Supporting Information for *ChemBioChem* Z 672

Comparison of fertilinβ peptide-substituted polymers and liposomes as inhibitors of in vitro fertilization

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Materials and Methods

Amino acids and coupling agents used were purchased from Advanced Chem Tech. (Louisville, KY) or PerSeptive Biosystems (Framingham, MA). Solvents were obtained from Fisher Scientific Inc (Springfield, NJ), and Aldrich (Milwaukee, WI). $Cl_2(PCy_3)_2Ru{=}CHPh$ was purchased from Fluka (Milwaukee, WI). CH_2Cl_2 was freshly distilled from CaH; CF_3CH_2OH, CH_3OH, and Et_2O, were used without further purification. LiCl was oven-dried and stored over P_2O_5 before use. All reactions were carried out under an N_2 or Ar atmosphere in oven-dried glassware. Moisture and oxygen-sensitive reagents were handled in an N_2-filled drybox. 5-Norbornene-*exo*-carboxylic acid was synthesized according to the literature.[1]

Analytical thin layer chromatography (TLC) was performed on precoated silica gel plates ($60F_{254}$), and flash chromatography on silica gel-60 (230-400 mesh). TLC spots were detected by UV light and by staining with phosphomolybdic acid (PMA). Peptides were purified by flash column chromatography on silica gel-60. The purities of all peptide monomers were assessed by RP-HPLC using a Vydac C_{18} column. Gradient elution was performed at 1 mL/min with acetonitrile and water (both containing TFA, 0.1%). The purity of the polymers was assessed by aqueous phase gel-filtration chromatography (BioSep-SEC-S2000) using 50 mM potassium, pH 7. Bruker AC-250, Gemini 2300, Inova500, and Inova600 MHz NMR spectrometers were used to perform NMR analysis, and spectra were recorded in $CDCl_3$ unless otherwise noted. Chemical shifts are quoted in parts per million (ppm) and ^1H NMR data are assumed to be first order.

Peptide synthesis

General procedure for amino acid coupling. A typical amino acid coupling was carried out in dry CH_2Cl_2 with TBTU/HOBt (1.1 eq / 0.37 eq) and DIEA (1.6 eq). Each reaction was carried out under Ar at a final concentration of 0.7 M in the amine compound, and a 1.1-fold excess of the carboxylic acid component. Upon completion of the reaction, the mixture was diluted with CH_2Cl_2, washed with 1N HCl and 5 % $NaHCO_3$, and the organic layer was dried with Na_2SO_4. The solvent was rotary evaporated. The peptide was purified by flash chromatography eluting with 10 % acetone/CH_2Cl_2 or 20 % EtOAc/CH_2Cl_2.

General procedure for Cbz hydrogenation. A methanolic solution of Cbz-protected peptide (0.3 M) and 10% Pd-C (0.05 eq) was stirred under an H_2 atmosphere for 2 h. The catalyst was removed by filtration and the amine used without further purification.

General procedure for Fmoc removal.[2] A solution of Fmoc-protected peptide in dry CH_2Cl_2 (0.5 M), was treated with octanethiol (0.1 eq) and a catalytic amount of DBU (0.001 eq). The reaction was stirred at RT for 16 h. The solvent was concentrated and the product was purified by flash column chromatography eluting with a step gradient of ranging from 2 % to 50 % EtOAc/CH_2Cl_2

Peptide 1

Z-VT(tBu)-OMe. H-T(tBu)-OMe and Z-Val-OH were coupled and purified to yield 1.26 g (97 %) of Z-VT(tBu)-OMe. ^{1}H-NMR (250 MHz): δ 7.36 (m, 5H), 6.42 (d, J= 10.0, 1H), 5.49 (d, J= 7.5, 1H), 5.11 (s, 2H), 4.47 (dd, J= 8.8 and 1.3, 1H), 4.25 (m, 1H), 4.22 (m, 1H), 3.69 (s, 3H), 2.12 (m, 1H), 1.15 (d, J = 5.0, 3H), 1.10 (s, 9H), 1.02 (d, J= 7.5, 3H), 0.97 (d, J = 5.0, 3H).

Z-D(tBu)VT(tBu)-OMe. Z-VT(tBu)-OMe was deprotected and coupled to Z-Asp (tBu)-OH to yield 0.822 g (98 %) of Z-D(tBu)VT(tBu)-OMe. ^{1}H-NMR (250 MHz): δ 7.31 (m, 5H), 7.12 (d, J= 10.0, 1H), 6.40 (d, J= 10.0, 1H), 6.05 (d, J= 7.5, 1H), 5.10 (s, 2H), 4.55 (m, 1H), 4.43 (dd, J = 8.8 and 1.3, 1H), 4.33 (dd, J = 7.5 and 5.0, 1H), 4.20 (m, 1H), 3.68 (s, 3H), 2.90 (dd, J= 17.0 and 5.0, 1H), 2.60 (dd, J= 17.5 and 5.0, 1H), 2.12 (m, 1H), 1.40 (s, 9H), 1.13 (d, J = 7.5, 3H), 1.10 (s, 9H), 0.96 (d, J= 5.0, 3H), 0.93 (d, J = 5.0, 3H).

Fmoc-C(Trt)D(tBu)VT(tBu)-OMe. Z-D(tBu)VT(tBu)-OMe was deprotected and coupled to Fmoc-Cys(Trt)-OH to yield 0.873 g (95 %) of Fmoc-C(Trt)D(tBu)VT(tBu)-OMe. ^{1}H-NMR (300 MHz): δ 7.74 (dd, J= 6.6 and 6.3, 2H), 7.56 (m, 2H), 7.43 (m, 7H), 7.38 (m, 2H), 7.25 (m, 13H), 7.13 (d, J= 8.7, 1H), 6.38 (d, J= 9.0, 1H), 4.96 (d, J= 6.0, 1H), 4.69 (m, 1H), 4.46 (dd, J= 8.7 and 1.3, 1H), 4.35 (d, J= 6.6, 2H), 4.22 (m, 3H), 3.69 (s, 3H), 2.90 (dd, J = 17.1 and 4.1, 1H), 2.73 (m, 2H), 2.54 (dd, J= 17.3 and 6.2, 1H), 2.00 (m, 1H), 1.39 (s, 9H), 1.14 (d, J= 6.3, 3H), 1.10 (s, 9H), 0.90 (d, J= 6.9, 3H), 0.85 (d, J= 6.6, 3H).

Fmoc-E(tBu)C(Trt)D(tBu)VT(tBu)-OMe. Fmoc-C(Trt)D(tBu)VT(tBu)-OMe was deprotected and coupled to Fmoc-Glu(tBu)-OH to yield 0.660 g (95 %) of Fmoc-E(tBu)C(Trt)D(tBu)VT(tBu)-OMe. ^{1}H-NMR (300 MHz): δ 7.76 (d, J= 6.0, 2H), 7.59(m, 2H), 7.40 (m, 7H), 7.28 (m, 10H), 7.14 (m, 4H), 6.53 (d, J= 6.0,1H), 6.42 (d, J= 7.0, 1H), 6.03 (d, J= 6.3, 1H), 4.73 (m, 1H), 4.46 (dd, J= 9.3 and 5.0, 1H), 4.35 (m, 2H), 4.22 (m, 1H), 4.13 (m, 4H), 3.91 (m, 1H), 3.68 (s, 3H), 2.81 (dd, J= 17 and 5.3, 1H), 2.62 (m, 3H), 2.38 (m, 3H), 2.08 (m, 2H), 1.86 (m, 1H), 1.45 (s, 9H), 1.40 (s, 9H), 1.13 (d, J= 6.3, 3H), 1.10 (s, 9H), 0.92 (d, J= 6.6, 3H), 0.88 (d, J= 6.9, 3H).

*Norbornene-E(tBu)C(Trt)D(tBu)VT(tBu)-OMe **1***. Fmoc-E(tBu)C(Trt)D(tBu)VT(tBu)-OMe was deprotected and coupled to 5-Norbornene-*exo*-carboxylic acid to yield 0.751 g (89 %) of norbornene-E(tBu)C(Trt)D(tBu)VT(tBu)-OMe, **1**. ^{1}H-NMR (250 MHz): δ 7.38 (m, 6H), 7.20 (m, 11H), 6.98 (dd, J= 8.1 and 6.3, 1H), 6.72 (dd, J= 7.2 and 3.3, 1H), 6.47 (dd, J= 9.0 and 2.7, 1H), 6.0 (m, 2H), 4.74 (m, 1H), 4.43 (dd, J= 8.7 and 1.8, 1H), 4.19 (m, 3H), 3.92 (m, 1H), 3.65 (s, 3H), 2.78 (m, 5H), 2.49 (m, 2H), 2.27 (m, 1H), 2.02 (m, 3H), 1.83 (m, 2H), 1.60 (m, 2H), 1.41 (d, J= 4.5, 9H), 1.39 (s, 9H), 1.24 (m, 2H), 1.11 (d, J= 6.3, 3H), 1.07 (s, 9H), 0.90 (d, J= 6.6, 3H), 0.87 (d, J= 6.9, 3H). ^{13}C (250 MHz) δ 18.04, 19.01, 20.82, 26.41, 27.97, 28.26, 30.44, 30.54, 30.77, 32.22, 32.96, 33.05, 36.72, 41.48, 44.39, 46.33, 46.98, 49.72, 51.97, 52.51, 52.61, 54.15, 57.73, 58.86, 73.95, 81.19, 81.31, 126.83, 128.02, 129.45, 135.87, 138.07, 144.15, 169.33, 169.96, 170.56, 170.76, 171.00, 171.36, 173.66, 176.73. MALDI, calcd for $(MNa)^{+}$1132.89, found 1133.96. HPLC purity was 98 %.

