

Supporting Information to Accompany: Characterization of the 4-(Benzothiazol-2-yl)phenylnitrenium Ion from a Putative Metabolite of a Model Anti-Tumor Drug

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Table S1. Rate constants for the decomposition of **8** at 30 °C collected by uv methods

| Reaction Conditions | pH | Wavelength (nm) and $10^3 k_o$ (s ⁻¹) ^a | Wavelength (nm) And $10^4 k_1$ (s ⁻¹) ^b |
|---------------------|------|--|--|
| Phosphate buffer | 7.30 | 319, 2.08 ± 0.01 250, 2.34 ± 0.02 221, 2.07 ± 0.02 Avg 2.16 ± 0.15 | 272, 4.21 ± 0.06 250, 2.02 ± 0.21 221, 2.63 ± 0.42 Avg 2.95 ± 1.13 |
| Phosphate buffer | 6.96 | 319, 2.20 ± 0.01 250, 2.29 ± 0.01 221, 2.15 ± 0.12 Avg 2.21 ± 0.07 | 272, 5.36 ± 0.09 250, 5.30 ± 0.10 Avg 5.33 ± 0.04 |
| Phosphate buffer | 6.52 | 319, 2.08 ± 0.01 272, 2.48 ± 0.09 250, 2.20 ± 0.01 Avg 2.25 ± 0.21 | 272, 7.60 ± 0.13 250, 7.29 ± 0.08 Avg 7.45 ± 0.22 |
| Phosphate buffer | 6.00 | 319, 2.41 ± 0.01 250, 2.73 ± 0.03 Avg 2.57 ± 0.23 | 272, 7.78 ± 0.15 250, 8.25 ± 0.12 221, 10.4 ± 0.08 Avg 8.82 ± 1.41 |
| Phosphate buffer | 5.57 | 319, 1.99 ± 0.01 272, 2.77 ± 0.02 250, 2.34 ± 0.01 221, 2.54 ± 0.03 Avg 2.41 ± 0.33 | 272, 8.17 ± 0.13 250, 8.50 ± 0.20 221, 9.53 ± 0.31 Avg 8.73 ± 0.71 |
| Acetate buffer | 4.66 | 319, 2.00 ± 0.01 250, 2.45 ± 0.03 215, 1.92 ± 0.20 Avg 2.12 ± 0.29 | 319, 5.83 ± 0.10 250, 6.51 ± 0.08 215, 11.3 ± 0.11 Avg 7.89 ± 3.00 |
| Formate buffer | 4.03 | 319, 2.21 ± 0.01 | 319, 8.37 ± 0.28 |

| | | | |
|---------------------------|------|--|--|
| | | 250, 2.43 ± 0.01 215, 2.42 ± 0.05 Avg 2.35 ± 0.12 | 274, 7.78 ± 0.07 250, 5.92 ± 0.03 220, 6.10 ± 0.06 215, 5.82 ± 0.12 Avg 6.80 ± 1.19 |
| Formate buffer | 3.50 | 319, 2.26 ± 0.03 250, 2.40 ± 0.01 221, 2.66 ± 0.12 Avg 2.44 ± 0.20 | 319, 7.94 ± 0.60 272, 5.95 ± 0.06 250, 5.37 ± 0.04 221, 6.18 ± 0.07 Avg 6.36 ± 1.11 |
| Formate buffer | 3.01 | 319, 2.02 ± 0.01 274, 3.11 ± 0.07 250, 2.21 ± 0.01 220, 2.26 ± 0.08 215, 2.04 ± 0.04 Avg 2.33 ± 0.45 | 319, 4.54 ± 0.13 274, 5.08 ± 0.05 250, 4.47 ± 0.02 220, 4.76 ± 0.05 215, 4.99 ± 0.10 Avg 4.77 ± 0.27 |
| 0.003 M HClO ₄ | 2.53 | 319, 2.07 ± 0.01 275, 2.67 ± 0.09 250, 2.08 ± 0.01 210, 1.81 ± 0.01 Avg 2.16 ± 0.36 | 319, 3.22 ± 0.09 275, 2.97 ± 0.03 250, 2.99 ± 0.02 221, 3.94 ± 0.21 210, 3.98 ± 0.07 Avg 3.42 ± 0.50 |
| 0.01 M HClO ₄ | 2.01 | 322, 1.68 ± 0.01 250, 1.69 ± 0.01 214, 1.58 ± 0.02 Avg 1.65 ± 0.06 | 322, 2.79 ± 0.08 250, 2.58 ± 0.01 220, 2.51 ± 0.01 214, 2.96 ± 0.04 Avg 2.71 ± 0.21 |
| 0.03 M HClO ₄ | 1.51 | 328, 1.19 ± 0.01 272, 1.21 ± 0.01 | 328, 2.91 ± 0.19 272, 1.92 ± 0.04 |

| | | | |
|---------------------------------|------|---|---|
| | | 250, 1.20 ± 0.01 210, 1.09 ± 0.02 Avg 1.17 ± 0.06 | 250, 2.02 ± 0.02 219, 2.37 ± 0.03 210, 2.76 ± 0.14 Avg 2.40 ± 0.44 |
| 0.1 M HClO ₄ | 1.01 | 349, 0.593 ± 0.002 250, 0.619 ± 0.004 219, 0.695 ± 0.018 213, 0.575 ± 0.021 Avg 0.621 ± 0.061 | ^c |
| Phosphate buffer 10.0 mM d-G | 7.1 | 344, 2.25 ± 0.03 314, 1.86 ± 0.01 Avg 2.06 ± 0.27 | |
| Phosphate buffer 5.0 mM d-G | 7.1 | 344, 2.27 ± 0.03 314, 1.95 ± 0.01 Avg 2.11 ± 0.23 | |
| Phosphate buffer 2.5 mM d-G | 7.1 | 344, 2.20 ± 0.03 314, 2.05 ± 0.01 Avg 2.13 ± 0.11 | |

^aRate constant for the decomposition of **8**.

