

Investigation of 8-aza-7-deaza purine nucleoside derivatives

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HNMR DMSO

Chemical structure: CN1=NC(=C(NC1CO)CO)OC

Peak list (ppm): 7.906, 6.867, 5.982, 5.970, 5.320, 5.306, 5.062, 5.048, 4.757, 4.743, 4.728, 4.534, 4.521, 4.508, 4.495, 4.183, 4.170, 4.158, 4.145, 3.991, 3.977, 3.862, 3.848, 3.561, 3.545, 3.532, 3.442, 3.427, 3.412, 3.398.

Integration values: 1.00, 1.98, 1.00, 1.03, 1.03, 1.04, 1.11, 1.04, 1.03, 1.04, 1.24, 1.04.

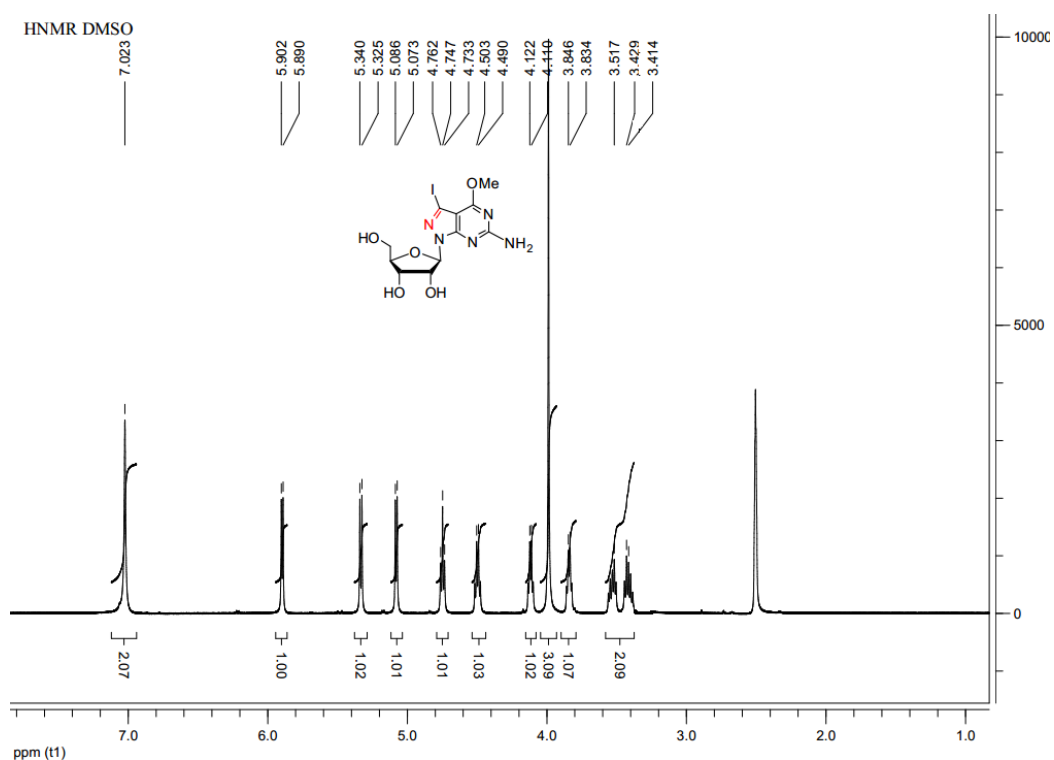
¹H NMR DMSO

Chemical structure: CN1C=NC2=C1N=CN2[C@@H]3O[C@H](CO)[C@@H](O)[C@H]3O

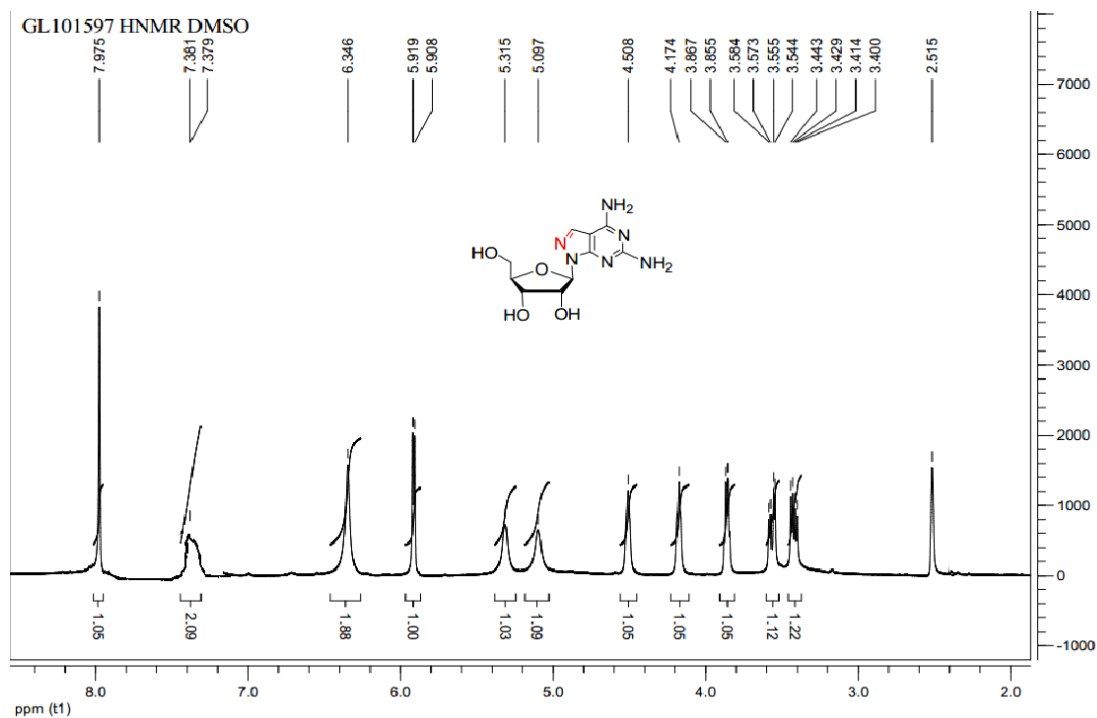
Chemical shifts (ppm): 8.535, 6.467, 5.756, 5.748, 4.320, 4.312, 4.309, 4.300, 4.168, 4.155, 4.142, 3.972, 3.711, 3.702, 3.681, 3.672, 3.561, 3.550, 3.531, 3.520, 3.171, 2.505.

Integration values: 1.01, 2.00, 1.03, 1.09, 1.09, 4.14, 1.08, 1.05, 1.08, 1.09.

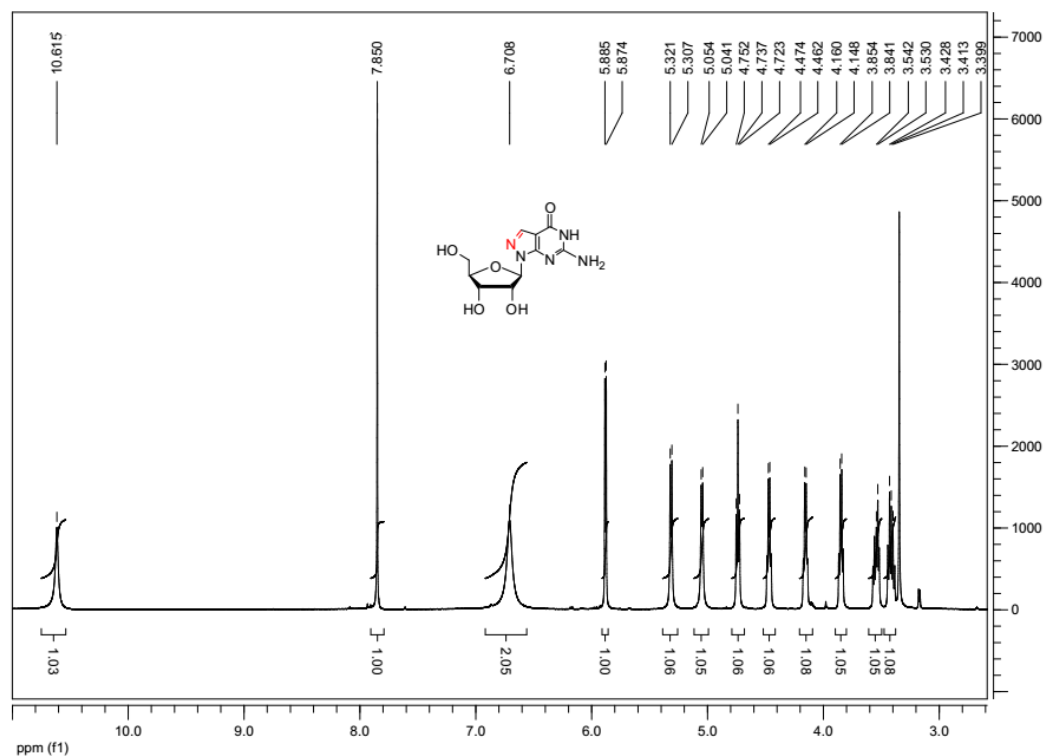
Product 3: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



