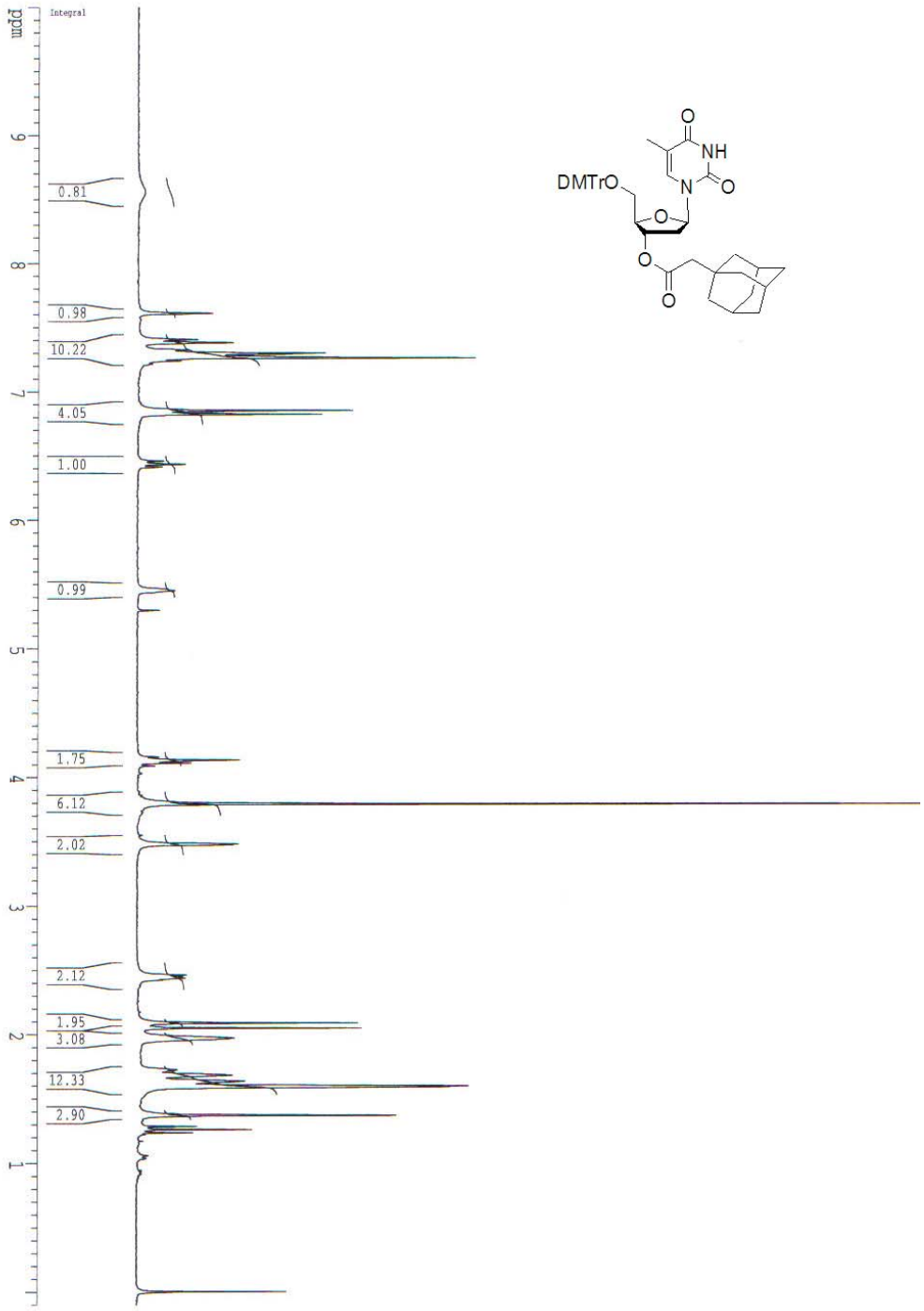
Simple and Efficient Solution Phase Synthesis of Oligonucleotides using Extractive Work-Up

Martijn C. de Koning, Amar B.T. Ghisaidoobe, Howard I. Duynstee, Paul B.W. Ten Kortenaar, Dmitri V. Filippov and Gijs A. van der Marel

Leiden Institute of Chemistry, Gorlaeus Laboratories, P.O.Box 9502, 2300 RA, Leiden, The Netherlands, and Diosynth B.V., P.O.Box 20, 5340 BH, Oss, The Netherlands

