Supporting information

One-step metal-free construction of fluorescent 5-aryl-2,3-dicyanofurans from simple aryl ketones with DDQ

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Contents

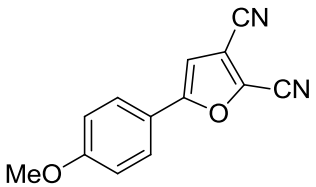
1) General information

All solvents and reagents were purchased from the suppliers and used without further purification unless otherwise noted. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded in $CDCl_3$ at room temperature on Bruker Avance III 400 spectrometer. The chemical-shift scale is based on internal TMS. IR spectra were recorded by Thermo Nicolet Avatar 360. MS spectra were performed on a Agilent 6890/5973 GC-MS. Elemental analyses were measured on a Perkin Elmer 2400 series analyzer. TLC analyses were performed on silica gel plates and column chromatography was conducted over silica gel (mesh 200-300) at increased pressure.

2) Experimental details and characterization data for the furans

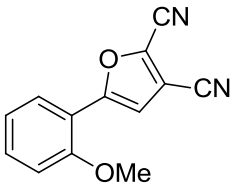
DCE (2 mL) was added to the ketone (0.5 mmol) and DDQ (340 mg, 1.5 mmol) in a sealed tube. The resulting dark reaction mixture was stirred at 100^{o}C for 13 h under N_2. After the mixture was cooled to room temperature, ethyl acetate (50 mL) added and the resulting mixture was washed with aqueous 5% $NaHCO_3$ (3 × 10 mL) and brine. Silica gel (1.0 g) was added and the mixture rotary evaporated. The resulting powder was added to the top of a short silica-gel column and purified using petroleum ether/ethyl acetate in a 1:10 ratio (volume ratio) as the eluent to afford the desired product.

5-(4-methoxyphenyl)furan-2,3-dicarbonitrile (2a)



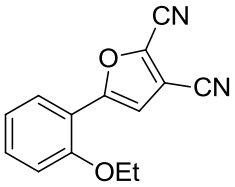
Light yellow solid, m.p. 116-117 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.64 (d, *J* = 8.8 Hz, 2 H), 6.94 (d, *J* = 8.8 Hz, 2 H), 6.78 (s, 1 H), 3.87 (s, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.8, 160.2, 129.2, 127.0 (2 C), 119.6, 114.9 (2 C), 110.1, 110.0, 105.4, 100.0, 55.6. IR (KBr) ν_{max}/cm^{-1} 3128, 3028, 2965, 2928, 2840, 2239, 2226, 1610, 1491, 1439, 1427, 1303, 1262, 1180, 1025, 840, 824, 813. MS(EI): 224 [M]$^{+}$,181. Anal. Calcd for $C_{13}H_8N_2O_2$ C 69.64, H 3.60, N 12.49%; Found: C 69.82, H 3.71, N 12.37%.

5-(2-methoxyphenyl)furan-2,3-dicarbonitrile (2b)



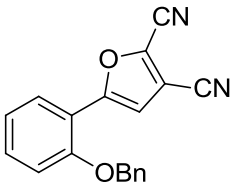
Light yellow solid, m.p. 129-130 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.84 (d, *J* = 7.8 Hz, 1 H), 7.45 (t, *J* = 7.7 Hz, 1 H), 7.23 (s, 1 H), 7.09 (t, *J* = 7.6 Hz, 1 H), 7.03 (d, *J* = 8.4 Hz, 1 H), 3.99 (s, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 156.6, 156.5, 132.0, 128.7, 127.1, 121.2, 115.9, 111.6, 111.4, 110.4, 110.0, 109.1, 55.7. IR (KBr) ν_{max}/cm^{-1} 3154, 3139, 2925, 2841, 2241, 2231, 1603, 1530, 1492, 1452, 1282, 1255, 1186, 1018, 756. MS(EI): 224 [M]$^{+}$, 181. Anal. Calcd for $C_{13}H_8N_2O_2$ C 69.64, H 3.60, N 12.49%; Found: C 69.73, H 3.79, N 12.30 %.

5-(2-ethoxyphenyl)furan-2,3-dicarbonitrile (2e)



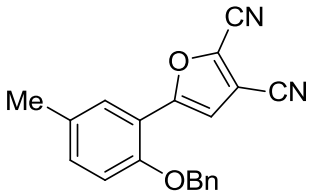
Light yellow solid, m.p. 115-116 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.84 (dd, *J* =7.9, 1.6 Hz, 1 H), 7.42 (dt, *J* = 7.6, 1.6 Hz, 1 H), 7.24 (s, 1 H), 7.07 (t, *J* = 7.6 Hz, 1 H), 7.00 (d, *J* = 8.4 Hz, 1 H), 4.21 (q, *J* = 7.0 Hz, 2 H), 1.56 (t, *J* = 7.0 Hz, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 156.6, 156.0, 131.9, 128.6, 127.0, 121.0, 115.8, 112.1, 111.4, 110.4, 109.9, 109.1, 64.3, 14.8. IR (KBr) ν_{max}/cm^{-1} 3164, 2925, 2854, 2244, 2225, 1603, 1527, 1494, 1456, 1289, 1255, 1184, 1019, 754. MS(EI): 238 $[M]^+$, 181. Anal. Calcd for $C_{14}H_{10}N_2O_2$ C 70.58, H 4.23, N 11.76%; Found: C 70.35, H 4.17, N 11.91%.

5-(2-(benzyloxy)phenyl)furan-2,3-dicarbonitrile (2f)



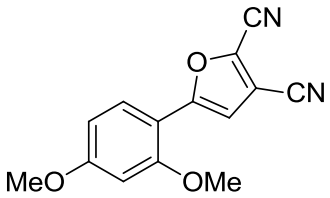
Light yellow solid, m.p. 125-126 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.87 (d, *J* = 6.8 Hz, 1 H), 7.45-7.41 (m, 6 H), 7.12-7.09 (m, 3 H), 5.22 (s, 2 H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 156.4, 155.7, 135.5, 132.0, 129.0 (2 C), 128.78, 128.76, 127.8 (2 C), 127.2, 121.4, 116.1, 112.6, 111.5, 110.3, 110.0, 109.1, 70.9. IR (KBr) ν_{max}/cm^{-1} 3161, 3030, 2937, 2880, 2854, 2243, 2227, 1603, 1522, 1491, 1444, 1375, 1306, 1289, 1247, 1166, 997, 828, 752, 697. MS(EI): 300 $[M]^+$, 181, 91. Anal. Calcd for $C_{19}H_{12}N_2O_2$ C 75.99, H 4.03, N 9.33%; Found: C 75.79, H 4.17, N 9.48%.

5-(2-(benzyloxy)-5-methylphenyl)furan-2,3-dicarbonitrile (2g)



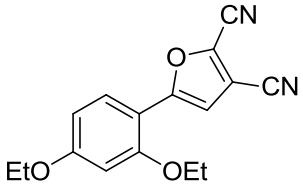
Light yellow solid, m.p. 131-132 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.55 (s, 1 H), 7.34-7.29 (m, 5 H), 7.11 (d, *J* = 8.8 Hz, 1 H), 7.01(s, 1 H), 6.88 (d, *J* = 8.8 Hz, 1 H), 5.08 (s, 2 H), 2.26 (s, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 156.5, 153.7, 135.7, 132.4, 130.7, 128.8 (2 C), 128.6, 128.5, 127.6 (2 C), 127.3，115.6, 112.5, 111.3, 110.2, 109.8, 109.0, 70.8, 20.4. IR (KBr) ν_{max}/cm^{-1} 3166, 3030, 2924, 2871, 2243, 2226, 1612, 1501, 1454, 1371, 1289, 1244, 1005, 817, 743, 697. MS(EI): 314 $[M]^+$, 195, 91. Anal. Calcd for $C_{20}H_{14}N_2O_2$ C 76.42, H 4.49, N 8.91%; Found: C 76.61, H 4.38, N 9.10 %.

5-(2,4-dimethoxyphenyl)furan-2,3-dicarbonitrile (2h)



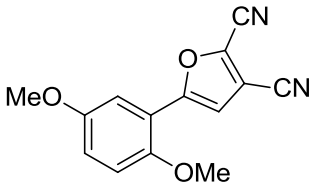
Yellow solid, m.p. 126-127 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.77 (d, *J* = 8.7 Hz, 1 H), 7.08 (s, 1 H), 6.62 (dd, *J* = 8.7, 2.3 Hz, 1 H), 6.54 (d, *J* = 2.3 Hz, 1 H), 3.96 (s, 3 H), 3.88 (s, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.9, 158.0, 156.8, 128.2, 127.9, 110.5, 109.9, 109.5, 109.3, 105.7, 98.7, 55.7, 55.6. IR (KBr) ν_{max}/cm^{-1} 3263, 2950, 2845, 2244, 2225, 1603, 1529, 1492, 1288, 1247, 1186, 1167, 1105, 1053, 1022, 832, 764. MS(EI): 254 $[M]^+$, 211. Anal. Calcd for $C_{14}H_{10}N_2O_3$ C 66.14, H 3.96, N 11.02%; Found: C 66.02, H 4.13, N 11.25%.

5-(2,4-diethoxyphenyl)furan-2,3-dicarbonitrile (2i)



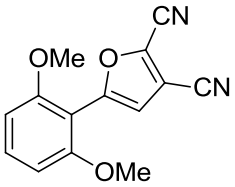
Yellow solid, m.p. 124-125 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.73 (d, *J* = 8.8 Hz, 1 H), 7.06 (s, 1 H), 6.58 (dd, *J* = 8.8, 2.2 Hz, 1 H), 6.05 (d, *J* = 2.0 Hz, 1 H), 4.15 (q, *J* = 7.0 Hz, 2 H), 4.08 (q, *J* = 7.0 Hz, 2 H), 1.55 (t, *J* = 7.0 Hz, 3 H), 1.45 (t, *J* = 7.0 Hz, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.2, 157.4, 157.0, 128.1, 127.8, 110.6, 109.9, 109.4, 109.3, 109.1, 106.1, 99.6, 64.3, 63.9, 14.73, 14.67. IR (KBr) ν_{max}/cm^{-1} 3163, 2983, 2974, 2937, 2883, 2244, 2223, 1610, 1499, 1427, 1397, 1327, 1292, 1266, 1202, 1045, 1045, 850, 820. MS(EI): 282 $[M]^+$, 225. Anal. Calcd for $C_{16}H_{14}N_2O_3$ C 68.07, H 5.00, N 9.92%; Found: C 68.27, H 5.14, N 9.80 %.

5-(2,5-dimethoxyphenyl)furan-2,3-dicarbonitrile (2j)



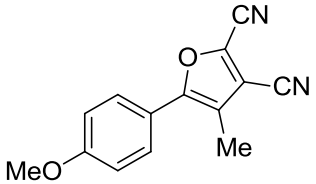
Yellow solid, m.p. 120-121 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.34 (d, *J* = 2.8 Hz, 1 H), 7.26 (s, 1 H), 7.01-6.94 (m, 2 H), 3.94 (s, 3 H), 3.85 (s, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 156.3, 153.7, 151.0, 128.6, 117.9, 116.1, 112.6, 111.8, 111.3, 110.3, 110.0, 109.2, 56.04, 55.98. IR (KBr) ν_{max}/cm^{-1} 3164, 2966, 2837, 2242, 2229, 1579, 1501, 1460, 1277, 1243, 1204, 1166, 1052, 845, 808, 738. MS(EI): 254 $[M]^+$, 211. Anal. Calcd for $C_{14}H_{10}N_2O_3$ C 66.14, H 3.96, N 11.02%; Found: C 65.96, H 3.89, N 11.26%.

5-(2,6-dimethoxyphenyl)furan-2,3-dicarbonitrile (2k)



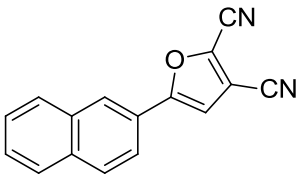
Yellow solid, m.p. 127-128 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.40 (t, *J* = 8.5 Hz, 1 H), 6.87 (s, 1 H), 6.63 (d, *J*= 8.5 Hz, 2 H), 3.84 (s, 6 H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 158.8 (2 C), 154.7, 132.7, 127.8, 118.9, 113.7, 110.6, 109.3, 108.8, 104.0 (2 C), 56.1 (2 C). IR (KBr) ν_{max}/cm^{-1} 3275, 3180, 2958, 2927, 2872, 2857, 2267, 2235, 1591, 1473, 1378, 1272, 1119, 1073, 782, 742. MS(EI): 254 $[M]^+$, 211. Anal. Calcd for $C_{14}H_{10}N_2O_3$ C 66.14, H 3.96, N 11.02%; Found: C 65.97, H 4.03, N 11.22%.

5-(4-methoxyphenyl)-4-methylfuran-2,3-dicarbonitrile (2l)



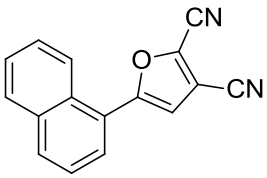
Light yellow solid, m.p. 120-121 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.59 (d, *J* = 8.9 Hz, 2 H), 7.02 (d, *J* = 8.9 Hz, 2 H), 3.88 (s, 3 H), 2.39 (s, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.0, 155.6, 128.1 (2 C), 127.8, 120.5, 117.3, 114.7 (2 C), 112.4, 110.1, 109.3, 55.5, 10.4. IR (KBr) ν_{max}/cm^{-1} 3071, 3018, 2965, 2931, 2839, 2242, 2222, 1612, 1504, 1458, 1404, 1307, 1273, 1255, 1177, 1040, 1021, 846, 836. MS(EI): 238 $[M]^+$, 195. Anal. Calcd for $C_{14}H_{10}N_2O_2$ C 70.58, H 4.23, N 11.76%; Found: C 70.37, H 4.34, N 11.62 %.