^bRate constant for the hydrolysis of **16** into **15**.

^cHydrolysis of **16** is detectable, but the product **15** slowly decomposes under the conditions

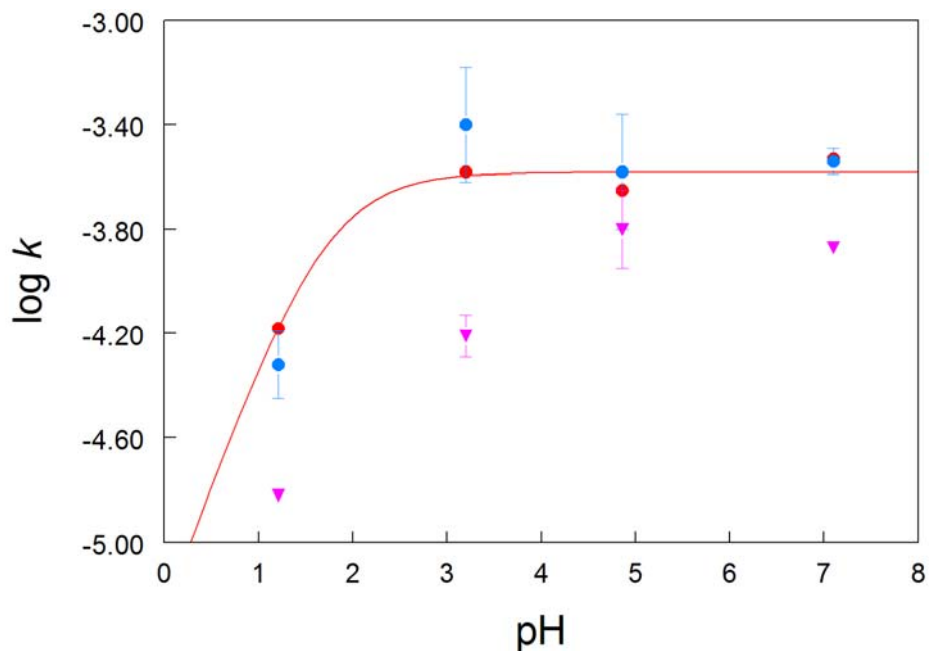


Figure S1. Rate constants obtained by HPLC methods for the disappearance of **8** and formation of **15** at 10 °C. Key: (●) Average k_0 for the disappearance of **8** obtained from a fit of HPLC peak area data for **8** at 212 nm and 330 nm to a first-order rate equation; (●) k_0 obtained from a fit of HPLC peak area data for the appearance of **15** at 212 nm to a double exponential rate equation; (▼) k_1 obtained from a fit of HPLC peak area data for the appearance of **15** at 212 nm to a double exponential rate equation. The k_0 from **8** were fit to eq 1 to obtain $k_0' = 2.6 \times 10^{-4} \text{ s}^{-1}$ and $\text{p}K_a = 1.7$.

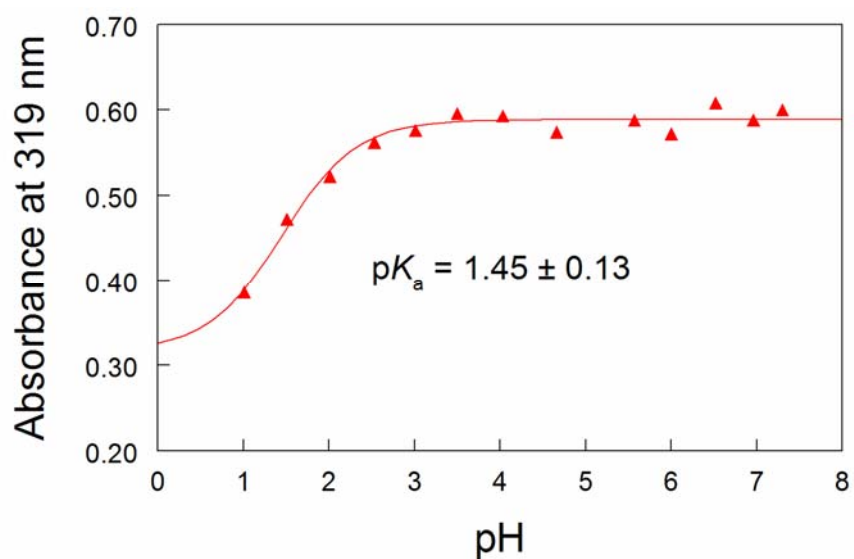


Figure S2. Initial absorbance of **8** at 319 nm vs. pH. Data were fit to a standard titration equation to obtain $\text{p}K_a$.

