Electronic Supplementary Material (ESI) for RSC Advances

Iodine-Catalyzed Oxidative System for Cyclization of Primary Alcohols with o-aminobenzamides to Quinazolinones using DMSO as Oxidant in Dimethyl Carbonate

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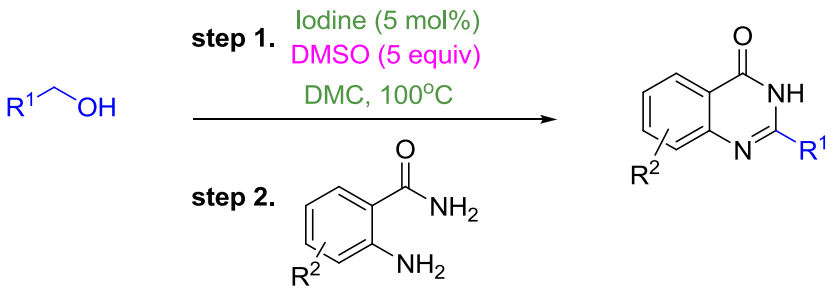


Experimental

All chemicals (AR grade) were obtained from commercial sources and were used without further purification. Petroleum ether (PE) refers to the fraction boiling in the 60-90 °C range. The progress of the reactions was monitored by TLC (silica gel, Polygram SILG/ UV 254 plates). Column chromatography was performed on Silicycle silica gel (200-300 mesh). Melting points were obtained using a Yamato melting point apparatus Model MP-21 and are uncorrected. ^{1}H and ^{13}C NMR spectra were obtained using a Bruker DRX 500 (500 MHz) spectrometer in $CDCl_3$ or DMSO-d_6 with TMS as the internal standard. All the products are known compounds and they were identified by comparison of their physical and spectral data with those reported in the literature.

General procedure for iodine-catalyzed cyclization primary alcohols with o-aminobenzamides

A mixture of primary alcohol **1** (1.2 mmol), and DMSO (5 mmol) were dissolved in DMC (1ml) at 100℃ in a sealed tube, then iodine (0.05 mmol, 5 mol%) was added. The reaction proceeded under an air atmosphere for the indicated time. Then the reaction solution was cooled to room temperature and o-aminobenzamide (1 mmol) was added. The reaction proceeded for the indicated time until complete consumption of starting material as monitored by TLC. The solution was diluted with DMC (5 ml), washed with H_2O (3 × 5 mL), and then the organic layer was separated and concentrated under vacuum and the crude product was purified by column

chromatography (PE:EtOAc, 1:1) or recrystallization (PE:EtOAc, 3:1) to provide the analytically pure product **3**.

2-Phenylquinazolin-4(*3H*)-one (3a)
White solid; mp 236-238°C (Lit.[13] 234-235°C).
^{1}H NMR (500 MHz, $CDCl_3$): δ = 11.58 (br s, 1 H), 8.35 (d, J=8.0Hz, 1 H), 8.26-8.28 (m, 2H), 7.80-7.87 (m, 2H), 7.60-7.61 (m, 3H), 7.52 (t, J=7.5, 1H).
MS (ESI, M/Z): 222 [M+].

2-(*p*-Tolyl)quinazolin-4(*3H*)-one (3b)
White solid; mp 256-257 °C (Lit.[13] 259-260 °C).
^{1}H NMR (500 MHz, DMSO-d_6): δ = 12.45 (br s, 1 H), 8.14 (d, J=8.0Hz, 1H), 8.09 (d, J=8.0Hz, 2H), 7.80-7.83 (m, 1H), 7.71 (d, J=8.0Hz, 1H), 7.48-7.51 (m, 1H), 7.34 (d, J=8.0Hz, 2H), 2.38 (s, 3H).

2-(4-Methoxyphenyl)quinazolin-4(*3H*)-one (3c)
White solid; mp 248-249 °C (Lit.[13] 247-248 °C).
^{1}H NMR (500 MHz, $CDCl_3$): δ = 11.17 (br s, 1 H), 8.33 (d, J=8.0Hz, 1H), 8.21 (d, J=9.0Hz, 2H), 7.79-7.82 (m, 1H), 7.47-7.55 (m, 2H), 7.08 (d, J=9.0Hz, 2H), 3.92 (s, 3H).

2-(4-N, N'-dimethylphenyl)quinazolin-4(*3H*)-one (3d)
White solid; mp 237-238 °C (Lit.[8l] 237-239 °C).
^{1}H NMR (500 MHz, DMSO-d_6): δ = 12.16 (br s, 1 H), 8.09 (t, J=8.0Hz, 3H), 7.76 (t, J=7.5Hz, 1H), 7.64 (d, J=8.0Hz, 1H), 7.41 (t, J=7.5Hz, 1H), 6.78 (d, J=9.0Hz, 2H), 3.00 (s, 6H).

2-(4-Hydroxyphenyl)quinazolin-4(*3H*)-one (3e)
White solid; mp >300 °C (Lit.[8n] >300 °C).
^{1}H NMR (500 MHz, DMSO-d_6): δ = 12.29 (br s, 1 H), 10.14 (s, 1H), 8.07-8.12 (m, 3H), 7.79 (t, J=7.5Hz, 1H), 7.67 (d, J=8.0Hz, 1H), 7.45 (t, J=7.5Hz, 1H), 7.88 (d, J=8.0Hz, 2H).

2-(2-Hydroxyphenyl)quinazolin-4(*3H*)-one (3f)
White solid; mp 253-255 °C (Lit.[8l] 252-254 °C).
^{1}H NMR (500 MHz, DMSO-d_6): δ = 13.77 (s, 1H), 12.46 (br s, 1 H), 8.22 (d, J=8.0Hz, 1H), 8.15 (d, J=8.0Hz, 1H), 7.84-7.87 (m, 1H), 7.76 (d, J=10Hz, 1H), 7.54 (t, J=7.5Hz, 1H), 7.45 (t, J=8.0Hz, 1H), 7.00 (d, J=8.0Hz, 1H), 6.95 (t, J=8.0Hz, 1H).

2-(4-Chlorophenyl)quinazolin-4(*3H*)-one (3g)
White solid; mp 298-300 °C (Lit.[13] 299-300 °C).
^{1}H NMR (500 MHz, DMSO-d_6): δ = 12.60 (br s, 1 H), 8.20 (d, J=9.0Hz, 2H), 8.14-8.16 (m, 1H), 7.82-7.85 (m, 1H), 7.74 (d, J=8.0Hz, 1H), 7.62 (d, J=8.5Hz, 2H), 7.51-7.54 (m, 1H).

2-(4-Bromophenyl)quinazolin-4(*3H*)-one (3h)
White solid; mp 294-296 °C (Lit.[13] 296-297 °C).
^{1}H NMR (500 MHz, DMSO-d_6): δ = 12.59 (br s, 1 H), 8.11-8.15 (m, 3H), 7.84 (t, J=8.0Hz, 1H), 7.75 (t, J=9.0Hz, 3H), 7.53 (t, J=7.0Hz, 1H).

