

Synthesis of 2,3,4-Trisubstituted Thiochromanes by Using an Organocatalytic Enantioselective Tandem Michael-Henry Reaction

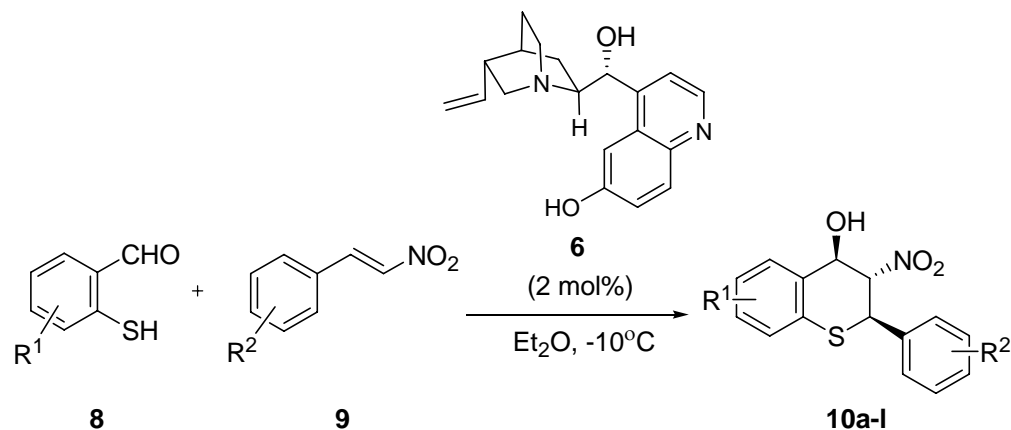
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Grant A. Broker and Edward R.T. Tiekink

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One UTSA Circle, San Antonio, TX 78249-0698

Supporting Information

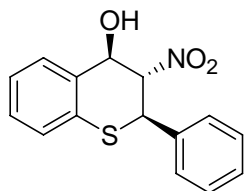
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¹H and ¹³C NMR data of compounds 10a-l.

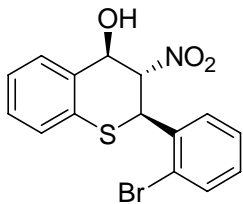
(2*R**,3*S**,4*R**)-3-Nitro-2-phenyl-thiochroman-4-ol (10a)



¹H NMR (500 MHz, Acetone-*d*₆) δ = 5.16 (d, *J* = 10.5 Hz, 1H), 5.37-5.43 (m, 2H), 5.83 (d, *J* = 8.0 Hz, 1H), 7.14 (dd, *J* = 7.0, 2.0 Hz, 1H), 7.25-7.28 (m, 2H), 7.39-7.44 (m, 3H), 7.53-7.55 (m, 2H),

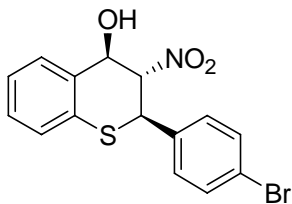
7.73 (dd, $J = 7.0, 1.5$ Hz, 1H). ^{13}C NMR (125 MHz, Acetone- d_6) $\delta = 46.8, 71.7, 93.5, 124.8, 125.4, 128.0, 128.5, 128.7$ (2C), 129.2 (2C), 129.4, 131.9, 135.4, 135.8.

(2*R,3*S**,4*R**)-2-(2-Bromophenyl)-3-nitro-thiochroman-4-ol (10b)**



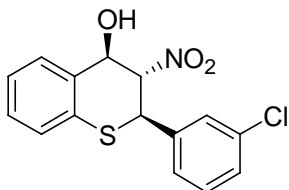
^1H NMR (300 MHz, Acetone- d_6) $\delta = 5.46$ (t, $J = 8.4$ Hz, 1H), 5.56 (t, $J = 10.1$ Hz, 1H), 5.67 (d, $J = 10.2$ Hz, 1H), 6.02 (d, $J = 8.4$ Hz, 1H), 7.14 (m, 1H), 7.24-7.34 (m, 3H), 7.47 (td, $J = 7.3, 0.9$ Hz, 1H), 7.67 (dd, $J = 7.8, 1.2$ Hz, 1H), 7.73- 7.76 (m, 2H). ^{13}C NMR (75 MHz, Acetone- d_6) $\delta = 45.5, 71.8, 92.2, 124.4, 125.0, 125.7, 128.0, 128.6, 128.8, 129.4, 131.0, 131.3, 133.8, 134.5, 135.9$.

(2*R,3*S**,4*R**)-2-(4-Bromophenyl)-3-nitro-thiochroman-4-ol (10c)**



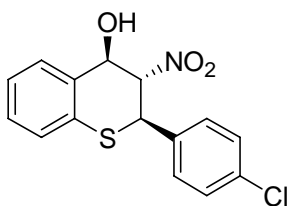
^1H NMR (500 MHz, Acetone- d_6) $\delta = 5.19$ (d, $J = 10.5$ Hz, 1H), 5.36-5.42 (m, 2H), 5.88 (d, $J = 8.0$ Hz, 1H), 7.14 (dd, $J = 7.0, 2.0$ Hz, 1H), 7.26, 7.28 (m, 2H), 7.51 (d, $J = 8.5$ Hz, 2H), 6.62 (d, $J = 8.5$ Hz, 2H), 7.74 (dd, $J = 7.0, 2.5$ Hz, 1H). ^{13}C NMR (125 MHz, Acetone- d_6) $\delta = 46.1, 71.5, 93.4, 122.9, 124.8, 125.5, 128.0, 128.5, 130.7$ (2C), 131.5, 132.4 (2C), 134.9, 135.8.

(2*R,3*S**,4*R**)-2-(3-Chlorophenyl)-3-nitro-thiochroman-4-ol (10d)**



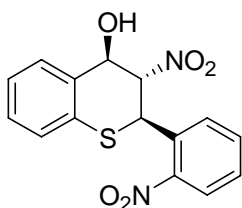
^1H NMR (500 MHz, Acetone- d_6) $\delta = 5.21$ (m, 1H), 5.41-5.43 (m, 2H), 5.90 (m, 1H), 7.16 (dd, $J = 7.5, 2.0$ Hz, 1H), 7.25-7.29 (m, 2H), 7.44-7.47 (m, 2H), 7.50-7.52 (m, 1H), 7.61 (d, $J = 2.0$ Hz, 1H), 7.74 (m, 1H). ^{13}C NMR (125 MHz, Acetone- d_6) $\delta = 46.2, 71.5, 93.3, 124.9, 125.6, 127.3, 127.9, 128.6, 128.7, 129.5, 131.0, 131.5, 134.4, 135.8, 137.9$.

(2*R,3*S**,4*R**)-2-(4-Chlorophenyl)-3-nitro-thiochroman-4-ol (10e)**



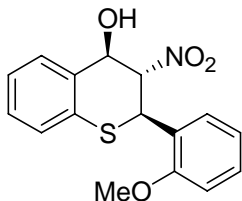
^1H NMR (300 MHz, Acetone- d_6) δ = 5.19 (d, J = 11.1, 1H), 5.35-5.46 (m, 2H), 5.71 (d, J = 8.4 Hz, 1H), 7.14 (m, 1H), 7.24-7.27 (m, 2H), 7.44 (d, J = 8.7 Hz, 2H), 7.56 (d, J = 8.7 Hz, 2H), 7.21 (m, 1H). ^{13}C NMR (75 MHz, Acetone- d_6) δ = 46.2, 71.6, 93.5, 124.8, 125.5, 127.9, 128.5, 129.3 (2C), 130.4 (2C), 131.6, 134.3, 134.7, 135.7.

(2R*,3S*,4R*)-3-Nitro-2-(2-nitrophenyl)-thiochroman-4-ol (10f)



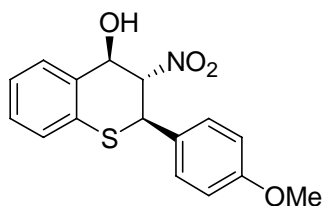
^1H NMR (500 MHz, Acetone- d_6) δ = 5.41 (t, J = 8.5 Hz, 1H), 5.50 (t, J = 10.0 Hz, 1H), 5.70 (d, J = 10.0 Hz, 1H), 6.03 (d, J = 8.0 Hz, 1H), 7.20 (m, 1H), 7.30-7.32 (m, 2H), 7.68 (td, J = 8.5, 1.5 Hz, 1H), 7.76 (m, 1H), 7.81 (td, J = 8.0, 1.5 Hz, 1H), 7.96 (td, J = 8.0, 1.5 Hz, 2H). ^{13}C NMR (125 MHz, Acetone- d_6) δ = 40.9, 71.6, 93.0, 125.1, 125.3, 126.0, 127.7, 128.7, 129.9, 130.1, 131.1, 133.7, 133.9, 136.2, 150.1.

(2R*,3S*,4R*)-2-(2-Methoxyphenyl)-3-nitro-thiochroman-4-ol (10g)



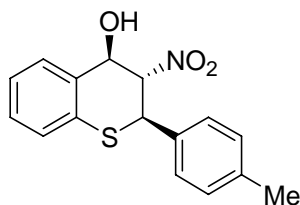
^1H NMR (300 MHz, Acetone- d_6) δ = 3.84 (s, 3H), 5.38 (m, 1H), 5.48-5.50 (m, 2H), 5.84 (d, J = 8.1 Hz, 1H), 6.96 (td, J = 7.5, 1.2 Hz, 1H), 7.05 (d, J = 8.4 Hz, 1H), 7.12 (m, 1H), 7.21-7.27 (m, 2H), 7.33 (td, J = 8.7, 1.8, 1H), 7.45 (dd, J = 7.5, 1.8 Hz, 1H), 7.72 (m, 1H). ^{13}C NMR (75 MHz, Acetone- d_6) δ = 41.0, 55.7, 72.0, 92.3, 112.0, 121.1, 123.3, 125.1, 125.4, 127.7, 128.3, 129.2, 130.5, 132.4, 136.1, 157.7.

(2R*,3S*,4R*)-2-(4-Methoxyphenyl)-3-nitro-thiochroman-4-ol (10h)



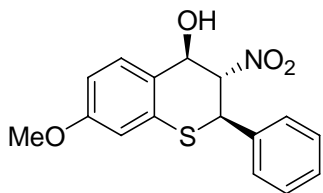
^1H NMR (500 MHz, Acetone- d_6) δ = 3.82 (s, 3H), 5.10 (d, J = 11.0 Hz, 1H), 5.33 (t, J = 10.3 Hz, 1H), 5.40 (m, 1H), 5.79 (d, J = 8.0 Hz, 1H), 6.95 (d, J = 9.0 Hz, 2H), 7.12 (dd, J = 7.5, 1.2 Hz, 1H), 7.23 (td, J = 7.0, 2.5 Hz, 1H), 7.26 (td, J = 7.0, 2.5 Hz, 1H), 7.45 (d, J = 9.0 Hz, 2H), 7.73 (m, 1H).
 ^{13}C NMR (125 MHz, Acetone- d_6) δ = 46.3, 55.0, 71.7, 93.7, 114.5 (2C), 124.7, 125.3, 126.8, 128.0, 128.4, 129.9 (2C), 132.1, 135.8, 160.6.

(2R*,3S*,4R*)-2-(4-Methylphenyl)-3-nitro-thiochroman-4-ol (10i)



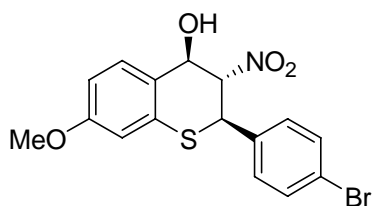
^1H NMR (300 MHz, Acetone- d_6) δ = 2.32 (s, 3H), 5.10 (d, J = 10.8 Hz, 1H), 5.32-5.40 (m, 2H), 5.85 (d, J = 7.8 Hz, 1H), 7.10 (m, 1H), 7.19-7.27 (m, 4H), 7.39 (d, J = 7.8 Hz, 2H), 7.71 (dd, J = 6.9, 2.4 Hz, 1H). ^{13}C NMR (75 MHz, Acetone- d_6) δ = 20.6, 46.6, 71.7, 93.6, 124.7, 125.3, 128.0, 128.4, 128.5 (2H), 129.8 (2H), 131.4, 132.1, 135.8, 139.2.

(2R*,3S*,4R*)-7-Methoxy-3-nitro-2-phenyl-thiochroman-4-ol (10j)



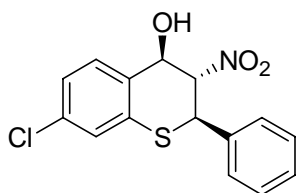
^1H NMR (300 MHz, Acetone- d_6) δ = 3.80 (s, 3H), 5.16 (m, 1H), 5.31-5.37 (m, 2H), 5.72 (d, J = 6.0, 1.8 Hz, 1H), 6.67 (d, J = 2.3 Hz, 1H), 6.81 (dd, J = 8.7, 2.3 Hz, 1H), 7.36-7.43 (m, 3H), 7.50-7.53 (m, 2H), 7.61 (d, J = 8.7 Hz, 1H). ^{13}C NMR (75 MHz, Acetone- d_6) δ = 47.0, 55.2, 71.6, 93.9, 109.0, 112.1, 128.7 (2C), 129.2 (2C), 129.3, 129.4, 132.8, 133.0, 135.1, 159.7.

(2R*,3S*,4R*)-2-(4-Bromophenyl)-7-Methoxy-3-nitro-thiochroman-4-ol (10k)



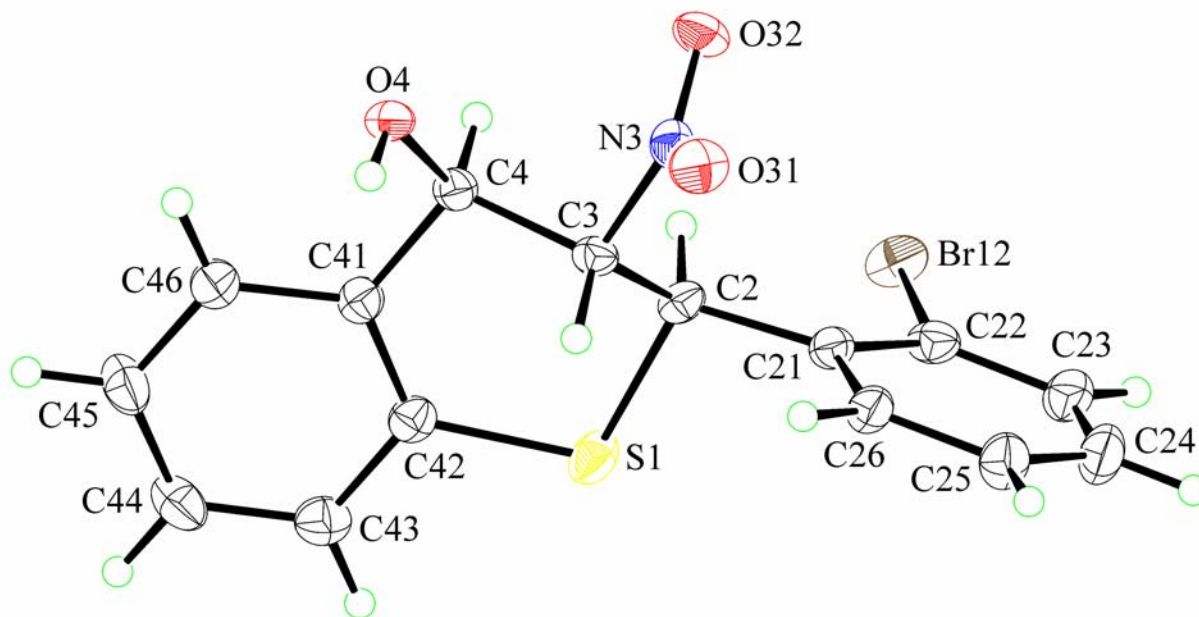
^1H NMR (300 MHz, Acetone- d_6) δ = 3.80 (s, 3H), 5.15 (m, 1H), 5.31-5.35 (m, 2H), 5.78 (d, J = 5.7 Hz, 1H), 6.68 (d, J = 2.4 Hz, 1H), 6.81 (dd, J = 8.9, 2.4 Hz, 1H), 7.49 (d, J = 8.6 Hz, 2H), 7.60 (d, J = 8.6 Hz, 2H), 7.61 (d, J = 8.9 Hz, 1H). ^{13}C NMR (75 MHz, Acetone- d_6) δ = 46.3, 55.2, 71.4, 93.7, 109.1, 112.2, 122.9, 127.7, 129.3, 130.7 (2C), 132.3 (2C), 132.8, 134.7, 159.6.

(2*R,3*S**,4*R**)-7-Chloro-3-nitro-2-phenylthiochroman-4-ol (10l)**



^1H NMR (300 MHz, Acetone- d_6) δ = 5.18 (m, 1H), 5.37-5.40 (m, 2H), 5.80 (m, 1H), 7.17 (d, J = 2.1 Hz, 1H), 7.22 (dd, J = 8.3, 2.1 Hz, 1H), 7.36-7.41 (m, 3H), 7.48-7.52 (m, 2H), 7.68 (d, J = 8.3 Hz, 1H). ^{13}C NMR (75 MHz, Acetone- d_6) δ = 47.2, 71.6, 93.2, 124.2, 125.6, 128.9 (2C), 129.5 (2C), 129.8, 129.9, 133.9, 134.6, 134.9, 135.0.

X-Ray data of compound 10b [(2*R*,3*S*,4*R*)-2-(2-Bromophenyl)-3-nitro-thiochroman-4-ol]



Crystal data for C₁₅H₁₂BrNO₃S (**10b**): $M = 366.23$, $T = 173(2)$ K, orthorhombic, $P2_12_12_1$, $a = 5.1673(10)$, $b = 11.594(2)$, $c = 24.283(5)$ Å, $V = 1454.8(5)$ Å³, $Z = 4$, $D_x = 1.672$ g cm⁻³, $\mu = 2.978$ mm⁻¹, no. of unique data (AFC12κ/SATURN724 CCD¹⁷ using Mo-Kα radiation so that $\theta_{\max} = 30.2^\circ$) = 2610, no. of parameters = 193, R (2485 data with $I \geq 2\sigma(I)$) = 0.047, wR (all data) = 0.129. The structure was solved by direct-methods (SHELXS-97¹⁸) and refined (anisotropic displacement parameters, H atoms in the riding model approximation and a weighting scheme $w = 1/[\sigma^2(F_o^2) + 0.069P^2 + 0.735P]$ where $P = (F_o^2 + 2F_c^2)/3$) with SHELXL-97 on F^2 .¹⁹ The absolute structure was determined based on differences in Friedel pairs included in the data set yielding a value for the Flack parameter²⁰ of -0.002(16). Fig. 1 was drawn with ORTEP-II²¹ at 35% displacement ellipsoids. Molecules aggregate in the crystal structure via a zig-zag chain of [O-H...O]_∞ hydrogen bonds aligned along the a -axis: O1-H...O1^{*i*} = 1.97 Å, O1...O1^{*i*} = 2.797(4) Å, with O-H...O angle of 169° for $i: \frac{1}{2}+x, \frac{1}{2}-y, -z$. CCDC deposition number: 647961.

Additional References:

17. CrystalClear User Manual, Rigaku/MSI Inc., The Woodlands, TX 77381, USA, **2005**;
18. Sheldrick, G. M. *Acta Crystallogr.* **1990**, *A46*, 467;
19. Sheldrick, G. M. SHELXL97. University of Göttingen, Germany, **1997**;
20. Flack, H. D. *Acta Crystallogr.* **1983**, *A39*, 876;
21. Johnson, C. K. *ORTEP-II, Report ORNL-5138*, Oak Ridge National Laboratory: Oak Ridge, TN, 1976.

