Supplementary Material (ESI) for Chemical Communications
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Supporting information

Silver-catalyzed intramolecular cyclization of 9-propargyladenine via N3 alkylation

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General procedures: ^1H and ^{13}C NMR spectra were obtained on a JEOL-DELTA2 500 model spectrometer operating at 500MHz. The spectra were recorded in DMSO-*d6* solution and the chemical shifts were referenced with respect to tetramethylsilane. High resolution (ESI+ mode) mass spectra were obtained on WATERS HAB 213 machine, Department of Chemistry, IIT Kanpur. Infrared spectra were obtained (KBr disk, 400 – 4000 cm^{-1}) on a Perkin-Elmer Model 1320 spectrometer.

Synthesis of 9-propargyladenine (1): The title compound was synthesized by following a literature procedure[1]. Adenine (2.0g, 1eq) was suspended in DMF (45mL) followed by addition of K_2CO_3 (4.09g, 2eq) and stirring under N_2 atmosphere for 30 min, after this propargyl bromide (4.40g, 2eq) is added and stirred for 15 hours under N_2 atmosphere. After this time DMF was evaporated at 60°C under high vacuum and compound was purified by column chromatography eluting with methanol/DCM to afford white powder (1.8g, 70% Yield). HRMS: $(M+1)^+$ calculated: 174.0780, Found: 174.0782(100); M.P.209-210 °C; IR (KBr): 2110 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6, 25 °C): δ (ppm) 3.43 (s, 1H, Acetylenic C-H), 4.98 (d, 2H, CH_2), 7.26 (s, 2H, NH_2), 8.12 (s, 1H, C8-H), 8.15 (s, 1H, C2-H); ^{13}C NMR (125 MHz, DMSO-d_6, 25 °C): δ (ppm) 32.76, 76.38, 78.86, 119.00, 140.62, 149.59, 153.23, 156.53.

Synthesis and characterization of cyclized product (2): In a 25 ml round bottom flask, wrapped with aluminum foil, 50 mg ligand (**1**) was dissolved in methanol and to this aqueous solution of silver nitrate (1 mole equivalent) was added drop wise with stirring. The complex started precipitating out immediately. Stirring was continued for another one hour. After this time, the precipitate was filtered carefully to avoid direct light, washed with water (4x 5 mL) and methanol (4 x 5 mL) to remove any traces of unreacted metal salt and ligand. The product so obtained was dried under high vacuum (50 mg, 73.5% Yield). HRMS: M^+ calculated: 174.0780, Found: 174.0787(100); M.P. > 300 ^oC; ^1H NMR (500 MHz, DMSO-d_6, 25 ^oC): δ (ppm) 5.01 (d, 2H, CH_2), 9.60, 9.54 (s, 2H, NH_2), 8.42 (s, 1H, C8-H), 8.67 (s, 1H, C2-H); ^{13}C NMR (125 MHz, DMSO-d_6, 25 ^oC): 43.46, 112.63, 116.62, 120.56, 141.86, 143.28, 157.29.

Synthesis of 6-*N*-acetyl-9-propargyl adenine: 9-Propargyladenine (1.0g, 1eq) was dissolved in DMF (25ml) followed by the addition of acetic anhydride (1.64ml, 3eq, d=1.08g/ml). The reaction mixture was refluxed at 160 ^oC for two hour with constant stirring. After this time DMF was evaporated at 60 ^oC under high vacuum and compound was purified by column chromatography eluting with methanol/DCM to afford white powder (800mg, 64.5% Yield). HRMS: $(M+1)^+$ calculated: 216.0885, Found: 216.0883 (100);M.P. 208-209^oC; IR (KBr): 2127 cm^{-1} ^1H NMR (500 MHz, DMSO-d_6, 25 ^oC): δ (ppm) 3.51 (s, 1H, acetylenic C-H), 5.14 (d, 2H, CH_2), 10.71 (s, 1H, N6-H), 8.51 (s, 1H, C8-H), 8.66 (s, 1H, C2-H), 2.24 (s, 3H, CH_3); ^{13}C NMR (125 MHz, DMSO-d_6, 25 ^oC): 24.31, 32.67, 76.27, 77.87, 123.07, 143.60, 149.48, 151.47, 151.80, 168.85.

Crystal structure refinement details for 1 and 2: Single Crystal of **1** and **2** were coated with light hydrocarbon oil and mounted in the 100 K dinitrogen stream of a Bruker SMART APEX CCD diffractometer equipped with CRYO Industries low-temperature apparatus and intensity data were collected using graphite-monochromated Mo Kα radiation. The data integration and reduction were processed with the SAINT software.[2] An absorption correction was applied.[3] Structures were solved by the direct method using SHELXS-97 and refined on *F*2 by a full-matrix least-squares technique using the SHELXL-97 program package.[4] Non-hydrogen atoms were refined anisotropically. In the refinement, hydrogens were treated as riding atoms using the SHELXL default parameters. Crystal structure refinement parameters are given in Table S1 whereas H-bonding parameters are provided in Table S2. CCDC contains the supplementary crystallographic data for this paper with a deposition number of CCDC **757808** and **757809**. Copies of this information can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK. [Fax: +44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk].

Table S1. Crystal structure refinement parameters for **1** and **2**.

Identification code	**Complex 1**	**Complex 2**
Empirical formula	$C_8H_7N_5$	$C_8H_8N_6O_3$
*M*r	173.19	236.20
crystal system	orthorhombic	triclinic
space group	*P 21 21 21*	*P -1*
a/Å	4.232(2)	5.536(3)
b/Å	8.030(5)	6.938(4)
c/Å	23.829(5)	12.729(7)
α/°	90.000(5)	93.040(10)
β/°	90.000(5)	97.088(10)
γ/°	90.000(5)	101.559(10)
Volume/ $Å^3$	809.8(7)	473.8(5)
Z	4	2
Dx /Mg m^{-3}	1.421	1.656
F(000)	360	244
μ/ mm^{-1}	0.096	0.132
θ range for data collection/ °	2.68 to 25.99	3.01 to 25.00
Limiting indices	-5 → h → 3, -9 → k →9, -29 → l → 27	-4 → h → 6, -8 → k → 6, -14 → l → 15
Reflections collected	4220	2428
unique reflections	1548	1643
R(int)	0.0672	0.0449
Completeness to *θ*	= 25.99, 98.9	= 25.00, 98.1
$T_{\max}$ / $T_{\min}$	0.9867/0.9811	0.9869 / 0.9767
Data / restraints / parameters	1548/0/118	1643 / 0 / 155
Goodness-of-fit on F^2	1.067	1.019
*R*1 and *R*2 [$I>2\sigma(I)$]	0.0516, 0.1163	0.0751,0.1776
*R*1 and *R*2 (all data)	0.1220, 0.1271	0.0896, 0.1971
Largest diff. peak and hole/e.A^{-3}	0.240 and -0.243	0.443 and -0.587

Table S2. (a) Hydrogen bond parameters for complexes **1** and **2**, **(b)** Observed bond lengths between constituent atoms for complexes **1** and **2**.

a

D—H…A^a	**H...A**	**D...A**	**D—H...A**
Complex 1			
N(6)—H(6A)...N(7)^{i}	2.19	3.043(4)	173
N(6)—H(6B)...N(1)^{ii}	2.09	2.907(4)	159
C(11)—H(11)...N(3)^{iii}	2.34	3.257(4)	169
Complex 2			
N(6)—H(6A)...O(1)^{iv}	2.01	2.859(3)	169
N(6)—H(6B)...N(7)^{v}	2.14	2.972(4)	162
C(8)—H(8)...O(3)^{vi}	2.16	3.042(4)	158
C(9)—H(9B)...O(2)^{vii}	2.50	3.390(4)	153
C(10)—H(10)...O(2)^{viii}	2.50	3.338(4)	149
C(11)—H(11)...O(1)^{ix}	2.46	3.278(4)	147
C(11)—H(11)...O(2)^{ix}	2.37	3.256(4)	159

b

Bond	Complex **1**	Complex **2**
C2—N1	1.33	1.31
C2—N3	1.33	1.35
C4—C5	1.38	1.37
C4—N3	1.34	1.35
C4—N9	1.36	1.33
C5—C6	1.41	1.41
C5—N7	1.38	1.39
C6—N1	1.34	1.39
C6—N6	1.33	1.32
C8—N7	1.32	1.32
C8—N9	1.36	1.37
C9—C10	1.46	1.49
C9—N9	1.46	1.48
C10—C11	1.16	1.31
C11—N3	---	1.44

[a] Symmetry of A: (i) 1-x,1/2+y,1/2-z; (ii) 1-x,-1/2+y,1/2-z; (iii) 1/2+x,1/2-y,-z; (iv) 1-x,1-y,-z; (v) 2-x,2-y,-z; (vi) 1+x,1+y,z; (vii) 2-x,1-y,1-z; (viii) 1-x,1-y,1-z; (ix) -1+x,y,z; where A= acceptor and D=donor

Figures:

Figure S1 shows the formation of adenine ribbon with Watson-Crick and Hoogsteen interaction and further interaction of adjacent adenine ribbons with C-H….N hydrogen bonds.