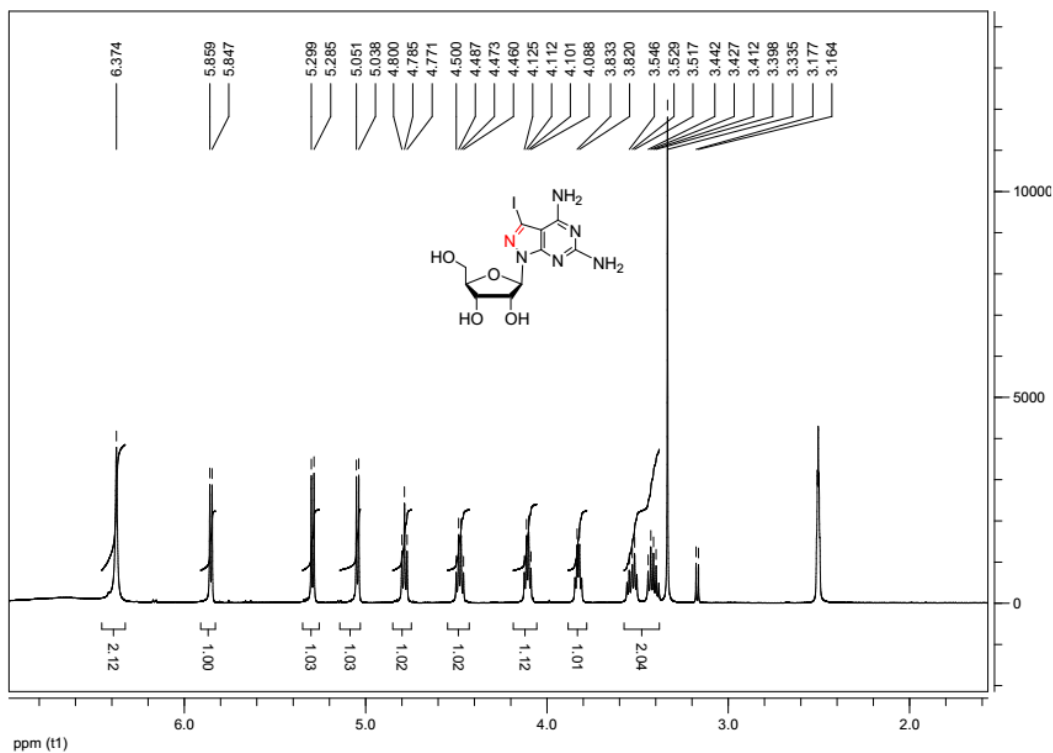
Product 5: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



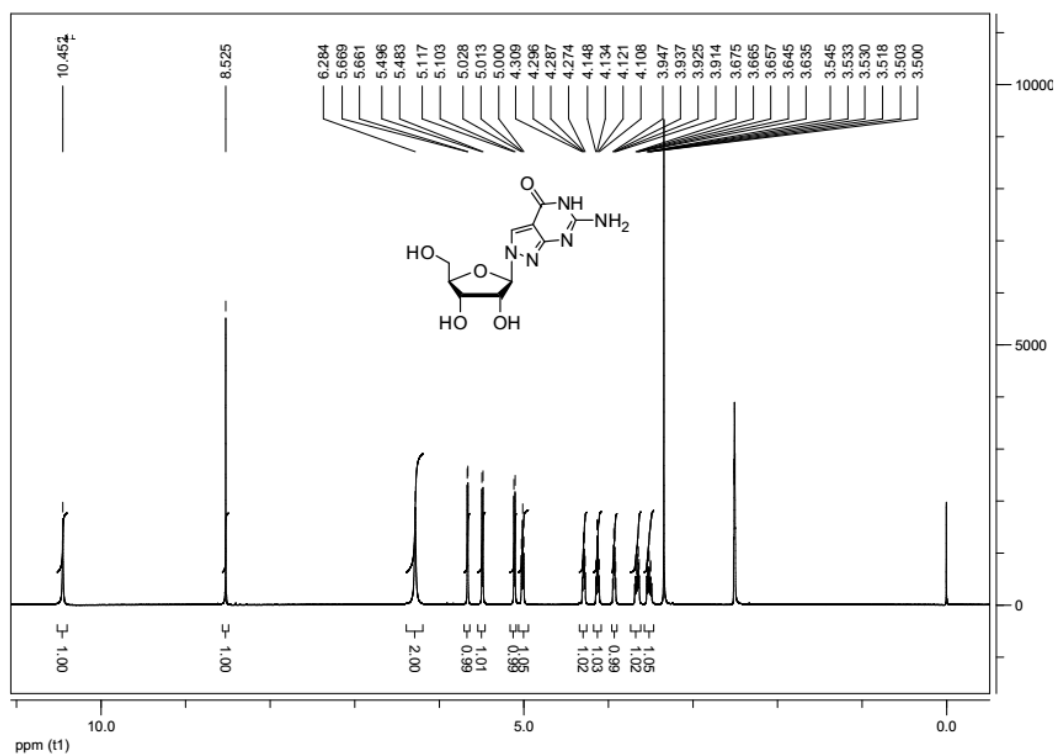
Product 7: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



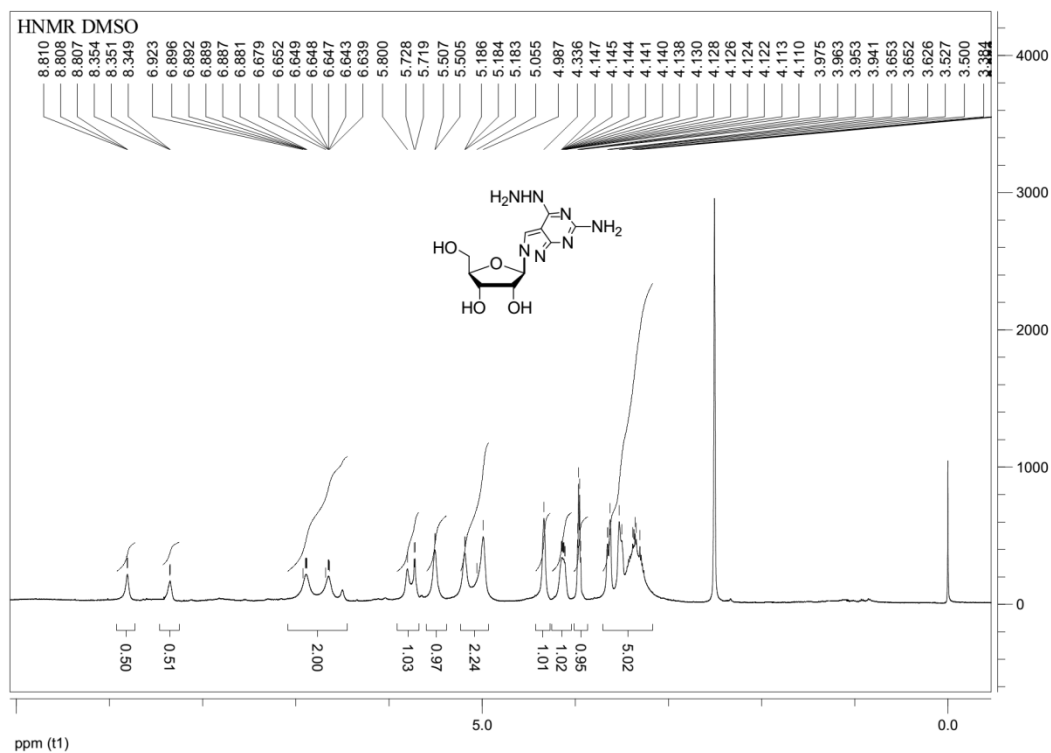
Product 8: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



