

## Supporting Information

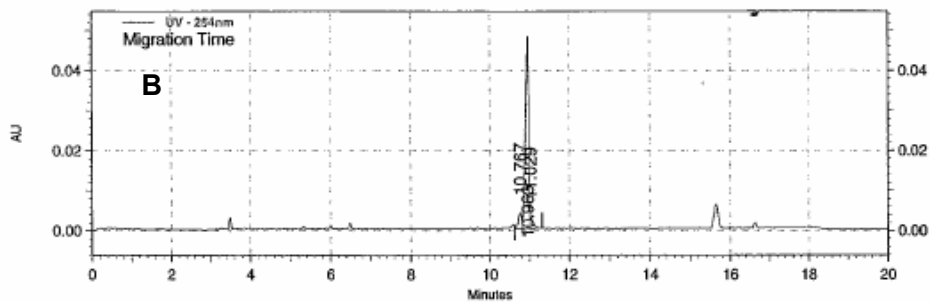
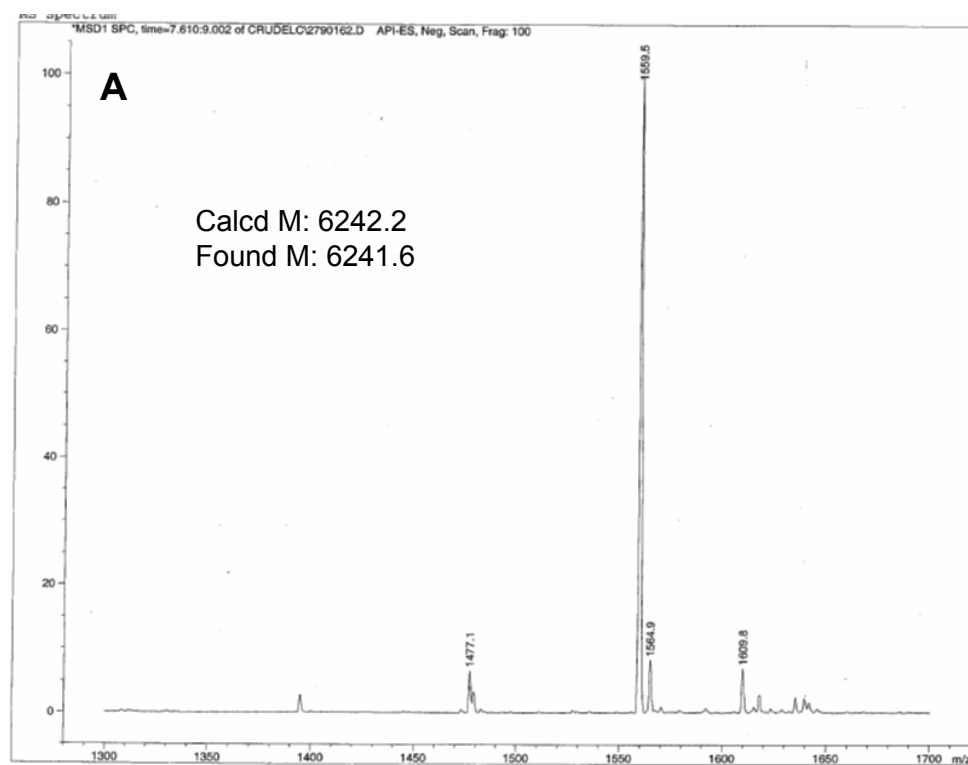
### Improving RNA Interference in Mammalian Cells by 4'-Thio modified siRNA: Effect on siRNA Activity and Nuclease Stability When Used in Combination with 2'-O-Alkyl Modifications

Prasad Dande \*, Thazha P. Prakash, Namir Sioufi, Hans Gaus, Russell Jarres, Andreas Berdeja, Eric E. Swayze, Richard H. Griffey, Balkrishen Bhat.

Department of Medicinal Chemistry and Antisense Core Research, Isis Pharmaceuticals Inc., Carlsbad CA 92008

#### Contents:

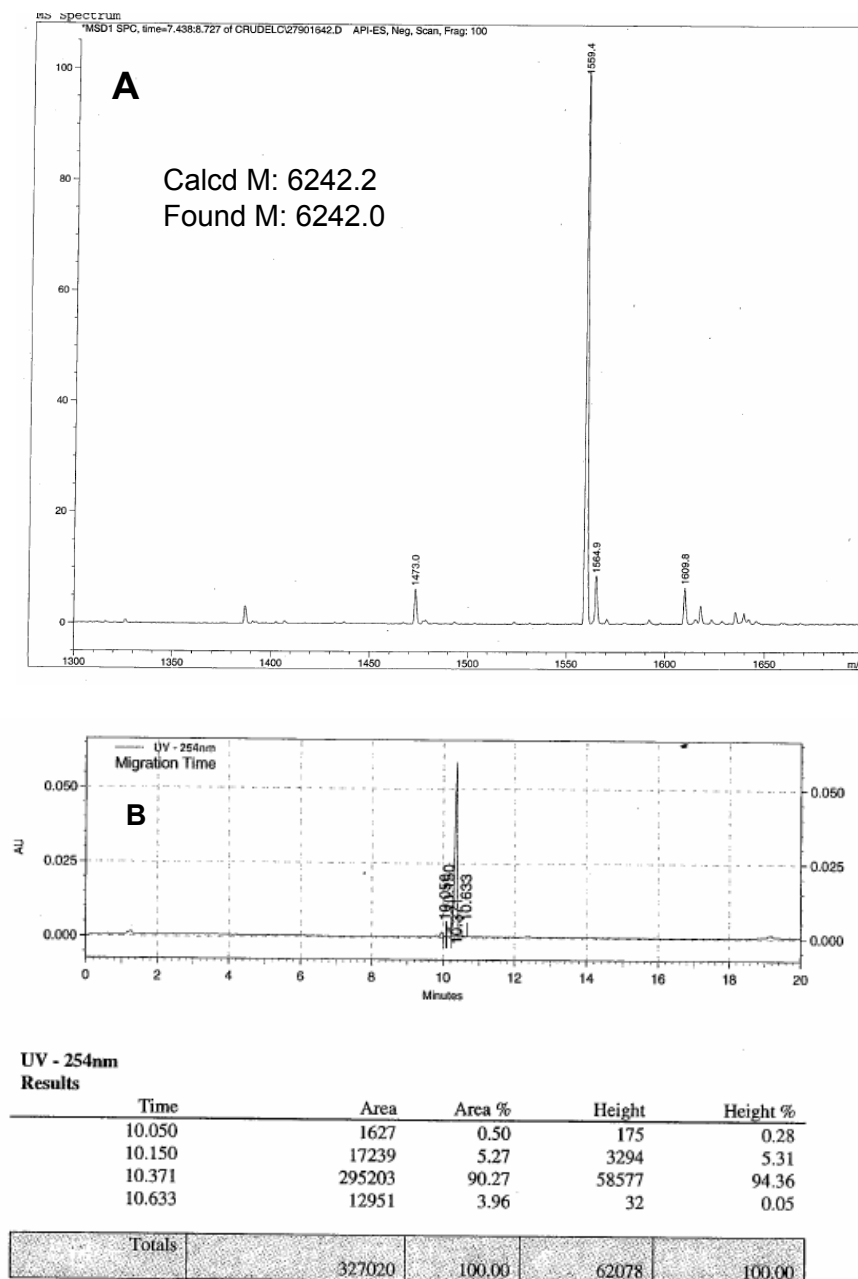
|    |  |     |
|----|--|-----|
| 1  | <b>Figure 1.</b> Electrospray mass spectrum and capillary electrophoresis profile of oligonucleotide 5'- AAGUAAGGACCAGAGACAA-3'.   | S2  |
| 2  | <b>Figure 2.</b> Electrospray mass spectrum and capillary electrophoresis profile of oligonucleotide 5'- AAGUAAGGACCAGAGACAA-3'.   | S3  |
| 3  | <b>Figure 3.</b> Electrospray mass spectrum and capillary electrophoresis profile of oligonucleotide 5'-AAGUAAGGACCAGAGACAA-3'.    | S4  |
| 4  | <b>Figure 4.</b> Electrospray mass spectrum and capillary electrophoresis profile of oligonucleotide 5'- UUCAUCCUGGUCUCUGUU-3'.    | S5  |
| 5  | <b>Figure 5.</b> Electrospray mass spectrum and capillary electrophoresis profile of oligonucleotide 3'- UUCAUCCUGGUCUCUGUU -5'.   | S6  |
| 6  | <b>Figure 6.</b> Electrospray mass spectrum and capillary electrophoresis profile of oligonucleotide 3'-UUCAUCCUGGUCUCUGUUU-P-5'.  | S7  |
| 7  | <b>Figure 7.</b> Electrospray mass spectrum and capillary electrophoresis profile of oligonucleotide 3'-UUCAUCCUGGUCUCUGUUU-P -5'. | S8  |
| 8  | <b>Figure 8.</b> Electrospray mass spectrum and capillary electrophoresis profile of oligonucleotide 3'-UUCAUCCUGGUCUCUGUUU-P -5'. | S9  |
| 9  | <b>Figure 9.</b> High resolution mass spectrum (FAB) of compound 7.  | S10 |
| 10 | <b>Figure 10.</b> High resolution mass spectrum (FAB) of compound 8.   | S11 |
| 11 | <b>Figure 11.</b> High resolution mass spectrum (FAB) of compound 14.  | S12 |
| 12 | <b>Figure 12.</b> <sup>31</sup> P NMR of compound 7 in CDCl <sub>3</sub> .   | S13 |
| 13 | <b>Figure 13.</b> <sup>31</sup> P NMR of compound 8 in CDCl <sub>3</sub>   | S14 |
| 14 | <b>Figure 14.</b> <sup>31</sup> P NMR of compound 14 in CDCl <sub>3</sub> .  | S15 |



