Room Temperature Ionic Liquid-DMSO Promoted and Improved One pot Synthesis of 5,6-Diaryl-1,2,4-triazines

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SUPPORTING INFORMATION

Experimental

Melting points were determined in capillaries using Veego programmable melting point apparatus and are uncorrected. IR (in cm^{-1}) spectra in KBr pellets on a Bruker ALPHA-T instrument; and ^1H NMR spectra were recorded in $CDCl_3$ on a Bruker Avance II spectrometer (400 MHz), using tetramethylsilane as an internal standard. Chemical shift data are reported in parts per million (δ in ppm) where *s*, *br*, *t* and *m* designate singlet, broad, triplet and multiplet, respectively. Elemental analyses were recorded on a Thermoscientific Flash-2000 CHN analyzer. Mass spectra were recorded on Thermoscientific DSQ-II mass spectrometer equipped with an electron impact ionization (EI) interface. Flash Column chromatography was carried out Combiflash R_F 200 (Teledyne Esco) using flash grade silica gel (230-400 mesh). Thin-layer chromatography (TLC) was performed on precoated Silica gel Merck plates. Compounds were visualized by illuminating with UV light (254 nm). All chemicals and solvents were of reagent grade and were purified and dried using standard methods.

General procedure for synthesis of 3-methylthio-1,2,4-triazines in IL+DMSO (1:10) (3a-3k)

A mixture of diketone **1a-k** (2.0 mmol), thiosemicarbazide **2** (2.0 mmol) and methyl iodide (2.4 mmol) in DMSO and $[Bbim]^+Br^-$ in 1:10 (0.5 g : 5 g) proportions was stirred at 70^oC for appropriate time durations as mentioned in table 4. The progress of the reaction was monitored by TLC with an eluent mixture of *n*-hexane and ethyl acetate (4.5 : 0.5). After completion, the reaction mixture was added to ice cold water. The precipitated product was filtered, washed with water and dried. In case of regioisomers, the mixture was subjected to flash chromatographic purification using 5 % ethyl acetate in *n*.hexane as eluent, to obtain first and second fractions as separated isomers (3d-3k).

Characterization data for 3-methylthio-5,6-disubstitutedphenyl-1,2,4-triazines (3a-3k′)

3-Methylthio-5,6-diphenyl-1,2,4-triazine (3a)[1]

mp: 119-20^{o}C (lit. 121-22^{o}C); IR (KBr, cm^{-1}) 3130, 2922, 1667, 1590; m/z 279.01 (M^+), ^1H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.80 (s, 3H, SC*H*$_3$); 7.34-7.59 (m, 10H, Ar-*H*), Anal. Calcd. for $C_{16}H_{13}N_3S$: C, 68.79; H, 4.69; N, 15.04; Found: C, 68.84; H, 4.66; N, 15.00.

3-Methylthio-5,6-di(4-chloro)-1,2,4-triazine (3b)[1]

mp: 134-36^{o}C (lit. 137-39^{o}C); IR (KBr, cm^{-1}) 3434, 3067, 1591, 839; m/z 346.93 (M^{+}), 349 (M^{+2}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.78 (s, 3H, SC*H*$_3$); 7.35-7.54 (m, 8H, Ar-*H*); Anal. Calcd. for $C_{16}H_{11}Cl_2N_3S$: C, 55.18; H, 3.18; N, 12.07; Found: C, 55.42; H, 3.11; N, 11.96.

3-Methylthio-5,6-di(*p*-tolyl)-1,2,4-triazine (3c)[2]

mp: 150-52^{o}C (lit. 149-51^{o}C); IR (KBr, cm^{-1}) 3127, 3032, 1675, 1607; m/z 307.02 (M^{+}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.38 (s, 3H, Ar-C*H*$_3$); 2.40 (s, 3H, Ar-C*H*$_3$); 2.77 (s, 3H, SC*H*$_3$); 7.14-7.50 (m, 8H, Ar-*H*); Anal. Calcd. for $C_{18}H_{17}N_3S$: C, 70.33; H, 5.57; N, 13.67; Found: C, 70.41; H, 5.36; N, 13.87.

3-Methylthio-5-(*p*-tolyl)-6-phenyl-1,2,4-triazine (3d)

mp: 105-07^{o}C; IR (KBr, cm^{-1}) 3434, 2972, 1636, 1608; m/z 293.01 (M^{+}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.39 (s, 3H, Ar-C*H*$_3$); 2.78 (s, 3H, SC*H*$_3$); 7.14-7.60 (m, 9H, Ar-*H*); Anal. Calcd. for $C_{17}H_{15}N_3S$: C, 69.59; H, 5.15; N, 14.32; Found: C, 69.48; H, 5.17; N, 14.27.

3-Methylthio-5-phenyl-6-(*p*-tolyl)-1,2,4-triazine (3d′)

mp: 92-94^{o}C; IR (KBr, cm^{-1}) 3434, 2972, 1636, 1608; m/z 293.01 (M^{+}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.39 (s, 3H, Ar-C*H*$_3$); 2.79 (s, 3H, SC*H*$_3$); 7.15-7.61 (m, 9H, Ar-*H*); Anal. Calcd. for $C_{17}H_{15}N_3S$: C, 69.59; H, 5.15; N, 14.32; Found: C, 69.51; H, 5.12; N, 14.29.

3-Methylthio-5-(4-methylthiophenyl)-6-phenyl-1,2,4-triazine (3f)

mp: 110-12^{o}C; IR (KBr, cm^{-1}) 3426, 3137, 2923, 1590; m/z 324.94 (M^{+}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.51 (s, 3H, Ar-C*H*$_3$); 2.78 (s, 3H, SC*H*$_3$); 7.16-7.72 (m, 9H, Ar-*H*); Anal. Calcd. for $C_{17}H_{15}N_3S_2$: C, 62.74; H, 4.65; N, 12.91; Found: C, 62.81; H, 4.66; N, 12.87.

3-Methylthio-5-phenyl-6-(4-methylthiophenyl)-1,2,4-triazine (3f′)

mp: 93-95^{o}C; IR (KBr, cm^{-1}) 3426, 3137, 2923, 1590; m/z 324.94 (M^{+}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.50 (s, 3H, Ar-C*H*$_3$); 2.77 (s, 3H, SC*H*$_3$); 7.15-7.56 (m, 9H, Ar-*H*); Anal. Calcd. for $C_{17}H_{15}N_3S_2$: C, 62.74; H, 4.65; N, 12.91; Found: C, 62.80; H, 4.72; N, 12.82.

3-Methylthio-5-(4-chlorophenyl)-6-(*p*-tolyl)-1,2,4-triazine (3g)

mp: 179-82^{o}C; IR (KBr, cm^{-1}) 3419, 3129, 1599, 834; m/z 326.98 (M^{+}), 329.06 (M^{+2}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.32 (s, 3H, Ar-C*H*$_3$); 2.70 (s, 3H, SC*H*$_3$); 7.09-7.52 (m, 8H, Ar-*H*); Anal. Calcd. for $C_{17}H_{14}ClN_3S$: C, 62.28; H, 4.30; N, 12.82; Found: C, 62.32; H, 4.33; N, 12.76.

3-Methylthio-5-(*p*-tolyl)-6-(4-chlorophenyl)-1,2,4-triazine (3g′)

mp: 165-68^{o}C; IR (KBr, cm^{-1}) 3419, 3129, 1599, 834; m/z 326.98 (M^{+}), 329.02 (M^{+2}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.41 (s, 3H, Ar-C*H*$_3$); 2.79 (s, 3H, SC*H*$_3$); 7.20-7.56 (m, 8H, Ar-*H*); Anal. Calcd. for $C_{17}H_{14}ClN_3S$: C, 62.28; H, 4.30; N, 12.82; Found: C, 62.31; H, 4.30; N, 12.79.

3-Methylthio-5-(4-chlorophenyl)-6-phenyl-1,2,4-triazine (3h)

mp: 118-20^{o}C; IR (KBr, cm^{-1}) 3422, 3134, 1591, 836; m/z 313.08 (M^{+}) 315.04 (M^{+2}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.79 (s, 3H, SC*H*$_3$); 7.19-7.49 (m, 9H, Ar-*H*); Anal. Calcd. for $C_{16}H_{12}ClN_3S$: C, 61.24; H, 3.85; N, 13.39; Found: C, 61.31; H, 3.96; N, 13.29.

3-Methylthio-5-phenyl-6-(4-chlorophenyl)-1,2,4-triazine (3h′)

mp: 131-33^{o}C; IR (KBr, cm^{-1}) 3422, 3134, 1591, 836; m/z 313.08 (M^{+}) 315.02 (M^{+2}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.79 (s, 3H, SC*H*$_3$); 7.32-7.67 (m, 9H, Ar-*H*); Anal. Calcd. for $C_{16}H_{12}ClN_3S$: C, 61.24; H, 3.85; N, 13.39; Found: C, 61.33; H, 3.99; N, 13.26.

3-Methylthio-5-(*p*-tolyl)-6-(4-nitrophenyl)-1,2,4-triazine (3i)

mp: 108-10^{o}C; IR (KBr, cm^{-1}) 3114, 1602, 1523; ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.41 (s, 3H, Ar-C*H*$_3$); 2.79 (s, 3H, SC*H*$_3$); 7.18-8.26 (m, 8H, Ar-*H*), Anal. Calcd. for $C_{17}H_{14}N_4O_2S$: C, 60.34; H, 4.17; N, 16.56; Found: C, 60.45; H, 4.19; N, 16.49.

3-Methylthio-5-(4-nitrophenyl)-6-(*p*-tolyl)-1,2,4-triazine (3i′)

mp: 148-50^{o}C; IR (KBr, cm^{-1}) 3114, 1602, 1523; ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.41 (s, 3H, Ar-C*H*$_3$); 2.79 (s, 3H, SC*H*$_3$); 7.18-8.26 (m, 8H, Ar-*H*), Anal. Calcd. for $C_{17}H_{14}N_4O_2S$: C, 60.34; H, 4.17; N, 16.56; Found: C, 60.40; H, 4.15; N, 16.48.

3-Methylthio-6-(4-nitrophenyl)-5-phenyl-1,2,4-triazine (3j)

mp: 155-57^{o}C; IR (KBr, cm^{-1}) 3424, 3127, 1600, 1509; m/z 324.08 (M^{+}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.79 (s, 3H, SC*H*$_3$); 7.38-8.25 (m, 9H, Ar-*H*); Anal. Calcd. for $C_{16}H_{12}N_4O_2S$: C, 59.25; H, 3.73; N, 17.27; Found: C, 59.32; H, 3.80; N, 17.19.

3-Methylthio-6-phenyl-5-(4-nitrophenyl)-1,2,4-triazine (3j′)

mp: 118-20^{o}C; IR (KBr, cm^{-1}) 3424, 3127, 1600, 1509; m/z 324.08 (M^{+}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.80 (s, 3H, SC*H*$_3$); 7.40-8.25 (m, 9H, Ar-*H*); Anal. Calcd. for $C_{16}H_{12}N_4O_2S$: C, 59.25; H, 3.73; N, 17.27; Found: C, 59.34; H, 3.81; N, 17.21.

