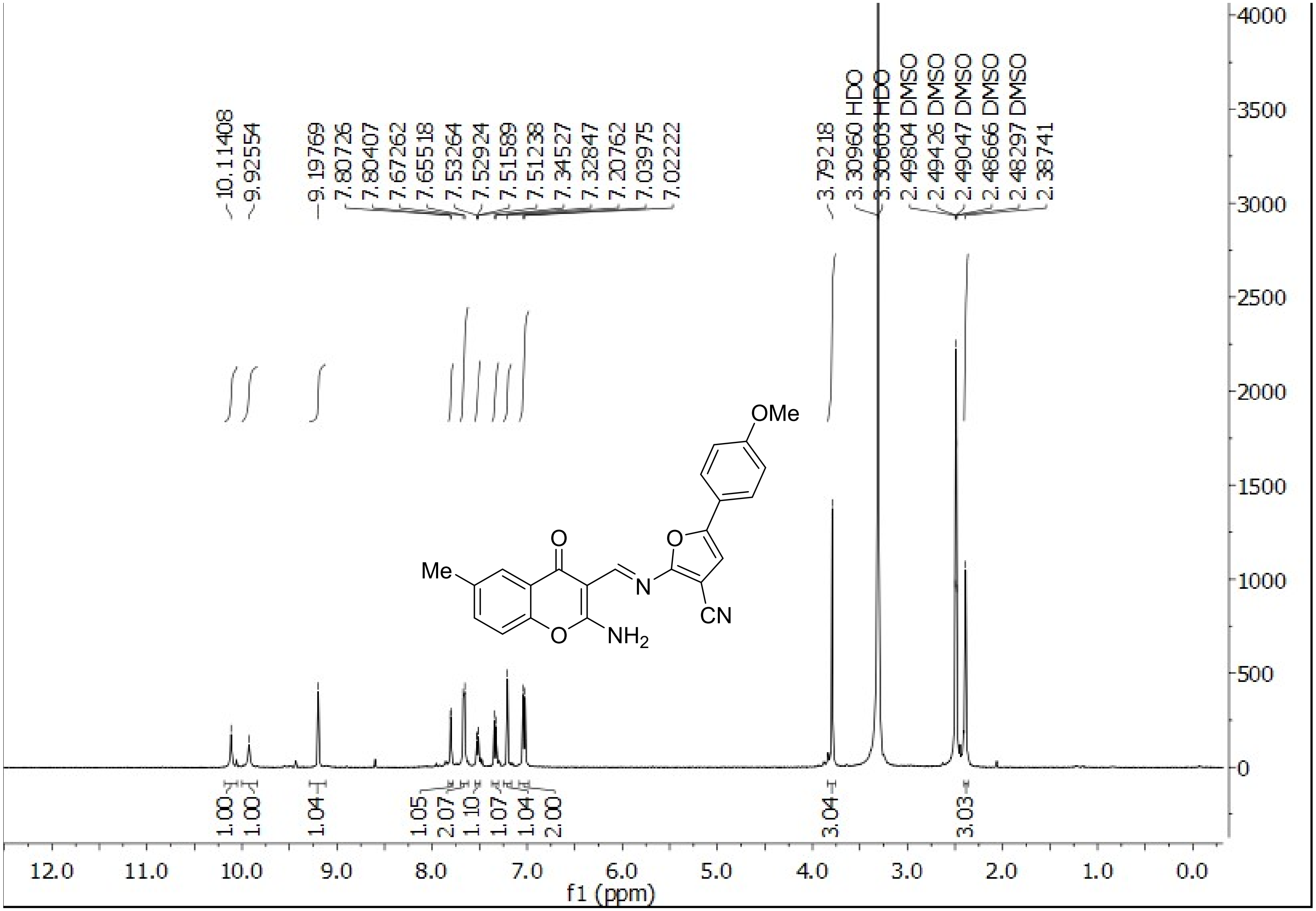
$^{13}C\{^1H\}$ NMR of **3h** (125 MHz, DMSO-d_6)