Peptide 2

Z-VD(tBu)-OMe. Asp(tBu)-OMe and Z-Val-OH were coupled and purified to yield 0.800 g (97 %) of Z-VD(tBu)-OMe. ^{1}H-NMR (300 MHz): δ 7.39 (m, 5H), 6.83 (d, J=8.7, 1H), 5.45 (d, J=8.4, 1H), 5.15 (s, 2H), 4.86 (m, 1H), 4.11 (m, 1H), 3.77 (s, 3H), 3.03 (dd, J=17.1 and 4.2, 1H), 2.74 (dd, J=17.1 and 4.2, 1H), 2.21 (m, 1H), 1.46 (s, 9H), 1.04 (d, J=6.6, 3H), 0.98 (d, J=6.9, 3H).

Z-E(tBu)VD(tBu)-OMe. Z-VD(tBu)-OMe was deprotected and coupled to Z-Glu(tBu)-OH to yield 0.751 g (98 %) of Z-E(tBu)VD(tBu)-OMe. ^{1}H-NMR (300 MHz): δ 7.35 (m, 5H), 7.10 (d, J=8.4, 1H), 7.00 (d, J=8.7, 1H), 5.87 (d, J=7.5, 1H), 5.13 (s, 2H), 4.85 (m, 1H), 4.35 (m, 2H), 3.76 (s, 3H), 2.96 (dd, J=17.0 and 4.8, 1H), 2.73 (dd, J=17.0 and 4.4, 1H), 2.42 (m, 2H), 2.15 (m, 2H), 1.98 (m, 1H), 1.46 (s, 9H), 1.46 (s, 9H), 0.99 (d, J=6.9, 3H), 0.97 (d, J=6.9, 3H).

Z-T(tBu)E(tBu)VD(tBu)-OMe. Z-E(tBu)VD(tBu)-OMe was deprotected and coupled to Z-Thr(tBu)-OH to yield 0.702 g (96 %) of Z-T(tBu)E(tBu)VD(tBu)-OMe. ^{1}H-NMR (500 MHz): δ 7.76 (d, J= 7.5, 1H), 7.35 (m, 5H), 6.99 (d, J=8.0, 1H), 6.88 (d, J=8.5, 1H), 5.90 (d, J=5.5, 1H), 5.15 (d, J=12.5, 1H), 5.10 (d, J=12.0, 1H), 4.82 (dt, J=9.0 and 4.5, 1H), 4.42 (dd, J=13.3 and 7.3, 1H), 4.28 (dd, J=8.3 and 5.8, 1H), 4.19 (bs, 2H), 3.75 (s, 3H), 2.93 (dd, J=16.8 and 4.8, 1H), 2.72 (dd, J=17.0 and 5.0, 1H), 2.43 (m, 1H), 2.36 (m, 1H), 2.23 (m, 1H), 2.13 (m, 1H), 1.97 (m, 1H), 1.45 (s, 9H), 1.44 (s, 9H), 1.28 (s, 9H) 1.08 (d, J= 6.5, 3H), 0.98 (d, J=8.0, 3H), 0.96 (d, J=7.0, 3H).

H-C(Trt)T(TBu)E(tBu)VD(tBu)-OMe. Z-T(tBu)E(tBu)VD(tBu)-OMe was deprotected and coupled to Fmoc-Cys(Trt)-OH. The Fmoc protecting group was then removed to yield 0.470 g (92 %) of H-C(Trt)T(TBu)E(tBu)VD(tBu)-OMe. ^{1}H-NMR (500 MHz): δ 7.81 (d, J= 6.3, 1H), 7.69(d, J= 7.4, 1H), 7.45 (m, 6H), 7.23 (m, 9H), 6.99 (d, J= 8.4, 1H), 6.90 (d, J= 8.4,1H), 4.81 (dt, J= 8.9 and 4.7, 1H), 4.37 (dd, J= 12.9 and 7.7, 1H), 4.23 (m, 2H), 4.11 (m, 1H), 3.71 (s, 3H), 2.90 (m, 2H), 2.70 (m, 2H), 2.34 (m, 4H), 2.24 (m, 2H), 1.93 (m, 1H), 1.42 (s, 18H), 1.24 (s, 9H), 0.97 (d, J= 6.6, 3H), 0.95 (d, J= 6.7, 3H).

Norbornene-C(Trt)T(tBu)E(tBu)VD(tBu)-OMe ***2***. H-C(Trt)T(TBu)E(tBu)VD(tBu)-OMe was coupled to 5-norbornene-exo-carboxylic acid to yield 0.400 g (90 %) of norbornene-C(Trt)T(tBu)E(tBu)VD(tBu)-

OMe, **2**. ^{1}H-NMR (600 MHz): δ 7.42 (m, 6H), 7.30 (m, 6H), 7.25 (m, 4H), 6.92 (m, 2H), 6.89 (d, J= 6.6, 1H), 6.13 (m, 2H), 5.67(d, J= 4.8, 1H), 4.82 (m, 1H), 4.37 (m, 1H), 4.27 (m, 2H), 4.13 (m, 1H), 4.00 (m, 1H), 3.72 (s, 3H), 2.87(m, 2H), 2.76 (m, 2H), 2.66 (m, 1H), 2.33 (m, 3H), 2.14 (m, 1H), 1.92 (m, 2H), 1.83 (d, J= 12.0 and 3.3, 1H), 1.43 (s, 9H), 1.42 (s, 9H), 1.27 (m, 2H), 1.17 (s, 9H), 1.07 (d, J= 6.6, 3H), 0.95 (d, J= 6.6, 3H), 0.93 (d, J= 6.6, 3H). ^{13}C (250 MHz) δ 17.68, 19.05, 19.87, 26.11, 26.51, 27.27, 27.99, 28.44, 30.45, 30.67, 31.90, 33.96, 37.18, 38.54, 41.54, 44.39, 46.21, 47.06, 47.67, 48.53, 49.26, 51.02, 52.47, 52.59, 58.55, 65.39, 66.67, 67.65, 68.05, 74.32, 80.77, 81.73, 126.79, 127.92, 129.39, 135.84, 138.24, 144.23, 169.98, 170.44, 170.59, 170.98, 171.12, 172.58, 175.02, 175.05. MALDI, calcd for $(MNa)^{+}$1132.89, found 1133.96. HPLC purity was 96 %.

Norbornyl oligopeptide polymers

General polymerization procedure. Catalyst $Cl_2(PCy_3)_2Ru$=CHPh was weighed in an N_2-filled drybox and dissolved in CH_2Cl_2/CH_3OH (3/1) to give a typical concentration of 0.03 M. Monomers were each dissolved in a minimum amount of CH_2Cl_2/CH_3OH (3/1) and LiCl (6 M) was added to the mixture. The desired portion of catalyst was added via syringe to the reaction bottle under an inert atmosphere. A typical reaction was carried out at an initial monomer concentration of 0.2 to 0.3 M. The reaction was stirred at RT for 3 to 4 h before quenching with ethyl vinyl ether and stirring for an additional 30 min. The solvent was removed and the product was washed with H_2O. Polymers were dissolved in CH_2Cl_2 and precipitated with cold Et_2O. Product was isolated by centrifugation and dried under vacuum in the presence of P_2O_5.

General deprotection and reduction procedure. Polymers were deprotected in a cocktail containing H_2O, TIS and TFA (2.5, 2.5, 95) for 5 h. The reaction mixtures were concentrated with N_2 and precipitated in cold Et_2O and centrifuged. Polymers were dissolved in H_2O at pH 6 and reduced with excess TCEP for 5 h with stirring at 37 C. Pure deprotected product was isolated by precipitation with 1

N HCl. Excess TCEP was removed by repeated washing with H_2O. A gray white solid was collected, dried and stored at -20 C.

Polymer **3**.Yield 22 mg (80 %) ^{1}H-NMR (D_2O, 500 MHz) δ 7.26 (m), 6.1 (m), 5.30 (bs), 4.40 – 4.00 (with max. at 4.42, 4.28, 4.13), 3.44 – 3.36 (with max. at 3.64, 3.57), 3.10 (bs), 2.85 (bs), 2.24 -2.74 (with max. at 2.58, 2.46, 2.38), 1.42 – 2.21 (with max. at 2.07, 1.90, 1.78, 1.58), 0.94 – 1.41 (with max. at 1.27, 1.19, 1.09), 0.83 (s). GFC purity was 96 %.

Polymer **4**.Yield 16 mg (77 %) ^{1}H-NMR (D_2O, 600 MHz) δ 7.20(m), 5.34 (bs), 4.67 (bs), 4.54 (bs), 3.96 – 4.20 (with max. at 4.10, 4.09, 4.00), 3.50 – 3.61 (with max. at 3.63, 3.58), 3.21 (s), 2.82 (bs), 2.58 (bs), 2.47 (bs), 1.40 - 2.20 (with max. at 2.17, 1.90, 1.78, 1.57), 0.97 – 1.40 (with max. at 1.26, 1.18, 1.08), 0.82 (s). GFC purity was 98 %.

