Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry


*Supporting Information*

# Cross-Coupling/Annulations of Quinazolinones with Alkynes for Access to Fused Polycyclic Heteroarenes under Mild Conditions

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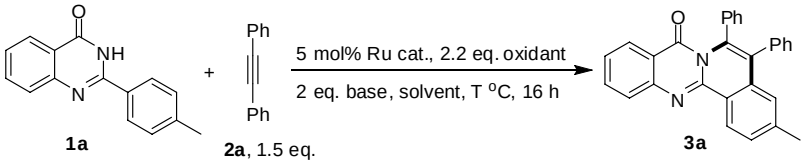
**General information:**

All reactions were performed in reaction tubes under air. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63 μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr (house vacuum) at 25–35°C. Commercial reagents and solvents were used as received. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale.

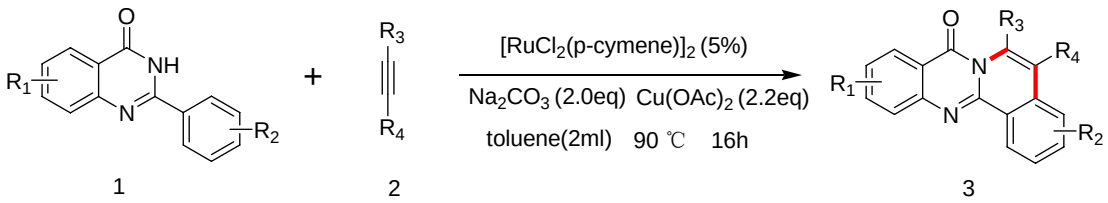
## Optimization table for the reaction:




| entry | solvent | oxidant | base | T ($^{o}$C) | yield (%)[b] |
|---|---|---|---|---|---|
| 1[c] | PhCl | $Cu(OAc)_2$ | $Na_2CO_3$ | 90 | 82 |
| 2 | PhCl | $Cu(OAc)_2$ | $Na_2CO_3$ | 90 | 81 |
| 3 | Toluene | $Cu(OAc)_2$ | $Na_2CO_3$ | 90 | 92 |
| 4 | DCE | $Cu(OAc)_2$ | $Na_2CO_3$ | 90 | 75 |
| 5 | THF | $Cu(OAc)_2$ | $Na_2CO_3$ | 90 | 55 |
| 6 | DMF | $Cu(OAc)_2$ | $Na_2CO_3$ | 90 | 42 |
| 7 | NMP | $Cu(OAc)_2$ | $Na_2CO_3$ | 90 | 21 |
| 8 | MeCN | $Cu(OAc)_2$ | $Na_2CO_3$ | 90 | 10 |
| 9 | MeOH | $Cu(OAc)_2$ | $Na_2CO_3$ | 90 | 16 |
| 10 | Toluene | $Cu(OAc)_2$*$H_2O$ | $Na_2CO_3$ | 90 | 86 |
| 11 | Toluene | $CuSO_4$ | $Na_2CO_3$ | 90 | 55 |
| 12 | Toluene | $CuNO_3$ | $Na_2CO_3$ | 90 | trace |
| 13 | Toluene | AgOAc | $Na_2CO_3$ | 90 | 87 |
| 14 | Toluene | $Ag_2CO_3$ | $Na_2CO_3$ | 90 | 29 |
| 15 | Toluene | $Ag_2O$ | $Na_2CO_3$ | 90 | 25 |
| 16 | Toluene | $Cu(OAc)_2$ | $K_2CO_3$ | 90 | 45 |
| 17 | Toluene | $Cu(OAc)_2$ | $Cs_2CO_3$ | 90 | 42 |
| 18 | Toluene | $Cu(OAc)_2$ | NaOH | 90 | 41 |
| 19 | Toluene | $Cu(OAc)_2$ | $K_3PO_4$ | 90 | 79 |
| 20 | Toluene | $Cu(OAc)_2$ | t-BuOK | 90 | 39 |
| 21 | Toluene | $Cu(OAc)_2$ | $Et_3N$ | 90 | 85 |
| 22 | Toluene | $Cu(OAc)_2$ | DBU | 90 | 45 |
| 23 | Toluene | $Cu(OAc)_2$ | DABCO | 90 | 42 |
| 24 | Toluene | $Cu(OAc)_2$ | $Na_2CO_3$ | 120 | 67 |
| 25 | Toluene | $Cu(OAc)_2$ | $Na_2CO_3$ | 60 | 87 |

[a] The reaction was carried out on a 0.2 mmol of **1a** with 1.5 equiv of **2a** in presence of 5 mol% catalyst $[RuCl_2(p\text{-cymene})]_2$, 2.2 equiv of oxidant and 2.0 equiv of base additive in 3 mL solvent at indicated temperature for 16 hours. [b] Isolated yield. [c] Under $N_2$ atmosphere.