SUPPORTING INFORMATION

SPECTRA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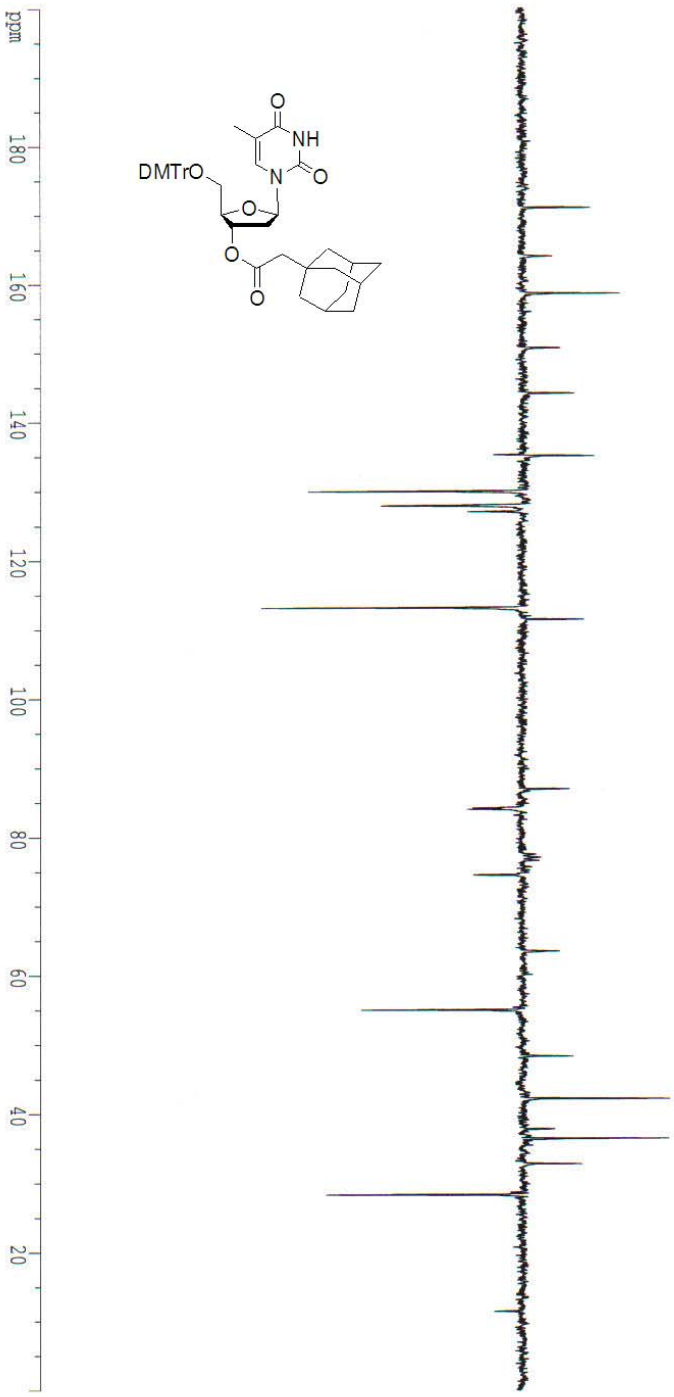
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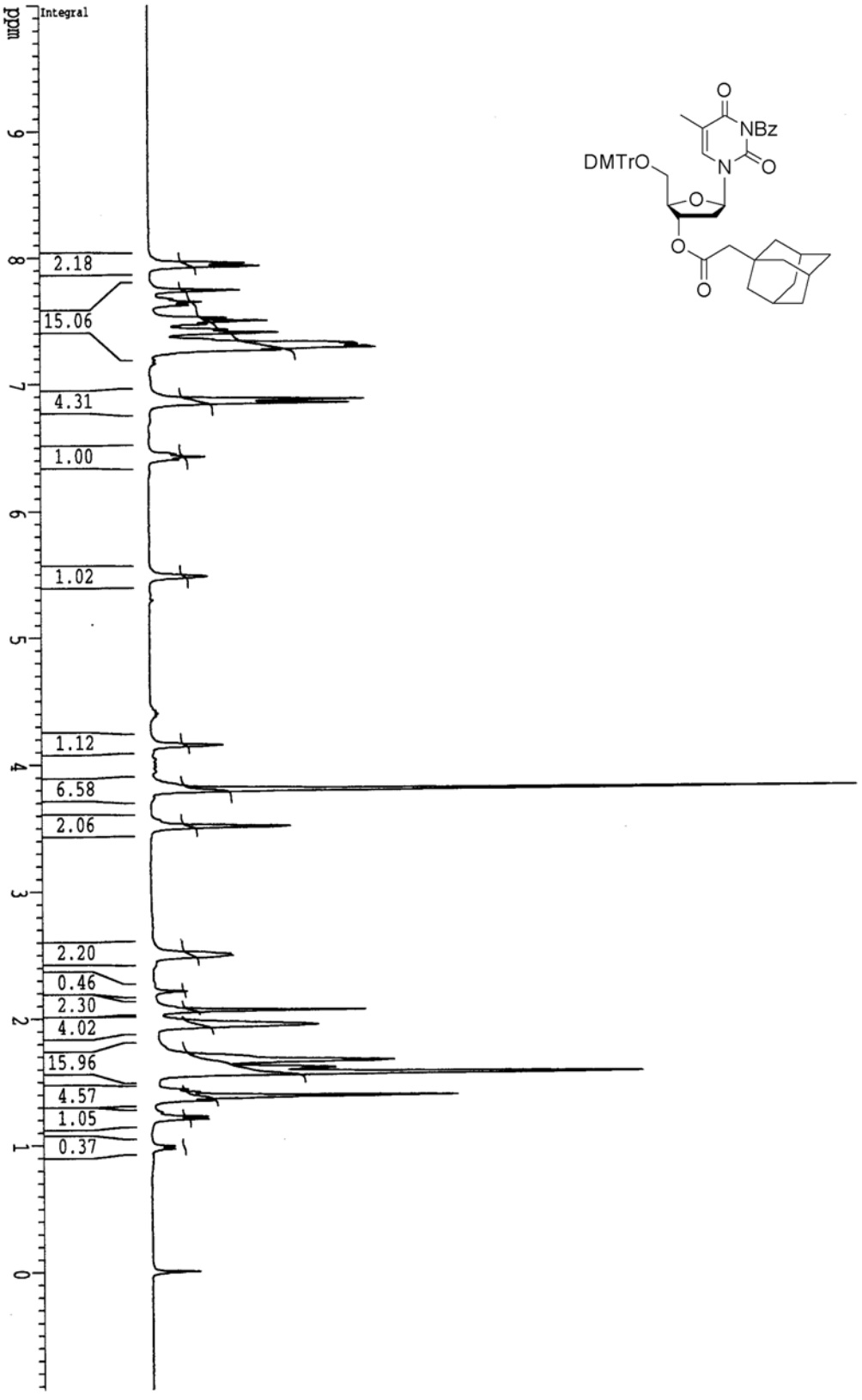
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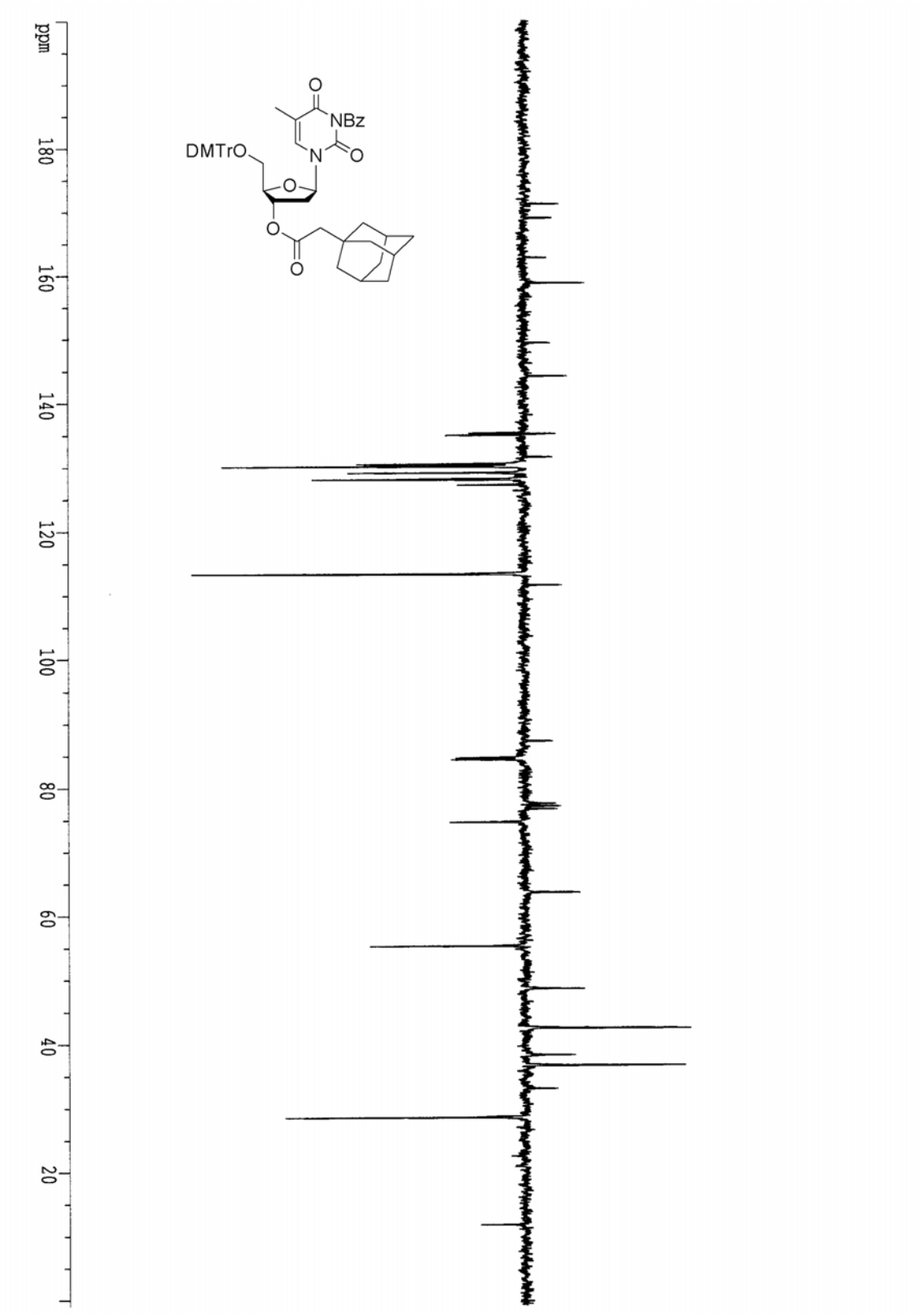
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DMTrO
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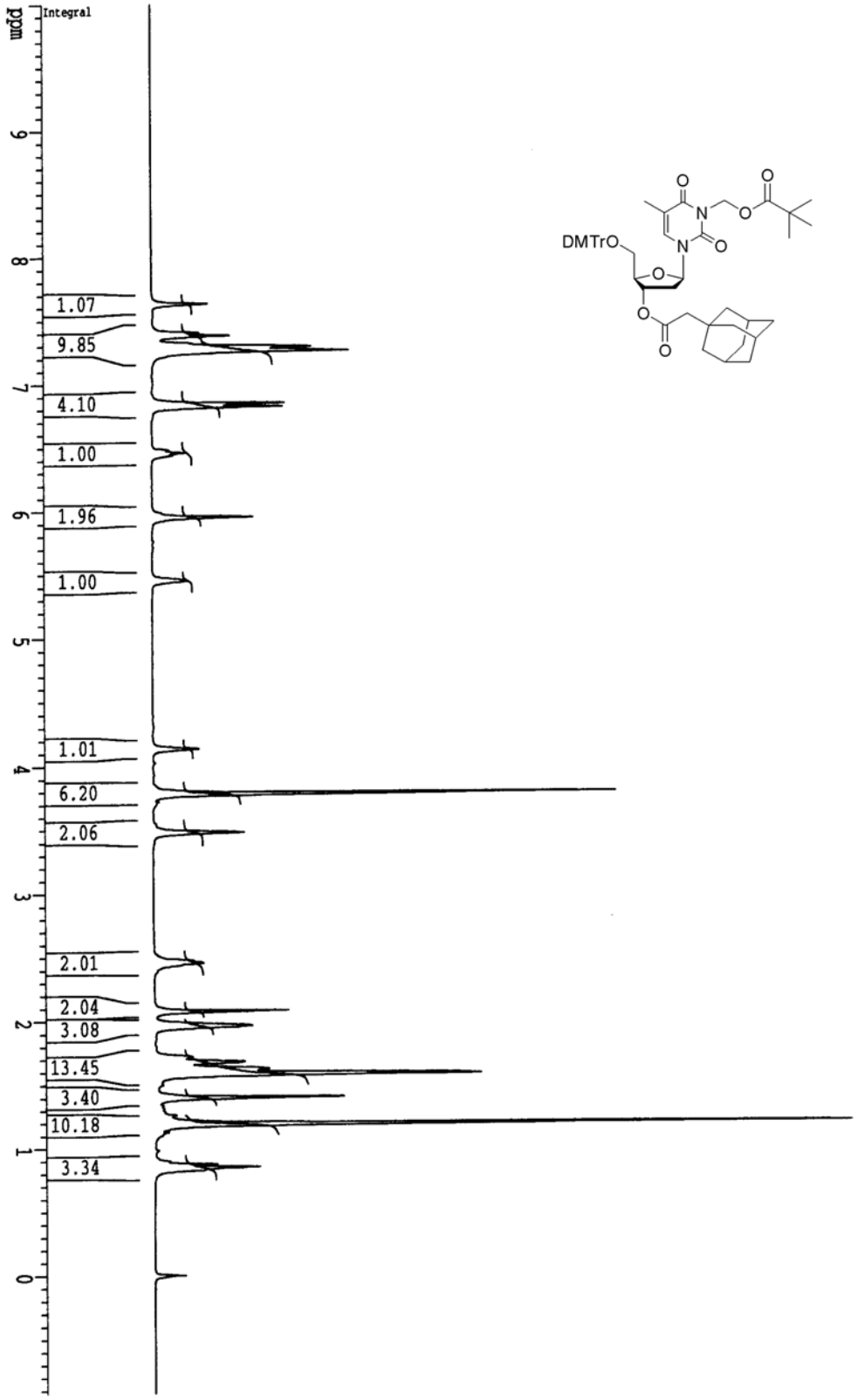
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DMTrO
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N
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O
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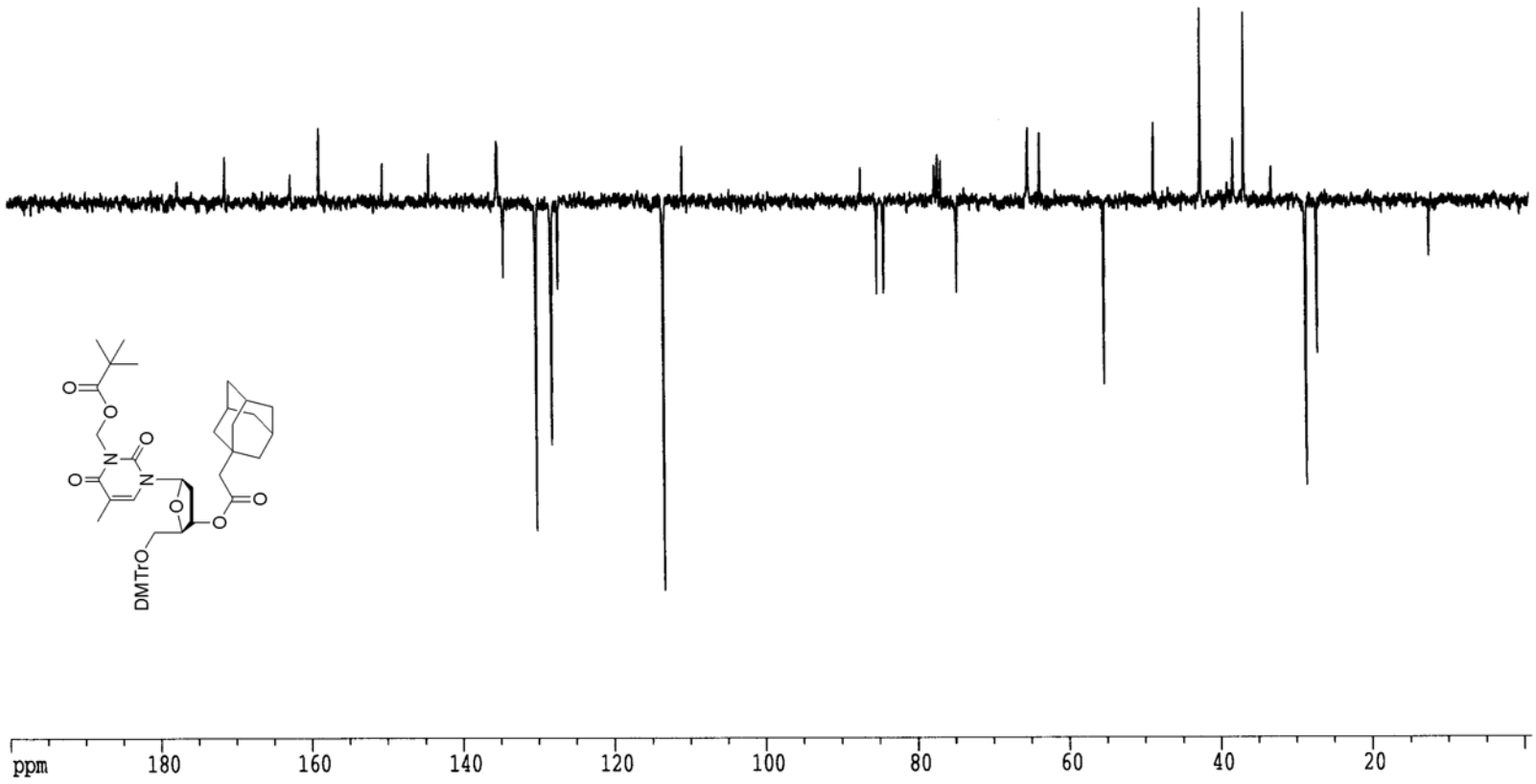
O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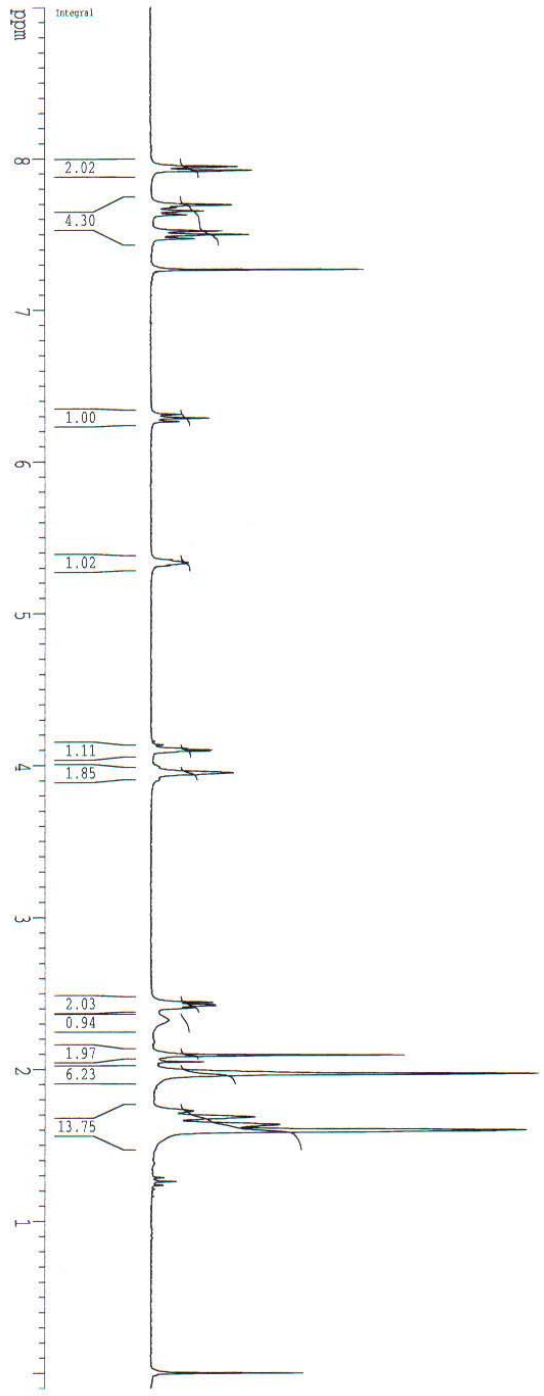
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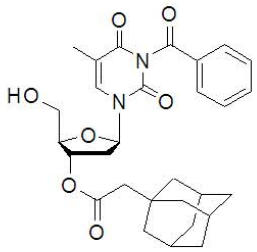
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N