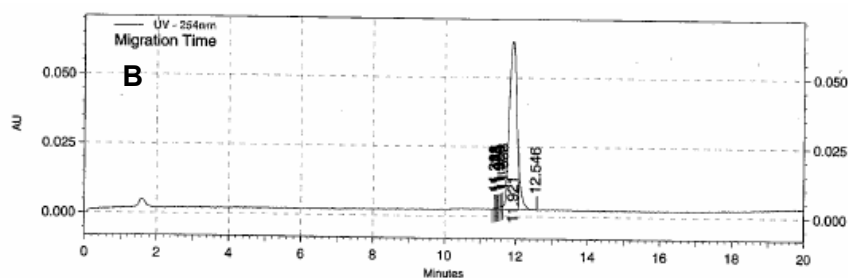
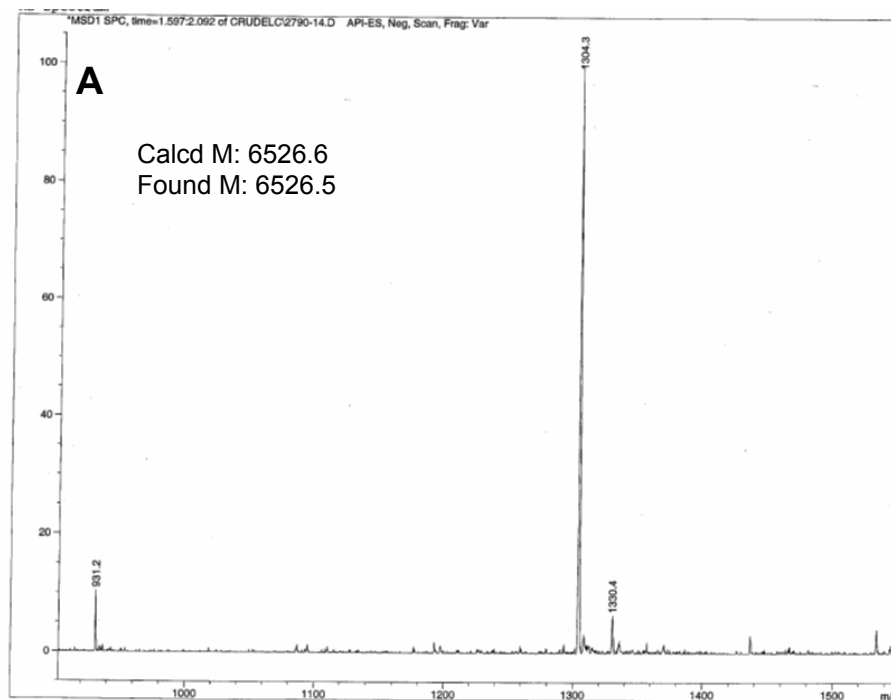
**UV - 254nm  
Results**

| Time   | Area   | Area % | Height | Height % |
|--------|--------|--------|--------|----------|
| 10.767 | 23116  | 7.92   | 3931   | 7.15     |
| 10.963 | 252780 | 86.61  | 48195  | 87.62    |
| 11.029 | 15964  | 5.47   | 2876   | 5.23     |
| Totals | 291860 | 100.00 | 55002  | 100.00   |

**Figure 1.** Electrospray mass spectrum (A) and capillary electrophoresis profile (B) of oligonucleotide 5'-AAGUAGGACCAGAGACAA-3'.

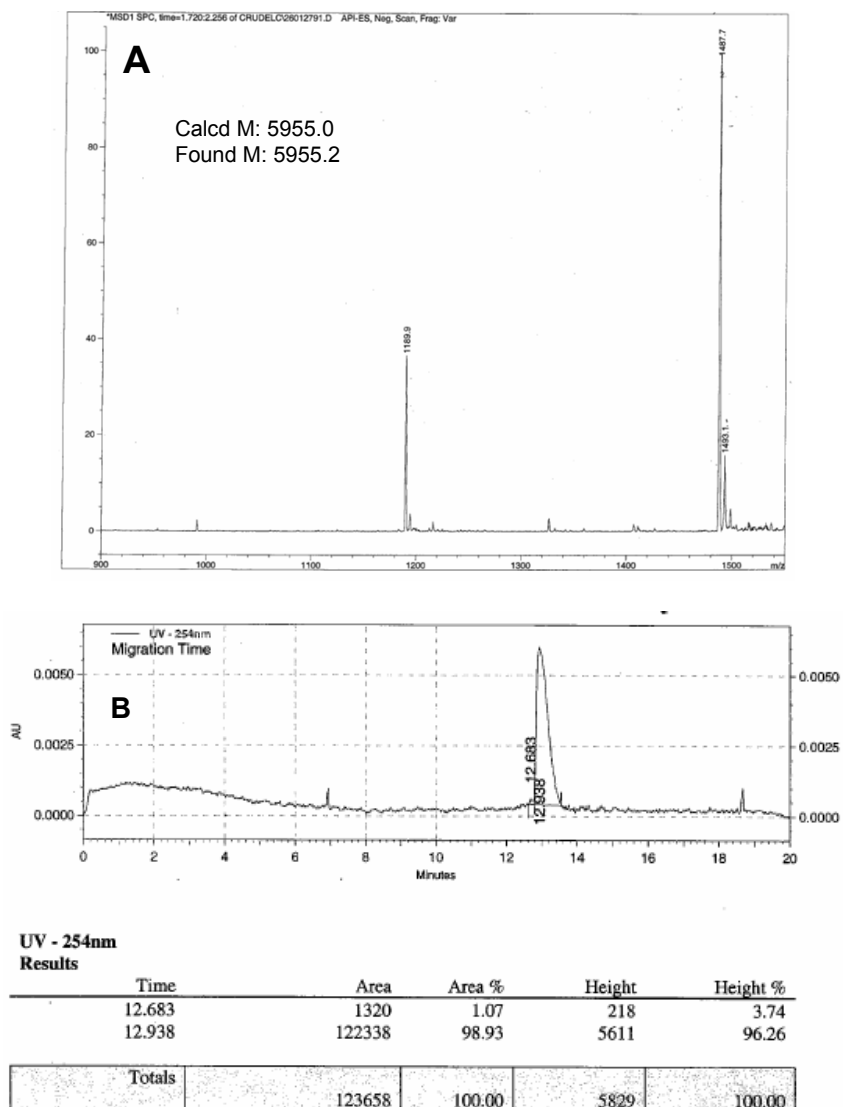


**Figure 2.** Electrospray mass spectrum (A) and capillary electrophoresis profile (B) of oligonucleotide 5'-**AA**GUAAGGACCAGAGAC**AA**-3'.