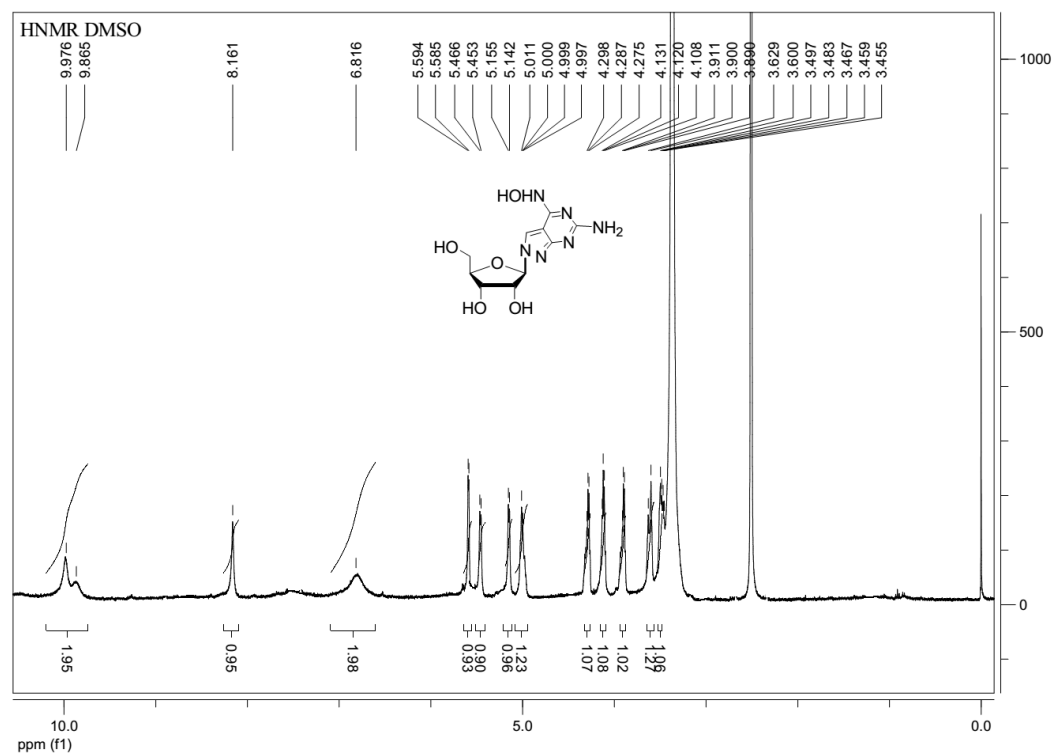
Product 10: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



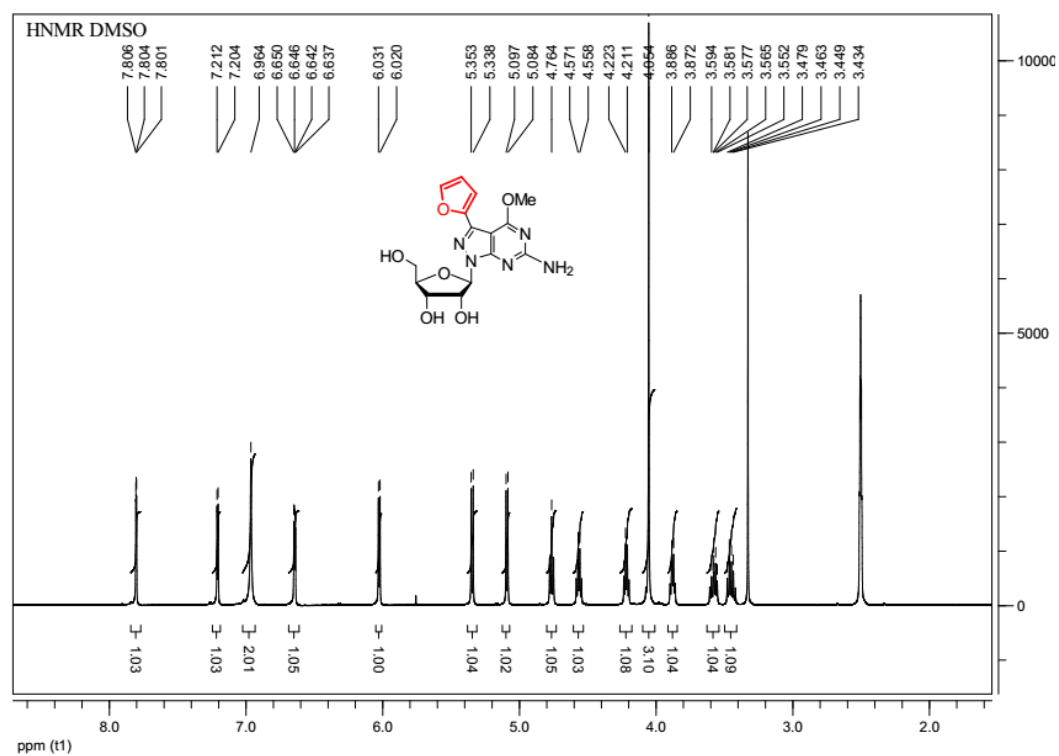
Product 12: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



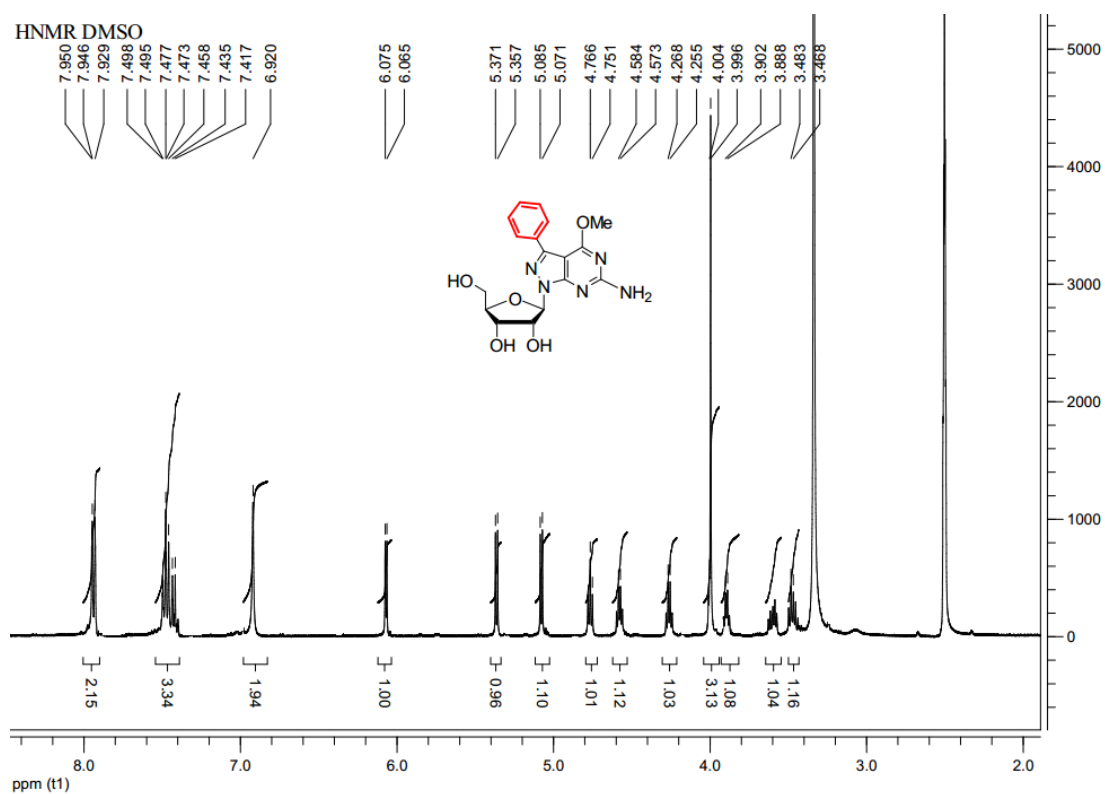
Product 13: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