5-(naphthalen-2-yl)furan-2,3-dicarbonitrile (2m)



Light yellow solid, m.p. 73-74 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.17 (s, 1 H), 7.89-7.85 (m, 2 H), 7.81 (t, *J* = 4.4 Hz, 1 H), 7.65 (dd, *J* = 8.6, 1.8 Hz, 1 H), 7.53-7.51 (m, 2 H), 6.96 (s, 1 H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.1, 134.2, 133.0, 130.0, 129.5, 128.7, 128.1, 128.0, 127.5, 125.5, 124.0, 121.7, 110.1, 110.0, 108.8, 107.3. IR (KBr) ν_{max}/cm^{-1} 3124, 3059, 2956, 2924, 2869, 2852, 2243, 2227, 1583, 1461, 1377, 1271, 1181, 1050, 893, 815, 752. MS(EI): 244 $[M]^+$, 215. Anal. Calcd for $C_{16}H_8N_2O$ C 78.68, H 3.30, N 11.47%; Found: C 78.56, H 3.41, N 11.59 %.

5-(naphthalen-1-yl)furan-2,3-dicarbonitrile (2n)



Light yellow solid, m.p. 98-99 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.06 (d, *J* = 8.3 Hz, 1 H), 7.95 (d, *J* = 8.3 Hz, 1 H), 7.88 (dd, *J* = 7.7, 1.6 Hz, 1 H), 7.69 (dd, *J* = 7.2, 1.1 Hz, 1 H), 7.59-7.48 (m, 3 H), 6.95 (s, 1 H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.8, 133.9, 132.0, 130.4, 129.9, 129.2, 128.3, 128.1, 126.9, 125.2, 124.2, 124.0, 111.2, 110.0, 109.7, 108.8. IR (KBr) ν_{max}/cm^{-1} 3129, 3057, 2956, 2924, 2851, 2238, 2229, 1598, 1583, 1526, 1509, 1390, 1366, 1237, 1119, 831, 803, 777. MS(EI): 244 $[M]^+$, 215. Anal. Calcd for $C_{16}H_8N_2O$ C 78.68, H 3.30, N 11.47%; Found: C 78.52, H 3.43, N 11.65%.

3) Fluorescence images

Figure S1. Fluorescence image of selected dicyanofurans (λ_{ex} = 365 nm, 5×10^{-3} M) in EtOAc: from left to right; **2j**, **2b**, **2e**, **2h**, **2l**, **2f**, and **2n**.

4) Description and figures of crystal structure and spatial packing

The molecular structure of **2f** is shown in Figure 1 together with the atom numbering scheme. The C1...C6 phenyl rings are disordered over two positions, inclined at 23.2(6)° to one another and with final occupancies 0.707(5) and 0.293(5). The central C8...C13 benzene ring is inclined to the principal disordered component of the phenyl ring at an angle of 76.12(6)° and to the furan ring at 4.32(17)° such that the phenyl-furan-dicarbonitrile unit is close to planar. The two carbonitrile substituents also lie close to the furan ring plane with a maximum deviation of only 0.155(5) Å for N2.

In the crystal structure pairs of C—H...N hydrogen bonds involving each of the carbonitrile (or cyano) substituents form $R^2_2(10)$ rings (Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. Angew. Chem. Int. Ed. **1995**, 34, 1555-1573.) and link adjacent molecules into chains. An additional pair of C3—H3...N2 hydrogen bonds generated $R^2_2(28)$ ring motifs giving rise to two dimensional sheets of molecules approximately parallel to (103), Figure 2. These sheets are stacked approximately at right angles to (103) by π...π stacking interactions between the central benzene rings and the furan rings of adjacent molecules with Cg1...Cg2^i = 3.597(2) (i = -1+X, Y, Z) to give a three dimensional network structure, Figure 3.

Platon revealed a solvent accessible void in the structure and the disordered solvent was removed using the SQUEEZE procedure details of which are recorded in the CIF file.

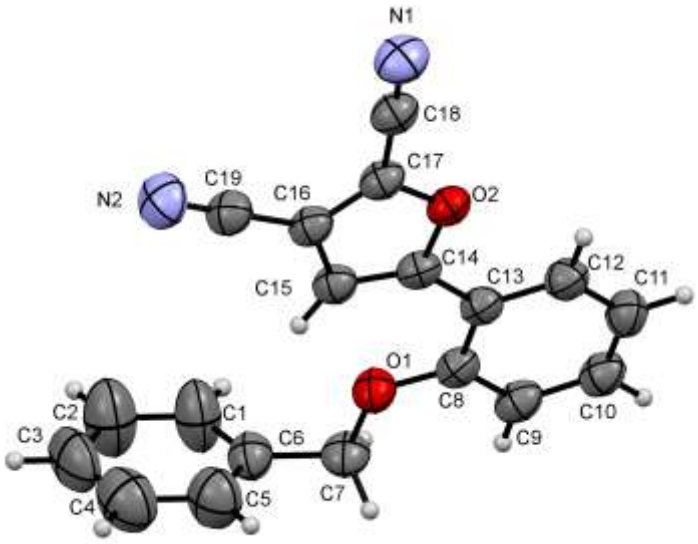


Figure S2. The structure of **2f** showing the atom numbering with ellipsoids drawn at the 50% probability level. For clarity, only the principal disorder component of the disordered C1...C6 phenyl ring is shown.

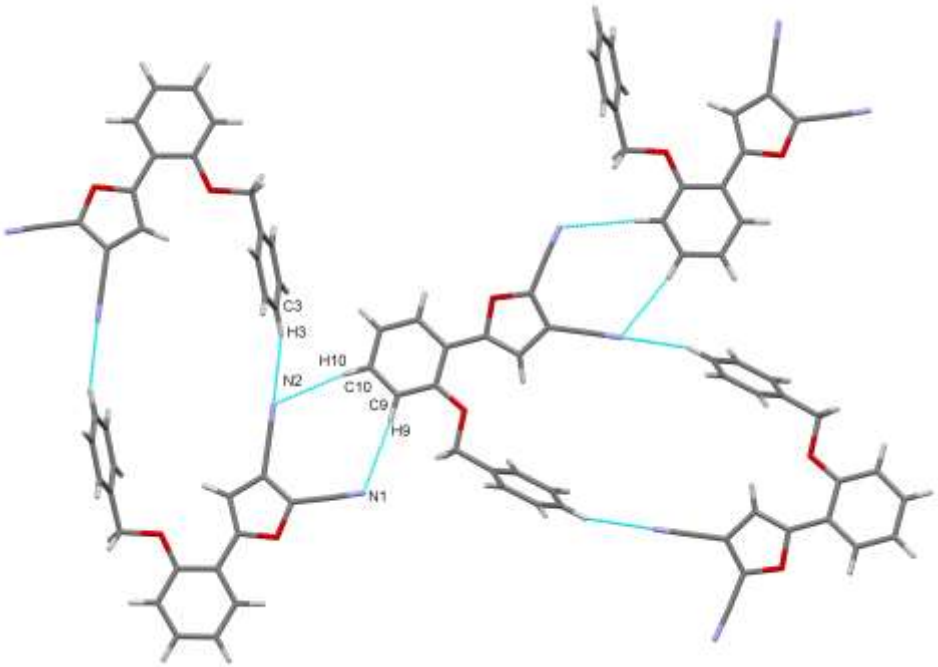


Figure S3. Sheets of molecule approximately parallel to (103) formed by C—H...N hydrogen bonds drawn as blue dashed lines.

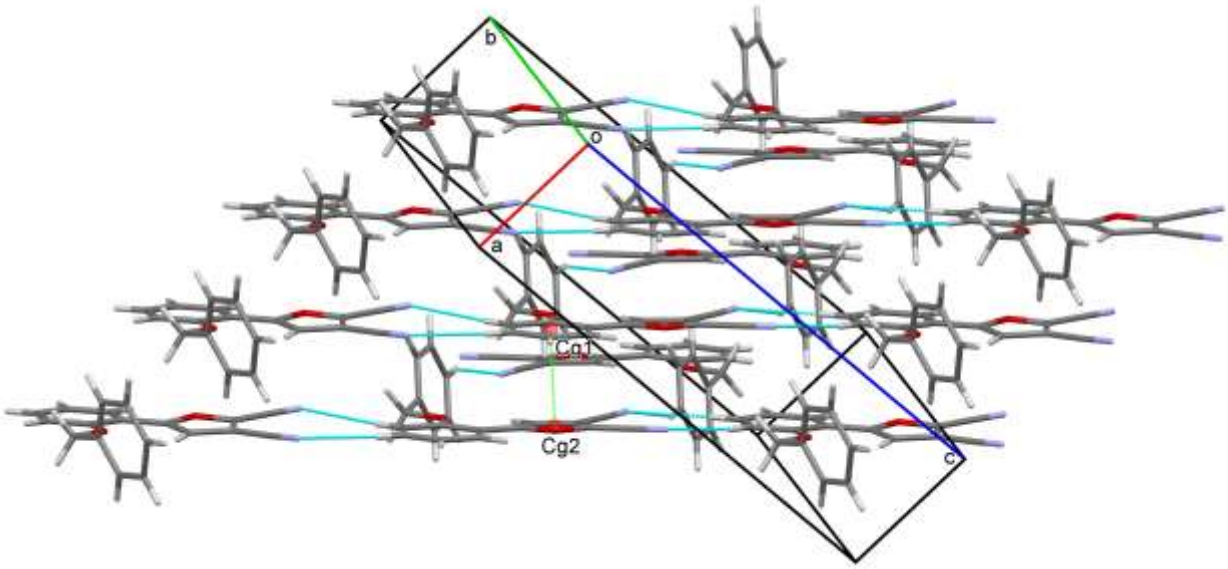


Figure S4. Sheets stacked at right angles to (103) by π...π stacking interactions. A representative π...π contact is shown as a dashed green line between the centroids of the C8...C13 benzene ring, Cg1 and that of the O2, C14...C17 furan ring, Cg2. The ring centroids are displayed as red spheres. Hydrogen bonds are drawn as blue dashed lines.

5) Crystallographic data for 2f

Crystal data

CCDC No.	952813
$C_{19}H_{12}N_2O_2$	D_x = 1.139 Mg m^{-3}
M_r = 300.31	Melting point: ? K
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 5198 reflections
a = 4.7455 (19) Å	θ = 1.6–27.8°
b = 23.385 (10) Å	μ = 0.08 mm^{-1}
c = 15.884 (7) Å	T = 113 (2) K
β = 96.403 (7)°	Cell measurement pressure: ? kPa
V = 1751.7 (12) Å^3	Prism, colourless
Z = 4	0.30 × 0.04 × 0.04 mm
F_{000} = 624	

Data collection

Rigaku Saturn724+ (2x2 bin mode) diffractometer	13869 measured reflections
Radiation source: fine-focus sealed tube	3085 independent reflections
Monochromator: graphite	2334 reflections with $I > 2\sigma(I)$
Detector resolution: 28.5714 pixels mm^{-1}	R_{int} = 0.044
T = 113(2) K	θ_{max} = 25.0°
P = ? kPa	θ_{min} = 1.6°
profile data from ω–scans	h = −5-5
Absorption correction: multi-scan CrystalClear-SM Expert 2.0 r2 (Rigaku, 2009)	k = −27-27
T_{min} = 0.801, T_{max} = 1.000	l = −18-18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.065	H-atom parameters constrained
$wR(F^2)$ = 0.201	$w = 1/[\sigma^2(F_o^2) + (0.1316P)^2 + 0.2092P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.99	$(\Delta/\sigma)_{max}$ = 0.030
3085 reflections	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
252 parameters	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
228 restraints	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
? constraints	Extinction coefficient: 0.041 (8)
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.4458 (8)	0.06056 (12)	0.7226 (2)	0.0942 (15)	0.707 (5)
H1	0.3780	0.0781	0.6703	0.113*	0.707 (5)
C2	0.3409 (8)	0.00737 (12)	0.7428 (2)	0.1211 (18)	0.707 (5)
H2	0.2029	−0.0112	0.7043	0.145*	0.707 (5)
C3	0.4380 (8)	−0.01864 (12)	0.8192 (2)	0.1057 (18)	0.707 (5)
H3	0.3664	−0.0550	0.8330	0.127*	0.707 (5)
C4	0.6400 (8)	0.00854 (12)	0.8755 (2)	0.1120 (19)	0.707 (5)
H4	0.7064	−0.0092	0.9278	0.134*	0.707 (5)
C5	0.7449 (8)	0.06172 (12)	0.8554 (2)	0.0925 (16)	0.707 (5)
H5	0.8826	0.0800	0.8944	0.111*	0.707 (5)
C6	0.6509 (17)	0.0889 (3)	0.7786 (3)	0.0622 (16)	0.707 (5)
C1'	0.455 (3)	0.0595 (6)	0.7755 (10)	0.100 (3)	0.293 (5)
H1'	0.3208	0.0796	0.7374	0.120*	0.293 (5)
C2'	0.393 (4)	0.0054 (7)	0.8006 (11)	0.114 (3)	0.293 (5)
H2'	0.2299	−0.0147	0.7760	0.136*	0.293 (5)
C3'	0.578 (4)	−0.0181 (7)	0.8626 (10)	0.099 (3)	0.293 (5)
H3'	0.5275	−0.0533	0.8866	0.119*	0.293 (5)
C4'	0.849 (3)	0.0078 (6)	0.8945 (9)	0.101 (3)	0.293 (5)
H4'	0.9744	−0.0099	0.9376	0.122*	0.293 (5)
C5'	0.915 (3)	0.0591 (5)	0.8590 (9)	0.087 (3)	0.293 (5)
H5'	1.0965	0.0762	0.8713	0.104*	0.293 (5)
C6'	0.701 (5)	0.0847 (10)	0.8037 (13)	0.076 (3)	0.293 (5)
C7	0.7516 (6)	0.14574 (11)	0.75980 (16)	0.0573 (7)	
H7A	0.9588	0.1486	0.7759	0.069*	
H7B	0.7111	0.1540	0.6985	0.069*	
O1	0.6024 (3)	0.18581 (7)	0.80883 (10)	0.0522 (5)	
C8	0.6390 (5)	0.24272 (10)	0.79634 (14)	0.0462 (6)	
C9	0.8096 (5)	0.26595 (11)	0.73906 (15)	0.0518 (6)	
H9	0.9094	0.2414	0.7050	0.062*	
C10	0.8337 (5)	0.32442 (11)	0.73166 (16)	0.0558 (7)	
H10	0.9518	0.3398	0.6928	0.067*	
C11	0.6885 (5)	0.36092 (11)	0.77994 (17)	0.0581 (7)	
H11	0.7061	0.4011	0.7740	0.070*	
C12	0.5167 (5)	0.33860 (10)	0.83709 (16)	0.0533 (6)	
H12	0.4164	0.3638	0.8701	0.064*	
C13	0.4896 (5)	0.27935 (10)	0.84665 (14)	0.0446 (6)	
C14	0.3100 (5)	0.25606 (10)	0.90667 (14)	0.0437 (6)	
C15	0.2494 (5)	0.20272 (10)	0.93483 (14)	0.0461 (6)	
H15	0.3247	0.1676	0.9174	0.055*	
C16	0.0522 (5)	0.20976 (10)	0.99524 (14)	0.0461 (6)	
C17	0.0044 (5)	0.26698 (10)	1.00093 (14)	0.0463 (6)	
O2	0.1604 (3)	0.29619 (6)	0.94823 (10)	0.0472 (5)	