3-Methylthio-5-(3-chlorophenyl)-6-(*p*-tolyl)-1,2,4-triazine (3k)

mp: 114-16^{o}C; IR (KBr, cm^{-1}) 3446, 1636, 1609, 1567; m/z 326.89 (M^{+}), 329.01 (M^{+2}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.40 (s, 3H, Ar-C*H*$_3$); 2.77 (s, 3H, SC*H*$_3$); 7.17-7.66 (m, 8H, Ar-*H*); Anal. Calcd. for $C_{17}H_{14}ClN_3S$: C, 62.28; H, 4.30; N, 12.82; Found: C, 62.33; H, 4.26; N, 12.78.

3-Methylthio-6-(3-chlorophenyl)-5-(*p*-tolyl)-1,2,4-triazine (3k′)

mp: 88-91^{o}C; IR (KBr, cm^{-1}) 3446, 1636, 1609, 1567; m/z 326.89 (M^{+}), 329 (M^{+2}); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 2.41 (s, 3H, Ar-C*H*$_3$); 2.79 (s, 3H, SC*H*$_3$); 7.19-7.71 (m, 8H, Ar-*H*); Anal. Calcd. for $C_{17}H_{14}ClN_3S$: C, 62.28; H, 4.30; N, 12.82; Found: C, 62.38; H, 4.25; N, 12.76.

References:

[1] Irannejad, H.; Amini, M.; Khodagholi, F.; Ansari, N.; Tusi, S. K.; Sharifzadeh, M.; Shafiee, A. Synthesis and *in vitro* evaluation of novel 1,2,4-triazine derivatives as neuroprotective agents. *Bioorg. Med. Chem.* **2010**, *18*, 4224-4230.

[2] Ansari, N.; Khodagholi, F.; Ramin, M.; Amini, M.; Irannejad, H.; Dargahi, L.; Amirabad, A. D. Inhibition of LPS-induced apoptosis in differentiated-PC12 cells by new triazine derivatives through NF-κB-mediated suppression of COX-2. *Neurochem. International* **2010**, *57*, 958-968.

Crystal data for 3e: $C_{16}H_{22}S_1N_3F_1$, CCDC-969264, *M*r = 297.36,*T*=293(2) K, space group = *P*21/c, *a* = 6.1928(2)Å, *b* = 10.6279(3)Å, *c* = 22.6088(8)Å, V = 1474.14(8)Å^3, Z = 4, R1 = 0.0768, wR2 = 0.2794[I >2σ (*I*)].

CCDC 969264 detailed crystal data

Table S1: Crystal data and structure refinement for CCDC 969264

Identification code	CCDC 969264
Empirical formula	$C_{16}H_{12}FN_3S$
Formula weight	297.36
Temperature/K	293(2)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	6.1928(2)
b/Å	10.6279(3)
c/Å	22.6088(8)
α/°	90.00
β/°	97.834(4)
γ/°	90.00
Volume/Å^3	1474.14(9)
Z	4
ρ_{calc}mg/mm^3	1.340
m/mm^{-1}	2.017
F(000)	616.0
Crystal size/mm^3	0.38 × 0.31 × 0.26
Radiation	Cu Kα (λ = 1.5418)
2Θ range for data collection	7.9 to 144.42°
Reflections collected	5832
Independent reflections	2860[R(int) = 0.0153]
Data/restraints/parameters	2860/0/191
Goodness-of-fit on F^2	1.084
Final R indexes [I>=2σ (I)]	R_1 = 0.0768, wR_2 = 0.2684
Final R indexes [all data]	R_1 = 0.0838, wR_2 = 0.2794
Largest diff. peak/hole / e Å^{-3}	1.28/-0.37

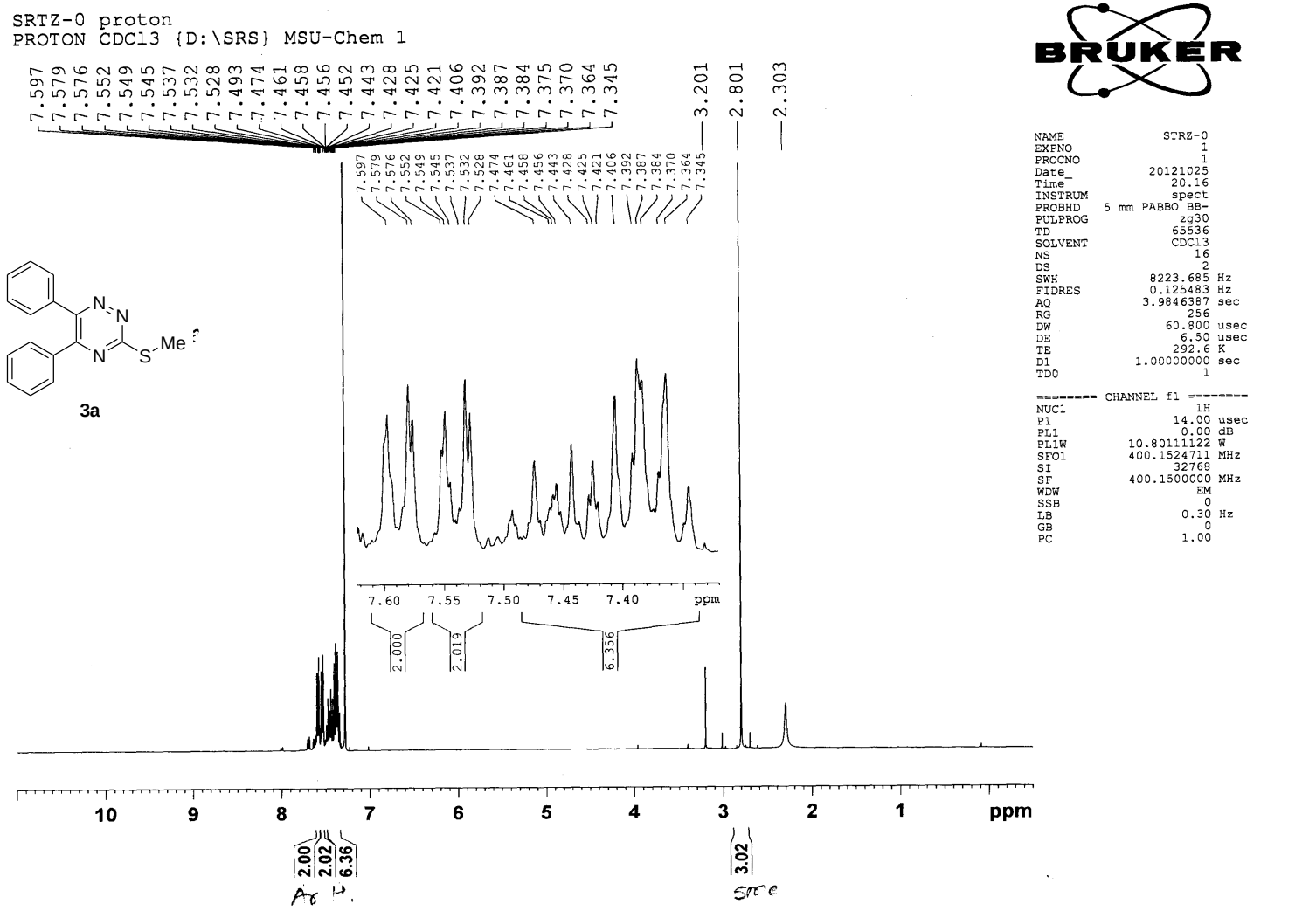
Table S2: Bond Lengths for CCDC 969264

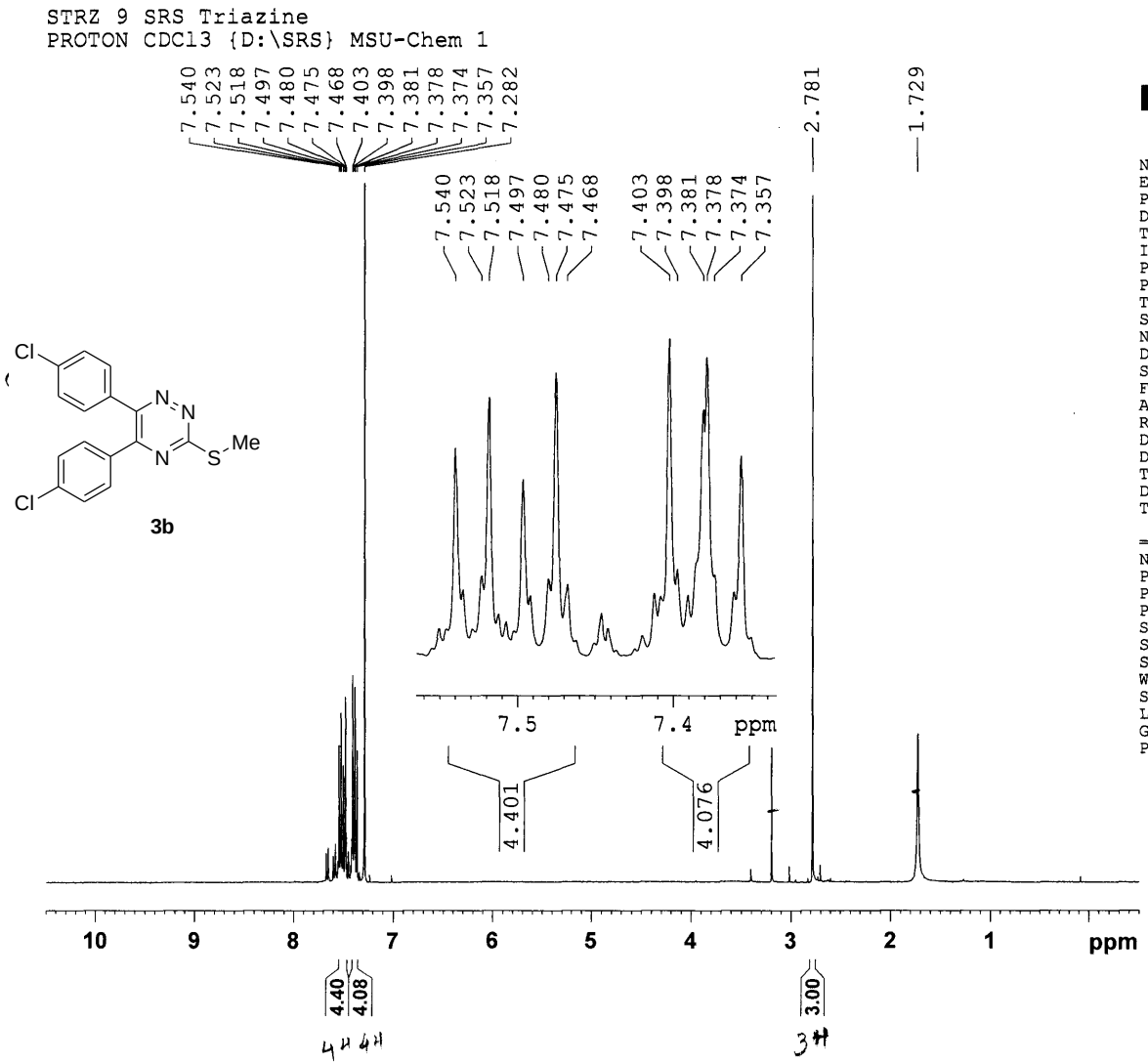
Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	C5	1.747(4)	N2	C5	1.348(4)
S1	C21	1.787(5)	C7	C12	1.387(4)
C15	C14	1.379(5)	C7	C8	1.369(5)
C15	C16	1.369(5)	C13	C18	1.396(4)
N3	N1	1.337(4)	C11	C12	1.382(6)
N3	C5	1.314(5)	C11	C10	1.361(6)
C14	C13	1.393(4)	C16	C17	1.377(5)
C2	N1	1.329(4)	C16	F1	1.325(4)
C2	C3	1.418(4)	C17	C18	1.370(5)
C2	C7	1.488(4)	C8	C9	1.393(5)
C3	N2	1.331(4)	C9	C10	1.364(6)
C3	C13	1.474(4)			

