Supporting Information

Eco-friendly diastereoselective synthesis of dihydrofuropyrido[2,3-d]pyrimidines via pyridinium ylides in water

Somayeh Ahadi,[a] Telma Kamranifard,[a] Mahsa Armaghan,[b] Hamid Reza Khavasi[a] and Ayoob Bazgir*[a]

[a]Department of Chemistry, Shahid Beheshti University G. C., Tehran 1983963113, Iran.
[b]Institute of Materials Research and Engineering, Agency for Science Technology and Research, 3 Research Link, S117602, Singapore
Email:a_bazgir@sbu.ac.ir

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General methods

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected IR spectra were recorded using a BOMEM MB-Series. ^{1}H and ^{13}C NMR spectra was recorded on a BRUKER DRX-300 AVANCE spectrometer. Chemical shifts are expressed in parts per million downfield from tetramethylsilane as an internal standard. Elemental analyses for C, H and N performed using a Heraeus CHN–O–Rapid analyzer. MS spectra were recorded on a Shimadzu QP 1100EX mass spectrometer operating at an ionization potential of 70 eV.

X-ray crystallography: The X-ray diffraction measurements were made on a STOE IPDS-II diffractometer with graphite monochromated Mo-Kα radiation. Cell constants and an orientation matrix for data collection were obtained by least-squares refinement of diffraction data from 4713 unique reflections for **4c**. Data were collected at a temperature of 298(2) K to a maximum 2θ value of 54.00° and in a series of ω scans in 1° oscillations and integrated using the Stoe X-AREA[1] software package. The data were corrected for Lorentz and Polarizing effects. The structures were solved by direct methods and refined on F^2 by full-matrix least-squares procedure. All hydrogen atoms were added at ideal positions and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}$. All refinements were performed using the X-STEP32 crystallographic software package.[2] Complete crystallographic data for compound **4c** has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication No. CCDC 955501. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, U.K. Fax: 0044 1223 336 033. Email: deposit@ccdc.cam.ac.uk

[1] Stoe & Cie, X–AREA, vesion 1.30: Program for the acquisition and analysis of data; Stoe & Cie GmbH: Darmatadt, Germany (2009).

[2] Stoe & Cie, X-STEP32, Version 1.07b: Crystallographic package; Stoe & Cie GmbH: Darmstadt, Germany (2000).

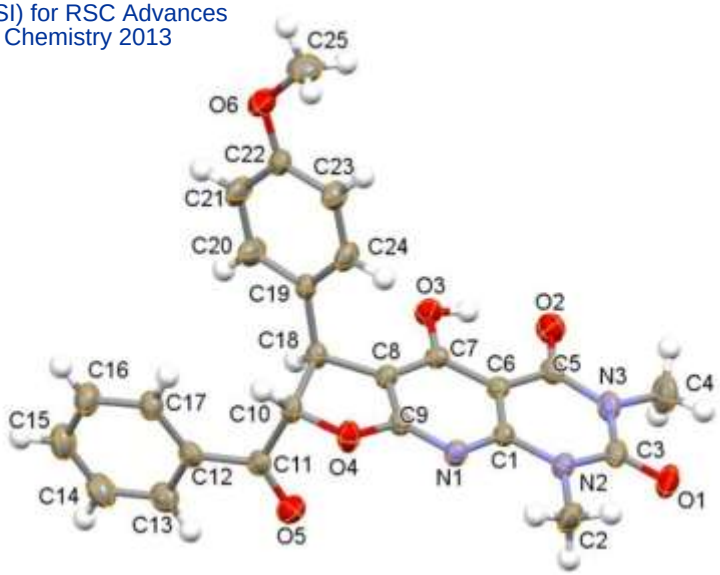
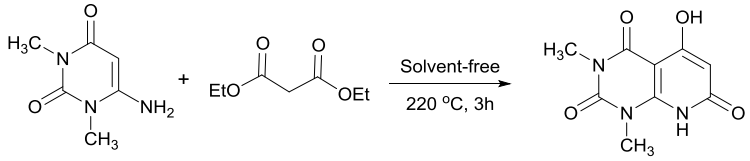


Fig. X-Ray crystal structure of **4c**

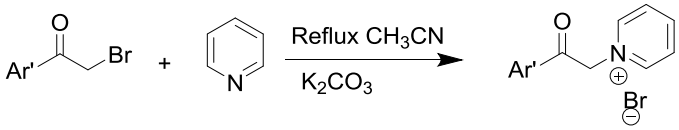
Synthesis of starting material 1:

A mixture of 6-amino-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione (1mmol) and diethyl malonate (4 mmol) was heated at 220 °C for 3h. Then, the precipitate washed with ethanol to produce pure product **1**.

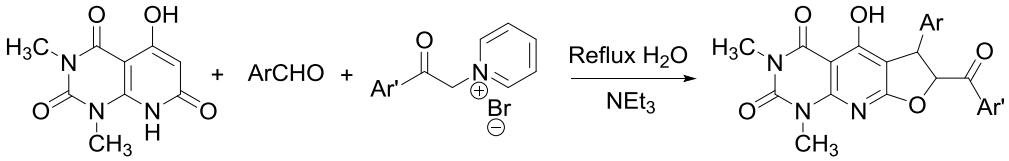


Synthesis of starting material 3:

A mixture of phenacyl bromide (1 mmol), pyridin (1.2 mmol) in MeCN (2 mL) in the presence of K_2CO_3 (30 mol%) was refluxing for 2 h. Then the precipitated product was filtered and washed with MeCN to produce pure product.



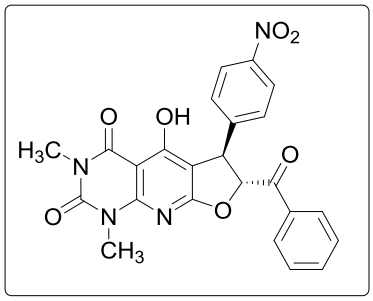
General procedure for the synthesis of 4



1 **2** **3** **4**

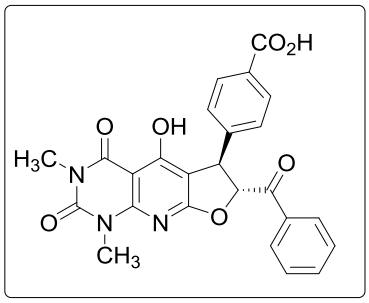
A mixture of 5-hydroxy-1,3-dimethylpyrido[2,3-*d*]pyrimidine-2,4,7(1*H*,3*H*,8*H*)-trione (1.0 mmol), aromatic aldehydes (1.0 mmol) and 1-(2-oxo-2-phenylethyl)pyridin-1-ium (1 mmol) in H_20 (2 mL) in the presence of NEt_3

(30 mol%) was refluxing for 48 h. After completion of the reaction (TLC), the reaction mixture was cooled to room temperature. Then, the precipitated product was filtered and washed with ethanol (5 ml) to afford the pure product **4**.



7-benzoyl-5-hydroxy-1,3-dimethyl-6-(4-nitrophenyl)-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (4a)

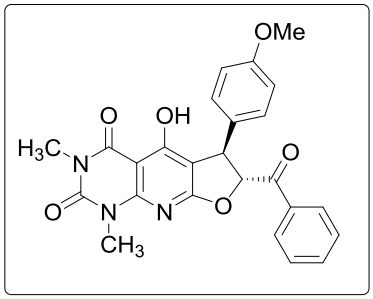
Brown powder (yield 97%); mp 198-205 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3379, 3051, 1678, 1600. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.30 (3H, s, CH_3), 3.56 (3H, s, CH_3), 5.04 (1H, d, *J*= 4.5 Hz, CH), 5.99 (1H, d, *J*= 4.62 Hz, CH), 7.39-7.60 (5H, m, H-Ar), 7.85 (2H, d, *J*= 7.61 Hz, H-Ar), 8.14 (2H, d, *J*= 8.13 Hz, H-Ar), 12.40 (1H, s, OH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 28.2, 30.5, 46.5, 88.6, 93.8, 100.0, 119.1, 122.2, 125.0, 129.7, 130.0, 135.1, 136.2, 155.1, 155.7, 164.8, 172.2, 194.7. Anal. Calcd for $C_{24}H_{18}N_4O_7$: C, 60.76; H, 3.82; N, 11.81. Found: C, 60.69; H, 3.77; N, 11.87.



7-benzoyl-5-hydroxy-1,3-dimethyl-2,4-dioxo-1,2,3,4,6,7-hexahydrofuro[3',2':5,6]pyrido[2,3-*d*]pyrimidin-6-yl)benzoic acid (4b)

White powder (yield 60%); mp 227-233 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3313, 3075, 3099, 1714, 1666, 1621. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.22 (3H, s, CH_3), 3.49 (3H, s, CH_3), 4.92 (1H, bs, CH), 6.47 (1H, bs, CH), 7.33-7.79 (5H, m, H-Ar), 7.89-7.94 (4H, m, H-Ar), 12.64 (2H,

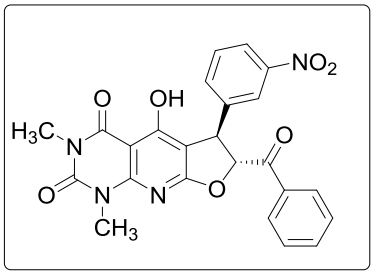
bs, 2OH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 28.2, 30.6, 46.5, 88.7, 93.8, 100.9, 128.6, 129.8, 129.9, 130.7, 131.0, 134.3, 135.1, 145.7, 151.0, 154.0, 165.1, 166.0, 167.8, 171.8, 194.3. MS (EI, 70 eV) m/z: 473 (M^+). Anal. Calcd for $C_{25}H_{19}N_3O_7$: C, 63.42; H, 4.05; N, 8.88. Found: C, 63.37; H, 4.09; N, 8.80.



7-benzoyl-5-hydroxy-6-(4-methoxyphenyl)-1,3-dimethyl-6,7-dihydrofuro[3',2':5,6]pyrido [2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (4c)

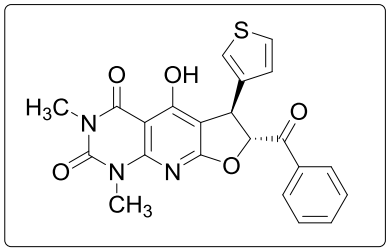
Cream powder (yield 84%); mp 194-204 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3598, 1701, 1633. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.22 (3H, s, CH_3), 3.48 (3H, s, CH_3), 3.73 (3H, s, OCH_3), 4.70 (1H, bs, CH), 6.36 (1H, bs, CH), 6.91 (2H, bs, H-Ar), 7.10 (2H, bs, H-Ar), 7.54 (2H, bs, H-Ar), 7.70 (1H, bs, H-Ar), 7.87 (2H, bs, H-Ar), 12.40 (1H, s, OH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 28.2, 30.5, 46.3, 56.0, 89.4, 93.7, 101.5, 115.1, 129.3, 129.8, 132.9, 134.2, 135.1, 151.0, 153.8, 159.5, 165.0, 166.0, 171.8, 194.5. MS (EI, 70 eV) m/z: 459 (M^+). Anal. Calcd for $C_{25}H_{21}N_3O_6$: C, 65.35; H, 4.61; N, 9.15. Found: C, 65.73; H, 4.56; N, 9.21.

Crystal data for **4c** $C_{25}H_{21}N_3O_6$ (CCDC 955501): M= 459.45 g/mol, monoclinic system, space group P21/a, a = 9.6442(12) Å, b = 14.0350(13) Å, c = 15.9534(18) Å, β = 91.025(9)^o, V= 2159.0(4) Å^3, Z = 4, Dc = 1.413 g.cm^{-3}, μ(Mo-Kα)= 0.103 mm^{-1}, crystal dimension of 0.25 x 0.20 x 0.15 mm. The structure was solved by using SHELXS. The structure refinement and data reduction was carried out with SHELXL of the X-Step32 suite of programs. The non-hydrogen atoms were refined anisotropically by full matrix least-squares on F^2 values to final R_1= 0.0641, wR_2= 0.1675 and S=0.958 with 313 parameters using 4713 independent reflection (θ range = 1.93 – 27.00^o). Hydrogen atoms were located from expected geometry and were not refined.



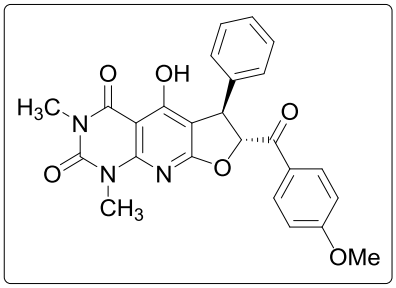
7-benzoyl-5-hydroxy-1,3-dimethyl-6-(3-nitrophenyl)-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (4d)

Cream powder (yield 81%); mp 187-194 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3452, 1708, 1624. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.22 (3H, s, CH_3), 3.48 (3H, s, CH_3), 5.14 (1H, bs, CH), 6.53 (1H, bs, CH), 7.54-7.70 (5H, m, H-Ar), 7.91 (2H, d, *J*= 7.08 Hz, H-Ar), 8.08 (1H, bs, H-Ar), 8.18 (1H, d, *J*= 6.23 Hz, H-Ar), 12.47 (1H, s, OH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 28.2, 30.6, 45.9, 88.3, 93.7, 100.6, 123.2, 123.4, 129.7, 130.0, 131.2, 131.2, 134.5, 135.1, 142.9, 165.1, 171.7, 194.1. MS (EI, 70 eV) m/z: 474 (M^+). Anal. Calcd for $C_{24}H_{18}N_4O_7$: C, 60.76; H, 3.82; N, 11.81. Found: C, 60.70; H, 3.78; N, 11.76.



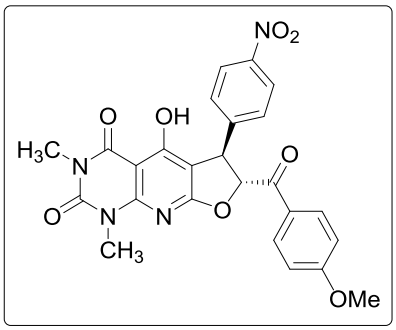
-7-benzoyl-5-hydroxy-1,3-dimethyl-6-(thiophen-3-yl)-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (4e)

Light cream powder (yield 100%); mp 185 ˚C (dec). IR (KBr) (ν_{max} /cm^{-1}): 3635, 1705, 1671, 1621. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.23 (3H, s, CH_3), 3.48 (3H, s, CH_3), 5.14 (1H, bs, CH), 6.44 (1H, bs, CH), 7.00 (2H, bs, H-Ar), 7.47-7.73 (4H, m, H-Ar), 7.96 (2H, d, *J*= 4.67 Hz, H-Ar), 12.56 (1H, s, OH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 28.2, 30.6, 41.9, 89.1, 93.7, 101.2, 126.4, 128.1, 129.9, 134.1, 135.2, 143.9, 151.0, 154.3, 163.2, 165.3, 166.0, 171.5, 194.1. MS (EI, 70 eV) m/z: 435 (M^+). Anal. Calcd for $C_{22}H_{17}N_3O_5S$: C, 60.68; H, 3.94; N, 9.65. Found: C, 60.78; H, 3.87; N, 9.73.



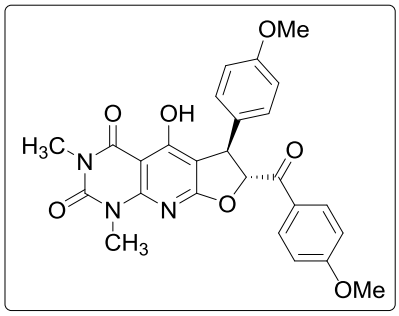
5-hydroxy-7-(4-methoxybenzoyl)-1,3-dimethyl-6-phenyl-6,7-dihydrofuro[3',2':5,6]pyrido [2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dionedione (4f)

Cream powder (yield 59%); mp 211 ˚C D. IR (KBr) (ν_{max} /cm^{-1}): 3531, 1709, 1671, 1678, 1615. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.22 (3H, s, CH_3), 3.49 (3H, s, CH_3), 3.85 (3H, s, OCH_3), 4.75 (1H, bs, CH), 6.37 (1H, bs, CH), 7.06-7.34 (7H, m, H-Ar), 7.86 (2H, bs, H-Ar), 12.35 (1H, bs, OH). MS (EI, 70 eV) m/z: 459 (M^+). Anal. Calcd for $C_{25}H_{21}N_3O_6$: C, 65.35; H, 4.61; N, 9.15. Found: C, 65.26; H, 4.67; N, 9.06.



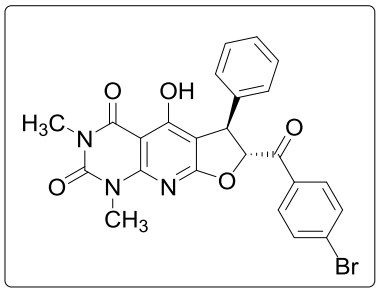
5-hydroxy-7-(4-methoxybenzoyl)-1,3-dimethyl-6-(4-nitrophenyl)-6,7-dihydrofuro[3',2':5,6] pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (4g)

Yellow powder (yield 60%); mp 195-200 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3602, 1704, 1672, 1625. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.22 (3H, s, CH_3), 3.48 (3H, s, CH_3), 3.85 (3H, s, OCH_3), 5.04 (1H, bs, CH), 6.46 (1H, bs, CH), 7.08 (2H, d, *J*= 8.21 Hz, H-Ar), 7.53 (2H, d, *J*= 7.76 Hz, H-Ar), 7.89 (2H, d, *J*= 7.92 Hz, H-Ar), 8.23 (2H, d, *J*= 7.60 Hz, H-Ar), 12.44 (1H, s, OH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 28.2, 30.6, 46.3, 56.6, 88.1, 93.8, 100.7, 115.2, 124.8, 127.0, 129.9, 132.4, 147.8, 148.4, 151.0, 154.1, 165.0, 166.0, 192.2. MS (EI, 70 eV) m/z: 504 (M^+). Anal. Calcd for $C_{25}H_{20}N_4O_8$: C, 59.52; H, 4.00; N, 11.11. Found: C, 59.61; H, 4.07; N, 11.07.



5-hydroxy-7-(4-methoxybenzoyl)-6-(4-methoxyphenyl)-1,3-dimethyl-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (4h)

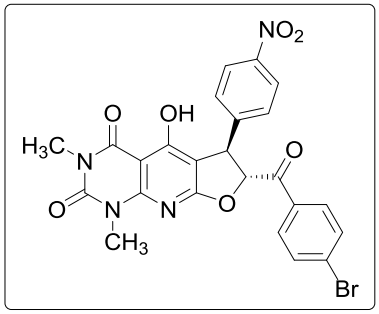
Yellow powder (yield 94%); mp 163-170 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3501, 1704, 1624. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.22 (3H, s, CH_3), 3.33 (3H, s, CH_3), 3.49 (3H, s, OCH_3), 3.85 (3H, s, OCH_3), 4.68 (1H, bs, CH), 6.32 (1H, bs, CH), 6.91 (2H, d, *J*= 8.02 Hz, H-Ar), 7.06-7.15 (4H, m, H-Ar), 7.86 (2H, d, *J*= 8.47 Hz, H-Ar), 12.40 (1H, s, OH). MS (EI, 70 eV) m/z: 489 (M^+). Anal. Calcd for $C_{26}H_{23}N_3O_7$: C, 63.80; H, 4.74; N, 8.58. Found: C, 63.74; H, 4.70; N, 8.52.



7-(4-bromobenzoyl)-5-hydroxy-1,3-dimethyl-6-phenyl-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (4i)

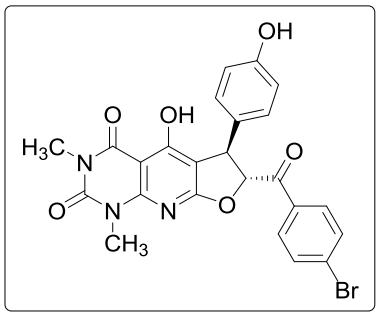
Cream powder (yield 60%); mp 202-210 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3075, 1706, 1670, 1628. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.22 (3H, s, CH_3), 3.48 (3H, s, CH_3), 4.82 (1H, d, *J*= 2.76 Hz, CH), 6.41 (1H, d, *J*= 2.97 Hz, CH), 7.21 (2H, d, *J*= 6.75 Hz, H-Ar), 7.30-7.36 (3H, m, H-Ar), 7.76-7.83 (4H, d, m, H-Ar), 12.42 (1H, s, OH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 28.2, 30.6, 46.5, 89.1, 93.8, 101.3, 128.2, 128.4, 129.3, 129.7, 131.8, 132.8, 133.4, 140.9, 151.0,

153.9, 165.1, 166.1, 171.8, 193.8. MS (EI, 70 eV) m/z: 509 (M^+, ^{81}Br), 507 (M^+, ^{79}Br). Anal. Calcd for $C_{24}H_{18}BrN_3O_5$: C, 56.71; H, 3.57; N, 8.27. Found: C, 56.63; H, 3.63; N, 8.34.



7-(4-bromobenzoyl)-5-hydroxy-1,3-dimethyl-6-(4-nitrophenyl)-6,7-dihydrofuro[3',2':5,6] pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (4j)

Cream powder (yield 80%); mp 259 ˚C (dec). IR (KBr) (ν_{max} /cm^{-1}): 3383, 1699, 1666, 1618. ^1H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.22 (3H, s, CH_3), 3.48 (3H, s, CH_3), 5.11 (1H, d, *J*= 3.12 Hz, CH), 6.49 (1H, d, *J*= 3.21 Hz, CH), 7.53 (2H, d, *J*= 8.13 Hz, H-Ar), 7.77-7.85 (4H, m, H-Ar), 8.23 (2H, d, *J*= 7.98 Hz, H-Ar), 12.46 (1H, s, OH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 28.2, 30.6, 45.9, 88.3, 93.8, 100.6, 124.8, 129.4, 129.9, 131.9, 132.9, 133.4, 147.8, 148.3, 151.0, 154.1, 165.2, 166.0, 171.6, 193.3. MS (EI, 70 eV) m/z: 554 (M^+, ^{81}Br), 552 (M^+, ^{79}Br). Anal. Calcd for $C_{24}H_{17}BrN_4O_7$: C, 52.10; H, 3.10; N, 10.13. Found: C, 52.05; H, 3.06; N, 10.05.