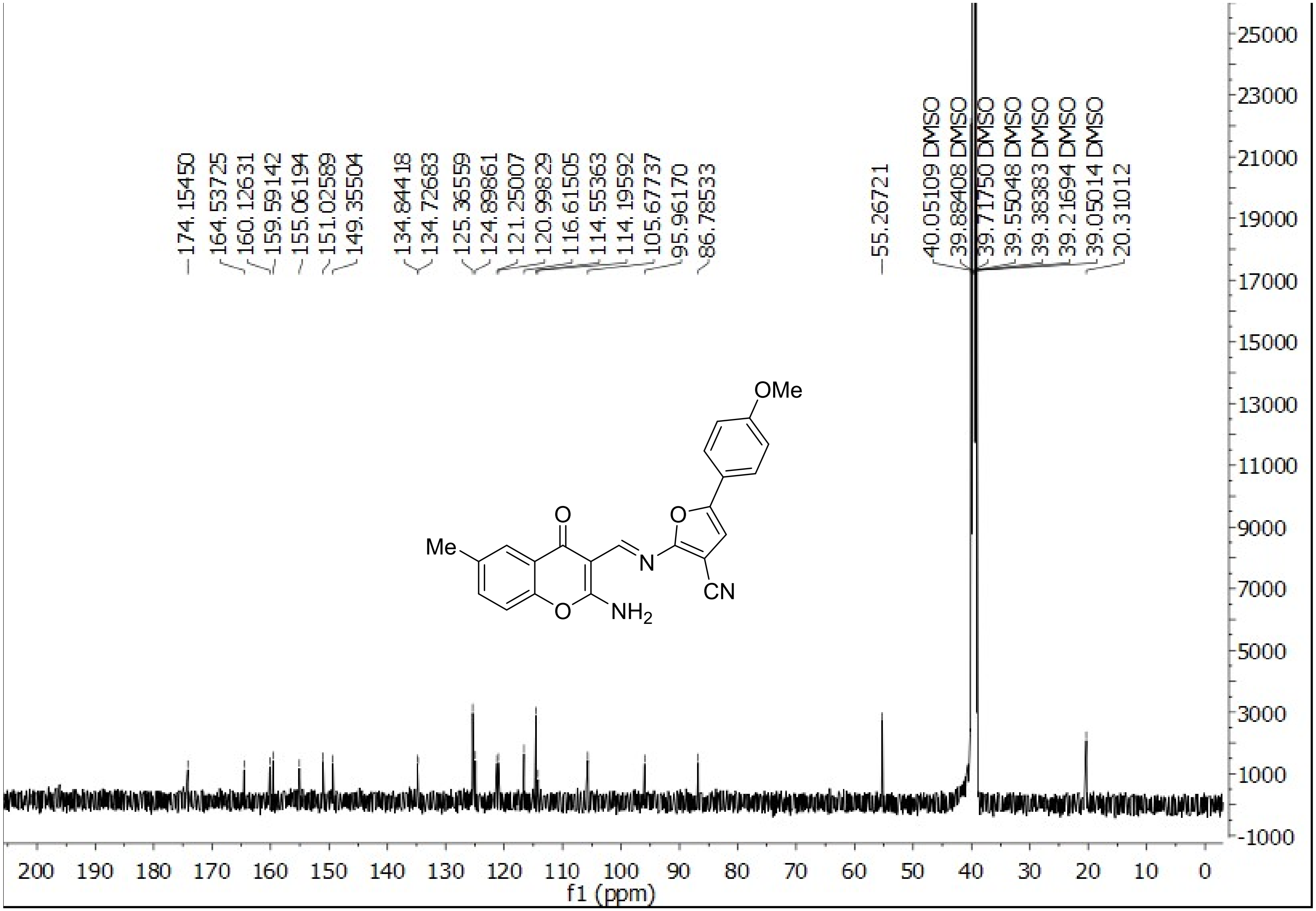
^{1}H NMR of **3i** (500 MHz, DMSO-d_6)



