An efficient approach for the synthesis of 5-hydroxy-chromeno[2,3-b]pyridines under catalyst and solvent free conditions

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SPECTRAL DATA

1-(4-fluorophenyl)-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVa)

Yellow solid, M.p: 236-240 °C, ν_{max} (DCM/cm^{-1}): 3318, 2219, 1565, 1149. δ_H (400 MHz, DMSO-d_6): δ 9.47 (s, 1H, OH), 8.06 (s, 1H, CH_{vinyl}), 7.65 (d, 1H, J = 8.4 Hz, ArH), 7.64 (m, 2H, ArH), 7.22 (m, 1H, ArH), 7.20 (m, 3H, ArH), 7.08(m, 1H, ArH), 6.20 (d, 1H, J = 4.4 Hz, CH), 5.50 (d, 1H, J = 5.6 Hz, NH). δ_C (100 MHz, DMSO-d_6): 158.49 (d, $^1J_{C\text{-}F}$ =238.30 Hz), 156.16, 153.39, 148.86, 141.68, 135.43 (d, $^4J_{C–F}$ = 1.91 Hz),, 133.70, 125.10, 124.12 (d, $^3J_{C–F}$ = 8.60 Hz), 123.60, 120.30, 117.45, 116.35, 115.13 (d, $^2J_{C–F}$ = 22.9 Hz),, 112.63. 91.42, 73.16. HRMS (ESI) m/z calcd for $C_{19}H_{12}FN_3O_2$ $[M+H]^+$ 334.0992, found: 334.0991.

1-(4-chlorophenyl)-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVb)

Yellow solid, M.p: 260-262 °C, ν_{max} (DCM/cm^{-1}): 3314, 2228, 1561, 1149. δ_H (400 MHz, DMSO-d_6): δ 9.56 (s, 1H, OH), 8.08 (s, 1H, CH_{vinyl}), 7.86 (d, 1H, J = 6.8 Hz, ArH), 7.68 (m, 2H, ArH), 7.45 (m, 3H, ArH), 7.16 (m, 2H, ArH), 6.20 (d, 1H, J = 5.6 Hz, CH), 5.51 (d, 1H, J = 4.4 Hz, NH). δ_C (100 MHz, DMSO-d_6): 156.75, 153.38, 148.83, 141.73, 138.21, 133.73, 128.39, 127.04, 125.16, 123.64, 123.26, 120.22, 117.45, 116.25, 113.05, 91.99, 73.12. HRMS (ESI) m/z calcd for $C_{19}H_{12}ClN_3O_2$ $[M+H]^+$ 350.0696, found: 350.0669.

1-(4-bromophenyl)-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVc)

Yellow solid, M.p: 270-272 °C, ν_{max} (DCM/cm^{-1}): 3315, 2225, 1560, 1148. δ_H (400 MHz, DMSO-d_6): δ 9.58 (s, 1H, OH), 8.11 (s, 1H, CH_{vinyl}), 7.90 (d, 1H, *J* = 7.6 Hz, ArH), 7.66 (m, 5H, ArH), 7.19 (m, 2H, ArH), 6.23 (d, 1H, *J* = 4.4 Hz, CH), 5.54 (d, 1H, *J* = 5.2 Hz, NH). δ_C (100 MHz, DMSO-d_6): 155.67, 153.30, 148.84, 141.76, 138.66, 133.75, 131.30, 125.19, 123.67, 123.58, 120.22, 117.46, 116.23, 115.06, 113.10, 92.08, 73.13. HRMS (ESI) m/z calcd for $C_{19}H_{12}BrN_3O_2$ $[M+H]^+$ 394.0191, found: 394.0164.

1-(3-chlorophenyl)-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVd**)**

Yellow solid, M.p: 188-190 °C, ν_{max} (DCM/cm^{-1}): 3362, 2217, 1586, 1147. δ_H (400 MHz, DMSO-d_6): δ 9.63 (s, 1H, OH), 8.11 (s, 1H, CH_{vinyl}), 7.87 (m, 2H, ArH), 7.64 (m, 1H, ArH), 7.38 (m, 2H, ArH), 7.13 (m, 5H, ArH), 6.23 (d, 1H, *J* = 5.6 Hz, CH), 5.52 (d, 1H, *J* = 5.2 Hz, NH). HRMS (ESI) m/z calcd for $C_{19}H_{12}ClN_3O_2$ $[M+H]^+$ 350.0696, found: 350.0681.

1-(4-methoxyphenyl)-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVe**)**

Yellow solid, M.p: 198-200 °C, ν_{max} (DCM/cm^{-1}): 3330, 2221, 1564, 1213. δ_H (400 MHz, DMSO-d_6): δ 9.28 (s, 1H, OH), 8.02 (s, 1H, CH_{vinyl}), 7.84(d, 1H, *J* = 7.6 Hz, ArH), 7.53 (m, 3H, ArH), 7.14 (m, 4H, ArH), 6.18 (s, 1H, CH), 5.50 (s, 1H, NH), 3.73 (s, 3H, OCH_3). δ_C (100 MHz, DMSO-d_6): 156.38, 155.81, 153.38, 148.89, 141.56, 133.56, 131.96, 125.07, 124.03, 123.50, 120.39, 117.42, 116.47, 113.67, 92.85, 73.21, 55.26. HRMS (ESI) m/z calcd for $C_{20}H_{15}N_3O_3$ $[M+H]^+$ 346.1192, found: 346.1191.

1-(4-methylphenyl)-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVf**)**

Yellow solid, M.p: 194-196 °C, ν_{max} (DCM/cm^{-1}): 3330, 2222, 1603, 1151. δ_H (400 MHz, DMSO-d_6): δ 9.34 (s, 1H, OH), 8.04 (s, 1H, CH_{vinyl}), 7.85 (d, 1H, *J* = 7.2 Hz, ArH), 7.52 (m, 3H, ArH), 7.16 (m, 4H, ArH), 6.19 (d, *J* = 4.0 Hz, 1H, CH), 5.50 (d, *J* = 4.0 Hz, 1H, NH), 2.27 (s, 3H, CH_3). δ_C (100 MHz, DMSO-d_6): 156.12, 153.39, 148.87, 141.65, 136.56, 133.65, 132.66, 128.97, 125.06, 123.57, 121.97, 120.38, 117.48, 116.45, 112.35, 91.38, 73.18, 20.64. HRMS (ESI) m/z calcd for $C_{20}H_{15}N_3O_2$ $[M+H]^+$ 330.1243, found: 330.1225.

1-(2-methylphenyl)-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVg**)**

Yellow solid, M.p: 198-200 °C, ν_{max} (DCM/cm^{-1}): 3401, 2217, 1559, 1152. δ_H (400 MHz, DMSO-d_6): δ 9.13 (s, 1H, OH), 8.02 (s, 1H, CH_{vinyl}), 7.56 (d, 1H, *J* = 6.8 Hz, ArH), 7.42 (m, 5H, ArH), 7.06 (m, 2H, ArH), 6.17 (d, 1H, *J* = 4.4 Hz, CH), 5.49 (s, 1H, NH), 2.14 (s, 3H, OCH_3). δ_C (100 MHz, DMSO-d_6): 157.09, 153.31, 148.90, 141.37, 137.28, 134.20, 133.50, 130.38, 126.60, 126.17, 126.06, 124.69, 123.38, 120.30, 117.38, 116.40, 90.31, 73.16, 18.09. HRMS (ESI) m/z calcd for $C_{20}H_{15}N_3O_2$ $[M+H]^+$ 330.1243, found: 330.1237.

5-hydroxy-2-imino-1-(pyridin-3-yl)-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVh)

Yellow solid, M.p: 226-228 °C, ν_{max} (DCM/cm^{-1}): 3321, 2221, 1562, 1200. δ_H (400 MHz, DMSO-d_6): δ 9.65 (s, 1H, OH), 8.68 (m, 1H, CH_{vinyl}), 8.29 (d, 1H, *J* = 4.4 Hz, ArH), 8.11 (m, 2H, ArH), 7.83 (d, 1H, *J* = 7.6 Hz, ArH), 7.46 (m, 2H, ArH), 7.18 (m, 1H, ArH), 7.09 (m, 1H, ArH), 6.21 (s, 1H, CH), 5.51 (s, 1H, NH). δ_C (100 MHz, DMSO-d_6): 155.84, 153.48, 148.89, 144.07, 143.21, 141.80, 136.06, 133.81, 129.14, 124.98, 123.58, 123.50, 120.20, 117.55, 116.25, 92.04, 73.15. HRMS (ESI) m/z calcd for $C_{18}H_{12}N_4O_2$ $[M+H]^+$ 317.1039, found: 317.1034.

Methyl 4-(3-cyano-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridin-1-yl) benzoate (IVi)

Yellow solid, M.p: 218-220 °C, ν_{max} (DCM/cm^{-1}): 3372, 2215, 1717, 1558, 1184. δ_H (400 MHz, DMSO-d_6): δ 9.80 (s, 1H, OH), 8.13 (s, 1H, CH_{vinyl}), 7.95 (m, 3H, ArH), 7.86 (m, 2H, ArH), 7.49 (m, 1H, ArH), 7.28 (m, 2H, ArH), 6.23 (d, 1H, *J* = 4.8 Hz, CH), 5.52 (d, 1H, *J* = 4.8 Hz, NH). δ_C (100 MHz, DMSO-d_6): 165.99, 155.35, 153.41, 148.89, 144.09, 141.86, 133.87, 130.07, 129.93, 125.27, 123.53, 121.80, 120.09, 117.49, 116.12, 113.95, 93.12, 73.09, 51.88. HRMS (ESI) m/z calcd for $C_{21}H_{15}N_3O_4$ $[M+H]^+$ 374.1141, found: 374.1131.

1-(4-iodophenyl)-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVj**)**

Yellow solid, M.p: 240-242 °C, ν_{max} (DCM/cm^{-1}): 3321, 2223, 1554, 1148. δ_H (400 MHz, DMSO-d_6): δ 9.55 (s, 1H, OH), 8.10 (s, 1H, CH_{vinyl}), 7.91 (d, 1H, *J* = 8.0 Hz, ArH), 7.71 (m, 2H, ArH), 7.54 (m, 3H, ArH), 7.21 (m, 1H, ArH), 7.09 (d, 1H, *J* = 8.0 Hz, ArH), 6.23 (d, 1H, *J*

= 5.2 Hz, CH), 5.53 (d, 1H, *J* = 5.6 Hz, NH). δ_C (100 MHz, DMSO-d_6): 155.60, 153.37, 148.83, 141.74, 139.17, 137.12, 133.74, 125.19, 123.74, 123.68, 120.22, 117.45, 116.24, 113.11, 92.17, 86.90, 73.12. HRMS (ESI) m/z calcd for $C_{19}H_{12}IN_3O_2$ $[M+H]^+$ 442.0052, found: 442.0039.

1-(3-bromophenyl)-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVk**)**

Yellow solid, M.p: 202-204 °C, ν_{max} (DCM/cm^{-1}): 3332, 2221, 1552, 1199. δ_H (400 MHz, DMSO-d_6): δ 9.62 (s, 1H, OH), 8.11 (s, 1H, CH_{vinyl}), 8.01 (s, 1H, ArH), 7.88 (d, 1H, *J* = 7.6 Hz, ArH), 7.66 (d, 1H, *J* = 7.6 Hz, ArH), 7.49 (m, 1H, ArH), 7.32 (m, 4H, ArH), 6.22 (d, 1H, *J* = 4.4 Hz, CH), 5.52 (d, 1H, *J* = 5.6 Hz, NH). δ_C (100 MHz, DMSO-d_6): 155.61, 153.46, 148.78, 141.84, 140.98, 133.90, 130.49, 125.82, 124.90, 123.90, 123.61, 121.92, 120.23, 117.62, 116.15, 92.34, 73.12. HRMS (ESI) m/z calcd for $C_{19}H_{12}BrN_3O_2$ $[M+H]^+$ 394.0191, found: 394.0190.

1-(2,3-dichlorophenyl)-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVl)

Yellow solid, M.p: 230-234 °C, ν_{max} (DCM/cm^{-1}): 3303, 2222, 1519, 1139. δ_H (400 MHz, DMSO-d_6): δ 9.48 (s, 1H, OH), 8.10 (s, 1H, CH_{vinyl}), 7.64 (m, 3H, ArH), 7.43 (m, 3H, ArH), 7.07 (m, 1H, ArH), 6.21 (d, 1H, *J* = 3.6 Hz, CH), 5.52 (d, 1H, *J* = 4.4 Hz, NH). δ_C (100 MHz, DMSO-d_6): 156.41, 153.38, 148.88, 141.36, 138.15, 133.77, 131.02, 128.17, 127.45, 126.26, 124.77, 123.54, 120.10, 117.46, 116.11, 113.04, 91.46, 73.10. HRMS (ESI) m/z calcd for $C_{19}H_{12}Cl_2N_3O_2$ $[M+H]^+$ 384.0307, found: 384.0308.

1-(3-cyano-4-fluorophenyl)-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVm)

Yellow solid, M.p: 234-236 °C, ν_{max} (DCM/cm^{-1}): 3314, 2227, 1524, 1153. δ_H (400 MHz, DMSO-d_6): δ 9.76 (s, 1H, OH), 8.12 (m, 2H, ArH and CH_{vinyl}), 8.10 (m, 1H, ArH), 7.81 (d, 1H, *J* = 8.0 Hz, ArH), 7.54 (m, 2H, ArH), 7.15 (m, 1H, ArH), 7.10 (m, 1H, ArH), 6.22 (d, 1H, *J* = 5.2 Hz, CH), 5.52 (d, 1H, *J* =4.8 Hz, NH). δ_C (100 MHz, DMSO-d_6): 158.34 (d, $^1J_{C-F}$ = 250.8 Hz) 155.64, 153.44, 148.78, 141.81, 136.36 (d, $^4J_{C-F}$ = 1.9 Hz), 133.88, 129.41 (d, $^3J_{C-F}$ = 8.6 Hz), 126.16, 124.95, 123.60, 120.11, 117.55, 116.73 (d, $^2J_{C-F}$ = 21 Hz), 116.07, 114.13, 113.55,111.94 (d, $^2J_{C-F}$ = 22.9 Hz), 99.59, 92.04, 73.09. HRMS (ESI) m/z calcd for $C_{20}H_{11}FN_4O_2$ $[M+H]^+$ 359.0944, found: 359.0942,

1-(2,4-dimethoxyphenyl)-5-hydroxy-2-imino-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVn)

Yellow solid, M.p: 220-224 °C, ν_{max} (DCM/cm^{-1}): 3377, 2207, 1559, 1147. δ_H (400 MHz, DMSO-d_6): δ 8.59 (s, 1H, OH), 7.98 (s, 1H, CH_{vinyl}), 7.80 (m, 1H, ArH), 7.88 (d, 1H, *J* = 7.6 Hz, ArH), 7.60 (d, 1H, *J* = 7.6 Hz, ArH), 7.46 (m, 1H, ArH), 7.15 (m, 1H, ArH), 7.06 (m, 1H, ArH), 6.66 (m, 1H, ArH), 6.58 (m, 1H, ArH), 6.17 (d, 1H, *J* = 4.8 Hz, CH), 5.49 (d, 1H, *J* = 4.4 Hz, NH), 3.76 (s, 6H, 2 OCH_3). δ_C (100 MHz, DMSO-d_6): 157.81, 156.81, 153.62, 153.36, 148.95, 141.02, 133.54, 125.58, 124.92, 123.48, 120.38, 117.44, 116.39, 112.15,111.54, 104.31, 99.11, 90.72, 73.18, 55.89, 55.41. HRMS (ESI) m/z calcd for $C_{21}H_{17}N_3O_4$ $[M+H]^+$ 376.1297, found: 376.1293.

7-bromo-5-hydroxy-2-imino-1-(4-methoxyphenyl)-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVo)

Yellow solid, M.p: 250-252 °C, ν_{max} (DCM/cm^{-1}): 3377, 2211, 1556, 1201. δ_H (400 MHz, DMSO-d_6): δ 9.39 (s, 1H, OH), 8.03 (s, 1H, CH_{vinyl}), 7.84 (s, 1H, ArH), 7.58 (d, 1H, *J* = 8.4 Hz, ArH), 7.46 (d, 2H, *J* = 8.8 Hz, ArH), 7.04 (d, 1H, *J* = 8.8 Hz, ArH), 6.92 (d, 2H, *J* = 8.4 Hz, ArH), 6.21 (d, 1H, *J* = 4.8 Hz,, CH), 5.51 (d, 1H, *J* = 5.2 Hz, NH). δ_C (100 MHz, DMSO-d_6): 156.41, 156.05, 152.53, 147.36, 141.87, 135.75, 131.65, 127.12, 124.48, 122.21, 119.87, 116.22, 114.98, 113.56, 111.81, 91.54, 73.36, 55.30. HRMS (ESI) m/z calcd for $C_{20}H_{14}BrN_3O_3$ $[M+H]^+$ 424.0297, found: 424.0286.

7-bromo-5-hydroxy-2-imino-1-(4-iodophenyl)-2,10*a*-dihydro-1*H*-chromeno[2,3-*b*]pyridine-3-carbonitrile (IVp)

Yellow solid, M.p: 240-244 °C, ν_{max} (DCM/cm^{-1}): 3327, 2223, 1551, 1197. δ_H (400 MHz, DMSO-d_6): δ 9.62 (s, 1H, OH), 8.10 (s, 1H, CH_{vinyl}), 7.88 (d, *J* = 5.6Hz, 1H, ArH), 7.67 (m, 3H, ArH), 7.46 (m, 2H, ArH), 7.07 (d, 1H, *J* =8.4 Hz, ArH), 6.25 (d, *J* = 5.2Hz, 1H, CH), 5.53 (d, *J* = 4.8 Hz, 1H, NH). HRMS (ESI) m/z calcd for $C_{19}H_{11}BrIN_3O_2$ $[M+H]^+$ 519.9158, found: 519.9134.

7-chloro-1-(4-fluorophenyl)-5-hydroxy-2-imino-8-methyl-2,10a-dihydro-1H-chromeno[2,3-b]pyridine-3-carbonitrile (IVq)

Yellow solid, M.p: 240-244 °C, ν_{max} (DCM/cm^{-1}): 3327, 2211, 1551, 1191. δ_H (400 MHz, DMSO-d_6): δ 9.50 (s, 1H, OH), 8.04 (s, 1H, CH_{vinyl}), 7.65 (s, 1H, ArH), 7.60 (m, 2H, ArH), 7.20 (m, 3H, ArH), 6.18 (d, J = 4.4 Hz, 1H, CH), 5.48 (d, J = 4.4 Hz, 1H, NH), 2.30 (s,3H, CH_3). δ_C (100 MHz, DMSO-d_6): 158.61 (d, $^1J_{C\text{-}F}$ =239.43Hz), 156.15, 151.89, 147.65, 141.77, 141.52 135.22, 127.80, 124.42 (d, $^3J_{C–F}$ = 7.60 Hz), 119.92, 119.44, 116.20, 114.99 (d, $^2J_{C–F}$ = 22.9 Hz),, 112.13, 91.62, 73.36. HRMS (ESI) m/z calcd for $C_{20}H_{13}ClFN_3O_2$ $[M+H]^+$ 382.0753, found: 382.0729.

1-(4-fluorophenyl)-5-hydroxy-2-imino-7-methyl-2,10a-dihydro-1H-chromeno[2,3-b]pyridine-3-carbonitrile (IVr)

Yellow solid, M.p: 240-244 °C, ν_{max} (DCM/cm^{-1}): 3347, 2223, 1561, 1190. δ_H (400 MHz, DMSO-d_6): δ 9.47 (s, 1H, OH), 8.04 (s, 1H, CH_{vinyl}), 7.66 (m, 3H, ArH), 7.28 (m, 3H, ArH), 6.97 (d, 1H, J =8.4 Hz, ArH), 6.15 (d, J = 5.6 Hz, 1H, CH), 5.47 (d, J = 5.6 Hz, 1H, NH). δ_C (100 MHz, DMSO-d_6): 158.44 (d, $^1J_{C\text{-}F}$ =238.30 Hz), 156.03, 151.27, 148.95, 141.61, 135.42 (d, $^4J_{C–F}$ = 2.90 Hz),, 134.29, 132.44, 125.02, 123.97 (d, $^3J_{C–F}$ = 7.60 Hz), 119.94, 117.32, 116.33, 115.02 (d, $^2J_{C–F}$ = 21.9 Hz), 112.74, 91.20, 73.02. HRMS (ESI) m/z calcd for $C_{20}H_{15}FN_3O_2$ $[M+H]^+$ 348.1148, found: 348.1121.

