Supplementary Material
A new methodology for the synthesis of *N*-acylbenzotriazoles

Anoop S. Singh, Anand K. Agrahari, Mala Singh, Nidhi Mishra and Vinod K. Tiwari*

Department of Chemistry, Institute of Science, Banaras Hindu University,
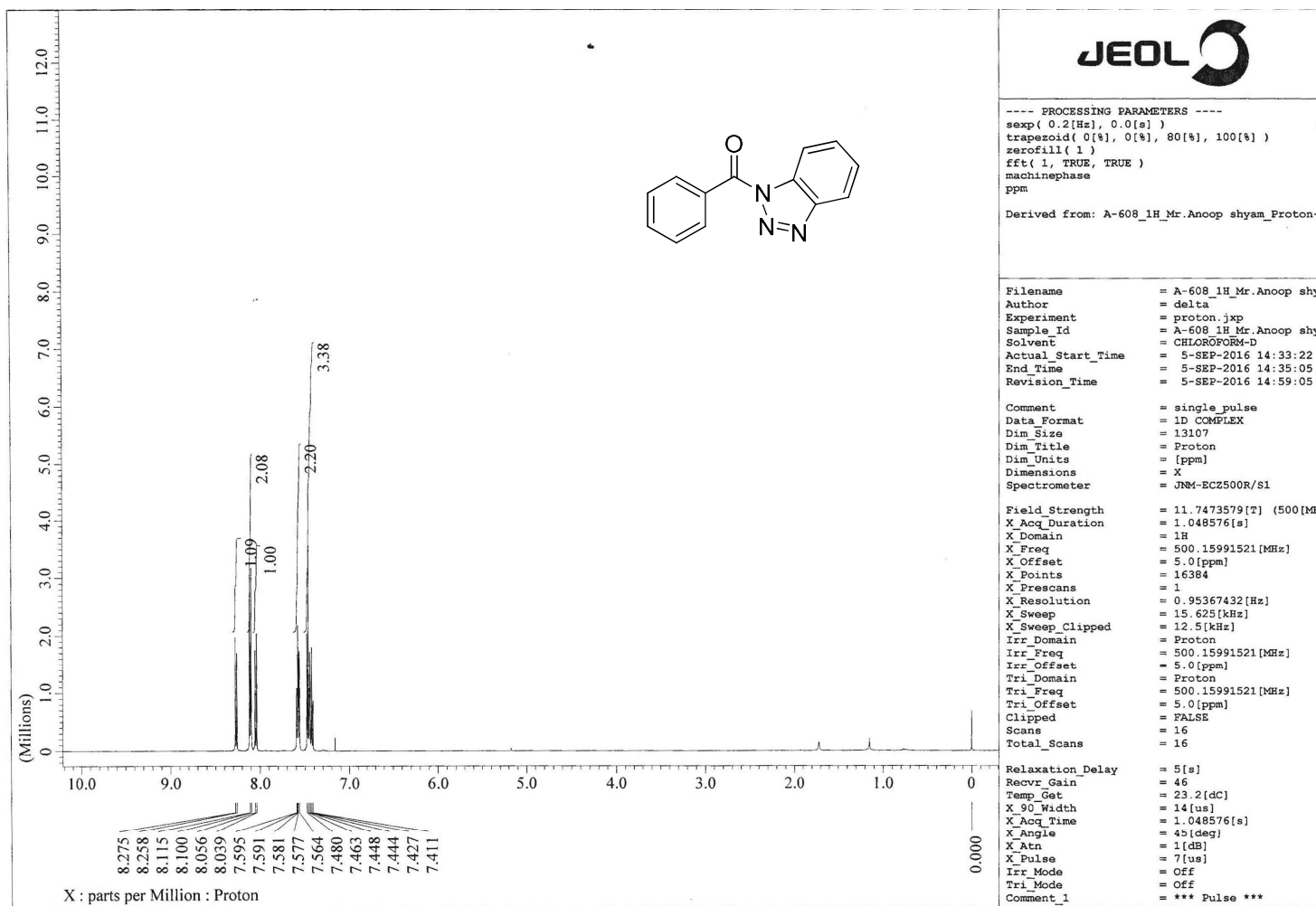*Varanasi, Uttar Pradesh-221 005, India*
Email: Tiwari_chem@yahoo.co.in

Dedicated to the late Prof. Alan R Katritzky for his excellent contributions to benzotriazole chemistry
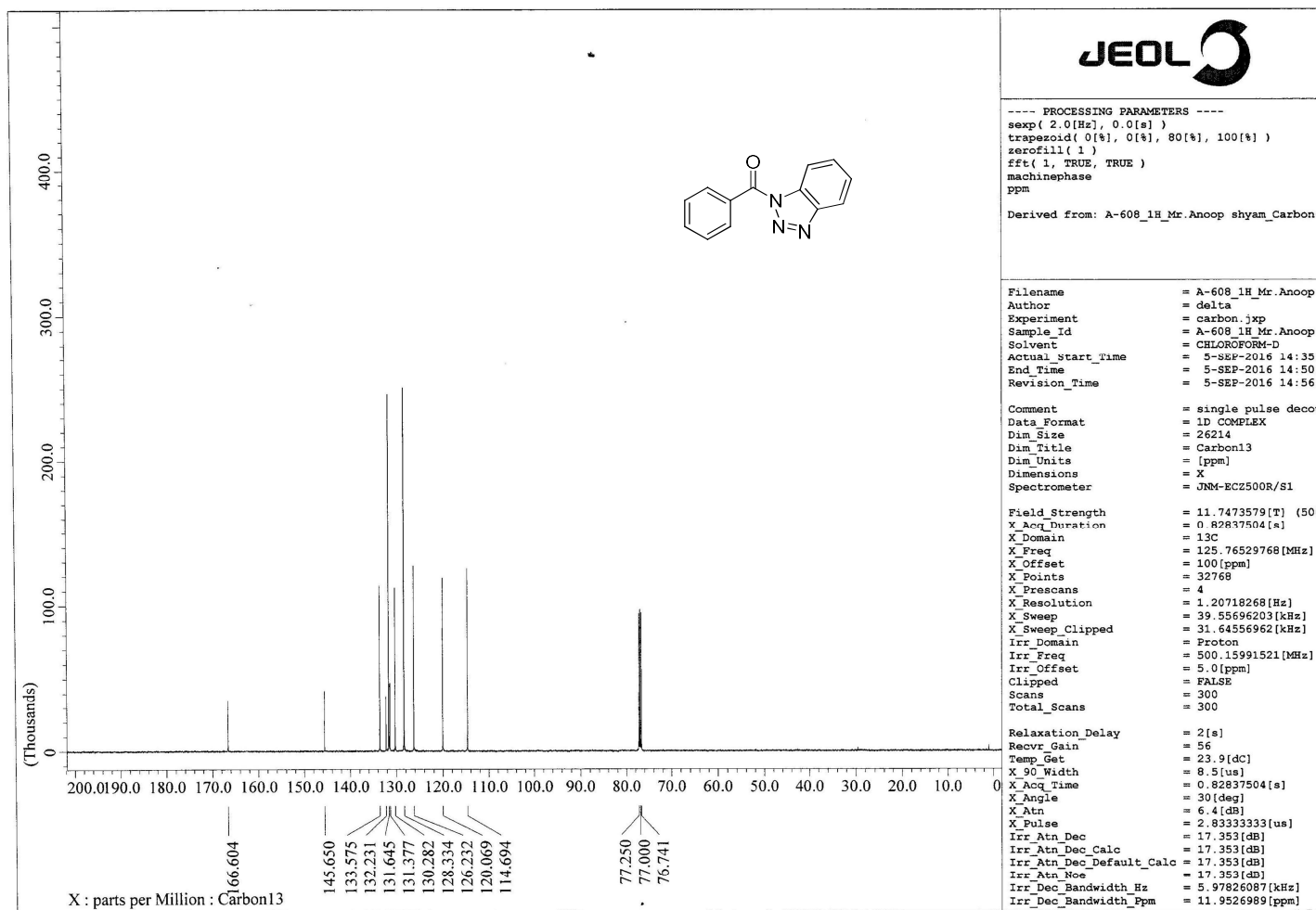
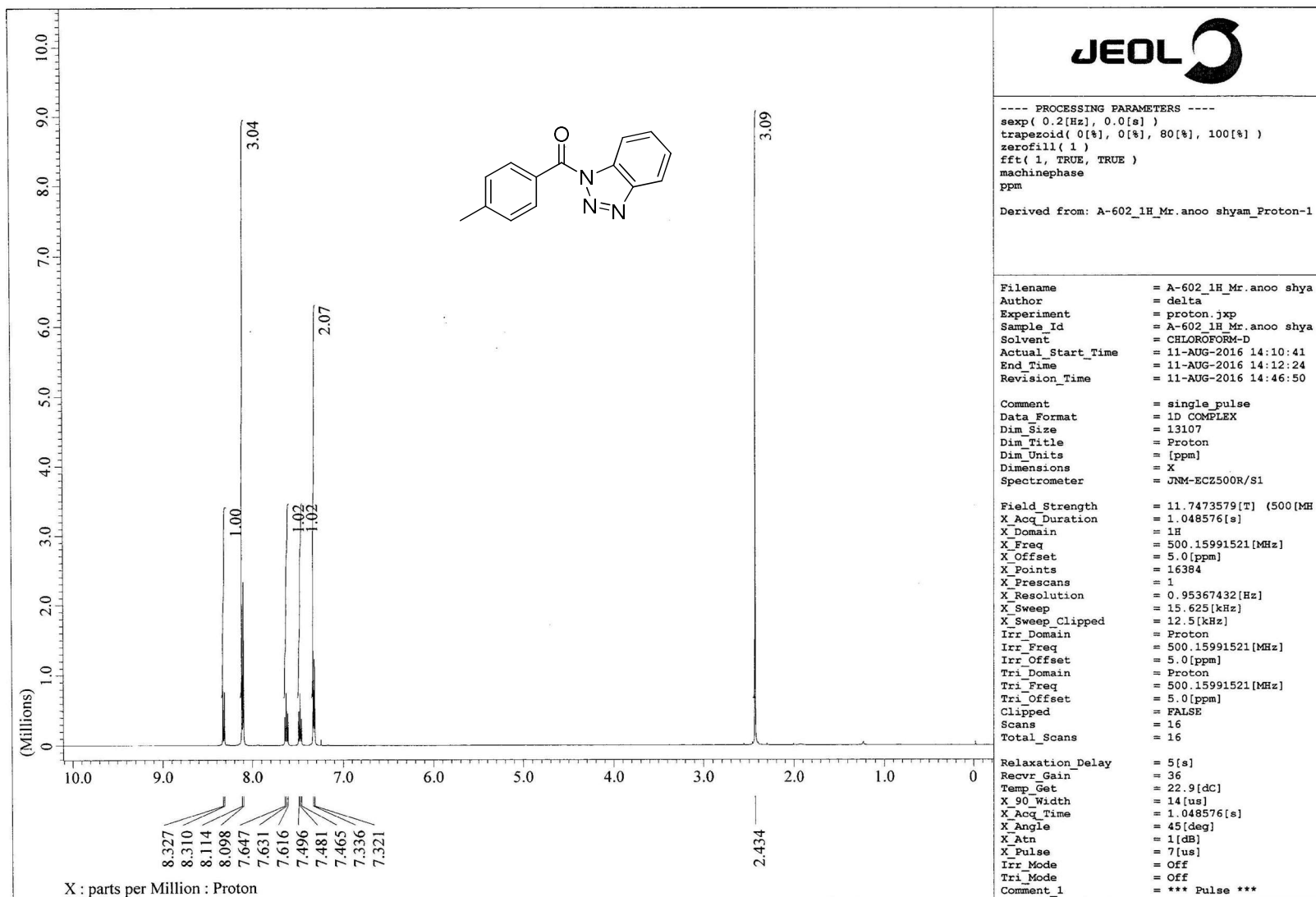**Reference**

[1] Kale, R. R.; Prasad, V.; Tiwari, V. K. *Lett. Org. Chem*. **2010**, *7*, 136.

[2] Singh, A. S.; Kumar, D.; Mishra, N.; Tiwari, V. K. *RSC Adv.* **2016**, *6*, 84512.

[3] Katritzky, A. R.; Suzuki, K.; Singh, S. K.; He, H-Y. *J. Org. Chem.* **2003** 68, 5720