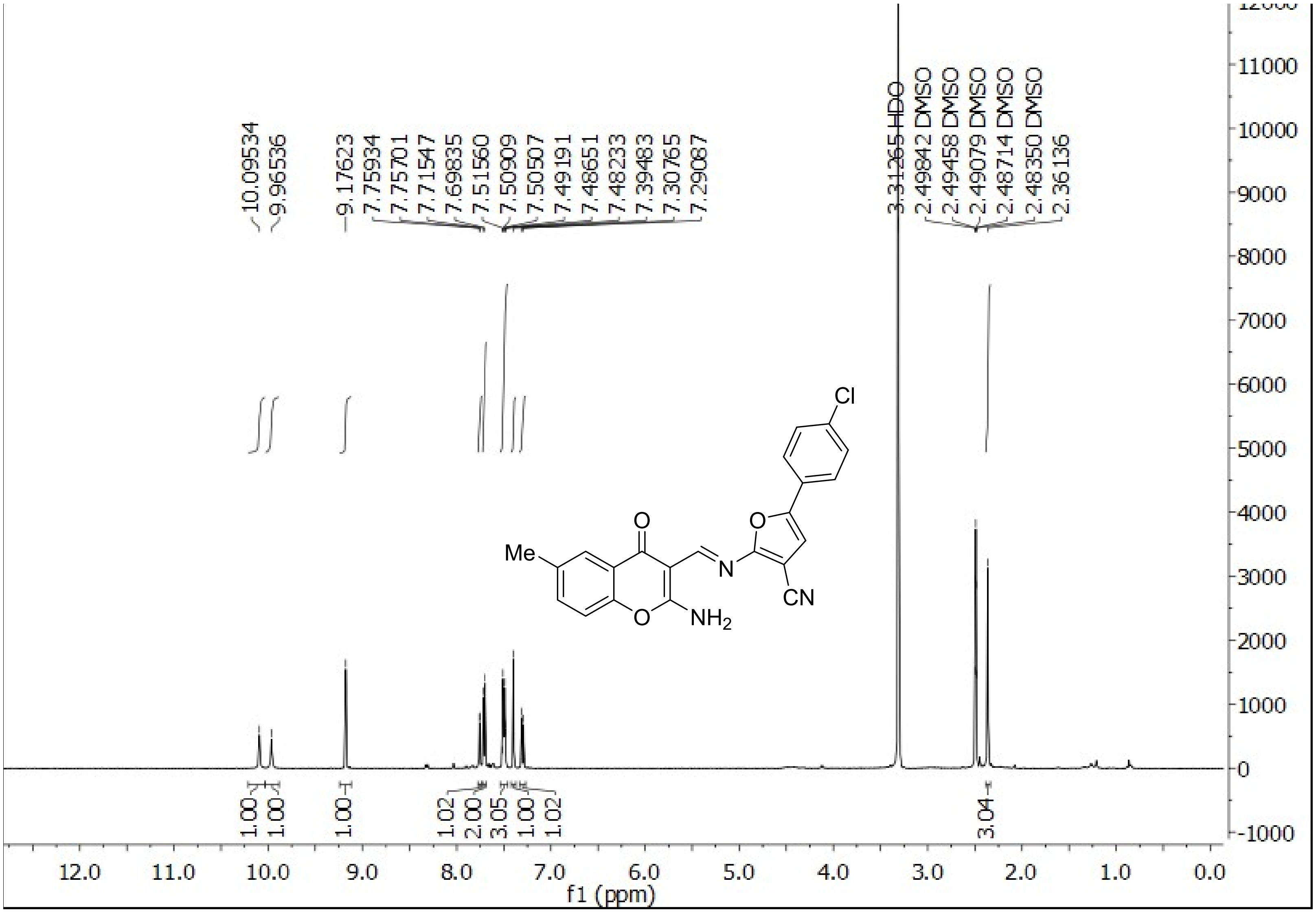
$^{13}C\{^{1}H\}$ NMR of **3i** (125 MHz, DMSO-d_6)