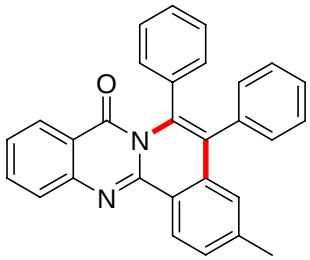
## General experimental procedure




1 2 3

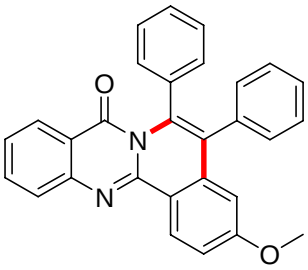
A mixture of quinazolinone (**1**) (0.2mmol, 1.0eq), the alkyne (**2**) (0.3mmol, 1.5eq), $[RuCl_2(p\text{-cymene})]_2$ (5%), $Na_2CO_3$ (0.4mmol, 2.0eq), $Cu(OAc)_2$ (0.44mmol, 2.2eq), toluene (2ml) were added to a reaction tube. The mixture was stirred at 90 $^oC$ for 16 hours. Afterwards, it was diluted with $CH_2Cl_2$ and transferred to a round bottom flask. Silica was added to the flask and volatiles were evaporated under reduced pressure. The purification was performed by flash column chromatography on silica gel.

## Characterization data



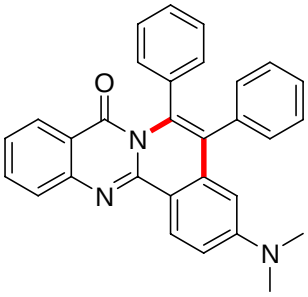

**3-methyl-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3a)**.

Compound was obtained as a white solid: mp 264-265 ℃; yield 92%; $^1H$ NMR (400 MHz, $CDCl_3$): δ 9.00 (d, $J$ = 8.4 Hz, 1H), 8.15 (d, $J$ = 8.0 Hz, 1H), 7.85 (d, $J$ = 7.6 Hz, 1H), 7.78 (t, $J$ = 7.2 Hz, 1H), 7.43 (d, $J$ = 8.0 Hz, 1H), 7.36 (t, $J$ = 7.2 Hz, 1H), 7.27-7.23 (m, 3H), 7.10-7.05 (m, 7H), 6.95 (s, 1H), 2.36 (s, 3H); $^{13}C$ NMR (100 MHz, $CDCl_3$): δ 161.4, 147.7, 147.0, 142.7, 137.2, 135.6, 135.3, 134.4, 133.9, 131.2, 129.8, 128.5, 128.0, 127.7, 127.2, 127.2, 126.8, 126.7, 126.2, 125.4, 124.9, 120.1, 22.0; HRMS calcd. For $C_{29}H_{20}KN_2O^+$ $[M + K]^+$ 451.1213, found: 451.1224; IR ($cm^{-1}$) ν 2922, 1706, 1599, 1541, 1487, 1444, 1292, 1155, 822, 770, 694.



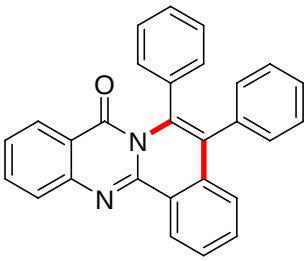

**3-methoxy-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3b).**

Compound was obtained as a white solid: mp 246-247 ℃; yield 95%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 9.04 (d, *J* = 8.8 Hz, 1H), 8.14 (d, *J* = 8.0 Hz, 1H), 7.84-7.76 (m, 2H), 7.73-7.70 (m, 1H), 7.54- 7.51 (m, 1H), 7.35 (t, *J* = 6.8 Hz, 1H), 7.28- 7.22 (m, 1H), 7.19 (d, *J* = 8.8 Hz, 1H), 7.13-7.11 (m, 3H), 7.08-7.05 (m, 4H), 6.57 (d, *J* = 6.4 Hz, 1H), 3.72 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 162.6, 161.4, 147.5, 147.2, 137.1, 135.9, 135.5, 134.4, 131.1, 129.3, 128.5, 128.1, 127.6, 127.3, 127.2, 127.2, 126.9, 126.5, 125.1, 120.7, 119.8, 116.3, 109.0, 55.4; HRMS calcd. For $C_{29}H_{20}N_2NaO_2^+$ [M + Na]$^+$ 451.1422, found: 451.1440; IR (cm$^{-1}$) v 2918, 1700, 1608, 1541, 1492, 1461, 1285, 1236, 857, 761, 702.