2-(4-Nitrophenyl)quinazolin-4(*3H*)-one (3i)
Brown solid; mp >300 °C (Lit.[13] >300 °C).
^{1}H NMR (500 MHz, DMSO-d_6): δ = 12.82 (br s, 1 H), 8.36-8.42 (m, 4H), 8.17-8.18 (m, 1H), 7.86-7.89 (m, 1H), 7.79 (d, J=8.0Hz, 1H), 7.56-7.59 (m, 1H).

2-(2-Nitrophenyl)quinazolin-4(*3H*)-one (3j)
Brown solid; mp 194-196 °C (Lit.[8n] 193-194 °C).
^{1}H NMR (500 MHz, DMSO-d_6): δ = 12.82 (br s, 1 H), 8.19 (q, J=8.0Hz, 2H), 7.81-7.92 (m, 4H), 7.65 (d, J=8.0Hz, 1H), 7.57 (t, J=7.5Hz, 1H).

2-styrylquinazolin-4(*3H*)-one (3k)
White solid; mp 222-224 °C (Lit.[8o] 220-228 °C).
^{1}H NMR (500 MHz, DMSO-d_6): δ = 12.33(br s, 1 H), 8.10 (d, J=8.0Hz, 1H), 7.95 (d, J=16Hz, 1H), 7.80 (t J=8.0Hz, 1H), 7.66 (t, J=8.0Hz, 3H), 7.39-7.49 (m, 4H), 7.00 (d, J=16Hz, 1H).

2-(2-Furanyl)-Quinazolinone-4(*3H*)-one (3l)
White solid; mp 218-221 °C (Lit.[10b] 219-220 °C).
^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.30 (d, J=8.0Hz, 1H), 8.14-8.16 (m, 2H), 7.82-7.86 (m, 1H), 7.76 (s, 1H), 7.54 (t, J=8.0Hz, 1H), 6.73 (q, J=1.5Hz, 1H).

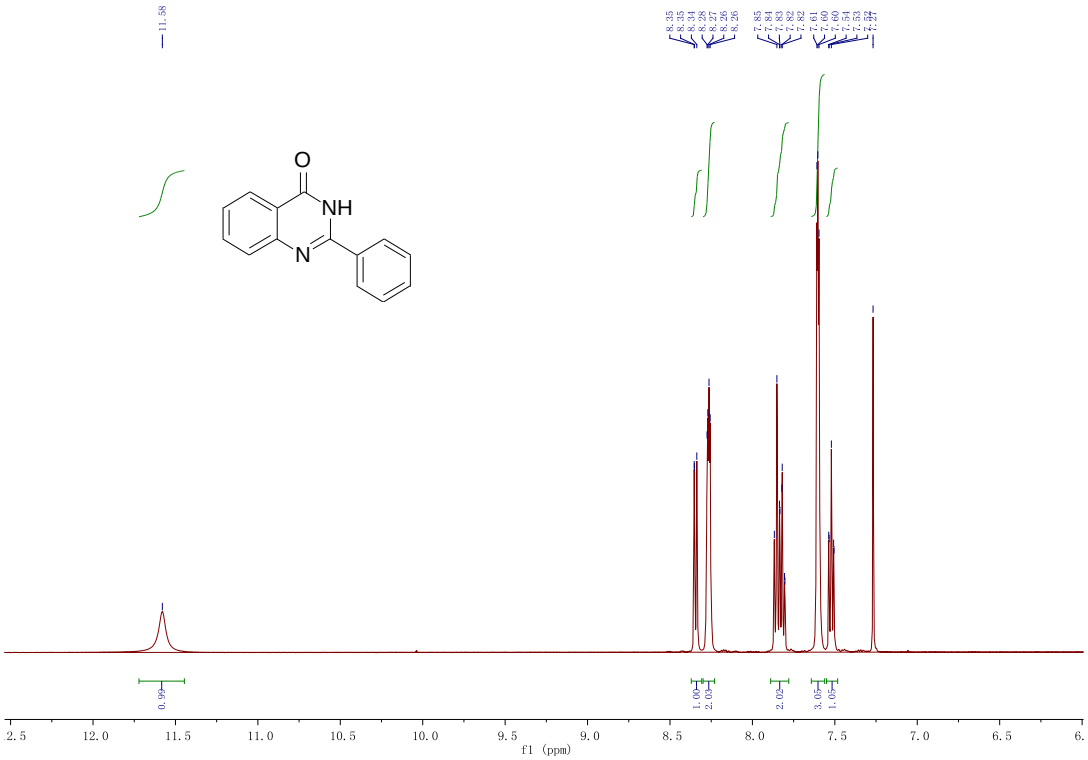
2-(2-Thienyl)-quinazolinone-4(*3H*)-one (3m)
White solid; mp 277-278 °C (Lit.[10b] 275-276 °C).
^{1}H NMR (500 MHz, DMSO-d_6): δ = 12.65 (br s, 1 H), 8.22 (d, J=3.0Hz, 1H), 8.12 (d, J=8.0Hz, 1H), 7.86 (d, J=5.0Hz, 1H), 7.78-7.81 (m, 1H), 7.64 (d, J=8.0Hz, 1H), 7.48 (t, J=8.0Hz, 1H), 7.22-7.24(m, 1H).

2-Benzylquinazolin-4(*3H*)-one (3n)
White solid; mp 245-247 °C (Lit.[13] 244-246 °C).
^{1}H NMR (500 MHz, $CDCl_3$): δ = 10.00 (br s, 1 H), 8.25 (d, J=8.0Hz, 1H), 7.78-7.81(m, 2H), 7.49 (t, J=8.0Hz, 1H), 7. 42 (d, J=7.0Hz, 2H), 7.36 (t, J=8.0Hz, 2H), 7.29-7.32 (m, 1H), 4.12 (s, 2H).

2-Propylquinazolinone-4(*3H*)-one (3o)
White solid; mp 201-202 °C (Lit.[10c] 200-202 °C).
^{1}H NMR (500 MHz, $CDCl_3$): δ = 12.01 (br s, 1 H), 8.31 (d, J=8.0Hz, 1H), 7.73-7.80 (m, 2H), 7.49 (t, J=7.5Hz, 1H), 2.81 (t, J=7.5Hz, 2H), 1.95 (h, J=7.5Hz, 2H), 1.10 (t, J=7.5Hz, 3H).