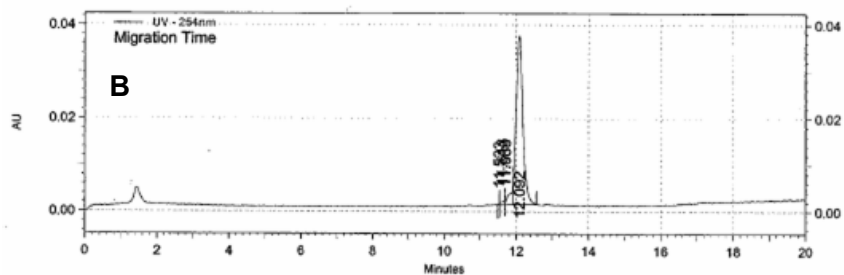
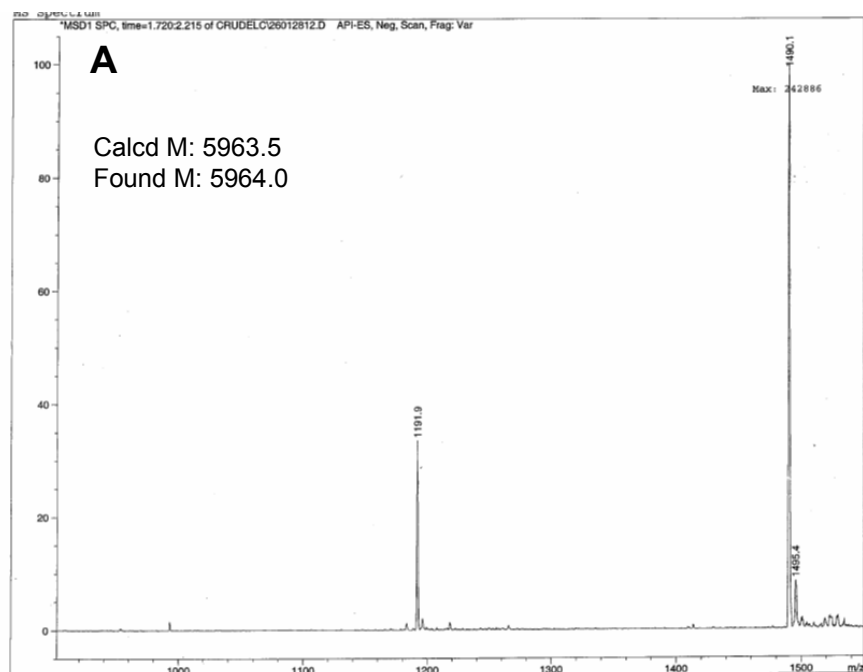


| UV - 254nm Results |        |        |        |          |
|--------------------|--------|--------|--------|----------|
| Time               | Area   | Area % | Height | Height % |
| 11.392             | 1147   | 0.12   | 377    | 0.59     |
| 11.433             | 1025   | 0.11   | 410    | 0.64     |
| 11.475             | 863    | 0.09   | 428    | 0.67     |
| 11.525             | 1021   | 0.11   | 571    | 0.89     |
| 11.550             | 1889   | 0.20   | 735    | 1.14     |
| 11.633             | 4242   | 0.45   | 1198   | 1.86     |
| 11.921             | 859611 | 90.98  | 60477  | 94.11    |
| 12.546             | 75064  | 7.94   | 67     | 0.10     |
| Totals             | 944862 | 100.00 | 64263  | 100.00   |

**Figure 3.** Electrospray mass spectrum (A) and capillary electrophoresis profile (B) of oligonucleotide 5'-*AAGUAAGGACCAGAGACA*-3'.



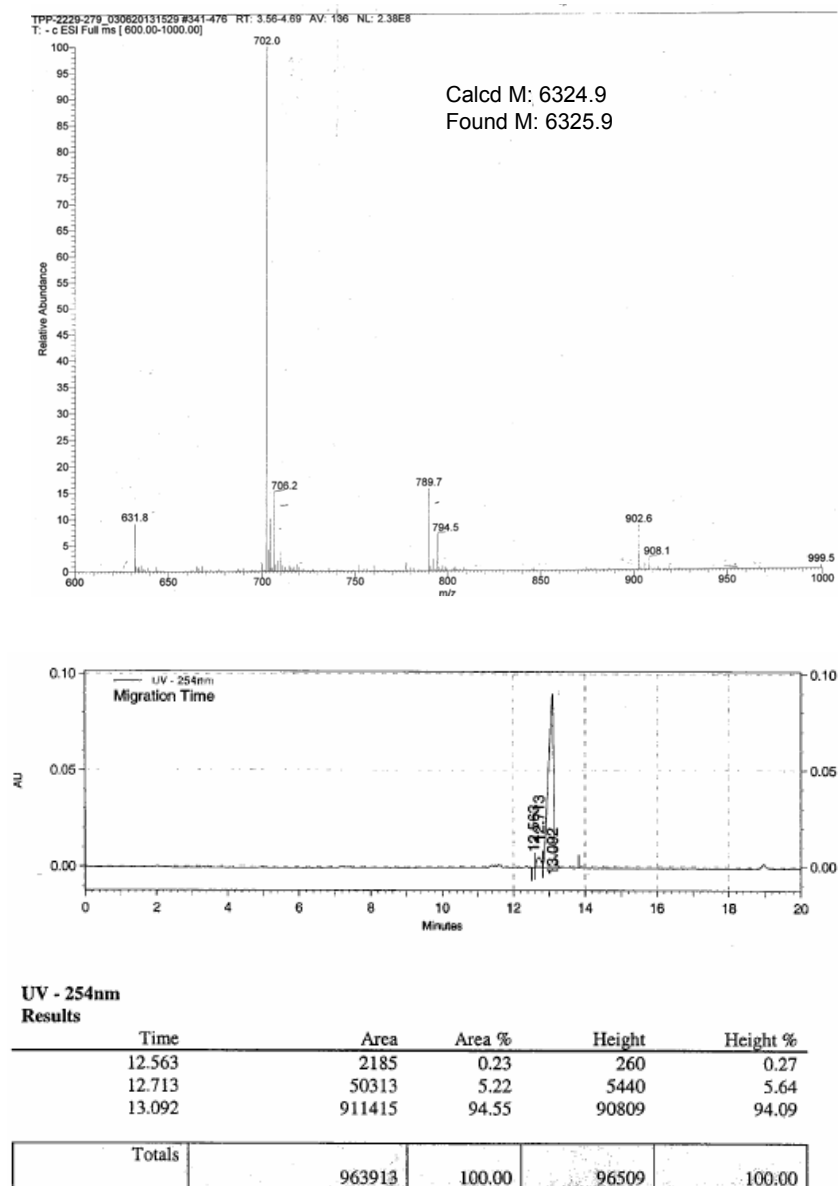
**Figure 4.** Electrospray mass spectrum (A) and capillary electrophoresis profile (B) of oligonucleotide 5'-**UU**CAUCCUGGUCUCUG**UU**-3'.