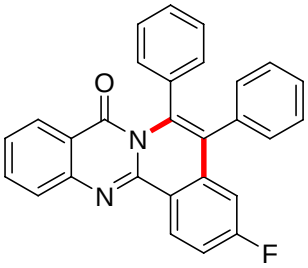

**3-(dimethylamino)-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3c).**

Compound was obtained as a yellow solid: mp 278-279 ℃; yield 96%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.91 (d, J = 9.2 Hz, 1H), 8.12 (d, J = 7.6 Hz, 1H), 7.79-7.71 (m, 3H), 7.52-7.51 (m, 1H), 7.31-7.22 (m, 3H), 7.10-6.97 (m, 6H), 6.98 (d, J = 8.8 Hz, 1H), 6.19 (s, 1H), 2.88 (s, 6H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.6, 152.6, 148.2, 147.7, 137.6, 136.1, 135.6, 134.2, 132.3, 131.1, 130.9, 128.9, 128.7, 128.5, 127.9, 127.2, 127.1, 126.6, 126.2, 124.2, 119.5, 116.0,113.5, 106.6, 39.1; HRMS calcd. For $C_{30}H_{24}N_3O^+$ [M + H]$^+$ 442.1919, found: 442.1920; IR (cm$^{-1}$) v 2960, 1728, 1608, 1537, 1492, 1467, 1290, 1124, 842, 778, 698.




**5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3d)**.

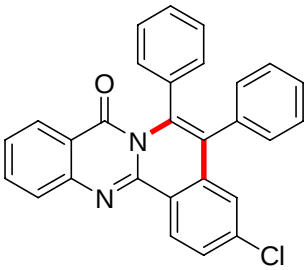
Compound was obtained as a white solid: mp 261-262 ℃; yield 77%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 9.11 (d, $J$ = 7.6 Hz, 1H), 8.16 (d, $J$ = 7.6 Hz, 1H), 7.88 (d, $J$ = 8.0 Hz, 1H), 7.81 (t, $J$ = 6.8 Hz, 1H), 7.62 (t, $J$ = 6.8 Hz, 1H), 7.55 (t, $J$ = 7.2 Hz, 1H), 7.40 (t, $J$ = 6.8 Hz,1H), 7.25 (m, 3H), 7.18 (d, $J$ = 7.6 Hz, 1H), 7.12-7.07 (m, 7H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.3, 147.5, 146.9, 137.0, 135.5, 135.2, 134.5, 133.9, 132.0, 131.2, 128.5, 128.3, 128.0, 127.8, 127.3, 127.2, 127.1, 126.9, 126.8, 126.2, 125.6, 120.3; HRMS calcd. For $C_{28}H_{19}N_2O^+$ $[M + H]^+$ 399.1497, found: 399.1506; IR ($cm^{-1}$) v 2930, 1697, 1607, 1546, 1487, 1468, 1298, 1137, 768, 696.




**3-fluoro-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3e).**

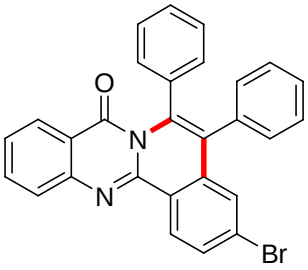
Compound was obtained as a white solid: mp 247-248 ℃; yield 67%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 9.13 (d, $J$ = 9.2 Hz, 1H), 8.16 (d, $J$ = 8.0 Hz, 1H), 7.87-7.82 (m, 2H), 7.41 (t, $J$ = 6.8 Hz, 1H), 7.33 (d, $J$ = 8.0 Hz, 1H), 7.35-7.36 (m, 3H), 7.14-7.06 (m, 7H), 6.84 (d, $J$ = 9.6 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 165.1 (d, $^{1}J_{CF}$ = 252 Hz), 161.2, 146.9, 146.8, 136.7, 136.6, 136.3 (d, $^{3}J_{CF}$ = 10 Hz), 135.0, 134.6, 131.0, 130.3, 130.2, 128.4, 128.3, 127.6, 127.2, 127.1, 127.0, 126.7, 125.7, 123.7, 120.1, 116.6 (d, $^{2}J_{CF}$ = 23 Hz),111.9 (d, $^{2}J_{CF}$ = 23 Hz); HRMS calcd. For $C_{28}H_{18}FN_2O^+$ $[M + H]^+$ 417.1403, found: 417.1423; IR ($cm^{-1}$) v 2919, 1704, 1613, 1549, 1489, 1469,

1292, 1202, 863, 766, 695.