Table S3: Bond Angles for CCDC 969264.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C5	S1	C21	102.4(2)	C14	C13	C18	118.5(3)
C16	C15	C14	119.2(3)	C18	C13	C3	118.9(3)
C5	N3	N1	117.4(3)	C10	C11	C12	119.7(4)
C15	C14	C13	120.4(3)	C15	C16	C17	122.1(3)
N1	C2	C3	121.0(3)	F1	C16	C15	117.2(3)
N1	C2	C7	114.5(3)	F1	C16	C17	120.7(4)
C3	C2	C7	124.5(3)	C18	C17	C16	118.5(3)
C2	N1	N3	119.9(3)	C11	C12	C7	120.1(3)
C2	C3	C13	125.6(3)	C17	C18	C13	121.3(3)
N2	C3	C2	118.6(3)	C7	C8	C9	119.7(3)
N2	C3	C13	115.8(3)	N3	C5	S1	120.2(2)
C3	N2	C5	116.1(3)	N3	C5	N2	126.8(3)
C12	C7	C2	119.1(3)	N2	C5	S1	112.9(3)
C8	C7	C2	121.3(3)	C10	C9	C8	120.0(4)
C8	C7	C12	119.7(3)	C11	C10	C9	120.8(4)
C14	C13	C3	122.4(3)				

^{1}H NMR spectra (**3a-3k'**)





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DE	6.50 usec
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D1	1.00000000 sec
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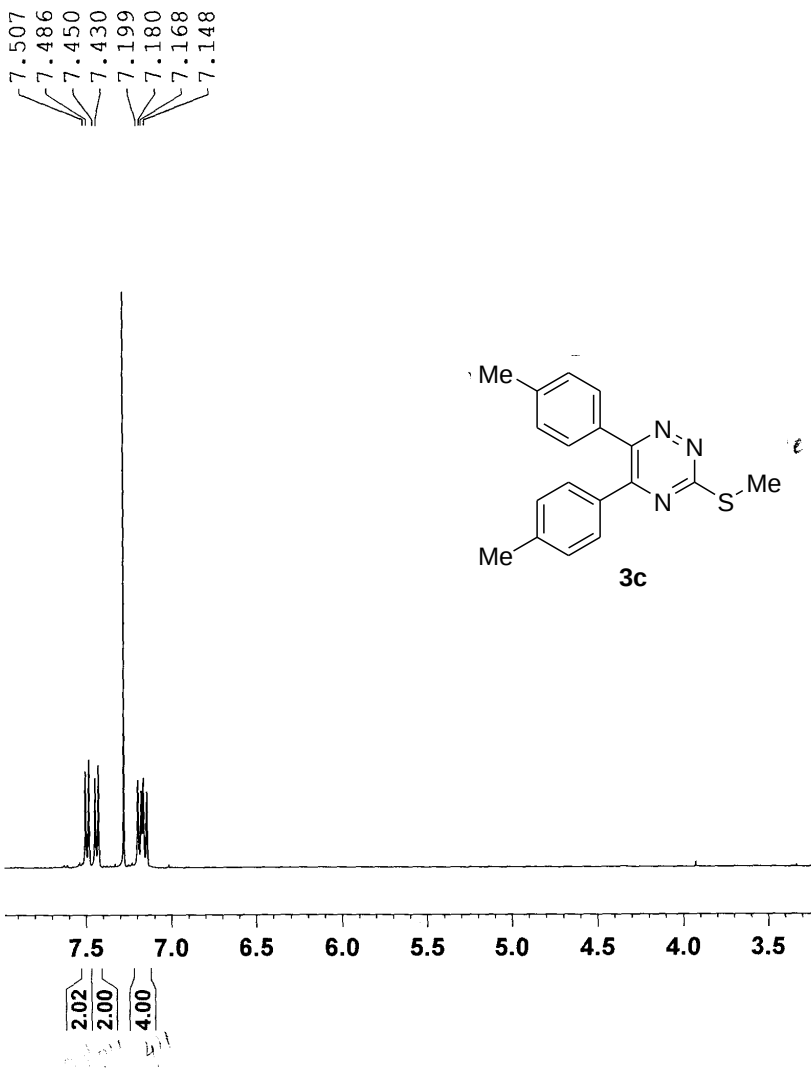

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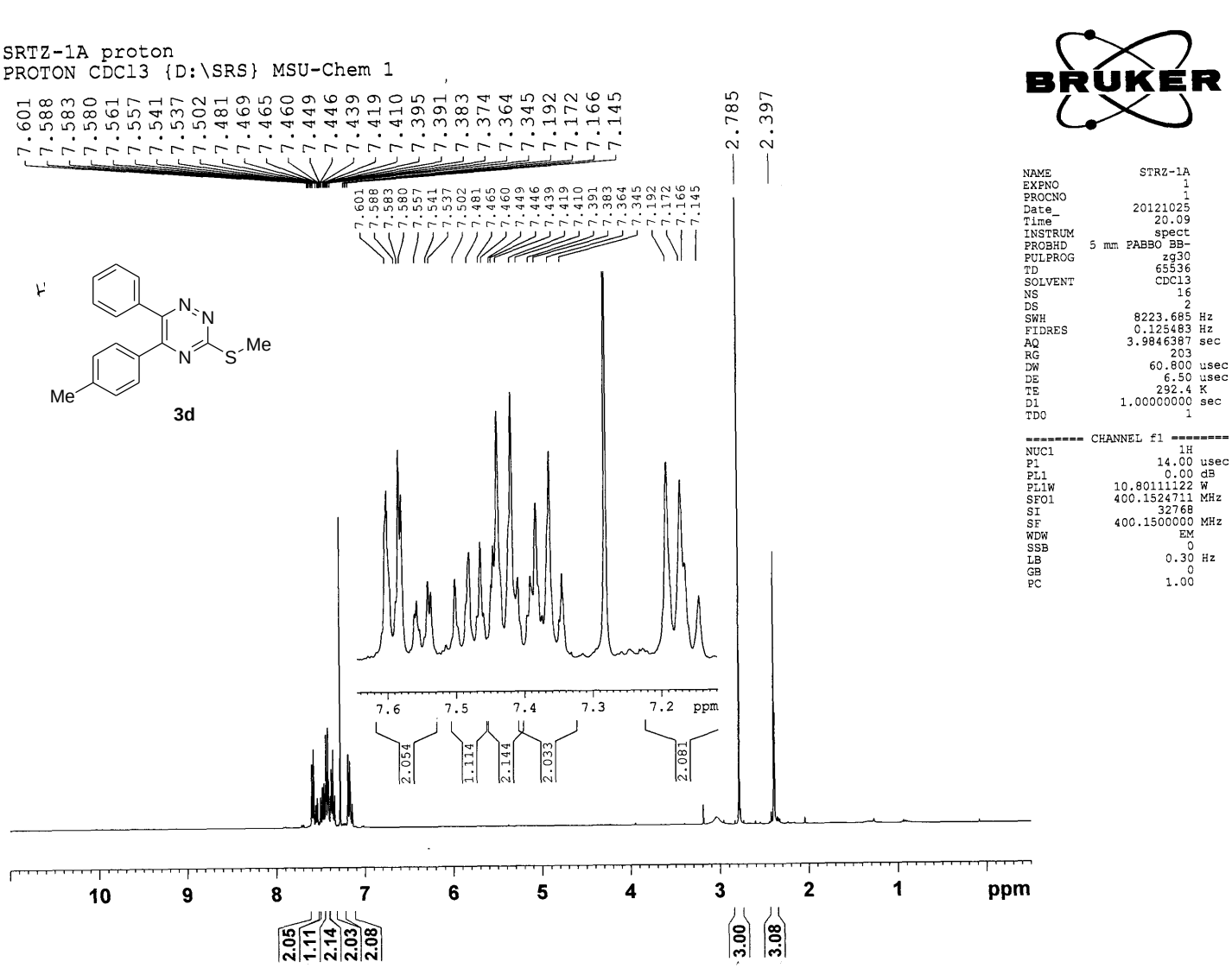

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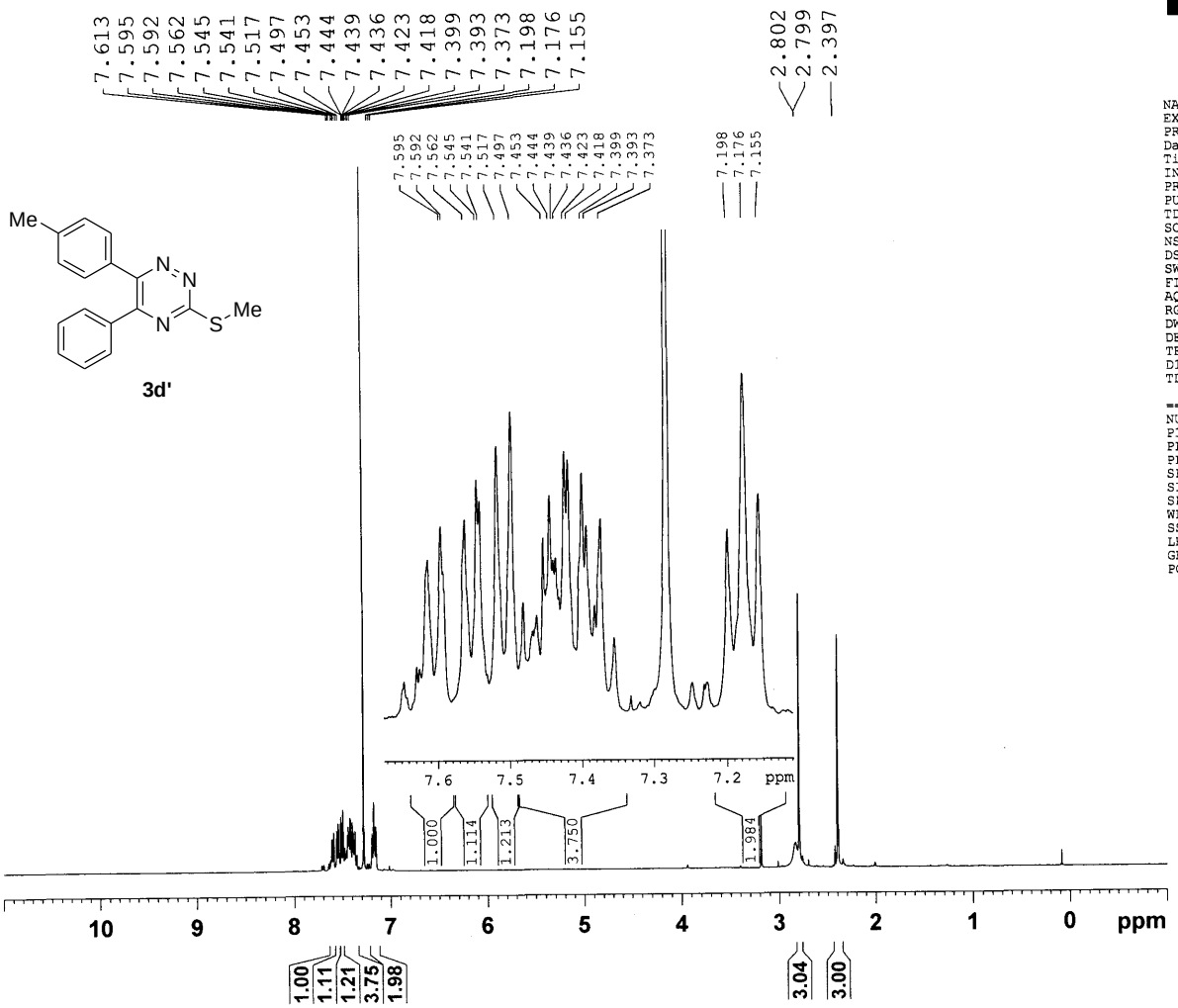

