

Supplementary data

Electronic Supplementary Information for *Chem. Commun.*

First total synthesis of murisolin

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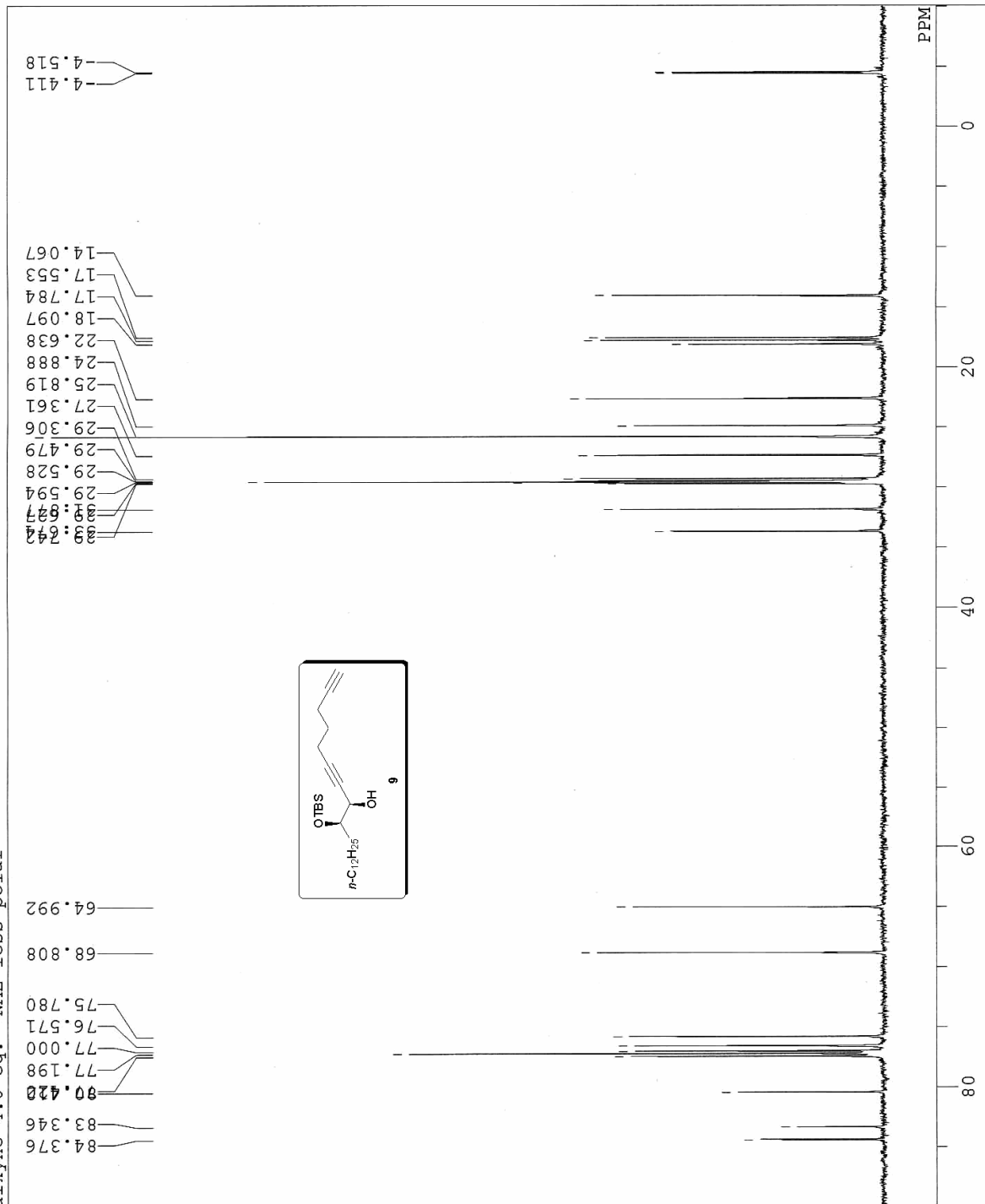
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General: Melting points are uncorrected. Optical rotations were measured using a JASCO DIP-360 digital polarimeter. ^1H NMR spectra were recorded in CDCl_3 solution with a JEOL JNM-GX500 spectrometer (500 MHz). ^{13}C NMR spectra were recorded in CDCl_3 solution with a JEOL JNM-AL300 spectrometer (75 MHz). All signals are expressed as ppm downfield from tetramethylsilane used as an internal standard (δ value). The following abbreviations are used: singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m). IR absorption spectra (FT: diffuse reflectance spectroscopy) were recorded with KBr powder with a Horiba FT-210 IR spectrometer, and only noteworthy absorptions (cm^{-1}) are listed. Mass spectra were obtained with a JEOL JMS-600H and a JEOL JMS-700 mass spectrometer.