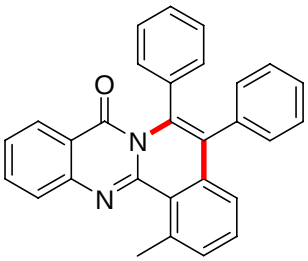

**3-chloro-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3f).**

Compound was obtained as a white solid: mp 236-237 ℃; yield 66%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 9.01 (d, *J* = 8.4 Hz, 1H), 8.14 (d, *J* = 8.0 Hz, 1H), 7.84-7.77 (m, 2H), 7.53 (d, *J* = 8.4Hz, 1H), 7.39 (t, *J* = 6.8 Hz, 1H), 7.26 (m, 3H), 7.12-7.06 (m, 8H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.1, 146.8, 146.7, 138.6, 136.7, 136.6, 135.3, 134.8, 134.6, 131.1, 128.9, 128.7, 128.4, 128.3, 127.6, 127.2, 127.1, 126.8, 126.6, 125.9, 125.7, 125.6, 120.2; HRMS calcd. For $C_{28}H_{18}ClN_2O^+$ $[M + H]^+$ 433.1108, found: 433.1115; IR ($cm^{-1}$) v 2959, 1701, 1613, 1549, 1488, 1469, 1292, 1117, 863, 775, 695.



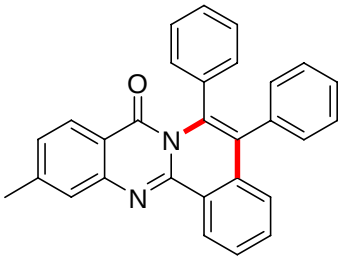

**3-bromo-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3g).**

Compound was obtained as a white solid: mp 223-224 ℃; yield 56%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.94 (d, *J* = 8.4 Hz, 1H), 8.14 (d, *J* = 8.0 Hz, 1H), 7.85-7.78 (m, 2H), 7.70 (d, *J* = 8.8 Hz, 1H), 7.40 (t, *J* = 6.8 Hz, 1H), 7.29-7.27 (m, 4H), 7.13-7.05 (m, 7H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.1, 147.0, 146.7, 136.7, 136.6, 135.5, 134.7, 134.6, 131.6, 131.0, 128.9, 128.7, 128.4, 128.3, 127.6, 127.2, 127.1, 126.8, 126.5, 126.1, 125.9, 120.3; HRMS calcd. For $C_{28}H_{18}BrN_2O^+$ $[M + H]^+$ 477.0603, found: 477.0597; IR ($cm^{-1}$) v 2930, 1712, 1604, 1544, 1479, 1330, 1288, 1175, 868, 744, 702.



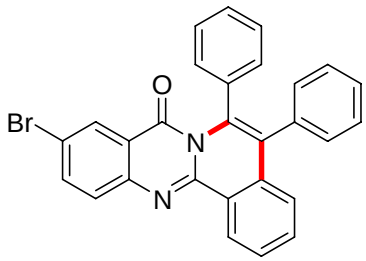

**1-methyl-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3h).**

Compound was obtained as a white solid: mp 226-227 ℃; yield 37%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.14 (d, *J* = 7.6 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.80 (t, *J* = 7.2 Hz, 1H), 7.44-7.37 (m, 3H), 7.26-7.24 (m, 3H), 7.09-7.06 (m, 8H), 3.25 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.6, 148.3, 146.2, 141.1, 137.0, 136.3, 135.6, 134.9, 134.2, 132.7, 131.3, 130.8, 128.5, 128.0, 127.2, 127.0, 126.9, 125.8, 125.7, 124.8, 120.1, 27.2; HRMS calcd. For $C_{29}H_{20}N_2NaO^+$ $[M + Na]^+$ 435.1473, found: 435.1486; IR ($cm^{-1}$) v 2926, 1689, 1606, 1552, 1490, 1443, 1282, 1139, 802, 764, 701.



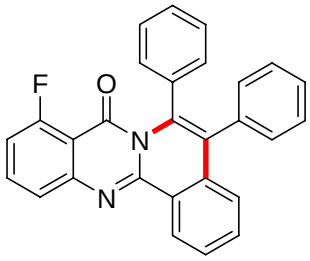

**11-methyl-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3i).**

Compound was obtained as a white solid: mp 251-252 ℃; yield 70%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 9.09 (d, *J* = 7.6 Hz, 1H), 8.05 (d, *J* = 7.6 Hz, 1H), 7.72-7.67 (m, 2H), 7.60 (t, *J* = 7.2 Hz, 1H), 7.56-7.52 (m, 2H), 7.25-7.18 (m, 3H), 7.17 (d, *J* = 7.6 Hz, 1H), 7.11-7.07 (m, 6H), 2.54 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.1, 147.6, 146.9, 145.4, 137.1, 135.6, 133.9, 132.3, 131.8, 131.1, 130.9, 128.8, 128.4, 128.2, 128.0, 127.4, 127.3, 127.2, 127.1, 127.0, 126.8, 126.4, 126.1, 117.9, 22.0; HRMS calcd. For $C_{29}H_{20}N_2NaO^+$ $[M + Na]^+$ 435.1473, found: 435.1464; IR ($cm^{-1}$) v 2960, 1690, 1610, 1544, 1483, 1442, 1282, 1074, 790, 700.



