Electronic Supplementary Material (ESI) for RSC Advances.

Electronic Supplementary Information

Aerobic photooxidative synthesis of 2-aryl-4-quinazolinones from aromatic aldehydes and aminobenzamide using catalytic amounts of molecular iodine

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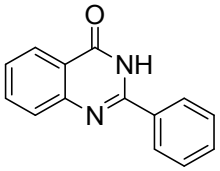
1. General Information

Unless otherwise noted, all reactants or reagents including dry solvents were obtained from commercial suppliers and used as received. The fluorescent lamp was a 23 W Twist lamp from Osram Sylvania Inc. Analytical thin-layer chromatography (TLC) was carried out using 0.25 mm commercial silica gel plates (Merck silica gel 60 F_{254}) and preparative thin-layer chromatography (PTLC) was carried out using 0.50 mm commercial silica gel plates (Merck silica gel 60 F_{254}). Flash column chromatography was performed with Kanto silica gel 60N (Spherical, Neutral, 40–50 mm). The developed chromatogram was analyzed by UV lamp (254 nm). ^{1}H NMR spectra was obtained on a JEOL ECA 500 spectrometer (500 MHz for ^{1}H NMR) or a JEOL AL 400 spectrometer (400MHz for ^{1}H NMR). Chemical shifts (δ) are expressed in parts per million and are internally referenced [2.50 ppm (DMSO) or 0.00 ppm (TMS) for ^{1}H NMR].

2. General Procedure

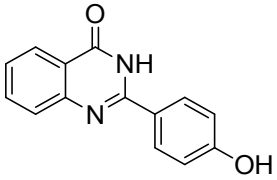
Synthesis of 2-phenyl-4-quinazolinone (3a) (Table 1): A solution of 2-aminobenzamide (**1a**, 0.3 mmol), benzaldehyde (**2a**, 0.3 mmol) and iodine (0.015 mmol) in dry EtOAc (5 mL) in a pyrex test tube, purged with an O_2 balloon, was stirred and irradiated externally with a fluorescent lamp for 1 h. The reaction mixture was concentrated under reduced pressure, and purification of the crude product by PTLC ($CHCl_3$:MeOH = 9:1) provided 2-phenyl-4-quinazolinone (**3a**) (Rf = 0.6, 57.3 mg, 86%).

2-phenyl-4-quinazolinone (3a)[S-1] **(Table 1)**



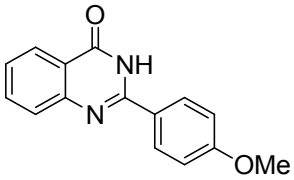
Prepared according to the general procedure: ^{1}H-NMR (500 MHz, DMSO-d6): δ 12.55 (br, 1H), 8.19-8.15 (m, 3H), 7.82 (t, *J* = 8.7 Hz, 1H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.58-7.50 (m, 5H).

2-(4-hydroxyphenyl)-4-quinazolinone (3b)[S-2] **(Table 2)**



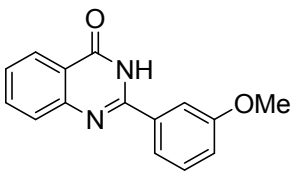
Prepared according to the general procedure: ^{1}H-NMR (500 MHz, DMSO-d6): δ 12.31 (br, 1H), 10.19 (s, 1H), 8.12-8.07 (m, 3H), 7.79 (t, *J* = 7.5 Hz, 1H), 7.68 (d, *J* = 8.8 Hz, 1H), 7.45 (t, *J* = 6.8 Hz, 1H), 6.90 (d, *J* = 7.8 Hz, 2H).

2-(4-methoxyphenyl)-4-quinazolinone (3c)[S-3] **(Table 2)**



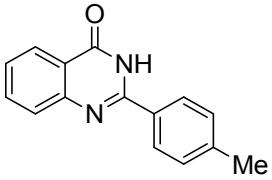
Prepared according to the general procedure: ^{1}H-NMR (400 MHz, CDCl3): δ 8.33-8.31 (m, 1H), 7.88-7.80 (m, 2H), 7.72-7.66 (m, 2H), 7.55-7.47 (m, 2H), 7.15-7.13 (m, 1H), 5.96 (s, 1H), 3.96 (s, 3H).

2-(3-methoxyphenyl)-4-quinazolinone (3d)[S-3] **(Table 2)**



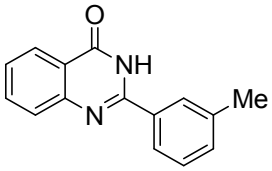
Prepared according to the general procedure: ^{1}H-NMR (500 MHz, $CDCl_3$): δ 10.72 (br, 1H), 8.32 (d, *J* = 8.0 Hz, 2H), 7.85-7.79 (m, 2H), 7.73-7.69 (m, 2H), 7.52-7.46 (m, 1H), 7.13 (d, *J* = 8.0 Hz, 1H), 3.95 (s, 3H).

2-(4-tolyl)-4-quinazolinone (3e)[S-3] **(Table 2)**



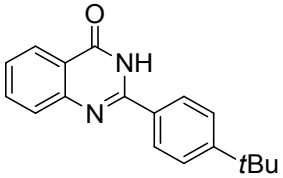
Prepared according to the general procedure: ^{1}H-NMR (500 MHz, DMSO-d6): δ 12.47 (br, 1H), 8.15 (d, *J* = 8.0 Hz, 1H), 8.10 (d, *J* = 8.0 Hz, 2H), 7.83 (t, *J* = 8.0 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 2H), 2.39 (s, 3H).

2-(3-tolyl)-4-quinazolinone (3f)[S-3] **(Table 2)**



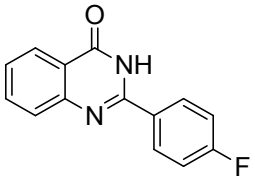
Prepared according to the general procedure: ^{1}H-NMR (500 MHz, $CDCl_3$): δ 10.79 (br, 1H), 8.24 (d, *J* = 8.0 Hz, 1H), 7.80 (d, *J* = 3.4 Hz, 2H), 7.57 (d, *J* = 6.9 Hz, 1H), 7.51-7.48 (m, 1H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.34 (d, *J* = 6.9 Hz, 2H), 2.52 (s, 3H).

2-(4-tert-butylphenyl)-4-quinazolinone (3g)[S-1] **(Table 2)**



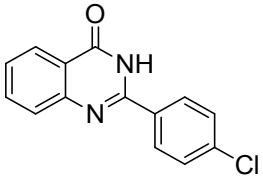
Prepared according to the general procedure: ^{1}H-NMR (500 MHz, $CDCl_3$): δ 11.80 (br, 1H), 8.34 (d, *J* = 8.0 Hz, 1H), 8.21 (d, *J* = 8.0 Hz, 2H), 7.84-7.78 (m, 2H), 7.59 (d, *J* = 8.6 Hz, 2H), 7.50 (t, *J* = 8.0 Hz, 1H), 1.39 (s, 9H).