Spectrum 1: ^{1}H NMR (500 MHz, CDCl$_3$) of compound **2a**

Spectrum 2: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2a**

Spectrum 3: ^{1}H NMR (500 MHz, CDCl$_3$) of compound **2b**

```
JEOL

---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: A-602_1H_Mr.anoo shyam_Carbon-1

Filename                      = A-602_1H_Mr.anoo s
Author                        = delta
Experiment                    = carbon.jxp
Sample_Id                     = A-602_1H_Mr.anoo s
Solvent                       = CHLOROFORM-D
Actual_Start_Time             = 11-AUG-2016 14:13:
End_Time                      = 11-AUG-2016 14:27:
Revision_Time                 = 11-AUG-2016 14:47:

Comment                       = single pulse decou
Data_Format                   = 1D COMPLEX
Dim_Size                      = 26214
Dim_Title                     = Carbon13
Dim_Units                     = [ppm]
Dimensions                    = X
Spectrometer                  = JNM-ECZ500R/S1

Field_Strength                = 11.7473579[T] (500
X_Acq_Duration                = 0.82837504[s]
X_Domain                      = 13C
X_Freq                        = 125.76529768[MHz]
X_Offset                      = 100[ppm]
X_Points                      = 32768
X_Prescans                    = 4
X_Resolution                  = 1.20718268[Hz]
X_Sweep                       = 39.55696203[kHz]
X_Sweep_Clipped               = 31.64556962[kHz]
Irr_Domain                    = Proton
Irr_Freq                      = 500.15991521[MHz]
Irr_Offset                    = 5.0[ppm]
Clipped                       = FALSE
Scans                         = 300
Total_Scans                   = 300

Relaxation_Delay              = 2[s]
Recvr_Gain                    = 56
Temp_Get                      = 23.7[dC]
X_90_Width                    = 8.5[us]
X_Acq_Time                    = 0.82837504[s]
X_Angle                       = 30[deg]
X_Atn                         = 6.4[dB]
X_Pulse                       = 2.83333333[us]
Irr_Atn_Dec                   = 17.353[dB]
Irr_Atn_Dec_Calc              = 17.353[dB]
Irr_Atn_Dec_Default_Calc      = 17.353[dB]
Irr_Atn_Noe                   = 17.353[dB]
Irr_Dec_Bandwidth_Hz          = 5.97826087[kHz]
Irr_Dec_Bandwidth_Ppm         = 11.9526989[ppm]
```
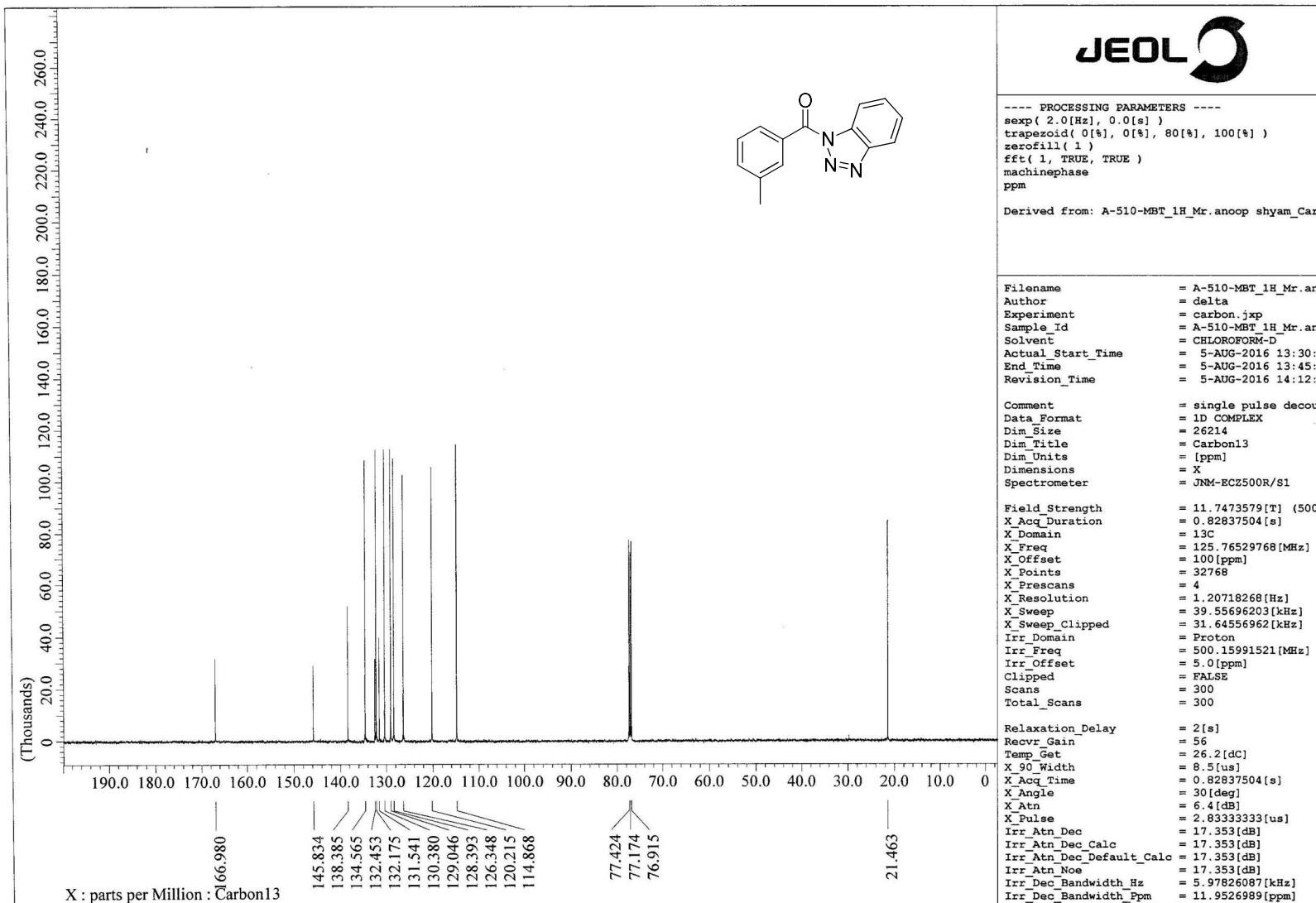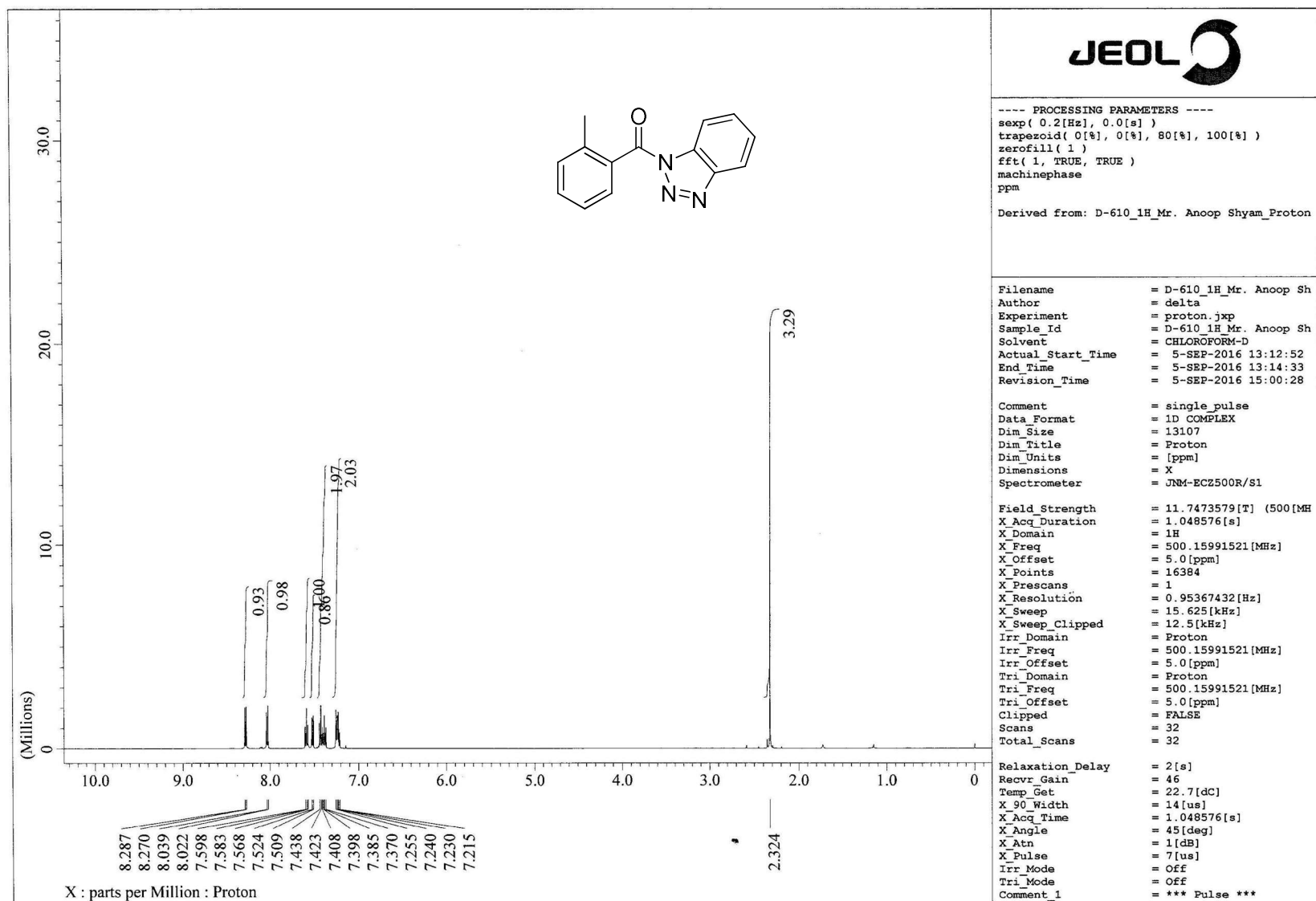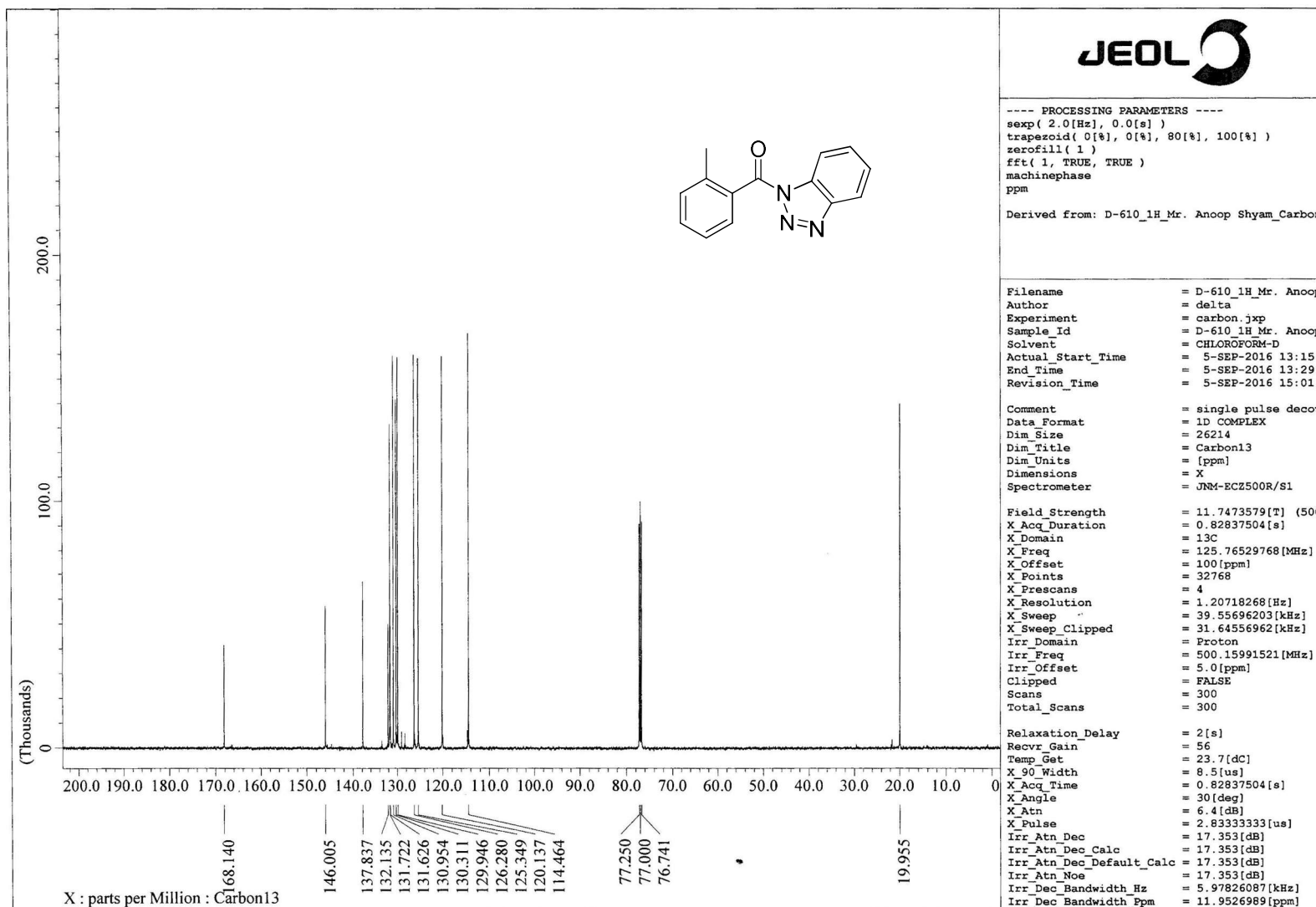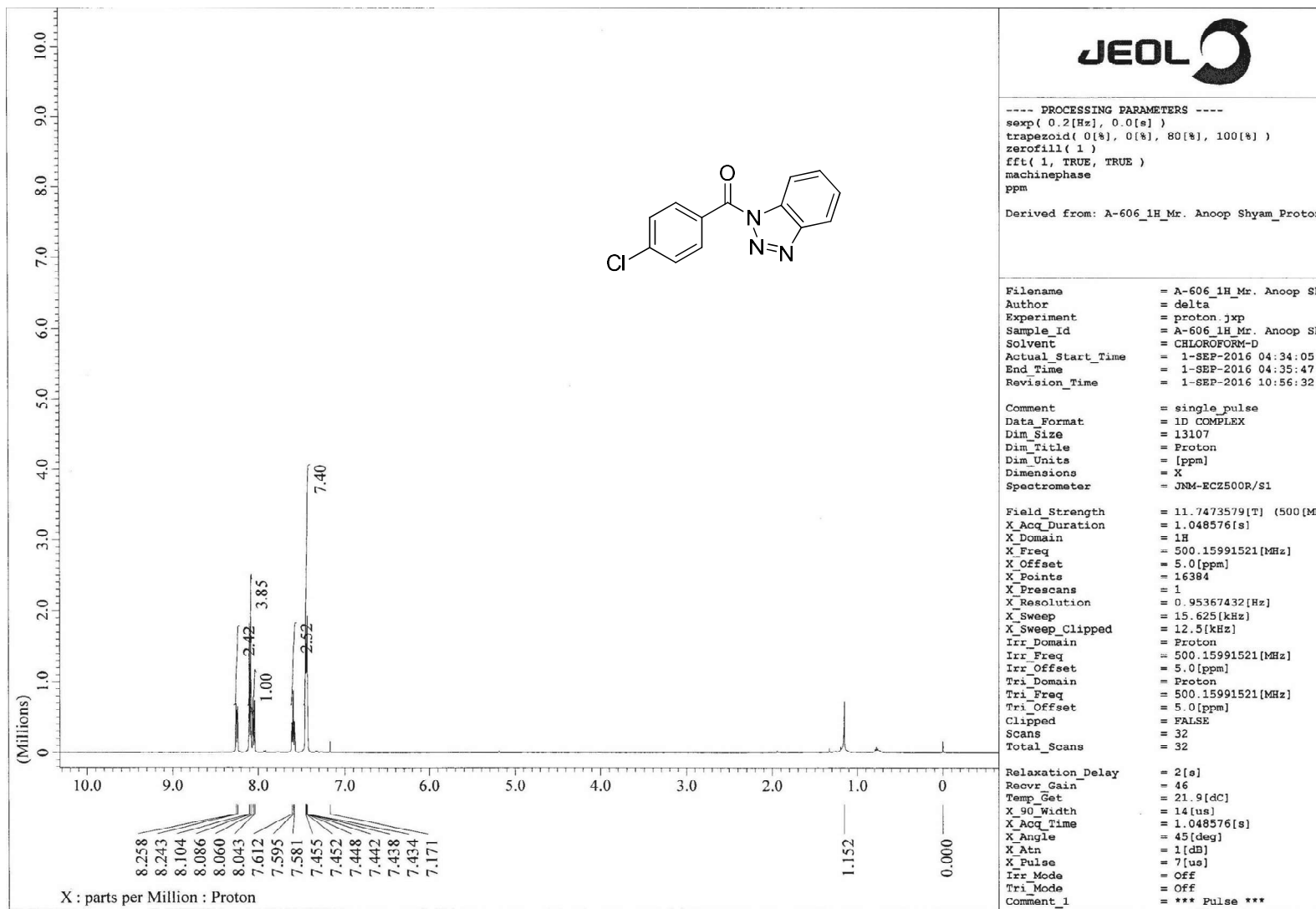
X : parts per Million : Carbon13