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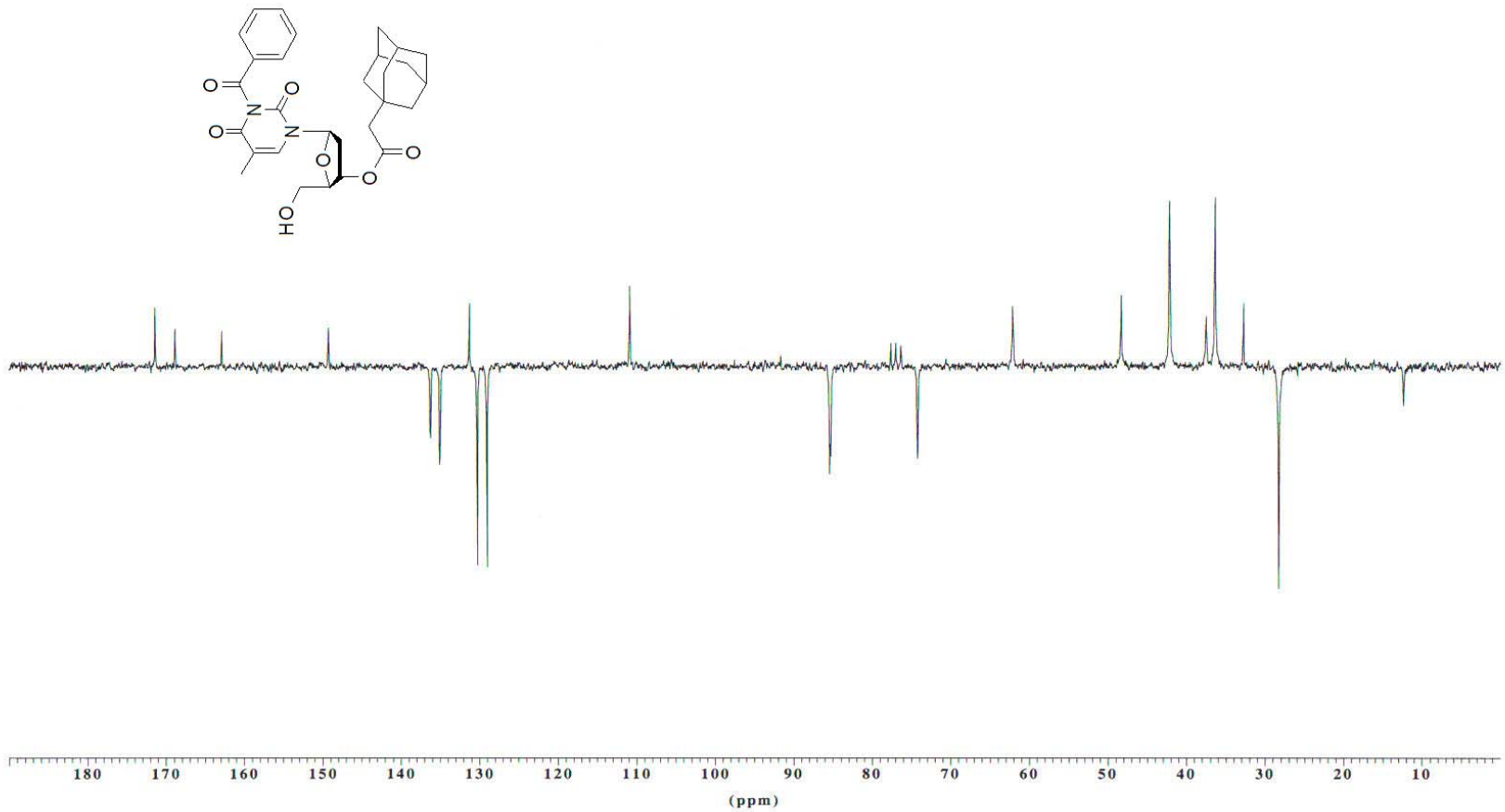
N
O
O
O
O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O
N
O
N
O
O
O
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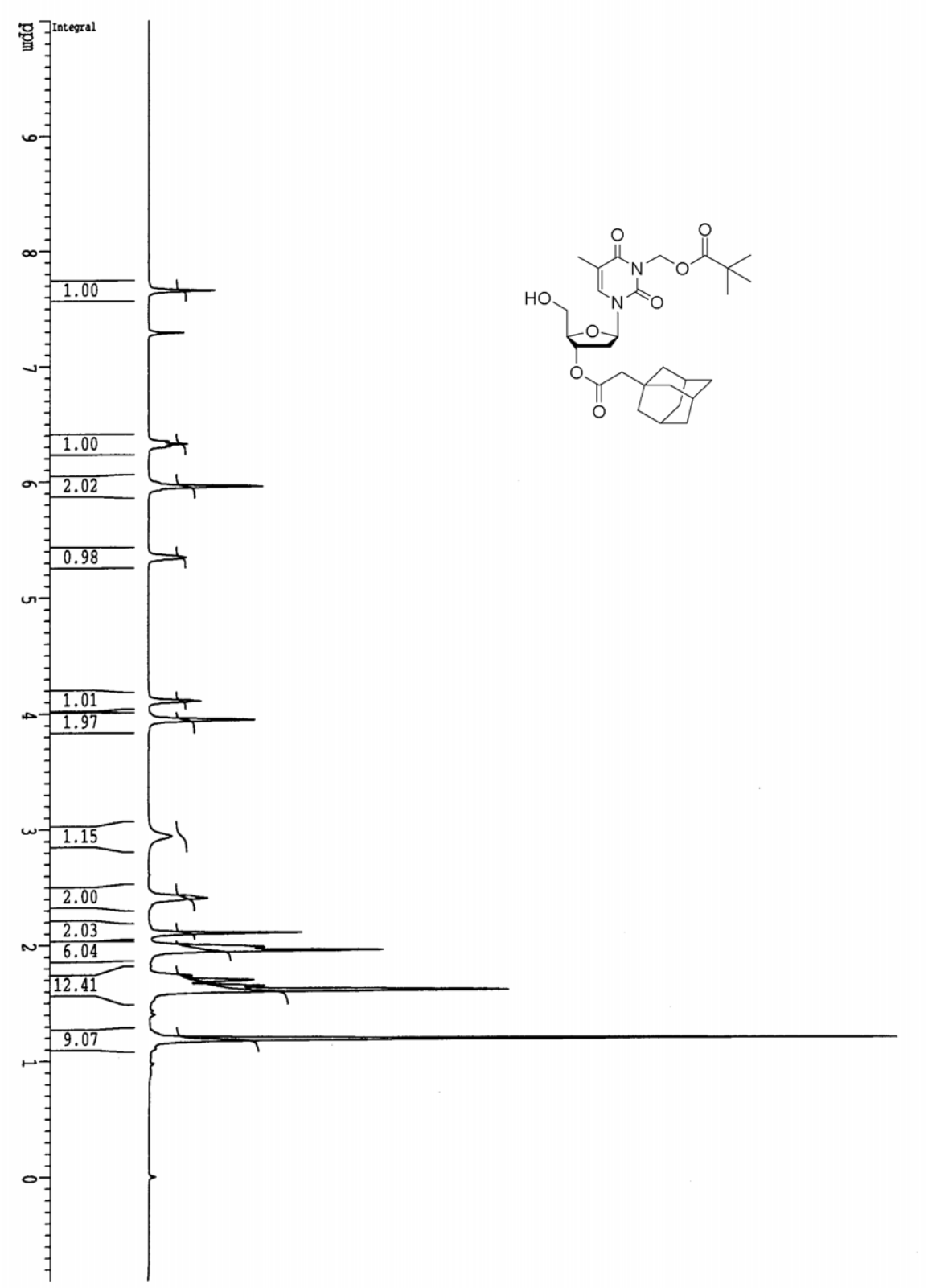
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)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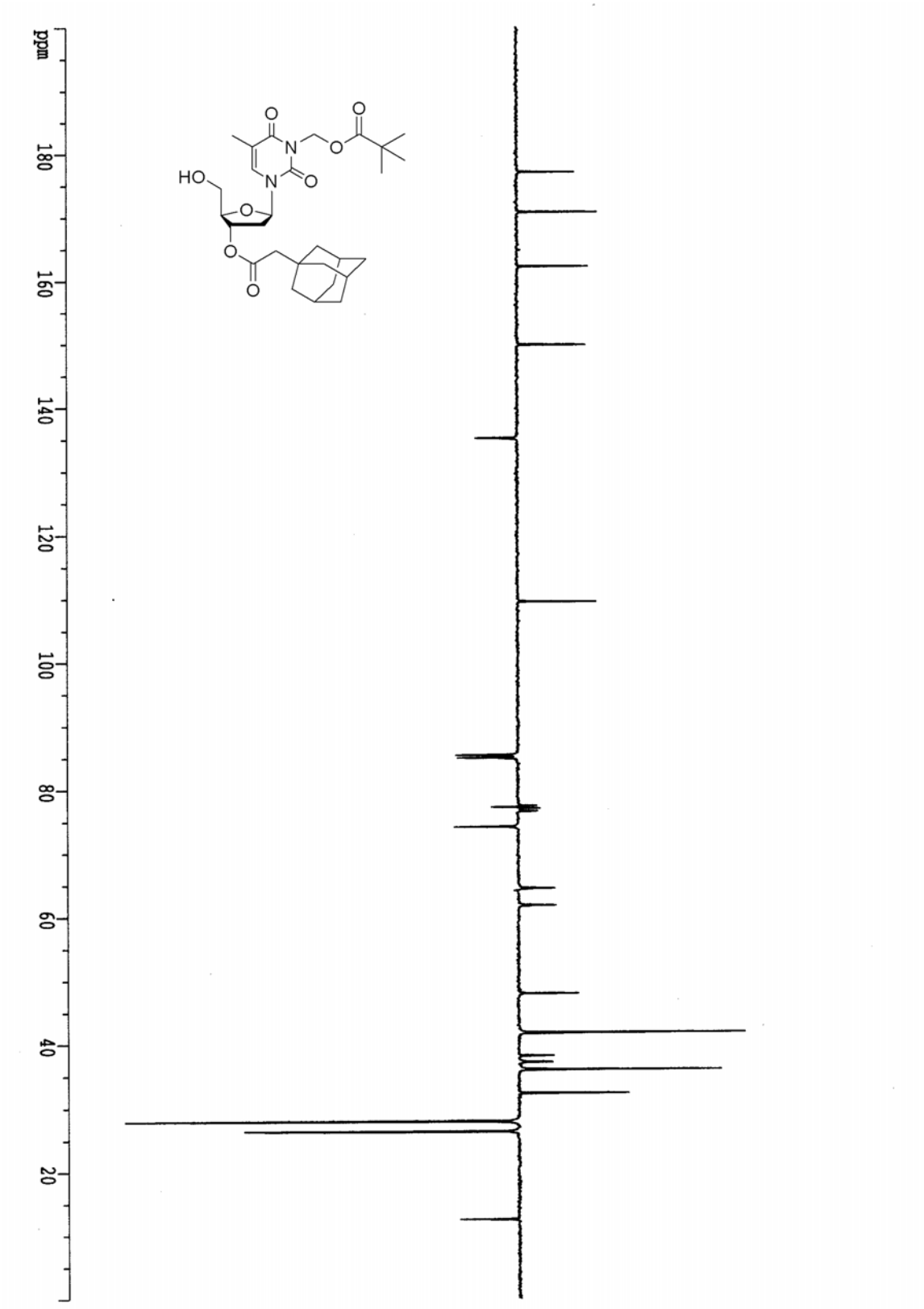
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HO
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N
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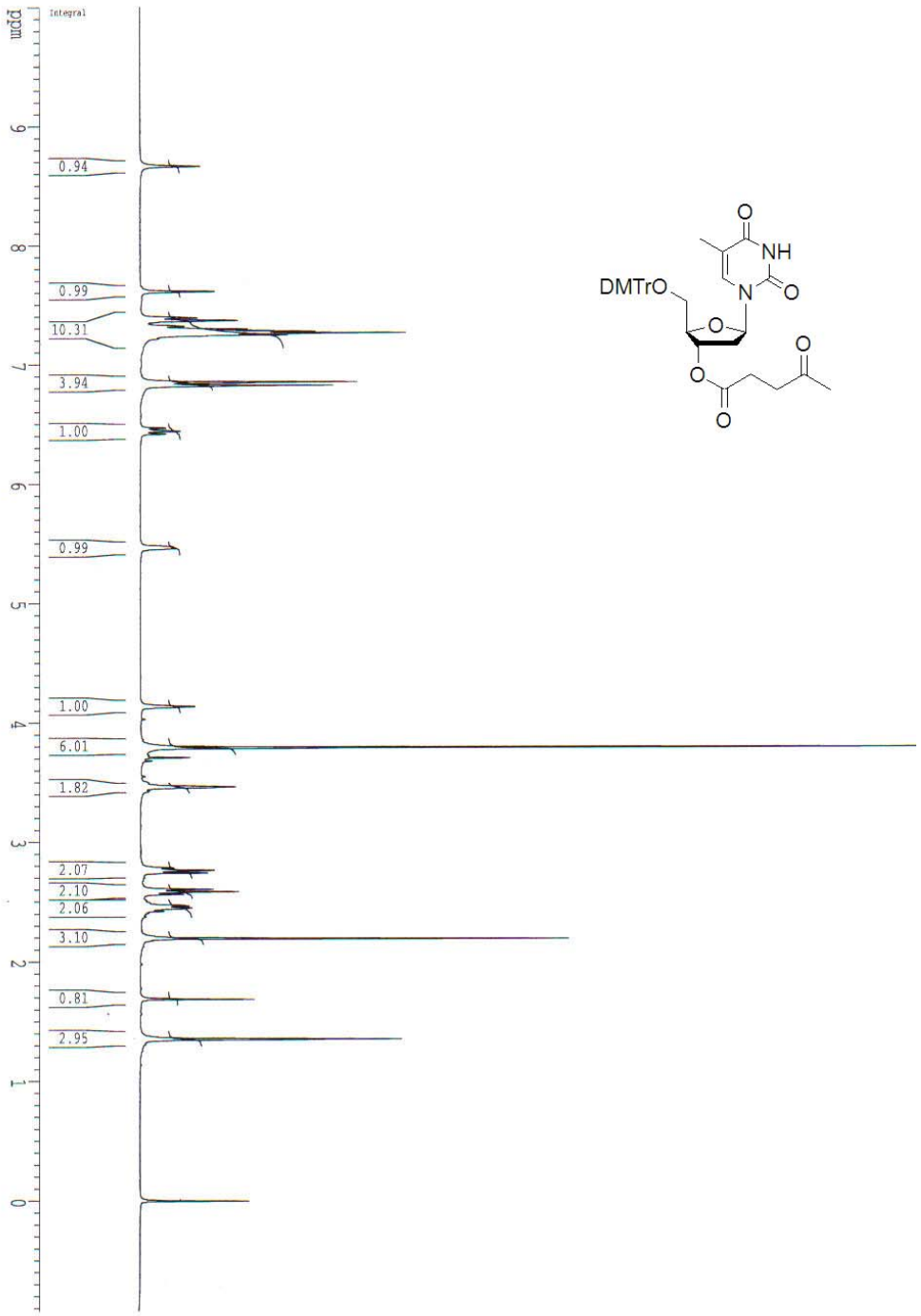
O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1.00
6.01
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2.06
3.10
0.81
2.95
DMTrO
NH
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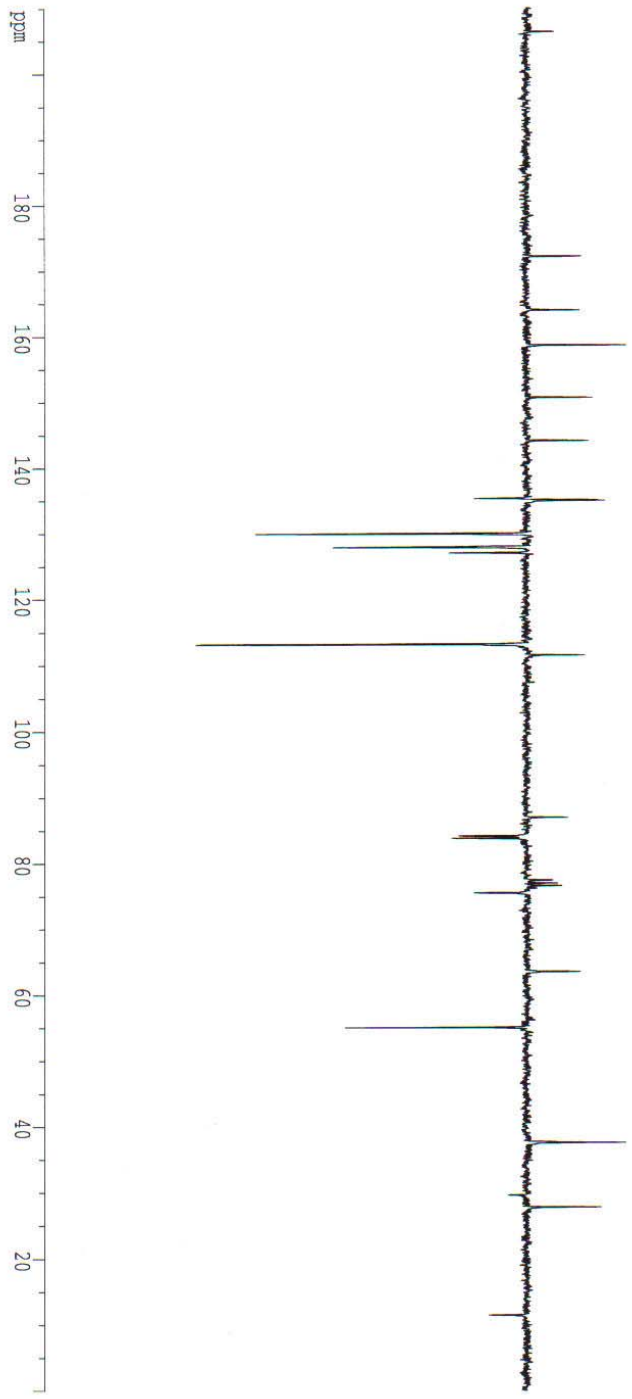
O
O
O
O
O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160
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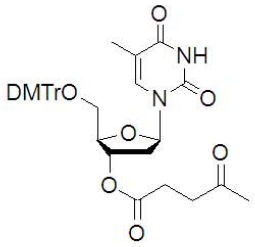
120
100
80
60
40
20