2-(4-fluorophenyl)-4-quinazolinone (3h)[S-1] **(Table 2)**



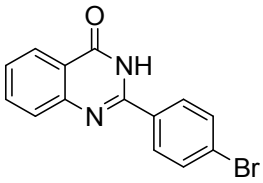
Prepared according to the general procedure. The crude product was purified by the filtration[S-1]: ^{1}H-NMR (500 MHz, DMSO-d6): δ 12.58 (br, 1H), 8.25 (dd, *J* = 5.4 Hz, 8.6 Hz, 2H), 8.15 (d, *J* = 7.5 Hz, 1H), 7.84 (t, *J* = 8.6 Hz, 1H), 7.73 (d, *J* = 8.2 Hz, 1H), 7.52 (t, *J* = 7.8 Hz, 1H), 7.39 (d, *J* = 8.6 Hz, 2H).

2-(4-chlorophenyl)-4-quinazolinone (3i)[S-1] **(Table 2)**



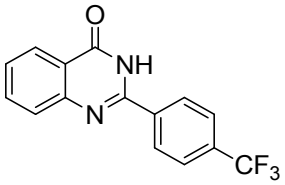
Prepared according to the general procedure. The crude product was purified by the filtration[S-1]: ^{1}H-NMR (500 MHz, DMSO-d6): δ 12.61 (br, 1H), 8.21-8.15 (m, 3H), 7.85 (t, *J* = 8.0 Hz, 1H), 7.75 (d, *J* = 8.6 Hz, 1H), 7.63 (d, *J* = 7.6 Hz, 2H), 7.54 (t, *J* = 7.1 Hz, 1H).

2-(4-bromophenyl)-4-quinazolinone (3j)[S-1] **(Table 2)**



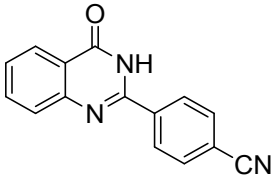
Prepared according to the general procedure. The crude product was purified by the filtration[S-1]: ^{1}H-NMR (500 MHz, DMSO-d6): δ 12.61 (br, 1H), 8.16-8.12 (m, 3H), 7.85 (t, *J* = 8.0 Hz, 1H), 7.78-7.74 (m, 3H), 7.54 (t, *J* = 7.2 Hz, 1H).

2-(4-trifluoromethylphenyl)-4-quinazolinone (3k)[S-1] **(Table 2)**



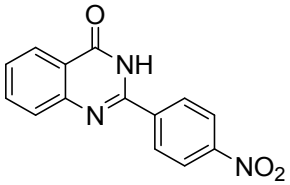
Prepared according to the general procedure. The crude product was purified by the filtration[S-1]: ^{1}H-NMR (500 MHz, DMSO-d6): δ 12.75 (br, 1H), 8.38-8.35 (m, 2H), 8.19-8.15 (m, 1H), 7.94-7.84 (m, 3H), 7.79-7.76 (m, 1H), 7.58-7.53 (m, 1H).

2-(4-cyanophenyl)-4-quinazolinone (3l)[S-3] **(Table 2)**



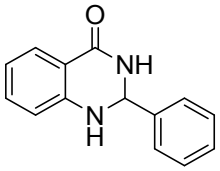
Prepared according to the general procedure. The crude product was purified by the filtration[S-1]: ^{1}H-NMR (500 MHz, DMSO-d6): δ 12.72 (br, 1H), 8.31 (d, *J* = 8.6 Hz, 2H), 8.15 (d, *J* = 8.0 Hz, 1H), 8.01 (d, *J* = 8.0 Hz, 2H), 7.84 (t, *J* = 7.5 Hz, 1H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.54 (t, *J* = 7.5 Hz, 1H).

2-(4-nitrophenyl)-4-quinazolinone (3m)[S-1] **(Table 2)**



Prepared according to the general procedure. The crude product was purified by the filtration[S-1]: ^{1}H-NMR (500 MHz, DMSO-d6): δ 12.84 (br, 1H), 8.41-8.40 (m, 4H), 8.19 (d, *J* = 6.9 Hz, 1H), 7.90-7.87 (m, 1H), 7.80 (d, *J* = 7.4 Hz, 1H), 7.60-7.57 (m, 1H).

2-phenyl-2,3-dihydroquinazolin-4-one (4a)[S-4] **(Scheme 2)**



Synthesis of 2-phenyl-2,3-dihydroquinazolin-4-one (4a): A solution of 2-aminobenzamide (**1a**, 0.3 mmol), benzaldehyde (**2a**, 0.3 mmol) and iodine (0.015 mmol) in dry Hexane (5 mL) in a pyrex test tube, purged with an O_2 balloon, was stirred and irradiated externally with a fluorescent lamp for 1 h. The reaction mixture was concentrated under reduced pressure, and purification of the crude product by PTLC ($CHCl_3$:MeOH = 20:1) provided 2-phenyl-2,3-dihydroquinazolin-4-one (**4a**) (Rf = 0.4, 33 mg, 49%).

^{1}H-NMR (500 MHz, DMSO-d6): δ 8.29 (s, 1H), 7.61 (d, *J* = 8.1 Hz, 1H), 7.50 (d, *J* = 6.8 Hz, 2H), 7.40-7.33 (m, 3H), 7.24 (t, *J* = 7.9 Hz, 1H), 7.11 (br, 1H), 6.75 (d, 8.6 Hz, 1H), 6.67 (t, *J* = 8.0 Hz, 1H), 5.75 (s, 1H).