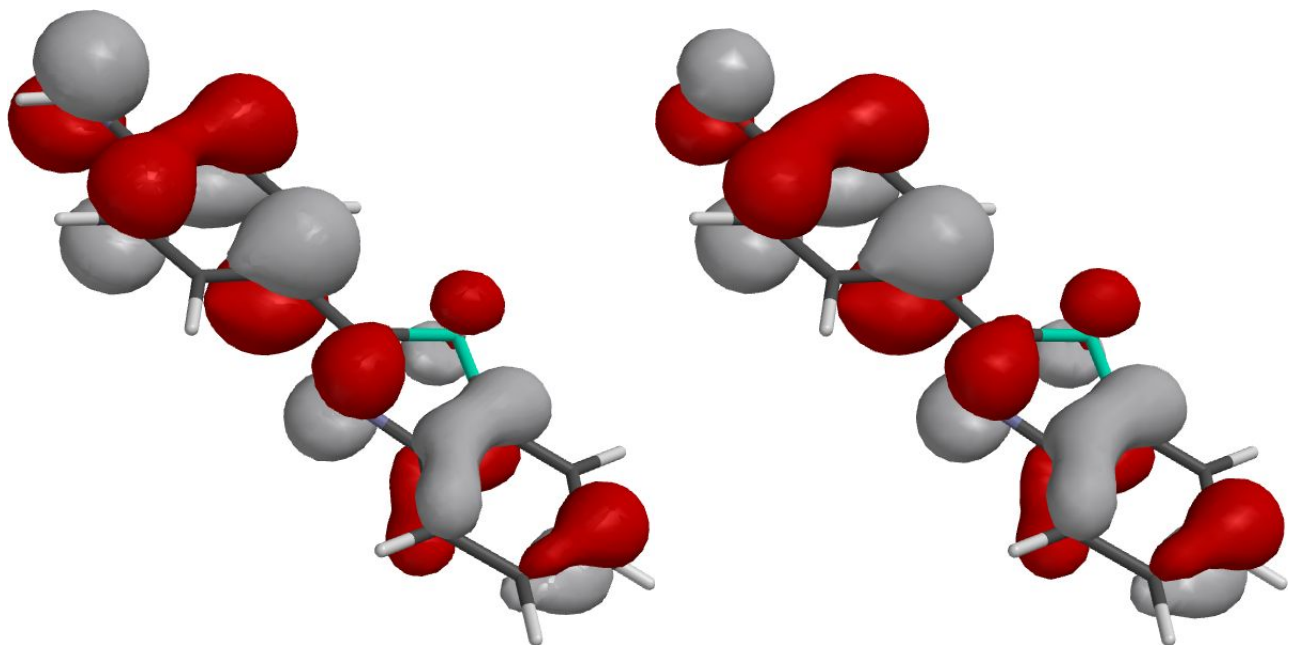


Figure S3. LUMOs for **11** (left) and **13** (right) at the B3LYP/6-31G(d) level.

Movie Descriptions

LUMO Movie of 11 and 13: LUMO_recording.avi shows the LUMOS of both cations alternately superimposed on each other. This works well since the two structures are nearly identical. Qualitative changes in LUMO coefficients are observed as the two structures alternate.

EDEP Surfaces Movie of 11 and 13: Densities_recording.avi shows the EDEP surfaces of both cations alternately superimposed on each other. Qualitative changes in the electron density surfaces are observed as the two structures alternate.

Table S2. 4-(benzothiazol-2-yl)phenylnitrenium ion **11**

B3LYP/6-31G(d) energy: -1008.217179 au

Z-Matrix

| Row | Symbol | N A | NB | NC | Bond | Angle | Dihedral | X | Y | Z |
|-----|--------|-----|----|----|-------|--------|----------|--------|--------|---|
| 1 | C | | | | | | | 4.272 | -0.188 | 0 |
| 2 | C | 1 | | | 2.838 | | | 1.441 | 0.008 | 0 |
| 3 | C | 1 | 2 | | 1.464 | 58.89 | | 3.431 | -1.386 | 0 |
| 4 | C | 1 | 3 | 2 | 1.463 | 117.54 | 0.00 | 3.599 | 1.111 | 0 |
| 5 | C | 4 | 1 | 3 | 1.360 | 121.06 | 0.00 | 2.242 | 1.198 | 0 |
| 6 | C | 3 | 1 | 4 | 1.357 | 120.84 | 0.00 | 2.078 | -1.286 | 0 |
| 7 | H | 3 | 1 | 4 | 1.088 | 118.39 | 180.00 | 3.917 | -2.359 | 0 |
| 8 | H | 4 | 1 | 3 | 1.086 | 116.76 | 180.00 | 4.235 | 1.991 | 0 |
| 9 | H | 5 | 4 | 1 | 1.086 | 119.90 | 180.00 | 1.762 | 2.172 | 0 |
| 10 | H | 6 | 3 | 1 | 1.084 | 121.69 | 180.00 | 1.442 | -2.164 | 0 |
| 11 | N | 1 | 4 | 5 | 1.294 | 116.99 | 180.00 | 5.566 | -0.179 | 0 |
| 12 | H | 11 | 1 | 4 | 1.028 | 111.60 | 180.00 | 5.951 | -1.132 | 0 |
| 13 | C | 2 | 1 | 11 | 1.423 | 178.29 | 180.00 | 0.019 | 0.064 | 0 |
| 14 | N | 13 | 2 | 1 | 1.328 | 122.19 | 0.00 | -0.732 | -1.031 | 0 |
| 15 | S | 13 | 2 | 1 | 1.785 | 123.16 | 180.00 | -0.898 | 1.596 | 0 |
| 16 | C | 15 | 13 | 2 | 1.731 | 88.57 | 180.00 | -2.361 | 0.670 | 0 |
| 17 | C | 16 | 15 | 13 | 2.793 | 168.94 | 0.00 | -4.391 | -1.249 | 0 |
| 18 | C | 14 | 13 | 2 | 1.346 | 111.82 | 180.00 | -2.045 | -0.737 | 0 |
| 19 | C | 16 | 15 | 13 | 1.396 | 129.30 | 180.00 | -3.686 | 1.110 | 0 |
| 20 | C | 19 | 16 | 15 | 1.390 | 117.48 | 180.00 | -4.683 | 0.142 | 0 |
| 21 | C | 17 | 16 | 15 | 1.376 | 62.43 | 0.00 | -3.090 | -1.698 | 0 |
| 22 | H | 19 | 16 | 15 | 1.085 | 121.64 | 0.00 | -3.935 | 2.166 | 0 |
| 23 | H | 20 | 19 | 16 | 1.086 | 118.88 | 180.00 | -5.722 | 0.459 | 0 |
| 24 | H | 21 | 17 | 16 | 1.084 | 122.42 | 180.00 | -2.839 | -2.753 | 0 |
| 25 | H | 17 | 16 | 15 | 1.085 | 177.54 | 180.00 | -5.211 | -1.960 | 0 |