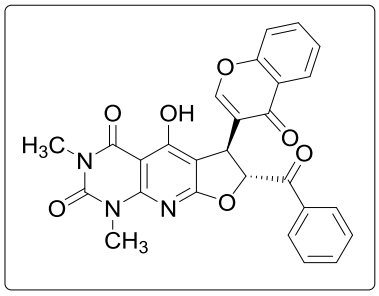


7-(4-bromobenzoyl)-5-hydroxy-6-(4-hydroxyphenyl)-1,3-dimethyl-6,7-dihydrofuro[3',2': 5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (4k)

Cream powder (yield 63%); mp 260 ˚C (dec). IR (KBr) (ν_{max} /cm^{-1}): 3343, 1705, 1657, 1615. ^1H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.21 (3H, s, CH_3), 3.47 (3H, s, CH_3), 4.64 (1H, bs, CH), 6.31 (1H, bs, CH), 6.70 (2H, bs, H-Ar), 6.97 (2H, bs, H-Ar), 7.78 (4H, bs, H-Ar), 9.44 (1H, s, OH), 12.36 (1H, bs, OH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 28.2, 30.5, 46.2, 89.5, 93.8,

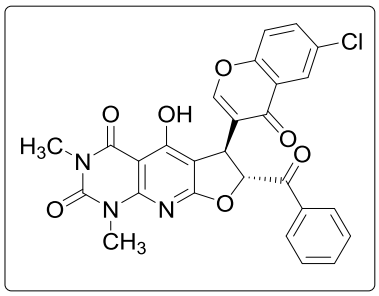
101.6, 116.5, 129.2, 131.2, 131.7, 132.8, 133.3, 151.1, 153.8, 157.7, 165.0, 166.1, 171.8, 194.0. MS (EI, 70 eV) m/z: 525 (M^+, ^{81}Br), 523 (M^+, ^{79}Br). Anal. Calcd for $C_{24}H_{18}BrN_3O_6$: C, 54.98; H, 3.46; N, 8.01. Found: C, 54.91; H, 3.41; N, 7.94.

*Due to very low solubility of the products **9**, we can not report the ^{13}C NMR date for these products.



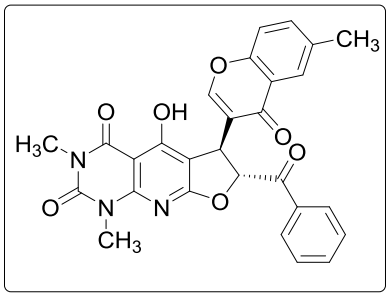
7-benzoyl-5-hydroxy-1,3-dimethyl-6-(4-oxo-4*H*-chromen-3-yl)-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (9a)

Cream powder (yield 70%); mp 260 ˚C (dec). IR (KBr) (ν_{max} /cm^{-1}): 3065, 1686, 1635. ^1H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.45 (6H, s, $2CH_3$), 4.85 (1H, bs, CH), 6.37 (1H, bs, CH), 7.54-7.76 (6H, m, H-Ar), 7.97 (3H, bs, H-Ar), 8.27 (1H, bs, H-Ar), 12.42 (1H, bs, OH). MS (EI, 70 eV) m/z: 497 (M^+). Anal. Calcd for $C_{27}H_{19}N_3O_7$: C, 65.19; H, 3.85; N, 8.45. Found: C, 65.09; H, 3.79; N, 8.52.



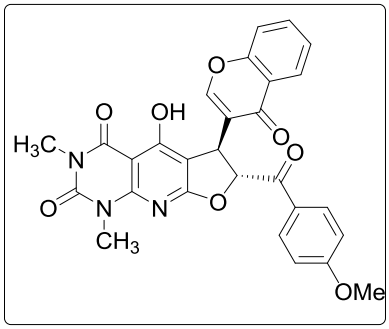
7-benzoyl-6-(6-chloro-4-oxo-4*H*-chromen-3-yl)-5-hydroxy-1,3-dimethyl-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (9b)

Cream powder (yield 60%); mp 237-245 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3428, 1708, 1676, 1632. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.23(3H, s, CH_3), 3.46 (3H, s, CH_3), 4.87 (1H, bs, CH) 6.38 (1H, bs, CH), 7.22-7.99 (8H, m, H-Ar), 8.35 (1H, bs, H-Ar), 12.35 (1H, bs, OH). MS (EI, 70 eV) m/z: 532 (M^+). Anal. Calcd for $C_{28}H_{21}N_3O_7$: C, 60.97; H, 3.41; N, 7.90. Found: C, 60.90; H, 3.48; N, 7.99.



7-benzoyl-5-hydroxy-1,3-dimethyl-6-(6-methyl-4-oxo-4*H*-chromen-3-yl)-6,7-dihydrofuro[3', 2':5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (9c)

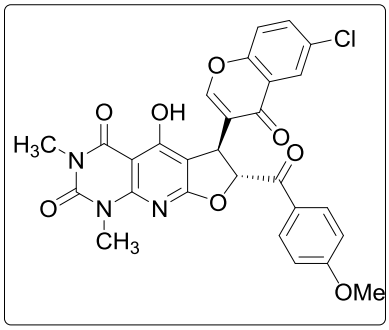
Cream powder (yield 100%); mp>260 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3467, 1710, 1618. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 2.40 (3H, s, CH_3), 3.38 (3H, s, CH_3), 3.47 (3H, s, CH_3), 4.86 (1H, bs, CH), 6.37 (1H, bs, CH), 7.56-7.78 (6H, m, H-Ar), 8.01 (2H, d, *J*= 6.09 Hz, H-Ar), 8.26 (1H, bs, H-Ar), 12.39 (1H, bs, OH). MS (EI, 70 eV) m/z: 511 (M^+). Anal. Calcd for $C_{28}H_{21}N_3O_7$: C, 65.75; H, 4.14; N, 8.22. Found: C, 65.82; H, 4.19; N, 8.16.



5-hydroxy-7-(4-methoxybenzoyl)-1,3-dimethyl-6-(4-oxo-4*H*-chromen-3-yl)-6,7-dihydrofuro [3',2':5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (9d)

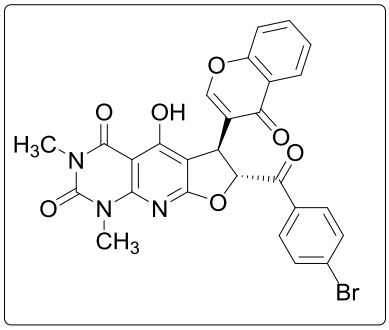
Light cream powder (yield 85%); mp 243 ˚C (dec). IR (KBr) (ν_{max} /cm^{-1}): 3087, 1700, 1670, 1636, 1608. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.23 (3H, s, CH_3), 3.47 (3H, s, CH_3), 3.85 (3H, s, CH_3), 4.83 (1H, bs, CH), 6.34 (1H, bs, CH), 7.08 (2H, d, *J*= 6.78 Hz, H-Ar), 7.49 (1H, bs,

H-Ar), 7.66 (1H, bs, H-Ar), 7.81 (1H, bs, H-Ar), 7.99 (3H, bs, H-Ar), 8.32 (1H, bs, H-Ar), 12.30 (1H, bs, OH). MS (EI, 70 eV) m/z: 527 (M^+). Anal. Calcd for $C_{28}H_{21}N_3O_8$: C, 63.76; H, 4.01; N, 7.97. Found: C, 63.69; H, 3.96; N, 7.91.



6-(6-chloro-4-oxo-4*H*-chromen-3-yl)-5-hydroxy-7-(4-methoxybenzoyl)-1,3-dimethyl-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (9e)

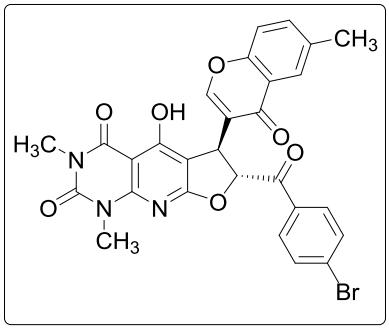
Cream powder (yield 90%); mp 244 ˚C (dec). IR (KBr) (ν_{max} /cm^{-1}): 3075, 1697, 1674, 1646, 1600. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.27 (3H, s, CH_3), 3.51 (3H, s, CH_3), 3.88 (3H, s, CH_3), 4.93 (1H, d, *J*= 4.74 Hz, CH), 6.24 (1H, d, *J*= 4.62 Hz, CH), 7.09 (2H, d, *J*= 7.45 Hz, H-Ar), 7.68-7.84 (2H, m, H-Ar), 7.97-8.02 (4H, m, H-Ar), 8.25 (1H, s, H-Ar), 12.40 (1H, bs, OH). MS (EI, 70 eV) m/z: 562 (M^+). Anal. Calcd for $C_{28}H_{20}ClN_3O_8$: C, 59.85; H, 3.59; N, 7.48. Found: C, 59.94; H, 3.52; N, 7.53.



7-(4-bromobenzoyl)-5-hydroxy-1,3-dimethyl-6-(4-oxo-4H-chromen-3-yl)-6,7-dihydrofuro [3',2':5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (9f)

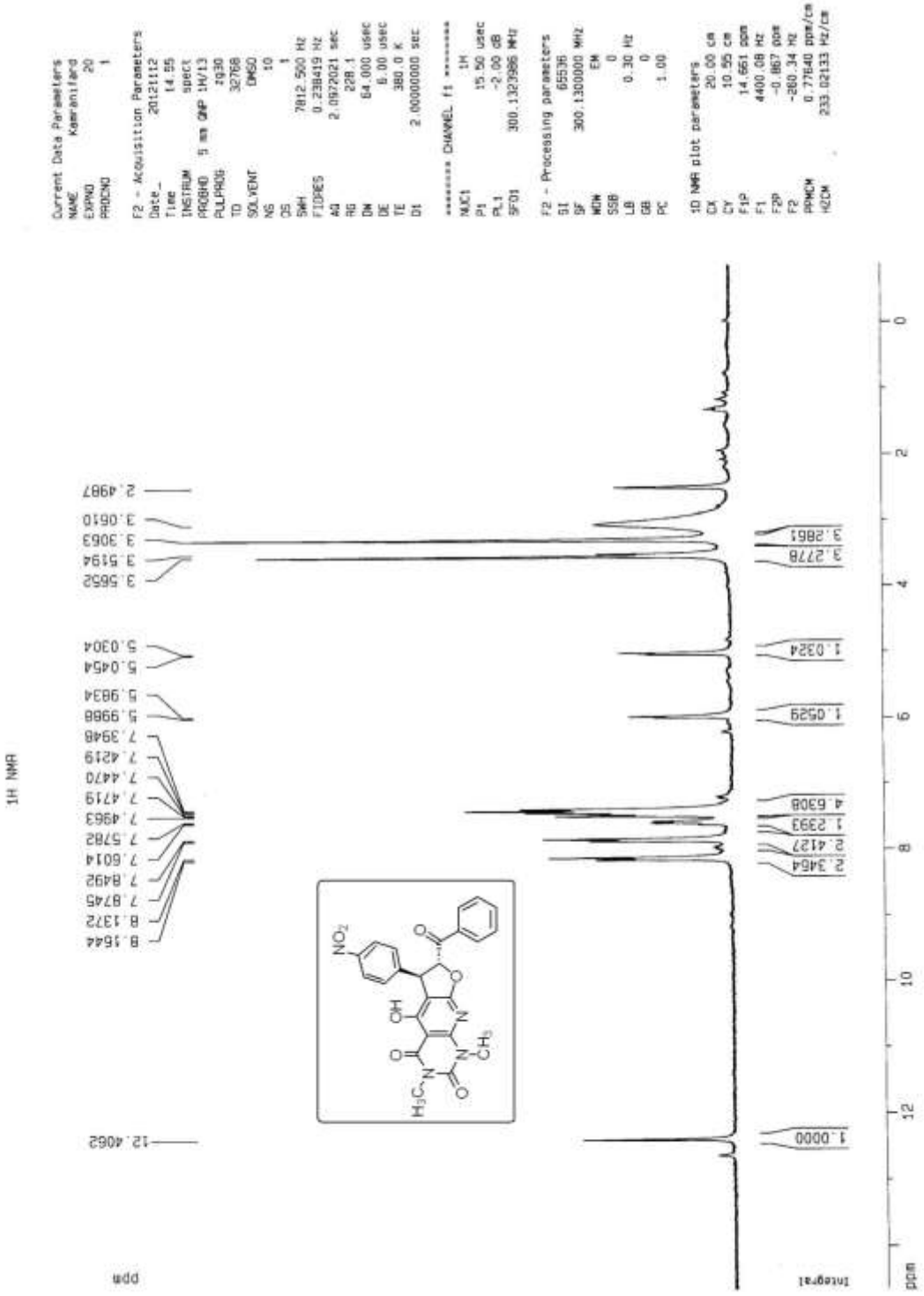
Cream powder (yield 60%); mp >260 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3428, 3075, 1690, 1624. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 2.78 (3H, s, CH_3), 3.03 (3H, s, CH_3), 4.38 (1H, d, *J*= 4.38 Hz, CH), 5.95 (1H, d, *J*= 3.54 Hz, CH), 6.94-7.07 (2H, m, H-Ar), 7.24-7.28 (3H, m, H-Ar), 7.48-

7.57 (3H, m, H-Ar), 7.89 (1H, s, H-Ar), 11.79 (1H, bs, OH). MS (EI, 70 eV) m/z: 577 (M^+, ^{81}Br), 575 (M^+, ^{79}Br). Anal. Calcd for $C_{27}H_{18}BrN_3O_7$: C, 56.27; H, 3.15; N, 7.29. Found: C, 56.17; H, 3.08; N, 7.21.



7-(4-bromobenzoyl)-5-hydroxy-1,3-dimethyl-6-(6-methyl-4-oxo-4*H*-chromen-3-yl)-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione (9g)