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TD0	1

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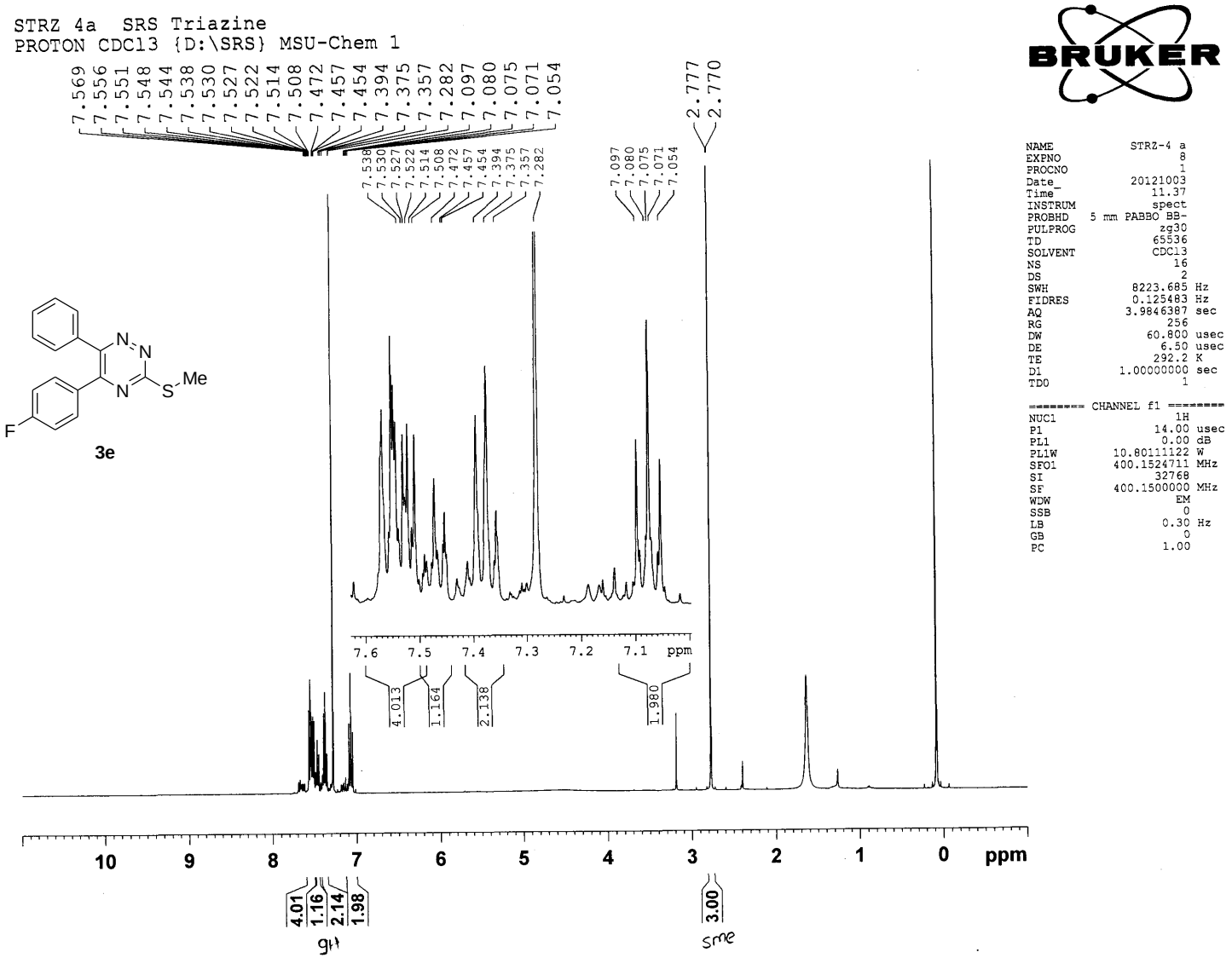
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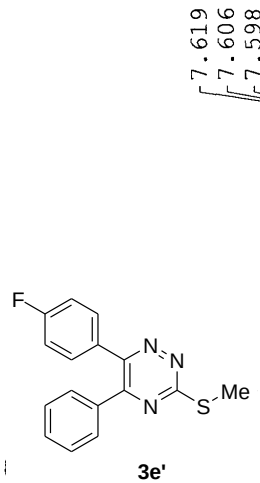
Date_ 20121003
Time_ 11.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
PL1W 10.80111122 W
SFO1 400.1524711 MHz
SI 32768
SF 400.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

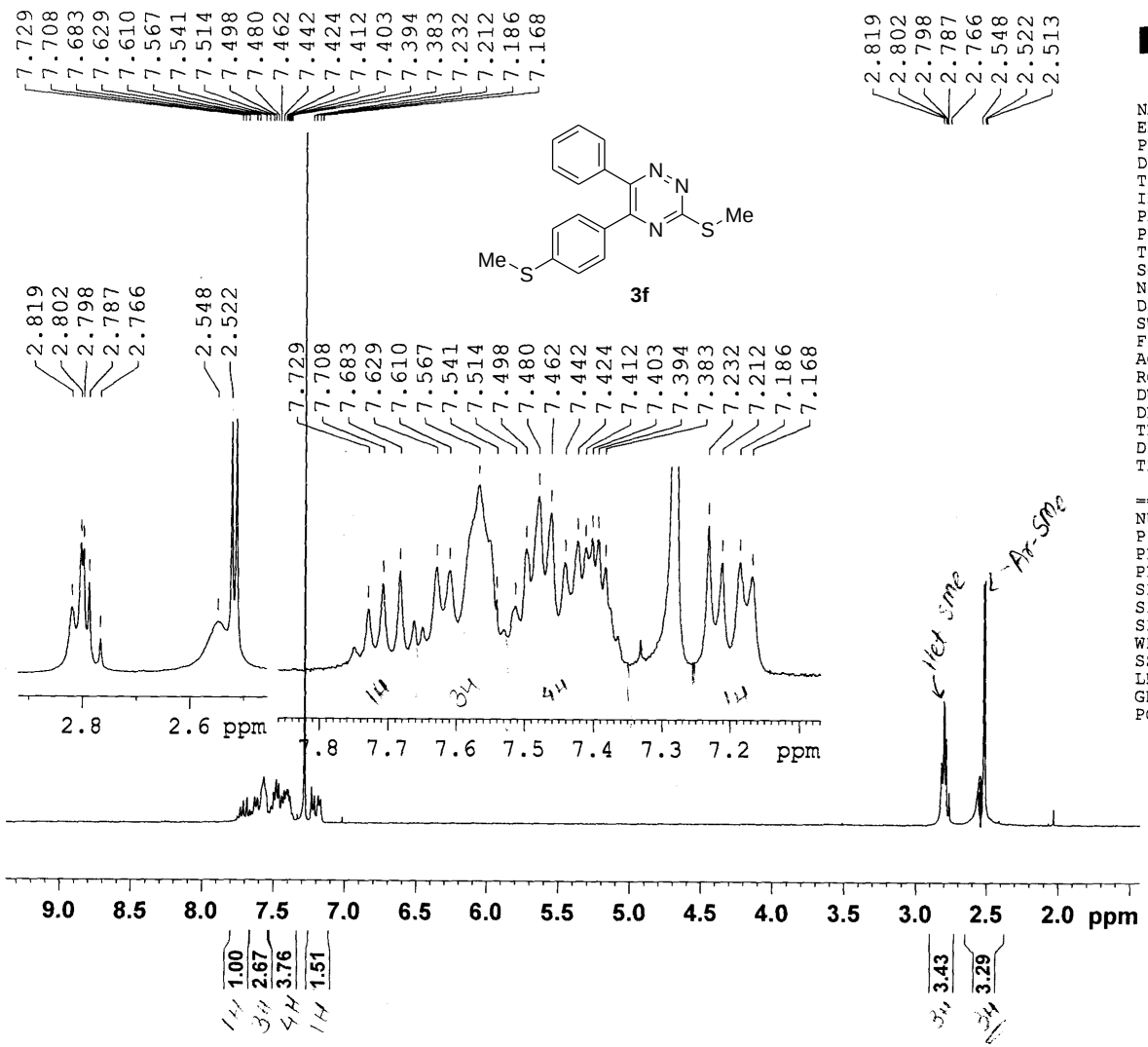
STRZ 4b SRS Triazine
PROTON CDCl3 {D:\SRS} MSU-Chem 1



3e'

NAME	STRZ-4 b
EXPNO	8
PROCNO	1
Date_	20121003
Time	11.45
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	256
DW	60.800 usec
DE	6.50 usec
TE	292.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
PL1W	10.80111122 W
SFO1	400.1524711 MHz
SI	32768
SF	400.1500000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