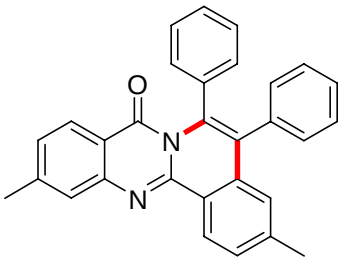

**10-bromo-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3j).**

Compound was obtained as a white solid: mp 230-231 ℃; yield 86%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 9.06 (d, $J$ = 8.0 Hz, 1H), 8.26 (s, 1H), 7.83 (d, $J$ = 8.4 Hz, 1H), 7.71 (d, $J$ = 8.4 Hz, 1H), 7.61-7.54 (m, 2H), 7.26-7.25 (m, 3H), 7.18 (d, $J$ = 7.6 Hz,1H), 7.13-7.06 (m, 7H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 160.1, 147.8, 145.7, 137.6, 136.7, 135.3, 135.1, 133.9, 132.3, 131.1, 131.0, 129.6, 128.7, 128.5, 128.2, 128.1, 127.4, 127.3, 127.2, 127.1, 126.3, 121.4, 118.8; HRMS calcd. For $C_{28}H_{18}BrN_2O^+$ $[M + H]^+$ 477.0603, found: 477.0582; IR ($cm^{-1}$) v 2930, 1711, 1600, 1542, 1482, 1466, 1288, 1134, 821, 758, 697.



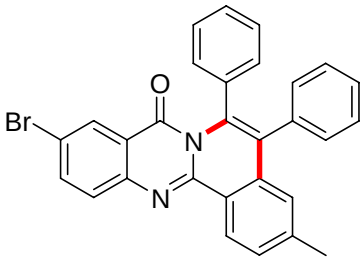

**9-fluoro-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3k).**

Compound was obtained as a white solid: mp 223-224 ℃; yield 82%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 9.07 (d, $J$ = 8.0 Hz, 1H), 7.74-7.69 (m, 2H), 7.67-7.59 (m, 3H), 7.54-7.52 (m, 1H), 7.30-7.26 (m, 2H), 7.21 (d, $J$ = 7.6 Hz, 1H), 7.11-7.06 (m, 6H), 7.03-7.01 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.0 (d, $^{1}J_{CF}$ = 265 Hz), 158.3, 148.9, 148.4, 136.4, 135.3, 135.0, 134.5 (d, $^{3}J_{CF}$ = 10 Hz), 134.2, 132.4, 132.3, 131.1, 131.0, 128.9, 128.5 (d, $^{4}J_{CF}$ = 5 Hz), 128.1, 127.8, 127.4, 127.1 (d, $^{4}J_{CF}$ = 4 Hz), 126.8, 126.3, 122.6 (d, $^{4}J_{CF}$ = 4 Hz), 111.7 (d, $^{2}J_{CF}$ = 21 Hz), 110.0 (d, $^{4}J_{CF}$ = 7 Hz); HRMS calcd. For $C_{28}H_{18}FN_2O^+$ $[M + H]^+$ 417.1403, found: 417.1413; IR ($cm^{-1}$) v 2933, 1710, 1611, 1544, 1488, 1444, 1284, 1141, 815, 777, 700.



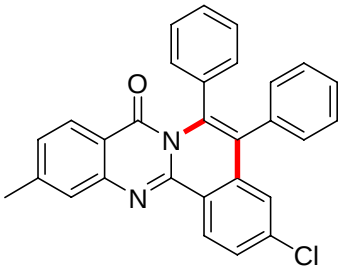

**3,11-dimethyl-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3l).**

Compound was obtained as a white solid: mp 252-253 ℃; yield 80%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.98 (d, *J* = 7.6 Hz, 1H), 8.05 (d, *J* = 7.6 Hz, 1H), 7.72-7.66 (m, 2H), 7.53 (s, 1H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.28-7.24 (m, 2H), 7.21 (d, *J* = 8.0 Hz, 1H), 7.12-7.02 (m, 6H), 6.95 (s, 1H), 2.55 (s, 3H), 2.38 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.1, 147.8, 147.1, 145.4, 142.6, 137.3, 135.7, 135.3, 133.9, 132.3, 131.2, 131.0, 129.8, 128.9, 128.5, 128.0, 127.5, 127.2, 127.1, 126.8, 126.3, 126.1, 125.0, 117.8, 22.1, 22.0; HRMS calcd. For $C_{30}H_{22}N_2NaO^+$ $[M + Na]^+$ 449.1630, found: 449.1652; IR ($cm^{-1}$) v 2918, 1729, 1610, 1543, 1487, 14639, 1287, 1124, 787, 695.