Brown powder (yield 65%); mp 245 ˚C (dec). IR (KBr) (ν_{max} /cm^{-1}): 3464, 1698, 1675, 1637. ^1H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 2.39 (3H, s, CH_3), 3.22 (3H, s, CH_3), 3.47 (3H, s, CH_3), 4.81 (1H, bs, CH), 6.37 (1H, bs, CH), 7.58-7.69 (2H, m, H-Ar), 7.79 (3H, bs, H-Ar), 7.91-7.93 (2H, m, H-Ar), 8.28 (1H, bs, H-Ar), 12.44 (1H, bs, OH). MS (EI, 70 eV) m/z: 591 (M^+, ^{81}Br), 589 (M^+, ^{79}Br). Anal. Calcd for $C_{28}H_{20}BrN_3O_7$: C, 56.96; H, 3.41; N, 7.12. Found: C, 56.84; H, 3.48; N, 7.02.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FIDRES 0.238419 Hz
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DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
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F2 - Processing parameters
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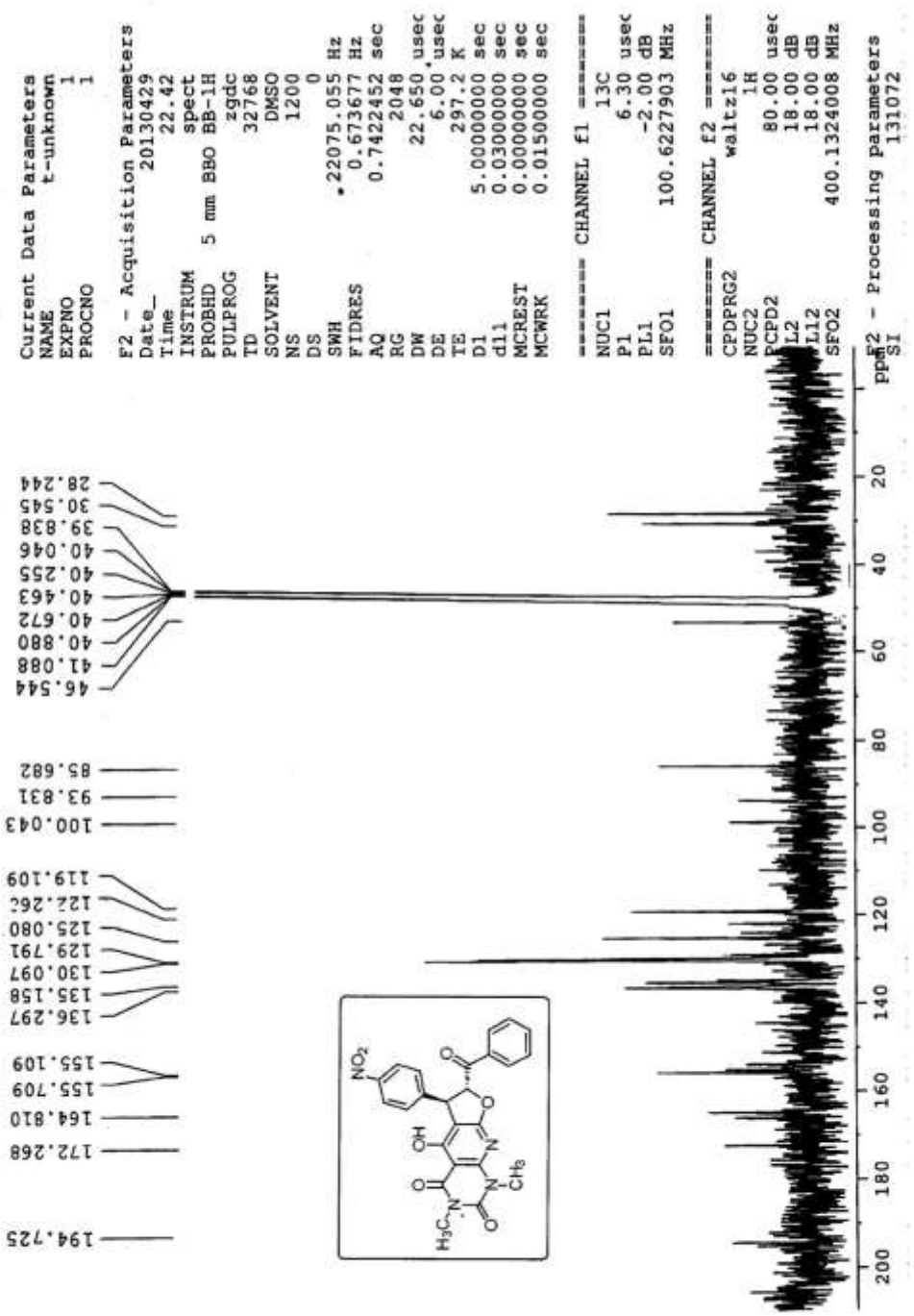
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PROCNO 1
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PULPROG zgdc
TD 32768
SOLVENT DMSO
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FIDRES 0.673677 Hz
AQ 0.7422452 sec
RG 2048
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DE 6.00 usec
TE 297.2 K
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d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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NO2
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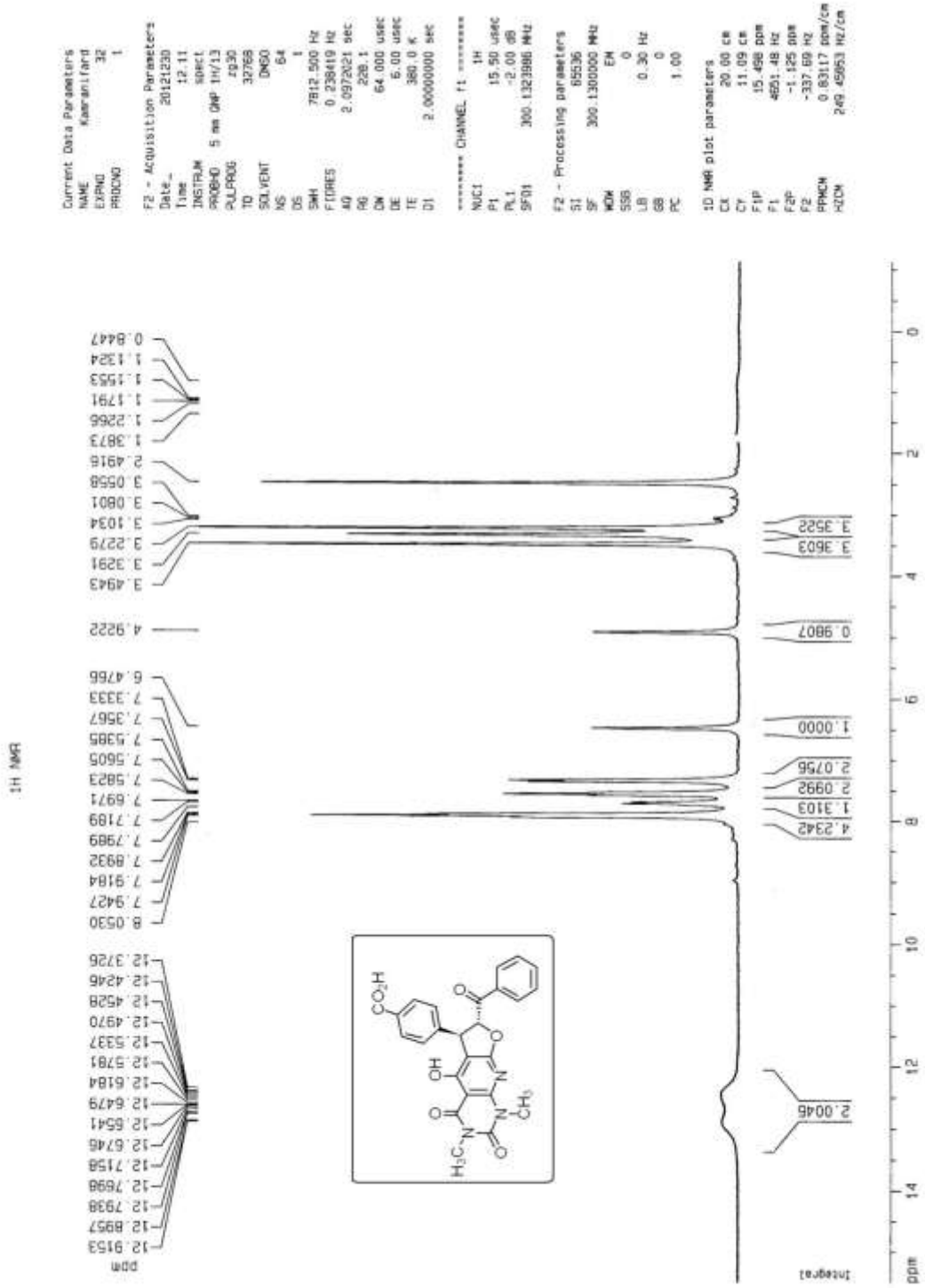
CH3
H3C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PROCNO 1
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FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
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P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
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SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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CY 11.09 cm
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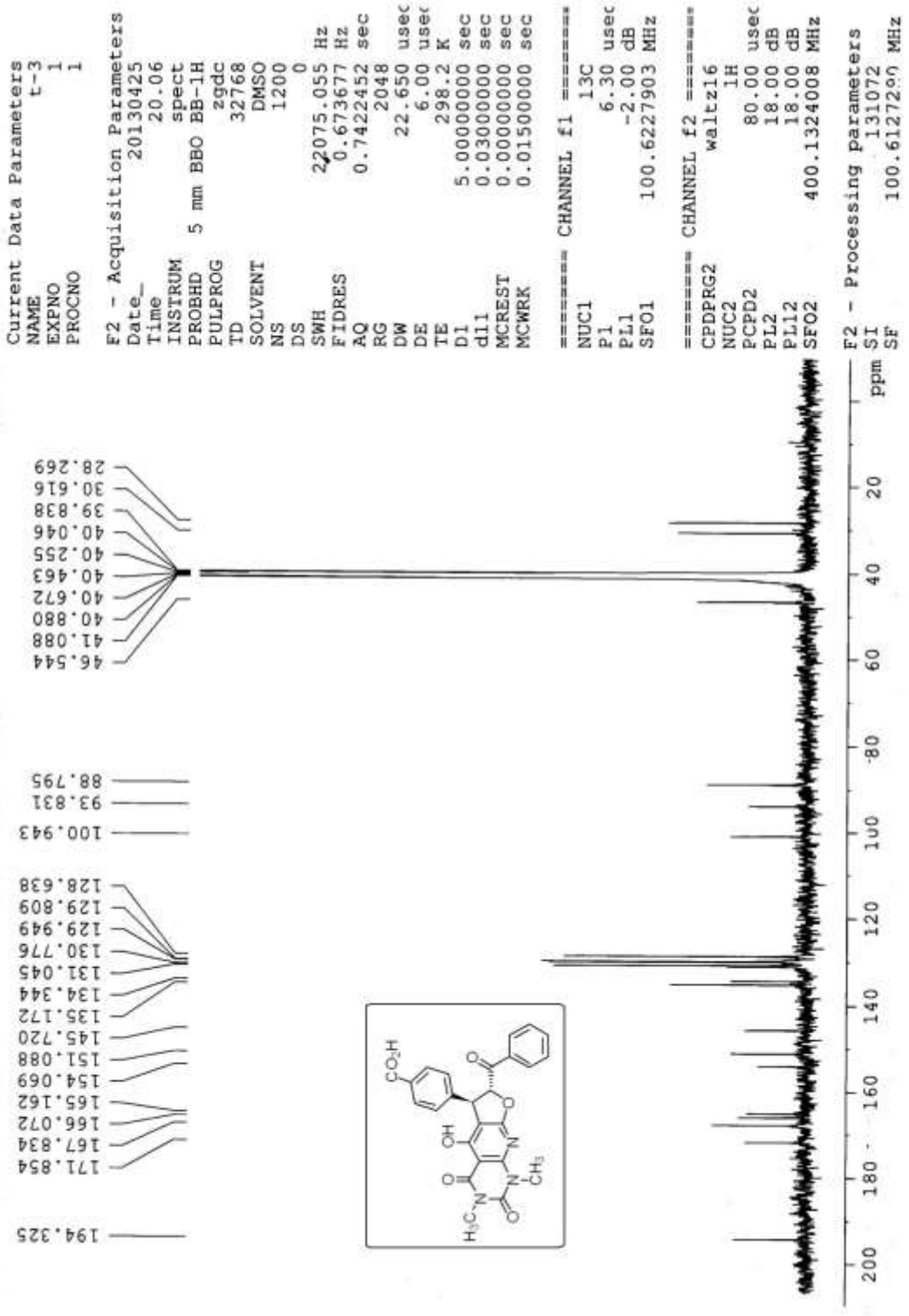
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TD 32768
SOLVENT DMSO
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FIDRES 0.673677 Hz
AQ 0.7422452 sec
RG 2048
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DE 6.00 usec
TE 298.2 K
D1 5.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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NUC1 13C
P1 6.30 usec
PL1 -2.00 dB
SFO1 100.6227903 MHz
======== CHANNEL f2 ========
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NUC2 1H
PCPD2 80.00 usec
PL2 18.00 dB
PL12 18.00 dB
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F2 - Processing parameters
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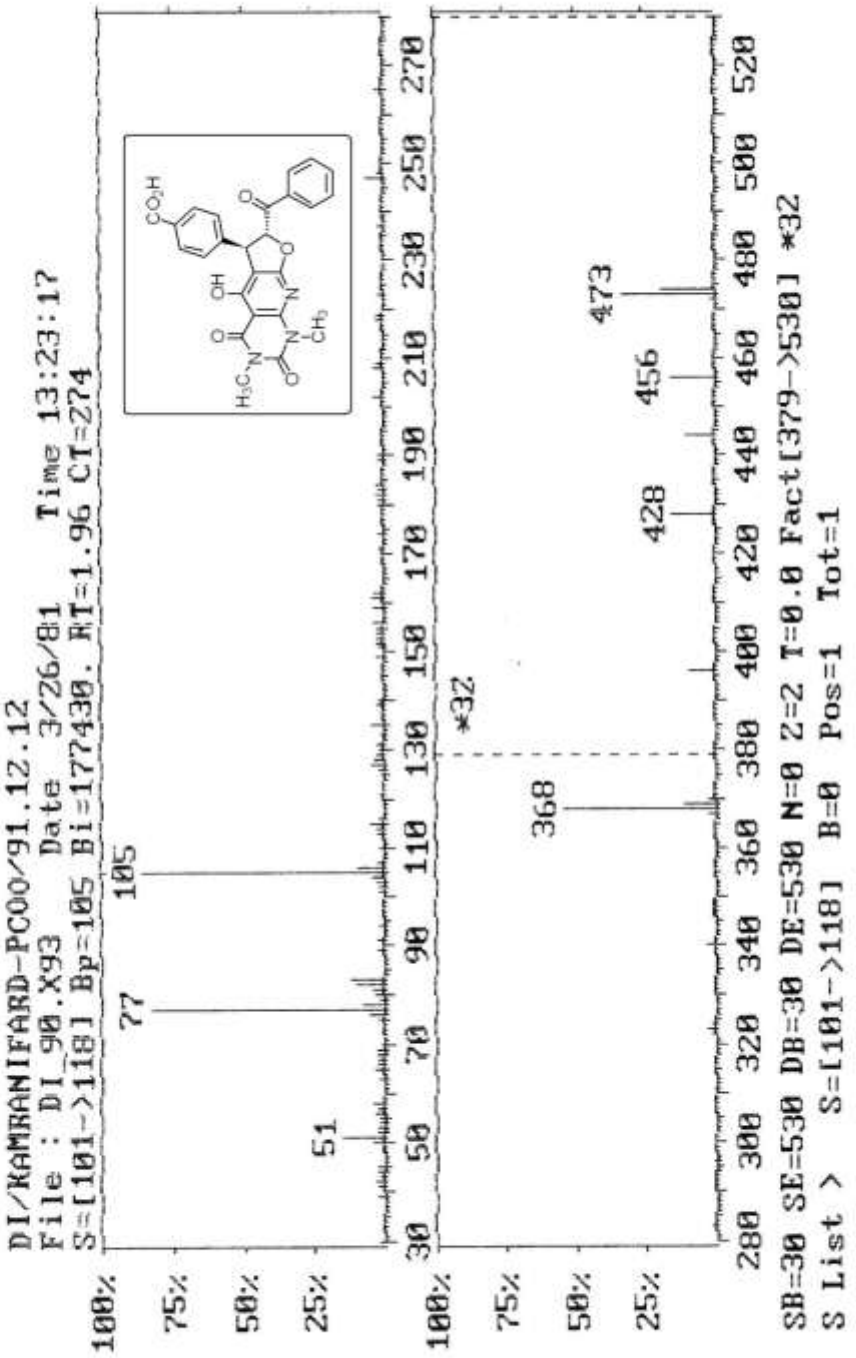
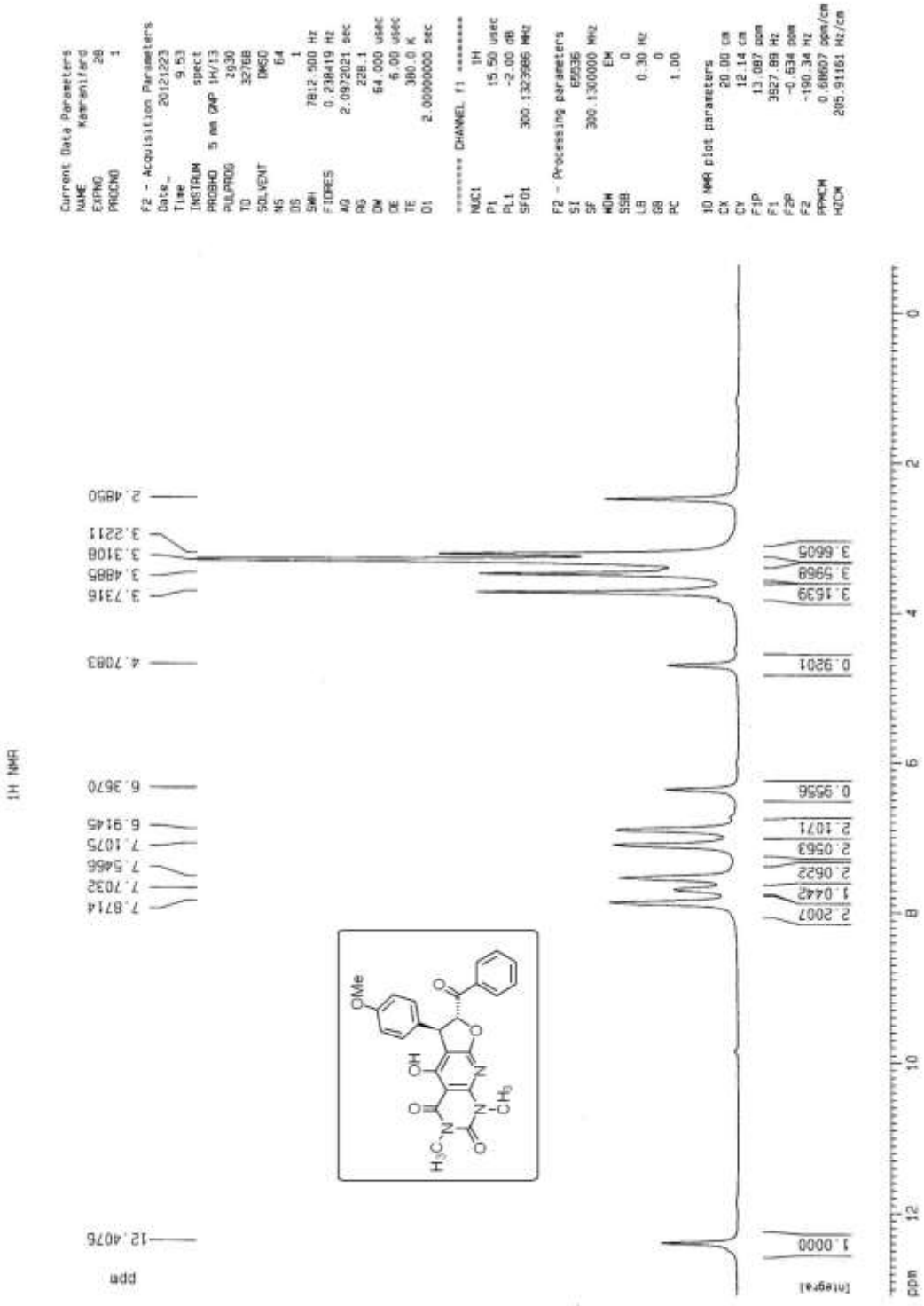
20