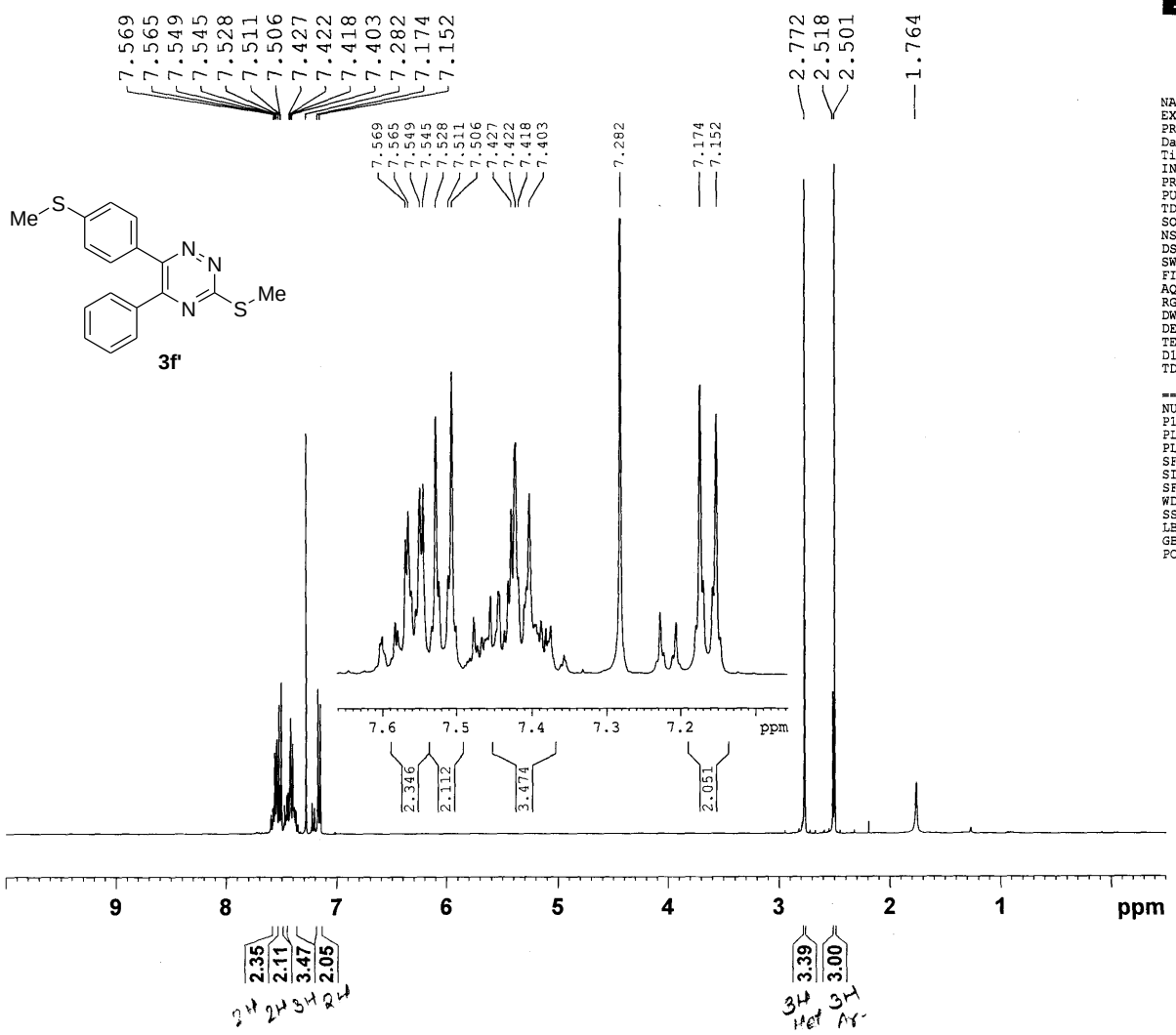
STRZ 5a SRS
PROTON CDCl3 {D:\SRS} MSU-Chem 1



BRUKER

NAME	STRZ-5a
EXPNO	3
PROCNO	1
Date_	20121113
Time	16.50
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	287
DW	60.800 usec
DE	6.50 usec
TE	292.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
PL1W	10.80111122 W
SFO1	400.1524711 MHz
SI	32768
SF	400.1500000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

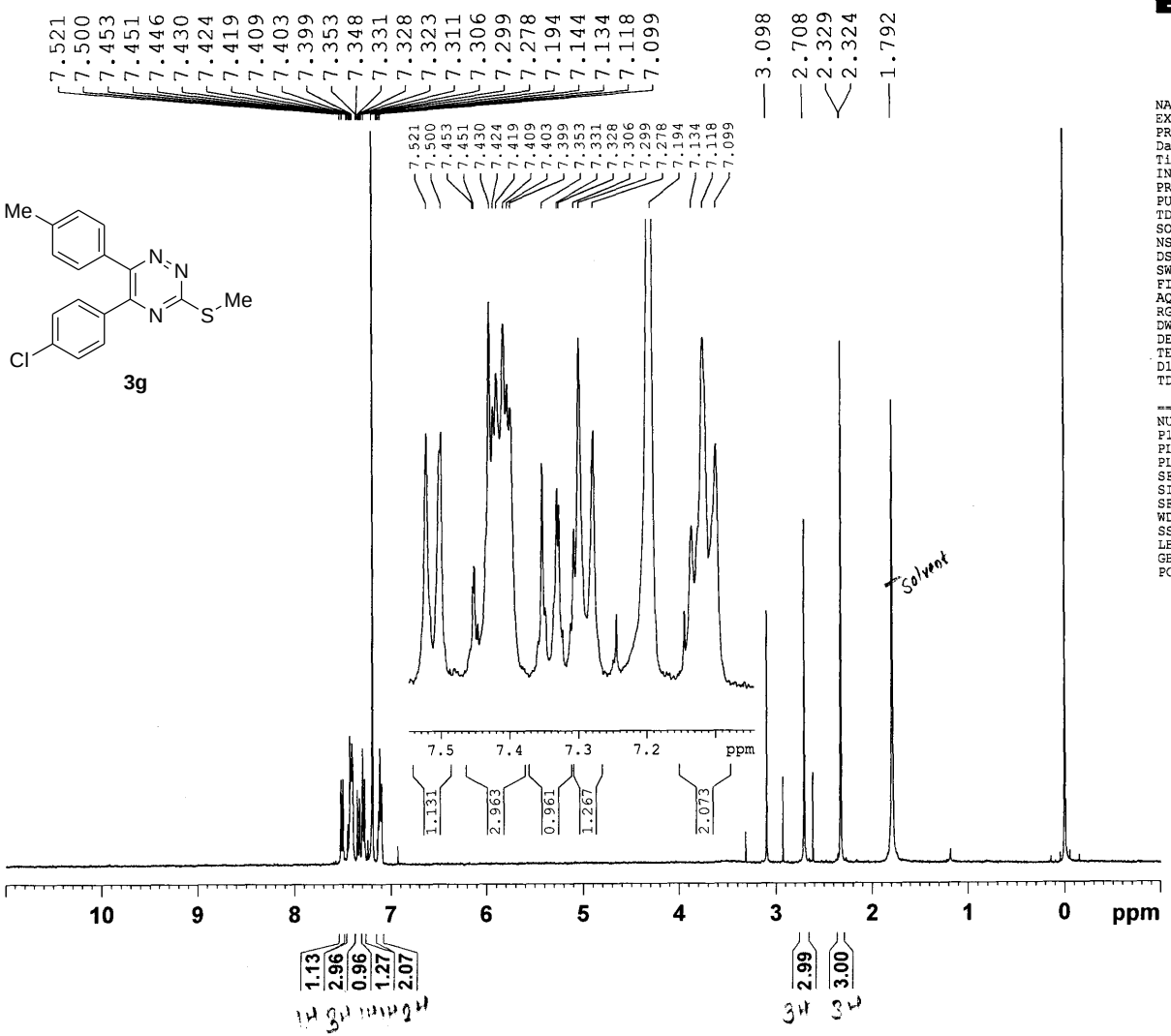
STRZ 5B Triazine
PROTON CDCl3 {D:\SRS} MSU-Chem 1



BRUKER

NAME	STRZ 5B
EXPNO	7
PROCNO	1
Date_	20120926
Time	18.41
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	228
DW	60.800 usec
DE	6.50 usec
TE	290.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
PL1W	10.80111122 W
SFO1	400.1524711 MHz
SI	32768
SF	400.1500000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

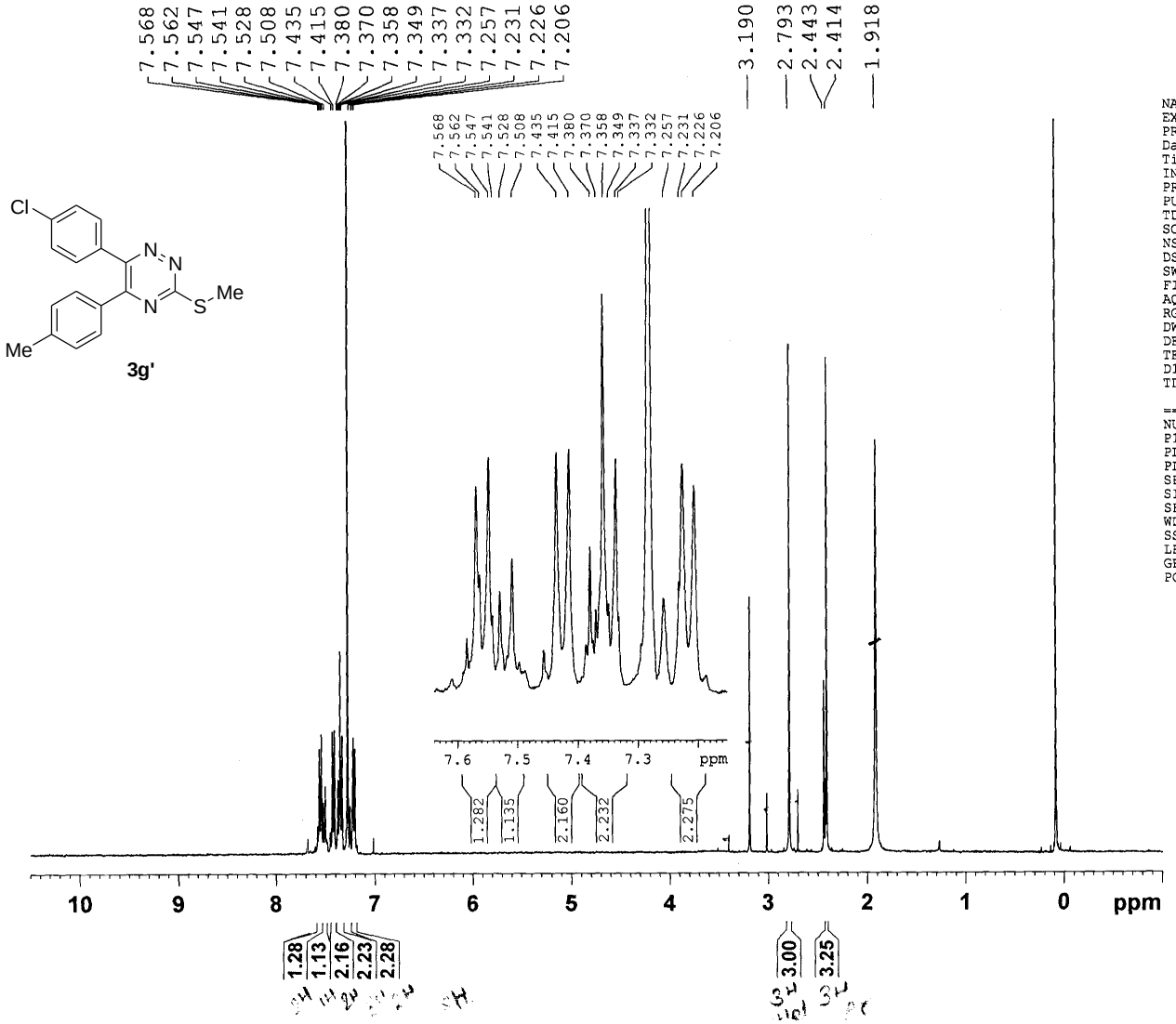
STRZ 6a SRS
PROTON CDCl3 {D:\SRS} MSU-Chem 1