O
NH
DMTrO
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O
O
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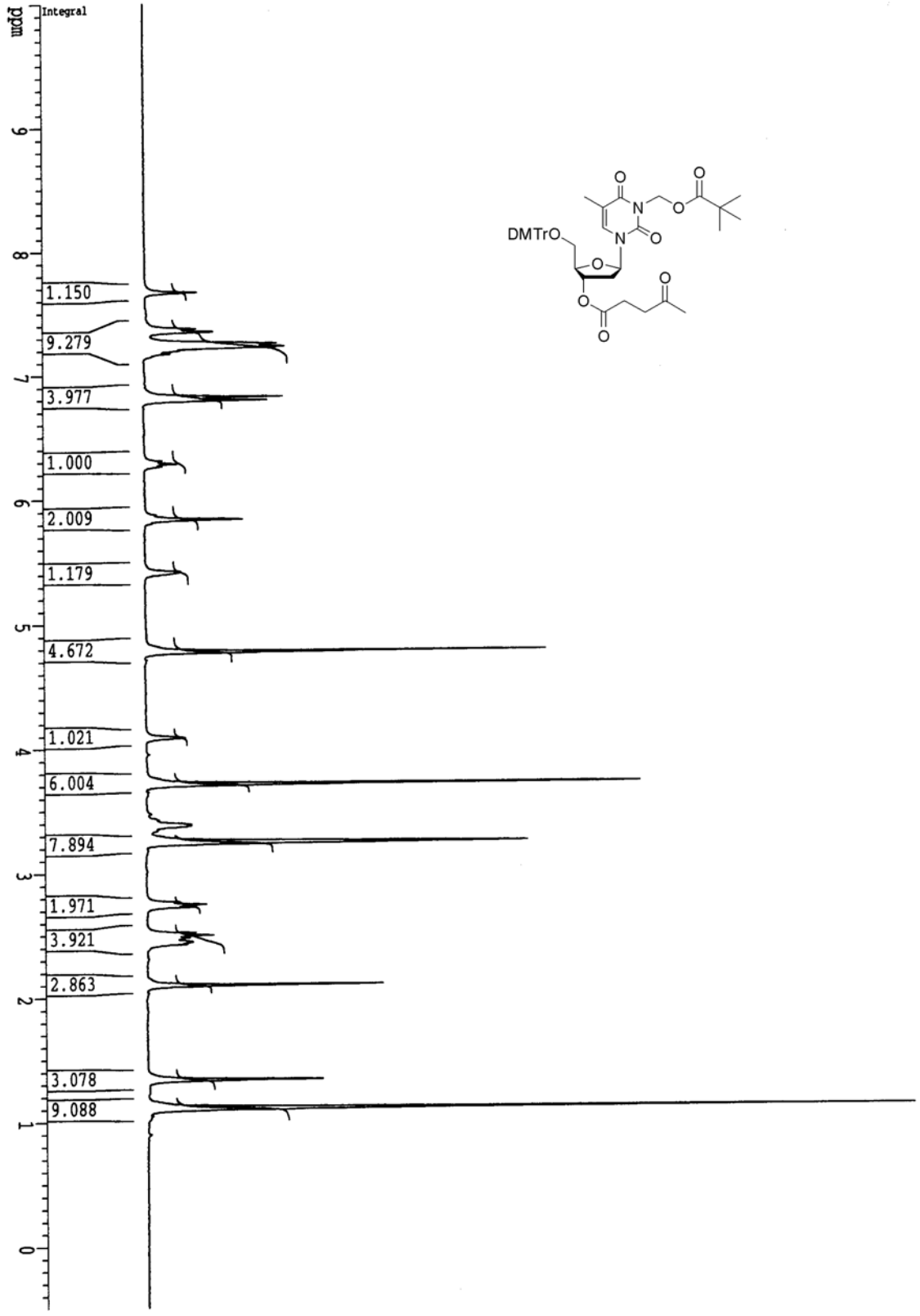
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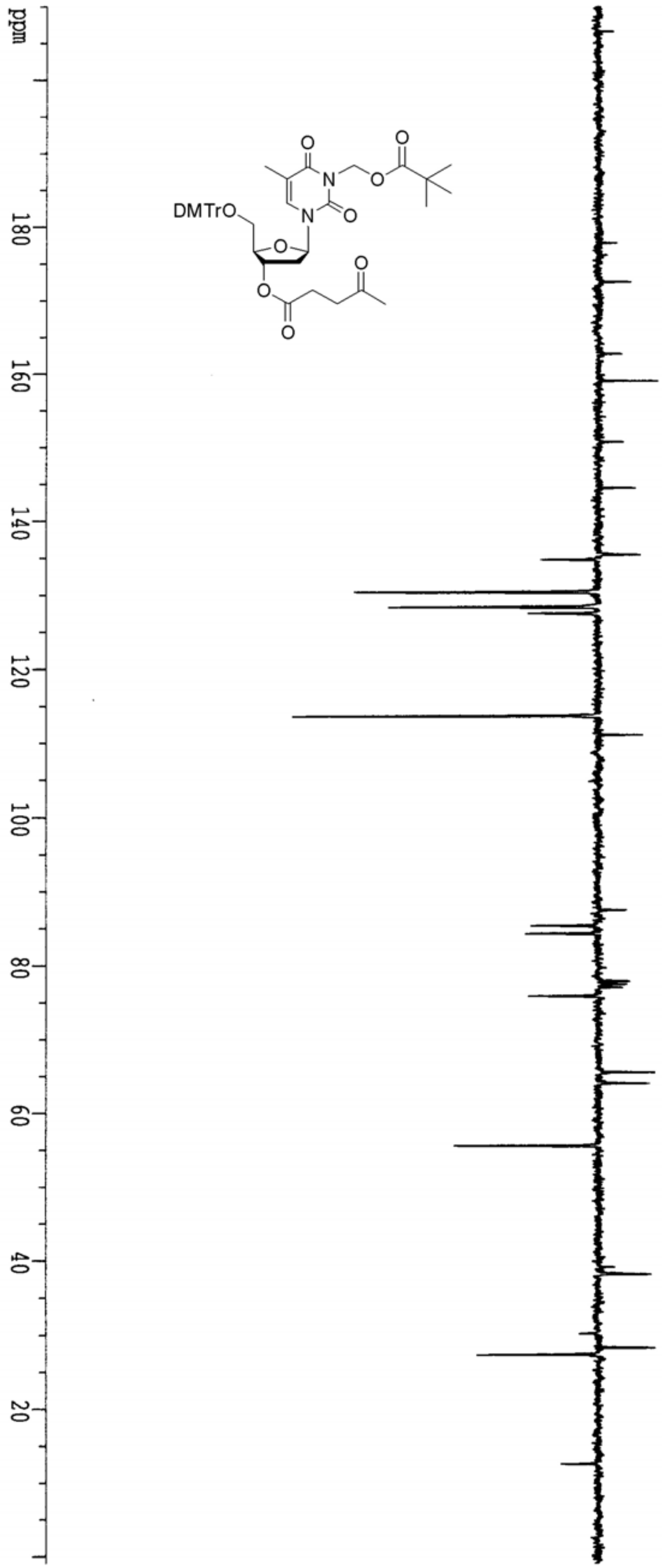
DMTrO
N
O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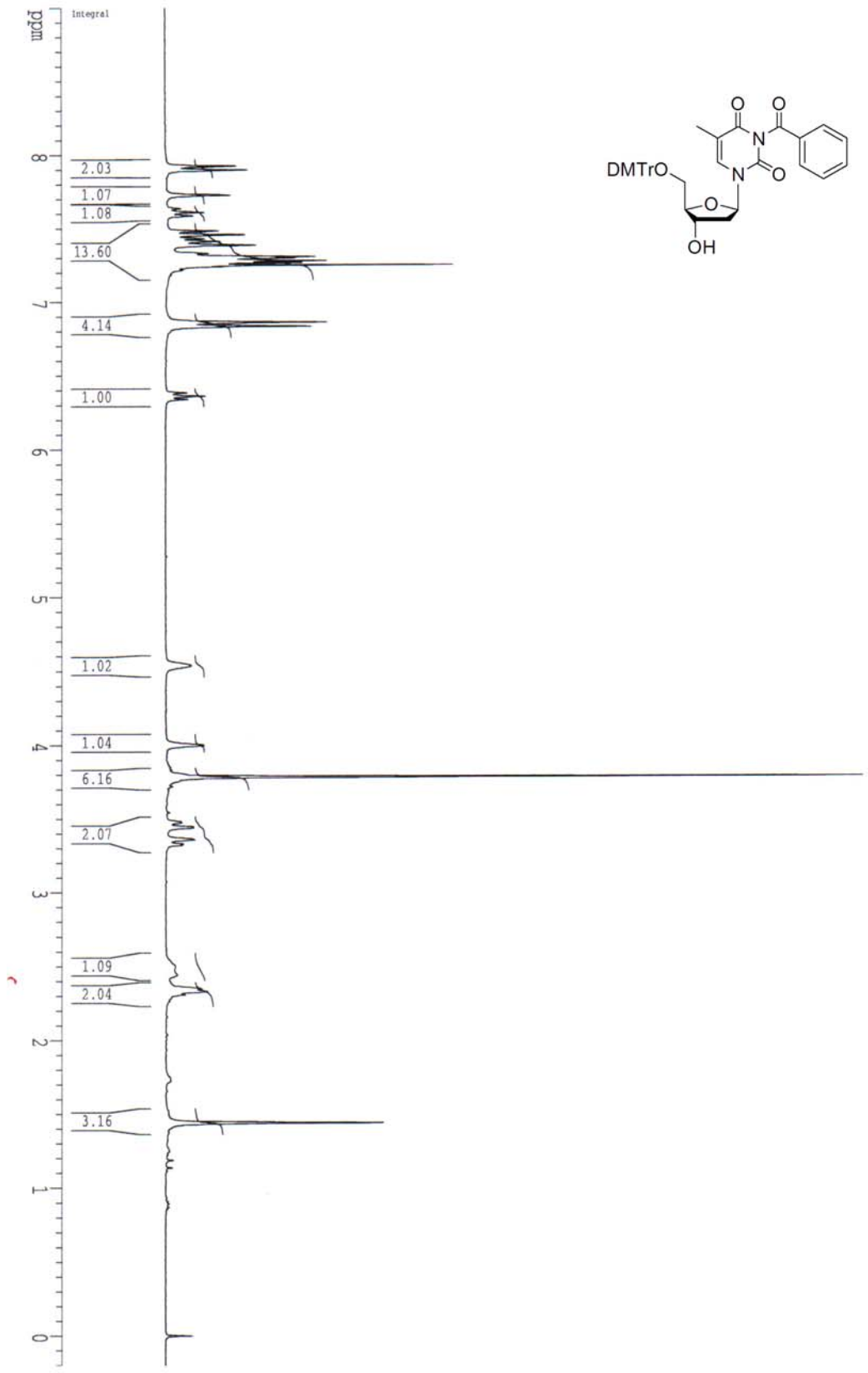
140
120
100
80
60
40
20
DMTrO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1.09
2.04
3.16
DMTrO
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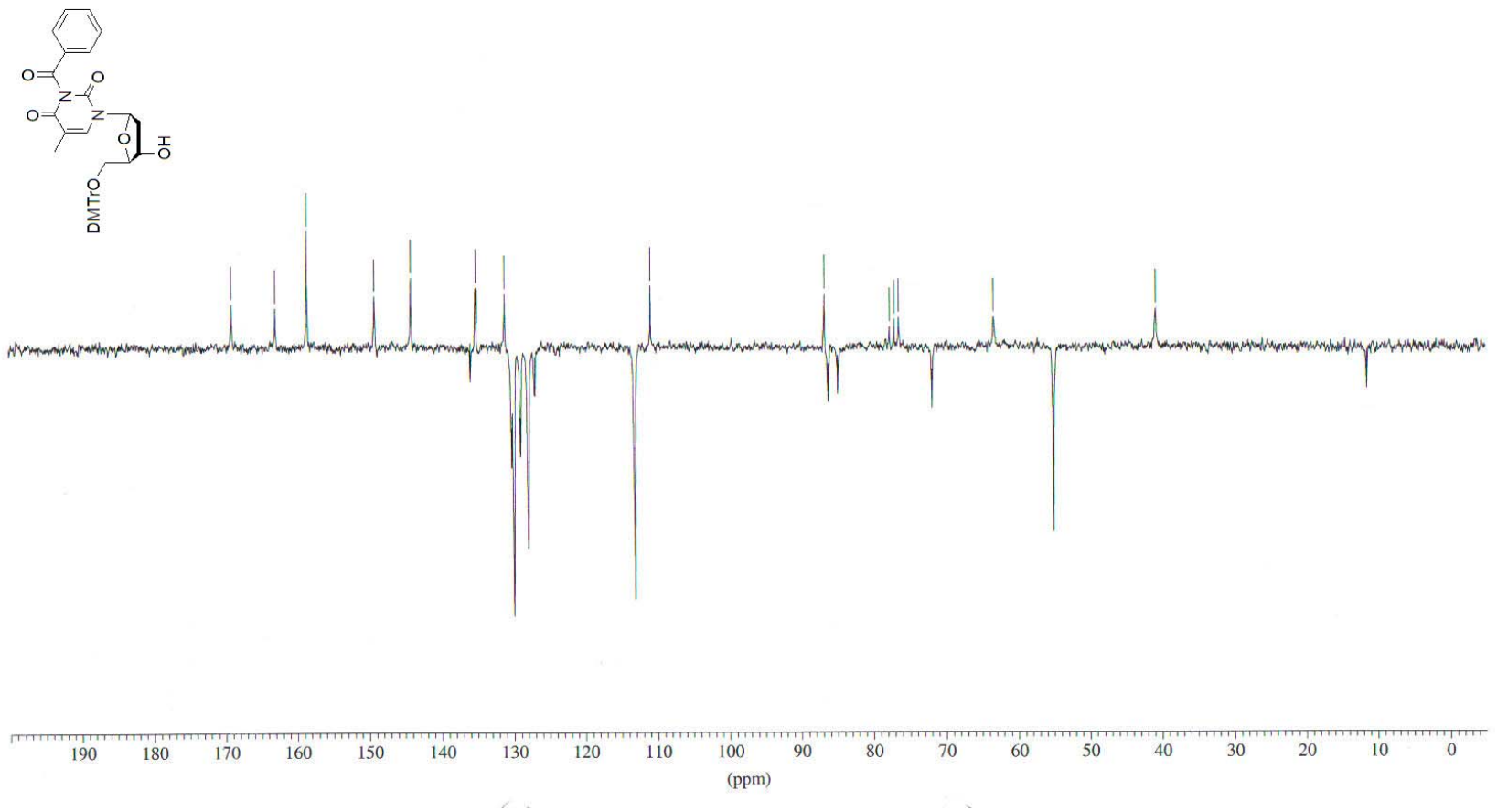
O
O
N
O
OH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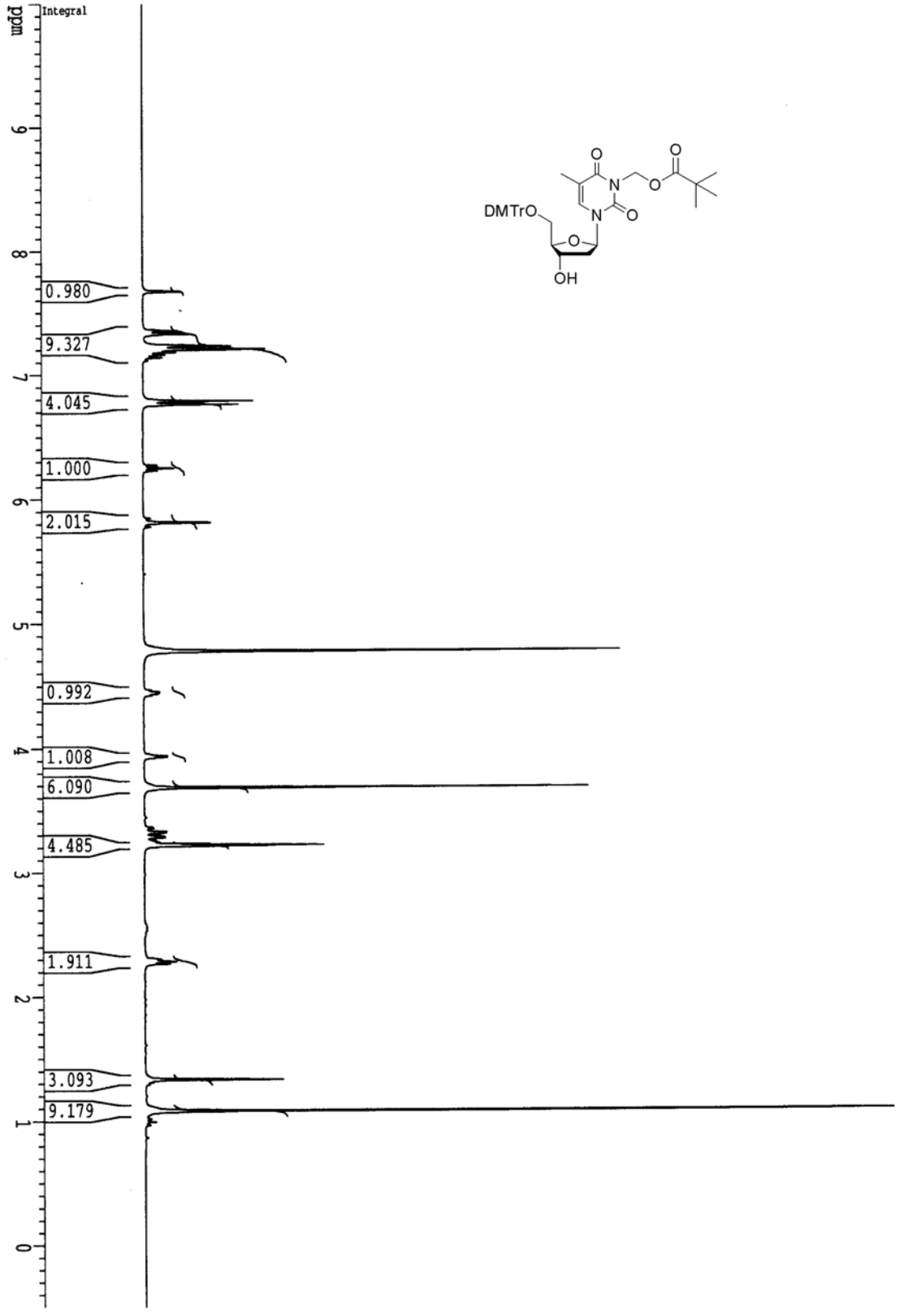