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PROCNO 1
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PULPROG zg30
TD 32768
SOLVENT DMSO
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SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
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SSB 0
LB 0.30 Hz
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N-CH3
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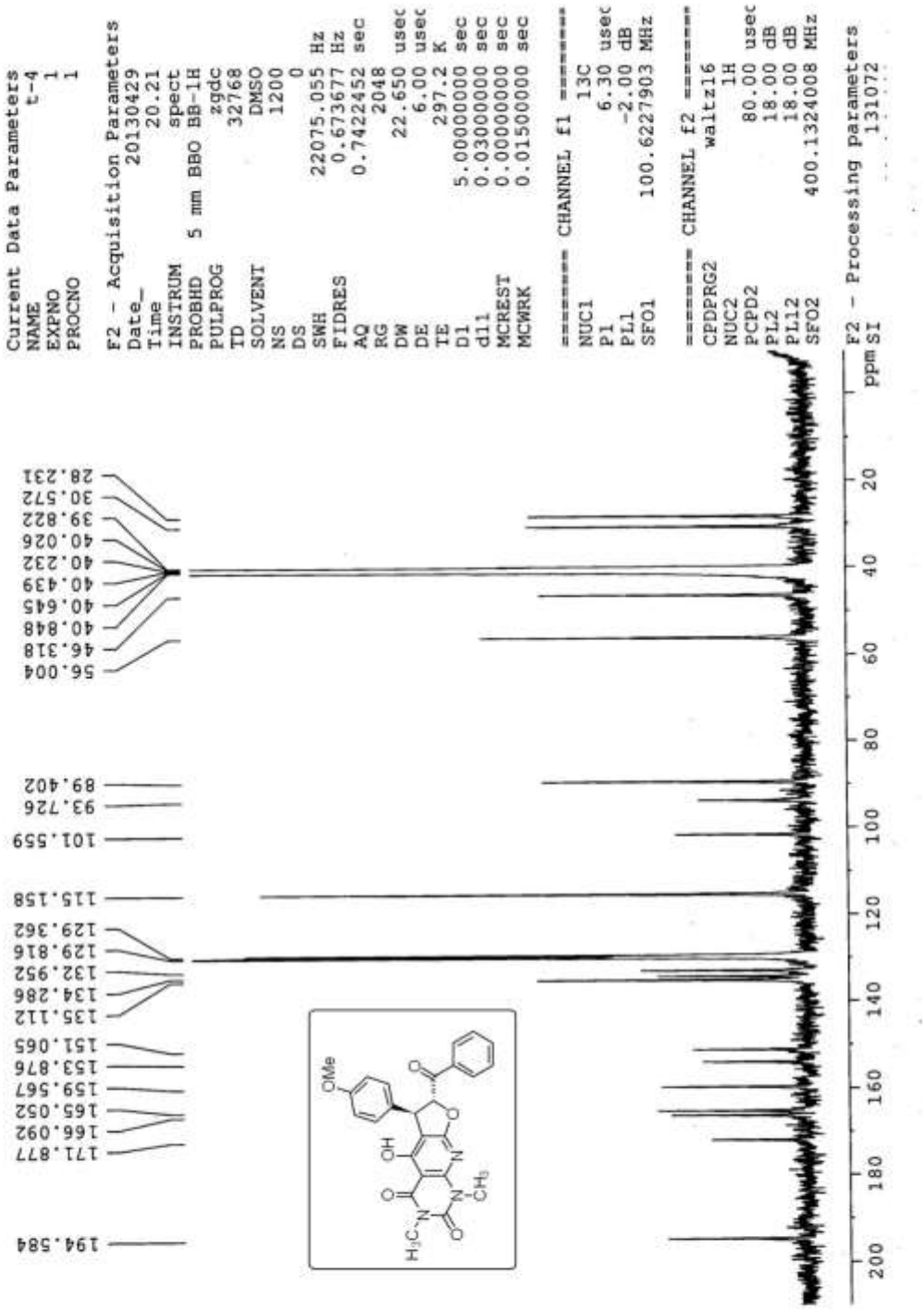
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PROCNO 1
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PULPROG zgdc
TD 32768
SOLVENT DMSO
NS 1200
DS 0
SWH 22075.055 Hz
FIDRES 0.673677 Hz
AQ 0.7422452 sec
RG 2048
DW 22.650 usec
DE 6.00 usec
TE 297.2 K
D1 5.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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P1 6.30 usec
PL1 -2.00 dB
SFO1 100.6227903 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 18.00 dB
PL12 18.00 dB
SFO2 400.1324008 MHz
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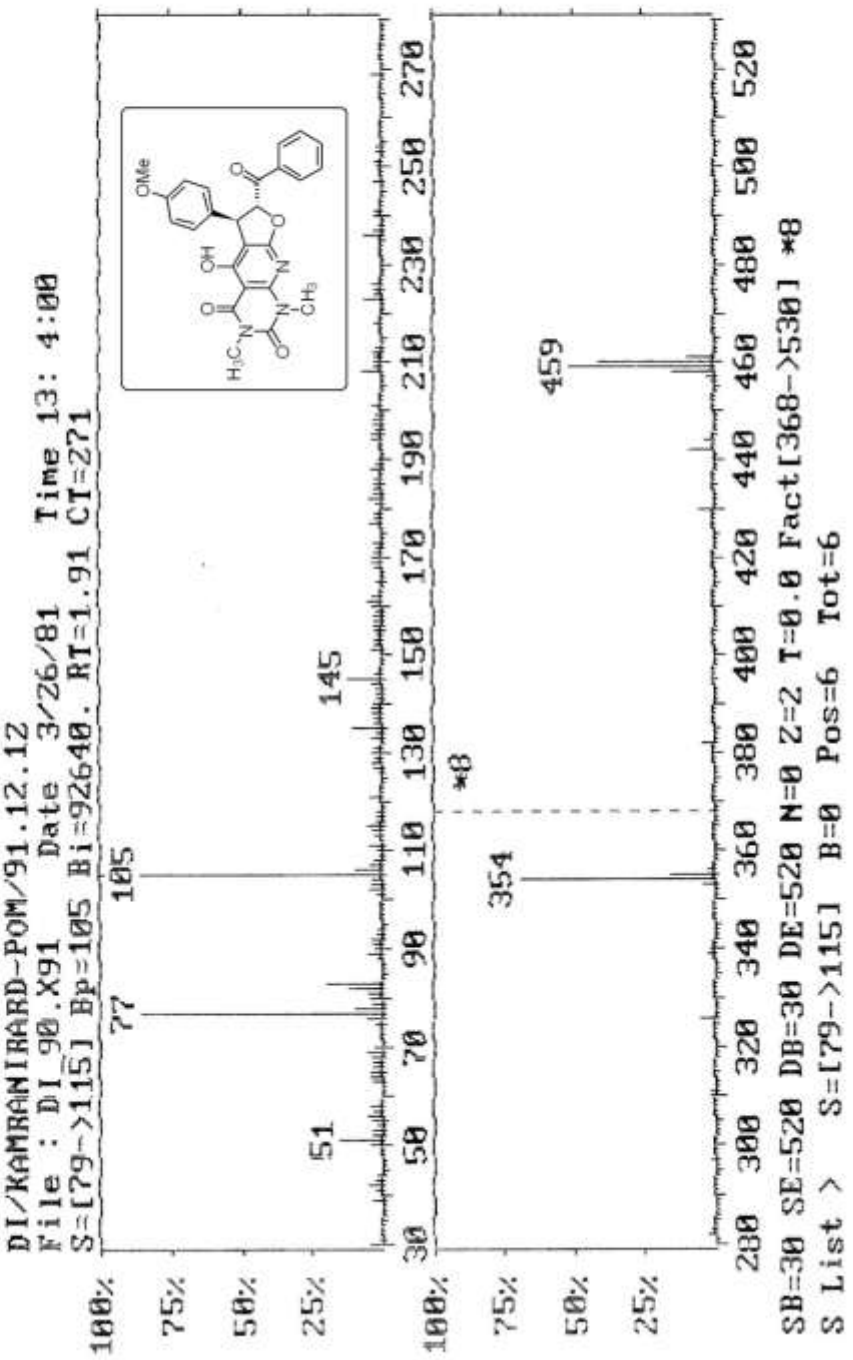
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50%
25%
77
105
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145
354
459
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OH
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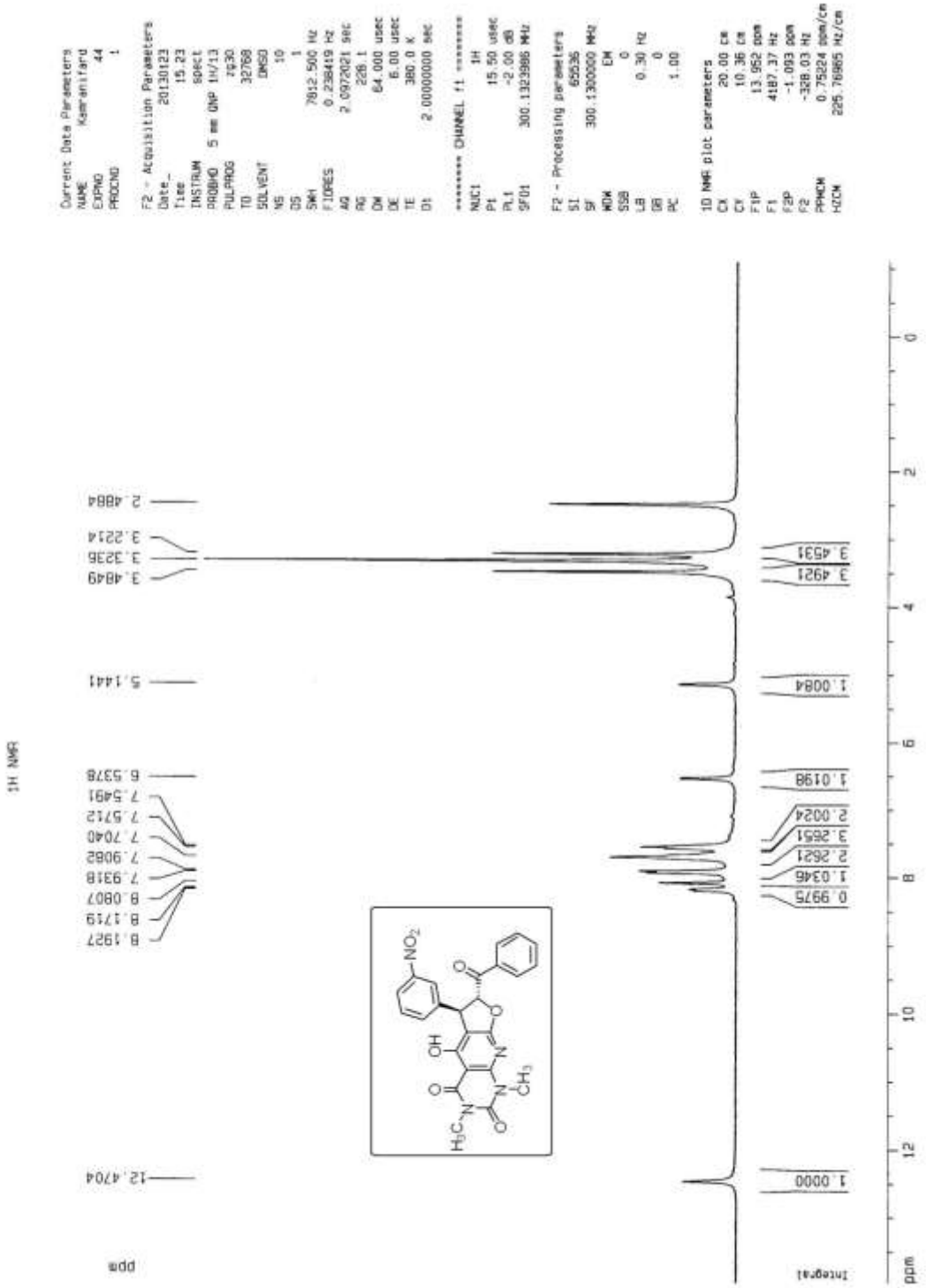
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SOLVENT DMSO
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SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
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P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
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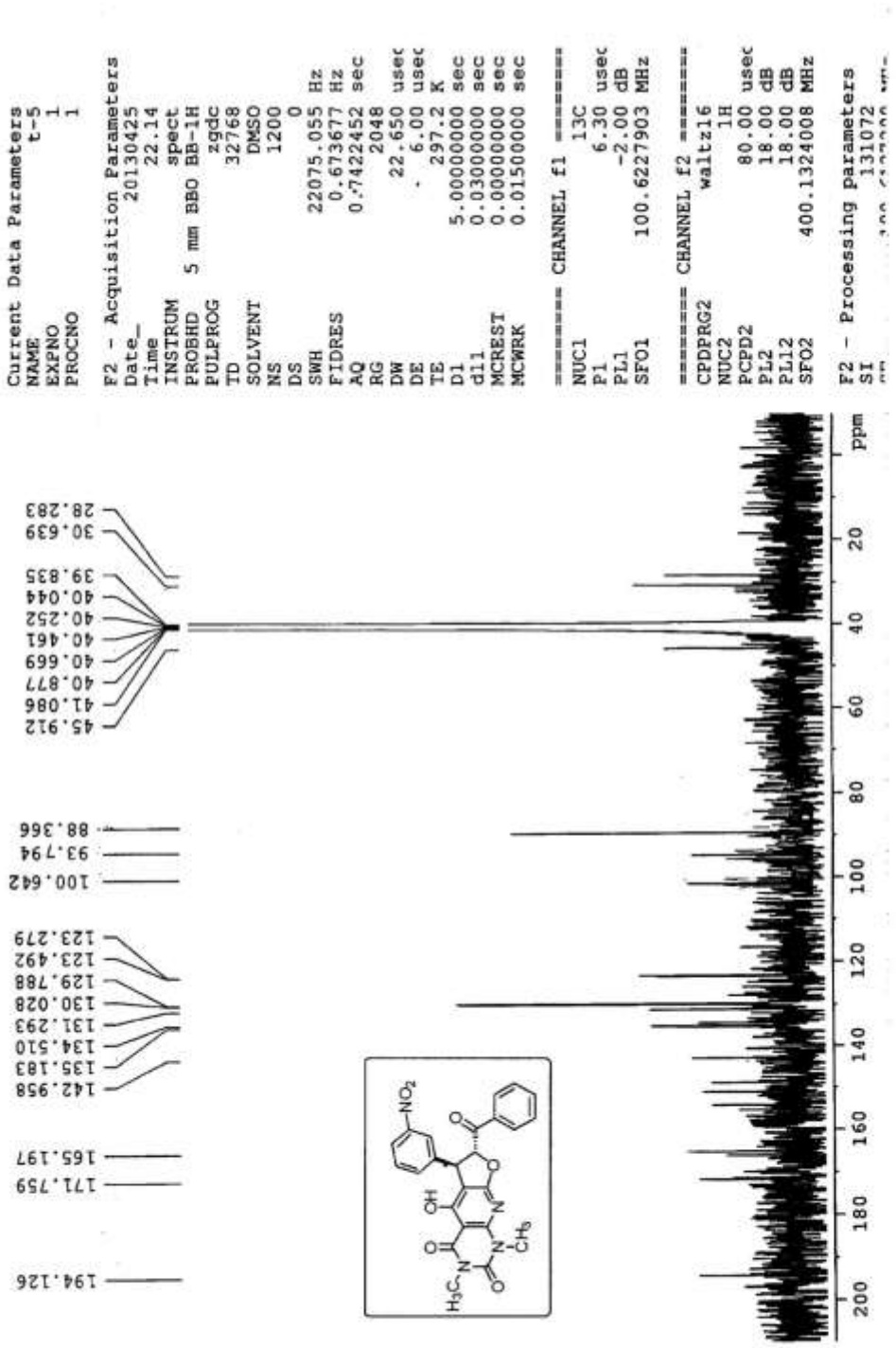
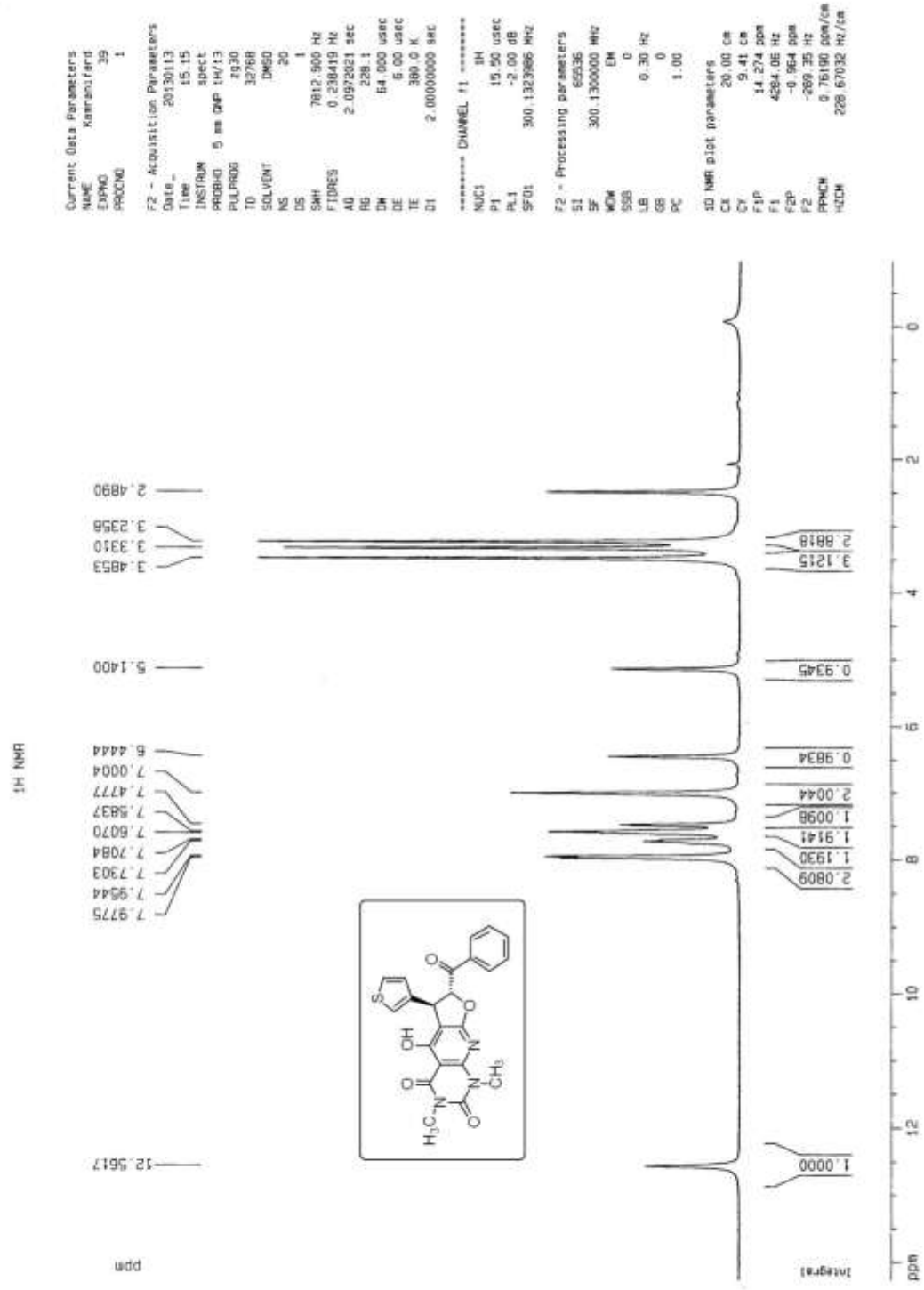
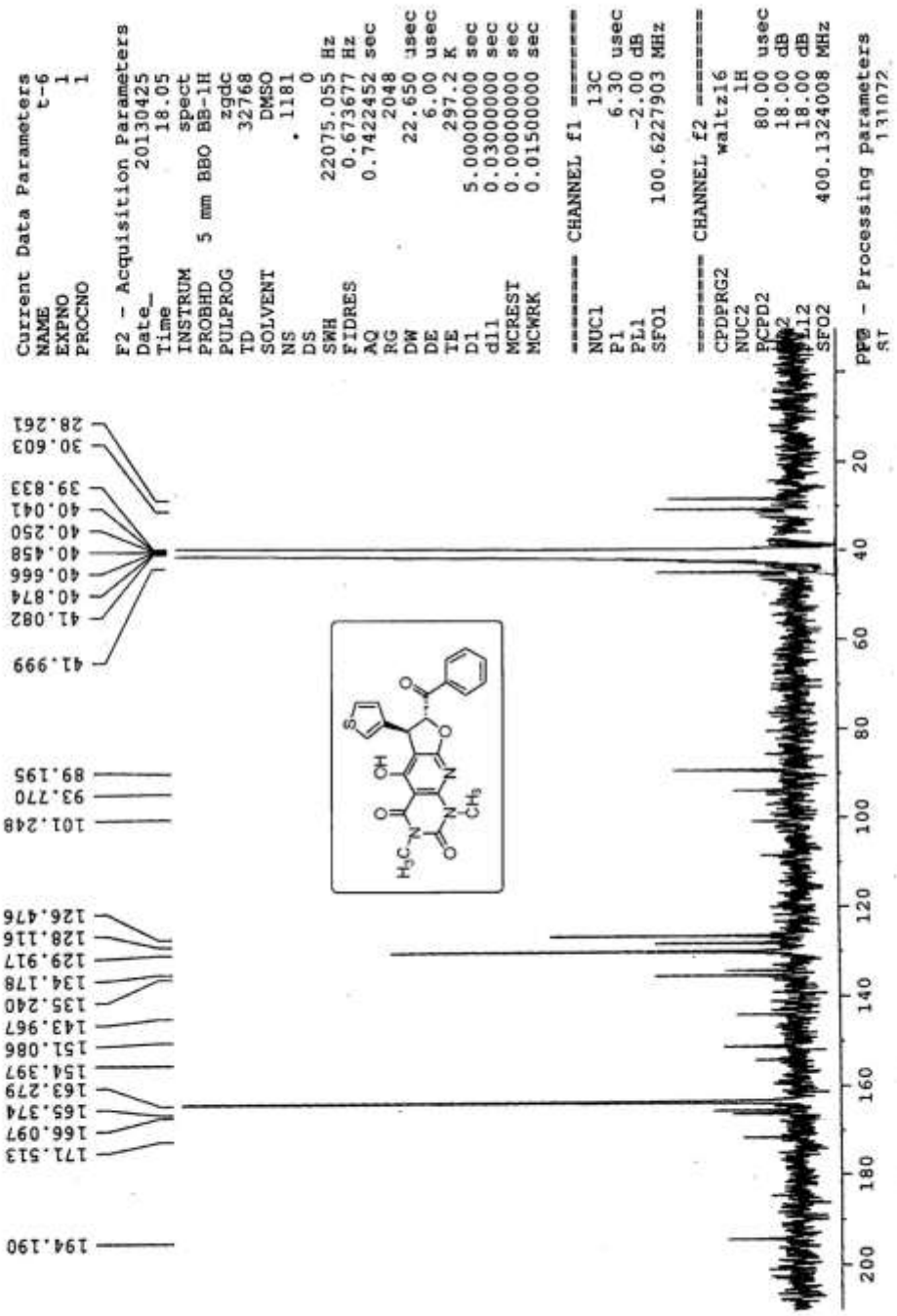
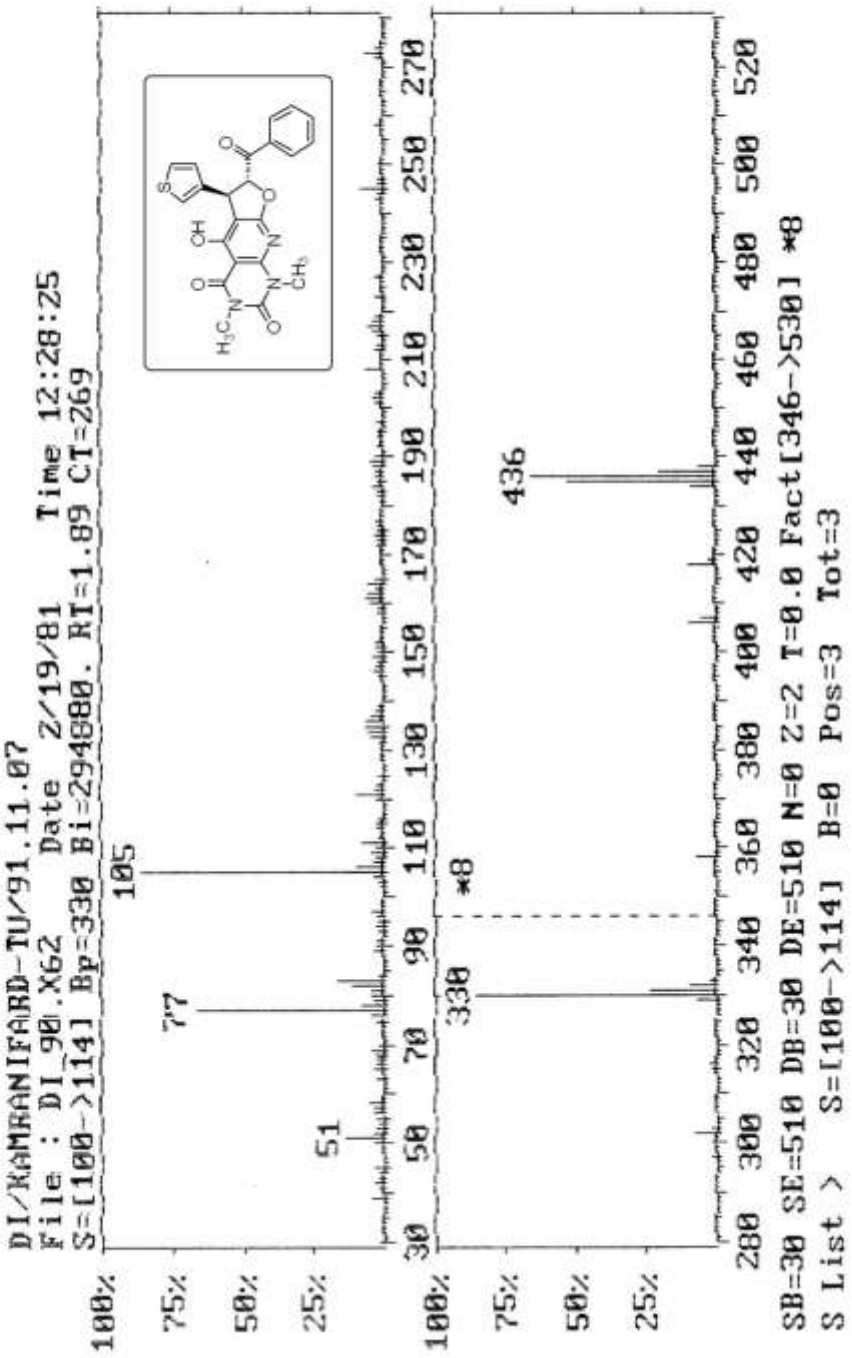
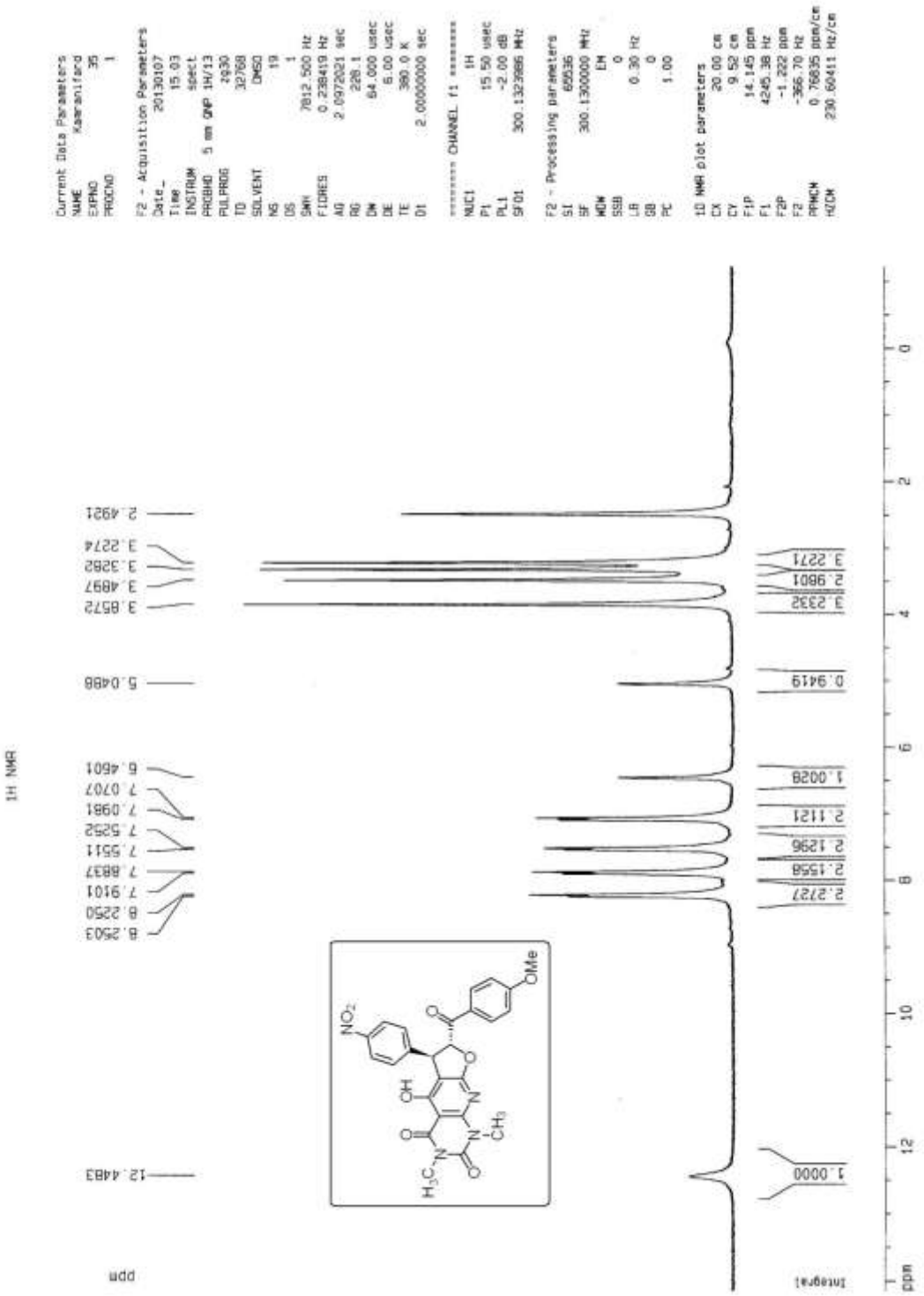
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.36 cm
F1P 13.952 ppm
F1 4187.37 Hz
F2P -1.093 ppm
F2 -328.03 Hz
PPMCM 0.75224 ppm/cm
HZCM 225.76865 Hz/cm
1H NMR
12.4704
8.1927
8.1719
8.0807
7.9318
7.9082
7.7040
7.5712
7.5491
6.5378
5.1441
3.4849
3.3236
3.2214
2.4884
1.0000
0.9975
1.0346
2.2621
3.2651
2.0024
1.0198
1.0084
3.4921
3.4531
Integral
ppm
0
2
4
6
8
10
12