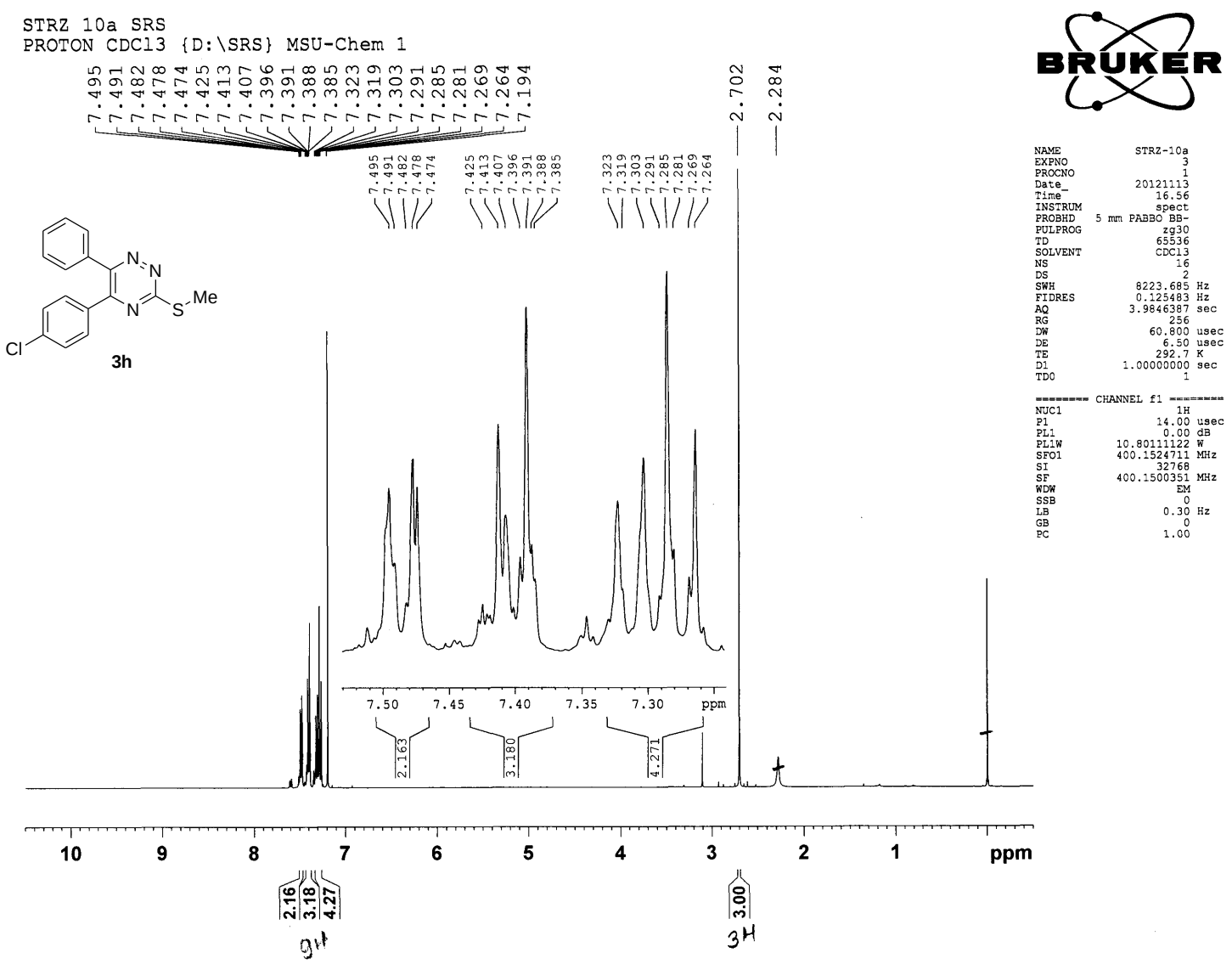
BRUKER

NAME	STRZ-6a
EXPNO	3
PROCNO	1
Date_	20121113
Time	17.03
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	287
DW	60.800 usec
DE	6.50 usec
TE	292.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
PL1W	10.80111122 W
SFO1	400.1524711 MHz
SI	32768
SF	400.1500349 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

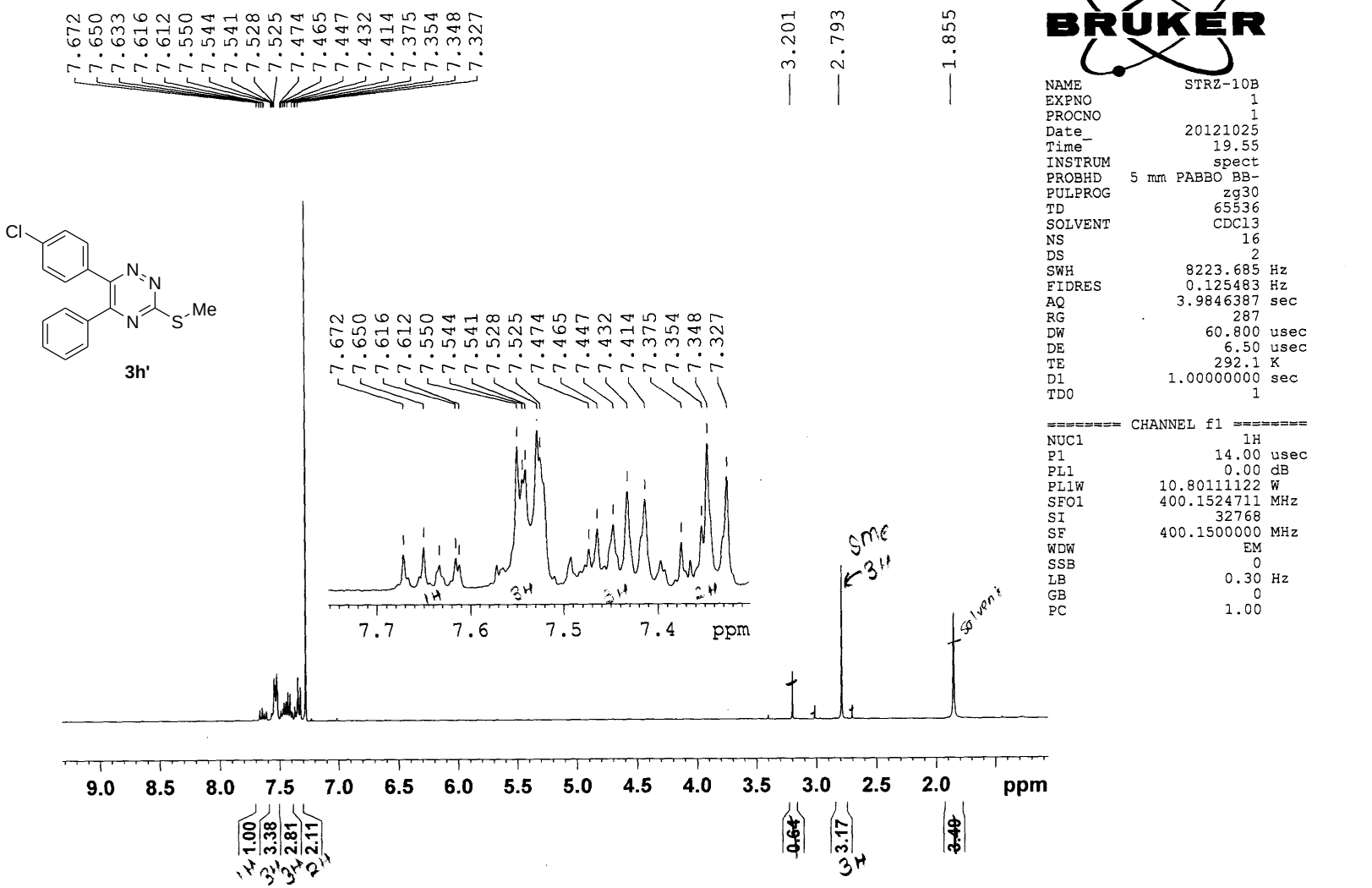
STRZ 6b SRS
PROTON CDCl3 {D:\SRS} MSU-Chem 1



NAME	STRZ-6b
EXPNO	3
PROCNO	1
Date_	20121113
Time	17.10
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	287
DW	60.800 usec
DE	6.50 usec
TE	292.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
PL1W	10.80111122 W
SFO1	400.1524711 MHz
SI	32768
SF	400.1500000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00


STRZ 10a SRS
PROTON CDCl3 {D:\SRS} MSU-Chem 1
7.495
7.491
7.482
7.478
7.474
7.425
7.413
7.407
7.396
7.391
7.388
7.385
7.323
7.319
7.303
7.291
7.285
7.281
7.269
7.264
7.194
2.702
2.284
N
N
N
S
Me
Cl
3h
7.50
7.45
7.40
7.35
7.30
ppm
2.163
3.180
4.271
10
9
8
7
6
5
4
3
2
1
ppm
2.16
3.18
4.27
9H
3.00
3H
BRUKER
NAME STRZ-10a
EXPNO 3
PROCNO 1
Date_ 20121113
Time 16.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
PL1W 10.80111122 W
SFO1 400.1524711 MHz
SI 32768
SF 400.1500351 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