Table S3. Biphenylnitrenium ion 12

B3LYP/6-31G(d) energy: -517.760693 au

Z-Matrix

| Row | Symbol | N A | NB | NC | Bond | Angle | Dihedral | X | Y | Z |
|-----|--------|-----|----|----|-------|--------|----------|--------|--------|---|
| 1 | C | | | | | | | 3.201 | 0.011 | 0 |
| 2 | C | 1 | | | 2.873 | | | 0.328 | 0.008 | 0 |
| 3 | C | 1 | 2 | | 1.463 | 58.59 | | 2.440 | -1.238 | 0 |
| 4 | C | 1 | 3 | 2 | 1.460 | 116.86 | 0.00 | 2.432 | 1.252 | 0 |
| 5 | C | 4 | 1 | 3 | 1.359 | 121.36 | 0.00 | 1.073 | 1.242 | 0 |
| 6 | C | 3 | 1 | 4 | 1.357 | 120.93 | 0.00 | 1.083 | -1.228 | 0 |
| 7 | H | 3 | 1 | 4 | 1.087 | 118.50 | 180.00 | 2.986 | -2.178 | 0 |
| 8 | H | 4 | 1 | 3 | 1.085 | 116.78 | 180.00 | 2.998 | 2.178 | 0 |
| 9 | H | 5 | 4 | 1 | 1.081 | 118.40 | 180.00 | 0.552 | 2.189 | 0 |
| 10 | H | 6 | 3 | 1 | 1.081 | 118.32 | 180.00 | 0.563 | -2.176 | 0 |
| 11 | C | 2 | 1 | 4 | 1.443 | 179.74 | 180.00 | -1.115 | 0.000 | 0 |
| 12 | C | 11 | 2 | 1 | 2.824 | 179.91 | 180.00 | -3.939 | -0.011 | 0 |
| 13 | C | 11 | 2 | 1 | 1.428 | 121.45 | 0.00 | -1.853 | -1.222 | 0 |
| 14 | C | 11 | 2 | 1 | 1.427 | 121.25 | 180.00 | -1.862 | 1.216 | 0 |
| 15 | C | 14 | 11 | 2 | 1.383 | 121.19 | 180.00 | -3.245 | 1.207 | 0 |
| 16 | C | 13 | 11 | 2 | 1.383 | 121.21 | 180.00 | -3.236 | -1.224 | 0 |
| 17 | H | 13 | 11 | 2 | 1.083 | 120.89 | 0.00 | -1.345 | -2.178 | 0 |
| 18 | H | 14 | 11 | 2 | 1.082 | 120.85 | 0.00 | -1.361 | 2.175 | 0 |
| 19 | H | 15 | 14 | 11 | 1.084 | 119.86 | 180.00 | -3.791 | 2.144 | 0 |
| 20 | H | 16 | 13 | 11 | 1.084 | 119.89 | 180.00 | -3.775 | -2.165 | 0 |
| 21 | H | 12 | 11 | 2 | 1.086 | 179.99 | 0.00 | -5.025 | -0.015 | 0 |
| 22 | N | 1 | 4 | 5 | 1.293 | 117.44 | 180.00 | 4.490 | 0.109 | 0 |
| 23 | H | 22 | 1 | 4 | 1.028 | 111.79 | 180.00 | 4.943 | -0.814 | 0 |

4-(benzothiazol-2-yl)quinolimine **16**

B3LYP/6-31G(d) energy: -1084.274591

Z-Matrix

| Row | Symbol | N A | NB | NC | Bond | Angle | Dihedral | X | Y | Z |
|-----|--------|-----|----|----|-------|--------|----------|--------|--------|--------|
| 1 | C | | | | | | | -0.045 | -0.497 | -0.110 |
| 2 | N | 1 | | | 1.293 | | | -1.051 | -1.288 | 0.075 |
| 3 | S | 1 | 2 | | 1.768 | 116.06 | | -0.430 | 1.218 | -0.300 |
| 4 | C | 3 | 1 | 2 | 1.757 | 88.34 | 0.24 | -2.126 | 0.807 | -0.100 |
| 5 | C | 4 | 3 | 1 | 2.775 | 169.34 | -1.46 | -4.634 | -0.327 | 0.251 |
| 6 | C | 2 | 1 | 3 | 1.389 | 111.49 | -0.11 | -2.252 | -0.590 | 0.086 |
| 7 | C | 4 | 3 | 1 | 1.397 | 129.28 | -179.89 | -3.246 | 1.642 | -0.114 |
| 8 | C | 7 | 4 | 3 | 1.393 | 117.99 | 179.70 | -4.498 | 1.059 | 0.064 |
| 9 | C | 5 | 4 | 3 | 1.390 | 61.43 | 1.24 | -3.521 | -1.159 | 0.262 |
| 10 | H | 7 | 4 | 3 | 1.085 | 121.15 | -0.37 | -3.146 | 2.713 | -0.258 |
| 11 | H | 8 | 7 | 4 | 1.086 | 119.27 | 179.92 | -5.383 | 1.689 | 0.058 |
| 12 | H | 9 | 5 | 4 | 1.085 | 121.74 | 179.90 | -3.611 | -2.231 | 0.405 |
| 13 | H | 5 | 4 | 3 | 1.087 | 179.01 | -178.84 | -5.624 | -0.754 | 0.387 |
| 14 | C | 1 | 2 | 6 | 1.533 | 120.68 | 178.11 | 1.388 | -1.042 | -0.116 |
| 15 | C | 14 | 1 | 2 | 1.518 | 108.14 | -102.96 | 2.030 | -0.698 | 1.216 |
| 16 | H | 15 | 14 | 1 | 1.087 | 114.87 | 63.25 | 1.560 | -1.157 | 2.082 |
| 17 | C | 14 | 1 | 2 | 1.513 | 110.20 | 133.96 | 2.158 | -0.474 | -1.288 |
| 18 | H | 17 | 14 | 1 | 1.087 | 115.02 | -64.71 | 1.789 | -0.769 | -2.267 |
| 19 | C | 17 | 14 | 1 | 1.339 | 123.35 | 115.70 | 3.222 | 0.327 | -1.151 |
| 20 | H | 19 | 17 | 14 | 1.086 | 121.73 | -179.76 | 3.753 | 0.723 | -2.011 |
| 21 | C | 15 | 14 | 1 | 1.339 | 123.51 | -117.41 | 3.101 | 0.096 | 1.337 |
| 22 | H | 21 | 15 | 14 | 1.089 | 120.62 | -179.26 | 3.531 | 0.302 | 2.316 |
| 23 | C | 19 | 17 | 14 | 1.472 | 122.34 | 1.32 | 3.758 | 0.706 | 0.167 |
| 24 | O | 14 | 1 | 2 | 1.423 | 108.01 | 15.26 | 1.320 | -2.457 | -0.255 |
| 25 | H | 24 | 14 | 1 | 0.978 | 106.21 | -13.86 | 0.389 | -2.704 | -0.083 |
| 26 | H | 23 | 19 | 17 | 1.903 | 148.89 | -178.42 | 5.033 | 1.683 | 1.188 |
| 27 | N | 23 | 19 | 17 | 1.289 | 118.42 | -177.86 | 4.760 | 1.515 | 0.212 |