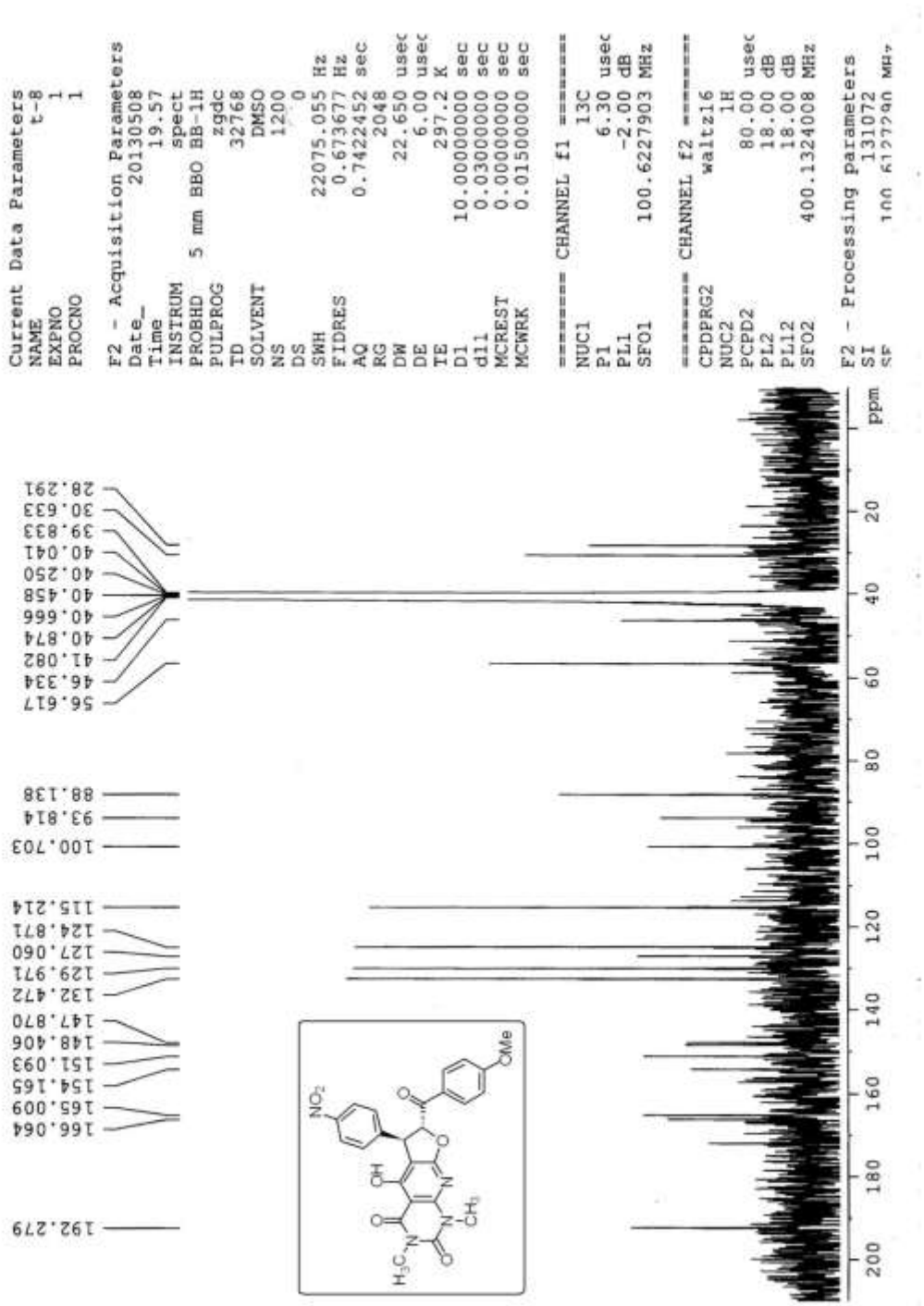


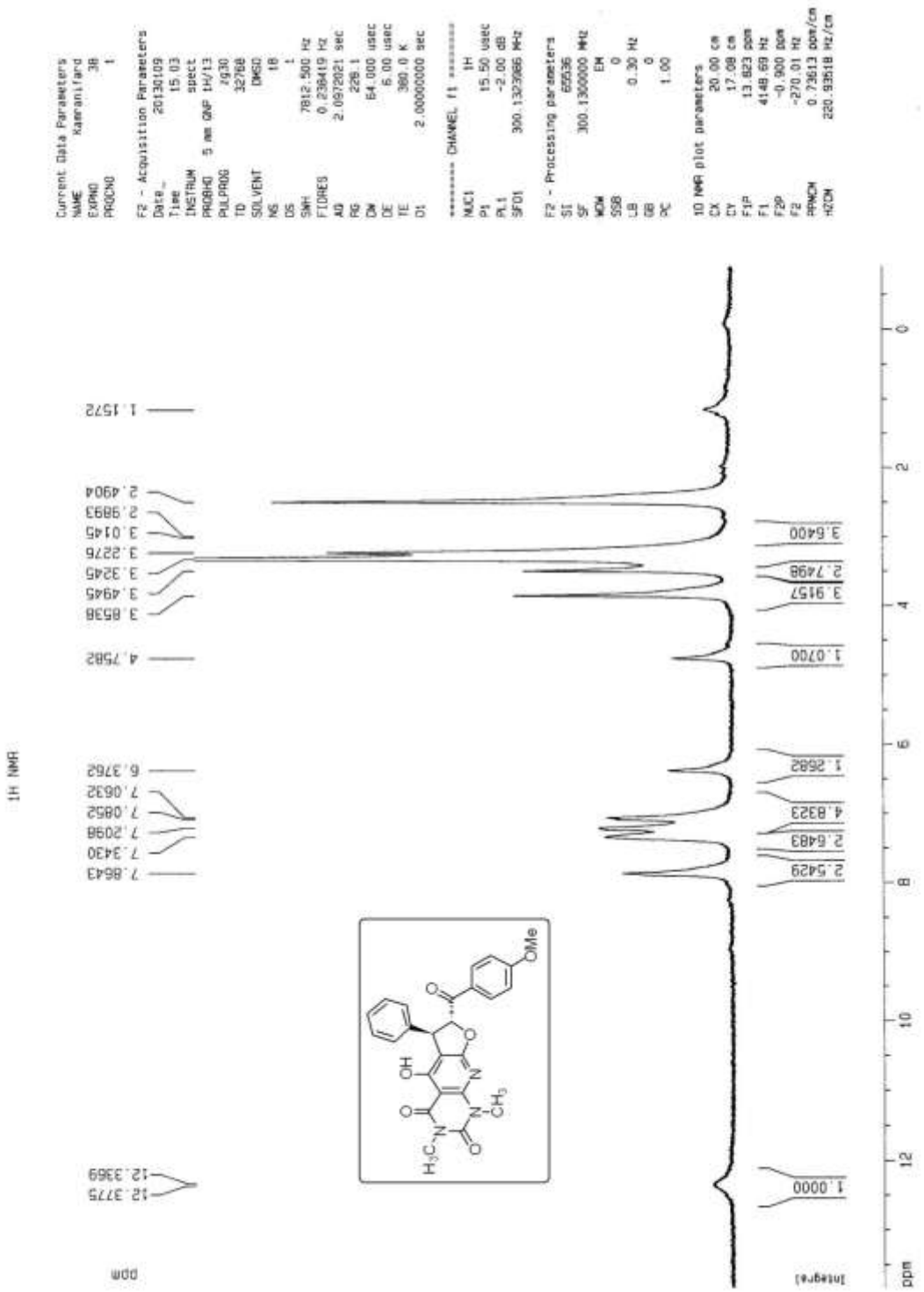

Current Data Parameters
NAME Kammanifard
EXPNO 39
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130113
Time 15.15
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 20
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 9.41 cm
F1P 14.274 ppm
F1 4284.06 Hz
F2P -0.964 ppm
F2 -289.35 Hz
PPMCM 0.76190 ppm/cm
HZCM 228.67032 Hz/cm
2.4890
3.2358
3.3310
3.4853
5.1400
6.4444
7.0004
7.4777
7.5837
7.6070
7.7084
7.7303
7.9544
7.9775
12.5617
2.8818
3.1215
0.9345
0.9834
2.0044
1.0098
1.9141
1.1930
2.0809
1.0000
1H NMR
ppm
Integral
ppm

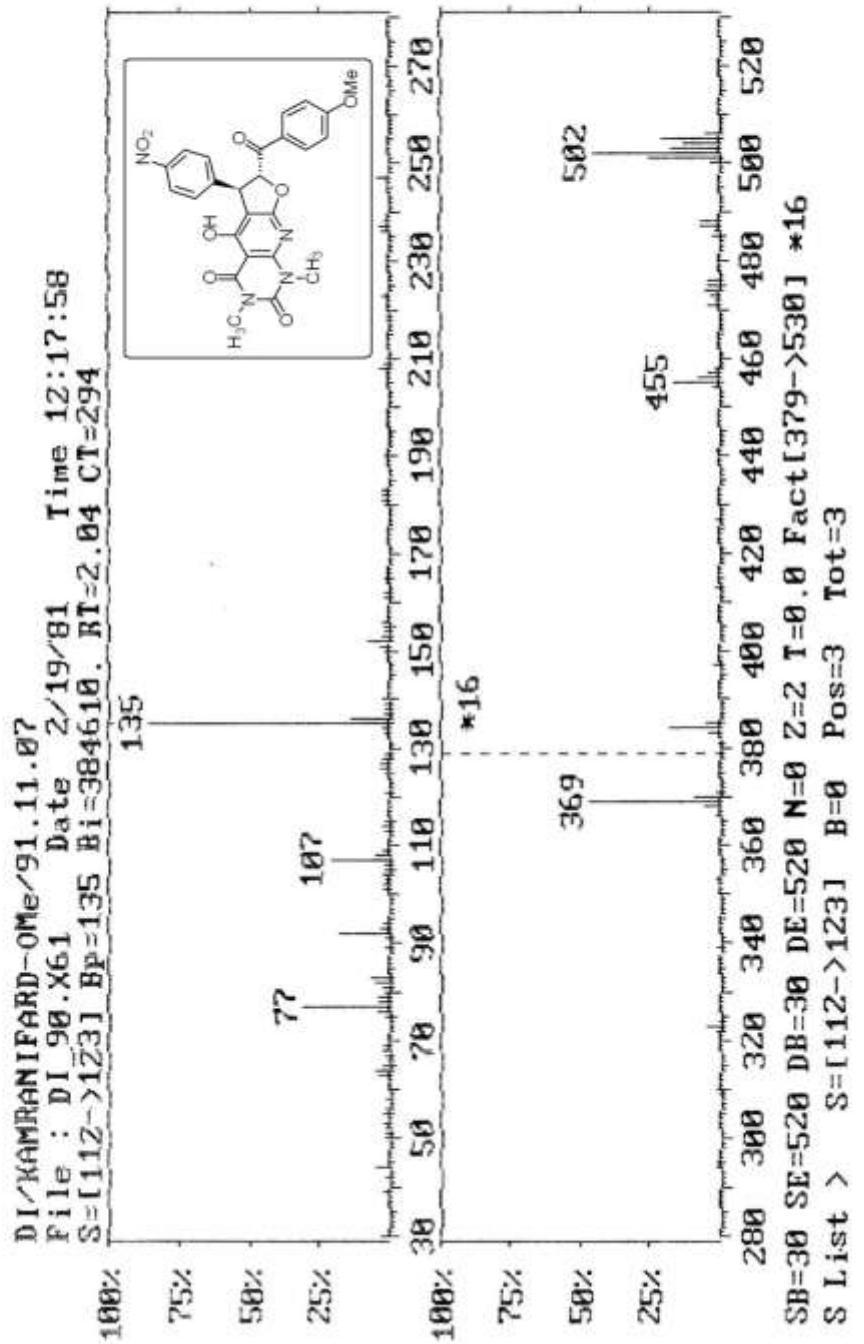

Current Data Parameters
NAME t-6
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130425
Time 18.05
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 32768
SOLVENT DMSO
NS 1181
DS 0
SWH 22075.055 Hz
FIDRES 0.673677 Hz
AQ 0.7422452 sec
RG 2048
DW 22.650 usec
DE 6.00 usec
TE 297.2 K
D1 5.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.30 usec
PL1 -2.00 dB
SFO1 100.6227903 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1324008 MHz
F2 - Processing parameters
SI 131072
28.261
30.603
39.833
40.041
40.250
40.458
40.666
40.874
41.082
41.999
89.195
93.770
101.248
126.476
128.116
129.917
134.178
135.240
143.967
151.086
154.397
163.279
165.374
166.097
171.513
194.190
OH
O
N
CH3
H3C
S
200
180
160
140
120
100
80
60
40
20

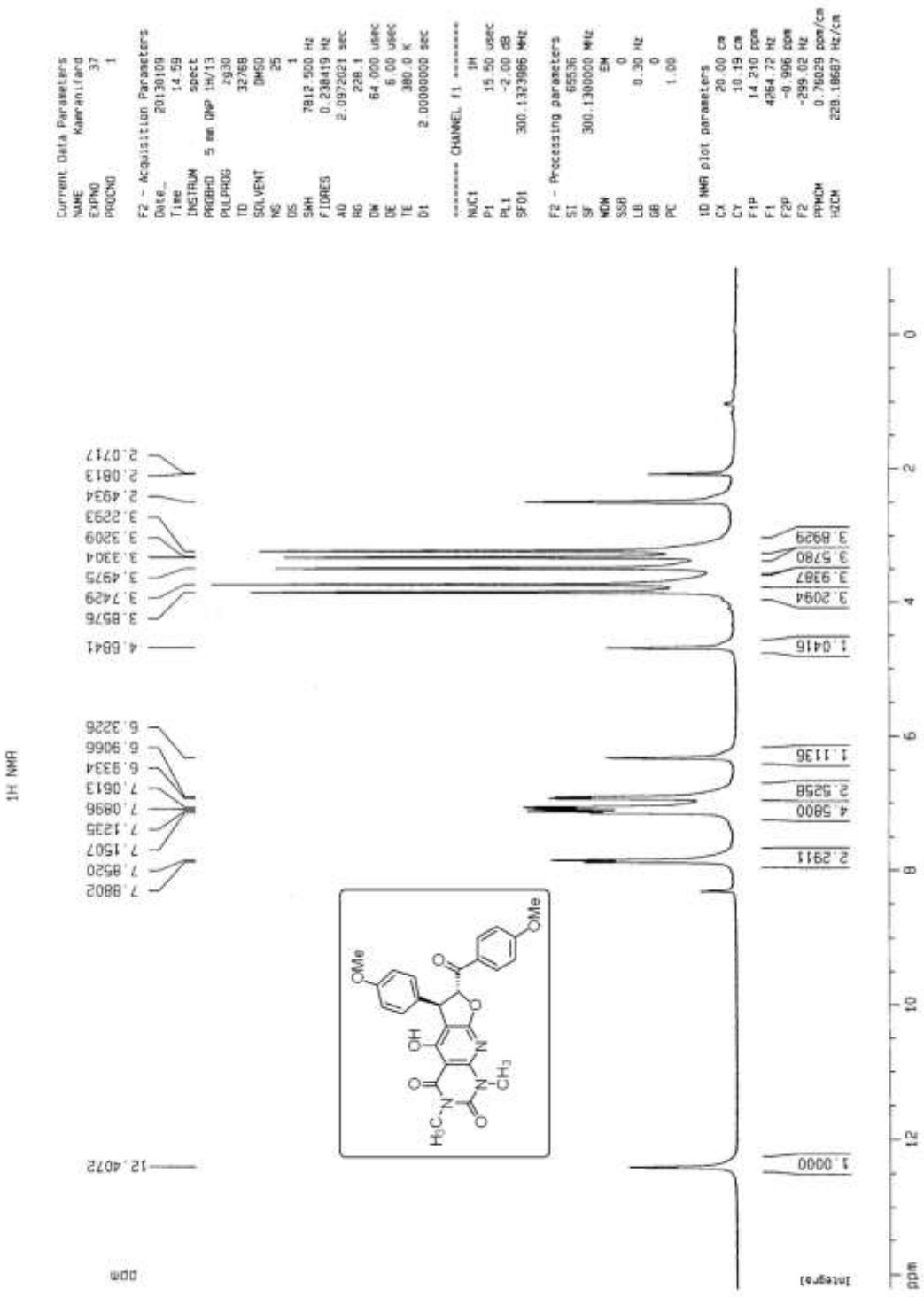

DI/RAMANIFARD-TU/91.11.07
File : DI_90.X62 Date 2/19/81 Time 12:28:25
S=[100->114] Bp=330 Bi=294880. RT=1.89 CT=269
105
77
51
330
*8
436
100%
75%
50%
25%
30 50 70 90 110 130 150 170 190 210 230 250 270
280 300 320 340 360 380 400 420 440 460 480 500 520
SB=30 SE=510 DB=30 DE=510 N=0 Z=2 T=0.0 Fact[346->530] *8
S List > S=[100->114] B=0 Pos=3 Tot=3

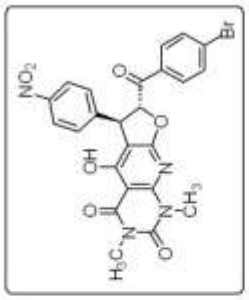

1H NMR
ppm
Integral
12.4483
8.2503
8.2250
7.9101
7.8837
7.5511
7.5252
7.0981
7.0707
6.4601
5.0488
3.8572
3.4897
3.3282
3.2274
2.4921
1.0000
2.2727
2.1558
2.1296
2.1121
1.0028
0.9419
3.2332
2.9801
3.2271
OMe
NO2
OH
CH3

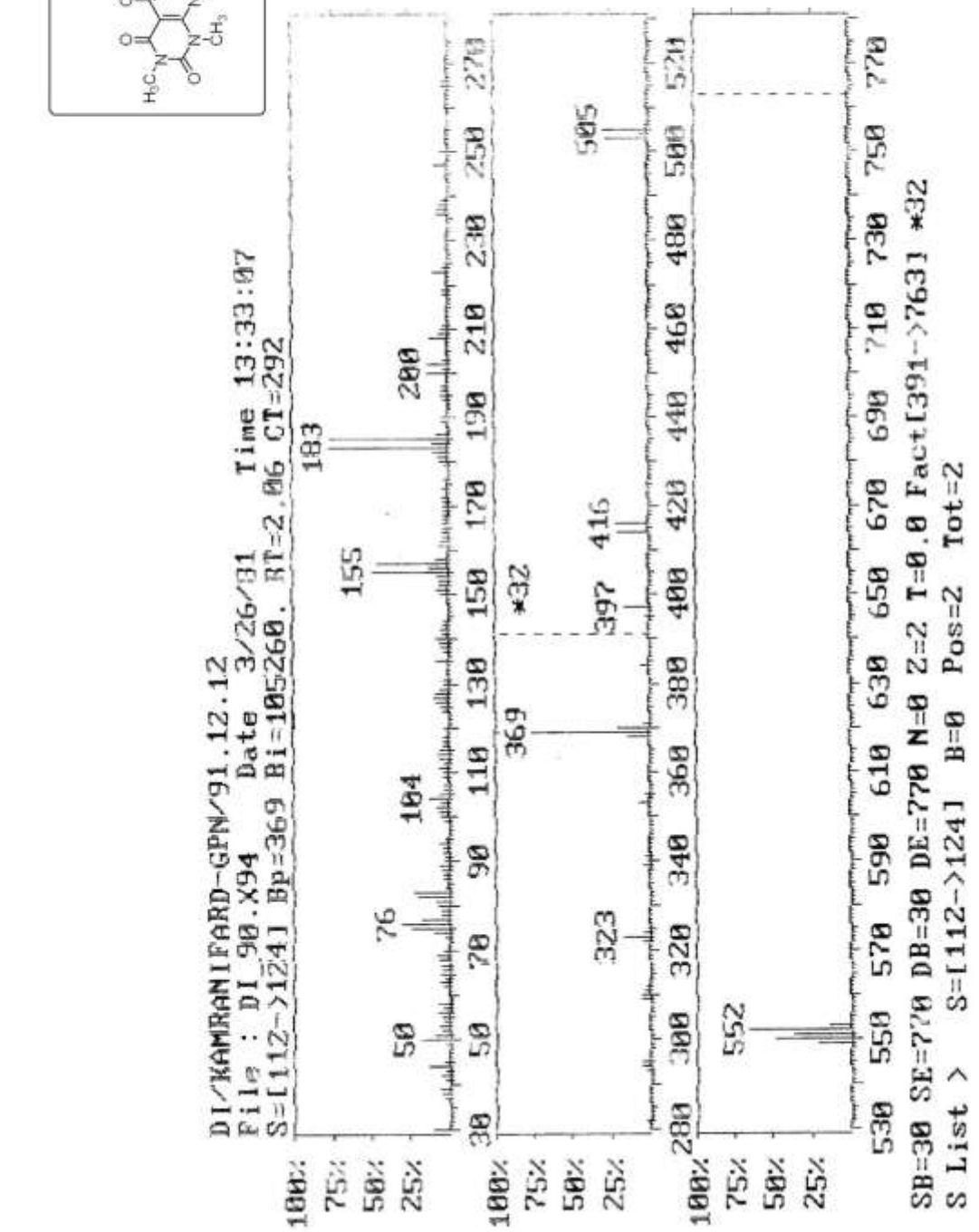

Current Data Parameters
NAME t-8
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130508
Time 19.57
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 32768
SOLVENT DMSO
NS 1200
DS 0
SWH 22075.055 Hz
FIDRES 0.673677 Hz
AQ 0.7422452 sec
RG 2048
DW 22.650 usec
DE 6.00 usec
TE 297.2 K
D1 10.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.30 usec
PL1 -2.00 dB
SFO1 100.6227903 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 18.00 dB
PL12 18.00 dB
SFO2 400.1324008 MHz
F2 - Processing parameters
SI 131072
SF 100.6127940 MHz
28.291
30.633
39.833
40.041
40.250
40.458
40.666
40.874
41.082
46.334
56.617
88.138
93.814
100.703
115.214
124.871
127.060
129.971
132.472
147.870
148.406
151.093
154.165
165.009
166.064
192.279
ppm
20
40
60
80
100
120
140
160
180
200
OMe
NO2
OH
N
O
CH3
H3C

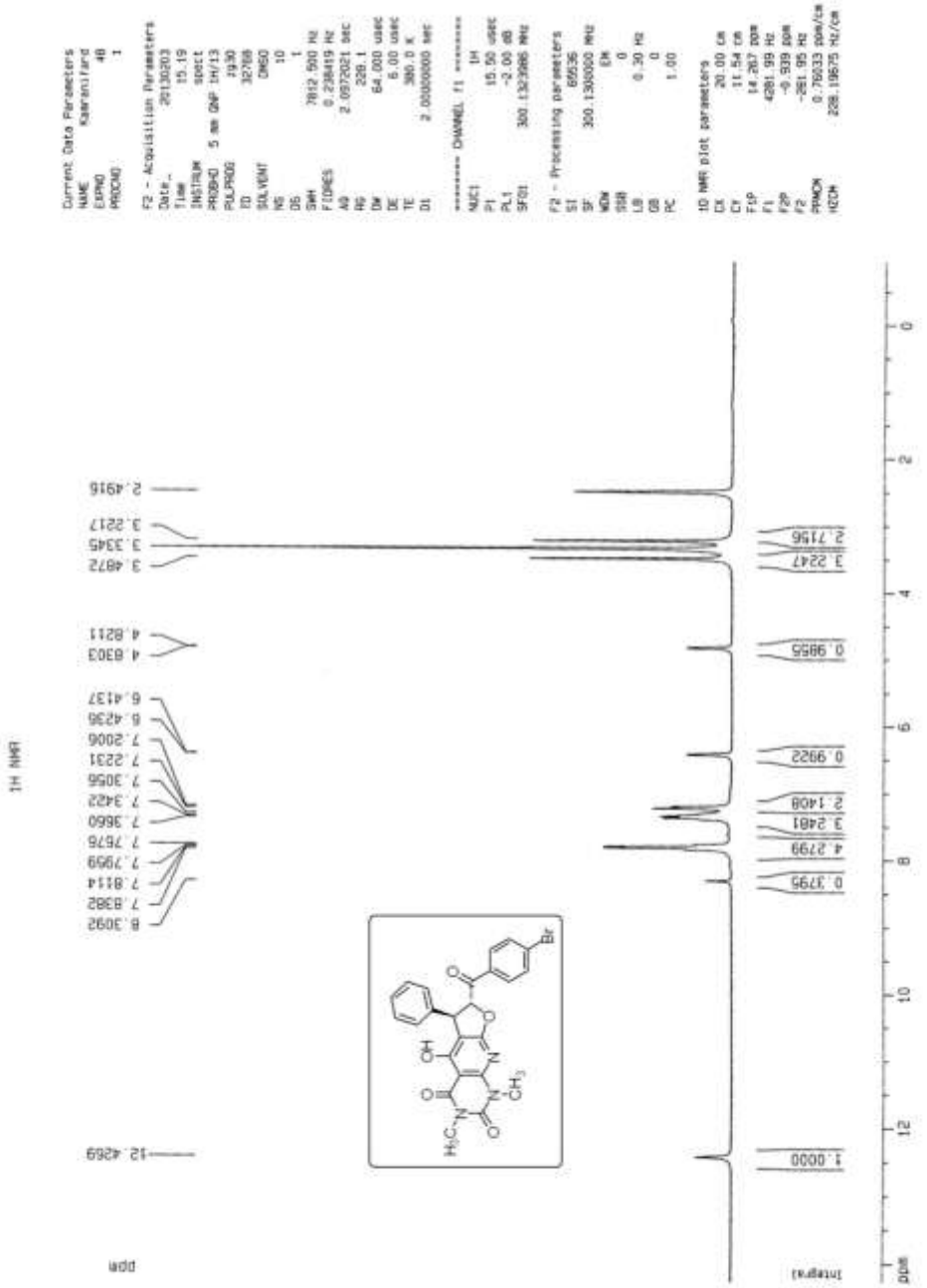

Current Data Parameters
NAME Kamranifard
EXPNO 38
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130109
Time 15.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1329866 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 17.08 cm
F1P 13.823 ppm
F1 4148.69 Hz
F2P -0.900 ppm
F2 -270.01 Hz
PPMCM 0.73613 ppm/cm
HZCM 220.93518 Hz/cm
1H NMR
12.3775
12.3369
7.8643
7.7430
7.2098
7.0852
7.0632
6.3762
4.7582
3.8538
3.4945
3.3245
3.2276
3.0145
2.9893
2.4904
1.1572
1.0000
1.2682
4.8323
2.6483
2.5429
2.7498
3.9157
3.6400
Integral
ppm
OMe
OH
N
O
H_3C
CH_3