In vitro fertilization assay

Eggs and sperm were isolated from ICR (Taconic farms) or CD-1 (Charles River Laboratories) mice as described in Yuan et al.[3] Zona pellucida were removed by treatment with acid Tyrode's solution for 30 sec and recovered at 37 °C, 5% CO_2 for 1 h. Zona-free eggs were loaded with Hoechst 33342 (1 μg/mL) for 30 min and washed through six drops of M16 medium (0.5% BSA). Eggs (20-30/40 μL) were incubated with peptide in M16 medium (3% BSA) for 45 min and then sperm were added (5 x 10^4/mL). After 45 min the eggs were washed in M16 medium (3% BSA) and mounted onto microscope slides. Fusion was scored by fluorescent labeling of sperm nuclei by Hoechst 33342 present in preloaded eggs. 3-7 experiments per peptide were performed and 25-100 eggs per peptide were used. A DAPI (465 nm) cutoff filter was used for fluorescence microscopy.

References

[1]Manning, D.D.; Strong, L.E.; Hu, X.; Beck, P.J.; Kiessling, L.L. *Tetrahedron* **1997**, *53*, 11937.
[2]Sheppek, J.E.; Kar, H. *Tetrahedron Lett.* **2000**, *41*, 5329 .
[3]Yuan, R.; Primakoff, P.; Myles, D. G. *J. Cell. Biol.* **1997**, *137*, 105.

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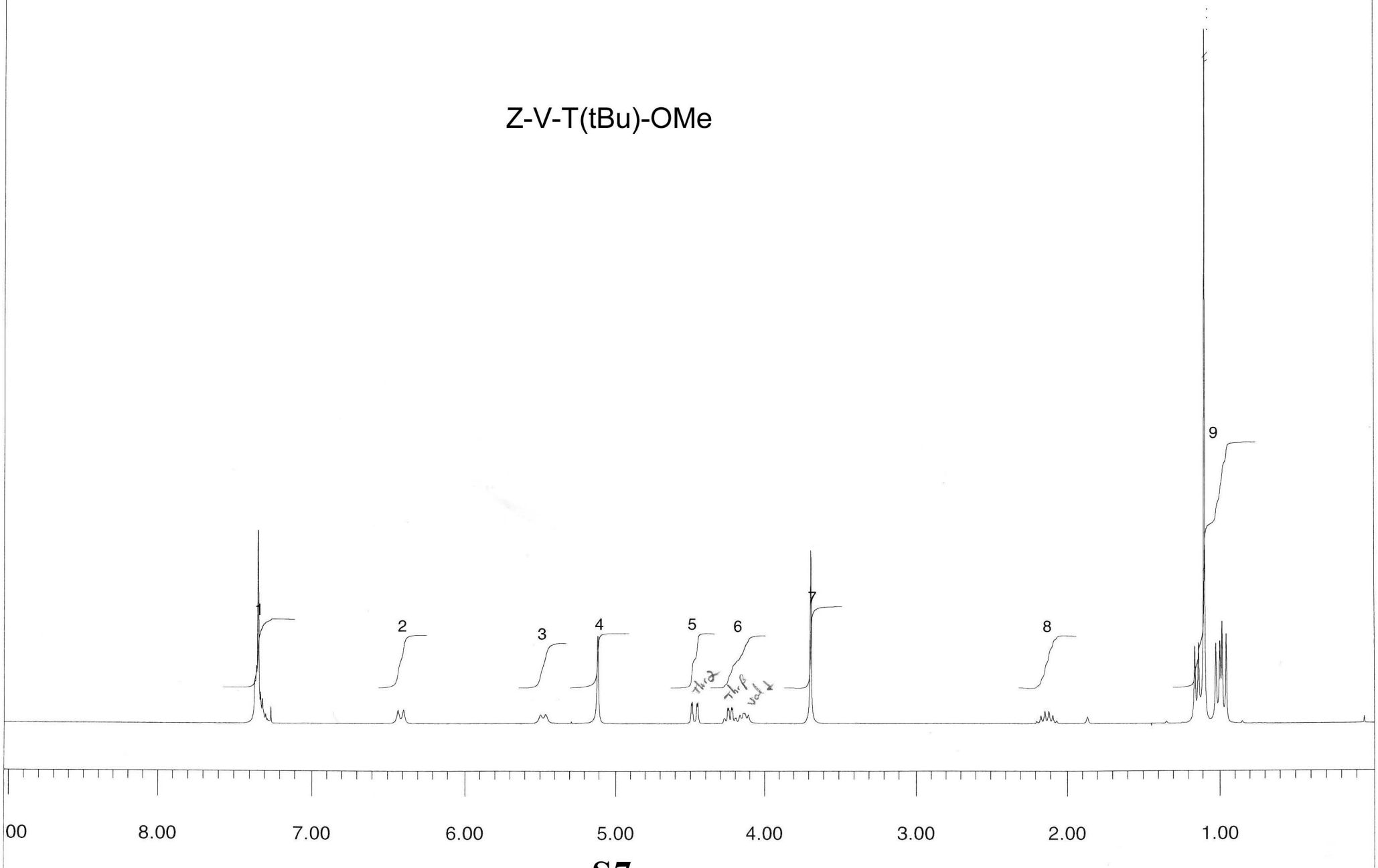
Z-V-T(tBu)-OMe



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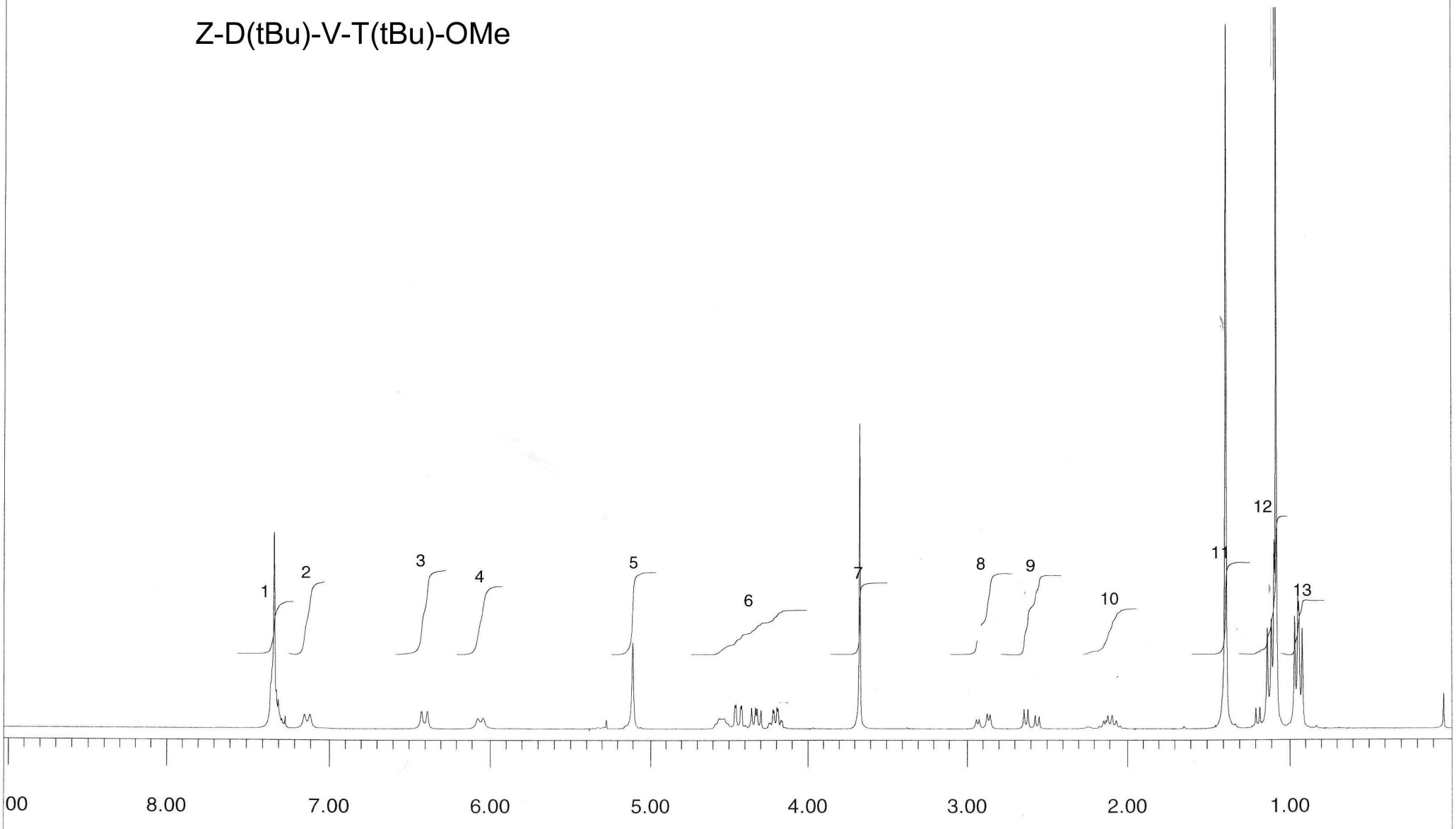
Z-D(tBu)-V-T(tBu)-OMe