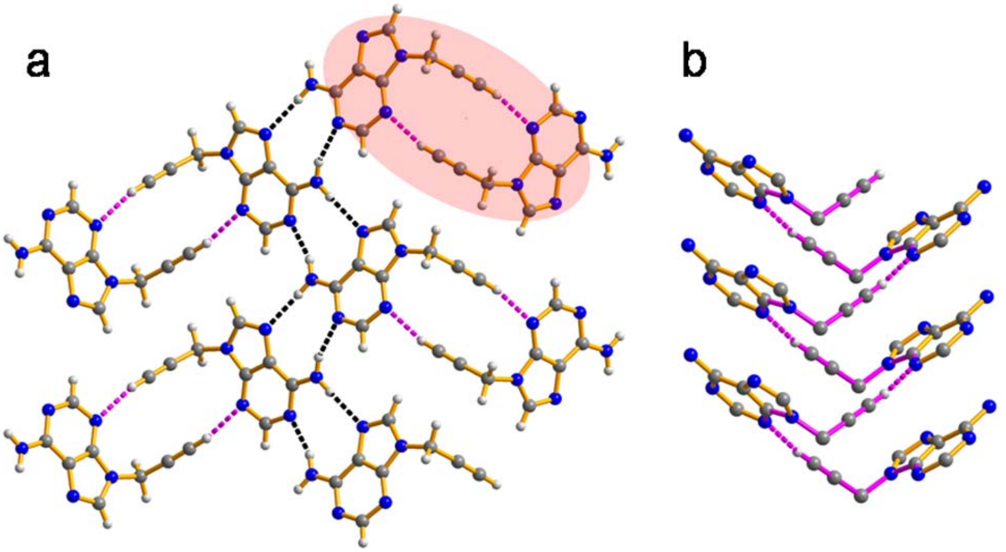


Figure S1: **(a)** View of crystal lattice of **1** along a-axis **(b)** different view of highlighted region shows helical structure.

Figure S2 shows the formation of adenine dimer with Hoogsteen face interaction which is further connected with nitrate counter anion with N-H….O and C-H….O hydrogen bonding.

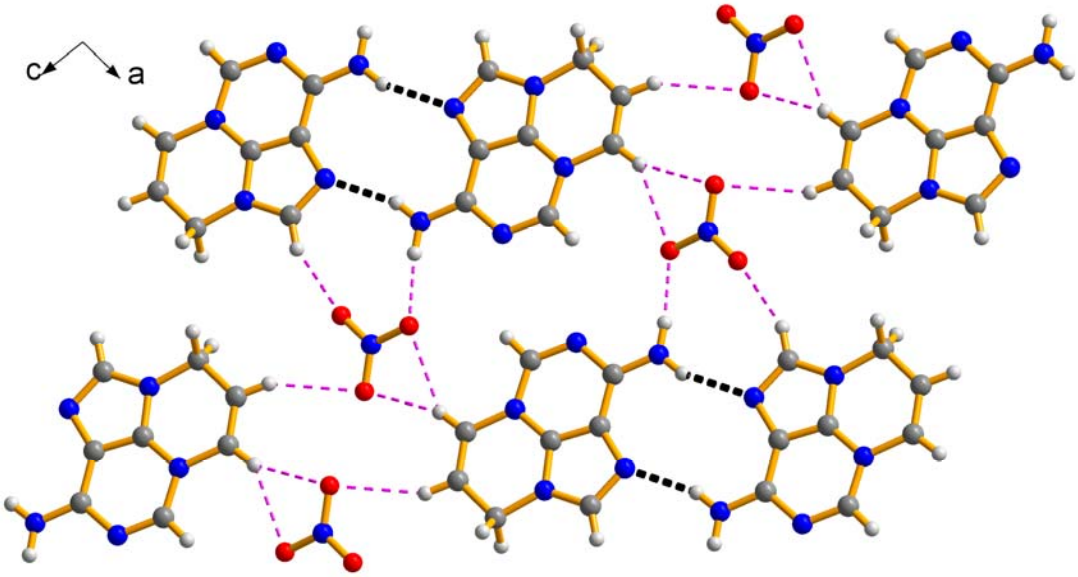
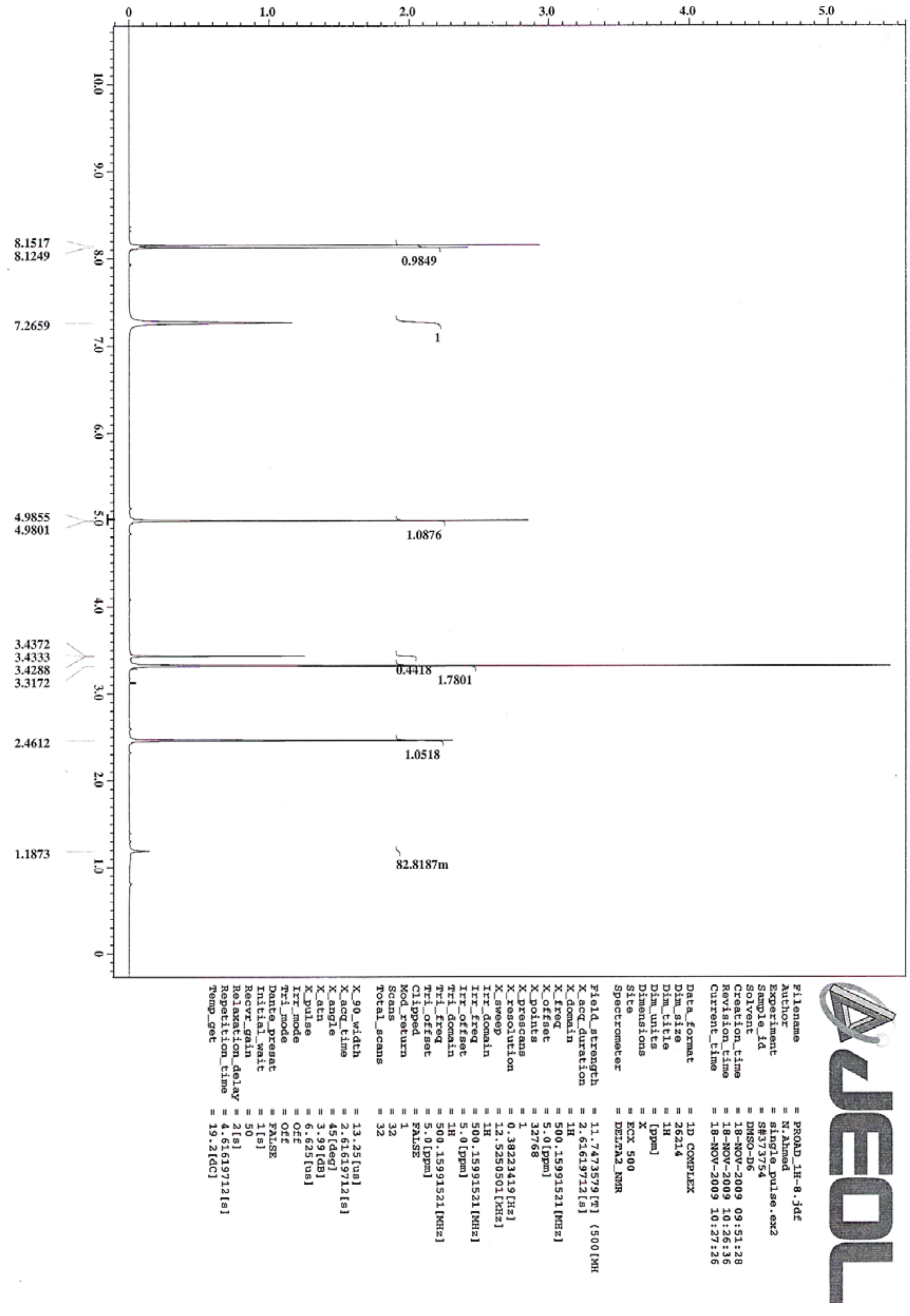


Figure S2: View of crystal lattice of **2** along b-axis.