(ppm)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DMTrO
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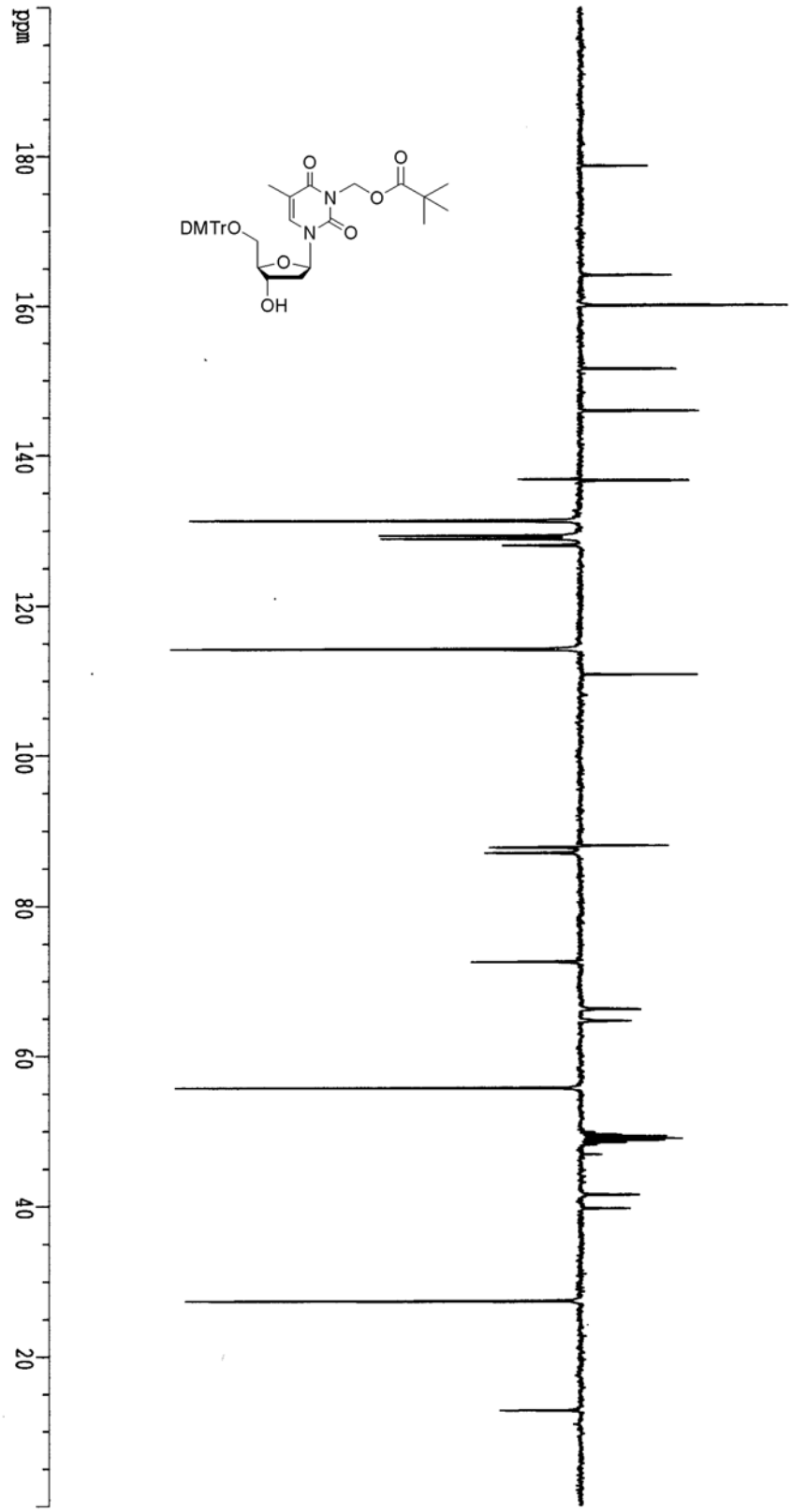
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60
40
20
DMTrO
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N
O
O
O
N
O
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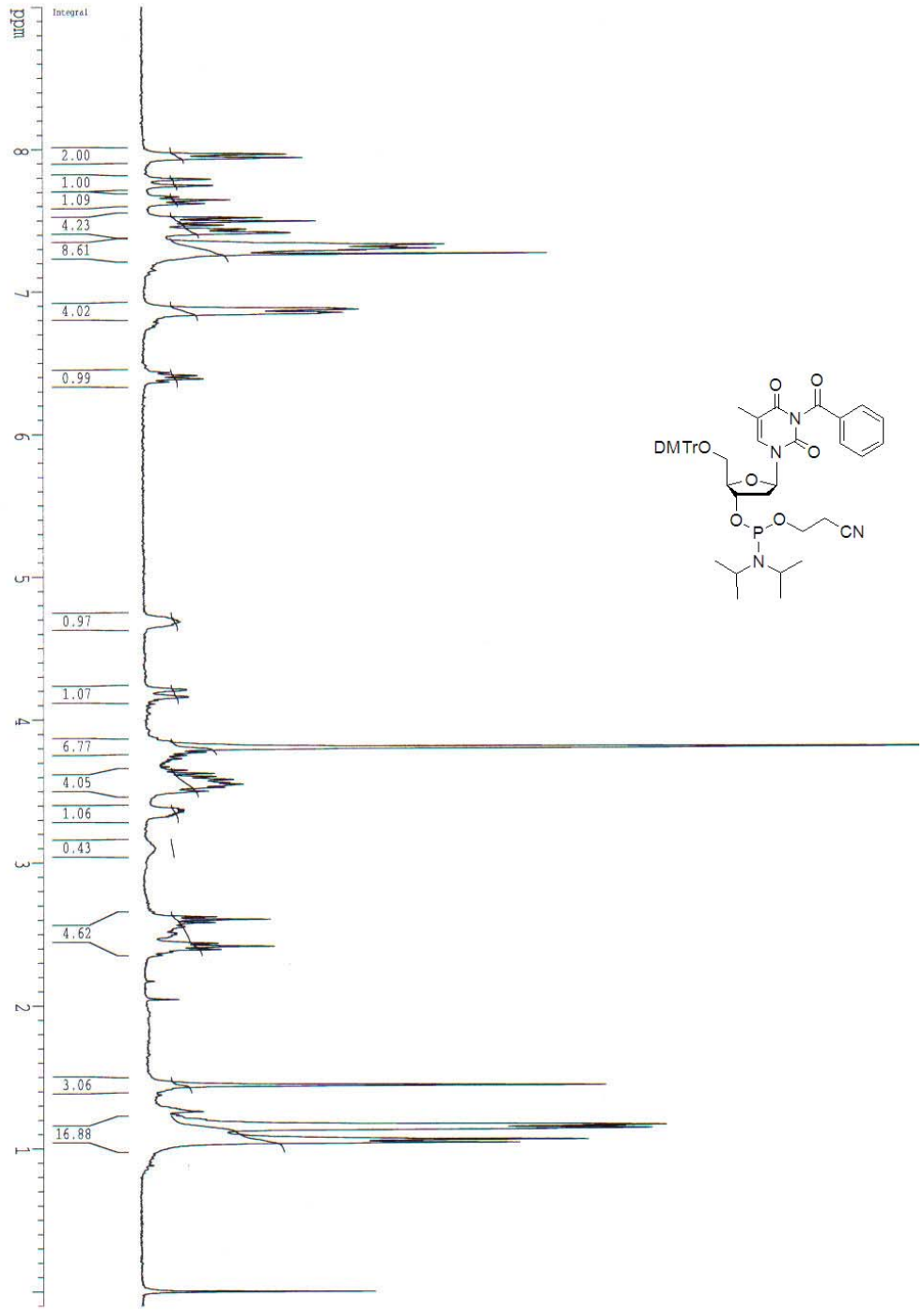
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3.06
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DMTrO
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O
O
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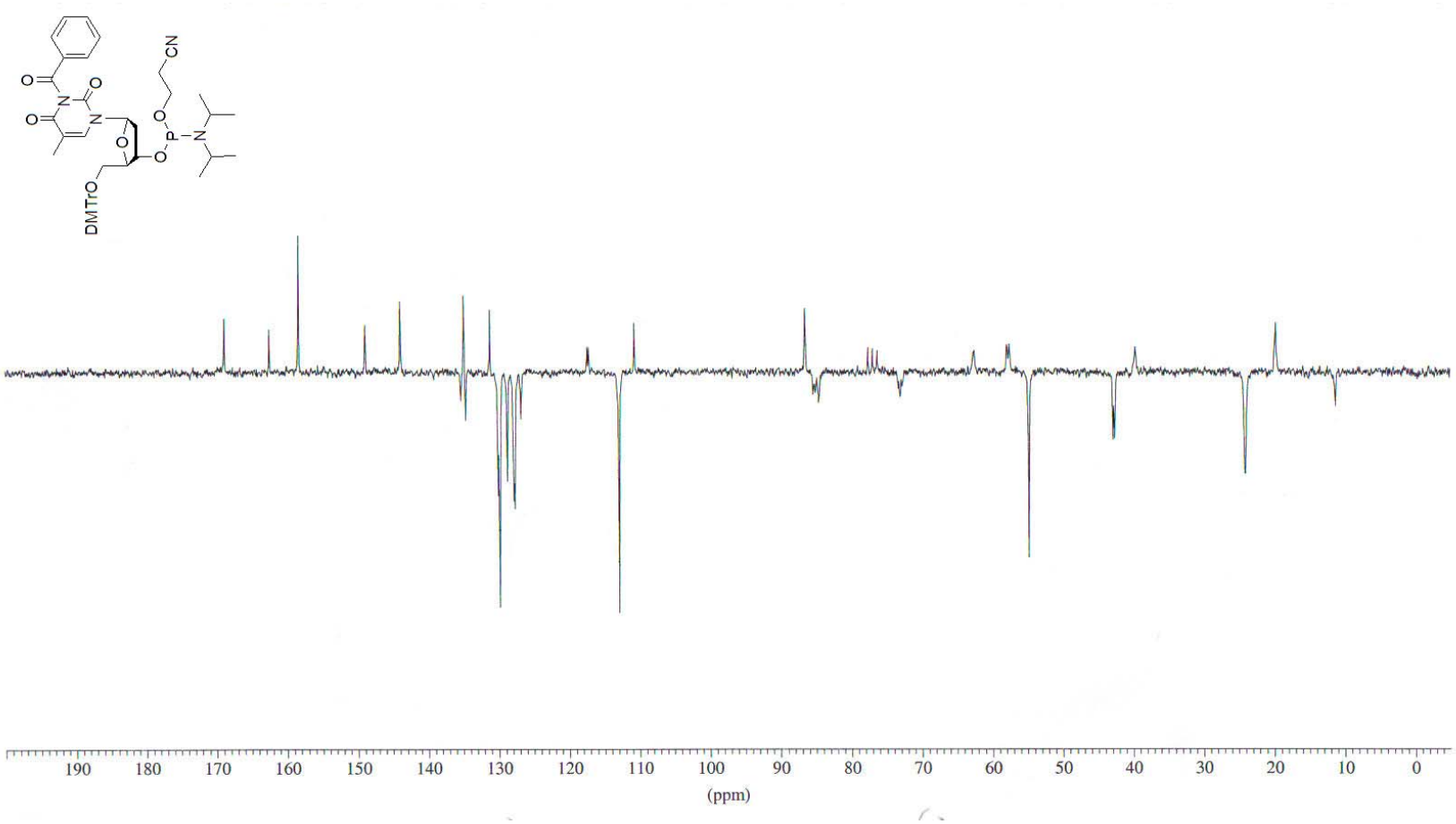
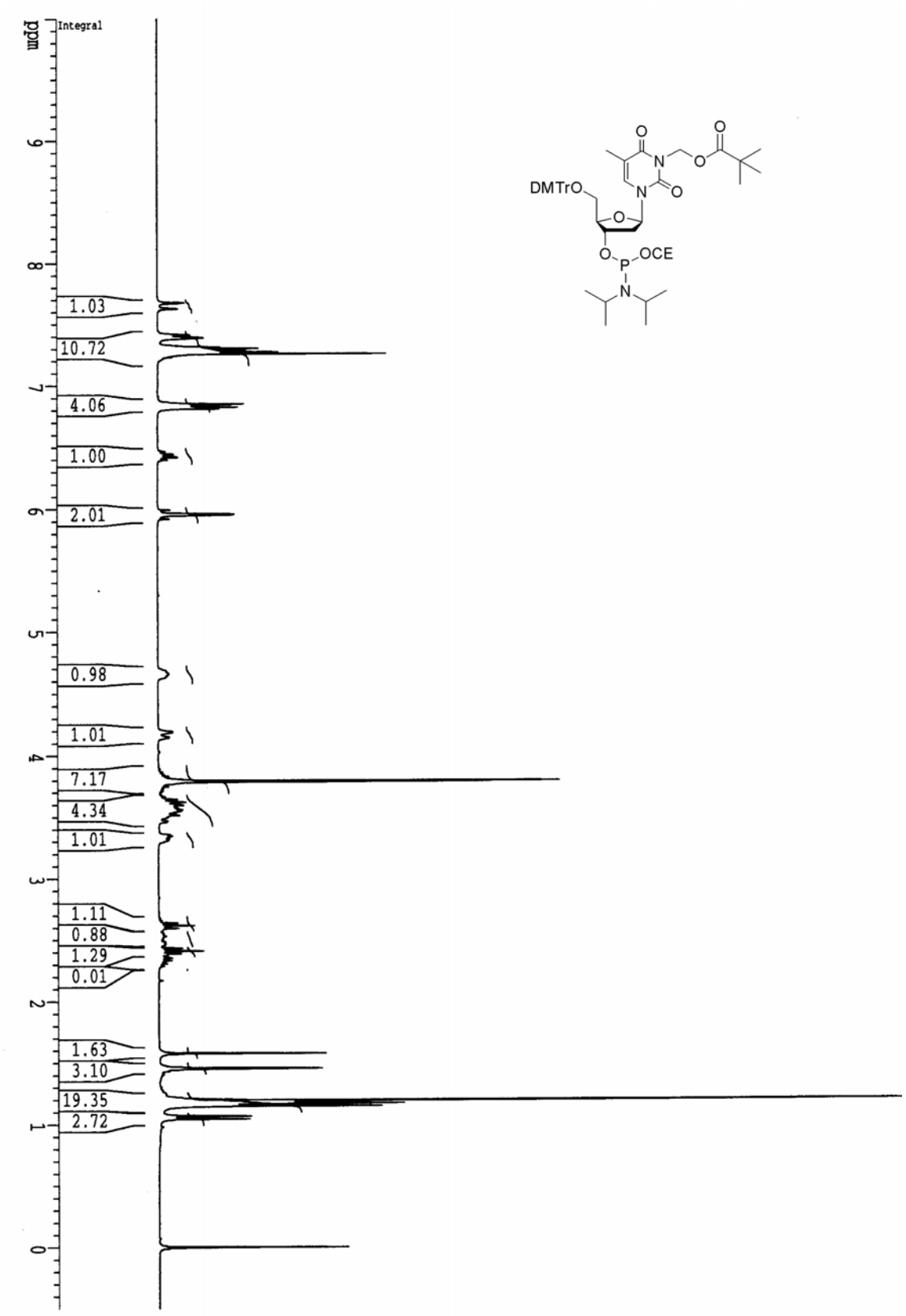
O
P
O
CN
N


