**Electronic supplementary materials** *Mendeleev Commun.*, 2015, **25**, 93–95

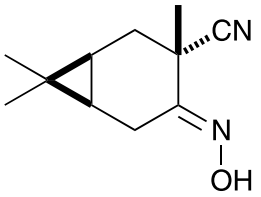
# Syntheses of α-cyano substituted oximes from terpenic hydrocarbons *via* nitroso chlorides: X-ray structures of 3-cyanocaran-4-one oxime, 2-cyanopinan-3-one oxime and 1-cyano-*p*-menth-8-en-2-one oxime

**Sergey N. Bizyaev, Yurii V. Gatilov and Alexey V. Tkachev**

Copies of $^{1}$H and $^{13}$C NMR, IR and full mass spectra (EI, 70 ev) for compounds obtained are presented below.

# 1H NMR

(1S,3S,6R)-3-cyano-caran-4-one E-oxime in CCl4˜CDCl3 2:1 v/v (BS-875-8)

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PROCNO                1

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PULPROG              zg
TD                16384
SOLVENT           CDCl3
NS                   64
DS                    2
SWH           10964.912 Hz
FIDRES         0.669245 Hz
AQ            0.7471604 sec
RG                   16
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TE                300.0 K
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PL1               -6.00 dB
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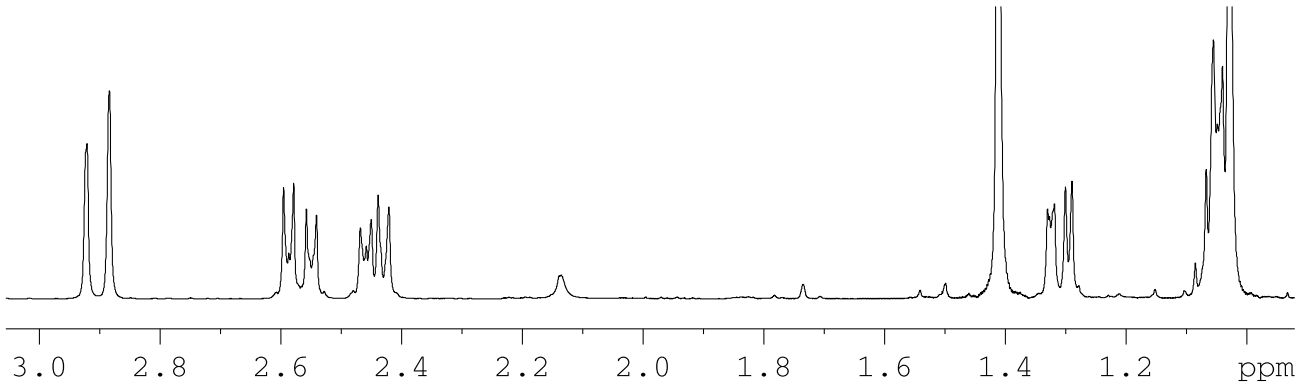
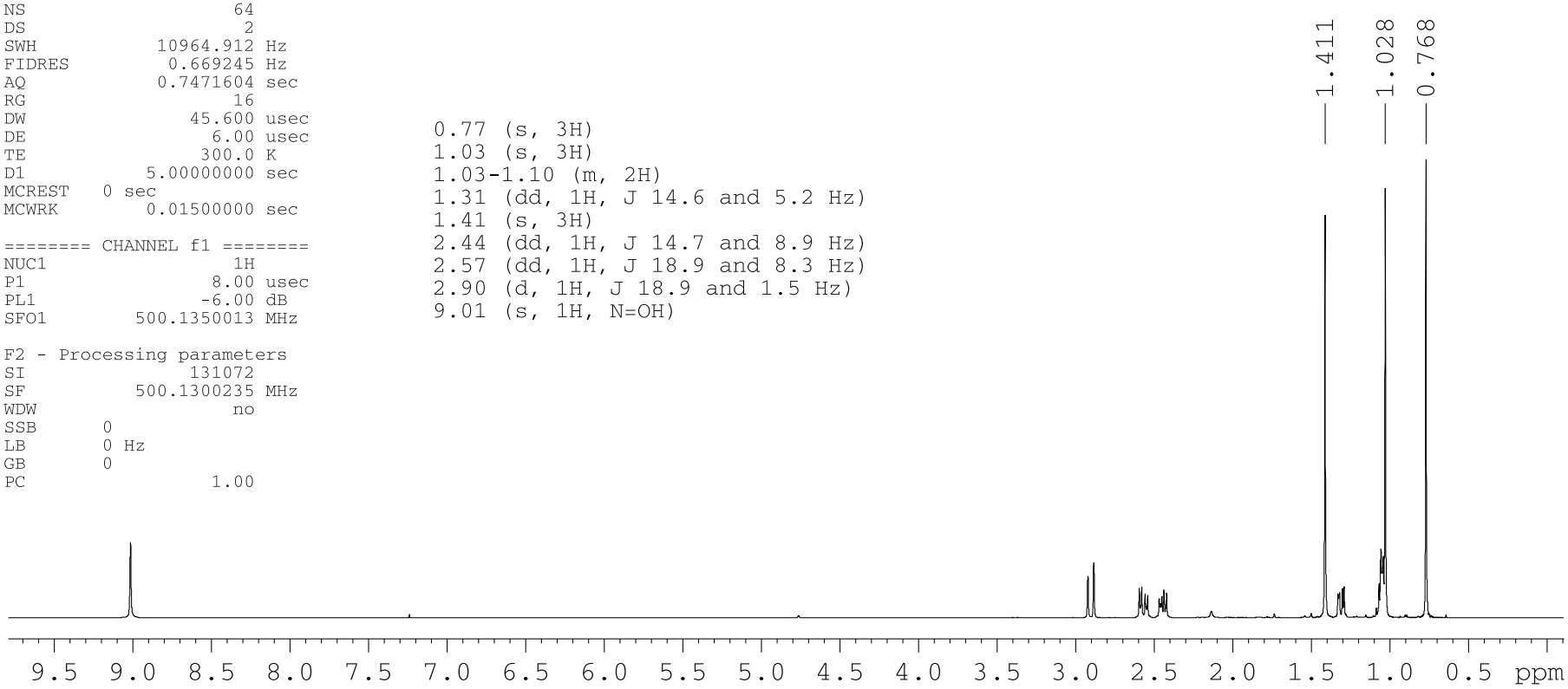
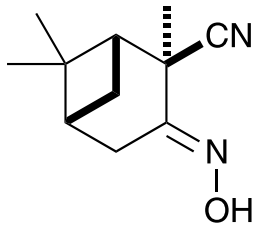



0.77 (s, 3H)
1.03 (s, 3H)
1.03-1.10 (m, 2H)
1.31 (dd, 1H, J 14.6 and 5.2 Hz)
1.41 (s, 3H)
2.44 (dd, 1H, J 14.7 and 8.9 Hz)
2.57 (dd, 1H, J 18.9 and 8.3 Hz)
2.90 (d, 1H, J 18.9 and 1.5 Hz)
9.01 (s, 1H, N=OH)