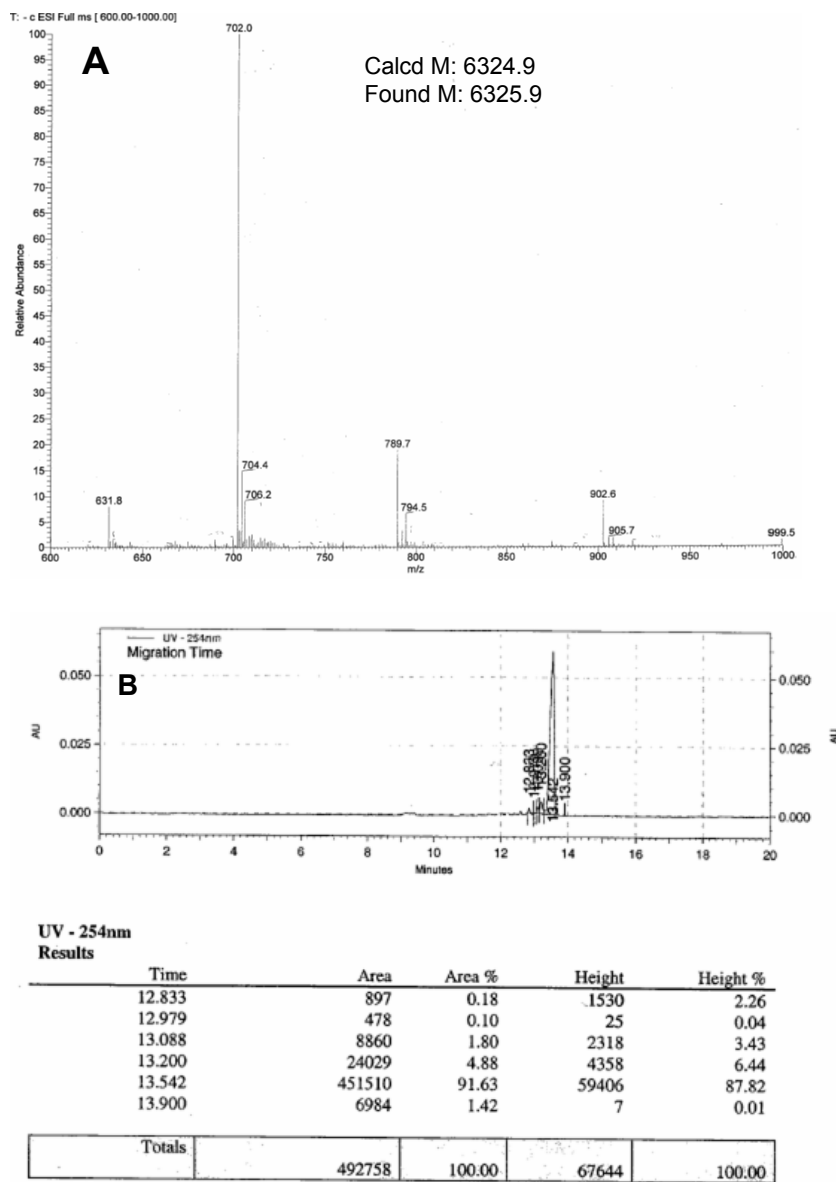
**UV - 254nm  
Results**

| Time   | Area   | Area % | Height | Height % |
|--------|--------|--------|--------|----------|
| 11.533 | 444    | 0.09   | 221    | 0.59     |
| 11.613 | 5560   | 1.08   | 734    | 1.94     |
| 11.688 | 28167  | 5.45   | 555    | 1.47     |
| 12.092 | 482900 | 93.39  | 36258  | 96.00    |
| Totals | 517071 | 100.00 | 37768  | 100.00   |

**Figure 5.** Electrospray mass spectrum (A) and capillary electrophoresis profile (B) of oligonucleotide 3'-*UU*CAUCCUGGUCUCUG*UU*-5'.

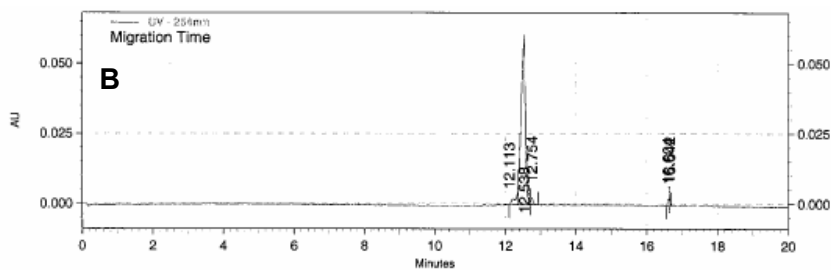
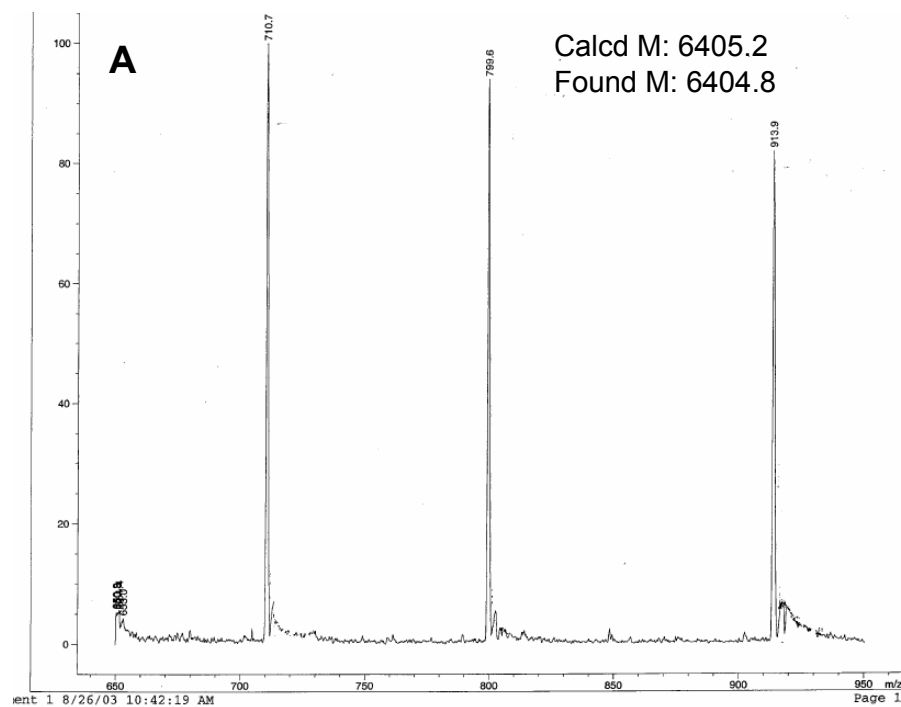


**Figure 6.** Electrospray mass spectrum (A) and capillary electrophoresis profile (B) of oligonucleotide 3'-**UUC**AUCCUGGUCUCUGUUU-P-5'.



**Figure 7.** Electrospray mass spectrum (A) and capillary electrophoresis profile (B) of oligonucleotide 3'-UUCAUUCCUGGUCUCUGUUU-P-5'.

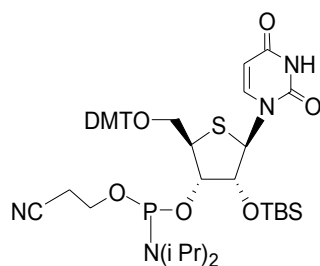




**UV - 254nm  
Results**

| Time   | Area   | Area % | Height | Height % |
|--------|--------|--------|--------|----------|
| 12.113 | 29422  | 5.28   | 6      | 0.01     |
| 12.538 | 505135 | 90.62  | 60756  | 89.20    |
| 12.754 | 10995  | 1.97   | 2517   | 3.70     |
| 16.604 | 6618   | 1.19   | 2250   | 3.30     |
| 16.642 | 5240   | 0.94   | 2584   | 3.79     |
| Totals | 557410 | 100.00 | 68113  | 100.00   |

**Figure 8.** Electrospray mass spectrum (A) and capillary electrophoresis profile (B) of oligonucleotide 3'-**UUCAU**UCCUGGUCUCU**GUUU**-P-5'.