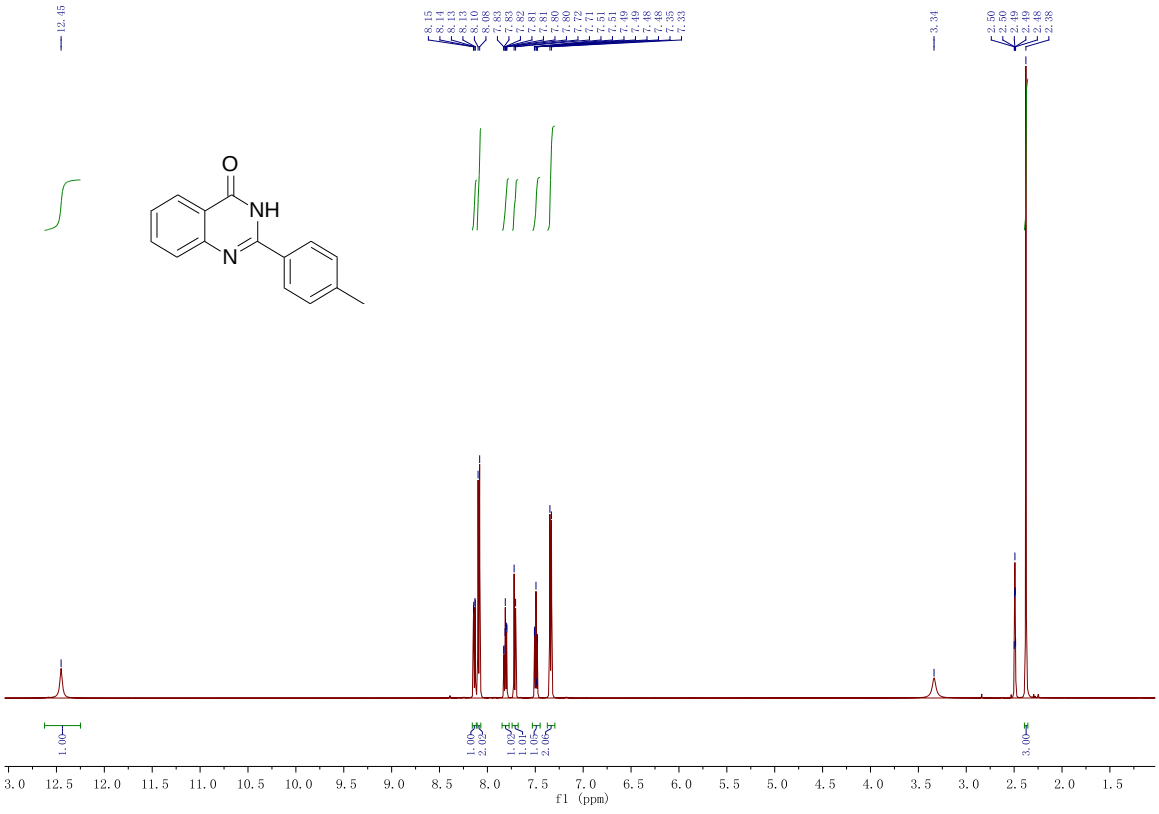
2-Phenylquinazolin-4(*3H*)-one (3a)

^{1}H NMR (500 MHz, $CDCl_3$)



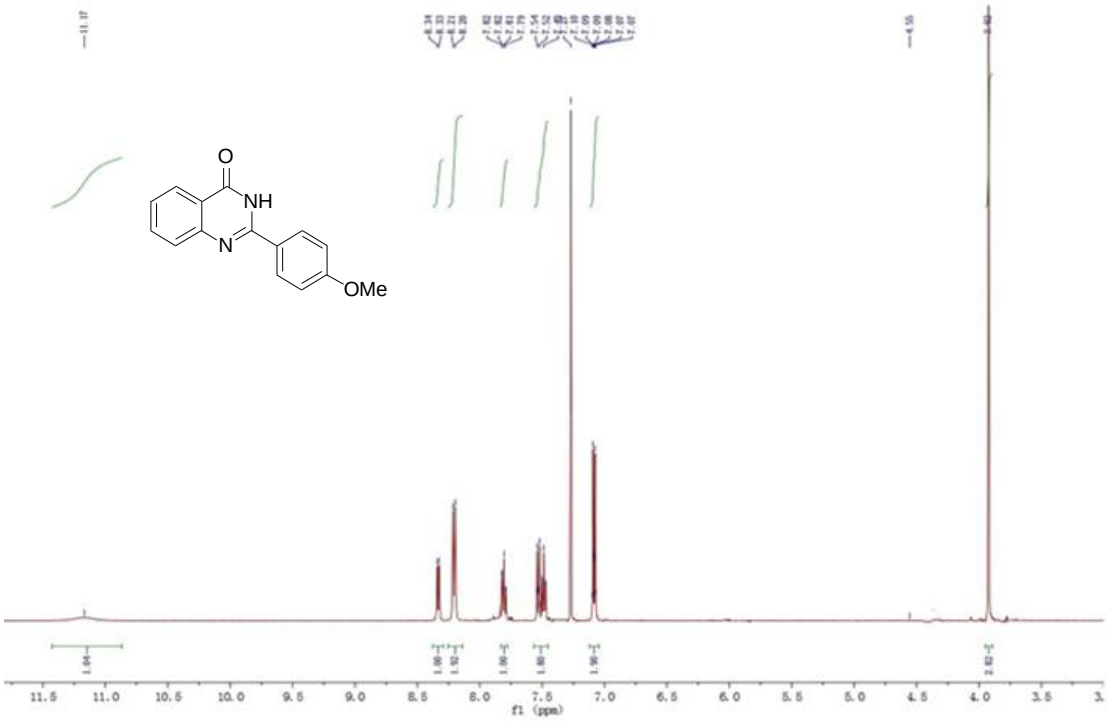
2-(*p*-Tolyl)quinazolin-4(*3H*)-one (3b)

^{1}H NMR (500 MHz, DMSO-d_6)



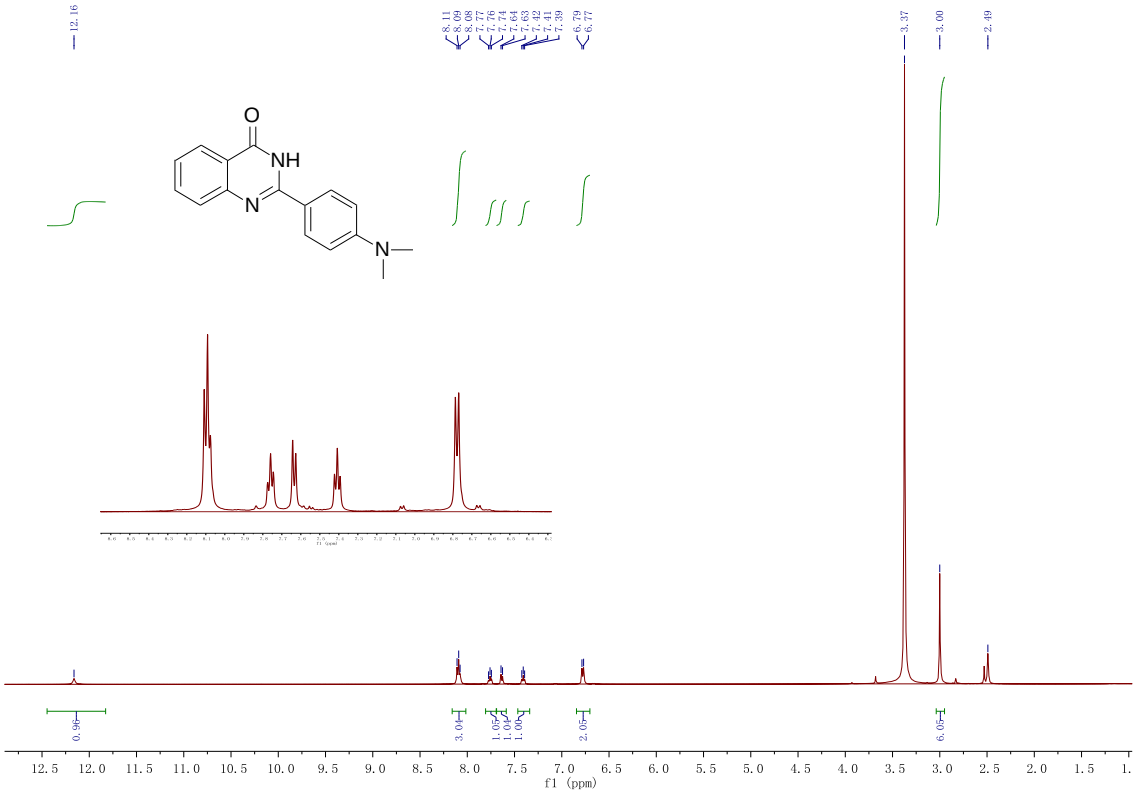
2-(4-Methoxyphenyl)quinazolin-4(*3H*)-one (3c)