# 1H NMR

(1R,2R,5R,E)-2-cyano-pinan-3-one E-oxime in CDCl3 (BS-739-1)

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TD                65536
SOLVENT           CDCl3
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DS                    2
SWH            8012.820 Hz
FIDRES        0.122266 Hz
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RG                  128
DW               62.400 usec
DE                6.50 usec
TE                673.2 K
D1           5.00000000 sec
TD0               11111

======== CHANNEL f1 ========
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PL1       0 dB
PL1W         9.52005005 W
SFO1        400.1338012 MHz

F2 - Processing parameters
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PC                  1.00
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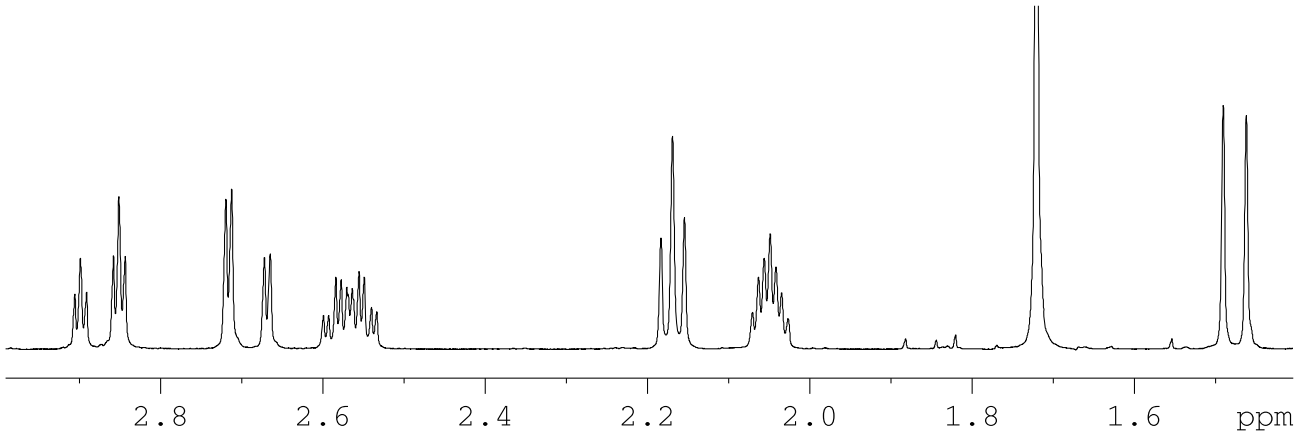


0.90 (s, 3H)
1.30 (s, 3H)
1.48 (d, 1H, 11.4 Hz)
1.72 (s, 3H)
2.05 (dddd, 1H, J 6.0, 6.0, 3.1 and 2.6 Hz)
2.17 (dd, 1H, J 6.0 and 6.0 Hz)
2.57 (dddd, J 11.4, 6.0, 6.0 and 3.1 Hz)
2.69 (dd, 1H, J 19.0 and 2.6 Hz)
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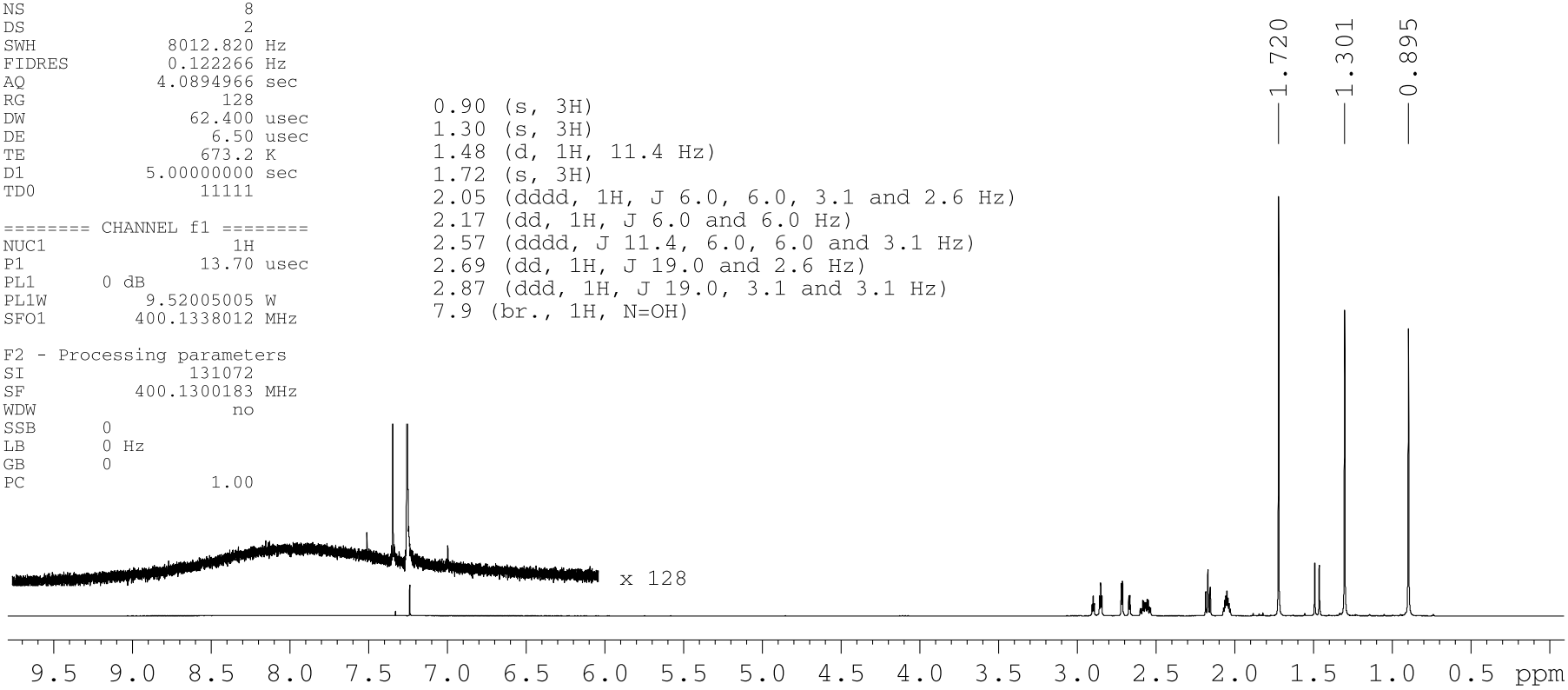
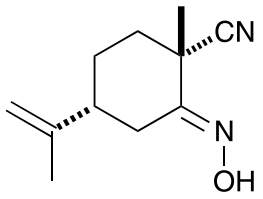
7.9 (br., 1H, N=OH)



# 1H NMR

(1S,4R)-1-cyano-p-menth-7-en-2-one E-oxime in DMSO-d6 (BS-879-4)

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PULPROG              zg
TD                32768
SOLVENT            DMSO
NS                   128
DS                     0
SWH           10964.912 Hz
FIDRES         0.334623 Hz
AQ            1.4942708 sec
RG                     9
DW               45.600 usec
DE                6.00 usec
TE                300.0 K
D1           5.00000000 sec
MCREST   0 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.00 usec
PL1               -6.00 dB
SFO1        500.1350013 MHz