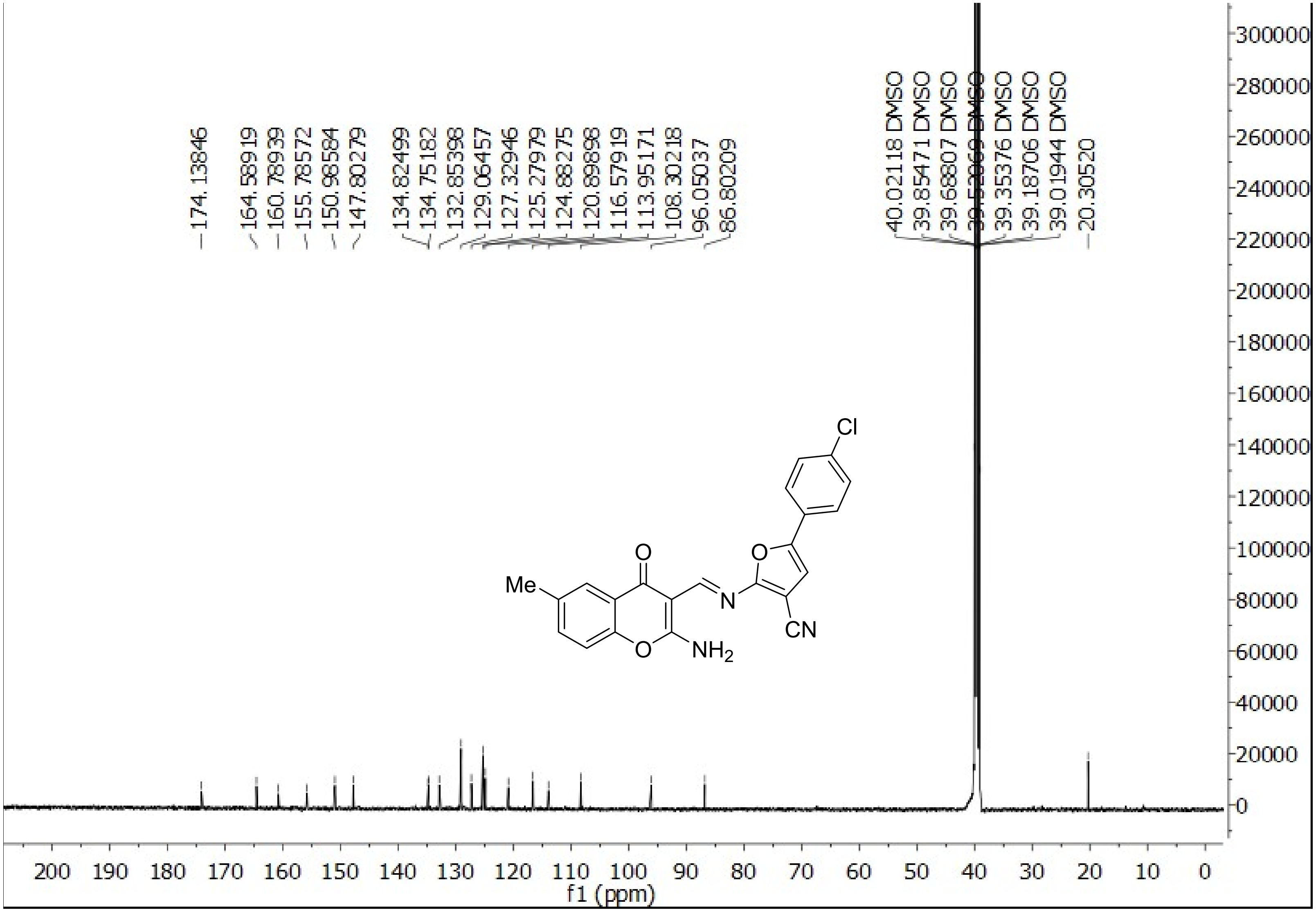
^{1}H NMR of **3j** (500 MHz, DMSO-d_6)