^{1}H NMR OF IVa

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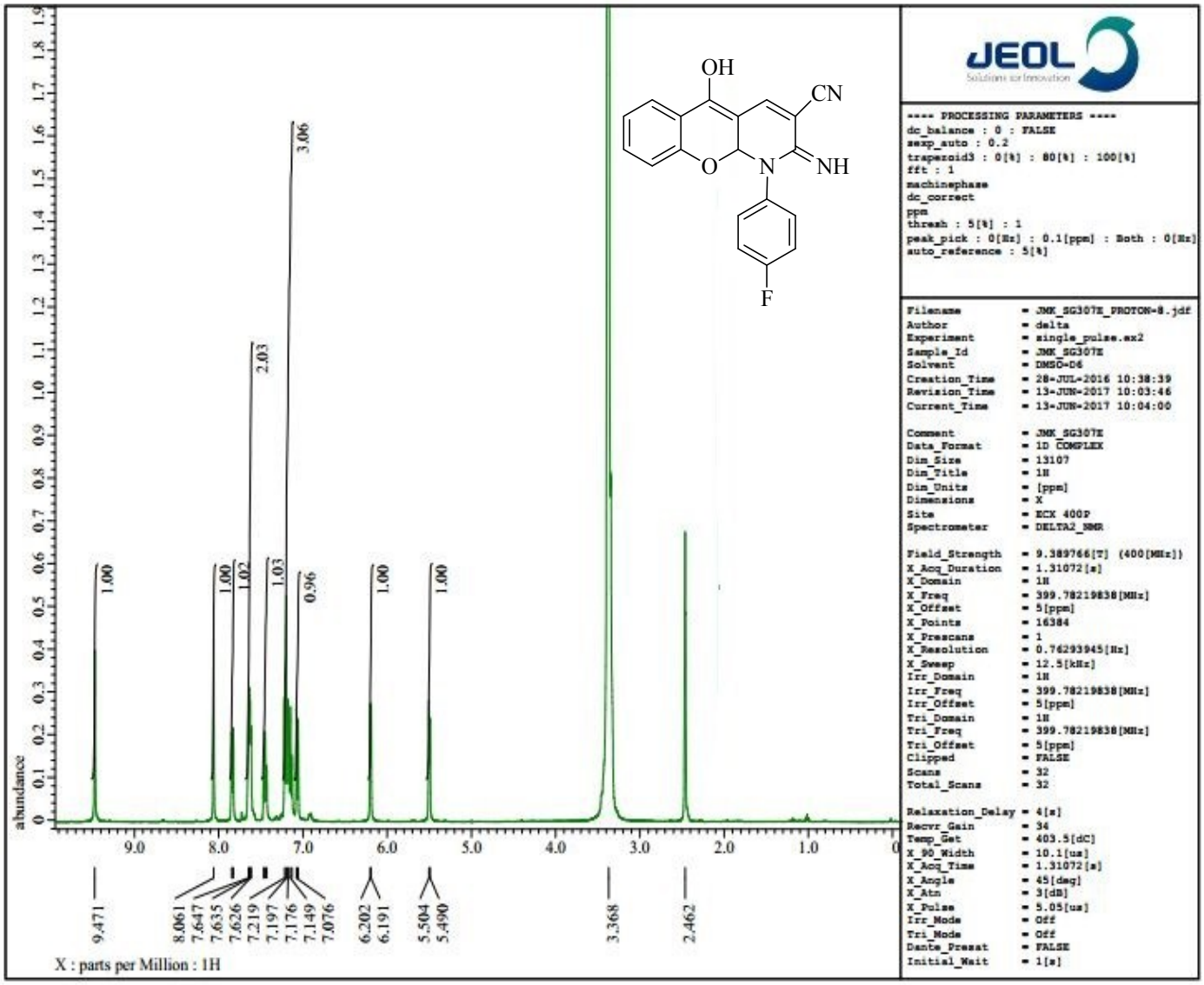
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JEOL
Solutions for Innovation
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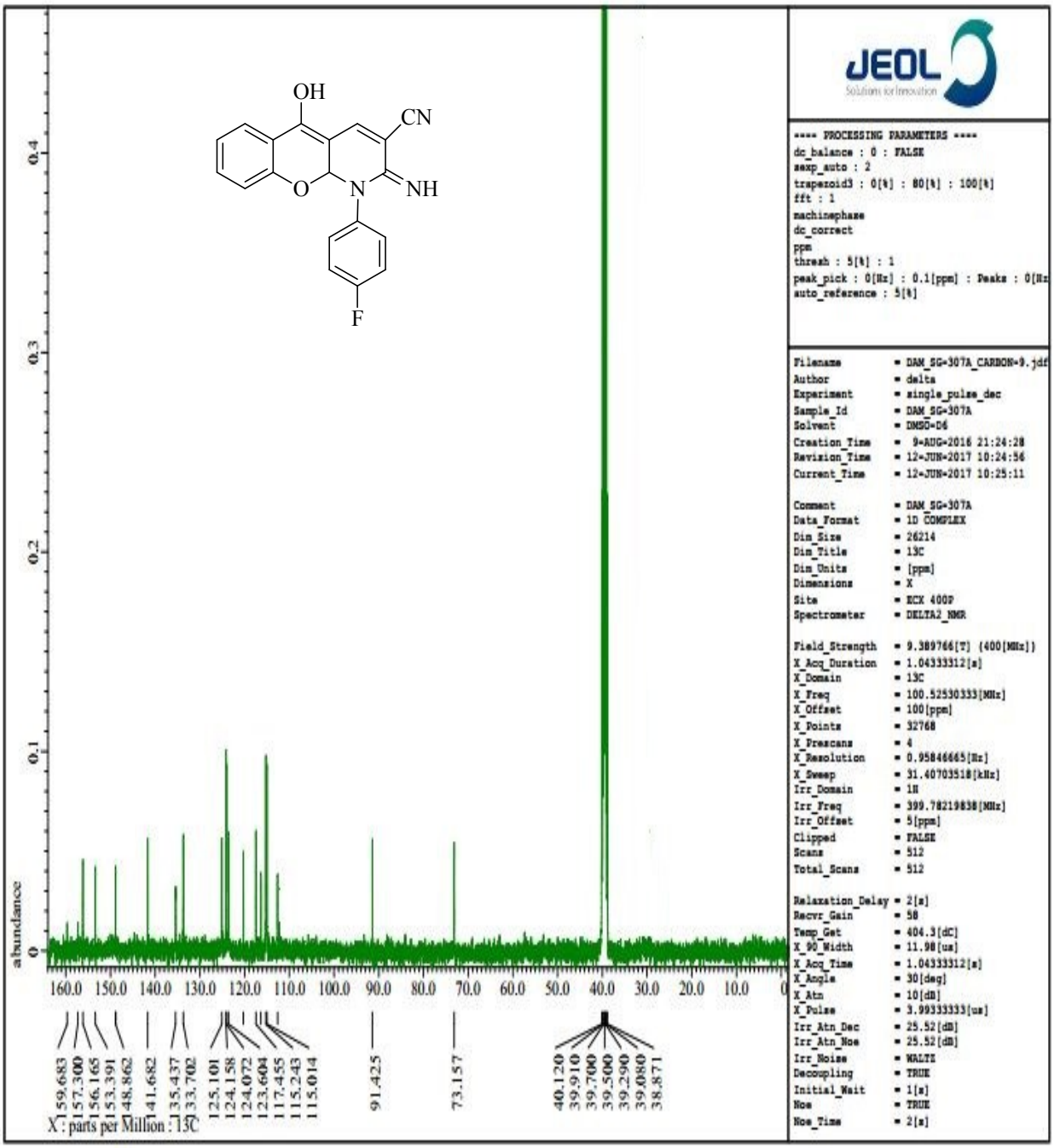
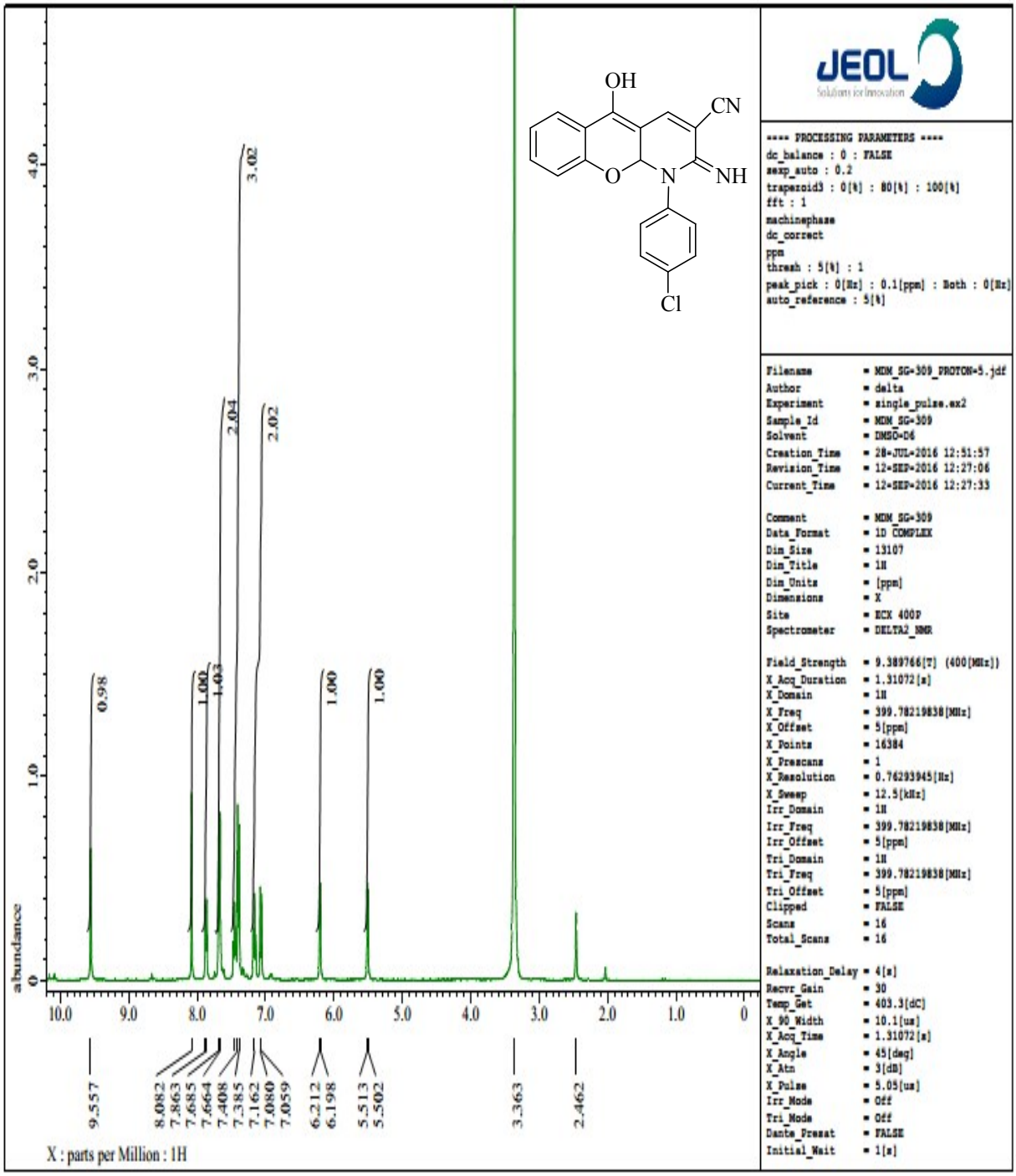
^{13}C NMR OF IVa



[1]H NMR OF IVb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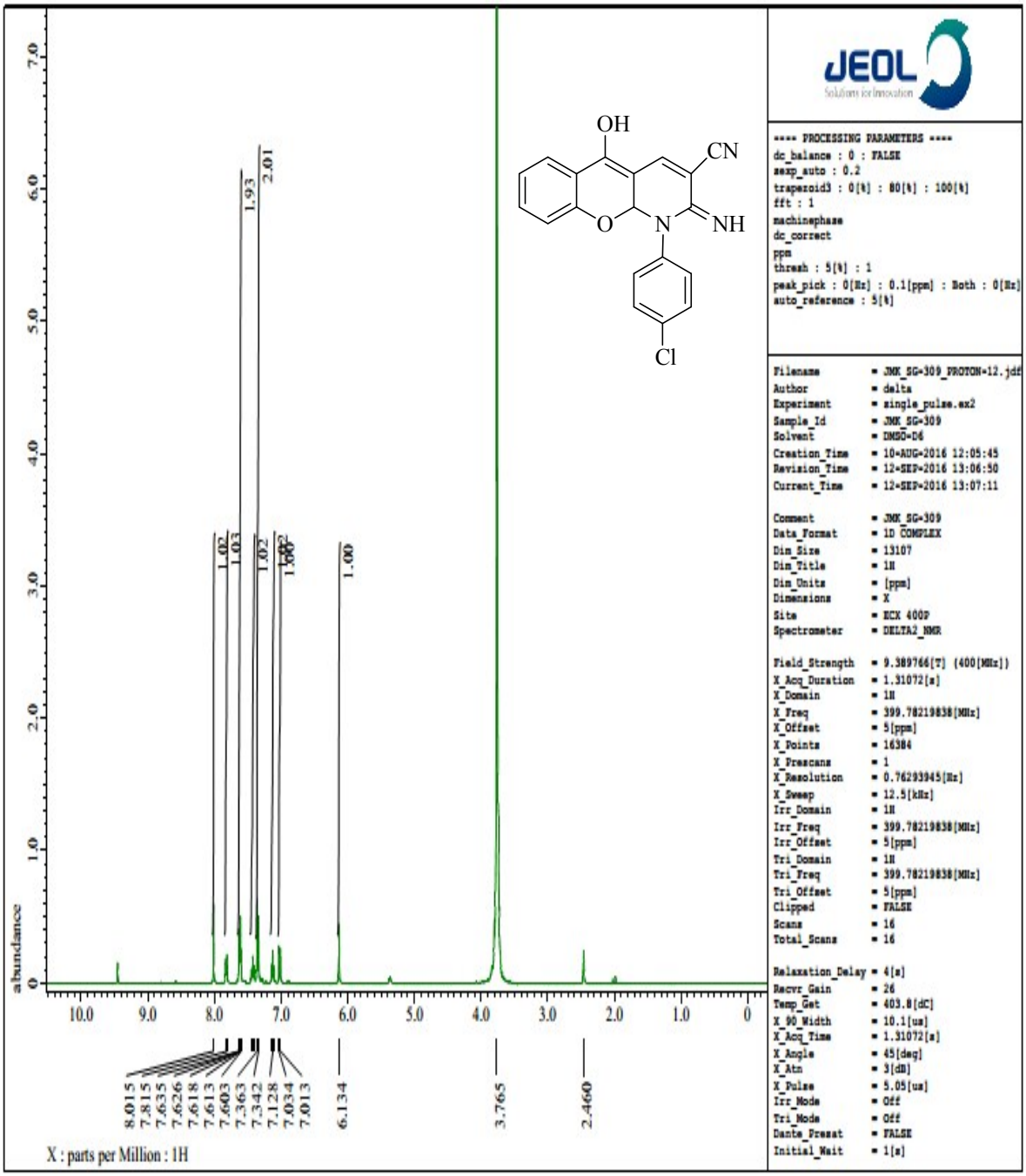
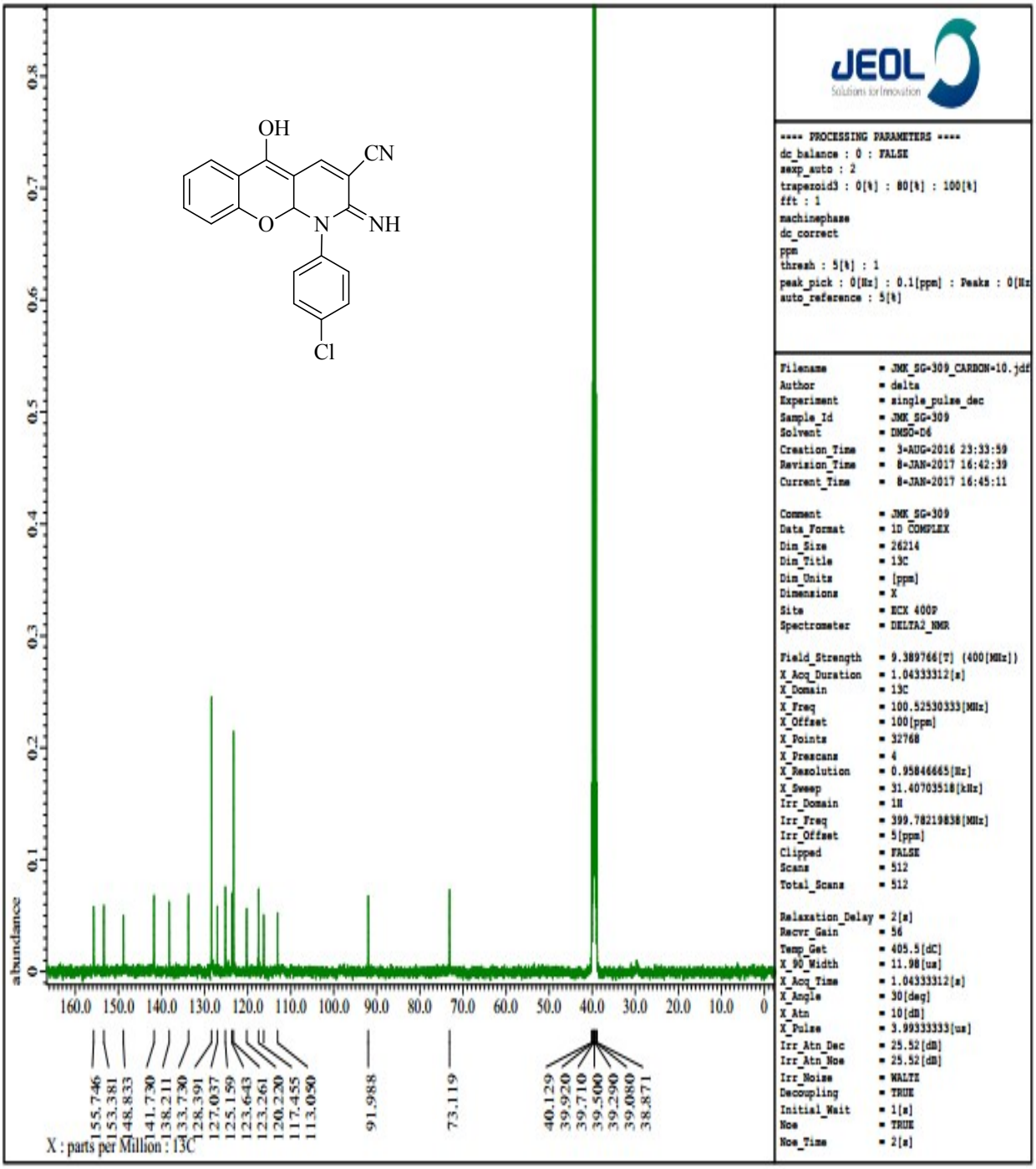
7.863
7.685
7.664
7.408
7.385
7.162
7.080
7.059
6.212
6.198
5.513
5.502
3.363
2.462
X : parts per Million : 1H
JEOL
Solutions for Innovation
**** PROCESSING PARAMETERS ****
dc_balance : 0 : FALSE
sexp_auto : 0.2
traperoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = MDM_SG-309_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = MDM_SG-309
Solvent = DMSO-D6
Creation_Time = 28-JUL-2016 12:51:57
Revision_Time = 12-SEP-2016 12:27:06
Current_Time = 12-SEP-2016 12:27:33
Comment = MDM_SG-309
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = NCX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.31072[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.76293945[Hz]
X_Sweep = 12.5[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 30
Temp_Get = 403.3[dC]
X_90_Width = 10.1[us]
X_Acq_Time = 1.31072[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 5.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