166.538 145.767 144.874 132.482 132.031 130.303 129.247 128.681 126.262 120.157 114.868 77.481 77.232 76.973 21.867

Spectrum 4: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2b**
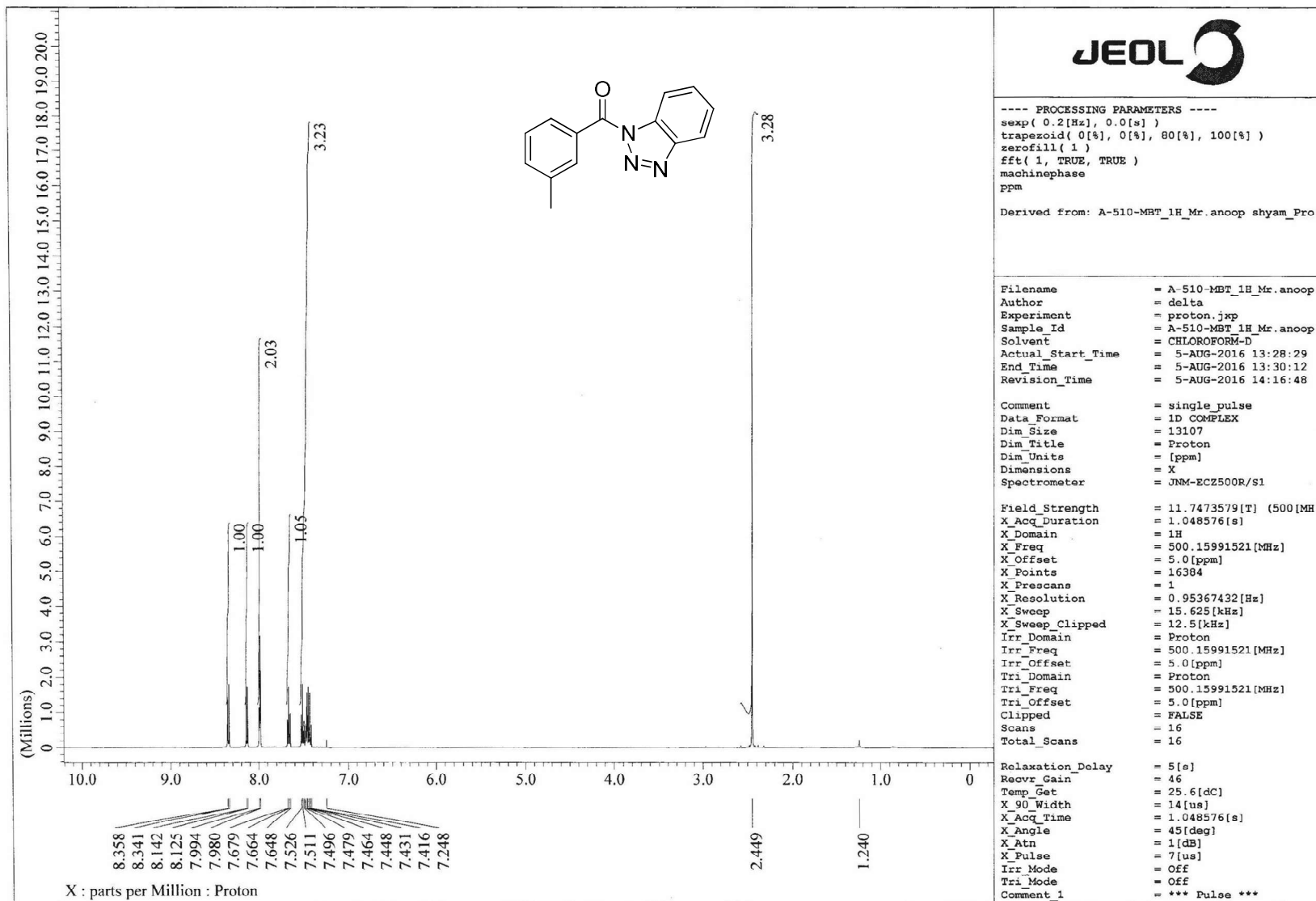
Spectrum 5: ^{1}H NMR (500 MHz, CDCl$_3$) of compound **2c**