4. References

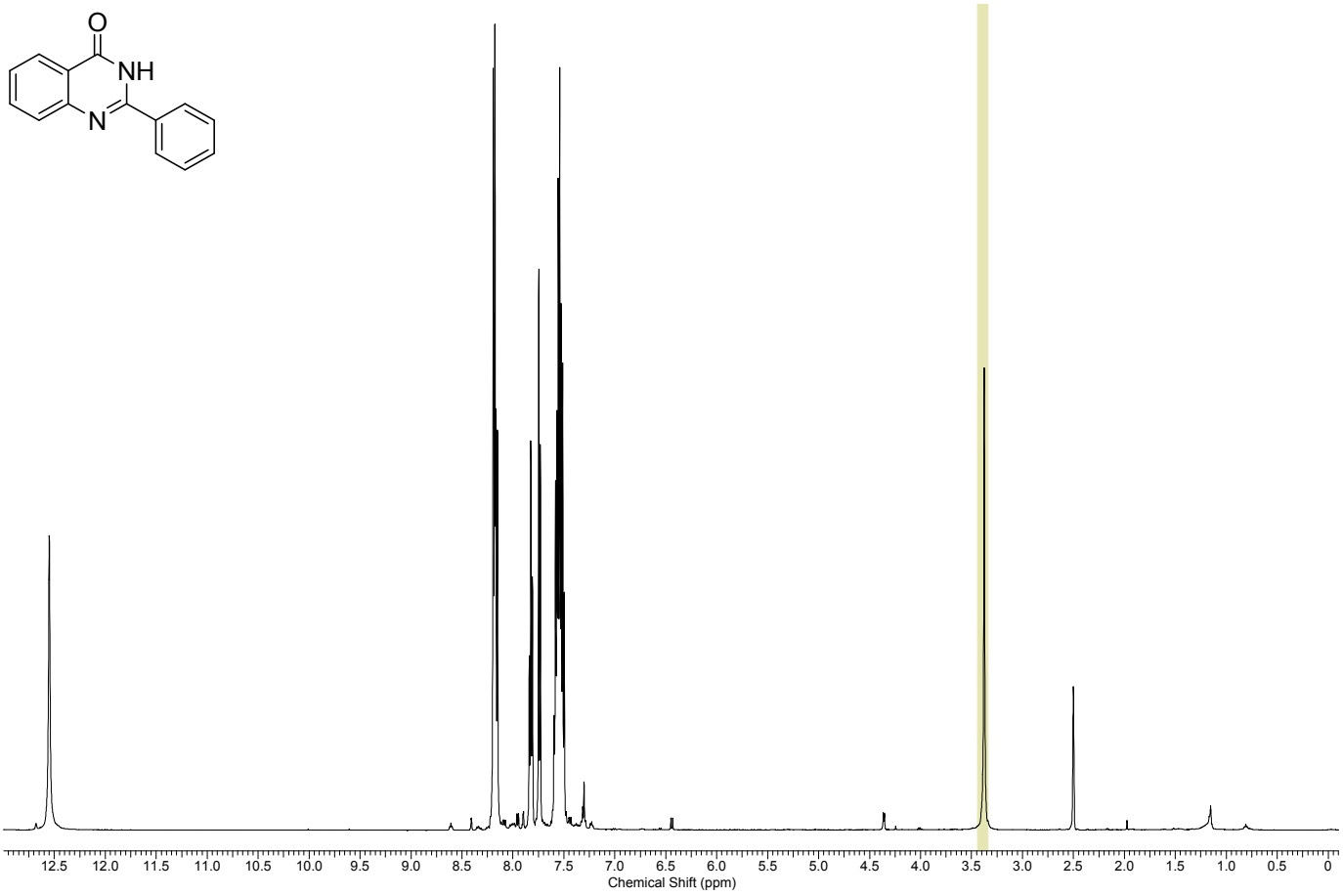
[S-1] M. Sharif, J. Opalach, P. Langer, M. Beller and X.-F. Wu, *RSC Adv.*, 2014, **4**, 8.

[S-2] G.-W. Wang, C.-B. Miao, and H. Kang, *Bull. Chem. Soc. Jpn.,* 2006, **79**, 1426.

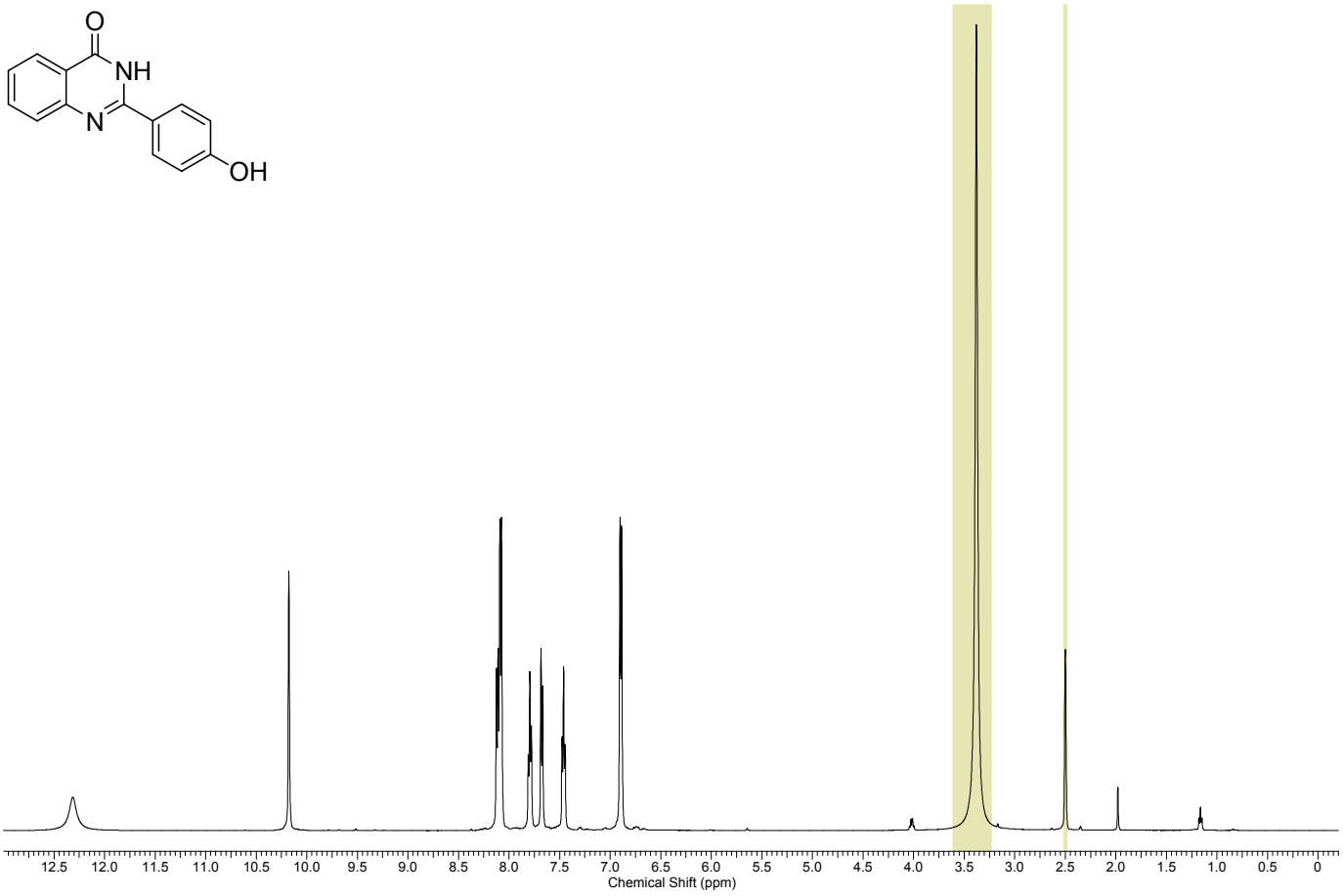
[S-3] J. Zhou and J. Fang, *J. Org. Chem.*, 2011, **76**, 7730.

[S-4] X.-F. Wu, S. Oschatz, A. Block, A. Spannenberg and P. Langer, *Org. Biomol. Chem.*, 2014, **12**, 1865.