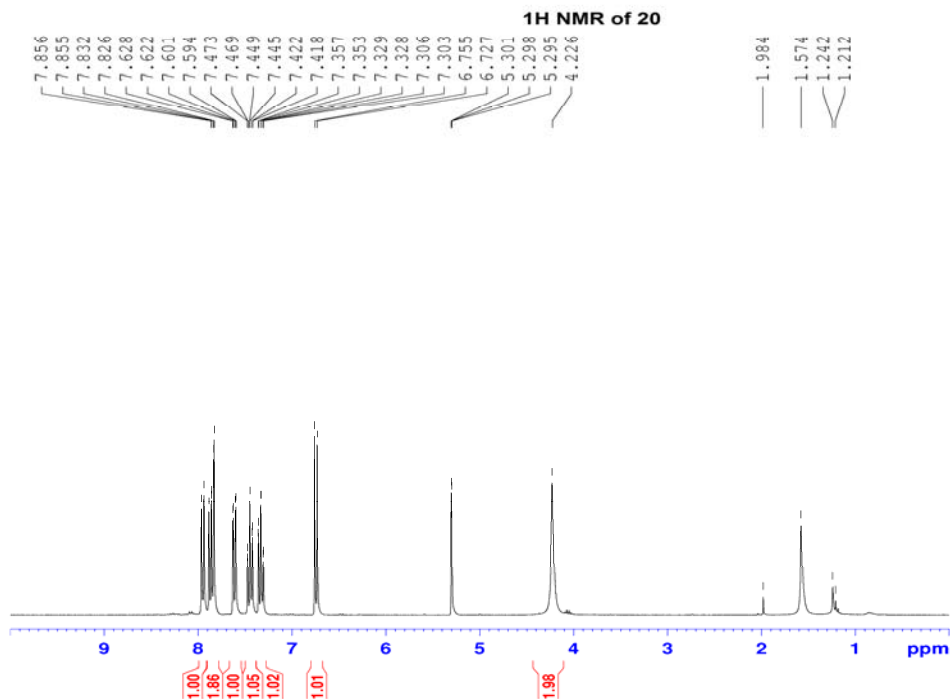
4-Phenylquinolimine **19a**

B3LYP/6-31G(d) energy: -593.819608 au

Z-Matrix

| Row | Symbol | NA | NB | NC | Bond | Angle | Dihedral | X | Y | Z |
|-----|--------|----|----|----|-------|--------|----------|--------|--------|--------|
| 1 | C | | | | | | | -0.289 | 0.869 | -0.019 |
| 2 | C | 1 | | | 1.513 | | | -1.038 | 0.496 | 1.242 |
| 3 | H | 2 | 1 | | 1.087 | 114.99 | | -0.555 | 0.796 | 2.169 |
| 4 | C | 1 | 2 | 3 | 1.513 | 112.14 | -177.45 | -1.046 | 0.477 | -1.269 |
| 5 | H | 4 | 1 | 2 | 1.088 | 114.96 | 177.61 | -0.567 | 0.761 | -2.204 |
| 6 | C | 4 | 1 | 2 | 1.340 | 123.69 | -1.93 | -2.223 | -0.163 | -1.255 |
| 7 | H | 6 | 4 | 1 | 1.087 | 121.95 | 179.70 | -2.745 | -0.427 | -2.171 |
| 8 | C | 2 | 1 | 4 | 1.341 | 123.75 | 2.02 | -2.215 | -0.146 | 1.247 |
| 9 | H | 8 | 2 | 1 | 1.089 | 120.77 | -179.61 | -2.715 | -0.385 | 2.184 |
| 10 | C | 6 | 4 | 1 | 1.472 | 122.16 | -0.05 | -2.891 | -0.547 | -0.001 |
| 11 | O | 1 | 4 | 6 | 1.442 | 109.07 | -122.37 | -0.076 | 2.295 | -0.025 |
| 12 | H | 11 | 1 | 4 | 0.971 | 106.38 | 59.03 | -0.956 | 2.703 | -0.060 |
| 13 | H | 10 | 6 | 4 | 1.905 | 148.95 | -179.19 | -4.364 | -1.378 | 0.875 |
| 14 | N | 10 | 6 | 4 | 1.289 | 118.50 | -178.83 | -4.011 | -1.181 | -0.070 |
| 15 | C | 1 | 11 | 2 | 1.535 | 106.89 | -118.30 | 1.098 | 0.211 | -0.010 |
| 16 | C | 15 | 1 | 11 | 2.806 | 178.92 | 166.41 | 3.611 | -1.038 | 0.019 |
| 17 | C | 15 | 1 | 11 | 1.401 | 119.46 | -178.30 | 1.198 | -1.186 | -0.039 |
| 18 | C | 15 | 1 | 11 | 1.398 | 121.32 | 2.10 | 2.266 | 0.978 | 0.035 |
| 19 | C | 16 | 15 | 1 | 1.395 | 59.69 | -164.54 | 3.516 | 0.353 | 0.048 |
| 20 | C | 17 | 15 | 1 | 1.394 | 120.53 | -179.64 | 2.446 | -1.807 | -0.024 |
| 21 | H | 17 | 15 | 1 | 1.087 | 119.70 | 0.45 | 0.295 | -1.790 | -0.075 |
| 22 | H | 18 | 15 | 1 | 1.084 | 119.40 | -0.43 | 2.192 | 2.059 | 0.057 |
| 23 | H | 19 | 16 | 15 | 1.087 | 120.08 | -179.94 | 4.417 | 0.960 | 0.081 |
| 24 | H | 20 | 17 | 15 | 1.087 | 119.70 | -179.93 | 2.508 | -2.892 | -0.048 |
| 25 | H | 16 | 15 | 1 | 1.087 | 179.88 | 160.45 | 4.585 | -1.520 | 0.029 |