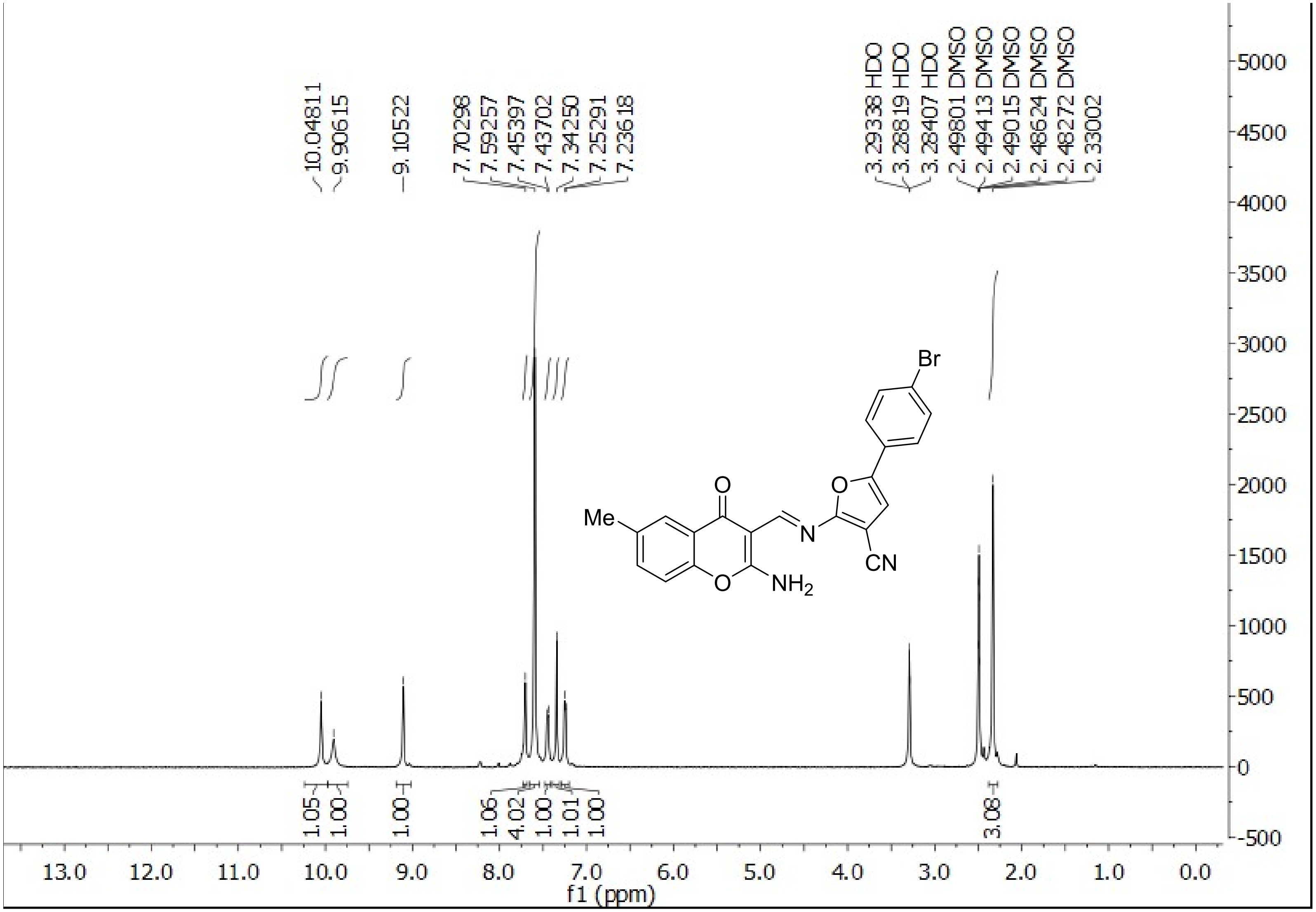
^{13}C{^{1}H} NMR of **3j** (125 MHz, DMSO-d_6)



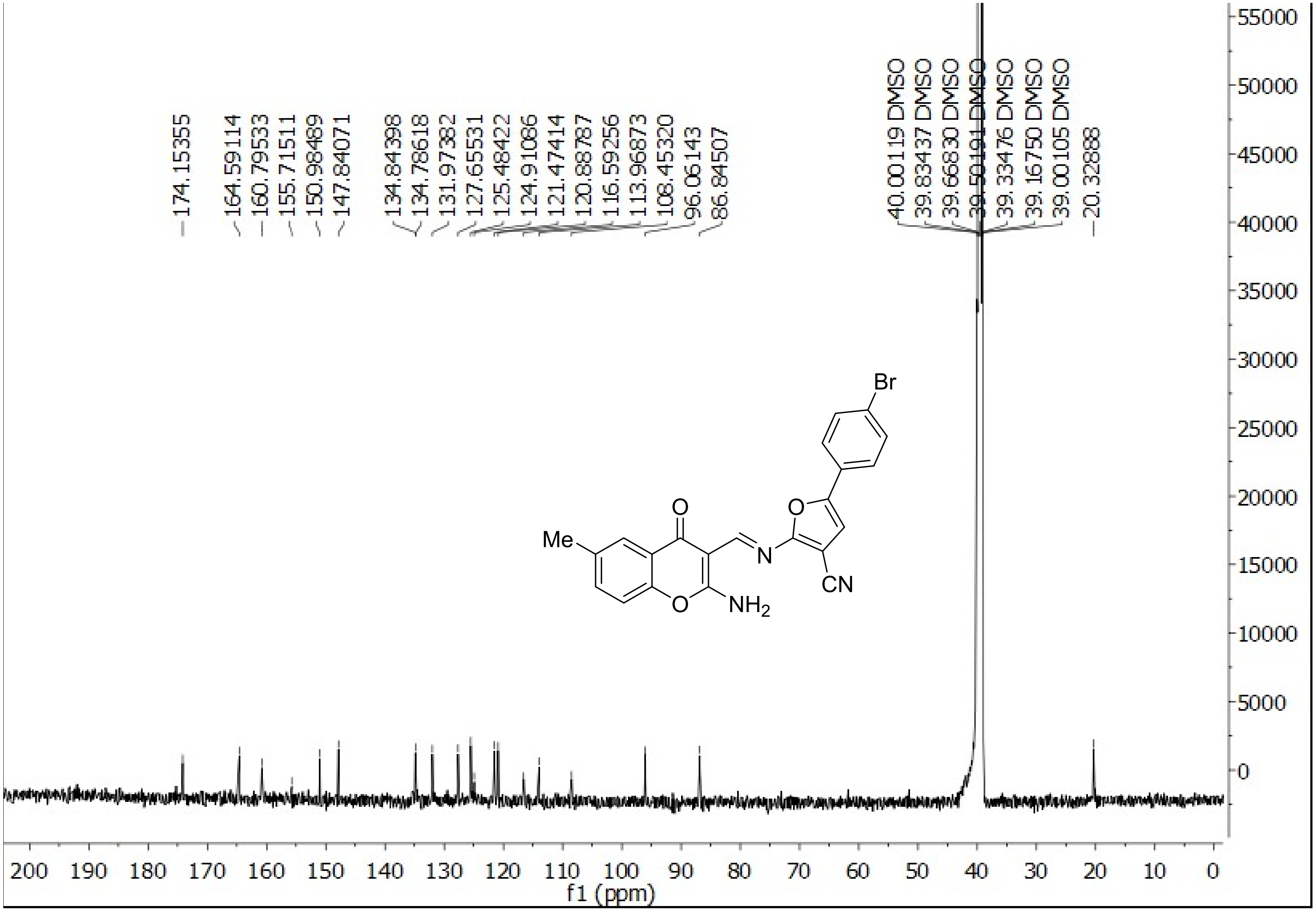
^{1}H NMR of **3k** (500 MHz, DMSO-d_6)