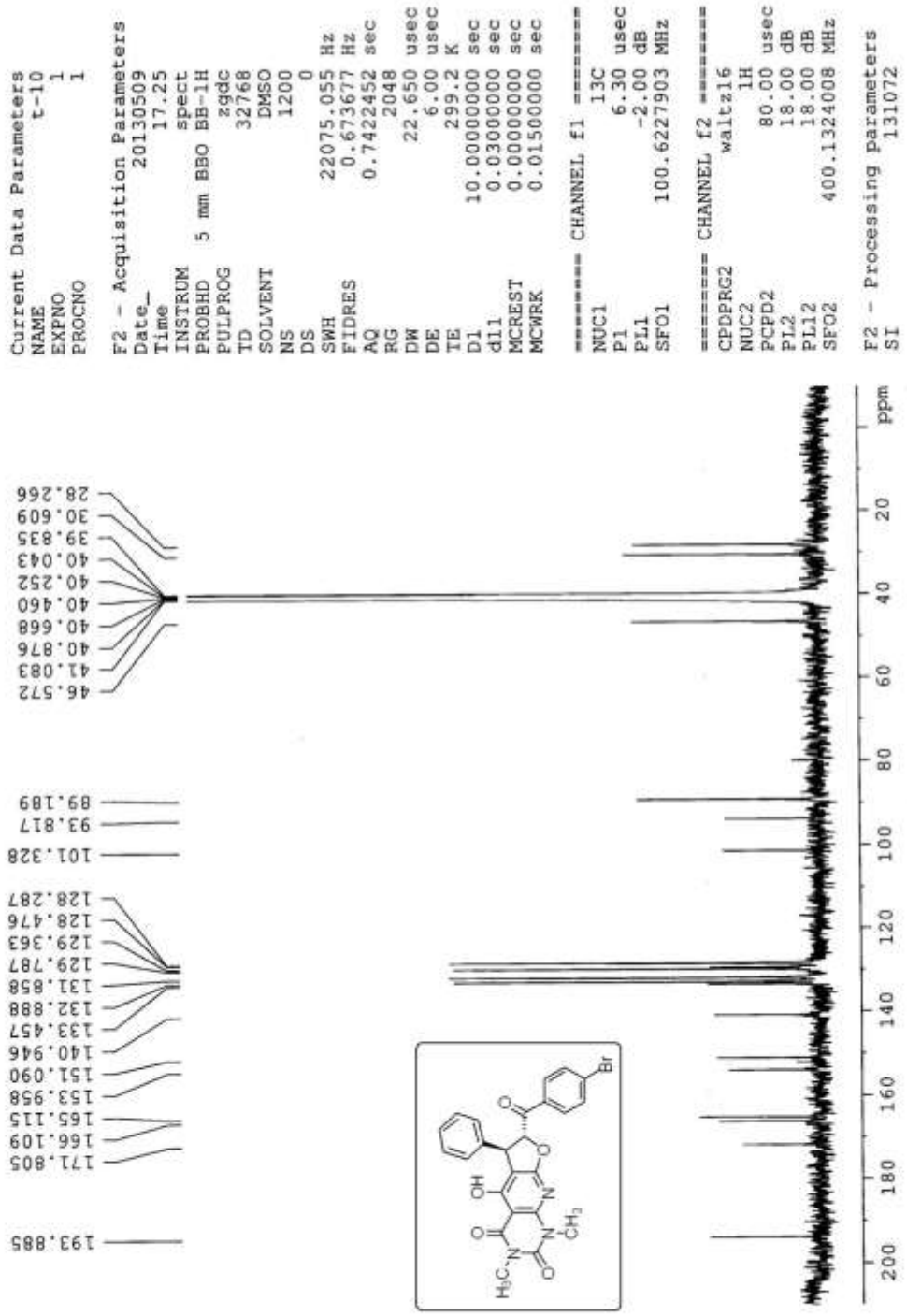


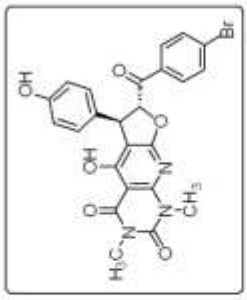

Current Data Parameters
NAME Kamranifard
EXPNO 37
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130109
Time 14.59
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 25
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.19 cm
F1P 14.210 ppm
F1 4264.72 Hz
F2P -0.996 ppm
F2 -299.02 Hz
PPMCM 0.76029 ppm/cm
HZCM 228.18687 Hz/cm
1H NMR
ppm
Integral
12.4072
7.8802
7.8520
7.1507
7.1235
7.0896
7.0613
6.9334
6.9066
6.8226
4.6841
3.8576
3.7429
3.4975
3.3304
3.3209
3.2293
3.0813
3.0717
1.0000
2.2911
4.5800
2.5258
1.1136
3.2094
3.9387
3.5780
3.8929
1.0416
OMe
OMe
OH
N
O
N
CH3
N
H3C
O
O


NO2
Br
O
OH
O
N
O
N
CH3
H3C
N
O

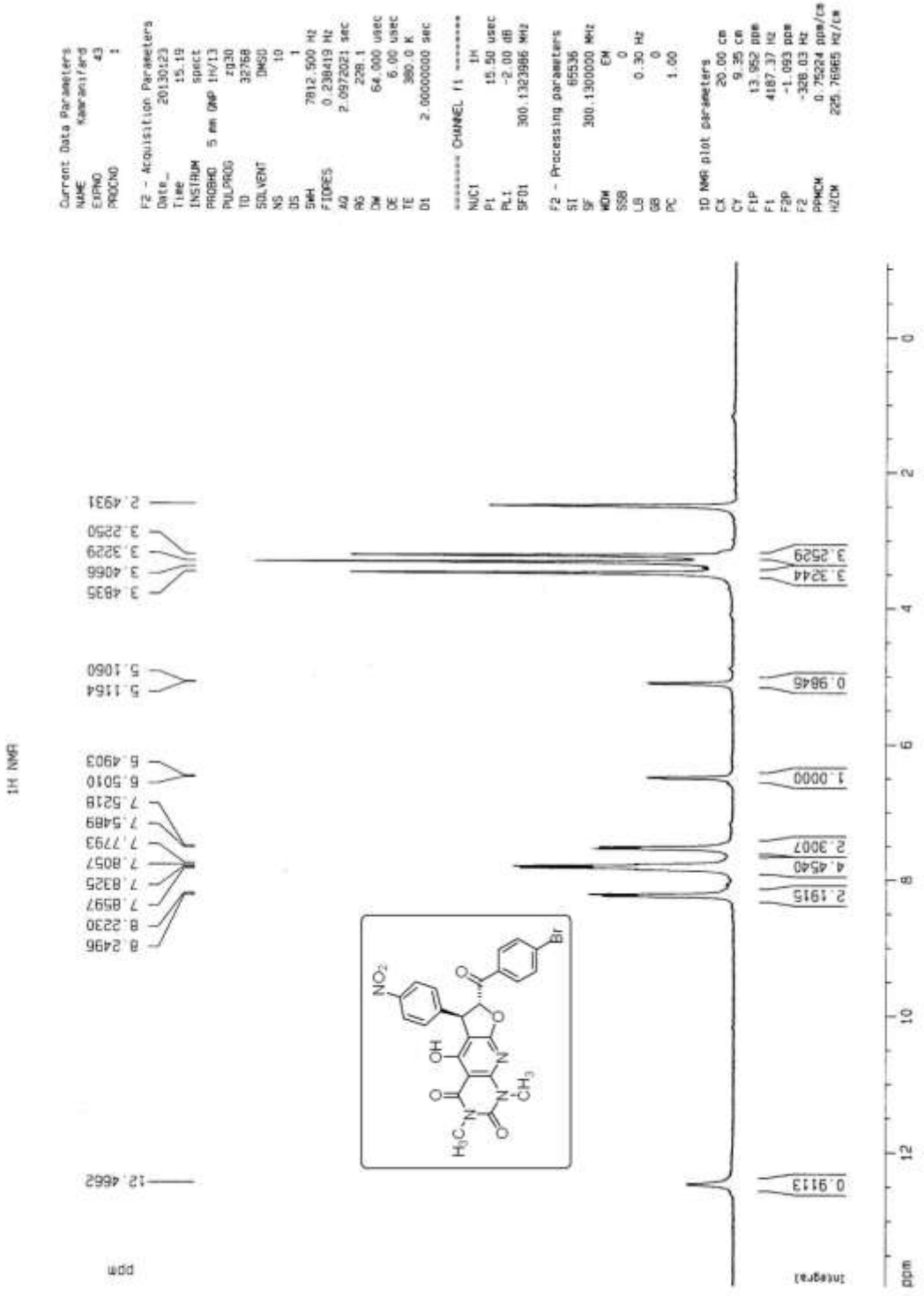

DI/KAMRANIFARD-GPN/91.12.12
File : DI_90.X94 Date 3/26/31 Time 13:33:07
S=[112->124] Bp=369 Bi=105260. RT=2.06 CT=292
100%
75%
50%
25%
30 50 70 90 110 130 150 170 190 210 230 250 270
183
155
200
76
104
50
369
*32
397
415
505
323
280 300 320 340 360 380 400 420 440 460 480 500 520
552
530 550 570 590 610 630 650 670 690 710 730 750 770
SB=30 SE=770 DB=30 DE=770 N=0 Z=2 T=0.0 Fact[391->763] *32
S List > S=[112->124] B=0 Pos=2 Tot=2

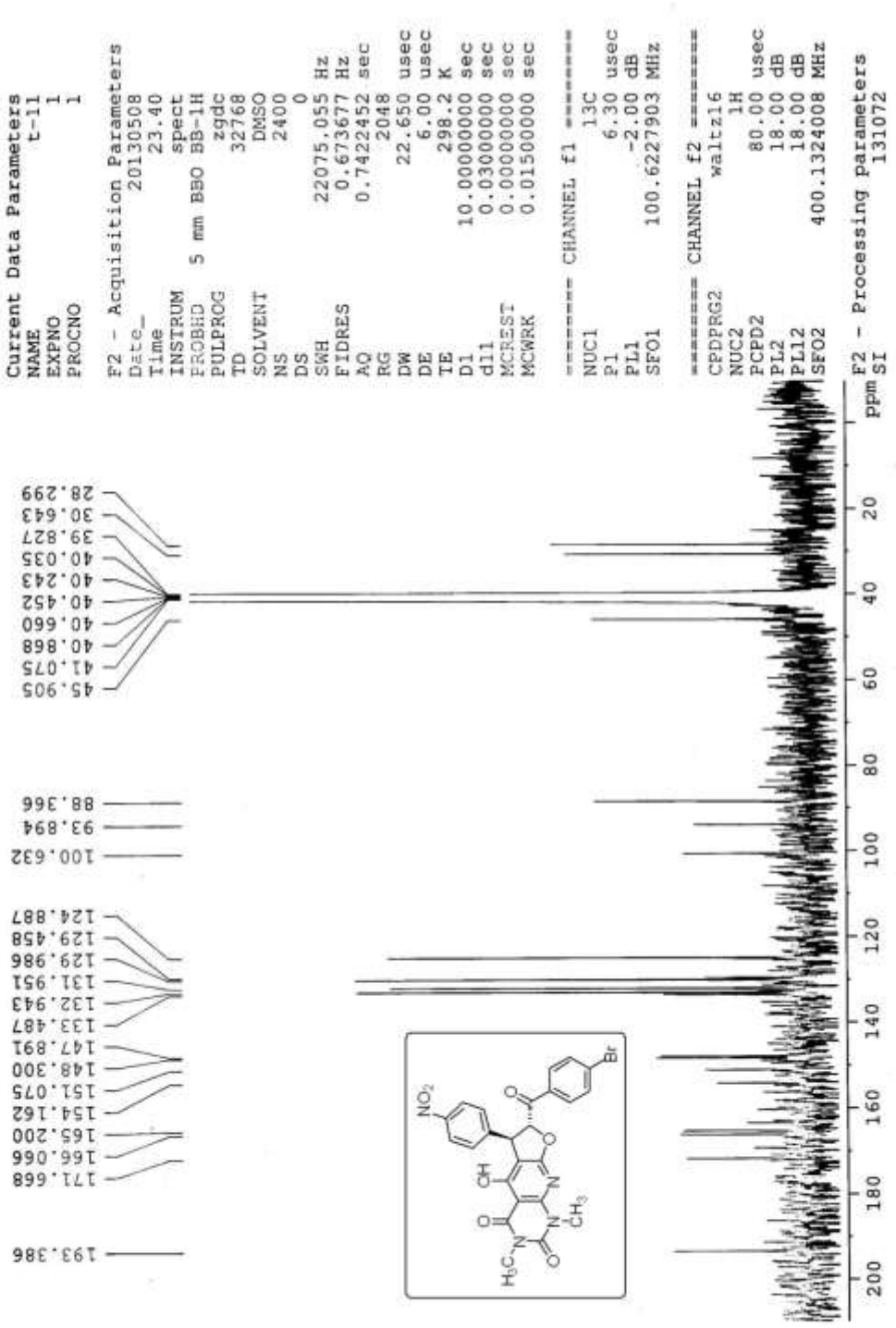

1H NMR
ppm
Integral
2.4916
3.2217
3.3345
3.4872
4.8211
4.8303
6.4137
6.4236
7.2006
7.2231
7.3056
7.3422
7.3660
7.7676
7.7959
7.8114
7.8382
8.3092
12.4269
2.7156
3.2247
0.9855
0.9922
2.1408
3.2481
4.2799
0.3795
1.0000
Br
OH
O
N
CH3
H3C

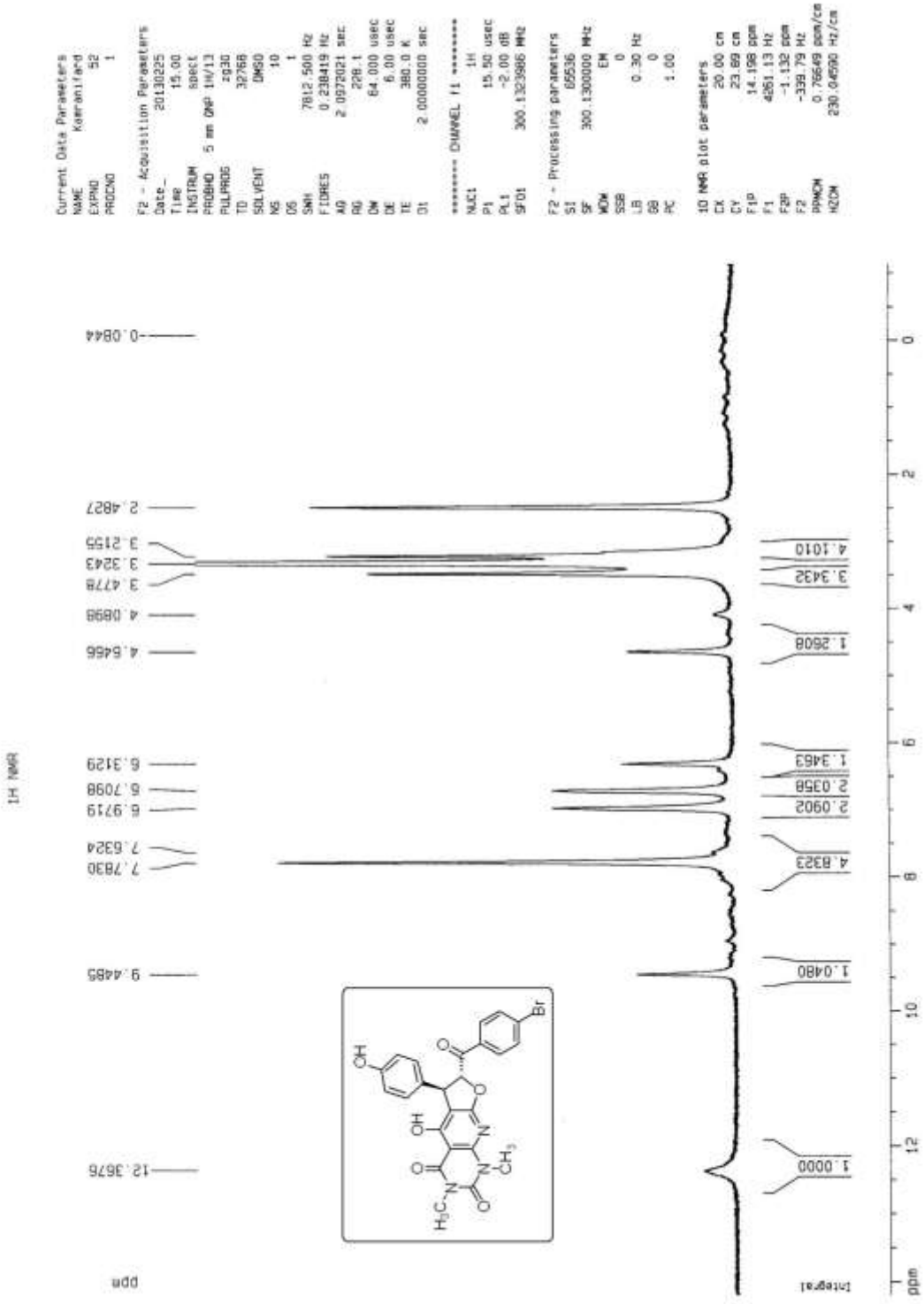

Current Data Parameters
NAME t-10
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130509
Time 17.25
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 32768
SOLVENT DMSO
NS 1200
DS 0
SWH 22075.055 Hz
FIDRES 0.673677 Hz
AQ 0.7422452 sec
RG 2048
DW 22.650 usec
DE 6.00 usec
TE 299.2 K
D1 10.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.30 usec
PL1 -2.00 dB
SFO1 100.6227903 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 18.00 dB
PL12 18.00 dB
SFO2 400.1324008 MHz
F2 - Processing parameters
SI 131072
28.266
30.609
39.835
40.043
40.252
40.460
40.668
40.876
41.083
46.572
89.189
93.817
101.328
128.287
128.476
129.363
129.787
131.858
132.888
133.457
140.946
151.090
153.958
165.115
166.109
171.805
193.885
ppm
200
180
160
140
120
100
80
60
40
20
Br
OH
O
N
CH3
H3C

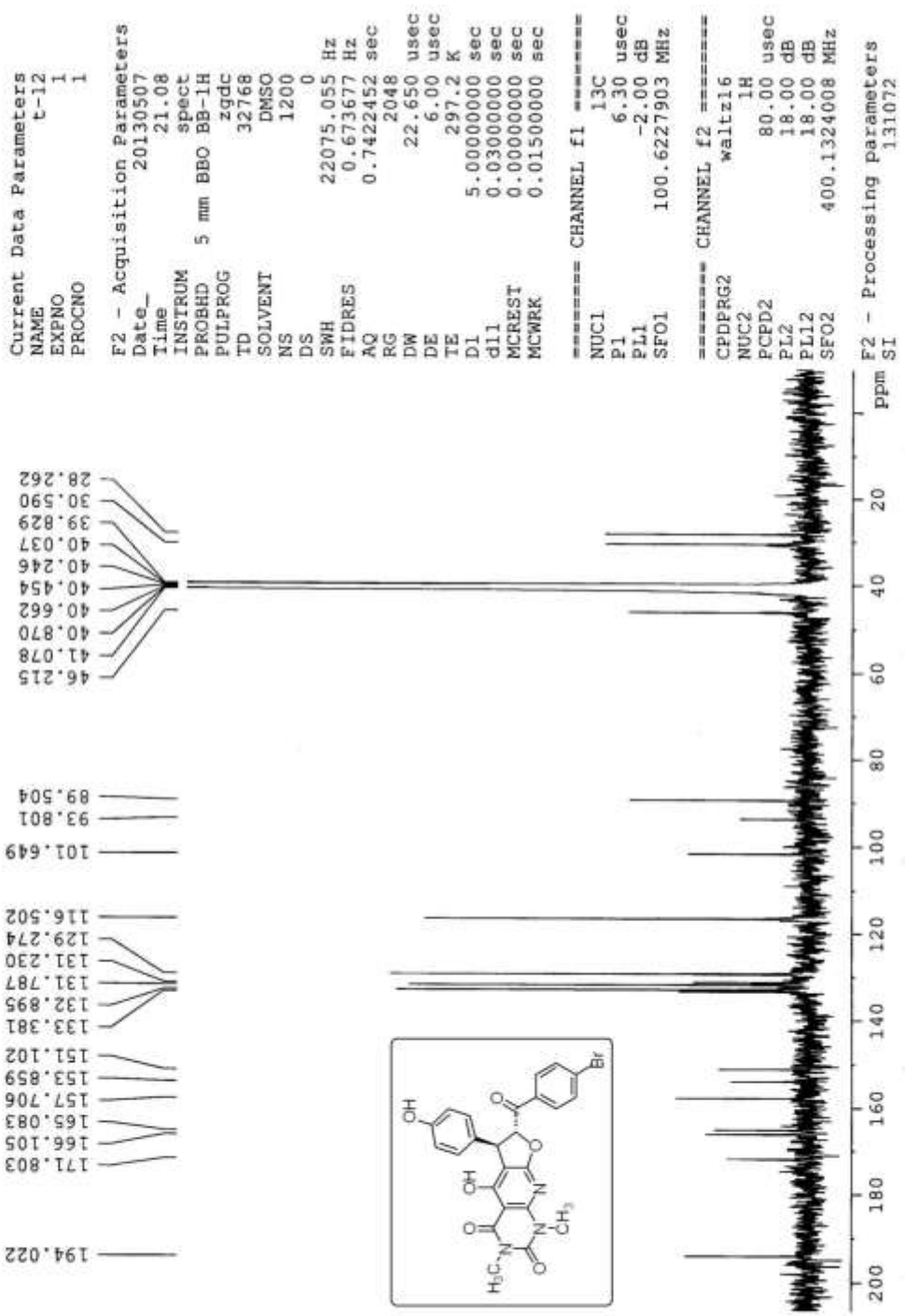

OH
OH
O
O
N
N
O
N
CH3
H3C
O
O
Br

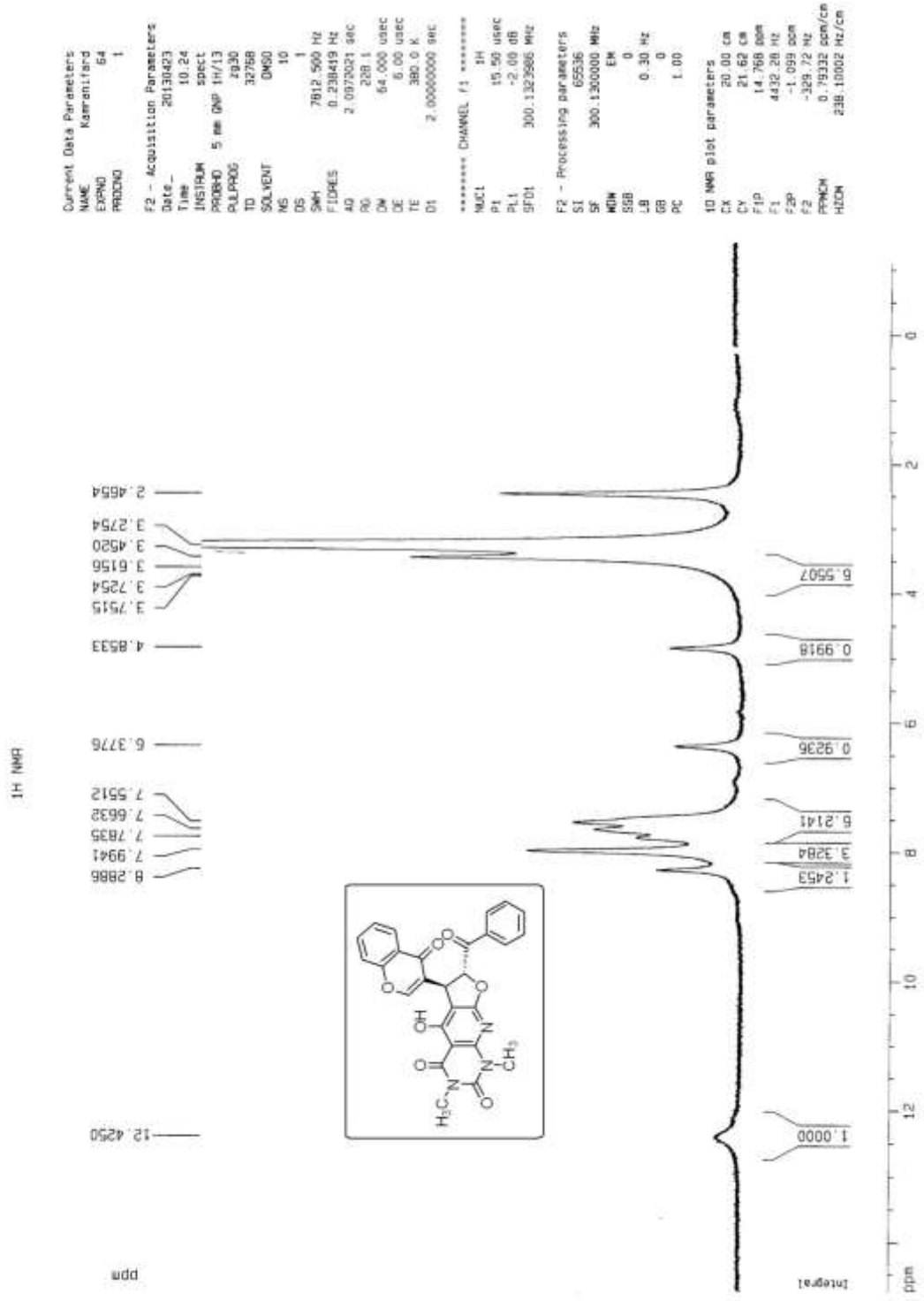
DI/KAMRANIFARD-BPOH/92.01.24
File : DI_91.X10 Date 5/ 7/81 Time 11:10:21
S=[104->114] Bp=340 Bi=59300. RT=1.89 CT=261
100%
75%
50%
25%
0
20
40
60
80
100
120
140
160
180
200
220
240
50
76
131
155
183
340
*128
368
494
250
270
290
310
330
350
370
390
410
430
450
470
490
523
500
520
540
560
580
600
620
640
660
680
700
720
740
SB=30 SE=720 DB=0 DE=720 N=0 Z=2 T=0.0 Fact[357->778]*128
S List > S=[104->114] B=0 Pos=1 Tot=1