F2 - Processing parameters
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PC                  1.00
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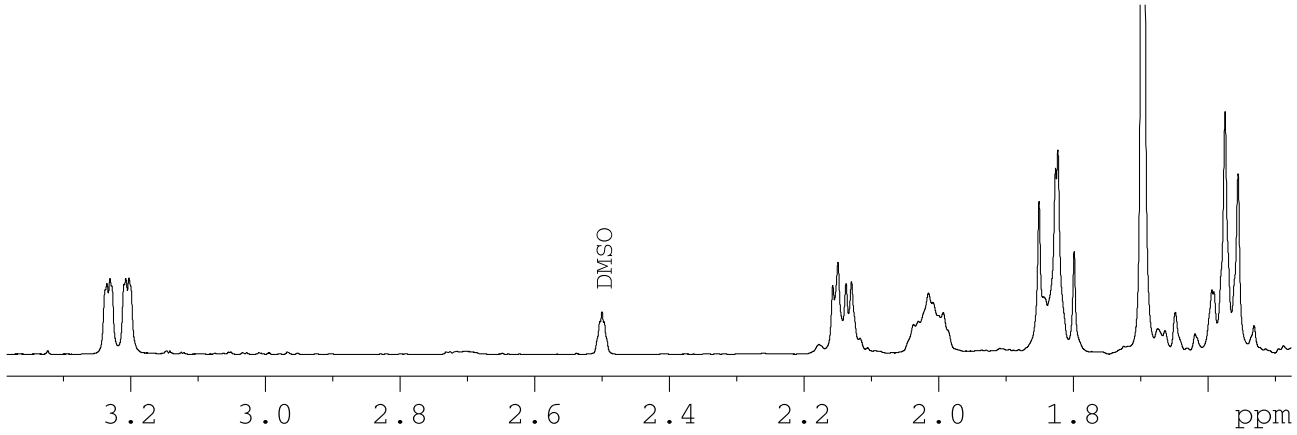
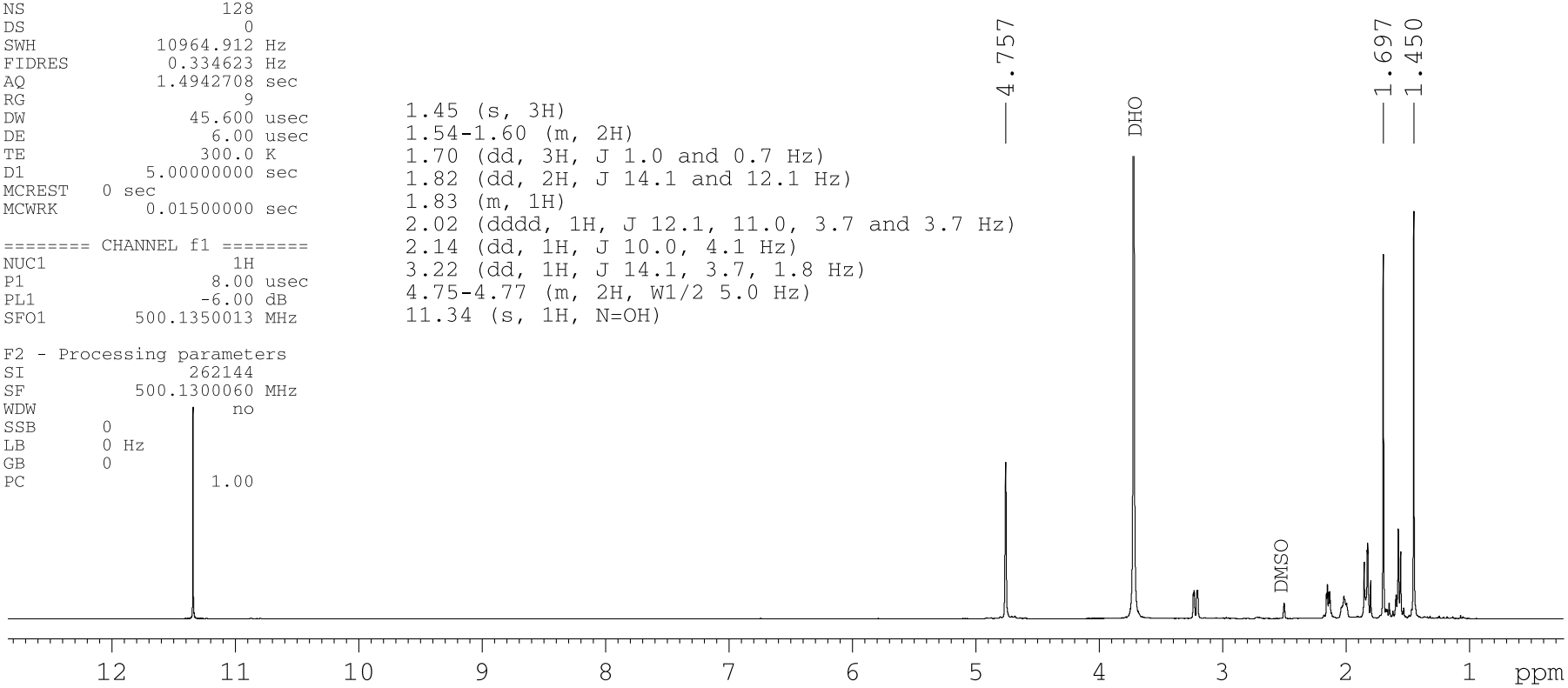
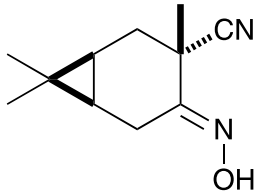



1.45 (s, 3H)
1.54-1.60 (m, 2H)
1.70 (dd, 3H, J 1.0 and 0.7 Hz)
1.82 (dd, 2H, J 14.1 and 12.1 Hz)
1.83 (m, 1H)
2.02 (dddd, 1H, J 12.1, 11.0, 3.7 and 3.7 Hz)
2.14 (dd, 1H, J 10.0, 4.1 Hz)
3.22 (dd, 1H, J 14.1, 3.7, 1.8 Hz)
4.75-4.77 (m, 2H, W1/2 5.0 Hz)
11.34 (s, 1H, N=OH)



# 13C NMR, J-modulation

(1S,3S,6R)-3-cyano-caran-4-one E-oxime in CCl4 : CDCl3 2:1 v/v (BS-875-8)

