

Synthesis of 2,6'-dioxo-1',5',6',7'-tetrahydrospiro
[indoline-3,4'-pyrazolo[3,4-*b*]pyridine]-5'-
carbonitriles via a one-pot, three-component reaction
in water

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ABSTRACT: A one-pot, three-component condensation reaction of an isatin, aminopyrazole and alkyl cyanoacetate in water to give 2,6'-dioxo-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-*b*]pyridine]-5'-carbonitrile with good yields, at 90 °C, using a Et₃N as catalyst, is described.

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S1

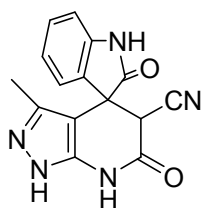
General information:

All chemicals were purchased and used without any further purification. IR spectra were recorded on a FT Infrared Spectroscopy instrument JASCO, FT/IR-6300 model as KBr discs. Mass spectra were recorded on a Shimadzu QP 1100 EX mass-Spectrometer operating at an ionization potential of 70 eV. The ^1H NMR, ^{13}C NMR, ROESY and HMBC spectra were recorded on a Bruker-AVANCE 400 MHz (topo spine) NMR instrument using $\text{DMSO-}d_6$ as solvent. Elemental analyses were performed using a Heraeus CHNS Rapid analyzer.

S2

General procedure for the synthesis of 2,6'-dioxo-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-b]pyridine]-5'-carbonitriles 4: A solution of isatin (1mmol), ethyl cyanoacetate (1 mmol) and Et_3N (0.1 mmol) in water (10 ml) in a balloon (25 mL) was stirred for 1 h at 60-70°C in oil bath. After production of intermediate 3-amin-pyrazole (1 mmol) was added in balloon and stirred for 5-12 h at 90°C in oil bath. Later than the completion of the reactions, which has been followed by TLC ($\text{EtOAc}:\textit{n}$ -hexene, 1:2), the reaction mixture was filtered as warm. The residue was washed with water and then recrystallized from ethanol and pure product was obtained.

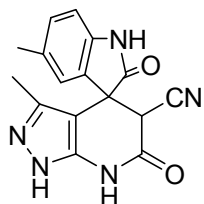
3'-methyl-2,6'-dioxo-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-b]pyridine]-5'-carbonitrile (4{1,1,1}): white powder (89%); IR (KBr): 3229, 3125, 3043, 2884, 2254, 1716, 1693, 1622, 1603 cm^{-1} ; MS (EI, 70 eV): m/z (%): 293 (M^+ , 100), 238 (67), 115 (61), 77 (72), 69 (81), 67 (89), 57 (91), 42 (85); Anal.Calcd for $\text{C}_{15}\text{H}_{11}\text{N}_5\text{O}_2$: C 61.43, H 3.78, N 23.88; Found: C 61.49, H 3.74, N 23.91. (major isomer) ^1H NMR (400 MHz, DMSO-d_6) δ : 1.37 (s, 3H, CH_3), 5.38 (s, 1H, CH), 6.98 (d, $^3J = 7.6$ Hz, 1H, CH_{arom}), 7.11 (ddd, $^3J = 7.2$ Hz, $^3J = 7.6$ Hz, $^4J = 0.8$ Hz, 1H, CH_{arom}), 7.35 (ddd, $^3J = 7.8$ Hz, $^3J = 7.8$ Hz, $^4J = 1.2$ Hz, 1H, CH_{arom}), 7.41 (d, $^3J = 7.2$ Hz, 1H, CH_{arom}), 10.76 (s, 1H, NH_{amide}), 11.01 (s, 1H, NH_{amide}), 12.14 (s, 1H, $\text{NH}_{\text{pyrazole}}$) ppm. ^{13}C NMR (100 MHz, DMSO-d_6) δ : 8.82, 42.50, 48.82, 97.27, 110.23, 115.19, 122.39, 124.63, 127.51, 129.84, 134.94, 142.58, 148.17, 162.13, 176.05 ppm. (minor isomer) ^1H NMR (400 MHz, DMSO-d_6) δ : 1.80 (s, 3H, CH_3), 4.88 (s, 1H, CH), 6.97-7.02 (m, 3H, CH_{arom}), 7.27-7.32 (m, 1H, CH_{arom}), 11.11 (s, 1H, NH_{amide}), 11.26 (s, 1H, NH_{amide}), 12.24 (s, 1H, $\text{NH}_{\text{pyrazole}}$) ppm. ^{13}C NMR (100 MHz, DMSO-d_6) δ : 9.08, 43.30, 48.27, 98.24, 110.47, 114.77, 122.68, 123.15, 128.96, 129.63, 134.20, 140.84, 147.64, 161.70, 175.31 ppm.



4{1,1,1}

3',5-dimethyl-2,6'-dioxo-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-b]pyridine]-5'-carbonitrile (4{1,1,2}): white powder (57%); IR (KBr): 3639, 3313, 3256, 3039, 2912, 2254, 1707, 1605, 1530 cm^{-1} ; MS (EI, 70 eV): m/z (%): 307 (M^+ , 100), 280 (49), 252 (82), 77 (75), 63 (53), 42 (61); Anal.Calcd for $\text{C}_{16}\text{H}_{13}\text{N}_5\text{O}_2$: C 60.53, H 4.26, N 22.79; Found: C 60.29,

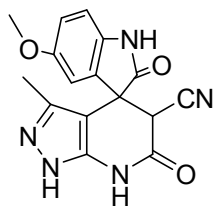
H 4.29, N 22.65. (major isomer)) ^1H NMR (400 MHz, DMSO- d_6) δ : 1.38 (s, 3H, CH_3), 2.29 (s, 3H, CH_3), 5.31(s, 1H, CH), 6.87 (d, $^3J = 7.6$ Hz, 1H, CH_{arom}), 7.16 (d, $^3J = 7.6$ Hz, 1H, CH_{arom}), 7.24 (s, 1H, CH_{arom}), 10.64 (s, 1H, NH_{amide}), 10.97 (s, 1H, NH_{amide}), 12.11 (s, 1H, $\text{NH}_{\text{pyrazole}}$) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ : 8.90, 20.73, 42.54, 48.88, 97.39, 109.96, 115.24, 125.06, 127.57, 130.05, 131.36, 134.96, 140.12, 148.15, 162.15, 176.04 ppm. (minor isomer) ^1H NMR (400 MHz, DMSO- d_6) δ : 1.82 (s, 3H, CH_3), 2.22 (s, 3H, CH_3), 4.88 (s, 1H, CH), 6.79 (s, 1H, CH_{arom}), 6.89 (d, $^3J = 7.6$ Hz, 1H, CH_{arom}), 7.10 (d, $^3J = 7.6$ Hz, 1H, CH_{arom}), 11.01 (s, 1H, NH_{amide}), 11.23 (s, 1H, NH_{amide}), 12.22 (s, 1H, $\text{NH}_{\text{pyrazole}}$) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ : 9.07, 20.61, 43.41, 48.41, 98.61, 110.28, 114.74, 123.45, 129.19, 129.96, 131.61, 134.18, 138.35, 147.57, 161.74, 175.27 ppm.



4{1,1,2}

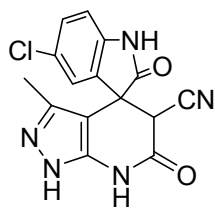
5-methoxy-3'-methyl-2,6'-dioxo-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-b]pyridine]-5'-carbonitrile (4{1,1,3}): white powder (76%); IR (KBr): 3628, 3407, 3237, 3039, 2906, 2254, 1709 cm^{-1} ; MS (EI, 70 eV): m/z (%): 323 (M^+ , 100), 268 (38), 77 (36), 63 (45), 52 (57), 42 (65); Anal.Calcd for $\text{C}_{16}\text{H}_{13}\text{N}_5\text{O}_3$: C 59.44, H 4.05, N 21.66; Found: C 59.62, H 4.08, N 21.55. (major isomer) ^1H NMR (400 MHz, DMSO- d_6) δ : 1.41 (s, 3H, CH_3), 3.71 (s, 3H, OCH_3), 5.34 (s, 1H, CH), 6.89-6.94 (bm, 2H, CH_{arom}), 7.10 (s, 1H, CH_{arom}), 10.59 (s, 1H, NH_{amide}), 11.00 (s, 1H, NH_{amide}), 12.15 (s, 1H, $\text{NH}_{\text{pyrazole}}$) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ : 8.83, 42.47, 49.30, 55.49, 97.33, 110.82, 111.14, 114.75, 115.16, 128.69, 135.04, 135.66, 148.08, 155.36, 162.11, 175.95 ppm. (minor isomer): ^1H NMR (400 MHz, DMSO- d_6) δ : 1.81(s, 3H, CH_3), 3.66 (s, 3H, OCH_3), 4.86 (s, 1H, CH), 6.52 (d, $^4J = 2$ Hz, 1H, CH_{arom}), 6.89-6.94 (bm, 2H, CH_{arom}),

10.96 (s, 1H, NH_{amide}), 11.25 (s, 1H, NH_{amide}), 12.26 (s, 1H, NH_{pyrazole}) ppm. ¹³C NMR (100 MHz, DMSO-d₆) δ: 9.04, 43.30, 48.58, 55.40, 98.42, 110.13, 111.09, 113.74, 115.16, 130.15, 134.01, 137.37, 147.54, 155.23, 161.72, 175.07 ppm.



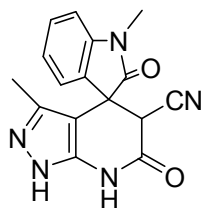
4{1,1,3}

5-chloro-3'-methyl-2,6'-dioxo-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-b]pyridine]-5'-carbonitrile (4{1,1,4}): white powder (57%); IR (KBr): 3633, 3418, 3323, 2906, 2254, 1716, 1690, 1619, 1529, 1477, 1306, 1174, 708 cm⁻¹; MS (EI, 70 eV): m/z (%): 329 (M⁺+2, 31), 327 (M⁺, 79), 166 (91), 75 (93), 69(95), 67 (90), 57 (100), 44 (87). Anal.Calcd for C₁₅H₁₀ClN₅O₂: C 54.97, H 3.08, N 21.37; Found: C 55.09, H 4.11, N 21.26. (major isomer) ¹H NMR (400 MHz, DMSO-d₆) δ: 1.44 (s, 3H, CH₃), 5.38 (s, 1H, CH), 7.02 (d, ³J = 8.0 Hz, 1H, CH_{arom}), 7.43 (dd, ³J = 8.4 Hz, ⁴J = 2.0 Hz, 1H, CH_{arom}), 7.54 (d, ⁴J = 1.6 Hz, 1H, CH_{arom}), 10.90 (s, 1H, NH_{amide}), 11.01 (s, 1H, NH_{amide}), 12.16 (s, 1H, NH_{pyrazole}) ppm. ¹³C NMR (100 MHz, DMSO-d₆) δ: 8.94, 42.22, 49.09, 96.66, 111.86, 114.95, 154.80, 126.48, 129.44, 129.89, 134.99, 141.49, 148.04, 161.77, 175.74 ppm. (minor isomer): ¹H NMR (400 MHz, DMSO-d₆) δ: 1.82 (s, 3H, CH₃), 4.92 (s, 1H, CH), 6.94 (d, ⁴J = 2.0 Hz, 1H, CH_{arom}), 7.04 (d, ³J = 8.8 Hz, 1H, CH_{arom}), 7.38 (dd, ³J = 8.4 Hz, ⁴J = 2.0 Hz, 1H, CH_{arom}), 11.24 (s, 1H, NH_{amide}), 11.26 (s, 1H, NH_{amide}), 12.28 (s, 1H, NH_{pyrazole}) ppm. ¹³C NMR (100 MHz, DMSO-d₆) δ: 9.04, 43.17, 48.43, 97.69, 112.26, 114.57, 122.85, 126.48, 129.77, 130.81, 134.99, 139.84, 148.04, 161.42, 174.93 ppm.



4{1,1,4}

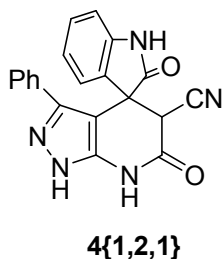
1,3'-dimethyl-2,6'-dioxo-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-b]pyridine]-5'-carbonitrile (4{1,1,5}): white powder (63%); IR (KBr): 3338, 3201, 3059, 2869, 2256, 1693, 1612, 1525, 1472, 1374 cm^{-1} . Anal.Calcd for $\text{C}_{16}\text{H}_{13}\text{N}_5\text{O}_2$: C 60.53, H 4.26, N 22.79; Found: C 60.36, H 4.22, N 22.70. (major isomer)) ^1H NMR (400 MHz, DMSO-d_6) δ : 1.34 (s, 3H, CH_3), 3.16 (s, 3H, CH_3), 5.42 (s, 1H, CH), 7.18-7.21 (m, 2H, CH_{arom}), 7.45-7.48 (m, 2H, CH_{arom}), 11.06 (s, 1H, NH_{amide}), 12.16 (s, 1H, $\text{NH}_{\text{pyrazole}}$) ppm. ^{13}C NMR (100 MHz, DMSO-d_6) δ : 8.88, 26.16, 42.61, 48.47, 96.97, 109.36, 115.01, 123.11, 124.30, 126.61, 130.02, 135.11, 143.96, 148.19, 162.04, 174.20 ppm. (minor isomer)) ^1H NMR (400 MHz, DMSO-d_6) δ : 1.71 (s, 3H, CH_3), 3.28 (s, 3H, CH_3), 5.42 (s, 1H, CH), 7.18-7.13 (m, 2H, CH_{arom}), 7.39-7.43 (m, 2H, CH_{arom}), 11.29 (s, 1H, NH_{amide}), 12.26 (s, 1H, $\text{NH}_{\text{pyrazole}}$) ppm. ^{13}C NMR (100 MHz, DMSO-d_6) δ : 9.01, 26.57, 43.09, 47.85, 98.10, 109.61, 114.72, 122.84, 123.39, 128.03, 129.79, 134.33, 142.31, 147.65, 161.62, 173.53 ppm.



4{1,1,5}

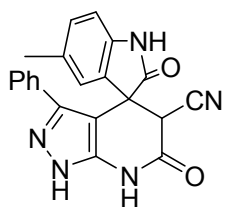
2,6'-dioxo-3'-phenyl-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-b]pyridine]-5'-carbonitrile (4{1,2,1}): white powder (83%); IR (KBr): 3597, 3508, 3398, 3165, 2261, 1734, 1708, 1618, 1469, 761, 701 cm^{-1} ; MS (EI, 70 eV): m/z (%): 355 (M^+ , 29), 300 (17), 77 (100), 57

(81), 51 (64), 43 (93); Anal.Calcd for C₂₀H₁₃N₅O₂: C 67.60, H 3.69, N 19.71; Found: C 67.41, H 3.67, N 19.79. (major isomer)) ¹H NMR (400 MHz, DMSO-d₆) δ: 5.33 (s, 1H, CH), 6.63 (t, ³J = 7.6 Hz, 1H, CH_{arom}), 6.83-7.38 (m, 7H, CH_{arom}), 10.96 (s, 1H, NH_{amide}), 11.18 (s, 1H, NH_{amide}), 12.74 (s, 1H, NH_{pyrazole}) ppm. ¹³C NMR (100 MHz, DMSO-d₆) δ: 43.78, 48.90, 97.75, 108.86, 114.94, 122.15, 124.44, 127.19, 127.25, 127.81, 128.46, 128.88, 129.46, 139.54, 142.71, 149.02, 162.05, 175.97 ppm. (minor isomer)) ¹H NMR (400 MHz, DMSO-d₆) δ: 4.91 (s, 1H, CH), 6.83-7.38 (m, 8H, CH_{arom}), 11.03 (s, 1H, NH_{amide}), 11.43 (s, 1H, NH_{amide}), 12.86 (s, 1H, NH_{pyrazole}) ppm. ¹³C NMR (100 MHz, DMSO-d₆) δ: 44.58, 48.70, 98.47, 110.55, 114.44, 122.80, 123.44, 126.70, 127.81, 127.90, 128.25, 128.88, 130.09, 138.68, 140.97, 148.44, 161.58, 174.88 ppm.



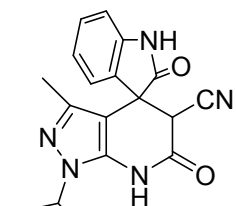
5-methyl-2,6'-dioxo-3'-phenyl-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-b]pyridine]-5'-carbonitrile (4{1,2,2}): white powder (77%); IR (KBr): 3453, 3246, 3026, 2912, 2868, 2266, 1704, 1622, 1594, 1488 cm⁻¹. MS (EI, 70 eV): m/z (%): 369 (M⁺, 89), 314 (42), 77 (100), 55 (50), 51 (58), 44 (62), 43 (81). Anal.Calcd for C₂₁H₁₅N₅O₂: C 68.28, H 4.09, N 18.96; Found: C 68.20, H 4.06, N 19.01. (major isomer) ¹H NMR (400 MHz, DMSO-d₆) δ: 1.90 (s, 3H, CH₃), 5.27 (s, 1H, CH), 6.70-7.37 (m, 8H, CH_{arom}), 10.82 (s, 1H, NH_{amide}), 11.15 (s, 1H, NH_{amide}), 12.71 (s, 1H, NH_{pyrazole}) ppm. ¹³C NMR (100 MHz, DMSO-d₆) δ: 20.31, 43.76, 48.86, 97.75, 109.57, 115.02, 125.13, 127.21, 127.32, 127.65, 128.44, 129.09, 129.53, 130.94, 138.57, 140.32, 148.94, 162.11, 175.91 ppm. (minor isomer) ¹H NMR (400 MHz, DMSO-d₆) δ: 2.23 (s, 3H, CH₃), 4.91 (s, 1H, CH), 6.70-7.37 (m, 8H, CH_{arom}), 10.92 (s, 1H, NH_{amide}), 11.41 (s, 1H, NH_{amide}), 12.85 (s, 1H, NH_{pyrazole}) ppm. ¹³C NMR (100 MHz, DMSO-d₆) δ: 20.59, 44.66, 48.77,

98.61, 110.33, 114.47, 123.79, 126.49, 127.32, 127.65, 128.10, 128.84, 130.44, 131.69, 138.62, 139.55, 148.43, 161.61, 174.79 ppm.



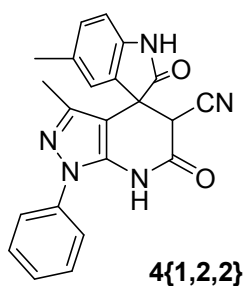
4{1,2,2}

3'-methyl-2,6'-dioxo-1'-phenyl-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-b]pyridine]-5'-carbonitrile (4{1,3,1}): white powder (83%). IR (KBr): 3571, 3165, 3092, 2258, 1734, 1711, 1597, 1557 cm^{-1} . MS (EI, 70 eV): m/z (%): 369 (M^+ , 100), 341 (87), 301 (32), 115 (47), 77 (81), 57 (61), 51 (66); Anal.Calcd for $\text{C}_{21}\text{H}_{15}\text{N}_5\text{O}_2$: C 68.28, H 4.09, N 18.96; Found: C 68.44, H 4.05, N 18.89. (major isomer)) ^1H NMR (400 MHz, DMSO-d_6) δ : 1.38 (s, 3H, CH_3), 5.59 (s, 1H, CH), 7.02-7.55 (m, 9H, CH_{arom}), 10.89 (s, 1H, NH_{amide}), 11.40 (s, 1H, NH_{amide}) ppm. ^{13}C NMR (100 MHz, DMSO-d_6) δ : 11.67, 42.53, 49.13, 98.75, 110.48, 114.61, 122.55, 123.09, 124.75, 126.95, 127.48, 129.34, 130.12, 137.34, 139.17, 142.83, 144.23, 162.93, 175.77 ppm. (minor isomer)) ^1H NMR (400 MHz, DMSO-d_6) δ : 1.84 (s, 3H, CH_3), 5.18 (s, 1H, CH), 7.02-7.55 (m, 9H, CH_{arom}), 11.25 (s, 1H, NH_{amide}), 11.58 (bs, 1H, NH_{amide}), ppm. ^{13}C NMR (100 MHz, DMSO-d_6) δ : 12.66, 43.54, 48.85, 100.15, 109.71, 110.60, 114.15, 122.66, 122.86, 123.14, 126.95, 128.19, 129.03, 129.97, 137.37, 140.91, 143.55, 162.42, 175.19 ppm.



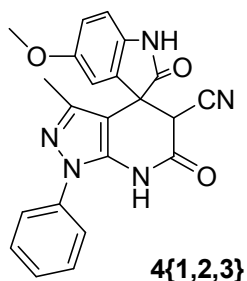
4{1,3,1}

3',5'-dimethyl-2,6'-dioxo-2'-phenyl-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-b]pyridine]-5'-carbonitrile (4{1,3,2}): white powder (71%). IR (KBr): 3572, 3502, 3180, 2938, 2252, 1718, 1595, 1496, 1319 cm^{-1} . MS (EI, 70 eV): m/z (%): 383 (M^+ , 100), 354 (72), 77 (95), 65 (40), 51 (67). Anal.Calcd for $\text{C}_{22}\text{H}_{17}\text{N}_5\text{O}_2$: C 68.92, H 4.47, N 18.27; Found: C 59.08, H 4.42, N 18.16. (major isomer) ^1H NMR (400 MHz, DMSO-d_6) δ : 1.39 (s, 3H, CH_3), 2.32 (s, 3H, CH_3), 5.52 (s, 1H, CH), 6.92 (d, $^3J = 7.6$ Hz, 1H, CH_{arom}), 7.20 (d, $^3J = 8.0$ Hz, 1H, CH_{arom}), 7.31 (s, 1H, CH_{arom}), 7.39-7.55 (m, 5H, CH_{arom}), 10.78 (s, 1H, NH_{amide}), 11.37 (s, 1H, NH_{amide}) ppm. ^{13}C NMR (100 MHz, DMSO-d_6) δ : 11.73, 20.71, 42.55, 49.16, 98.85, 110.20, 114.68, 123.11, 125.17, 127.03, 127.47, 129.34, 130.32, 131.56, 137.36, 139.14, 140.36, 144.27, 162.94, 175.75 ppm. (minor isomer) ^1H NMR (400 MHz, DMSO-d_6) δ : 1.84 (s, 3H, CH_3), 2.23 (s, 3H, CH_3), 5.16 (s, 1H, CH), 6.83 (s, 1H, CH_{arom}), 6.93 (d, $^3J = 7.2$ Hz, 1H, CH_{arom}), 7.14 (d, $^3J = 8.0$ Hz, 1H, CH_{arom}), 7.39-7.55 (m, 5H, CH_{arom}), 11.15 (s, 1H, NH_{amide}), 11.54 (s, 1H, NH_{amide}) ppm. ^{13}C NMR (100 MHz, DMSO-d_6) δ : 11.68, 20.61, 43.60, 48.93, 100.32, 110.38, 114.19, 123.65, 125.17, 127.03, 127.47, 129.34, 130.32, 131.80, 138.46, 139.14, 140.36, 143.54, 162.49, 175.14 ppm.



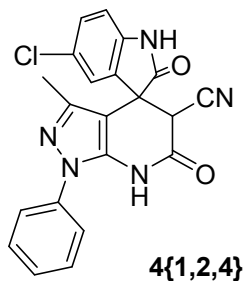
5-methoxy-3'-methyl-2,6'-dioxo-1'-phenyl-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-b]pyridine]-5'-carbonitrile (4{1,3,3}): white powder (78%). IR (KBr): 3564, 3496, 3174, 3067, 2935, 2252 (weak), 1720, 1615, 1556, 1497, 1312, 1212 cm^{-1} . MS (EI, 70 eV): m/z (%): 399 (M^+ , 100), 356 (79), 77 (78), 65 (24), 51 (55); Anal.Calcd for $\text{C}_{22}\text{H}_{17}\text{N}_5\text{O}_3$: C

66.16, H 4.29, N 17.53; Found: C 65.98, H 4.32, N 17.48. (major isomer) ^1H NMR (400 MHz, DMSO- d_6) δ : 1.43 (s, 3H, CH_3), 3.75 (s, 3H, OCH_3), 5.56 (s, 1H, CH), 6.94-7.55 (m, 8H, CH_{arom}), 10.72 (s, 1H, NH_{amide}), 11.40 (br,s, 1H, NH_{amide}) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ : 11.69, 42.49, 49.59, 55.55, 98.78, 111.06, 111.23, 114.62, 115.04, 123.08, 127.50, 128.17, 129.36, 135.93, 137.31, 139.12, 144.25, 155.47, 162.88, 175.65 ppm. (minor isomer) ^1H NMR (400 MHz, DMSO- d_6) δ : 1.85 (s, 3H, CH_3), 3.67 (s, 3H, OCH_3), 5.16 (s, 1H, CH), 6.94-7.55 (m, 8H, CH_{arom}), 11.09 (s, 1H, NH_{amide}), 11.40 (br,s, 1H, NH_{amide} (This signal overlap with major signal)) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ : 11.69, 43.54, 49.15, 56.01, 100.23, 110.36, 111.16, 114.03, 114.17, 123.08, 127.50, 128.17, 129.42, 134.07, 137.37, 143.56, 155.34, 162.46, 174.91 ppm.

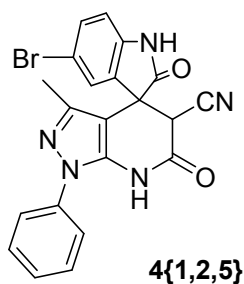


5-chloro-3'-methyl-2,6'-dioxo-1'-phenyl-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo [3,4-b]pyridine]-5'-carbonitrile (4{1,3,4}): white powder (72%). IR (KBr): 3574, 3503, 3172, 2965, 2266, 1724, 1619, 1563, 1479 cm^{-1} . MS (EI, 70 eV): m/z (%): 405 ($\text{M}^+ + 2$, 44), 403 (M^+ , 100), 375 (46), 77 (87), 51 (65), 43 (43); Anal.Calcd for $\text{C}_{21}\text{H}_{14}\text{ClN}_5\text{O}_2$: C 62.46, H 3.49, N 17.34; Found: C 62.57, H 3.48, N 17.39. (major isomer) ^1H NMR (400 MHz, DMSO- d_6) δ : 1.44 (s, 3H, CH_3), 5.63 (s, 1H, CH), 7.05 (s, 1H, CH_{arom}), 7.42-7.55 (m, 6H, CH_{arom}), 7.64 (s, 1H, CH_{arom}), 11.07 (s, 1H, NH_{amide}), 11.47 (br,s, 1H, NH_{amide}) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ : 11.82, 42.24, 49.37, 98.11, 112.05, 114.42, 123.16, 124.99, 126.58, 127.58, 128.97, 129.37, 130.12, 137.26, 139.21, 141.77, 144.03, 162.56, 175.46 ppm. (minor isomer) ^1H NMR (400

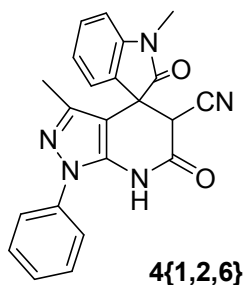
MHz, DMSO- d_6) δ : 1.85 (s, 3H, CH₃), 5.23 (s, 1H, CH), 7.07 (s, 1H, CH_{arom}), 7.42-7.55 (m, 7H, CH_{arom}), 11.42 (s, 1H, NH_{amide}), 11.47 (s, 1H, NH_{amide} (This signal overlap with major signal)), ppm. ¹³C NMR (100 MHz, DMSO- d_6) δ : 11.70, 43.45, 49.01, 98.11, 112.27, 114.42, 123.22, 124.99, 126.58, 127.58, 128.97, 129.30, 130.03, 137.26, 139.21, 141.77, 143.50, 162.13, 175.46 ppm.

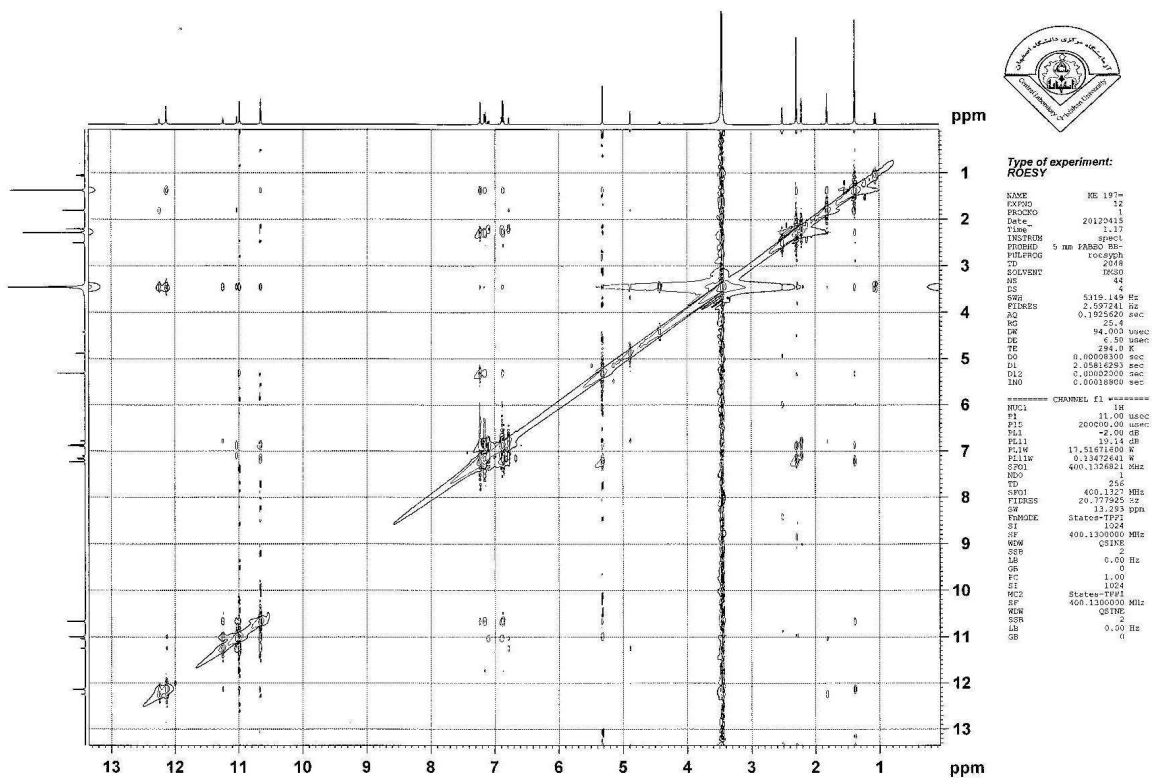


5-bromo-3'-methyl-2,6'-dioxo-1'-phenyl-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo [3,4-b]pyridine]-5'-carbonitrile (4{1,3,5}): white powder (64%). IR (KBr): 3577, 3504, 3071, 2938, 1723, 1616, 1499, 1318 cm^{-1} . MS (EI, 70 eV): m/z (%): 449 ($M^+ + 2$, 100), 447 (M^+ , 96), 445 (91), 418 (85), 152 (48), 141 (62), 127 (60), 114 (64). Anal. Calcd for $\text{C}_{21}\text{H}_{14}\text{BrN}_5\text{O}_2$: C 56.27, H 3.15, N 15.62. Found: C 55.98, H 3.17, N 15.57. (major isomer)) ¹H NMR (400 MHz, DMSO- d_6) δ : 1.44 (s, 3H, CH₃), 5.64 (s, 1H, CH), 7.01 (d, $^3J = 8.0$ Hz, 1H, CH_{arom}), 7.40-7.61 (m, 6H, CH_{arom}), 7.76 (s, 1H, CH_{arom}), 11.07 (s, 1H, NH_{amide}), 11.46 (s, 1H, NH_{amide}), ppm. ¹³C NMR (100 MHz, DMSO- d_6) δ : 11.82, 42.22, 49.30, 98.13, 112.53, 114.14, 114.43, 123.17, 123.22, 127.59, 127.72, 129.32, 129.37, 132.95, 137.25, 139.18, 142.17, 144.02, 162.56, 175.33 ppm. (minor isomer)) ¹H NMR (400 MHz, DMSO- d_6) δ : 1.85 (s, 3H, CH₃), 5.24 (s, 1H, CH), 7.01 (d, $^3J = 8.0$ Hz, 1H, CH_{arom}), 7.03 (s, 1H, CH_{arom}), 7.15 (s, 1H, CH_{arom}), 7.40-7.61 (m, 6H, CH_{arom}), 11.43 (s, 1H, NH_{amide}), 11.52 (s, 1H, NH_{amide}) ppm. ¹³C NMR (100 MHz, DMSO- d_6) δ : 11.71, 43.49, 48.93, 112.77, 123.22, 125.89, 140.34, 143.50, 162.13, 174.70 ppm (some of other carbons signals did not appeared in ¹³C NMR).



1,3'-dimethyl-2,6'-dioxo-1'-phenyl-1',5',6',7'-tetrahydrospiro[indoline-3,4'-pyrazolo[3,4-b]pyridine]-5'-carbonitrile (4m): white powder (55%); IR (KBr): 3172, 3101, 2952, 2882, 2258, 1732, 1697, 1612, 1561, 1497, 1378, 751 cm^{-1} . MS (EI, 70 eV): m/z (%): 383 (M^+ , 100), 357 (85), 354 (42), 77 (87), 65 (33), 51 (62). Anal. Calcd for $\text{C}_{22}\text{H}_{17}\text{N}_5\text{O}_2$: C 68.92, H 4.47, N 18.27; Found: C 69.11, H 4.45, N 18.38. (major isomer) ^1H NMR (400 MHz, DMSO-d_6) δ : 1.34 (s, 3H, CH_3), 3.21 (s, 3H, CH_3), 5.63 (s, 1H, CH), 7.23-7.56 (m, 9H, CH_{arom}), 11.48 (br, s, 1H, NH_{amide}) ppm. ^{13}C NMR (100 MHz, DMSO-d_6) δ : 11.72, 26.28, 42.59, 48.74, 98.42, 109.61, 114.46, 123.15, 123.17, 123.26, 124.44, 126.07, 127.56, 129.36, 130.27, 137.32, 139.27, 144.27, 162.83, 173.92 ppm. (minor isomer) ^1H NMR (400 MHz, DMSO-d_6) δ : 1.74 (s, 3H, CH_3), 3.34 (s, 3H, CH_3), 5.24 (s, 1H, CH), 7.08-7.56 (m, 9H, CH_{arom}), 11.48 (br, s, 1H, NH_{amide} (This minor signal is under the major signal)) ppm. ^{13}C NMR (100 MHz, DMSO-d_6) δ : 11.60, 26.73, 43.31, 48.44, 99.80, 109.75, 114.09, 122.96, 123.15, 123.59, 124.44, 126.07, 127.36, 129.36, 130.11, 138.77, 143.50, 144.19, 162.32, 173.39 ppm.