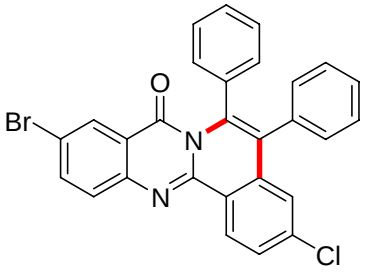



**10-bromo-3-methyl-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3m).**

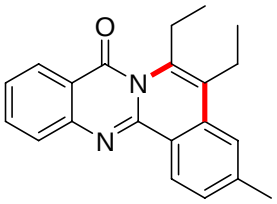
Compound was obtained as a white solid: mp 254-255 ℃; yield 95%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.90 (d, *J* = 8.0 Hz, 1H), 8.23 (s, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 1H), 7.40 (d, *J* = 7.6 Hz, 1H), 7.28-7.21 (m, 3H), 7.12-7.05 (m, 7H), 6.93 (s, 1H), 2.35 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 160.1, 147.9, 145.9, 143.0, 137.4, 136.9, 135.5, 135.3, 134.0, 131.1, 129.9, 129.5, 128.6, 128.5, 128.1, 128.0, 127.3, 127.2, 127.1, 126.9, 126.2, 124.8, 121.3, 118.4, 21.9; HRMS calcd. For $C_{29}H_{20}BrN_2O^+$ $[M + H]^+$ 491.0759, found: 491.0740; IR ($cm^{-1}$) v 2923, 1689, 1597, 1541, 1489, 1463, 1292, 1153, 840, 728, 695.




**3-chloro-11-methyl-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3n).** Compound was obtained as a white solid: mp 286-287 ℃; yield 57%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.94 (d, *J* = 8.8 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.56 (s, 1H), 7.47 (d, *J* = 8.8 Hz, 1H), 7.20-7.15 (m, 4H), 7.05-6.96 (m, 8H), 2.47 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.0, 147.0, 146.8, 145.7, 138.5, 136.8, 136.7, 135.3, 134.8, 131.1, 128.8, 128.7, 128.4, 128.2, 127.6, 127.5, 127.2, 127.1, 127.0, 126.4, 125.8, 125.6, 117.9, 22.1; HRMS calcd. For $C_{29}H_{20}ClN_2O^+$ $[M + H]^+$ 447.1264, found: 447.1294; IR ($cm^{-1}$) v 2919, 1689, 1608, 1544, 1482, 1442, 1287, 1197, 875, 787, 708.

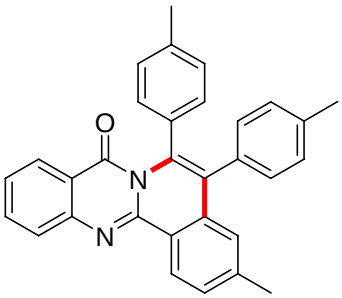



**10-bromo-3-chloro-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3o).** Compound was obtained as a white solid: mp 232-233 ℃; yield 84%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.95 (d, *J* = 8.4 Hz, 1H), 8.23 (s, 1H), 7.82 (d, *J* = 7.2 Hz, 1H), 7.66 (d, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.30-7.24 (m, 3H), 7.17-7.04 (m, 8H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 159.9, 147.1, 145.5, 138.9, 137.7, 136.5, 136.4, 135.3, 134.6, 131.0, 129.6, 128.9, 128.6, 128.4, 128.3, 127.7, 127.3, 127.3, 127.1, 125.7, 125.5, 121.4, 119.0; HRMS calcd. For $C_{28}H_{17}BrClN_2O^+$ $[M + H]^+$ 511.0213, found: 511.0233; IR ($cm^{-1}$) v 2927, 1705, 1617, 1541, 1479, 1444, 1287, 1217, 885, 828, 696.