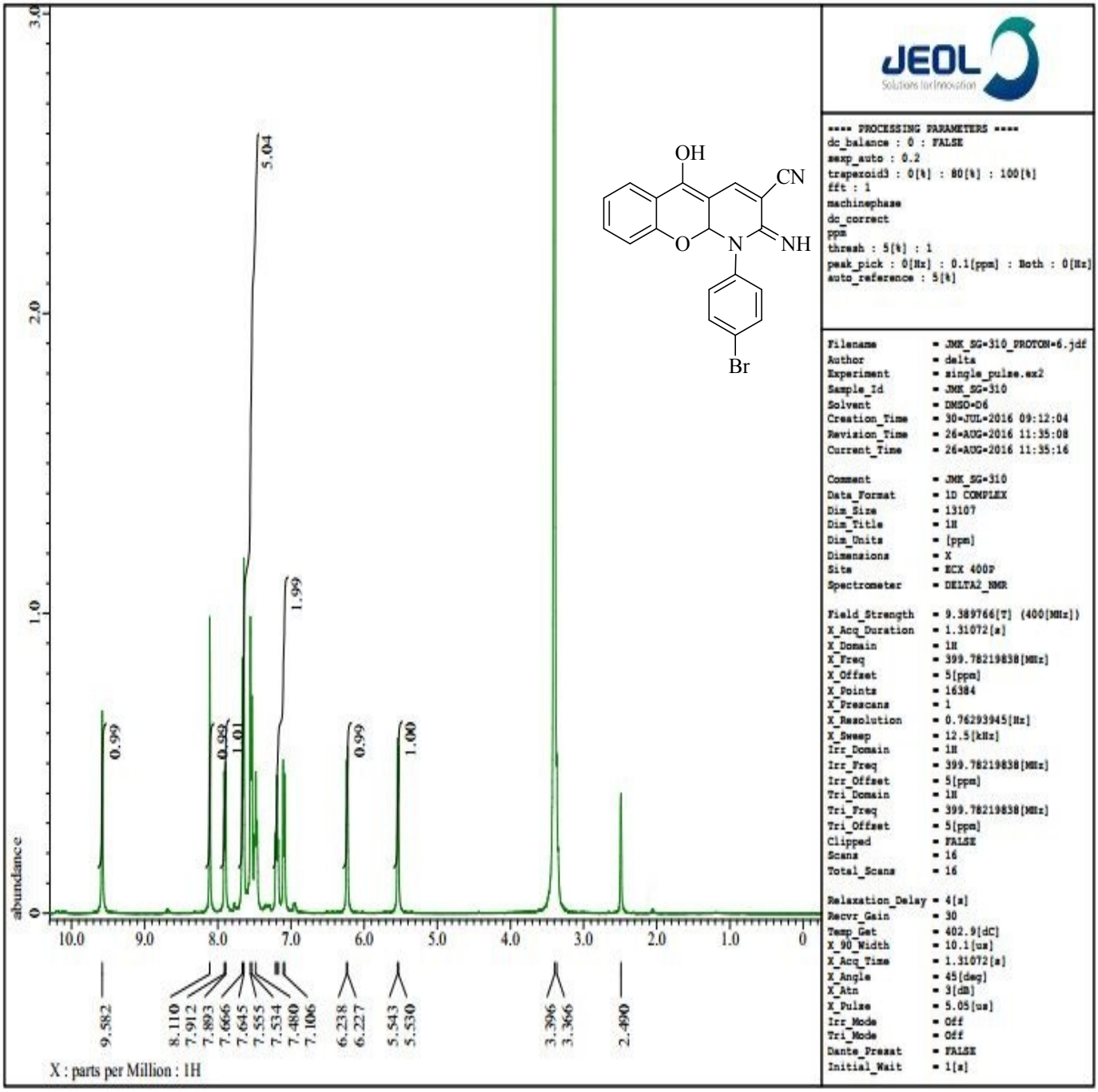
^{1}H NMR OF IVb (D_2O exchange)



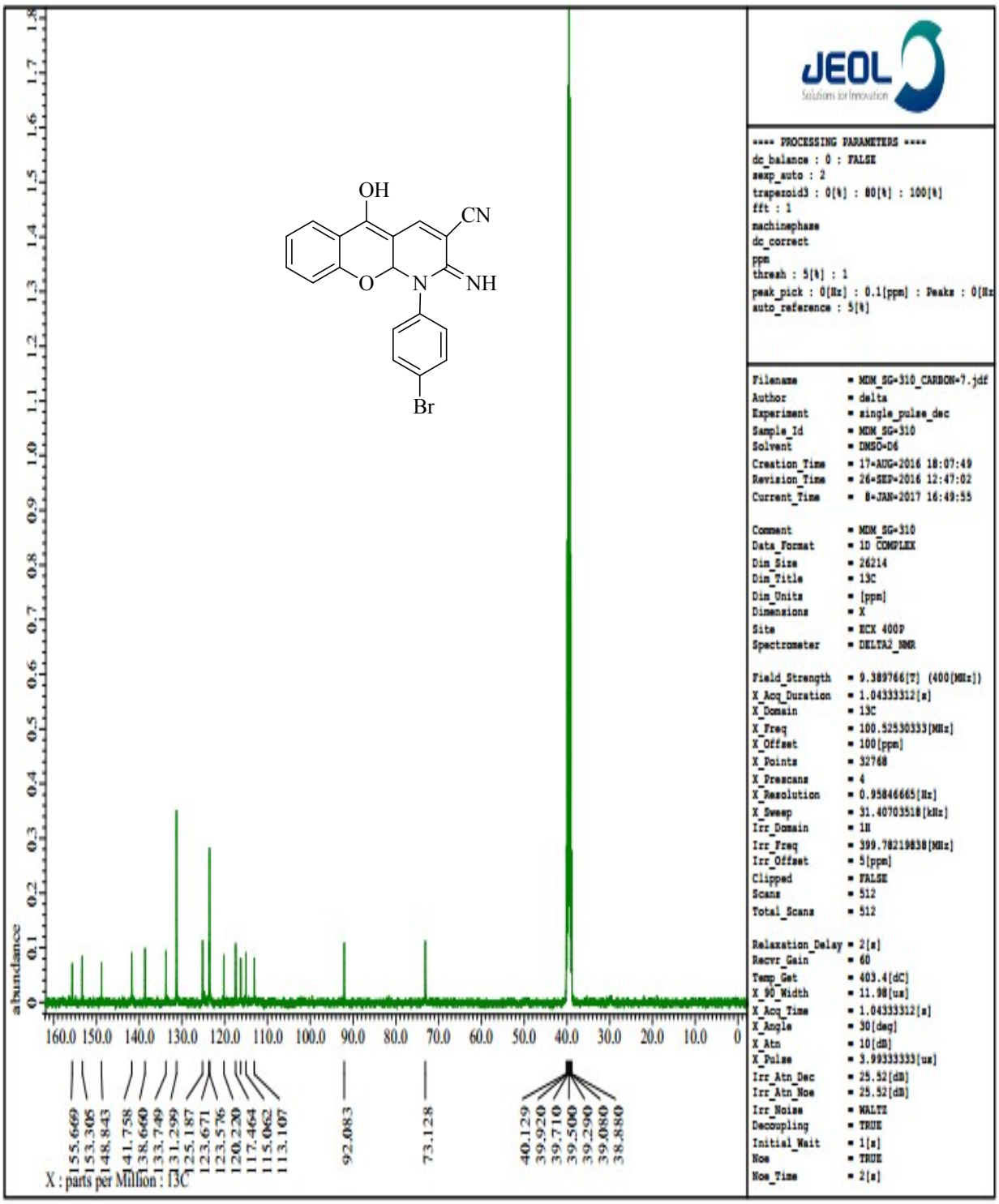
^{13}C NMR OF IVb



[1]H NMR OF IVc

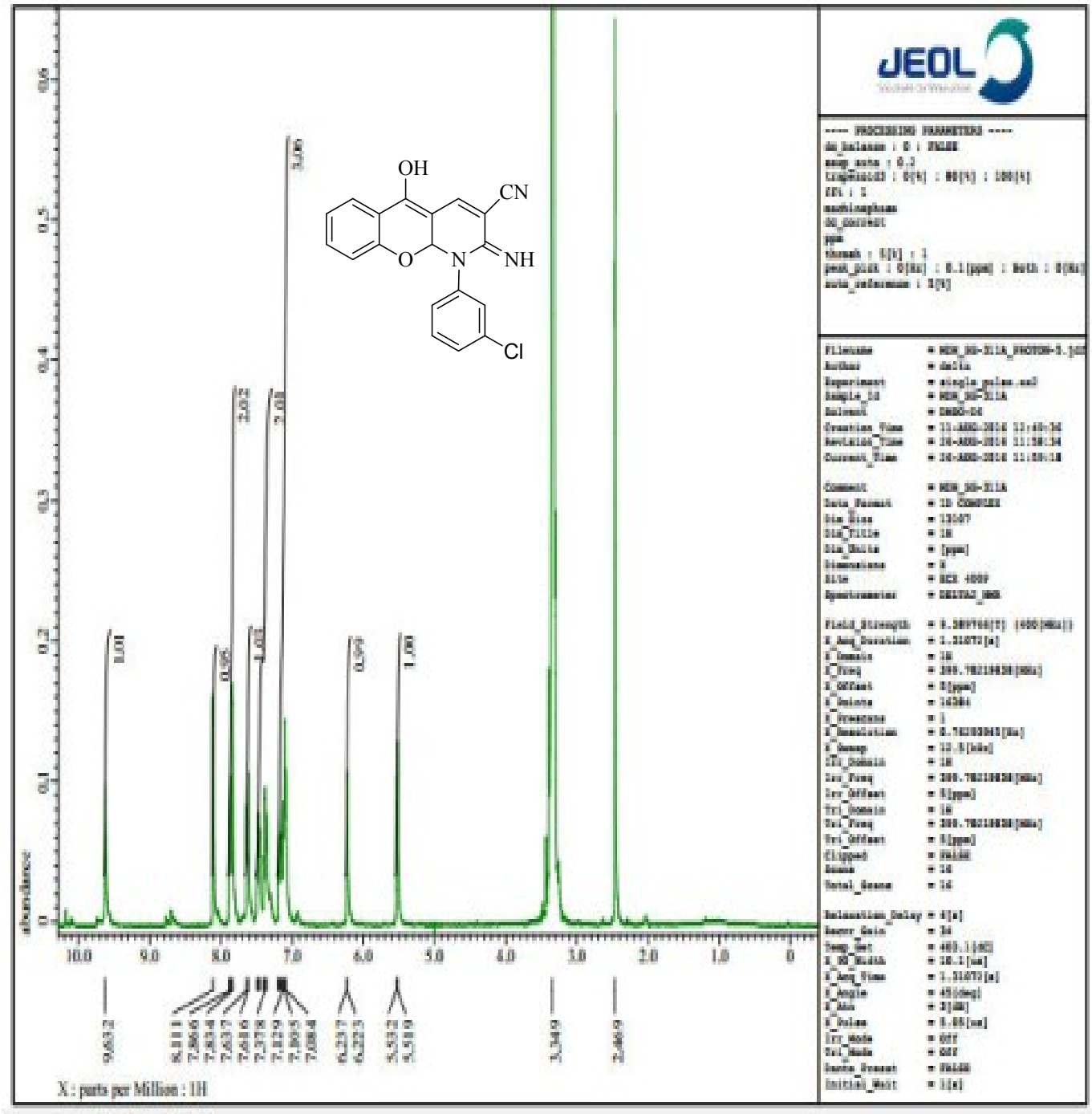

OH
CN
NH
O
N
Br
5.04
1.99
0.99
0.98
1.01
0.99
1.00
abundance
X : parts per Million : 1H
9.582
8.110
7.912
7.893
7.666
7.645
7.555
7.534
7.480
7.106
6.238
6.227
5.543
5.530
3.396
3.366
2.490
**** PROCESSING PARAMETERS ****
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-310_PROTON-6.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-310
Solvent = DMSO-D6
Creation_Time = 30-JUL-2016 09:12:04
Revision_Time = 26-AUG-2016 11:35:08
Current_Time = 26-AUG-2016 11:35:16
Comment = JMK_SG-310
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.31072[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.76293945[Hz]
X_Sweep = 12.5[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 30
Temp_Get = 402.9[dC]
X_90_Width = 10.1[us]
X_Acq_Time = 1.31072[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 5.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