^{1}H NMR (500 MHz, $CDCl_3$)



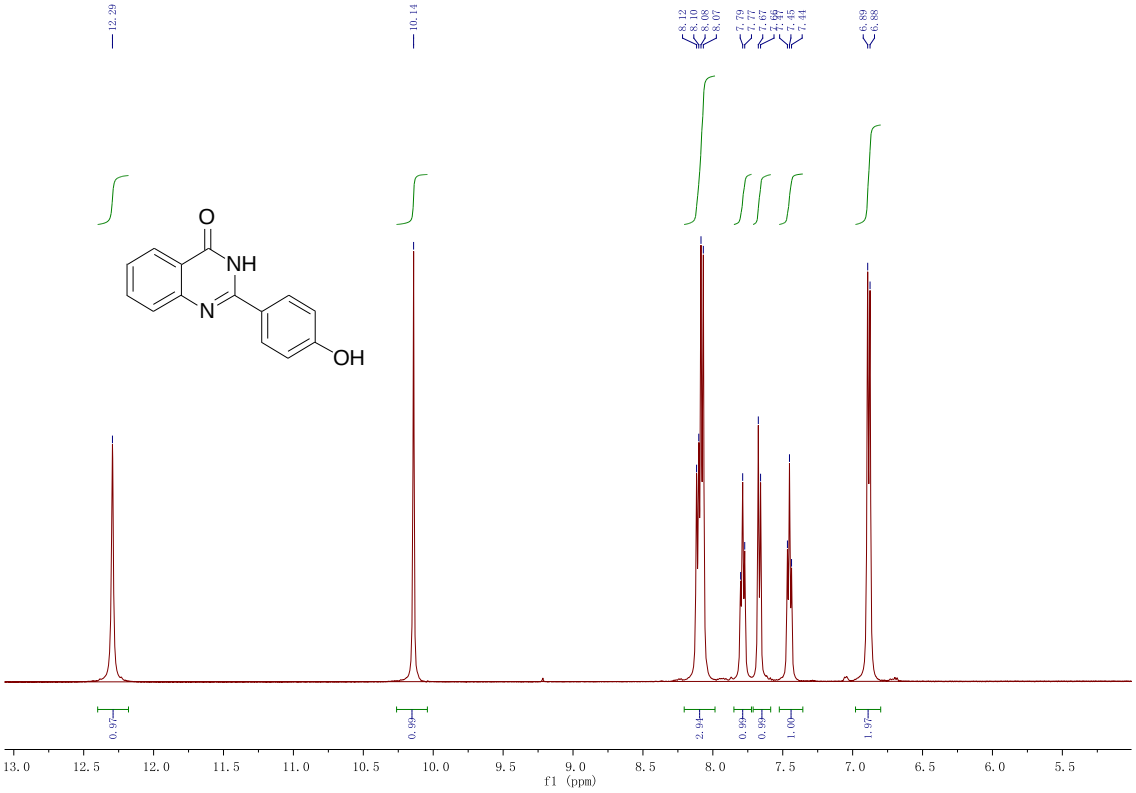
2-(4-N, N'-dimethylphenyl)quinazolin-4(*3H*)-one (3d)

^{1}H NMR (500 MHz, DMSO-d_6)



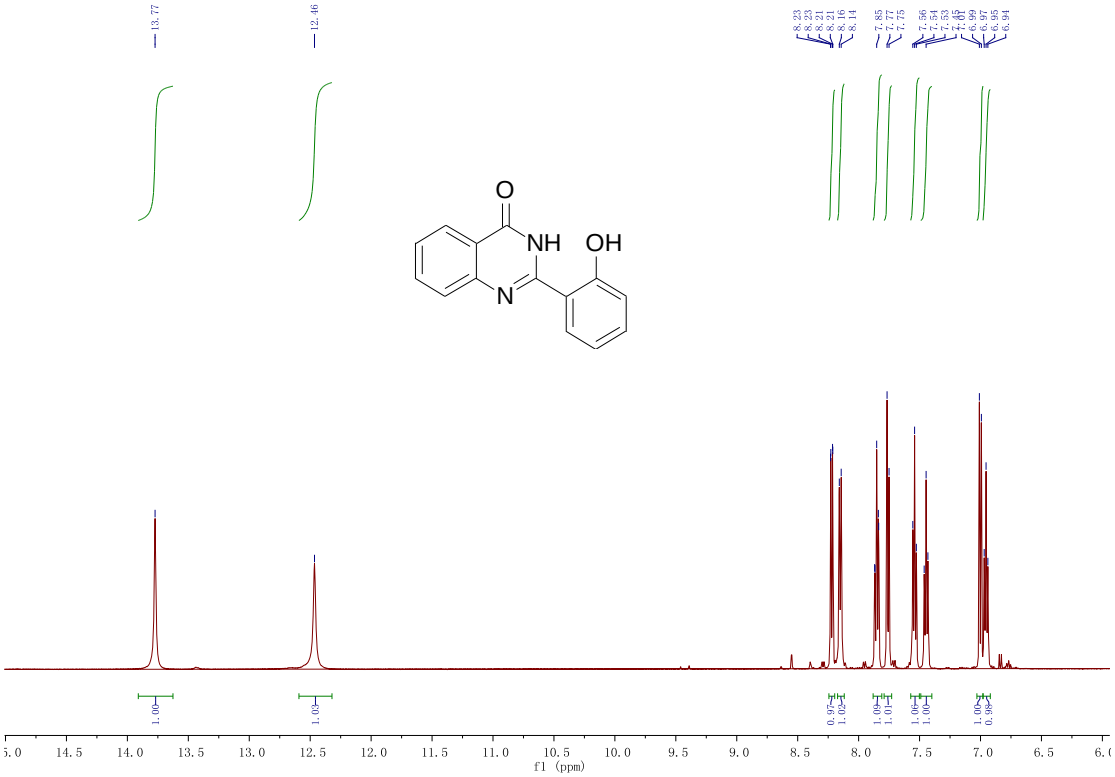
2-(4-Hydroxyphenyl)quinazolin-4(*3H*)-one (3e)