Figure S3: ^{1}H NMR of 9-propargyladenine (**1**).



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Revision_time     = 18-NOV-2009 10:26:36
Current_time      = 18-NOV-2009 10:27:26

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Dim_units         = [ppm]
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Spectrometer      = DELTA2_NMR

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Recvr_gain        = 50
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Temp_get          = 19.2[dC]
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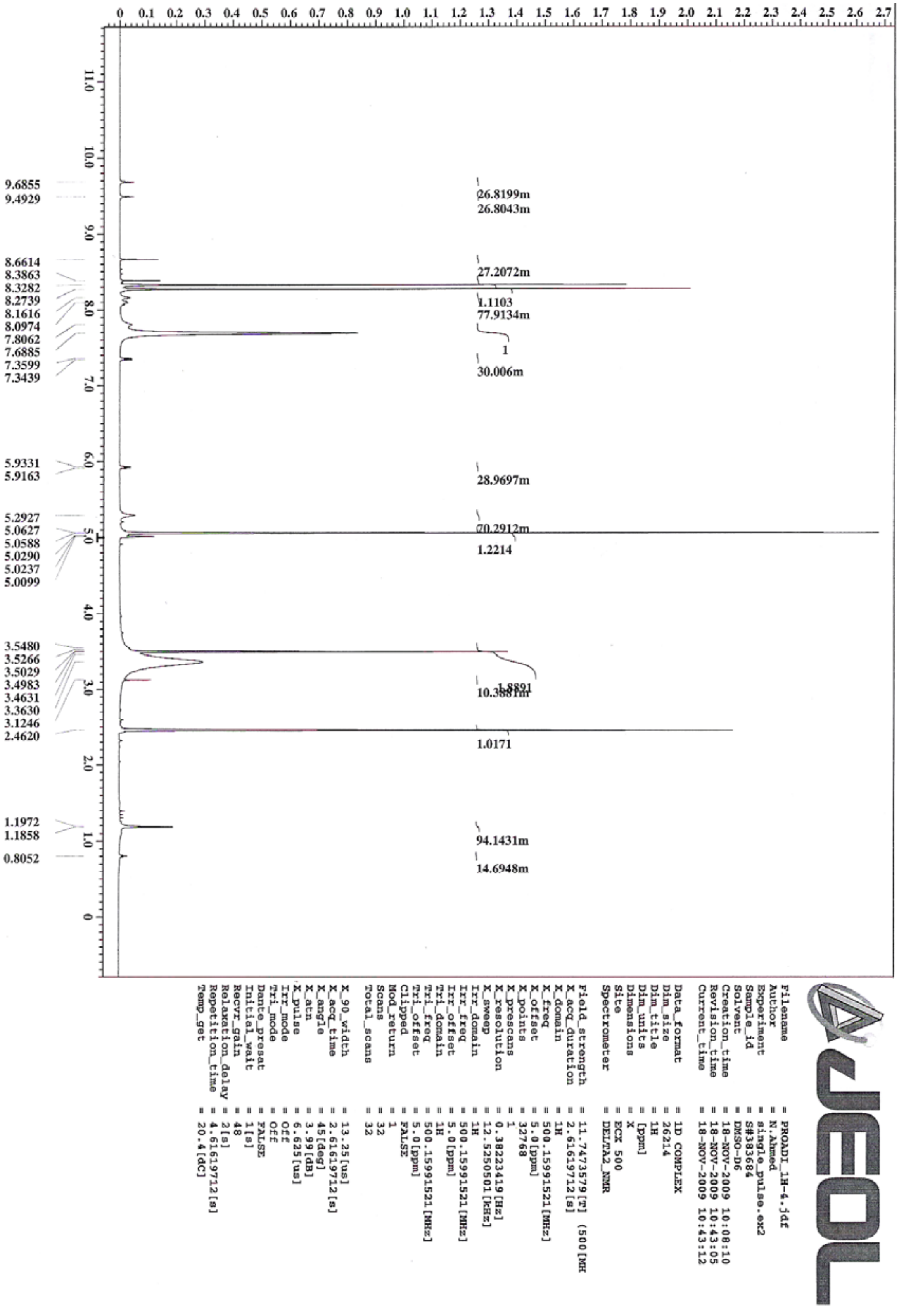
Figure S4: ^{1}H NMR of 9-propargyladenine (**1**) just after addition of silver nitrate.



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Solvent           = DMSO-D6
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Revision_time     = 18-NOV-2009 10:43:05
Current_time      = 18-NOV-2009 10:43:12

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Dim_units         = [ppm]
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Spectrometer      = DELTA2_NMR

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X_offset          = 5.0[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.38223419[Hz]
X_sweep           = 12.5250501[kHz]
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Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
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Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
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Total_scans       = 32

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X_acq_time        = 2.61619712[s]
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X_atn             = 3.99[dB]
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Tri_mode          = Off
Dante_presat      = FALSE
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Relaxation_delay  = 2[s]
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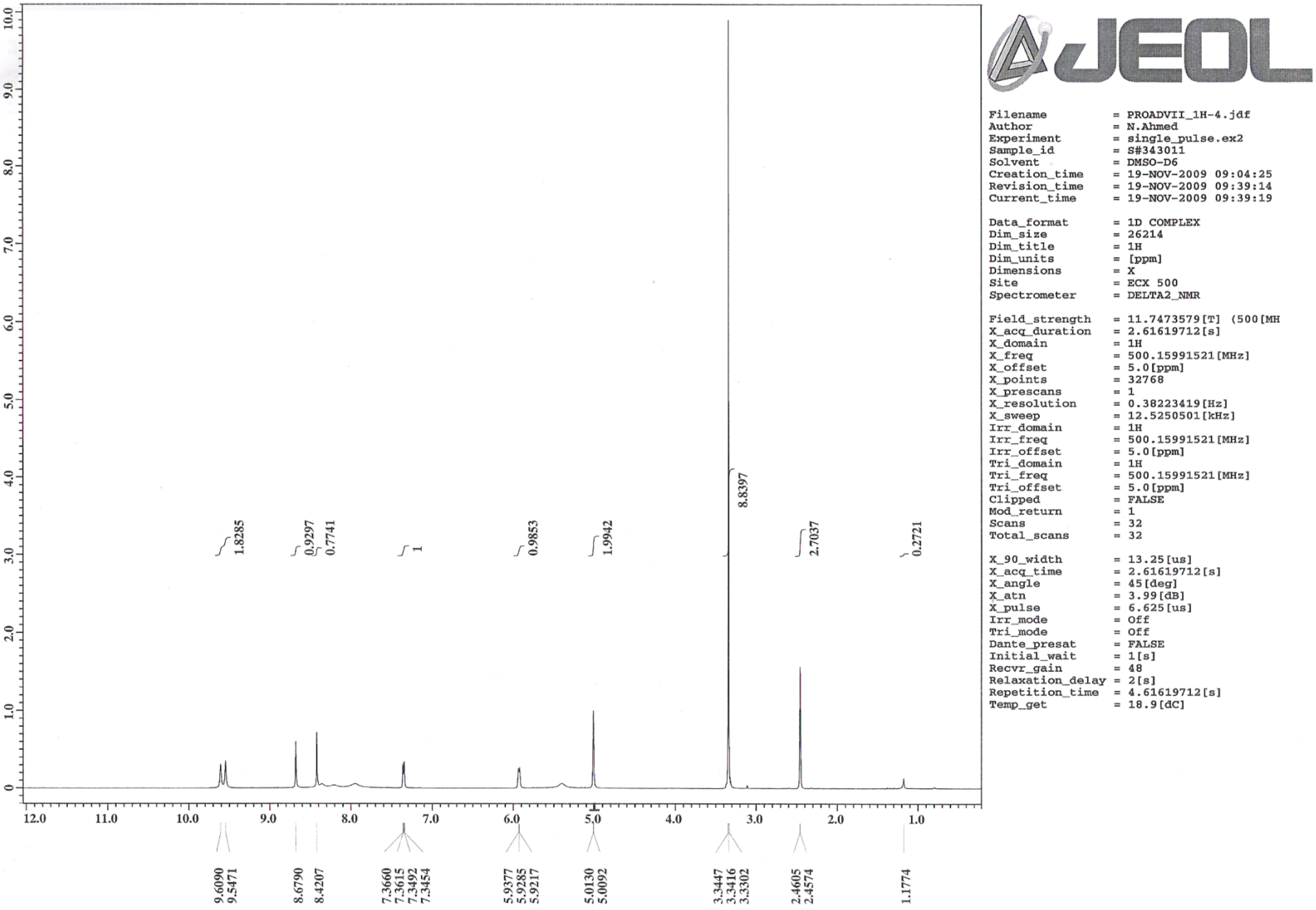
Figure S5: ^{1}H NMR of cyclized derivative of 9-propargyladenine (**2**).