O
O
N
O
N
O
O
P
N
CN
DMTrO
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
(ppm)

DMTrO
O
N
O
O
O
O
P
O
CN
N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1.01
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4.34
1.01
1.11
0.88
1.29
0.01
1.63
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19.35
2.72
DMTrO
O
N
O
O
O
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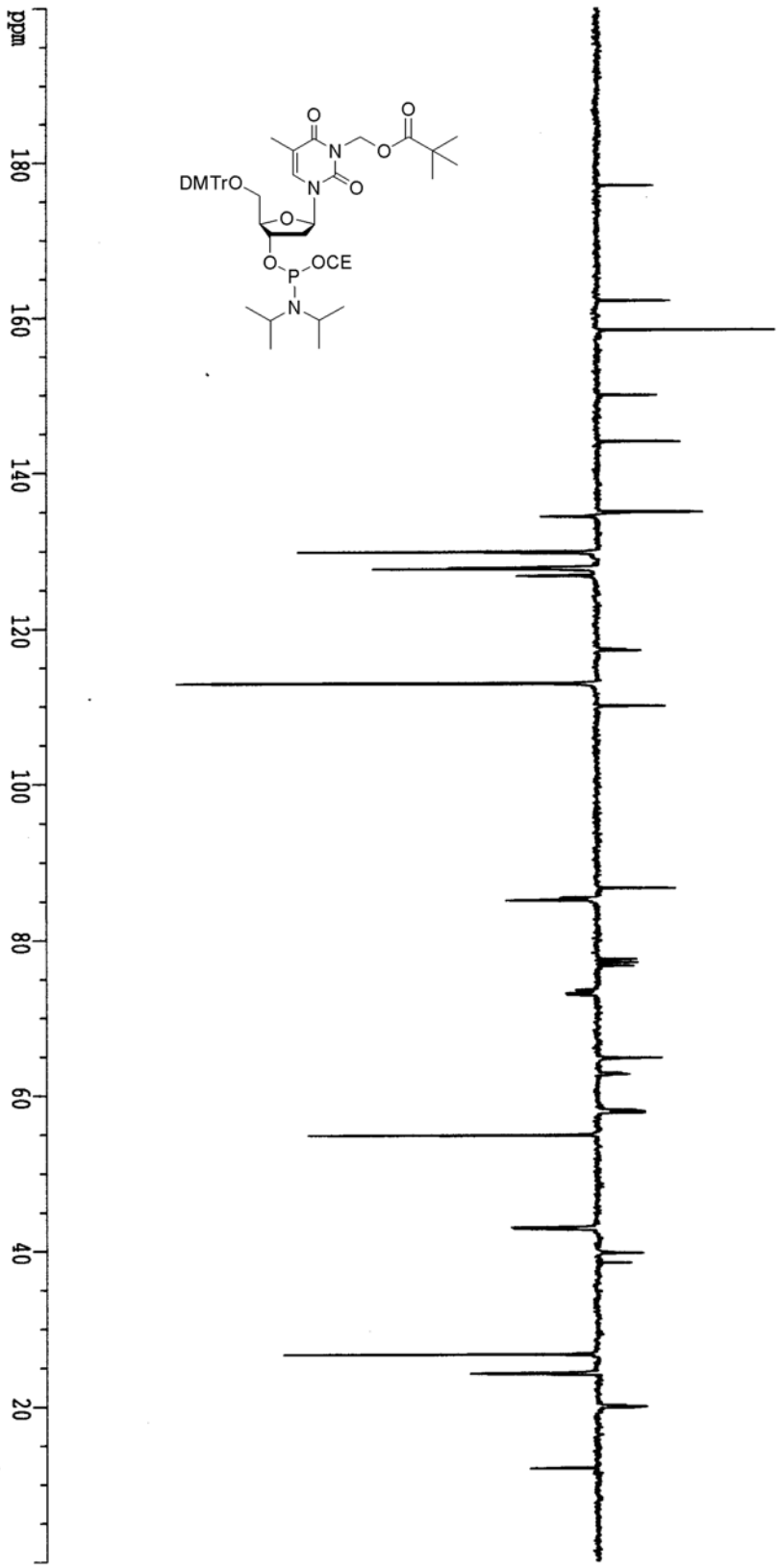
O
O
P
OCE
N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160
140
120
100
80
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40
20
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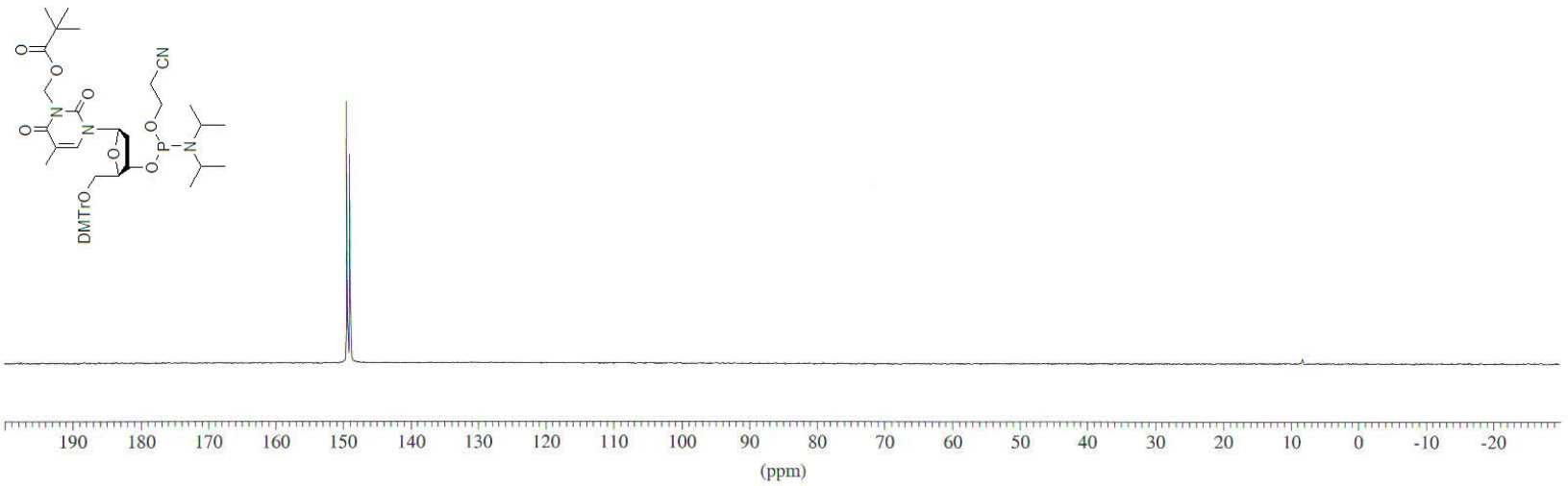
N
O
O
O
O
N
O
O
P
OCE
N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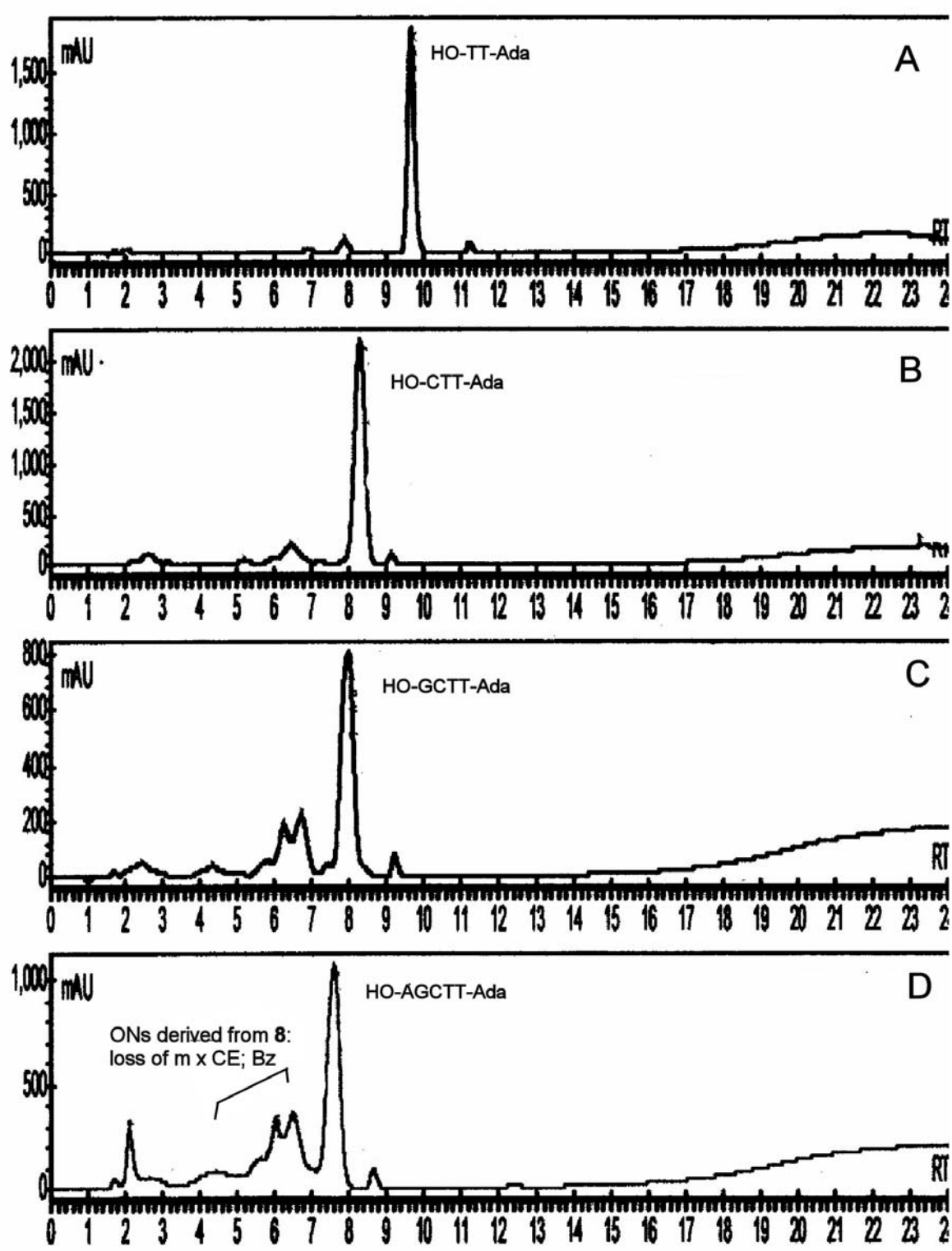
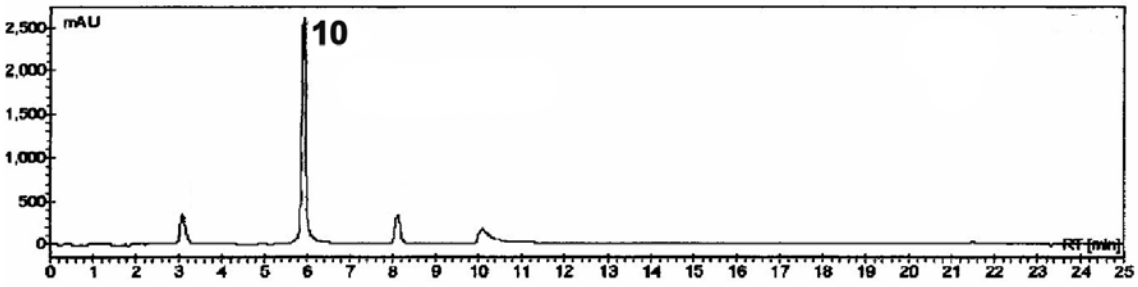
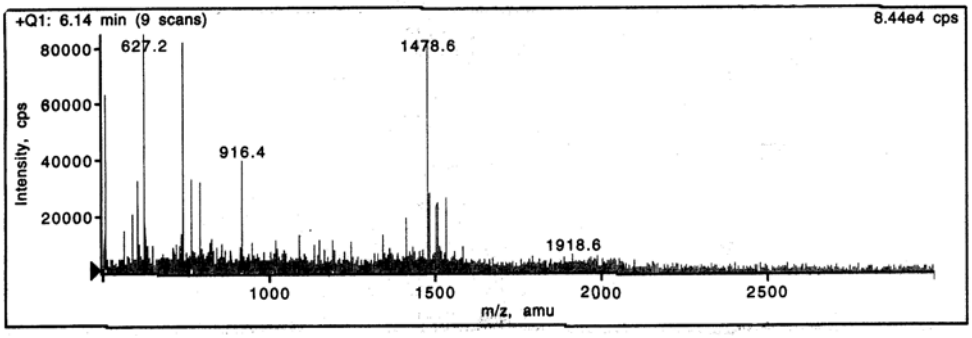
(ppm)