^{1}H NMR (500 MHz, DMSO-d_6)



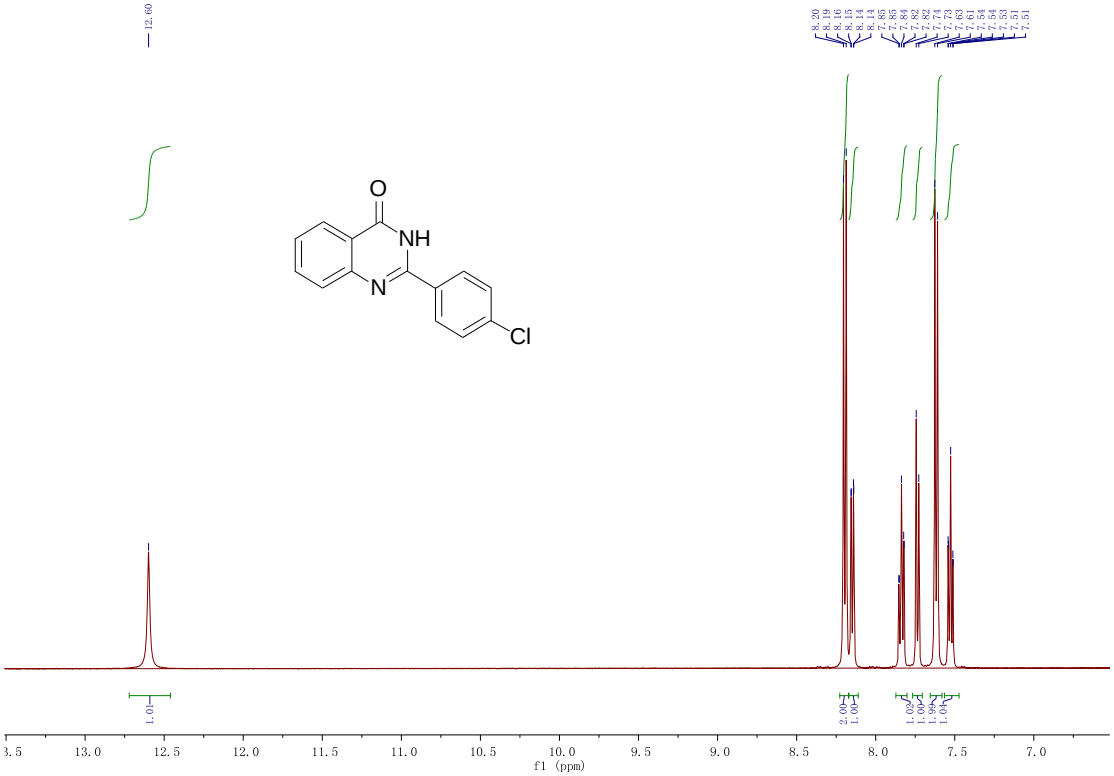
2-(2-Hydroxyphenyl)quinazolin-4(*3H*)-one (3f)

^{1}H NMR (500 MHz, DMSO-d_6)



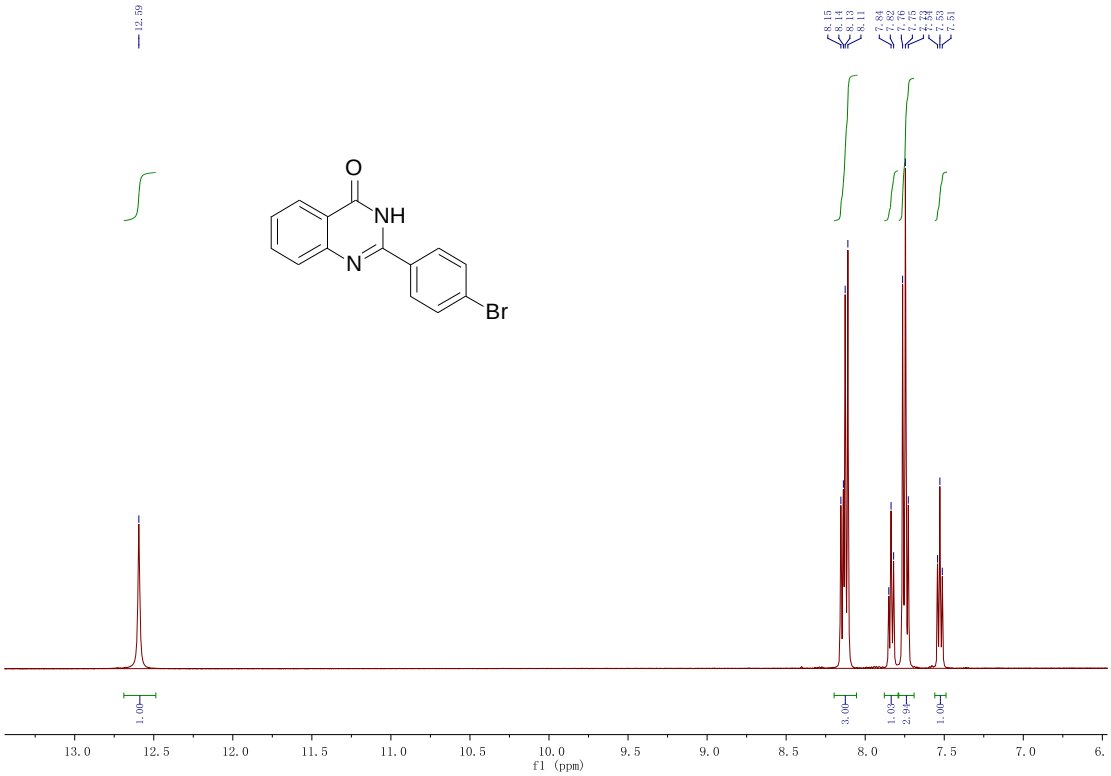
2-(4-Chlorophenyl)quinazolin-4(*3H*)-one (3g)

^{1}H NMR (500 MHz, DMSO-d_6)