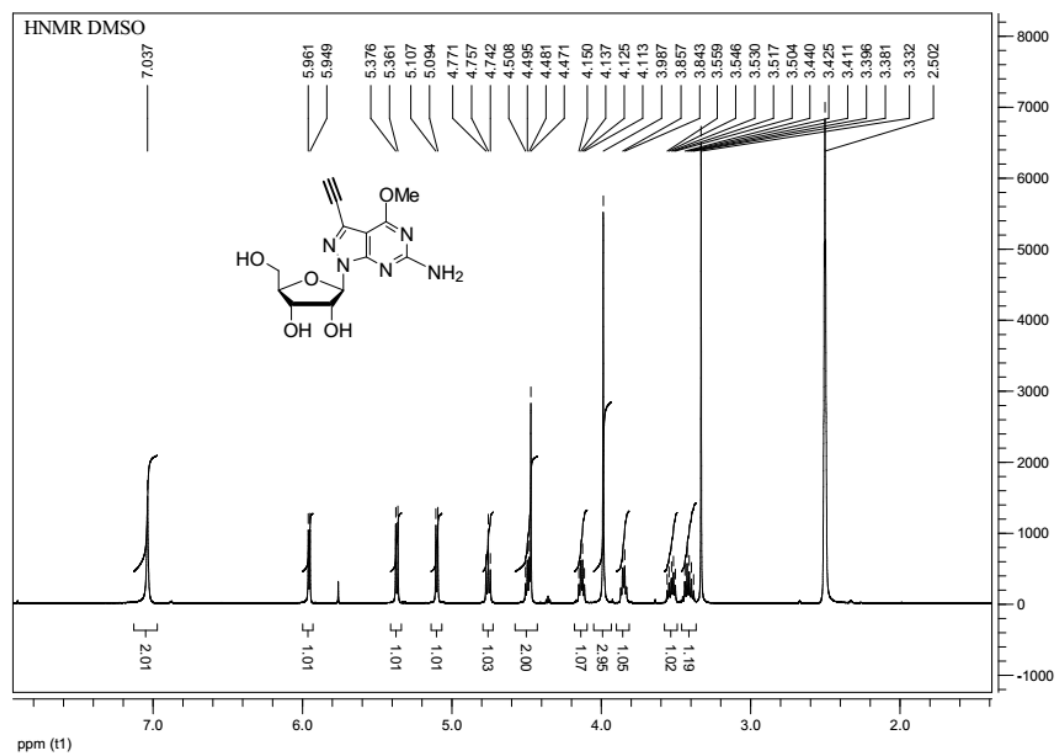
Product 14: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



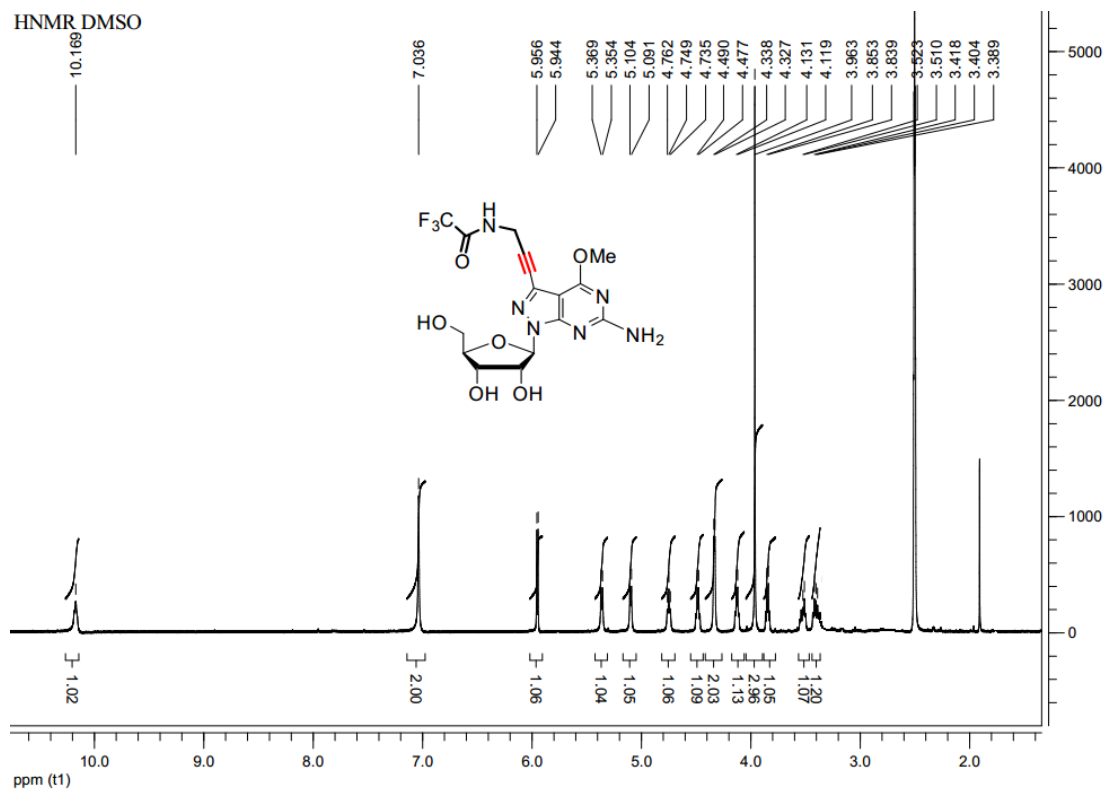
Product 15: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



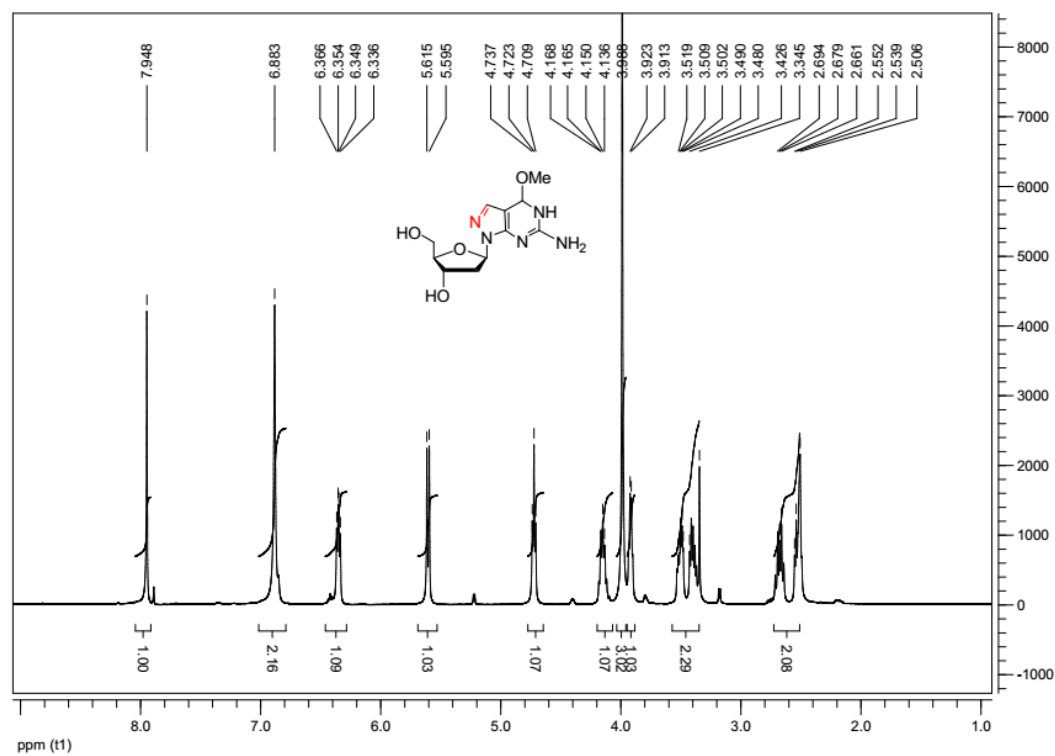
Product 16: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