^{13}C NMR OF IVc

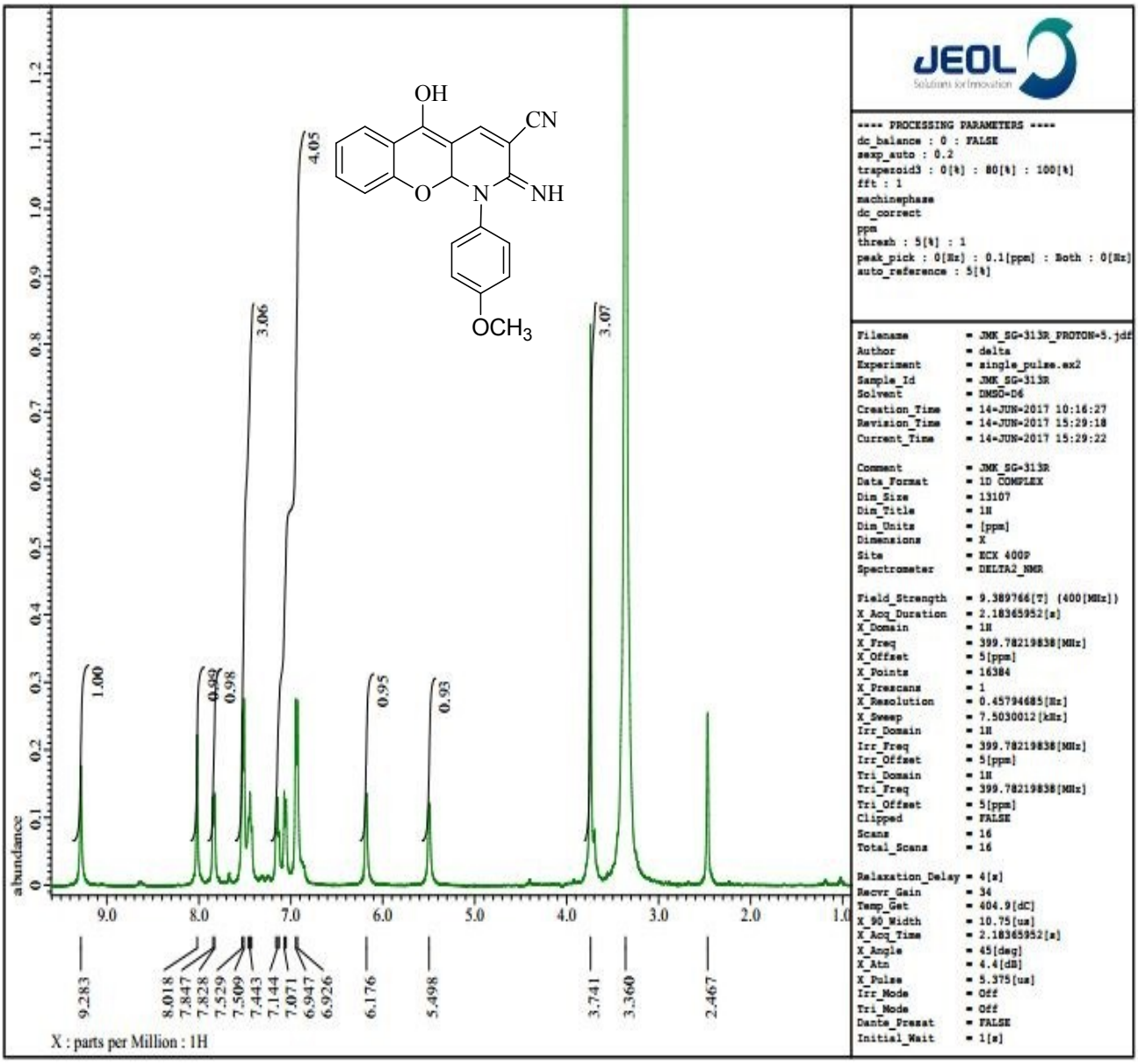


--

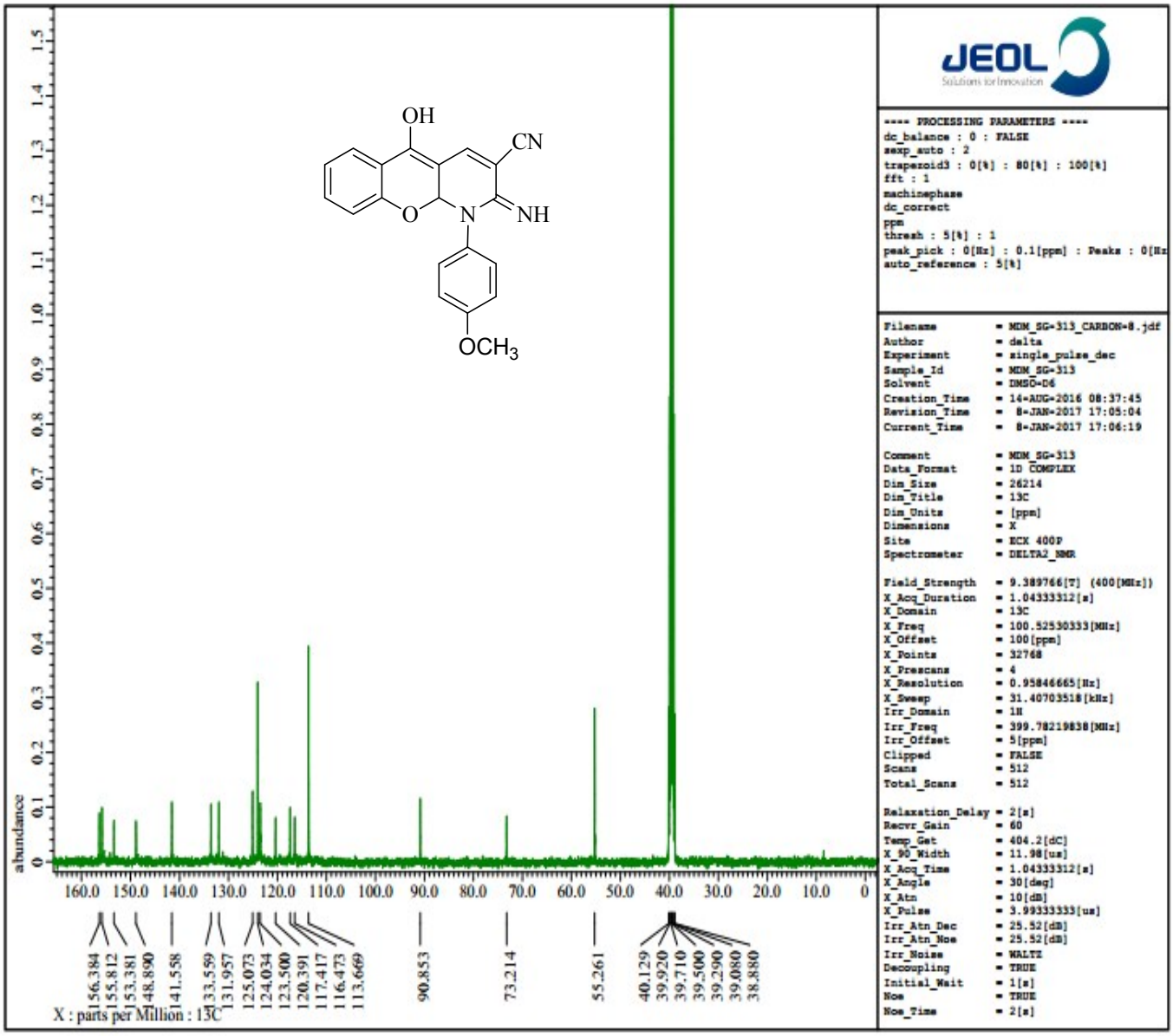
[1]H NMR OF IVd



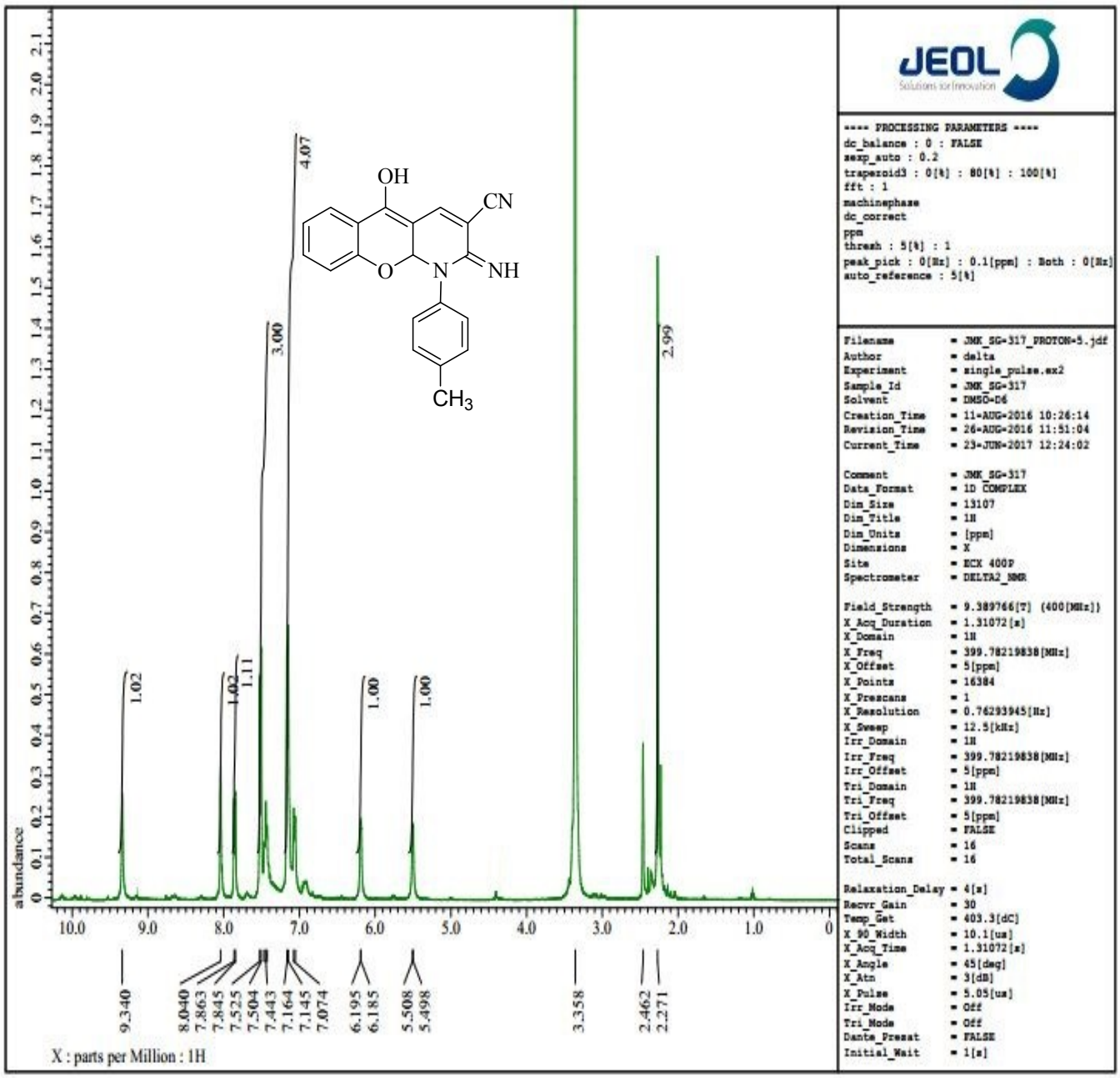
^{1}H NMR OF IVe


OH
CN
NH
O
N
OCH3
4.05
3.06
3.07
1.00
0.98
0.95
0.93
abundance
X : parts per Million : 1H
9.283
8.018
7.847
7.828
7.529
7.509
7.443
7.144
7.071
6.947
6.926
6.176
5.498
3.741
3.360
2.467
**** PROCESSING PARAMETERS ****
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-313R_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-313R
Solvent = DMSO-D6
Creation_Time = 14-JUN-2017 10:16:27
Revision_Time = 14-JUN-2017 15:29:18
Current_Time = 14-JUN-2017 15:29:22
Comment = JMK_SG-313R
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 34
Temp_Get = 404.9[dC]
X_90_Width = 10.75[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 5.375[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

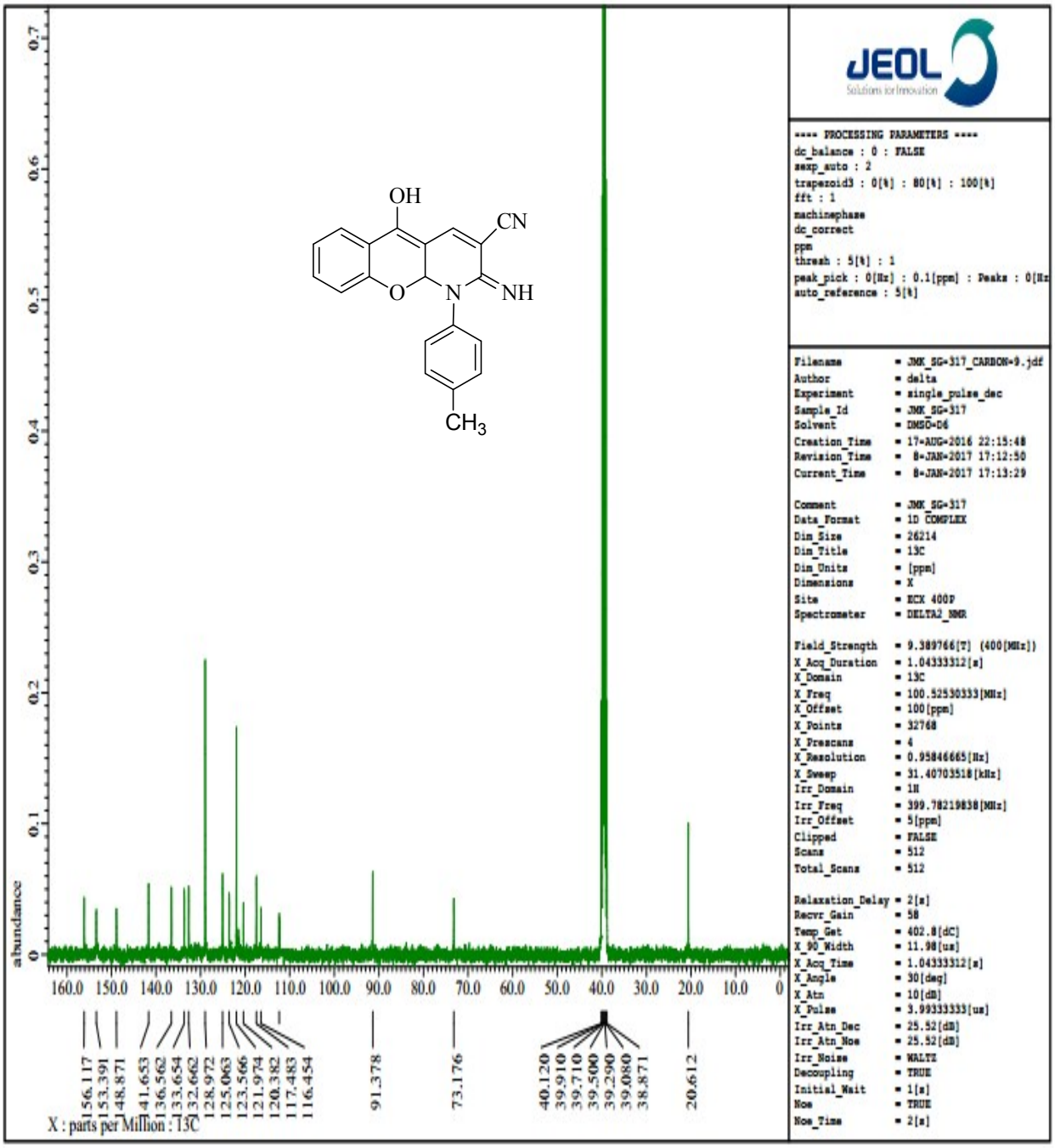
^{13}C NMR OF IVe



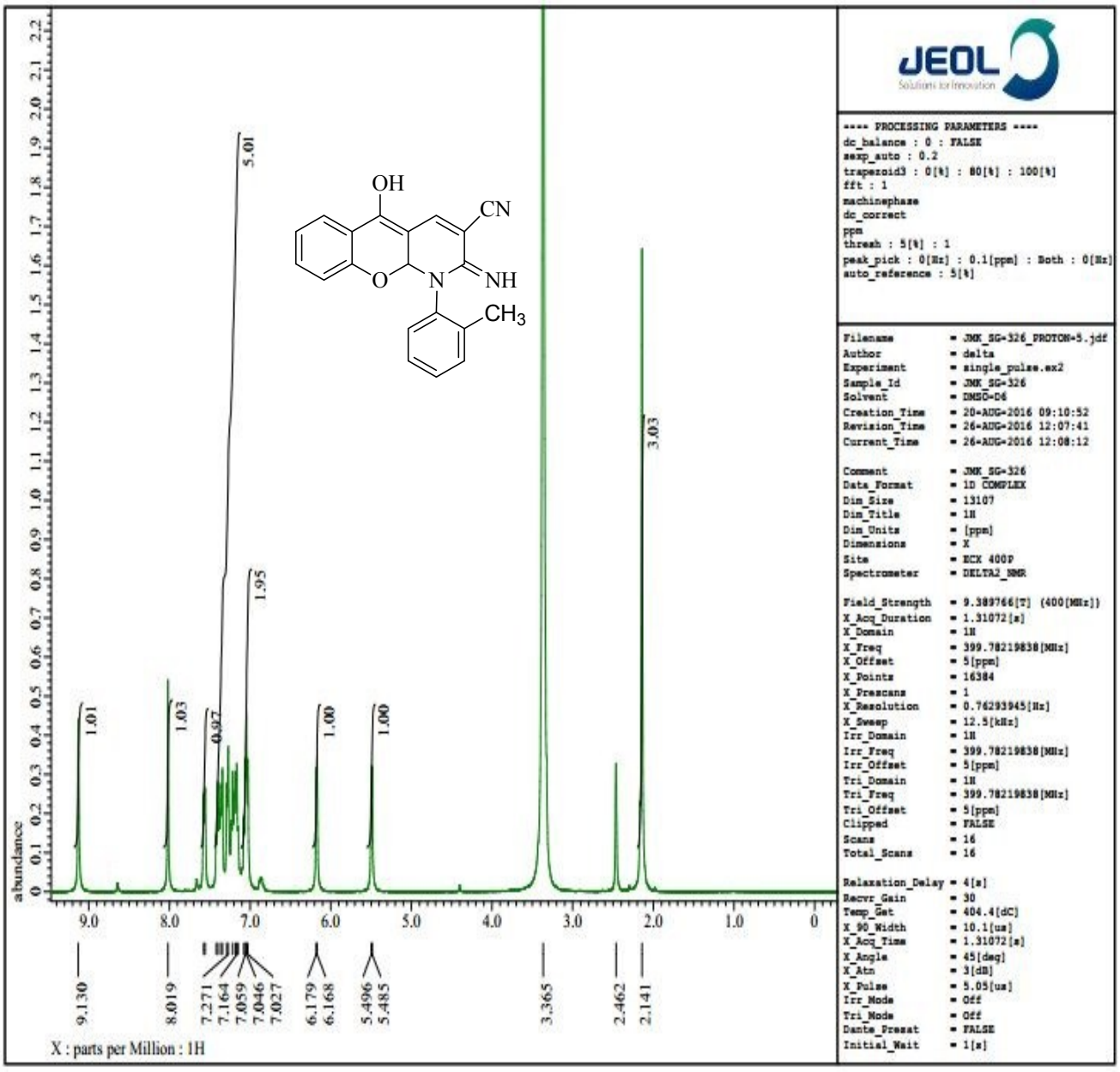
^{1}H NMR OF IVf


OH
CN
O
N
NH
CH3
4.07
3.00
2.99
1.02
1.02
1.11
1.00
1.00
abundance
9.340
8.040
7.863
7.845
7.525
7.504
7.443
7.164
7.145
7.074
6.195
6.185
5.508
5.498
3.358
2.462
2.271
X : parts per Million : 1H
JEOL
Solutions for Innovation
==== PROCESSING PARAMETERS ====
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-317_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-317
Solvent = DMSO-D6
Creation_Time = 11-AUG-2016 10:26:14
Revision_Time = 26-AUG-2016 11:51:04
Current_Time = 23-JUN-2017 12:24:02
Comment = JMK_SG-317
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.31072[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.76293945[Hz]
X_Sweep = 12.5[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 30
Temp_Get = 403.3[dC]
X_90_Width = 10.1[us]
X_Acq_Time = 1.31072[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 5.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

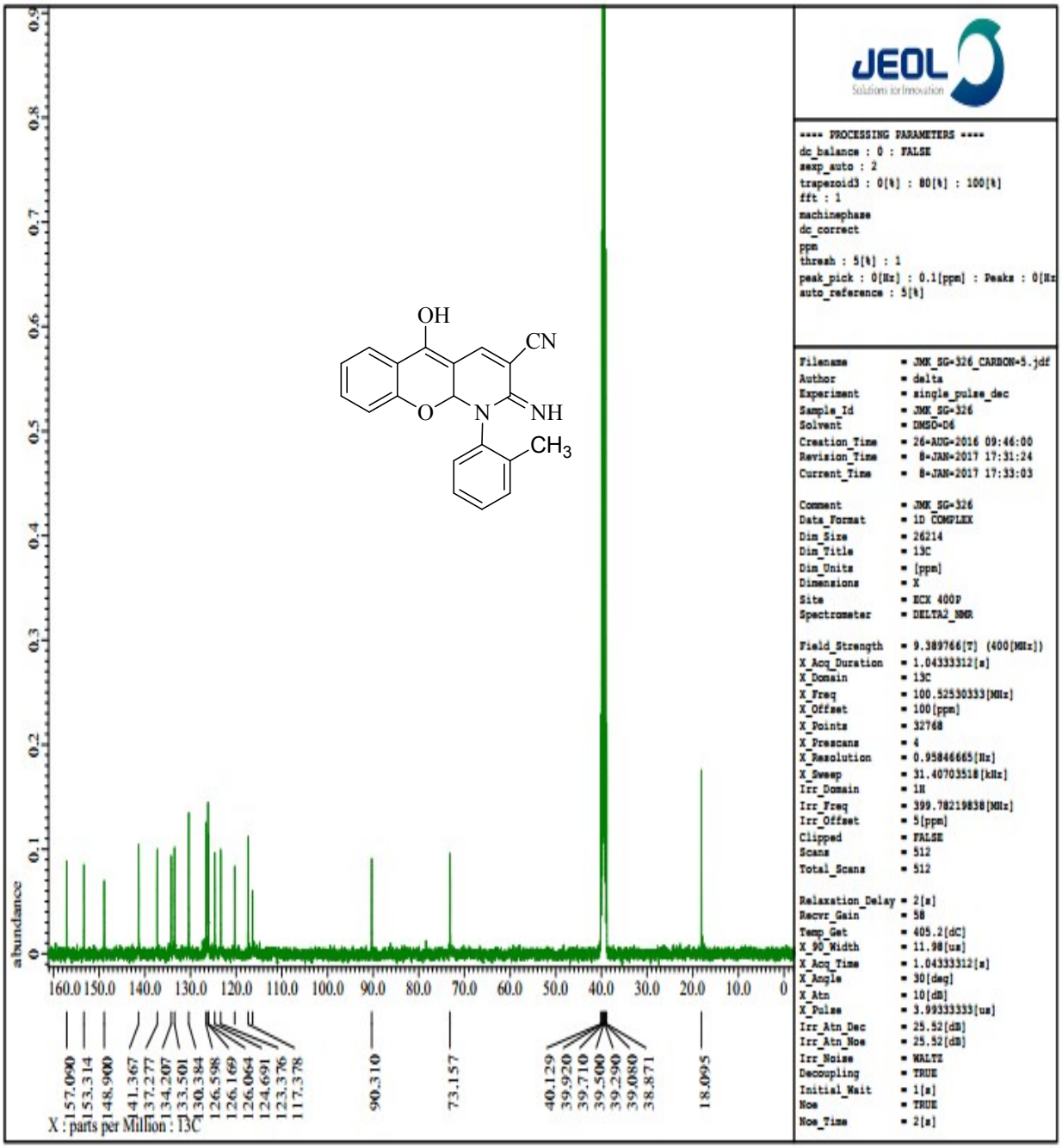
^{13}C NMR OF IVf



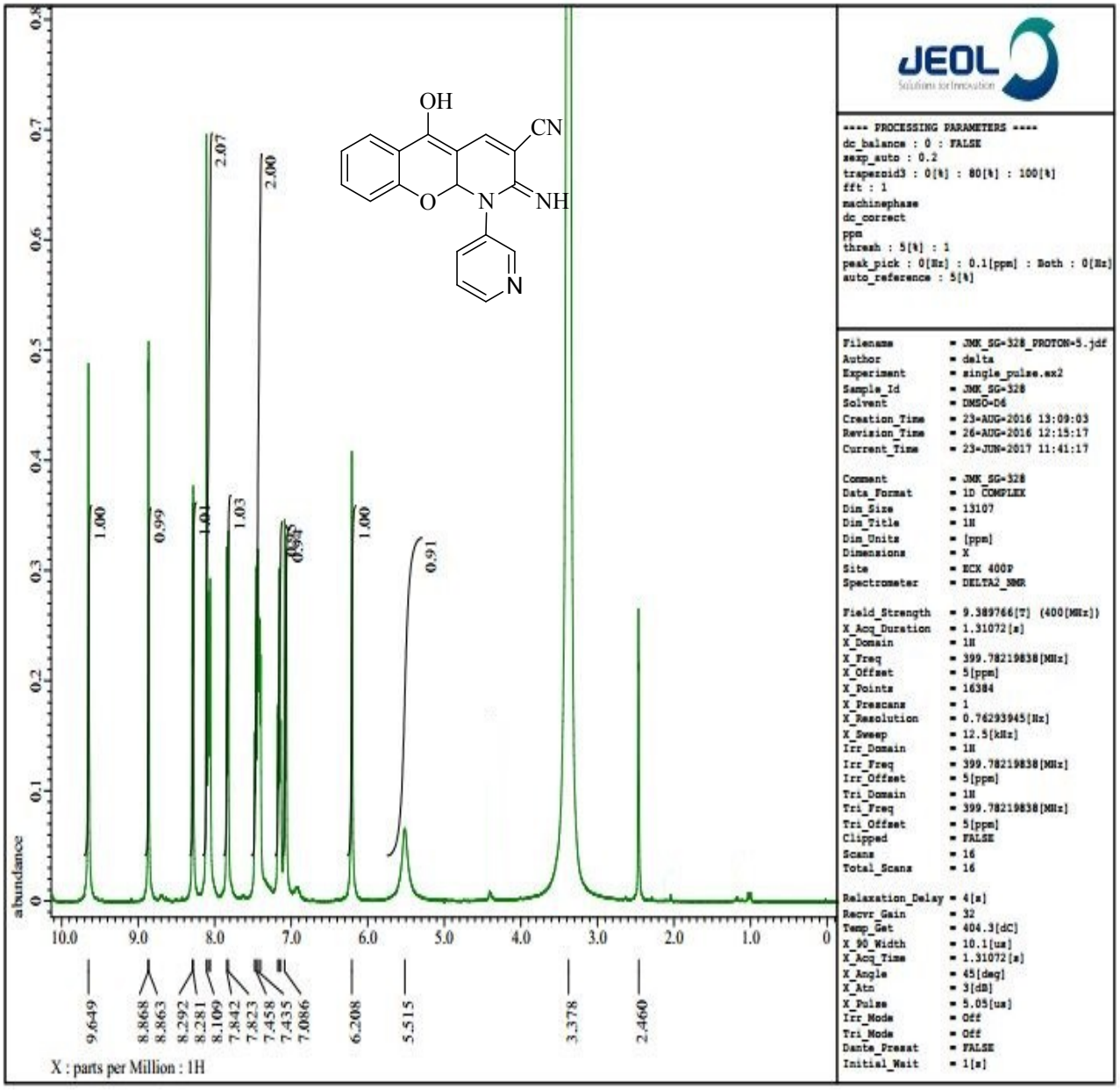
^{1}H NMR OF IVg


OH
CN
NH
O
N
CH3
5.01
1.95
3.03
1.01
1.03
0.97
1.00
1.00
abundance
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.130
8.019
7.271
7.164
7.059
7.046
7.027
6.179
6.168
5.496
5.485
3.365
2.462
2.141
X : parts per Million : 1H
JEOL
Solutions for Innovation
**** PROCESSING PARAMETERS ****
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-326_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-326
Solvent = DMSO-D6
Creation_Time = 20-AUG-2016 09:10:52
Revision_Time = 26-AUG-2016 12:07:41
Current_Time = 26-AUG-2016 12:08:12
Comment = JMK_SG-326
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.31072[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.76293945[Hz]
X_Sweep = 12.5[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 30
Temp_Get = 404.4[dC]
X_90_Width = 10.1[us]
X_Acq_Time = 1.31072[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 5.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