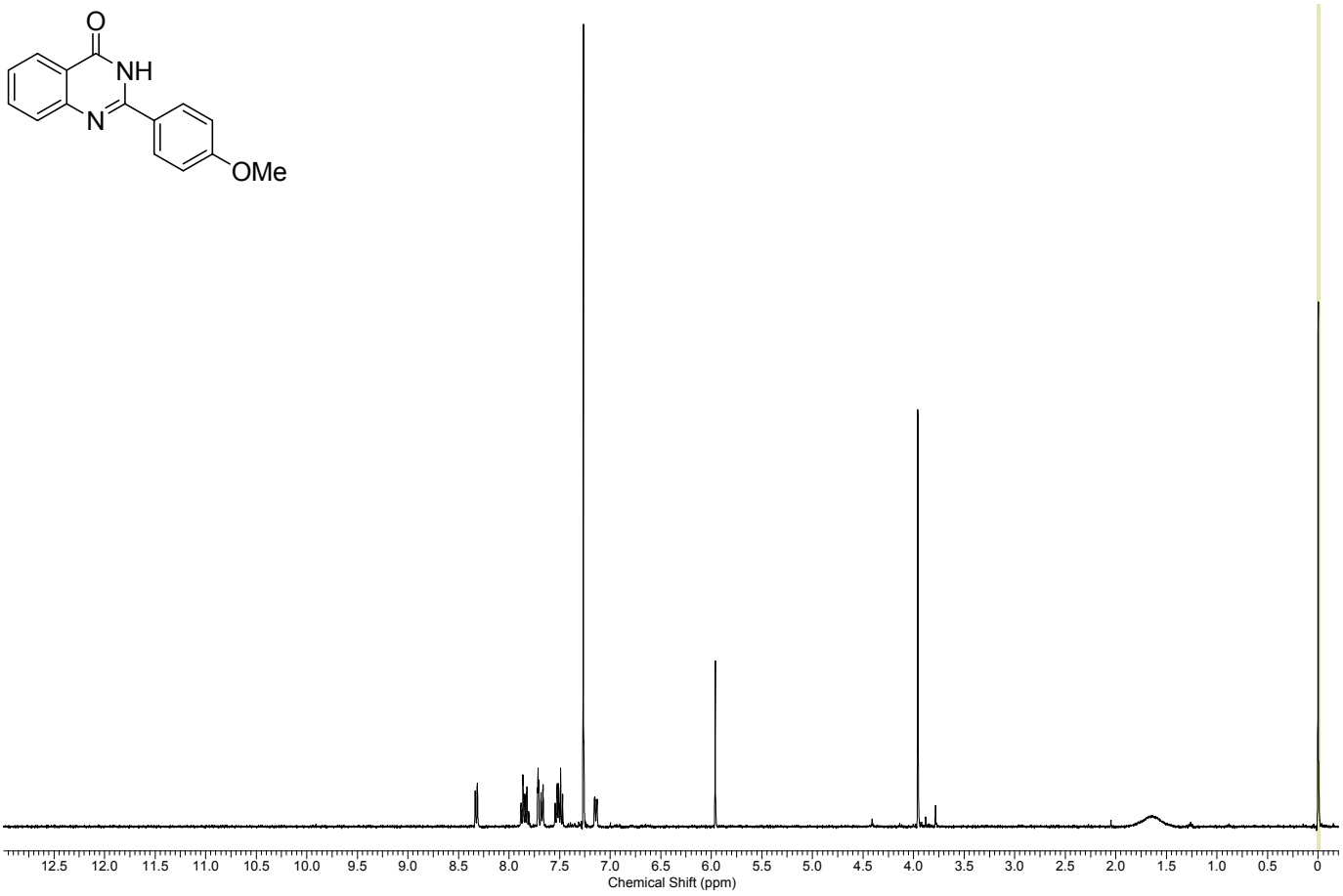
2-phenyl-4-quinazolinone (3a)[S-1] **(Table 1)**



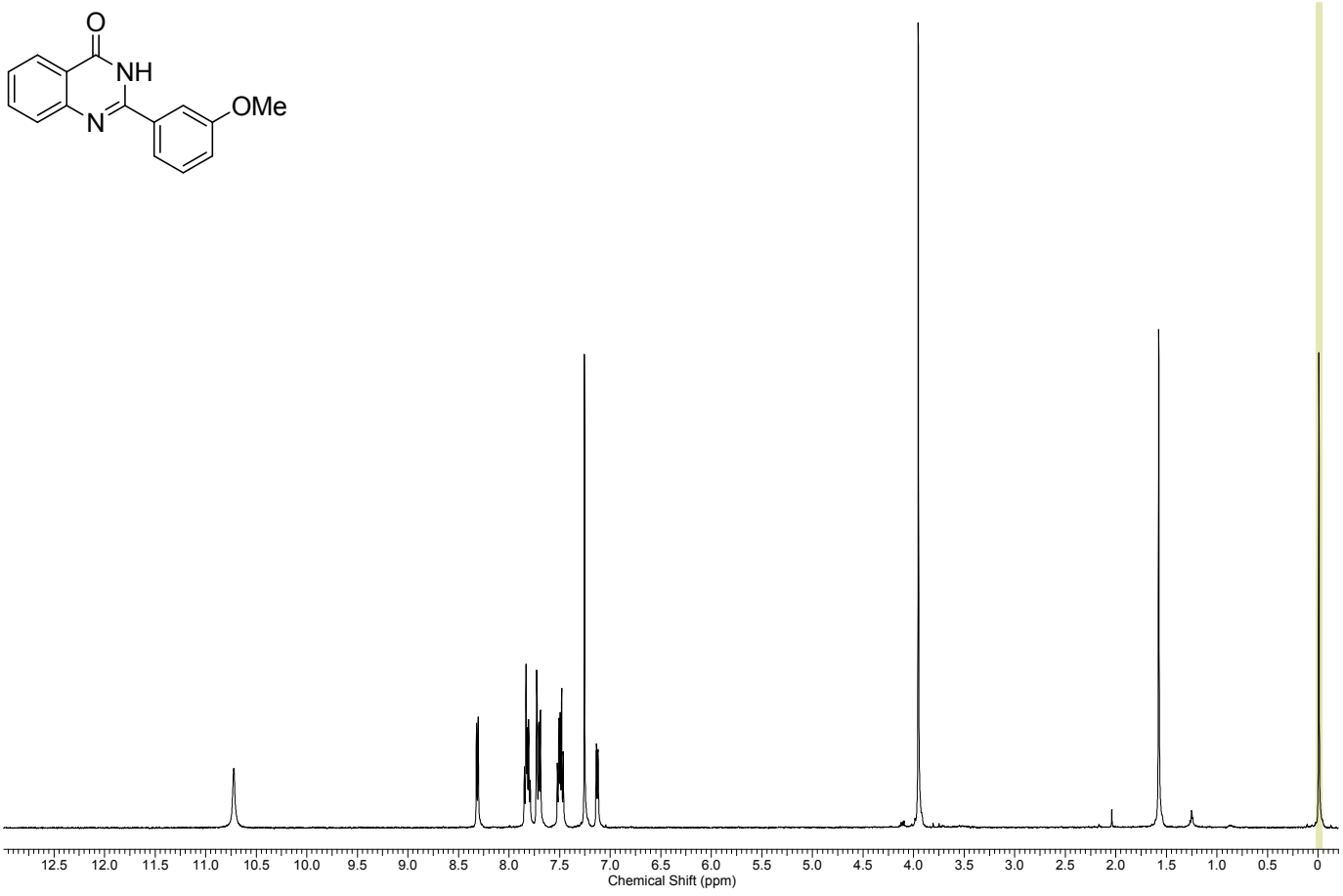
2-(4-hydroxyphenyl)-4-quinazolinone (3b)[S-2] **(Table 2)**