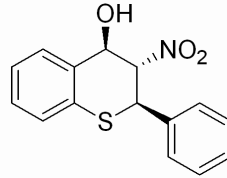
VII-DR-146-1H

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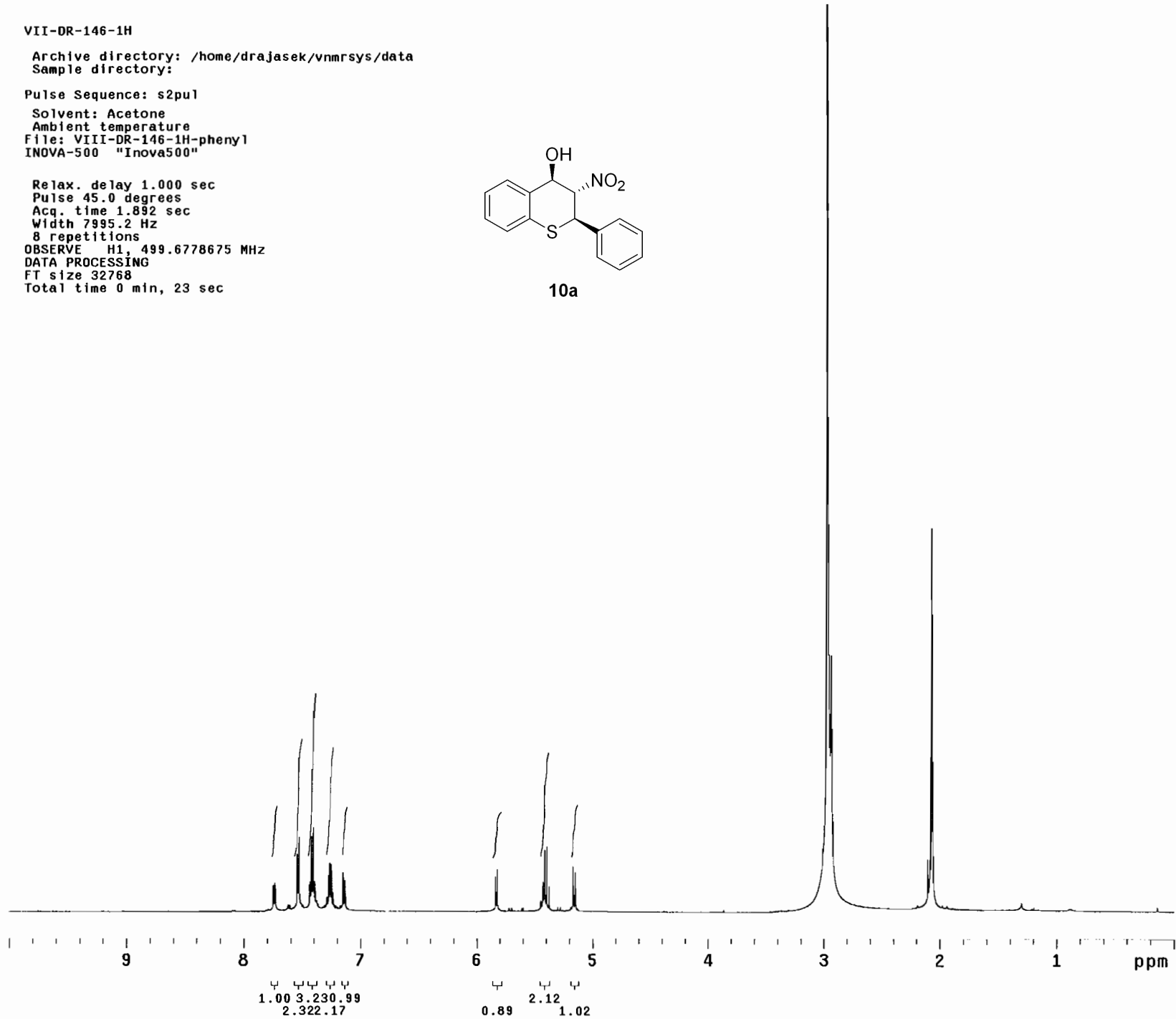
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8 repetitions
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DATA PROCESSING
FT size 32768
Total time 0 min, 23 sec



10a



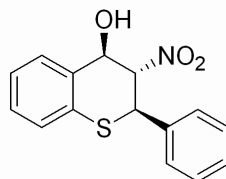
VIII-DR-146-13C

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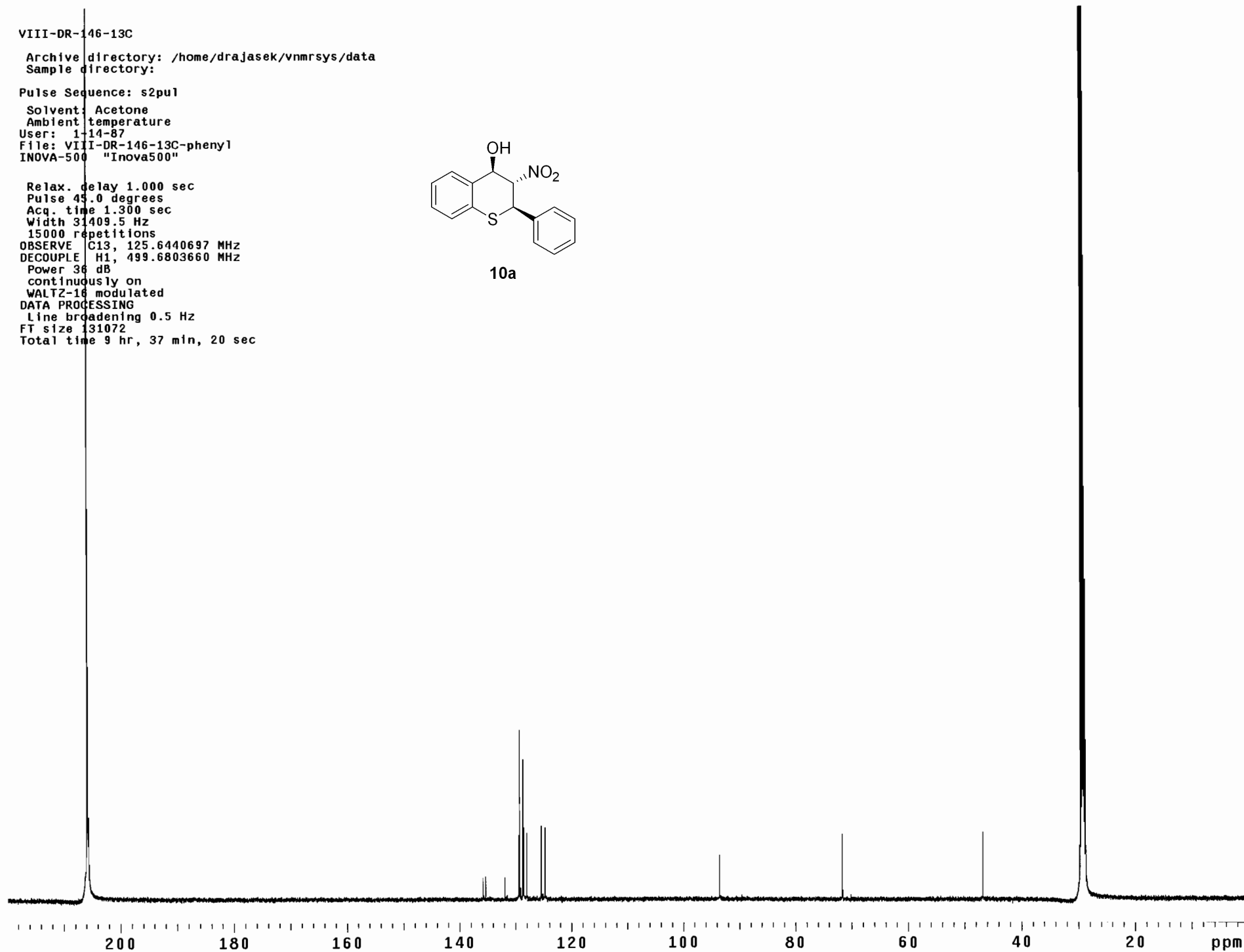
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Solvent: Acetone
Ambient temperature
User: 1-14-87
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INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
15000 repetitions
OBSERVE C13, 125.6440697 MHz
DECOUPLE H1, 499.6803660 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 9 hr, 37 min, 20 sec



10a



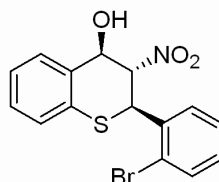
IX-DR-21-1H

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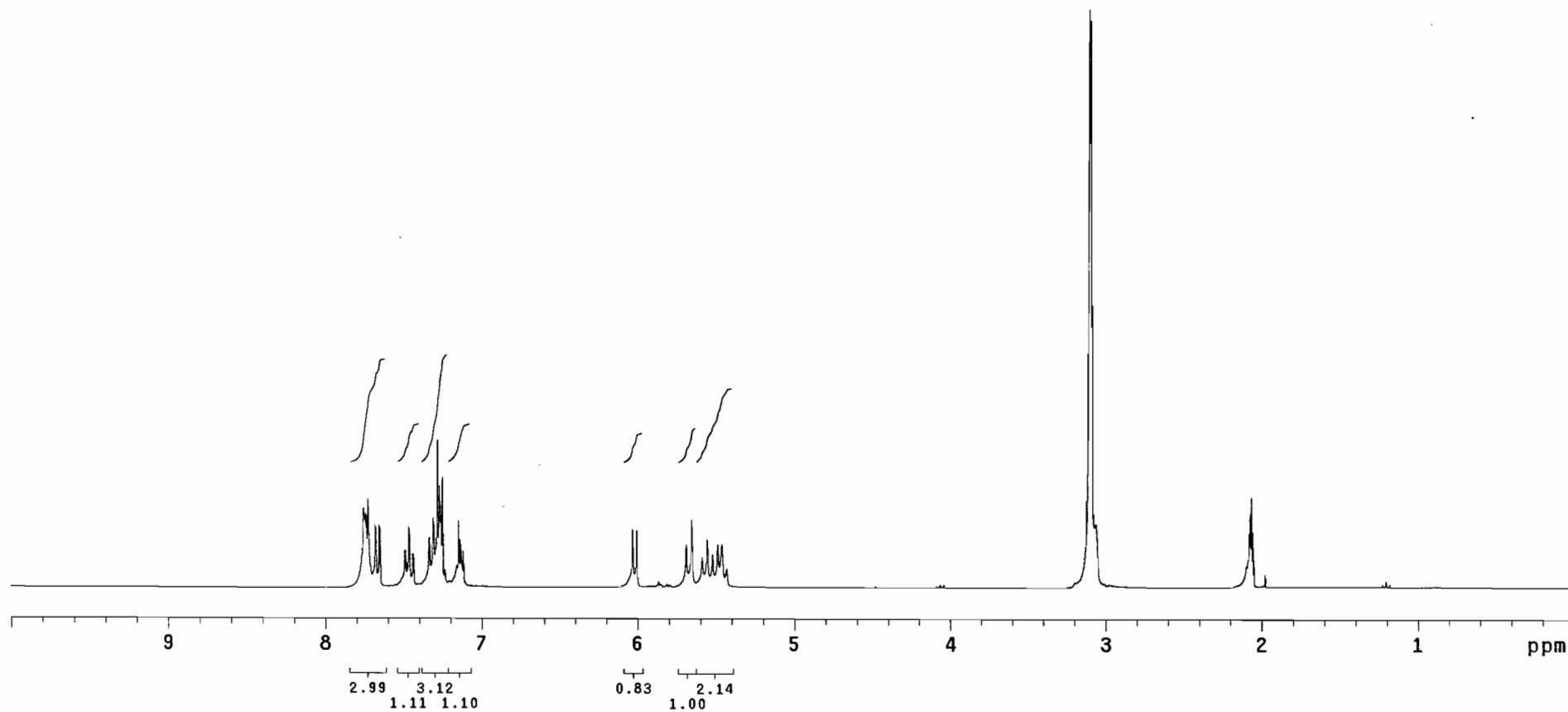
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Solvent: Acetone
Ambient temperature
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Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
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8 repetitions
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DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec



10b



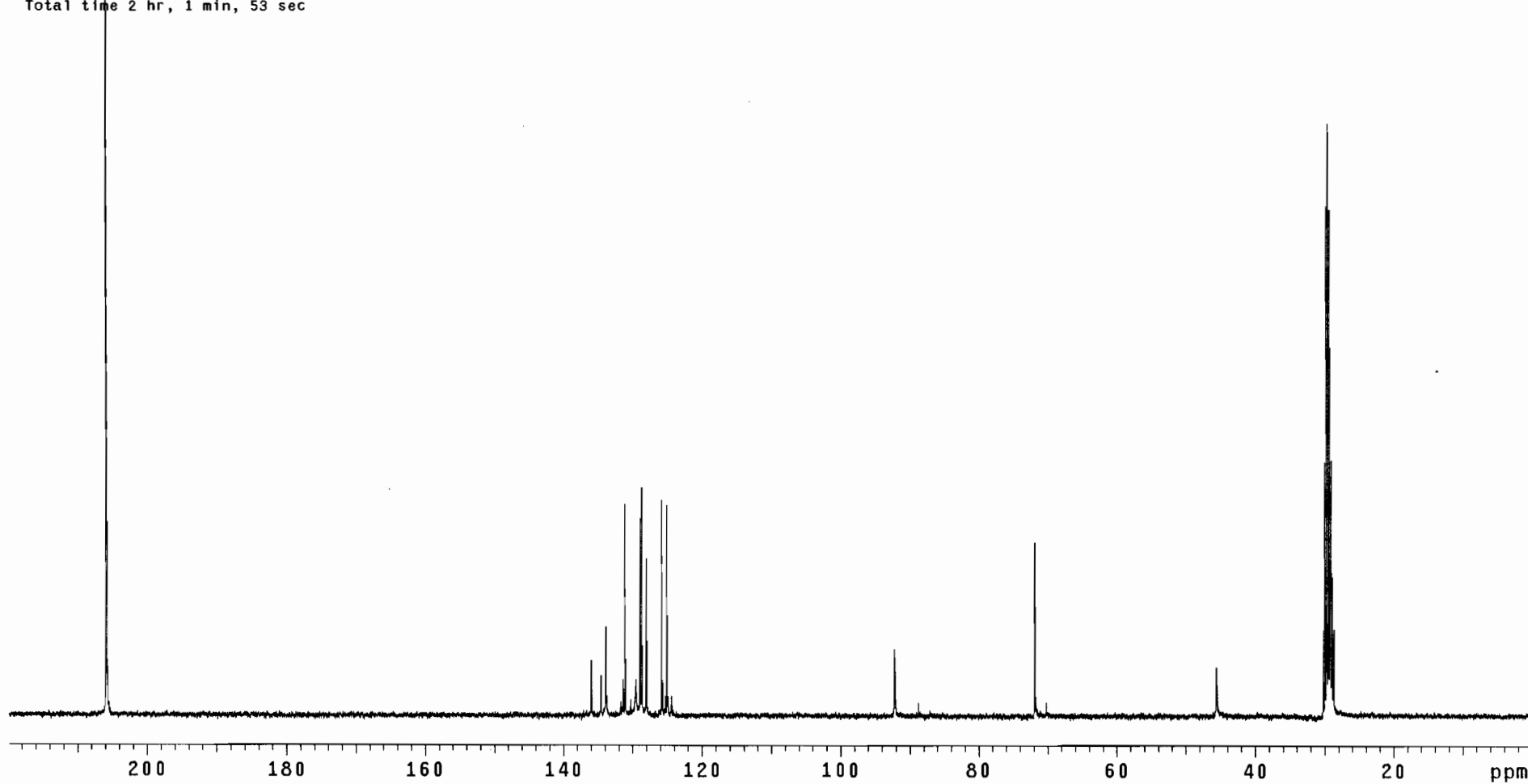
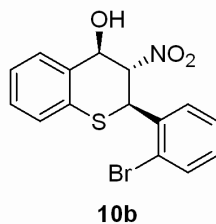
IX-DR-21-13C

Archive directory: /export2/home/drajasek/vnmrSYS/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: Acetone
Ambient temperature
File: IX-DR-21-13C-2-bromo
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
2500 repetitions
OBSERVE C13, 75.3713792 MHz
DECOUPLE H1, 299.7483521 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 1 min, 53 sec



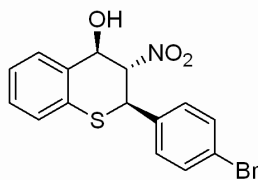
VIII-DR-149-1H

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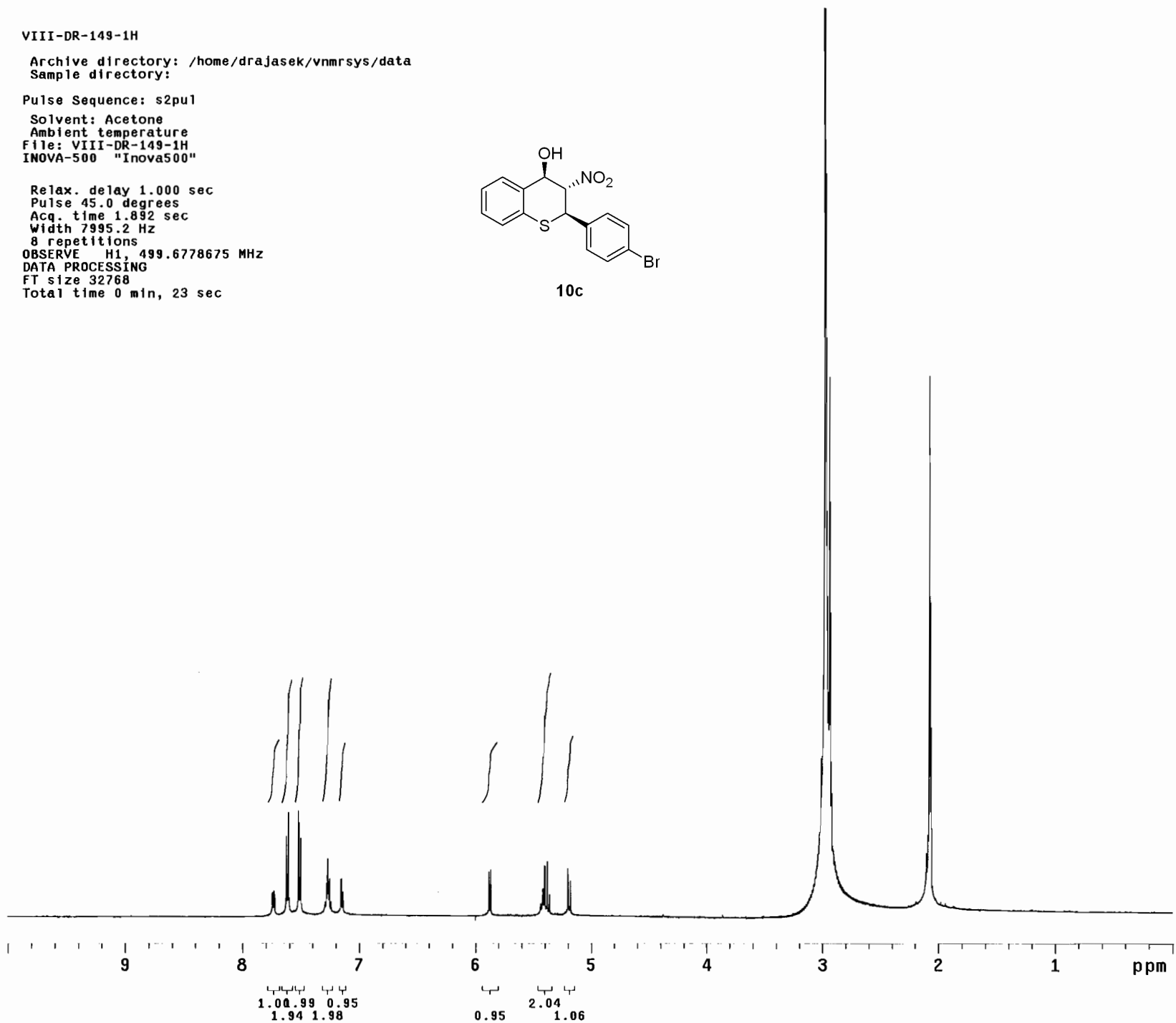
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Solvent: Acetone
Ambient temperature
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INOVA-500 "Inova500"

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Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7995.2 Hz
8 repetitions
OBSERVE H1, 499.6778675 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 23 sec



10c



VIII-DR-149-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

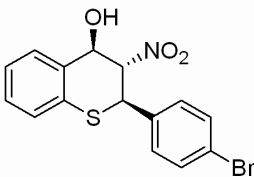
Solvent: Acetone
Ambient temperature

User: 1-14-87

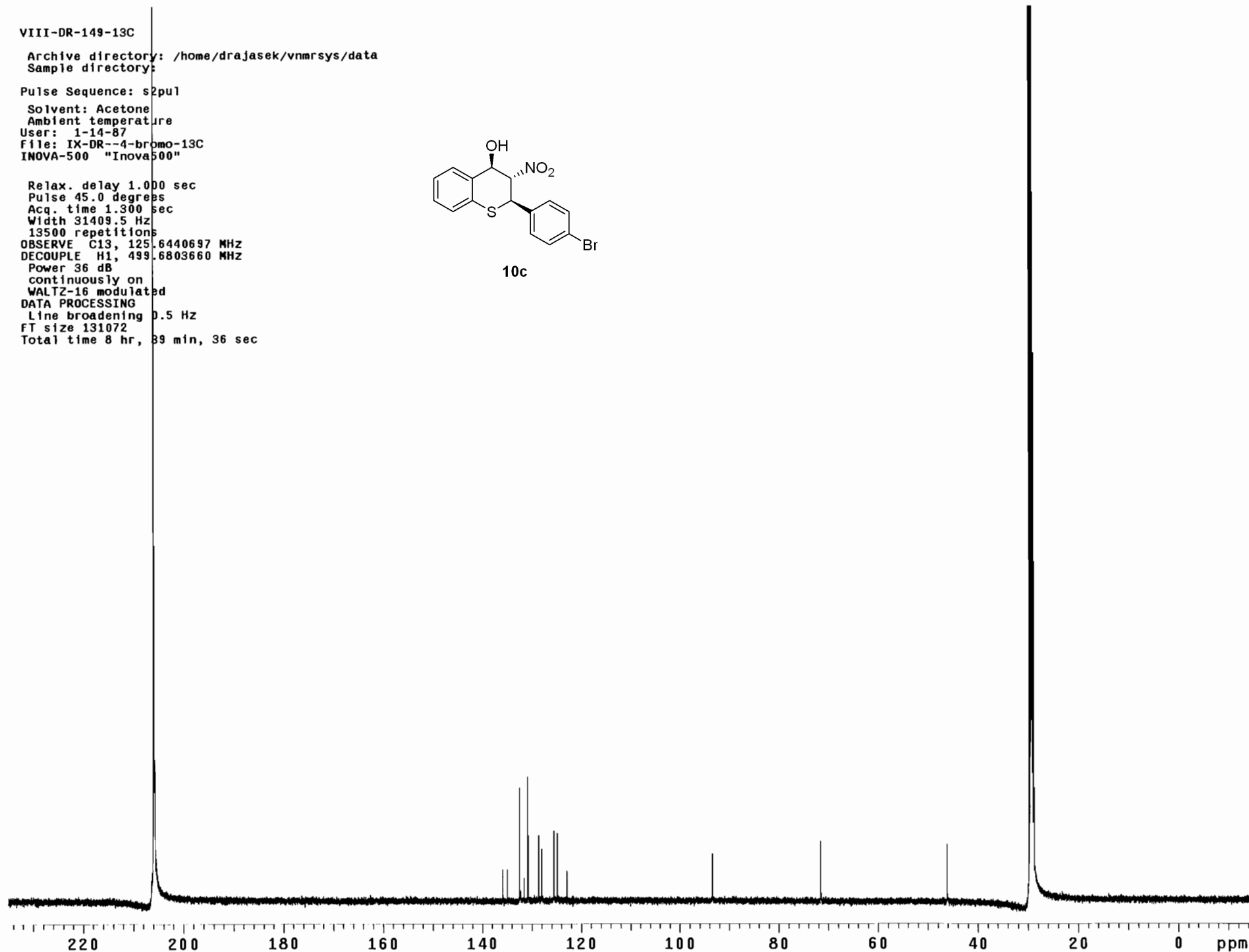
File: IX-DR--4-bromo-13C

INOVA-500 "Inova500"