Fmoc-C(Trt)-D(tBu)-V-T(tBu)-OMe

STANDARD 1H OBSERVE

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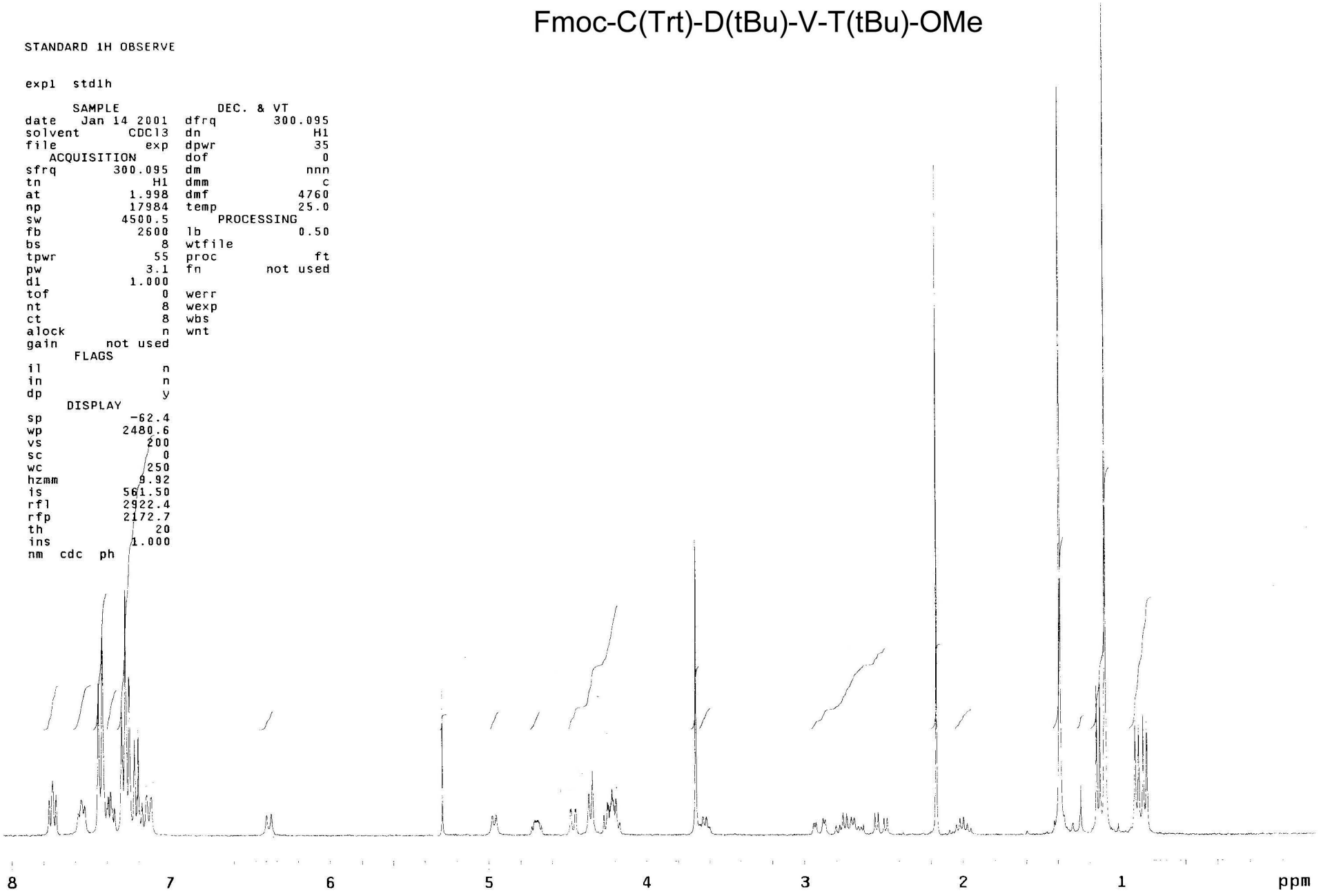

Fmoc-E(tBu)-C(Trt)-D(tBu)-V-T(tBu)-OMe

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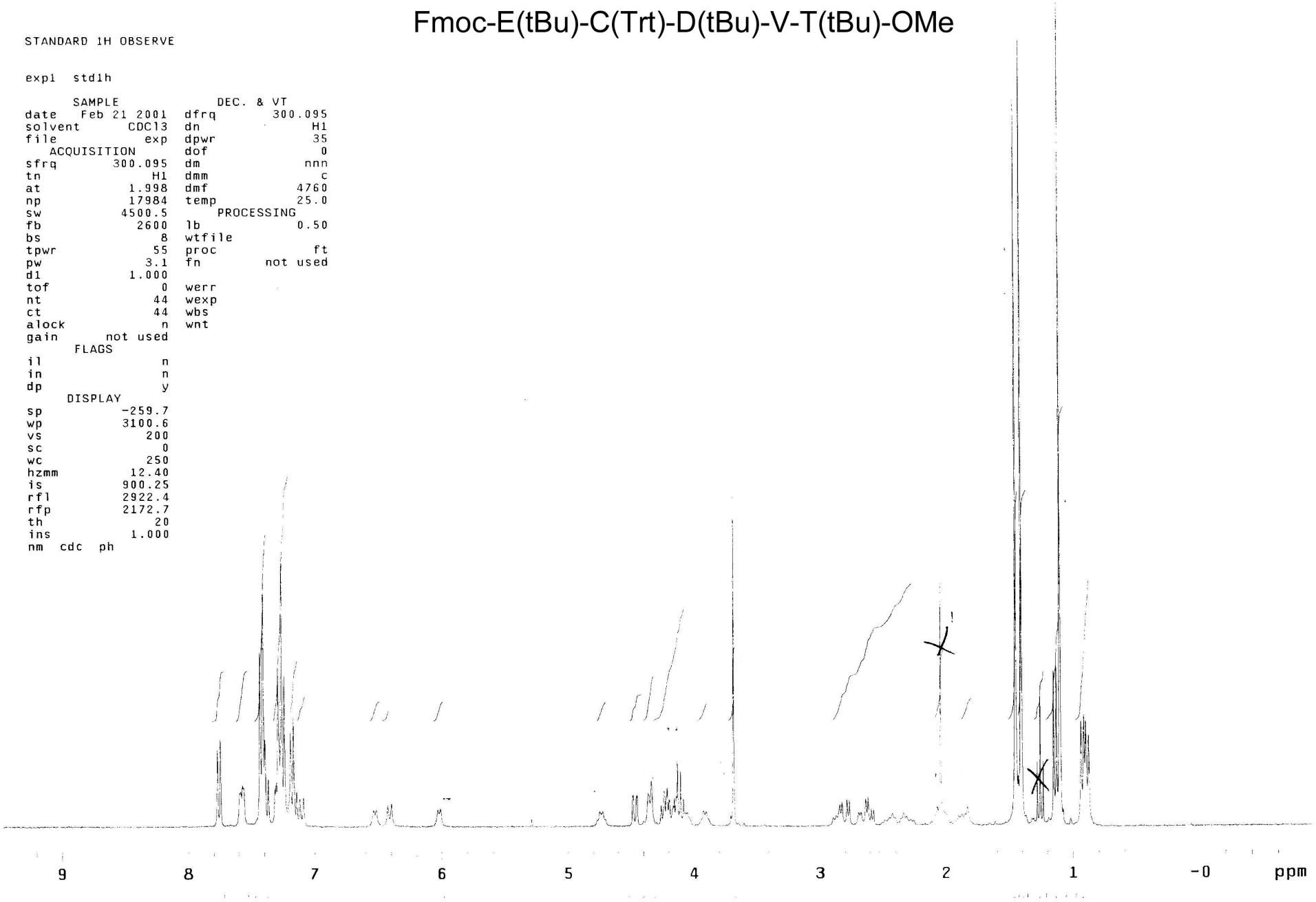
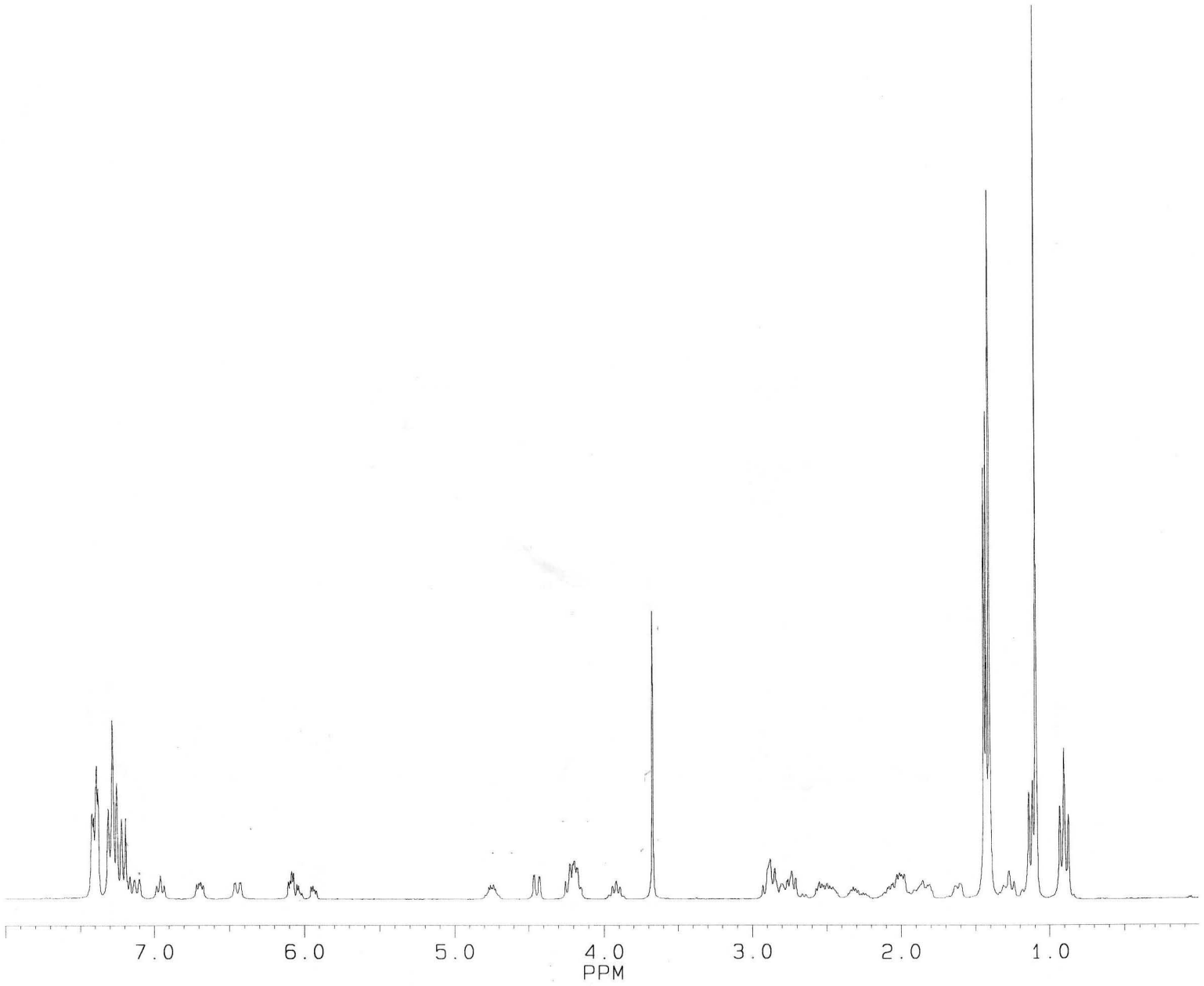