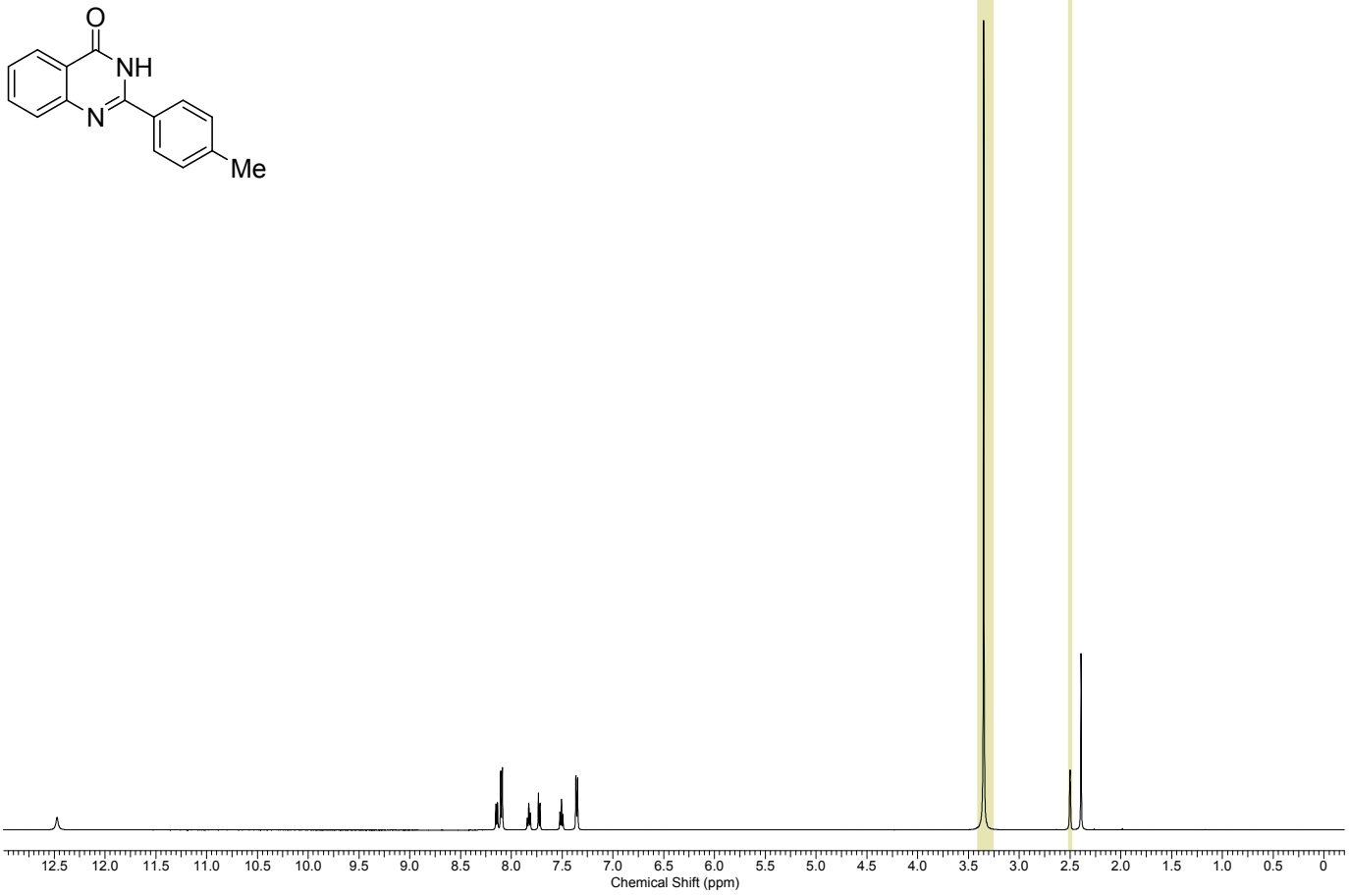
2-(4-methoxyphenyl)-4-quinazolinone (3c)[S-3] **(Table 2)**



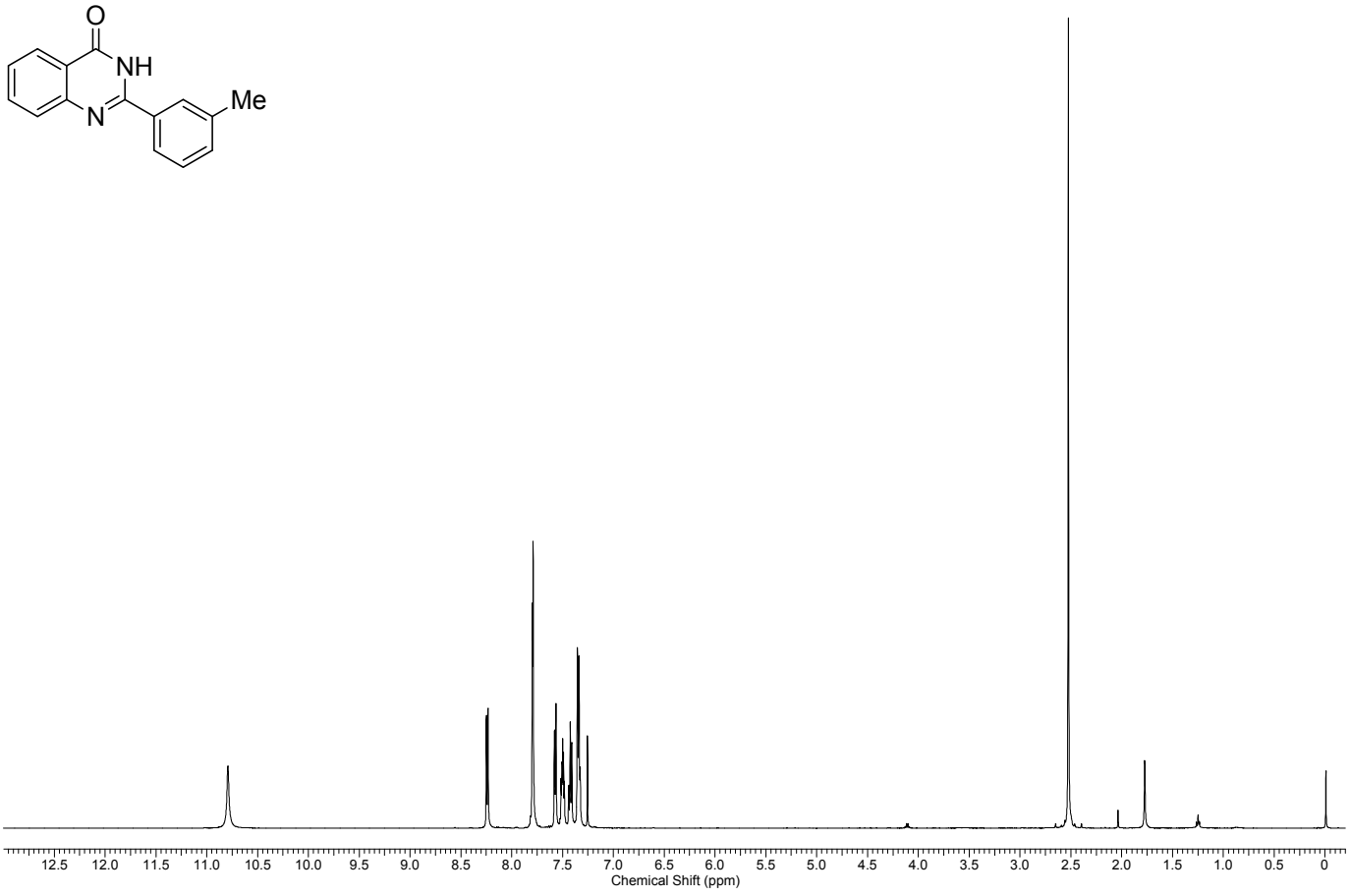
2-(3-methoxyphenyl)-4-quinazolinone (3d)[S-3] **(Table 2)**