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Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
13500 repetitions
OBSERVE C13, 125.6440697 MHZ
DECOUPLE H1, 499.6803660 MHZ
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 8 hr, 89 min, 36 sec



10c



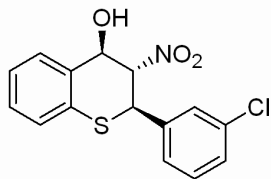
IX-DR-08-1H

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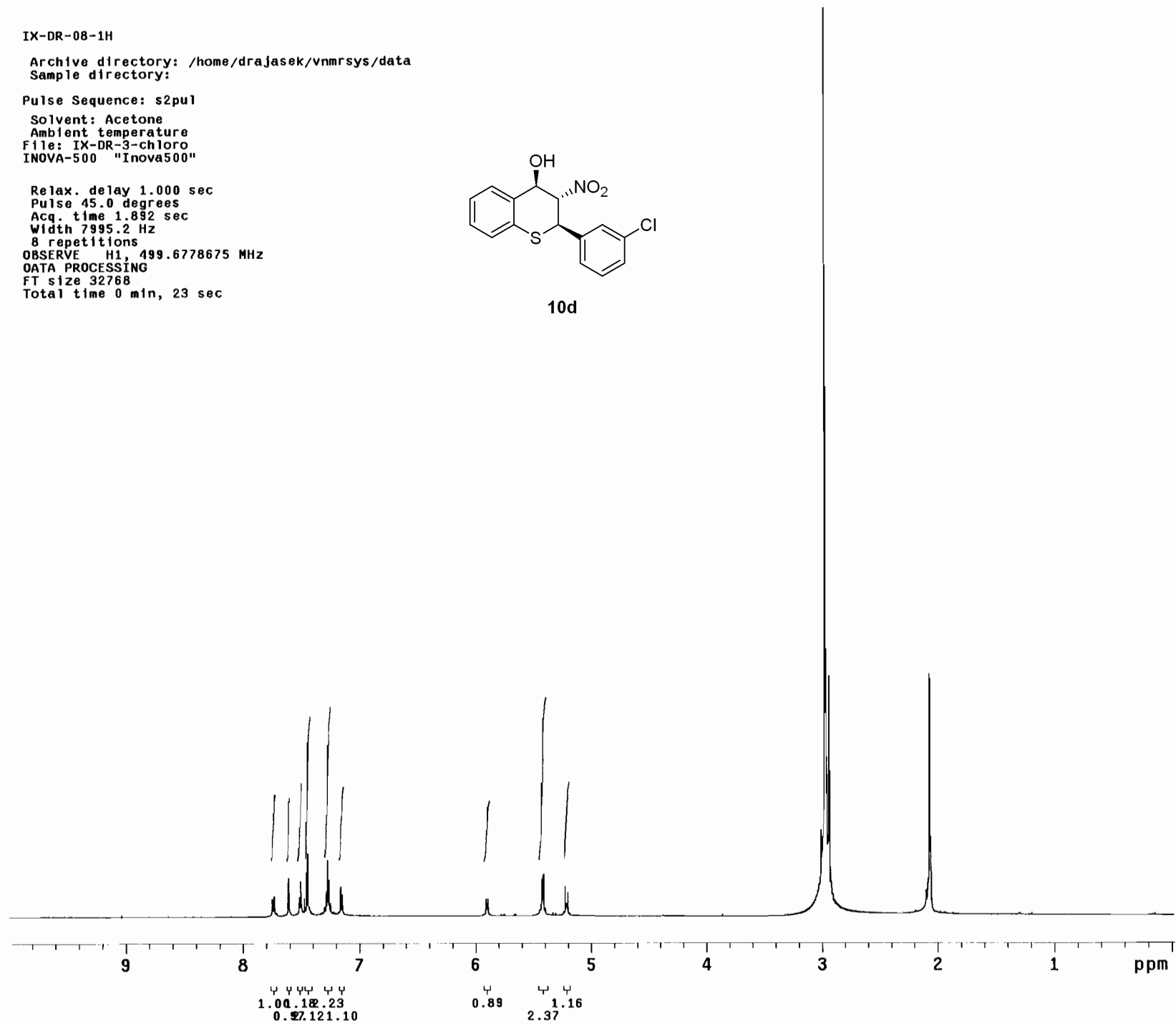
Pulse Sequence: s2pul

Solvent: Acetone
Ambient temperature
File: IX-DR-3-chloro
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7995.2 Hz
8 repetitions
OBSERVE H1, 499.6778675 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 23 sec



10d



IX-DR-08-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

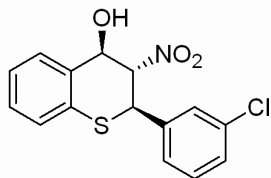
Solvent: Acetone
Ambient temperature

User: 1-14-87

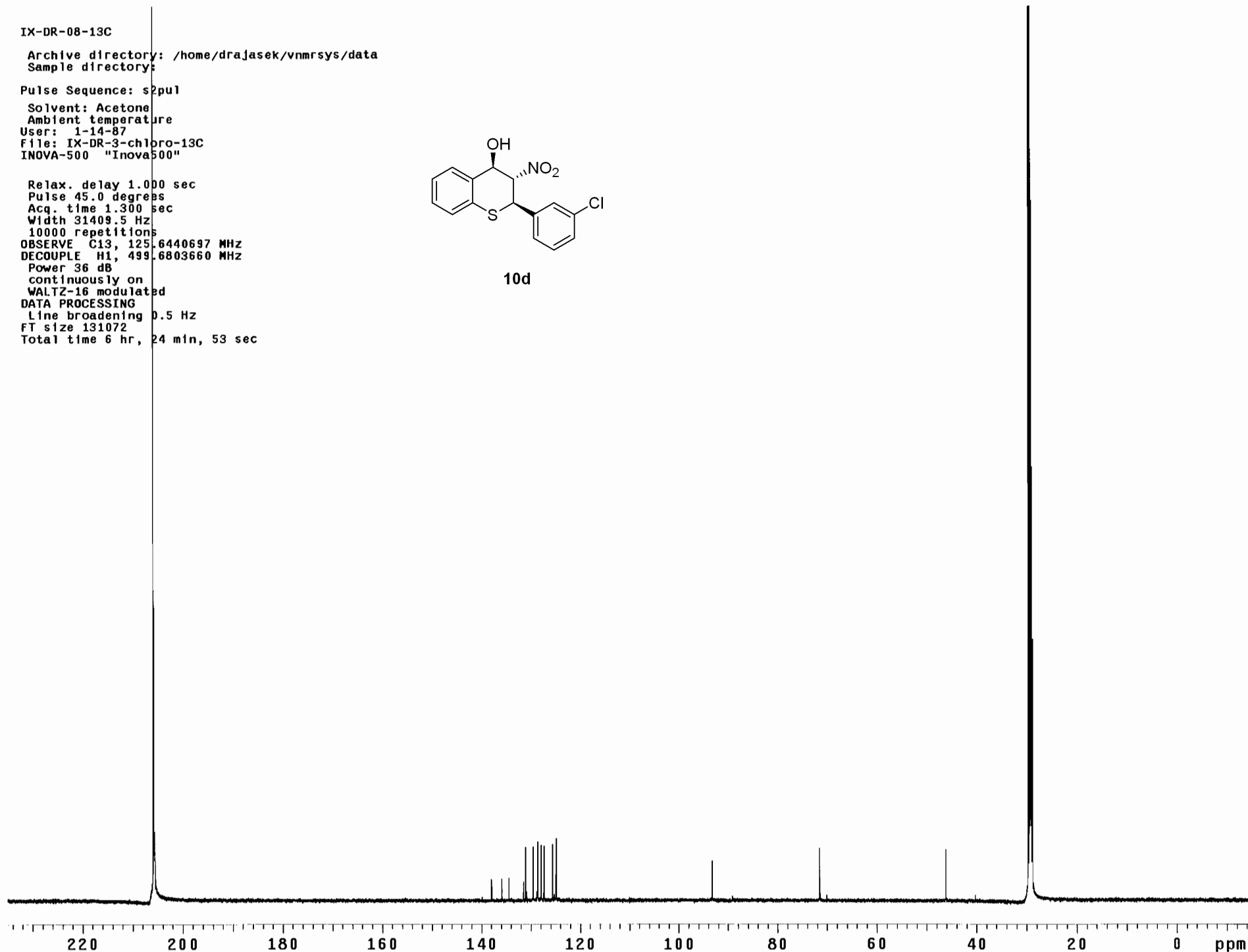
File: IX-DR-3-chloro-13C

INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
10000 repetitions
OBSERVE C13, 125.6440697 MHz
DECOUPLE H1, 499.6803660 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 6 hr, 24 min, 53 sec



10d



IX-DR-06-1H

Archive directory: /export2/home/drajasek/vnmrsys/data
Sample directory:

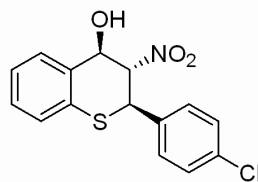
Pulse Sequence: s2pu1

Solvent: Acetone
Ambient temperature

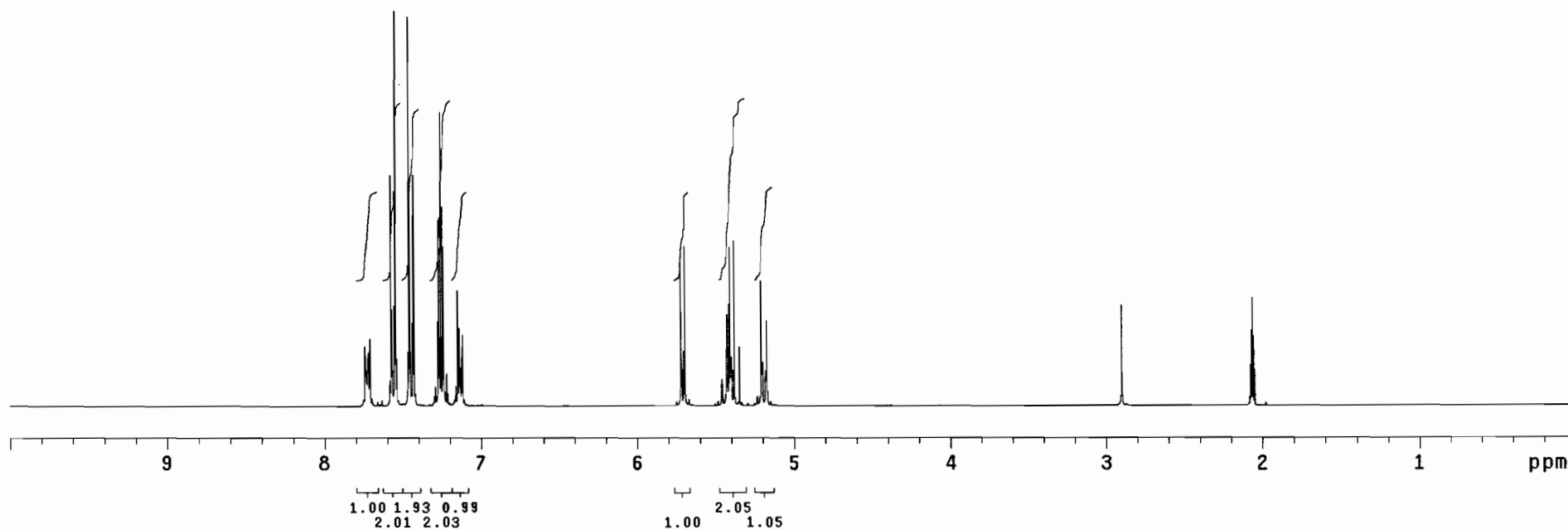
File: IX-DR-06-1H

Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
8 repetitions
OBSERVE H1, 299.7468175 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec



10e

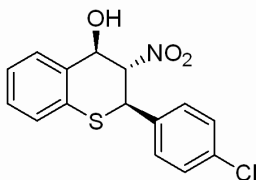


IX-DR-06-13C

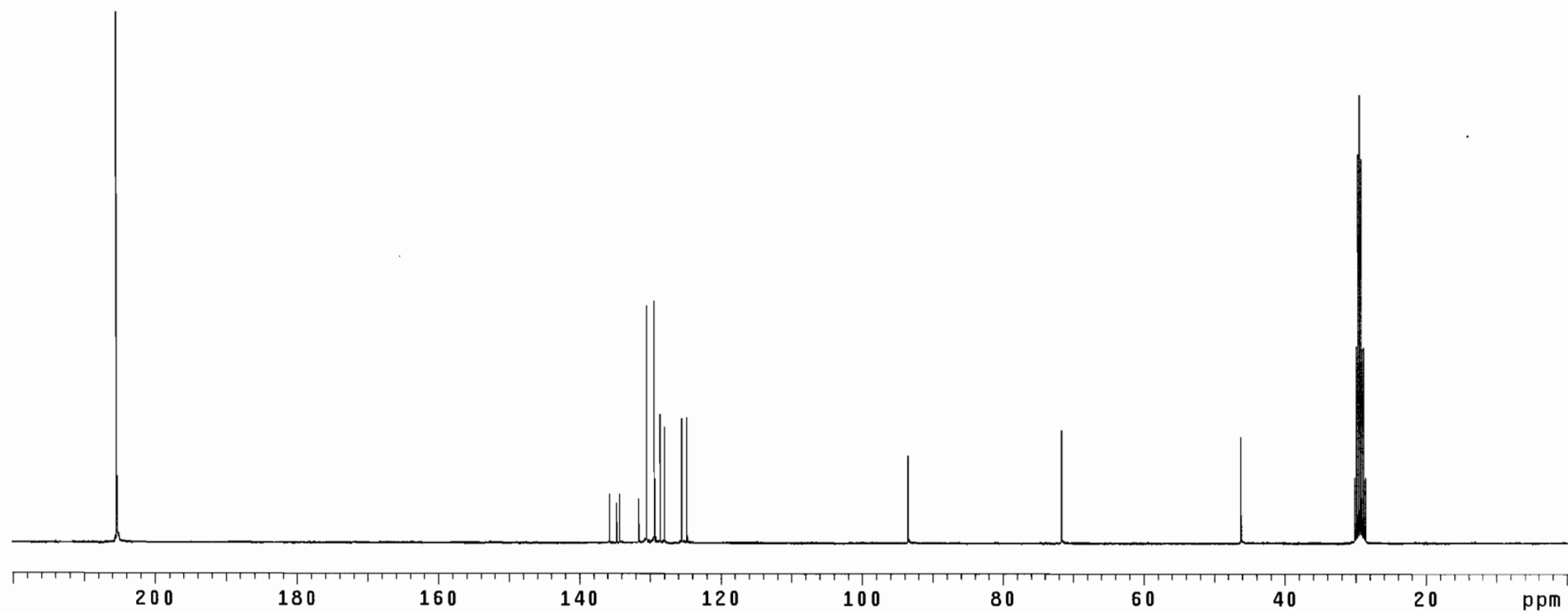
Archive directory: /export2/home/drajasek/vnmrsys/data
Sample directory:
File: CARBON

Pulse Sequence: s2pu1
Solvent: Acetone
Ambient temperature
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
5000 repetitions
OBSERVE C13, 75.3713792 MHz
DECOUPLE H1, 299.7483521 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 4 hr, 3 min, 14 sec



10e



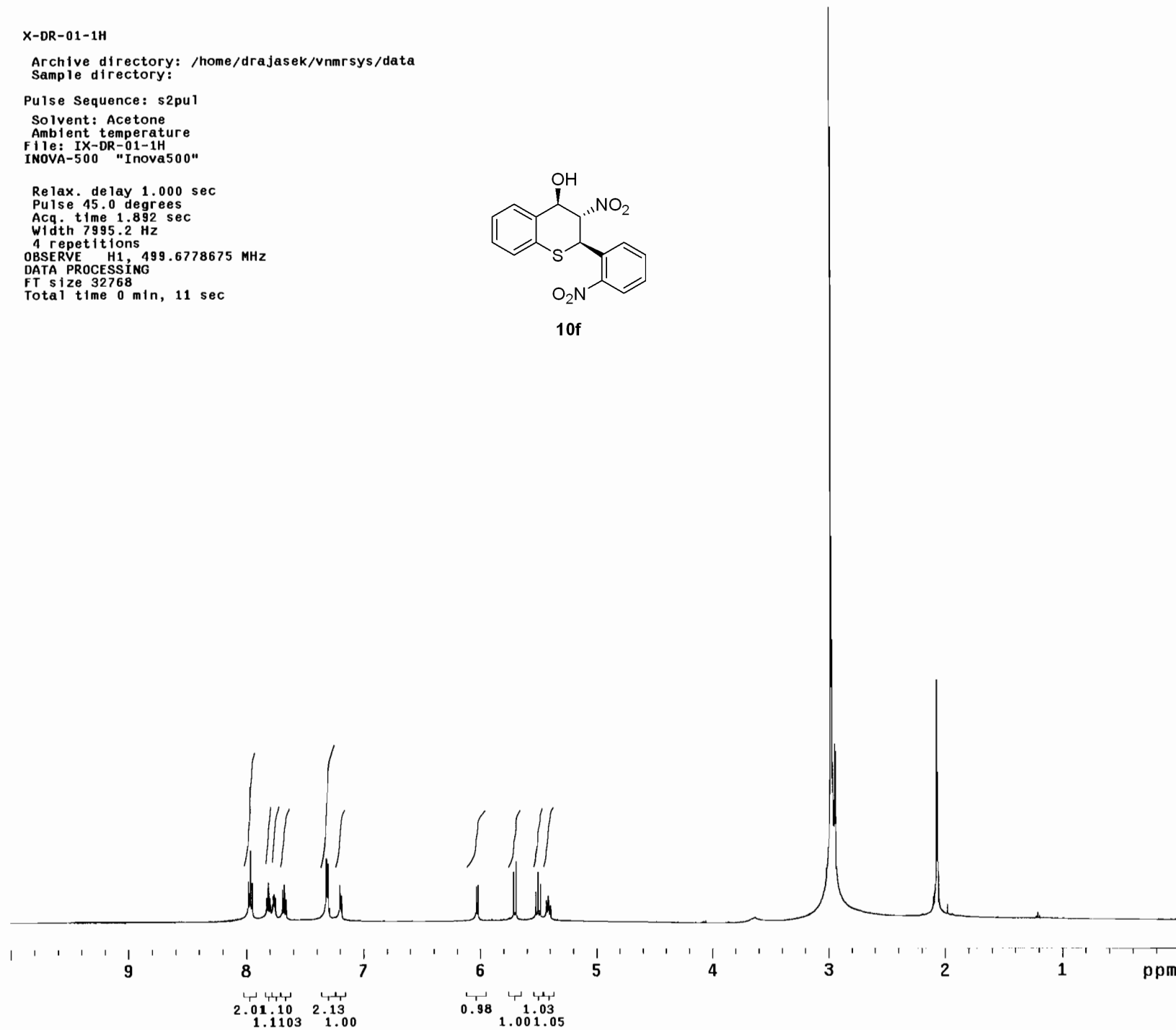
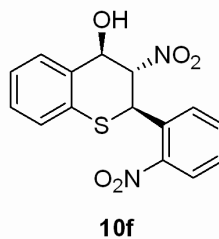
X-DR-01-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: Acetone
Ambient temperature
File: IX-DR-01-1H
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7995.2 Hz
4 repetitions
OBSERVE H1, 499.6778675 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 11 sec



IX-DR-01-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: Acetone
Ambient temperature