Norbornene-E(tBu)-C(Trt)-D(tBu)-V-T(tBu)-OMe

1



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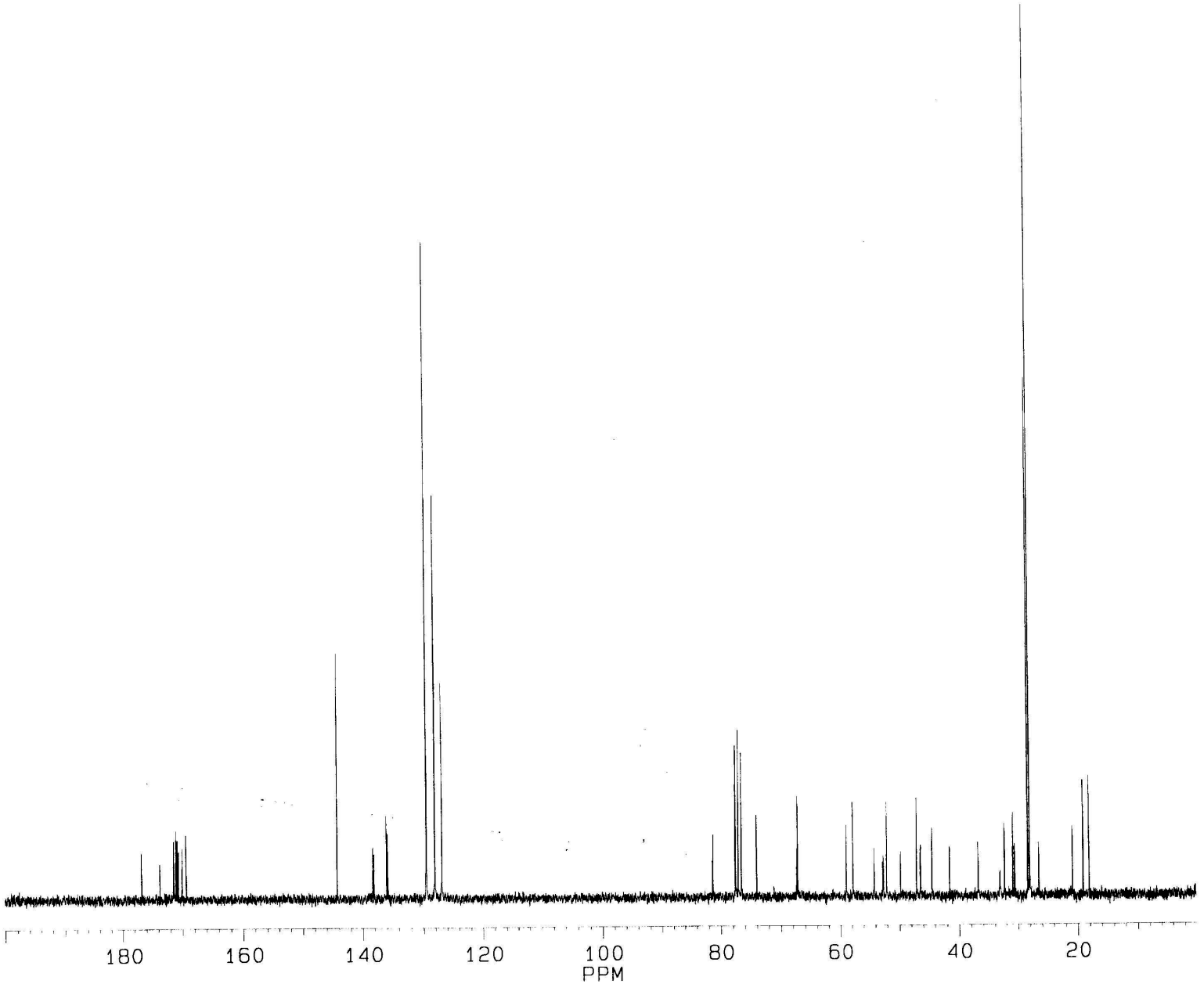
PPM/CM .400
SR 2856.30

Norbornene-E(tBu)-C(Trt)-D(tBu)-V-T(tBu)-OMe



BRUKER

XHUI.030
DATE 20-12-1

SF 62.910
SY 62.0
O1 3218.018
SI 32768
TD 32768
SW 15625.000
HZ/PT .954

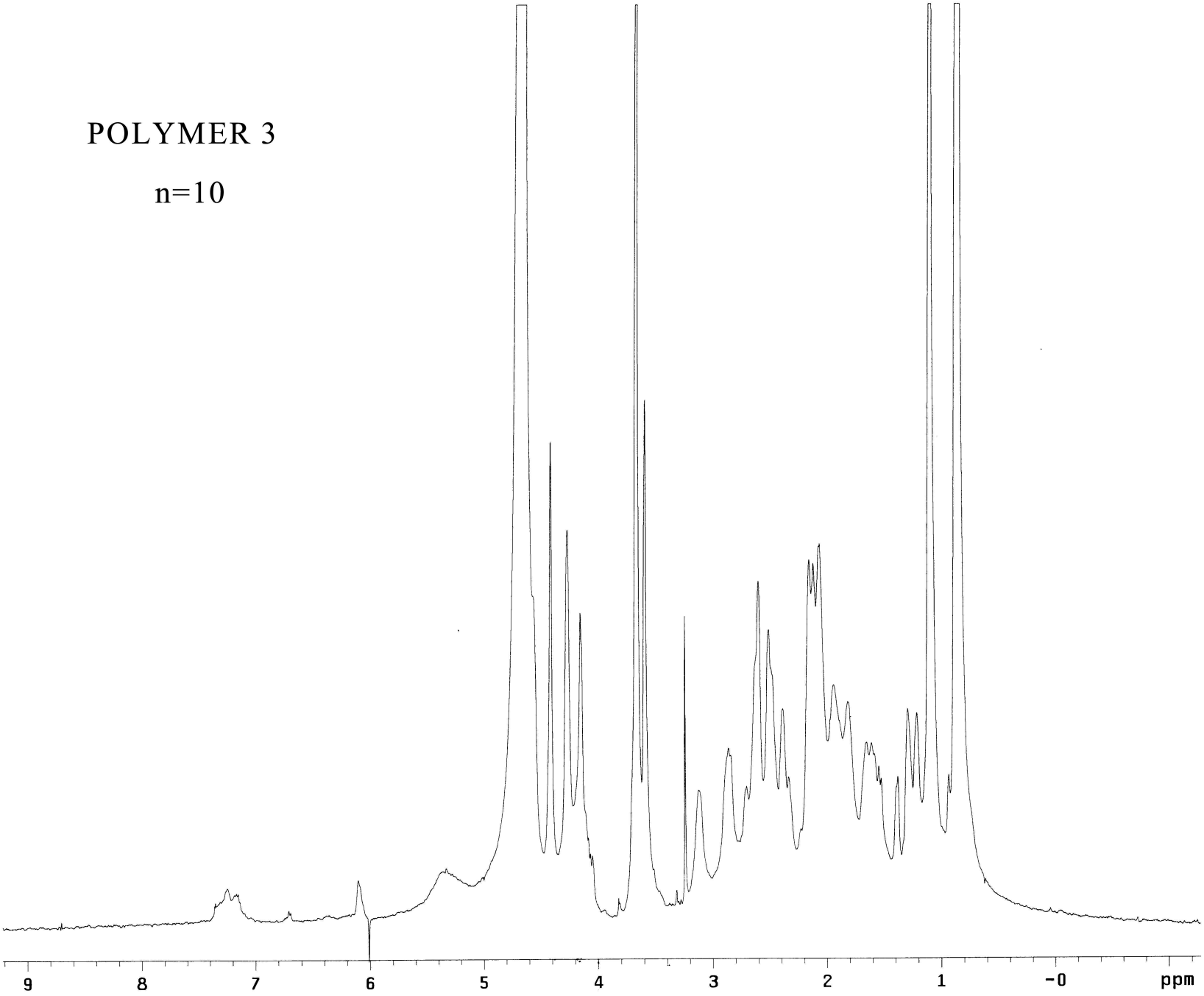
PW 5.0
RD 1.000
AQ 1.049
RG 400
NS 817
TE 297