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Experiment          = single_pulse.ex2
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Revision_time       = 19-NOV-2009 09:39:14
Current_time        = 19-NOV-2009 09:39:19

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Dim_size            = 26214
Dim_title           = 1H
Dim_units           = [ppm]
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Site                = ECX 500
Spectrometer        = DELTA2_NMR

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X_points            = 32768
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X_sweep             = 12.5250501[kHz]
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Irr_freq            = 500.15991521[MHz]
Irr_offset          = 5.0[ppm]
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Tri_freq            = 500.15991521[MHz]
Tri_offset          = 5.0[ppm]
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Total_scans         = 32

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X_acq_time          = 2.61619712[s]
X_angle             = 45[deg]
X_atn               = 3.99[dB]
X_pulse             = 6.625[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 48
Relaxation_delay    = 2[s]
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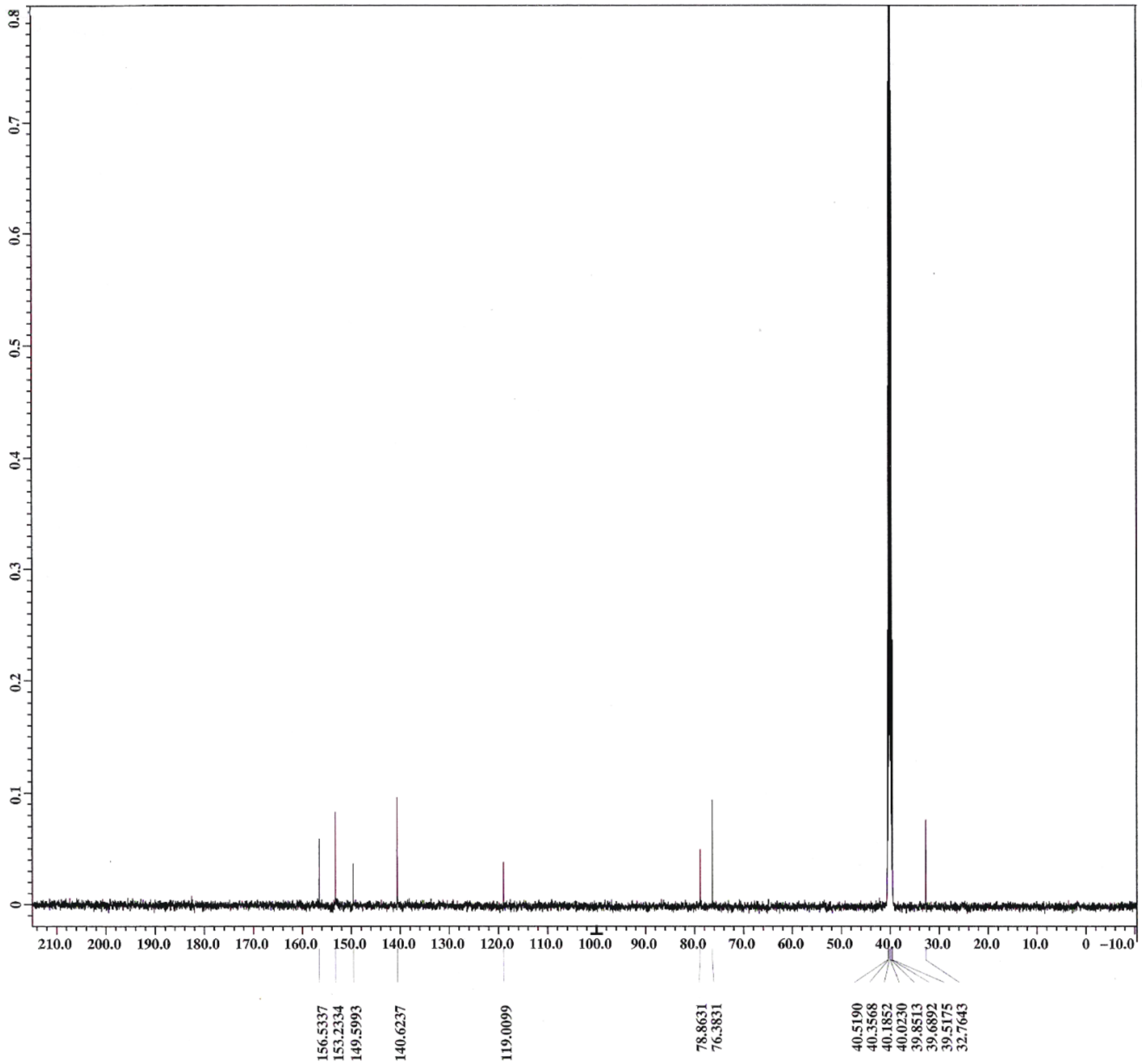
Figure S6: ^{13}C NMR of 9-propargyladenine (**1**).



JEOL

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Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#448701
Solvent           = DMSO-D6
Creation_time     = 24-NOV-2009 13:58:49
Revision_time     = 24-NOV-2009 14:33:33
Current_time      = 24-NOV-2009 14:33:41

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Initial_wait      = 1[s]
Noe               = TRUE
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Recvr_gain        = 60
Relaxation_delay  = 1.19[s]
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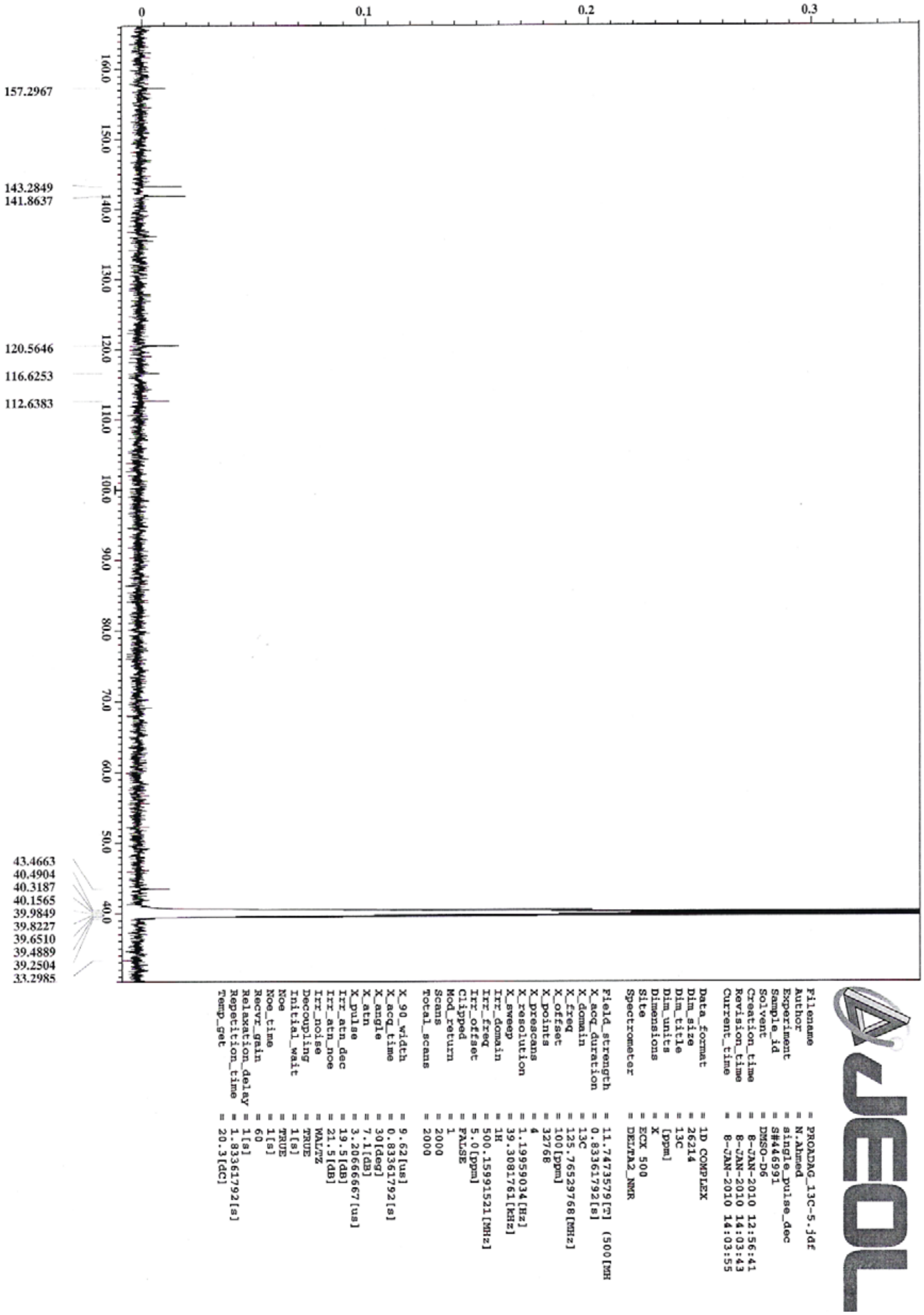
Figure S7: ^{13}C NMR of cyclized derivative of 9-Propargyl adenine (**2**).