7

AUG-16-2005 17:06

MASS SPEC. UC BERKELEY

510 642 9295 P.03

Elemental Composition

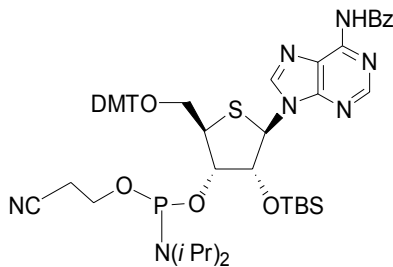
Date : 16-AUG-2005

Heteroatom Max: 40  
Limits:

Ion: Both Even and Odd

| 877.381470 |       | 10.0 |            | -0.5 | 0   | 0   | 0 | 0  | 0  | 0 | 0 | 0 |
|------------|-------|------|------------|------|-----|-----|---|----|----|---|---|---|
|            |       |      |            | 20.0 | 200 | 400 | 6 | 10 | 1  | 1 | 1 | 1 |
| Mass       | PPM   | mDa  | Calc. Mass | DBE  | C   | H   | N | O  | Si | P | S |   |
| 877.381470 | -0.7  | -0.6 | 877.380872 | 18.0 | 47  | 64  | 1 | 9  | 1  | 1 | 1 | 1 |
|            | -2.2  | -1.9 | 877.379529 | 18.5 | 45  | 62  | 4 | 8  | 1  | 1 | 1 | 1 |
|            | 3.7   | 3.2  | 877.384711 | 19.0 | 44  | 60  | 5 | 10 | 1  | 1 | 1 | 1 |
|            | 4.0   | 3.5  | 877.384939 | 19.0 | 45  | 60  | 5 | 9  |    | 1 | 1 | 1 |
|            | 5.5   | 4.8  | 877.386282 | 18.5 | 47  | 62  | 2 | 10 |    | 1 | 1 | 1 |
|            | -6.8  | -6.0 | 877.375507 | 14.5 | 40  | 62  | 6 | 10 | 1  | 1 | 1 | 1 |
|            | -7.2  | -6.3 | 877.375194 | 19.0 | 44  | 59  | 5 | 10 | 1  |   | 1 | 1 |
|            | 7.2   | 6.3  | 877.387770 | 18.5 | 45  | 61  | 4 | 10 | 1  |   | 1 | 1 |
|            | 7.5   | 6.6  | 877.388083 | 14.0 | 41  | 64  | 5 | 10 | 1  | 1 | 1 | 1 |
|            | -8.8  | -7.8 | 877.373706 | 19.0 | 46  | 60  | 3 | 10 |    | 1 | 1 | 1 |
|            | -10.4 | -9.1 | 877.372363 | 19.5 | 44  | 58  | 6 | 9  |    | 1 | 1 | 1 |
|            | 10.6  | 9.3  | 877.390763 | 18.5 | 44  | 62  | 6 | 7  | 1  | 1 | 1 | 1 |
|            | -10.6 | -9.3 | 877.372135 | 19.5 | 43  | 58  | 6 | 10 | 1  | 1 |   |   |

**Figure 9.** High resolution mass spectrum (FAB) of compound 7. HRMS (FAB) calc. for  $C_{45}H_{61}N_4O_8PSSi = 876.3717$ , found = 877.3815  $[M + H]^+$



8

JUL-02-2004 18:02

MASS SPEC. UC BERKELEY

510 642 9295 P.09

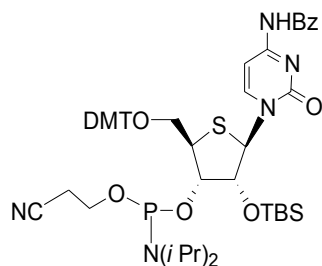
Elemental Composition

Date : 2-JUL-2004

Heteroatom Max: 40 Ion: Both Even and Odd  
Limits:

| Mass        | PPM  | mDa  | Calc. Mass  | DBE  | C  | H  | N | O  | Si | P | S |
|-------------|------|------|-------------|------|----|----|---|----|----|---|---|
| 1004.434280 | 0.0  | 0.0  | 1004.434305 | 25.0 | 55 | 69 | 4 | 8  | 1  | 1 | 1 |
|             | -0.3 | -0.3 | 1004.433992 | 29.5 | 59 | 66 | 3 | 8  | 1  |   | 1 |
|             | 0.4  | 0.4  | 1004.434643 | 38.5 | 67 | 62 | 1 | 6  | 1  |   |   |
|             | 0.4  | 0.4  | 1004.434687 | 35.5 | 60 | 58 | 7 | 8  |    |   |   |
|             | -0.4 | -0.4 | 1004.433841 | 34.5 | 62 | 63 | 5 | 4  |    | 1 | 1 |
|             | 0.6  | 0.6  | 1004.434871 | 38.5 | 68 | 62 | 1 | 5  |    |   | 1 |
|             | -0.7 | -0.7 | 1004.433613 | 34.5 | 61 | 63 | 5 | 5  | 1  | 1 |   |
|             | 0.7  | 0.7  | 1004.434955 | 34.0 | 63 | 65 | 2 | 6  | 1  | 1 |   |
|             | 0.7  | 0.7  | 1004.435000 | 31.0 | 56 | 61 | 8 | 8  |    | 1 |   |
|             | -0.7 | -0.8 | 1004.433529 | 39.0 | 66 | 60 | 4 | 4  |    |   | 1 |
|             | 0.9  | 0.9  | 1004.435184 | 34.0 | 64 | 65 | 2 | 5  |    | 1 | 1 |
|             | -1.0 | -1.0 | 1004.433300 | 39.0 | 65 | 60 | 4 | 5  | 1  |   |   |
|             | 1.0  | 1.0  | 1004.435329 | 34.5 | 60 | 62 | 7 | 4  | 1  |   | 1 |
|             | 1.1  | 1.1  | 1004.435335 | 29.0 | 61 | 68 |   | 9  | 1  |   | 1 |
|             | -1.3 | -1.3 | 1004.432962 | 25.5 | 53 | 67 | 7 | 7  | 1  | 1 | 1 |
|             | 1.4  | 1.4  | 1004.435642 | 30.0 | 56 | 65 | 8 | 4  | 1  | 1 | 1 |
|             | 1.4  | 1.4  | 1004.435647 | 24.5 | 57 | 71 | 1 | 9  | 1  | 1 | 1 |
|             | -1.6 | -1.6 | 1004.432649 | 30.0 | 57 | 64 | 6 | 7  | 1  |   | 1 |
|             | 1.7  | 1.8  | 1004.436030 | 35.0 | 62 | 60 | 4 | 9  |    |   |   |
|             | -1.8 | -1.8 | 1004.432504 | 29.5 | 61 | 67 | 1 | 8  |    | 1 | 1 |
|             | -1.8 | -1.8 | 1004.432499 | 35.0 | 60 | 61 | 8 | 3  |    | 1 | 1 |
|             | -1.8 | -1.8 | 1004.432454 | 38.0 | 67 | 65 | 2 | 1  | 1  | 1 | 1 |
|             | -2.0 | -2.0 | 1004.432276 | 29.5 | 60 | 67 | 1 | 9  | 1  | 1 |   |
|             | -2.0 | -2.0 | 1004.432270 | 35.0 | 59 | 61 | 8 | 4  | 1  | 1 |   |
|             | 2.0  | 2.0  | 1004.436293 | 39.0 | 64 | 61 | 6 | 2  | 1  | 1 |   |
|             | 2.1  | 2.1  | 1004.436343 | 30.5 | 58 | 63 | 5 | 9  |    | 1 |   |
|             | -2.1 | -2.1 | 1004.432191 | 34.0 | 65 | 64 |   | 8  |    |   | 1 |
|             | -2.1 | -2.1 | 1004.432186 | 39.5 | 64 | 58 | 7 | 3  |    |   | 1 |
|             | 2.2  | 2.2  | 1004.436521 | 39.0 | 65 | 61 | 6 | 1  |    | 1 | 1 |
|             | -2.3 | -2.3 | 1004.431963 | 34.0 | 64 | 64 |   | 9  | 1  |   |   |
|             | -2.3 | -2.3 | 1004.431957 | 39.5 | 63 | 58 | 7 | 4  | 1  |   |   |
|             | 2.4  | 2.4  | 1004.436672 | 34.0 | 62 | 64 | 4 | 5  | 1  |   | 1 |
|             | -2.5 | -2.5 | 1004.431812 | 39.0 | 67 | 61 | 2 | 5  |    | 1 |   |
|             | 2.7  | 2.7  | 1004.436985 | 29.5 | 58 | 67 | 5 | 5  | 1  | 1 | 1 |
|             | 3.1  | 3.1  | 1004.437368 | 40.0 | 63 | 56 | 8 | 5  |    |   |   |
|             | 3.1  | 3.1  | 1004.437373 | 34.5 | 64 | 62 | 1 | 10 |    |   |   |
|             | -3.1 | -3.1 | 1004.431161 | 30.0 | 59 | 65 | 4 | 7  |    | 1 | 1 |
|             | -3.2 | -3.2 | 1004.431111 | 38.5 | 65 | 63 | 5 |    | 1  | 1 | 1 |
|             | -3.3 | -3.3 | 1004.430933 | 30.0 | 58 | 65 | 4 | 8  | 1  | 1 |   |
|             | 3.3  | 3.4  | 1004.437635 | 38.5 | 66 | 63 | 3 | 3  | 1  | 1 |   |
|             | 3.4  | 3.4  | 1004.437685 | 30.0 | 60 | 65 | 2 | 10 |    | 1 |   |
|             | -3.4 | -3.4 | 1004.430849 | 34.5 | 63 | 62 | 3 | 7  |    |   | 1 |
|             | 3.5  | 3.6  | 1004.437831 | 30.5 | 56 | 62 | 7 | 9  | 1  |   |   |
|             | 3.6  | 3.6  | 1004.437864 | 38.5 | 67 | 63 | 3 | 2  |    | 1 | 1 |
|             | -3.6 | -3.7 | 1004.430620 | 34.5 | 62 | 62 | 3 | 8  | 1  |   |   |
|             | 3.7  | 3.7  | 1004.438009 | 39.0 | 63 | 60 | 8 | 1  | 1  |   | 1 |
|             | 3.7  | 3.7  | 1004.438015 | 33.5 | 64 | 66 | 1 | 6  | 1  |   | 1 |
|             | 3.8  | 3.8  | 1004.438059 | 30.5 | 57 | 62 | 7 | 8  |    |   | 1 |