User: 1-14-87

File: IX-DR-01-13C

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

2816 repetitions

OBSERVE C13, 125.6440697 MHz

DECOUPLE H1, 499.6803660 MHz

Power 36 dB

continuously on

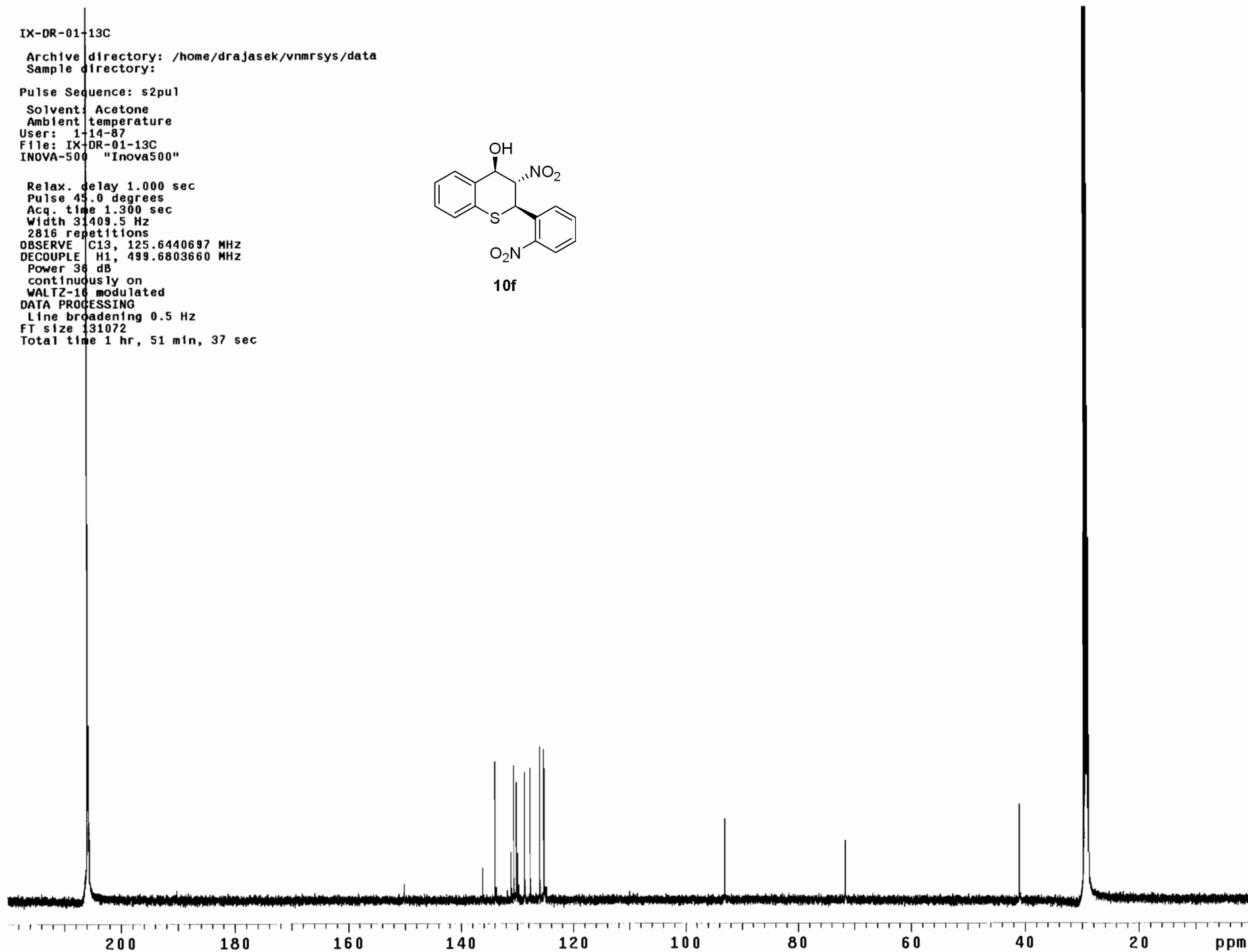
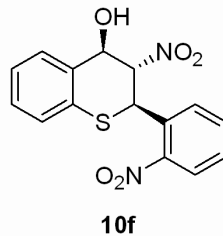
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 1 hr, 51 min, 37 sec



IX-DR-38-1H

Archive directory: /export2/home/drajasek/vnmrsys/data
Sample directory:

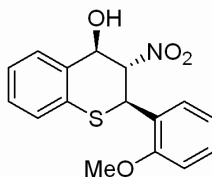
Pulse Sequence: s2pu1

Solvent: Acetone
Ambient temperature

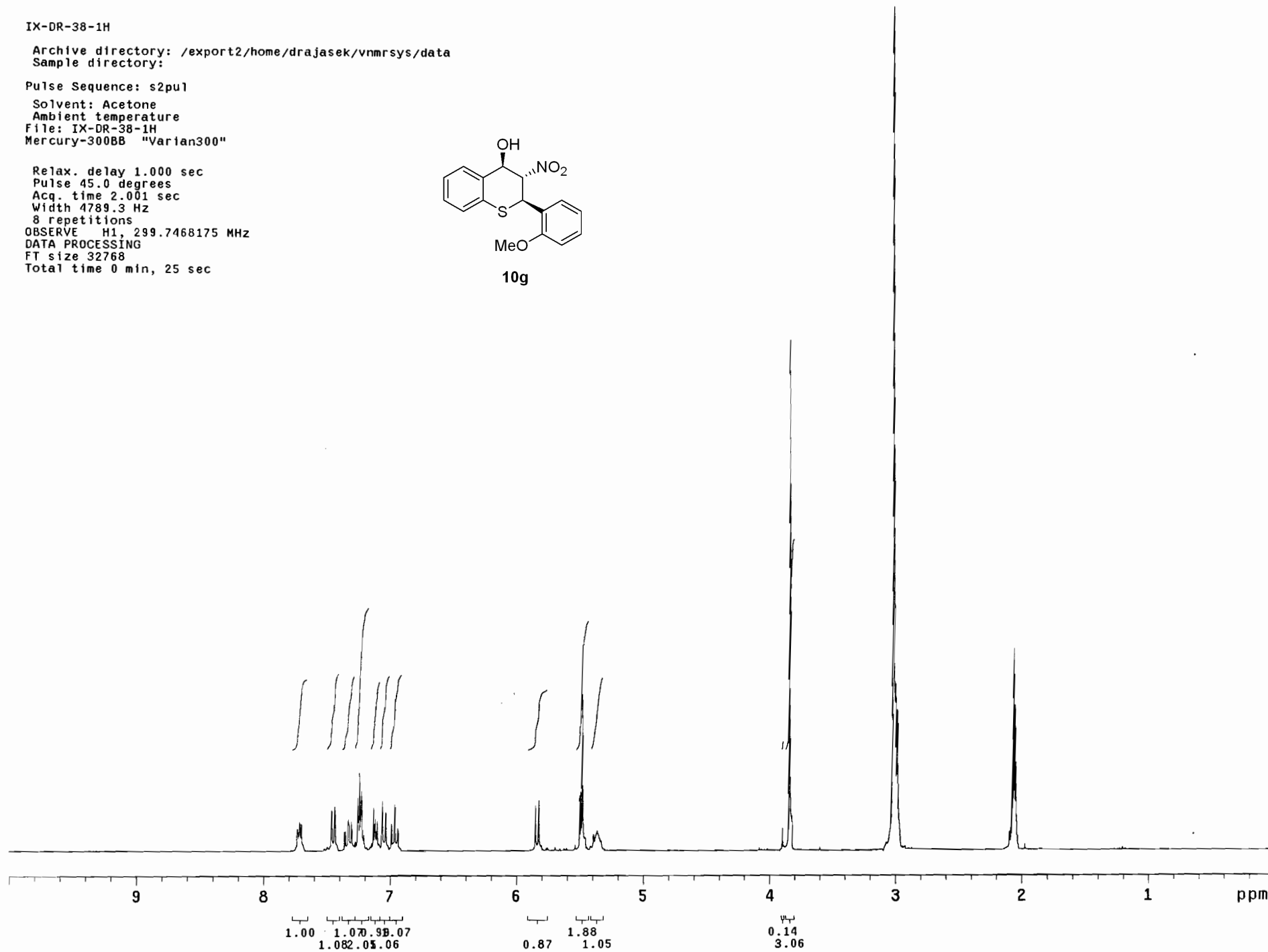
File: IX-DR-38-1H

Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
8 repetitions
OBSERVE H1, 299.7468175 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec



10g



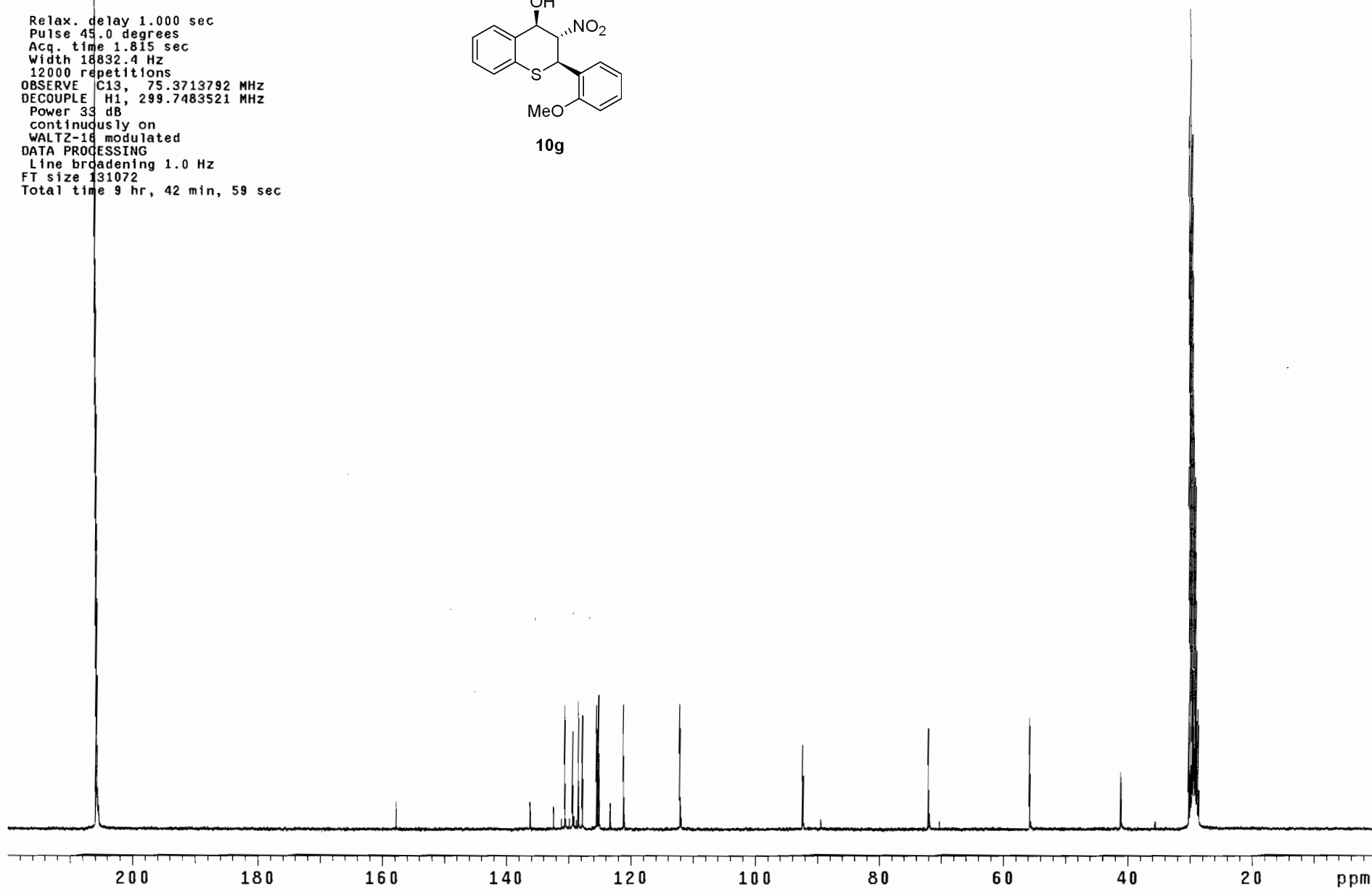
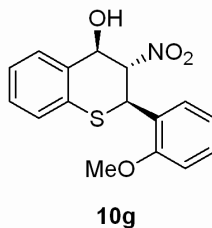
IX-DR-38-13C

Archive directory: /export2/home/drajasek/vnmrsys/data
Sample directory:
File: CARBON

Pulse Sequence: s2pu1

Solvent: Acetone
Ambient temperature
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
12000 repetitions
OBSERVE C13, 75.3713792 MHz
DECOUPLE H1, 299.7483521 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 9 hr, 42 min, 59 sec



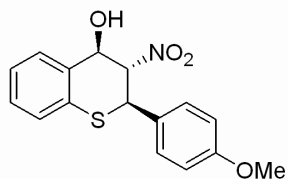
VIII-DR-145-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

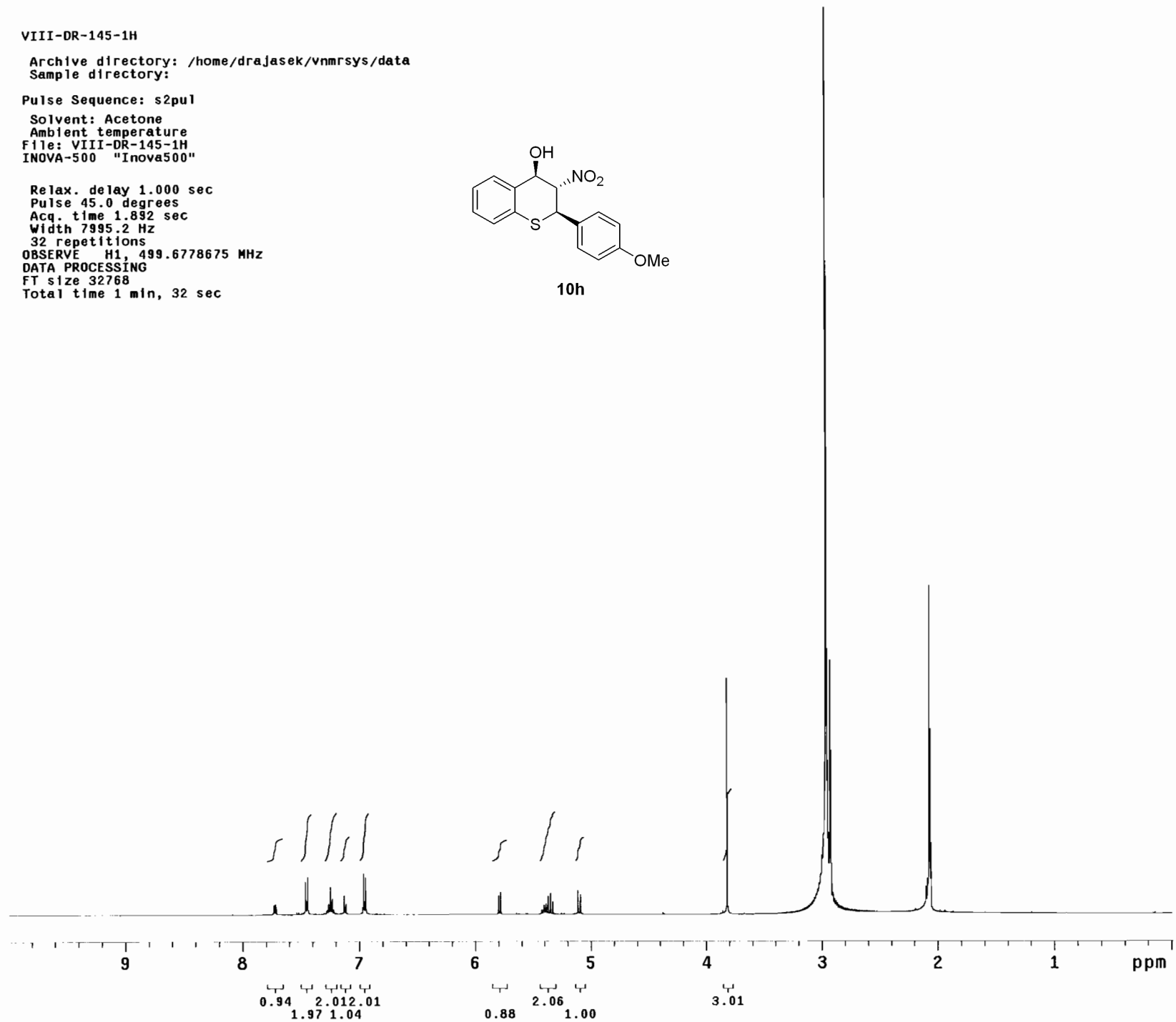
Pulse Sequence: s2pul

Solvent: Acetone
Ambient temperature
File: VIII-DR-145-1H
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7995.2 Hz
32 repetitions
OBSERVE H1, 499.6778675 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 32 sec



10h



VIII-DR-145-13C

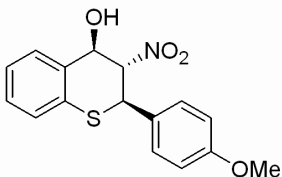
Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

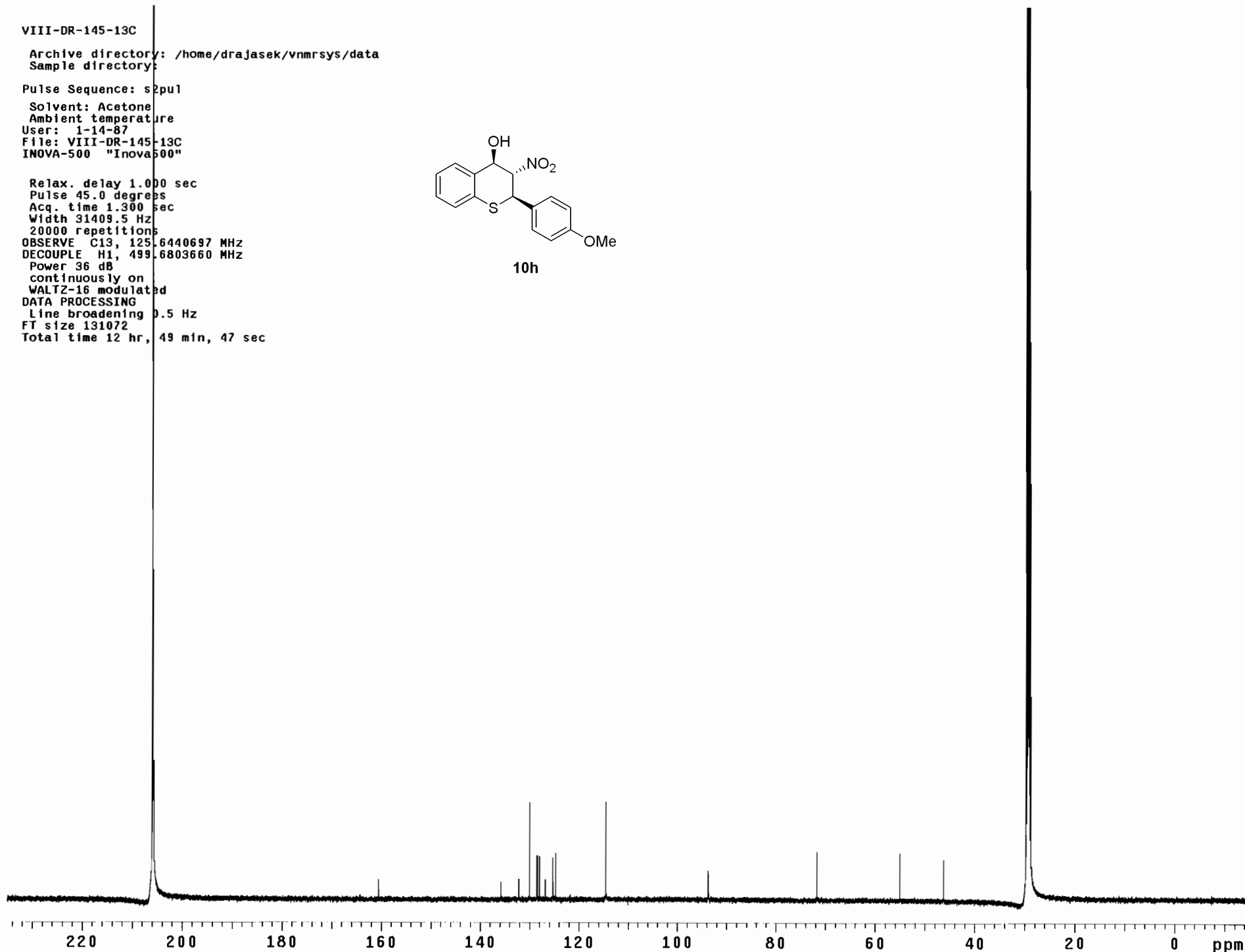
Solvent: Acetone
Ambient temperature

User: 1-14-87
File: VIII-DR-145-13C
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
20000 repetitions
OBSERVE C13, 125.6440697 MHz
DECOUPLE H1, 499.6803660 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 12 hr, 49 min, 47 sec



10h



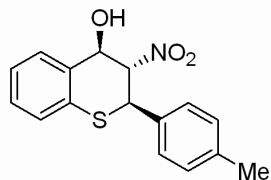
IX-DR-03-1H

Archive directory: /export2/home/drajasek/vnmrsys/data
Sample directory:

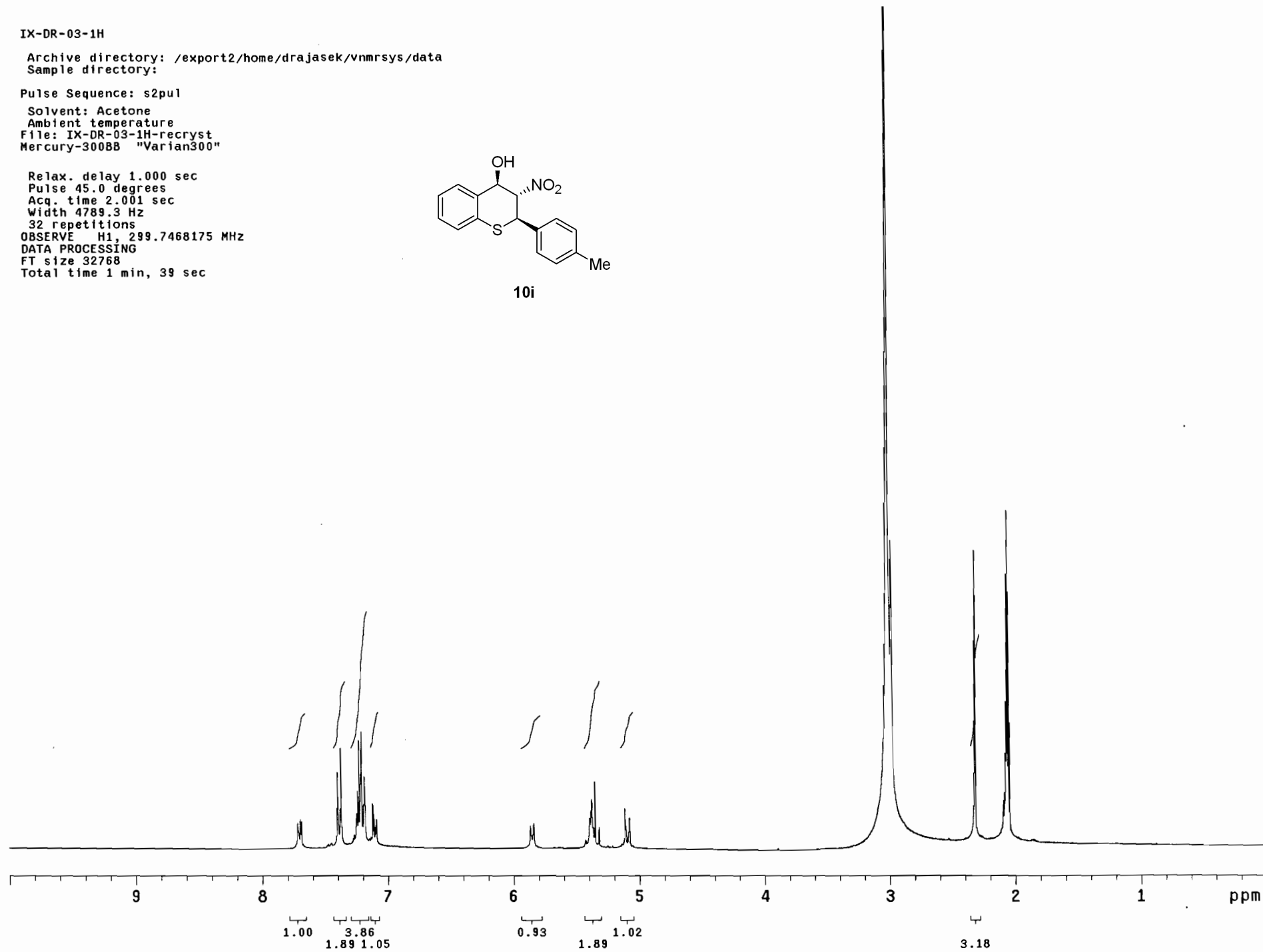
Pulse Sequence: s2pul

Solvent: Acetone
Ambient temperature
File: IX-DR-03-1H-recryst
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
32 repetitions
OBSERVE H1, 299.7468175 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 39 sec



10i



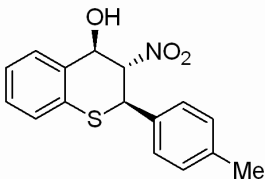
IX-DR-03-13C

Archive directory: /export2/home/drajasek/vnmrsys/data
Sample directory:

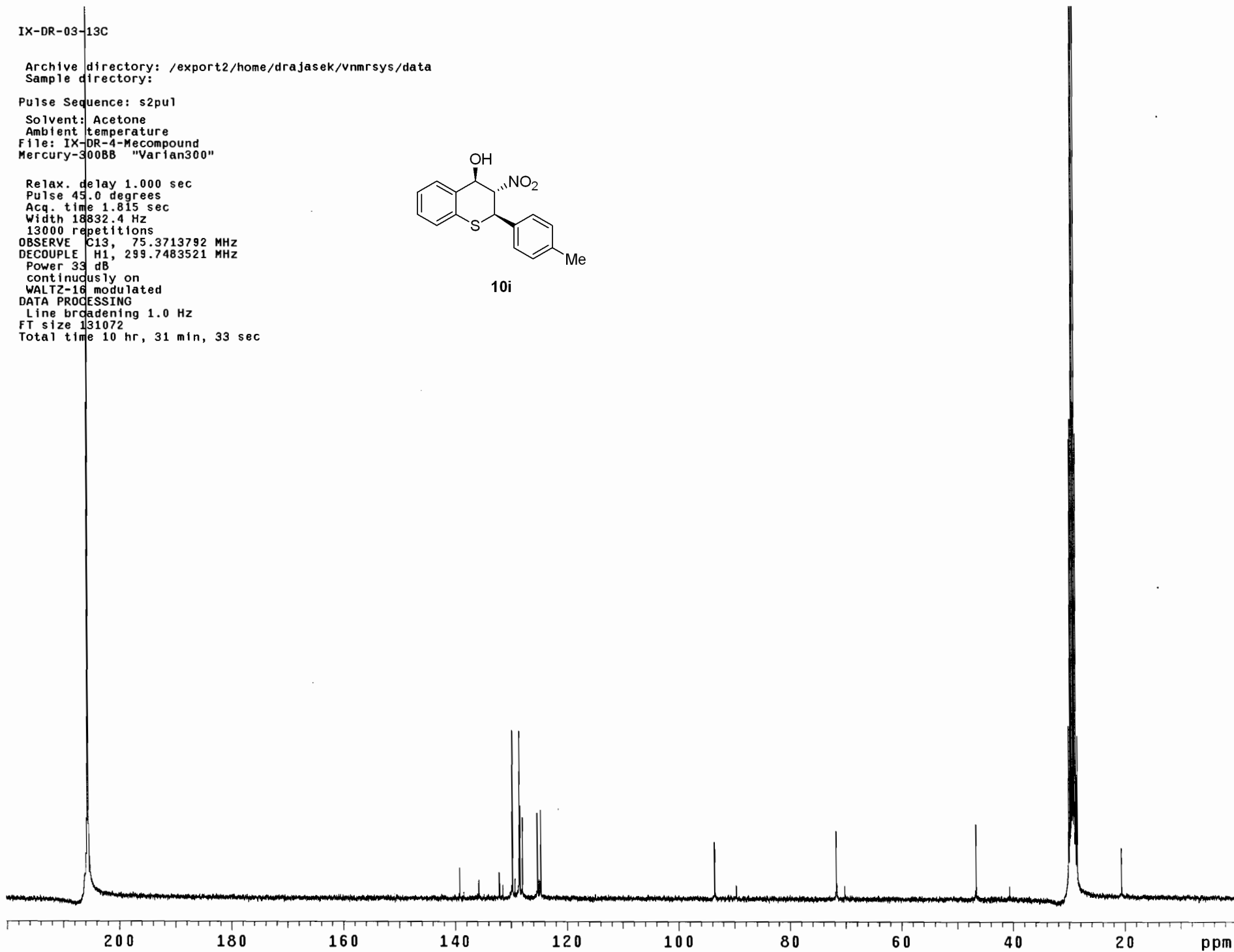
Pulse Sequence: s2pu1

Solvent: Acetone
Ambient temperature
File: IX-DR-4-Mecomound
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
13000 repetitions
OBSERVE C13, 75.3713792 MHz
DECOUPLE H1, 299.7483521 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 10 hr, 31 min, 33 sec



10i



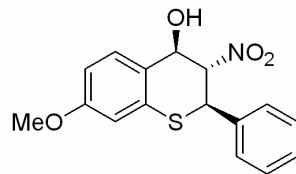
IX-DR-16-1H

Archive directory: /export2/home/drajasek/vnmrsys/data
Sample directory:

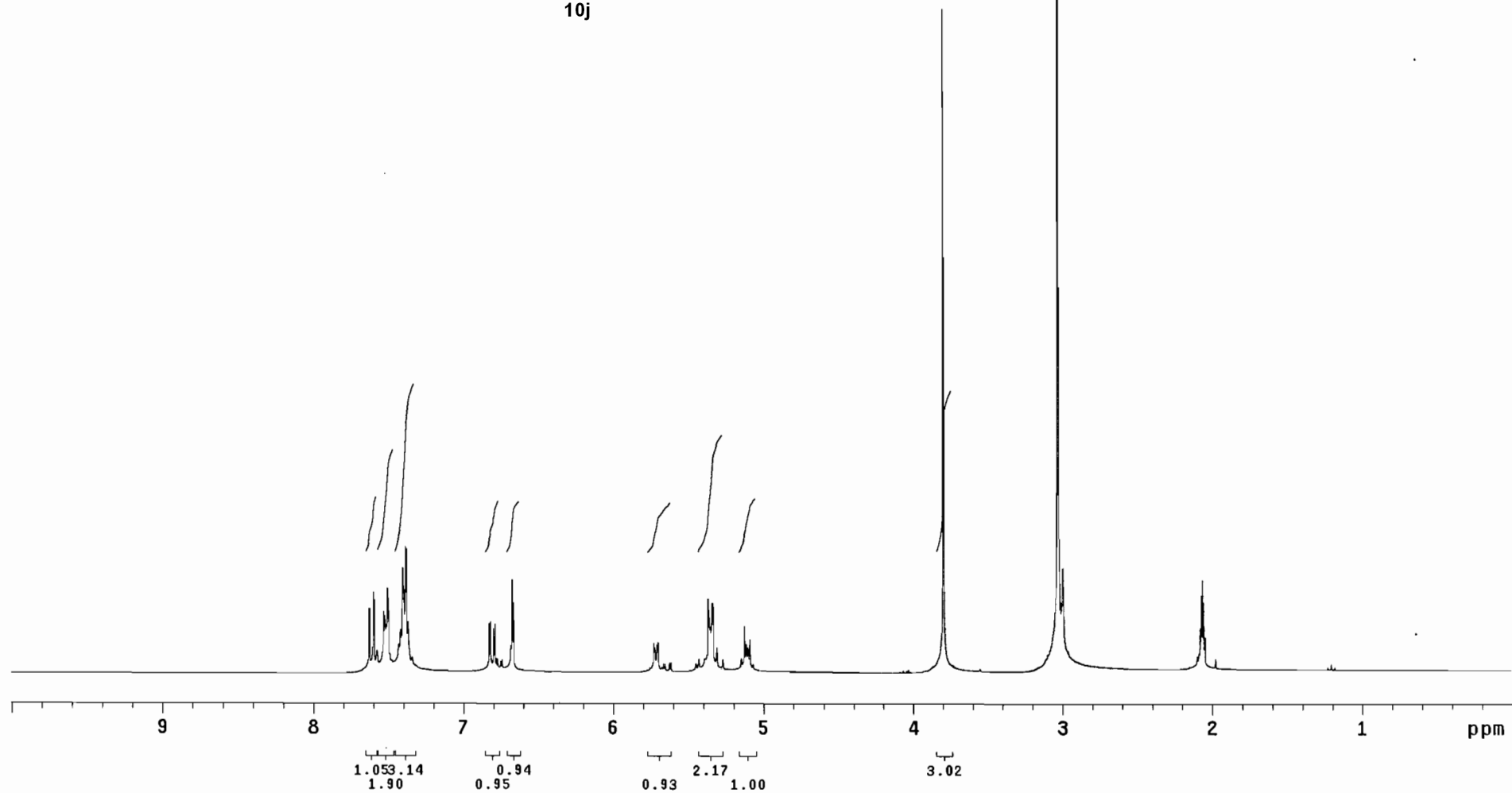
Pulse Sequence: s2pu1

Solvent: Acetone
Ambient temperature
File: IX-DR-16-1Ha
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
8 repetitions
OBSERVE H1, 299.7468175 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec



10j



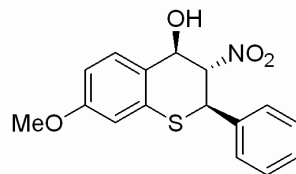
IX-DR-16-13C

Archive directory: /export2/home/drajasek/vnmrsys/data
Sample directory:

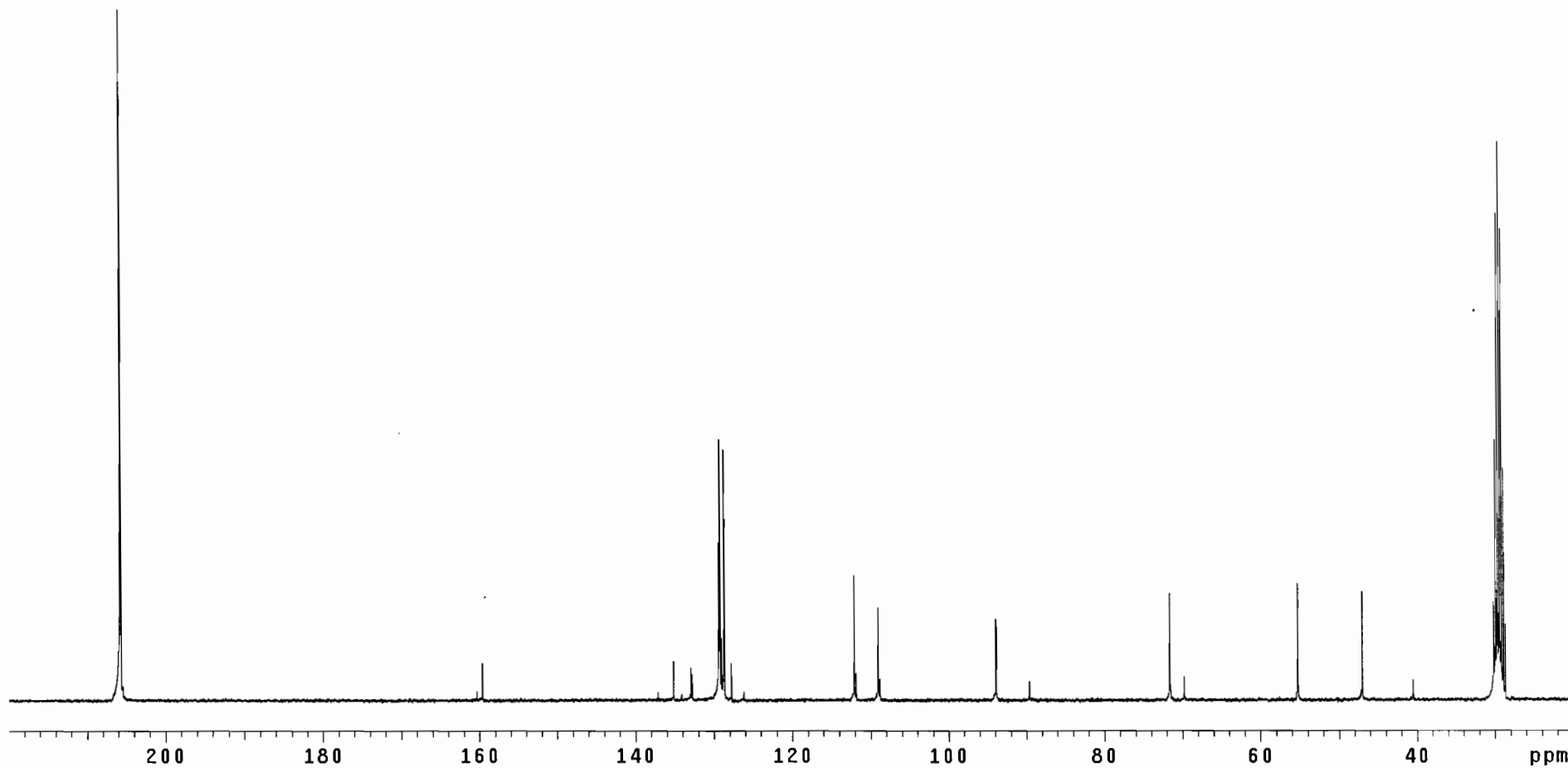
Pulse Sequence: s2pu1

Solvent: Acetone
Ambient temperature
File: IX-DR-16-13Ca
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
7000 repetitions
OBSERVE C13, 75.3713792 MHz
DECOUPLE H1, 299.7483521 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 5 hr, 40 min, 18 sec



10j

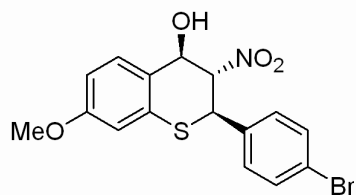


IX-DR-14-1H

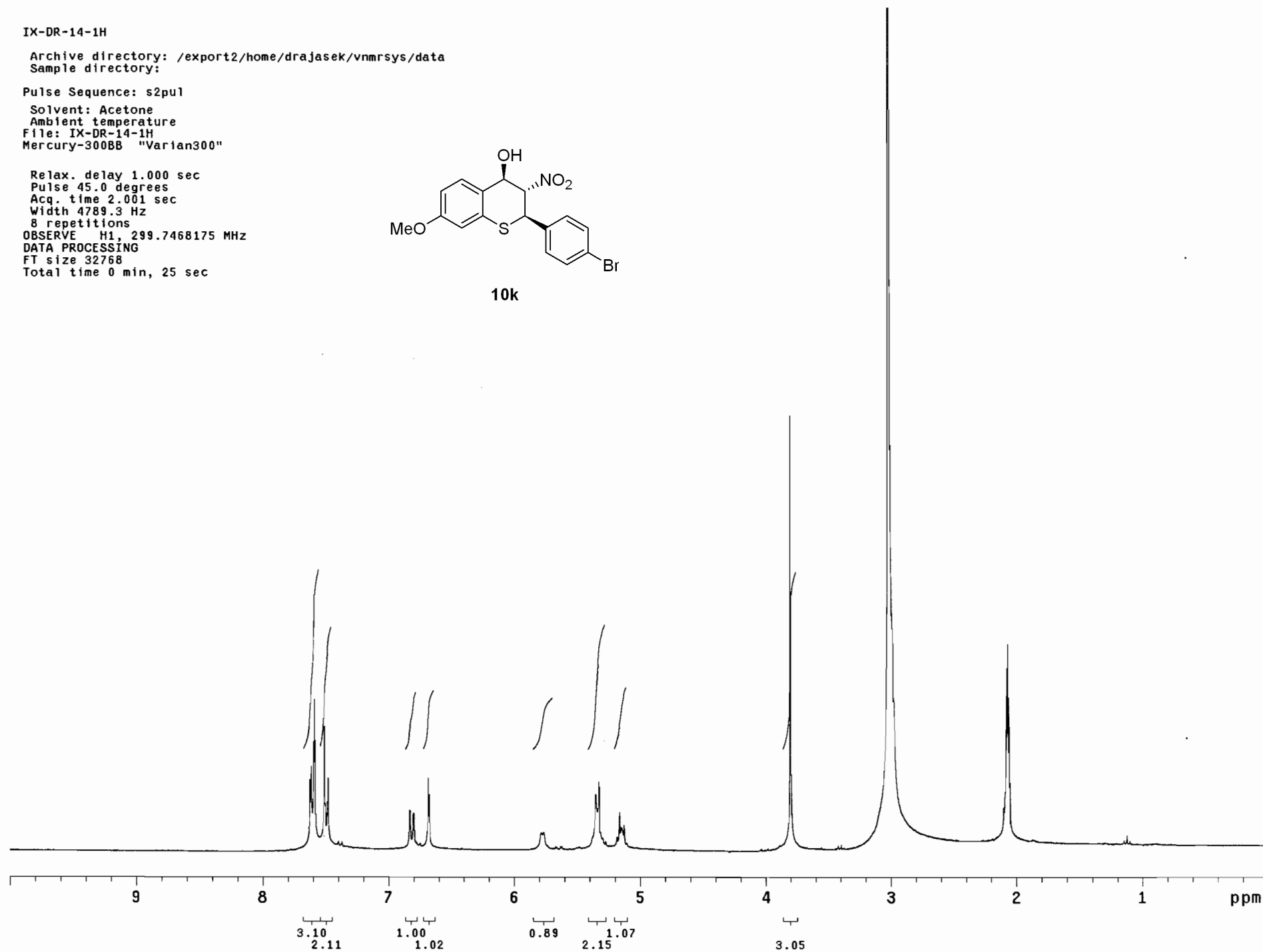
Archive directory: /export2/home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1
Solvent: Acetone
Ambient temperature
File: IX-DR-14-1H
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
8 repetitions
OBSERVE H1, 299.7468175 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec



10k



IX-DR-14-13C

Archive directory: /export2/home/drajasek/vnmrsys/data

Sample directory:

Pulse Sequence: s2pul

Solvent: Acetone

Ambient temperature

File: IX-DR-14-13C

Mercury-300BB "Varian300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 18832.4 Hz