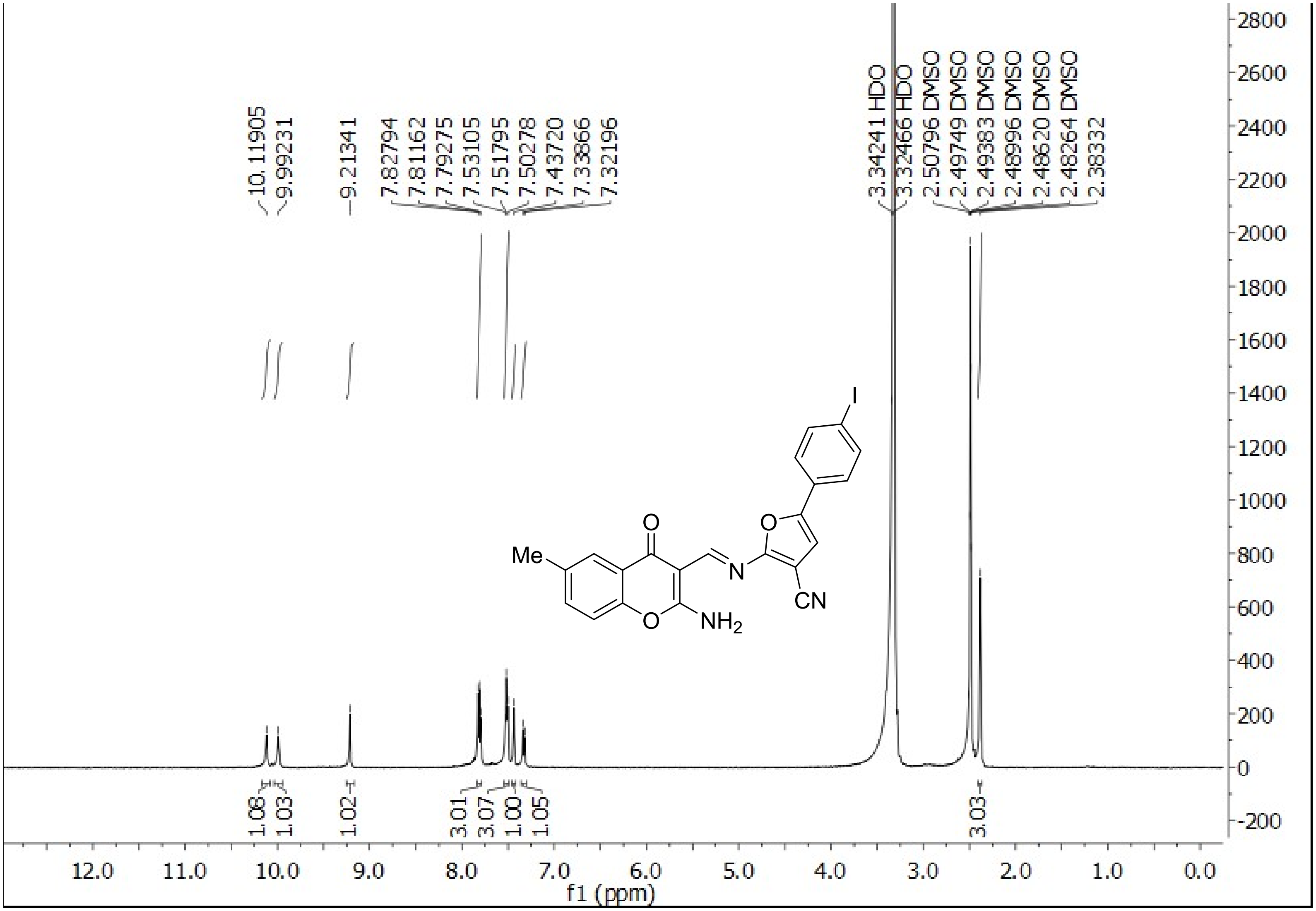
^{13}C{^{1}H} NMR of **3k** (125 MHz, DMSO-d_6)