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PROCNO                1

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INSTRUM           spect
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PULPROG            jmod
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ            1.0912244 sec
RG               5792.6
DW              16.650 usec
DE               6.00 usec
TE              300.0 K
CNST2       145.0000000
CNST11        1.0000000
D1            4.00000000 sec
d20           0.00689655 sec
DELTA         0.00000955 sec
MCREST   0 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                7.50 usec
p2               15.00 usec
PL1              -1.00 dB
SFO1        125.7715724 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            84.00 usec
PL2             120.00 dB
PL12             17.00 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                131072
SF          125.7578092 MHz
WDW                 EM
SSB      0
LB                2.00 Hz
GB       0
PC                1.00
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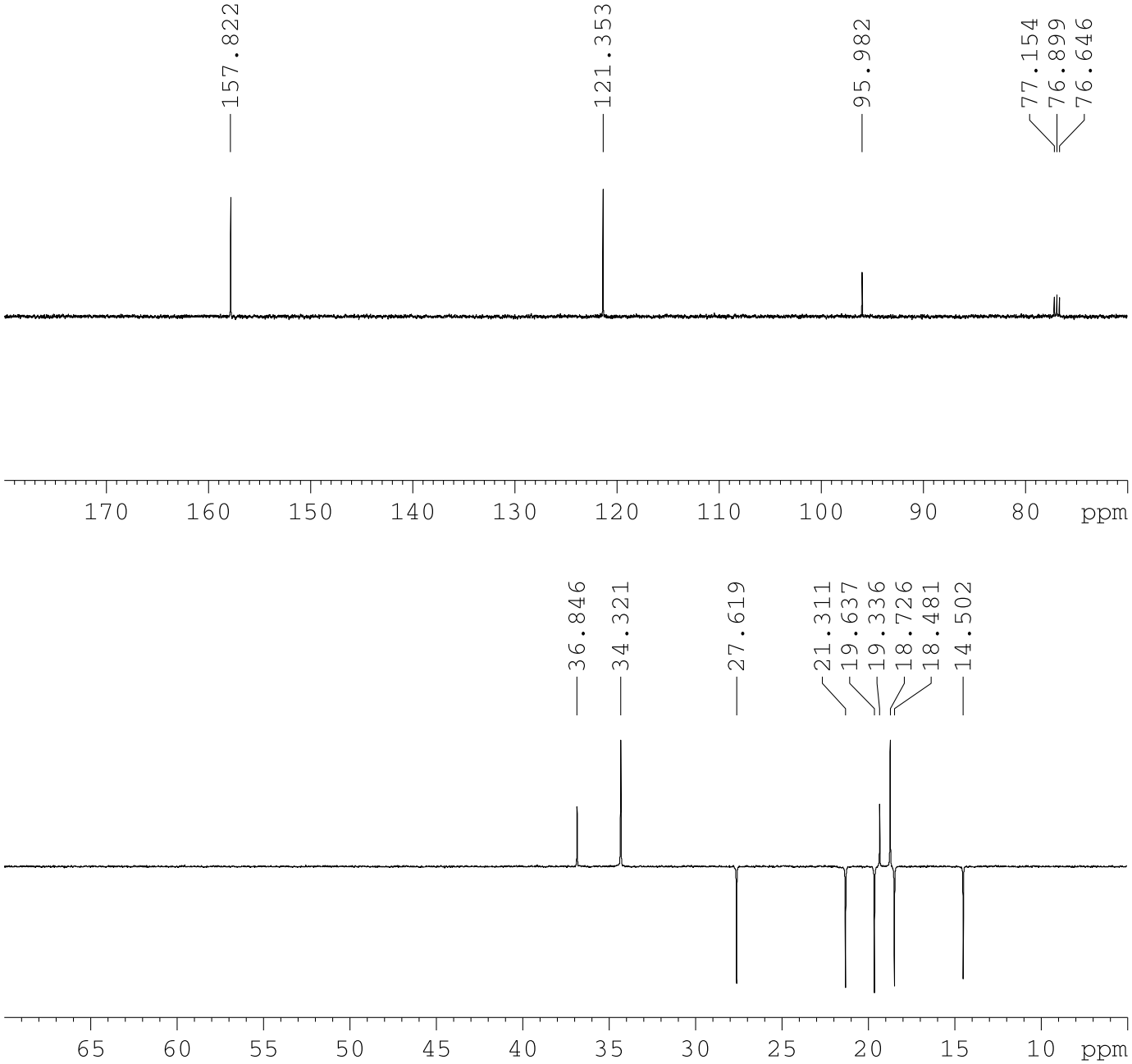
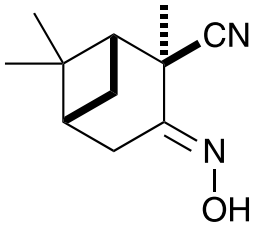


# 13C NMR, J-modulation

(1R,2R,5R,E)-2-cyano-pinan-3-one E-oxime in CDCl3 (BS-739-1)

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PROCNO                1

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PULPROG            jmod
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE               673.2 K
CNST2        145.0000000
CNST11         1.0000000
D1             5.00000000 sec
D20            0.00689655 sec
TD0                11111

======== CHANNEL f1 ========
NUC1                13C
P1                 9.60 usec
P2                19.20 usec
PL1       0 dB
PL1W        36.42235184 W
SFO1       100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              86.00 usec
PL2       0 dB
PL12              16.00 dB
PL2W          9.52005005 W
PL12W         0.23913284 W
SFO2        400.1316005 MHz

F2 - Processing parameters
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SF           100.6127846 MHz
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GB        0
PC                 1.40
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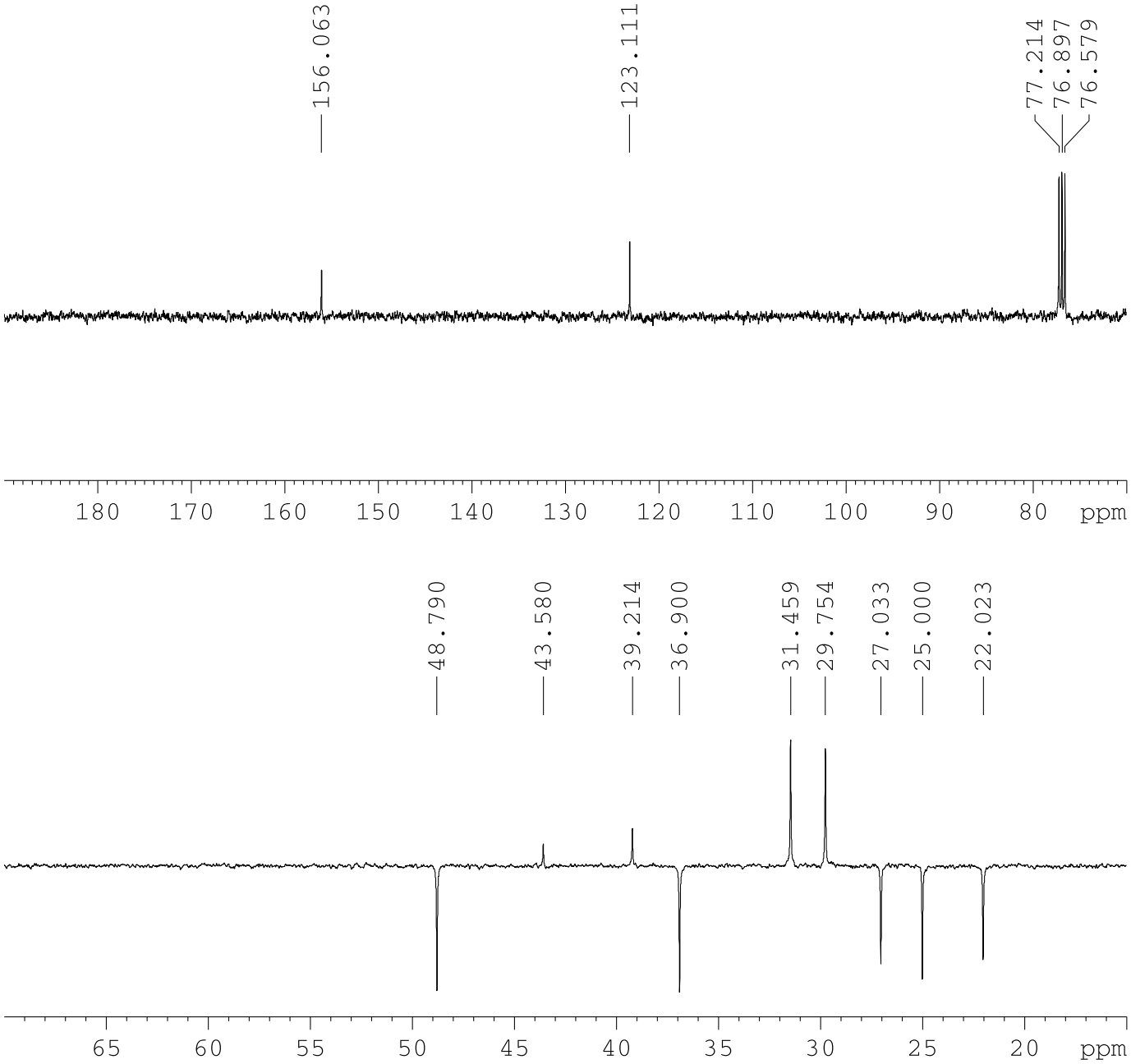
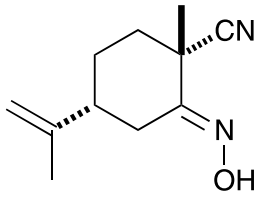