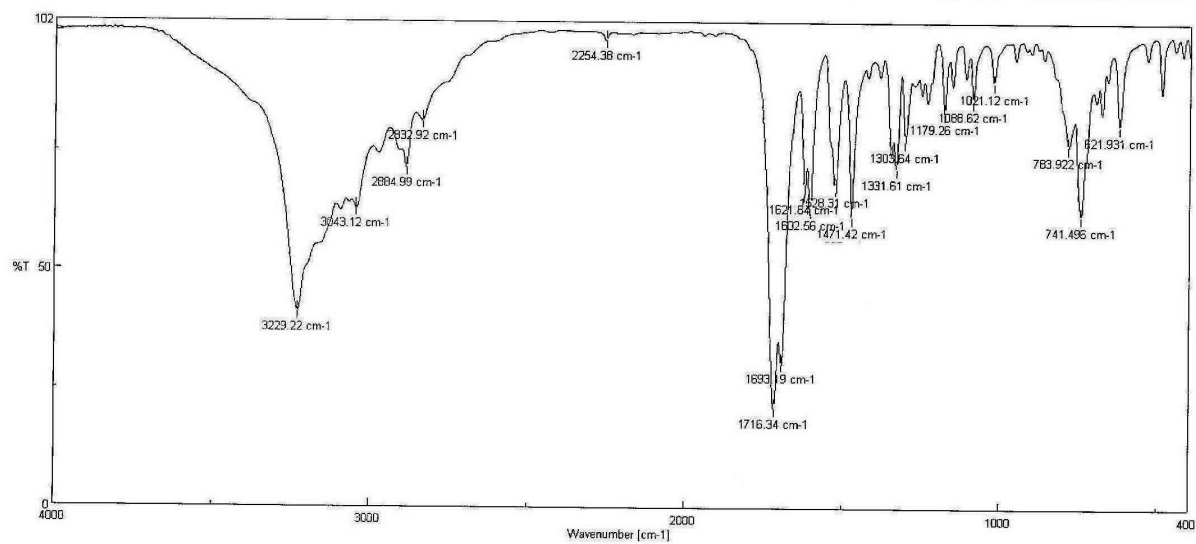
S-4: ROSEY spectrum of compound 4{1,1,2}

Sample Code: *11900830-122 (KE-122)*

Central Laboratory of the University of Isfahan

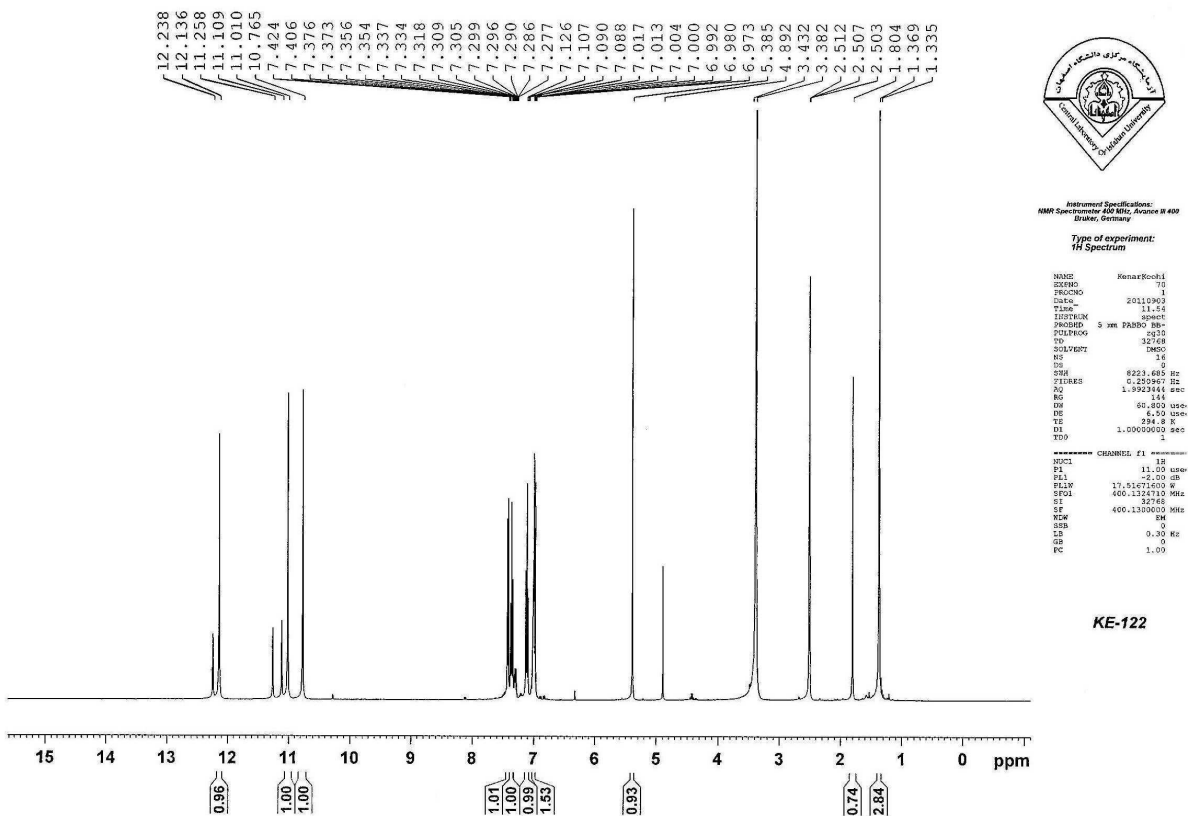
Instrument Specifications

FT Infrared Spectroscopy, JASCO, F7/IR-6300 (400-4000 cm⁻¹), Japan

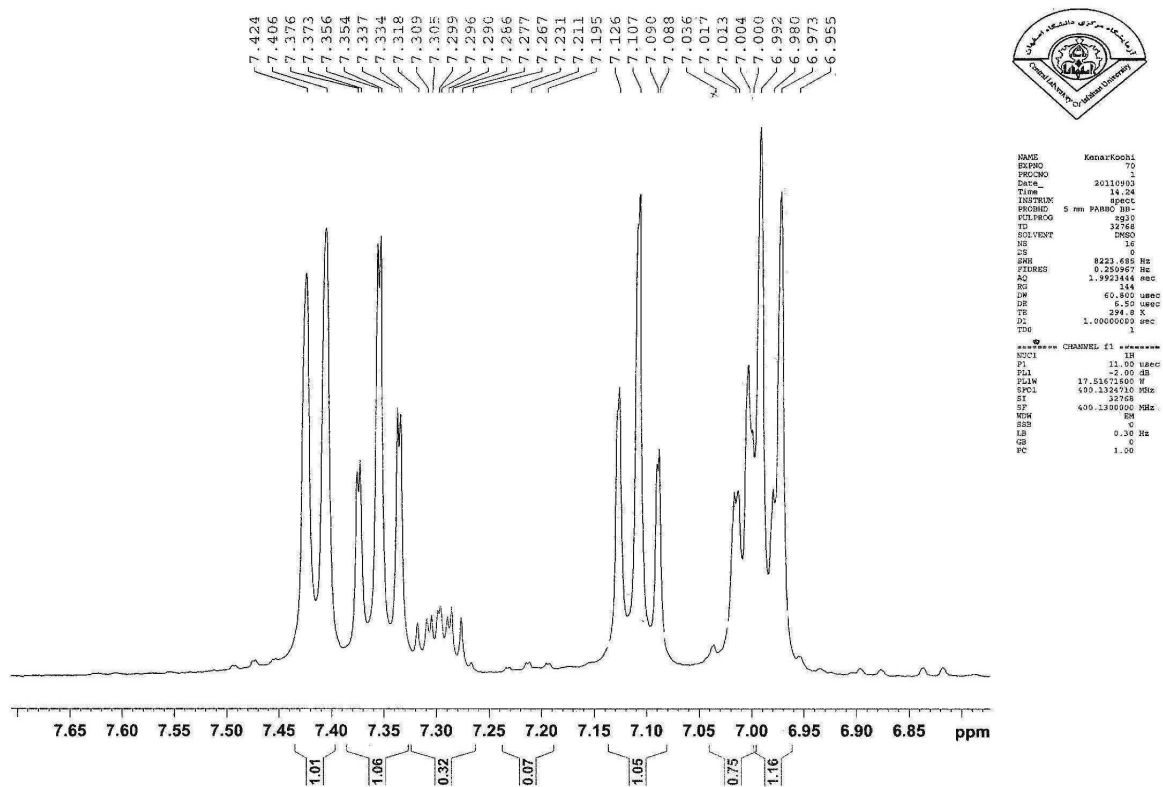


Date: 90/08/30
No.:

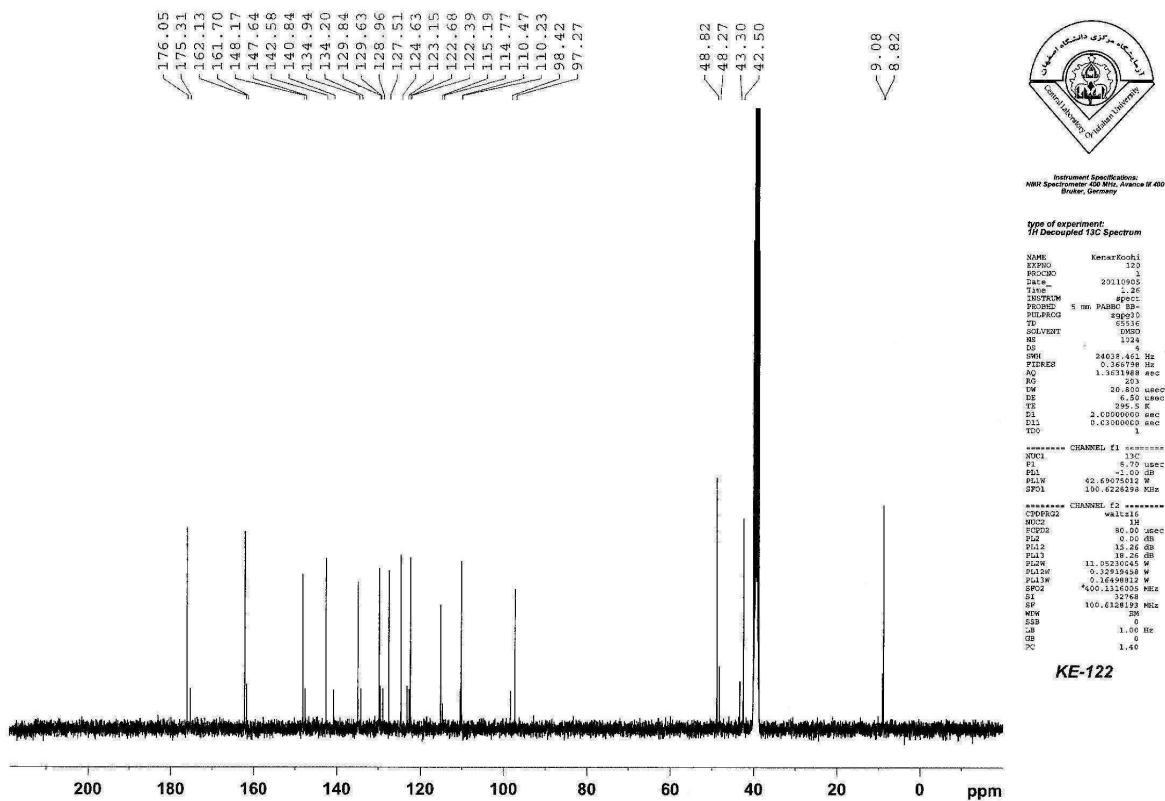
S-6: IR spectrum of compound **4{1,1,1}**



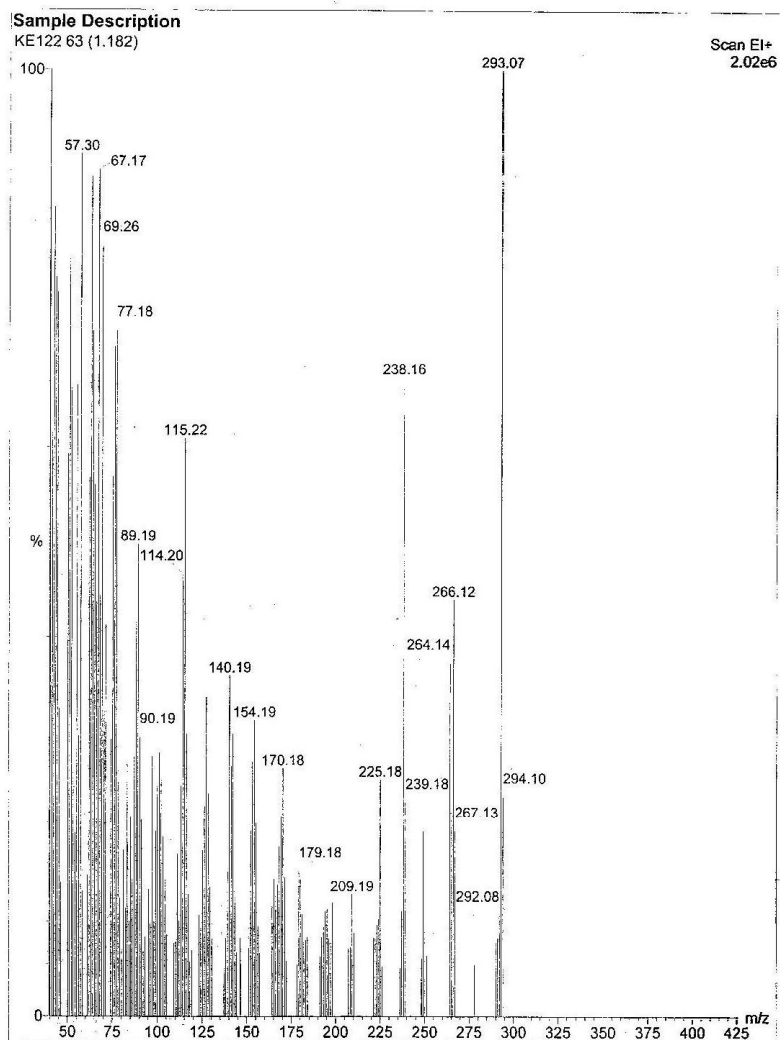
S-7: ^1H NMR spectrum of compound 4{1,1,1}



S-7: Expand of ^1H NMR spectrum of compound 4{1,1,1}



S-8: ^{13}C NMR spectrum of compound 4{1,1,1}



S-9: Mass spectrum of compound 4{1,1,1}

Sample Description

KE122 63 (1.182)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	Scan EI+
1	40.18	1.03e6	51.02	1.31	67	117.22	2.59e5	12.86	0.33	133	248.15	3.93e5	19.51	0.50	
2	41.26	1.25e6	61.79	1.58	68	118.19	1.17e5	5.79	0.15	134	250.14	1.90e5	9.45	0.24	
3	42.24	1.72e6	85.37	2.19	69	119.21	1.39e5	6.91	0.18	135	251.14	1.28e5	6.35	0.16	
4	43.25	1.57e6	78.05	2.00	70	123.22	2.15e5	10.67	0.27	136	264.14	7.50e5	37.20	0.95	
5	44.20	1.54e6	76.42	1.96	71	124.21	1.87e5	9.30	0.24	137	265.13	4.34e5	21.54	0.55	
6	45.25	6.55e5	32.52	0.83	72	125.21	3.52e5	17.48	0.45	138	266.12	8.85e5	43.90	1.13	
7	46.24	2.87e5	14.23	0.36	73	126.18	4.46e5	22.15	0.57	139	267.13	3.53e5	19.51	0.50	
8	60.21	1.20e6	59.35	1.52	74	127.20	6.80e5	33.74	0.87	140	278.10	1.09e5	5.39	0.14	
9	51.21	1.62e6	80.49	2.06	75	128.19	4.75e5	23.58	0.60	141	290.07	1.57e5	7.77	0.20	
10	52.21	1.34e6	66.67	1.71	76	129.19	2.74e5	13.62	0.35	142	291.08	1.66e5	8.23	0.21	
11	53.20	5.90e5	29.27	0.75	77	130.19	1.67e5	8.28	0.21	143	292.08	2.15e5	10.67	0.27	
12	54.21	3.89e5	19.31	0.50	78	137.23	1.02e5	5.08	0.13	144	293.07	2.02e6	100.00	2.57	
13	55.24	1.34e6	66.67	1.71	79	138.16	1.67e5	8.28	0.21	145	294.10	4.67e5	23.17	0.59	
14	56.28	5.98e5	29.67	0.76	80	139.19	3.07e5	15.24	0.39						
15	57.30	1.84e6	91.06	2.34	81	140.19	7.25e5	35.98	0.92						
16	58.43	2.66e5	13.21	0.34	82	141.19	5.32e5	26.42	0.68						
17	61.18	2.99e5	14.84	0.38	83	142.20	6.02e5	29.88	0.77						
18	62.19	1.15e6	56.91	1.46	84	143.19	2.74e5	13.62	0.35						
19	63.18	1.79e6	88.62	2.27	85	144.18	1.38e5	6.76	0.17						
20	64.17	1.46e6	72.36	1.86	86	146.49	1.57e5	8.28	0.21						
21	65.19	1.13e6	56.10	1.44	87	151.20	1.44e5	7.16	0.18						
22	66.19	8.81e5	43.70	1.12	88	152.18	3.93e5	19.51	0.50						
23	67.17	1.80e6	89.43	2.29	89	153.18	5.41e5	26.83	0.69						
24	68.19	8.97e5	44.51	1.14	90	154.19	6.31e5	31.30	0.80						
25	69.26	1.64e6	81.30	2.09	91	155.19	4.10e5	20.33	0.52						
26	70.44	6.47e5	32.11	0.82	92	156.18	1.91e5	9.50	0.24						
27	71.27	8.31e5	41.26	1.06	93	157.17	1.34e5	6.68	0.17						
28	74.17	5.90e5	29.27	0.75	94	164.17	2.33e5	11.59	0.30						
29	75.17	1.15e6	56.91	1.46	95	165.17	2.91e5	14.43	0.37						
30	76.18	1.43e6	70.73	1.81	96	166.17	2.62e5	13.01	0.33						
31	77.18	1.46e6	72.36	1.86	97	167.20	2.79e5	13.62	0.35						
32	78.15	5.00e5	24.30	0.64	98	168.18	3.60e5	17.89	0.46						
33	79.18	2.51e5	12.45	0.32	99	169.19	4.26e5	21.14	0.54						
34	80.19	1.44e5	7.16	0.18	100	170.18	5.28e5	26.22	0.67						
35	81.23	3.52e5	17.48	0.45	101	171.18	2.95e5	14.63	0.38						
36	82.36	2.30e5	11.43	0.29	102	172.19	1.18e5	5.84	0.15						
37	83.30	5.45e5	27.03	0.69	103	178.17	1.09e5	5.39	0.14						
38	84.32	2.02e5	10.01	0.26	104	179.18	3.07e5	15.24	0.39						
39	85.25	4.26e5	21.14	0.54	105	180.16	2.91e5	14.43	0.37						
40	86.15	2.91e5	14.43	0.37	106	181.17	2.17e5	10.77	0.28						
41	87.17	5.57e5	27.64	0.71	107	182.18	1.62e5	8.03	0.21						
42	88.18	8.40e5	41.67	1.07	108	183.19	1.62e5	8.03	0.21						
43	89.19	1.00e6	49.80	1.28	109	184.19	1.69e5	8.38	0.22						
44	90.19	5.94e5	29.47	0.76	110	191.18	1.26e5	6.25	0.16						
45	91.20	4.18e5	20.73	0.53	111	192.18	1.67e5	8.28	0.21						
46	92.20	1.36e5	6.76	0.17	112	193.18	1.96e5	9.71	0.25						
47	93.19	1.69e5	8.38	0.22	113	194.18	2.24e5	11.13	0.29						
48	95.26	2.70e5	13.41	0.34	114	195.18	2.28e5	11.33	0.29						
49	96.23	2.02e5	10.01	0.26	115	196.18	1.65e5	8.18	0.21						
50	97.28	5.53e5	27.44	0.70	116	197.18	1.57e5	7.77	0.20						
51	98.21	2.06e5	10.21	0.26	117	198.19	2.42e5	11.99	0.31						
52	99.18	3.93e5	19.51	0.50	118	207.18	1.43e5	7.11	0.18						
53	100.16	4.92e5	24.39	0.63	119	208.19	1.46e5	7.27	0.19						
54	101.19	5.61e5	27.85	0.71	120	209.19	2.59e5	12.86	0.33						
55	102.19	5.16e5	25.61	0.66	121	210.18	1.77e5	8.79	0.23						
56	103.18	3.81e5	18.90	0.48	122	221.18	1.67e5	8.28	0.21						
57	104.20	2.91e5	14.43	0.37	123	222.19	1.66e5	8.23	0.21						
58	105.28	1.74e5	8.64	0.22	124	223.20	1.95e5	9.65	0.25						
59	109.25	1.56e5	7.72	0.20	125	224.19	2.07e5	10.26	0.26						
60	110.22	1.59e5	7.88	0.20	126	225.18	5.04e5	25.00	0.64						
61	111.19	3.44e5	17.07	0.44	127	226.18	1.04e5	5.18	0.13						
62	112.16	2.04e5	10.11	0.26	128	236.18	1.01e5	5.03	0.13						
63	113.19	4.92e5	24.39	0.63	129	237.18	2.23e5	11.08	0.28						
64	114.20	9.38e5	46.54	1.19	130	238.16	1.36e6	67.48	1.73						
65	115.22	1.23e6	60.98	1.56	131	239.18	4.55e5	22.56	0.58						
66	116.21	6.02e5	29.88	0.77	132	248.16	1.23e5	6.10	0.16						

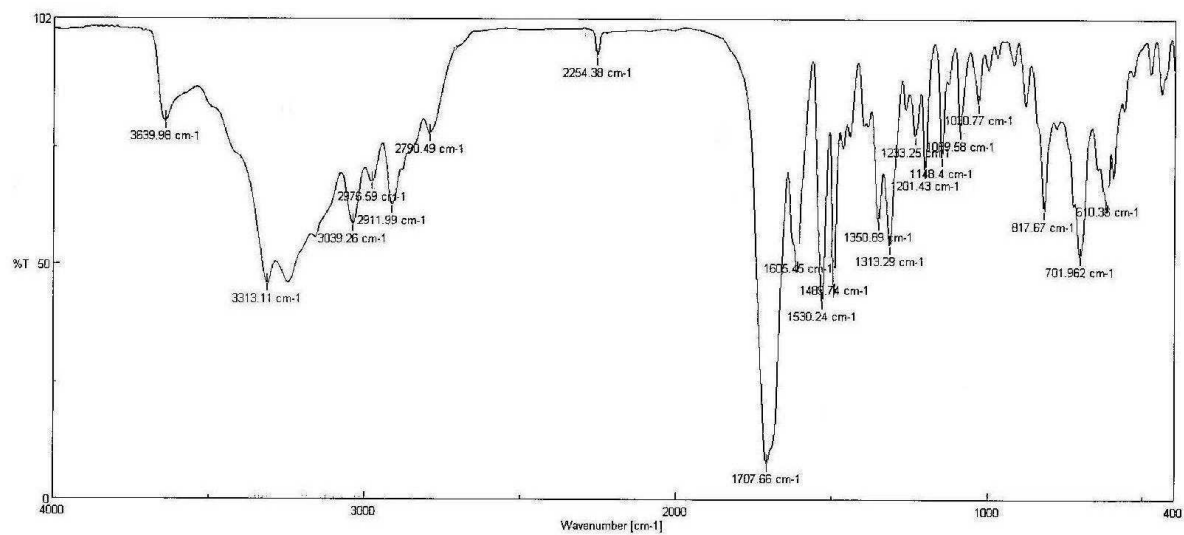
S-10: Sample description presented by mass instrument to compound 4{1,1,1}

Sample Code: *II900830-125 (KE-197)*

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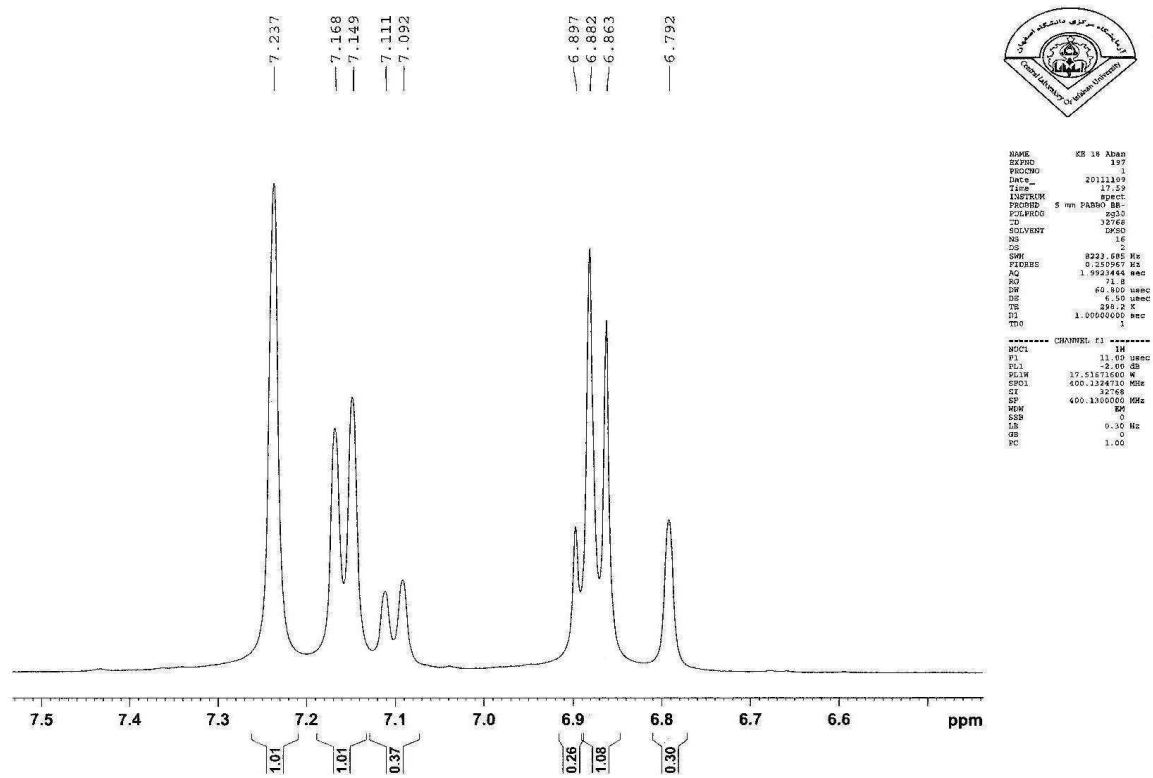
Instrument Specifications

FT Infrared Spectroscopy, JASCO, FT/IR-6300 (400–4000 cm^{-1}), Japan

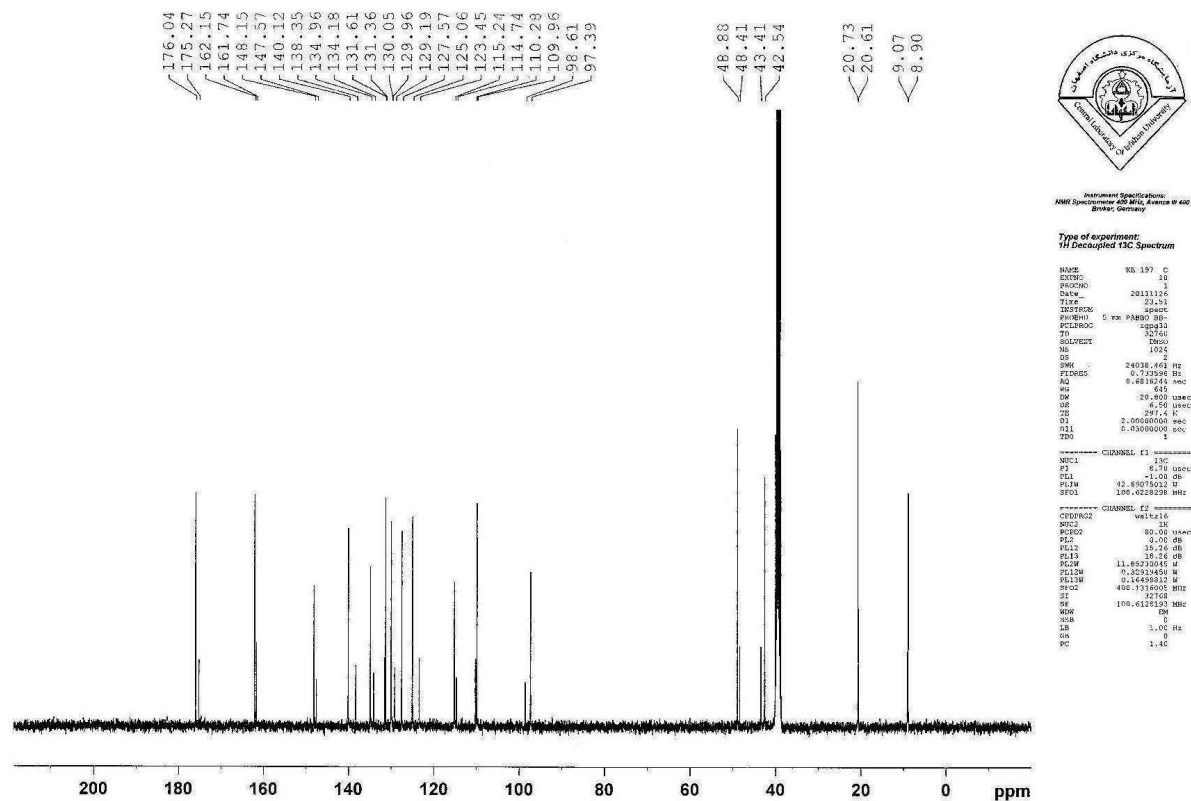


Date: 90/09/05
No.:

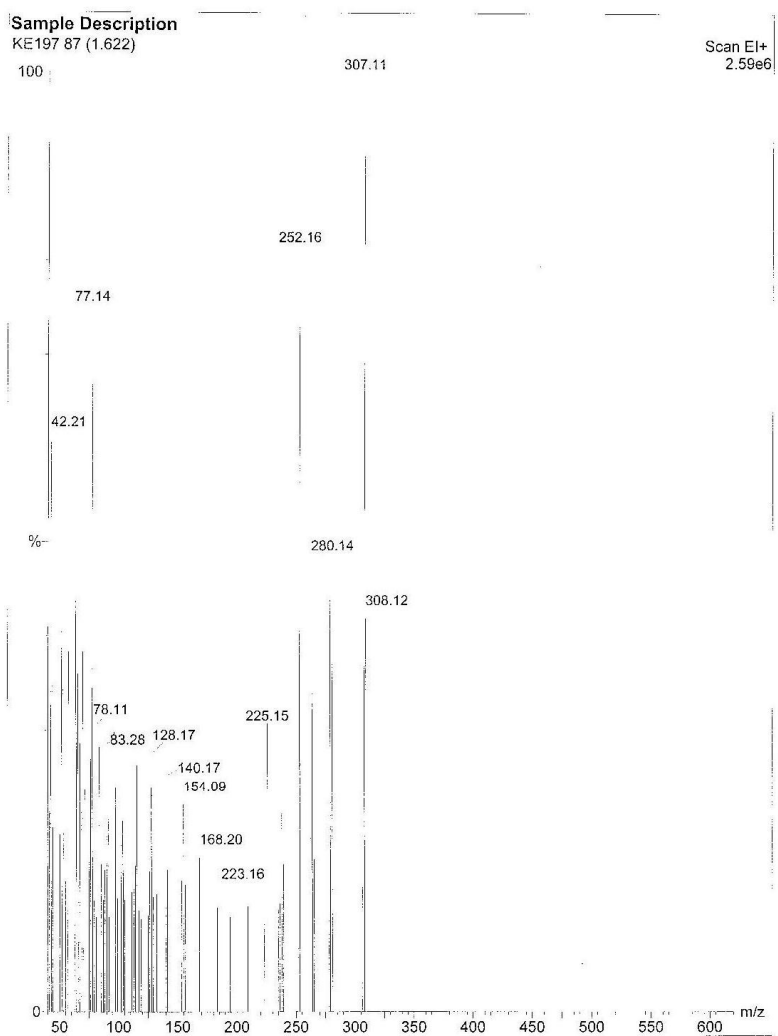
S-12: IR spectrum of compound **4{1,1,2}**



S-13: Expand of ^1H NMR spectrum of compound 4{1,1,2}



S-14. ^{13}C NMR spectrum of compound 4{1,1,2}



S-15: Mass spectrum of compound 4{1,1,2}

KE197 87 (1.622)

KE197 87 (1.622)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC
1:	40.14	4.01e5	15.51	0.54	67:	153.16	3.60e5	13.92	0.48					
2:	41.23	5.53e5	21.36	0.74	68:	154.09	5.69e5	21.99	0.76					
3:	42.21	1.57e6	60.76	2.10	69:	155.18	5.65e5	21.94	0.76					
4:	43.22	9.26e5	35.76	1.24	70:	156.19	3.48e5	13.45	0.47					
5:	44.17	5.08e5	19.62	0.68	71:	167.20	3.40e5	13.13	0.45					
6:	45.21	2.95e5	11.39	0.39	72:	168.20	4.22e5	16.30	0.56					
7:	50.17	4.87e5	18.83	0.65	73:	169.19	2.66e5	10.28	0.36					
8:	51.18	1.25e6	48.10	1.67	74:	183.19	2.87e5	11.08	0.38					
9:	52.17	8.89e5	34.34	1.19	75:	184.18	3.89e5	14.87	0.52					
10:	53.17	4.92e5	18.99	0.66	76:	194.18	2.61e5	10.09	0.35					
11:	55.18	5.78e5	22.31	0.77	77:	209.15	2.91e5	11.23	0.39					
12:	57.21	9.29e5	38.29	1.33	78:	210.16	2.68e5	10.28	0.36					
13:	62.14	4.55e5	17.56	0.61	79:	223.16	3.28e5	12.66	0.44					
14:	63.16	1.38e6	53.16	1.84	80:	225.15	7.95e5	30.70	1.06					
15:	64.13	8.48e5	32.75	1.13	81:	235.17	2.95e5	11.39	0.39					
16:	65.12	9.34e5	36.08	1.25	82:	236.16	2.99e5	11.55	0.40					
17:	66.14	6.06e5	23.42	0.81	83:	237.15	5.49e5	21.20	0.73					
18:	67.13	9.91e5	38.29	1.33	84:	238.15	3.93e5	15.19	0.53					
19:	68.14	5.78e5	22.31	0.77	85:	239.16	4.06e5	15.66	0.54					
20:	69.22	9.91e5	38.29	1.33	86:	251.16	3.52e5	13.61	0.47					
21:	70.37	5.12e5	19.78	0.69	87:	252.16	2.11e6	81.65	2.83					
22:	71.24	6.14e5	23.73	0.82	88:	253.17	7.95e5	30.70	1.06					
23:	74.12	3.32e5	12.82	0.44	89:	263.14	9.05e5	34.97	1.21					
24:	75.12	7.70e5	29.75	1.03	90:	264.14	6.14e5	23.73	0.82					
25:	76.13	1.11e6	43.04	1.49	91:	265.13	4.18e5	16.14	0.56					
26:	77.14	1.95e6	75.32	2.61	92:	278.16	1.19e6	44.94	1.56					
27:	78.11	7.85e5	30.22	1.05	93:	279.15	7.21e5	27.85	0.96					
28:	79.12	3.07e5	11.87	0.41	94:	280.14	1.28e6	48.73	1.69					
29:	81.17	2.61e5	10.09	0.35	95:	281.14	6.72e5	25.95	0.90					
30:	83.28	7.29e5	28.16	0.98	96:	292.10	4.92e5	18.99	0.66					
31:	85.14	4.06e5	15.66	0.54	97:	306.11	3.44e5	13.29	0.46					
32:	87.11	3.07e5	11.87	0.41	98:	307.11	2.59e6	100.00	3.46					
33:	88.12	3.89e5	15.03	0.52	99:	308.12	1.09e6	41.77	1.45					
34:	89.14	7.21e5	27.85	0.96										
35:	90.13	4.96e5	19.15	0.66										
36:	91.12	5.37e5	20.73	0.72										
37:	91.98	2.62e5	10.13	0.35										
38:	97.21	6.18e5	23.89	0.83										
39:	99.07	3.11e5	12.03	0.42										
40:	100.12	2.62e5	10.13	0.35										
41:	101.15	5.08e5	19.62	0.68										
42:	102.15	5.94e5	22.94	0.79										
43:	103.13	5.24e5	20.25	0.70										
44:	104.08	4.55e5	17.56	0.61										
45:	105.05	3.07e5	11.87	0.41										
46:	110.29	2.62e5	10.13	0.35										
47:	111.19	3.32e5	12.82	0.44										
48:	112.10	2.74e5	10.60	0.37										
49:	113.06	5.82e5	22.47	0.78										
50:	114.15	4.01e5	15.51	0.54										
51:	115.18	6.80e5	26.27	0.91										
52:	116.17	3.89e5	15.03	0.52										
53:	117.18	2.79e5	10.76	0.37										
54:	118.11	2.59e5	10.01	0.35										
55:	119.07	2.60e5	10.05	0.35										
56:	125.11	2.86e5	10.28	0.36										
57:	126.15	3.85e5	14.87	0.52										
58:	127.16	6.18e5	23.89	0.83										
59:	128.17	7.19e5	27.53	0.95										
60:	129.17	3.15e5	12.18	0.42										
61:	130.17	3.28e5	12.86	0.44										
62:	132.15	3.24e5	12.50	0.43										
63:	139.14	2.99e5	11.55	0.40										
64:	140.17	6.51e5	25.16	0.87										
65:	141.17	3.89e5	15.03	0.52										
66:	142.18	3.07e5	11.87	0.41										