C18	−0.1737 (5)	0.29796 (11)	1.05094 (16)	0.0532 (7)
N1	−0.3228 (5)	0.32090 (11)	1.09242 (14)	0.0670 (7)
C19	−0.0860 (5)	0.16674 (11)	1.03907 (15)	0.0543 (7)
N2	−0.2031 (5)	0.13183 (10)	1.07249 (15)	0.0711 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.141 (4)	0.059 (2)	0.083 (3)	−0.025 (2)	0.013 (3)	0.010 (2)
C2	0.173 (4)	0.080 (3)	0.111 (4)	−0.029 (3)	0.018 (4)	0.016 (3)
C3	0.146 (4)	0.066 (3)	0.112 (4)	0.008 (3)	0.047 (3)	0.032 (3)
C4	0.141 (4)	0.094 (3)	0.106 (3)	0.010 (4)	0.031 (3)	0.027 (3)
C5	0.112 (4)	0.084 (3)	0.085 (3)	0.003 (3)	0.024 (3)	0.023 (2)
C6	0.083 (3)	0.051 (2)	0.058 (3)	0.006 (2)	0.034 (3)	0.004 (2)
C1'	0.120 (4)	0.075 (4)	0.105 (5)	−0.012 (4)	0.017 (4)	0.005 (4)
C2'	0.142 (4)	0.088 (4)	0.113 (4)	−0.016 (4)	0.019 (4)	0.016 (4)
C3'	0.114 (5)	0.077 (5)	0.109 (5)	−0.016 (5)	0.029 (4)	0.005 (5)
C4'	0.117 (5)	0.085 (4)	0.105 (5)	−0.015 (5)	0.025 (5)	0.016 (4)
C5'	0.096 (6)	0.074 (5)	0.095 (5)	−0.023 (5)	0.028 (5)	0.012 (4)
C6'	0.087 (5)	0.061 (5)	0.085 (6)	−0.007 (4)	0.033 (5)	0.004 (5)
C7	0.0577 (15)	0.0615 (15)	0.0558 (15)	0.0017 (12)	0.0205 (12)	−0.0066 (11)
O1	0.0545 (10)	0.0513 (10)	0.0540 (10)	−0.0025 (7)	0.0206 (8)	−0.0039 (7)
C8	0.0394 (13)	0.0554 (14)	0.0438 (13)	−0.0050 (10)	0.0040 (10)	0.0035 (9)
C9	0.0446 (14)	0.0690 (16)	0.0428 (13)	−0.0033 (11)	0.0095 (10)	0.0038 (11)
C10	0.0490 (14)	0.0686 (16)	0.0508 (14)	−0.0059 (12)	0.0109 (11)	0.0121 (12)
C11	0.0563 (16)	0.0558 (15)	0.0629 (16)	−0.0078 (12)	0.0103 (12)	0.0131 (11)
C12	0.0455 (14)	0.0577 (15)	0.0575 (15)	−0.0001 (11)	0.0102 (11)	0.0053 (11)
C13	0.0380 (12)	0.0536 (13)	0.0424 (12)	−0.0007 (10)	0.0051 (10)	0.0045 (10)
C14	0.0360 (12)	0.0538 (13)	0.0414 (12)	0.0012 (9)	0.0051 (9)	−0.0008 (9)
C15	0.0424 (13)	0.0537 (13)	0.0427 (13)	0.0014 (10)	0.0068 (10)	0.0023 (9)
C16	0.0434 (13)	0.0566 (14)	0.0390 (12)	−0.0008 (10)	0.0086 (10)	0.0046 (9)
C17	0.0408 (12)	0.0597 (15)	0.0395 (12)	−0.0014 (10)	0.0097 (10)	0.0010 (10)
O2	0.0416 (9)	0.0553 (10)	0.0456 (9)	−0.0019 (7)	0.0093 (7)	−0.0007 (7)
C18	0.0472 (15)	0.0650 (16)	0.0483 (14)	−0.0019 (11)	0.0089 (11)	−0.0017 (11)
N1	0.0649 (15)	0.0791 (16)	0.0602 (14)	−0.0001 (12)	0.0212 (12)	−0.0068 (11)
C19	0.0541 (15)	0.0634 (16)	0.0472 (14)	0.0005 (12)	0.0131 (11)	0.0064 (11)
N2	0.0807 (17)	0.0724 (15)	0.0639 (15)	−0.0039 (12)	0.0251 (12)	0.0122 (12)

Geometric parameters (Å, °)

C1—C2	1.3900	C7—H7A	0.9900
C1—C6	1.408 (8)	C7—H7B	0.9900
C1—H1	0.9500	O1—C8	1.360 (3)
C2—C3	1.3900	C8—C9	1.393 (3)
C2—H2	0.9500	C8—C13	1.415 (3)
C3—C4	1.3900	C9—C10	1.378 (4)

C3—H3	0.9500	C9—H9	0.9500
C4—C5	1.3900	C10—C11	1.382 (4)
C4—H4	0.9500	C10—H10	0.9500
C5—C6	1.405 (6)	C11—C12	1.388 (3)
C5—H5	0.9500	C11—H11	0.9500
C6—C7	1.455 (8)	C12—C13	1.401 (3)
C1'—C6'	1.34 (3)	C12—H12	0.9500
C1'—C2'	1.37 (2)	C13—C14	1.454 (3)
C1'—H1'	0.9500	C14—C15	1.367 (3)
C2'—C3'	1.36 (2)	C14—O2	1.387 (3)
C2'—H2'	0.9500	C15—C16	1.423 (3)
C3'—C4'	1.46 (2)	C15—H15	0.9500
C3'—H3'	0.9500	C16—C17	1.362 (3)
C4'—C5'	1.375 (17)	C16—C19	1.424 (3)
C4'—H4'	0.9500	C17—O2	1.361 (3)
C5'—C6'	1.40 (2)	C17—C18	1.421 (3)
C5'—H5'	0.9500	C18—N1	1.152 (3)
C6'—C7	1.62 (2)	C19—N2	1.150 (3)
C7—O1	1.452 (3)		
C2—C1—C6	121.1 (3)	C6'—C7—H7A	98.0
C2—C1—H1	119.5	C6—C7—H7A	110.4
C6—C1—H1	119.5	O1—C7—H7B	110.4
C1—C2—C3	120.0	C6'—C7—H7B	125.1
C1—C2—H2	120.0	C6—C7—H7B	110.4
C3—C2—H2	120.0	H7A—C7—H7B	108.6
C4—C3—C2	120.0	C8—O1—C7	118.39 (18)
C4—C3—H3	120.0	O1—C8—C9	124.7 (2)
C2—C3—H3	120.0	O1—C8—C13	115.47 (19)
C3—C4—C5	120.0	C9—C8—C13	119.8 (2)
C3—C4—H4	120.0	C10—C9—C8	120.1 (2)
C5—C4—H4	120.0	C10—C9—H9	119.9
C4—C5—C6	121.2 (4)	C8—C9—H9	119.9
C4—C5—H5	119.4	C11—C10—C9	120.9 (2)
C6—C5—H5	119.4	C11—C10—H10	119.5
C5—C6—C1	117.7 (6)	C9—C10—H10	119.5
C5—C6—C7	120.8 (5)	C10—C11—C12	119.8 (2)
C1—C6—C7	121.4 (4)	C10—C11—H11	120.1
C6'—C1'—C2'	121.0 (16)	C12—C11—H11	120.1
C6'—C1'—H1'	119.5	C11—C12—C13	120.7 (2)
C2'—C1'—H1'	119.5	C11—C12—H12	119.7
C3'—C2'—C1'	116.3 (16)	C13—C12—H12	119.7
C3'—C2'—H2'	121.9	C12—C13—C8	118.7 (2)
C1'—C2'—H2'	121.9	C12—C13—C14	120.6 (2)
C2'—C3'—C4'	123.8 (15)	C8—C13—C14	120.7 (2)

C2'—C3'—H3'	118.1	C15—C14—O2	108.91 (19)
C4'—C3'—H3'	118.1	C15—C14—C13	135.8 (2)
C5'—C4'—C3'	116.7 (13)	O2—C14—C13	115.29 (19)
C5'—C4'—H4'	121.6	C14—C15—C16	107.1 (2)
C3'—C4'—H4'	121.6	C14—C15—H15	126.5
C4'—C5'—C6'	116.6 (15)	C16—C15—H15	126.5
C4'—C5'—H5'	121.7	C17—C16—C15	106.6 (2)
C6'—C5'—H5'	121.7	C17—C16—C19	124.9 (2)
C5'—C6'—C1'	124.6 (19)	C15—C16—C19	128.4 (2)
C5'—C6'—C7	120.9 (16)	O2—C17—C16	110.26 (19)
C1'—C6'—C7	114.1 (15)	O2—C17—C18	119.1 (2)
O1—C7—C6'	103.5 (8)	C16—C17—C18	130.7 (2)
O1—C7—C6	106.8 (3)	C17—O2—C14	107.18 (17)
C6'—C7—C6	15.8 (9)	N1—C18—C17	177.1 (3)
O1—C7—H7A	110.4	N2—C19—C16	178.1 (3)
C6—C1—C2—C3	0.1 (4)	C8—C9—C10—C11	−0.6 (3)
C1—C2—C3—C4	0.0	C9—C10—C11—C12	0.3 (4)
C2—C3—C4—C5	0.0	C10—C11—C12—C13	0.3 (4)
C3—C4—C5—C6	−0.1 (4)	C11—C12—C13—C8	−0.5 (3)
C4—C5—C6—C1	0.2 (7)	C11—C12—C13—C14	179.9 (2)
C4—C5—C6—C7	177.0 (3)	O1—C8—C13—C12	−179.9 (2)
C2—C1—C6—C5	−0.2 (7)	C9—C8—C13—C12	0.3 (3)
C2—C1—C6—C7	−177.0 (4)	O1—C8—C13—C14	−0.3 (3)
C6'—C1'—C2'—C3'	8(3)	C9—C8—C13—C14	179.9 (2)
C1'—C2'—C3'—C4'	−8(3)	C12—C13—C14—C15	−175.5 (2)
C2'—C3'—C4'—C5'	1(2)	C8—C13—C14—C15	4.9 (4)
C3'—C4'—C5'—C6'	8(2)	C12—C13—C14—O2	3.6 (3)
C4'—C5'—C6'—C1'	−9(3)	C8—C13—C14—O2	−175.98 (18)
C4'—C5'—C6'—C7	178.6 (13)	O2—C14—C15—C16	0.8 (2)
C2'—C1'—C6'—C5'	1(3)	C13—C14—C15—C16	179.9 (2)
C2'—C1'—C6'—C7	173.8 (14)	C14—C15—C16—C17	−0.3 (2)
C5'—C6'—C7—O1	−102.7 (17)	C14—C15—C16—C19	176.4 (2)
C1'—C6'—C7—O1	84.1 (16)	C15—C16—C17—O2	−0.3 (2)
C5'—C6'—C7—C6	153 (5)	C19—C16—C17—O2	−177.2 (2)
C1'—C6'—C7—C6	−20 (3)	C15—C16—C17—C18	179.7 (2)
C5—C6—C7—O1	−76.2 (6)	C19—C16—C17—C18	2.8 (4)
C1—C6—C7—O1	100.4 (5)	C16—C17—O2—C14	0.8 (2)
C5—C6—C7—C6'	4(3)	C18—C17—O2—C14	−179.19 (19)
C1—C6—C7—C6'	−179 (4)	C15—C14—O2—C17	−1.0 (2)
C6'—C7—O1—C8	170.2 (8)	C13—C14—O2—C17	179.69 (18)
C6—C7—O1—C8	−173.8 (3)	O2—C17—C18—N1	171 (5)
C7—O1—C8—C9	0.4 (3)	C16—C17—C18—N1	−9(6)
C7—O1—C8—C13	−179.5 (2)	C17—C16—C19—N2	94 (8)
O1—C8—C9—C10	−179.6 (2)	C15—C16—C19—N2	−82 (8)

C13—C8—C9—C10	0.3 (3)

Hydrogen-bond geometry (Å, °)

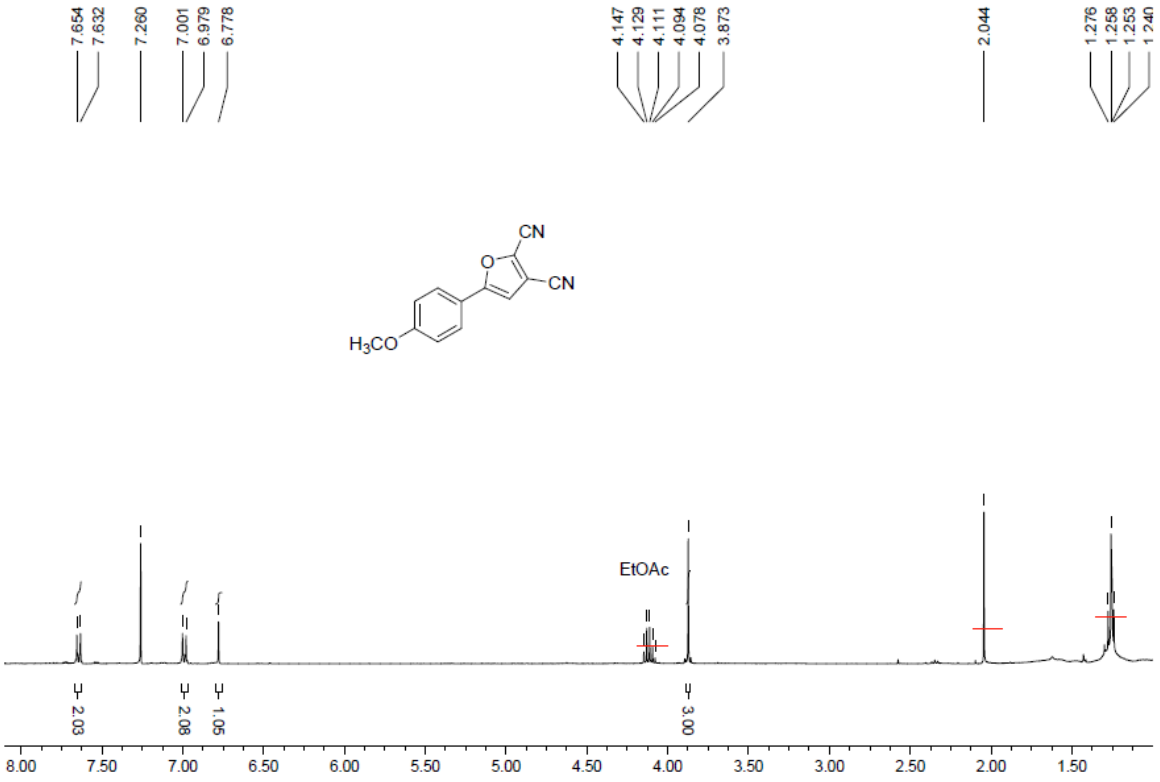
D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···N1^{i}	0.95	2.73	3.674 (4)	174
C10—H10···N2^{i}	0.95	2.73	3.676 (4)	172

Symmetry codes: (i) *x*+3/2, −*y*+1/2, *z*−1/2.