**Figure 10.** High resolution mass spectrum (FAB) of compound **8**. HRMS (FAB) calc. for  $C_{53}H_{66}N_7O_7PSSi = 1003.4251$ , found = 1004.434  $[M + H]^+$



14

AUG-16-2005 17:07

MASS SPEC. UC BERKELEY

510 642 9295 P.09

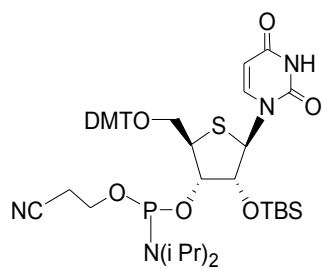
Elemental Composition

Date : 16-AUG-2005

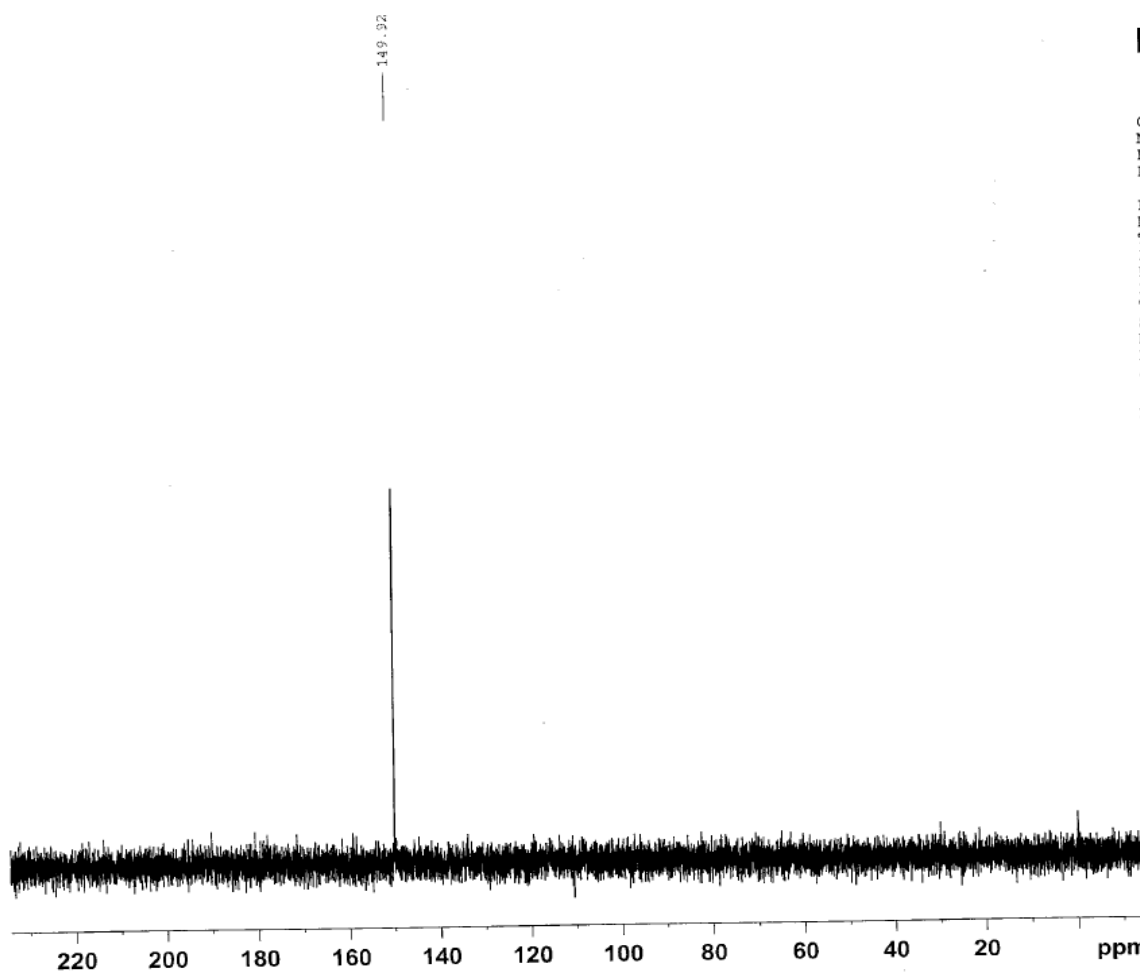
Heteroatom Max: 40 Ion: Both Even and Odd  
Limits:

| Mass       | PPM  | mDa  | Calc. Mass | DBE  | C  | H  | N | O  | Si | P | S |
|------------|------|------|------------|------|----|----|---|----|----|---|---|
| 980.419920 | 0.0  | 0.0  | 980.419928 | 28.0 | 58 | 65 | 2 | 8  |    | 1 | 1 |
|            | 0.0  | 0.0  | 980.419878 | 36.5 | 64 | 63 | 3 | 1  | 1  | 1 | 1 |
|            | -0.2 | -0.2 | 980.419699 | 28.0 | 57 | 65 | 2 | 9  | 1  | 1 | 1 |
|            | -0.3 | -0.3 | 980.419615 | 32.5 | 62 | 62 | 1 | 8  |    |   | 1 |
|            | -0.5 | -0.5 | 980.419387 | 32.5 | 61 | 62 | 1 | 9  | 1  |   |   |
|            | 0.7  | 0.7  | 980.420579 | 37.0 | 66 | 61 |   | 6  |    | 1 |   |
|            | -0.7 | -0.7 | 980.419236 | 37.5 | 64 | 59 | 3 | 5  |    | 1 |   |
|            | 0.8  | 0.8  | 980.420724 | 37.5 | 62 | 58 | 5 | 5  | 1  |   |   |
|            | 1.1  | 1.0  | 980.420953 | 37.5 | 63 | 58 | 5 | 4  |    |   | 1 |
|            | 1.1  | 1.1  | 980.421036 | 33.0 | 58 | 61 | 6 | 5  | 1  | 1 |   |
|            | 1.3  | 1.3  | 980.421220 | 36.0 | 66 | 65 | 2 | 1  | 1  | 1 | 1 |
|            | -1.4 | -1.3 | 980.418585 | 28.5 | 56 | 63 | 5 | 7  |    | 1 | 1 |
|            | 1.4  | 1.3  | 980.421265 | 33.0 | 59 | 61 | 6 | 4  |    | 1 | 1 |
|            | -1.4 | -1.4 | 980.418535 | 37.0 | 62 | 61 | 6 |    | 1  | 1 | 1 |
|            | 1.5  | 1.5  | 980.421416 | 28.0 | 56 | 64 | 4 | 8  | 1  |   | 1 |
|            | -1.6 | -1.6 | 980.418356 | 28.5 | 55 | 63 | 5 | 8  | 1  | 1 | 1 |
|            | -1.7 | -1.6 | 980.418273 | 33.0 | 60 | 60 | 4 | 7  |    |   | 1 |
|            | 1.8  | 1.8  | 980.421728 | 23.5 | 52 | 67 | 5 | 8  | 1  | 1 | 1 |
|            | -1.9 | -1.9 | 980.418044 | 33.0 | 59 | 60 | 4 | 8  | 1  |   |   |
|            | -2.1 | -2.0 | 980.417893 | 38.0 | 62 | 57 | 6 | 4  |    | 1 |   |
|            | 2.2  | 2.1  | 980.422067 | 37.0 | 64 | 60 | 2 | 6  | 1  |   |   |
|            | 2.4  | 2.4  | 980.422296 | 37.0 | 65 | 60 | 2 | 5  |    |   | 1 |
|            | 2.5  | 2.5  | 980.422379 | 32.5 | 60 | 63 | 3 | 6  | 1  | 1 |   |
|            | -2.6 | -2.5 | 980.417393 | 24.0 | 51 | 64 | 6 | 10 | 1  |   | 1 |
|            | 2.7  | 2.7  | 980.422608 | 32.5 | 61 | 63 | 3 | 5  |    | 1 | 1 |
|            | -2.8 | -2.7 | 980.417198 | 32.0 | 61 | 65 | 2 | 4  | 1  | 1 | 1 |
|            | 2.9  | 2.8  | 980.422759 | 27.5 | 58 | 66 | 1 | 9  | 1  |   | 1 |
|            | -3.1 | -3.0 | 980.416885 | 36.5 | 65 | 62 | 1 | 4  | 1  |   | 1 |
|            | 3.2  | 3.2  | 980.423071 | 23.0 | 54 | 69 | 2 | 9  | 1  | 1 | 1 |
|            | -3.4 | -3.4 | 980.416556 | 33.0 | 61 | 61 | 2 | 8  |    | 1 |   |
|            | 3.6  | 3.5  | 980.423454 | 33.5 | 59 | 58 | 5 | 9  |    |   |   |
|            | -3.7 | -3.7 | 980.416243 | 37.5 | 65 | 58 | 1 | 8  |    |   |   |
|            | 3.9  | 3.8  | 980.423722 | 32.0 | 62 | 65 |   | 7  | 1  | 1 |   |
|            | 3.9  | 3.8  | 980.423767 | 29.0 | 55 | 61 | 6 | 9  |    | 1 |   |
|            | -4.1 | -4.0 | 980.415905 | 24.0 | 53 | 65 | 4 | 10 |    | 1 | 1 |
|            | 4.1  | 4.0  | 980.423951 | 32.0 | 63 | 65 |   | 6  |    | 1 | 1 |
|            | -4.1 | -4.1 | 980.415855 | 32.5 | 59 | 63 | 5 | 3  | 1  | 1 | 1 |
|            | 4.3  | 4.2  | 980.424096 | 32.5 | 59 | 62 | 5 | 5  | 1  |   | 1 |
|            | -4.4 | -4.3 | 980.415593 | 28.5 | 57 | 62 | 3 | 10 |    |   | 1 |
|            | -4.5 | -4.4 | 980.415543 | 37.0 | 63 | 60 | 4 | 3  | 1  |   | 1 |
|            | 4.6  | 4.5  | 980.424408 | 28.0 | 55 | 65 | 6 | 5  | 1  | 1 | 1 |
|            | -4.8 | -4.7 | 980.415213 | 33.5 | 59 | 59 | 5 | 7  |    | 1 |   |
|            | 5.0  | 4.9  | 980.424797 | 33.0 | 61 | 60 | 2 | 10 |    |   |   |
|            | -5.1 | -5.0 | 980.414901 | 38.0 | 63 | 56 | 4 | 7  |    |   |   |
|            | 5.2  | 5.1  | 980.425059 | 37.0 | 63 | 61 | 4 | 3  | 1  | 1 |   |
|            | 5.3  | 5.2  | 980.425109 | 28.5 | 57 | 63 | 3 | 10 |    | 1 |   |
|            | 5.5  | 5.4  | 980.425288 | 37.0 | 64 | 61 | 4 | 2  |    | 1 | 1 |
|            | -5.5 | -5.4 | 980.414518 | 27.5 | 58 | 67 | 1 | 7  | 1  | 1 | 1 |
|            | 5.6  | 5.5  | 980.425439 | 32.0 | 61 | 64 | 2 | 6  | 1  |   | 1 |
|            | -5.8 | -5.7 | 980.414250 | 29.0 | 55 | 60 | 6 | 9  |    |   | 1 |
|            | -5.8 | -5.7 | 980.414205 | 32.0 | 62 | 64 |   | 7  | 1  |   | 1 |

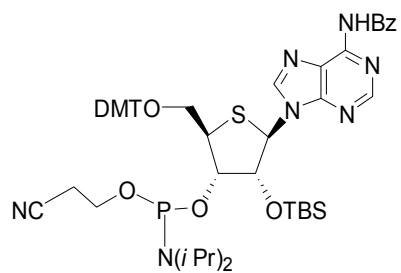
**Figure 11.** High resolution mass spectrum (FAB) of compound **14**. HRMS (FAB) calc. for  $C_{52}H_{66}N_5O_8PSSi = 979.4139$ , found = 980.4199  $[M + H]^+$