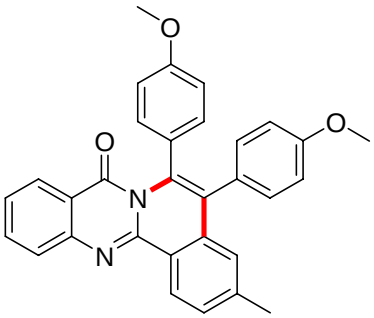

**5,6-diethyl-3-methyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3p).**

Compound was obtained as a white solid: mp 103-104 ℃; yield 98%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.86 (d, *J* = 8.0 Hz, 1H), 8.31 (d, *J* = 7.6 Hz, 1H), 7.76-7.73 (m, 2H), 7.48 (s, 1H), 7.40 (t, *J* = 6.4 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 3.23 (q, *J* = 7.2 Hz, 2H), 2.91 (q, *J* = 7.2 Hz, 2H), 2.52 (s, 3H), 1.30 (t, *J* = 7.2 Hz, 3H), 1.25 (t, *J* = 7.6 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 162.4, 147.7, 146.8, 142.4, 137.5, 134.0, 132.7, 128.7, 127.5, 126.7, 126.4, 125.1, 125.0, 124.6, 123.0, 120.0, 23.5, 22.1, 20.5, 14.5, 14.2; HRMS calcd. For $C_{21}H_{20}N_2NaO^+$ $[M + Na]^+$ 339.1473, found: 339.1493; IR ($cm^{-1}$) v 2964, 2873, 1684, 1603, 1541, 1468, 1296, 1180, 820, 764, 698.

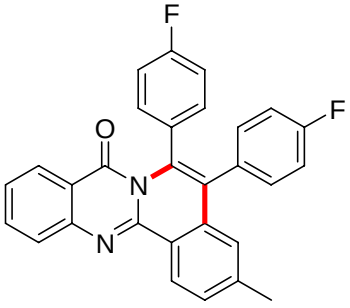



**3-methyl-5,6-dip-tolyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3q).**

Compound was obtained as a white solid: mp 298-299 ℃; yield 93%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.97 (d, *J* = 8.0 Hz, 1H), 8.16 (d, *J* = 7.6 Hz, 1H), 7.85 (d, *J* = 8.0Hz, 1H), 7.78 (t, *J* = 8.0 Hz, 1H), 7.41 (d, *J* = 8.4 Hz, 1H), 7.37 (t, *J* = 8.0 Hz, 1H), 7.08 (d, *J* = 7.6 Hz, 2H), 6.96-6.93 (m, 7H), 2.36 (s, 3H), 2.35 (s, 3H), 2.26 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.5, 147.8, 147.1, 142.6, 136.7, 136.2, 135.3, 134.3, 134.2, 132.6, 131.0, 129.6, 128.9, 128.7, 128.3, 128.0, 127.7, 127.2, 127.1, 126.6, 126.2, 125.2, 124.9, 120.2, 22.0, 21.4, 21.3; HRMS calcd. For $C_{31}H_{24}N_2NaO^+$ $[M + Na]^+$ 463.1786, found: 463.1791; IR ($cm^{-1}$) v 2921, 1706, 1602, 1542, 1491, 1467, 1291, 1123, 831, 769, 694.

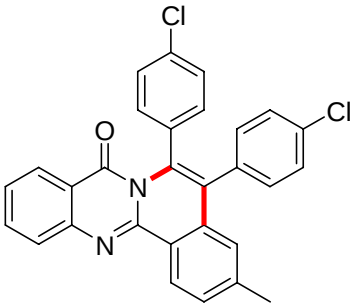


**5,6-bis(4-methoxyphenyl)-3-methyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3r).** Compound was obtained as a white solid: mp 276-278 ℃; yield 90%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.95 (d, $J$ = 8.4 Hz, 1H), 8.15 (d, $J$ = 8.0 Hz, 1H), 7.84 (d, $J$ = 8.0 Hz, 1H), 7.78 (t, $J$ = 7.2Hz, 1H), 7.42-7.35 (m, 2H), 6.99-6.95 (m, 5H), 6.82 (d, $J$ = 8.4 Hz, 2H), 6.67 (d, $J$ = 8.8 Hz, 2H), 3.81 (s, 3H), 3.74 (s, 3H), 2.36 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.6, 158.5, 158.0, 147.8, 147.1, 142.6, 135.4, 134.4, 134.3, 132.2, 129.7, 129.6, 129.6, 127.9, 127.5, 127.1, 127.1, 126.6, 126.1, 125.2, 124.9, 120.2, 113.5, 112.7, 55.2, 55.0, 22.0; HRMS calcd. For $C_{31}H_{25}N_2O_3^+$ $[M + H]^+$ 473.1865, found: 473.1894; IR ($cm^{-1}$) v 2923, 1701, 1601, 1545, 1510, 1468, 1289, 1247, 1176, 830, 768, 694.