# 13C NMR, J-modulation

(1S,4R)-1-cyano-p-menth-7-en-2-one E-oxime in DMSO-d6 (BS-879-4)

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Current Data Parameters
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PROCNO                1

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PULPROG            jmod
TD                65536
SOLVENT           CDCl3
NS                  592
DS                    2
SWH           28985.508 Hz
FIDRES         0.442284 Hz
AQ            1.1305460 sec
RG               7298.2
DW              17.250 usec
DE               6.00 usec
TE              300.0 K
CNST2       160.0000000
CNST11        1.0000000
D1            4.00000000 sec
d20           0.00625000 sec
DELTA         0.00000955 sec
MCREST   0 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 7.50 usec
p2                15.00 usec
PL1               -1.00 dB
SFO1        125.7720754 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            84.00 usec
PL2             120.00 dB
PL12             17.00 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                65536
SF          125.7577969 MHz
WDW                 EM
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LB                1.00 Hz
GB        0
PC                1.00
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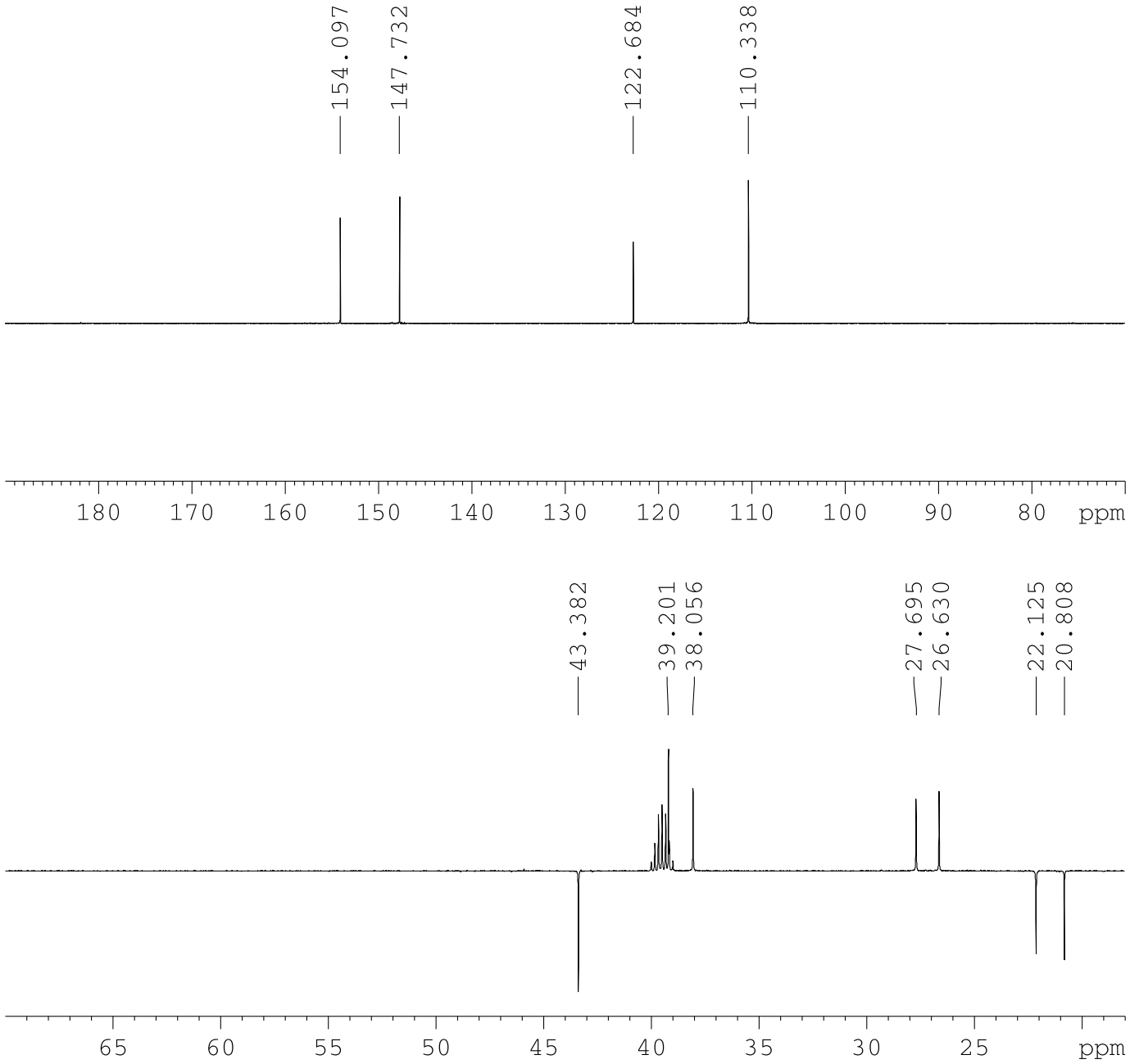
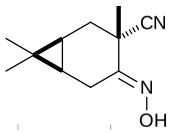
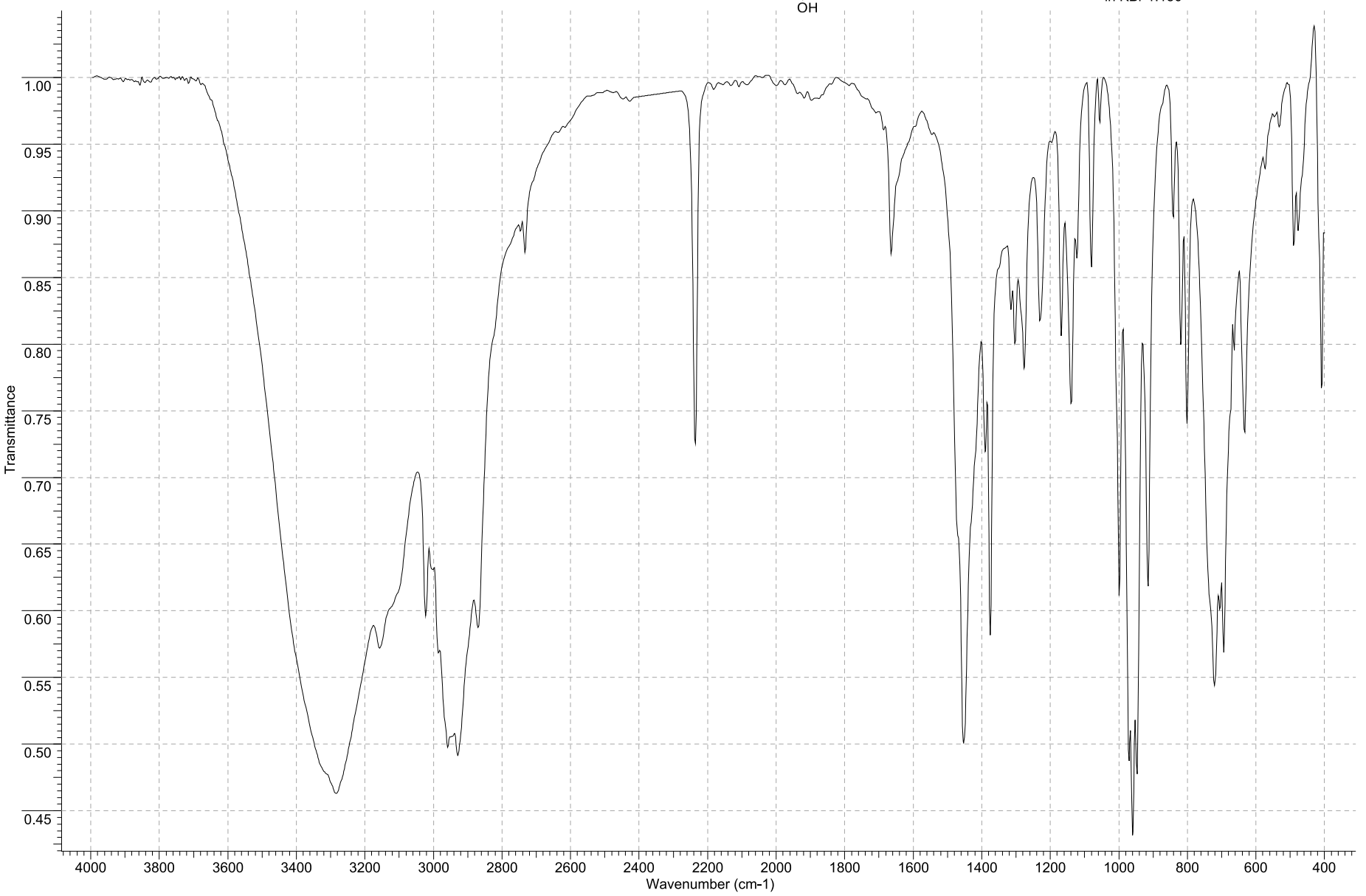
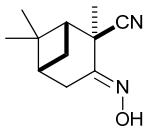