SRTZ-10B proton
PROTON CDCl3 {D:\SRS} MSU-Chem 1

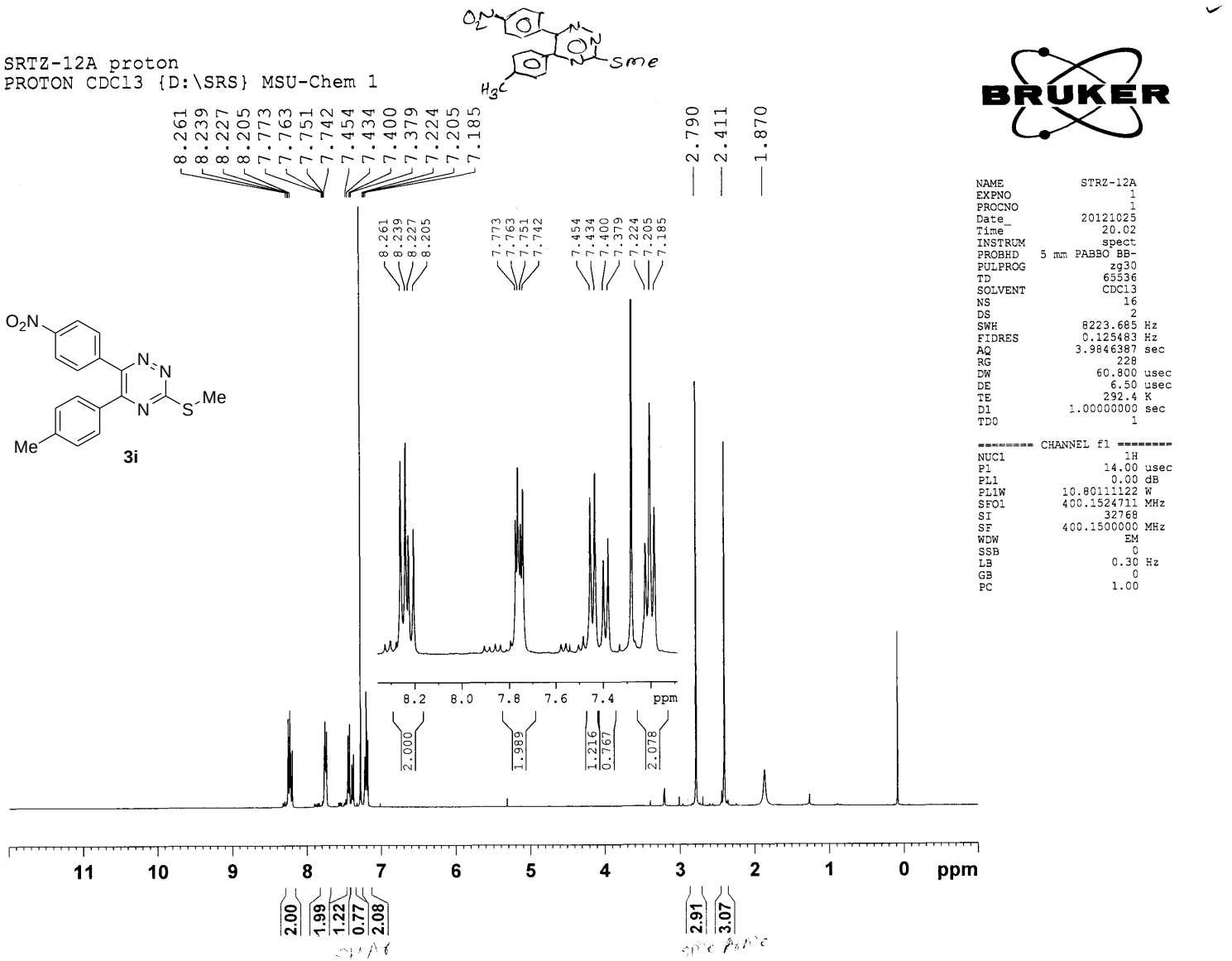


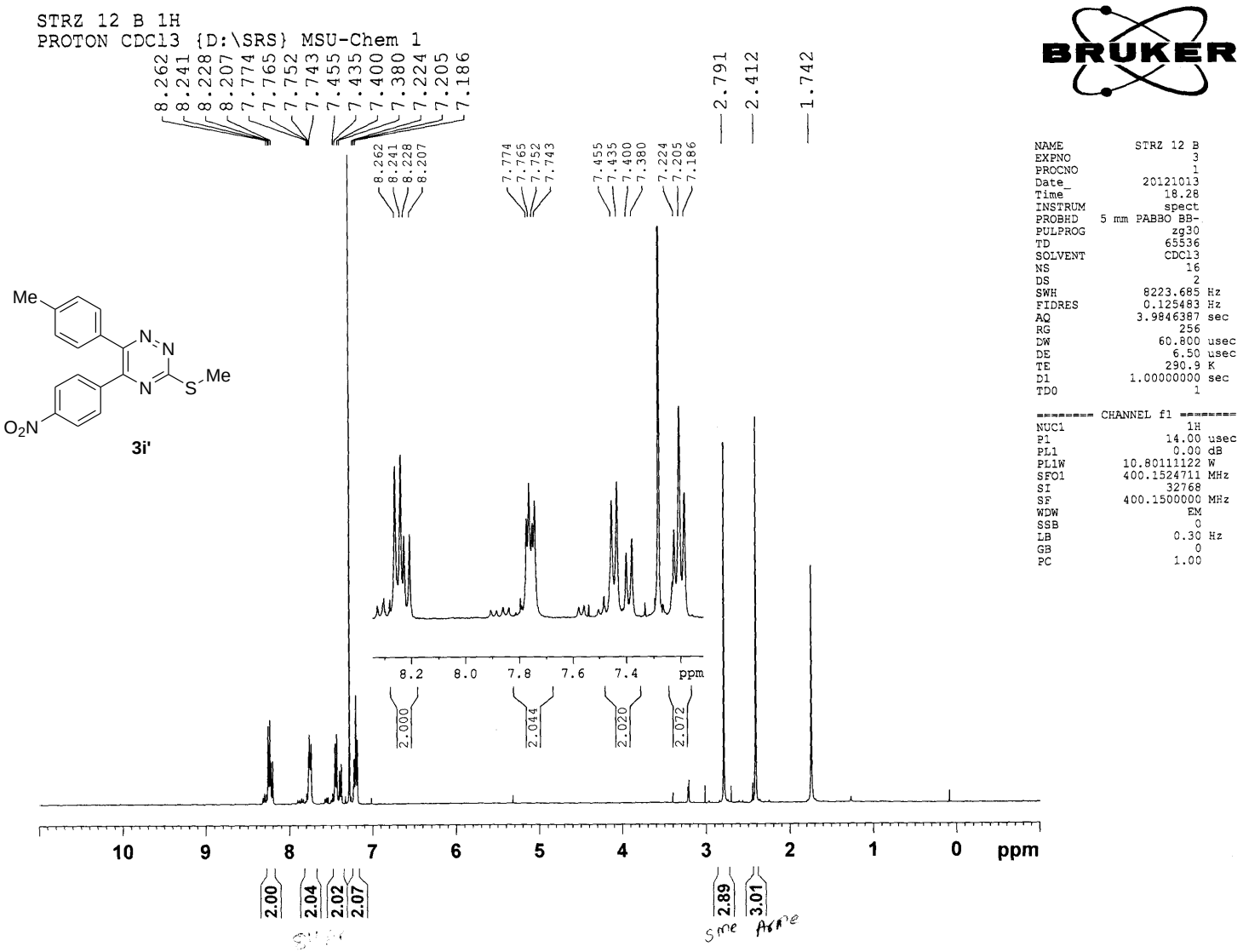
BRUKER

NAME	STRZ-10B
EXPNO	1
PROCNO	1
Date_	20121025
Time	19.55
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	287
DW	60.800 usec
DE	6.50 usec
TE	292.1 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

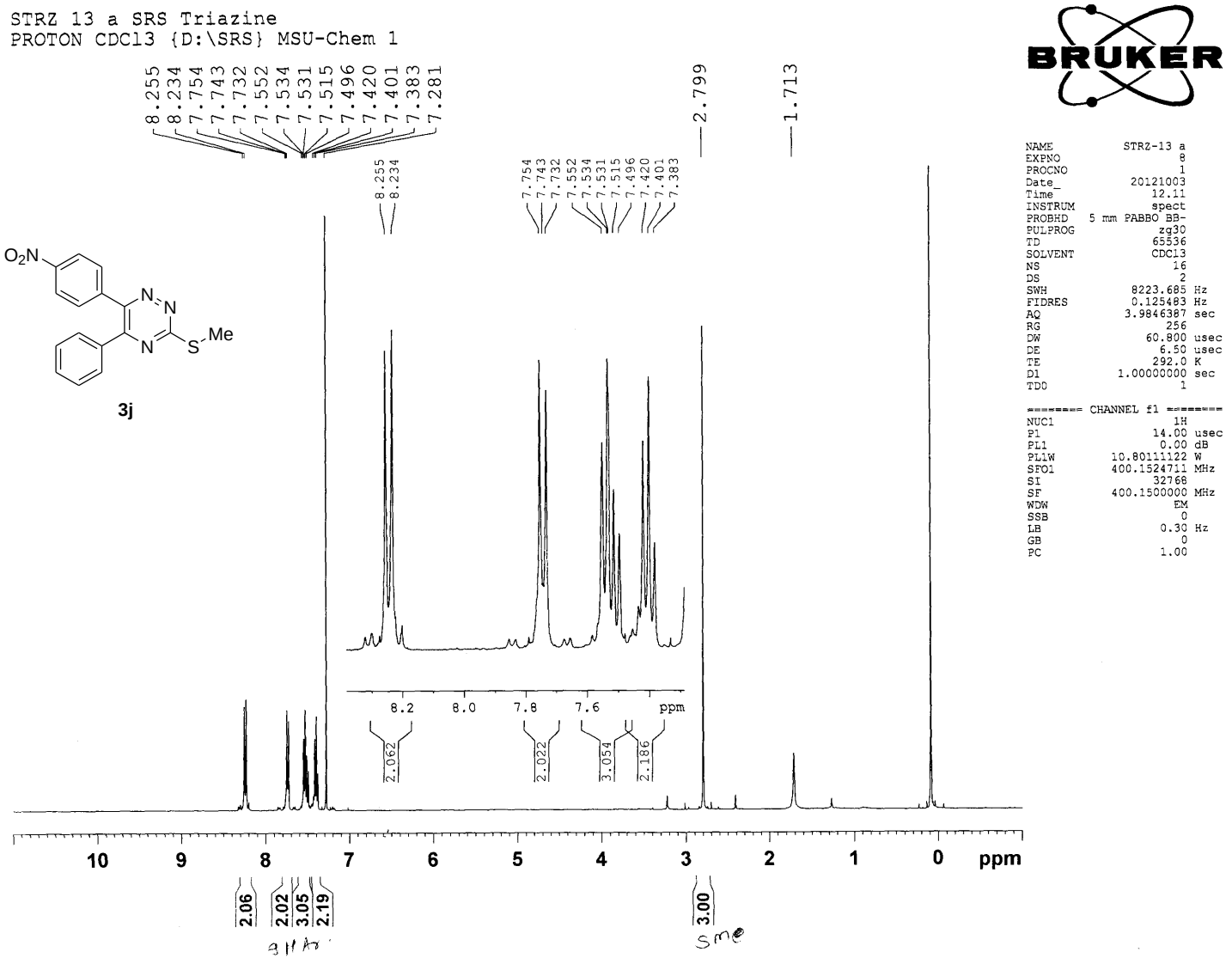
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
PL1W	10.80111122 W
SFO1	400.1524711 MHz
SI	32768
SF	400.1500000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00


SRTZ-12A proton
PROTON CDCl3 {D:\SRS} MSU-Chem 1
8.261
8.239
8.227
8.205
7.773
7.763
7.751
7.742
7.454
7.434
7.400
7.379
7.224
7.205
7.185
2.790
2.411
1.870
3i
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.00
1.99
1.22
0.77
2.08
2.91
3.07
8.2 8.0 7.8 7.6 7.4 ppm
2.000
1.989
1.216
0.767
2.078
NAME STRZ-12A
EXPNO 1
PROCNO 1
Date_ 20121025
Time 20.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 228
DW 60.800 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
PL1W 10.80111122 W
SFO1 400.1524711 MHz
SI 32768
SF 400.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