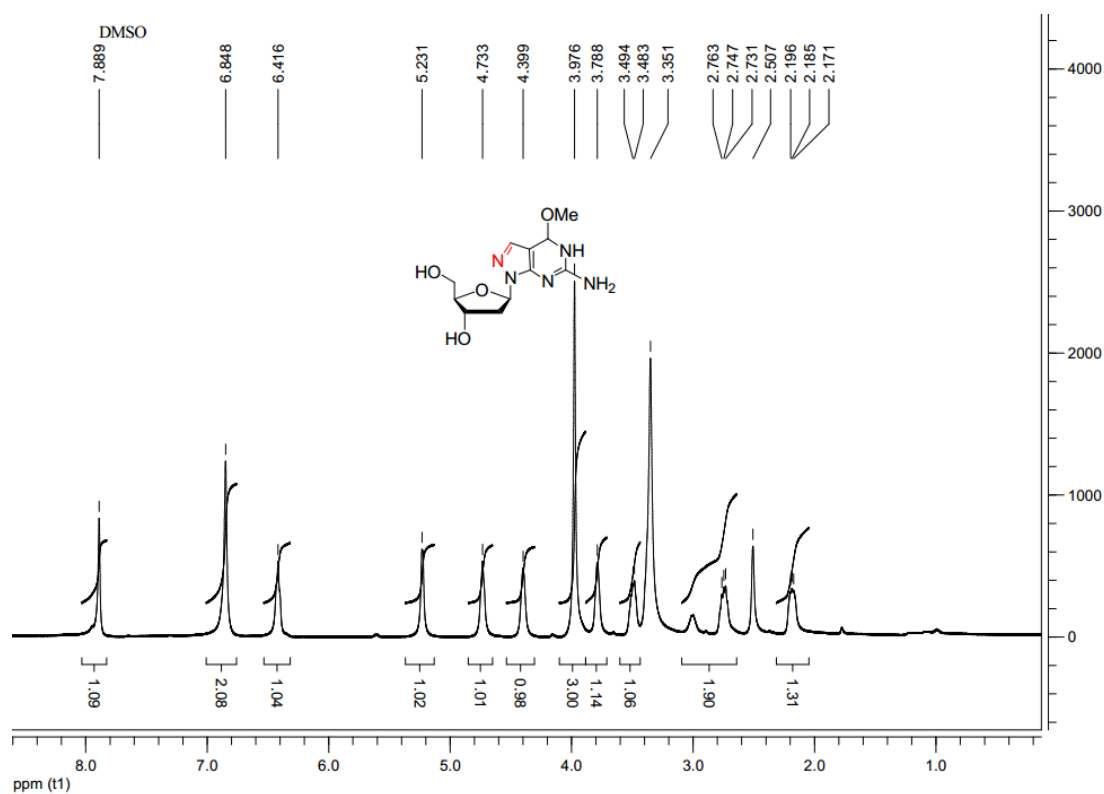
Product 17: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



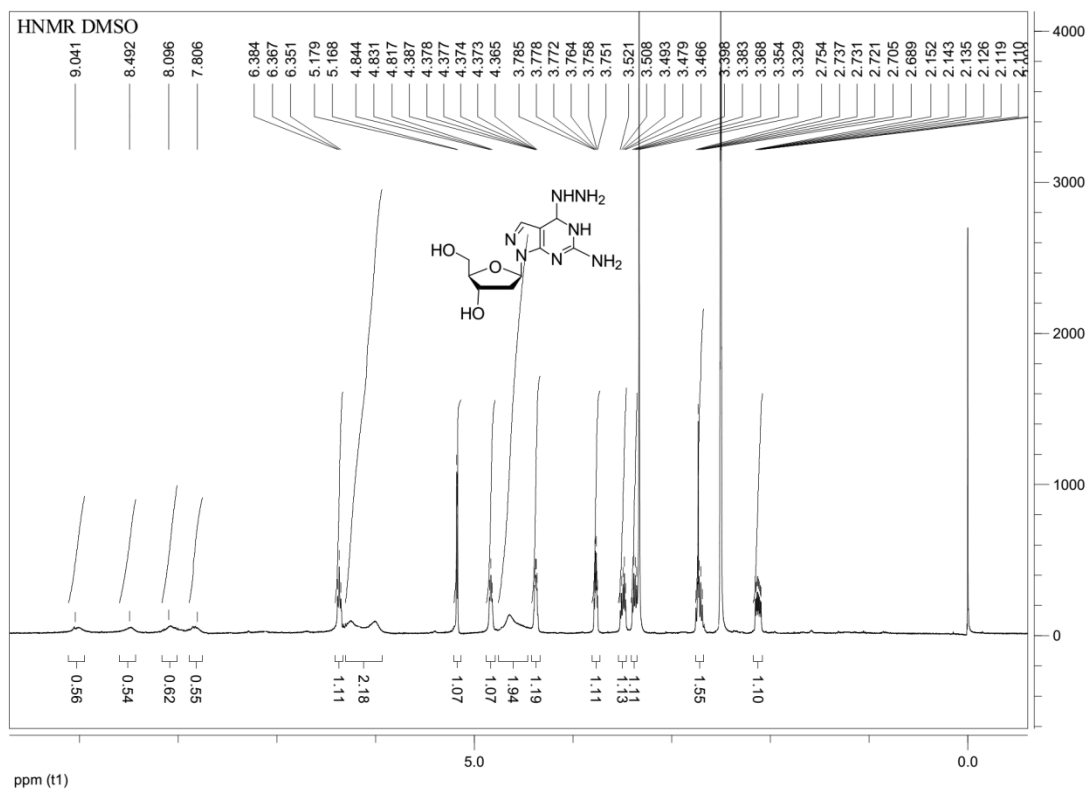
Product 18: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



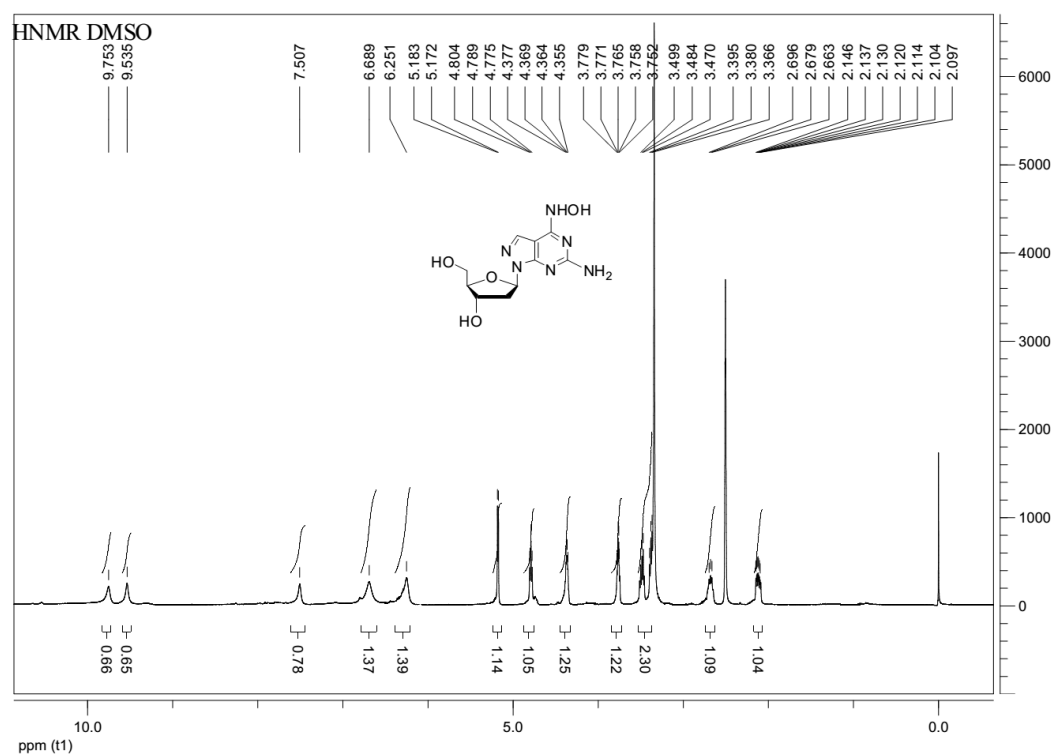
Product 19: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