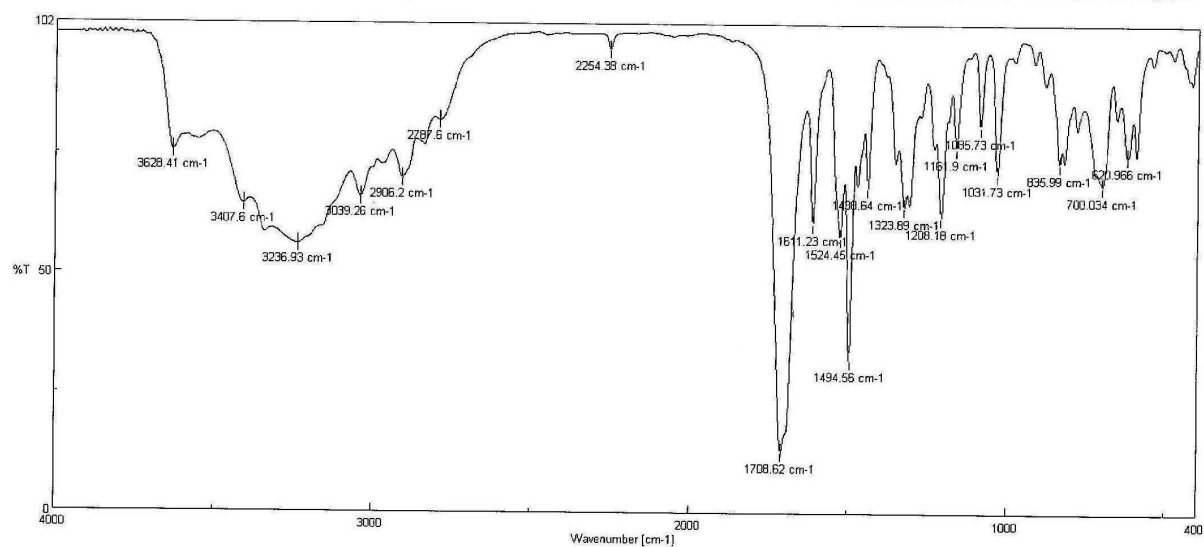
S-16: Sample description presented by mass instrument to compound **4{1,1,2}**

Sample Code: H900830-124 (KE-129)

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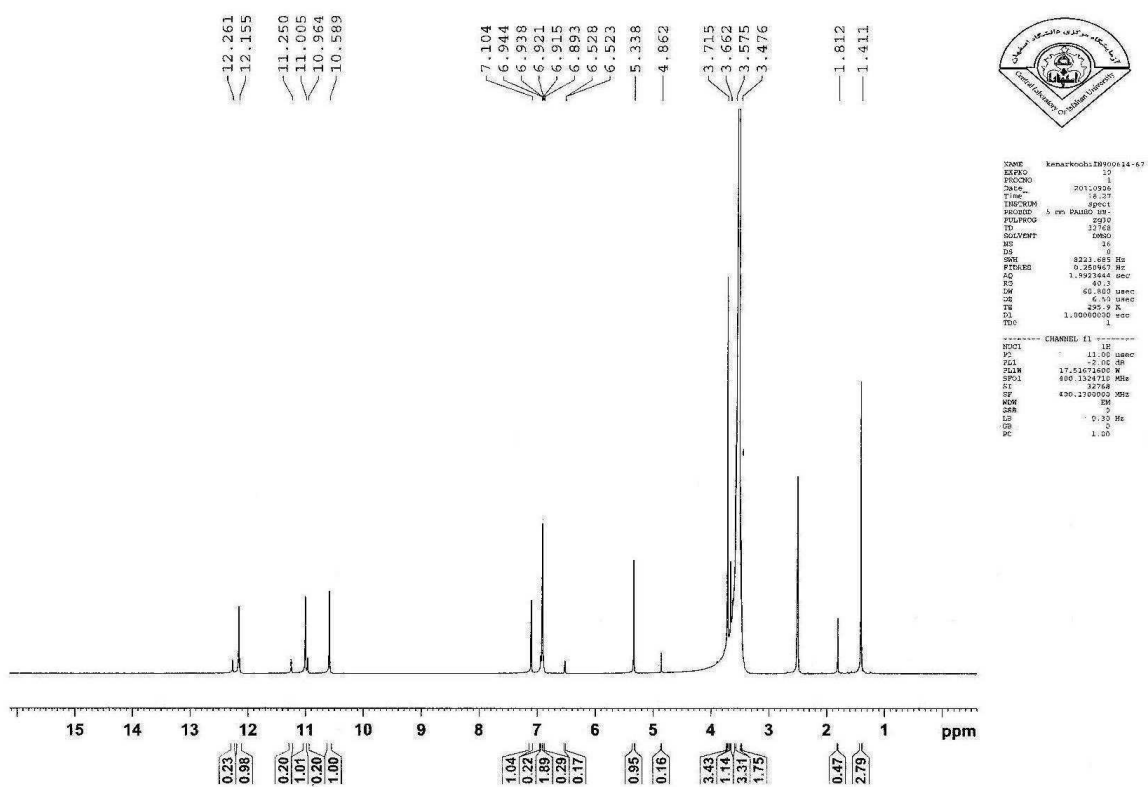
Instrument Specifications

FT Infrared Spectroscopy, JASCO, FT/IR-6300 (400-4000 cm^{-1}), Japan

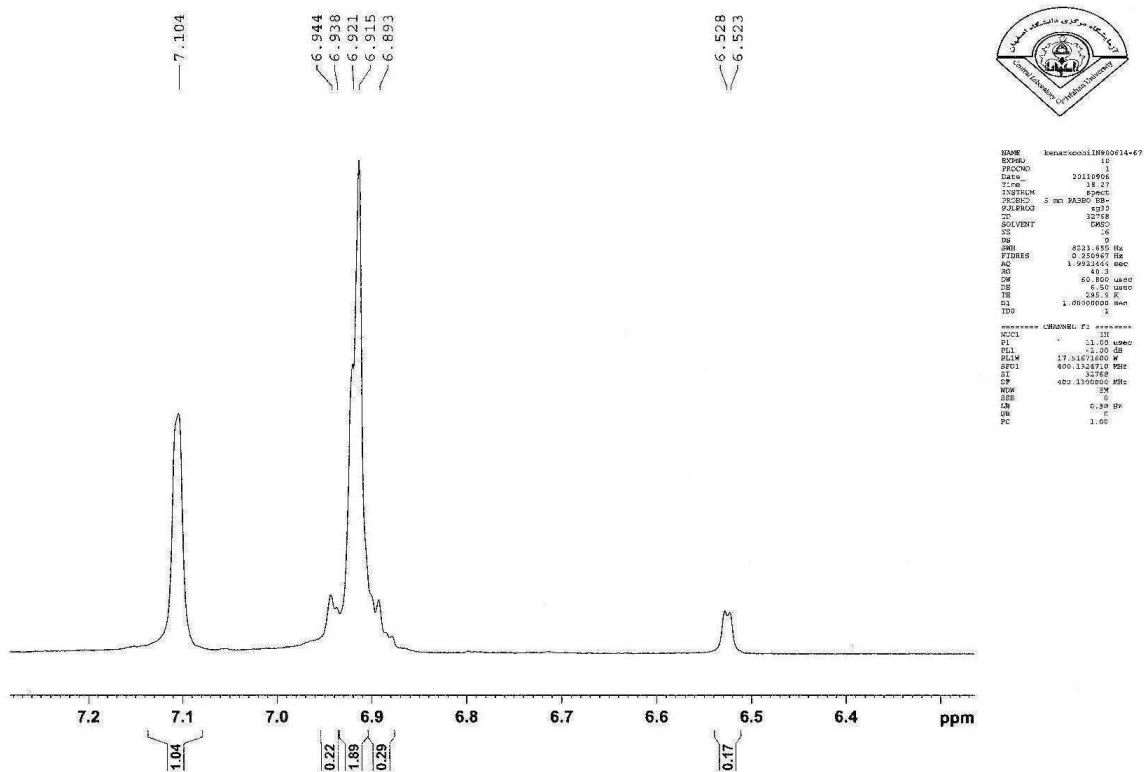


Date: 90/08/30
No.:

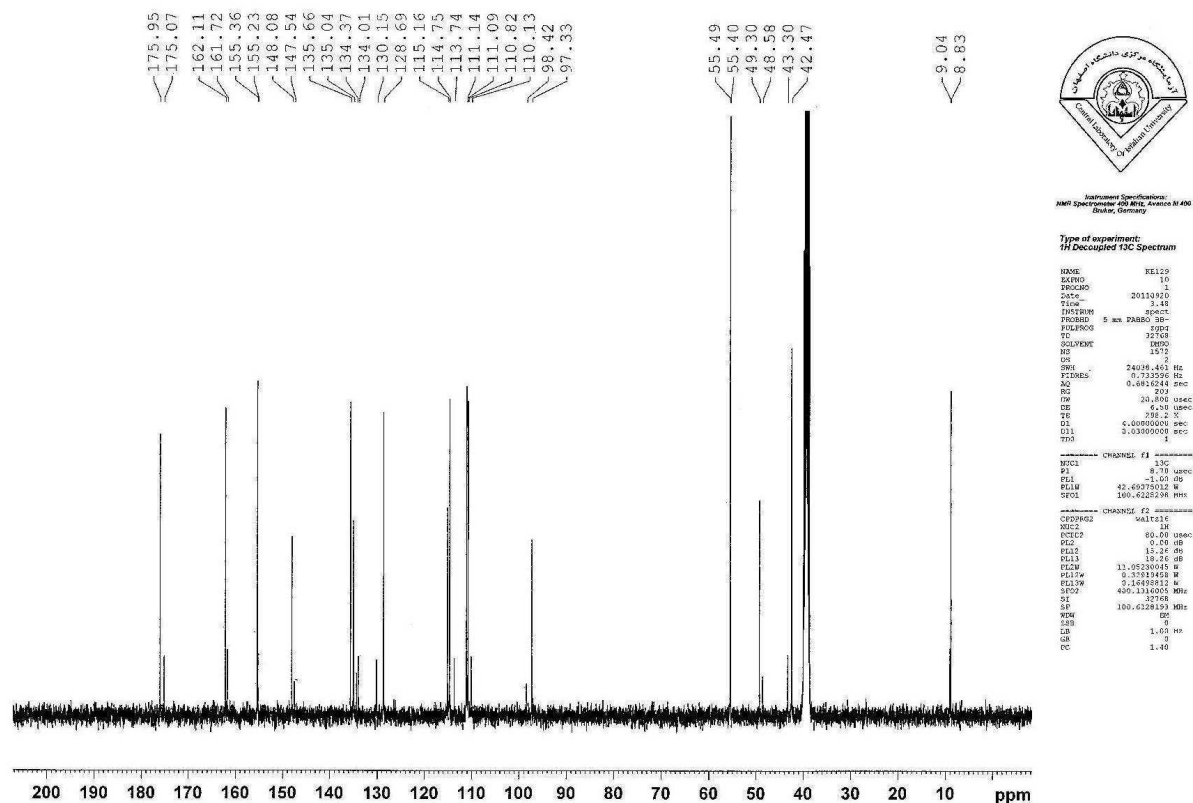
S-17: IR spectrum of compound 4{1,1,3}



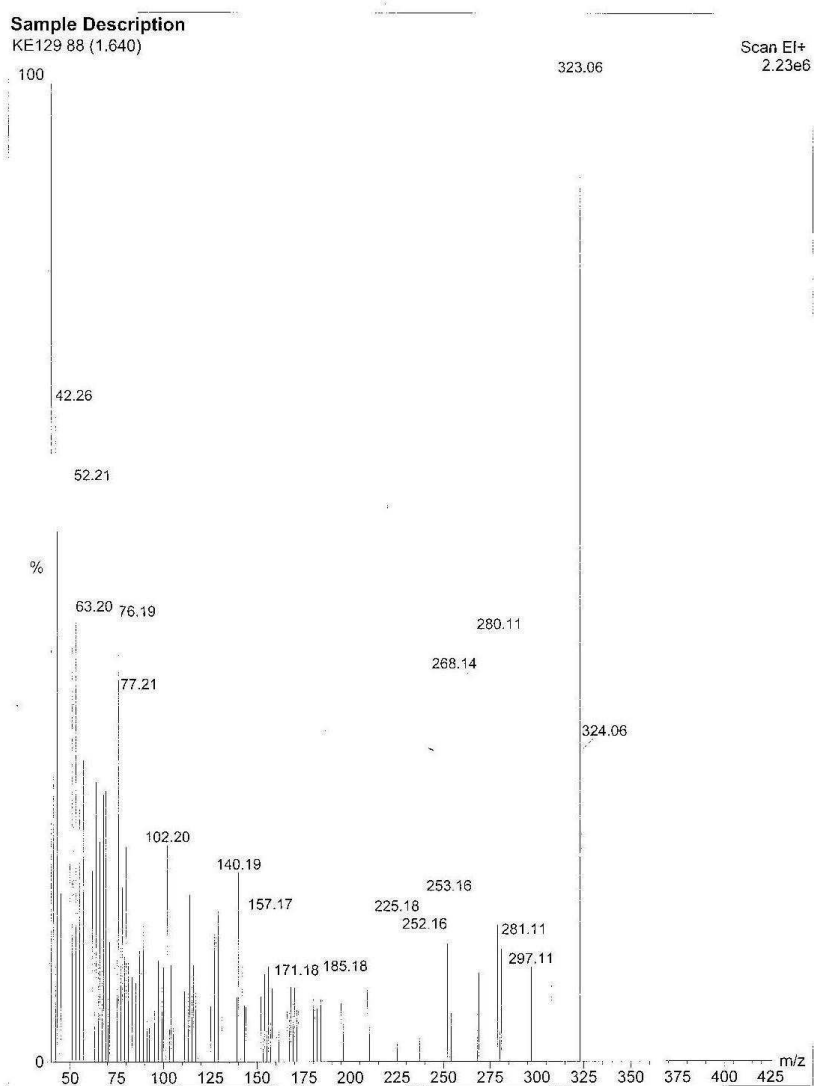
S-18: ^1H NMR spectrum of compound 4{1,1,3}



S-18: Expand of ^1H NMR spectrum of compound 4{1,1,3}



S-19: ^{13}C NMR spectrum of compound 4{1,1,3}



S-20: Mass spectrum of compound 4{1,1,3}

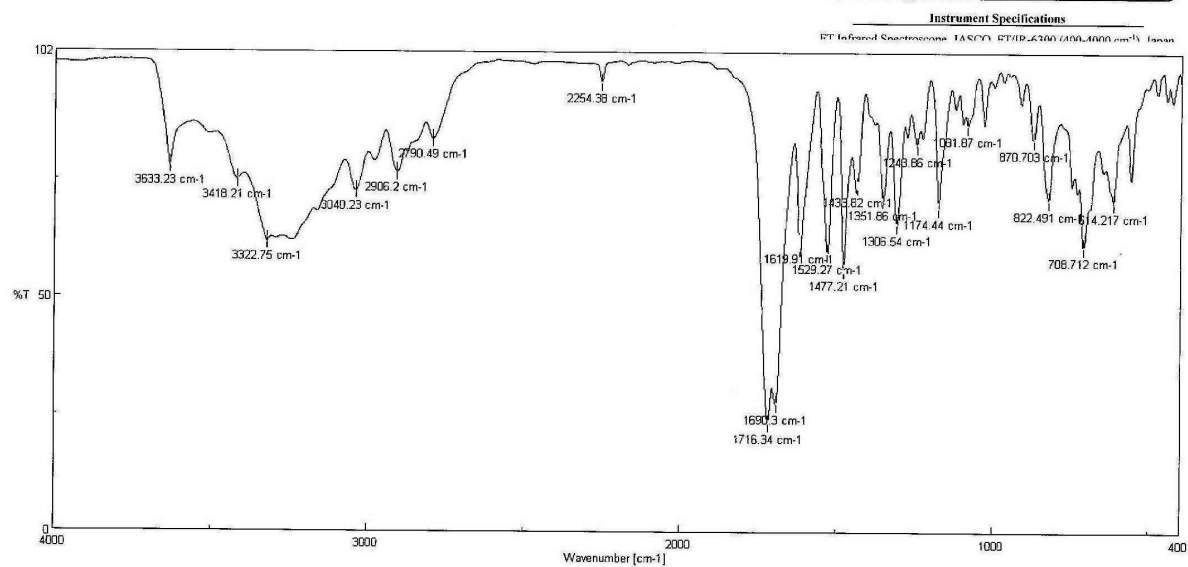
Sample Description
KE129 88 (1.640)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC
1:	40.18	5.41e5	24.26	1.11	67:	129.20	3.40e5	15.26	0.70					
2:	41.27	6.47e5	29.04	1.32	68:	130.19	1.96e5	8.78	0.40					
3:	42.26	1.46e6	65.44	2.98	69:	131.31	1.15e5	5.15	0.23					
4:	43.26	1.20e6	53.68	2.45	70:	132.23	1.38e5	6.20	0.28					
5:	44.21	9.13e5	40.99	1.87	71:	139.20	1.47e5	6.62	0.30					
6:	45.25	3.81e5	17.10	0.78	72:	140.19	4.26e5	19.12	0.87					
7:	46.24	1.60e5	7.17	0.33	73:	141.19	2.70e5	12.13	0.55					
8:	50.23	4.83e5	21.69	0.99	74:	142.20	2.17e5	9.74	0.44					
9:	51.22	9.58e5	43.01	1.96	75:	143.19	1.26e5	5.65	0.26					
10:	52.21	1.28e6	57.35	2.61	76:	144.18	1.23e5	5.51	0.25					
11:	53.21	9.95e5	44.67	2.04	77:	152.19	1.46e5	6.57	0.30					
12:	54.22	3.69e5	16.54	0.75	78:	153.20	2.06e5	9.24	0.42					
13:	55.24	5.69e5	25.55	1.17	79:	154.20	1.97e5	8.82	0.40					
14:	56.28	2.33e5	10.48	0.48	80:	155.19	2.38e5	10.66	0.49					
15:	57.29	6.80e5	30.51	1.39	81:	156.18	2.14e5	9.60	0.44					
16:	62.19	4.30e5	19.30	0.89	82:	157.17	3.36e5	15.07	0.69					
17:	63.20	1.01e6	45.22	2.06	83:	158.20	1.64e5	7.35	0.34					
18:	64.19	6.31e5	28.31	1.29	84:	161.58	1.29e5	5.79	0.26					
19:	65.19	5.82e5	26.10	1.19	85:	166.17	1.14e5	5.10	0.23					
20:	66.19	4.96e5	22.24	1.01	86:	167.20	1.76e5	7.90	0.36					
21:	67.18	7.78e5	34.93	1.59	87:	168.19	1.88e5	7.54	0.34					
22:	68.18	6.02e5	27.02	1.23	88:	169.19	1.75e5	7.86	0.36					
23:	69.23	6.10e5	27.39	1.25	89:	170.18	1.66e5	7.44	0.34					
24:	70.42	2.41e5	10.80	0.49	90:	171.18	1.87e5	8.41	0.38					
25:	71.30	2.70e5	12.13	0.55	91:	172.19	1.14e5	5.10	0.23					
26:	74.18	2.37e5	10.62	0.48	92:	179.18	1.18e5	5.28	0.24					
27:	75.18	5.65e5	25.37	1.16	93:	180.17	1.42e5	6.39	0.29					
28:	76.19	9.71e5	43.57	1.99	94:	181.17	1.16e5	5.19	0.24					
29:	77.21	8.07e5	36.21	1.65	95:	182.18	1.19e5	5.33	0.24					
30:	78.22	3.93e5	17.85	0.89	96:	183.18	1.51e5	6.76	0.31					
31:	79.20	2.42e5	10.85	0.49	97:	184.17	1.55e5	6.84	0.32					
32:	80.18	4.83e5	21.69	0.99	98:	185.18	1.71e5	7.87	0.35					
33:	81.21	2.29e5	10.29	0.47	99:	195.18	1.33e5	5.97	0.27					
34:	83.25	1.91e5	8.59	0.39	100:	196.18	1.14e5	5.10	0.23					
35:	85.25	1.77e5	7.95	0.36	101:	209.19	1.61e5	7.22	0.33					
36:	86.16	1.23e5	5.51	0.25	102:	210.19	1.19e5	5.33	0.24					
37:	87.17	2.52e5	11.31	0.52	103:	225.18	3.32e5	14.89	0.68					
38:	88.19	3.11e5	13.97	0.64	104:	237.17	1.67e5	7.49	0.34					
39:	89.19	3.11e5	13.97	0.64	105:	252.16	2.66e5	11.95	0.54					
40:	90.19	2.70e5	12.13	0.55	106:	253.16	3.77e5	16.91	0.77					
41:	91.15	2.20e5	9.88	0.45	107:	254.15	1.30e5	5.84	0.27					
42:	92.31	2.33e5	10.48	0.48	108:	268.14	8.52e5	38.24	1.74					
43:	95.25	1.19e5	5.33	0.24	109:	269.15	1.99e5	8.92	0.41					
44:	97.29	2.29e5	10.25	0.47	110:	279.13	3.07e5	13.79	0.63					
45:	98.30	1.51e5	6.78	0.31	111:	280.11	9.67e5	43.38	1.98					
46:	99.20	1.68e5	7.54	0.34	112:	281.11	2.54e5	11.40	0.52					
47:	100.18	2.13e5	9.58	0.44	113:	296.10	1.67e5	7.49	0.34					
48:	101.19	3.32e5	14.89	0.68	114:	297.11	2.13e5	9.56	0.44					
49:	102.20	4.87e5	21.88	1.00	115:	308.07	1.74e5	7.81	0.36					
50:	103.19	2.99e5	13.42	0.61	116:	322.05	1.32e5	5.93	0.27					
51:	104.19	2.18e5	9.79	0.45	117:	323.06	2.23e6	100.00	4.56					
52:	105.14	1.57e5	7.03	0.32	118:	324.06	7.00e5	31.43	1.43					
53:	106.23	1.36e5	6.11	0.28										
54:	111.26	1.59e5	7.12	0.32										
55:	112.29	1.78e5	8.00	0.36										
56:	113.21	2.33e5	10.48	0.48										
57:	114.19	3.77e5	16.91	0.77										
58:	115.22	3.03e5	13.60	0.62										
59:	116.21	2.18e5	9.79	0.45										
60:	117.23	1.69e5	7.58	0.35										
61:	118.27	1.35e5	6.07	0.28										
62:	120.23	1.33e5	5.97	0.27										
63:	125.27	1.25e5	5.61	0.26										
64:	126.19	1.81e5	8.13	0.37										
65:	127.17	2.95e5	13.24	0.60										
66:	128.19	3.32e5	14.89	0.68										

S-21: Sample description presented by mass instrument to compound 4{1,1,3}

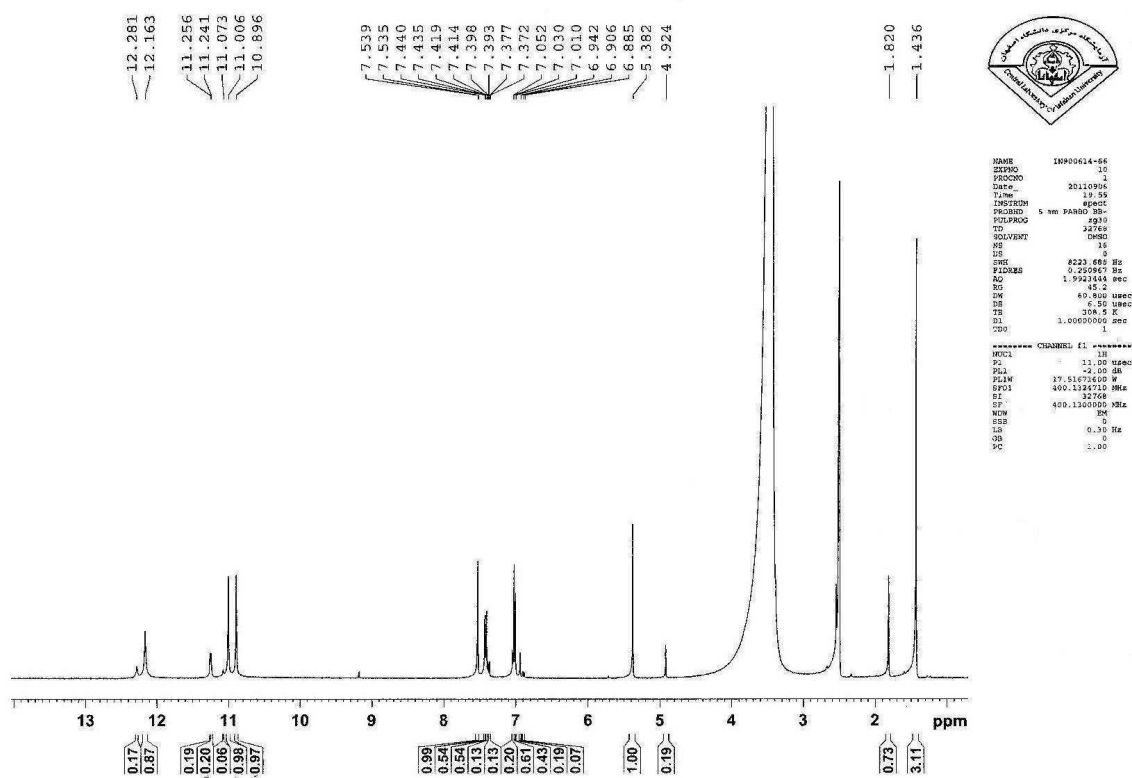
Sample Code: *11900830-123 (KE-128)*

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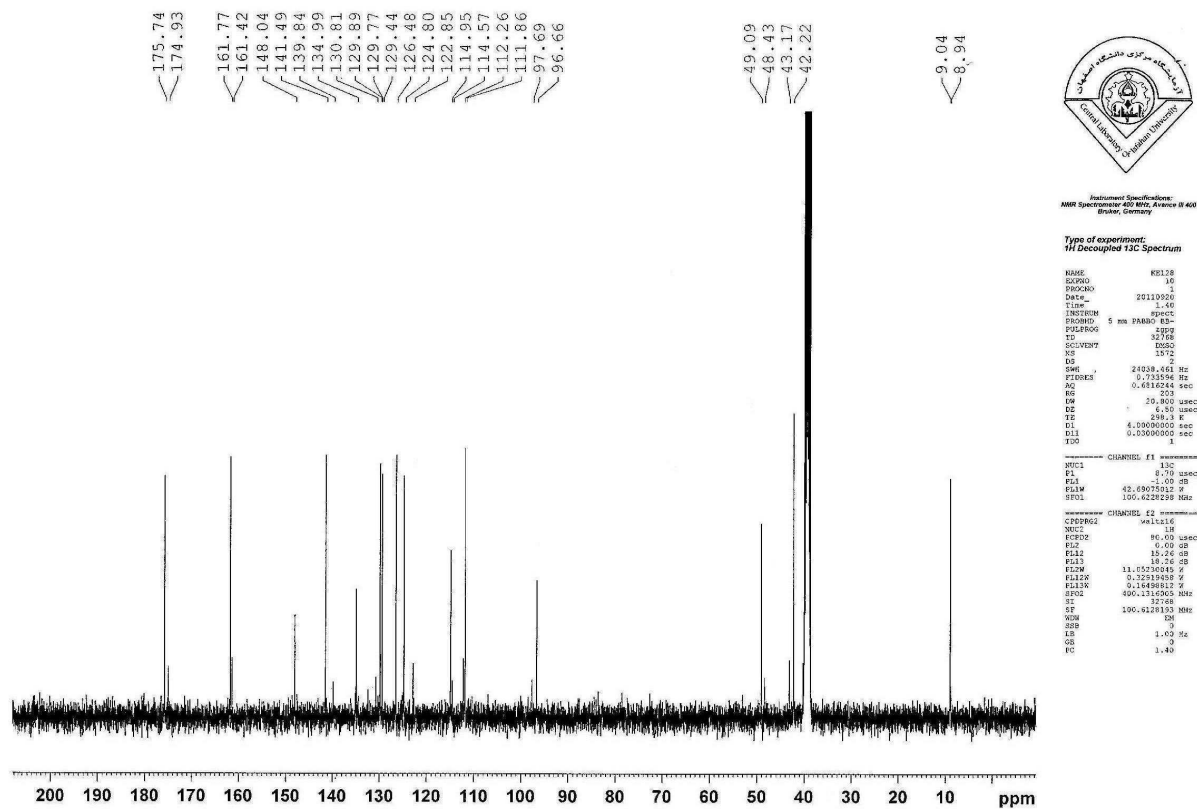


Date: 90/08/30
No.:

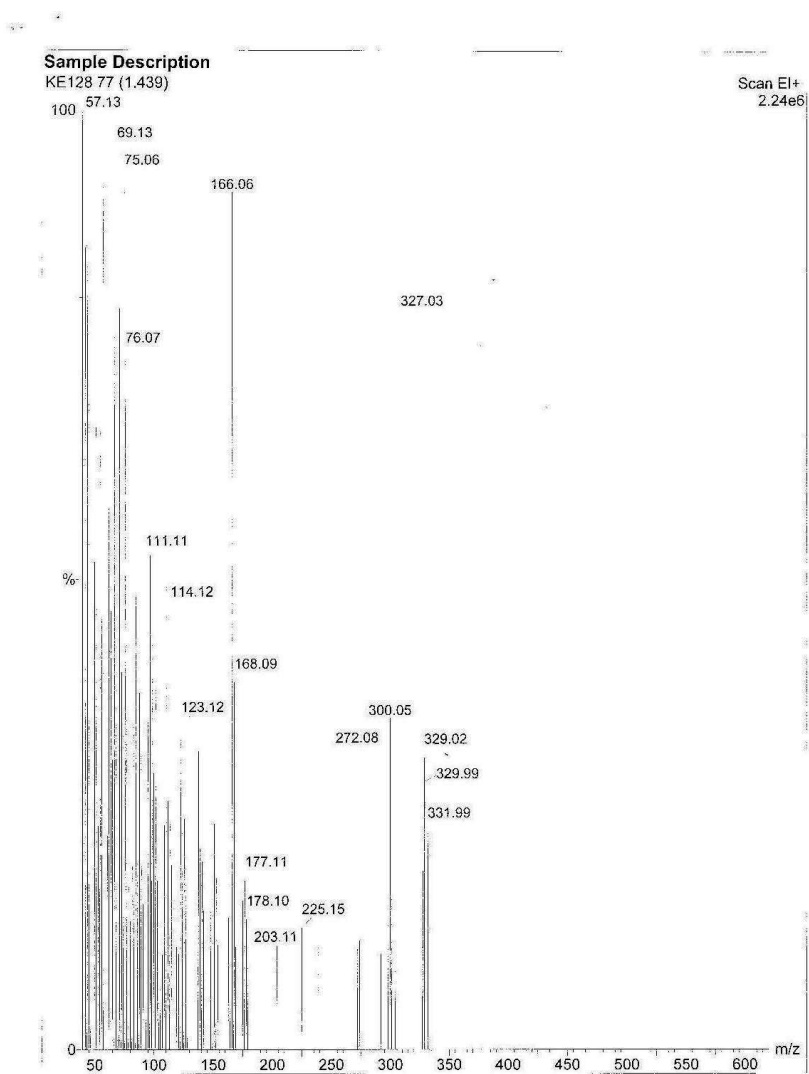
S-22: IR spectrum of compound 4{1,1,4}



S-23: ^1H NMR spectrum of compound 4{1,1,4}



S-24: ^{13}C NMR spectrum of compound 4{1,1,4}



S-25: Mass spectrum of compound **4{1,1,4}**

Sample Description

KE128 77 (1.439)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC
1:	40.05	1.16e6	51.82	1.10	67:	115.12	4.42e5	19.71	0.42					
2:	41.15	1.70e6	75.91	1.61	68:	119.27	2.46e5	10.95	0.23					
3:	42.14	1.92e6	85.40	1.81	69:	121.04	2.29e5	10.22	0.22					
4:	43.15	2.02e6	89.78	1.90	70:	122.09	2.66e5	11.56	0.25					
5:	44.09	1.97e6	87.59	1.86	71:	123.12	7.74e5	34.49	0.73					
6:	45.14	1.56e6	69.34	1.47	72:	124.11	6.06e5	27.01	0.57					
7:	46.13	6.80e5	30.29	0.64	73:	125.11	5.98e5	26.64	0.56					
8:	50.10	1.16e6	51.82	1.10	74:	126.10	5.53e5	24.64	0.52					
9:	51.10	1.49e6	66.42	1.41	75:	127.12	5.41e5	24.09	0.51					
10:	52.09	1.11e6	48.64	1.05	76:	128.10	2.66e5	11.86	0.25					
11:	53.10	5.37e5	23.91	0.51	77:	137.11	3.93e5	17.52	0.37					
12:	54.12	3.85e5	17.15	0.36	78:	138.09	7.13e5	31.75	0.67					
13:	55.13	1.93e6	86.13	1.83	79:	139.10	4.83e5	21.53	0.46					
14:	56.14	1.03e6	45.80	0.97	80:	140.12	7.29e5	32.48	0.69					
15:	57.13	2.24e6	100.00	2.12	81:	141.13	4.51e5	20.07	0.43					
16:	58.12	3.03e5	13.50	0.29	82:	142.11	3.32e5	14.78	0.31					
17:	61.09	5.12e5	22.81	0.48	83:	147.99	3.40e5	15.15	0.32					
18:	62.08	1.29e6	57.66	1.22	84:	149.09	3.69e5	16.42	0.35					
19:	63.08	2.00e6	89.05	1.89	85:	150.10	3.73e5	16.61	0.35					
20:	64.07	1.05e6	46.72	0.99	86:	151.12	5.41e5	24.09	0.51					
21:	65.08	6.92e5	30.84	0.65	87:	152.12	4.34e5	19.34	0.41					
22:	66.08	7.17e5	31.93	0.68	88:	153.12	4.14e5	18.43	0.39					
23:	67.08	2.03e6	90.51	1.92	89:	154.15	2.51e5	11.18	0.24					
24:	68.09	1.03e6	48.18	1.02	90:	163.18	3.15e5	14.05	0.30					
25:	69.13	2.15e6	95.62	2.03	91:	164.10	3.40e5	15.15	0.32					
26:	70.20	9.95e5	44.34	0.94	92:	165.10	3.69e5	16.42	0.35					
27:	71.14	1.77e6	78.83	1.67	93:	166.06	2.05e6	91.24	1.93					
28:	72.08	2.59e5	11.54	0.24	94:	167.10	4.83e5	21.53	0.46					
29:	73.05	9.01e5	40.15	0.85	95:	168.09	8.77e5	39.05	0.83					
30:	74.06	1.21e6	54.01	1.15	96:	169.12	2.46e5	10.95	0.23					
31:	75.06	2.08e6	92.70	1.97	97:	175.09	3.56e5	15.88	0.34					
32:	76.07	1.65e6	73.72	1.56	98:	176.10	3.85e5	17.15	0.36					
33:	77.07	9.95e5	44.34	0.94	99:	177.11	4.06e5	18.07	0.38					
34:	78.07	4.30e5	19.16	0.41	100:	178.10	3.11e5	13.87	0.29					
35:	79.11	4.01e5	17.88	0.38	101:	179.13	2.83e5	12.59	0.27					
36:	80.09	3.07e5	13.69	0.29	102:	203.11	2.51e5	11.18	0.24					
37:	81.13	9.63e5	42.88	0.91	103:	204.11	2.47e5	10.99	0.23					
38:	82.12	4.38e5	19.53	0.41	104:	225.15	2.91e5	12.96	0.27					
39:	83.13	1.18e6	52.55	1.11	105:	239.11	2.50e5	11.13	0.24					
40:	84.10	3.97e5	17.70	0.38	106:	272.08	7.25e5	32.30	0.68					
41:	85.13	1.08e6	48.18	1.02	107:	273.08	2.66e5	11.86	0.25					
42:	86.07	5.41e5	24.09	0.51	108:	274.07	2.61e5	11.63	0.25					
43:	87.08	9.22e5	41.06	0.87	109:	292.09	2.28e5	10.17	0.22					
44:	88.07	8.52e5	37.96	0.80	110:	298.06	4.96e5	22.08	0.47					
45:	89.05	7.74e5	34.49	0.73	111:	299.07	3.11e5	13.87	0.29					
46:	90.07	4.42e5	19.71	0.42	112:	300.05	7.91e5	35.22	0.75					
47:	91.05	3.48e5	15.51	0.33	113:	301.06	3.60e5	16.06	0.34					
48:	93.13	2.79e5	12.41	0.26	114:	302.02	5.98e5	26.64	0.56					
49:	95.13	7.82e5	34.85	0.74	115:	304.02	2.40e5	10.68	0.23					
50:	96.13	4.38e5	19.53	0.41	116:	327.03	1.77e6	78.83	1.67					
51:	97.14	1.18e6	52.55	1.11	117:	328.03	4.26e5	18.98	0.40					
52:	98.11	5.90e5	26.28	0.56	118:	329.02	6.96e5	31.02	0.66					
53:	99.09	9.79e5	43.61	0.92	119:	329.99	6.39e5	28.47	0.60					
54:	100.08	6.59e5	29.38	0.62	120:	331.99	5.20e5	23.18	0.49					
55:	101.10	5.61e5	25.00	0.53										
56:	102.09	8.64e5	38.50	0.82										
57:	103.05	4.06e5	18.07	0.38										
58:	104.08	2.32e5	10.36	0.22										
59:	105.18	3.07e5	13.69	0.29										
60:	107.16	2.27e5	10.13	0.21										
61:	109.14	5.37e5	23.91	0.51										
62:	110.11	5.24e5	23.36	0.50										
63:	111.11	1.20e6	53.28	1.13										
64:	112.09	5.94e5	26.46	0.56										
65:	113.08	8.38e5	37.23	0.79										
66:	114.12	1.05e6	46.72	0.99										