¹H NMR of 20:



```

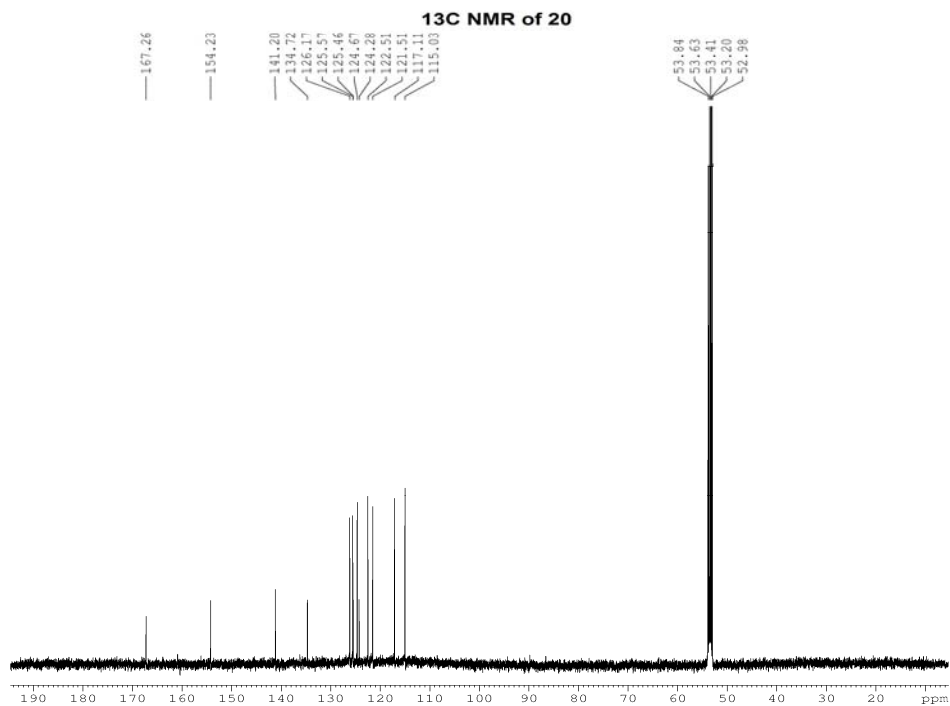
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¹³C NMR of 20:



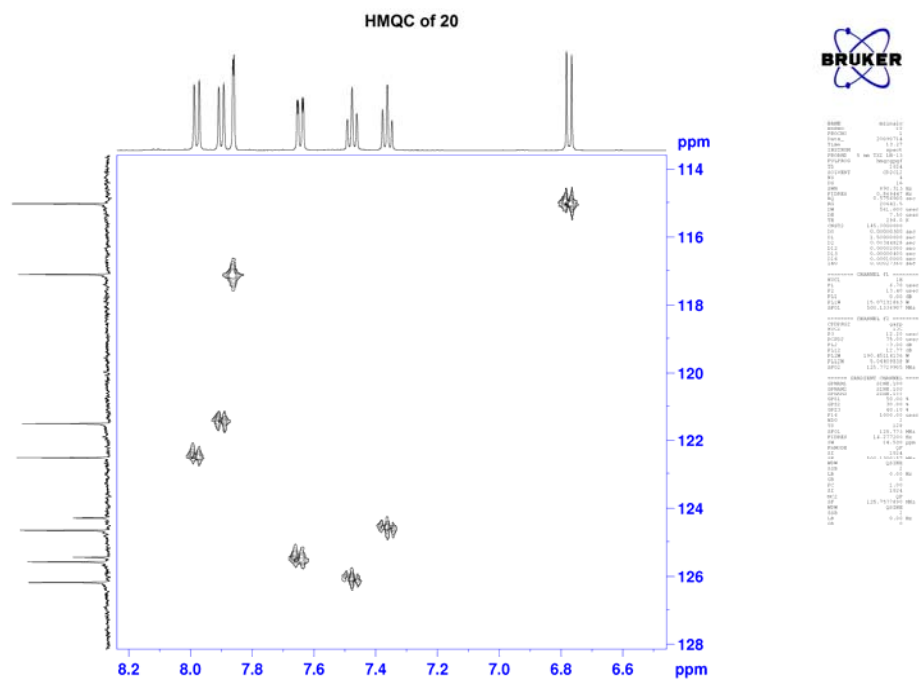
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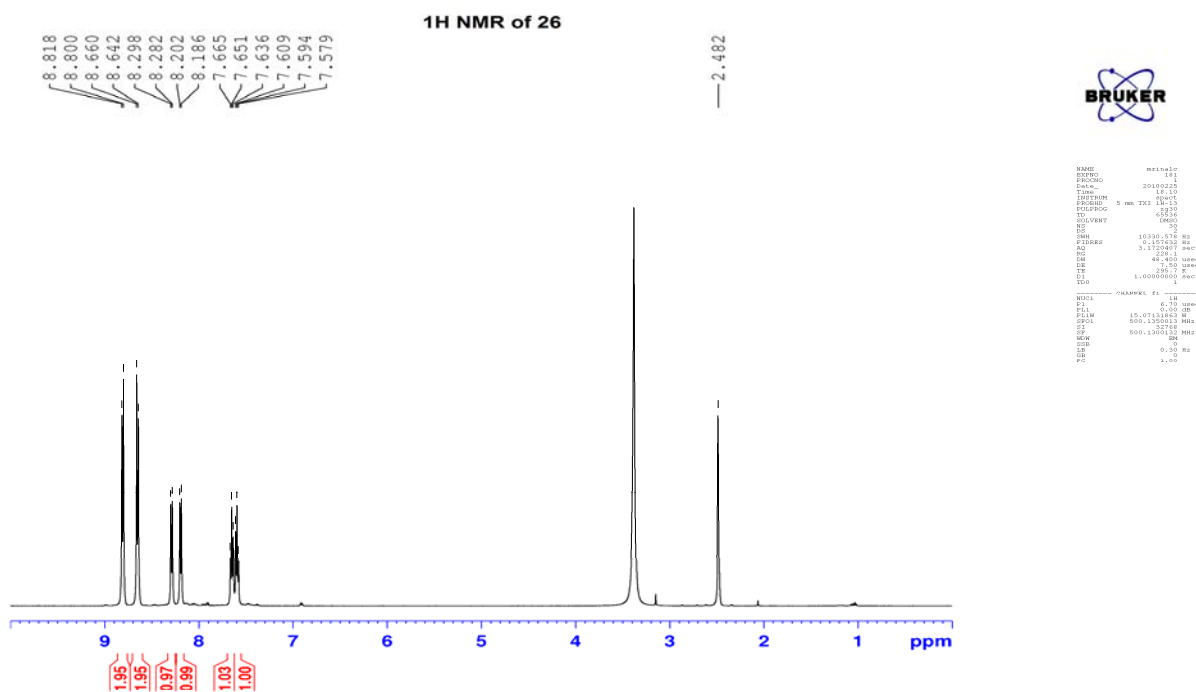