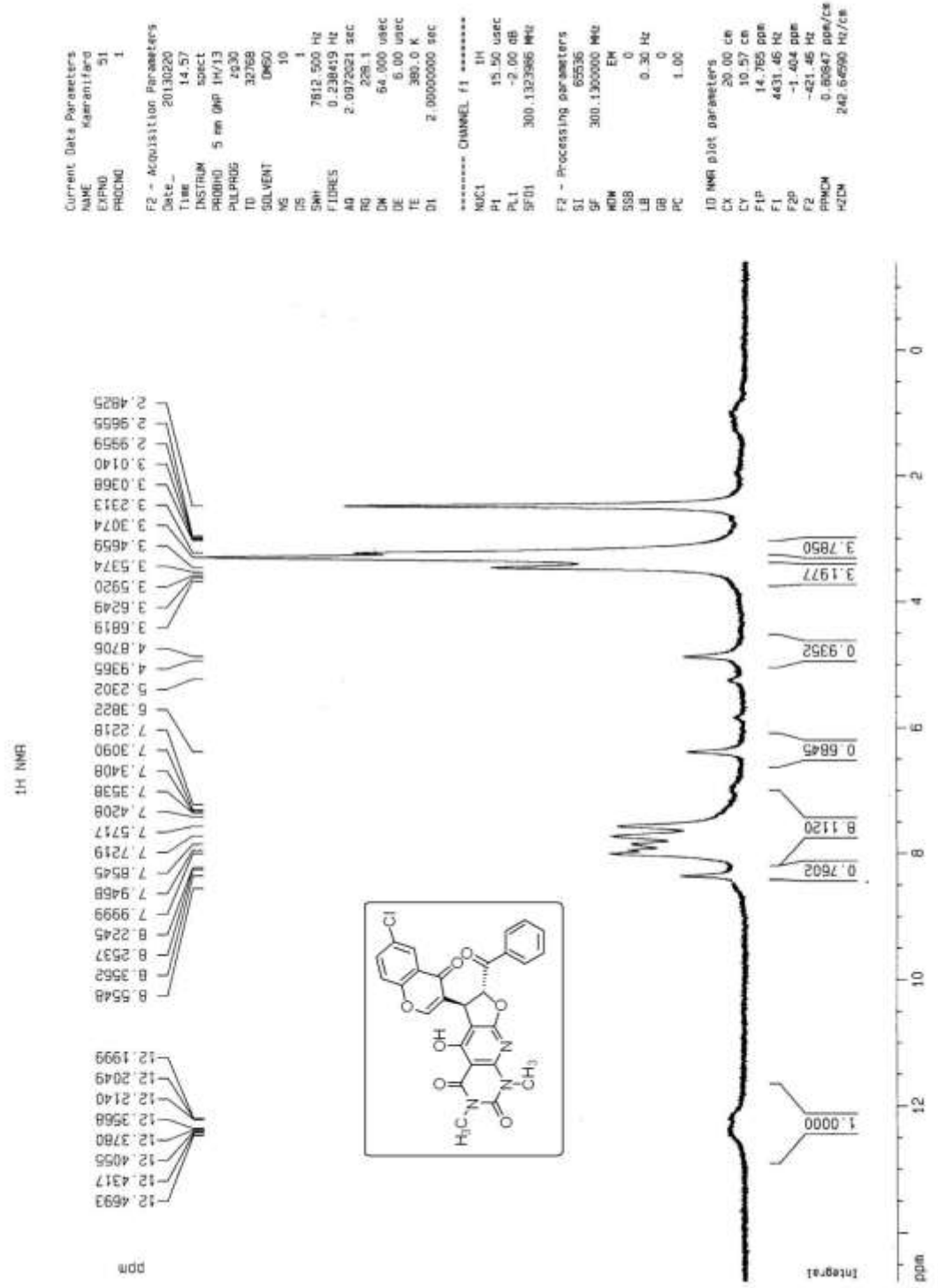

Current Data Parameters
NAME Kamenitzfard
EXPNO 43
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130123
Time 15.19
INSTRUM spect
PROBHD 5 mm DMP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 9.35 cm
F1P 13.952 ppm
F1 4187.37 Hz
F2P -1.093 ppm
F2 -328.03 Hz
PPMCM 0.75224 ppm/cm
HZCM 225.76965 Hz/cm
1H NMR
ppm
integral
ppm
12.4662
8.2496
8.2230
7.8597
7.8325
7.8057
7.7793
7.5489
7.5218
6.5010
6.4903
5.1164
5.1060
3.4835
3.4066
3.3229
3.2250
2.4931
0.9113
2.1915
4.4540
2.3007
1.0000
0.9846
3.2529
3.3244
Br
NO2
OH
O
N
CH3
H3C

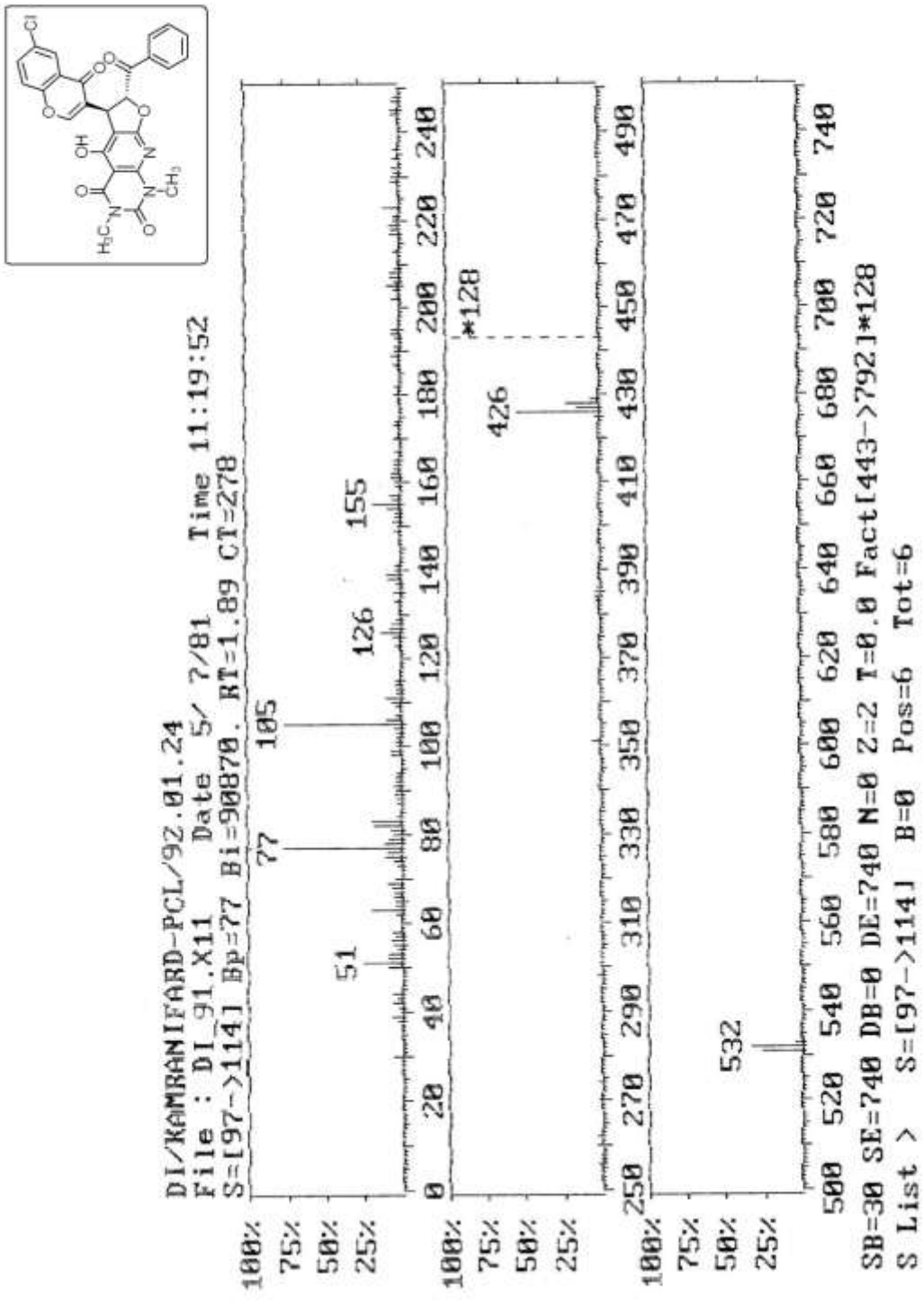

Current Data Parameters
NAME t-11
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130508
Time 23.40
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 32768
SOLVENT DMSO
NS 2400
DS 0
SWH 22075.055 Hz
FIDRES 0.673677 Hz
AQ 0.7422452 sec
RG 2048
DW 22.650 usec
DE 6.00 usec
TE 298.2 K
D1 10.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.30 usec
PL1 -2.00 dB
SFO1 100.6227903 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 18.00 dB
PL12 18.00 dB
SFO2 400.1324008 MHz
F2 - Processing parameters
SI 131072
ppm
28.299
30.643
39.827
40.035
40.243
40.452
40.660
40.868
41.075
45.905
88.366
93.894
100.632
124.887
129.458
129.986
131.951
132.943
133.487
147.891
148.300
151.075
154.162
165.200
166.066
171.668
193.386
Br
NO2
OH
O
N
CH3
H3C

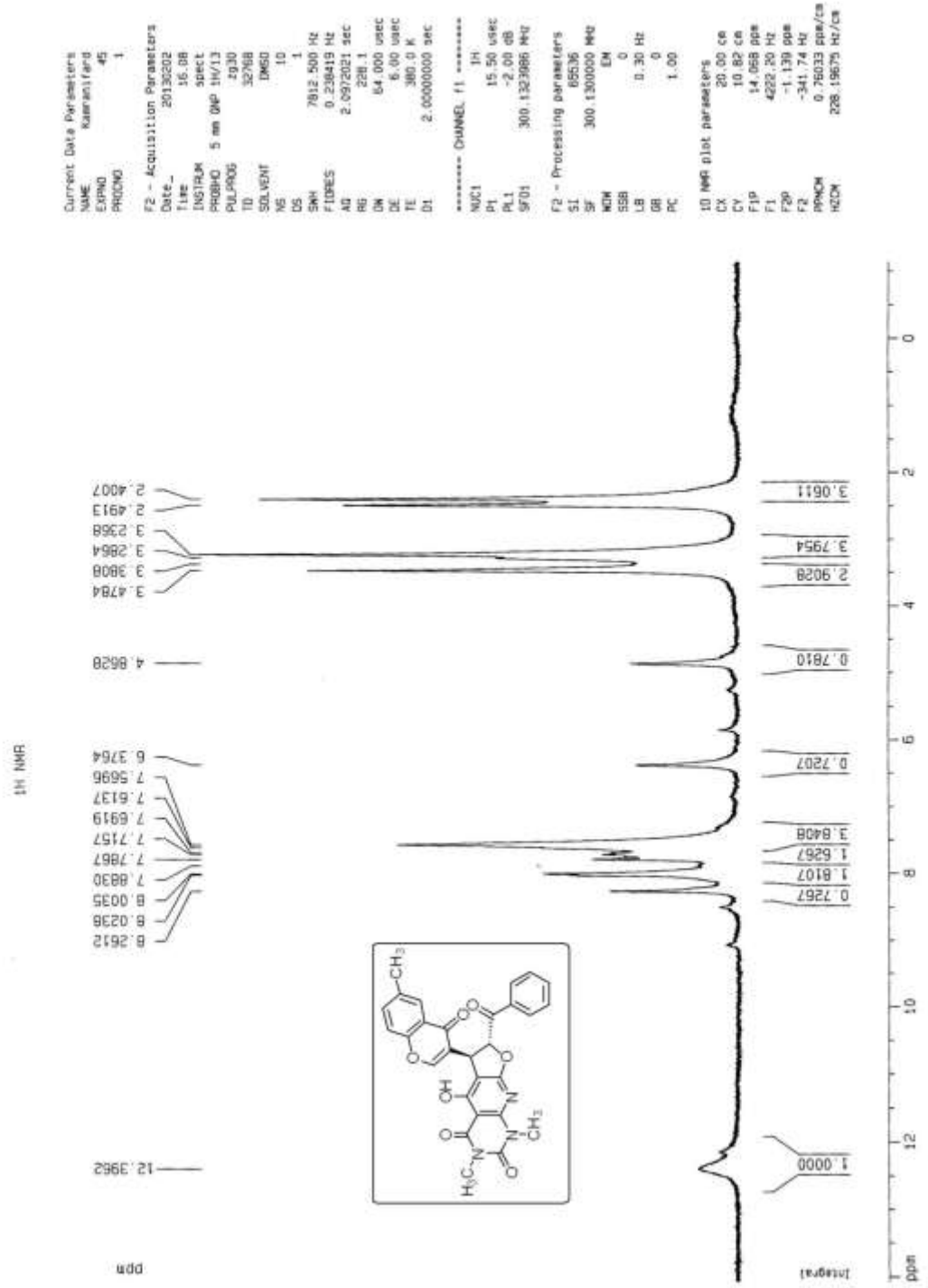

Current Data Parameters
NAME Kamranifard
EXPNO 52
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130225
Time 15.00
INSTRUM spect
PROBHD 5 mm CNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 23.69 cm
F1P 14.198 ppm
F1 4261.13 Hz
F2P -1.132 ppm
F2 -339.79 Hz
PPMCM 0.76649 ppm/cm
HZCM 230.04590 Hz/cm
0.0844
2.4827
3.2155
3.3243
3.4778
4.0898
4.5456
6.3129
6.7098
6.9719
7.6324
7.7830
9.4485
12.3676
4.1010
3.3432
1.2608
1.3463
2.0358
2.0902
4.8323
1.0480
1.0000
Br
OH
OH
O
N
N
CH3
H3C
O
O
1H NMR
ppm
Integral
ppm

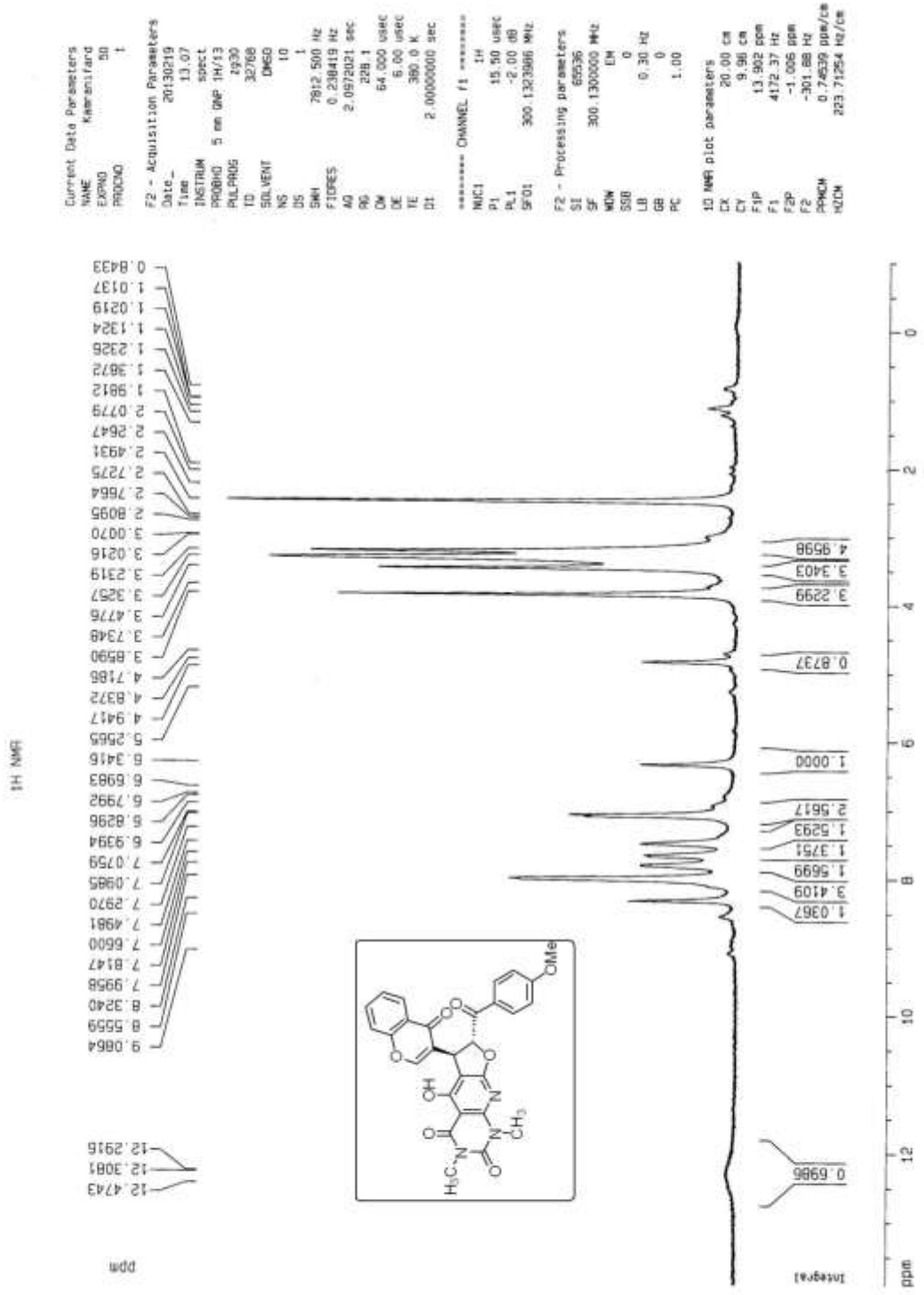

Current Data Parameters
NAME t-12
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130507
Time 21.08
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 32768
SOLVENT DMSO
NS 1200
DS 0
SWH 22075.055 Hz
FIDRES 0.673677 Hz
AQ 0.7422452 sec
RG 2048
DW 22.650 usec
DE 6.00 usec
TE 297.2 K
D1 5.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.30 usec
PL1 -2.00 dB
SFO1 100.6227903 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 18.00 dB
PL12 18.00 dB
SFO2 400.1324008 MHz
F2 - Processing parameters
SI 131072
ppm
28.262
30.590
39.829
40.037
40.246
40.454
40.662
40.870
41.078
46.215
89.504
93.801
101.649
116.502
129.274
131.230
131.787
132.895
133.381
151.102
153.859
157.706
165.083
166.105
171.803
194.022
OH
Br
O
OH
O
N
N
CH3
H3C
N
O