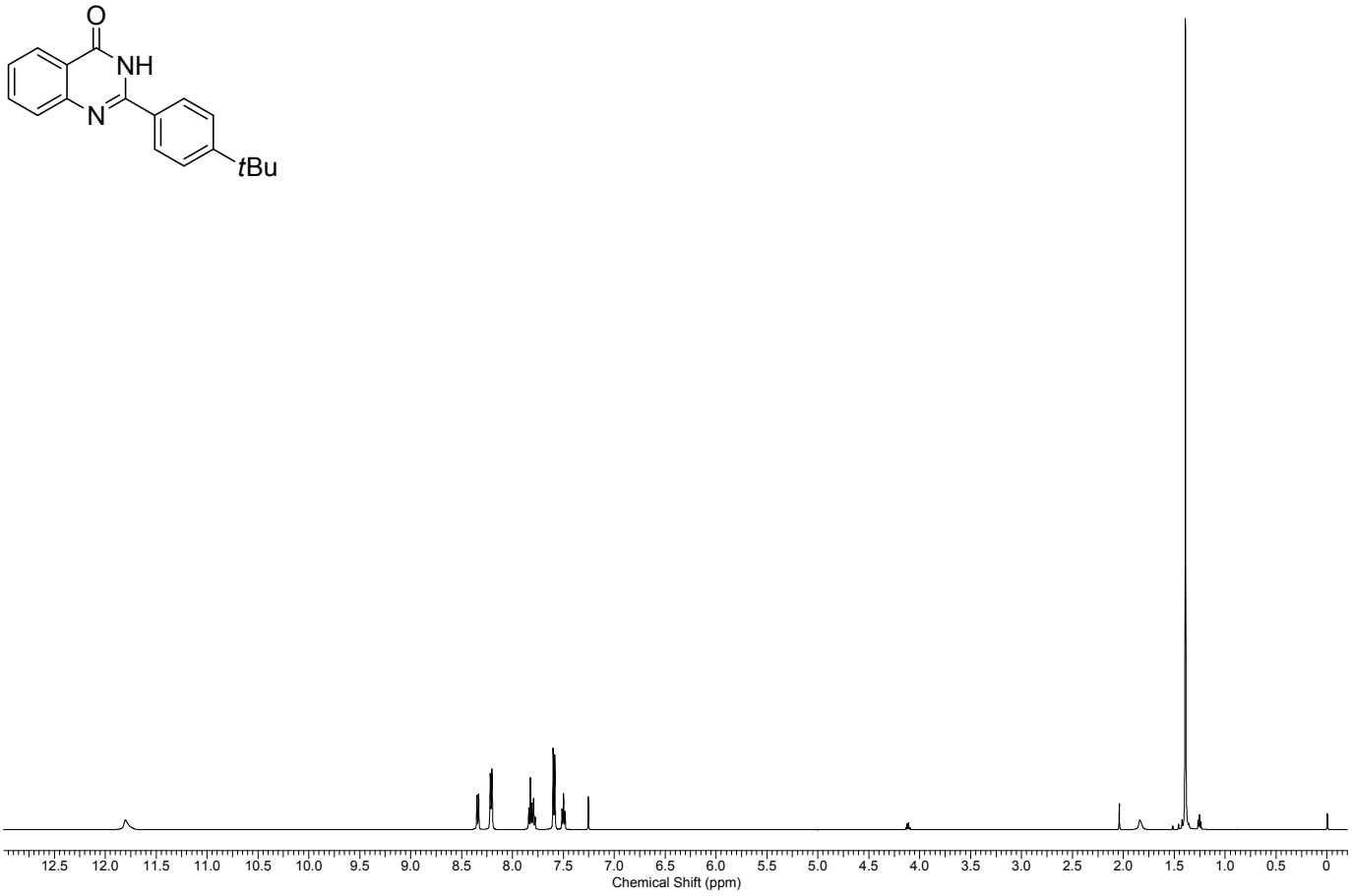
2-(4-tolyl)-4-quinazolinone (3e)[S-3] **(Table 2)**



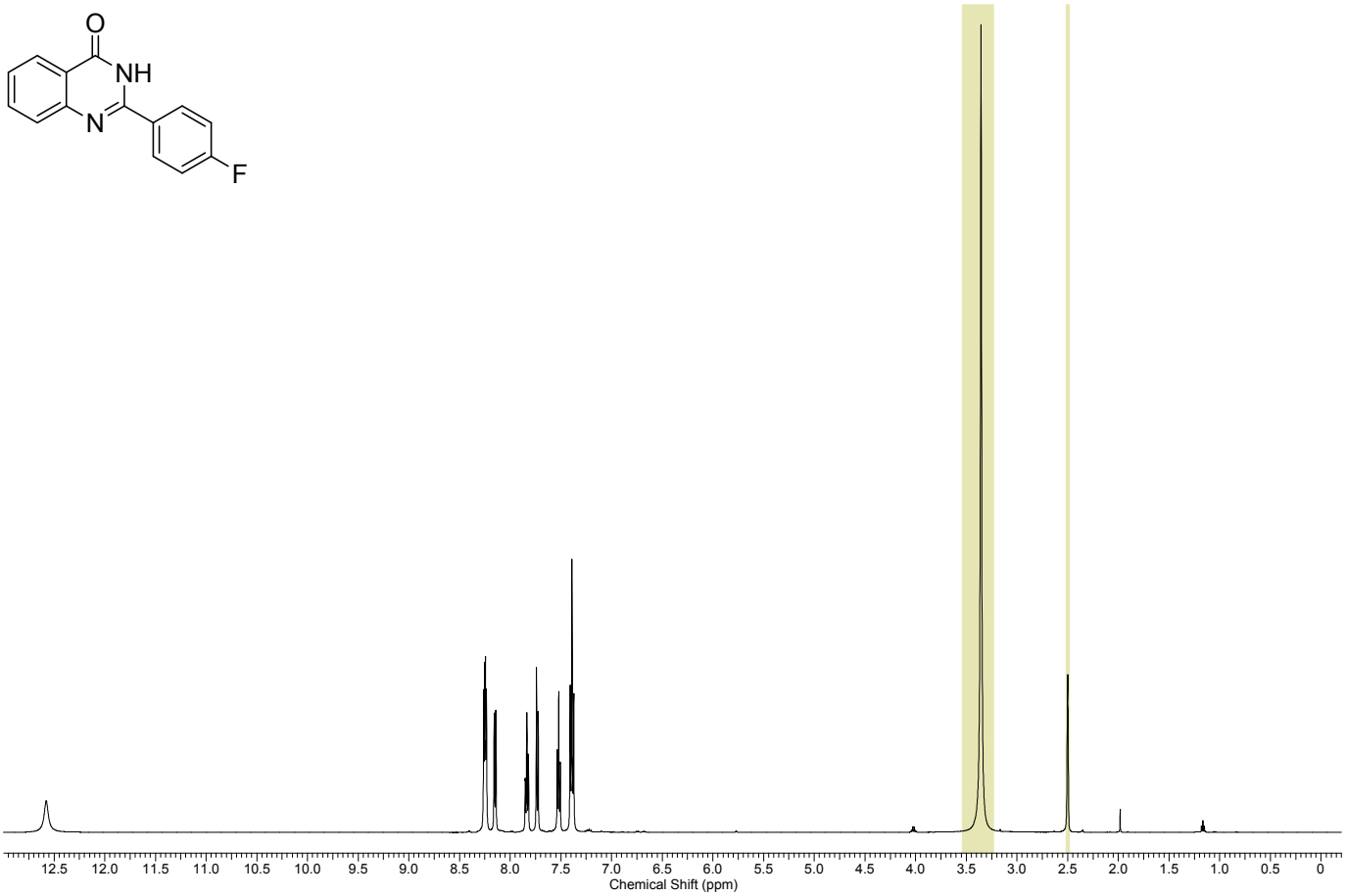
2-(3-tolyl)-4-quinazolinone (3f)[S-3] **(Table 2)**