Scan EI+

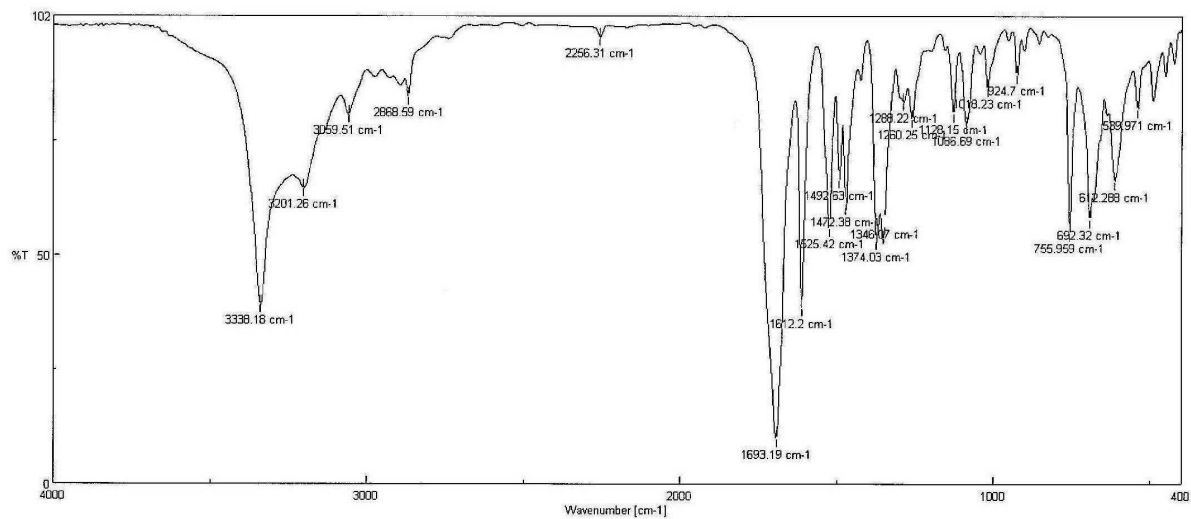
S-26: Sample description presented by mass instrument to compound 4{1,1,4}

Sample Code: H901111-29 (KE-215)

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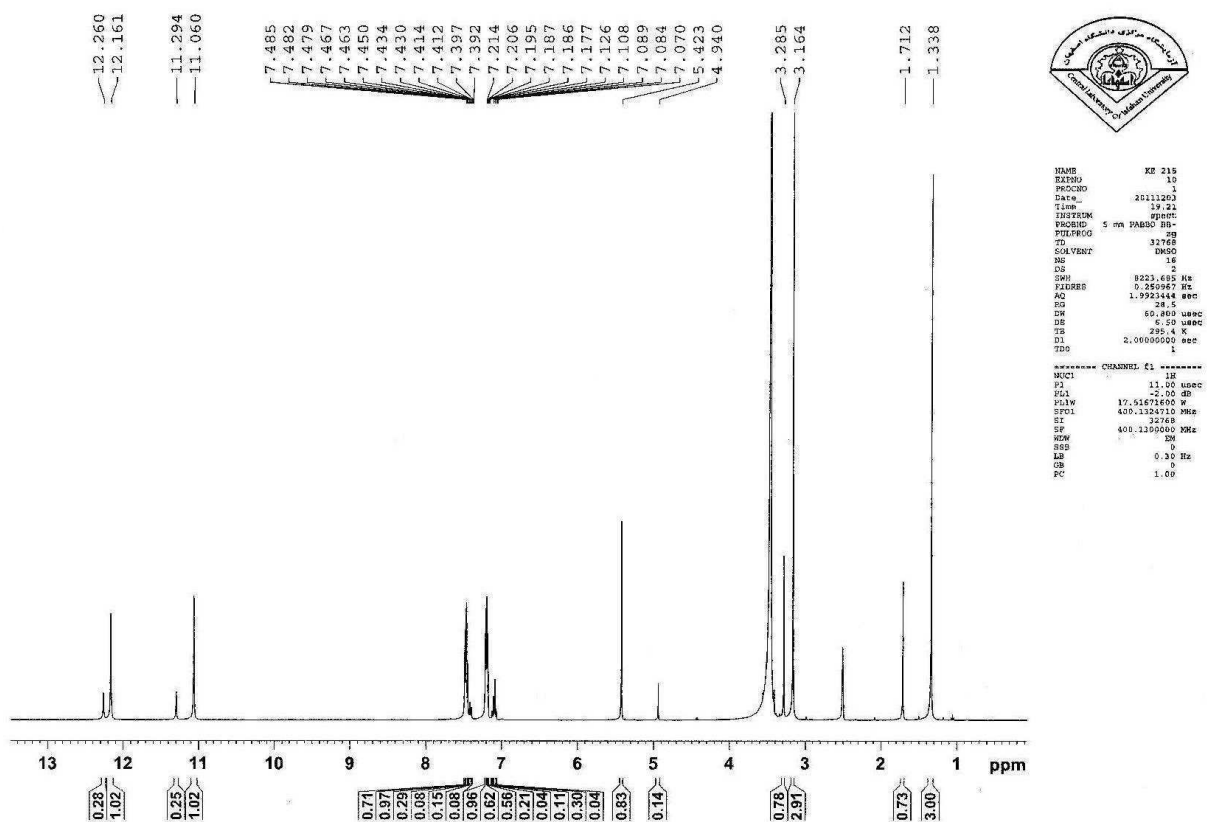
Instrument Specifications

FT Infrared Spectroscope, JASCO, FT/IR-6300 (400-4000 cm^{-1}), Japan

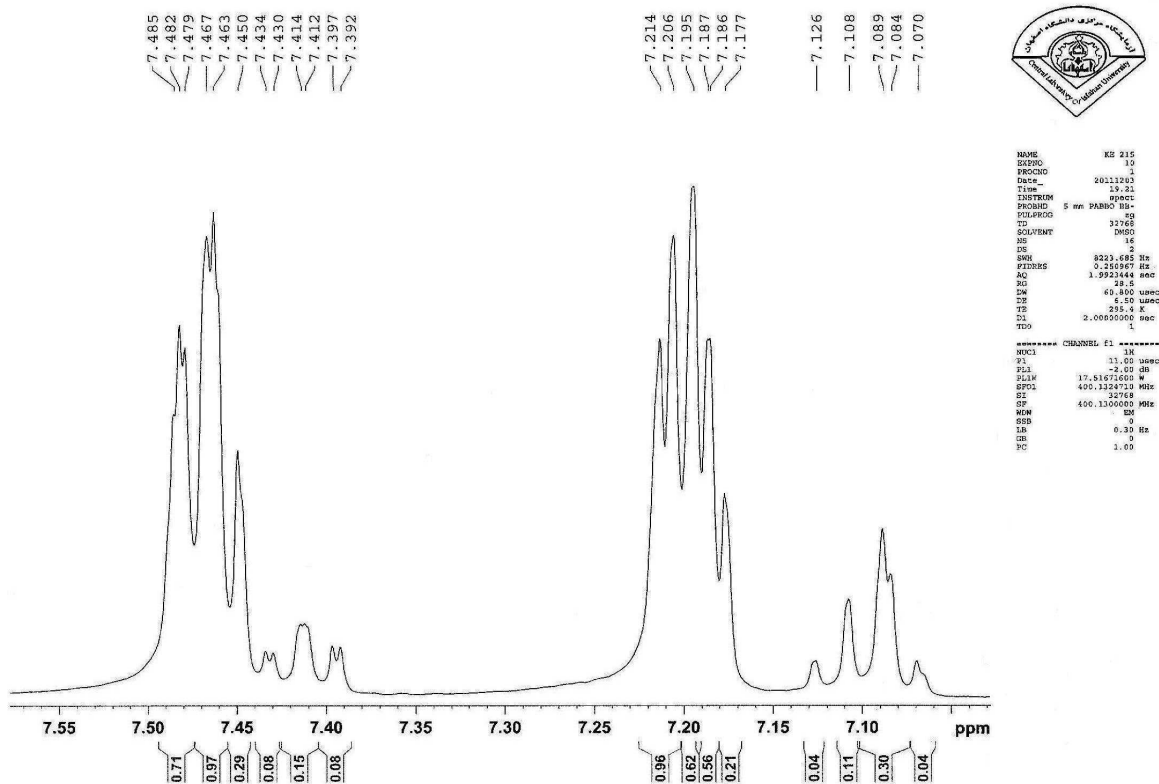


Date: 90/11/11
No.:

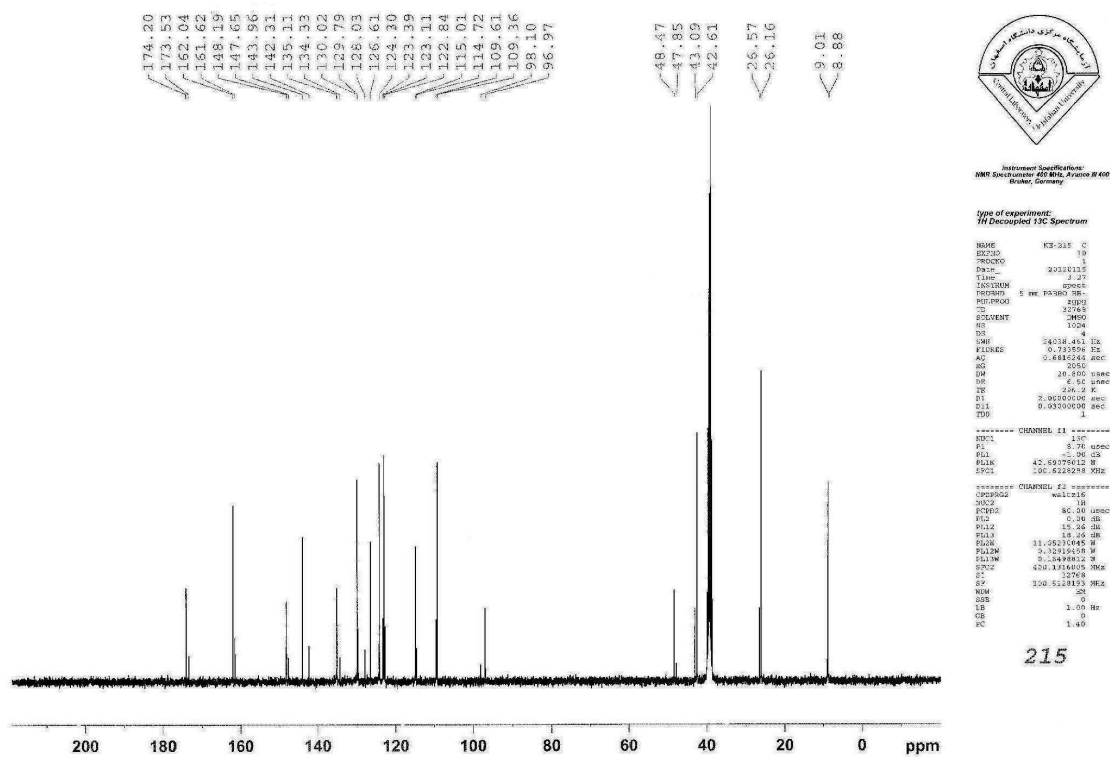
S-27: IR spectrum of compound 4{1,1,5}



S-28: ^1H NMR spectrum of compound 4{1,1,5}



S-28: Expand of ^1H NMR spectrum of compound 4{1,1,5}



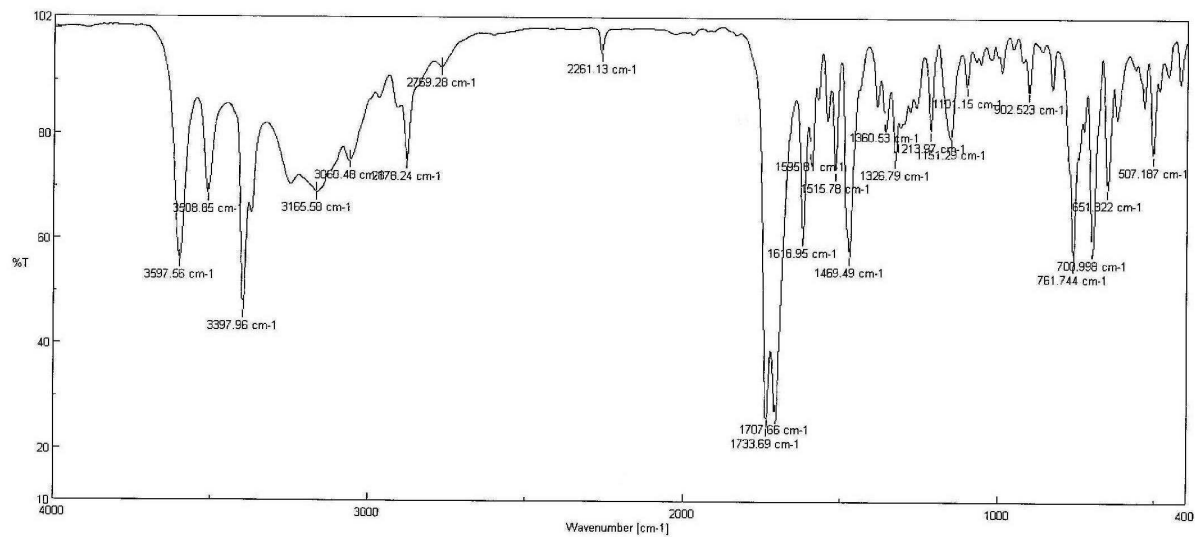
S-29: ^{13}C NMR spectrum of compound 4{1,1,5}

Sample Code: 11900830-127 (KE-180)

Central Laboratory of the University of Isfahan

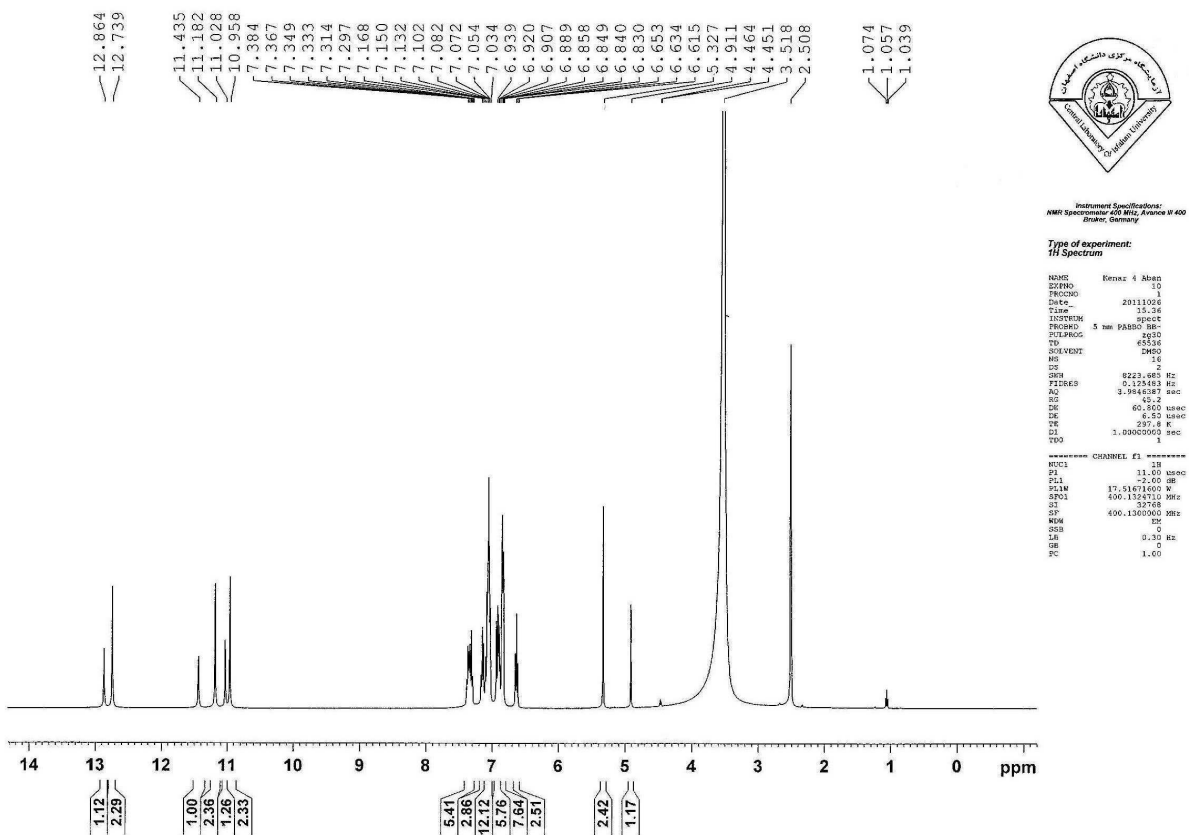
Instrument Specifications

FT Infrared Spectroscopy, JASCO, FT/IR-6300 (400-4000 cm^{-1}), Japan

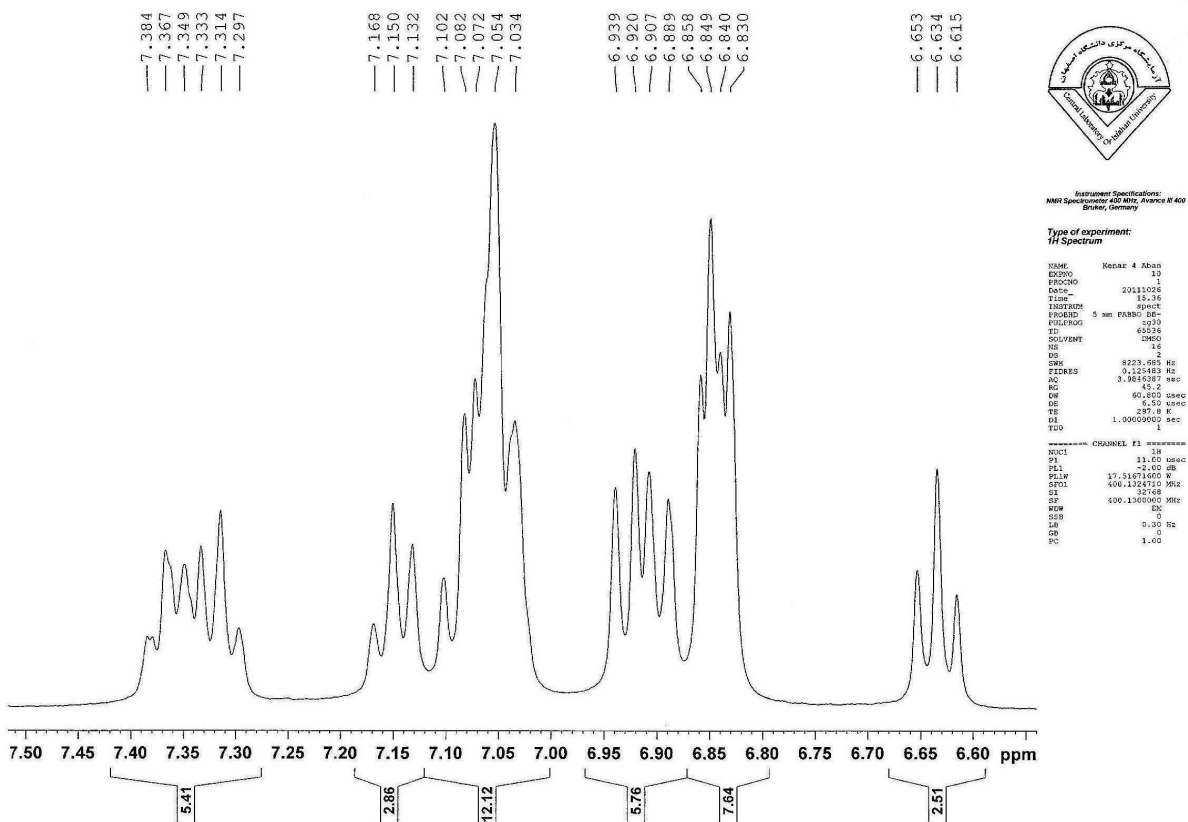


Date: 90/08/30
No.:

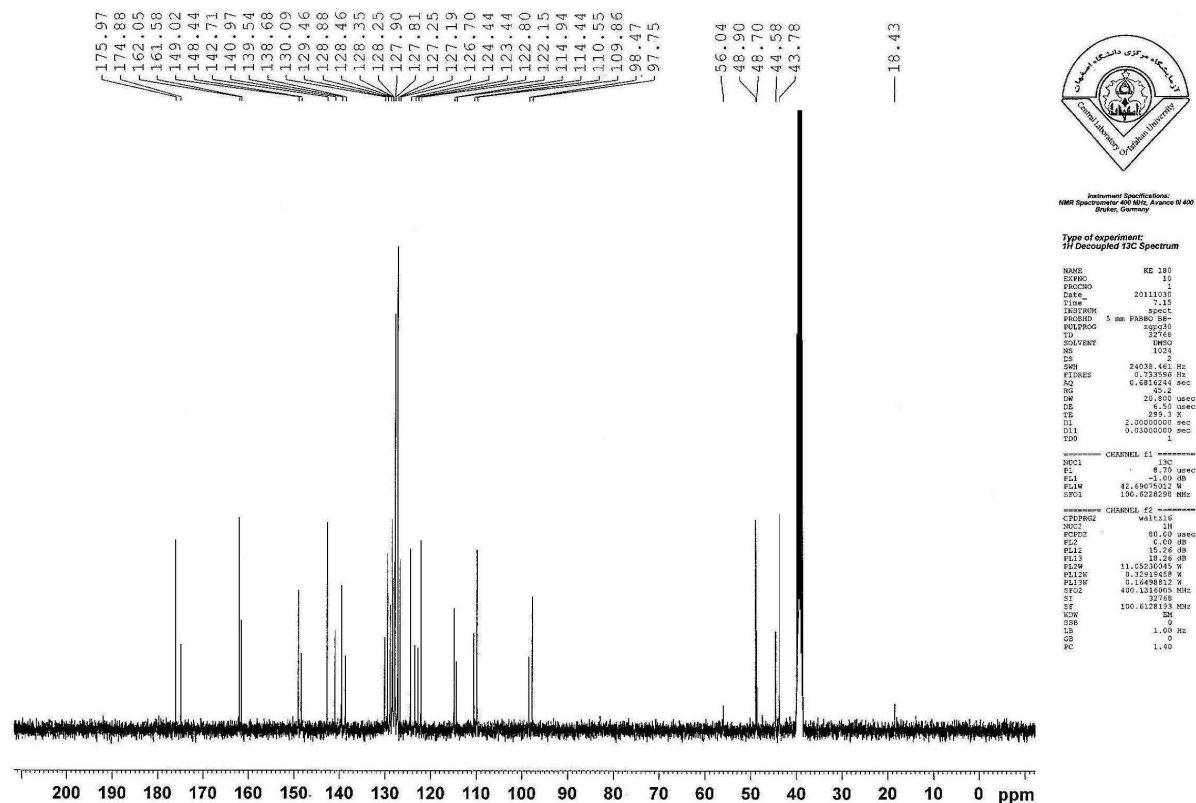
S-30: IR spectrum of compound 4{1,2,1}



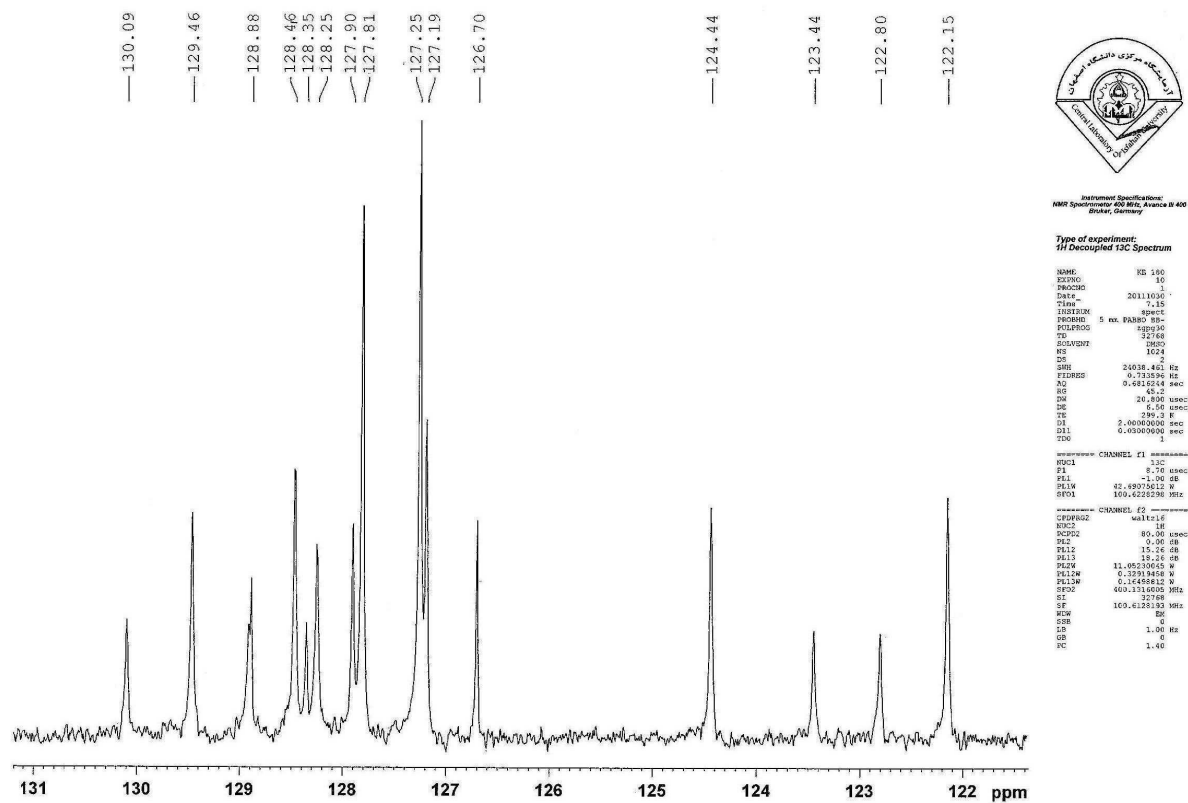
S-31: ^1H NMR spectrum of compound 4{1,2,1}



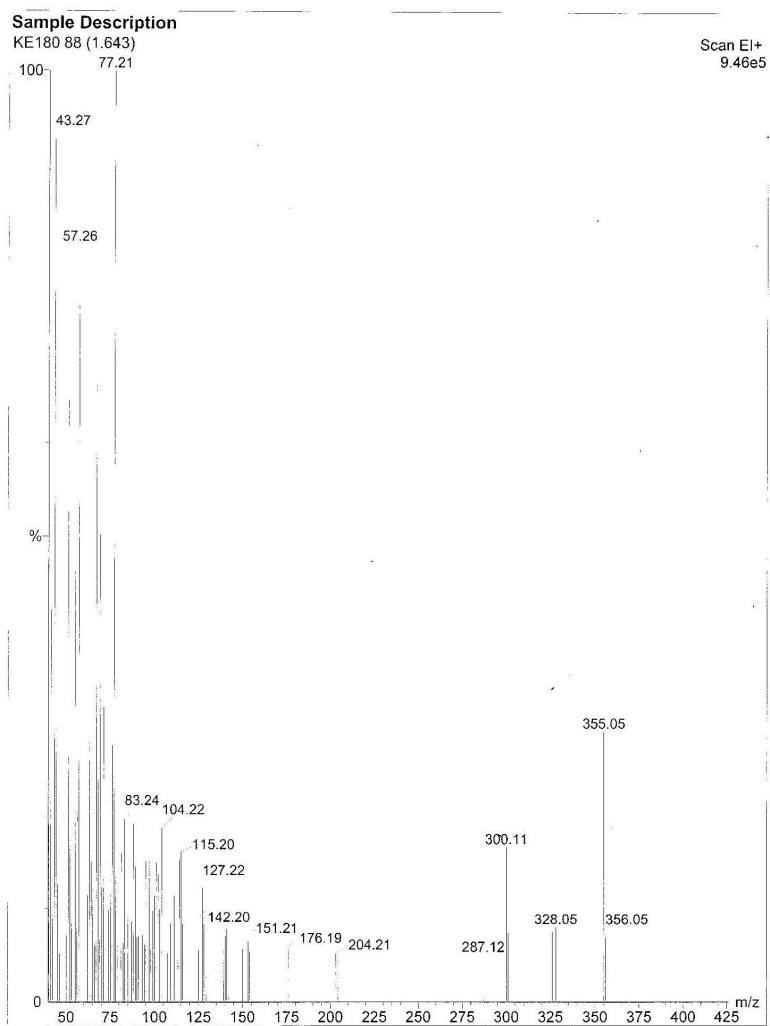
S-31: Expand of ^1H NMR spectrum of compound 4{1,2,1}



S-32: ^{13}C NMR spectrum of compound 4{1,2,1}



S-32: Expand of ^{13}C NMR spectrum of compound 4{1,2,1}



S-33: Mass spectrum of compound **4{1,2,1}**

Sample Description															Scan EI+				
KE180 88 (1.643)																			
No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC
1:	40.19	1.79e5	18.94	1.05	67:	141.22	7.48e4	7.90	0.44										
2:	41.28	3.97e5	41.99	2.32	68:	142.20	7.78e4	8.23	0.45										
3:	42.28	1.56e5	16.45	0.91	69:	150.20	5.35e4	5.65	0.31										
4:	43.27	8.77e5	92.64	5.12	70:	151.21	6.66e4	7.03	0.39										
5:	44.23	2.87e5	30.30	1.67	71:	152.20	5.61e4	5.93	0.33										
6:	45.25	1.19e5	12.55	0.69	72:	153.21	6.09e4	6.44	0.36										
7:	46.24	4.86e4	5.14	0.28	73:	154.22	5.09e4	5.38	0.30										
8:	50.23	1.85e5	19.59	1.08	74:	176.19	5.68e4	5.98	0.33										
9:	51.23	6.10e5	64.50	3.56	75:	203.21	4.89e4	5.17	0.29										
10:	52.22	2.09e5	22.08	1.22	76:	204.21	4.97e4	5.25	0.29										
11:	53.22	9.42e4	9.96	0.55	77:	287.12	4.76e4	5.03	0.28										
12:	54.23	7.07e4	7.47	0.41	78:	300.11	1.58e5	16.67	0.92										
13:	55.25	5.12e5	54.11	2.99	79:	301.10	6.96e4	7.36	0.41										
14:	56.25	1.94e5	20.45	1.13	80:	326.07	7.07e4	7.47	0.41										
15:	57.26	7.70e5	81.39	4.50	81:	327.07	5.04e4	5.33	0.29										
16:	62.19	1.09e5	11.47	0.63	82:	328.05	7.58e4	8.01	0.44										
17:	63.21	2.66e5	28.14	1.55	83:	355.05	2.74e5	29.00	1.60										
18:	64.20	1.43e5	15.15	0.84	84:	356.05	6.50e4	6.87	0.38										
19:	65.21	1.35e5	14.29	0.79															
20:	66.20	5.69e4	6.01	0.33															
21:	67.19	8.27e5	86.23	3.66															
22:	68.20	3.19e5	33.77	1.87															
23:	69.24	4.75e5	50.22	2.77															
24:	70.25	1.17e5	12.34	0.68															
25:	71.25	2.99e5	31.60	1.75															
26:	74.17	9.32e4	9.85	0.54															
27:	75.18	1.86e5	19.70	1.09															
28:	76.17	2.60e5	27.49	1.52															
29:	77.21	9.46e5	100.00	5.53															
30:	78.20	1.34e5	14.18	0.78															
31:	79.22	5.76e4	6.09	0.34															
32:	81.27	1.52e5	16.02	0.88															
33:	82.26	5.94e4	6.28	0.35															
34:	83.24	1.86e5	19.70	1.09															
35:	85.25	1.37e5	14.50	0.80															
36:	87.18	8.09e4	8.55	0.47															
37:	88.19	1.81e5	19.16	1.06															
38:	89.19	1.38e5	14.61	0.81															
39:	90.19	6.50e4	6.87	0.38															
40:	91.20	6.66e4	7.03	0.39															
41:	93.31	6.76e4	7.14	0.39															
42:	94.42	5.79e4	6.11	0.34															
43:	95.28	1.43e5	15.15	0.84															
44:	97.26	1.52e5	16.02	0.88															
45:	98.22	5.27e4	5.57	0.31															
46:	99.23	9.22e4	9.74	0.54															
47:	100.18	1.08e5	11.36	0.63															
48:	101.21	1.42e5	15.04	0.83															
49:	102.19	1.30e5	13.74	0.76															
50:	103.15	9.42e4	9.96	0.55															
51:	104.22	1.77e5	18.72	1.03															
52:	107.39	4.92e4	5.19	0.29															
53:	109.24	7.99e4	8.44	0.47															
54:	111.23	1.08e5	11.36	0.63															
55:	113.24	7.48e4	7.90	0.44															
56:	114.19	1.45e5	15.37	0.85															
57:	115.20	1.53e5	16.13	0.89															
58:	116.23	7.88e4	8.33	0.46															
59:	123.20	5.76e4	6.09	0.34															
60:	125.24	5.22e4	5.52	0.30															
61:	126.19	8.81e4	9.31	0.51															
62:	127.22	1.16e5	12.23	0.68															
63:	128.25	1.15e5	12.12	0.67															
64:	129.18	5.07e4	5.36	0.30															
65:	139.21	5.48e4	5.79	0.32															
66:	140.20	6.66e4	7.03	0.39															

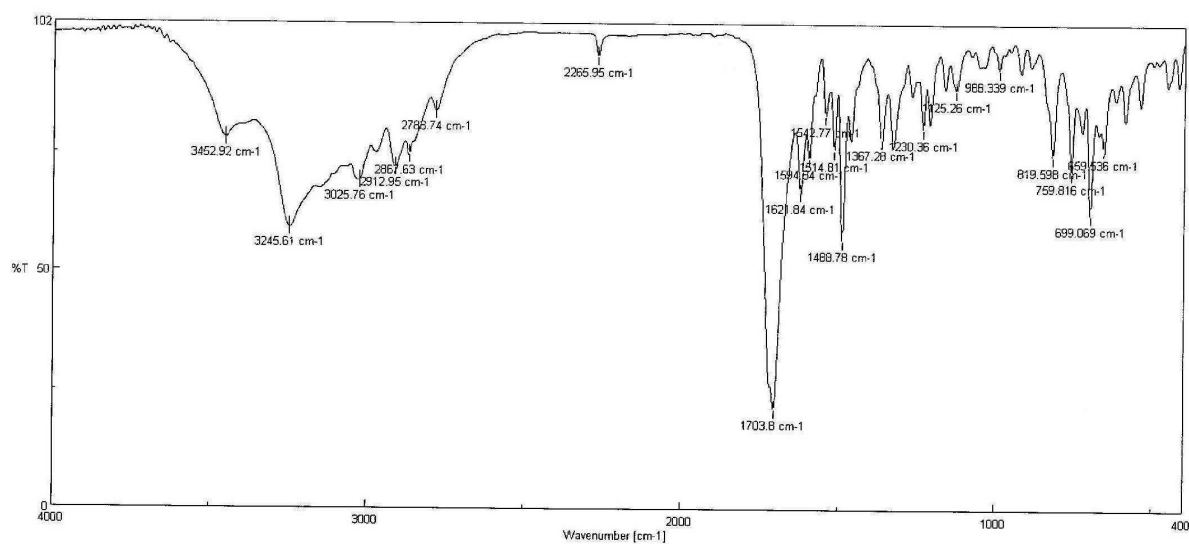
S-34: Sample description presented by mass instrument to compound 4{1,2,1}

Sample Code: *I1900830-128 (KE-198)*

Central Laboratory of the University of Isfahan

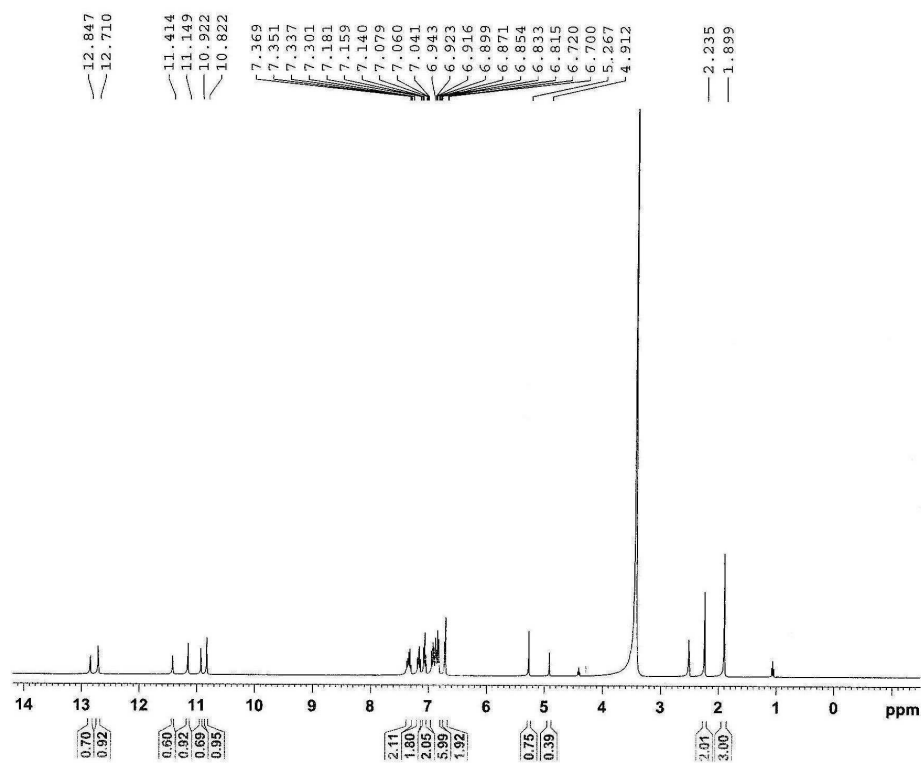
Instrument Specifications

FT Infrared Spectroscopy, JASCO, FT/IR-6300 (400-4000 cm^{-1}), Japan



Date: 90/09/05
No.:

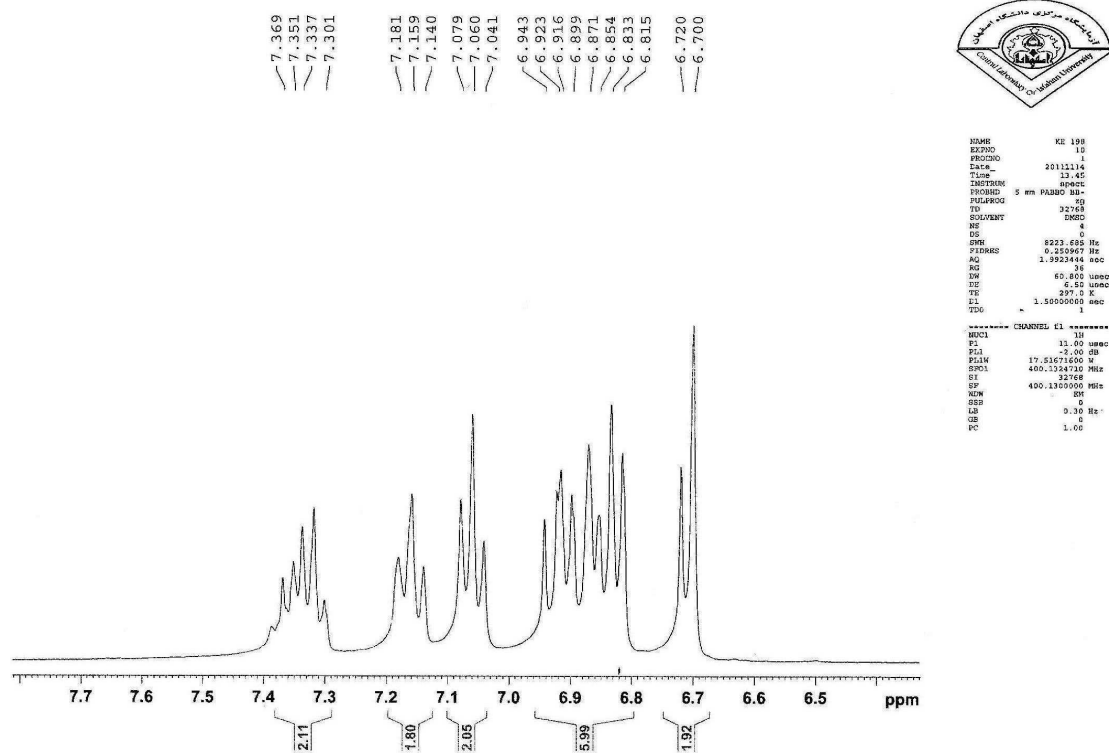
S-35: IR spectrum of compound 4{1,2,2}



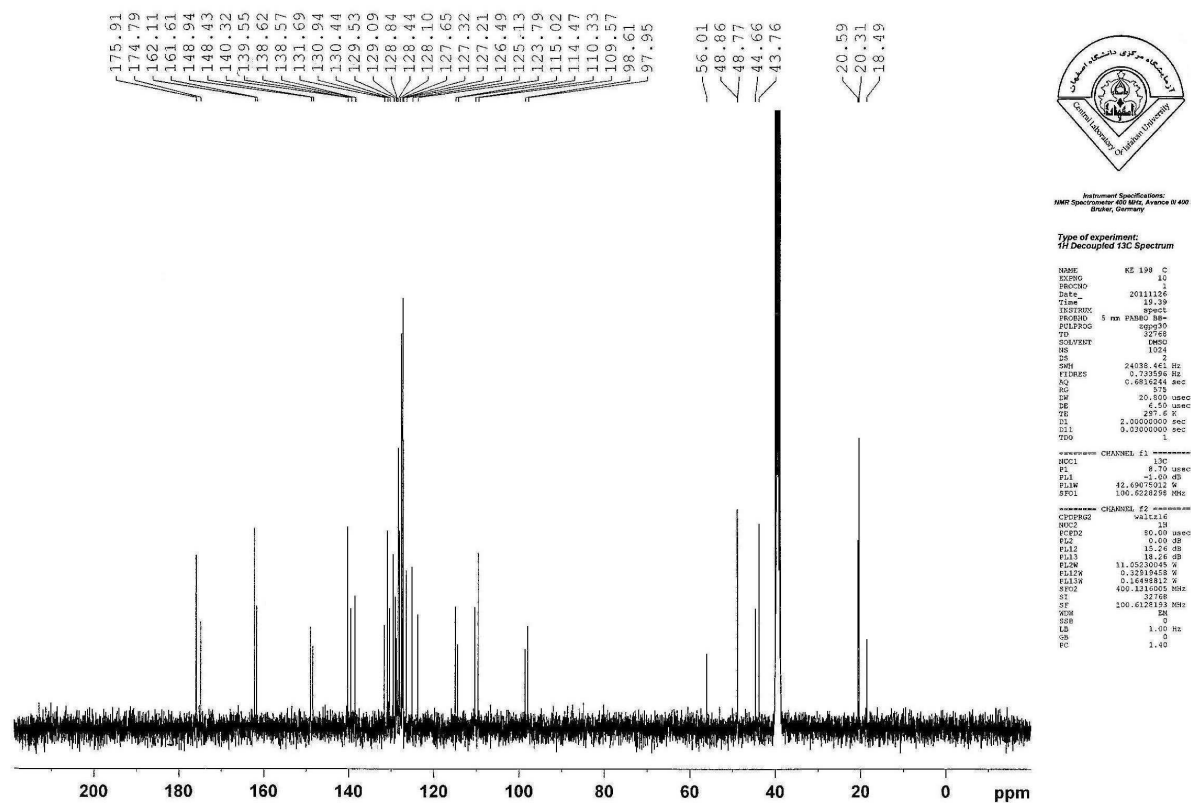
NAME: EE 158
 EXPNO: 1
 PROCNO: 1
 Date_: 20111114
 Time: 13.45
 INSTRUM: spect
 PULPROG: 5 mm WALTZ16
 TD: 32768
 SFO: 400.1306033 MHz
 SOLVENT: DMSO
 NS: 4
 DS: 4
 SWH: 2223.655 Hz
 FIDRES: 0.250967 Hz
 AQ: 1.9921444 sec
 RG: 36
 CW: 60.400 usec
 DE: 6.50 usec
 SE: 297.0 Hz
 LB: 1.50000000 sec
 TDO: 1

===== CHANNEL f1 =====
 NUC1: 1H
 P1: 11.00 usec
 PL1: -2.00 dB
 PL12: 17.51674603 W
 SFO1: 400.1306033 MHz
 SI: 32768
 SF: 400.1306033 MHz
 MDK: DM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00

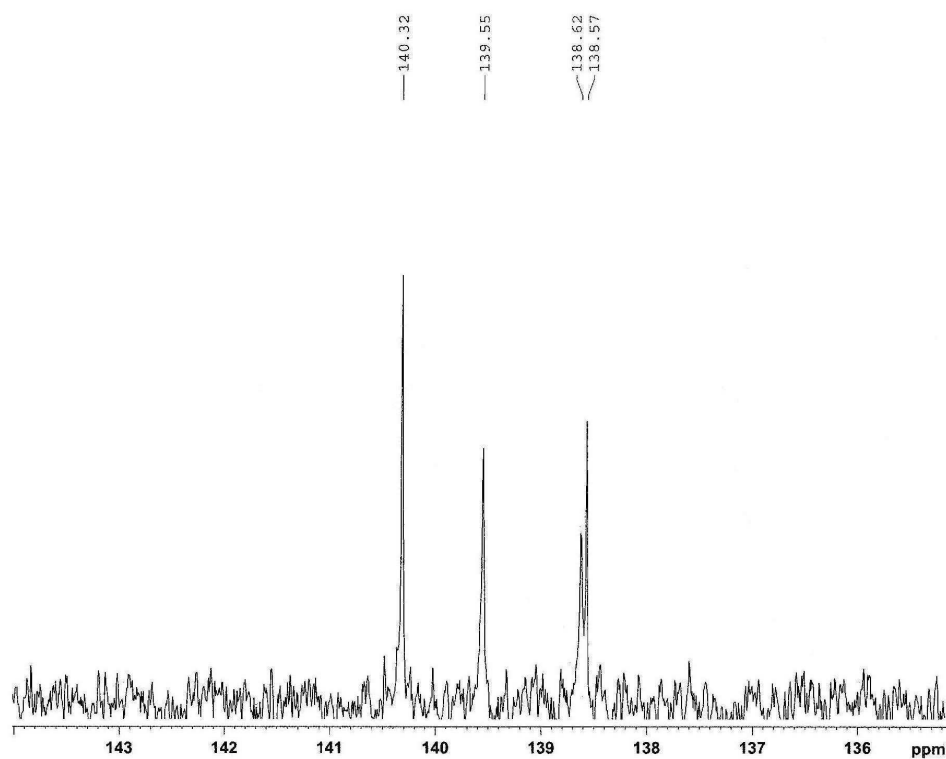
S-36: ^1H NMR spectrum of compound 4{1,2,2}



S-36: Expand of ^1H NMR spectrum of compound **4{1,2,2}**



S-37: ^{13}C NMR spectrum of compound 4{1,2,2}



Instrument Specifications:
NMR Spectrometer 400 MHz, Avance II 400
Bruker, Germany

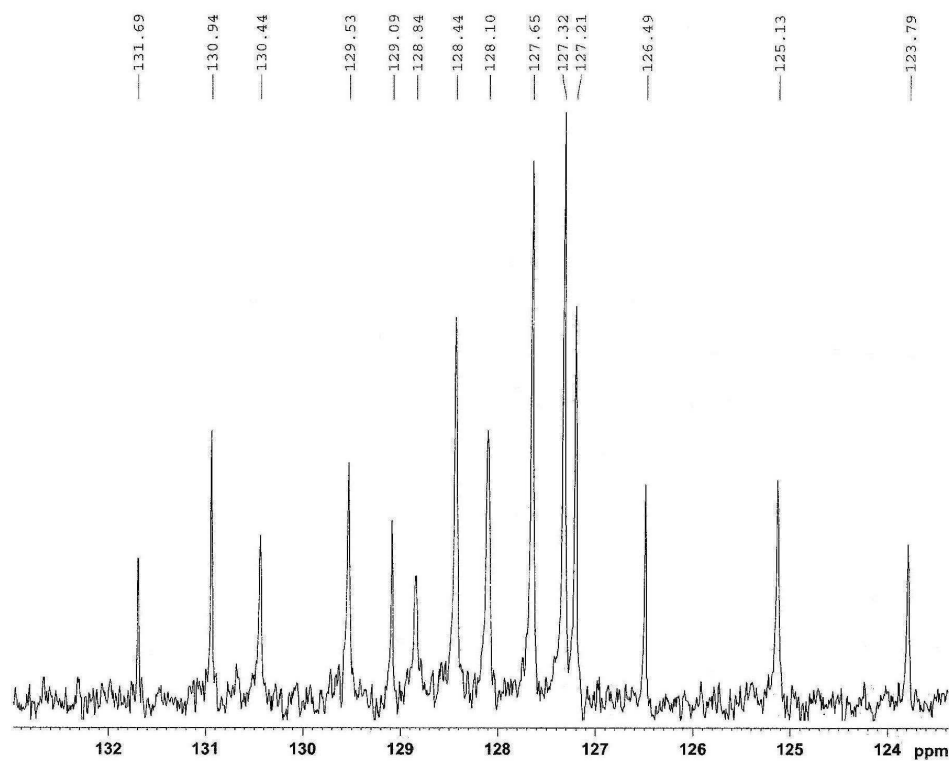
Type of experiment:
1H Decoupled 13C Spectrum

NAME KE 199 C
EXPNO 10
PROCNO 1
DATA 20111118
TIME 22.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 1024
DS 2
SWH 24939.461 Hz
FIDRES 0.733396 Hz
AQ 0.6816244 sec
RG 175
RM 20.800 usec
SE 6.50 usec
TE 297.6 K
D1 2.0000000 sec
D11 0.0200000 sec
TD0 1

----- CHANNEL F1 -----
NUC1 13C
P1 8.70 usec
PL1 -1.20 dB
SFO1 42.6975012 MHz
SFO2 100.6262998 MHz

----- CHANNEL F2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.24 dB
PL13 18.24 dB
PCPD 11.0820040 MHz
PL12W 0.32919448 W
PL13W 0.16459712 W
SFO2 400.1314005 MHz
SI 32768
SF 100.6182133 MHz
RM 305
SE2 0
LA 1.60 Hz
GB 0
PC 1.60

S-37: Expand of ^{13}C NMR spectrum of compound 4{1,2,2}



Instrument Specifications:
NMR Spectrometer: 400 MHz, Avance II 400
Bruker, Germany

Type of experiment:
1D Decoupled ^{13}C Spectrum

```
NAME      KE 188 C
EXPNO     1
PROCNO    1
F2 -      20111128
TIME      22.09
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        32768
SOLVENT   DMSO
NS         1024
DS         2
FIDRES    24038.461 Hz
AQ         0.733896 Hz
RG         675
SW         20.800 MHz
DE         6.50 MHz
TE         297.6 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

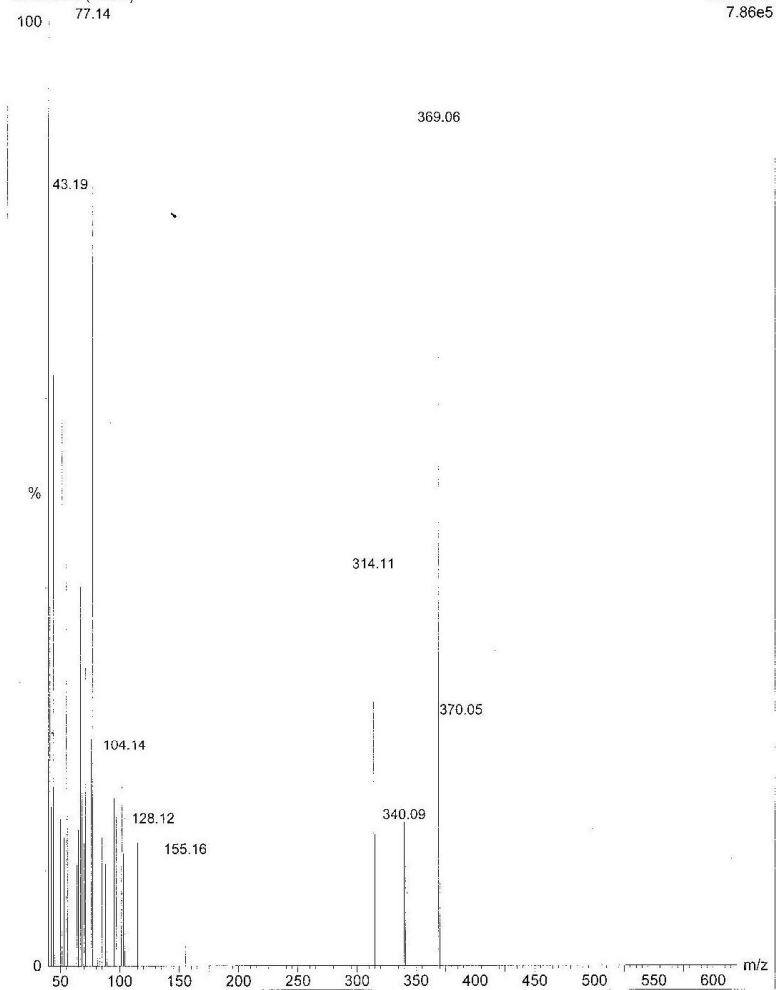
===== CHANNEL f1 =====
NUC1       13C
P1         8.10 MHz
PL1        -1.00 dB
PL1H       42.69075012 W
RF1        100.6228899 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 MHz
PL2        0.00 dB
PL2H       15.30 dB
PL12       16.26 dB
PL12H      11.08370045 W
PL12M      0.32939458 W
PL12M      0.16499812 W
RF12       400.3160005 MHz
ST         32768
SF         400.6128893 MHz
WDW         EM
GB         0
GB         1.00 Hz
PC         1.40
```

S-37: Expand of ^{13}C NMR spectrum of compound 4{1,2,2}

Sample Description
KE198 76 (1.421)

Scan EI+
7.86e5



S-38: Mass spectrum of compound 4{1,2,2}

Sample Description										Scan EI+				
KE198 76 (1.421)					No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC
No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC
1:	40.08	1.72e5	21.88	1.07										
2:	41.20	2.99e5	38.02	1.87										
3:	42.19	1.32e5	16.80	0.82										
4:	43.19	6.35e5	80.73	3.96										
5:	44.14	4.92e5	62.50	3.07										
6:	45.17	1.46e5	18.62	0.91										
7:	50.15	1.22e5	15.49	0.76										
8:	51.15	4.55e5	57.81	2.84										
9:	52.14	2.05e5	26.04	1.28										
10:	53.14	1.06e5	13.54	0.66										
11:	55.17	3.93e5	50.00	2.45										
12:	56.18	1.39e5	17.71	0.87										
13:	57.18	5.61e5	71.35	3.50										
14:	63.13	1.94e5	24.61	1.21										
15:	64.12	8.40e4	10.68	0.52										
16:	65.14	1.13e5	14.32	0.70										
17:	67.12	3.15e5	40.10	1.97										
18:	68.12	2.02e5	25.65	1.26										
19:	69.17	3.60e5	45.83	2.25										
20:	70.16	1.01e5	12.89	0.63										
21:	71.18	2.49e5	31.64	1.55										
22:	75.11	1.37e5	17.45	0.86										
23:	76.11	1.89e5	24.09	1.18										
24:	77.14	7.86e5	100.00	4.91										
25:	78.14	1.27e5	16.15	0.79										
26:	81.21	1.37e5	17.45	0.86										
27:	83.14	1.65e5	20.96	1.03										
28:	85.19	1.06e5	13.54	0.66										
29:	88.12	8.40e4	10.68	0.52										
30:	89.11	1.12e5	14.19	0.70										
31:	94.42	7.99e4	10.16	0.50										
32:	95.28	1.39e5	17.71	0.87										
33:	97.19	1.24e5	15.76	0.77										
34:	101.10	1.18e5	14.97	0.73										
35:	102.09	1.34e5	17.06	0.84										
36:	103.10	9.32e4	11.85	0.58										
37:	104.14	1.77e5	22.53	1.11										
38:	111.19	7.88e4	10.03	0.49										
39:	114.12	8.70e4	11.07	0.54										
40:	115.12	1.02e5	13.02	0.64										
41:	128.12	1.16e5	14.71	0.72										
42:	140.18	8.70e4	11.07	0.54										
43:	155.16	9.01e4	11.46	0.56										
44:	314.11	3.28e5	41.67	2.04										
45:	315.10	1.09e5	13.80	0.68										
46:	340.09	1.19e5	15.10	0.74										
47:	341.09	1.01e5	12.89	0.63										
48:	342.07	1.09e5	13.80	0.68										
49:	369.06	7.00e5	89.06	4.37										
50:	370.05	1.98e5	25.13	1.23										

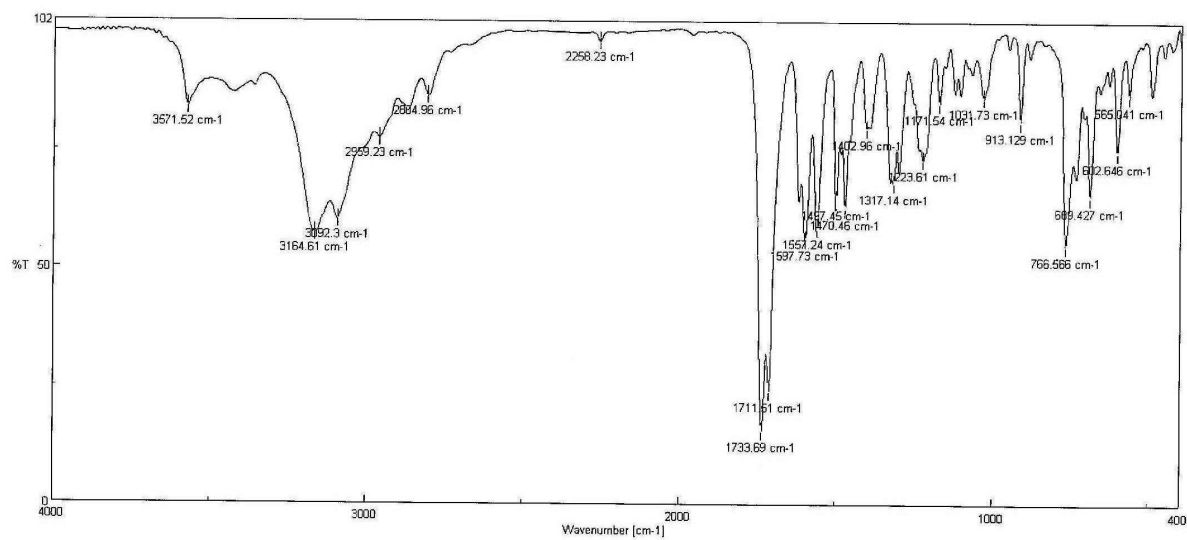
S-39: Sample description presented by mass instrument to compound 4{1,2,2}

Sample Code: H900830-129 (KE-183)

Central Laboratory of the University of Isfahan

Instrument Specifications

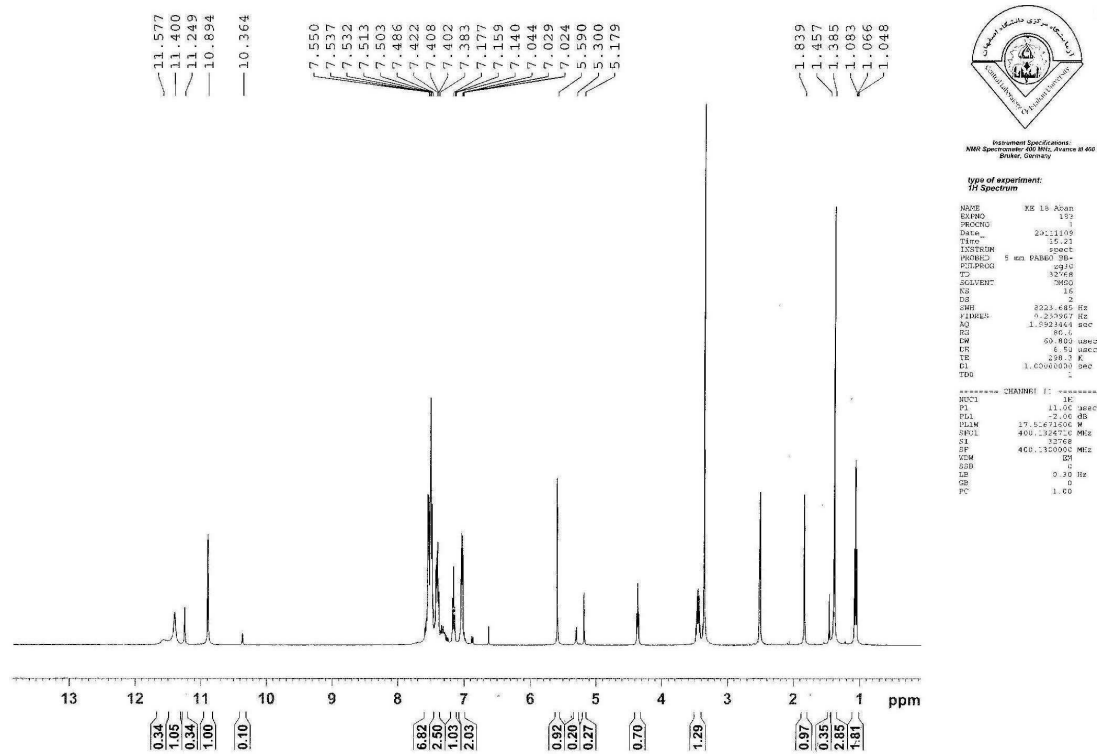
FT Infrared Spectroscopy, JASCO, FT/IR-6300 (400-4000 cm^{-1}), Japan



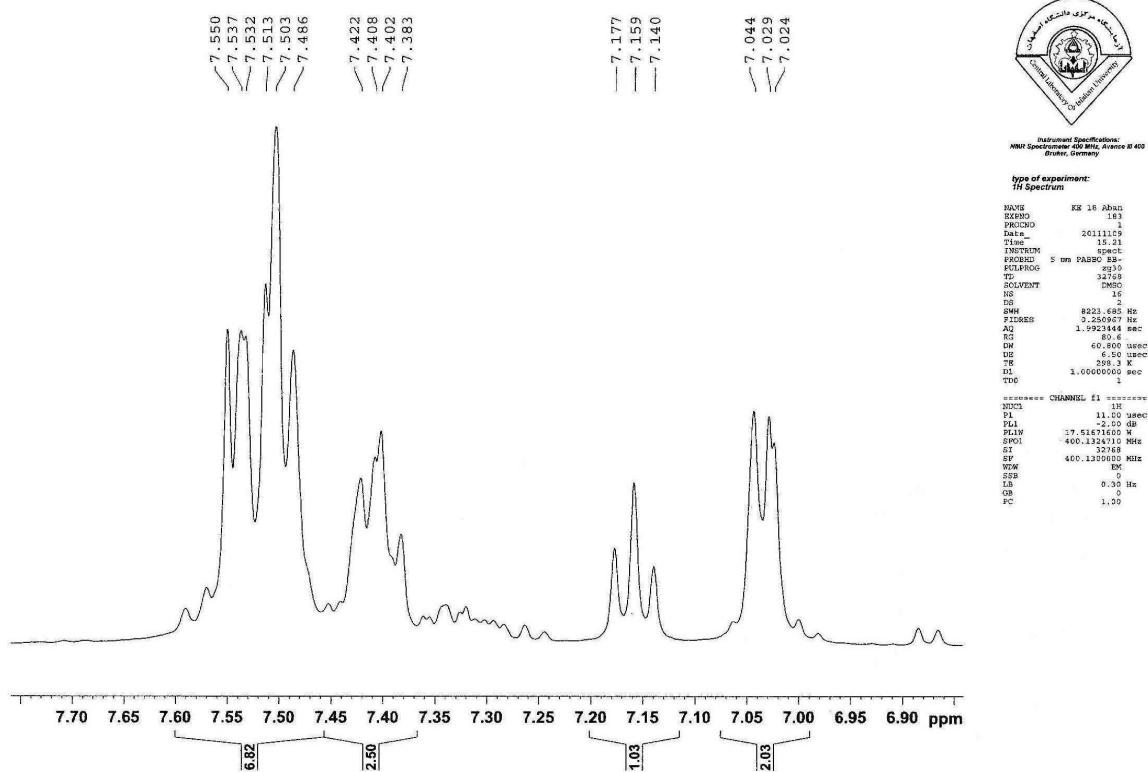
Date: 90/08/30

No.:

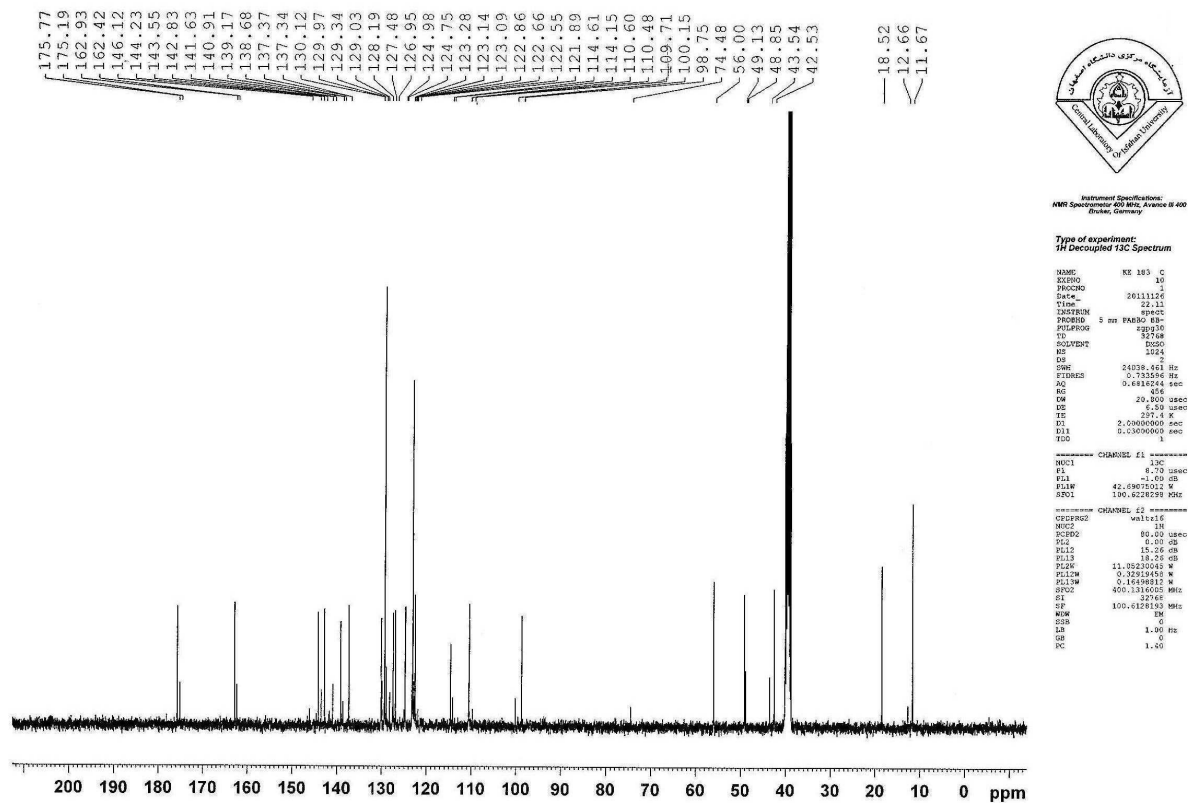
S-40: IR spectrum of compound 4{1,3,1}



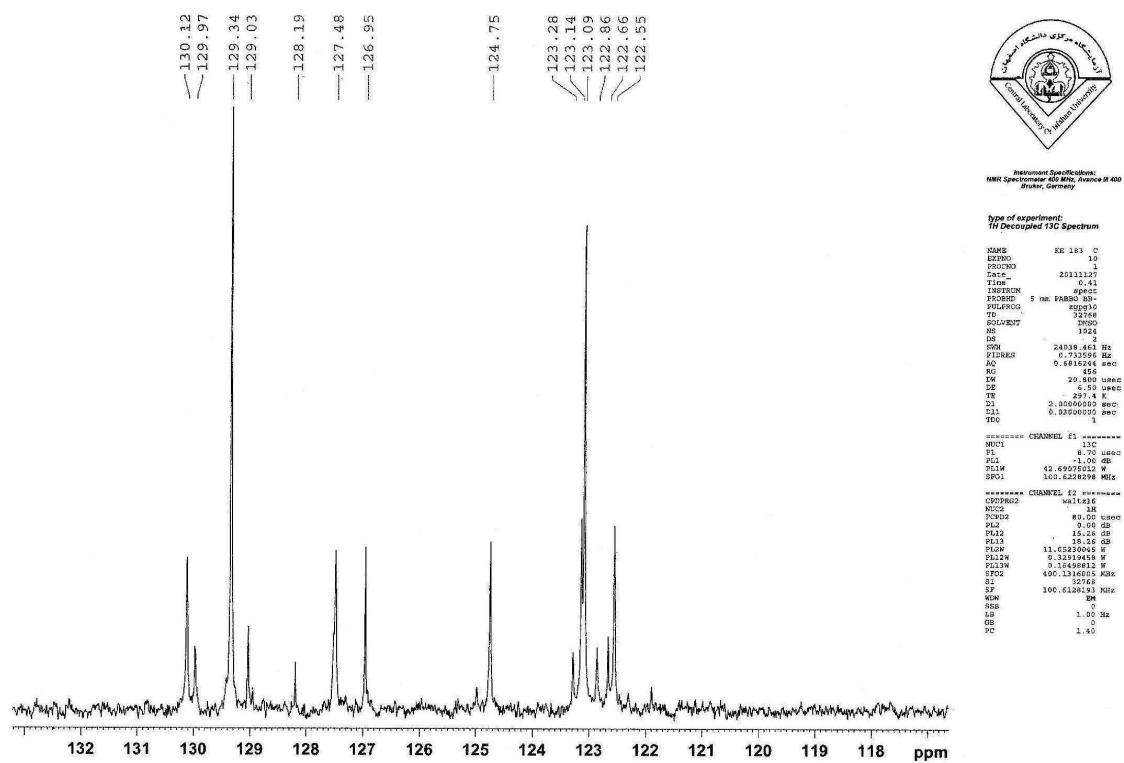
S-41: ^1H NMR spectrum of compound 4{1,3,1}



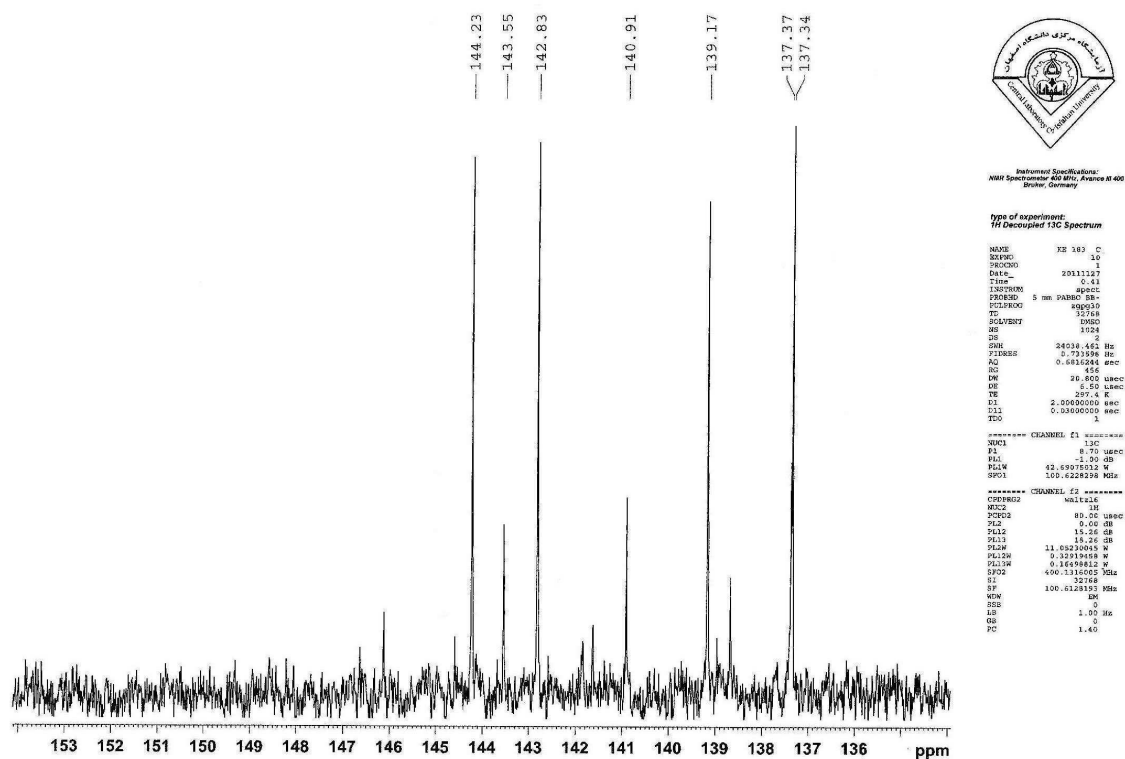
S-41: Expand of ^1H NMR spectrum of compound 4{1,3,1}