5000 repetitions

OBSERVE C13, 75.8713792 MHz

DECOUPLE H1, 299.7483521 MHz

Power 33 dB

continuously on

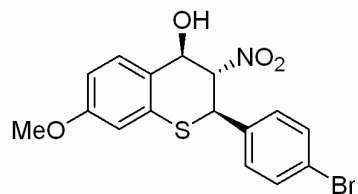
WALTZ-16 modulated

DATA PROCESSING

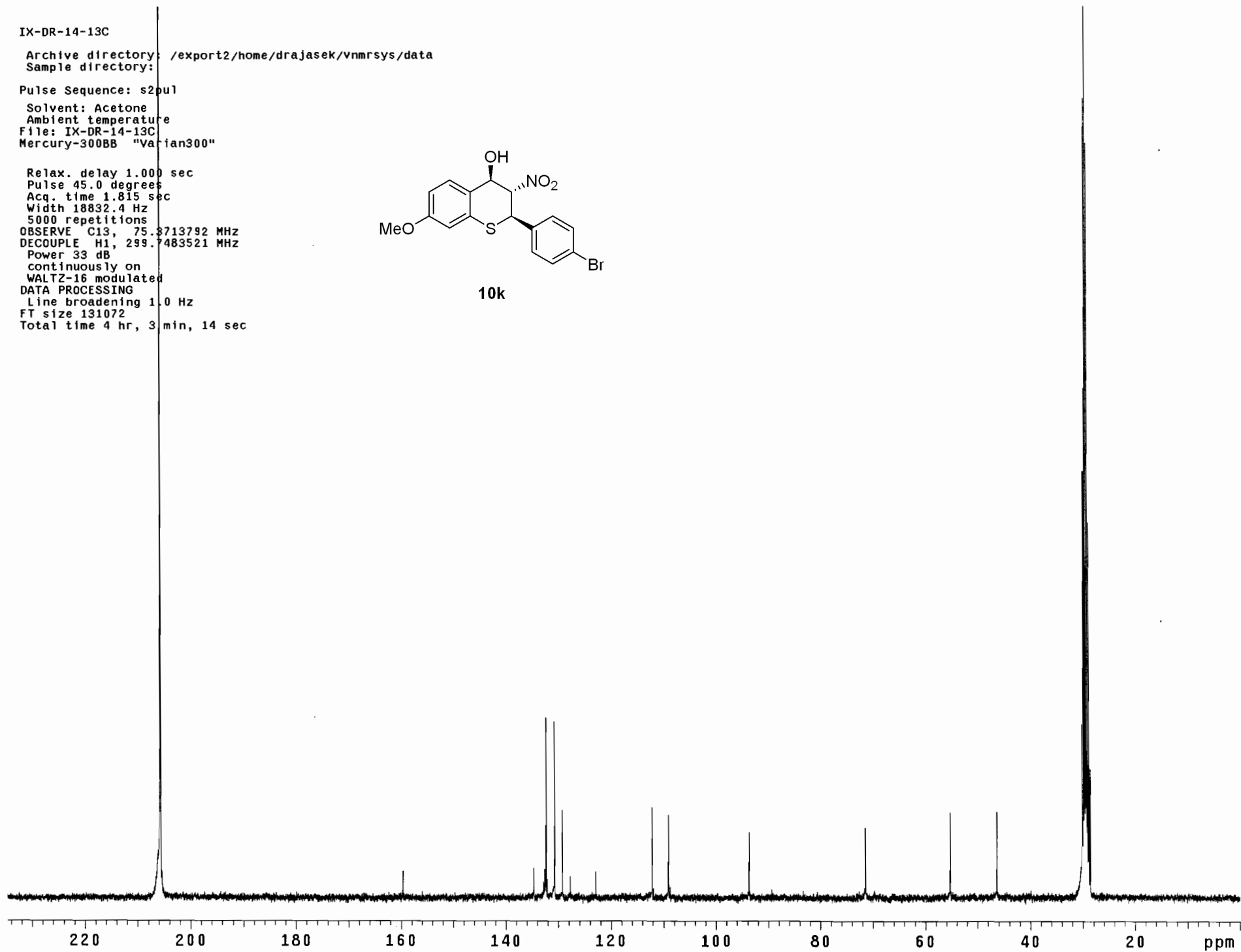
Line broadening 1.0 Hz

FT size 131072

Total time 4 hr, 3 min, 14 sec



10k



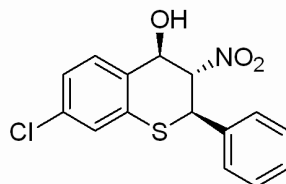
IX-DR-20-1H

Archive directory: /export2/home/drajasek/vnmrsys/data
Sample directory:

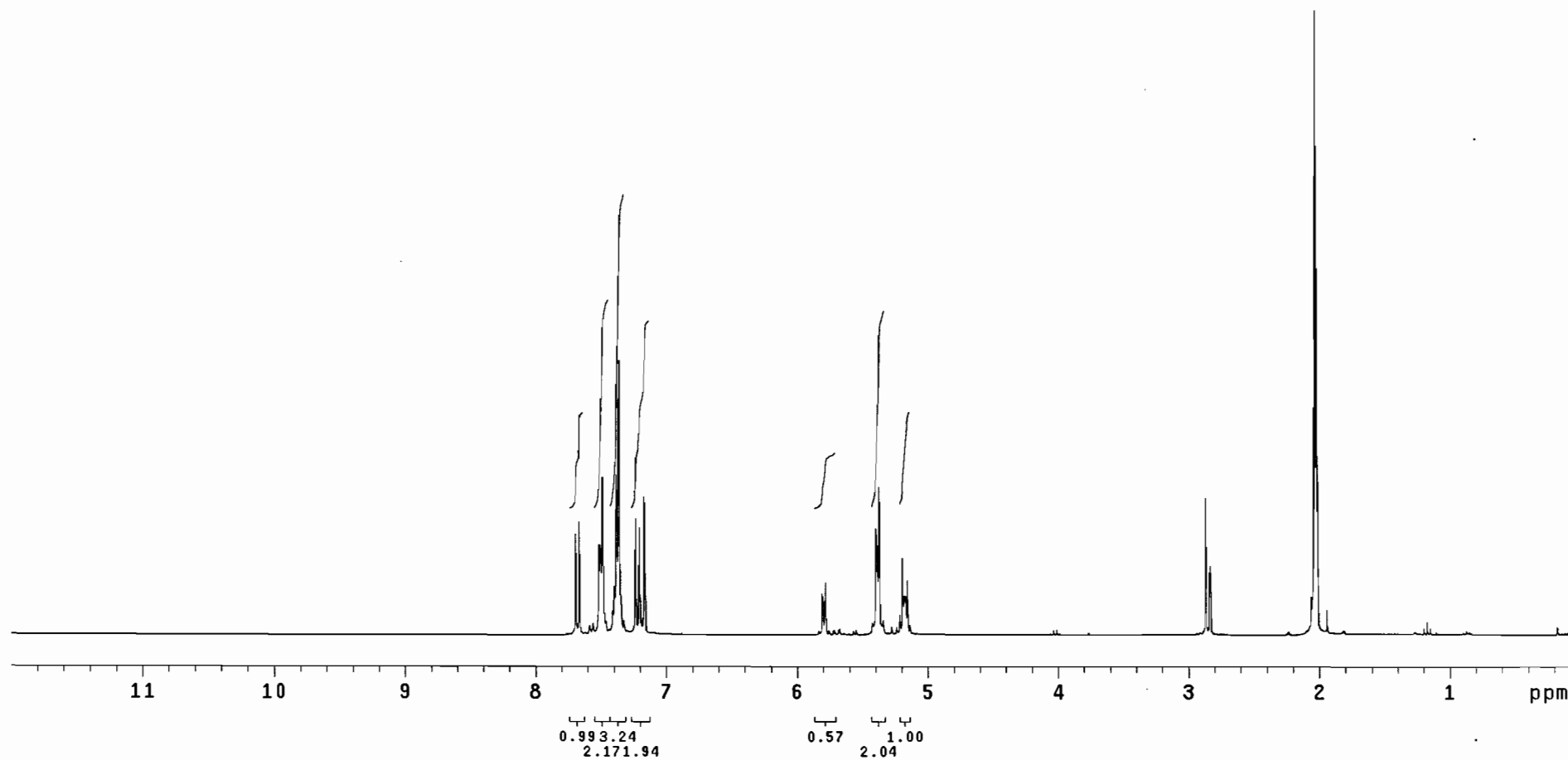
Pulse Sequence: s2pu1

Solvent: Acetone
Ambient temperature
File: IX-DR-20-1H
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
8 repetitions
OBSERVE H1, 299.7469979 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec



10I



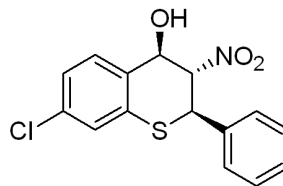
IX-DR-20-13C

Archive directory: /export2/home/drajasek/vnmrsys/data
Sample directory:

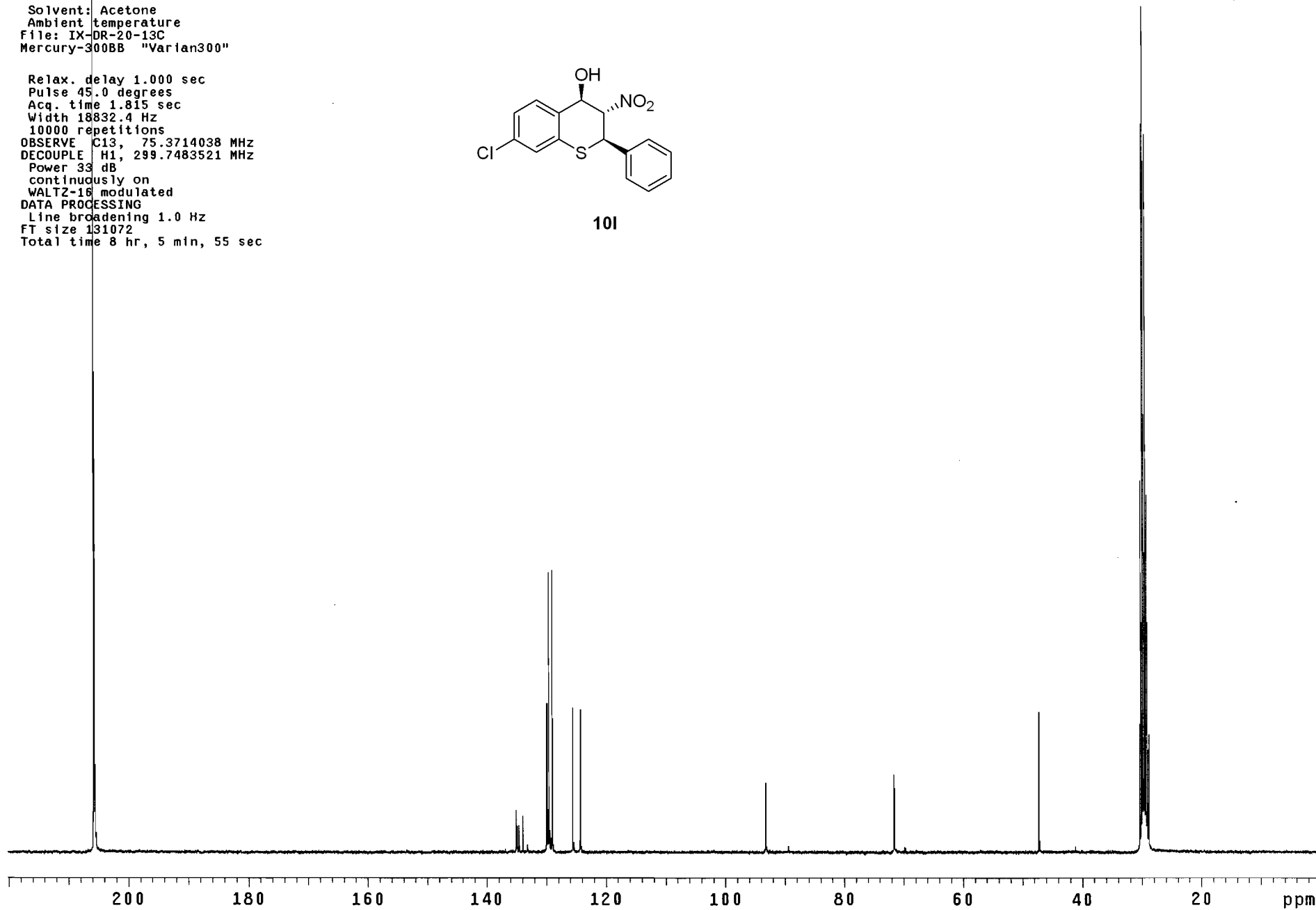
Pulse Sequence: s2pu1

Solvent: Acetone
Ambient temperature
File: IX-DR-20-13C
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
10000 repetitions
OBSERVE C13, 75.3714038 MHz
DECOUPLE H1, 299.7483521 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 8 hr, 5 min, 55 sec

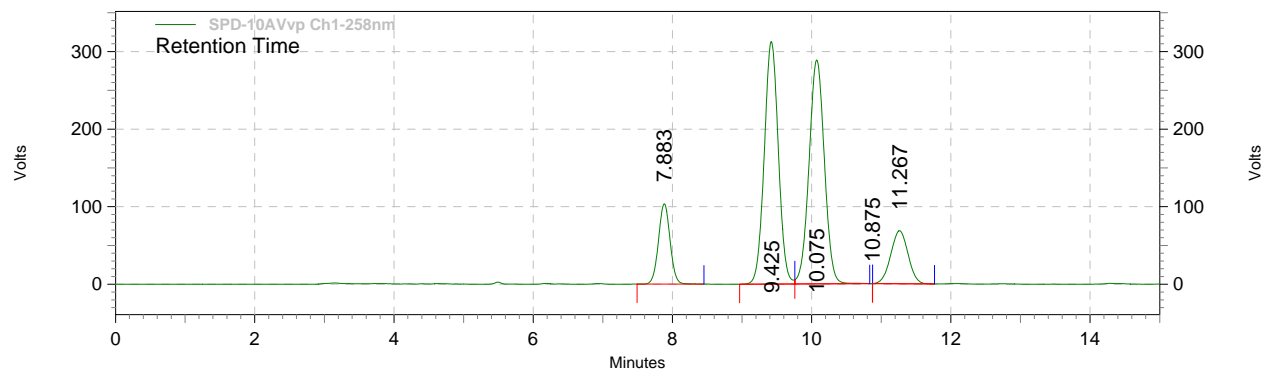


101



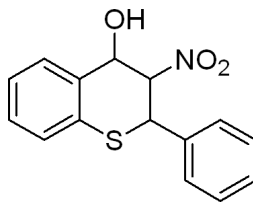
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\VIII-DR-84-B
User: System
Acquired: 3/21/2007 11:57:54 AM
Printed: 5/16/2007 2:10:38 PM



SPD-10AVvp Ch1-258nm Results

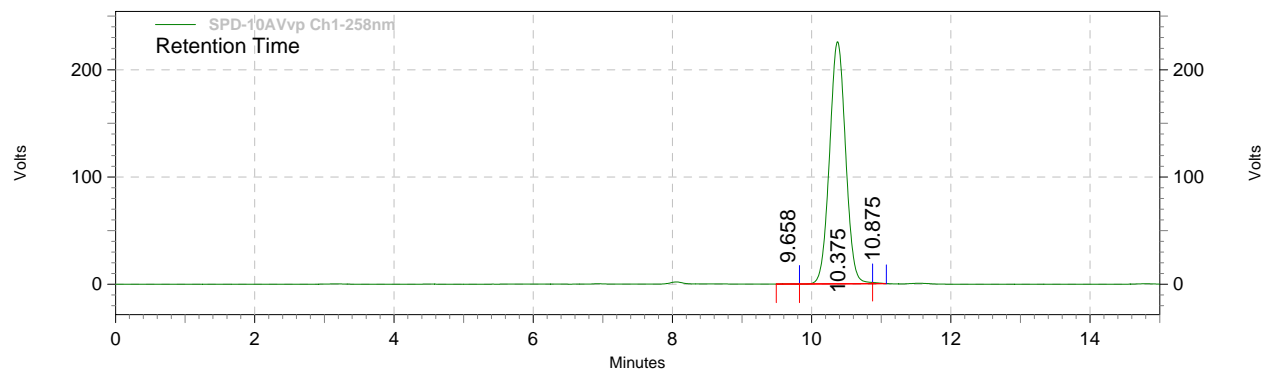
Retention Time	Area	Area %	Height	Height %
7.883	1197571	10.68	103625	13.41
9.425	4401885	39.27	312347	40.41
10.075	4430923	39.53	288675	37.35
10.875	0	0.00	0	0.00
11.267	1178436	10.51	68292	8.84
Totals	11208815	100.00	772939	100.00



10a, racemic

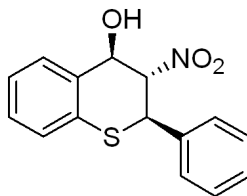
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\VIII-DR-146-2
User: System
Acquired: 4/15/2007 11:43:43 AM
Printed: 5/16/2007 2:11:11 PM



SPD-10AVvp
Ch1-258nm
Results

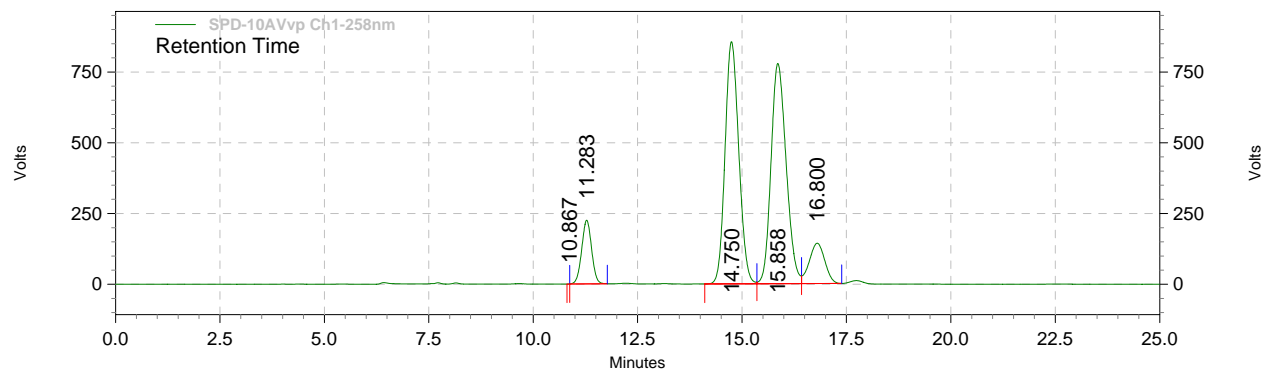
Retention Time	Area	Area %	Height	Height %
9.658	788	0.02	112	0.05
10.375	3604068	99.81	225665	99.49
10.875	6129	0.17	1049	0.46
Totals	3610985	100.00	226826	100.00



10a, >99%ee

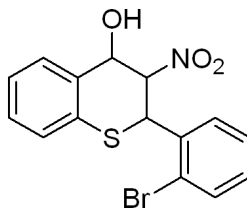
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\VIII-DR-148-B
User: System
Acquired: 4/16/2007 9:25:42 AM
Printed: 5/16/2007 2:11:38 PM



SPD-10AVvp Ch1-258nm Results

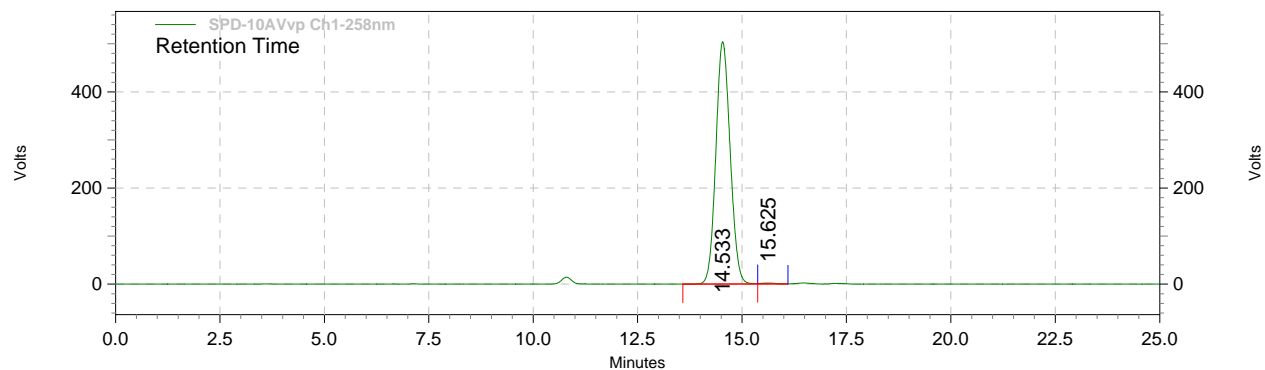
Retention Time	Area	Area %	Height	Height %
10.867	519	0.00	289	0.01
11.283	3694827	7.94	224820	11.23
14.750	19558310	42.00	855489	42.74
15.858	19628064	42.15	778337	38.89
16.800	3681084	7.91	142590	7.12
Totals	46562804	100.00	2001525	100.00



10b, racemic

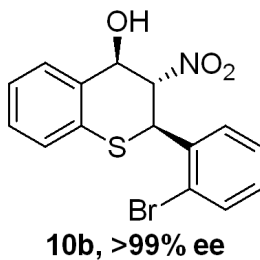
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-05-D
User: System
Acquired: 5/17/2007 3:06:11 PM
Printed: 5/17/2007 3:43:28 PM



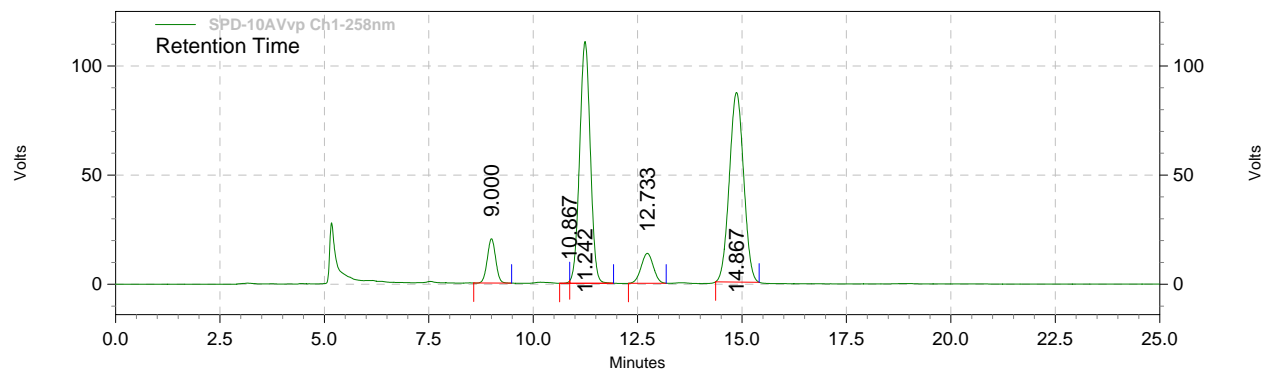
SPD-10AVvp
Ch1-258nm
Results

Retention Time	Area	Area %	Height	Height %
14.533	11999158	99.63	503967	99.62
15.625	44167	0.37	1905	0.38
Totals	12043325	100.00	505872	100.00