Murisolin (**1**): mp 72.5–73.5 °C; $[\alpha]^{23}_{\text{D}} +20.7$ (*c* 0.39, MeOH), $[\alpha]^{22}_{\text{D}} +21.5$ (*c* 0.36, CHCl_3); IR (KBr): 3435, 3419, 2916, 2848, 1751, 1470, 1319, 1201, 1119, 1074, 1028, 960, 847, 721 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.88 (t, 3H, $J = 7.0$ Hz), 1.25–1.57 (m, 42H), 1.44 (d, 3H, $J = 6.7$ Hz), 1.63–1.71 (m, 2H), 1.94–2.02 (m, 2H), 2.40 (dd, 1H, $J = 15.3, 8.6$ Hz), 2.47–2.58 (m, 3H), 2.51–2.55 (m, 1H), 3.41 (td, 2H, $J = 6.7, 4.9$ Hz), 3.80 (q, 2H, $J = 6.7$ Hz), 3.82–3.87 (m, 1H), 5.07 (qd, 1H, $J = 6.7, 1.2$ Hz), 7.20 (d, 1H, $J = 1.2$ Hz); ^{13}C NMR (75 MHz, CDCl_3) δ 14.1, 19.1, 22.7, 25.5 (2C), 25.6, 28.7 (2C), 29.3, 29.4, 29.5 (4C), 29.57, 29.62 (4C), 29.65, 29.69, 31.9, 33.3, 33.4 (2C), 37.4, 70.0, 74.0 (2C), 78.0, 82.6 (2C), 131.2, 151.8, 174.6; MS (FAB): m/z : 581.5 ($[\text{M}+\text{H}]^+$). HRMS (FAB): calcd for $\text{C}_{35}\text{H}_{65}\text{O}_6$ ($[\text{M}+\text{H}]^+$): 581.4781. Found: 581.4786.