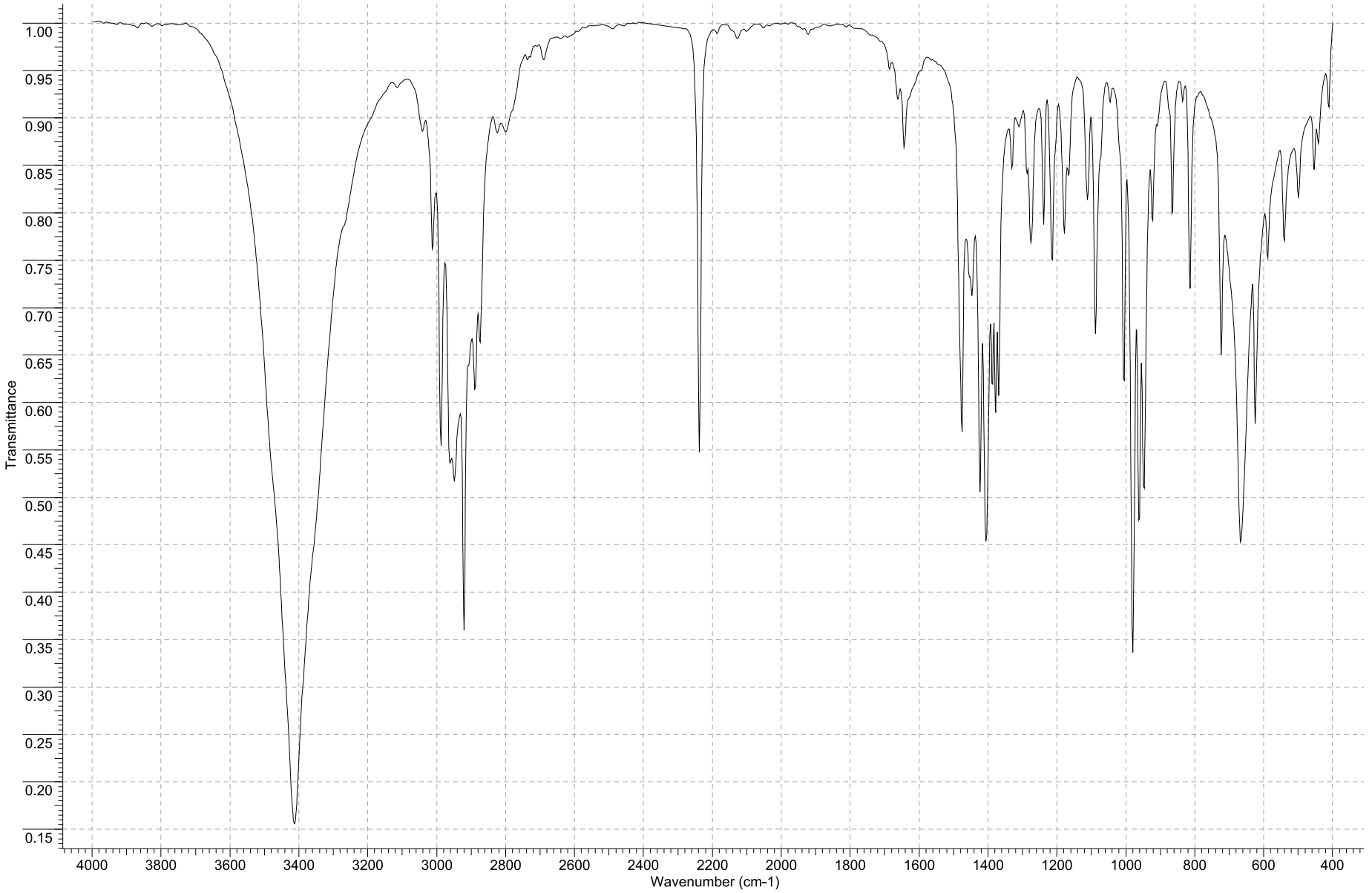
(1*S*,3*S*,6*R*,*E*)-4-(hydroxyimino)-3,7,7-trimethylbicyclo[4.1.0]heptane-3-carbonitrile
(1*S*,3*S*,6*R*)-3-cyano-caran-4-one *E*-oxime