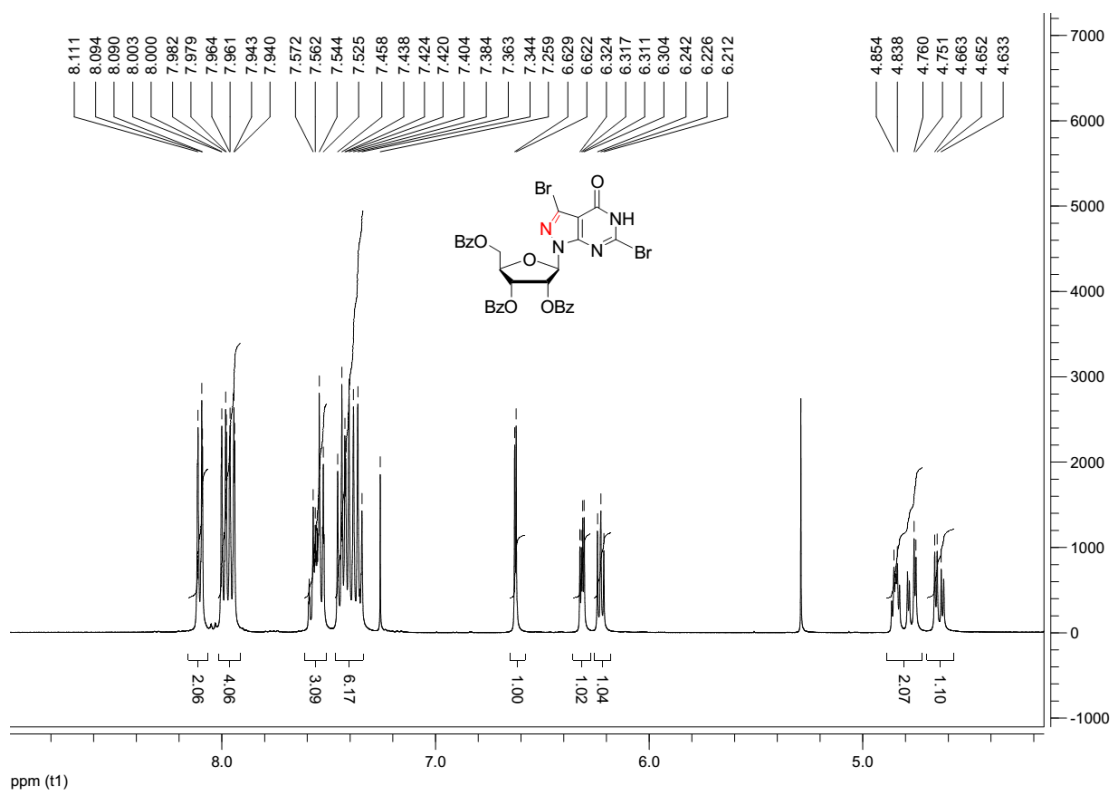
Product 22: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



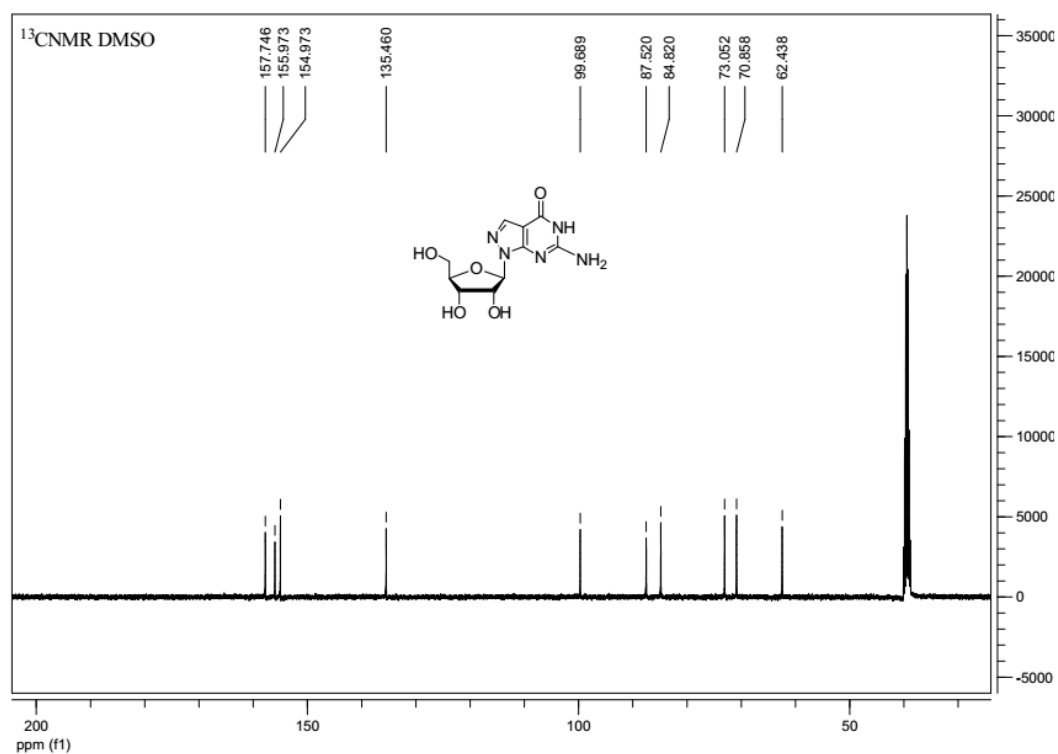
Product 23: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



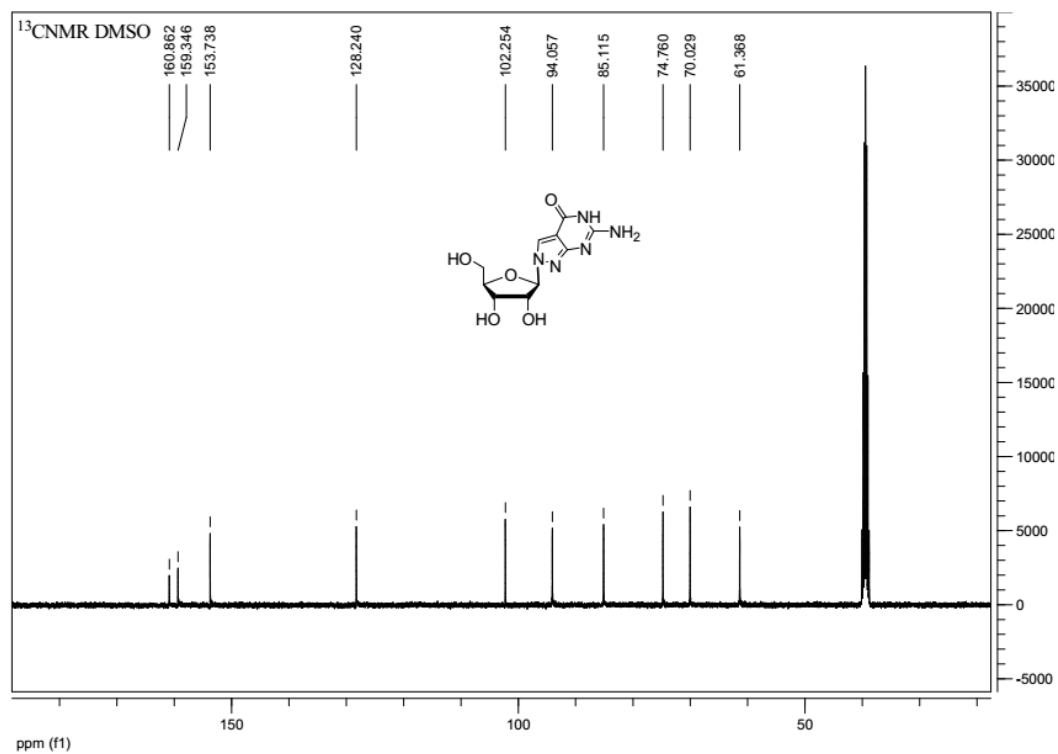
Product 27: ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



Product 7: ^{13}C NMR (400 MHz, $\text{DMSO}-d_6$)



Product 10: ^{13}C NMR (400 MHz, $\text{DMSO}-d_6$)

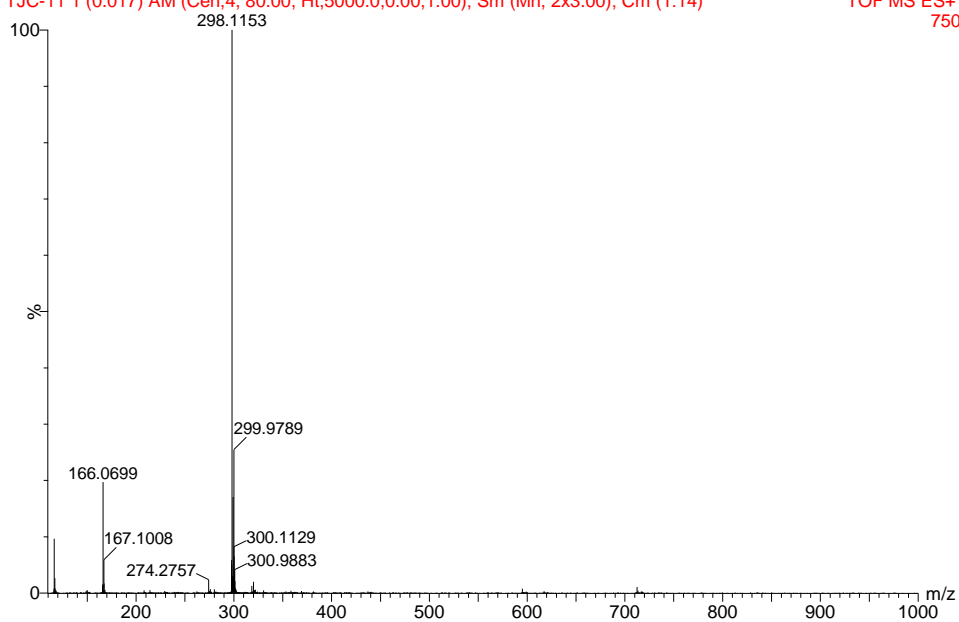


Product 2: HRMS spectra

2

TJC-11 1 (0.017) AM (Cen,4, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x3.00); Cm (1:14)