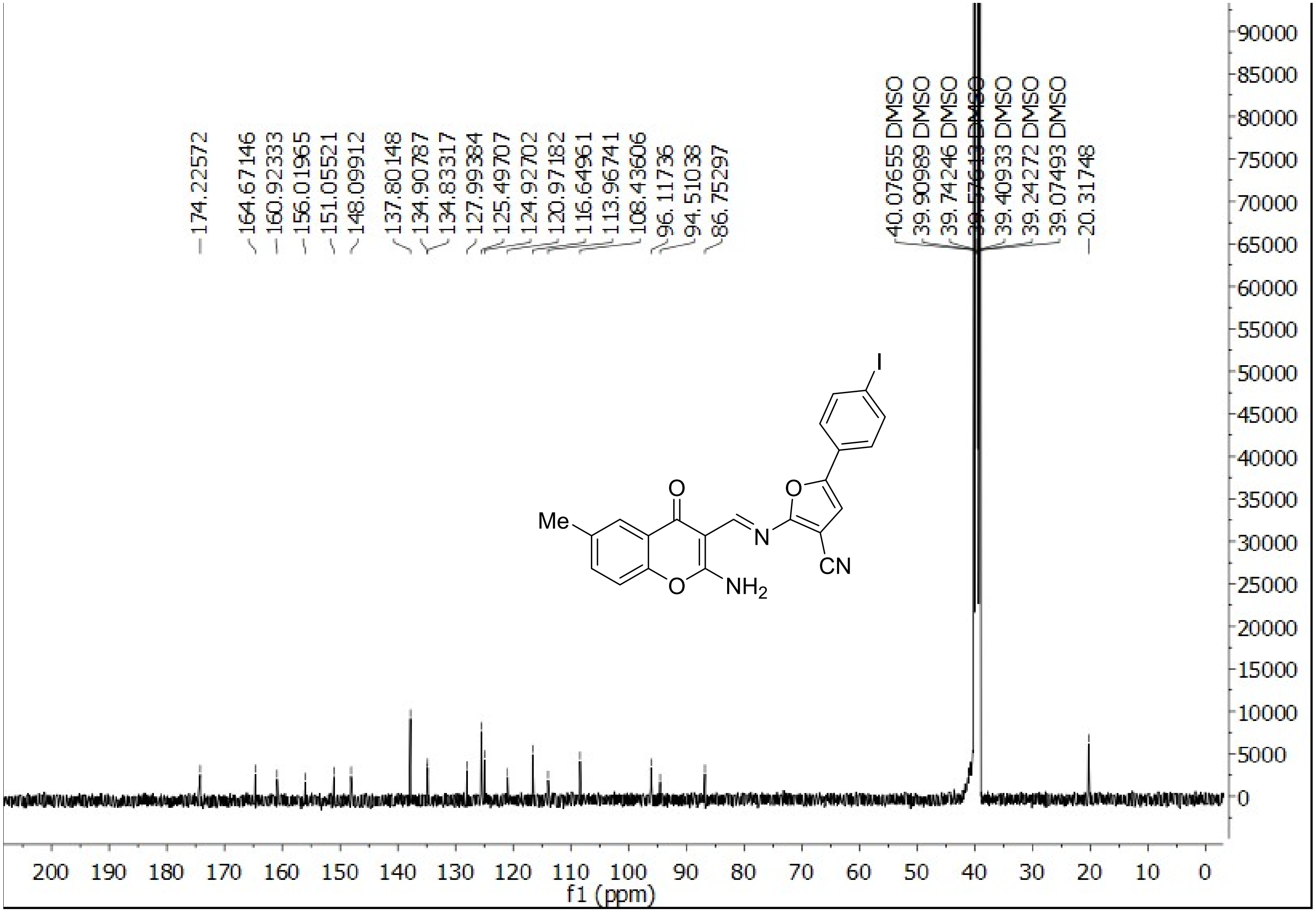
^{1}H NMR of **3l** (500 MHz, DMSO-d_6)