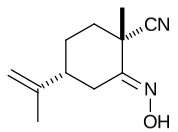



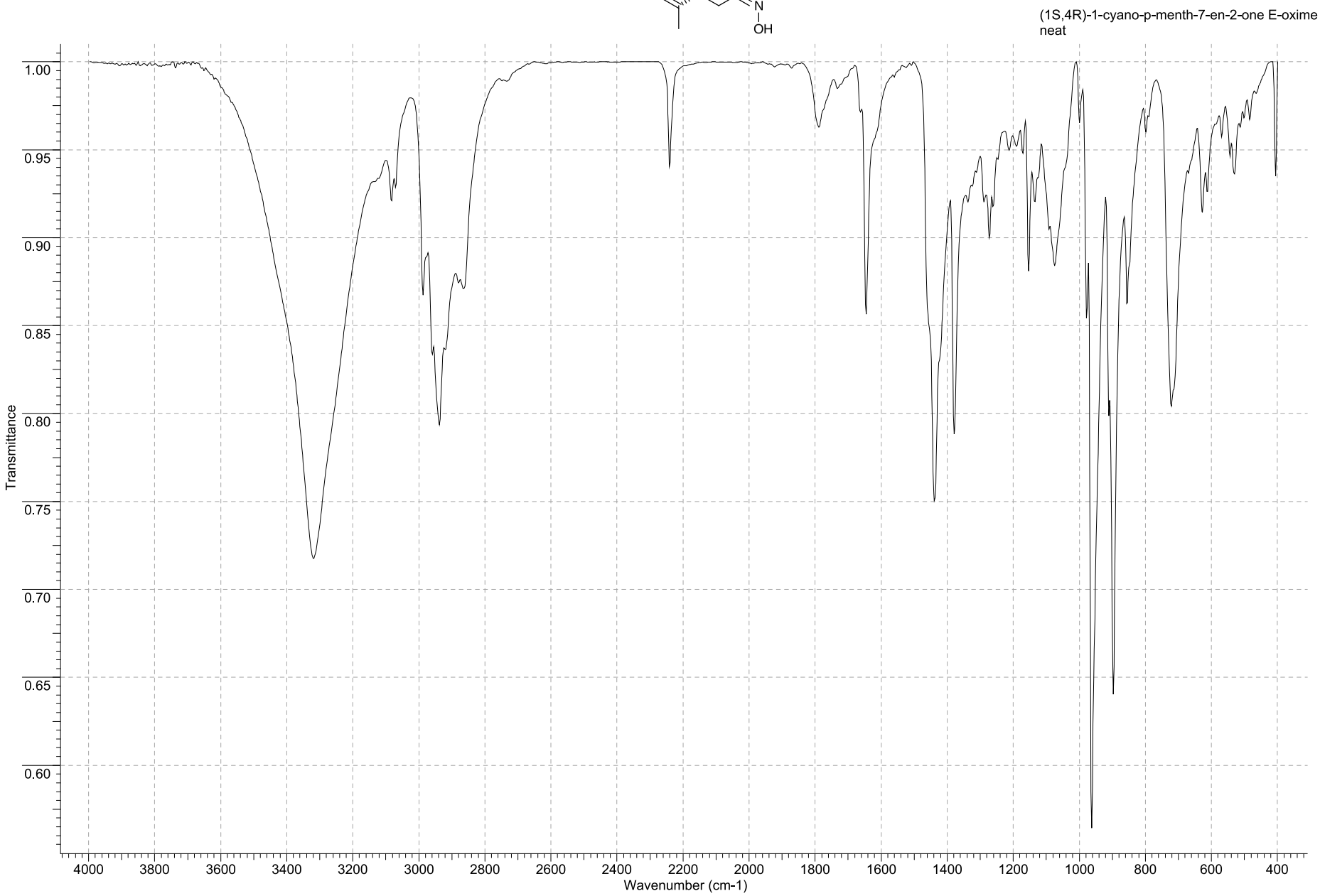
(1*R*,2*R*,5*R*,*E*)-3-(hydroxyimino)-2,6,6-trimethylbicyclo[3.1.1]heptane-2-carbonitrile
(1*R*,2*R*,5*R*,*E*)-2-cyano-pinan-3-one *E*-oxime $C_{11}H_{16}N_2O$ 70 eV