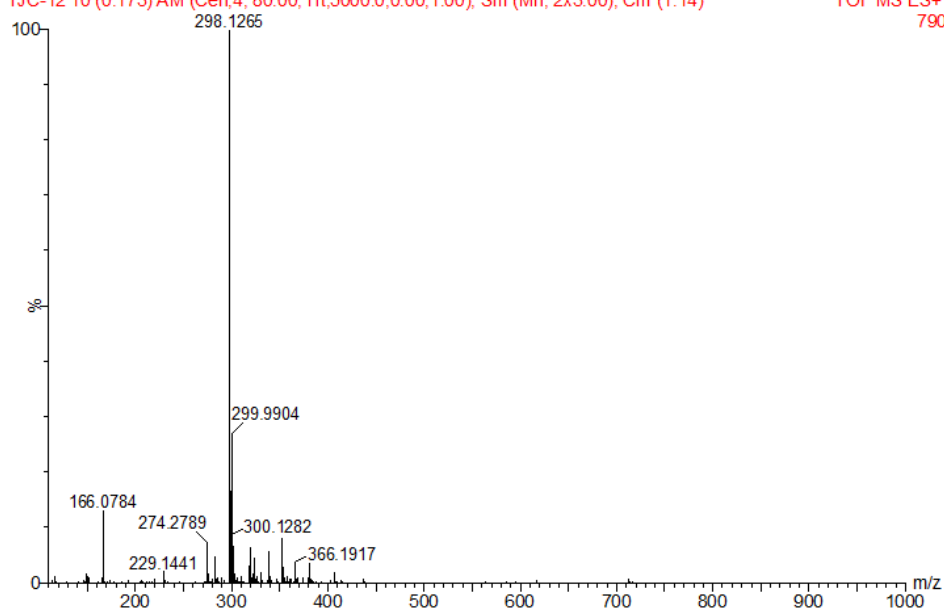
TOF MS ES+
750



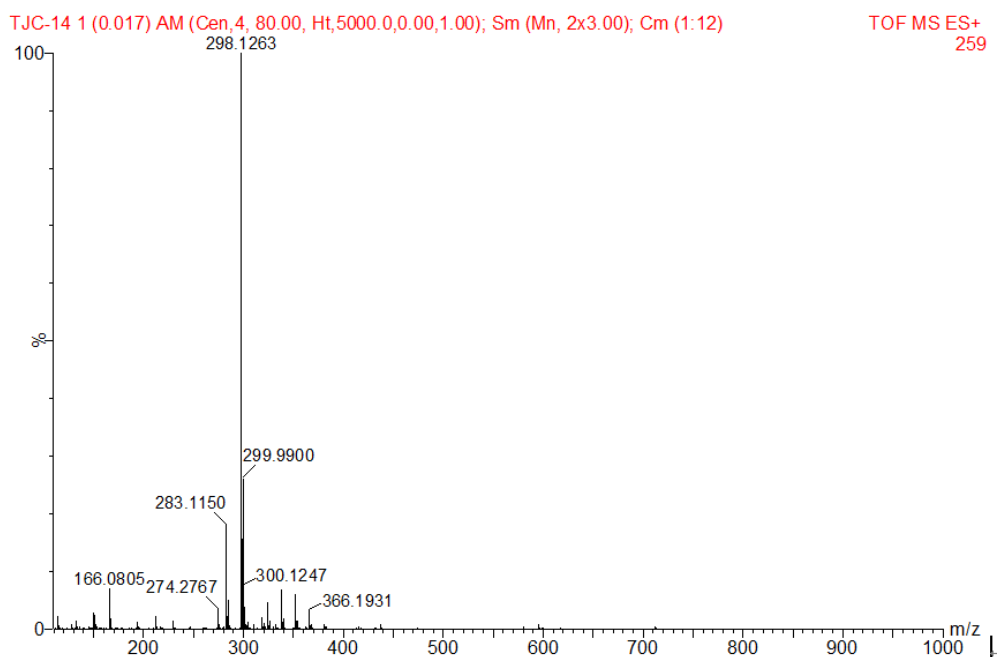
Product 6: HRMS spectra

TJC-12 10 (0.173) AM (Cen,4, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x3.00); Cm (1:14)