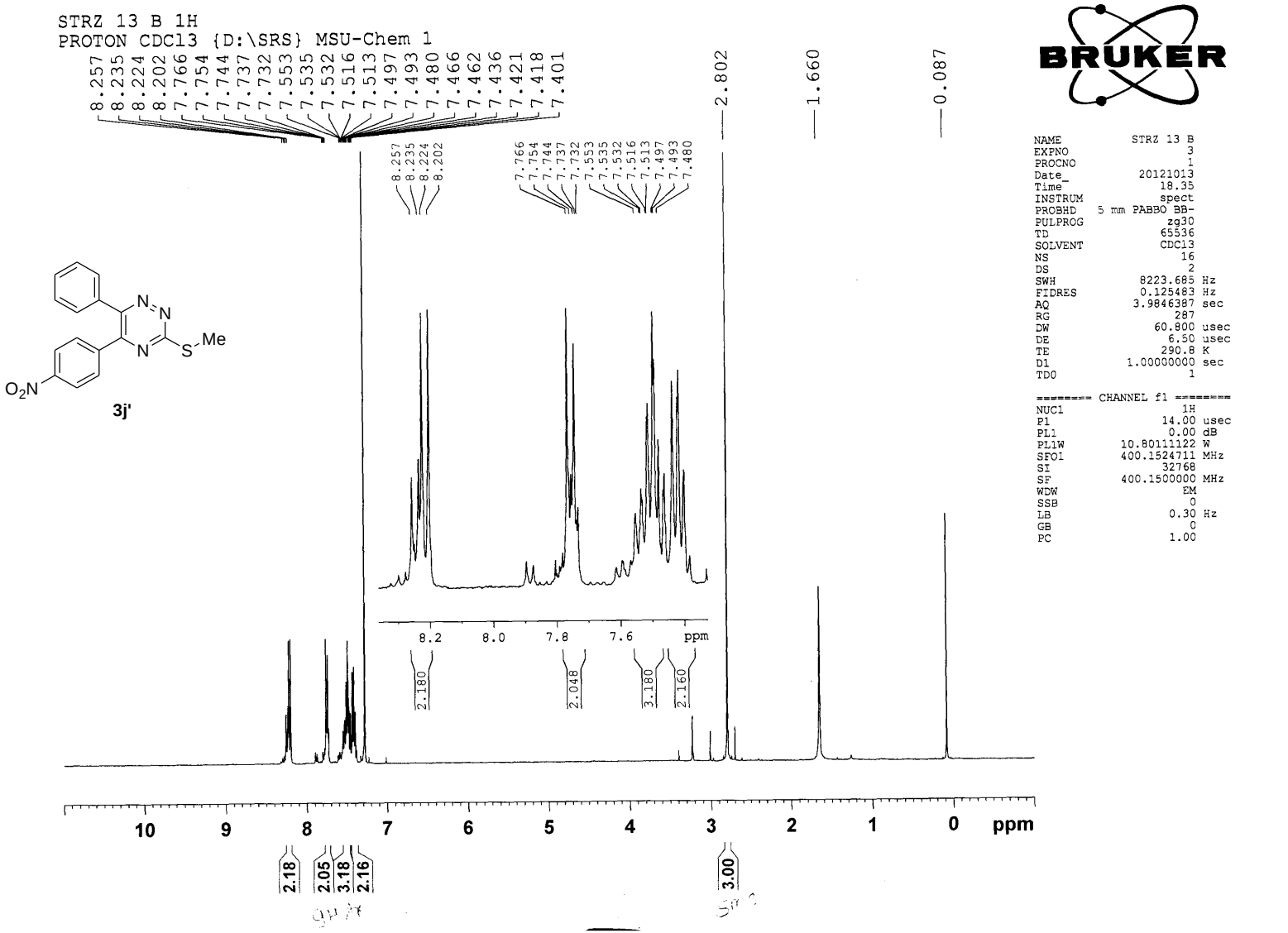
BRUKER

NAME	STRZ 12 B
EXPNO	3
PROCNO	1
Date_	20121013
Time	18.28
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	256
DW	60.800 usec
DE	6.50 usec
TE	290.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
PL1W	10.80111122 W
SFO1	400.1524711 MHz
SI	32768
SF	400.1500000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



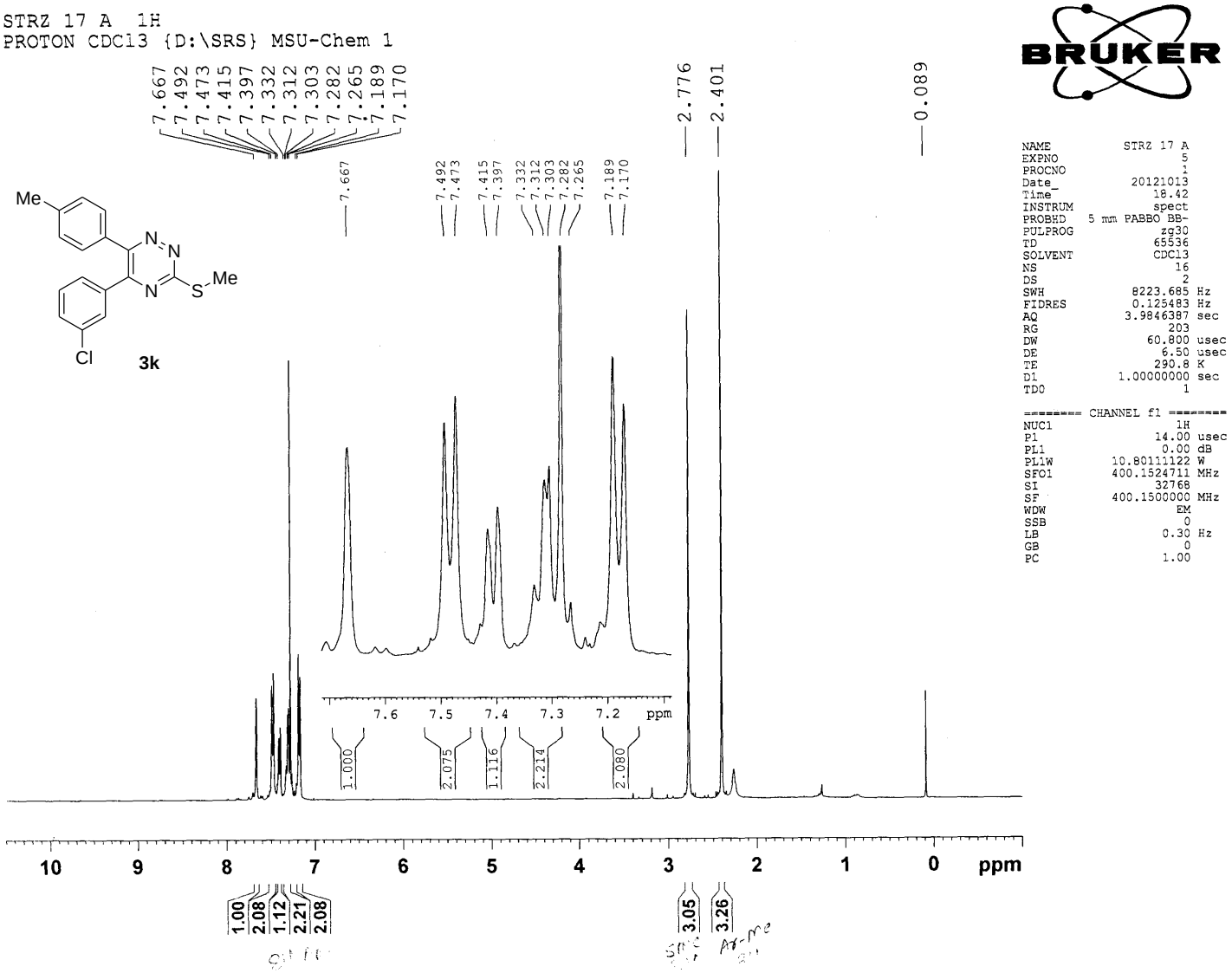
BRUKER

NAME	STRZ-13 a
EXPNO	8
PROCNO	1
Date_	20121003
Time	12.11
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	256
DW	60.800 usec
DE	6.50 usec
TE	292.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
PL1W	10.80111122 W
SFO1	400.1524711 MHz
SI	32768
SF	400.1500000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



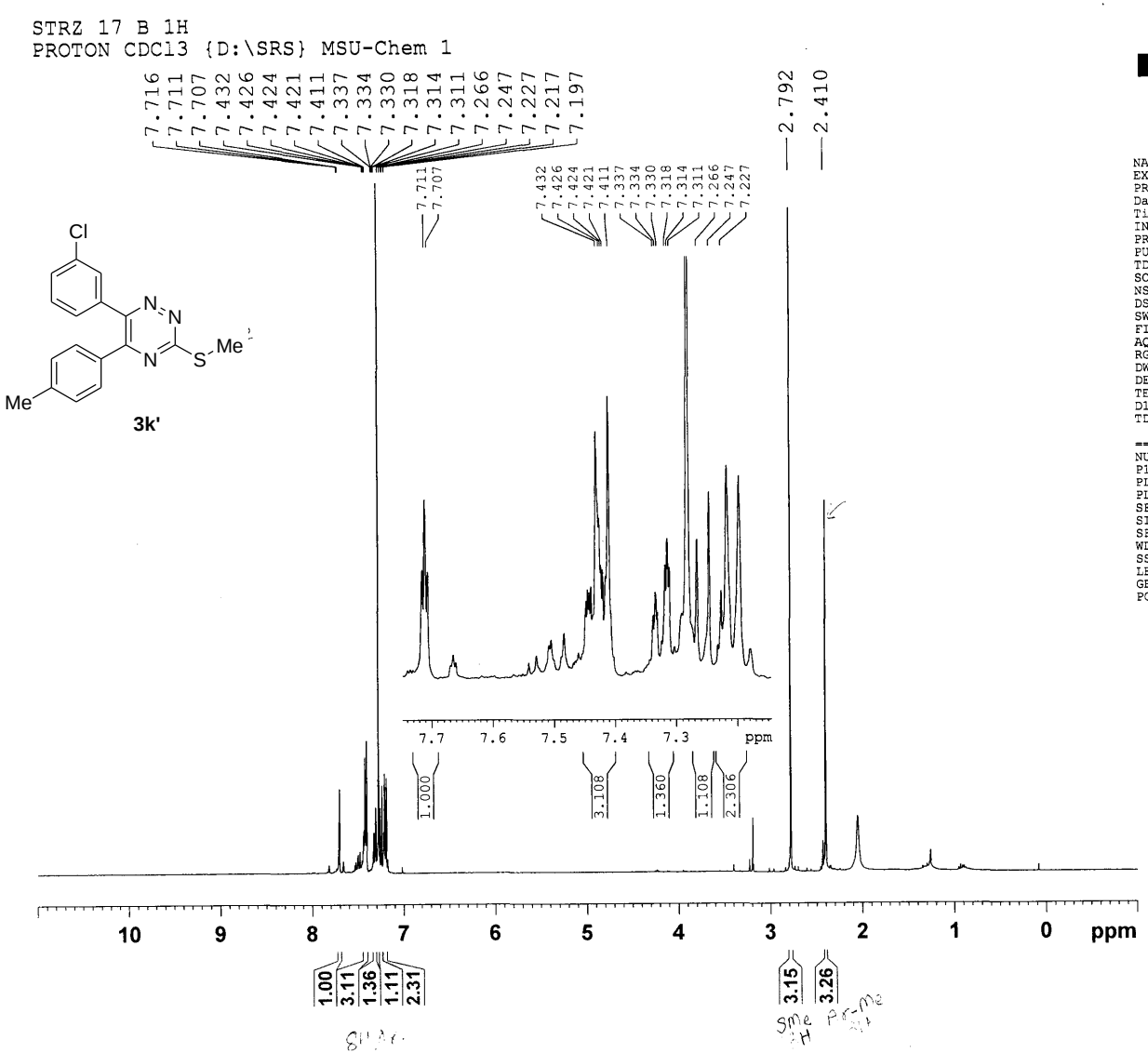
BRUKER

NAME	STRZ 13 B
EXPNO	3
PROCNO	1
Date_	20121013
Time	18.35
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	287
DW	60.800 usec
DE	6.50 usec
TE	290.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
PL1W	10.80111122 W
SFO1	400.1524711 MHz
SI	32768
SF	400.1500000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

NAME	STRZ 17 A
EXPNO	5
PROCNO	1
Date_	20121013
Time	18.42
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	290.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
PL1W	10.80111122 W
SFO1	400.1524711 MHz
SI	32768
SF	400.1500000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

NAME	STRZ 17 B
EXPNO	3
PROCNO	1
Date_	20121013
Time	18.21
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	256
DW	60.800 usec
DE	6.50 usec
TE	290.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	0.00 dB
PL1W	10.80111122 W
SFO1	400.1524711 MHz
SI	32768
SF	400.1500000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00