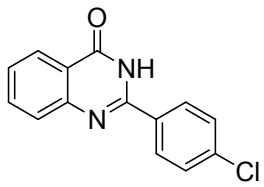
2-(4-tert-butylphenyl)-4-quinazolinone (3g)[S-1] **(Table 2)**



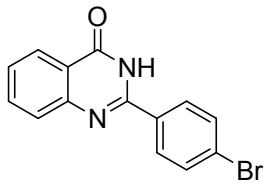
2-(4-fluorophenyl)-4-quinazolinone (3h)[S-1] **(Table 2)**



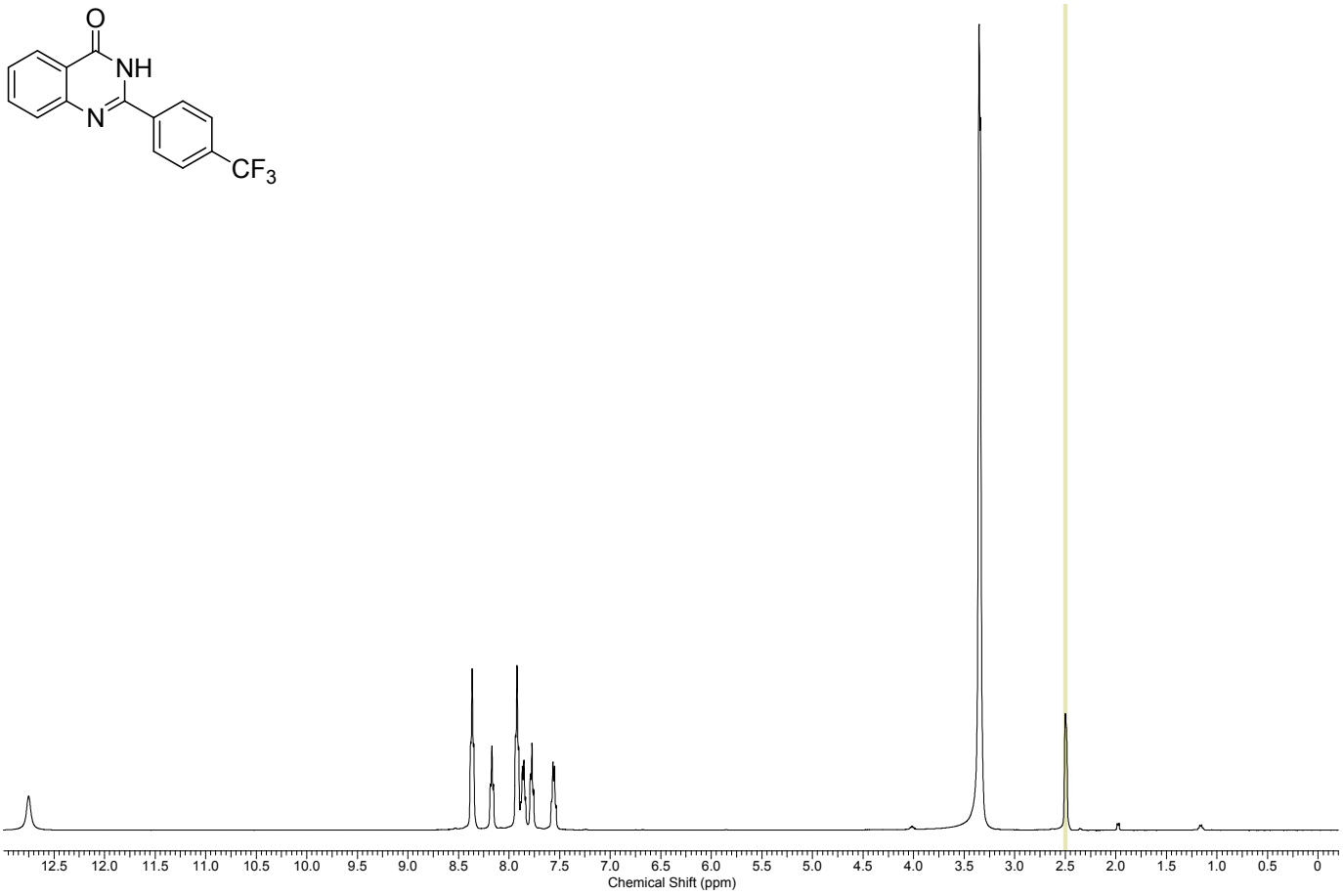
2-(4-chlorophenyl)-4-quinazolinone (3i)[S-1] **(Table 2)**