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Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
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Figure S8: HRMS of 9-propargyladenine (**1**).

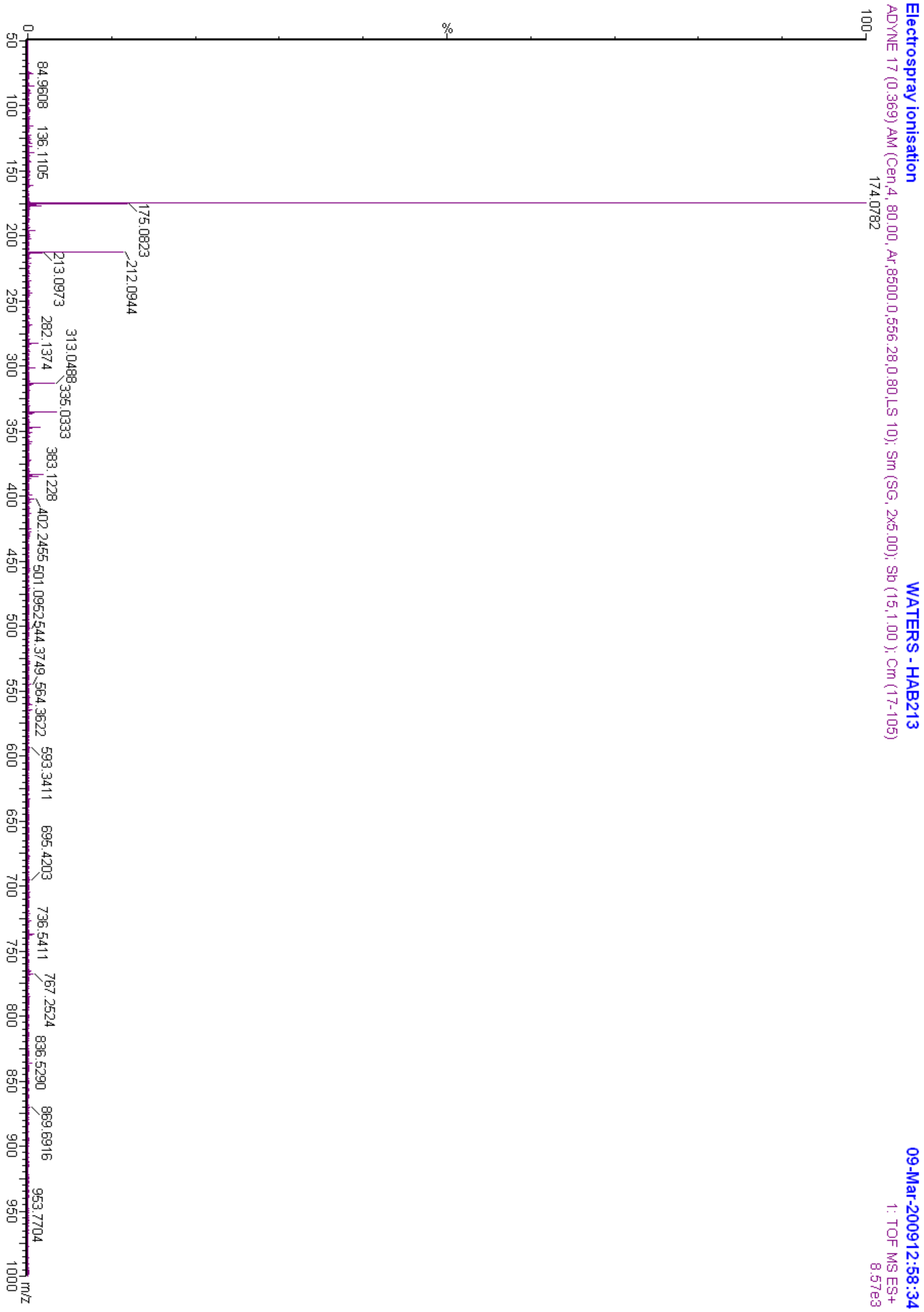


Figure S9: HRMS of cyclized derivative of 9-propargyladenine (**2**).