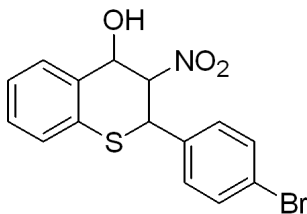
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\VIII-DR-147-RACEMIC
User: System
Acquired: 4/15/2007 11:14:44 AM
Printed: 5/16/2007 2:12:43 PM



SPD-10AVvp Ch1-258nm Results

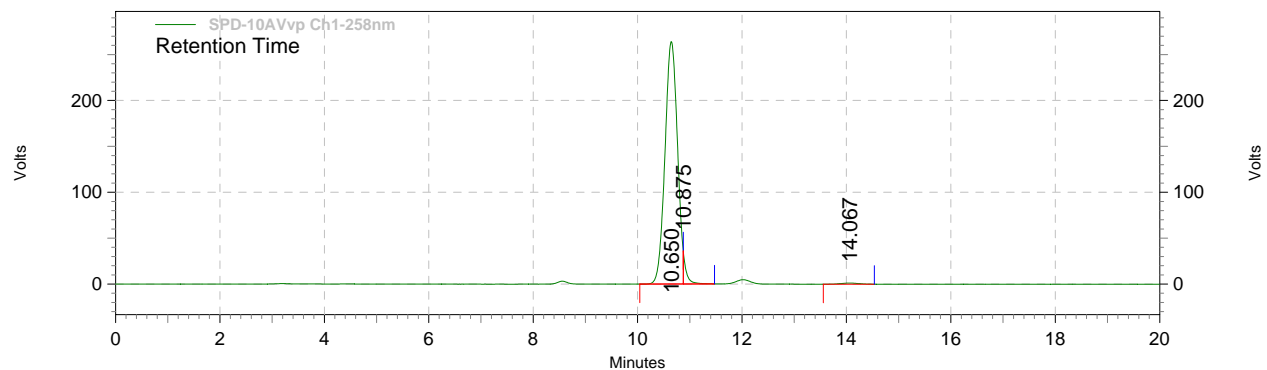
Retention Time	Area	Area %	Height	Height %
9.000	282849	6.16	20309	8.74
10.867	2990	0.07	969	0.42
11.242	1961037	42.73	110644	47.59
12.733	277677	6.05	13712	5.90
14.867	2064754	44.99	86842	37.36
Totals	4589307	100.00	232476	100.00



10c, racemic

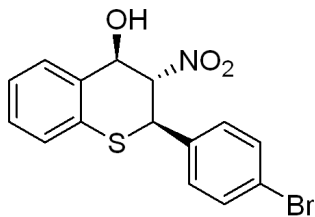
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\VIII-DR-149-C
User: System
Acquired: 4/16/2007 10:33:32 AM
Printed: 5/16/2007 2:13:32 PM



SPD-10AVvp
Ch1-258nm
Results

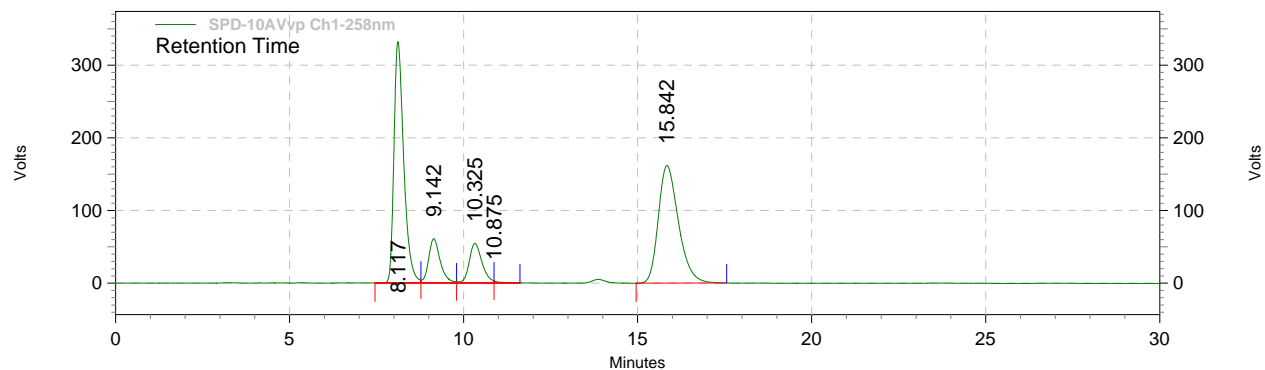
Retention Time	Area	Area %	Height	Height %
10.650	4350335	96.33	263932	87.61
10.875	136925	3.03	36076	11.98
14.067	28930	0.64	1243	0.41
Totals	4516190	100.00	301251	100.00



10c, 99% ee

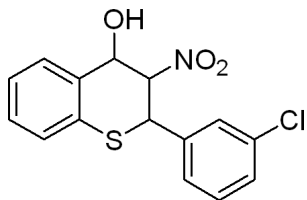
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-07-RACEMIC-A
User: System
Acquired: 4/20/2007 10:48:27 AM
Printed: 5/16/2007 2:14:00 PM



SPD-10AVvp Ch1-258nm Results

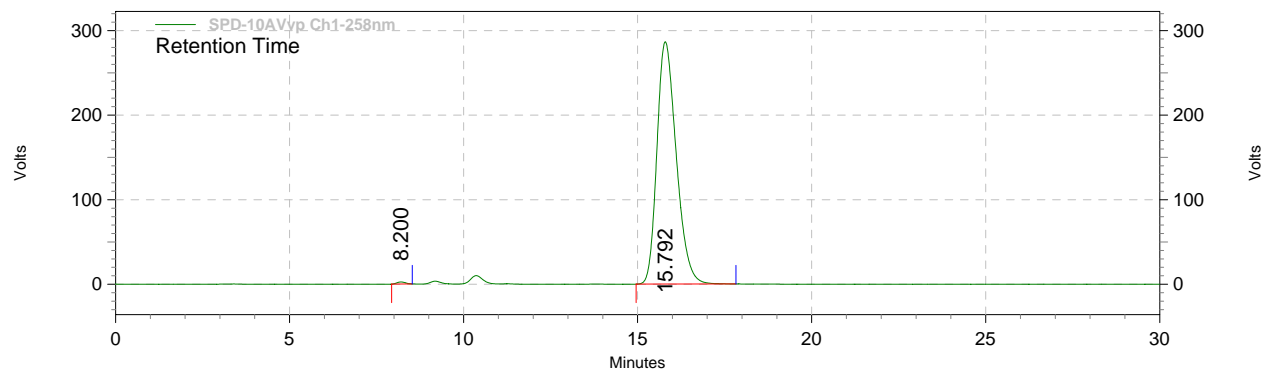
Retention Time	Area	Area %	Height	Height %
8.117	6484786	41.00	331989	54.25
9.142	1411334	8.92	60919	9.95
10.325	1391025	8.79	54515	8.91
10.875	33150	0.21	2757	0.45
15.842	6497740	41.08	161792	26.44
Totals	15818035	100.00	611972	100.00



10d, racemic

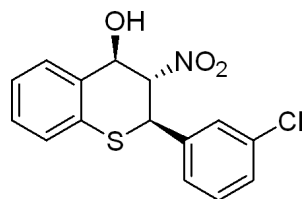
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-08-A
User: System
Acquired: 4/20/2007 5:10:44 PM
Printed: 5/16/2007 2:14:45 PM



SPD-10AVvp
Ch1-258nm
Results

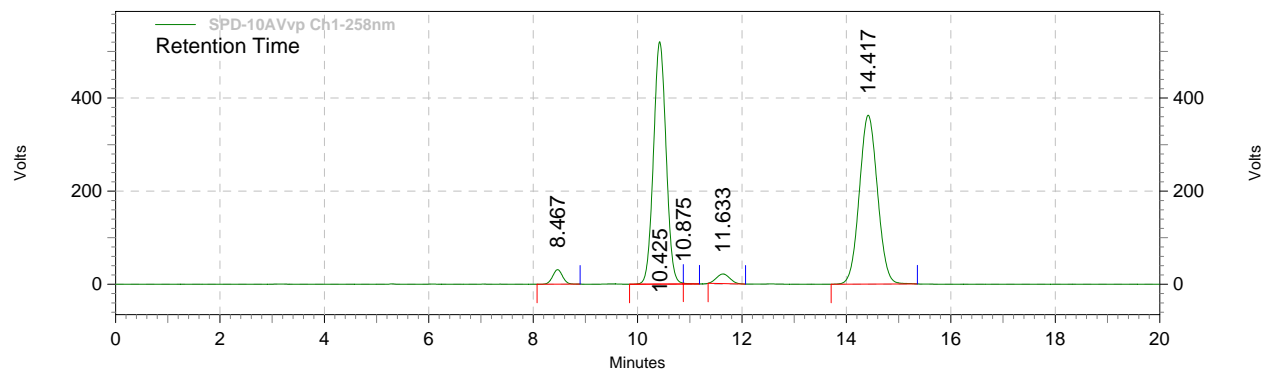
Retention Time	Area	Area %	Height	Height %
8.200	41681	0.38	2486	0.86
15.792	11056539	99.62	286553	99.14
Totals	11098220	100.00	289039	100.00



10d, >99% ee

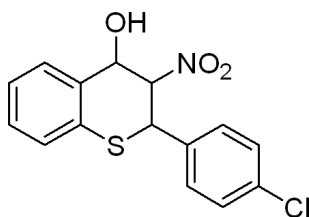
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-02-RACEMIC
User: System
Acquired: 4/19/2007 2:23:17 PM
Printed: 5/16/2007 2:15:13 PM



SPD-10AVvp Ch1-258nm Results

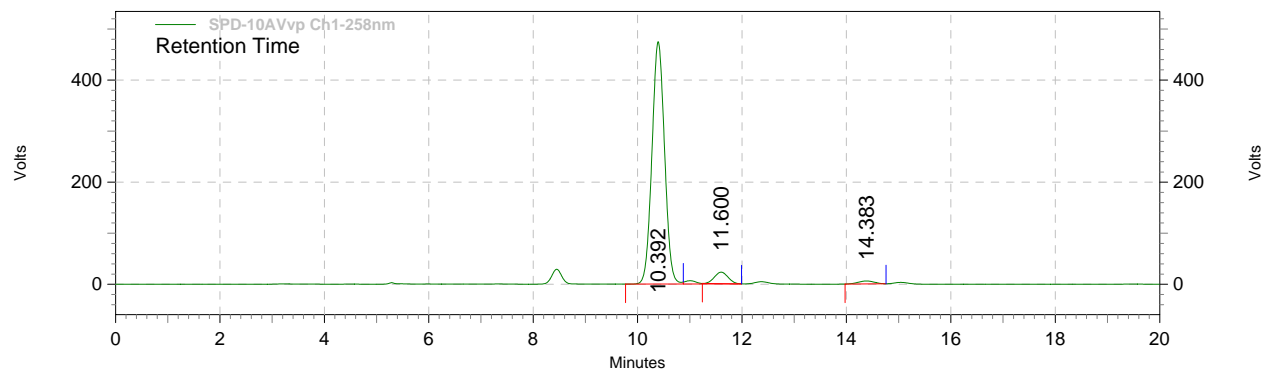
Retention Time	Area	Area %	Height	Height %
8.467	415312	2.27	31386	3.35
10.425	8715388	47.72	520588	55.53
10.875	11186	0.06	1946	0.21
11.633	367489	2.01	20688	2.21
14.417	8755795	47.94	362861	38.71
Totals	18265170	100.00	937469	100.00



10e, racemic

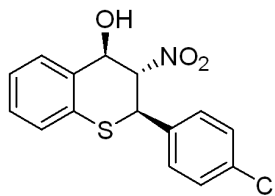
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-06-A
User: System
Acquired: 4/19/2007 4:03:36 PM
Printed: 5/16/2007 2:15:52 PM



SPD-10AVvp Ch1-258nm Results

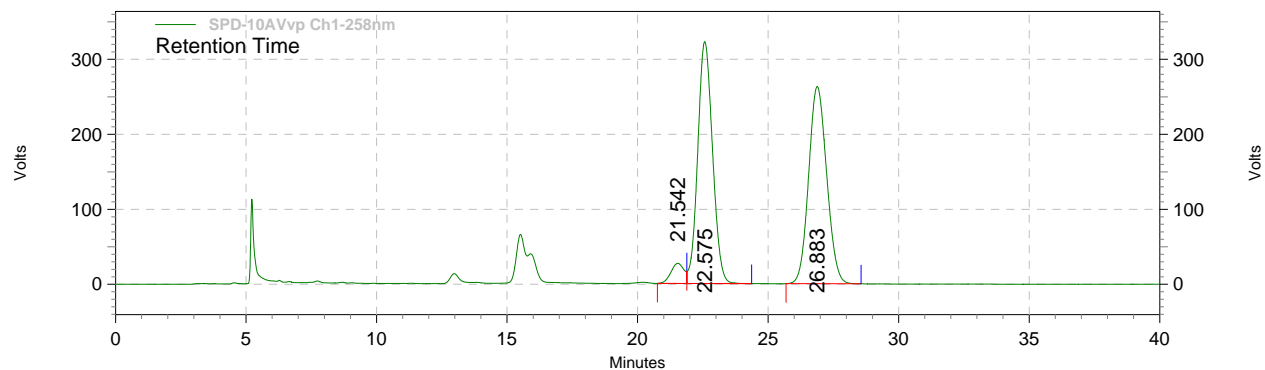
Retention Time	Area	Area %	Height	Height %
10.392	7960637	93.74	474629	94.38
11.600	409113	4.82	22559	4.49
14.383	122348	1.44	5702	1.13
Totals	8492098	100.00	502890	100.00



10e, 97% ee

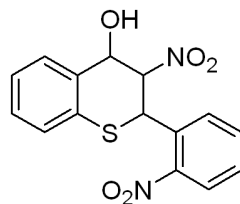
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\VIII-DR-152-A
User: System
Acquired: 4/18/2007 12:54:13 PM
Printed: 5/16/2007 2:17:22 PM



SPD-10AVvp Ch1-258nm Results

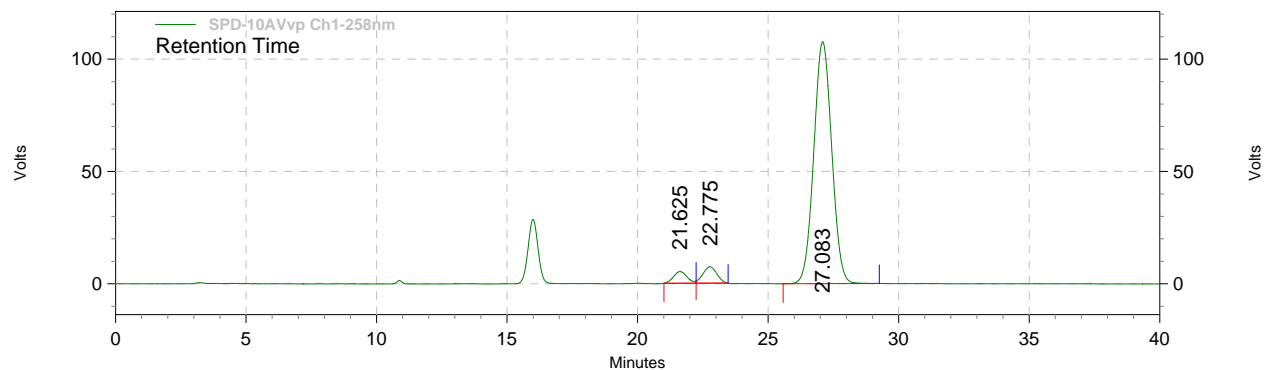
Retention Time	Area	Area %	Height	Height %
21.542	923393	3.51	26716	4.36
22.575	12719040	48.36	322422	52.67
26.883	12658999	48.13	263044	42.97
Totals	26301432	100.00	612182	100.00



10f, racemic

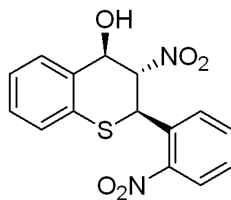
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-01-B
User: System
Acquired: 4/18/2007 2:24:02 PM
Printed: 5/16/2007 2:18:39 PM



SPD-10AVvp
Ch1-258nm
Results

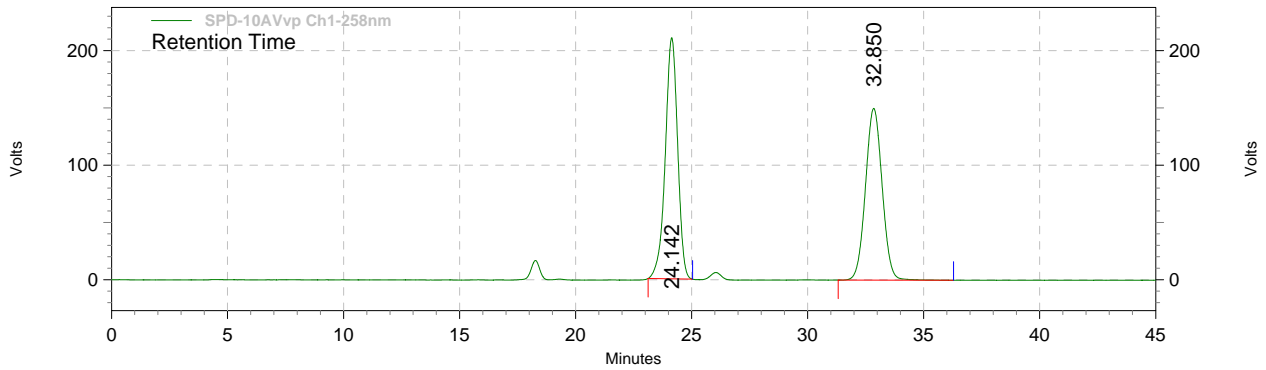
Retention Time	Area	Area %	Height	Height %
21.625	186581	3.33	5199	4.33
22.775	269471	4.81	7293	6.07
27.083	5149258	91.86	107660	89.60
Totals	5605310	100.00	120152	100.00



10f, 90% ee

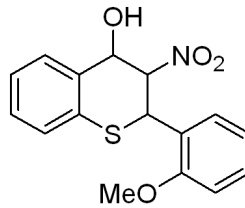
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\VIII-DR-138-4
User: System
Acquired: 4/13/2007 6:27:31 PM
Printed: 5/16/2007 2:09:38 PM



SPD-10AVvp
Ch1-258nm
Results

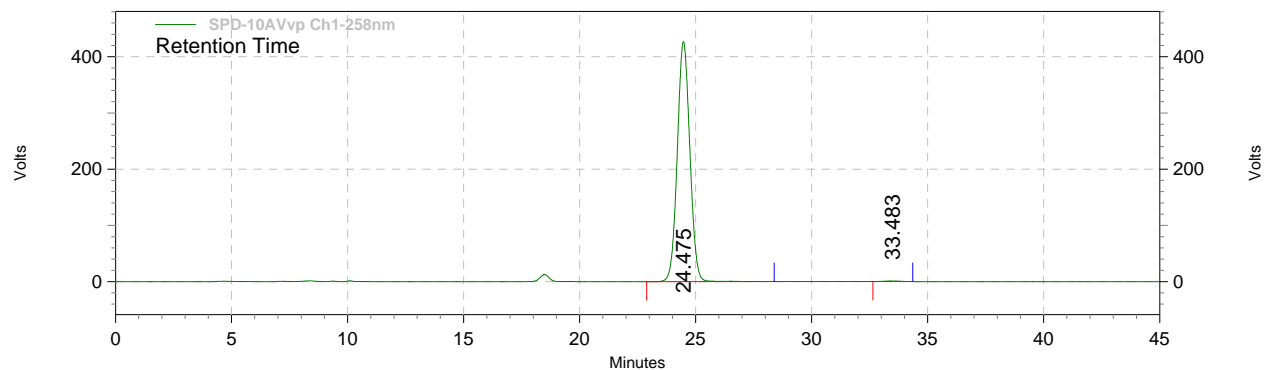
Retention Time	Area	Area %	Height	Height %
24.142	7799441	51.04	210515	58.39
32.850	7480239	48.96	149992	41.61
Totals	15279680	100.00	360507	100.00



10g, racemic

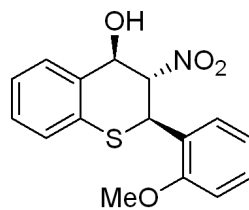
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-38
User: System
Acquired: 5/16/2007 1:20:59 PM
Printed: 5/16/2007 2:06:20 PM



SPD-10AVvp Ch1-258nm Results

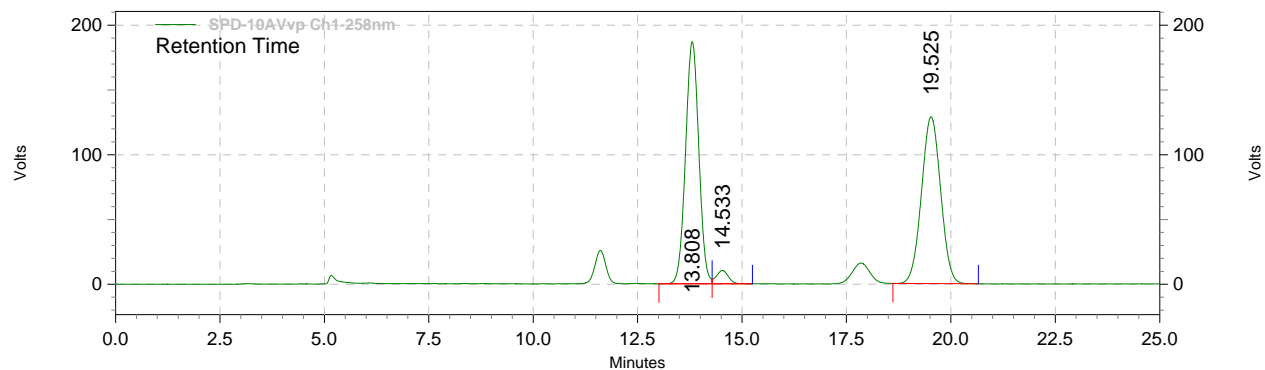
Retention Time	Area	Area %	Height	Height %
24.475	16052203	99.70	426720	99.76
33.483	48585	0.30	1009	0.24
Totals	16100788	100.00	427729	100.00