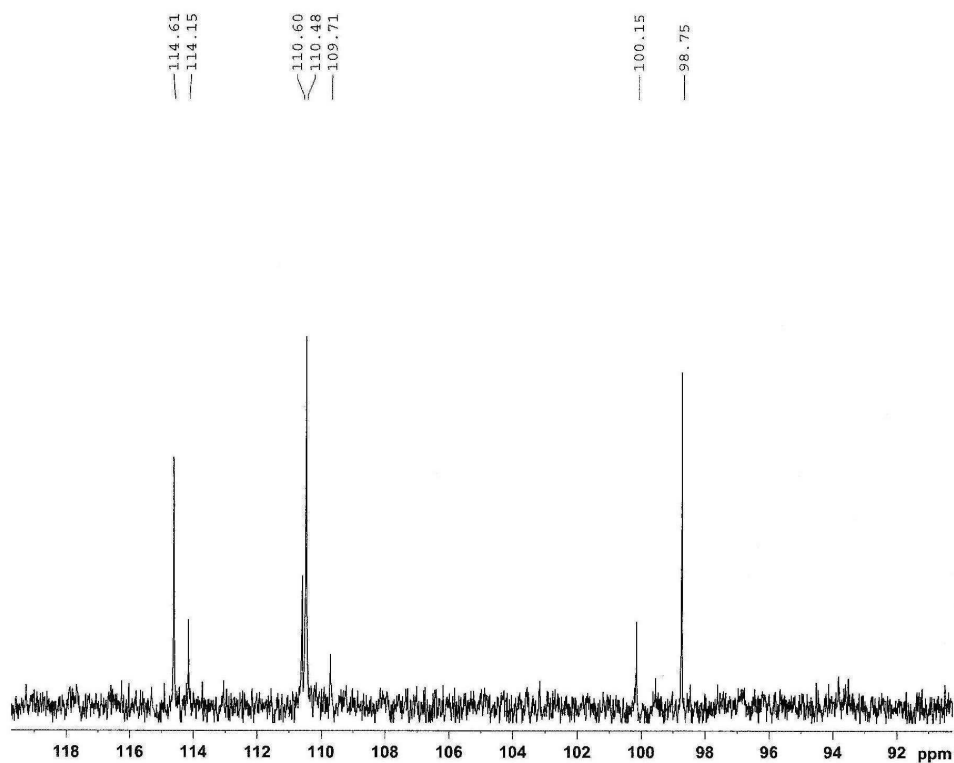
S-42: ^{13}C NMR spectrum of compound 4{1,3,1}



S-42: Expand of ^{13}C NMR spectrum of compound 4{1,3,1}



S-42: Expand of ^{13}C NMR spectrum of compound 4{1,3,1}

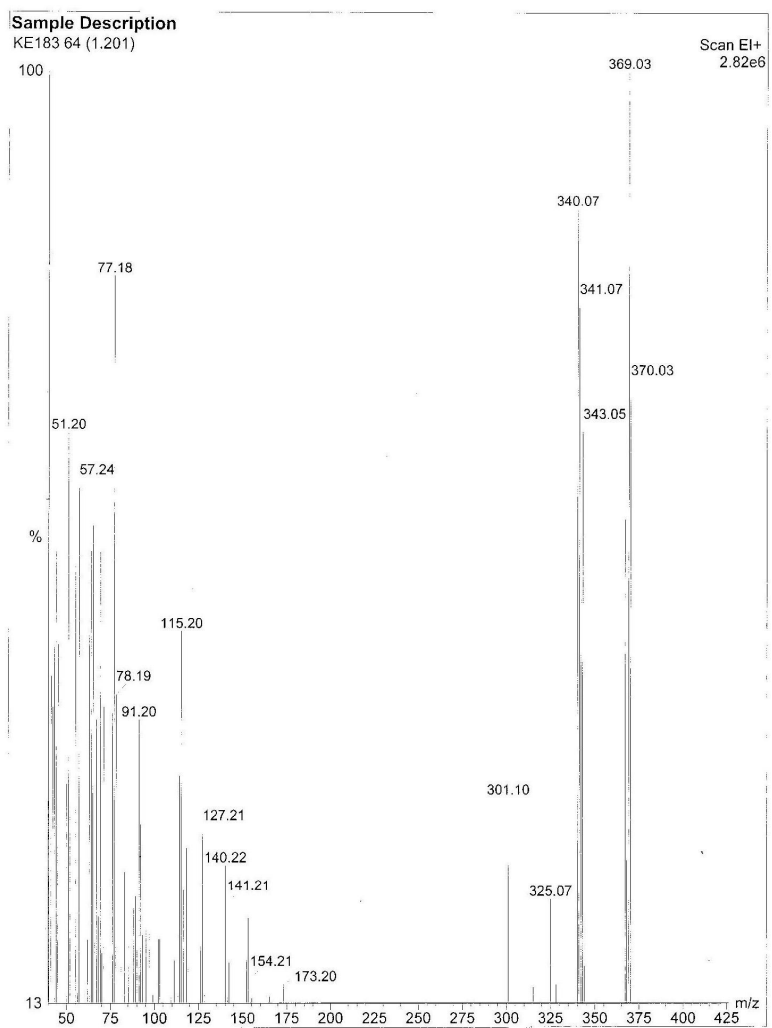


Instrument Specifications:
NMR Spectrometer 400 MHz, Avance II 400
Bruker, Germany

Type of experiment:
1H Decoupled ^{13}C Spectrum

NAME	KE 143 C
EXPNO	10
PROCNO	1
Date_	20111107
Time	0.41
INSTRUM	gpcp
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	32768
SOLVENT	DMSO
NS	1024
DS	4
SVF	24036.461 Hz
FIDRES	0.713056 Hz
AQ	0.5815246 sec
RG	650
DM	20.800 usec
DE	6.10 usec
TE	297.4 K
D1	2.00000000 sec
D11	0.03000000 sec
D12	1
===== CHANNEL f1 =====	
NUC1	^{13}C
PT	8.70 usec
PL1	0.00 dB
PL12	12.00 dB
PL14	12.00 dB
PL16	12.00 dB
PL18	12.00 dB
PL20	12.00 dB
PL22	12.00 dB
PL24	12.00 dB
PL26	12.00 dB
PL28	12.00 dB
PL30	12.00 dB
PL32	12.00 dB
PL34	12.00 dB
PL36	12.00 dB
PL38	12.00 dB
PL40	12.00 dB
PL42	12.00 dB
PL44	12.00 dB
PL46	12.00 dB
PL48	12.00 dB
PL50	12.00 dB
PL52	12.00 dB
PL54	12.00 dB
PL56	12.00 dB
PL58	12.00 dB
PL60	12.00 dB
PL62	12.00 dB
PL64	12.00 dB
PL66	12.00 dB
PL68	12.00 dB
PL70	12.00 dB
PL72	12.00 dB
PL74	12.00 dB
PL76	12.00 dB
PL78	12.00 dB
PL80	12.00 dB
PL82	12.00 dB
PL84	12.00 dB
PL86	12.00 dB
PL88	12.00 dB
PL90	12.00 dB
PL92	12.00 dB
PL94	12.00 dB
PL96	12.00 dB
PL98	12.00 dB
PL100	12.00 dB

S-42: Expand of ^{13}C NMR spectrum of compound 4{1,3,1}



S-43: Mass spectrum of compound 4{1,3,1}

Sample Description
KE183 64 (1.201)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	Scan	El+
1:	40.17	7.05e5	25.00	0.68	67:	115.20	1.34e6	47.67	1.29	133:	285.12	1.43e5	5.09	0.14		
2:	41.27	1.23e6	43.60	1.18	68:	116.24	6.68e5	23.69	0.64	134:	292.07	1.43e5	5.09	0.14		
3:	42.26	1.15e6	40.70	1.10	69:	117.22	3.15e5	11.19	0.30	135:	295.12	2.02e5	7.16	0.19		
4:	43.25	1.64e6	58.14	1.58	70:	118.21	7.78e5	27.62	0.75	136:	297.10	1.79e5	6.36	0.17		
5:	44.20	1.58e6	55.23	1.50	71:	119.20	5.08e5	18.02	0.49	137:	298.11	1.81e5	6.43	0.17		
6:	45.25	1.31e6	45.51	1.26	72:	121.31	1.99e5	7.05	0.19	138:	299.11	2.38e5	8.43	0.23		
7:	46.24	5.94e5	21.08	0.57	73:	122.37	1.59e5	5.63	0.15	139:	300.10	2.37e5	8.39	0.23		
8:	50.22	9.46e5	33.58	0.91	74:	123.25	2.74e5	9.74	0.26	140:	301.10	8.09e5	32.27	0.87		
9:	51.20	1.87e6	66.28	1.80	75:	125.23	2.87e5	10.17	0.28	141:	302.10	2.07e5	7.34	0.20		
10:	52.21	1.05e6	37.21	1.01	76:	126.19	5.16e5	18.31	0.50	142:	303.13	1.51e5	5.34	0.14		
11:	53.20	3.80e5	12.79	0.35	77:	127.21	8.15e5	28.92	0.78	143:	312.12	1.60e5	5.67	0.15		
12:	54.22	2.70e5	9.59	0.26	78:	128.24	5.98e5	21.22	0.58	144:	313.11	2.38e5	8.43	0.23		
13:	55.23	1.52e6	54.07	1.47	79:	129.24	3.48e5	12.35	0.33	145:	314.10	4.55e5	16.13	0.44		
14:	56.24	6.51e5	23.11	0.63	80:	130.18	1.93e5	6.83	0.19	146:	315.10	4.10e5	14.53	0.39		
15:	57.24	1.72e6	61.05	1.66	81:	131.19	1.62e5	5.74	0.16	147:	323.07	2.17e5	7.70	0.21		
16:	58.25	2.24e5	7.96	0.22	82:	135.43	2.30e5	8.18	0.22	148:	324.07	3.52e5	12.50	0.34		
17:	61.18	1.44e5	5.12	0.14	83:	137.23	1.51e5	5.34	0.14	149:	325.07	6.43e5	22.82	0.62		
18:	62.19	5.37e5	19.04	0.52	84:	138.20	1.85e5	6.58	0.18	150:	326.07	3.24e5	11.48	0.31		
19:	63.19	1.51e6	53.49	1.45	85:	139.20	3.40e5	12.06	0.33	151:	327.05	3.07e5	10.90	0.30		
20:	64.19	1.56e6	55.23	1.50	86:	140.22	7.33e5	26.02	0.71	152:	328.06	4.18e5	14.83	0.40		
21:	65.20	1.62e6	57.56	1.56	87:	141.21	6.31e5	22.38	0.61	153:	329.06	1.43e5	5.09	0.14		
22:	66.19	7.54e5	26.74	0.73	88:	142.20	4.75e5	16.86	0.46	154:	338.06	2.11e5	7.49	0.20		
23:	67.19	1.11e6	39.53	1.07	89:	143.19	2.74e5	9.74	0.26	155:	339.07	2.33e5	8.28	0.22		
24:	68.20	5.98e5	21.22	0.58	90:	145.19	1.91e5	6.80	0.18	156:	340.07	2.46e6	87.21	2.36		
25:	69.23	1.56e6	55.23	1.50	91:	147.24	2.11e5	7.49	0.20	157:	341.07	2.20e6	77.91	2.11		
26:	70.25	5.00e5	17.73	0.48	92:	148.36	1.62e5	5.74	0.16	158:	342.06	1.43e6	50.58	1.37		
27:	71.24	1.15e6	40.70	1.10	93:	149.29	1.59e5	5.63	0.15	159:	343.05	1.87e6	66.28	1.80		
28:	74.16	3.56e5	12.65	0.34	94:	151.20	2.29e5	8.14	0.22	160:	344.05	4.67e5	16.57	0.45		
29:	75.17	7.37e5	26.16	0.71	95:	152.19	4.79e5	17.01	0.46	161:	354.02	1.64e5	5.81	0.16		
30:	76.18	1.13e6	40.12	1.09	96:	153.20	5.94e5	21.08	0.57	162:	366.01	3.32e5	11.77	0.32		
31:	77.18	2.28e6	80.81	2.19	97:	154.21	4.30e5	15.26	0.41	163:	367.03	1.64e6	58.14	1.58		
32:	78.19	1.18e6	41.86	1.13	98:	155.21	3.81e5	13.52	0.37	164:	368.03	7.45e5	26.45	0.72		
33:	79.19	2.83e5	10.03	0.27	99:	156.19	1.67e5	5.92	0.16	165:	369.03	2.82e6	100.00	2.71		
34:	80.19	1.65e5	5.85	0.16	100:	157.22	1.93e5	6.83	0.19	166:	370.03	1.98e6	70.35	1.91		
35:	81.23	5.57e5	19.77	0.54	101:	163.19	2.66e5	9.45	0.26	167:	371.04	3.40e5	12.06	0.33		
36:	82.20	2.49e5	8.83	0.24	102:	164.16	3.28e5	11.63	0.32							
37:	83.23	7.17e5	25.44	0.69	103:	165.18	3.85e5	13.66	0.37							
38:	84.23	1.70e5	6.03	0.16	104:	166.19	2.18e5	7.74	0.21							
39:	85.25	5.49e5	19.48	0.53	105:	167.20	2.51e5	8.90	0.24							
40:	87.16	2.79e5	9.88	0.27	106:	168.19	2.99e5	10.61	0.29							
41:	88.18	6.18e5	21.95	0.60	107:	169.20	3.36e5	11.92	0.32							
42:	89.19	6.51e5	23.11	0.63	108:	170.18	2.66e5	9.45	0.26							
43:	90.18	5.08e5	18.02	0.49	109:	172.18	1.97e5	6.98	0.19							
44:	91.20	1.11e6	39.53	1.07	110:	173.20	4.18e5	14.83	0.40							
45:	92.19	8.40e5	29.80	0.81	111:	177.18	1.51e5	5.34	0.14							
46:	93.21	5.49e5	19.48	0.53	112:	178.17	1.69e5	6.00	0.16							
47:	94.21	1.59e5	5.63	0.15	113:	179.18	3.69e5	13.06	0.35							
48:	95.26	5.61e5	19.91	0.54	114:	180.17	3.28e5	11.63	0.32							
49:	96.24	2.37e5	8.39	0.23	115:	181.18	1.88e5	6.61	0.18							
50:	97.25	6.92e5	24.56	0.67	116:	184.49	2.95e5	10.47	0.28							
51:	98.24	1.55e5	5.49	0.15	117:	190.17	2.05e5	7.27	0.20							
52:	99.22	3.88e5	13.81	0.37	118:	191.18	1.76e5	6.25	0.17							
53:	100.18	3.69e5	13.08	0.35	119:	192.18	1.45e5	5.16	0.14							
54:	101.18	5.73e5	20.35	0.55	120:	193.19	1.43e5	5.09	0.14							
55:	102.18	5.37e5	19.04	0.52	121:	194.19	1.83e5	6.50	0.18							
56:	103.18	5.37e5	19.04	0.52	122:	195.17	1.83e5	6.50	0.18							
57:	104.19	4.34e5	15.41	0.42	123:	205.17	1.55e5	5.49	0.15							
58:	105.20	2.52e5	8.94	0.24	124:	225.16	1.47e5	5.23	0.14							
59:	106.22	1.62e5	5.74	0.16	125:	229.17	1.89e5	6.72	0.18							
60:	107.30	1.72e5	6.10	0.17	126:	243.16	1.44e5	5.12	0.14							
61:	109.22	4.46e5	15.84	0.43	127:	244.17	1.76e5	6.25	0.17							
62:	110.25	1.62e5	5.74	0.16	128:	256.14	1.89e5	6.72	0.18							
63:	111.25	4.79e5	17.01	0.46	129:	260.18	1.89e5	6.72	0.18							
64:	112.19	1.90e5	6.76	0.18	130:	273.15	2.87e5	10.17	0.28							
65:	113.22	4.81e5	15.99	0.43	131:	274.14	1.51e5	5.34	0.14							
66:	114.19	9.67e5	34.30	0.93	132:	284.10	1.45e5	5.16	0.14							

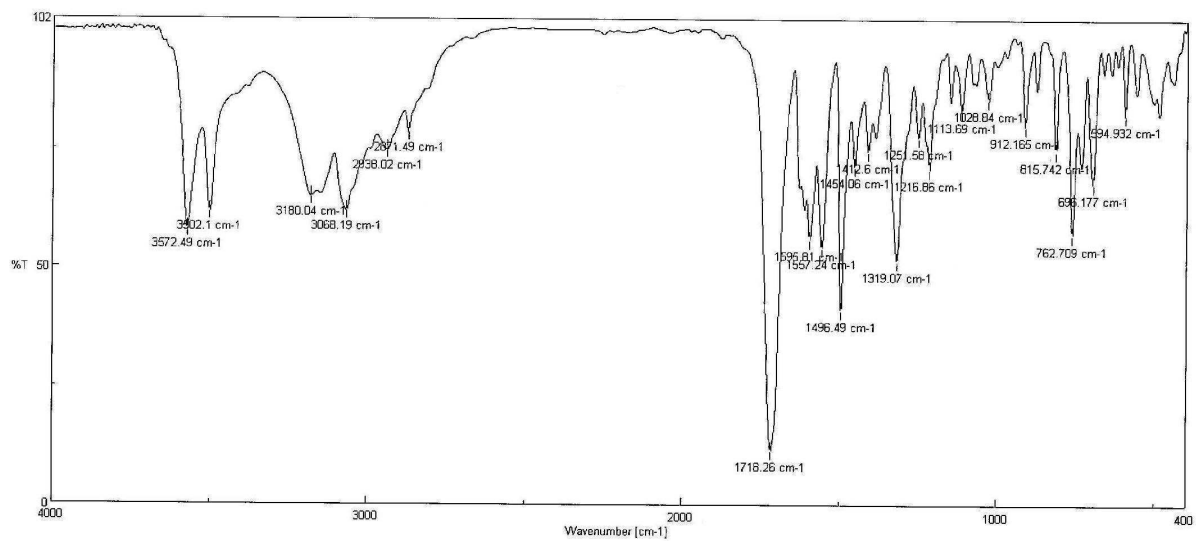
S-44: Sample description presented by mass instrument to compound 4{1,3,1}

Sample Code: *II900830-131 (KE-199)*

Central Laboratory of the University of Isfahan

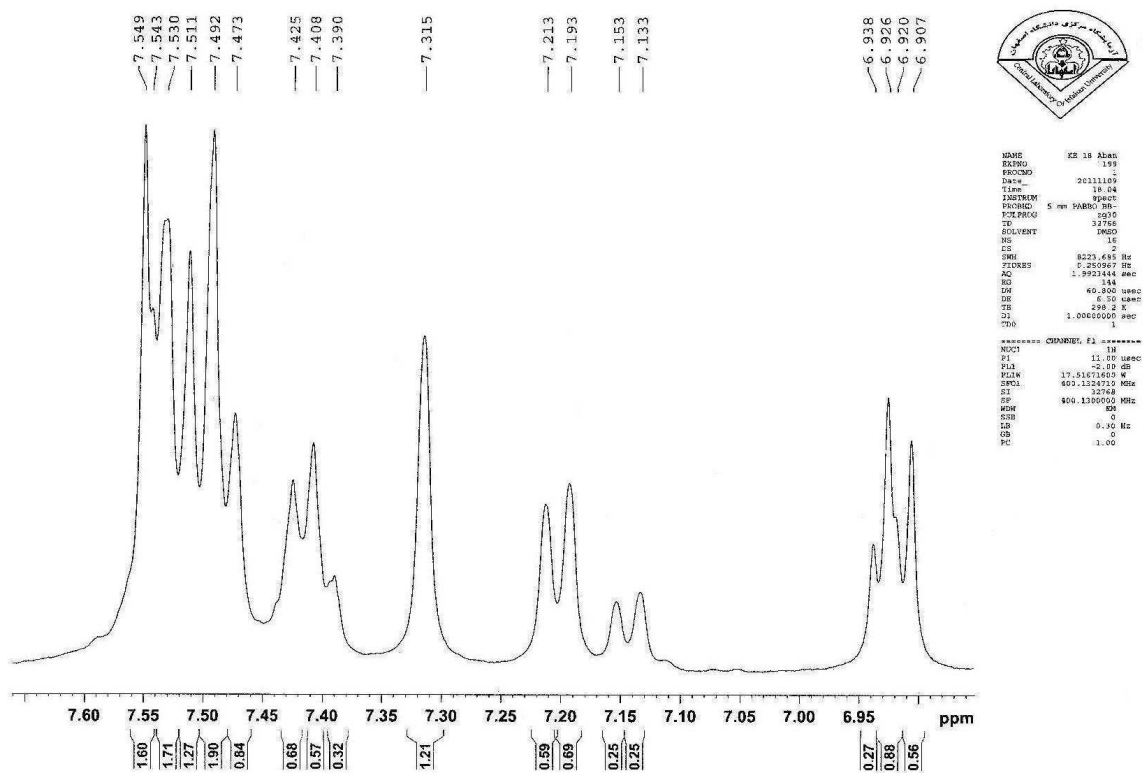
Instrument Specifications

FT Infrared Spectroscopy, JASCO, FT/IR-6300 (400-4000 cm^{-1}), Japan

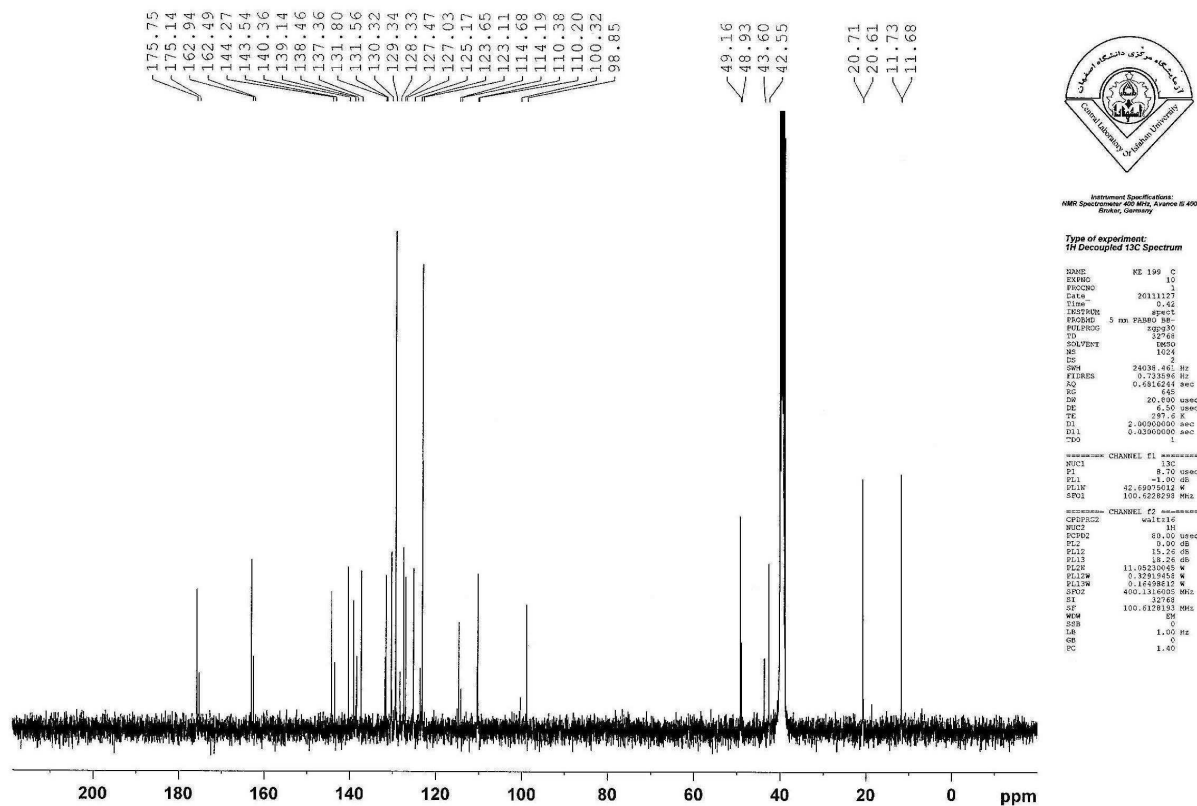


Date: 90/09/05
No.:

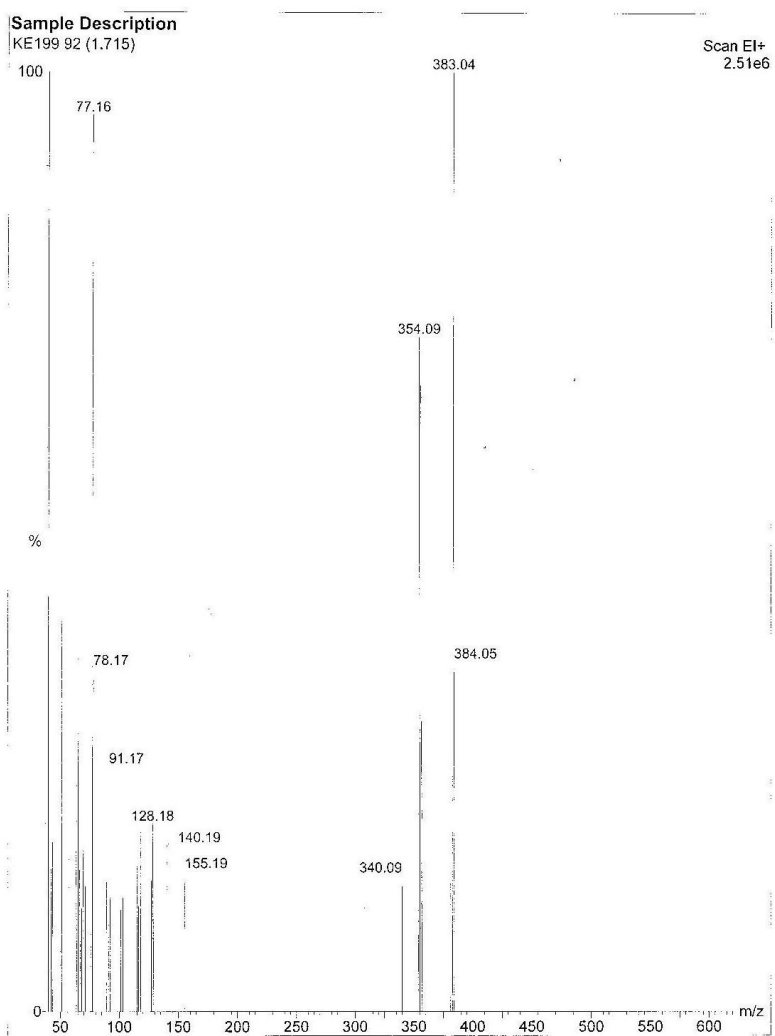
S-45: IR spectrum of compound 4{1,3,2}



S-46: Expand of ^1H NMR spectrum of compound 4{1,3,2}



S-47: ^{13}C NMR spectrum of compound 4{1,3,2}



S-48: Mass spectrum of compound 4{1,3,2}

Sample Description					Scan El+				
KE199 92 (1.715)									
No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC
1:	41.25	3.03e5	12.09	0.63					
2:	42.24	3.81e5	15.20	0.80					
3:	43.25	4.51e5	17.97	0.94					
4:	50.19	2.83e5	11.27	0.59					
5:	51.20	1.69e6	67.32	3.53					
6:	52.19	4.67e5	18.63	0.98					
7:	55.20	3.32e5	13.24	0.69					
8:	57.22	5.08e5	20.26	1.06					
9:	63.16	6.27e5	25.00	1.31					
10:	64.17	6.96e5	27.78	1.46					
11:	65.18	9.95e5	39.71	2.08					
12:	66.17	3.77e5	15.03	0.79					
13:	67.15	4.06e5	16.18	0.85					
14:	68.16	2.74e5	10.95	0.57					
15:	69.20	4.38e5	17.48	0.92					
16:	71.20	3.32e5	13.24	0.69					
17:	75.13	3.60e5	14.38	0.75					
18:	76.15	6.06e5	24.18	1.27					
19:	77.16	2.39e6	95.42	5.00					
20:	78.17	8.89e5	35.46	1.86					
21:	89.16	3.44e5	13.73	0.72					
22:	91.17	6.27e5	25.00	1.31					
23:	92.16	3.03e5	12.09	0.63					
24:	101.17	2.70e5	10.78	0.57					
25:	102.16	3.32e5	13.24	0.69					
26:	103.16	3.03e5	12.09	0.63					
27:	114.18	2.58e5	10.29	0.54					
28:	115.19	3.89e5	15.52	0.81					
29:	116.16	2.79e5	11.11	0.58					
30:	118.19	4.79e5	19.12	1.00					
31:	127.19	3.48e5	13.89	0.73					
32:	128.18	5.00e5	19.93	1.05					
33:	129.14	2.82e5	10.46	0.55					
34:	140.19	4.42e5	17.65	0.93					
35:	141.19	2.60e5	10.38	0.54					
36:	155.19	3.44e5	13.73	0.72					
37:	339.10	2.91e5	11.60	0.61					
38:	340.09	3.32e5	13.24	0.69					
39:	354.09	1.80e6	71.90	3.77					
40:	355.09	1.67e6	68.67	3.50					
41:	356.07	7.74e5	30.88	1.62					
42:	357.08	9.54e5	38.07	2.00					
43:	381.03	3.40e5	13.56	0.71					
44:	382.03	2.51e5	10.01	0.52					
45:	383.04	2.51e6	100.00	5.24					
46:	384.05	9.05e5	36.11	1.89					

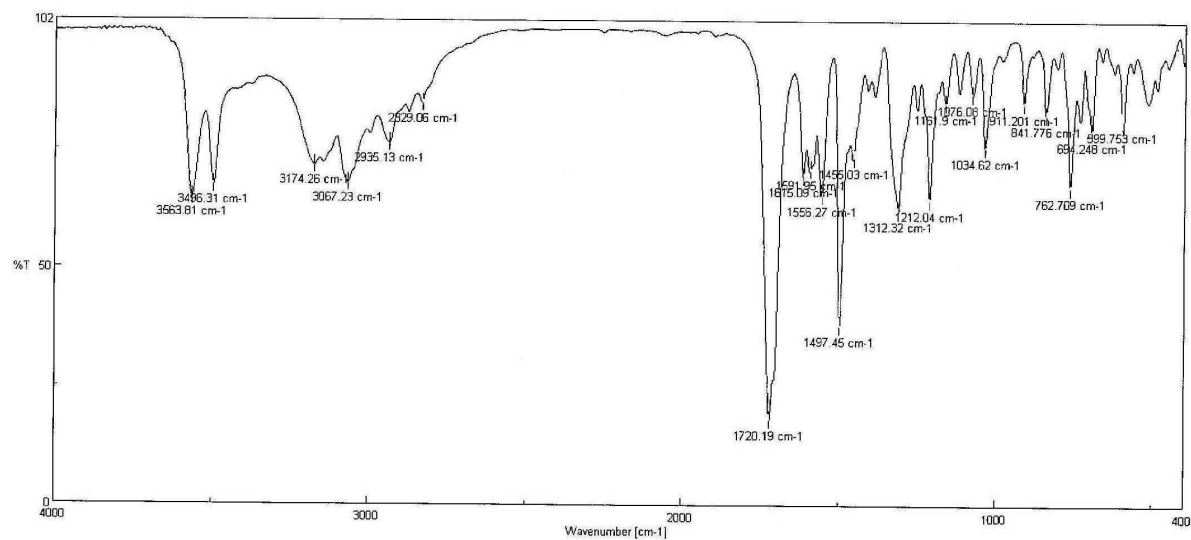
S-49: Sample description presented by mass instrument to compound 4{1,3,2}

Sample Code: *II900830-132 (KE-202)*

Central Laboratory of the University of Isfahan

Instrument Specifications

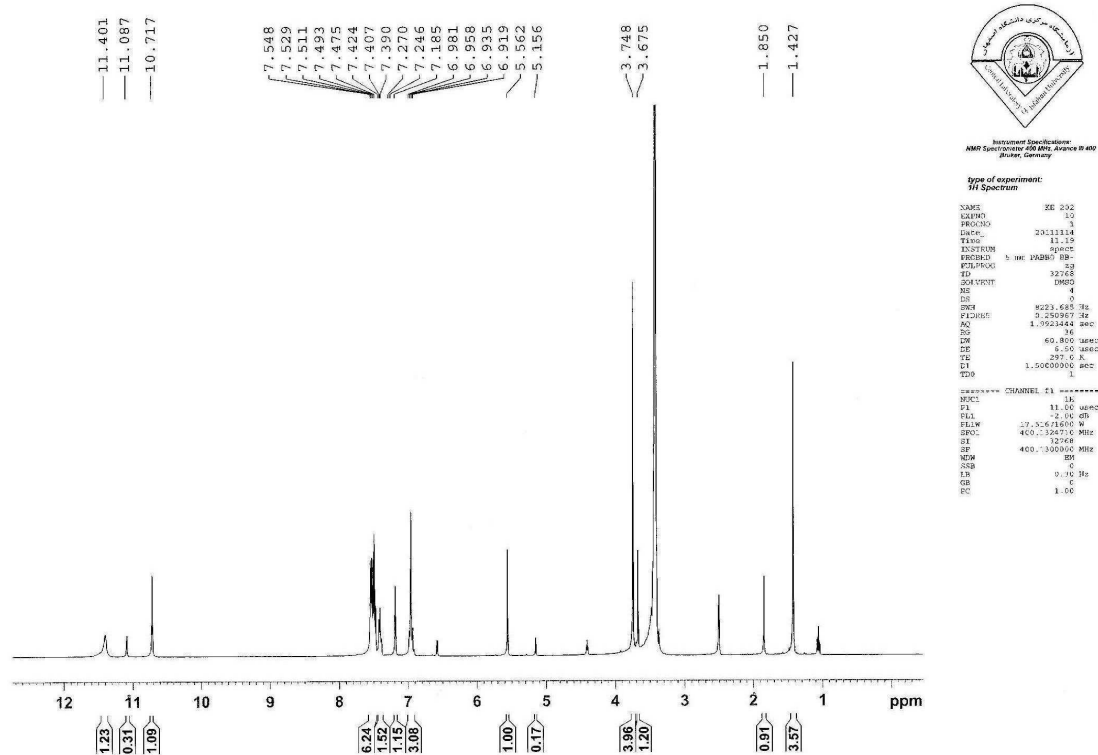
FT Infrared Spectroscopy, JASCO, FT/IR-6300 (400-4000 cm⁻¹), Japan



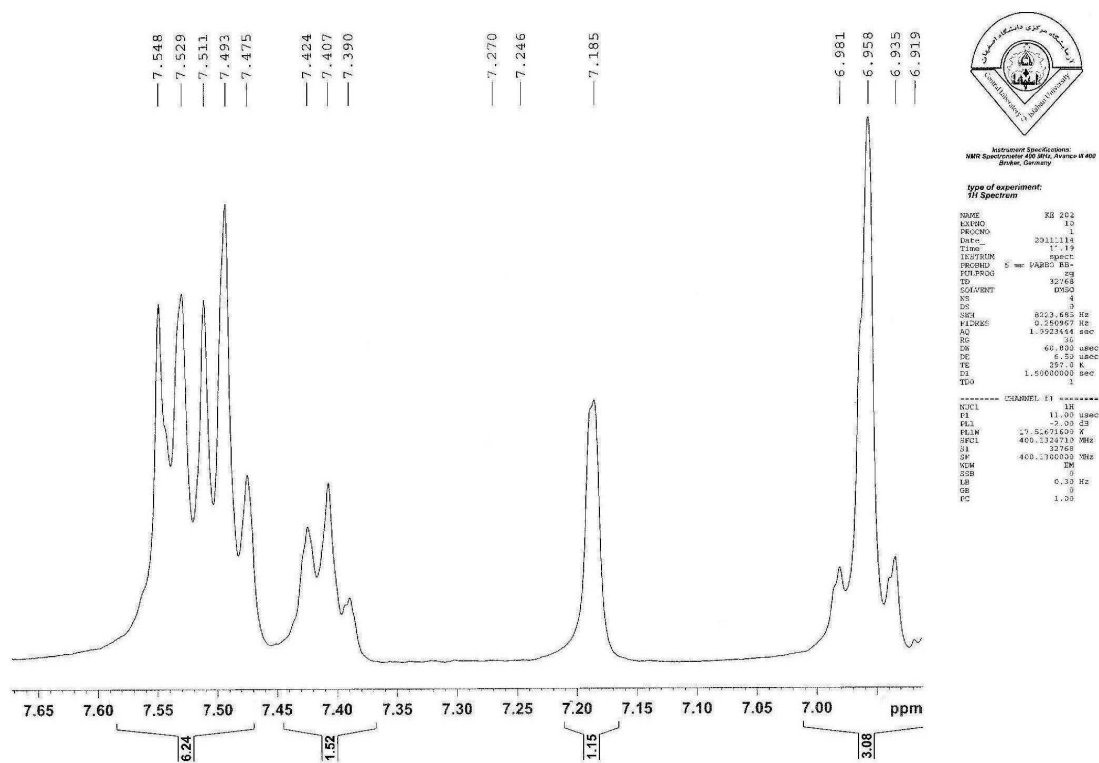
Date: 90/09/05

No.:

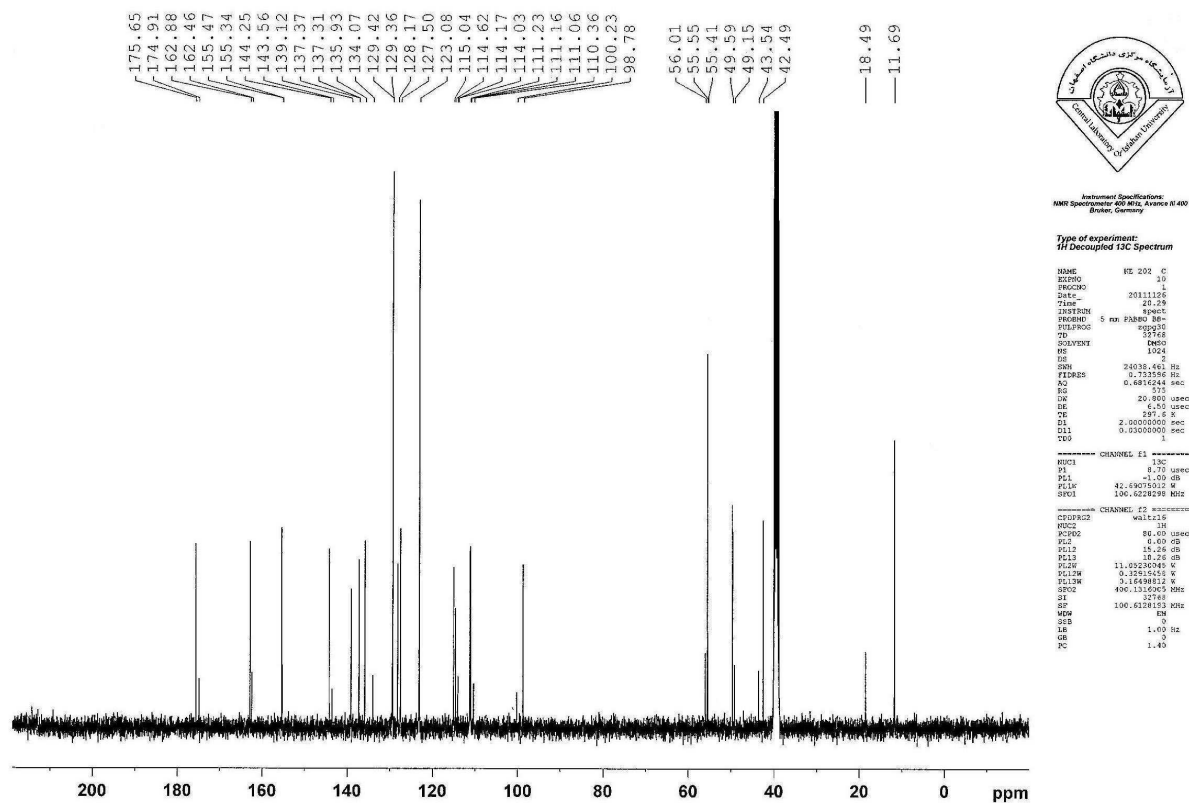
S-50: IR spectrum of compound 4{1,3,3}



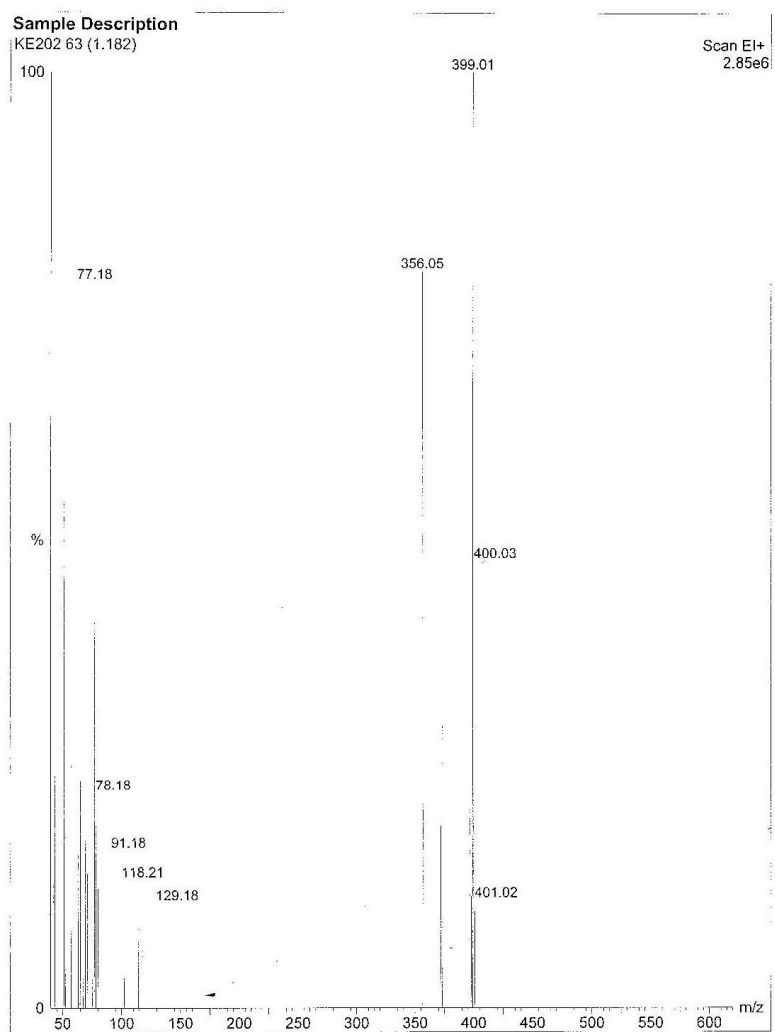
S-51: ^1H NMR spectrum of compound 4{1,3,3}



S-51: Expand of ^1H NMR spectrum of compound 4{1,3,3}



S-52: ^{13}C NMR spectrum of compound 4{1,3,3}



S-53: Mass spectrum of compound 4{1,3,3}

Sample Description														
KE202 63 (1.182)										Scan El+				
No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC
1:	41.27	3.60e5	12.64	0.75										
2:	42.26	3.89e5	13.65	0.81										
3:	43.26	7.09e5	24.86	1.48										
4:	44.21	4.22e5	14.80	0.88										
5:	51.21	1.56e6	54.60	3.24										
6:	52.21	5.28e5	18.53	1.10										
7:	53.20	3.69e5	12.93	0.77										
8:	55.22	5.45e5	19.11	1.13										
9:	57.23	7.74e5	27.16	1.61										
10:	63.18	4.87e5	17.10	1.01										
11:	64.18	5.69e5	19.97	1.19										
12:	65.19	6.92e5	24.28	1.44										
13:	67.19	3.77e5	13.22	0.78										
14:	69.21	5.16e5	18.10	1.07										
15:	71.21	4.10e5	14.37	0.85										
16:	75.16	3.28e5	11.49	0.68										
17:	76.17	5.53e5	19.40	1.15										
18:	77.18	2.21e6	77.59	4.60										
19:	78.18	6.23e5	21.84	1.30										
20:	80.16	3.65e5	12.79	0.76										
21:	91.18	4.46e5	15.66	0.93										
22:	102.18	3.40e5	11.93	0.71										
23:	114.19	2.91e5	10.20	0.61										
24:	118.21	3.89e5	13.65	0.81										
25:	129.18	2.87e5	10.06	0.60										
26:	356.05	2.24e6	78.74	4.67										
27:	357.05	8.19e5	28.74	1.71										
28:	371.08	5.73e5	20.11	1.19										
29:	372.05	5.53e5	19.40	1.15										
30:	373.05	8.64e5	30.32	1.80										
31:	397.01	8.10e5	21.41	1.27										
32:	398.02	3.40e5	11.93	0.71										
33:	399.01	2.85e6	100.00	5.94										
34:	400.03	1.33e6	46.55	2.76										
35:	401.02	2.95e5	10.34	0.61										

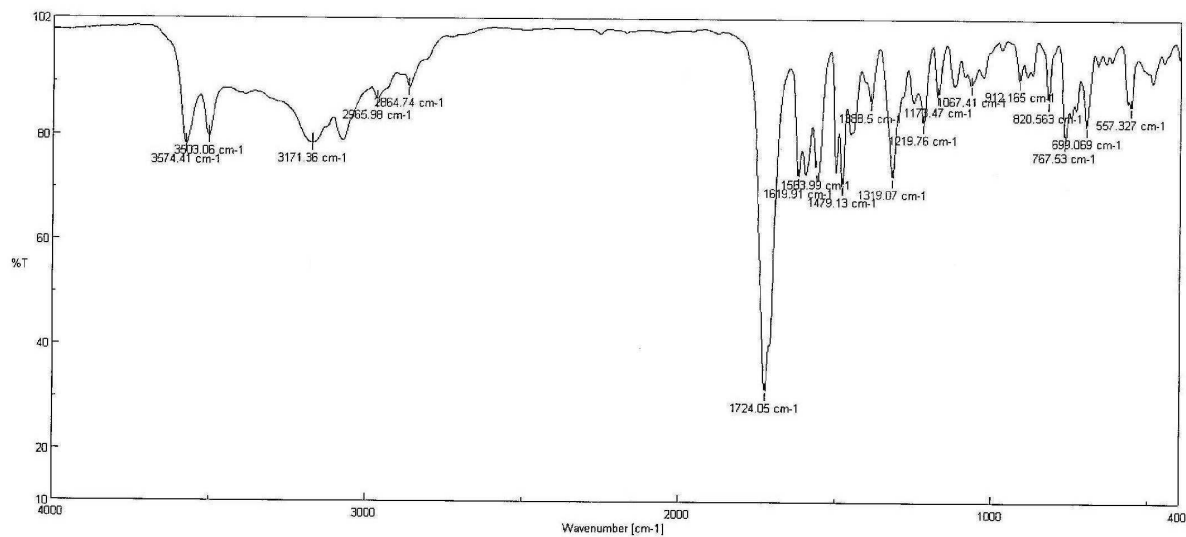
S-54: Sample description presented by mass instrument to compound **4{1,3,3}**

Sample Code: 11900830-133 (KE-204)

Central Laboratory of the University of Isfahan

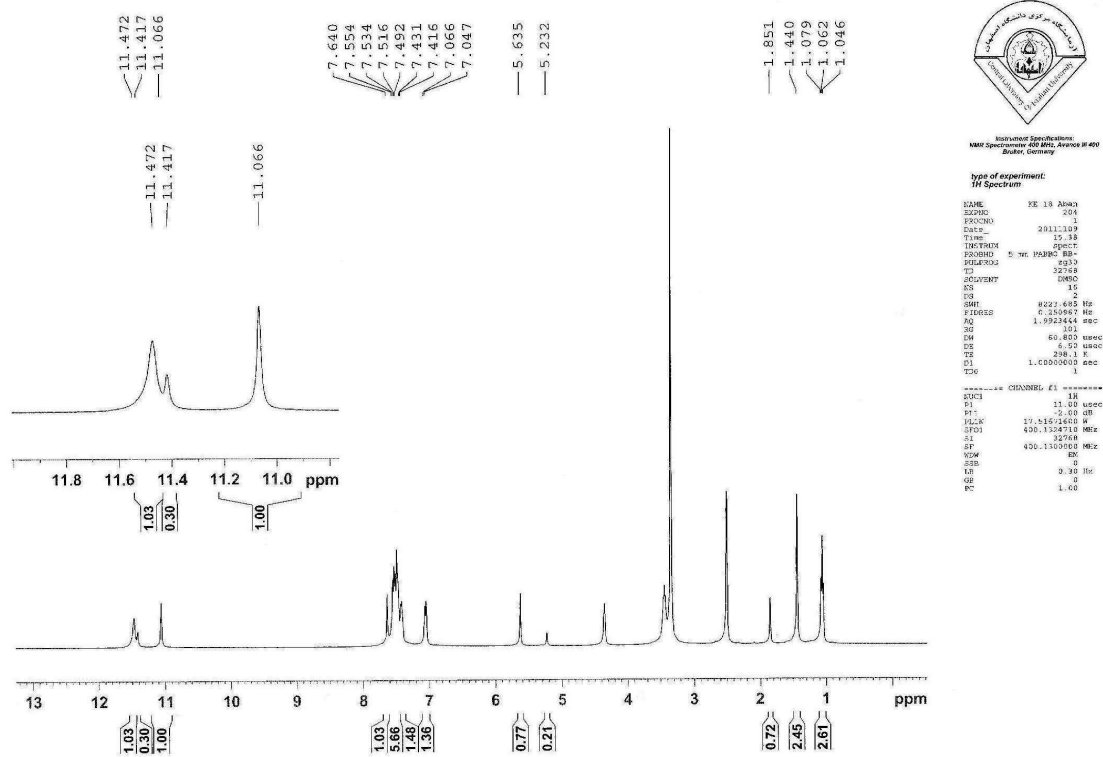
Instrument Specifications

FT Infrared Spectroscopy, JASCO, FT/IR-6300 (400-4000 cm^{-1}), Japan

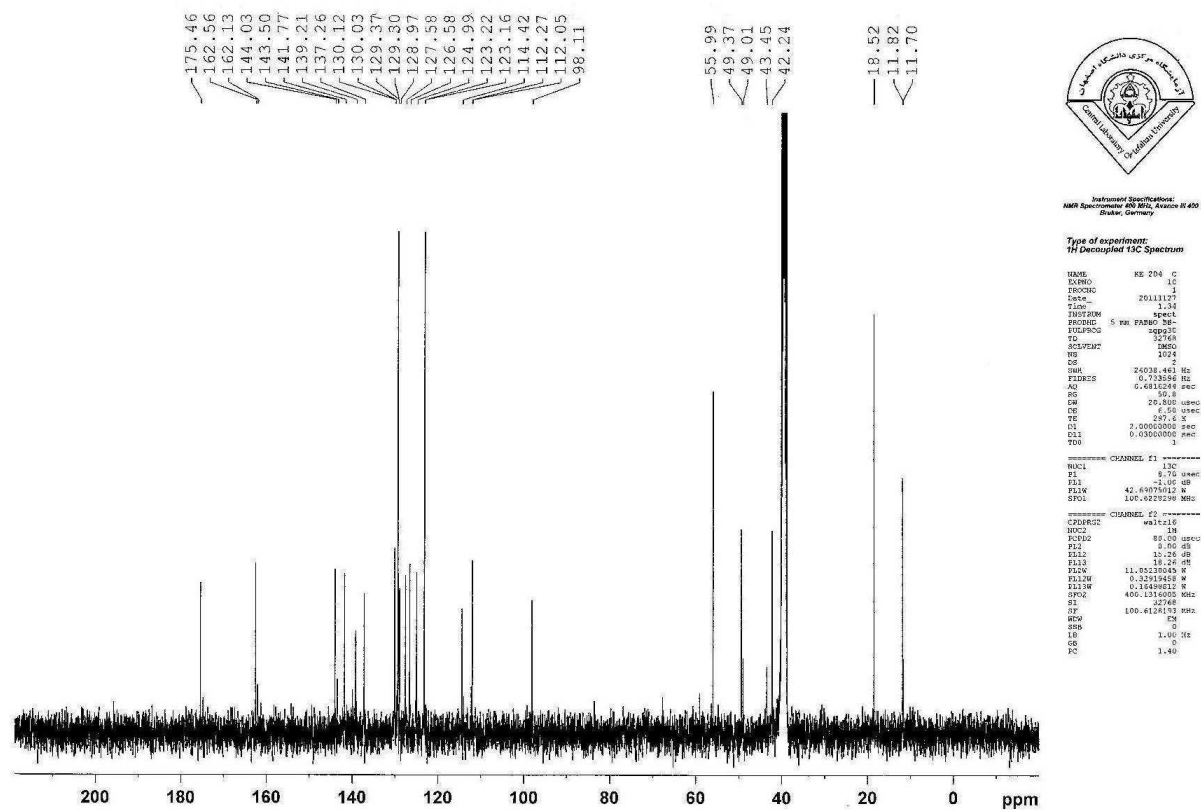


Date: 90/09/05
No.:

S-55: IR spectrum of compound 4{1,3,4}



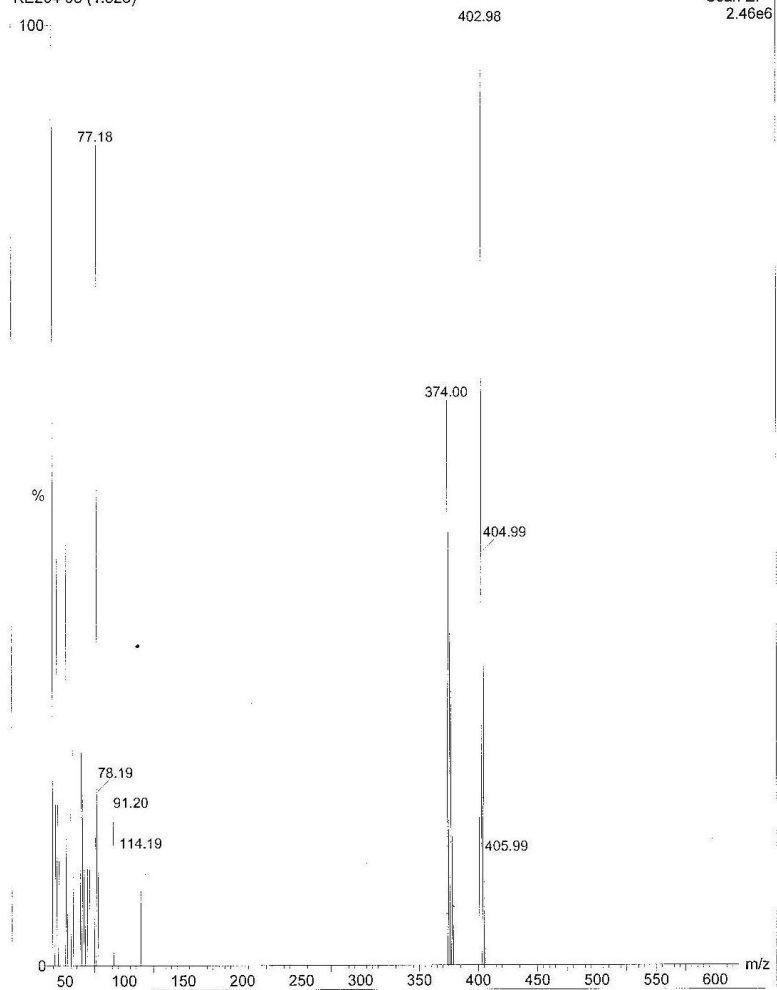
S-56: ^1H NMR spectrum of compound 4{1,3,4}



S-57: ^{13}C NMR spectrum of compound 4{1,3,4}

Sample Description
KE204 98 (1.823)

Scan EI+
2.46e6



S-58: Mass spectrum of compound 4{1,3,4}

Sample Description														
KE204 98 (1.823)										Scan E1+				
No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC
1:	41.27	3.40e5	13.83	0.83										
2:	42.25	4.22e5	17.17	1.03										
3:	43.25	1.06e6	43.33	2.61										
4:	44.20	4.22e5	17.17	1.03										
5:	45.24	2.74e5	11.17	0.67										
6:	50.21	2.91e5	11.83	0.71										
7:	51.21	1.61e6	65.33	3.93										
8:	52.21	3.36e5	13.67	0.82										
9:	55.22	3.97e5	16.17	0.97										
10:	57.24	5.73e5	23.33	1.40										
11:	63.19	4.92e5	20.00	1.20										
12:	64.19	5.57e5	22.67	1.36										
13:	65.20	6.47e5	26.33	1.58										
14:	66.19	2.62e5	10.67	0.64										
15:	67.19	3.24e5	13.17	0.79										
16:	69.22	3.60e5	14.67	0.98										
17:	71.21	2.51e5	10.21	0.61										
18:	75.16	2.91e5	11.83	0.71										
19:	76.17	3.38e5	13.67	0.82										
20:	77.18	2.15e6	87.33	5.25										
21:	78.19	4.55e5	18.50	1.11										
22:	91.20	3.77e5	15.33	0.92										
23:	114.19	2.99e5	12.17	0.73										
24:	118.23	2.62e5	10.67	0.64										
25:	374.00	1.47e6	60.00	3.61										
26:	375.02	1.13e6	48.00	2.77										
27:	376.00	1.06e6	43.33	2.61										
28:	377.00	1.04e6	42.50	2.56										
29:	377.99	3.36e5	13.67	0.82										
30:	378.99	2.46e5	10.00	0.60										
31:	400.96	3.85e5	15.67	0.94										
32:	402.98	2.46e6	100.00	6.01										
33:	403.98	7.91e5	32.17	1.93										
34:	404.99	1.08e6	44.00	2.65										
35:	405.99	2.62e5	10.67	0.64										

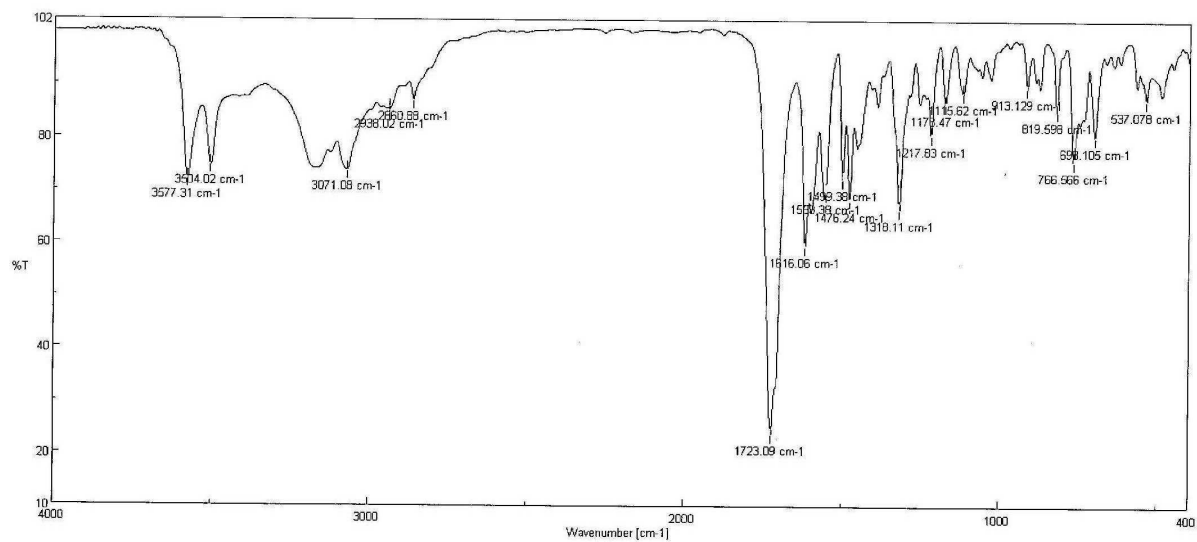
S-59: Sample description presented by mass instrument to compound 4{1,3,4}

Sample Code: H900830-130 (KE-186)

Central Laboratory of the University of Isfahan

Instrument Specifications

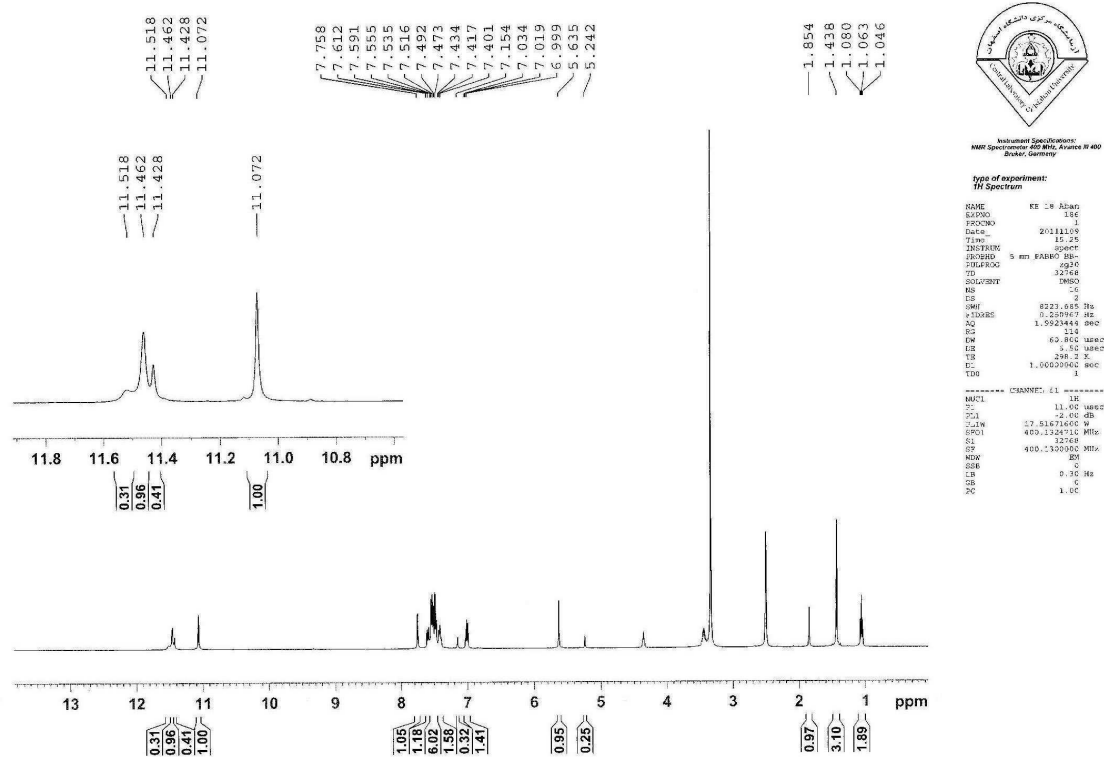
FT Infrared Spectroscopy, JASCO, FT/IR-6300 (400-4000 cm^{-1}), Japan



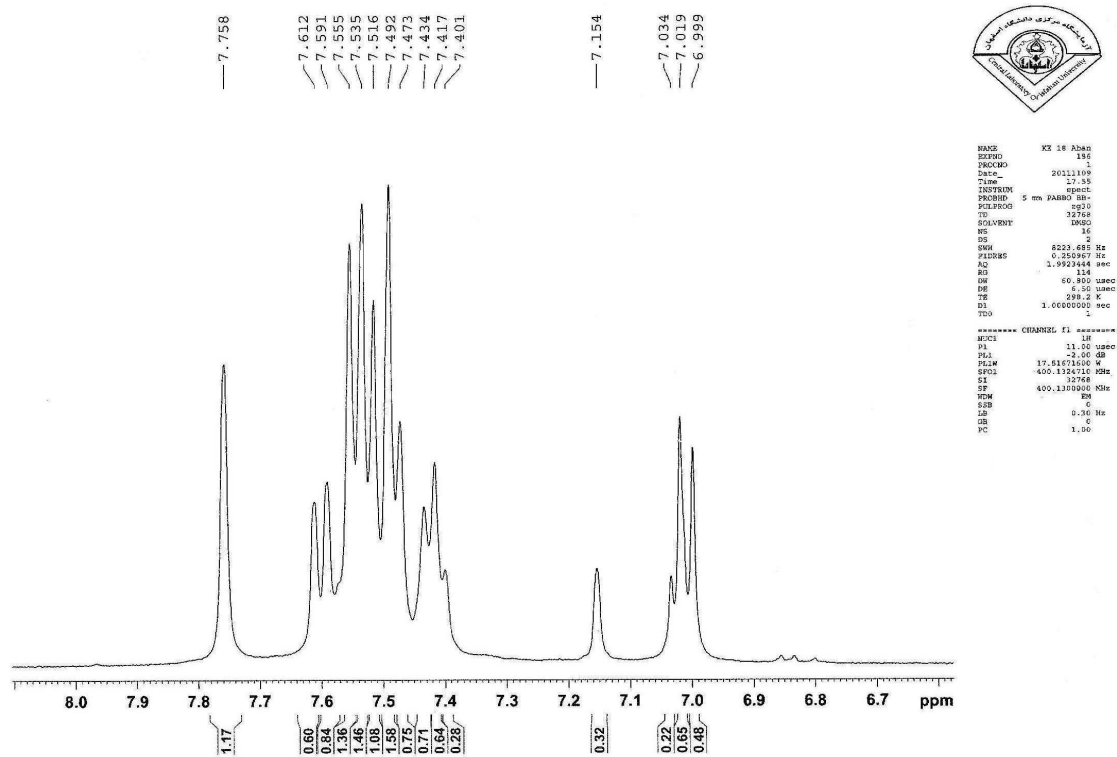
Date: 90/09/05

No.:

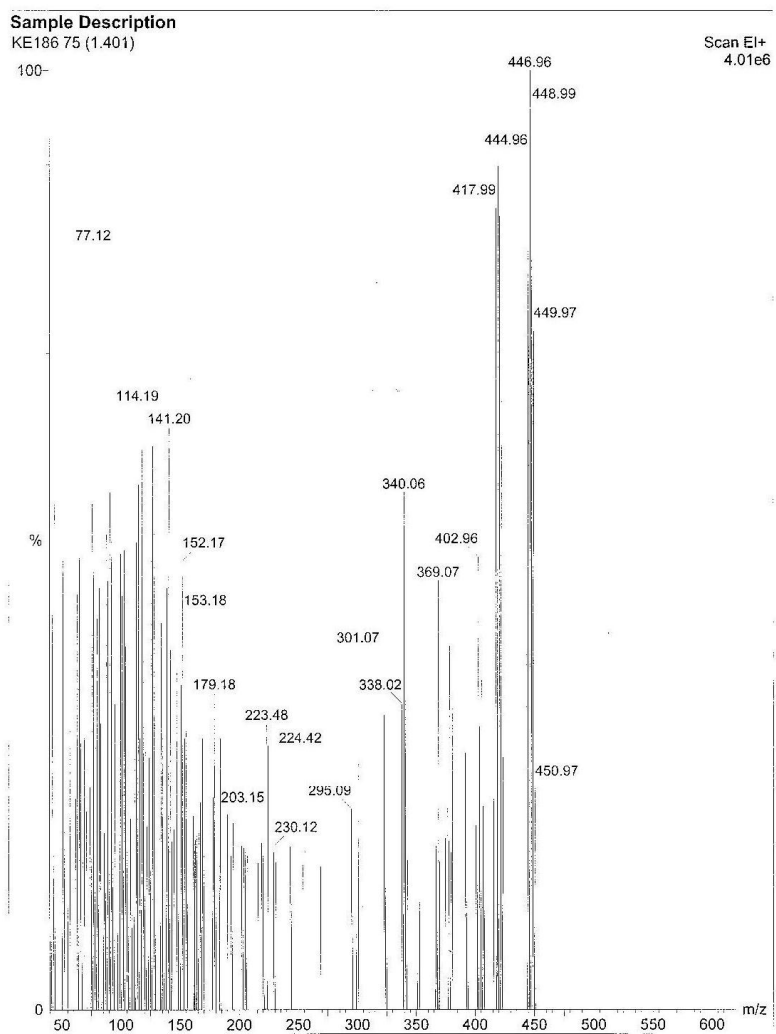
S-60: IR spectrum of compound 4{1,3,5}



S-61: ^1H NMR spectrum of compound 4{1,3,5}



S-61: Expand of ^1H NMR spectrum of compound 4{1,3,5}



S-63: Mass spectrum of compound 4{1,3,5}

Sample Description					Scan EI+				
KE186 75 (1.401)									
No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC
1:	40.17	1.25e6	31.02	0.39	87:	125.17	1.49e6	37.14	0.47
2:	41.24	1.43e6	35.51	0.45	88:	126.17	1.93e6	48.16	0.60
3:	42.23	1.69e6	42.04	0.53	89:	127.24	2.41e6	60.00	0.75
4:	43.23	1.67e6	41.63	0.52	90:	128.26	2.13e6	53.06	0.67
5:	44.15	2.16e6	53.88	0.65	91:	129.19	1.23e6	30.61	0.38
6:	50.19	1.51e6	40.00	0.50	92:	133.37	7.41e5	18.47	0.23
7:	51.15	2.46e6	61.22	0.77	93:	134.39	1.65e6	41.22	0.52
8:	52.18	1.56e6	38.78	0.49	94:	135.35	1.36e6	33.88	0.43
9:	55.21	8.48e5	21.12	0.27	95:	136.31	6.23e5	15.51	0.19
10:	57.22	1.23e6	30.61	0.38	96:	138.16	9.01e5	22.45	0.28
11:	62.16	9.01e5	22.45	0.28	97:	139.16	1.80e6	44.90	0.56
12:	63.16	1.80e6	44.90	0.56	98:	140.19	2.38e6	59.18	0.74
13:	64.16	1.88e6	46.94	0.59	99:	141.20	2.49e6	62.04	0.78
14:	65.18	1.93e6	48.16	0.60	100:	142.22	1.54e6	38.37	0.48
15:	66.18	1.15e6	28.57	0.36	101:	143.18	7.21e5	17.96	0.23
16:	67.17	1.23e6	30.61	0.38	102:	145.17	7.70e5	19.18	0.24
17:	68.16	6.64e5	16.53	0.21	103:	147.39	1.52e6	37.96	0.48
18:	69.21	1.16e6	28.98	0.36	104:	148.38	1.65e6	41.22	0.52
19:	71.23	8.48e5	21.12	0.27	105:	150.16	6.64e5	16.53	0.21
20:	74.16	9.50e5	23.67	0.30	106:	151.17	1.39e6	34.69	0.44
21:	75.15	1.85e6	46.12	0.58	107:	152.17	1.92e6	47.76	0.60
22:	76.16	2.16e6	53.88	0.68	108:	153.18	1.67e6	41.63	0.52
23:	77.12	3.28e6	81.63	1.02	109:	154.18	1.16e6	28.98	0.36
24:	78.17	2.02e6	50.20	0.63	110:	155.24	1.20e6	29.80	0.37
25:	79.13	1.13e6	28.16	0.35	111:	156.26	7.86e5	19.59	0.25
26:	80.09	1.67e6	41.63	0.52	112:	161.40	8.27e5	20.61	0.26
27:	81.16	1.44e6	35.92	0.45	113:	162.36	8.52e5	21.22	0.27
28:	82.09	1.80e6	44.90	0.56	114:	163.18	7.25e5	18.06	0.23
29:	83.20	1.23e6	30.61	0.38	115:	164.14	9.71e5	24.18	0.30
30:	85.21	1.02e6	25.51	0.32	116:	165.17	1.11e6	27.76	0.35
31:	86.16	7.58e5	18.88	0.24	117:	166.17	7.58e5	18.88	0.24
32:	87.15	1.80e6	44.90	0.56	118:	167.21	8.85e5	22.04	0.28
33:	88.15	2.03e6	50.61	0.64	119:	168.17	8.93e5	22.24	0.28
34:	89.15	1.84e6	45.71	0.57	120:	169.10	1.16e6	28.98	0.36
35:	90.15	1.88e6	46.94	0.59	121:	170.11	1.08e6	26.94	0.34
36:	91.16	2.21e6	55.10	0.69	122:	177.14	8.36e5	20.82	0.26
37:	92.17	2.05e6	51.02	0.64	123:	178.17	9.05e5	22.55	0.28
38:	93.18	1.98e6	49.39	0.62	124:	179.18	1.36e6	33.88	0.43
39:	94.26	7.95e5	19.80	0.25	125:	180.17	7.82e5	19.49	0.24
40:	95.25	1.31e6	32.65	0.41	126:	183.41	8.69e5	22.24	0.28
41:	97.21	1.13e6	28.16	0.35	127:	184.25	1.16e6	28.98	0.36
42:	99.15	1.84e6	45.71	0.57	128:	190.18	8.38e5	20.82	0.26
43:	100.15	1.95e6	48.57	0.61	129:	193.18	6.59e5	16.43	0.21
44:	101.13	1.77e6	44.08	0.55	130:	194.14	8.19e5	20.41	0.26
45:	102.14	2.06e6	51.43	0.65	131:	195.15	7.99e5	19.90	0.25
46:	103.13	1.97e6	48.98	0.61	132:	202.17	7.00e5	17.45	0.22
47:	104.15	1.56e6	38.78	0.49	133:	203.15	8.72e5	21.73	0.27
48:	105.15	1.26e6	31.43	0.39	134:	204.14	6.92e5	17.24	0.22
49:	106.20	7.70e5	19.18	0.24	135:	205.14	8.44e5	21.02	0.26
50:	107.30	7.95e5	19.80	0.25	136:	206.13	6.39e5	15.92	0.20
51:	108.32	8.15e5	20.31	0.25	137:	207.12	6.68e5	16.63	0.21
52:	109.23	7.29e5	18.16	0.23	138:	216.14	6.27e5	15.61	0.20
53:	111.16	1.04e6	25.92	0.33	139:	219.11	7.13e5	17.76	0.22
54:	112.13	1.03e6	26.94	0.34	140:	220.08	6.76e5	16.84	0.21
55:	113.17	2.00e6	49.80	0.62	141:	221.08	6.39e5	15.92	0.20
56:	114.19	2.59e6	64.49	0.81	142:	222.21	6.64e5	16.53	0.21
57:	115.20	2.24e6	55.92	0.70	143:	223.48	1.23e6	30.61	0.38
58:	116.16	1.16e6	28.98	0.36	144:	224.42	1.13e6	28.16	0.35
59:	117.14	1.16e6	29.39	0.37	145:	229.14	6.72e5	16.73	0.21
60:	118.17	2.39e6	59.59	0.75	146:	230.12	7.00e5	17.45	0.22
61:	119.17	1.51e6	37.55	0.47	147:	231.12	6.31e5	15.71	0.20
62:	120.35	8.44e5	21.02	0.26	148:	243.12	6.96e5	17.35	0.22
63:	121.38	1.13e6	28.16	0.35	149:	244.12	6.51e5	16.22	0.20
64:	122.31	7.82e5	19.49	0.24	150:	254.12	6.18e5	15.41	0.19
65:	123.17	7.33e5	18.27	0.23	151:	269.10	6.10e5	15.20	0.19
66:	124.14	1.08e6	26.94	0.34	152:	295.09	8.56e5	21.33	0.27
					153:	296.08	6.23e5	15.51	0.19
					154:	299.06	6.64e5	16.53	0.21
					155:	301.07	1.56e6	38.78	0.49
					156:	323.03	1.26e6	31.43	0.39
					157:	324.04	1.08e6	26.94	0.34
					158:	325.04	8.15e5	20.31	0.25
					159:	338.02	1.31e6	32.65	0.41
					160:	339.06	1.11e6	27.76	0.35
					161:	340.06	2.21e6	55.10	0.69
					162:	341.07	1.36e6	33.88	0.43
					163:	342.01	9.30e5	23.16	0.29
					164:	343.04	6.39e5	15.92	0.20
					165:	350.97	8.03e5	20.00	0.25
					166:	352.96	6.47e5	16.12	0.20
					167:	366.01	6.14e5	15.31	0.19
					168:	367.05	7.05e5	17.55	0.22
					169:	368.05	1.18e6	29.39	0.37
					170:	369.07	1.84e6	45.71	0.57
					171:	370.02	6.31e5	15.71	0.20
					172:	374.93	7.33e5	18.27	0.23
					173:	376.95	8.68e5	21.63	0.27
					174:	377.95	7.21e5	17.96	0.23
					175:	378.95	1.56e6	38.78	0.49
					176:	379.95	6.72e5	16.73	0.21
					177:	380.95	1.44e6	35.92	0.45
					178:	391.95	1.10e6	27.35	0.34
					179:	392.95	9.22e5	22.96	0.29
					180:	393.95	9.18e5	22.86	0.29
					181:	400.95	7.86e5	19.59	0.25
					182:	401.94	7.05e5	17.55	0.22
					183:	402.96	1.93e6	48.16	0.60
					184:	403.95	1.21e6	30.20	0.38
					185:	404.96	1.85e6	46.12	0.58
					186:	405.94	1.41e6	35.10	0.44
					187:	406.94	8.68e5	21.63	0.27
					188:	407.93	9.79e5	24.39	0.31
					189:	415.95	8.97e5	22.35	0.28
					190:	417.99	3.42e6	85.31	1.07
					191:	418.00	3.29e6	82.04	1.03
					192:	419.99	3.60e6	89.80	1.13
					193:	421.00	3.39e6	84.48	1.06
					194:	421.97	2.79e6	69.39	0.87
					195:	422.98	2.74e6	68.16	0.86
					196:	423.97	1.08e6	26.94	0.34
					197:	443.93	1.52e6	37.96	0.48
					198:	444.96	3.64e6	90.61	1.14
					199:	445.94	2.83e6	70.61	0.89
					200:	446.96	4.01e6	100.00	1.26
					201:	447.99	3.23e6	80.41	1.01
					202:	448.99	3.83e6	95.51	1.20
					203:	449.97	2.90e6	72.24	0.91
					204:	450.97	9.42e5	23.47	0.29