Spectrum 6: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2c**

Spectrum 7: ^{1}H NMR (500 MHz, CDCl$_3$) of compound **2d**

Spectrum 8: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2d**

Spectrum 9: ^{1}H NMR (500 MHz, CDCl$_3$) of compound **2e**
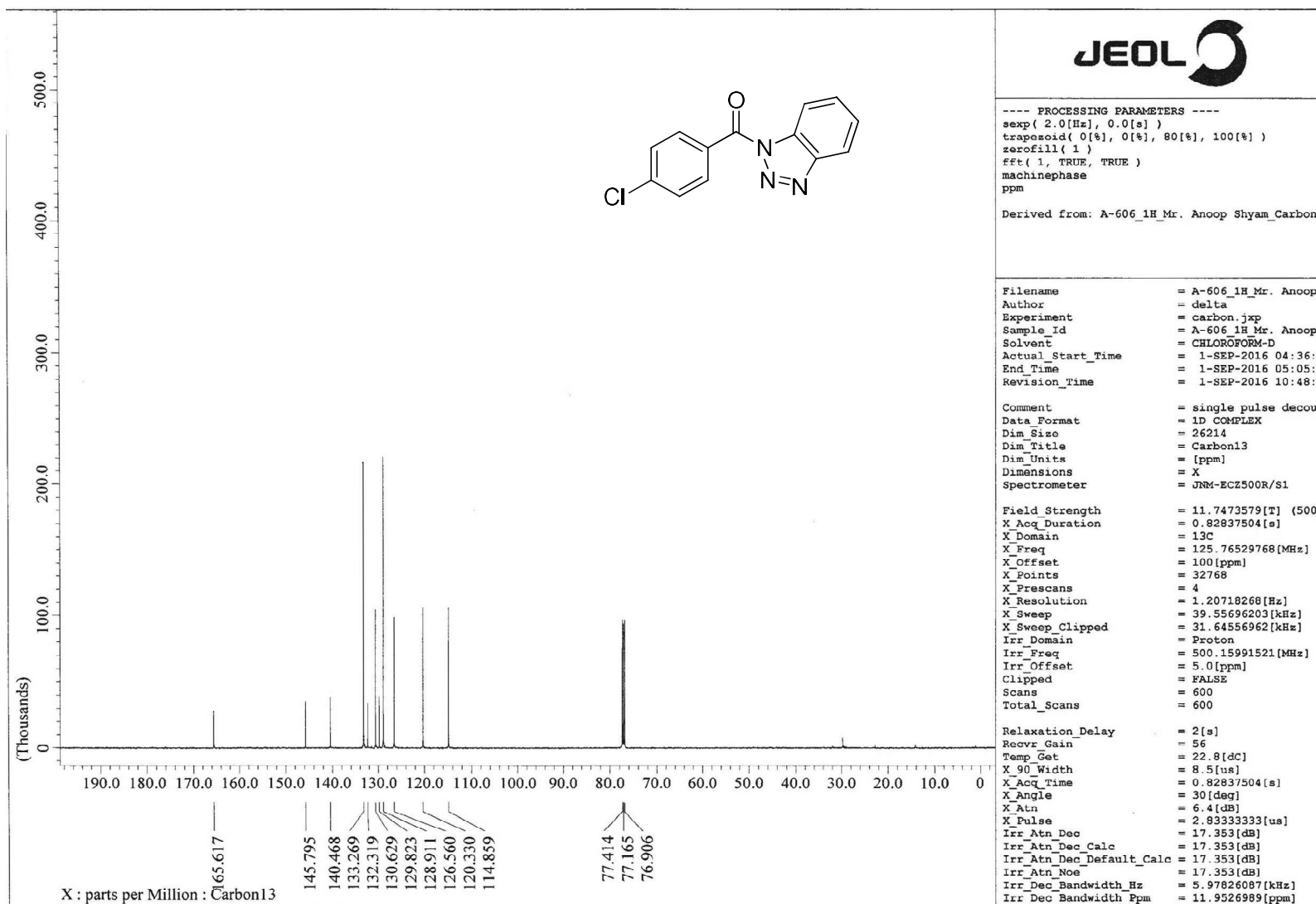
Spectrum 10: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2e**
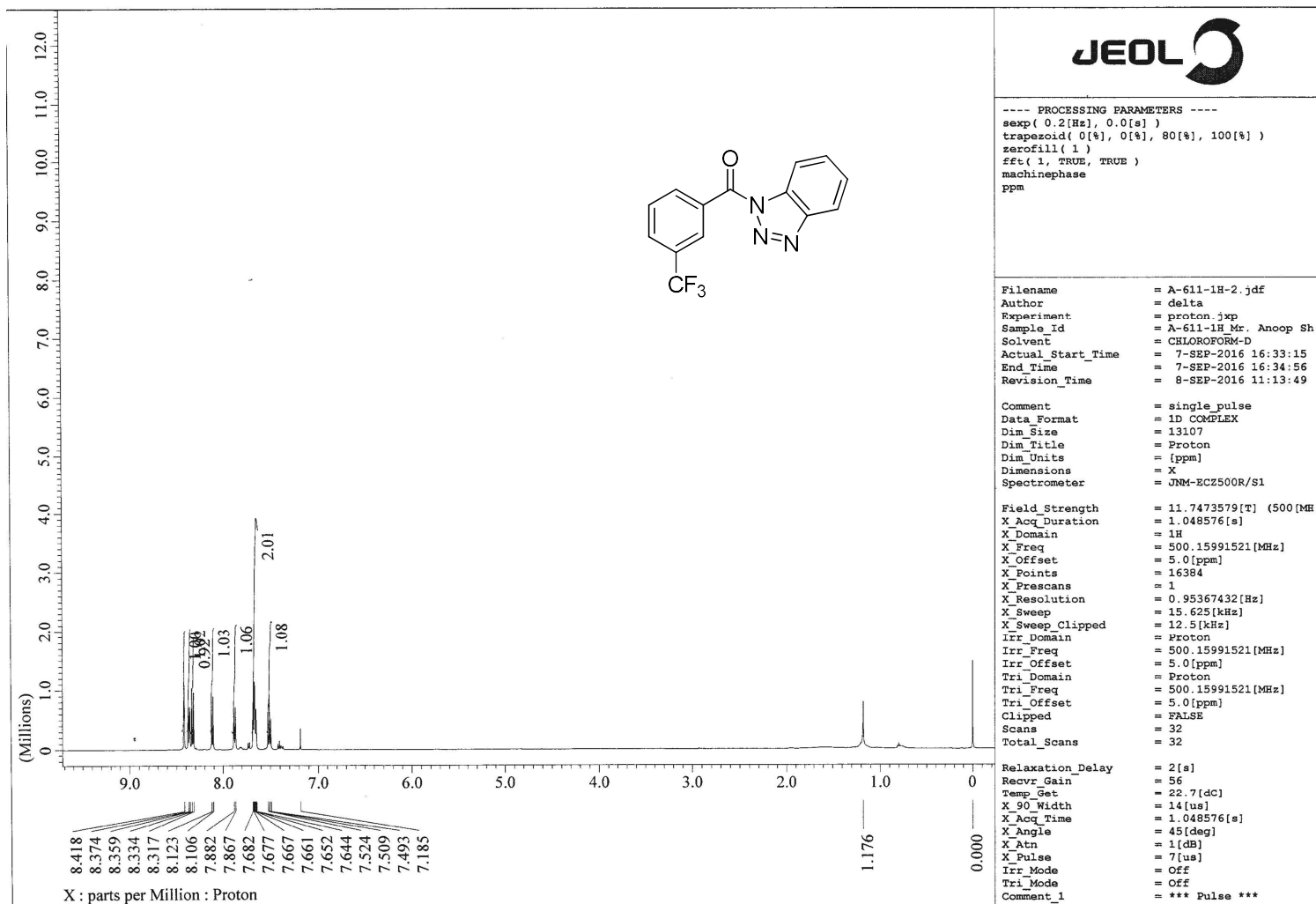
Spectrum 11: ^{1}H NMR (500 MHz, CDCl$_3$) of compound **2f**
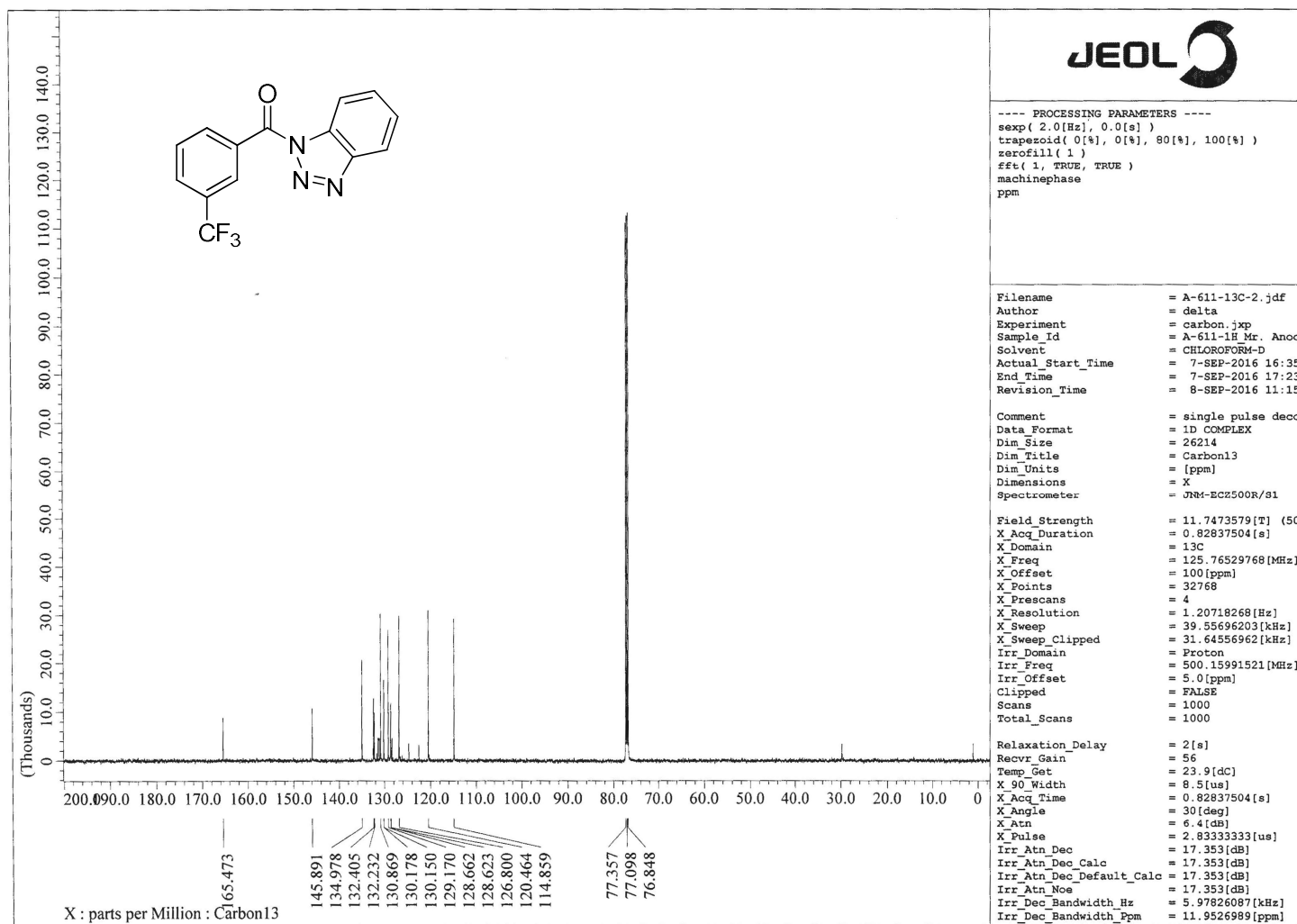
Spectrum 12: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2f**
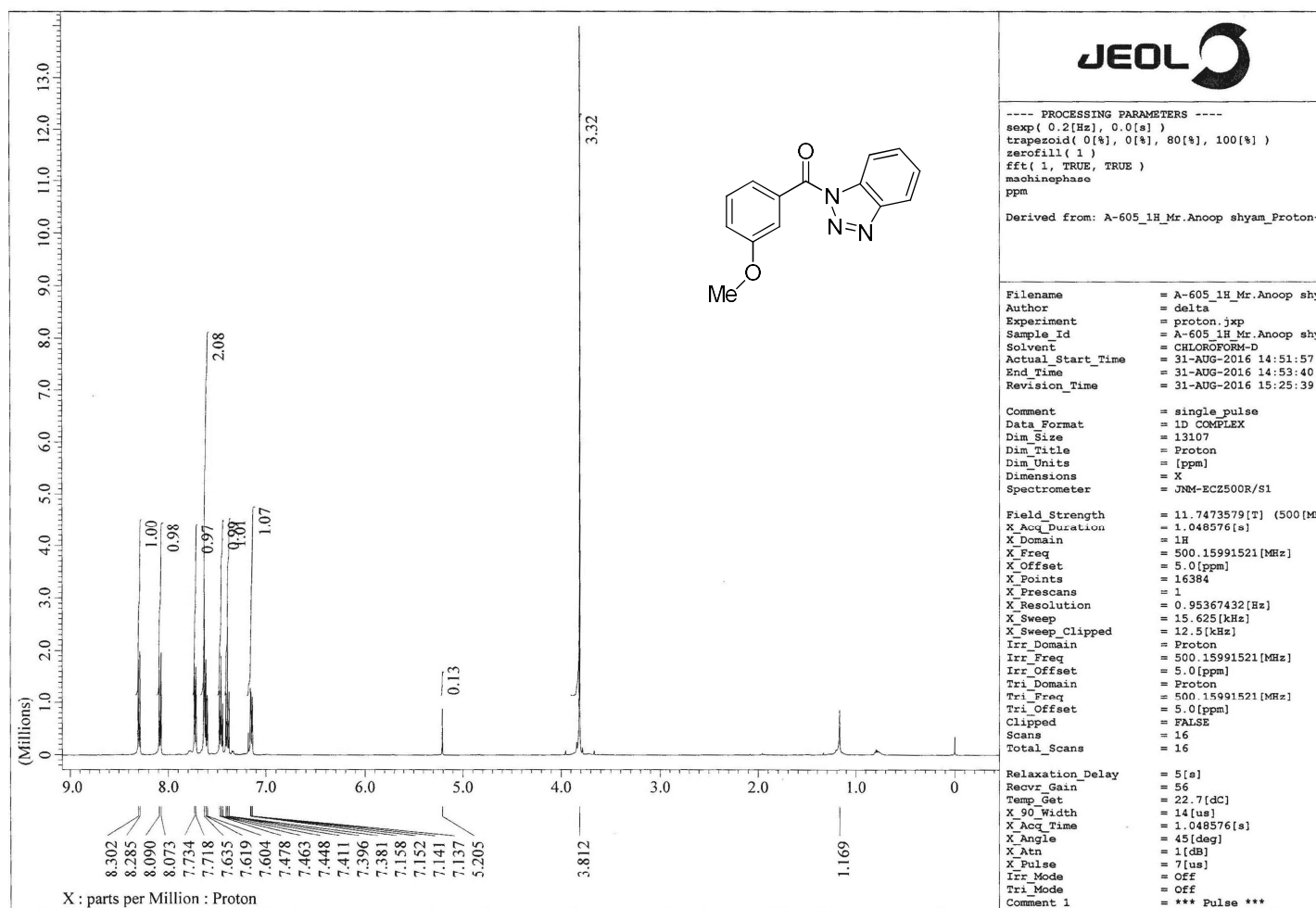
Spectrum 13: ^{1}H NMR (500 MHz, CDCl₃) of compound **2g**
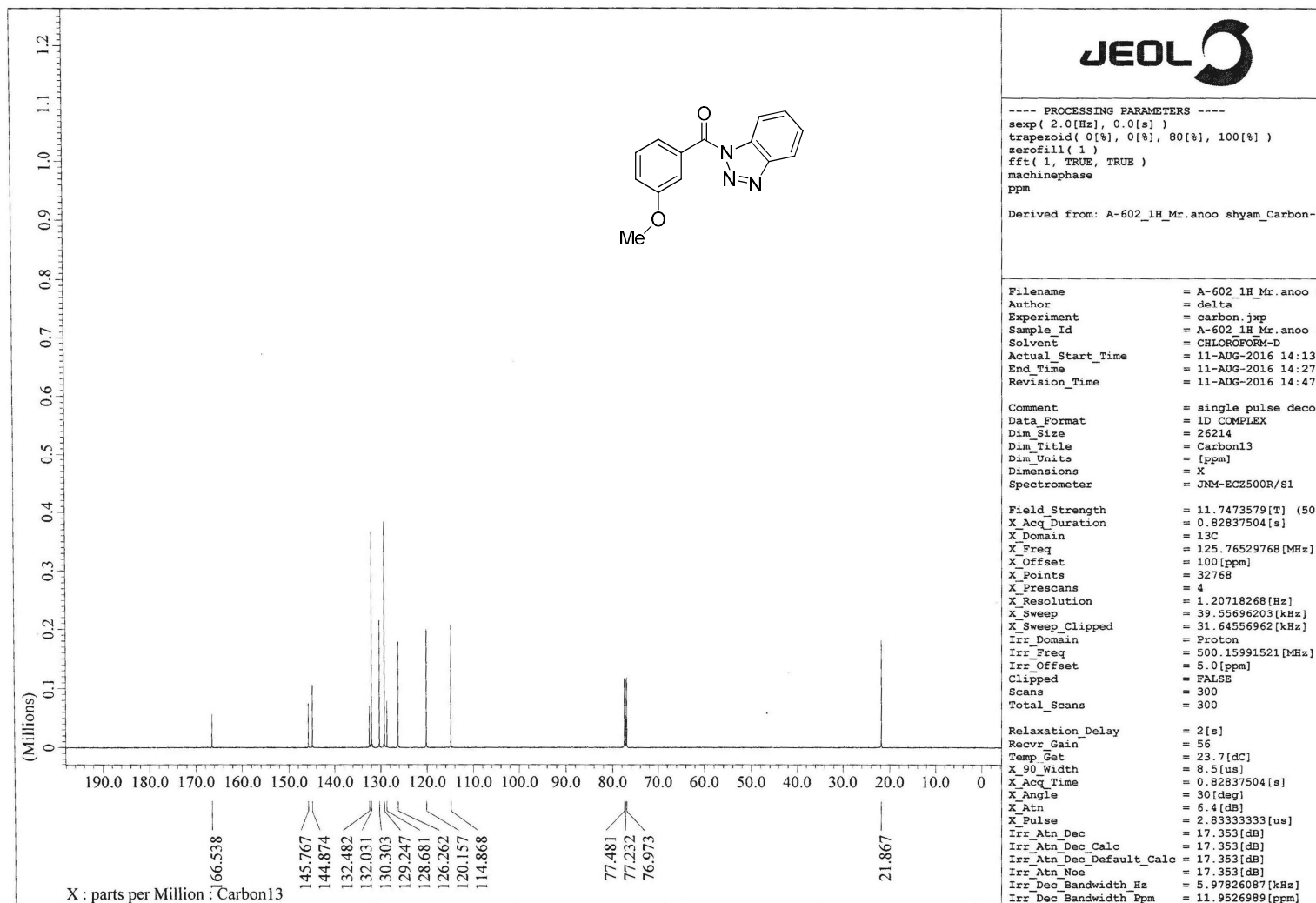
Spectrum 14: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2g**
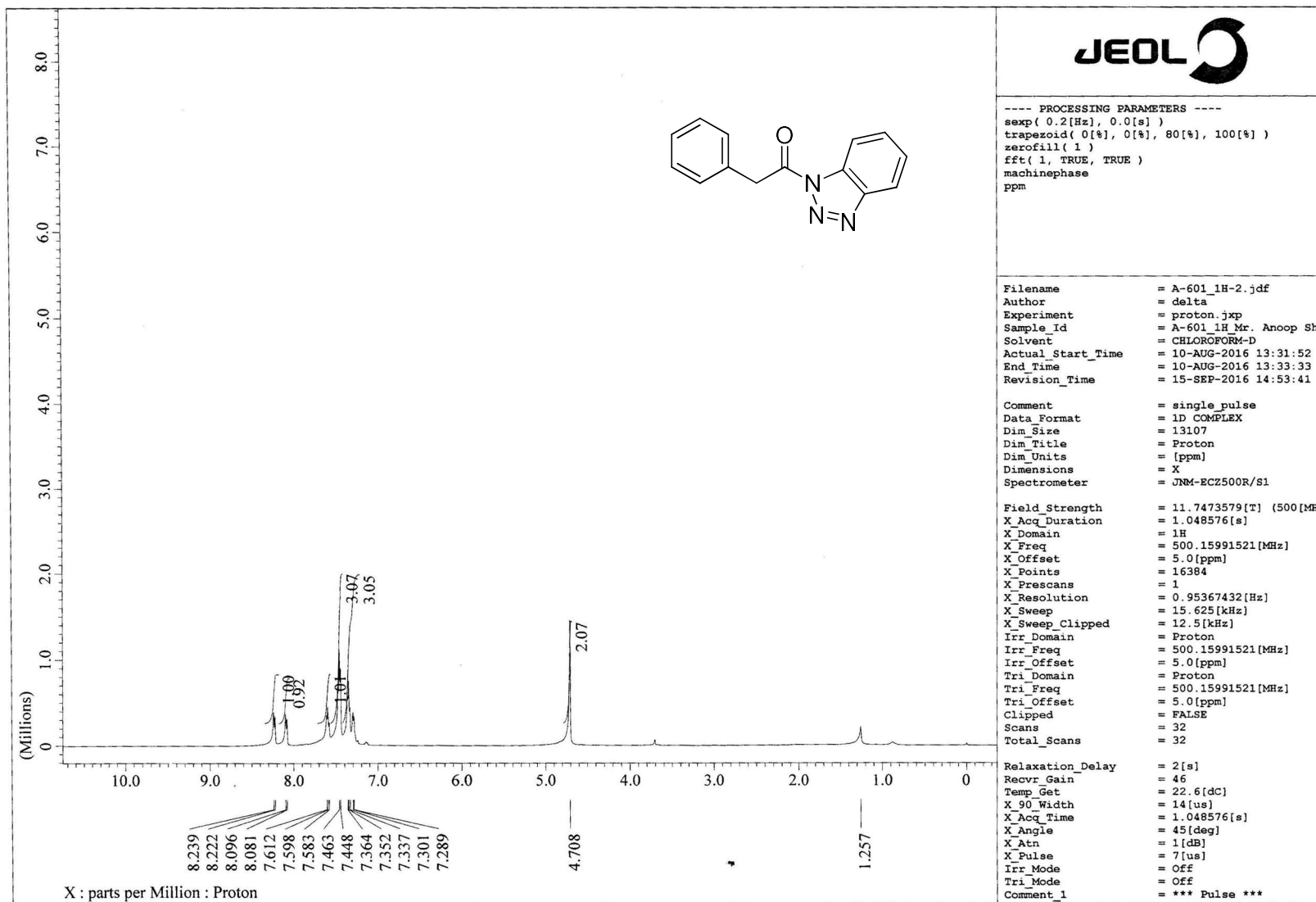
Spectrum 15: ^{1}H NMR (500 MHz, CDCl$_3$) of compound **2h**
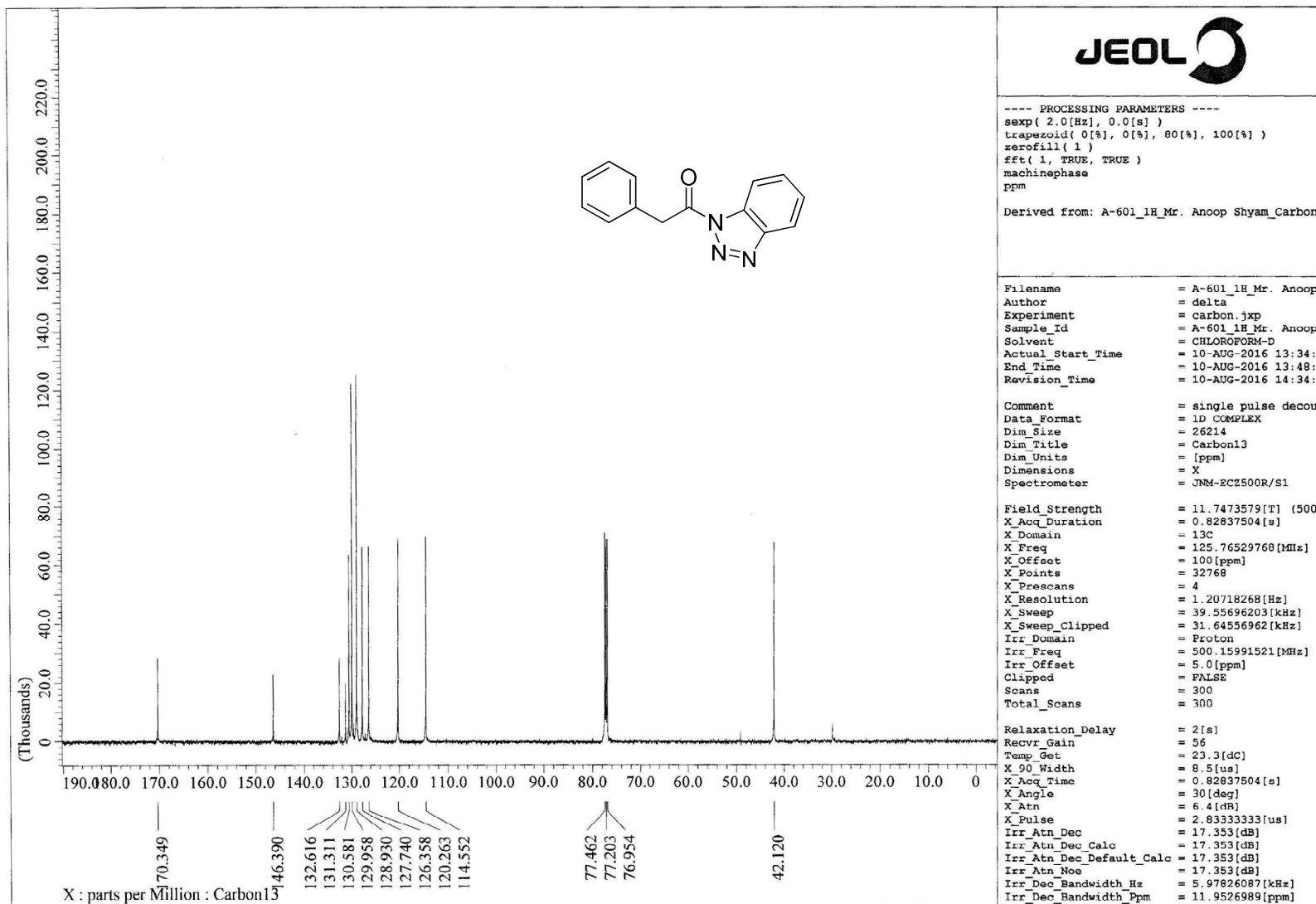
Spectrum 16: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2h**
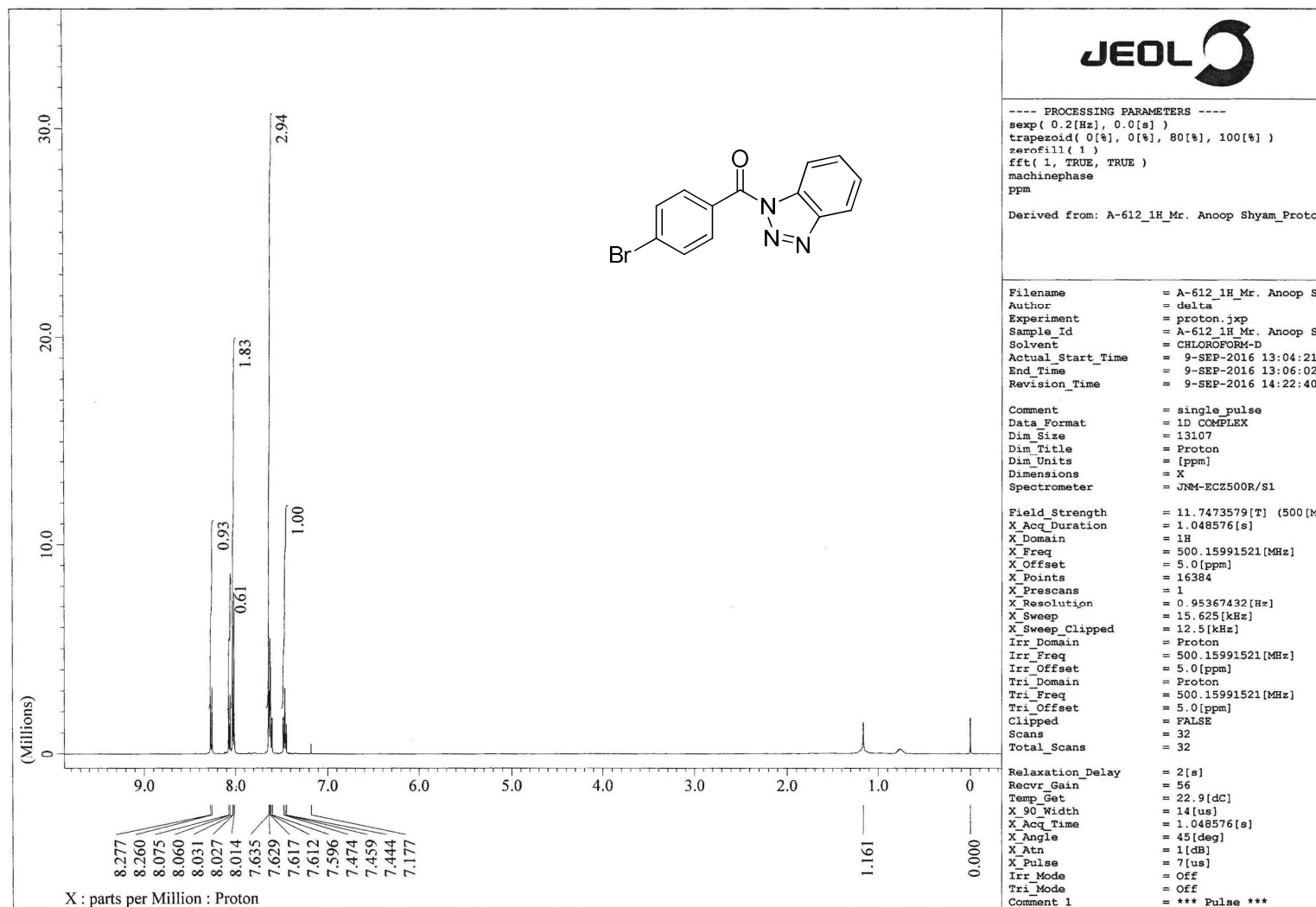
Spectrum 17: ^{1}H NMR (500 MHz, CDCl$_3$) of compound **2i**
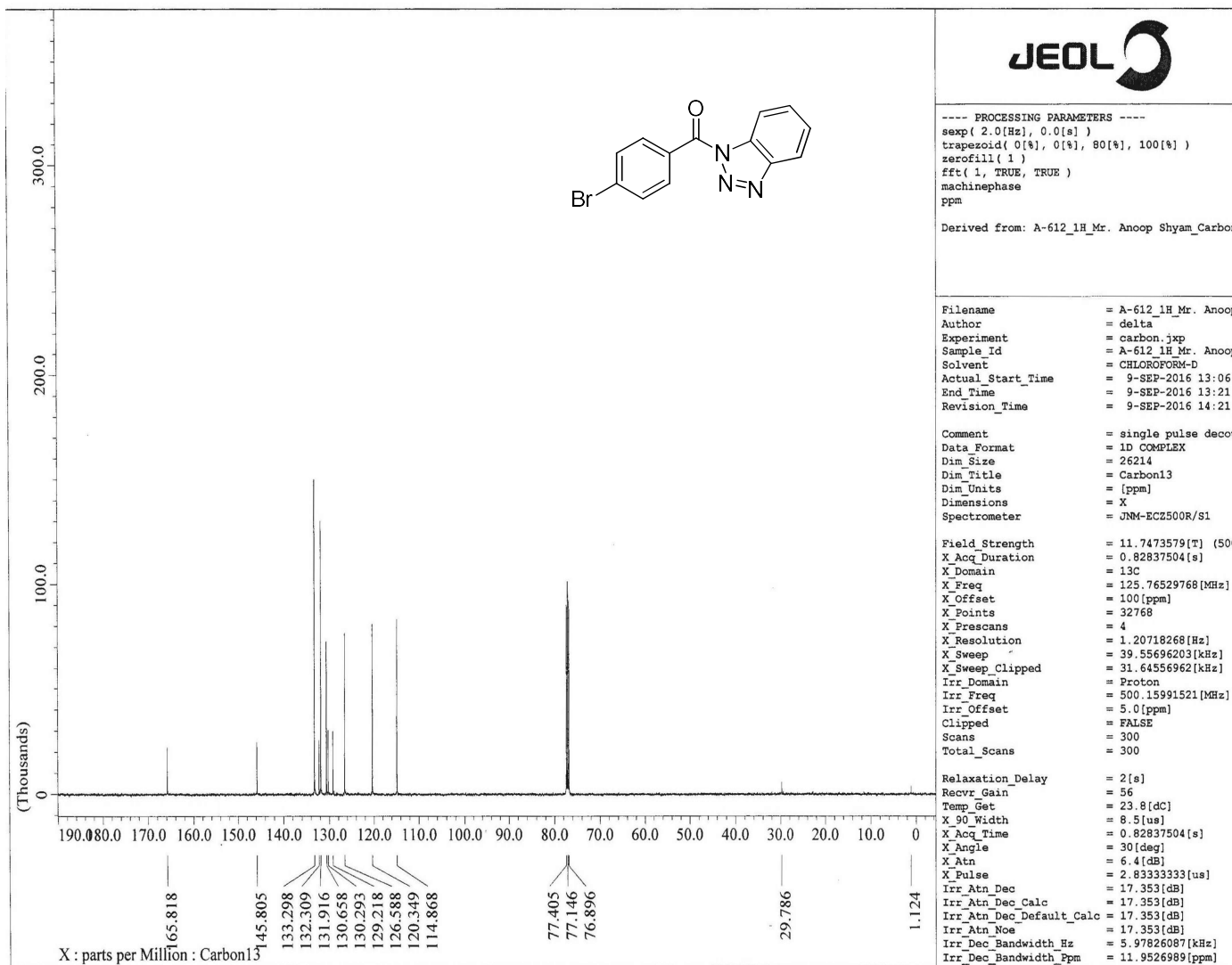
Spectrum 18: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2i**
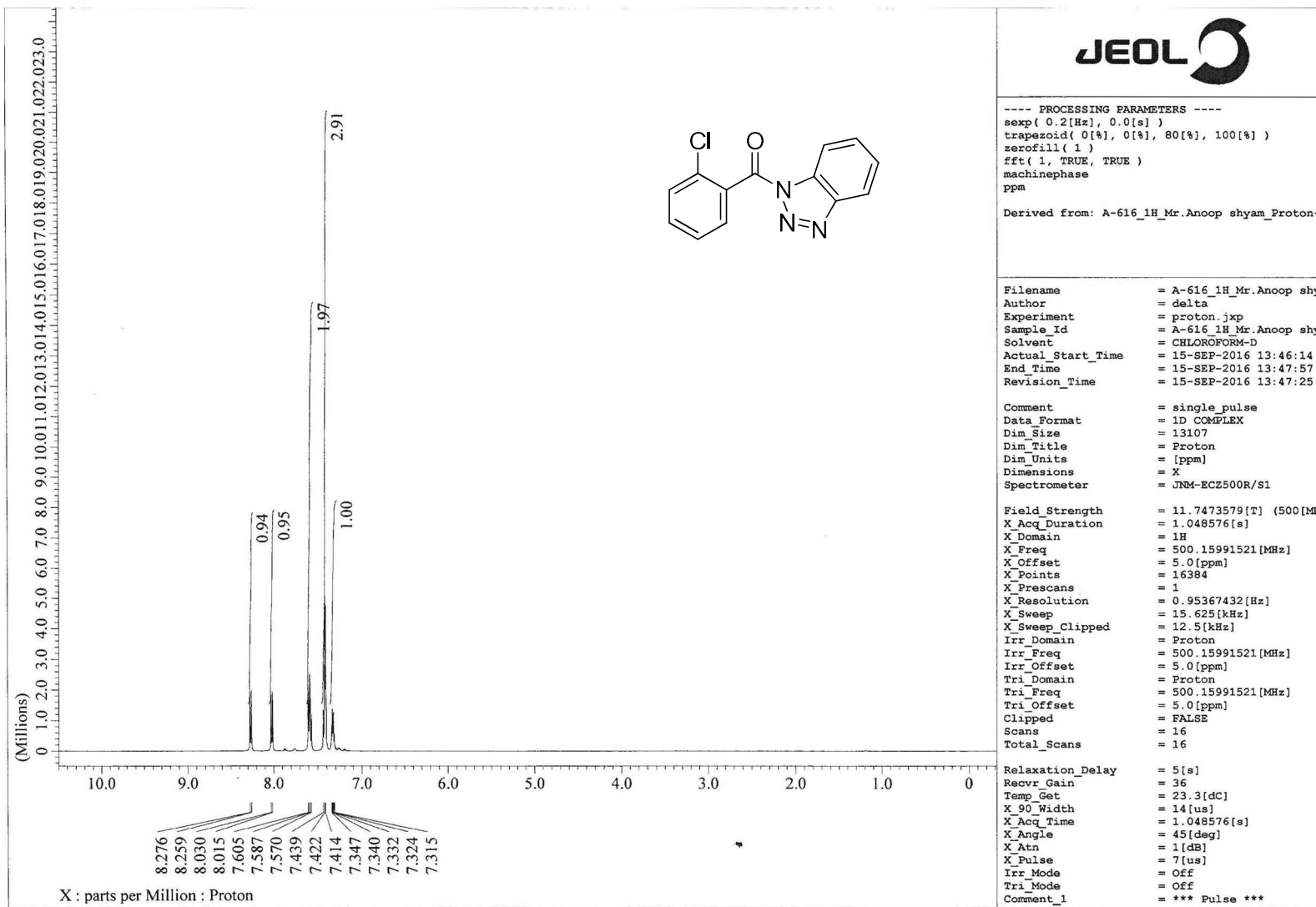
Spectrum 19: ^{1}H NMR (500 MHz, CDCl$_3$) of compound **2j**