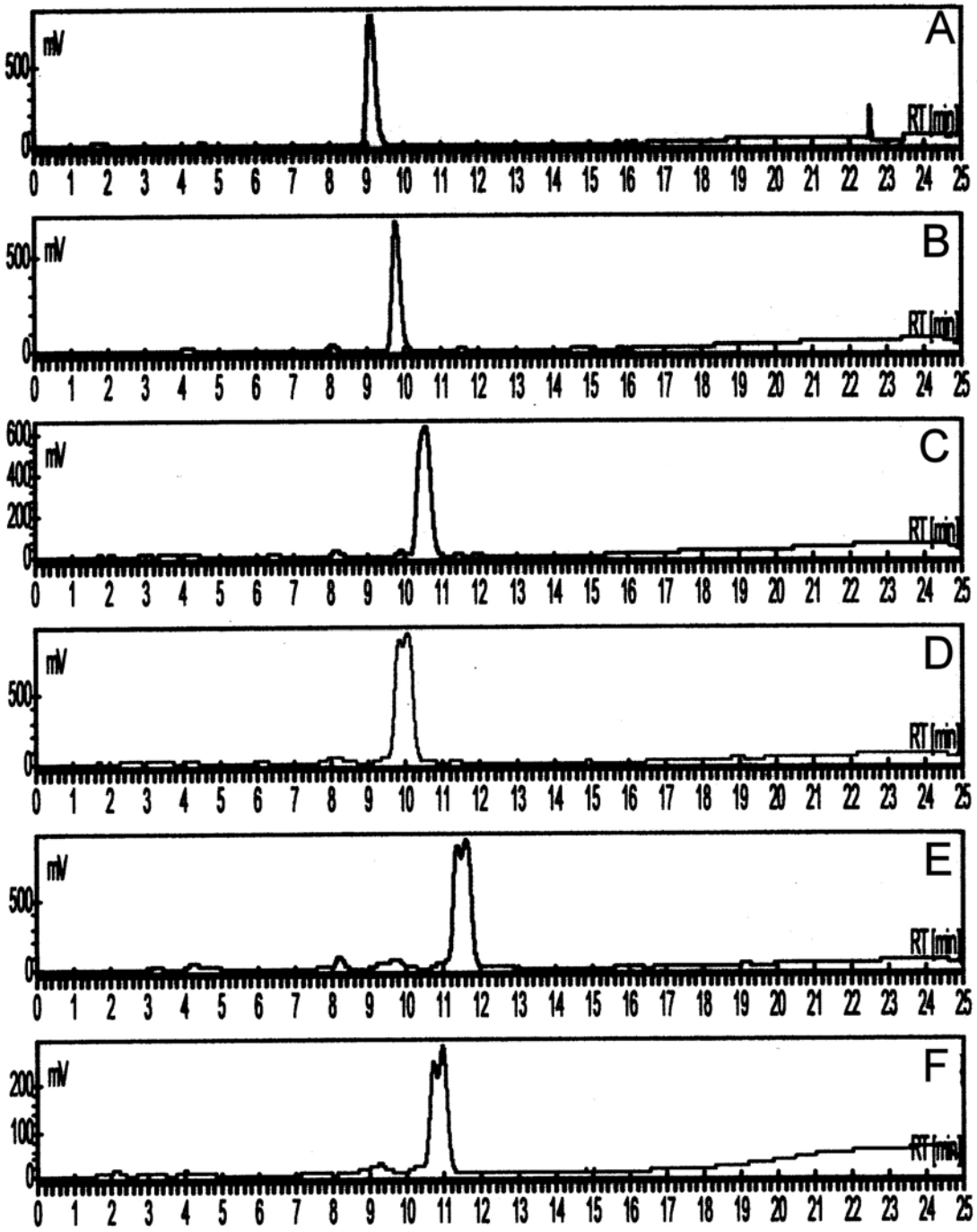
HPLC traces of the growing oligonucleotide chain in the synthesis of pentamer **8** after each cycle. A: $^{HO}dT^{Bz}dT^{Bz}{}_{Ada}$; B: $^{HO}dC^{Bz}dT^{Bz}dT^{Bz}{}_{Ada}$; C: $^{HO}dG^{iBu}dC^{Bz}dT^{Bz}dT^{Bz}{}_{Ada}$; D: $^{HO}dA^{Bz}dG^{iBu}dC^{Bz}dT^{Bz}dT^{Bz}{}_{Ada}$.