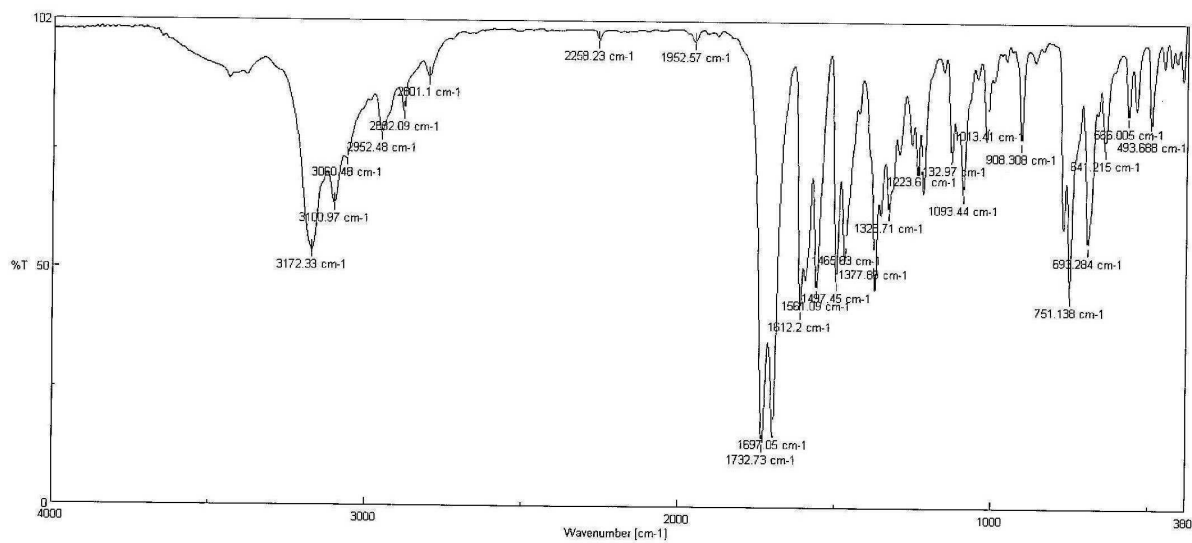
S-64: Sample description presented by mass instrument to compound 4{1,3,5}

Sample Code: IT900830-134 (KE-213)

Central Laboratory of the University of Isfahan

Instrument Specifications

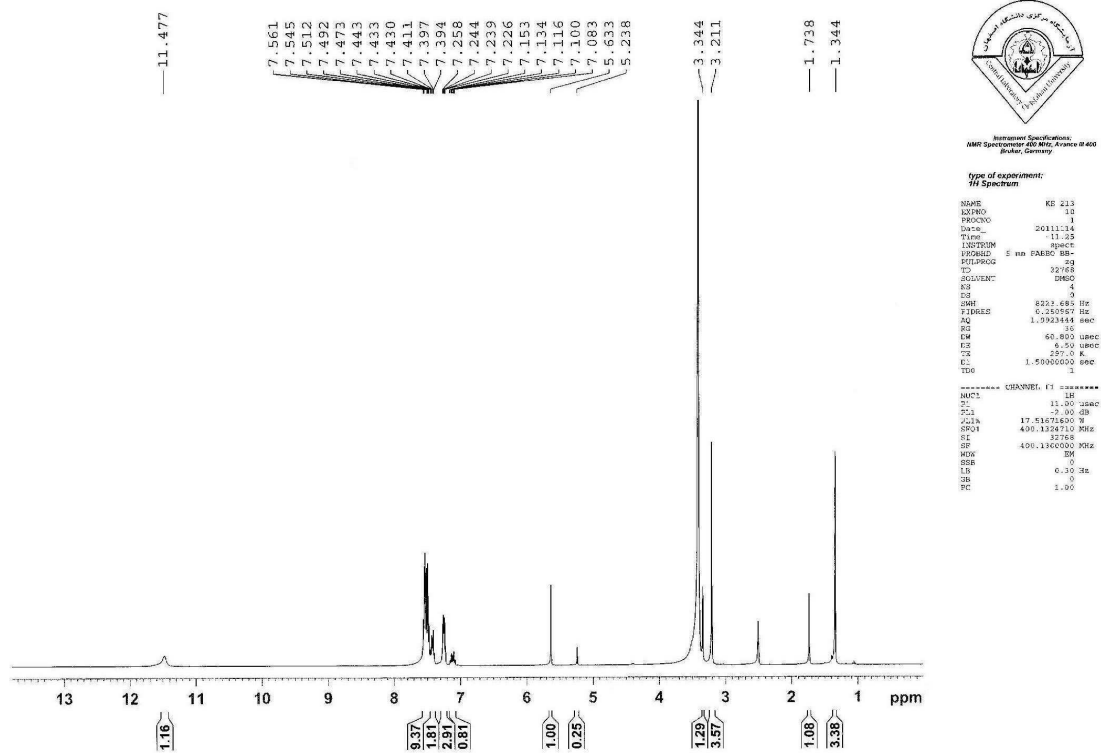
FT Infrared Spectroscopy, JASCO, FT/IR-6300 (4000-400 cm⁻¹), Japan



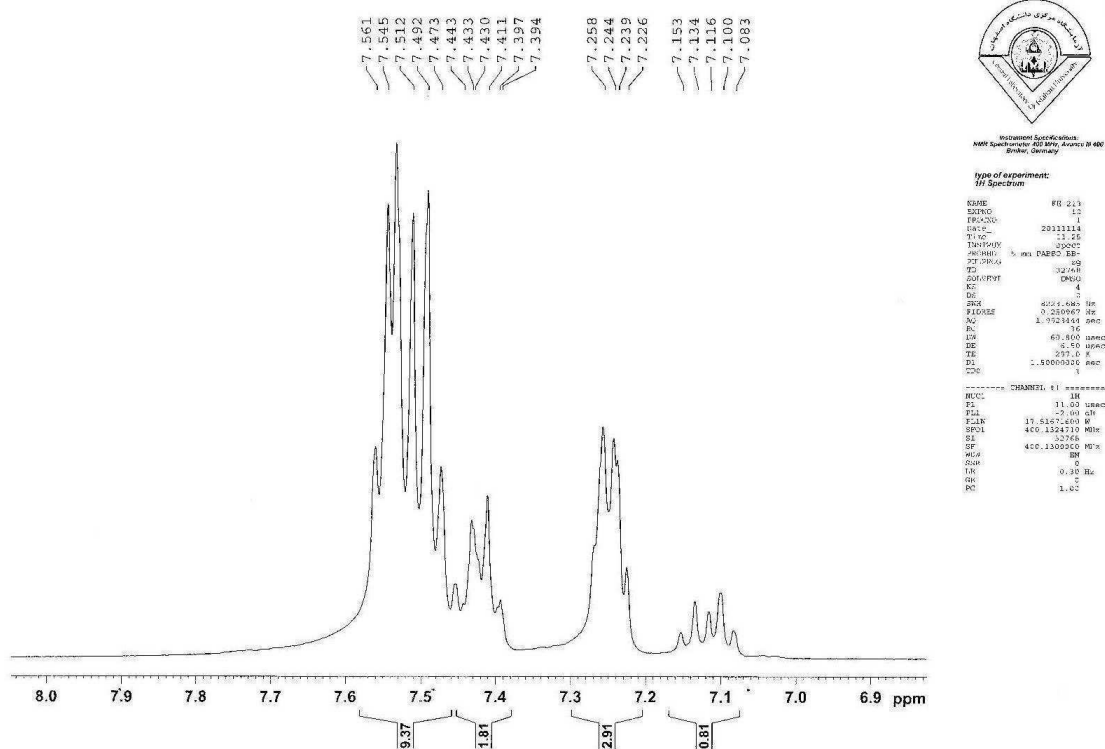
Date: 90/09/05

No.:

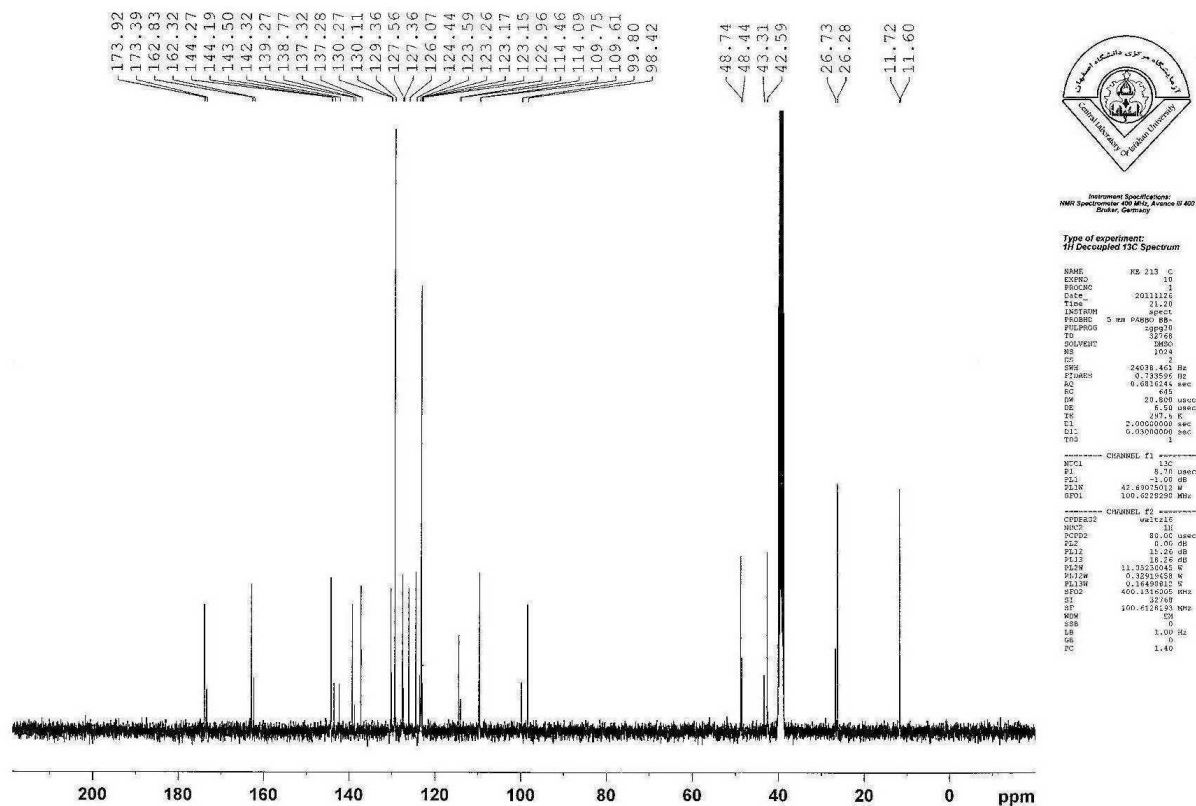
S-65: IR spectrum of compound 4{1,3,6}



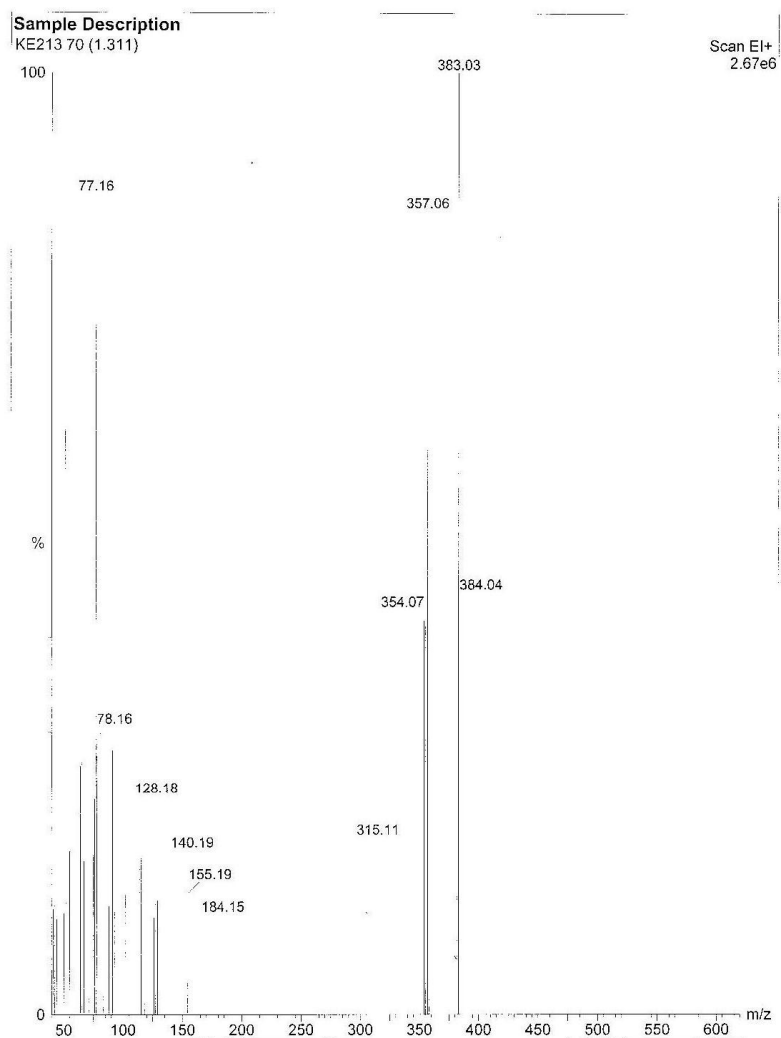
S-66: ^1H NMR spectrum of compound 4{1,3,6}



S-66: Expand of ^1H NMR spectrum of compound 4{1,3,6}



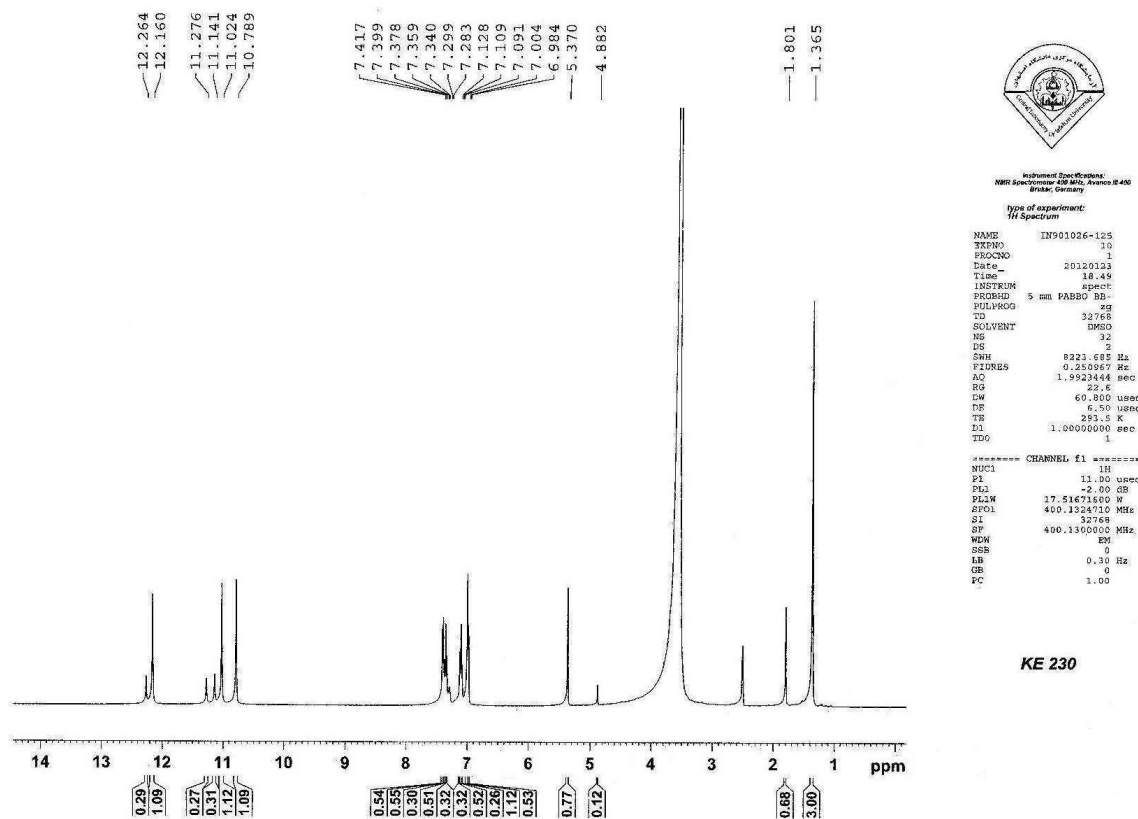
S-67: ^{13}C NMR spectrum of compound 4{1,3,6}



S-68: Mass spectrum of compound 4{1,3,6}

Sample Description										Scan El+				
KE213 70 (1.311)					No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC
1:	41.25	2.99e5	11.20	0.57										
2:	42.25	4.51e5	16.87	0.86										
3:	43.25	7.41e5	27.76	1.42										
4:	44.20	2.70e5	10.12	0.52										
5:	50.19	2.87e5	10.74	0.55										
6:	51.20	1.65e6	61.96	3.17										
7:	52.20	3.19e5	11.96	0.61										
8:	55.20	4.63e5	17.33	0.89										
9:	57.22	7.17e5	26.84	1.37										
10:	63.16	6.31e5	23.62	1.21										
11:	64.16	7.05e5	26.38	1.35										
12:	65.18	8.77e5	32.82	1.68										
13:	66.17	3.73e5	13.96	0.71										
14:	67.15	4.34e5	16.26	0.83										
15:	69.20	5.37e5	20.09	1.03										
16:	71.20	4.18e5	15.64	0.80										
17:	75.14	4.51e5	16.87	0.86										
18:	76.15	6.10e5	22.85	1.17										
19:	77.16	2.33e6	87.12	4.46										
20:	78.16	7.86e5	29.45	1.51										
21:	83.21	2.91e5	10.89	0.56										
22:	88.15	3.07e5	11.50	0.59										
23:	89.16	3.69e5	13.80	0.71										
24:	91.16	7.50e5	28.07	1.44										
25:	92.17	3.28e5	12.27	0.63										
26:	93.19	2.91e5	10.89	0.56										
27:	101.17	3.81e5	14.26	0.73										
28:	102.16	3.40e5	12.73	0.65										
29:	114.18	4.14e5	15.49	0.79										
30:	115.19	4.42e5	16.56	0.85										
31:	116.18	2.83e5	10.58	0.54										
32:	118.20	4.79e5	17.94	0.92										
33:	126.18	2.74e5	10.28	0.53										
34:	127.18	4.92e5	18.40	0.94										
35:	128.18	6.18e5	23.16	1.19										
36:	129.15	3.24e5	12.12	0.62										
37:	140.19	4.34e5	16.26	0.83										
38:	154.20	2.83e5	10.58	0.54										
39:	155.19	3.44e5	12.88	0.66										
40:	184.15	2.83e5	10.58	0.54										
41:	315.11	5.00e5	18.71	0.96										
42:	354.07	1.11e6	41.72	2.13										
43:	355.09	1.10e6	41.10	2.10										
44:	356.07	3.28e5	12.27	0.63										
45:	357.06	2.28e6	85.28	4.36										
46:	358.07	5.98e5	22.39	1.15										
47:	382.03	3.36e5	12.58	0.64										
48:	383.03	2.67e6	100.00	5.12										
49:	384.04	1.16e6	43.56	2.23										

S-69: Sample description presented by mass instrument to compound 4{1,3,6}



S-70: ^1H NMR spectrum of compound $4\{2,1,1\}^e$

S71

Production and separation of 5{-,1,1} and 4{1,1,1} via three component reaction (Entries 12 and 13, Table1): A solution of isatin (1mmol), ethyl cyanoacetate (1 mmol) and Et₃N (0.1 mmol) in aprotic solvents (10 ml) such as ethyl acetate and acetonitrile in a balloon (25 mL) was stirred for 1 h at 60-70°C in an oil bath. Then 3-amin-pyrazole (1 mmol) was added in balloon and stirred for 24 h under reflux condition in an oil bath. After 24 h, which has been followed by TLC (EtOAc:*n*-hexene, 1:2), the solvent was removed using vacuum. The participate was washed with water prior to chromatographic separation (EtOAc:*n*-hexene (4:1)). The products 5{-,1,1} and 4{1,1,1} were obtained as pure compounds.

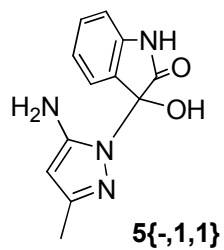
S72

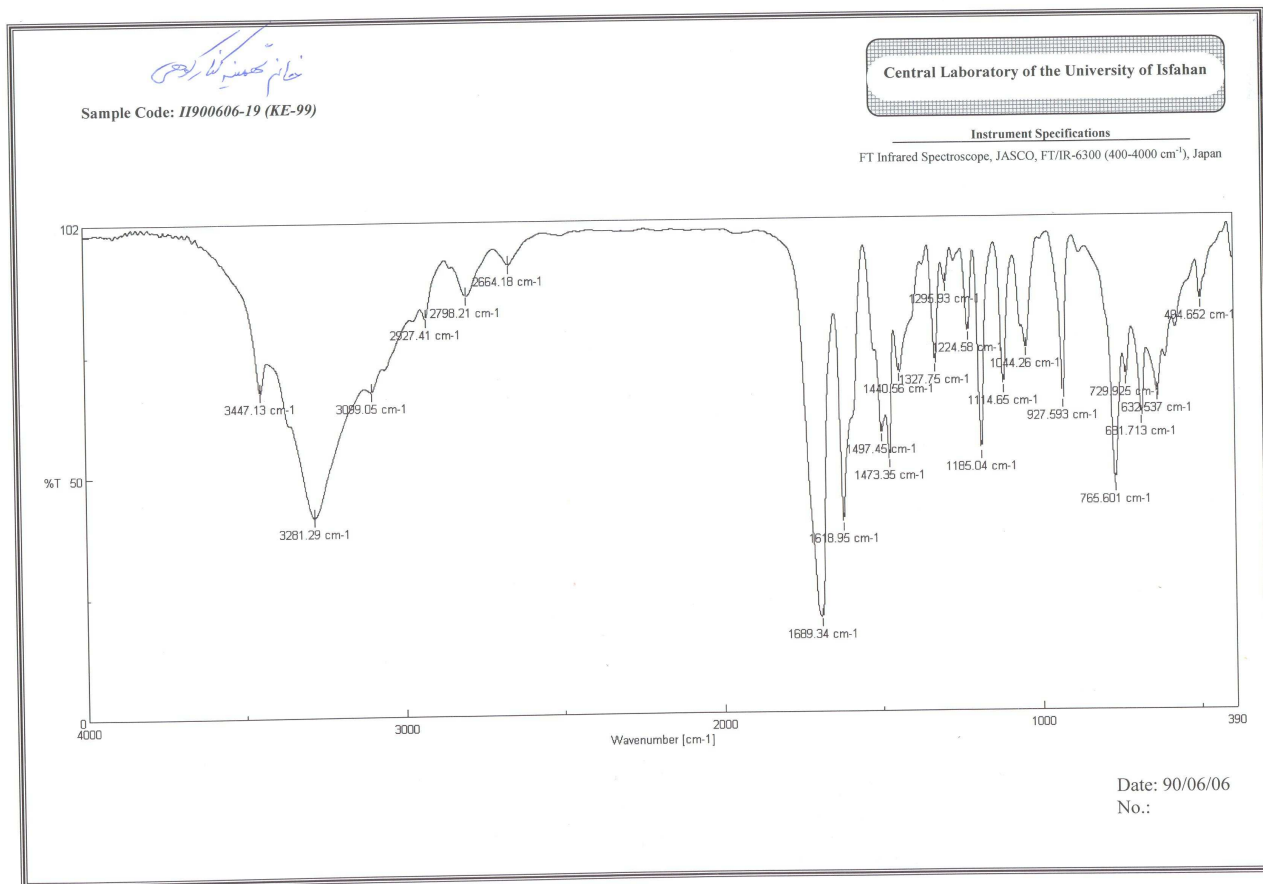
Procedure for the synthesis of 3-(5-amino-3-methyl-1H-pyrazol-1-yl)-3-hydroxyindolin-2-one 5{-,1,1}: A solution of isatin (1mmol), 3-amin-pyrazole (1 mmol) in acetonitrile (10 ml) in a balloon (25 mL) was stirred for 20 min at 60-70°C in oil bath. After the completion of the reactions, which has been followed by TLC (EtOAc:*n*-hexene, 1:2), the reaction mixture was filtered as warm and pure product was obtained.

S73

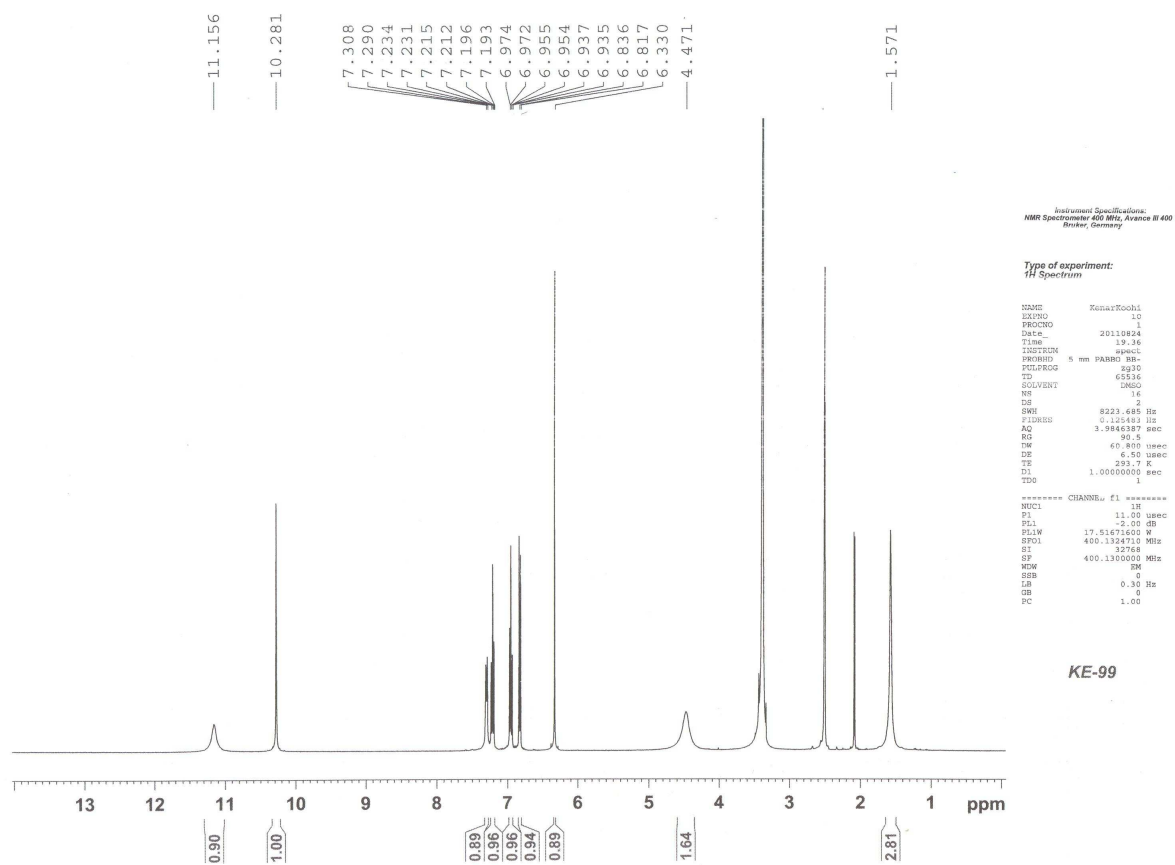
Representative data 5{-,1,1}: white powder (87%); IR (KBr): 3447, 3281, 2927, 1689, 1619, 1497, 1473, 1185, 765 cm⁻¹; Anal.Calcd for C₁₂H₁₂N₄O₂: C 59.01, H 4.95, N 22.94; Found: C 58.81, H 4.91, N 23.01. ¹H NMR (400 MHz, DMSO-d₆) δ: 1.57 (s, 3H, CH₃), 4.47 (s, 2H, NH₂), 6.33 (s, 1H, CH_{pyrazole}), 6.83 (d, ³J = 7.6 Hz, 1H, CH_{arom}), 6.93-6.97 (m, 1H, CH_{arom}), 7.19-7.23

(m, 1H, CH_{arom}), 7.30 (d, $^3J = 7.2$ Hz, 1H, CH_{arom}), 10.28 (s, 1H, NH_{amide}), 11.16 (bs, 1H, OH) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ : 10.77, 74.14, 109.57, 121.68, 124.78, 129.06, 133.24, 141.27, 178.30 ppm.

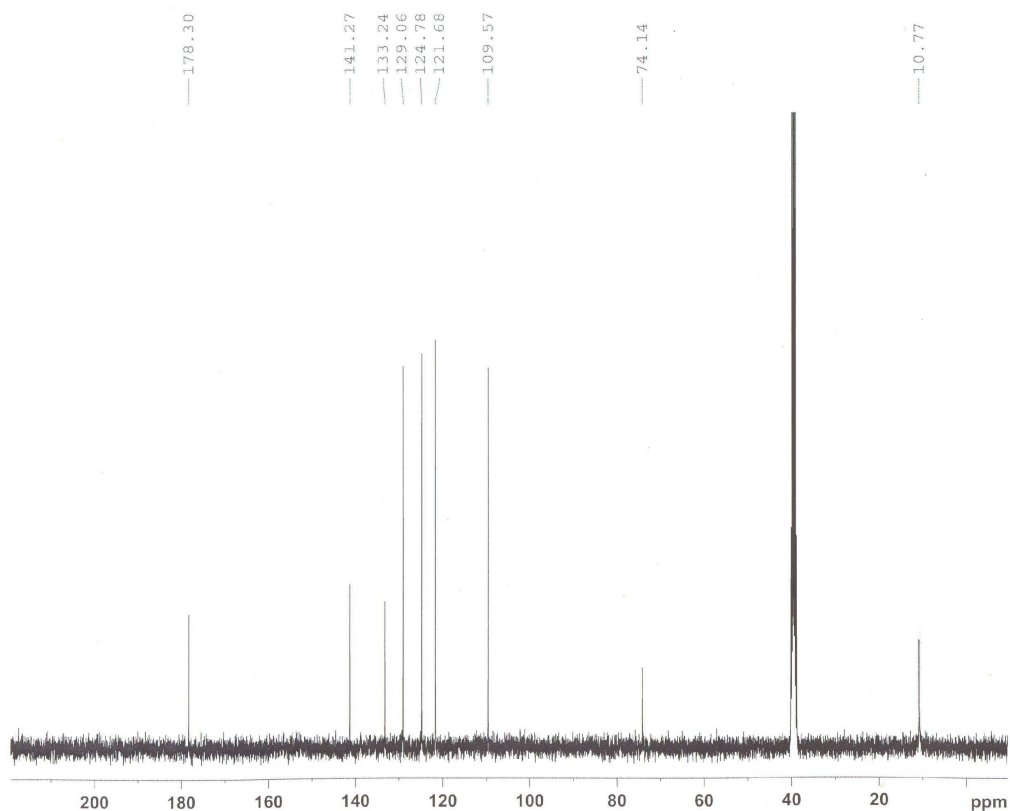




S-74: IR spectrum of compound 5{-,1,1}



S-75: ^1H NMR spectrum of compound **5{-,1,1}**



Instrument Specifications:
NMR Spectrometer 400 MHz, Avance III 400
Bruker, Germany

Type of experiment:
1H Decoupled 13C Spectrum

NAME IN910224-14..16
EXPNO 16
PROCNO 1
Date_ 20120516
Time 0.45
INSTRUM spect
PROBHD 5 mm PABBO-BB
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 2048
DS 4
SWH 26041.606 Hz
FIDRES 0.794720 Hz
AQ 0.6291950 sec
RG 2050
DE 19.430 umsec
DM 5.50
TE 304.1 K
CL 1.00000000 sec
C11 0.03000000 sec
C10 1

***** CHANNEL f1 *****
NUC1 13C
P1 12.70 umsec
PL1 -1.00 dB
PL1X 42.85910012 K
SFO1 100.628364 MHz

***** CHANNEL f2 *****
CPOB2 waltz16
NUC2 1H
PCPD2 80.00 umsec
PL2 0.00 dB
PL12 15.26 dB
PL13 15.26 dB
PL2W 11.05230045 W
PL1W 9.32291450 W
PL13W 9.16498812 W
SFO2 400.1116000 MHz
SI 32768
SF 100.6128183 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

END OF FILE
C:\NMR\N1\N1000001.D

S-76: ^{13}C NMR spectrum of compound 5{-,1,1}