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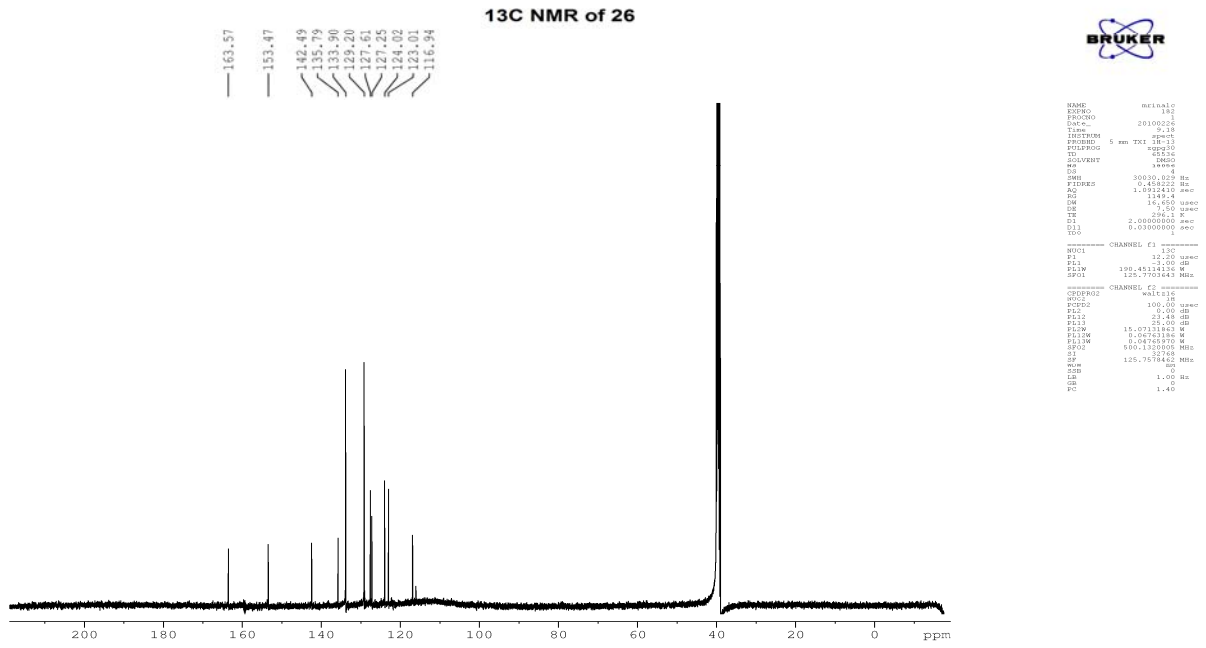
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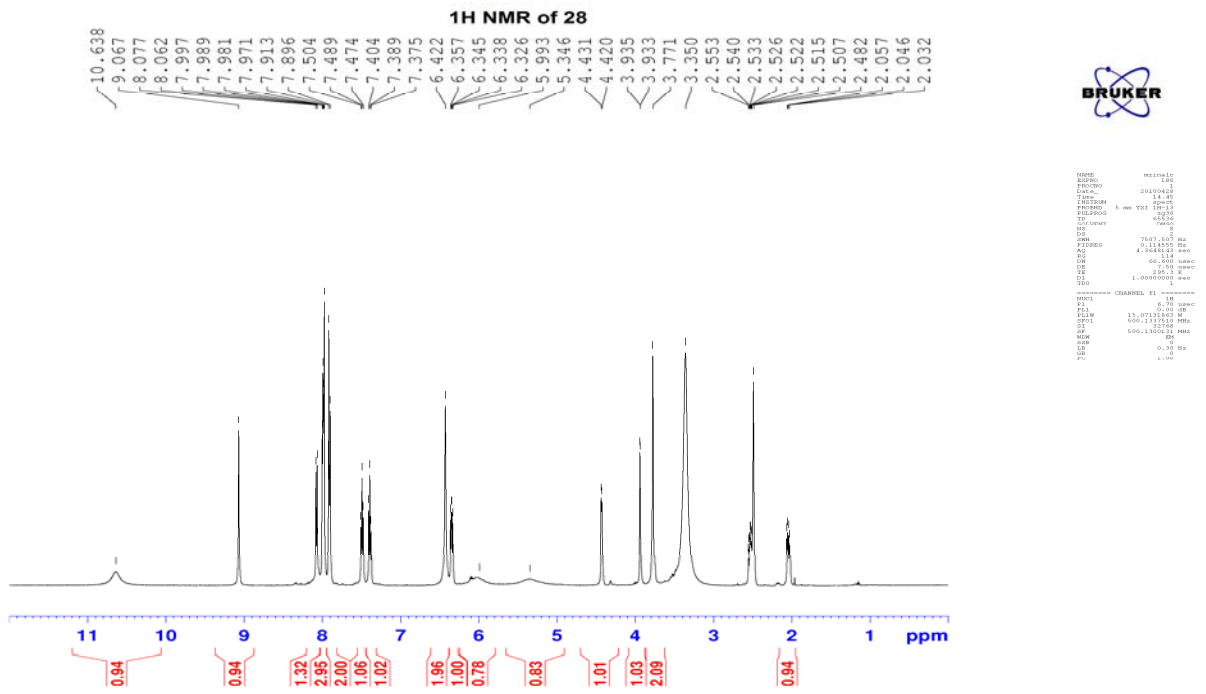
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