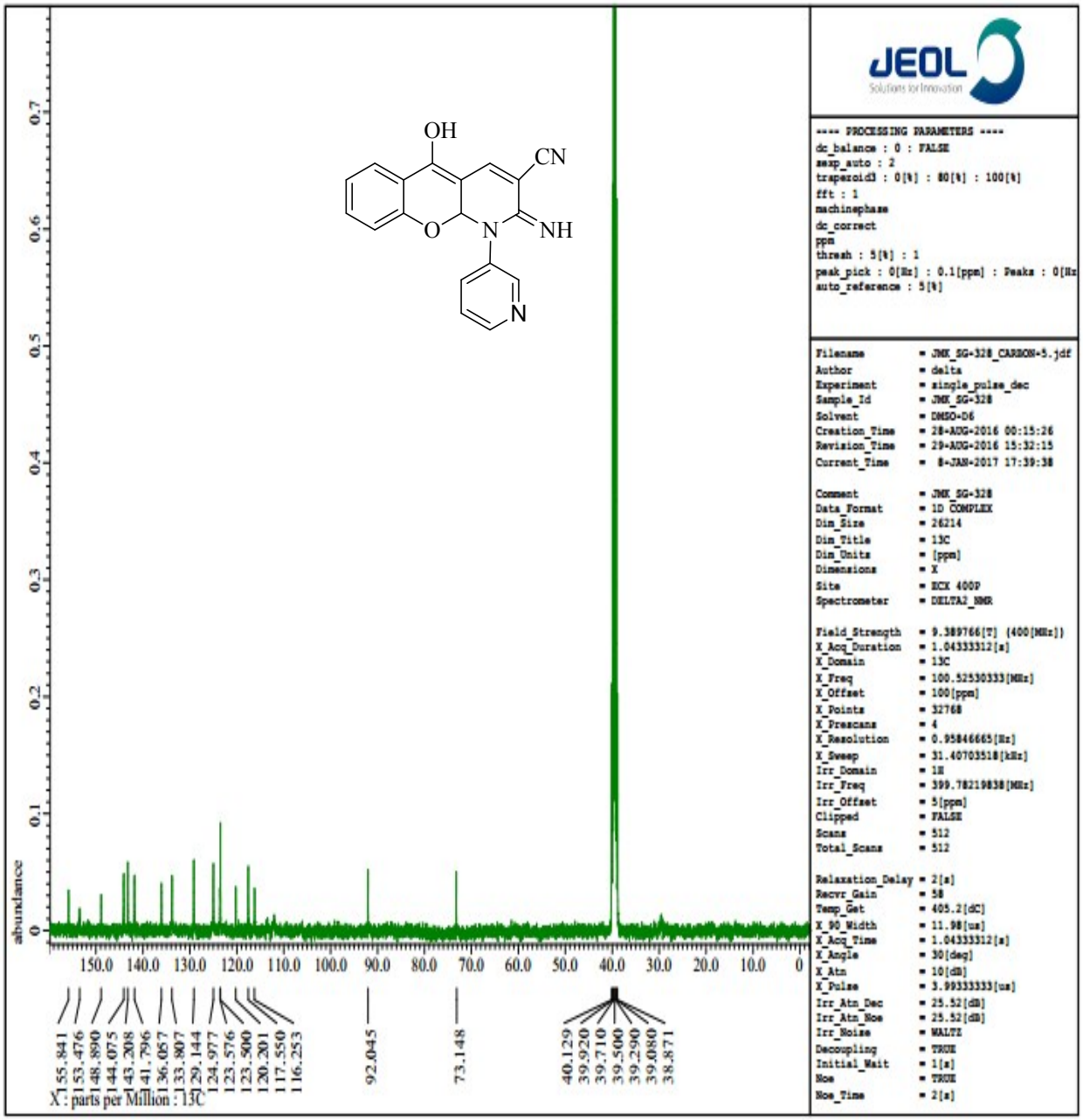
^{13}C NMR OF IVg



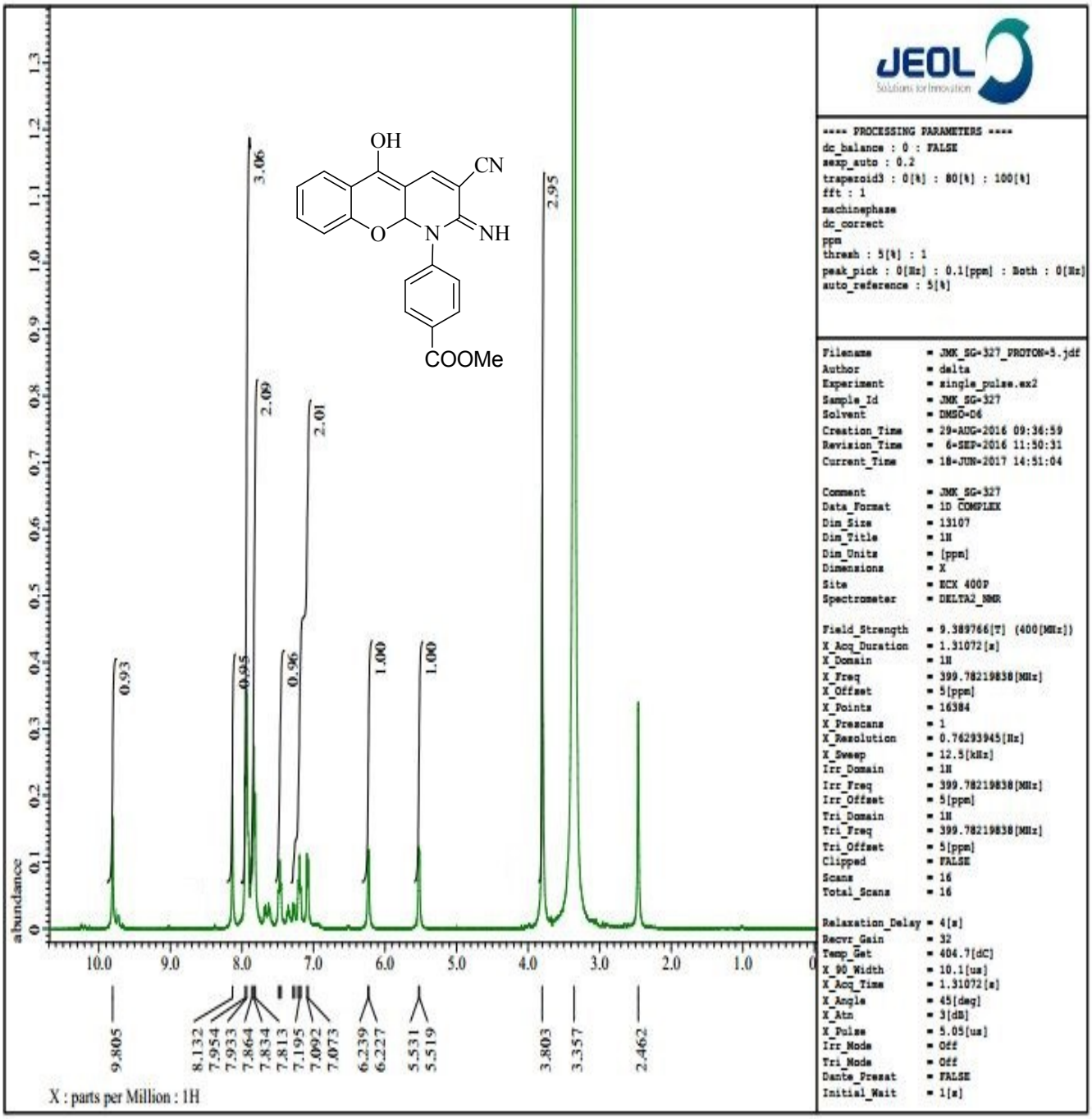
^{1}H NMR OF IVh


OH
CN
NH
O
N
N
2.07
2.00
1.03
1.01
1.00
0.99
1.00
0.91
abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.649
8.868
8.863
8.292
8.281
8.109
7.842
7.823
7.458
7.435
7.086
6.208
5.515
3.378
2.460
X : parts per Million : 1H
JEOL
Solutions for Innovation
**** PROCESSING PARAMETERS ****
dc_balance : 0 : FALSE
zexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-328_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-328
Solvent = DMSO-D6
Creation_Time = 23-AUG-2016 13:09:03
Revision_Time = 26-AUG-2016 12:15:17
Current_Time = 23-JUN-2017 11:41:17
Comment = JMK_SG-328
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.31072[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.76293945[Hz]
X_Sweep = 12.5[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 32
Temp_Get = 404.3[dC]
X_90_Width = 10.1[us]
X_Acq_Time = 1.31072[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 5.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

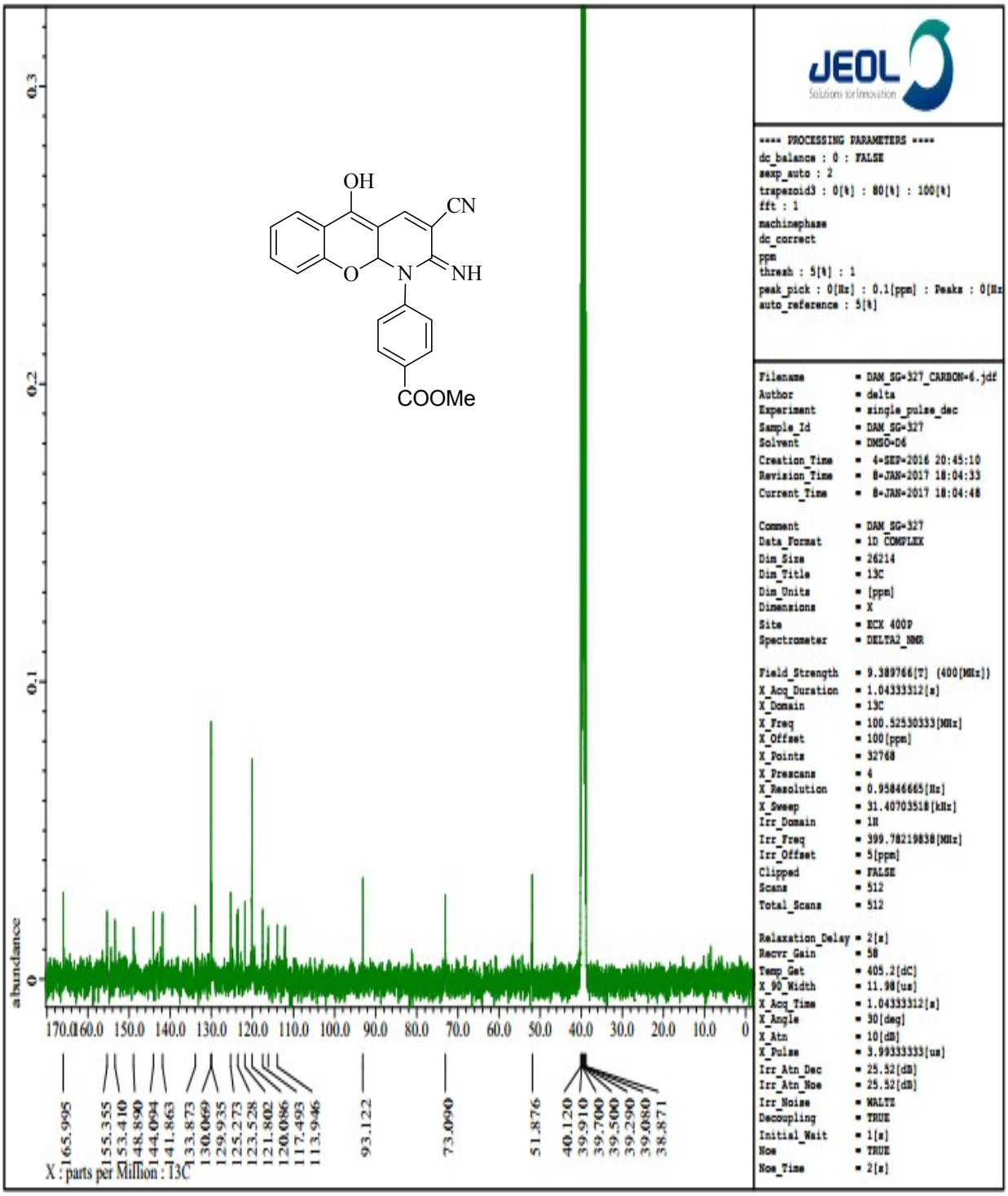
^{13}C NMR OF IVh



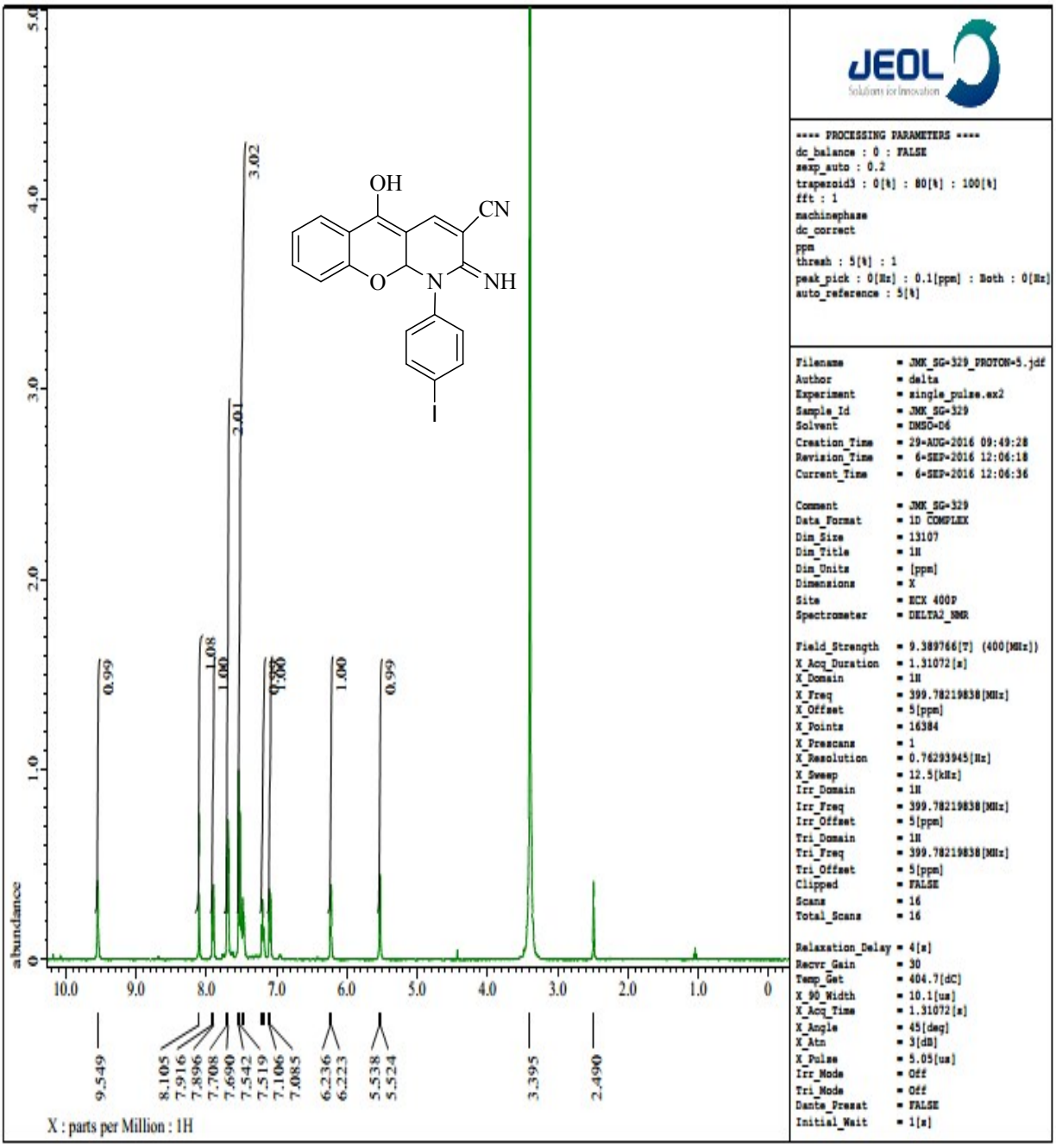
^{1}H NMR OF IVi


OH
CN
NH
O
N
COOMe
3.06
2.09
2.01
2.95
0.93
0.95
0.96
1.00
1.00
abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
9.805
8.132
7.954
7.933
7.864
7.834
7.813
7.195
7.092
7.073
6.239
6.227
5.531
5.519
3.803
3.357
2.462
X : parts per Million : 1H
JEOL
Solutions for Innovation
**** PROCESSING PARAMETERS ****
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
zachinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-327_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-327
Solvent = DMSO-D6
Creation_Time = 29-AUG-2016 09:36:59
Revision_Time = 6-SEP-2016 11:50:31
Current_Time = 18-JUN-2017 14:51:04
Comment = JMK_SG-327
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.31072[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.76293945[Hz]
X_Sweep = 12.5[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 32
Temp_Get = 404.7[dC]
X_90_Width = 10.1[us]
X_Acq_Time = 1.31072[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 5.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

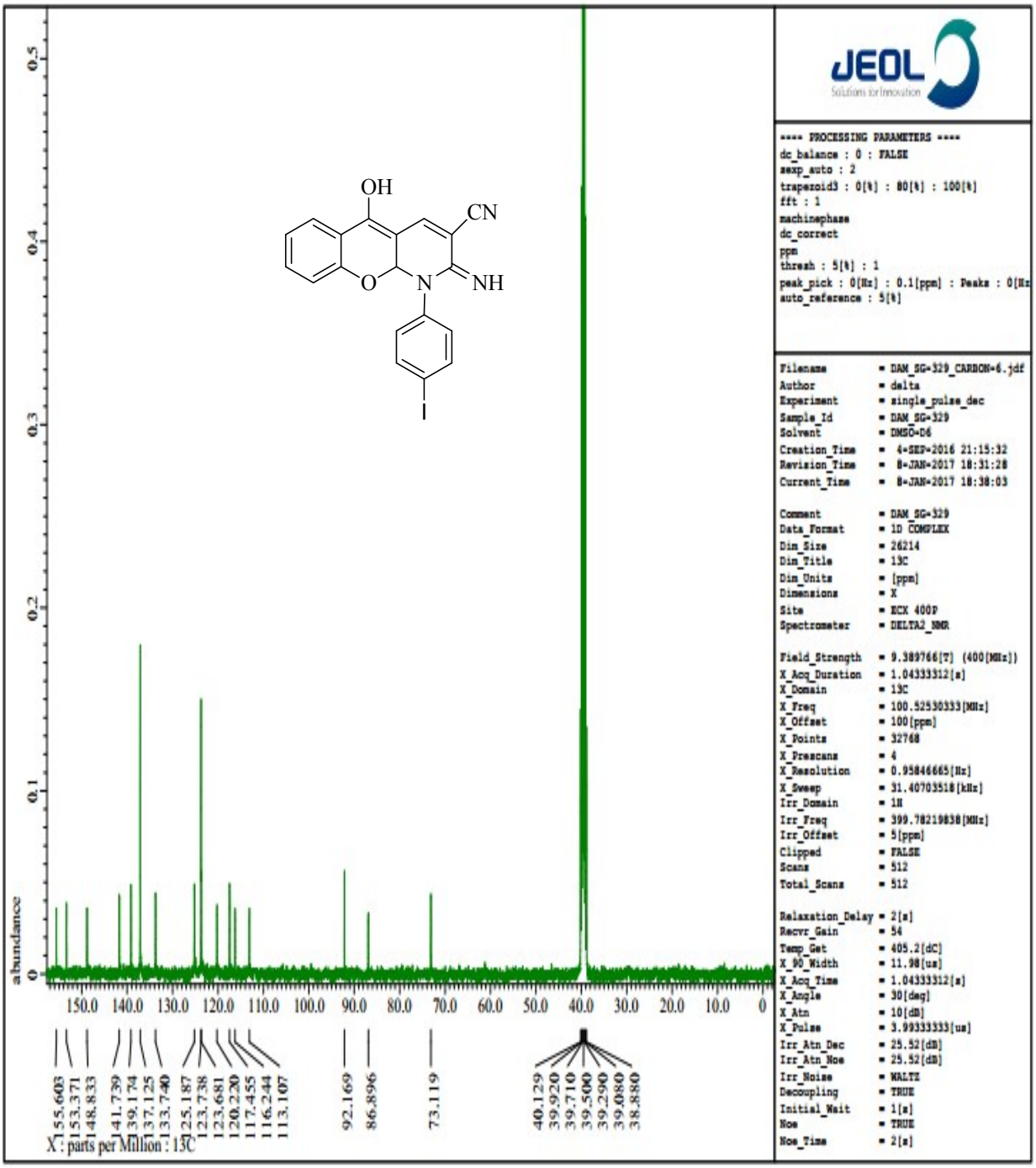
^{13}C NMR OF IVi



^{1}H NMR OF IVj


OH
CN
NH
O
N
I
3.02
2.01
1.08
1.00
1.00
0.99
0.99
abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.549
8.105
7.916
7.896
7.708
7.690
7.542
7.519
7.106
7.085
6.236
6.223
5.538
5.524
3.395
2.490
X : parts per Million : 1H
JEOL
Solutions for Innovation
**** PROCESSING PARAMETERS ****
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-329_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-329
Solvent = DMSO-D6
Creation_Time = 29-AUG-2016 09:49:28
Revision_Time = 6-SEP-2016 12:06:18
Current_Time = 6-SEP-2016 12:06:36
Comment = JMK_SG-329
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.31072[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.76293945[Hz]
X_Sweep = 12.5[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 30
Temp_Get = 404.7[dC]
X_90_Width = 10.1[us]
X_Acq_Time = 1.31072[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 5.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