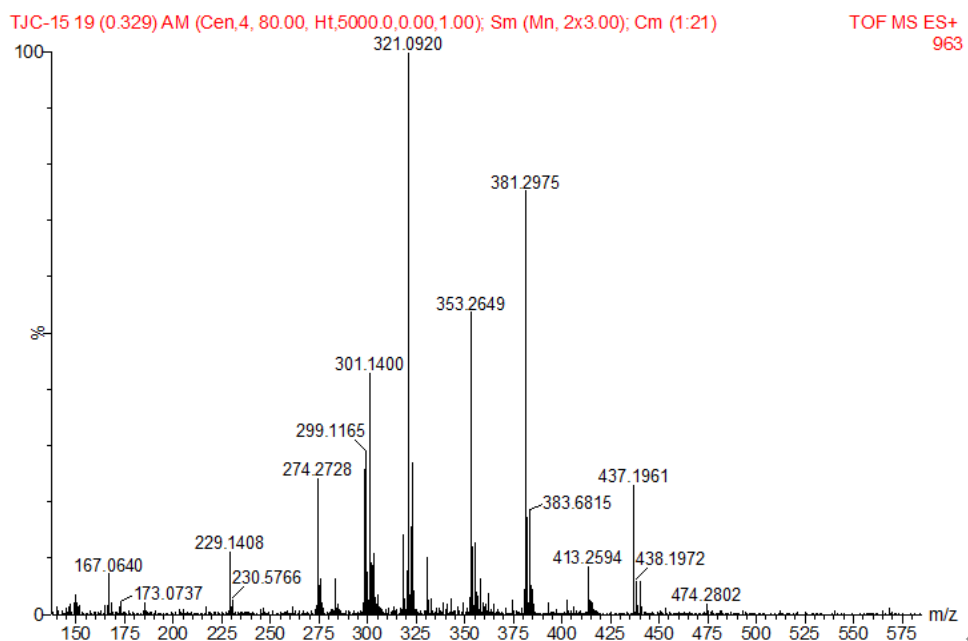
TOF MS ES+
790



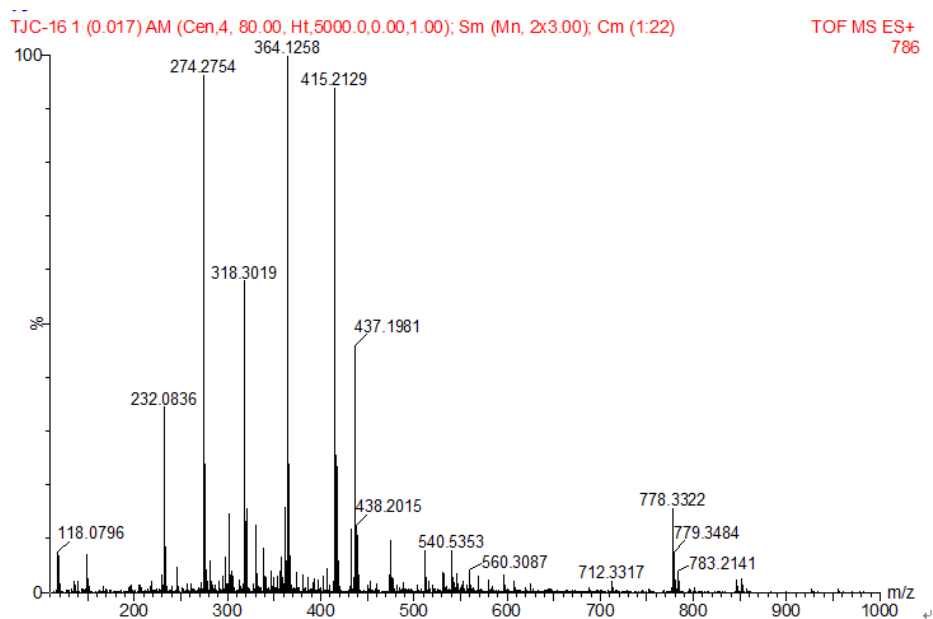
Product 12: HRMS spectra



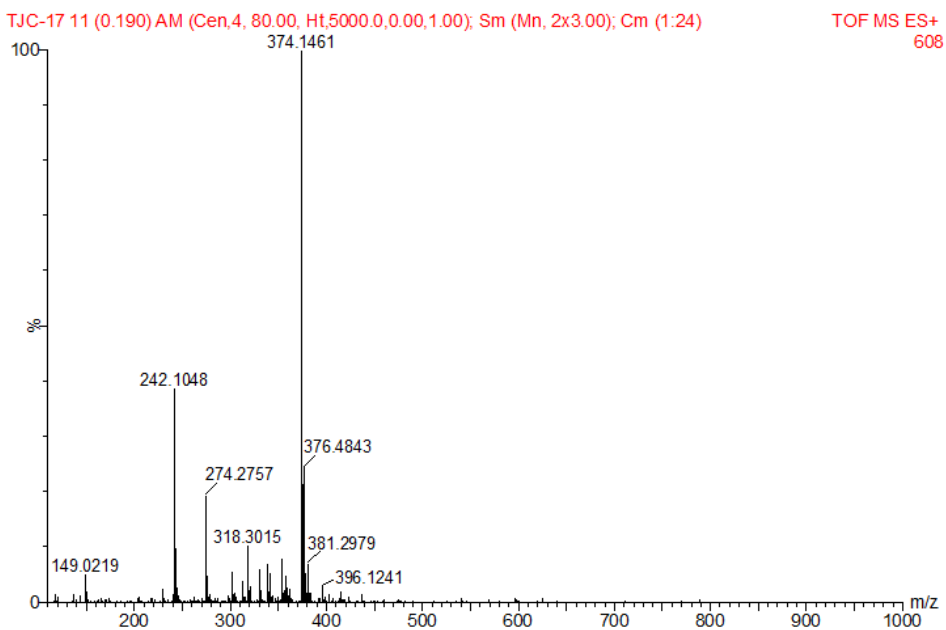
Product 13: HRMS spectra



Product 14: HRMS spectra



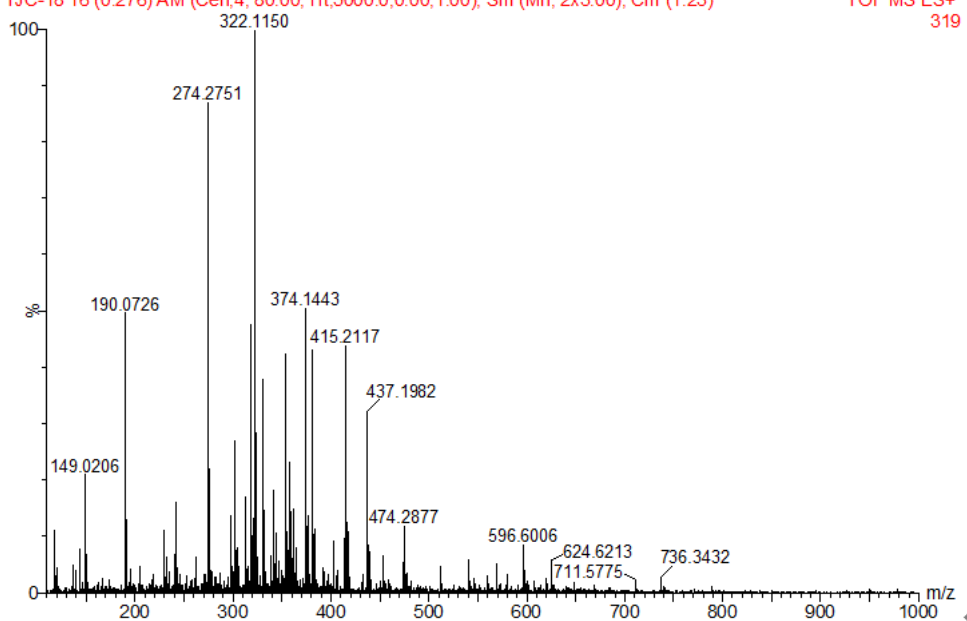
Product 15: HRMS spectra



Product 16: HRMS spectra

TJC-18 16 (0.276) AM (Cen.4, 80.00, Ht.5000.0,0.00,1.00); Sm (Mn, 2x3.00); Cm (1:23)

TOF MS ES+
319



Crystal data and structure refinement for compound 7

| | |
|---|--|
| Identification code | 201804211 |
| Empirical formula | C ₁₀ H ₁₃ N ₅ O ₅ |
| Formula weight | 283.25 |
| Temperature/K | 293(2) |
| Crystal system | orthorhombic |
| Space group | P2 ₁ 2 ₁ 2 ₁ |
| a/Å | 4.7669(2) |
| b/Å | 11.0409(5) |
| c/Å | 21.6334(9) |
| α /° | 90 |
| β /° | 90 |
| γ /° | 90 |
| Volume/Å ³ | 1138.59(8) |
| Z | 4 |
| $\rho_{\text{calc}}/\text{cm}^3$ | 1.652 |
| μ/mm^{-1} | 1.157 |
| F(000) | 592.0 |
| Crystal size/mm ³ | 0.15 × 0.11 × 0.1 |
| Radiation | CuK α (λ = 1.54184) |
| 2 Θ range for data collection/° | 8.174 to 134.138 |
| Index ranges | -3 ≤ h ≤ 5, -13 ≤ k ≤ 13, -25 ≤ l ≤ 25 |
| Reflections collected | 4100 |
| Independent reflections | 2032 [R_{int} = 0.0304, R_{sigma} = 0.0405] |
| Data/restraints/parameters | 2032/0/184 |
| Goodness-of-fit on F ² | 1.036 |
| Final R indexes [$I \geq 2\sigma(I)$] | R_1 = 0.0366, wR_2 = 0.0880 |
| Final R indexes [all data] | R_1 = 0.0408, wR_2 = 0.0924 |
| Largest diff. peak/hole / e Å ⁻³ | 0.14/-0.21 |
| Flack parameter | 0.2(2) |

Crystal data and structure refinement for compound 8

| | |
|---|--|
| Identification code | 201804210 |
| Empirical formula | C ₁₀ H ₁₅ IN ₆ O ₅ |
| Formula weight | 426.18 |
| Temperature/K | 293(2) |
| Crystal system | monoclinic |
| Space group | P2 ₁ |
| a/Å | 5.08845(13) |
| b/Å | 12.7514(2) |
| c/Å | 11.3200(2) |
| α/° | 90 |
| β/° | 91.4146(19) |
| γ/° | 90 |
| Volume/Å ³ | 734.28(3) |
| Z | 2 |
| ρ _{calc} /cm ³ | 1.928 |
| μ/mm ⁻¹ | 17.478 |
| F(000) | 420.0 |
| Crystal size/mm ³ | 0.15 × 0.13 × 0.11 |
| Radiation | CuKα (λ = 1.54184) |
| 2θ range for data collection/° | 7.812 to 134.094 |
| Index ranges | -3 ≤ h ≤ 6, -15 ≤ k ≤ 15, -13 ≤ l ≤ 12 |
| Reflections collected | 5309 |
| Independent reflections | 2616 [R _{int} = 0.0300, R _{sigma} = 0.0391] |
| Data/restraints/parameters | 2616/3/213 |
| Goodness-of-fit on F ² | 1.062 |
| Final R indexes [I ≥ 2σ (I)] | R ₁ = 0.0286, wR ₂ = 0.0701 |
| Final R indexes [all data] | R ₁ = 0.0296, wR ₂ = 0.0711 |
| Largest diff. peak/hole / e Å ⁻³ | 0.56/-0.62 |
| Flack parameter | -0.021(5) |