FW 19600
O2 4100.000
DP 20H CPD

LB .200
GB 0.0
CX 20.00
CY 15.00
F1 200.000P
F2 .004P
HZ/CM 629.091
PPM/CM 10.000
SR -4040.63

POLYMER 3

n=10

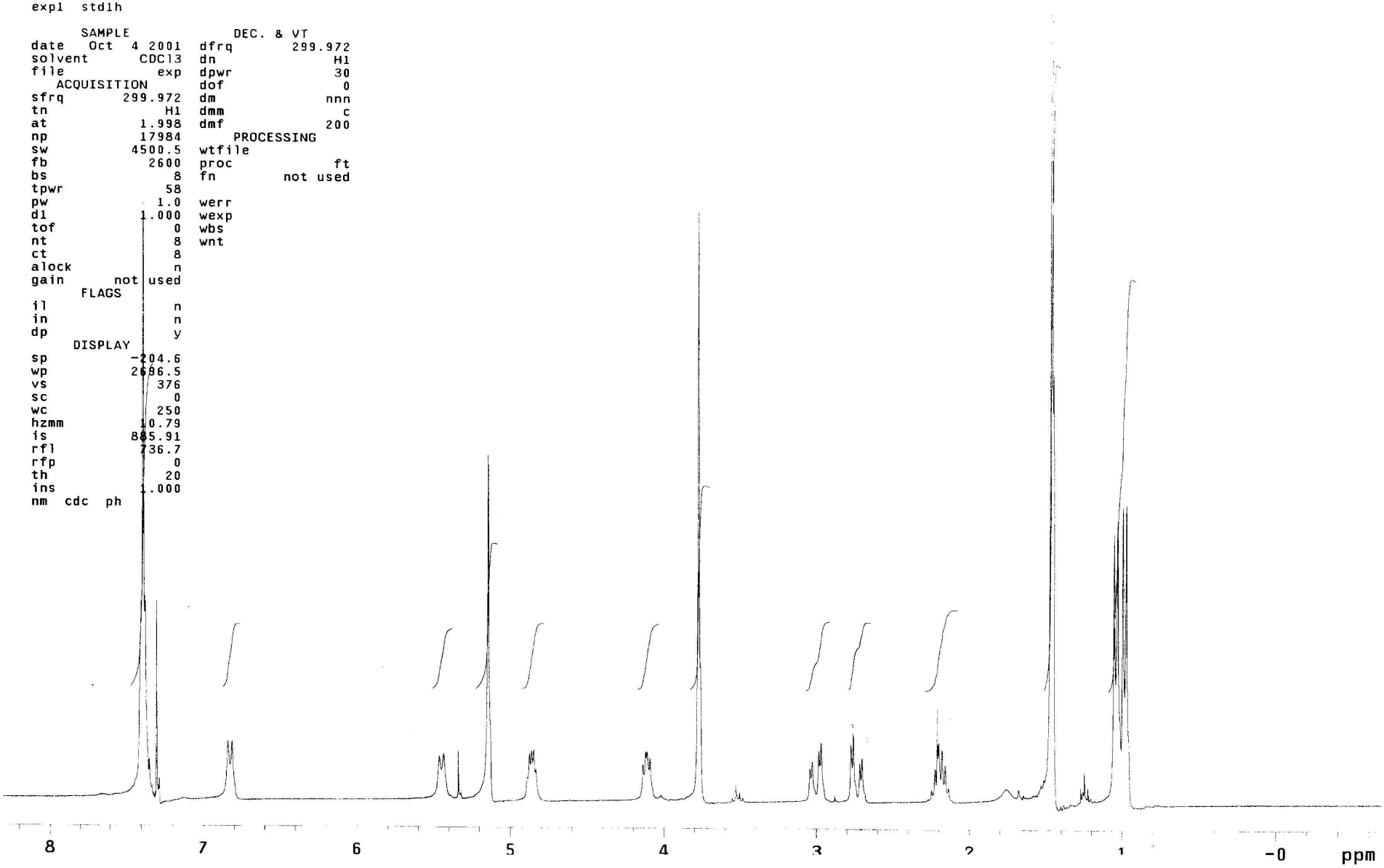


STANDARD 1H OBSERVE

Z-V-D(tBu)-OMe

exp1 std1h

SAMPLE		DEC. & VT	
date	Oct 4 2001	dfrq	299.972
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.972	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	17984	PROCESSING	
sw	4500.5	wtfile	
fb	2600	proc	ft
bs	8	fn	not used
tpwr	58		
pw	1.0	werr	
d1	1.000	wexp	
tof	0	wbs	
nt	8	wnt	
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-204.6		
wp	2696.5		
vs	376		
sc	0		
wc	250		
hzmm	10.79		
is	885.91		
rfl	736.7		
rfp	0		
th	20		
ins	1.000		
nm cdc ph			