^{13}C NMR OF IVj



^{1}H NMR OF IVk

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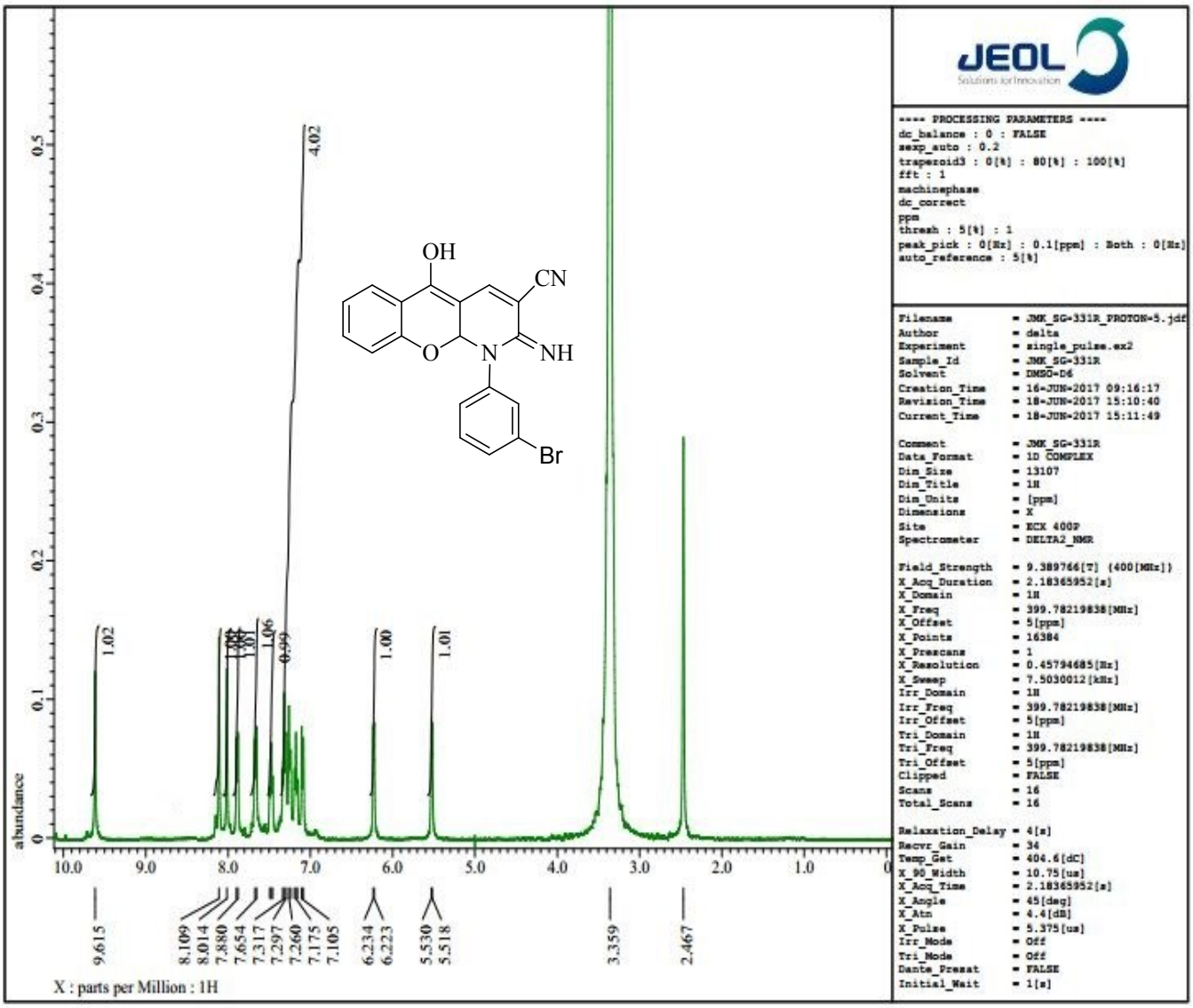
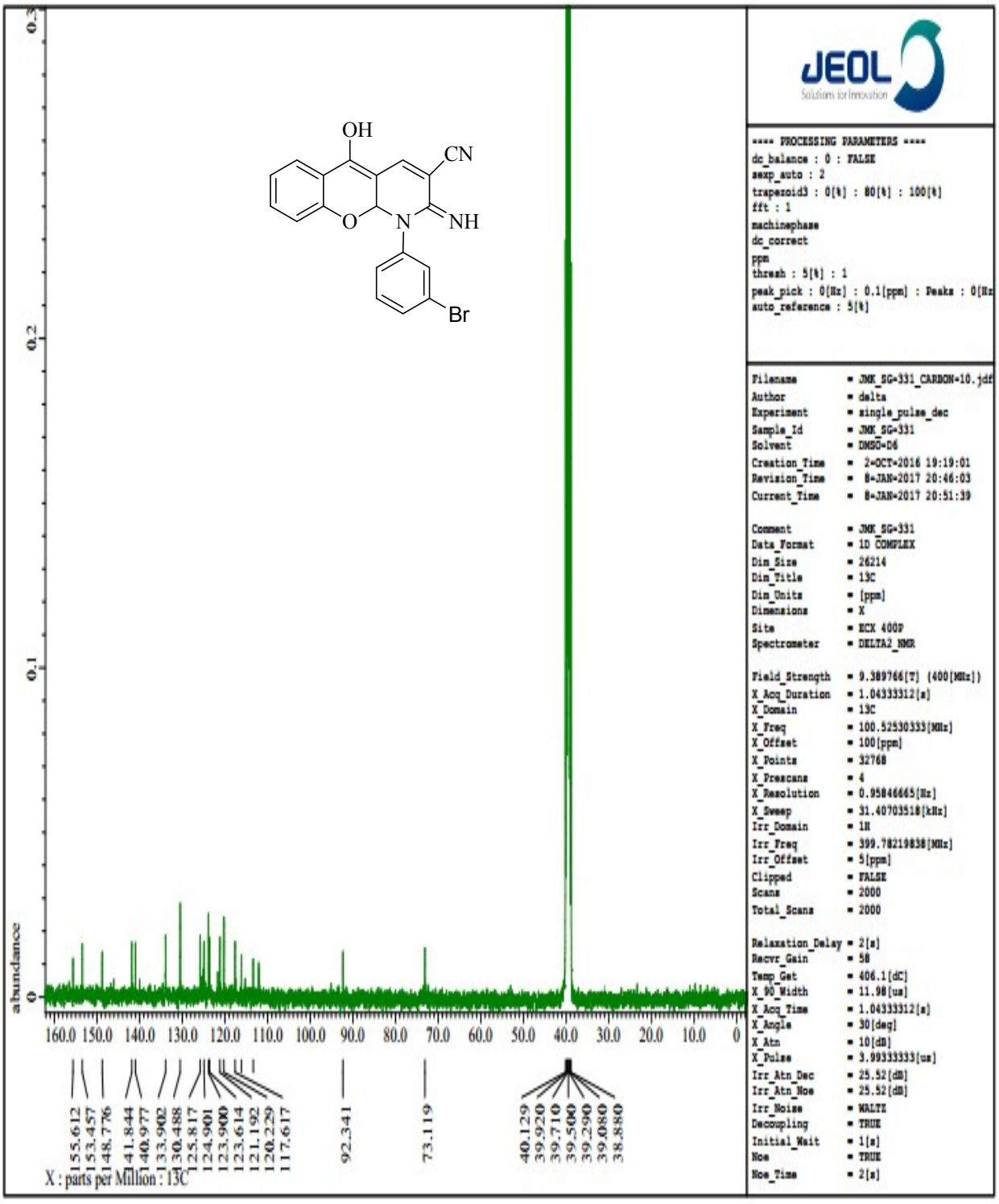
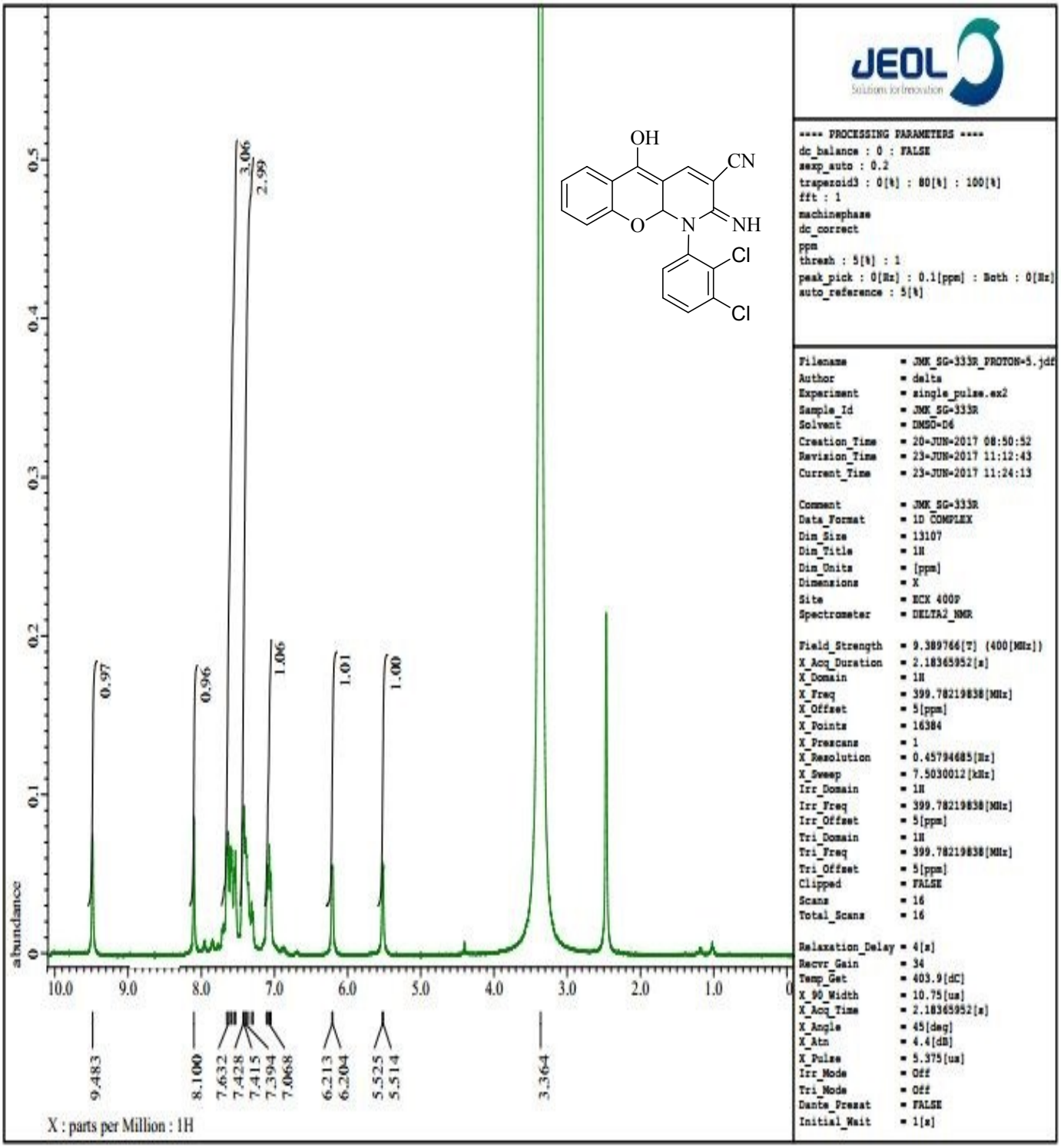

OH
CN
NH
O
N
Br
4.02
1.02
1.06
0.99
1.00
1.01
abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
9.615
8.109
8.014
7.880
7.654
7.317
7.297
7.260
7.175
7.105
6.234
6.223
5.530
5.518
3.359
2.467
X : parts per Million : 1H
JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-331R_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-331R
Solvent = DMSO-D6
Creation_Time = 16-JUN-2017 09:16:17
Revision_Time = 18-JUN-2017 15:10:40
Current_Time = 18-JUN-2017 15:11:49
Comment = JMK_SG-331R
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 34
Temp_Get = 404.6[dC]
X_90_Width = 10.75[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 5.375[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

-

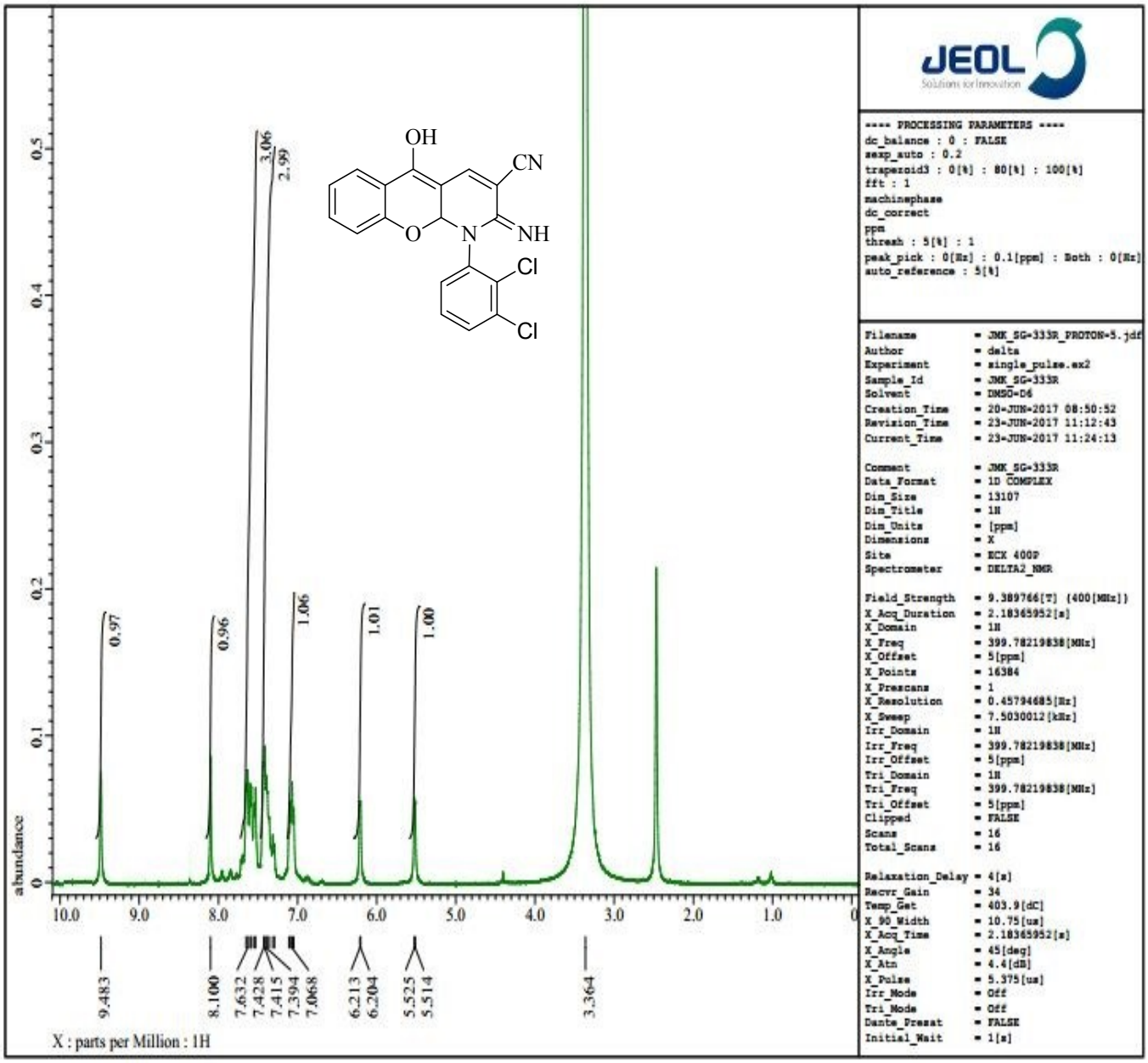
^{13}C NMR OF IVk



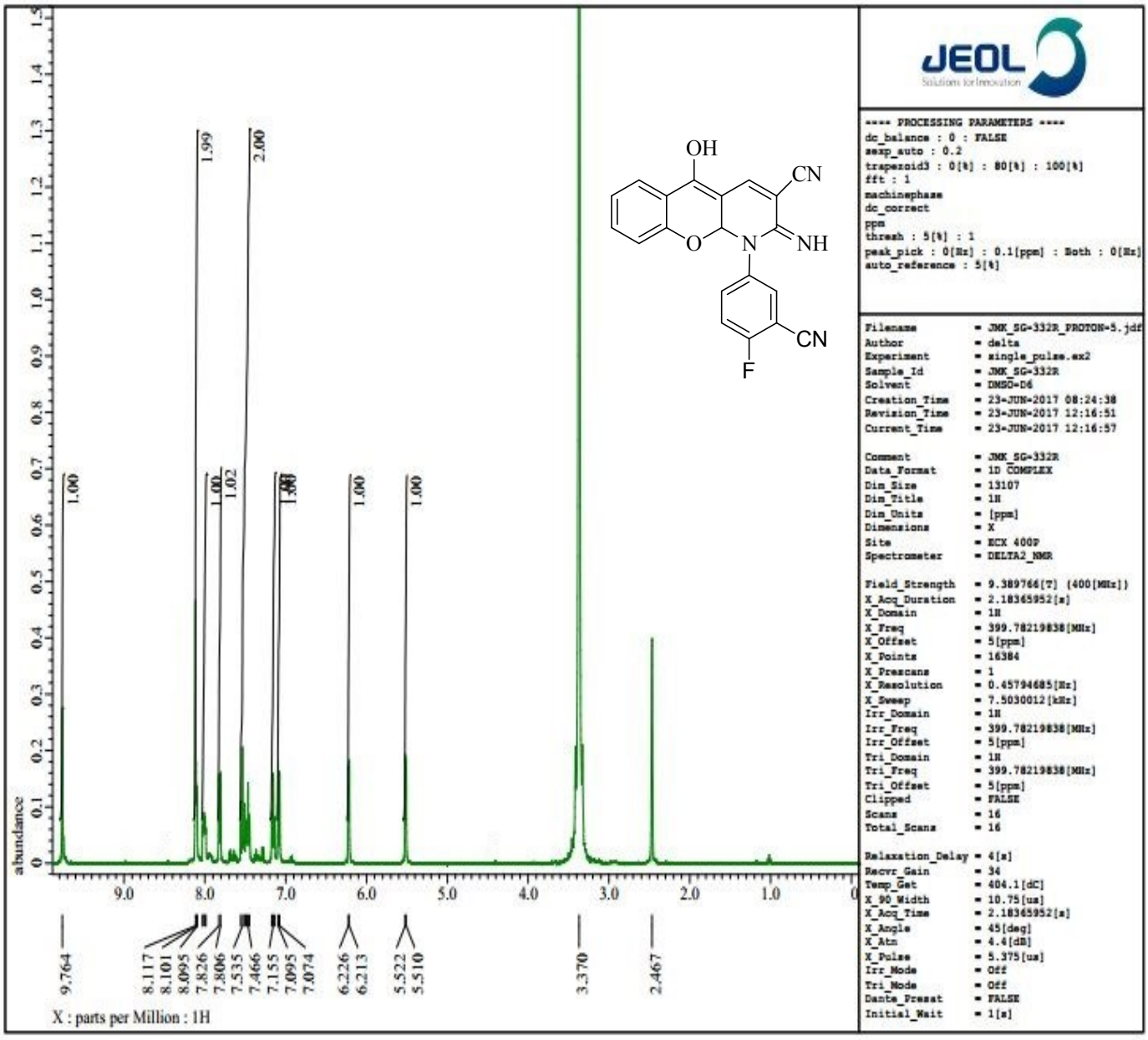
[1]H NMR OF IVl


OH
CN
NH
O
N
Cl
Cl
3.06
2.99
0.97
0.96
1.06
1.01
1.00
abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
9.483
8.100
7.632
7.428
7.415
7.394
7.068
6.213
6.204
5.525
5.514
3.364
X : parts per Million : 1H
JEOL
Solutions for Innovation
==== PROCESSING PARAMETERS ====
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-333R_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-333R
Solvent = DMSO-D6
Creation_Time = 20-JUN-2017 08:50:52
Revision_Time = 23-JUN-2017 11:12:43
Current_Time = 23-JUN-2017 11:24:13
Comment = JMK_SG-333R
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 34
Temp_Get = 403.9[dC]
X_90_Width = 10.75[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 5.375[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