^{13}C{^{1}H} NMR of **3l** (125 MHz, DMSO-*d$_6$*)



^{1}H NMR of **3m** (500 MHz, DMSO-d_6)



^{13}C{^{1}H} NMR of **3m** (125 MHz, DMSO-d_6)

Crystal structure description of compound 3e

The X-ray diffraction measurement was made on a STOE IPDS-II diffractometer with graphite monochromated Mo-Kα radiation. For crystal determination of **3e**, plate yellow crystal was chosen using a polarizing microscope and was mounted on a glass fiber which was used for data collection. Cell constants and orientation matrices for data collection were obtained by least-squares refinement of diffraction data from 4046 unique reflections for **3e**. Data were collected to a maximum 2θ value of 50° in a series of ω scans in 1° oscillations and integrated using the Stoe X-AREA[3] software package. The data were corrected for Lorentz and Polarizing effects. The structure was solved by direct methods[4] and subsequent difference Fourier maps and then refined on F^2 by a full-matrix least-squares procedure using anisotropic displacement parameters[5]. All hydrogen atoms attached to carbon were added in idealized positions. Hydrogen atoms of N–H were found in difference Fourier maps. The atomic factors were taken from the International Tables for X-ray Crystallography[6]. All refinements were performed using the X-STEP32 crystallographic software package[7]. CCDC No. 2478275 contains crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

*Crystal data for **3e***. $C_{24}H_{19}ClN_4O_4$, M = 462.88, yellow plate, crystal dimensions: 0.30×0.20×0.20 mm^3; monoclinic, space group $P2_1/c$; a = 16.683(3), b = 8.7068(17), c = 16.643(3) Å; β = 107.37(3)°; V = 2307.2(8) Å^3; T = 298(2) K; Z = 4; D_{calc} = 1.333 g cm^{-3}; μ = 0.204 mm^{-1} (for Mo Kα, λ = 0.71073 Å); F(000) = 960; reflections collected = 10979; reflections independent = 4046 [R_{int} = 0. 0.1615]; ϑ range 2.50 to 25.00; full-matrix least-squares on F^2; parameters = 306; restraints = 3; R_1 = 0.0843; wR_2 = 0.1579 [I>2sigma(I)]; GooF = S = 0.957; largest difference in peak and hole, $\Delta\rho_{\max}$ and $\Delta\rho_{\min}$ = 0.198 and -0.238 e.Å^3.

References

1. Farajpour, B.; Alizadeh, A. Chemoselective and One-Pot Synthesis of Novel Coumarin-Based Cyclopenta[*c*]pyrans *via* Base-Mediated Reaction of *α*,*β*-Unsaturated Coumarins and *β*-Ketodinitriles. *RSC Adv.* **2022**, *12*, 7262-7267.
2. Yan, J.; Cheng, M.; Hu, F.; Hu, Y. Direct Synthesis of Functional Azaxanthones by Using a Domino Three-Component Reaction. *Org. Lett.* **2012**, *14*, 3206-3209.
3. Stoe & Cie, X–AREA: Program for the Acquisition and Analysis of Data, Version 1.30; Stoe & Cie GmbH: Darmatadt, Germany, 2005.
4. Sheldrick, G. M. SHELX97. Program for Crystal Structure Solution. University of Göttingen, Germany, 1997.
5. Sheldrick, G. M. SHELX97. Program for Crystal Structure Refinement. University of Göttingen, Germany, 1997.
6. International Tables for X-ray Crystallography, Vol C, Kluwer Academic Publisher, Doordrecht, The Netherlands,1995.
7. Stoe & Cie, X-STEP32: Crystallographic Package, Version 1.07b; Stoe & Cie GmbH: Darmstadt, Germany, 2000.