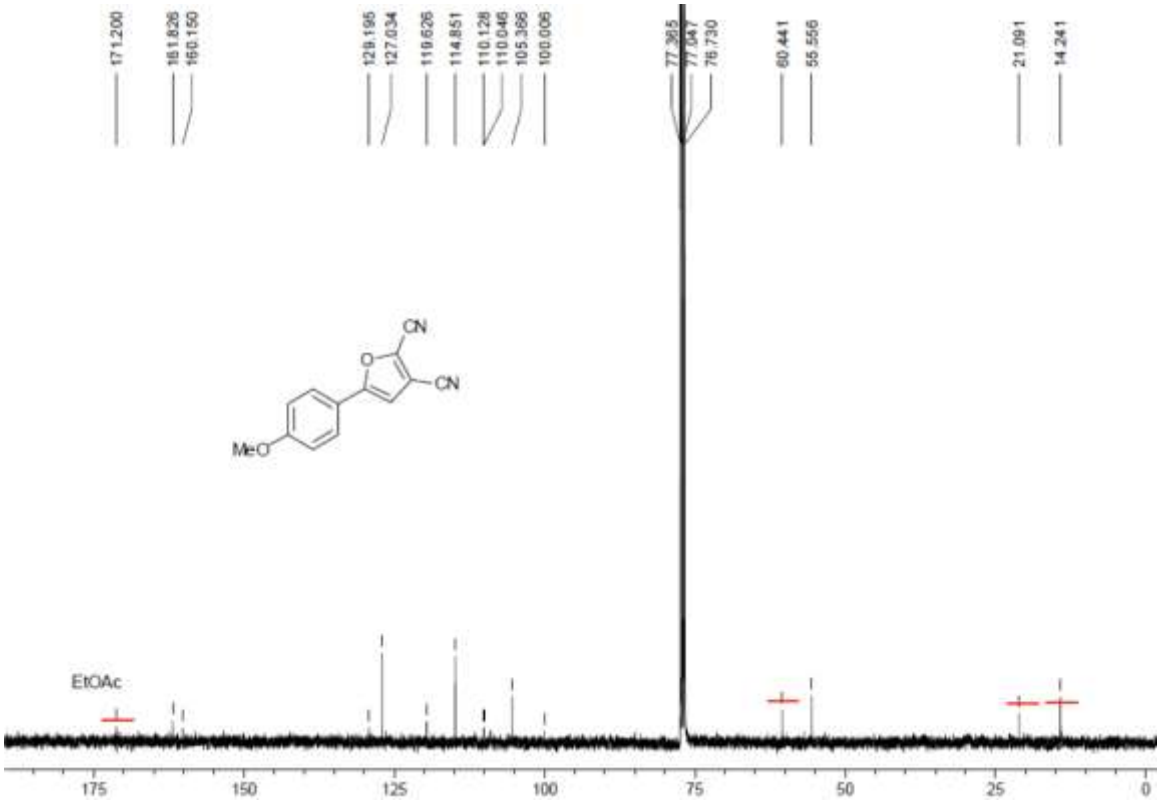
Softwares used for crystallographic data

Data collection: *CrystalClear*SM Expert 2.0 r2 (Rigaku, 2009); cell refinement: *CrystalClear*SM Expert 2.0 r2 (Rigaku, 2009); data reduction: *CrystalClear*SM Expert 2.0 r2 (Rigaku, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*, *enCIFer* (Allen *et al.*, 2004), *PLATON* (Spek, 2009), PublCIF (Westrip 2010).

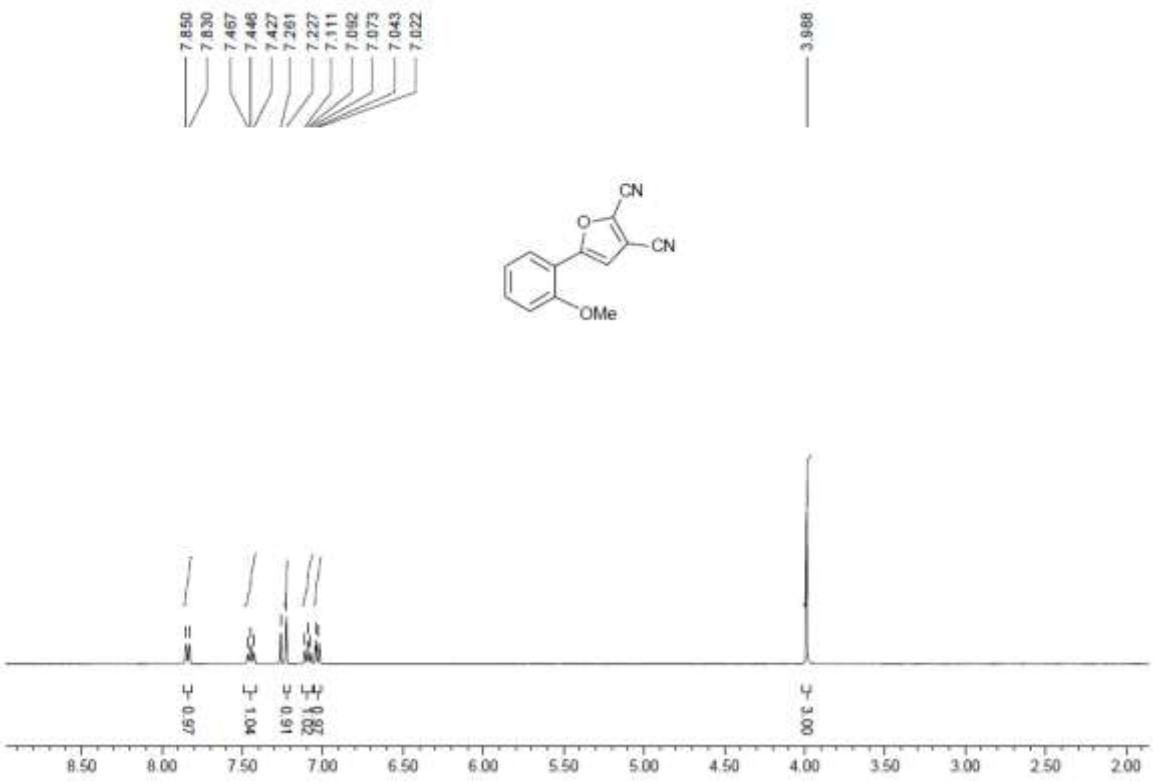
6) ^{1}H and ^{13}C NMR spectra of the synthesized 2,3-dicyanofurans