---- PROCESSING PARAMETERS ----
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

Derived from: A-616_1H_Mr.Anoop shyam_Carbon-

Filename   = A-616_1H_Mr.Anoop
Author   = delta
Experiment   = carbon.jxp
Sample_Id   = A-616_1H_Mr.Anoop
Solvent   = CHLOROFORM-D
Actual_Start_Time   = 15-SEP-2016 13:48:
End_Time   = 15-SEP-2016 14:03:
Revision_Time   = 15-SEP-2016 13:59:

Comment   = single pulse decou
Data_Format   = 1D COMPLEX
Dim_Size   = 26214
Dim_Title   = Carbon13
Dim_Units   = [ppm]
Dimensions   = X
Spectrometer   = JNM-ECZ500R/S1

Field_Strength   = 11.7473579[T] (500
X_Acq_Duration   = 0.82837504[s]
X_Domain   = 13C
X_Freq   = 125.76529768[MHz]
X_Offset   = 100[ppm]
X_Points   = 32768
X_Prescans   = 4
X_Resolution   = 1.20718268[Hz]
X_Sweep   = 39.55696203[kHz]
X_Sweep_Clipped   = 31.64556962[kHz]
Irr_Domain   = Proton
Irr_Freq   = 500.15991521[MHz]
Irr_Offset   = 5.0[ppm]
Clipped   = FALSE
Scans   = 300
Total_Scans   = 300