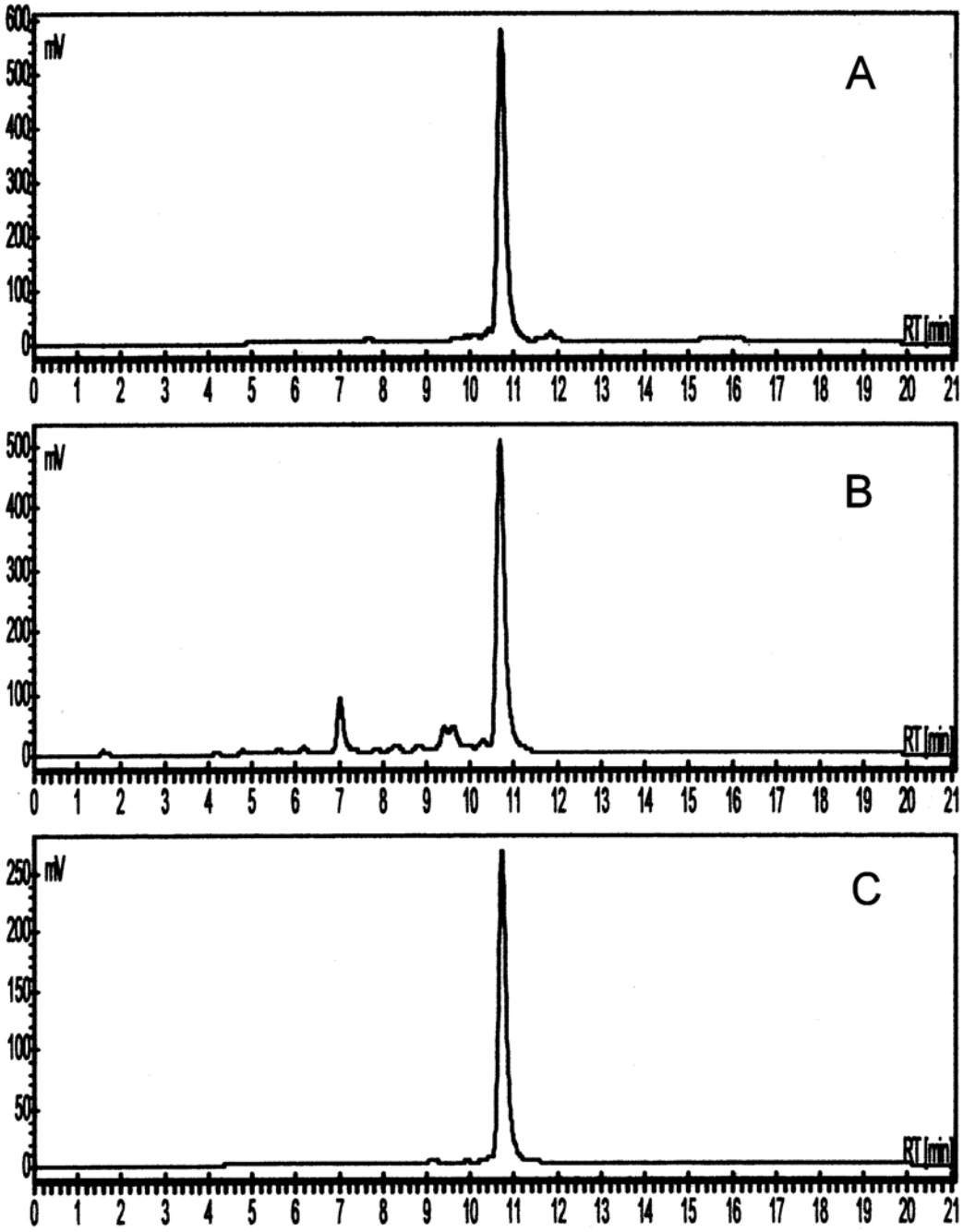


LC/MS chromatograms (254 nm, 0-40% B) of crude, deblocked pentamer oligonucleotide **10** (5'- AGCTT – 3')

Calculated mass for **10:** $[M+H]^+$: 1478.3



HPLC traces of the growing oligonucleotide chain after each cycle in the synthesis of hexamer **9**. A: Starting compound **17**; B: $^{HO}dT^{Pom}dT^{Pom}{}_{Ada}$; C: $^{HO}dC^{Bz}dT^{Pom}dT^{Pom}{}_{Ada}$; D: $^{HO}dG^{iBu}dC^{Bz}dT^{Pom}dT^{Pom}{}_{Ada}$; E: $^{HO}dT^{Pom}dG^{iBu}dC^{Bz}dT^{Pom}dT^{Pom}{}_{Ada}$; F: $^{HO}dA^{Bz}dT^{Pom}dG^{iBu}dC^{Bz}dT^{Pom}dT^{Pom}{}_{Ada}$



Mono-Q ion exchange chromography traces of hexamer oligonucleotide **11**.

A: Crude sample after deblocking of **9**

B: Crude reference sample prepared by standard solid phase synthesis procedures

C: Oligonucleotide **11** after purification and desalting.