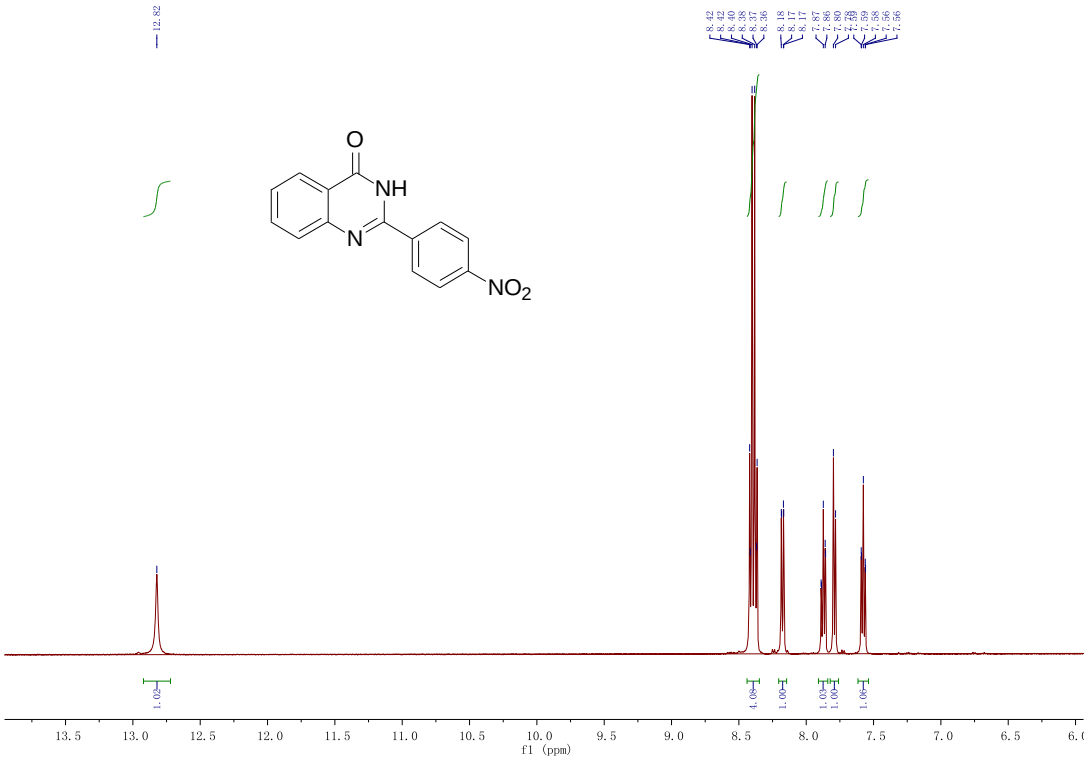
2-(4-Bromophenyl)quinazolin-4(*3H*)-one (3h)

^{1}H NMR (500 MHz, DMSO-d_6)



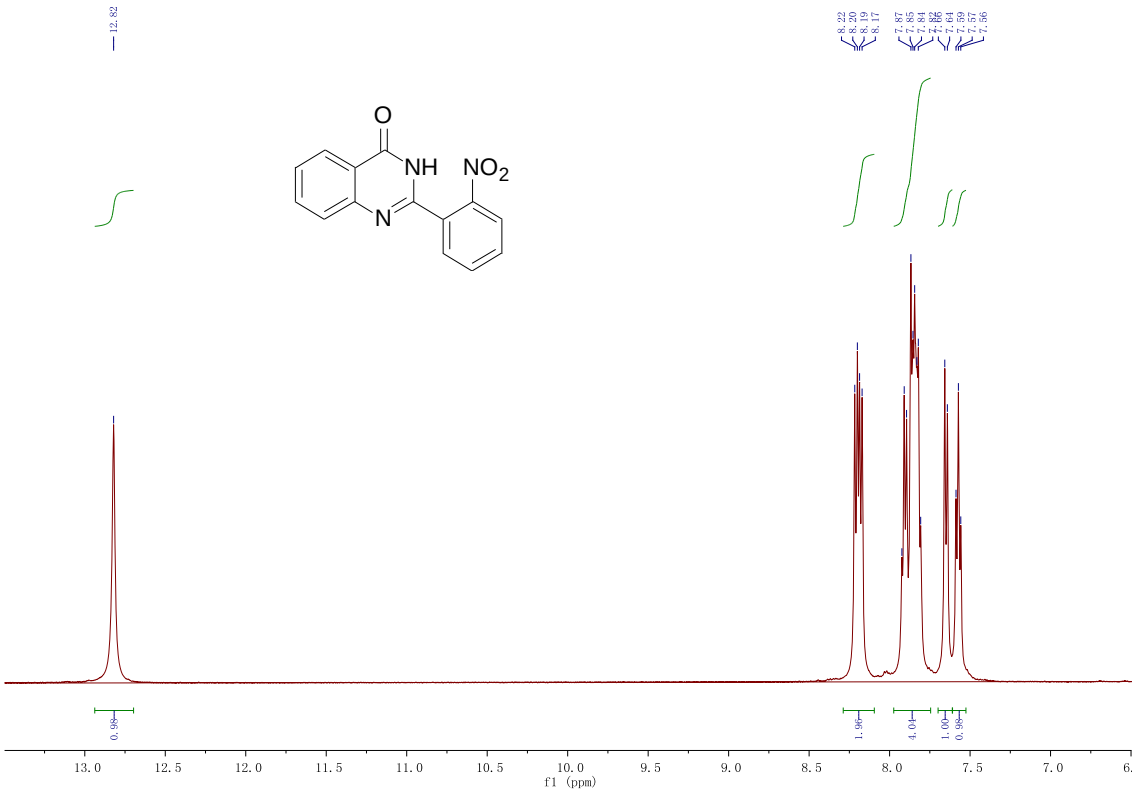
2-(4-Nitrophenyl)quinazolin-4(*3H*)-one (3i)

^{1}H NMR (500 MHz, DMSO-d_6)