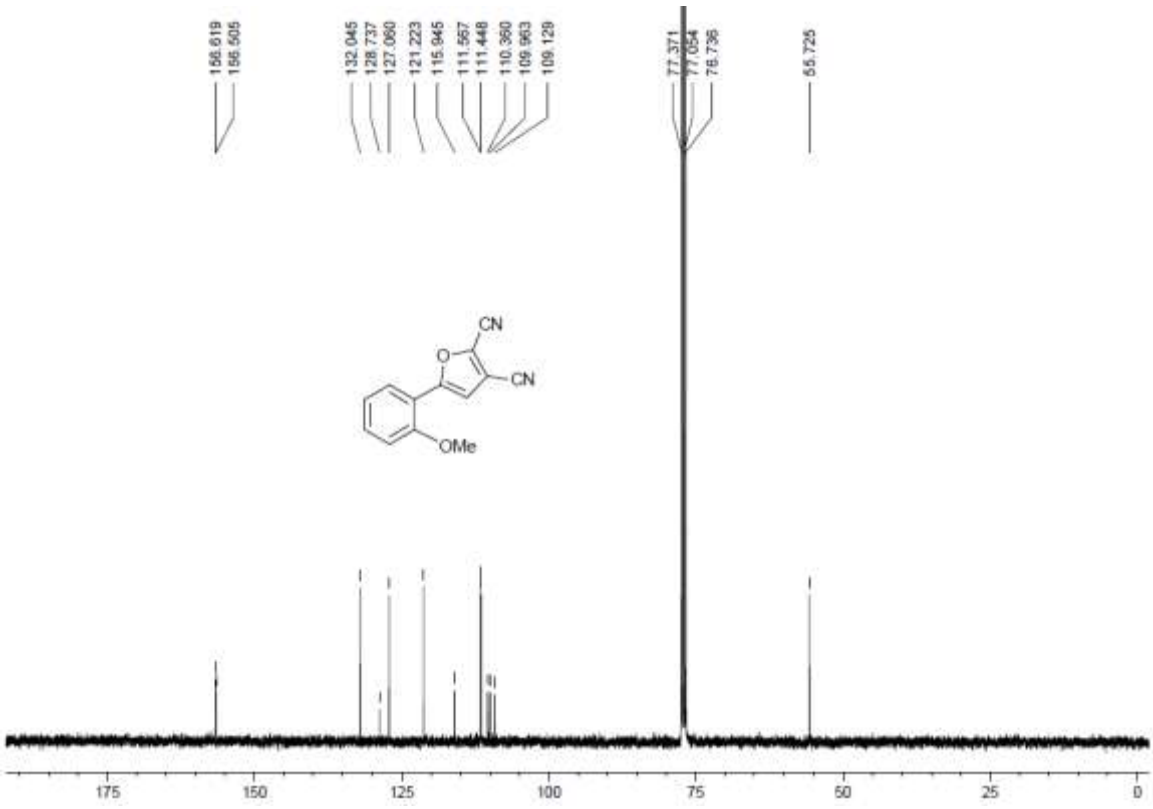
^{1}H NMR spectrum of **2a**



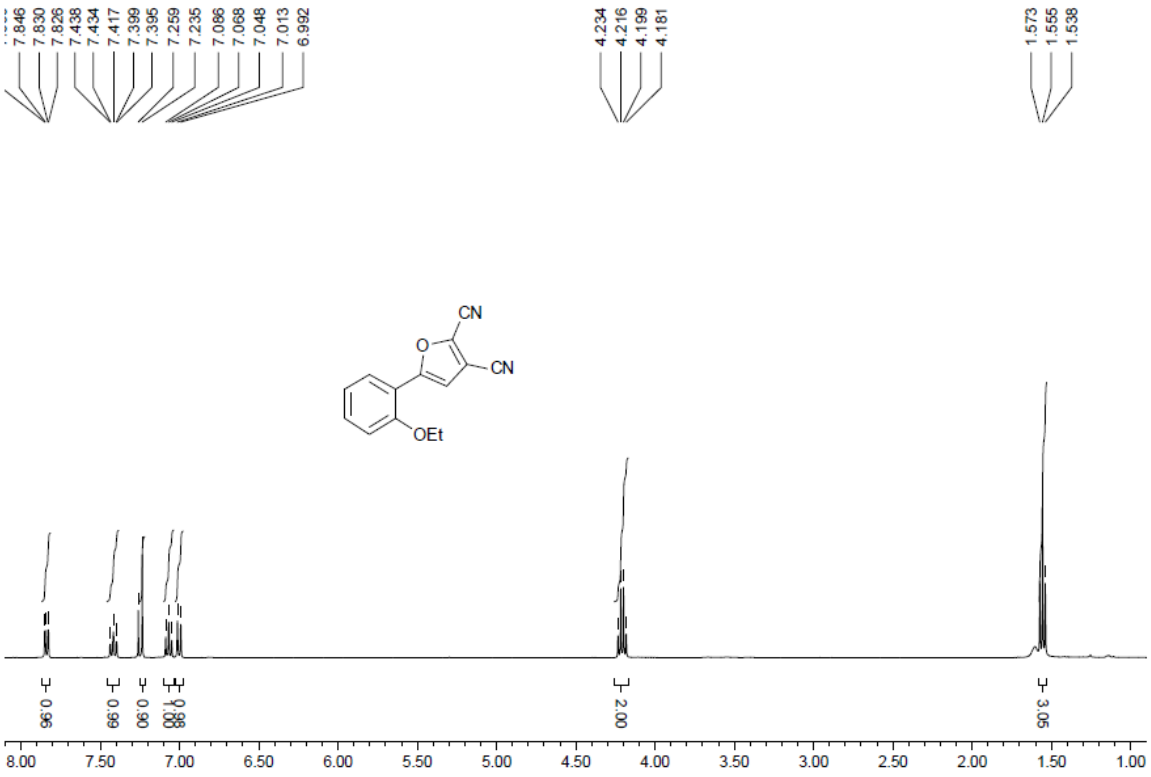
^{13}C NMR spectrum of **2a**



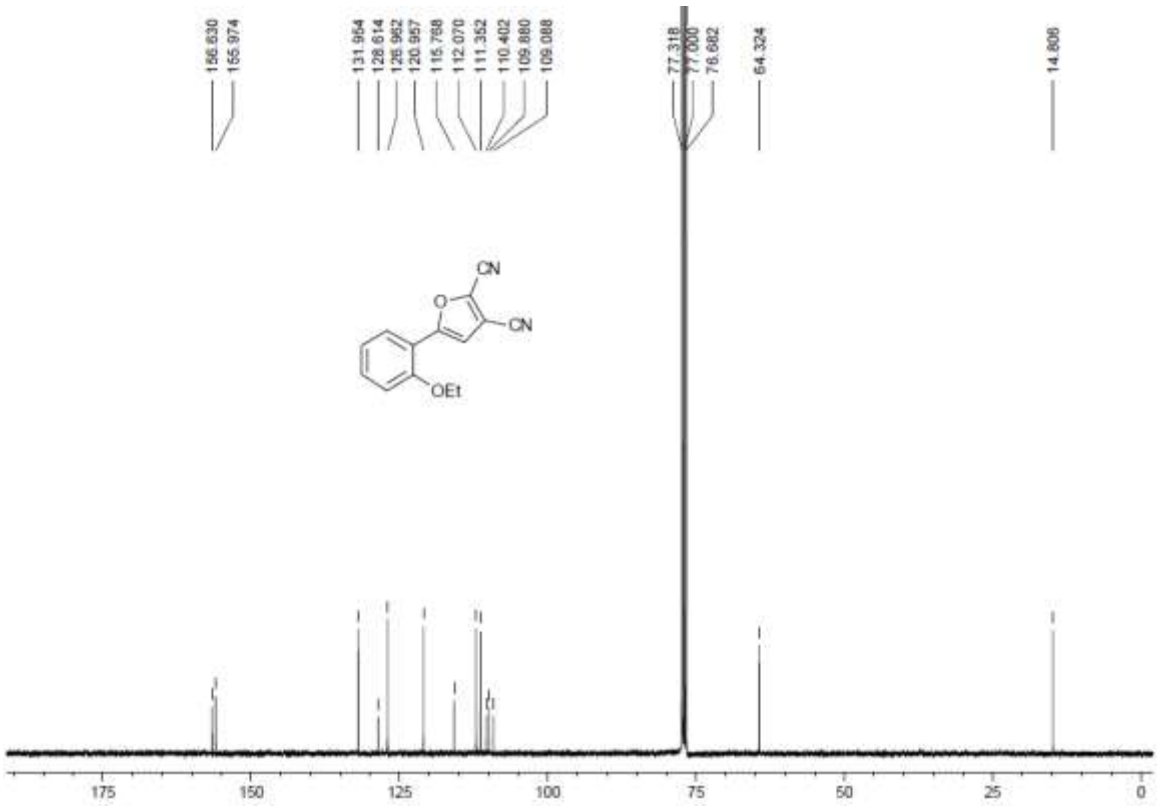
^{1}H NMR spectrum of **2b**



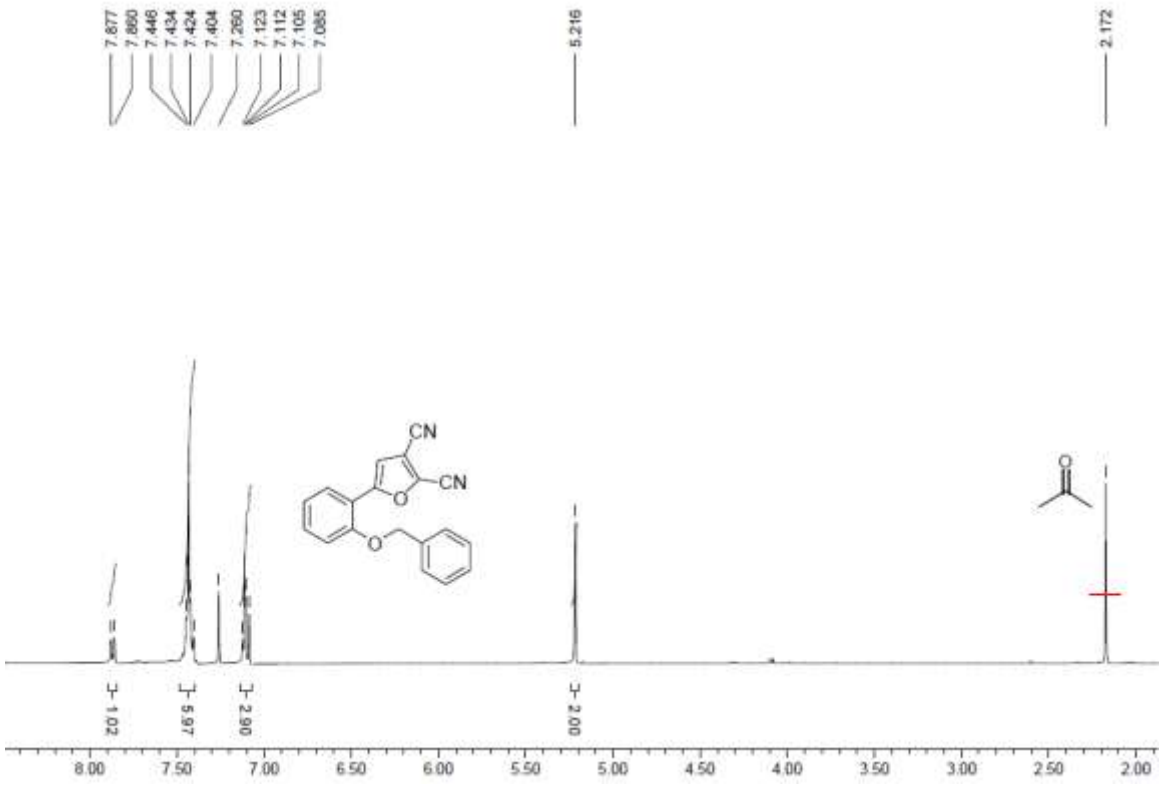
^{13}C NMR spectrum of **2b**



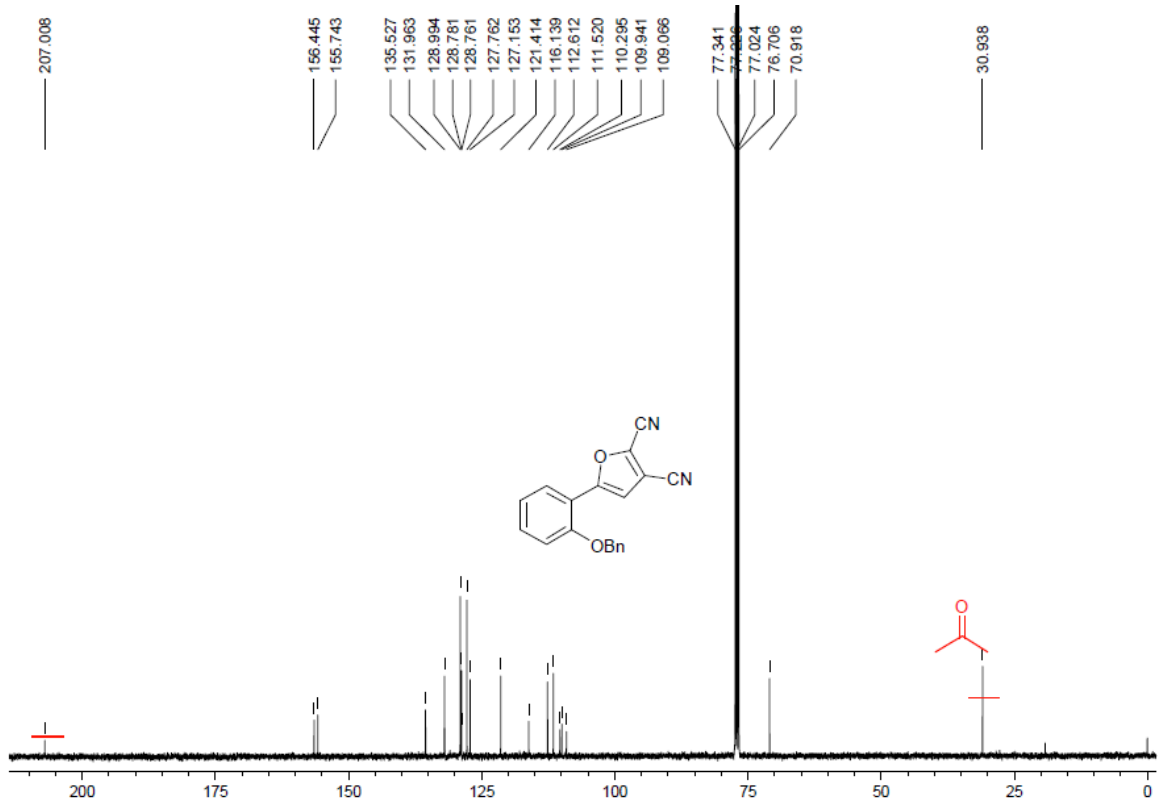
^{1}H NMR spectrum of **2e**



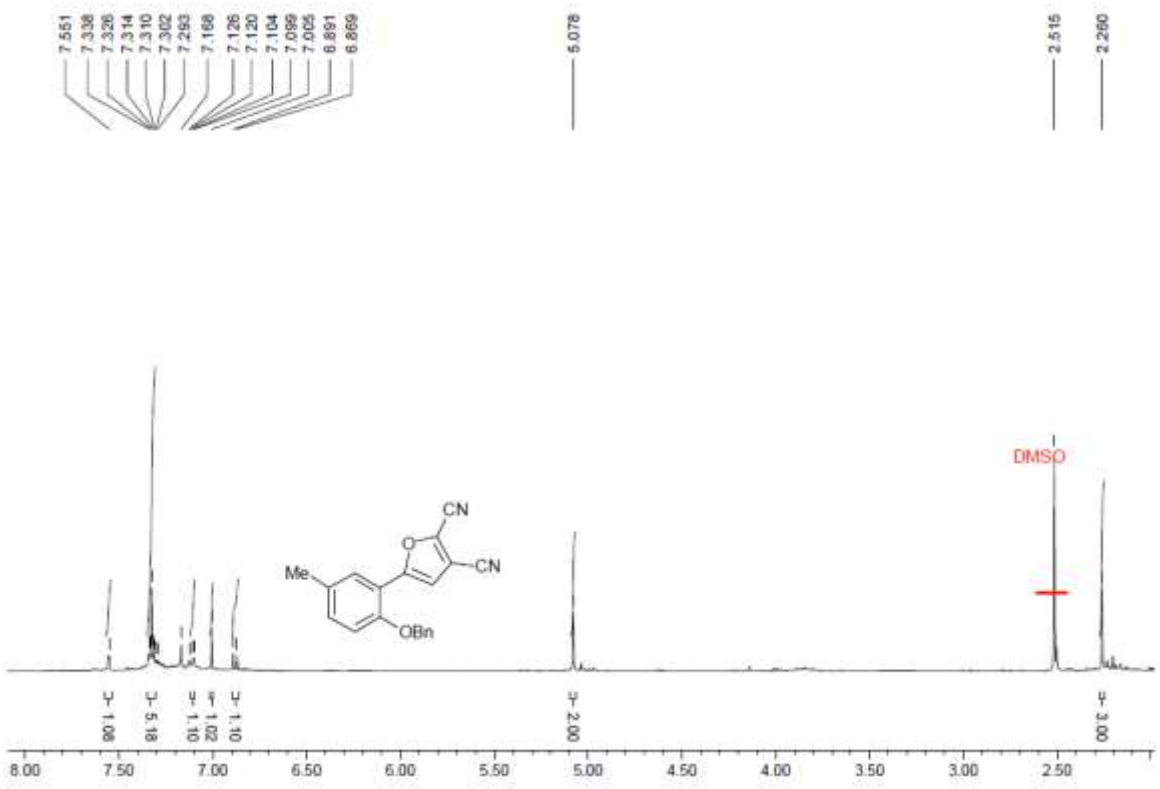