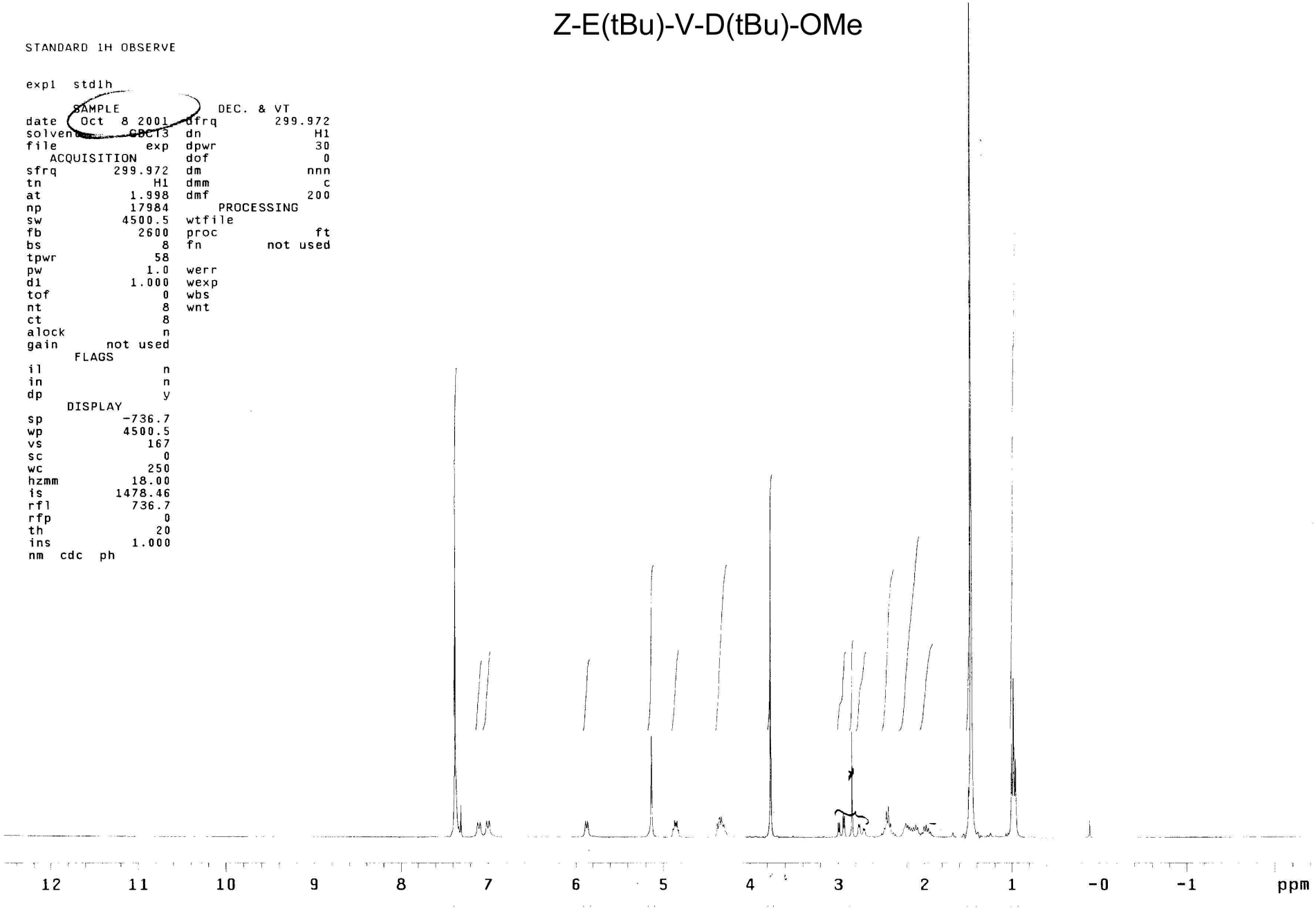


Z-E(tBu)-V-D(tBu)-OMe

STANDARD 1H OBSERVE

expl stdlh

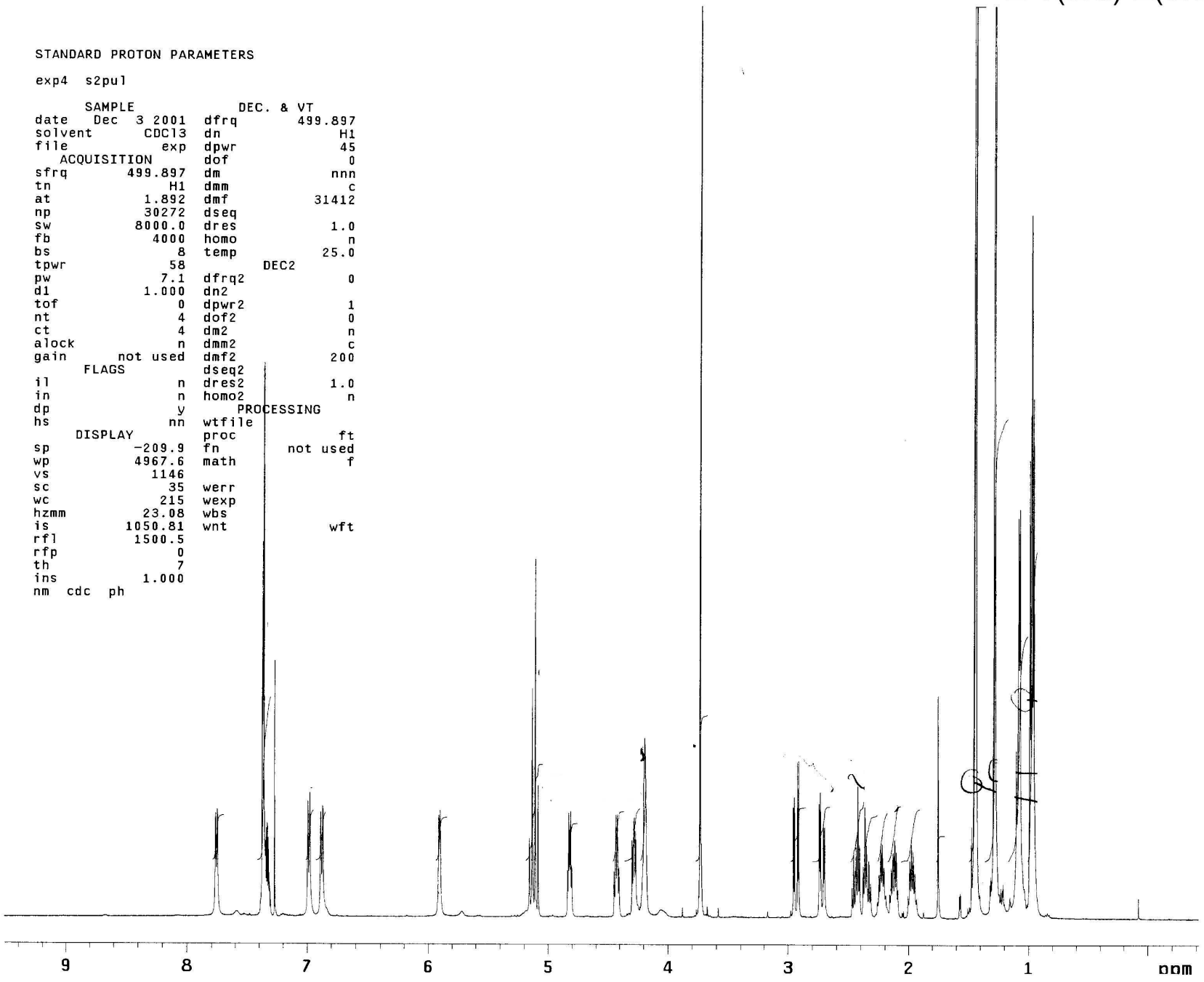
SAMPLE		DEC. & VT	
date	Oct 8 2001	dfrq	299.972
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.972	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	17984	PROCESSING	
sw	4500.5	wtfile	
fb	2600	proc	ft
bs	8	fn	not used
tpwr	58		
pw	1.0	werr	
d1	1.000	wexp	
tof	0	wbs	
nt	8	wnt	
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-736.7		
wp	4500.5		
vs	167		
sc	0		
wc	250		
hzmm	18.00		
is	1478.46		
rfl	736.7		
rfp	0		
th	20		
ins	1.000		
nm cdc ph			



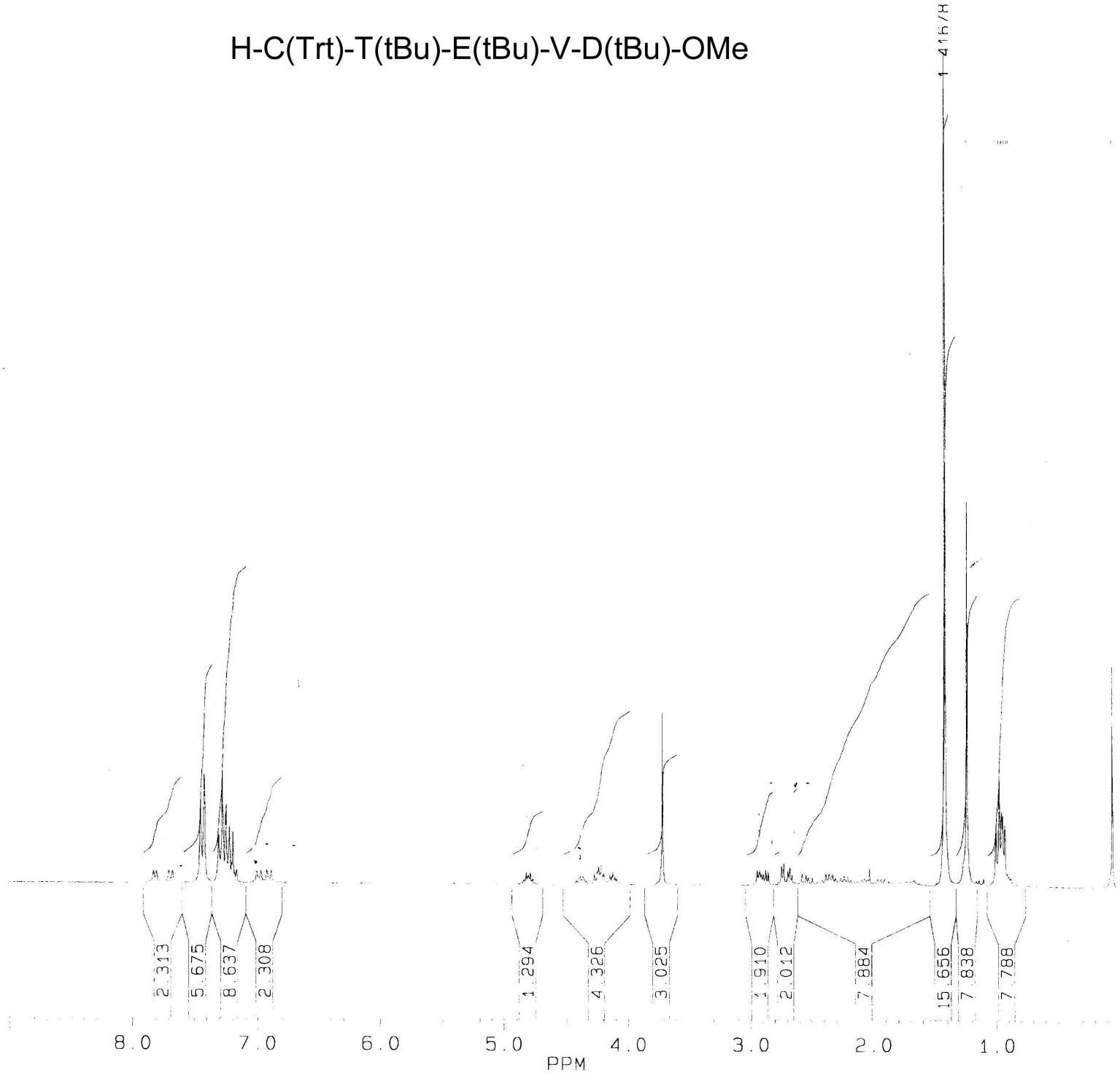
STANDARD PROTON PARAMETERS

exp4 s2pul

SAMPLE		DEC. & VT	
date	Dec 3 2001	dfrq	499.897
solvent	CDCl3	dn	H1
file	exp	dpwr	45
ACQUISITION		dof	0
sfrq	499.897	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	31412
np	30272	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	8	temp	25.0
tpwr	58	DEC2	
pw	7.1	dfrq2	0
d1	1.000	dn2	
tof	0	dpwr2	1
nt	4	dof2	0
ct	4	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	200
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	PROCESSING	
hs	nn	wtfile	
DISPLAY		proc	ft
sp	-209.9	fn	not used
wp	4967.6	math	f
vs	1146		
sc	35	werr	
wc	215	wexp	
hzmm	23.08	wbs	
is	1050.81	wnt	wft
rfl	1500.5		
rfp	0		
th	7		
ins	1.000		
nm cdc ph			