10g, >99% ee

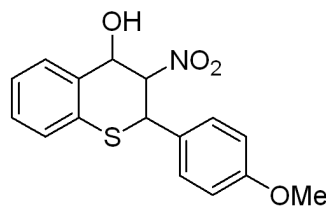
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\VIII-DR-142-RACEMIC-1
User: System
Acquired: 4/14/2007 11:47:43 AM
Printed: 5/16/2007 2:19:26 PM



SPD-10AVvp
Ch1-258nm
Results

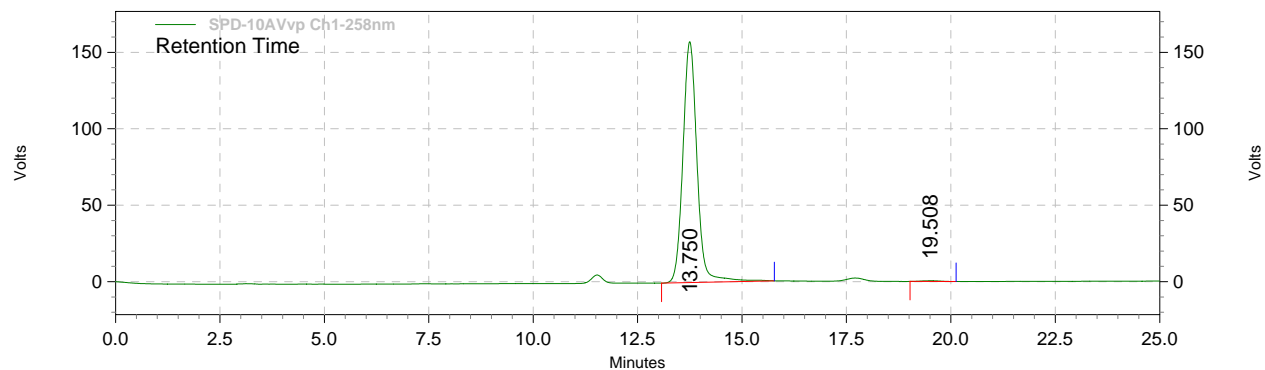
Retention Time	Area	Area %	Height	Height %
13.808	4131189	48.90	186874	57.31
14.533	205243	2.43	10319	3.16
19.525	4112555	48.68	128862	39.52
Totals	8448987	100.00	326055	100.00



10h, racemic

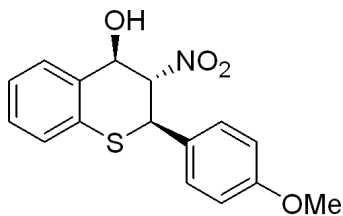
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\VIII-DR-145-4
User: System
Acquired: 4/14/2007 10:44:28 PM
Printed: 5/16/2007 2:20:06 PM



SPD-10AVvp Ch1-258nm Results

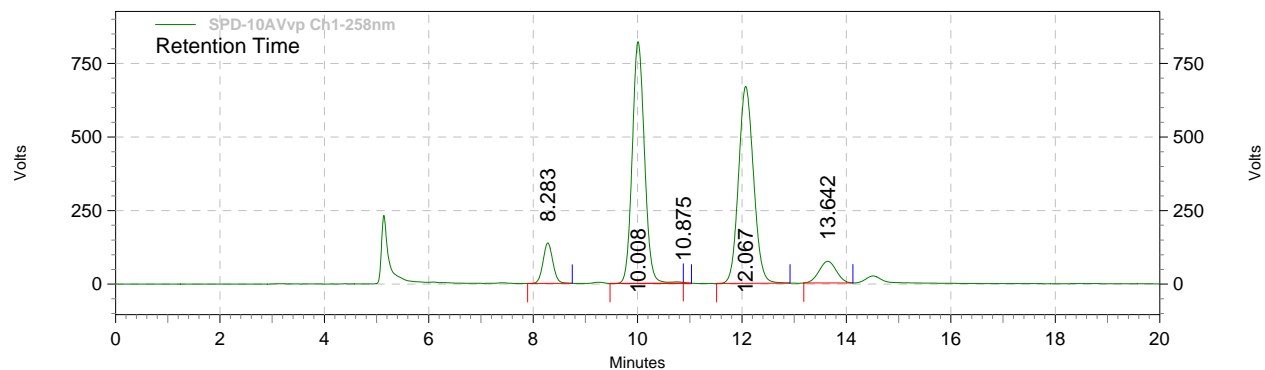
Retention Time	Area	Area %	Height	Height %
13.750	3694473	99.62	157457	99.70
19.508	13923	0.38	472	0.30
Totals	3708396	100.00	157929	100.00



10h, >99% ee

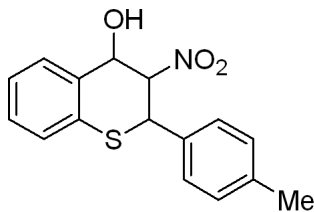
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\VIII-DR-151-RACEMIC
User: System
Acquired: 4/16/2007 1:09:38 PM
Printed: 5/16/2007 2:20:50 PM



SPD-10AVvp Ch1-258nm Results

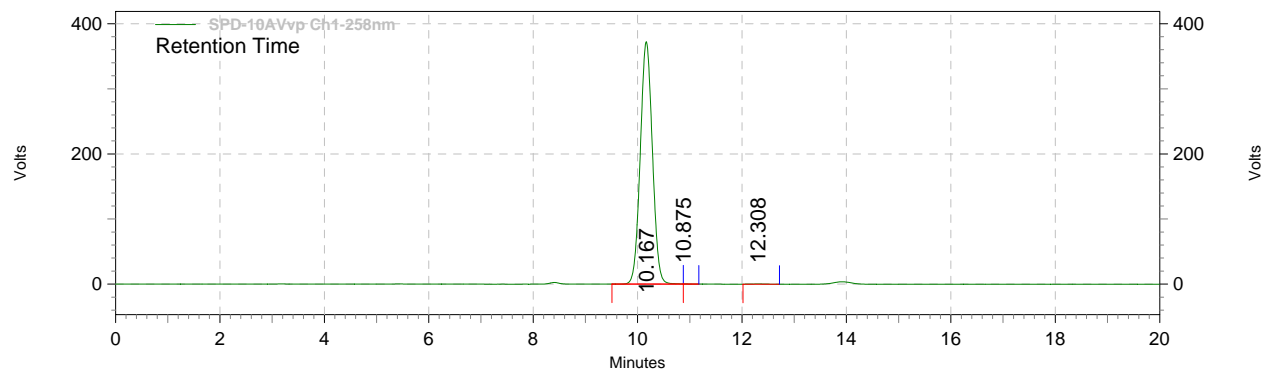
Retention Time	Area	Area %	Height	Height %
8.283	1783570	5.90	137363	8.06
10.008	13400938	44.32	821210	48.19
10.875	11825	0.04	2956	0.17
12.067	13350748	44.16	668984	39.25
13.642	1687753	5.58	73694	4.32
Totals	30234834	100.00	1704207	100.00



10i, racemic

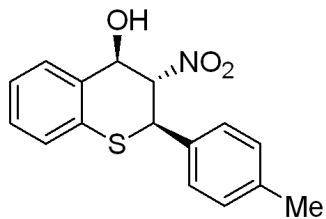
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-03-A
User: System
Acquired: 4/19/2007 10:49:00 AM
Printed: 5/16/2007 2:21:21 PM



SPD-10AVvp
Ch1-258nm
Results

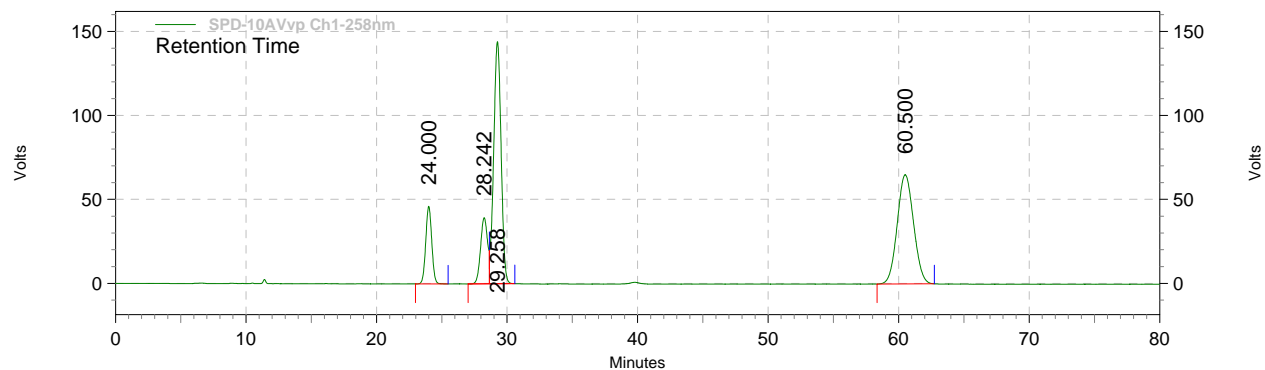
Retention Time	Area	Area %	Height	Height %
10.167	6001800	99.87	372330	99.84
10.875	1842	0.03	257	0.07
12.308	6022	0.10	332	0.09
Totals	6009664	100.00	372919	100.00



10i, >99% ee

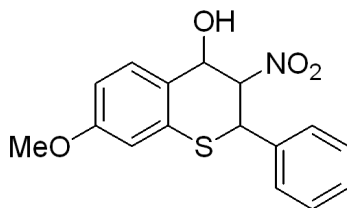
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-12-A
User: System
Acquired: 4/25/2007 4:52:03 PM
Printed: 5/16/2007 2:23:50 PM



SPD-10AVvp Ch1-258nm Results

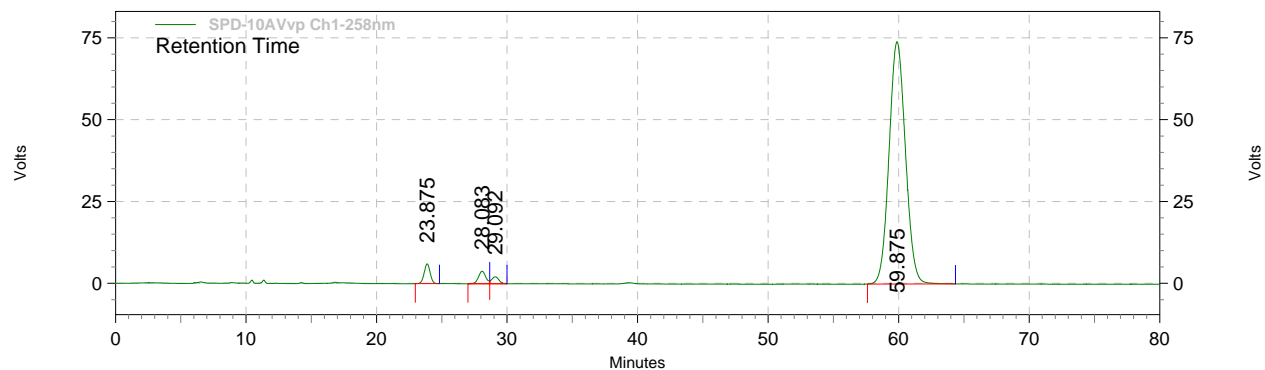
Retention Time	Area	Area %	Height	Height %
24.000	1480220	10.36	46247	15.69
28.242	1458891	10.21	39439	13.38
29.258	5701129	39.89	144117	48.89
60.500	5652327	39.55	64981	22.04
Totals	14292567	100.00	294784	100.00



10j, racemic

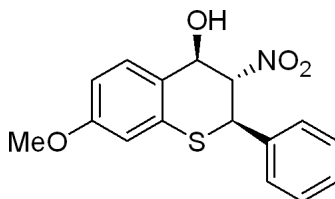
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-15-A
User: System
Acquired: 4/27/2007 1:24:36 PM
Printed: 5/16/2007 2:24:35 PM



SPD-10AVvp Ch1-258nm Results

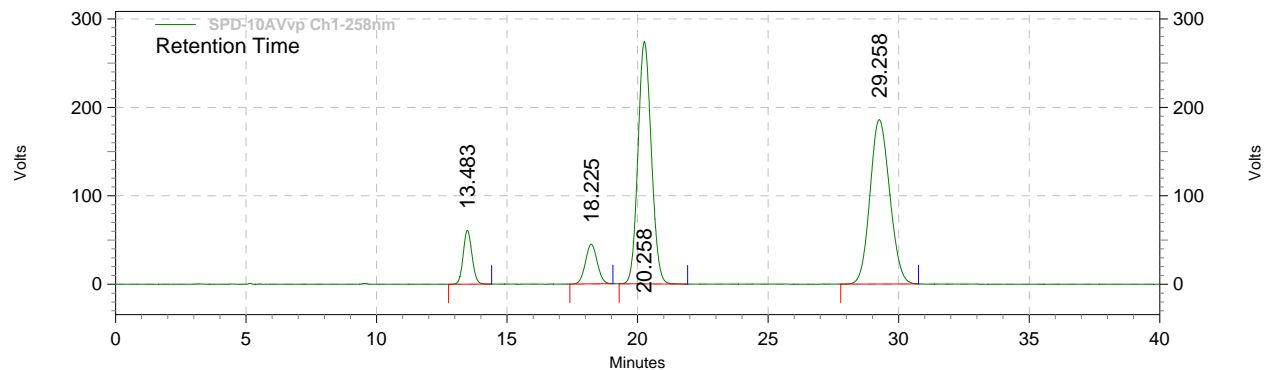
Retention Time	Area	Area %	Height	Height %
23.875	193782	2.82	6042	7.03
28.083	147961	2.15	3815	4.44
29.092	79914	1.16	2122	2.47
59.875	6457377	93.87	74025	86.07
Totals	6879034	100.00	86004	100.00



10j, 98% ee

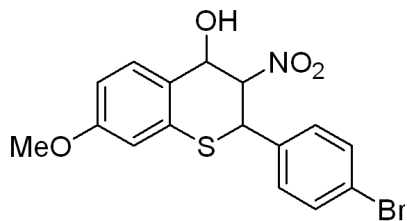
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-13-RACEMIC
User: System
Acquired: 4/26/2007 10:42:50 AM
Printed: 5/16/2007 2:25:09 PM



SPD-10AVvp Ch1-258nm Results

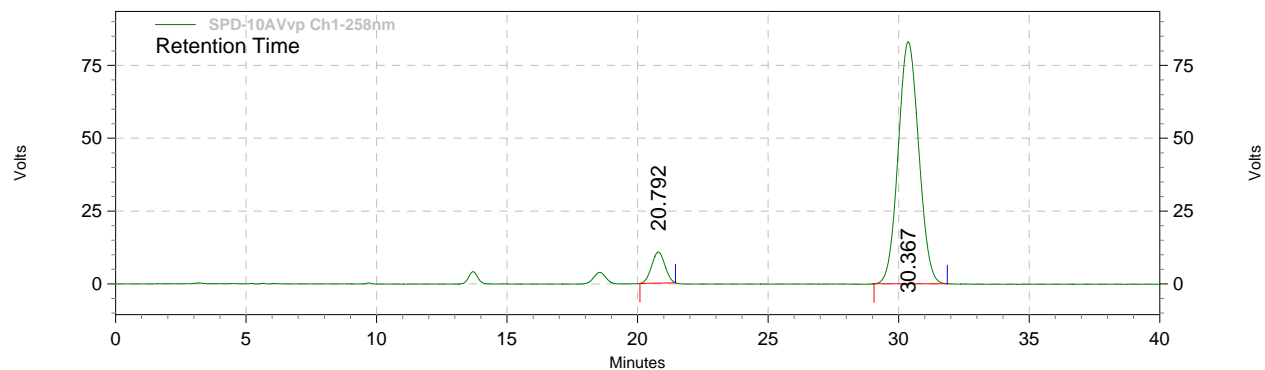
Retention Time	Area	Area %	Height	Height %
13.483	1422640	6.37	60877	10.77
18.225	1407883	6.30	44872	7.94
20.258	9745782	43.64	273762	48.42
29.258	9753374	43.68	185853	32.87
Totals	22329679	100.00	565364	100.00



10k, racemic

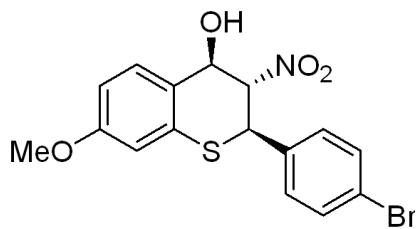
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-14-PURE-A
User: System
Acquired: 4/26/2007 2:08:51 PM
Printed: 5/16/2007 2:25:33 PM



SPD-10AVvp Ch1-258nm Results

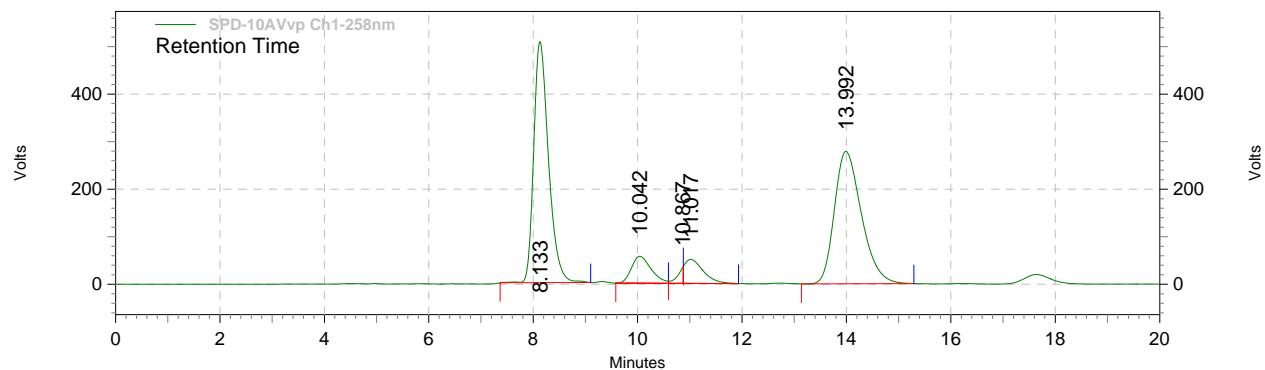
Retention Time	Area	Area %	Height	Height %
20.792	377246	7.66	10674	11.38
30.367	4549549	92.34	83094	88.62
Totals	4926795	100.00	93768	100.00



10k, 85% ee

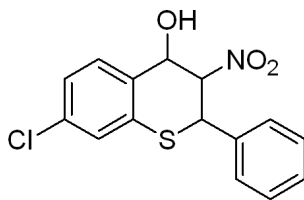
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-19-RACEMIC-C
User: System
Acquired: 5/1/2007 3:49:19 PM
Printed: 5/14/2007 5:17:52 PM



SPD-10AVvp Ch1-258nm Results

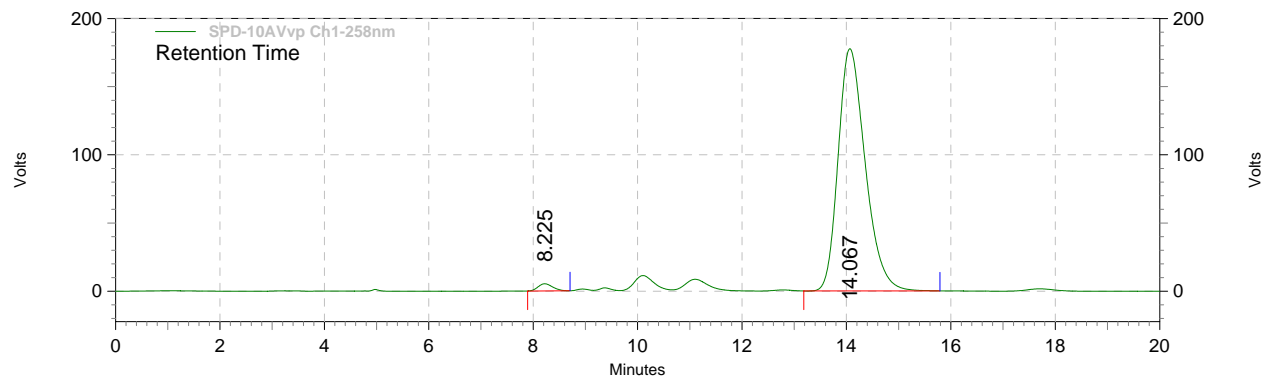
Retention Time	Area	Area %	Height	Height %
8.133	9996151	43.51	506832	54.70
10.042	1483825	6.46	56698	6.12
10.867	249143	1.08	34028	3.67
11.017	1237678	5.39	50479	5.45
13.992	10007617	43.56	278594	30.07
Totals	22974414	100.00	926631	100.00



10I, racemic

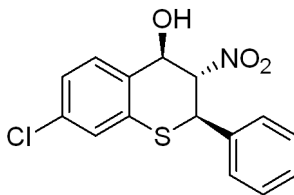
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IX-DR-20-A
User: System
Acquired: 5/1/2007 4:48:12 PM
Printed: 5/16/2007 2:27:14 PM



SPD-10AVvp
Ch1-258nm
Results

Retention Time	Area	Area %	Height	Height %
8.225	102818	1.58	5295	2.89
14.067	6410581	98.42	177649	97.11
Totals	6513399	100.00	182944	100.00



10, 97% ee