Relaxation_Delay   = 2[s]
Recvr_Gain   = 56
Temp_Get   = 24.5[dC]
X_90_Width   = 8.5[us]
X_Acq_Time   = 0.82837504[s]
X_Angle   = 30[deg]
X_Atn   = 6.4[dB]
X_Pulse   = 2.83333333[us]
Irr_Atn_Dec   = 17.353[dB]
Irr_Atn_Dec_Calc   = 17.353[dB]
Irr_Atn_Dec_Default_Calc = 17.353[dB]
Irr_Atn_Noe   = 17.353[dB]
Irr_Dec_Bandwidth_Hz = 5.97826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.9526989[ppm]

Spectrum 20: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2j**
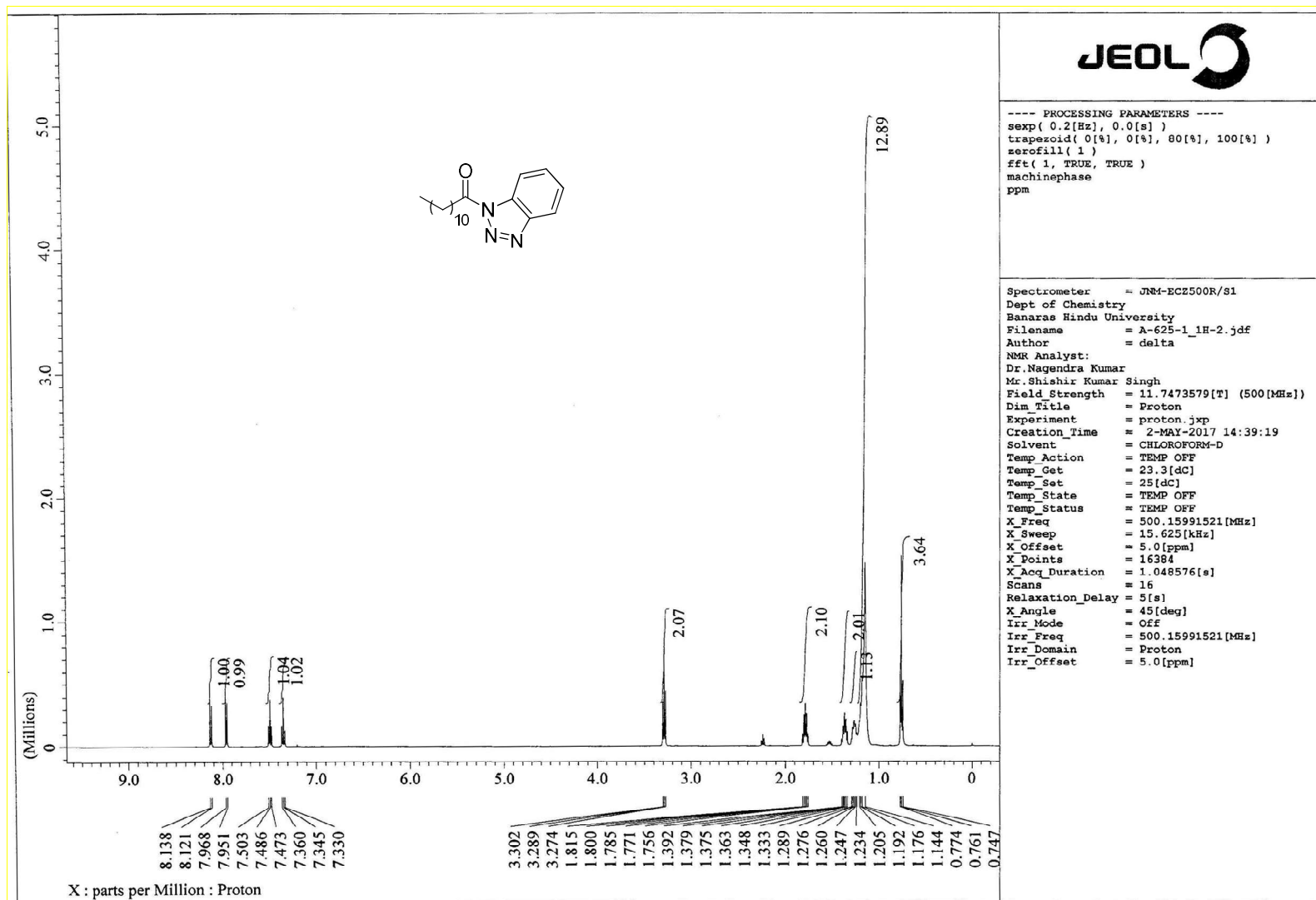
Spectrum 21: ^{1}H NMR (500 MHz, CDCl₃) of compound **2k**
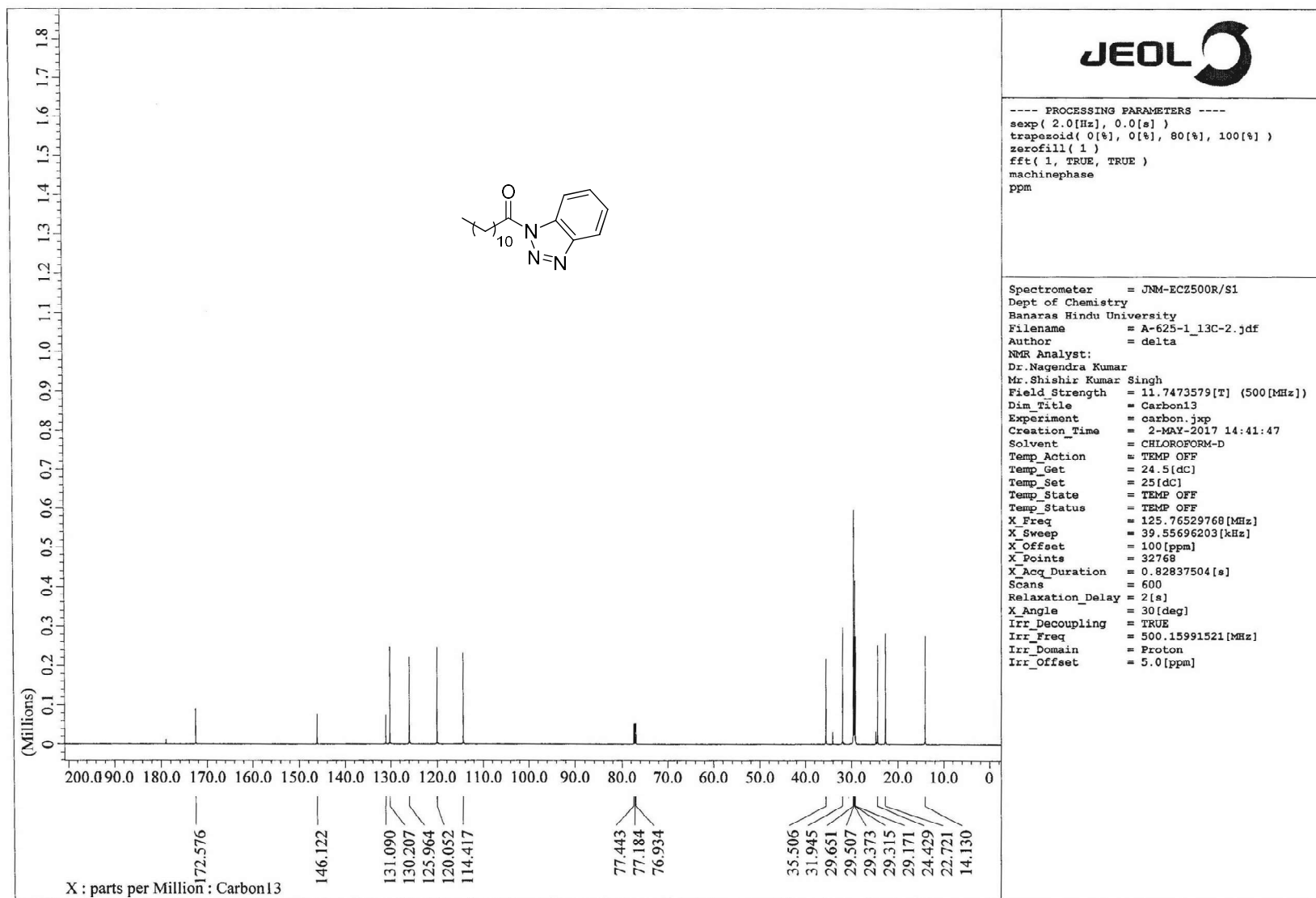
Spectrum 22: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2k**

C:\WINNMR98\COMMON_DEFAULT.ALS
MS-ABT-1 1H Mr. Anoop Shyam
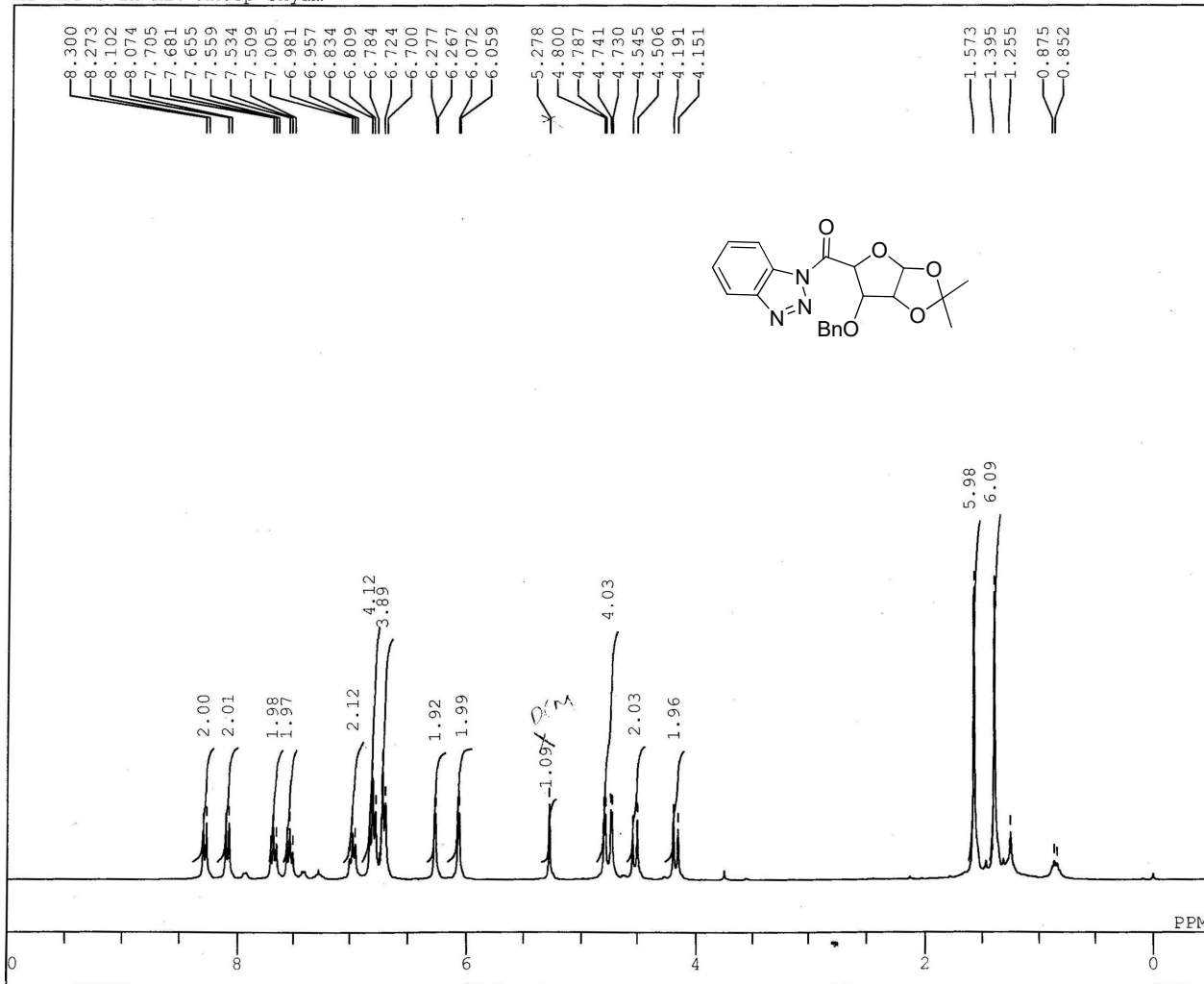
JEOL AL300 FTNMR
CHEMISTRY DEPARTMENT
Banaras Hindu University,
VARANASI-221005

Operator : Nagendra Kumar
          Shishir Singh

DFILE   C:\WINNMR98\COMMON_
COMNT   MS-ABT-1_1H_Mr. Anoo
DATIM   Fri Jul 01 15:58:34
OBNUC   1H
EXMOD   NON
OBFRQ   300.40 MHz
OBSET   130.00 KHz
OBFIN   1150.0 Hz
POINT   32768
FREQU   9505.7 Hz
SCANS   534
ACQTM   3.447 sec
PD   1.547 sec
PW1   5.2 us
IRNUC   1H
CTEMP   23.4 c
SLVNT   CDCL3
EXREF   0.00 ppm
BF   1.20 Hz
RGAIN   13

Peak labels (ppm): 8.300, 8.273, 8.102, 8.074, 7.705, 7.681, 7.655, 7.559, 7.534, 7.509, 7.005, 6.981, 6.957, 6.834, 6.809, 6.784, 6.724, 6.700, 6.277, 6.267, 6.072, 6.059, 5.278, 4.800, 4.787, 4.741, 4.730, 4.545, 4.506, 4.191, 4.151, 1.573, 1.395, 1.255, 0.875, 0.852

Integrations: 2.00, 2.01, 1.98, 1.97, 2.12, 4.12, 3.89, 1.92, 1.99, 1.09, 4.03, 2.03, 1.96, 5.98, 6.09

Spectrum 23: ^{1}H NMR (300 MHz, CDCl$_3$) of compound **2l**

C:\WINNMR98\Data\MS-ABT-1_13C1BCM_E2.ALS
MS-ABT-1 13C Mr. Anoop Shyam

165.762

145.612
135.862
130.818
130.562
127.909
127.661
127.365
126.285
120.046
114.104
112.843