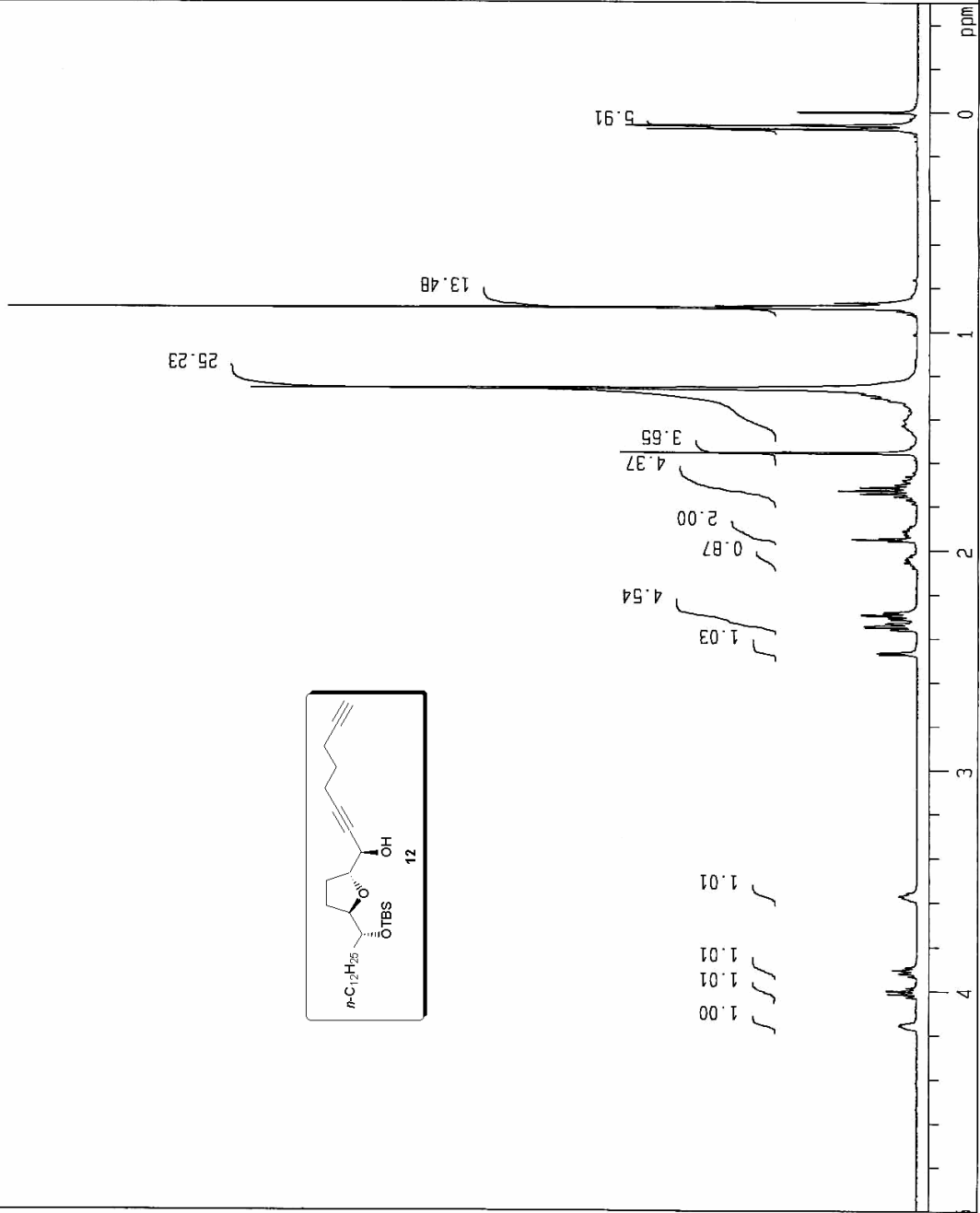
alkyne 4.0 eq. -NME less polar



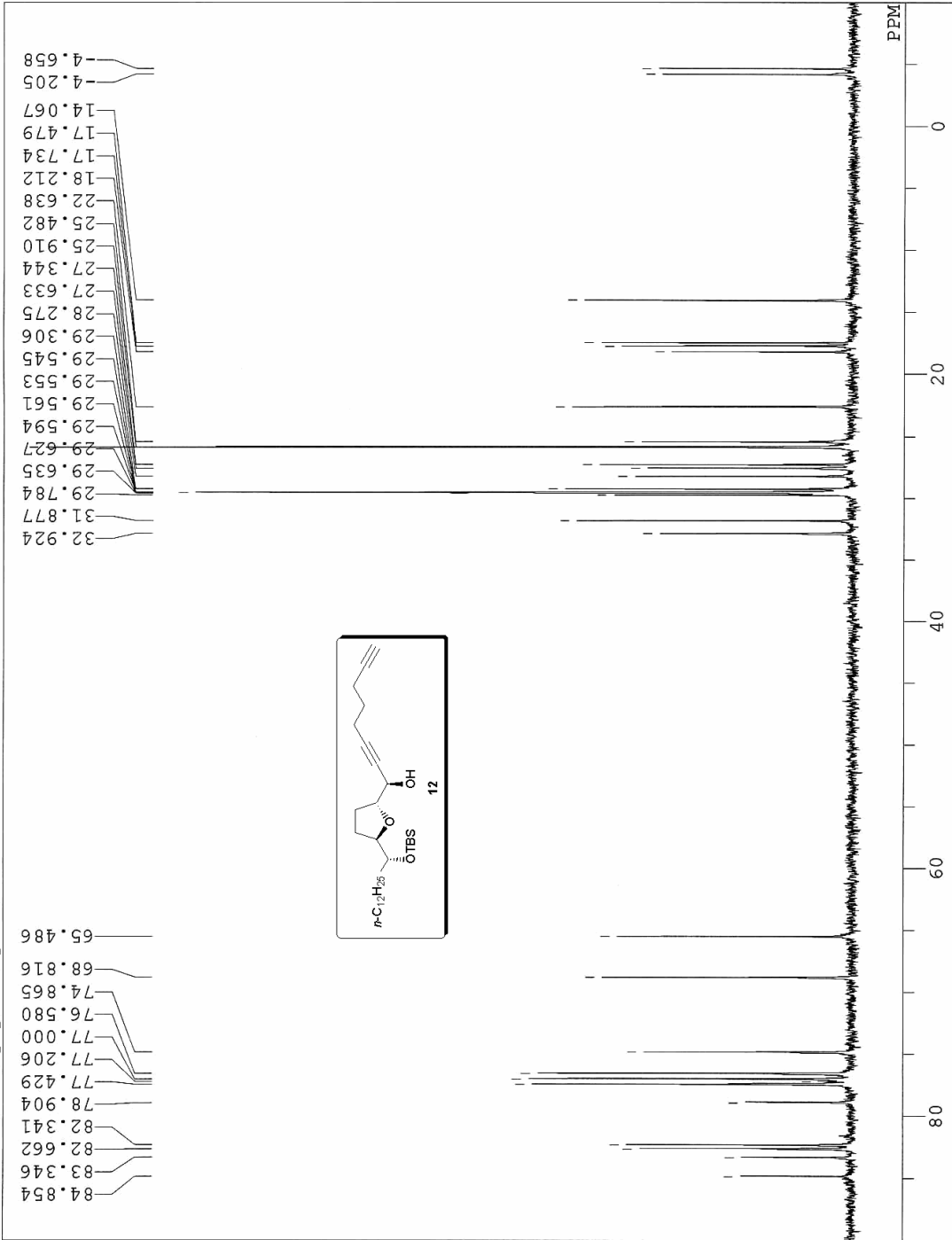
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OBFIN 1840.0 Hz
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SCANS 500
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.3 us
IRNUC 1H
CTEMP 23.7 c
SIVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 25

1H Line

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INVTL : 100.0 usec
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DUMMY : 1 times
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RESOL : 0.61 Hz
PW1 : 2.63 usec
1H :
OBNUC : 500.00 MHz
OBFRQ : 162167.63 Hz
OBSET : 23
RGAIN :
SCANS : 16 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 26.6 C