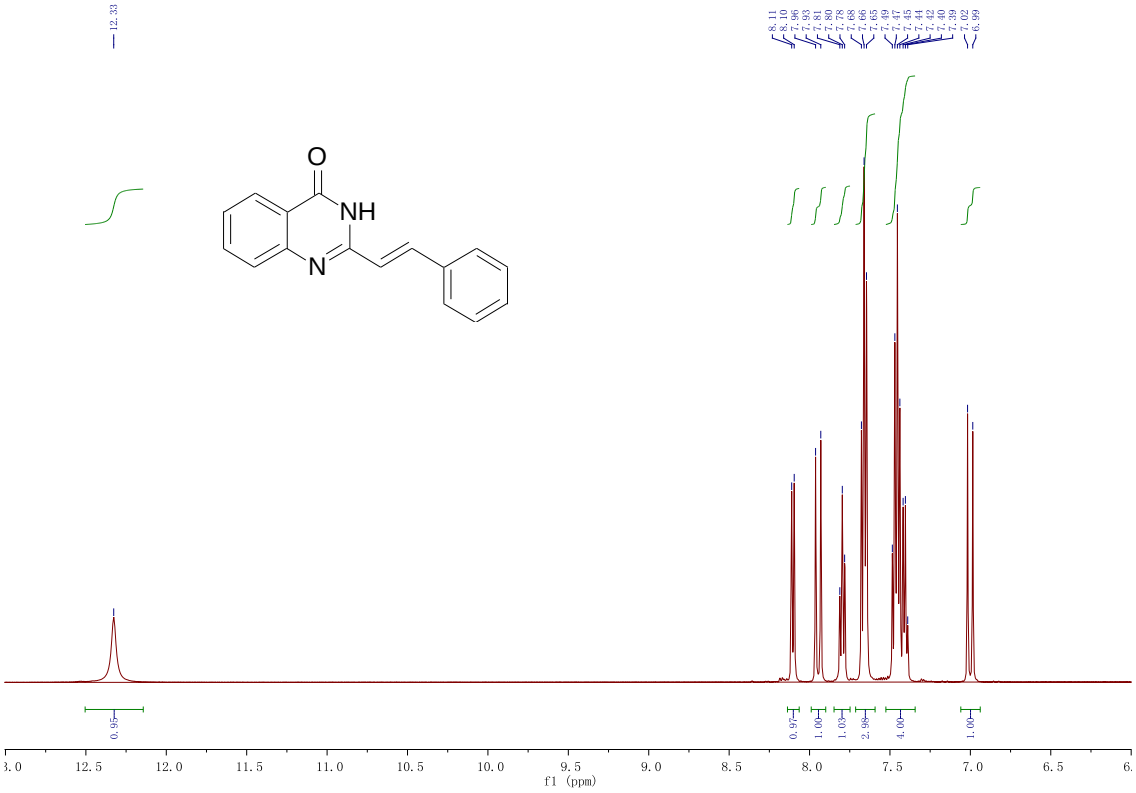
2-(2-Nitrophenyl)quinazolin-4(*3H*)-one (3j)

^{1}H NMR (500 MHz, DMSO-d_6)



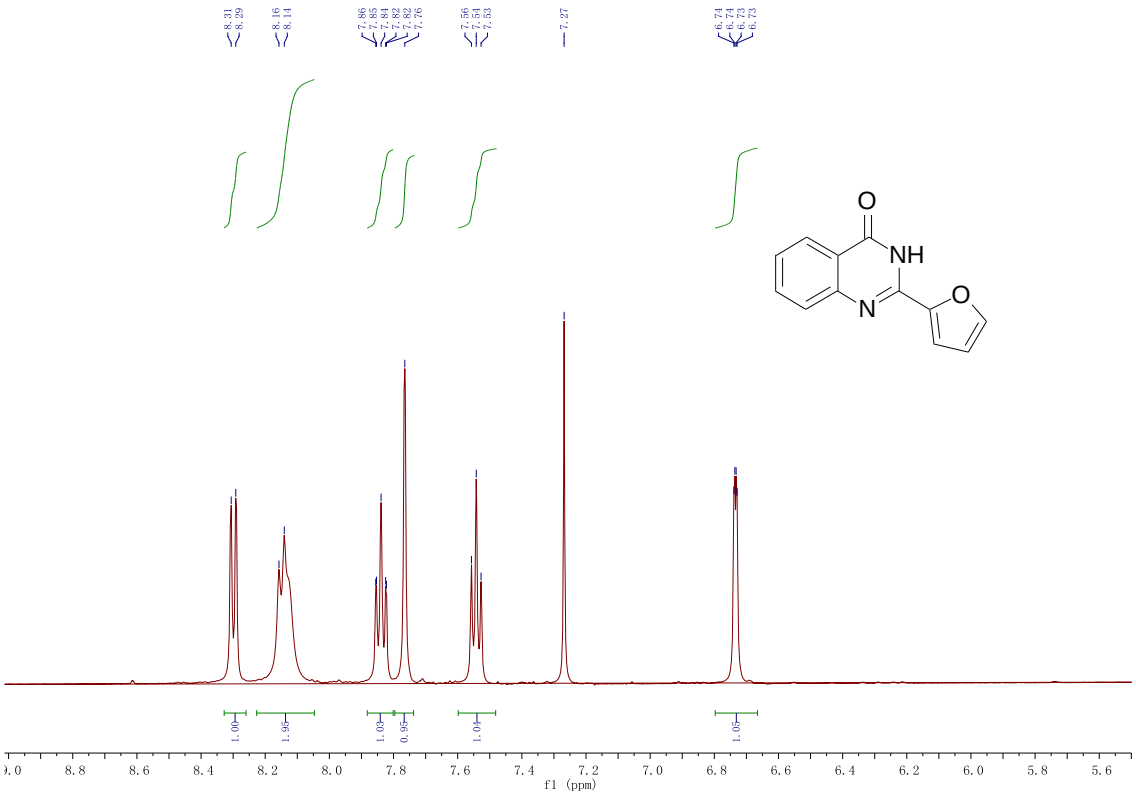
2-styrylquinazolin-4(*3H*)-one (3k)

^{1}H NMR (500 MHz, DMSO-d_6)



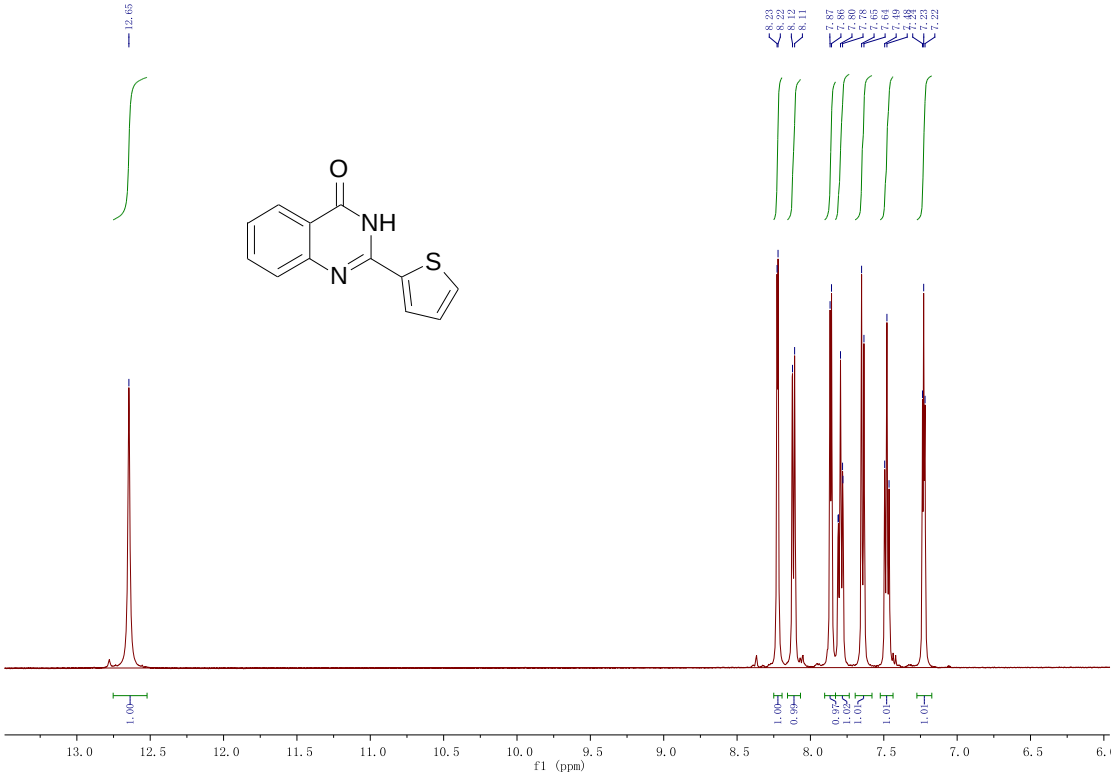
2-(2-Furanyl)-Quinazolinone-4(*3H*)-one (3l)