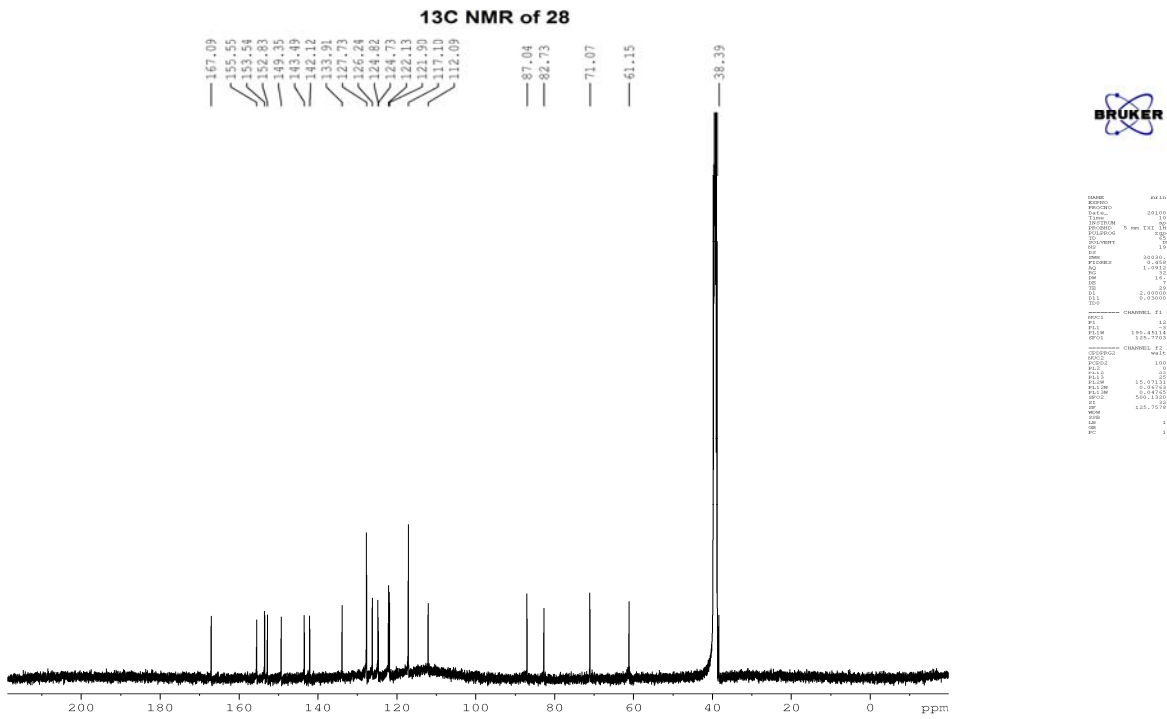
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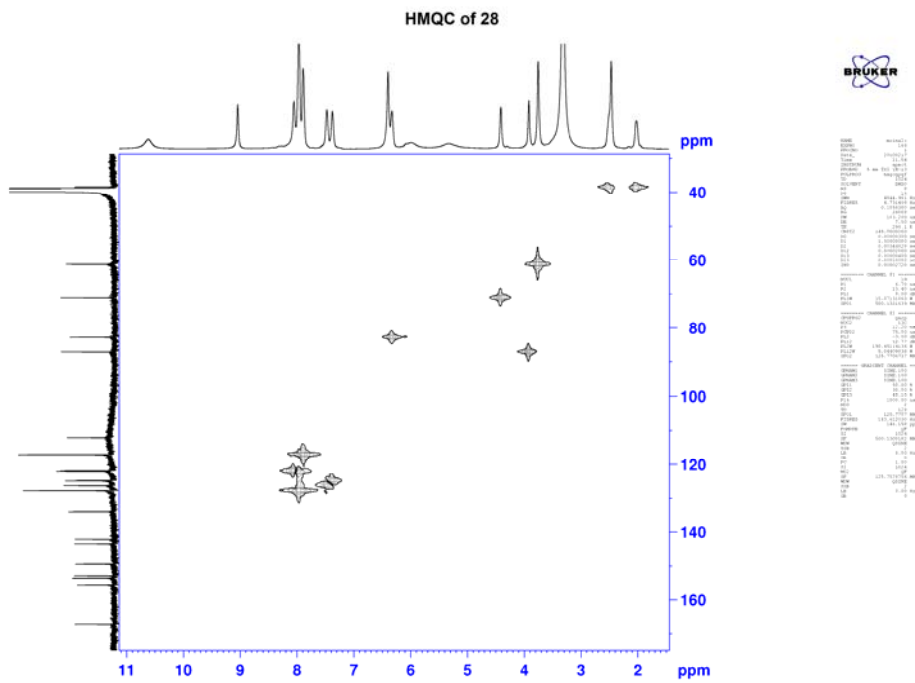
¹H NMR of 28:



¹³C NMR of **28**:



HMQC of **28**:



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