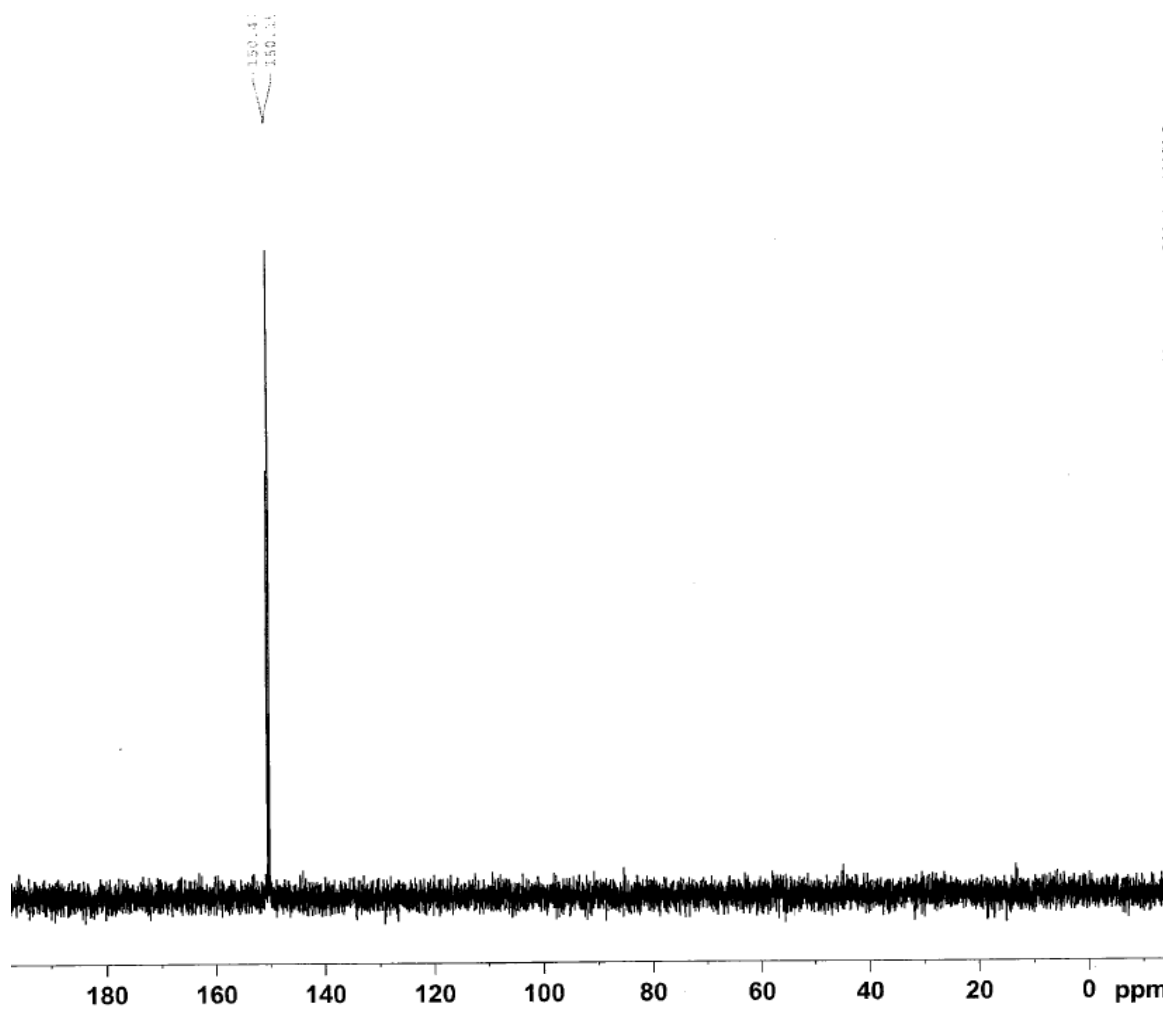
7



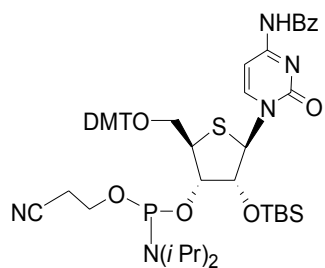
**Figure 12.**  $^{31}\text{P}$  NMR of compound **7** in  $\text{CDCl}_3$ .  $^{31}\text{P}$  NMR spectra were performed using external standard 85%  $\text{H}_3\text{PO}_4$



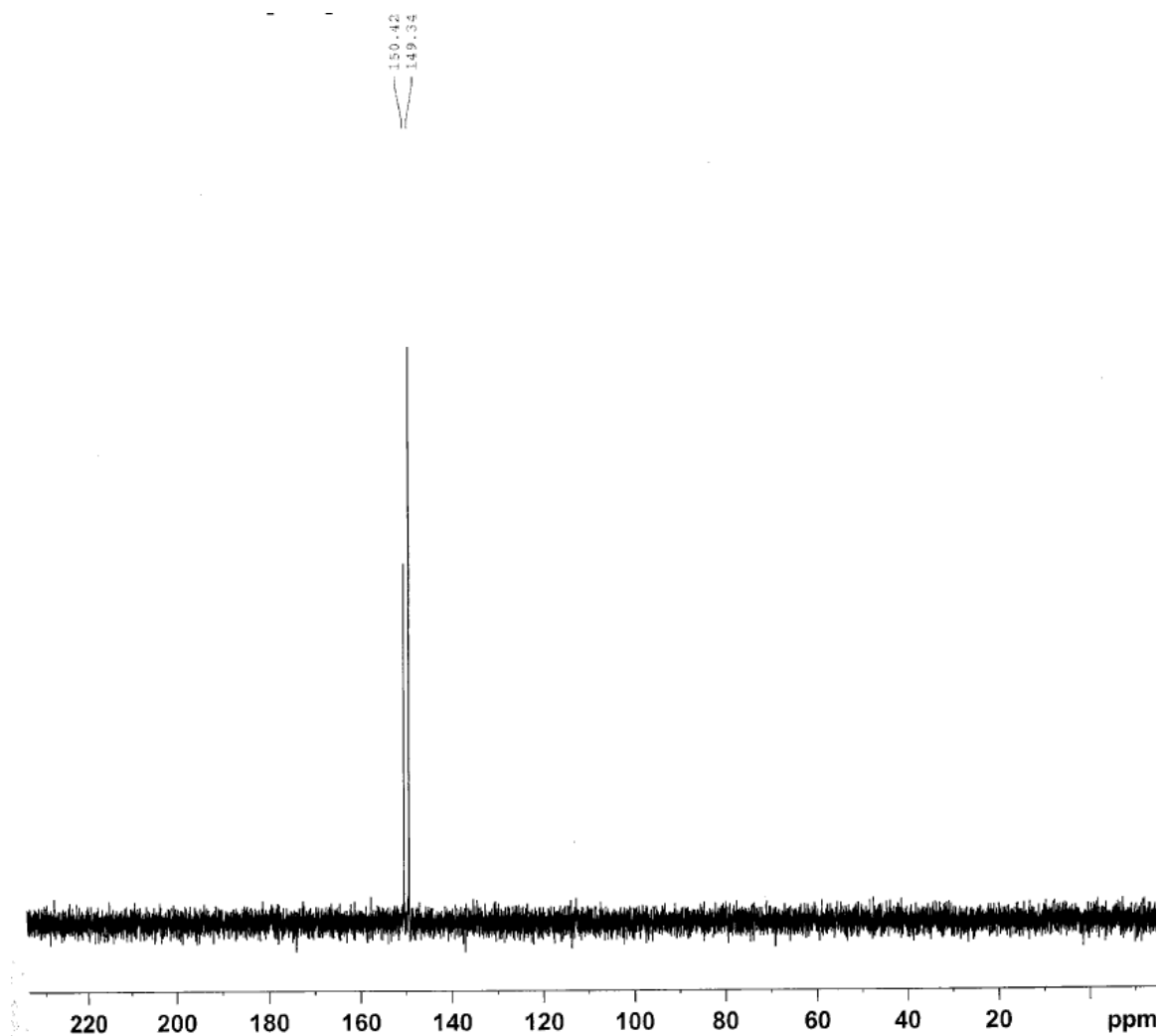
**8**



**Figure 13.**  $^{31}\text{P}$  NMR of compound **8** in  $\text{CDCl}_3$ .  $^{31}\text{P}$  NMR spectra were performed using external standard 85%  $\text{H}_3\text{PO}_4$



**14**



**Figure 14.**  $^{31}\text{P}$  NMR of compound **14** in  $\text{CDCl}_3$ .  $^{31}\text{P}$  NMR spectra were performed using external standard 85%  $\text{H}_3\text{PO}_4$