105.961

83.165
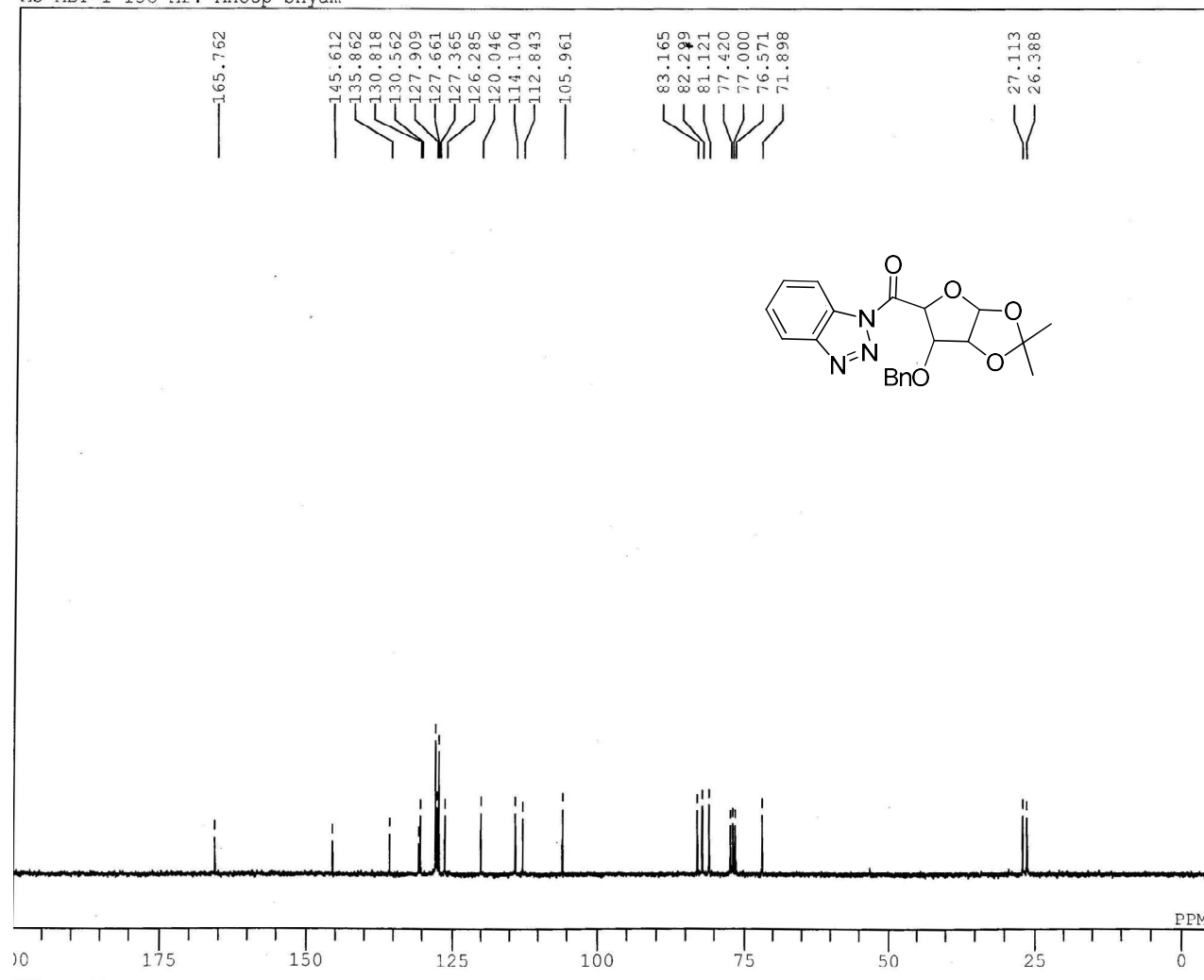82.229
81.121
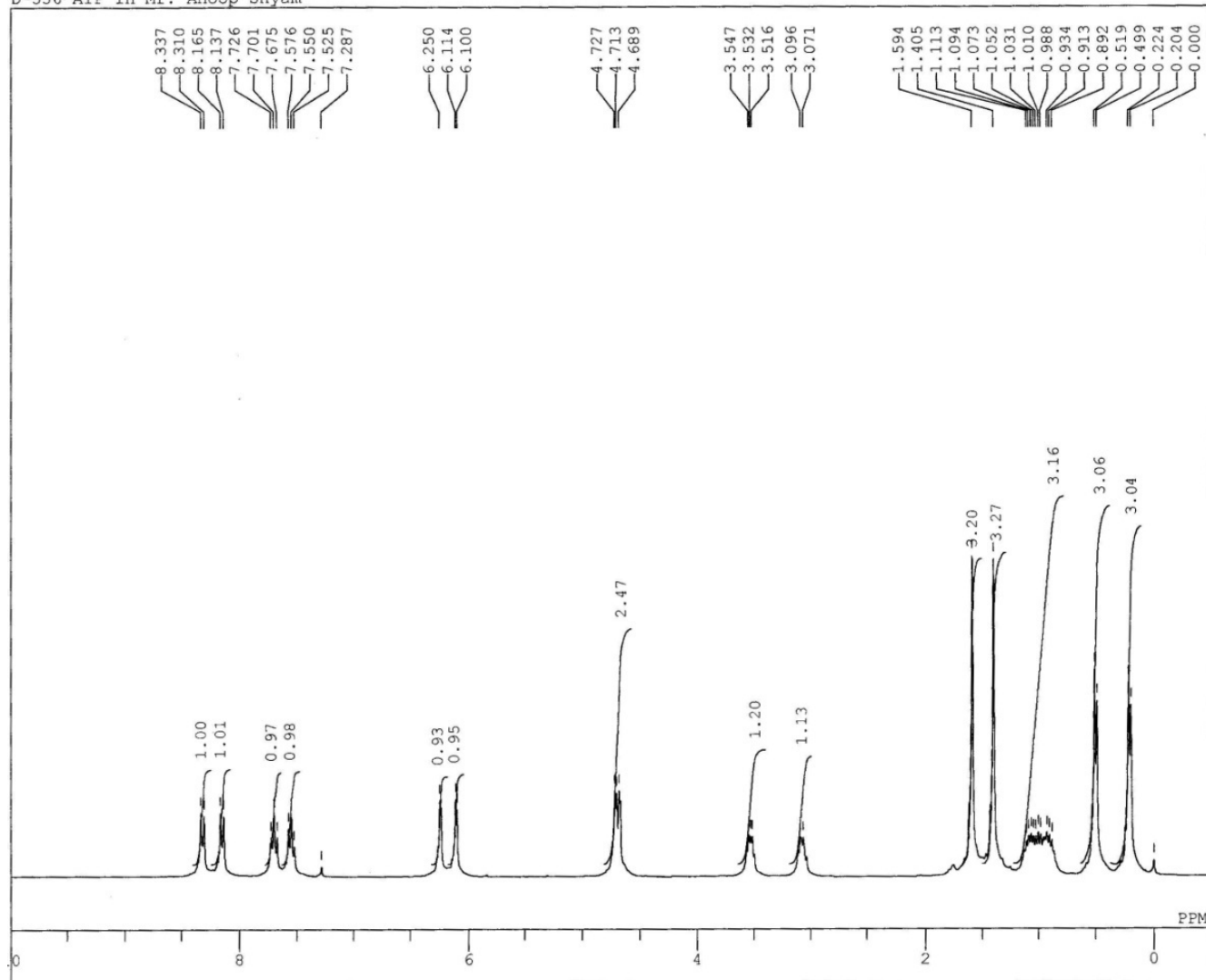77.420
77.000
76.571
71.898

27.113
26.388

JEOL AL300 FTNMR
CHEMISTRY DEPARTMENT
Banaras Hindu University,
VARANASI-221005

Operator : Nagendra Kumar
            Shishir Singh

DFILE   C:\WINNMR98\Data\MS-
COMNT   MS-ABT-1_13C_Mr. Ano
DATIM   Fri Jul 01 15:06:56
OBNUC   13C
EXMOD   BCM
OBFRQ   75.45 MHz
OBSET   124.00 KHz
OBFIN   1840.0 Hz
POINT   32768
FREQU   20408.1 Hz
SCANS   256
ACQTM   1.606 sec
PD   1.394 sec
PW1   5.0 us
IRNUC   1H
CTEMP   26.5 c
SLVNT   CDCL3
EXREF   77.00 ppm
BF   1.20 Hz
RGAIN   15

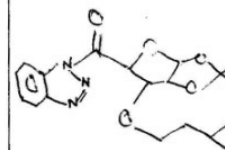**Spectrum 24:** ^{13}C NMR (75 MHz, CDCl$_3$) of compound **2l**

C:\WINNMR98\Data\D-536-AIP_1H1NON_E4.als
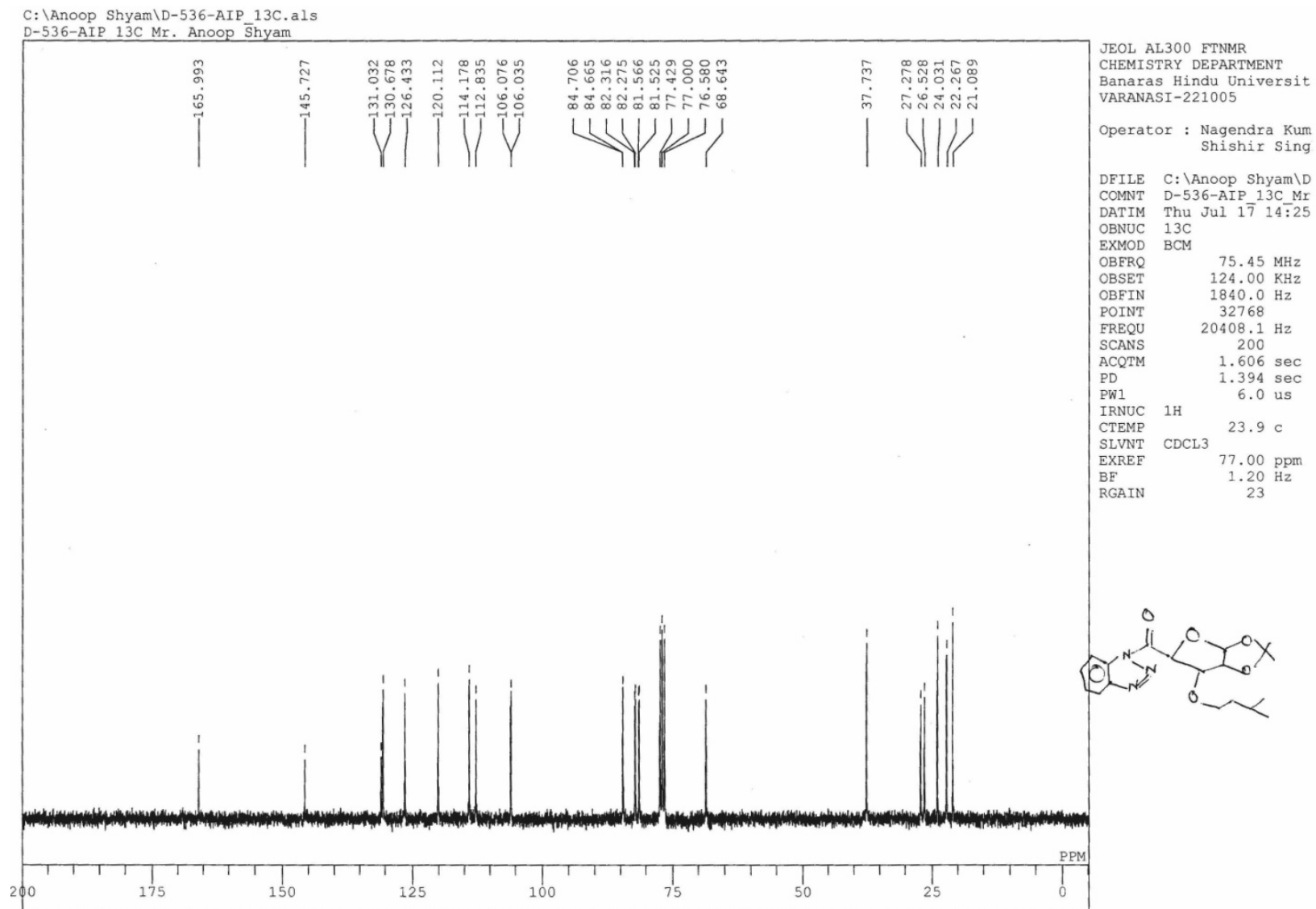D-536-AIP 1H Mr. Anoop Shyam

JEOL AL300 FTNMR
CHEMISTRY DEPARTMENT
Banaras Hindu Universit
VARANASI-221005

Operator : Nagendra Kum
          Shishir Sing

DFILE   C:\WINNMR98\Data
COMNT   D-536-AIP_1H_Mr.
DATIM   Thu Jul 17 14:14
OBNUC   1H
EXMOD   NON
OBFRQ   300.40 MHz
OBSET   130.00 KHz
OBFIN   1150.0 Hz
POINT   32768
FREQU   6016.8 Hz
SCANS   16
ACQTM   5.446 sec
PD   1.547 sec
PW1   5.2 us
IRNUC   1H
CTEMP   22.6 c
SLVNT   CDCL3
EXREF   0.00 ppm
BF   1.20 Hz
RGAIN   15

Spectrum 25: ^{1}H NMR (500 MHz, CDCl$_3$) of compound **2m**

```
C:\Anoop Shyam\D-536-AIP_13C.als
D-536-AIP 13C Mr. Anoop Shyam
```