^{13}C NMR spectrum of **2e**



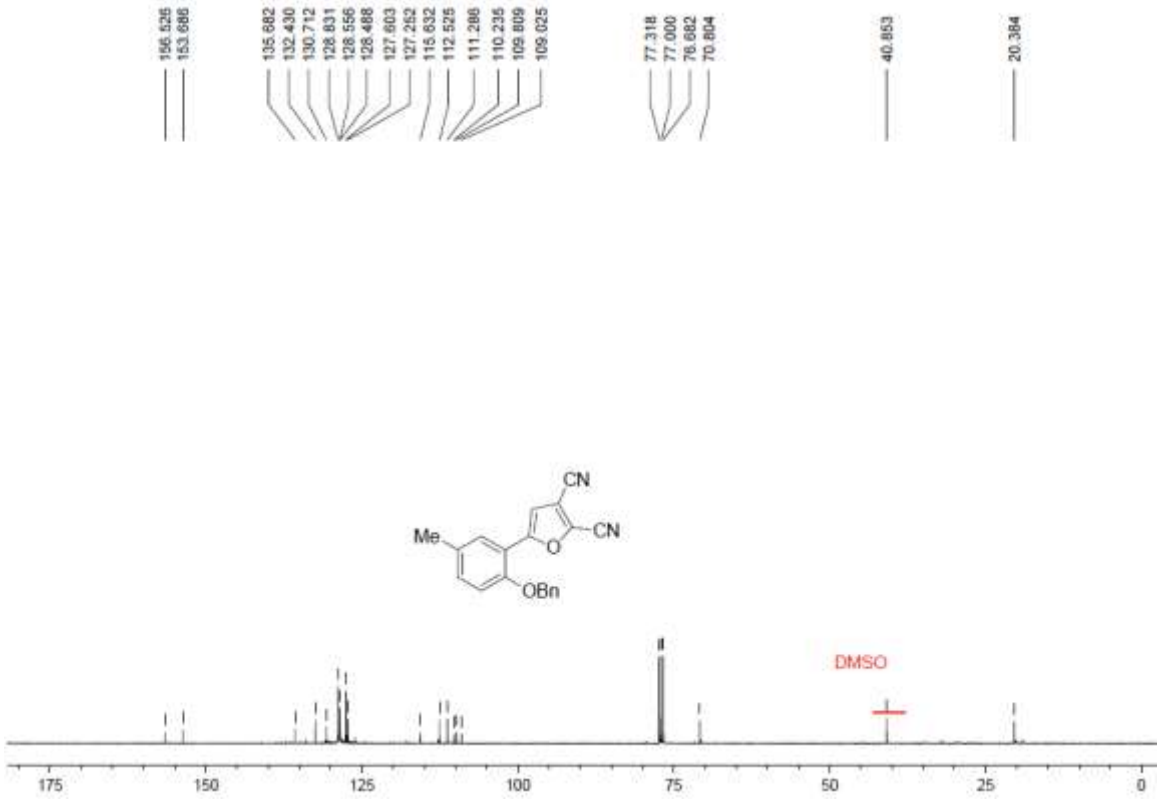
^{1}H NMR spectrum of **2f**



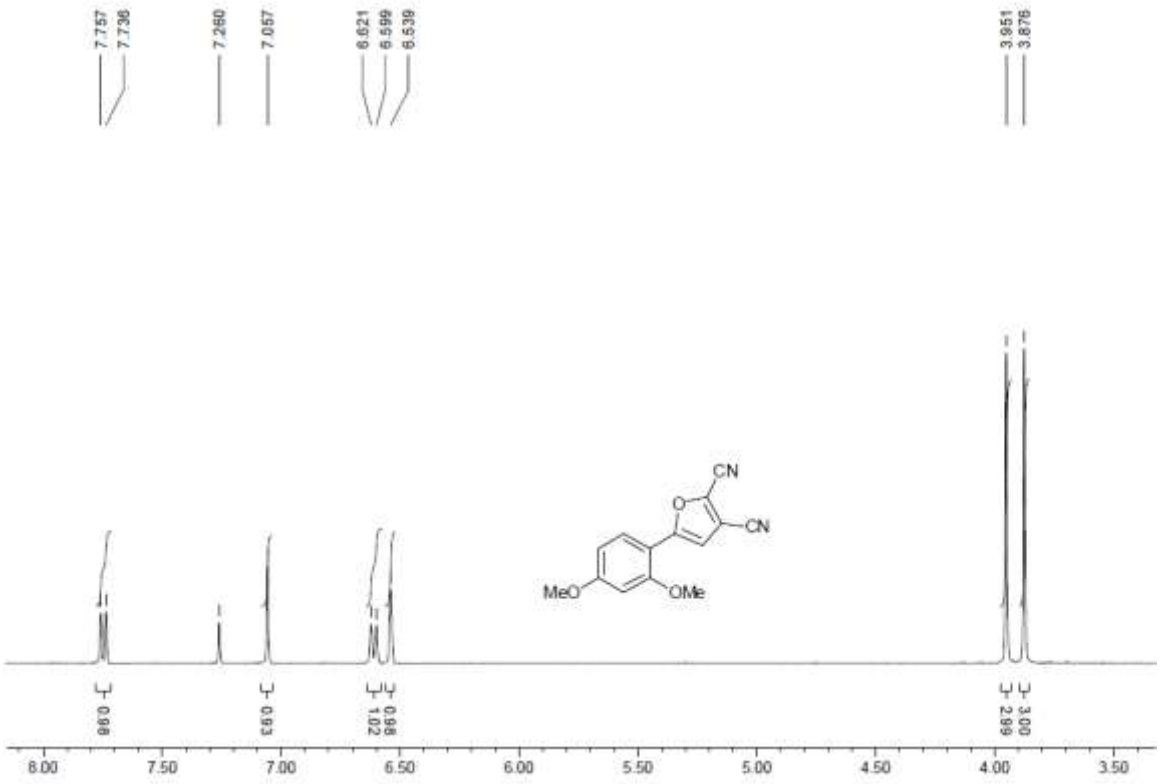
^{13}C NMR spectrum of **2f**



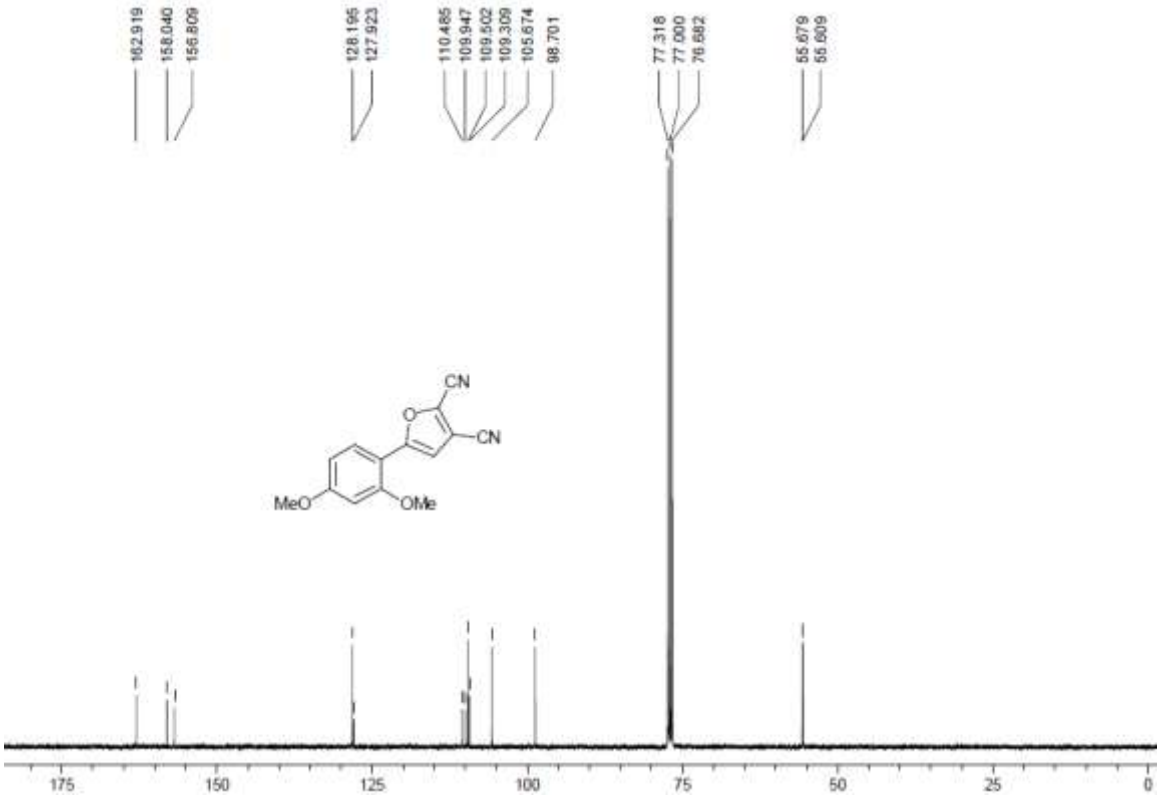
^{1}H NMR spectrum of **2g**



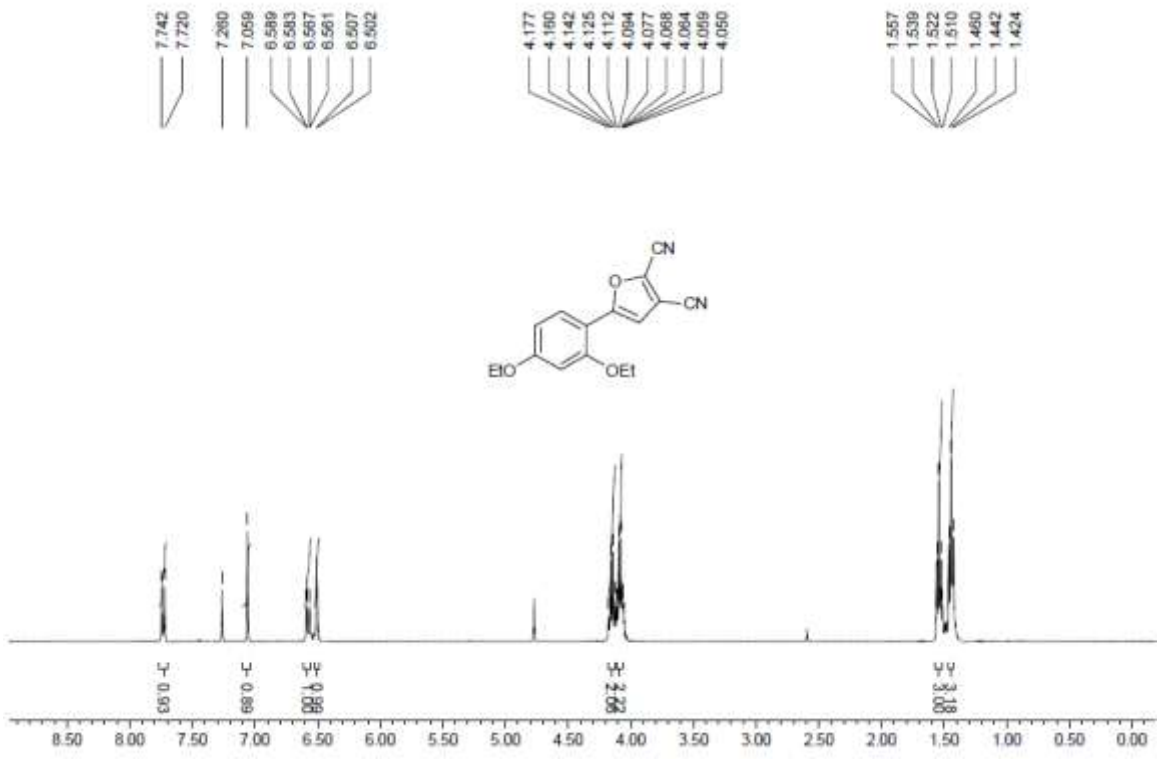
^{13}C NMR spectrum of **2g**



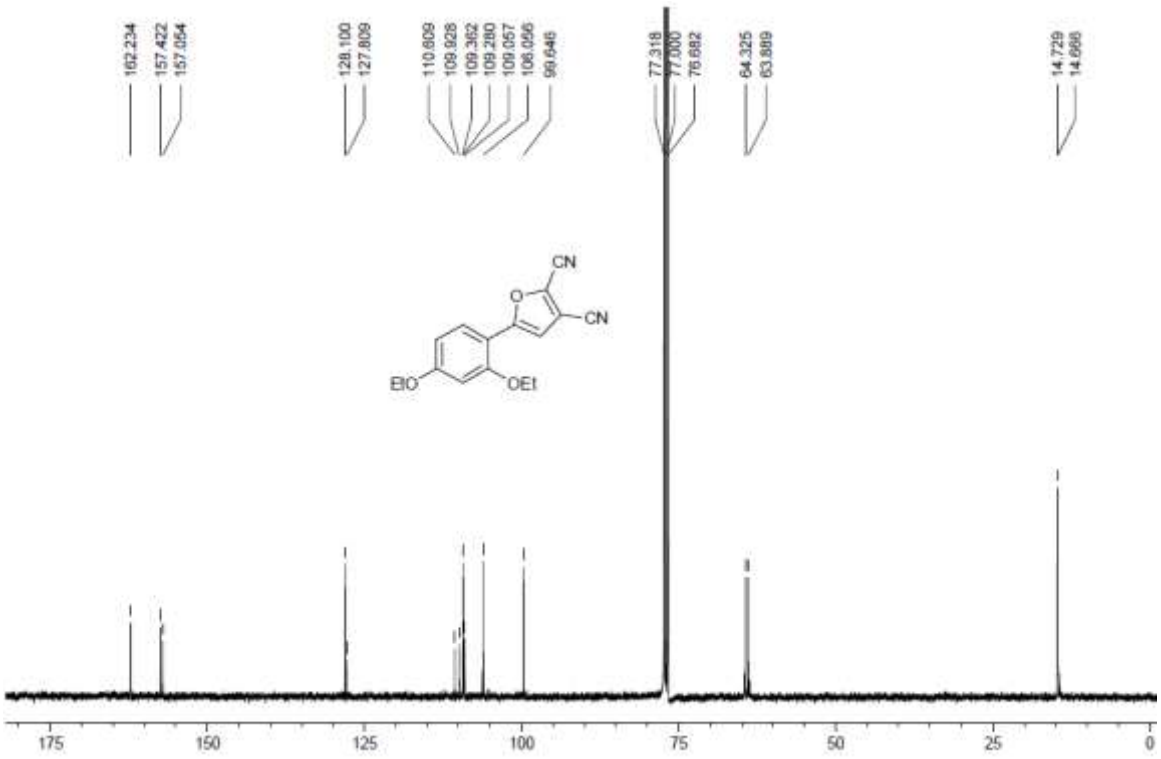
^{1}H NMR spectrum of **2h**