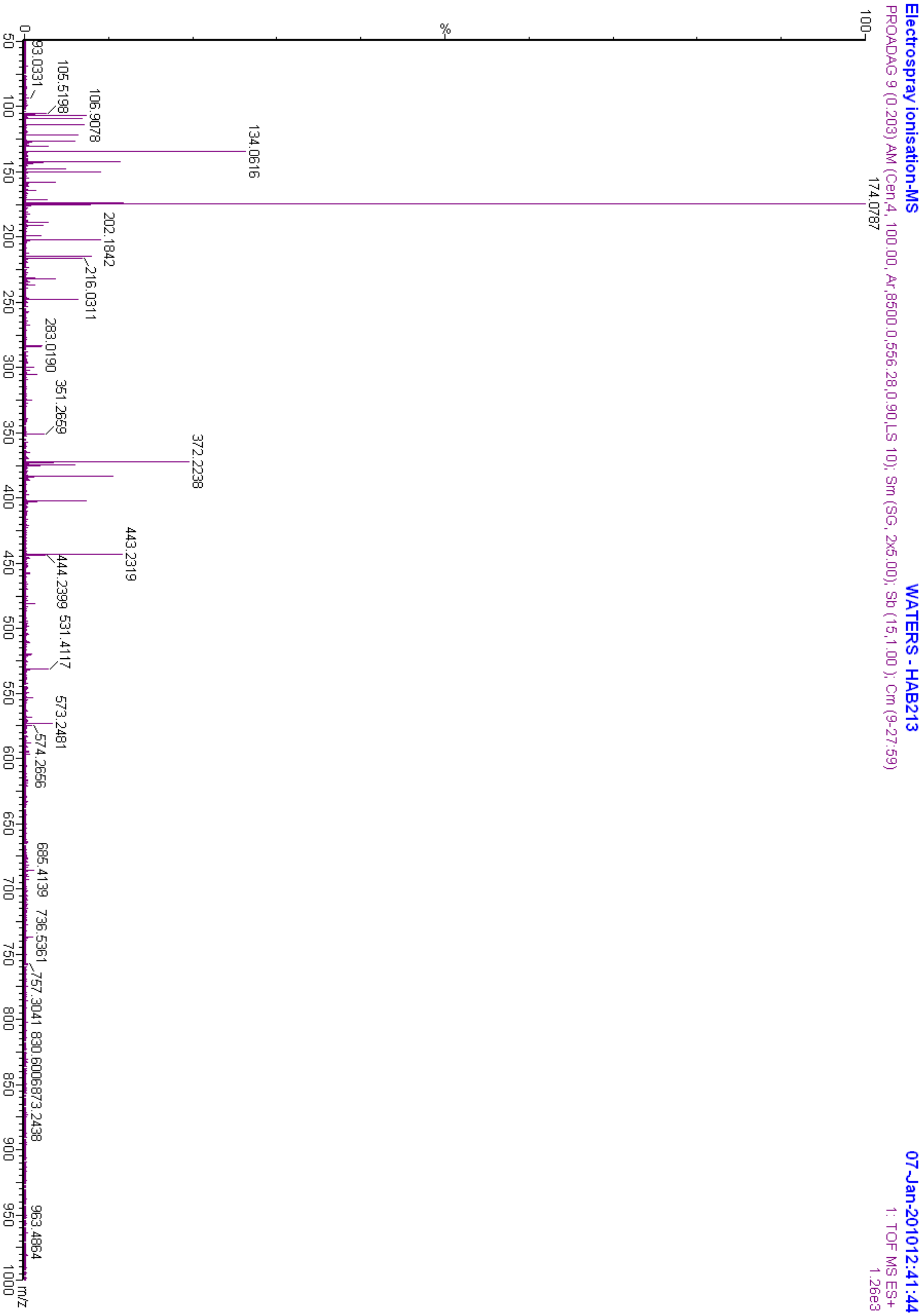
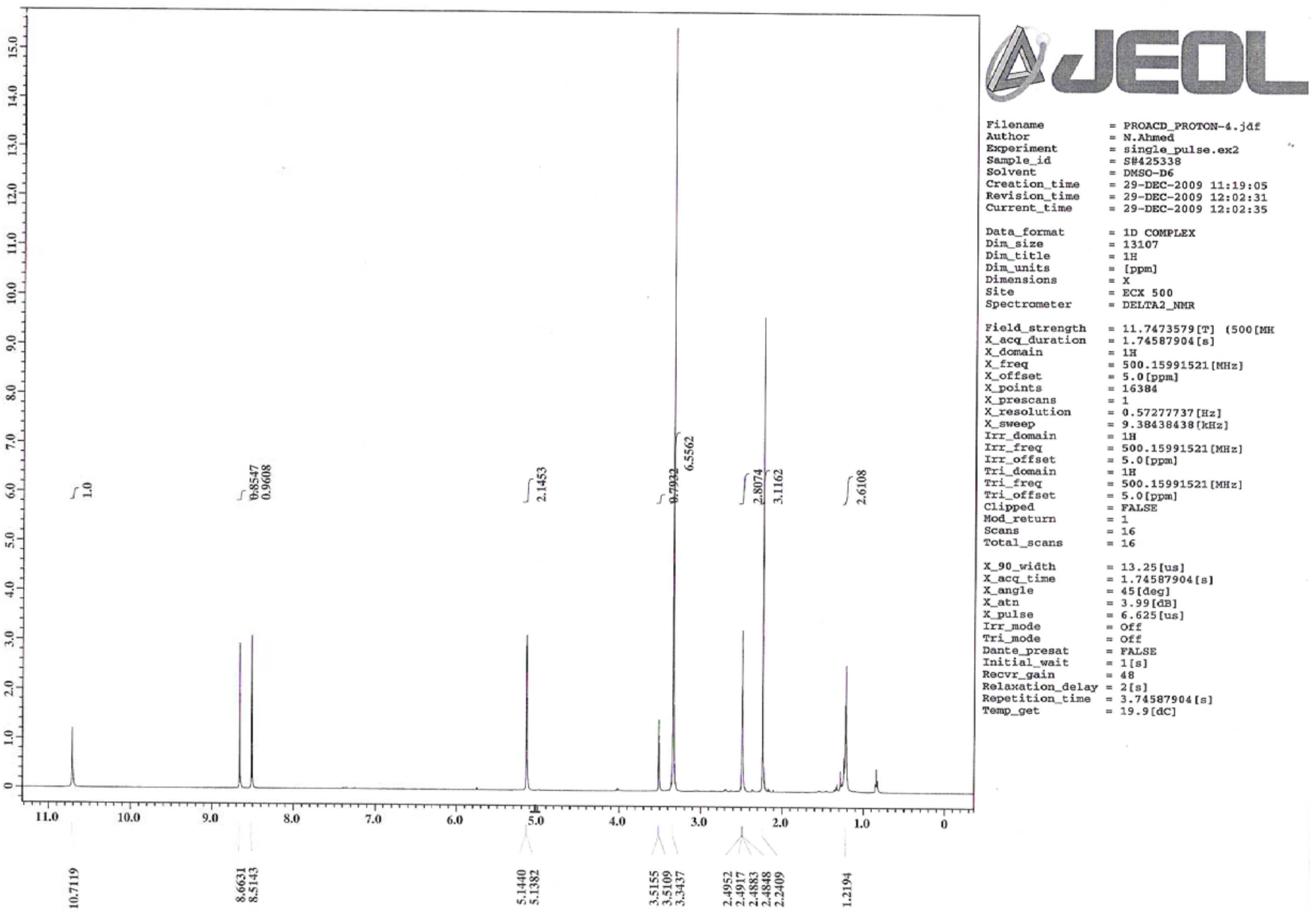


Figure S10: 1H NMR of 6-*N*-acetyl-9-propargyladenine.



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X_atn              = 3.99[dB]
X_pulse            = 6.625[us]
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Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
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Relaxation_delay   = 2[s]
Repetition_time    = 3.74587904[s]
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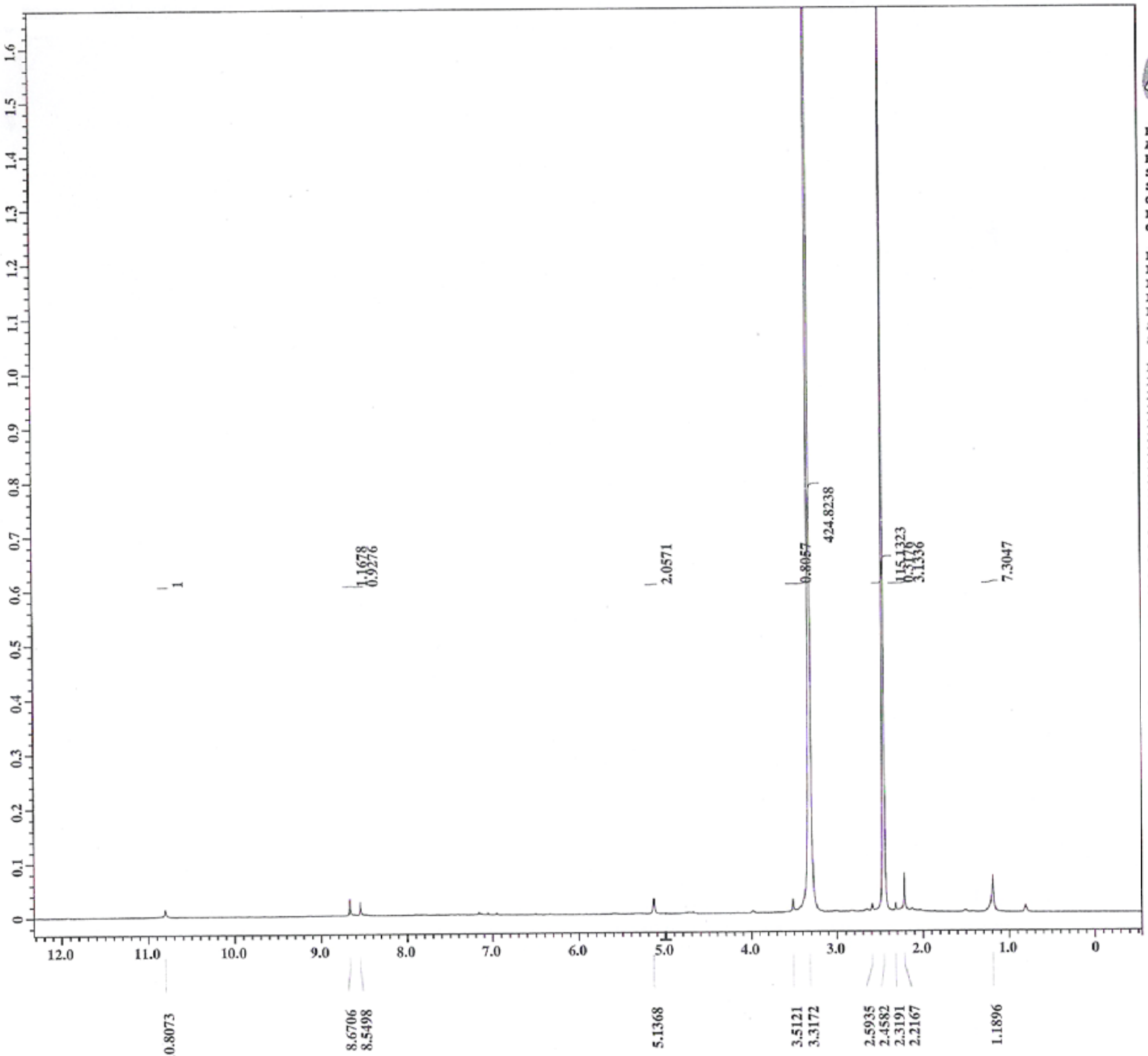
Figure S11: ^{1}H NMR of 6-*N*-acetyl-9-propargyladenine with $AgNO_3$ after 10 h.