^{1}H NMR (500 MHz, $CDCl_3$)



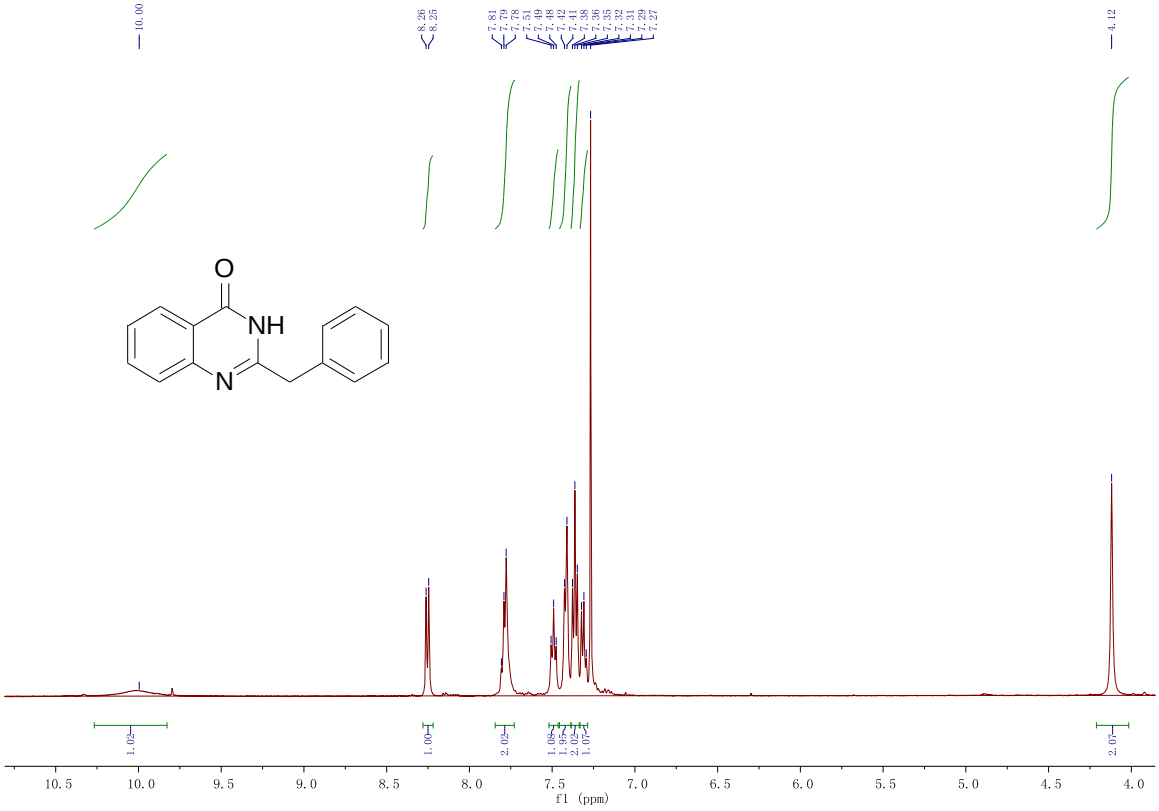
2-(2-Thienyl)-quinazolinone-4(*3H*)-one (3m)

^{1}H NMR (500 MHz, DMSO-d_6)



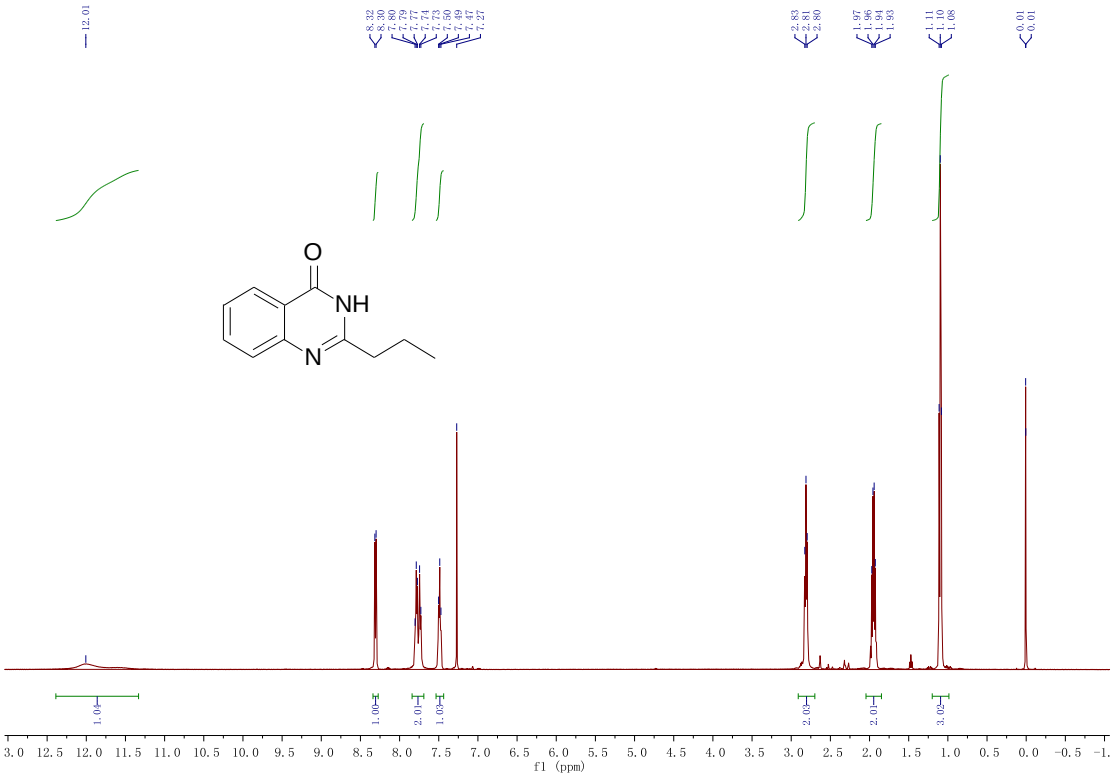
2-Benzylquinazolin-4(*3H*)-one (3n)

^{1}H NMR (500 MHz, $CDCl_3$)



2-Propylquinazolinone-4(*3H*)-one (3o)

^{1}H NMR (500 MHz, $CDCl_3$)



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