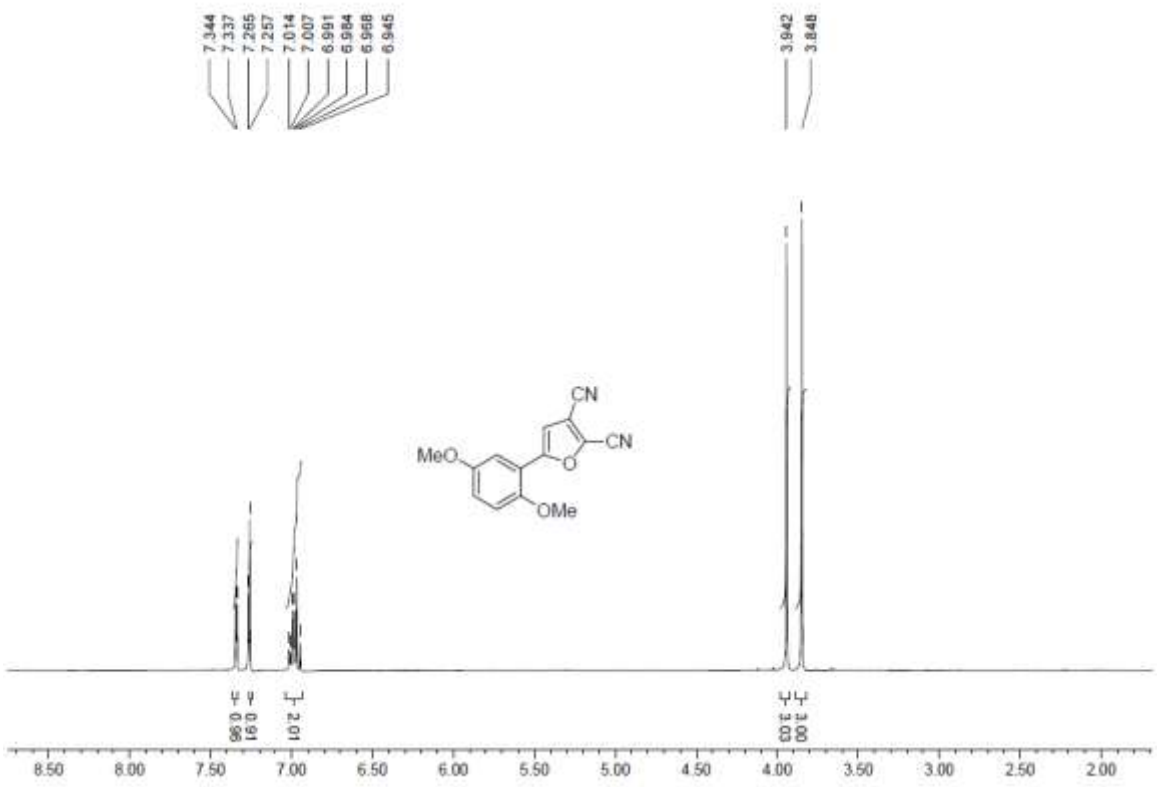
^{13}C NMR spectrum of **2h**



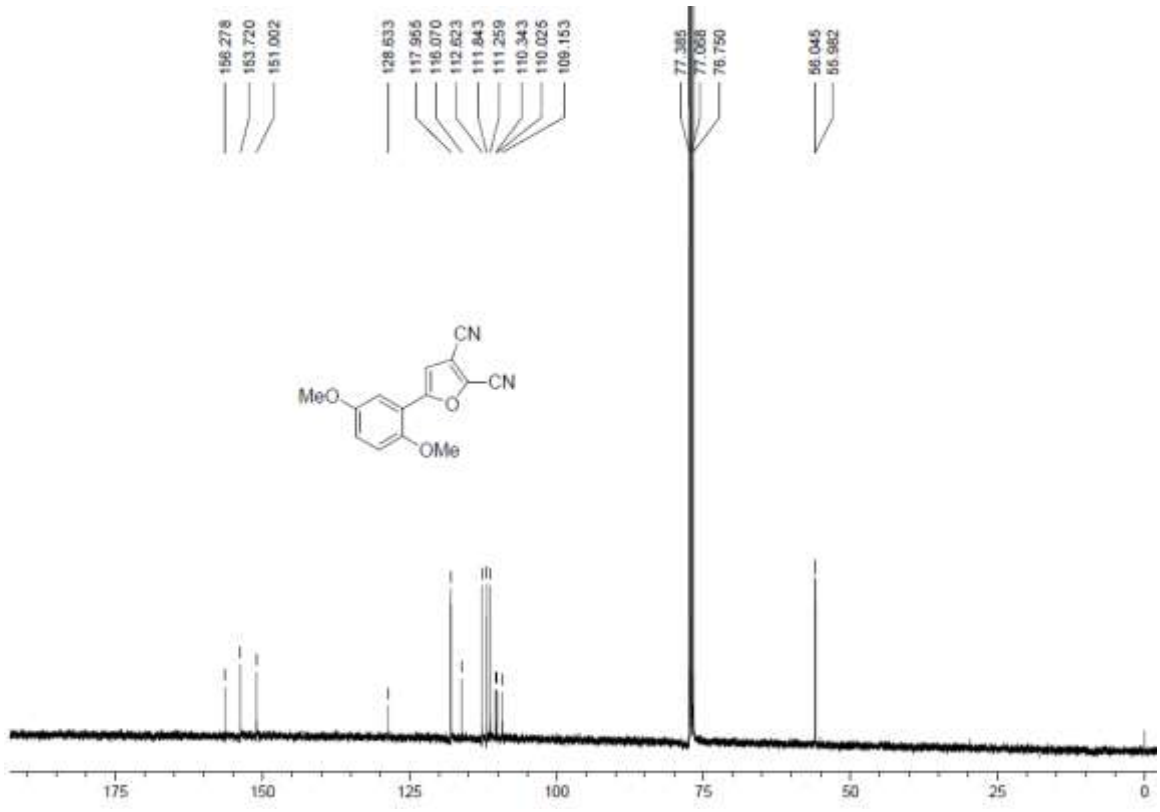
^{1}H NMR spectrum of **2i**



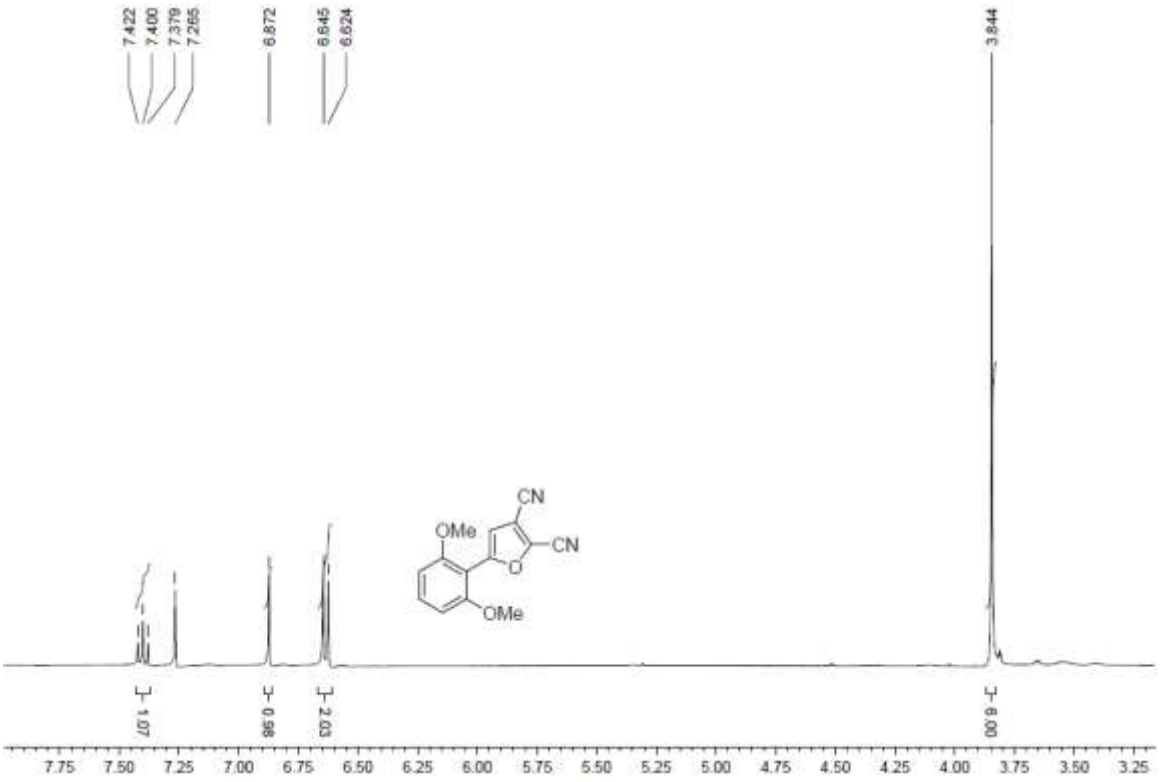
^{13}C NMR spectrum of **2i**



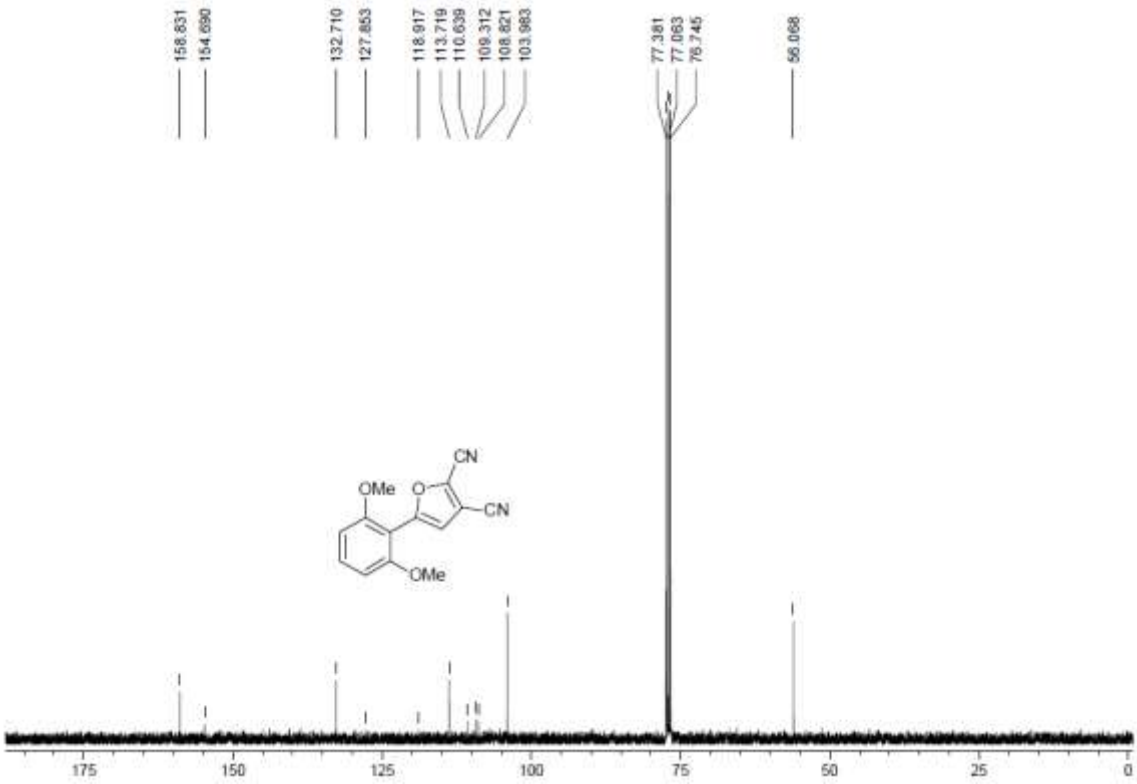
^{1}H NMR spectrum of **2j**



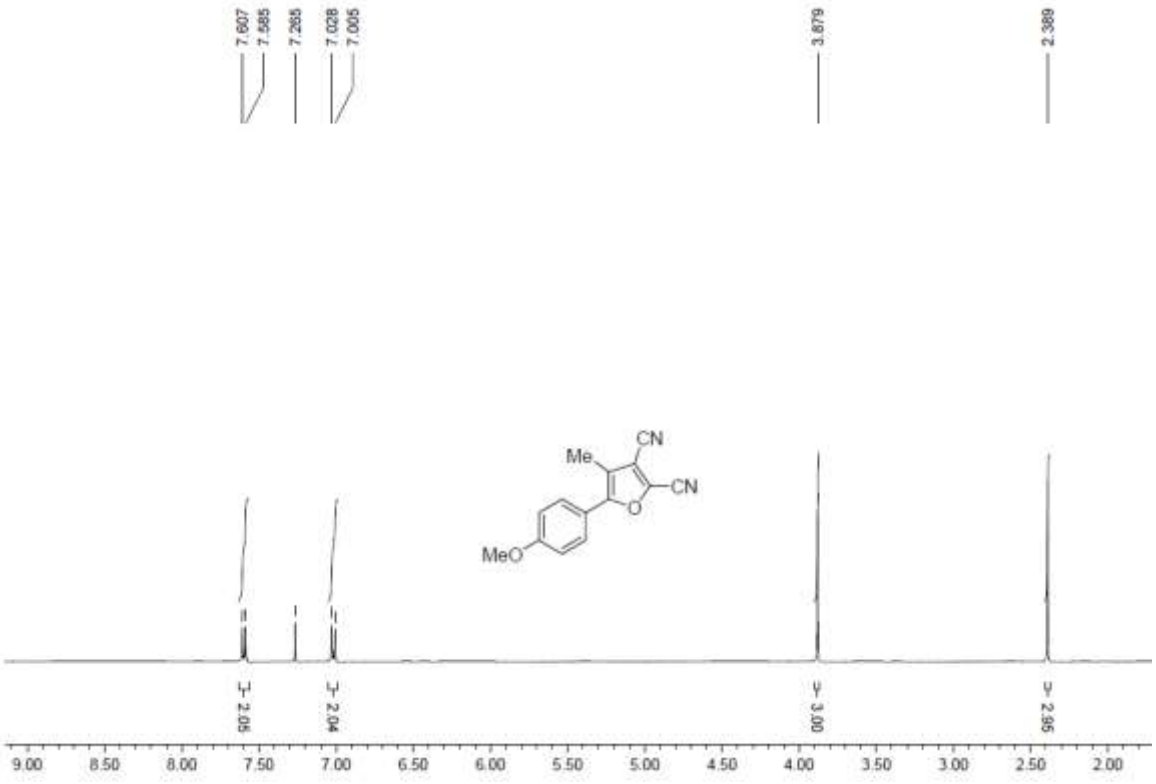
^{13}C NMR spectrum of **2j**



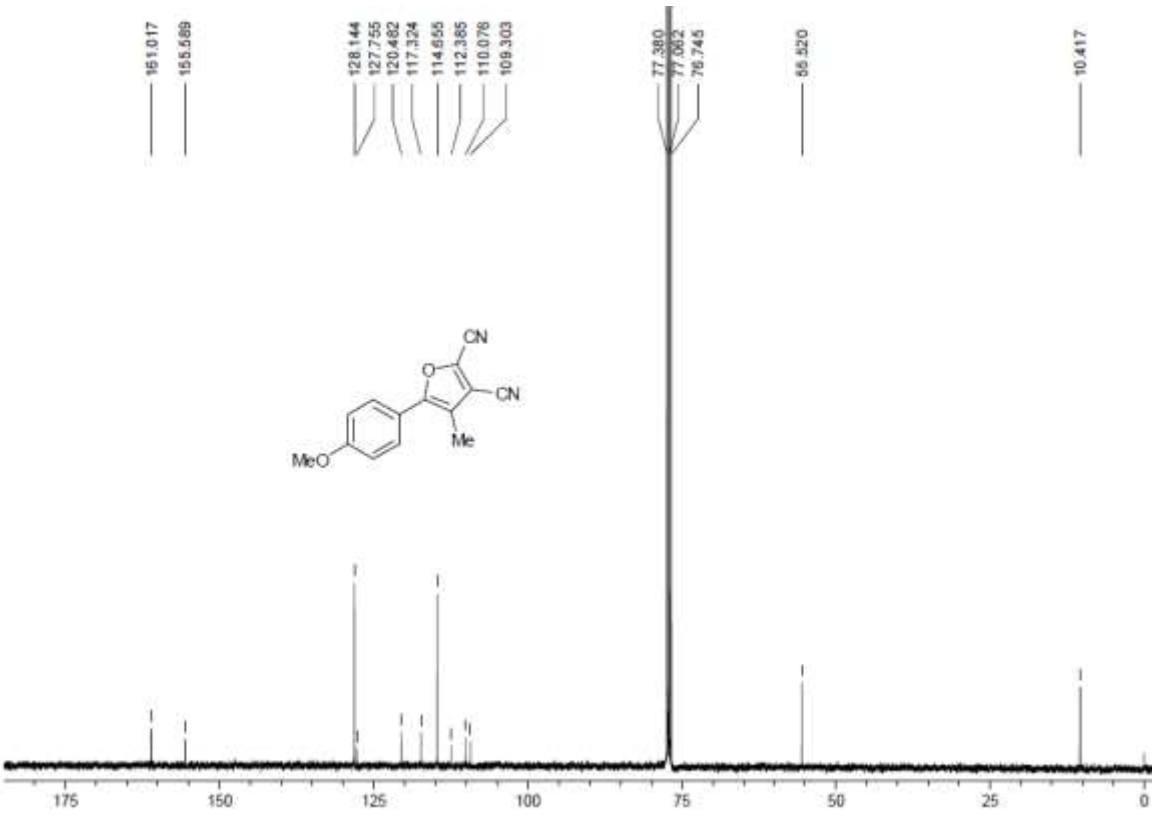