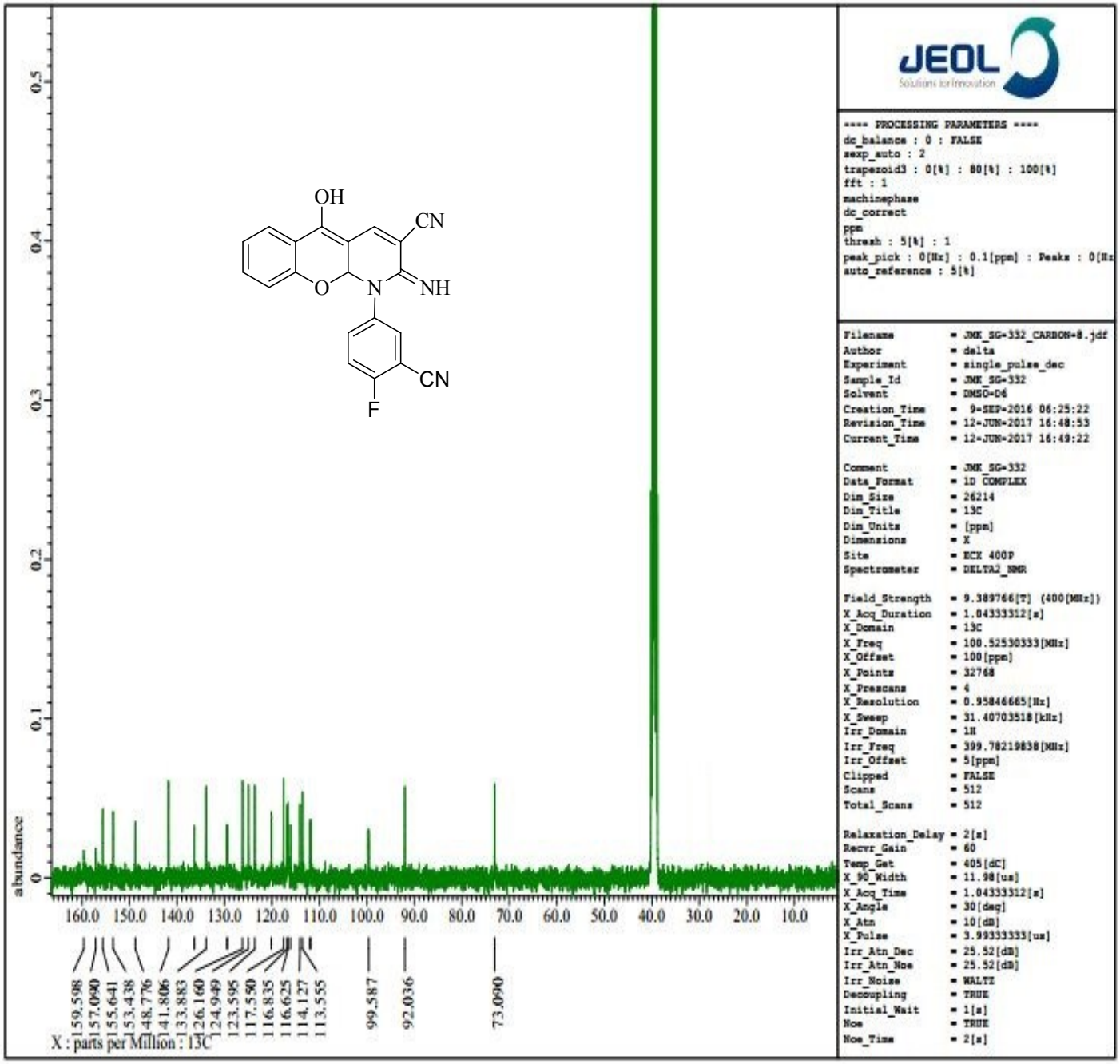
^{13}C NMR OF IVl



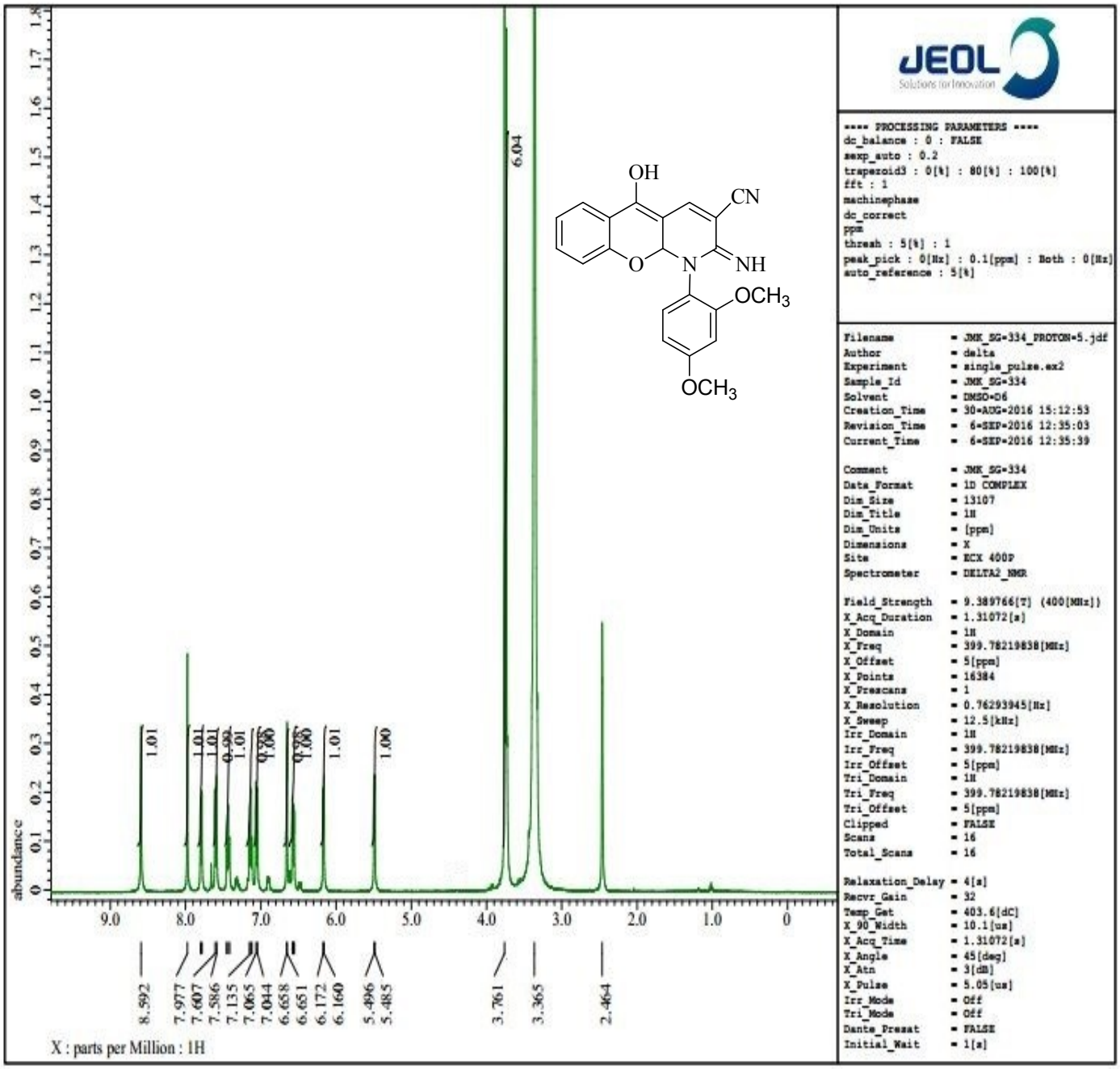
^{1}H NMR OF IVm


OH
CN
NH
O
N
CN
F
abundance
X : parts per Million : 1H
9.764
8.117
8.101
8.095
7.826
7.806
7.535
7.466
7.155
7.095
7.074
6.226
6.213
5.522
5.510
3.370
2.467
1.00
1.99
2.00
1.00
1.02
1.00
1.00
1.00
JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-332R_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-332R
Solvent = DMSO-D6
Creation_Time = 23-JUN-2017 08:24:38
Revision_Time = 23-JUN-2017 12:16:51
Current_Time = 23-JUN-2017 12:16:57
Comment = JMK_SG-332R
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 34
Temp_Get = 404.1[dC]
X_90_Width = 10.75[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 5.375[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

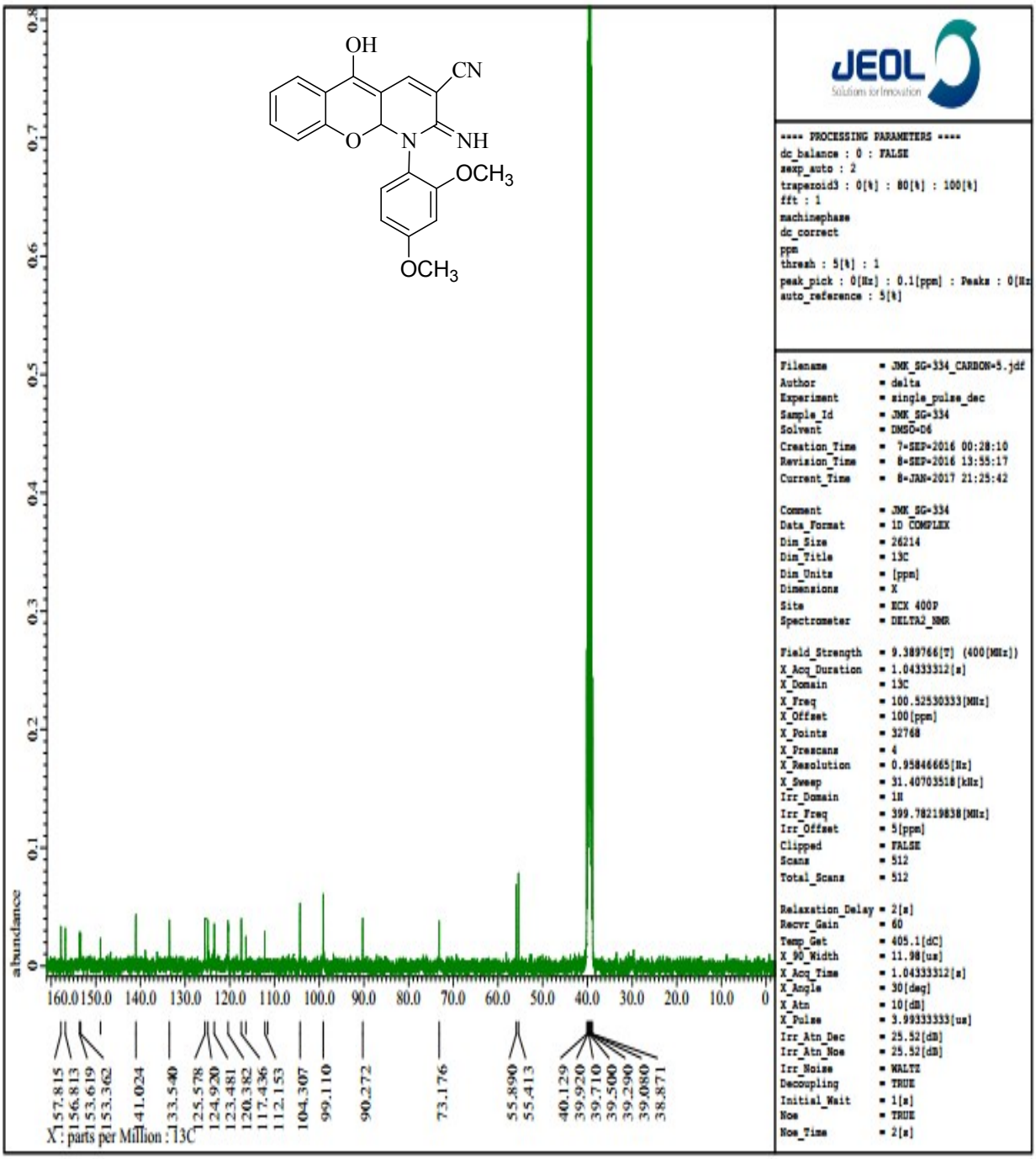
^{13}C NMR OF IVm



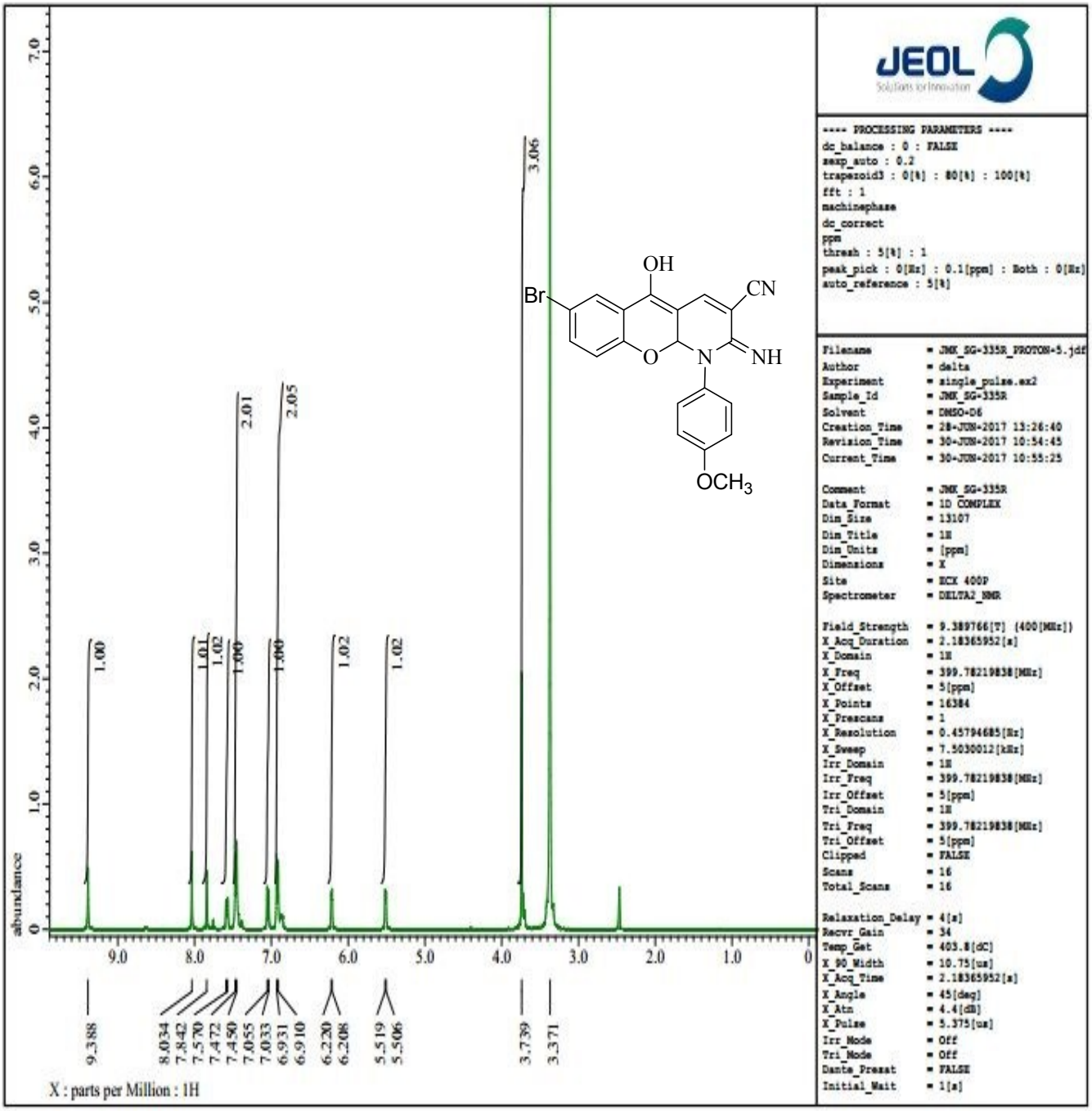
^{1}H NMR OF IVn


OH
CN
O
N
NH
OCH3
OCH3
6.04
1.01
1.01
1.01
0.99
1.01
1.00
1.00
1.00
1.01
1.00
abundance
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.592
7.977
7.607
7.586
7.135
7.065
7.044
6.658
6.651
6.172
6.160
5.496
5.485
3.761
3.365
2.464
X : parts per Million : 1H
JEOL
Solutions for Innovation
==== PROCESSING PARAMETERS ====
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-334_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-334
Solvent = DMSO-D6
Creation_Time = 30-AUG-2016 15:12:53
Revision_Time = 6-SEP-2016 12:35:03
Current_Time = 6-SEP-2016 12:35:39
Comment = JMK_SG-334
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.31072[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.76293945[Hz]
X_Sweep = 12.5[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 32
Temp_Get = 403.6[dC]
X_90_Width = 10.1[us]
X_Acq_Time = 1.31072[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 5.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