H-C(Trt)-T(tBu)-E(tBu)-V-D(tBu)-OMe



BRUKER

PENG.302
DATE 2-3-2

SF 250.133
SY 100.0
O1 4300.000
SI 16384
TD 16384
SW 3496.503
HZ/PT .427

PW 3.0
RD 1.000
AQ 2.343
RG 40
NS 96
TE 297

FW 4400
O2 3210.000
DP 63L PO

LB .100
GB 0.0
CX 20.00
CY 15.00
F1 9.000P
F2 .001P
HZ/CM 112.552
PPM/CM .450
SR 2856.30

Norbornene-C(Trt)-T(tBu)-E(tBu)-V-D(tBu)-OMe

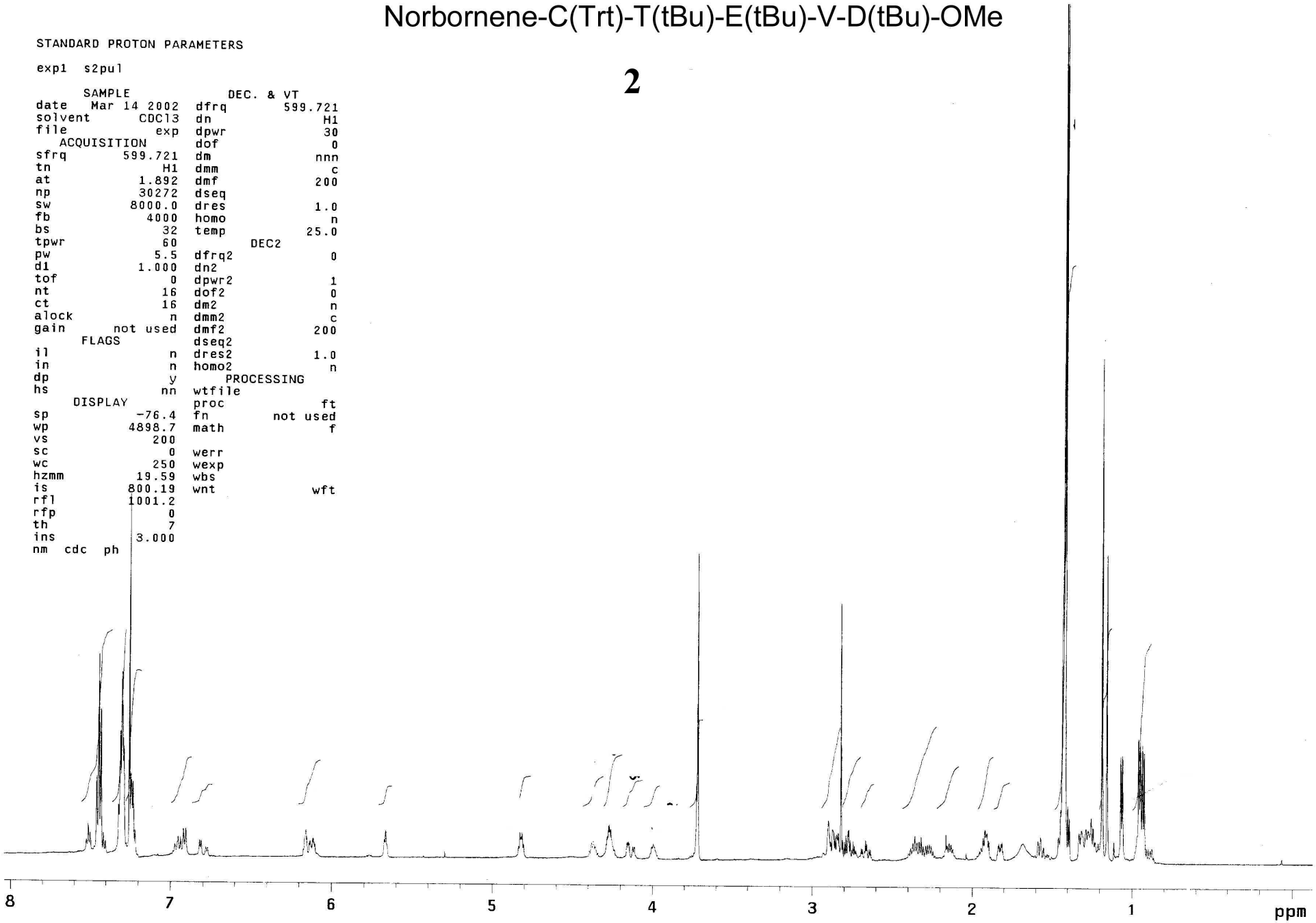
2

STANDARD PROTON PARAMETERS

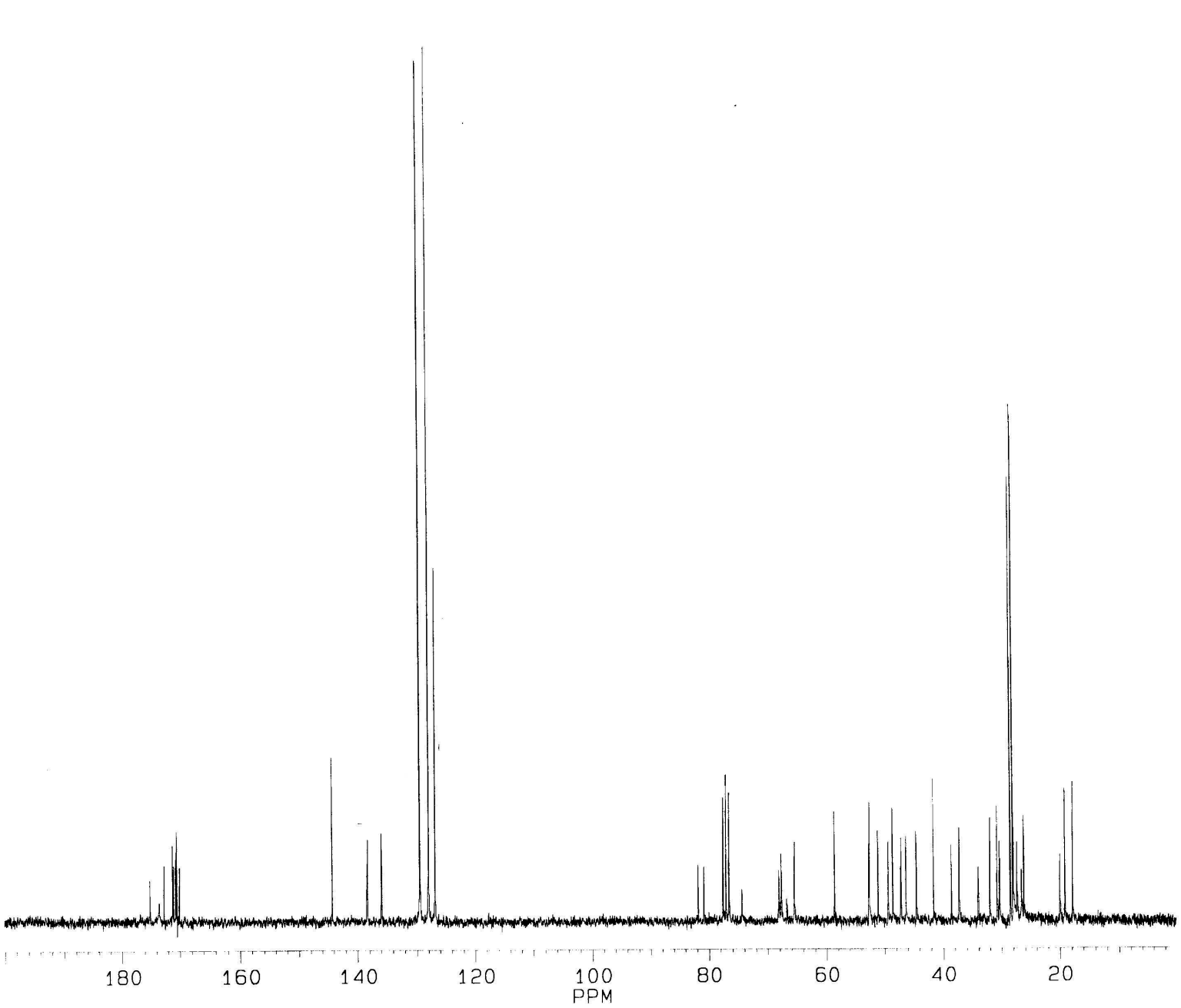
exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 14 2002	dfrq	599.721
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.721	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	32	temp	25.0
tpwr	60	DEC2	
pw	5.5	dfrq2	0
d1	1.000	dn2	
tof	0	dpwr2	1
nt	16	dof2	0
ct	16	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	200
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	PROCESSING	
hs	nn	wtfile	
DISPLAY		proc	ft
sp	-76.4	fn	not used
wp	4898.7	math	f
vs	200		
sc	0	werr	
wc	250	wexp	
hzmm	19.59	wbs	
is	800.19	wnt	wft
rfl	1001.2		
rfp	0		
th	7		
ins	3.000		

nm cdc ph



Norbornene-C(Trt)-T(tBu)-E(tBu)-V-D(tBu)-OMe



BRUKER

SAMPSON.028
DATE 20-12-1

SF 62.896
SY 62.0
O1 3218.018
SI 32768
TD 32768
SW 15625.000
HZ/PT .954

PW 5.0
RD 1.000
AQ 1.049
RG 3200
NS 3600
TE 297

FW 19600
O2 4100.000
DP 20H PO

LB 1.000
GB 0.0
CX 20.00
CY 15.00
F1 200.046P
F2 .004P
HZ/CM 629.091
PPM/CM 10.002
SR -4041.58

POLYMER 4
n=10