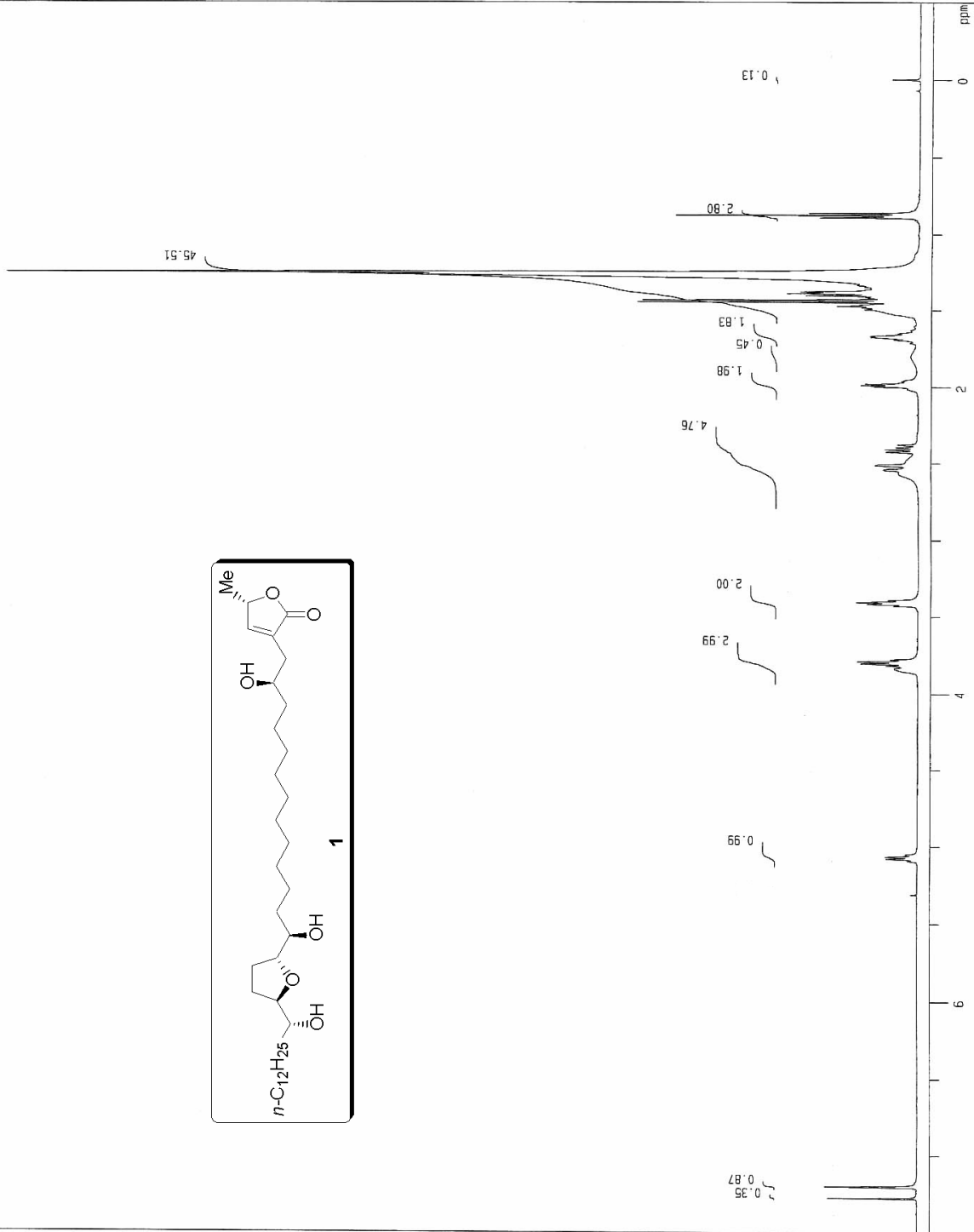
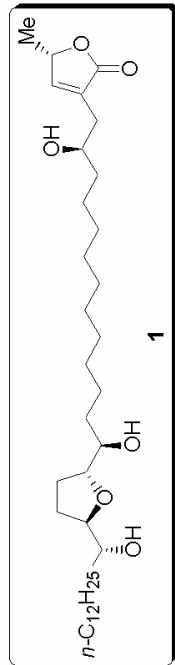
HT-255 mono-alkynyl compound



DFILE F:\HT-255 THF-ALDEHY.
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 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 300
 ACQTM 1.606 sec
 PD 1.394 sec
 PWL 4.3 us
 IRNUC 1H
 CTEMP 23.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 24

1H Line

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DEADT : 58.7 usec
INTEVL : 100.0 usec
TIMES : 16 times
DUMMY : 1 times
PR : 5.3616 SEC
ACQTM : 1638.3999 msec
PREDL : 0.01000 msec
INVTM : 1000.0000 msec
RESOL : 0.61 Hz
PMT : 1H 500.00 MHz
ORNUC : 162167.63 Hz
ORSET : 17
SCANS : 15 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 14.0 C



HT-266 murisolin

F:\HT-266 MURISOLIN\
HT-266 murisolin
Sat Jun 28 04:31:55
13C
EXMOD BCM

OBFRO 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 5000
ACQTM 1.606 sec
PD 1.394 sec
PWL 4.3 us
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 24