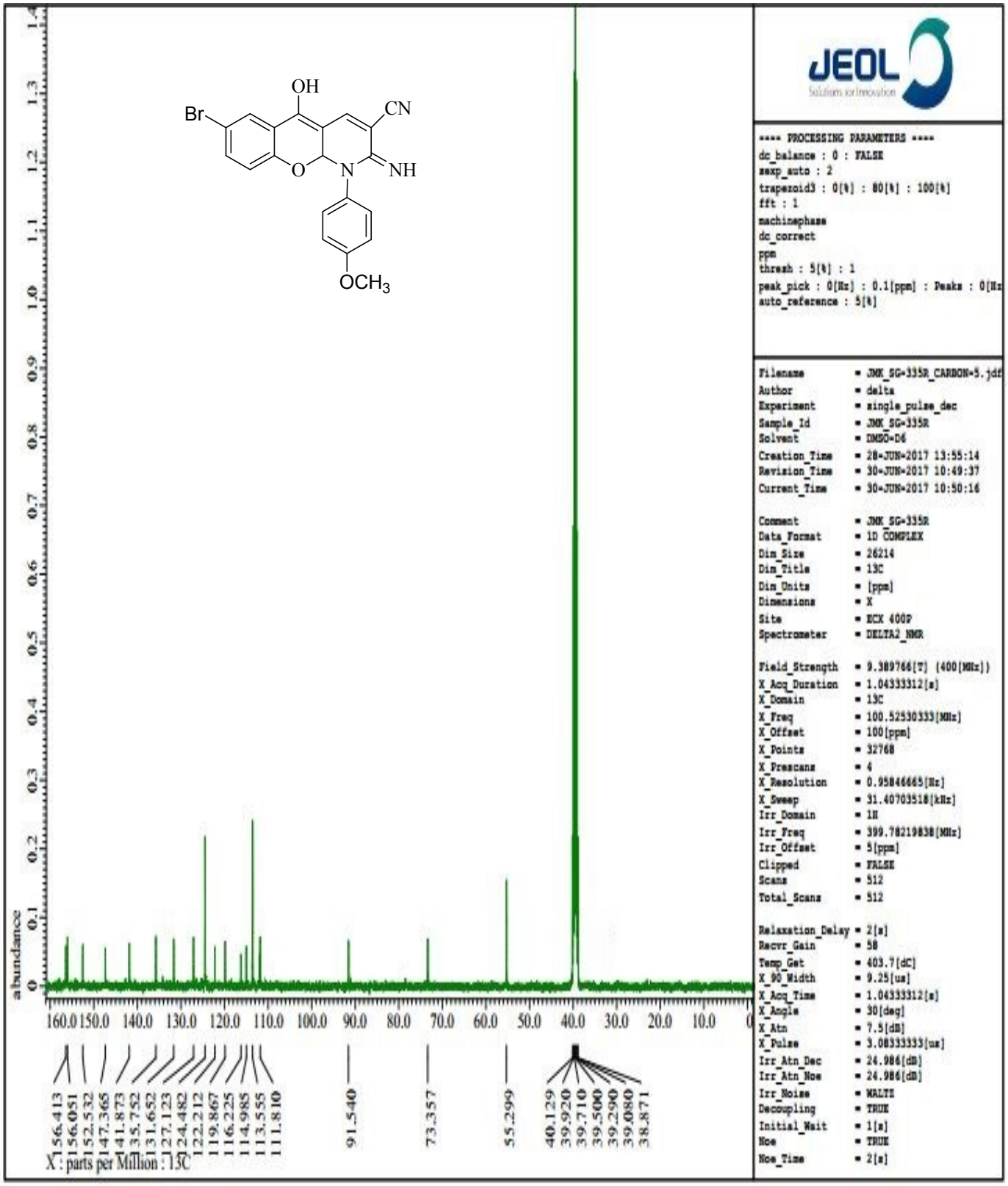
^{13}C NMR OF IVn



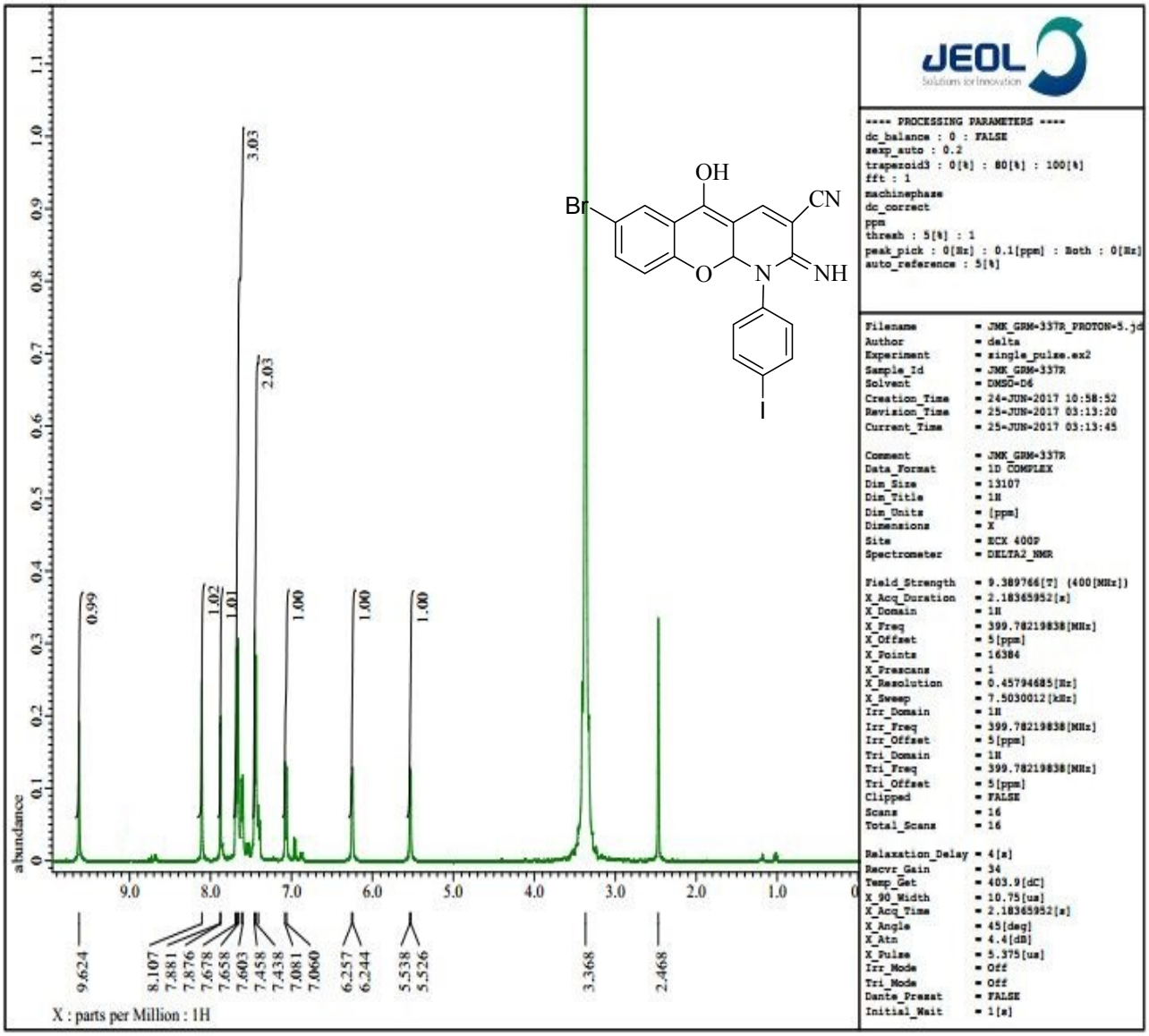
^{1}H NMR OF IVo


OH
Br
CN
O
N
NH
OCH3
abundance
X : parts per Million : 1H
**** PROCESSING PARAMETERS ****
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-335R_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-335R
Solvent = DMSO-D6
Creation_Time = 28-JUN-2017 13:26:40
Revision_Time = 30-JUN-2017 10:54:45
Current_Time = 30-JUN-2017 10:55:25
Comment = JMK_SG-335R
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = NCX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 34
Temp_Get = 403.8[dC]
X_90_Width = 10.75[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 5.375[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

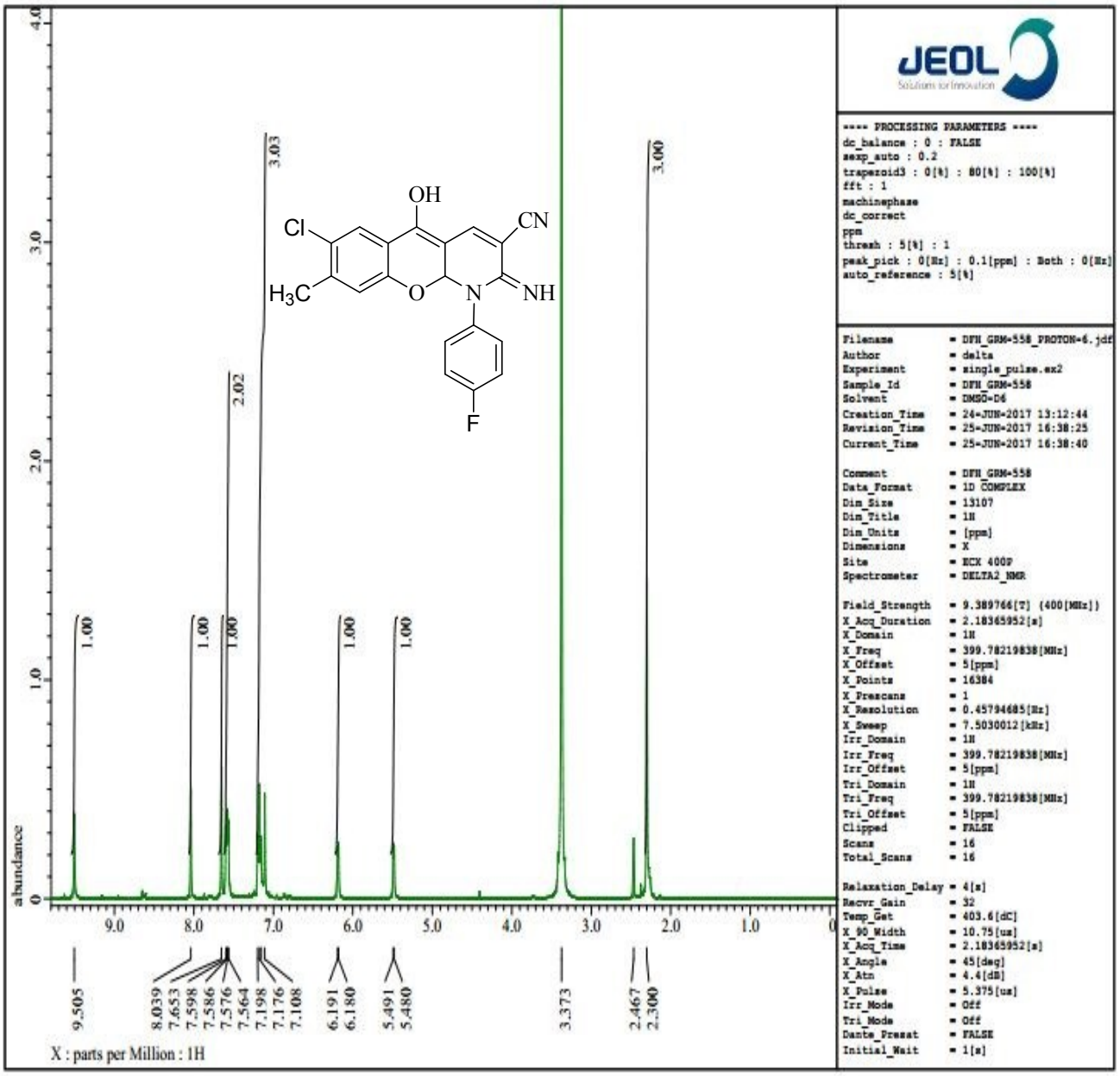
-^{13}C NMR OF IVo



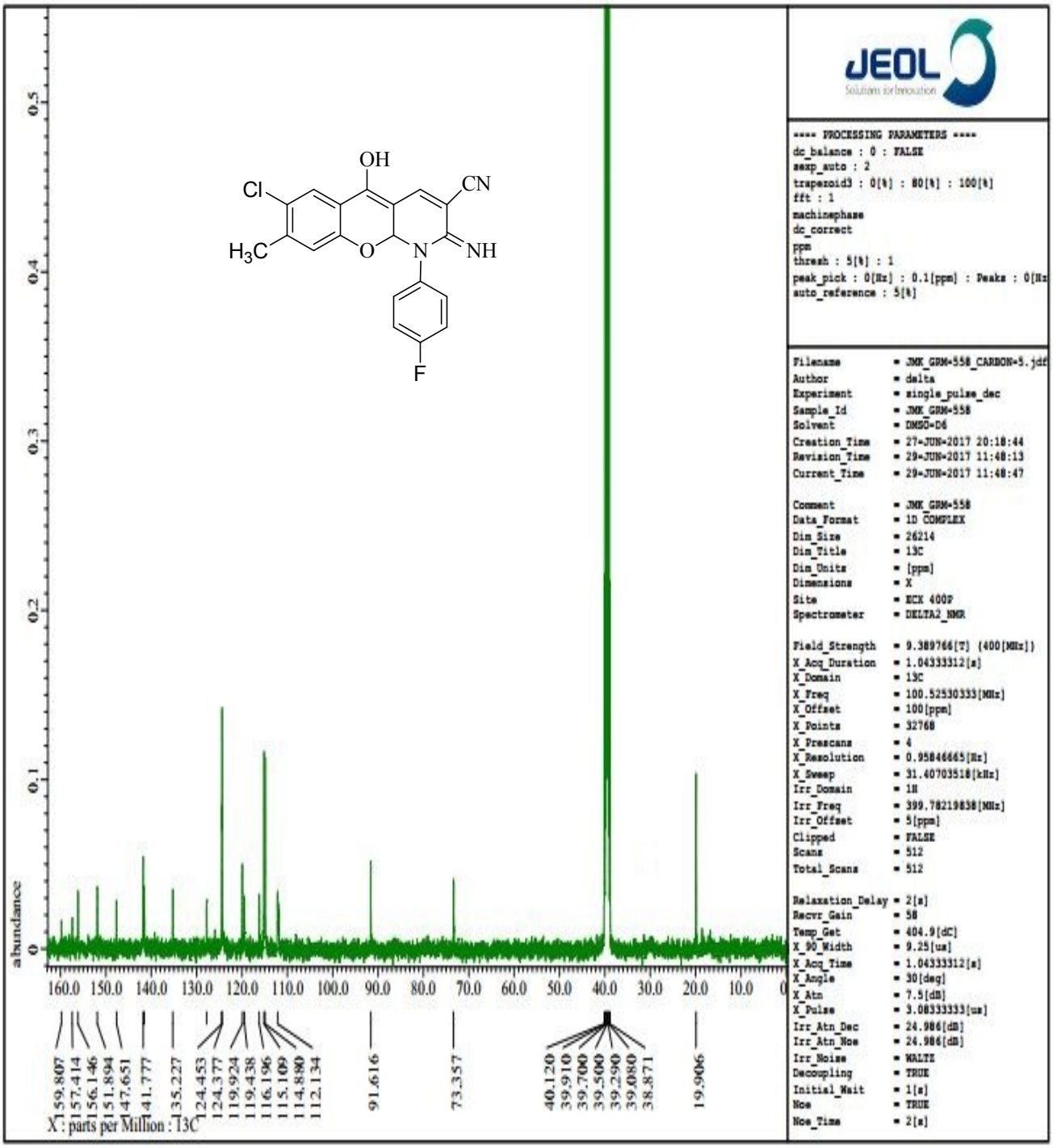
^{1}H NMR OF IVp


OH
CN
Br
O
N
NH
I
3.03
2.03
1.02
1.01
0.99
1.00
1.00
1.00
abundance
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
9.624
8.107
7.881
7.876
7.678
7.658
7.603
7.458
7.438
7.081
7.060
6.257
6.244
5.538
5.526
3.368
2.468
X : parts per Million : 1H
JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
zexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_GRM-337R_PROTON-5.jd
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_GRM-337R
Solvent = DMSO-D6
Creation_Time = 24-JUN-2017 10:58:52
Revision_Time = 25-JUN-2017 03:13:20
Current_Time = 25-JUN-2017 03:13:45
Comment = JMK_GRM-337R
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 34
Temp_Get = 403.9[dC]
X_90_Width = 10.75[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 5.375[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

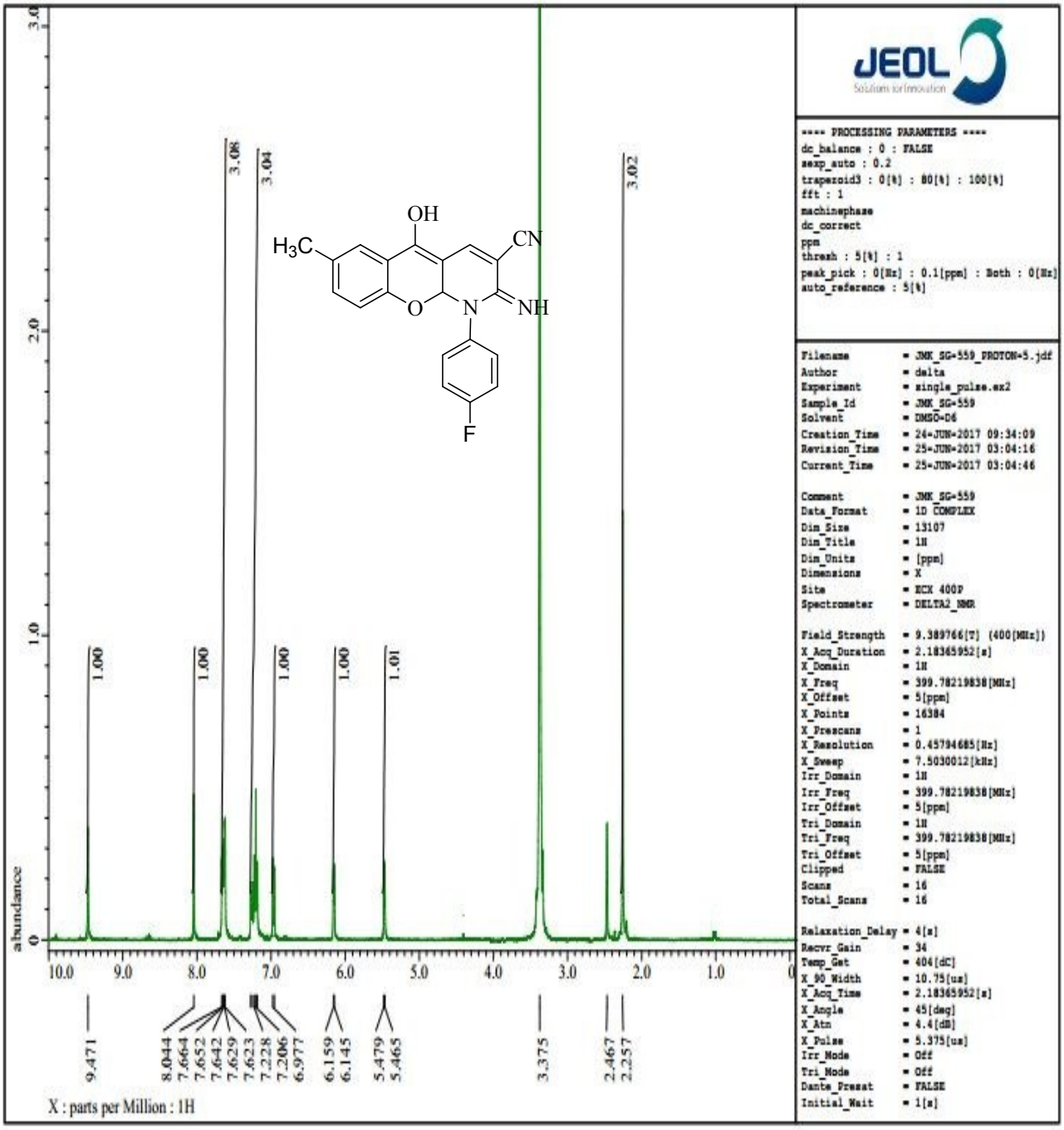
[1]H NMR OF IVq


OH
CN
Cl
H3C
O
N
NH
F
3.03
2.02
1.00
3.00
abundance
X : parts per Million : 1H
9.505
8.039
7.653
7.598
7.586
7.576
7.564
7.198
7.176
7.108
6.191
6.180
5.491
5.480
3.373
2.467
2.300
JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = DFH_GRM-558_PROTON-6.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = DFH_GRM-558
Solvent = DMSO-D6
Creation_Time = 24-JUN-2017 13:12:44
Revision_Time = 25-JUN-2017 16:38:25
Current_Time = 25-JUN-2017 16:38:40
Comment = DFH_GRM-558
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 32
Temp_Get = 403.6[dC]
X_90_Width = 10.75[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 5.375[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

^{13}C NMR OF IVq



[1]H NMR OF IVr


OH
CN
H_3C
O
N
NH
F
3.08
3.04
3.02
1.00
1.00
1.00
1.00
1.01
abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
9.471
8.044
7.664
7.652
7.642
7.629
7.623
7.228
7.206
6.977
6.159
6.145
5.479
5.465
3.375
2.467
2.257
X : parts per Million : 1H
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---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
Filename = JMK_SG-559_PROTON-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = JMK_SG-559
Solvent = DMSO-D6
Creation_Time = 24-JUN-2017 09:34:09
Revision_Time = 25-JUN-2017 03:04:16
Current_Time = 25-JUN-2017 03:04:46
Comment = JMK_SG-559
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 4[s]
Recvr_Gain = 34
Temp_Get = 404[dC]
X_90_Width = 10.75[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 5.375[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]

*

^{13}C NMR OF IVr