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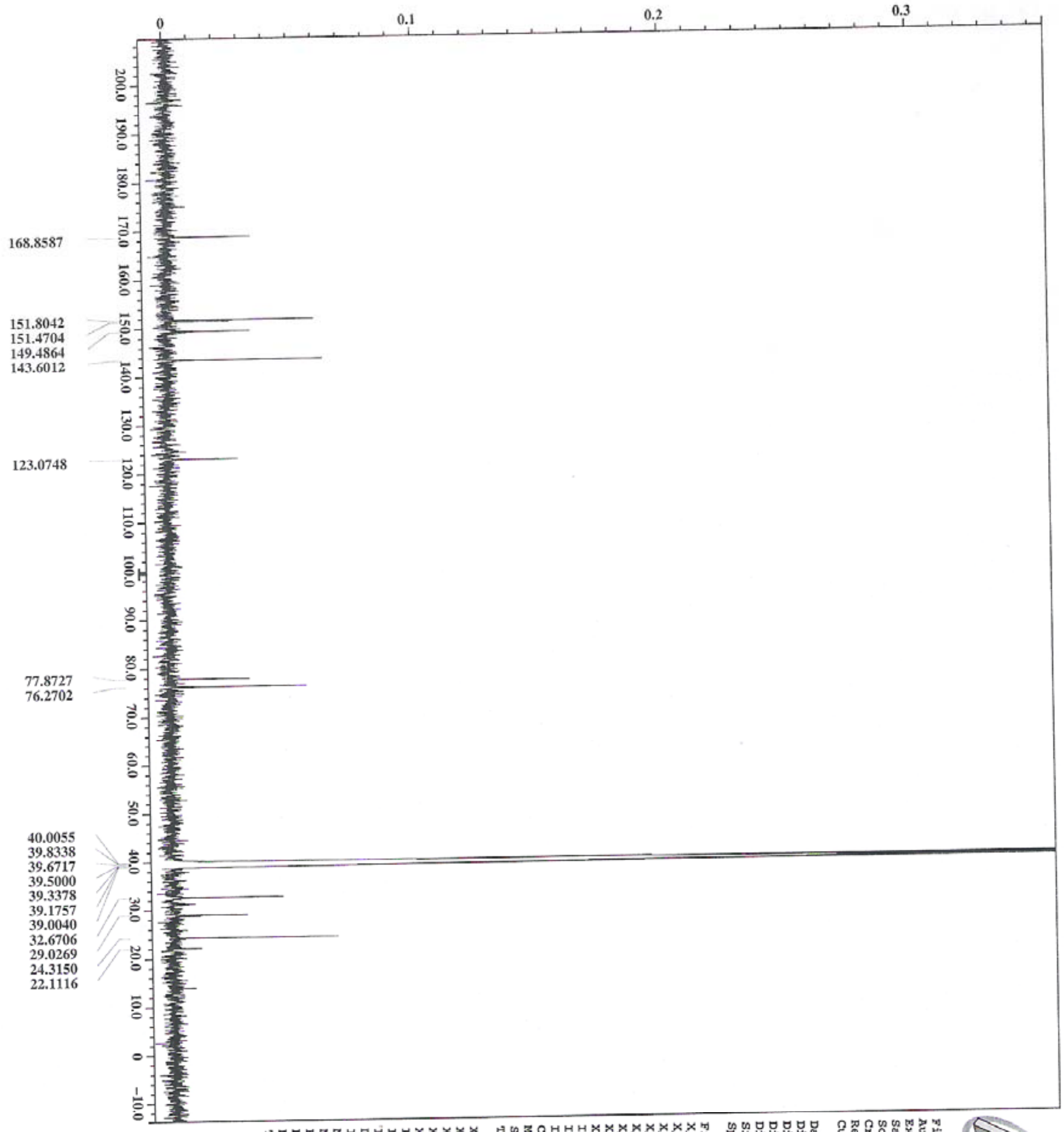
X_acq_duration    = 2.61619712[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 32768
X_prescans        = 2
X_resolution      = 0.38223419[Hz]
X_sweep           = 12.5250501[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 13.25[us]
X_acq_time        = 2.61619712[s]
X_angle           = 45[deg]
X_atn             = 3.99[dB]
X_pulse           = 6.625[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 48
Relaxation_delay  = 2[s]
Repetition_time   = 4.61619712[s]
Temp_get          = 19.9[dC]
```

Figure S12: ^{13}C NMR of 6-*N*-acetyl-9-propargyladenine.



```
Filename          = PROACAD_CARBON-4.jdf
Author            = N.Ahmed
Experiment        = single_pulse_dec
Sample_id         = S#531903
Solvent           = DMSO-D6
Creation_time     = 29-DEC-2009 14:53:57
Revision_time     = 14-JAN-2010 12:58:47
Current_time      = 14-JAN-2010 12:58:55

Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1024
Total_scans       = 1024