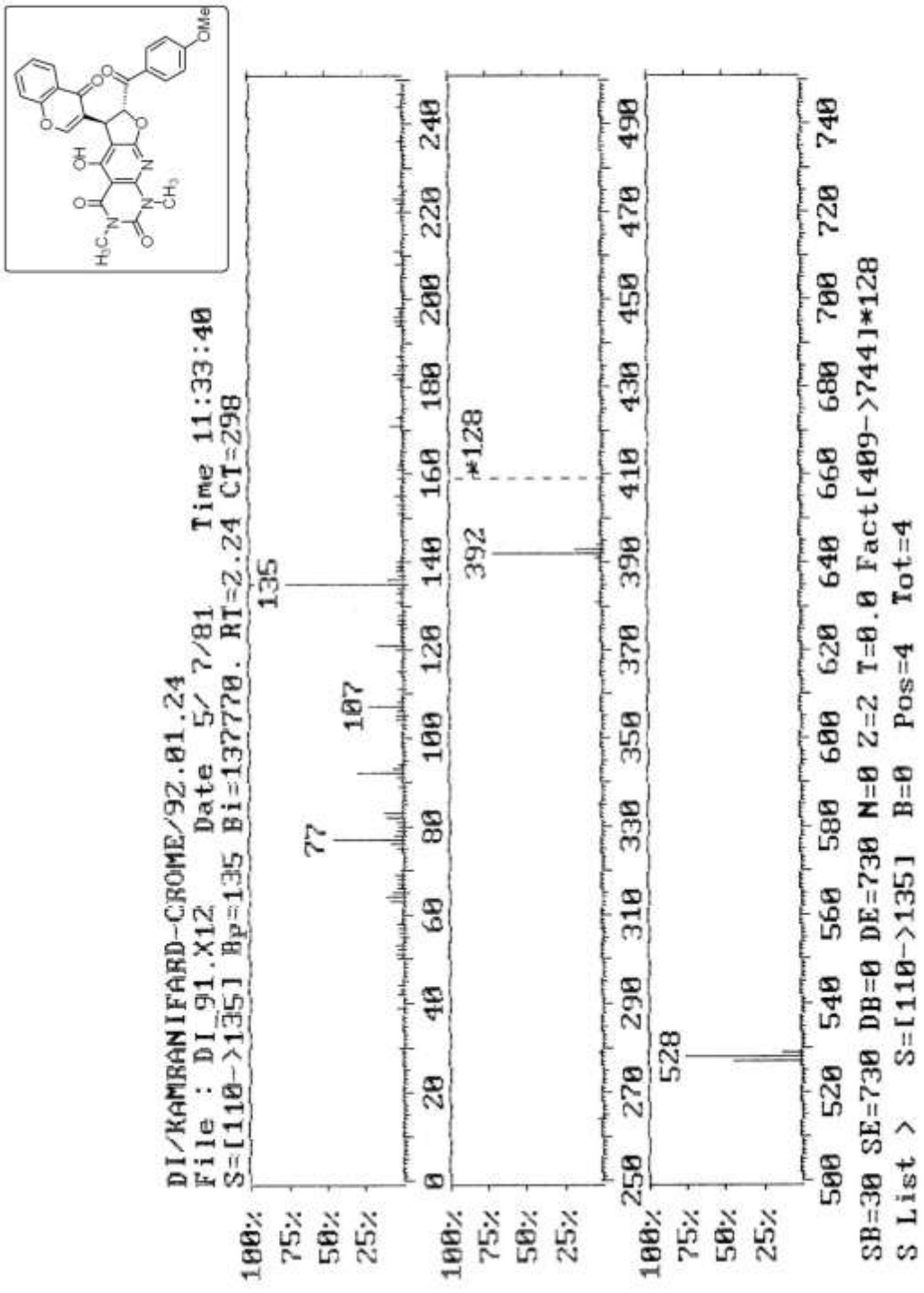

1H NMR
ppm
Integral
12.4250
8.2886
7.9941
7.7835
7.6632
7.5512
6.3776
4.8533
3.7515
3.7254
3.6156
3.4520
3.2754
2.4654
1.0000
1.2453
3.3284
5.2141
0.9236
0.9918
6.5507
0
2
4
6
8
10
12
ppm

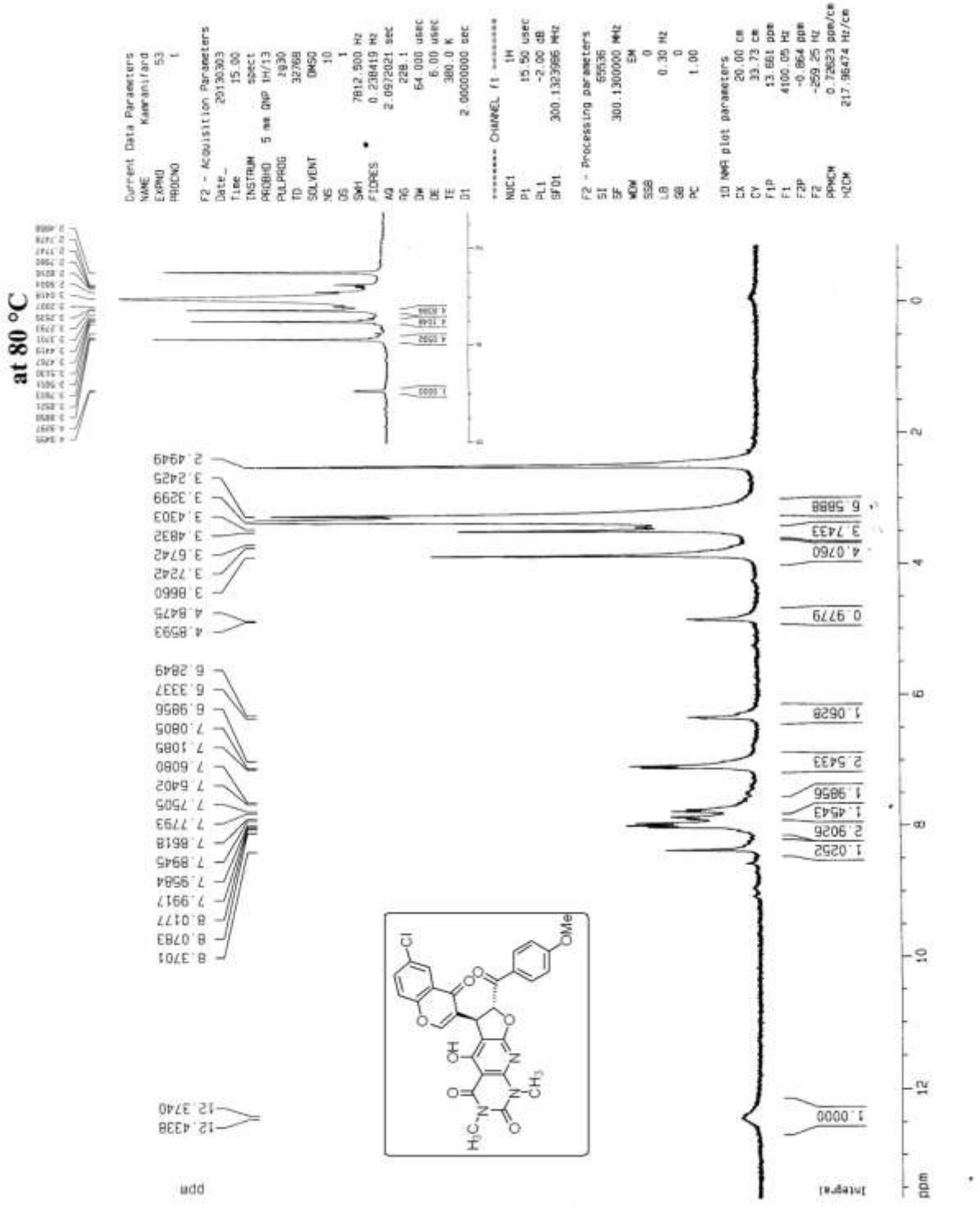

Current Data Parameters
NAME Kamaniford
EXPNO 51
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130220
Time 14.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 389.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.57 cm
F1P 14.765 ppm
F1 4431.46 Hz
F2P -1.404 ppm
F2 -421.45 Hz
PPMCM 0.80947 ppm/cm
HZCM 242.64590 Hz/cm
1H NMR
ppm
Integral
2.4825
2.9655
2.9959
3.0140
3.0368
3.2313
3.3074
3.4659
3.5374
3.5920
3.6249
3.6819
4.8706
4.9365
5.2302
6.3822
7.2218
7.3090
7.3408
7.3538
7.4208
7.5717
7.7219
7.8545
7.9468
7.9999
8.2245
8.2537
8.3562
8.5548
12.1999
12.2049
12.2140
12.3568
12.3780
12.4055
12.4317
12.4693
3.7850
3.1977
0.9352
0.8845
8.1120
0.7602
1.0000
Cl
OH
O
N
N
CH3
H3C
N
O

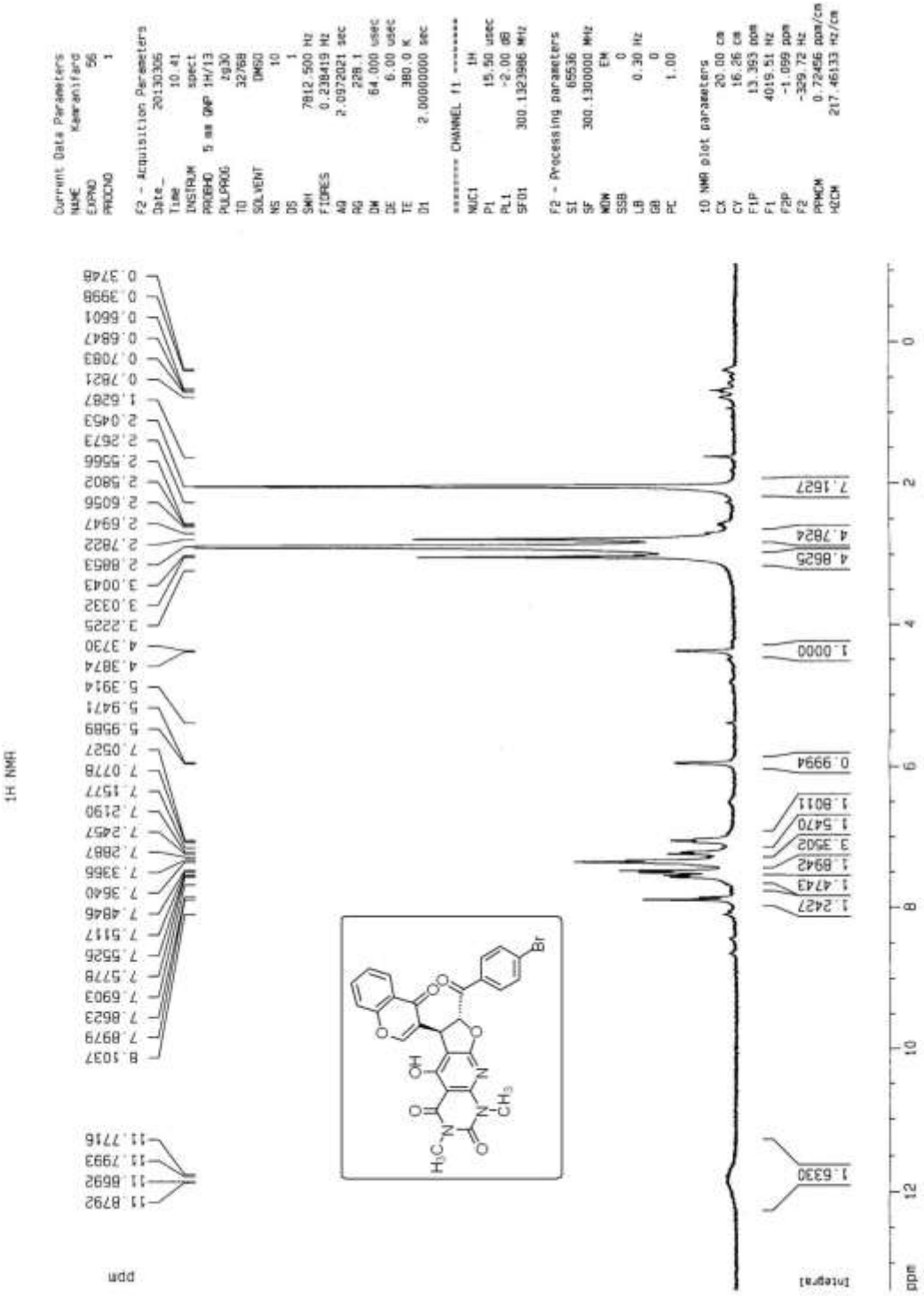


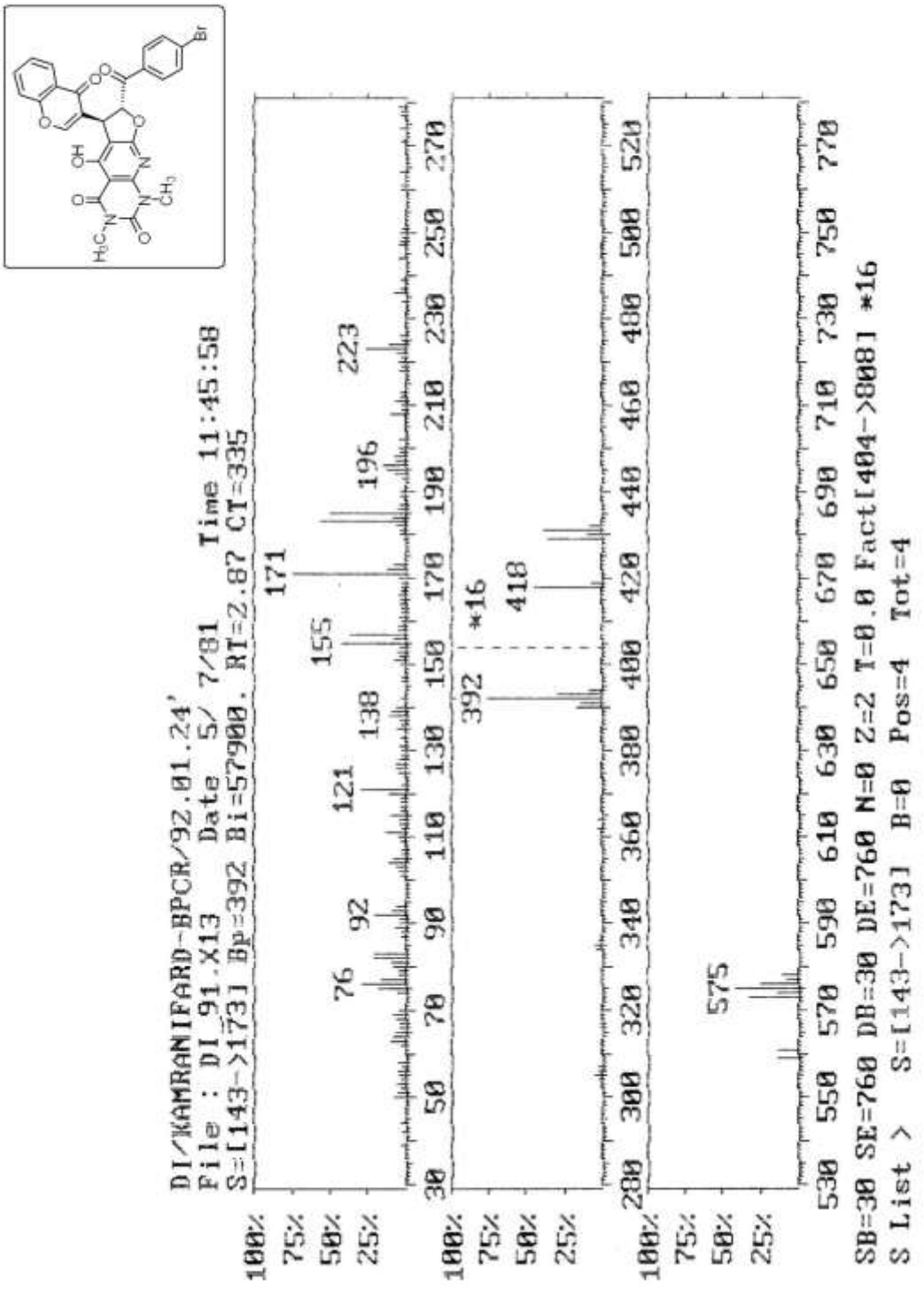

Current Data Parameters
NAME Kamranlfard
EXPNO 45
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130202
Time 16.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.82 cm
F1P 14.068 ppm
F1 4222.20 Hz
F2P -1.139 ppm
F2 -341.74 Hz
PPMCM 0.76033 ppm/cm
HZCM 228.19675 Hz/cm
1H NMR
2.4007
2.4913
3.2368
3.2864
3.3808
3.4784
4.8628
6.3764
7.5696
7.6137
7.6919
7.7157
7.7867
7.8830
8.0035
8.0238
8.2612
12.3962
3.0611
3.7954
2.9028
0.7810
0.7207
3.8408
1.6267
1.8107
0.7267
1.0000
integral
ppm

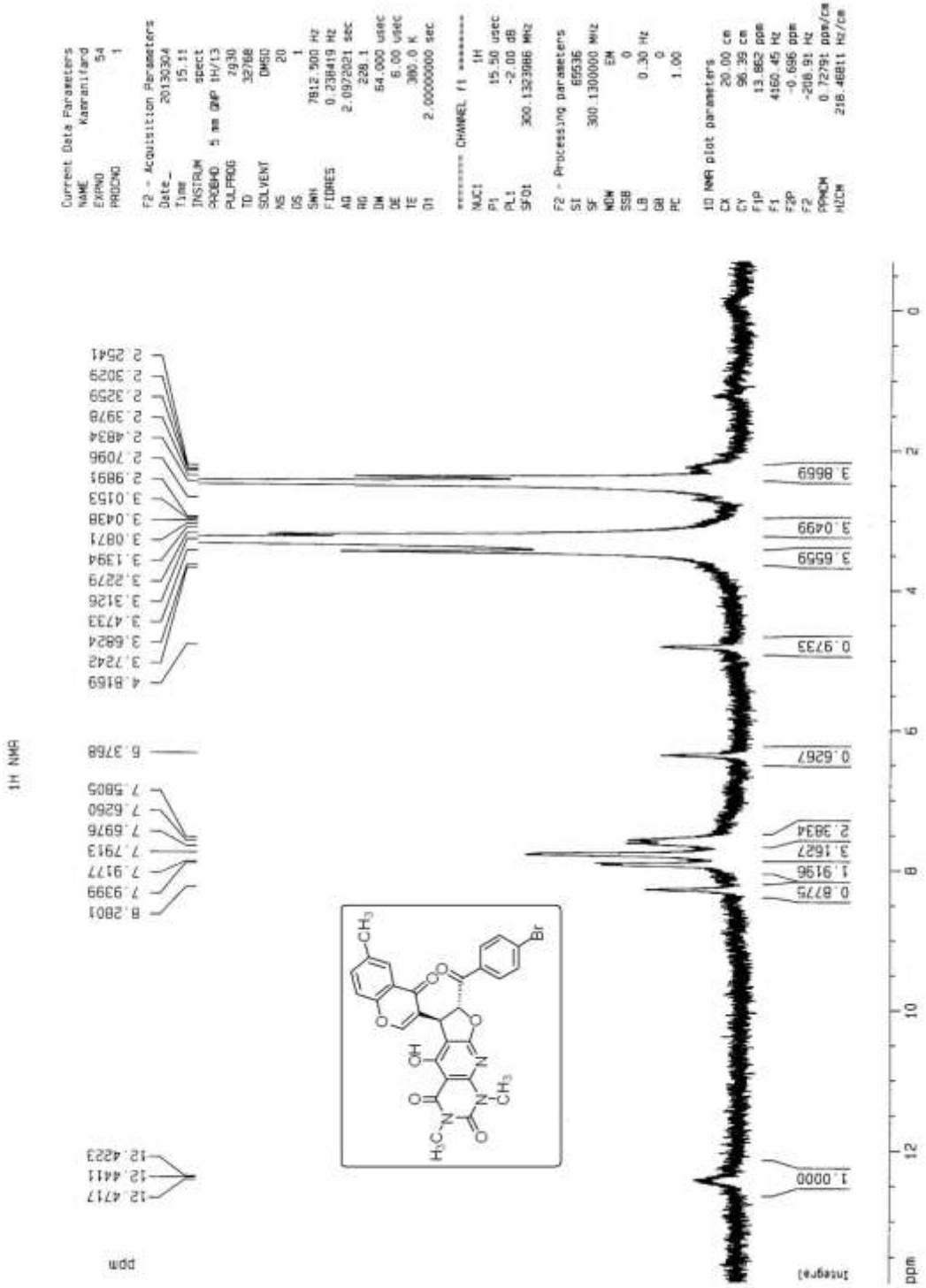

Current Data Parameters
NAME Kamenitard
EXPNO 50
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130219
Time 13.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.09720211 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 360.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 9.96 cm
F1P 13.902 ppm
F1 4172.37 Hz
F2P -1.006 ppm
F2 -301.88 Hz
PPMCM 0.74639 ppm/cm
HZCM 223.71254 Hz/cm
1H NMR
ppm
Integral
OMe
OH
N
CH3
H3C
O


DI/KAMRANIFARD-CROME/92.01.24
File : DI_91.X12 Date 5/ 7/81 Time 11:33:40
S=[110->135] Bp=135 Bi=137770. RT=2.24 CT=298
135
77
107
392
*128
528
100%
75%
50%
25%
0 20 40 60 80 100 120 140 160 180 200 220 240
250 270 290 310 330 350 370 390 410 430 450 470 490
500 520 540 560 580 600 620 640 660 680 700 720 740
SB=30 SE=730 DB=0 DE=730 N=0 Z=2 T=0.0 Fact[409->744]*128
S List > S=[110->135] B=0 Pos=4 Tot=4


at 80 °C
Current Data Parameters
NAME Kawanisiard
EXPNO 53
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130303
Time 15.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 13.73 cm
F1P 13.661 ppm
F1 4100.05 Hz
F2P -0.864 ppm
F2 -259.25 Hz
PPMCM 0.72623 ppm/cm
HZCM 217.96474 Hz/cm
2.4949
3.2425
3.3299
3.4303
3.4832
3.6742
3.7242
3.8660
4.8475
4.8593
6.2849
6.3337
6.9856
7.0805
7.1085
7.6080
7.6402
7.7505
7.7793
7.8618
7.8945
7.9584
7.9917
8.0177
8.0783
8.3701
12.4238
12.3740
1.0000
1.0252
2.9026
1.4543
1.9856
2.5433
1.0628
0.9779
4.0760
3.7433
5.5888
Integral
ppm
Cl
OMe
OH
O
N
CH3
H3C


Current Data Parameters
NAME Kamanifard
EXPNO 56
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130305
Time 10.41
INSTRUM spect
PROBHD 5 mm DMP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SF01 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 16.26 cm
F1P 13.393 ppm
F1 4019.51 Hz
F2P -1.099 ppm
F2 -329.72 Hz
PPMCM 0.72456 ppm/cm
HZCM 217.46133 Hz/cm
1H NMR
ppm
Integral
Br
OH
N
O
CH3
H3C




Current Data Parameters
NAME Kamranifard
EXPNO 54
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130304
Time 15.11
INSTRUM spect
PROBHD 5 mm BBF 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 20
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1329986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 96.39 cm
F1P 13.862 ppm
F1 4160.45 Hz
F2P -0.696 ppm
F2 -208.91 Hz
PPMCM 0.72791 ppm/cm
HZCM 218.46811 Hz/cm
1H NMR
ppm
Integral
12.4717
12.4411
12.4223
8.2801
7.9399
7.9177
7.7913
7.6976
7.6260
7.5805
5.3768
4.8169
3.7242
3.6824
3.4733
3.3126
3.2279
3.1394
3.0871
3.0438
3.0153
2.9891
2.7096
2.4834
2.3978
2.3259
2.3029
2.2541
1.0000
0.8775
1.9196
3.1627
2.3834
0.8267
0.9733
3.6559
3.0499
3.8669
Br
CH3
OH
N
O
H3C