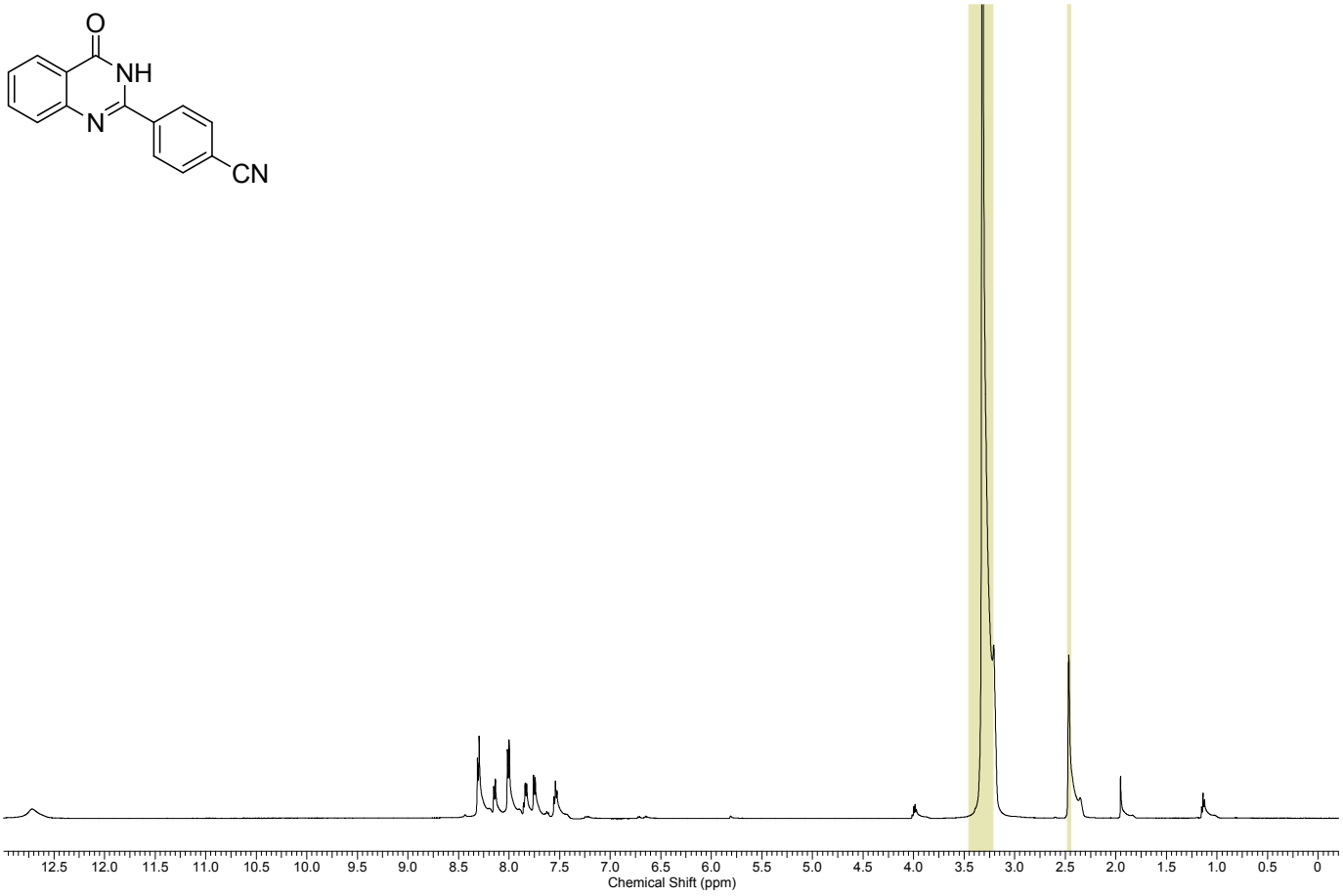
2-(4-bromophenyl)-4-quinazolinone (3j)[S-1] **(Table 2)**



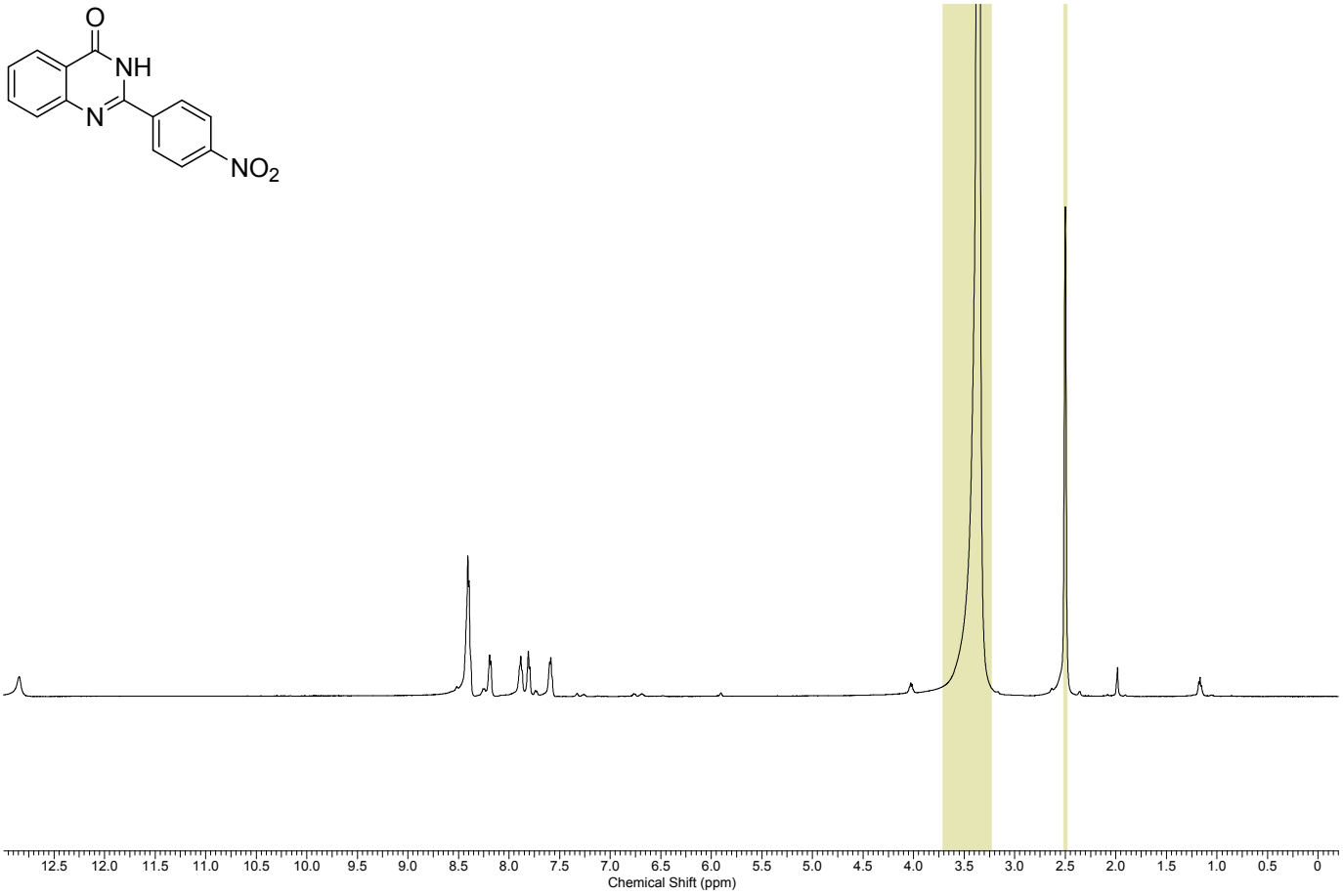
2-(4-trifluoromethylphenyl)-4-quinazolinone (3k)[S-1] (Table 2)



2-(4-cyanophenyl)-4-quinazolinone (3l)[S-3] (Table 2)



2-(4-nitrophenyl)-4-quinazolinone (3m)[S-1] **(Table 2)**



2-phenyl-2,3-dihydroquinazolin-4-one (4a)[S-4] **(Scheme 2)**