^{1}H NMR spectrum of **2k**



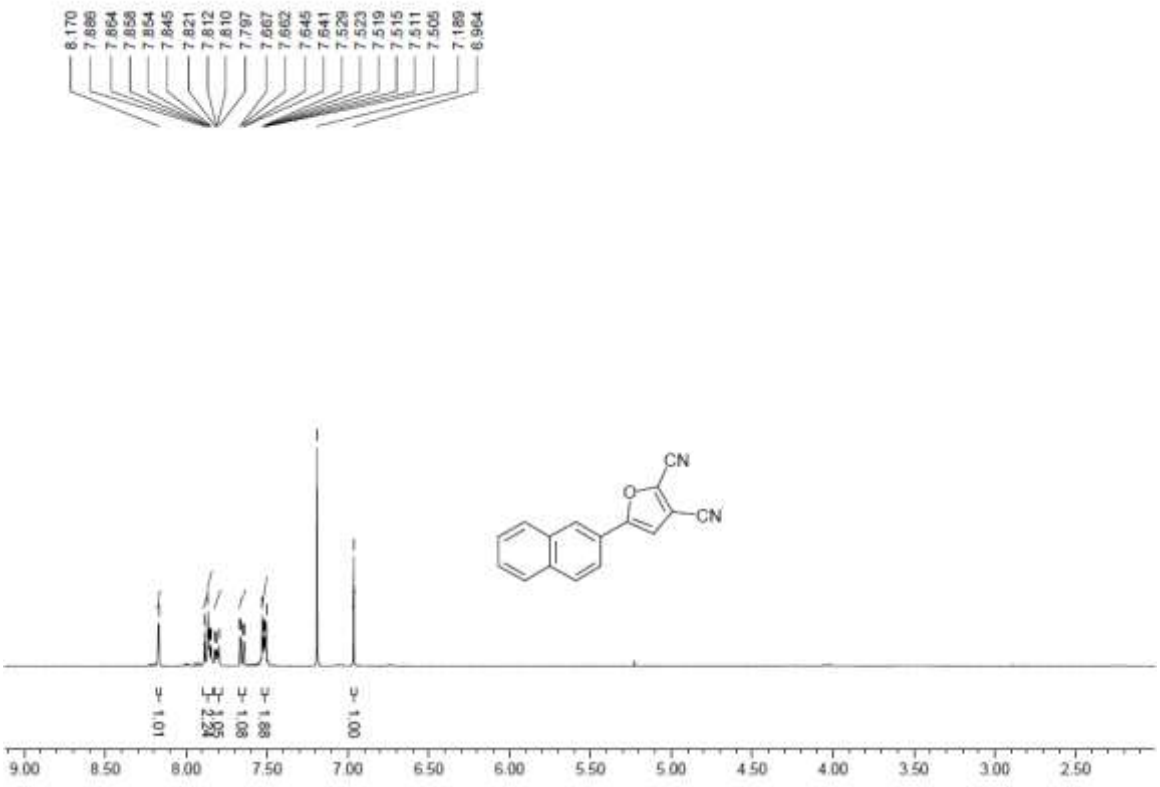
^{13}C NMR spectrum of **2k**



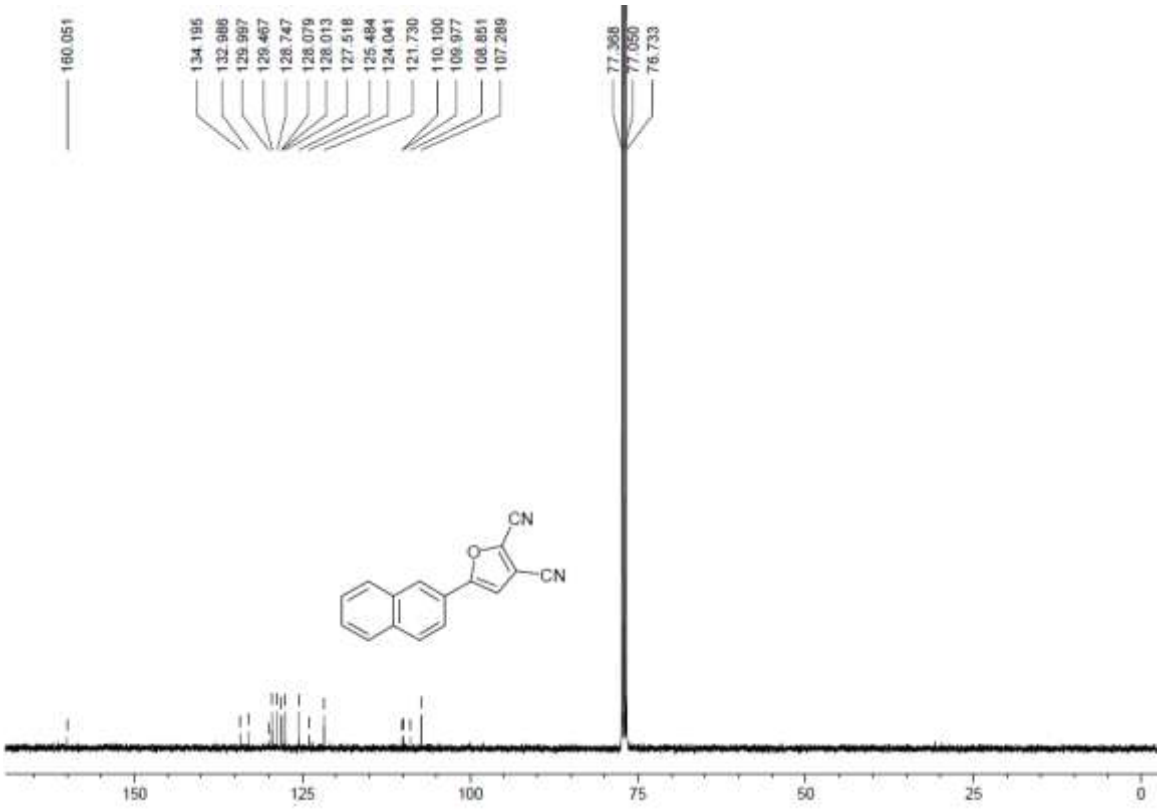
^{1}H NMR spectrum of **2l**



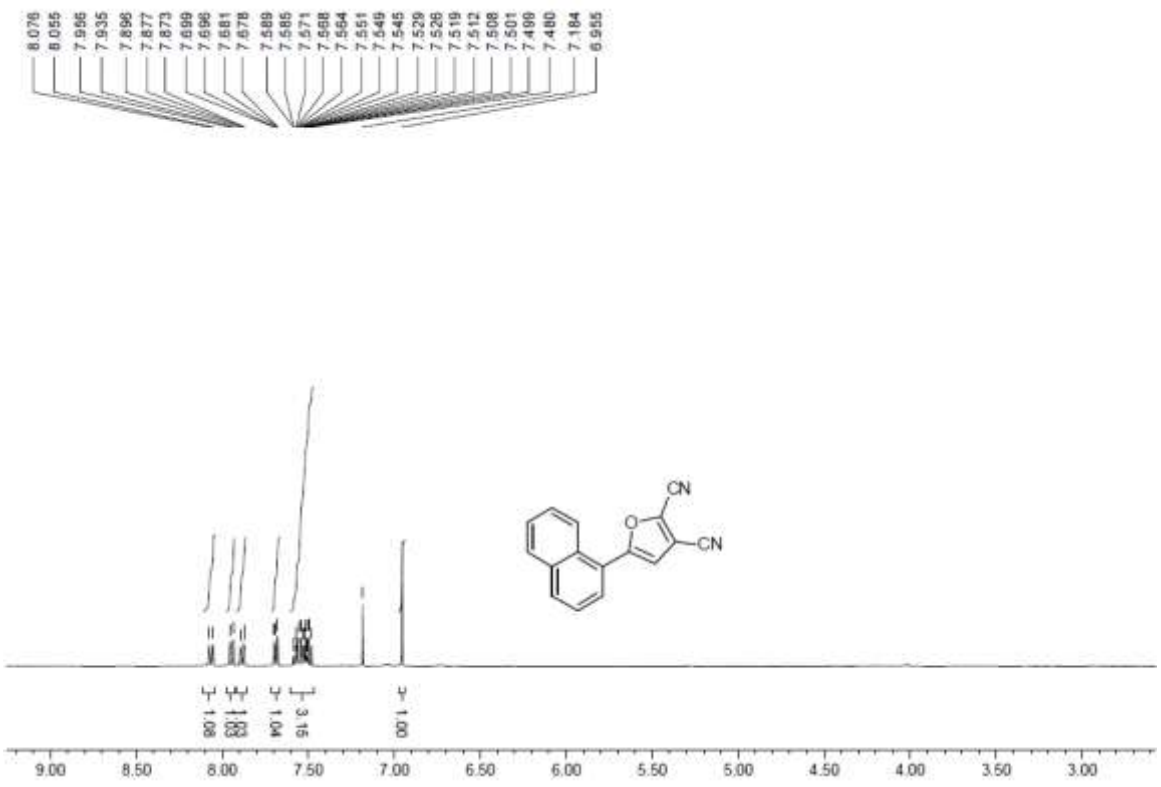
^{13}C NMR spectrum of **2l**



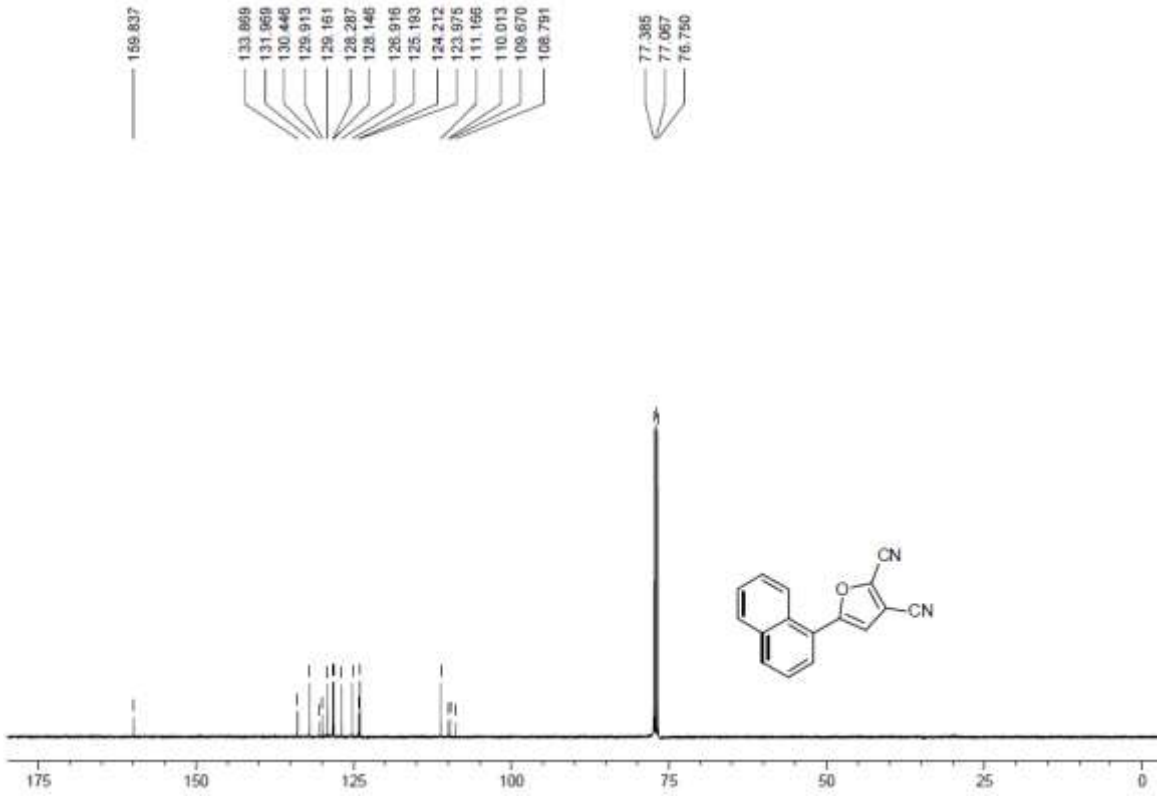
^{1}H NMR spectrum of **2m**



^{13}C NMR spectrum of **2m**



^{1}H NMR spectrum of **2n**



^{13}C NMR spectrum of **2n**