X_90_width        = 9.62[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 7.1[dB]
X_pulse           = 3.20666667[us]
Irr_atn_dec       = 21.5[dB]
Irr_atn_noe       = 21.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1.5[s]
Recvr_gain        = 60
Relaxation_delay  = 1.5[s]
Repetition_time   = 2.33361792[s]
Temp_get          = 22[dC]
```

Figure S13: HRMS 6-*N*-acetyl-9-propargyladenine.

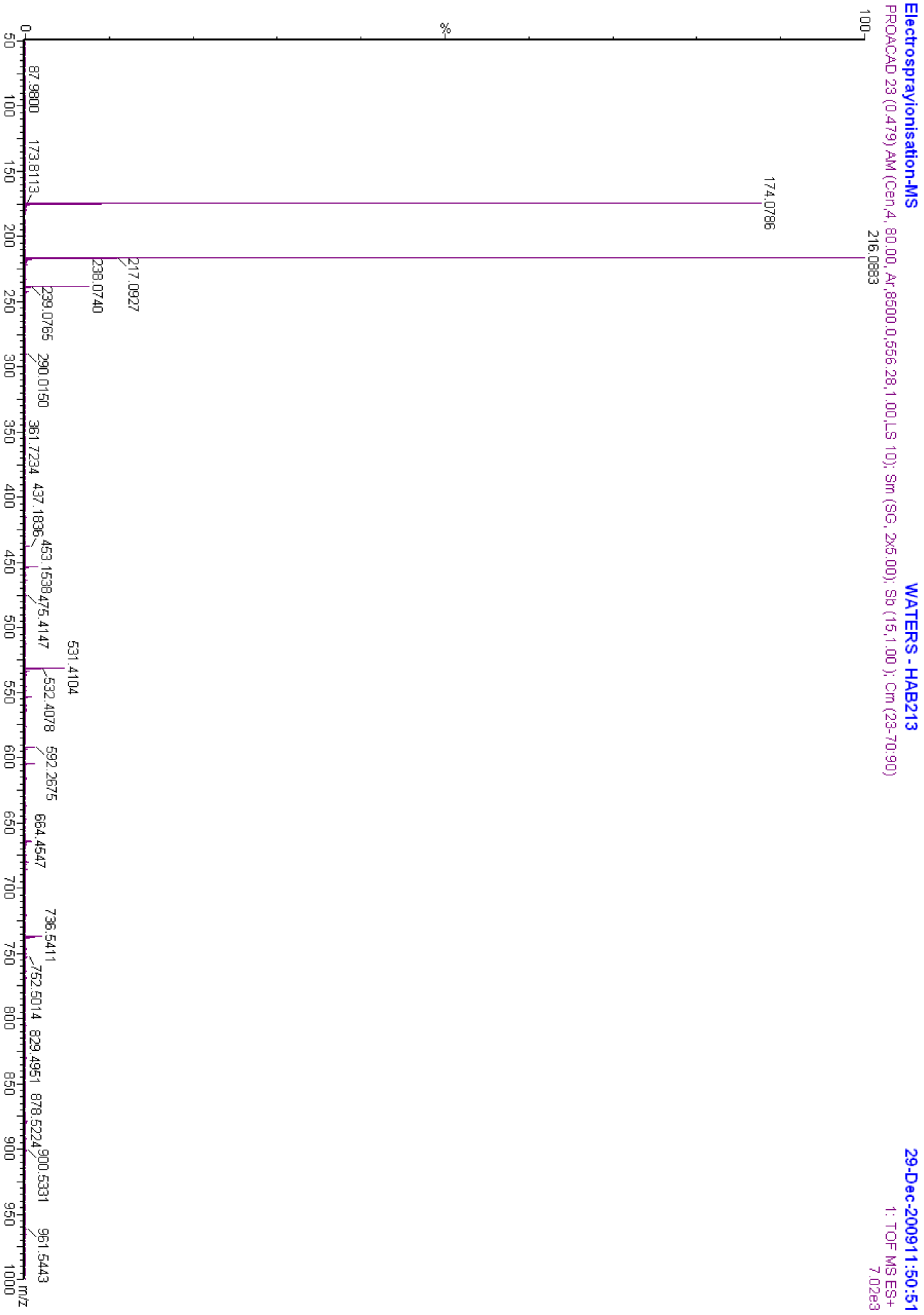


Figure S14: IR spectra of 9-propargyladenine (**1**)

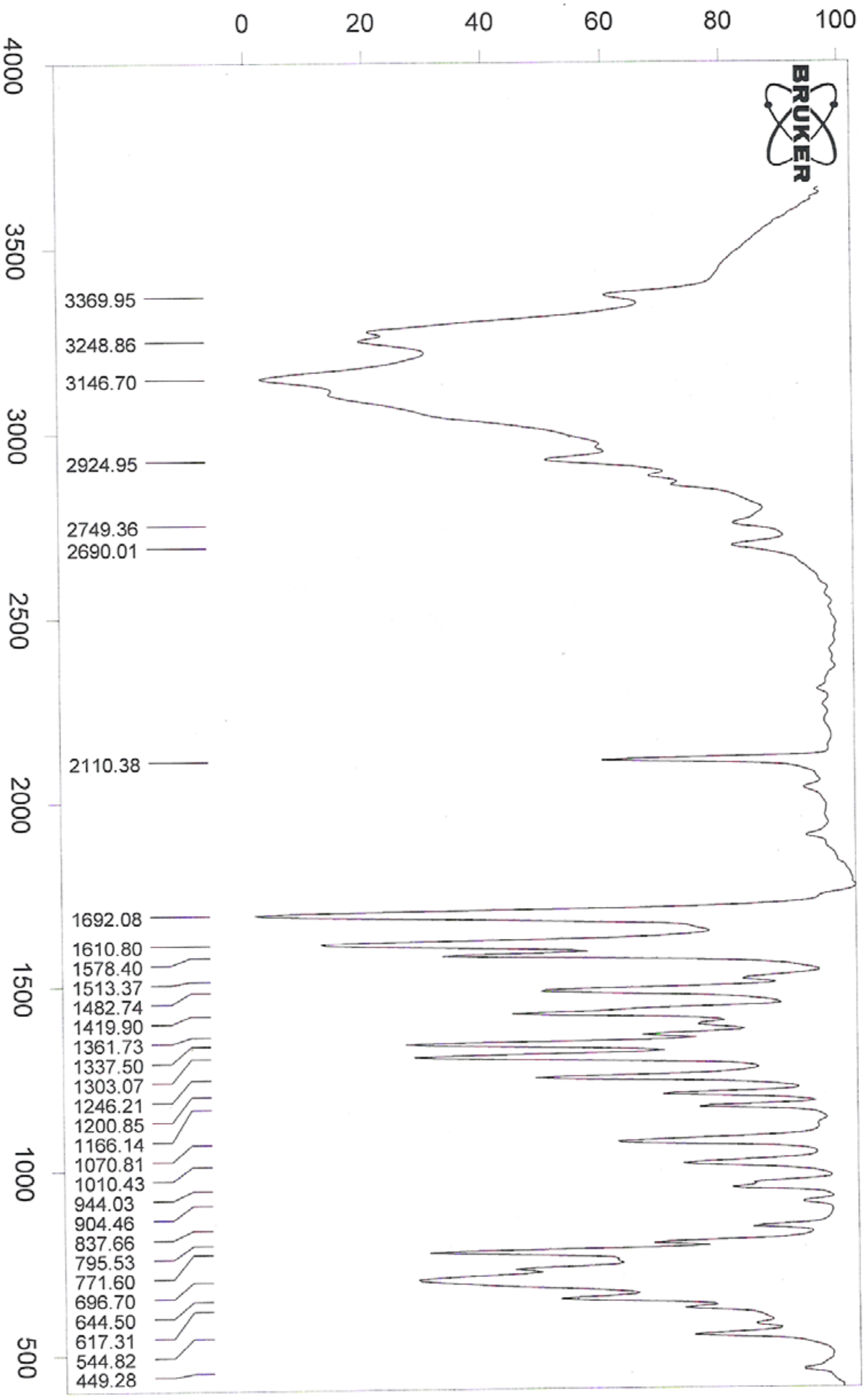
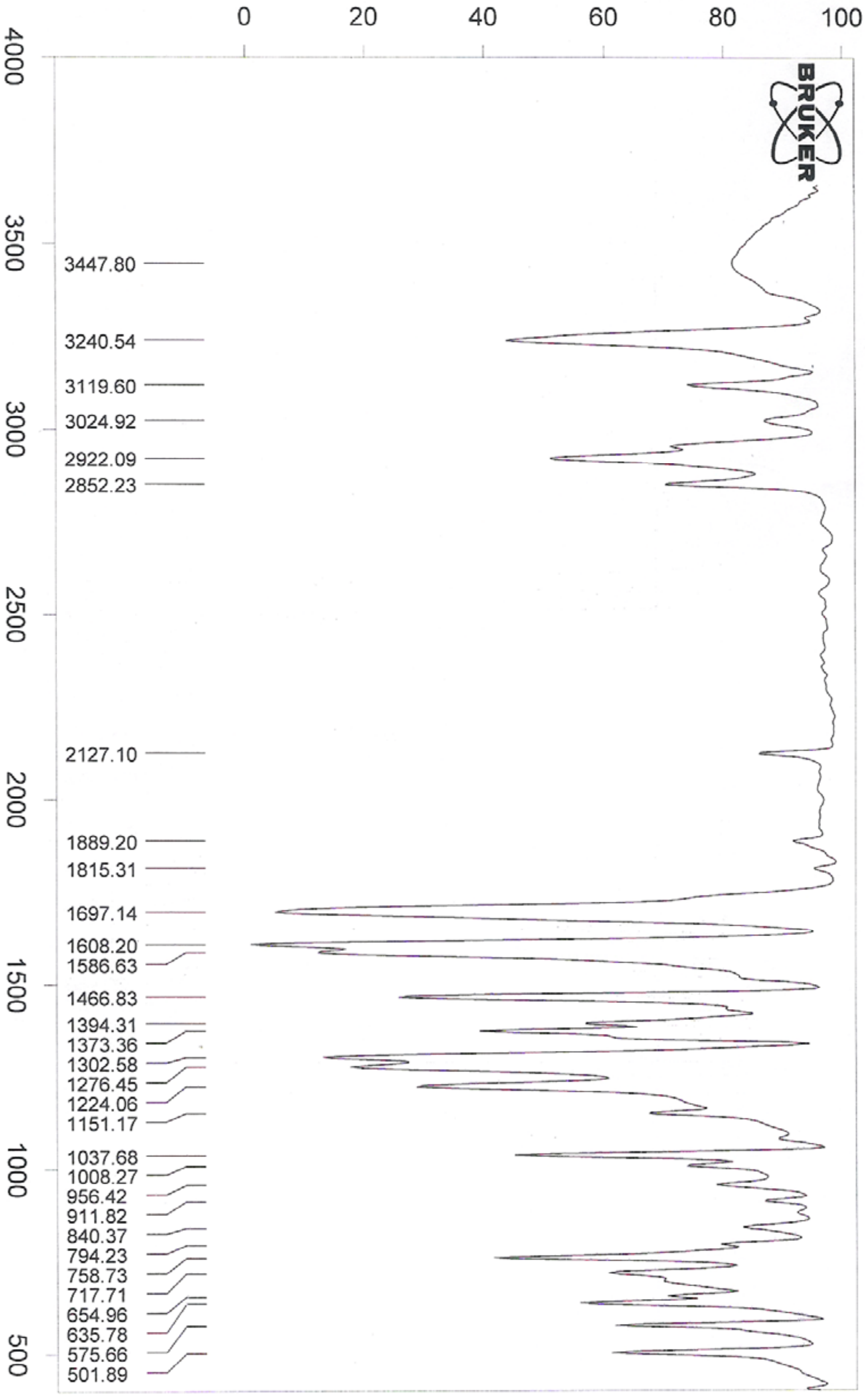


Figure S15: IR spectra of 6-N-acetyl-9-propargyladenine.



Reference:

1. R. V. Joshi, J. Zemlicka, *Tetrahedron* 1993, **49**, 2353.
2. *SAINT*+, 6.02 ed.; Bruker AXS, Madison, WI, 1999.
3. G. M. Sheldrick,. *SADABS 2.0*; University of Göttingen: Göttingen, Germany, 2000.
4. G. M. Sheldrick, *SHELXL-97: Program for Crystal Structure Refinement*; University of Göttingen: Göttingen, Germany, 1997.