Peak labels (left to right):
165.993; 145.727; 131.032, 130.678, 126.433; 120.112; 114.178, 112.835; 106.076, 106.035; 84.706, 84.665, 82.316, 82.275, 81.566, 81.525, 77.429, 77.000, 76.580, 68.643; 37.737; 27.278, 26.528, 24.031, 22.267, 21.089

```
JEOL AL300 FTNMR
CHEMISTRY DEPARTMENT
Banaras Hindu Universit
VARANASI-221005

Operator : Nagendra Kum
          Shishir Sing

DFILE   C:\Anoop Shyam\D
COMNT   D-536-AIP_13C_Mr
DATIM   Thu Jul 17 14:25
OBNUC   13C
EXMOD   BCM
OBFRQ        75.45 MHz
OBSET       124.00 KHz
OBFIN      1840.0 Hz
POINT        32768
FREQU     20408.1 Hz
SCANS          200
ACQTM       1.606 sec
PD          1.394 sec
PW1           6.0 us
IRNUC   1H
CTEMP        23.9 c
SLVNT   CDCL3
EXREF       77.00 ppm
BF           1.20 Hz
RGAIN          23
```
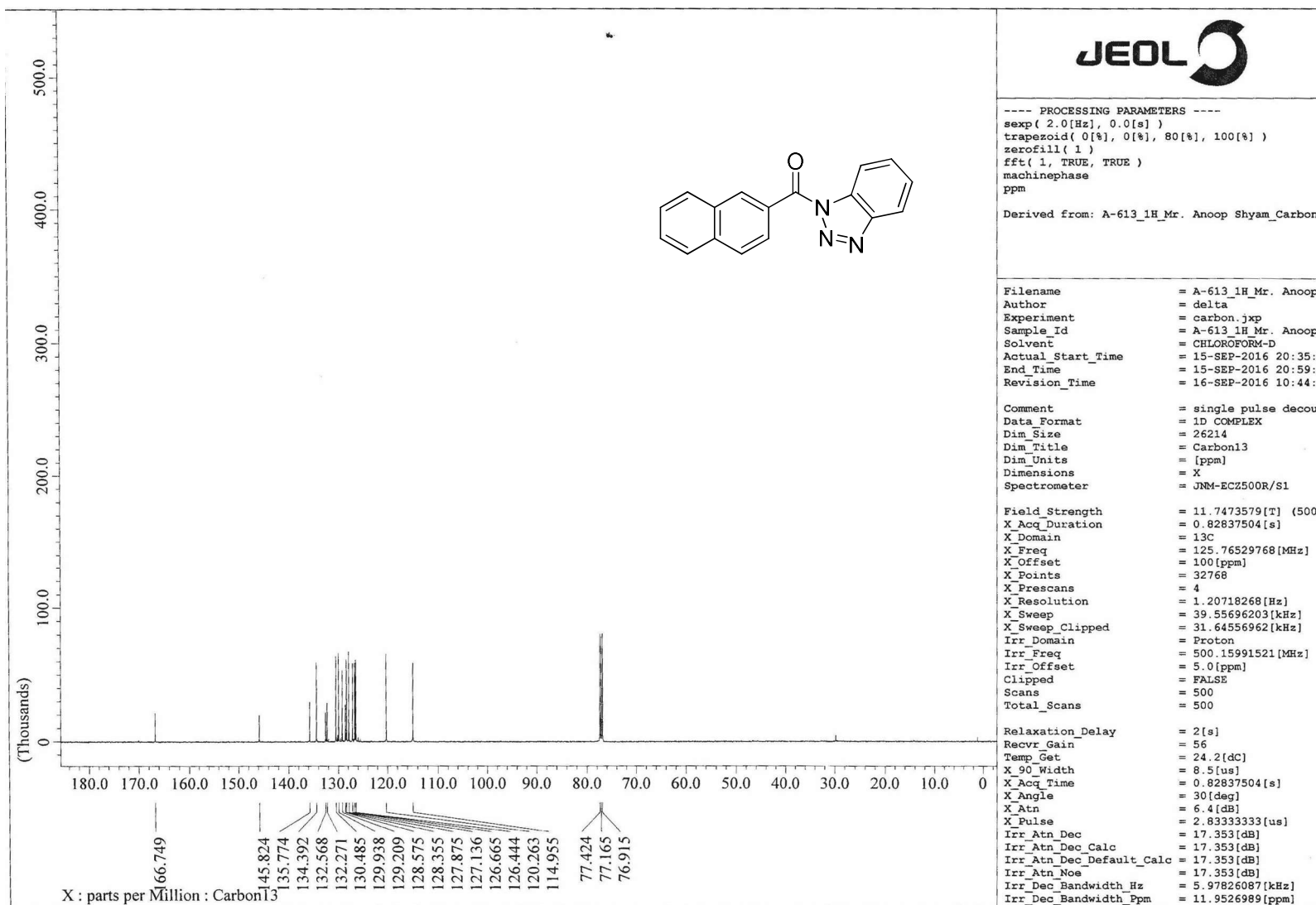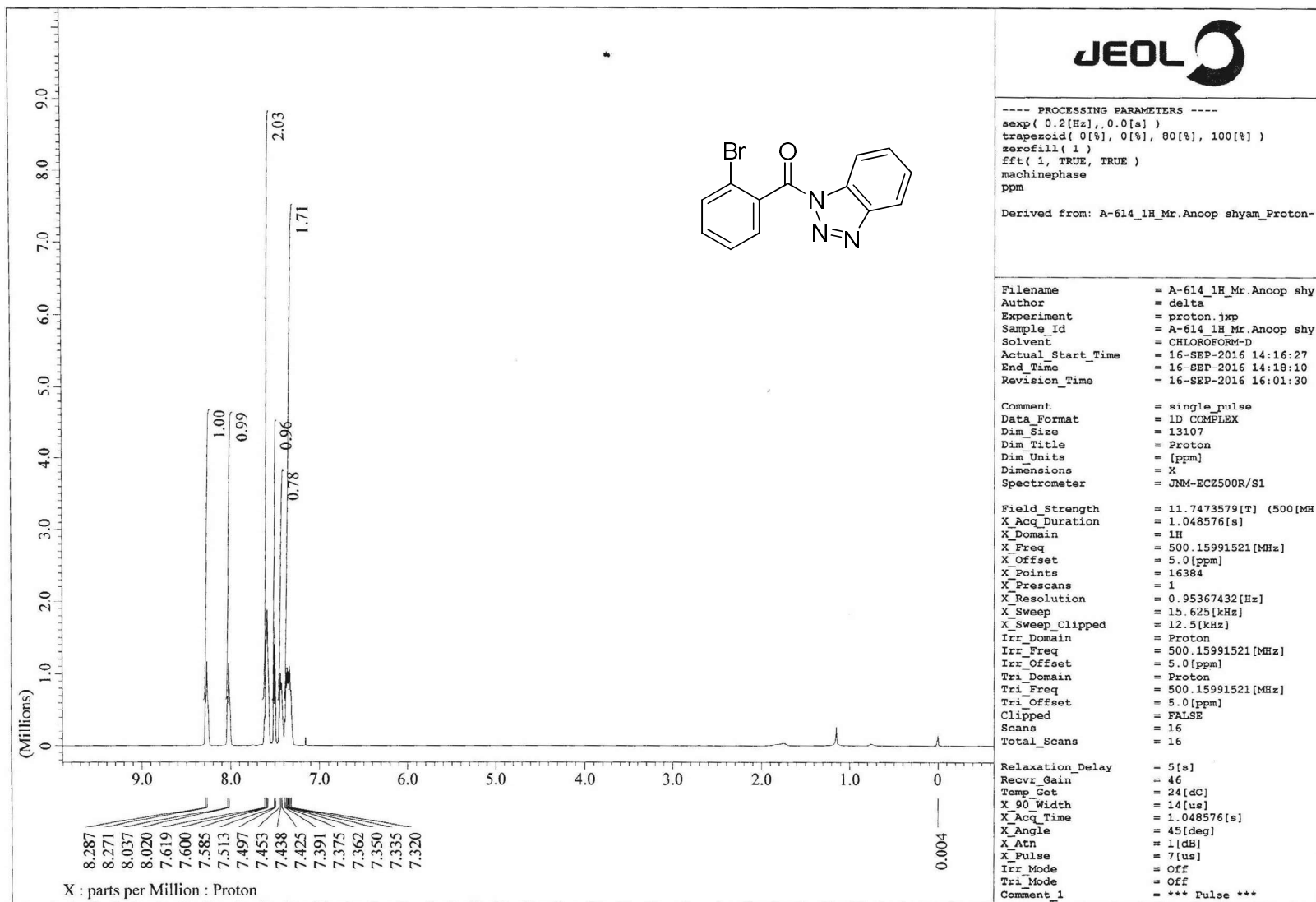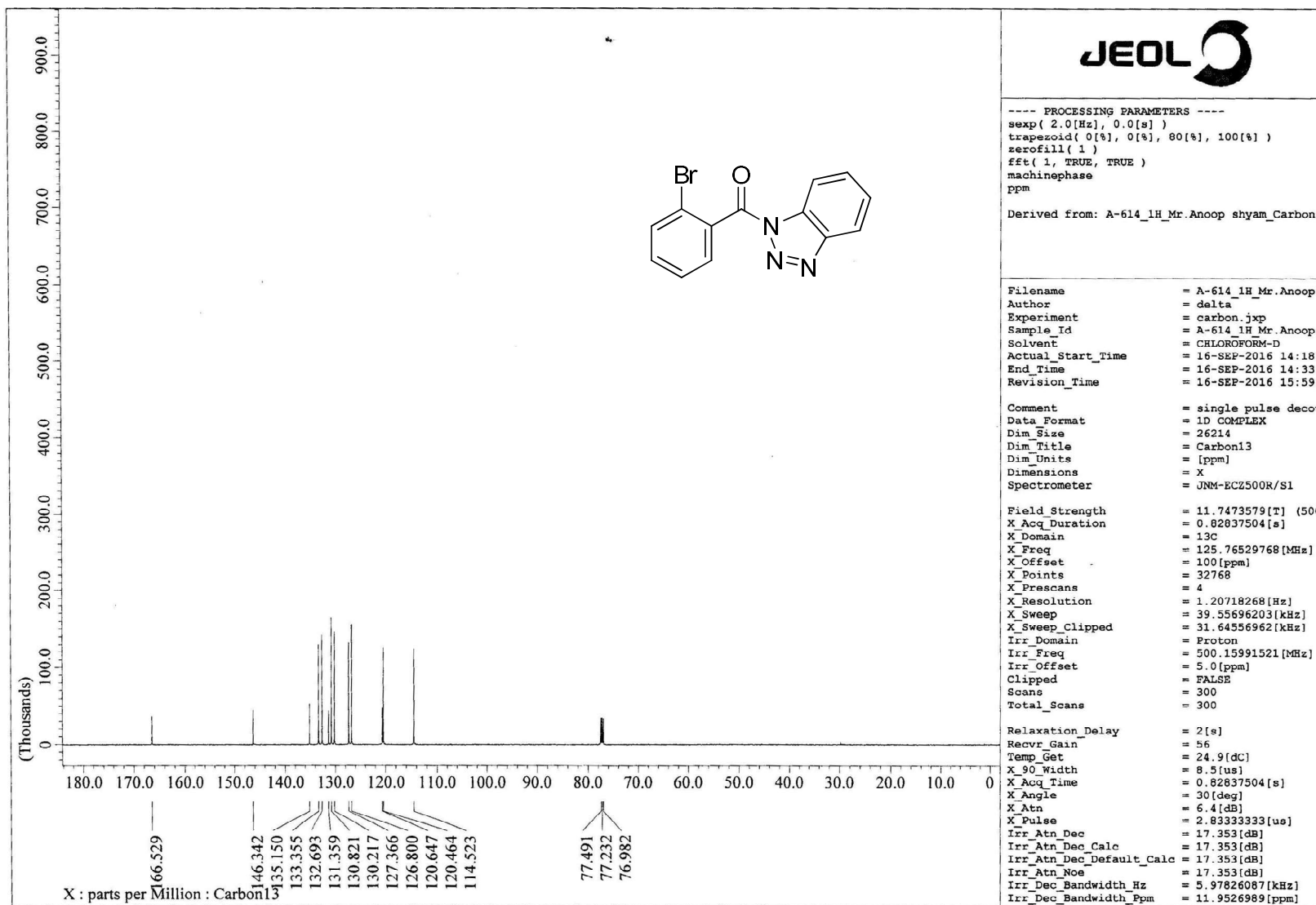
Spectrum 26: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2m**
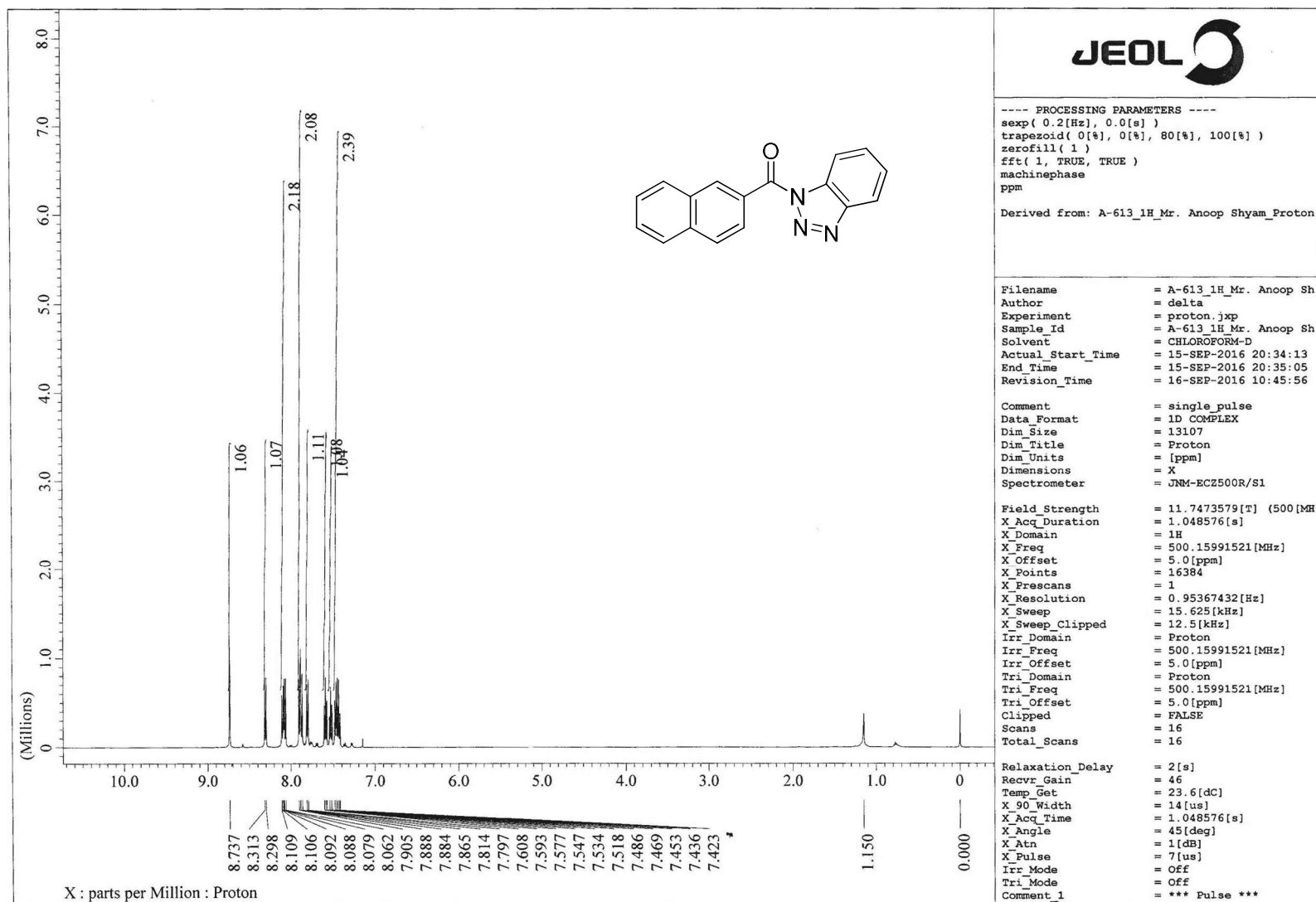
Spectrum 27: ^{1}H NMR (500 MHz, CDCl$_3$) of compound **2n**

Spectrum 28: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2n**

Spectrum 29: ^{1}H NMR (500 MHz, CDCl$_3$) of compound **2o**

Spectrum 30: ^{13}C NMR (125 MHz, CDCl$_3$) of compound **2o**