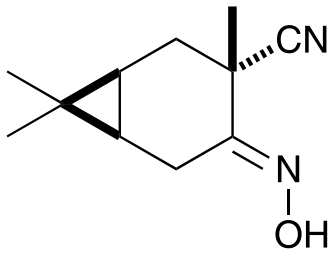



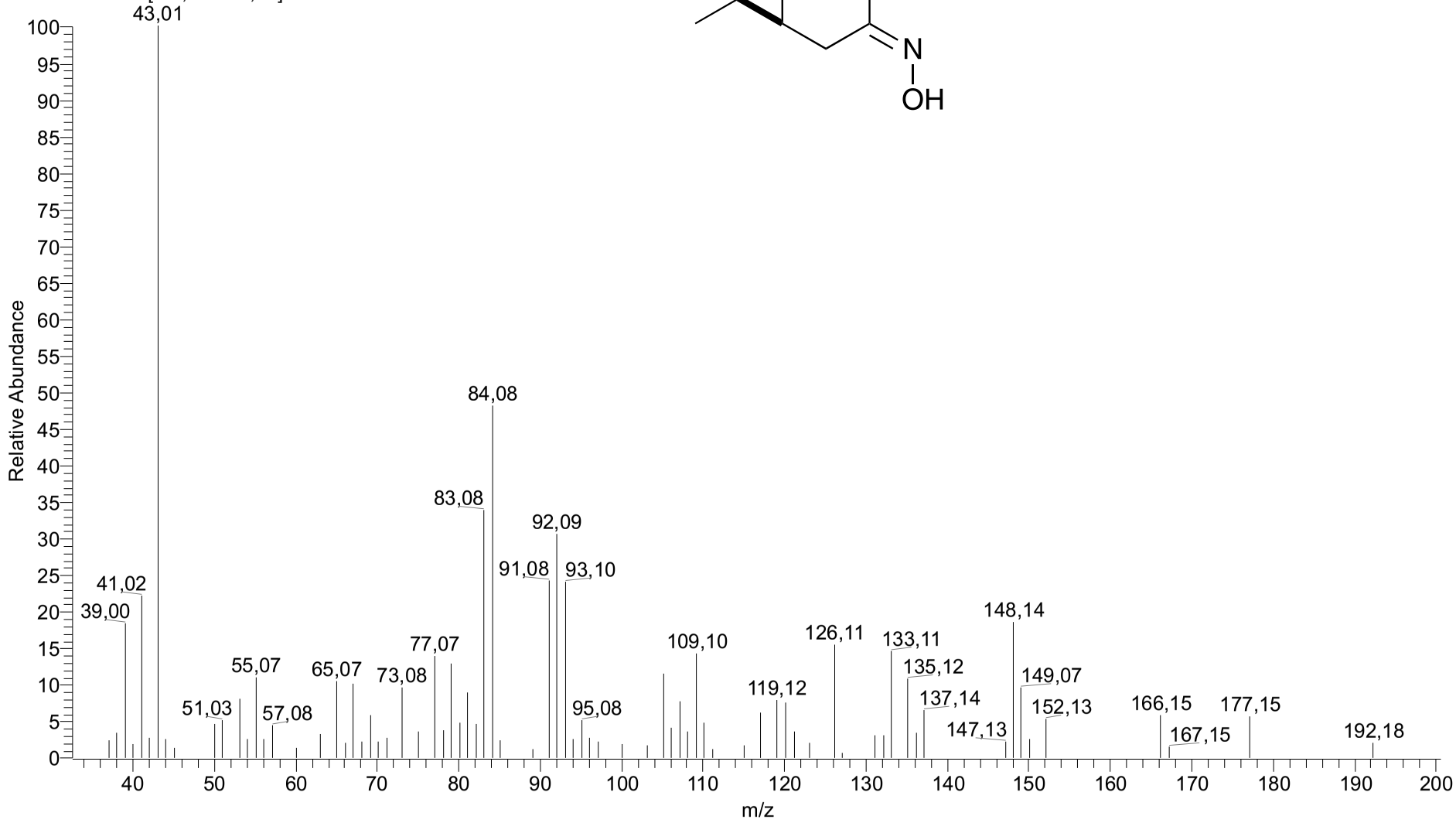
(1*S*,4*R*,*E*)-2-(hydroxyimino)-1-methyl-4-(prop-1-en-2-yl)cyclohexanecarbonitrile
(1*S*,4*R*)-1-cyano-p-menth-7-en-2-one *E*-oxime



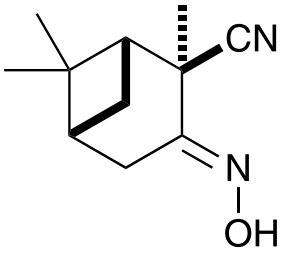



(1*S*,3*S*,6*R*)-3-cyano-caran-4-one *E*-oxime $C_{11}H_{16}N_2O$ 70 eV



(1*R*,2*R*,5*R*,*E*)-2-cyano-pinan-3-one *E*-oxime $C_{11}H_{16}N_2O$ 70 eV

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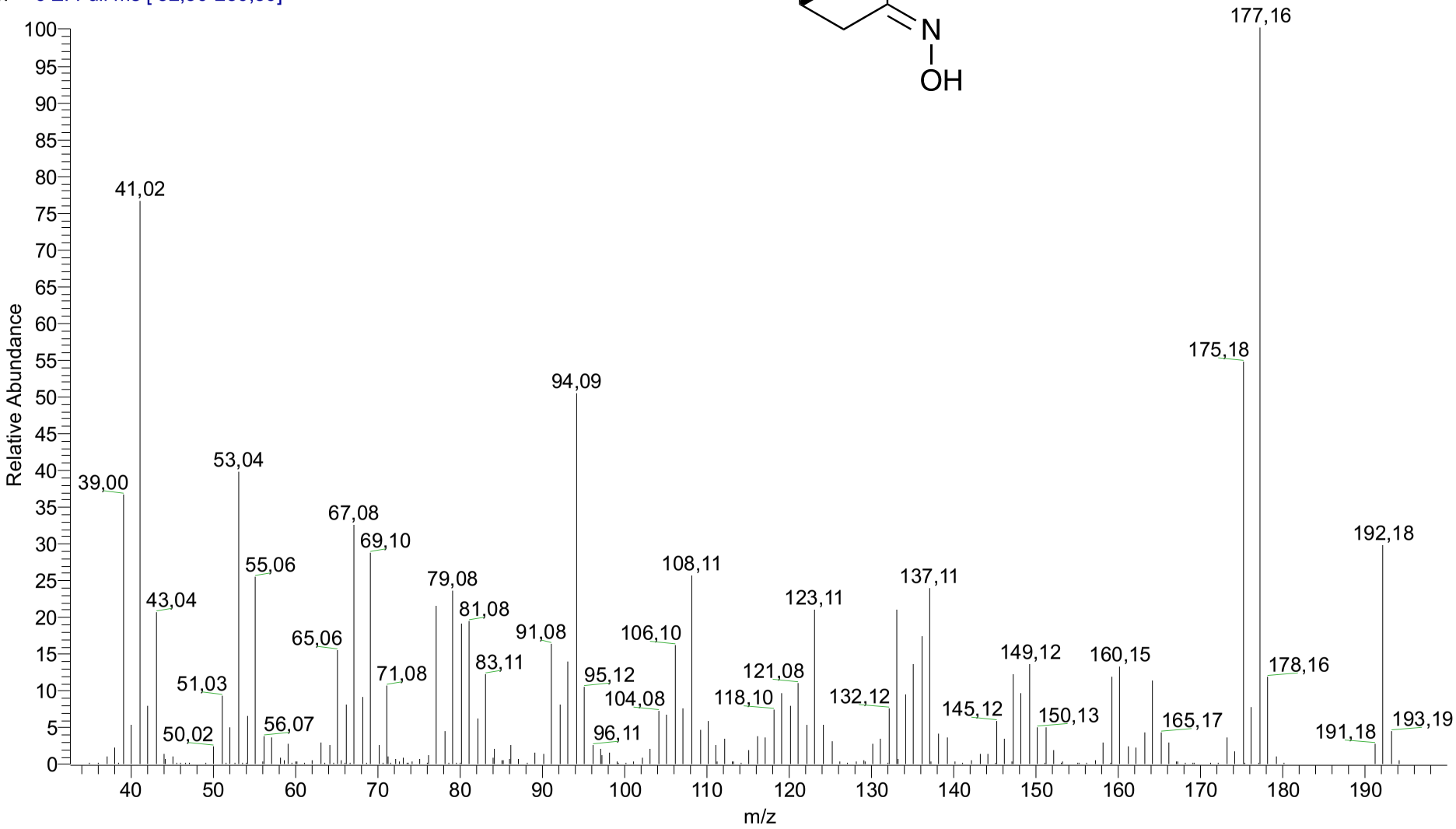

(1*S*,4*R*)-1-cyano-p-menth-7-en-2-one *E*-oxime $C_{11}H_{16}N_2O$ 70 eV