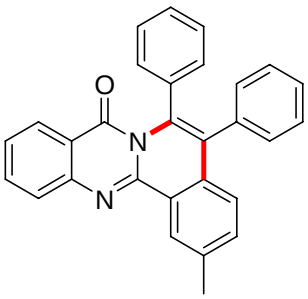


**5,6-bis(4-fluorophenyl)-3-methyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3s).** Compound was obtained as a white solid: mp 249-250 ℃; yield 90%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.96 (d, $J$ = 7.6 Hz, 1H), 8.12 (d, $J$ = 7.6 Hz, 1H), 7.81-7.77 (m, 2H), 7.43-7.36 (m, 2H), 7.05-6.95 (m, 6H), 6.85- 6.81 (m, 3H), 2.34 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.9 (d, $^{1}J_{CF}$ = 246 Hz), 161.4 (d, $^{1}J_{CF}$ = 245 Hz), 161.2, 147.4, 146.9, 142.9, 134.7, 134.5, 133.6, 133.1 (d, $^{4}J_{CF}$ = 4 Hz), 132.8 (d, $^{3}J_{CF}$ = 8.0 Hz), 131.4 (d, $^{4}J_{CF}$ = 3 Hz), 130.2 (d, $^{3}J_{CF}$ = 8.0 Hz), 130.1, 127.2, 127.1, 127.1,126.8,

125.9, 125.5, 124.9, 120.0, 115.3 (d, $^{2}J_{CF}$ = 21 Hz), 114.5 (d, $^{2}J_{CF}$ = 22 Hz), 22.0; HRMS calcd. For $C_{29}H_{19}F_2N_2O^+$ $[M + H]^+$ 449.1465, found: 449.1488; IR ($cm^{-1}$) v 2921, 1681, 1600, 1548, 1508, 1467, 1293, 1223, 858, 778, 696.



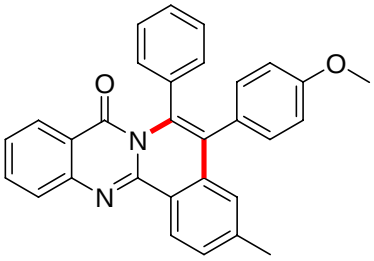

**5,6-bis(4-chlorophenyl)-3-methyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (3t).** Compound was obtained as a light yellow solid: mp 256-257 ℃; yield 88%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.96 (d, *J* = 8.0 Hz, 1H), 8.12 (d, *J* = 7.2 Hz, 1H), 7.84-7.76 (m, 2H), 7.43 (d, *J* = 7.6 Hz, 1H), 7.37 (t, *J* = 6.8 Hz, 1H), 7.28 (d, *J* = 7.6 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 6.98 (t, *J* = 9.6 Hz, 4H), 6.84 (s, 1H), 2.35 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.2, 147.3, 146.9, 143.0, 135.5, 134.6, 133.8, 133.6, 133.3, 132.8, 132.4, 131.0, 130.2, 129.7, 128.9, 128.6, 127.7, 127.3, 127.1, 126.8, 125.9, 125.6, 125.0, 119.9, 22.0; HRMS calcd. For $C_{29}H_{19}Cl_2N_2O^+$ $[M + H]^+$ 481.0874, found: 481.0883; IR ($cm^{-1}$) v 2927, 1708, 1602, 1542, 1489, 1467, 1291, 1095, 831, 768, 697.



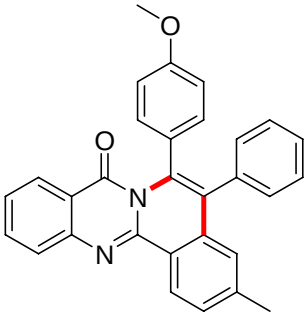

**2-methyl-5,6-diphenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (5a).** Compound was obtained as a white solid: mp 214-215 ℃; yield 49%; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.93 (s, 1H), 8.17 (d, *J* = 8.0 Hz, 1H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.82 (t, *J* = 7.2 Hz, 1H), 7.73-7.71 (m, 1H), 7.54-7.52 (m, 1H), 7.40 (t, *J* = 8.0 Hz, 2H),

7.27-7.25 (m, 2H), 7.13-7.10 (m, 3H), 7.08-7.06 (m, 4H), 2.58 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.4, 147.6, 146.9, 138.7, 137.1, 135.7, 134.4, 133.4, 132.3, 131.7, 131.1, 131.0, 128.9, 128.5, 128.0, 127.8, 127.2, 127.2, 126.9, 126.8, 126.7, 126.3, 125.6, 120.2, 21.7; HRMS calcd. For $C_{29}H_{21}N_2O^+$ $[M + H]^+$ 413.1654, found: 413.1653; IR ($cm^{-1}$) v 2920, 1694, 1604, 1548, 1471, 1445, 1286, 1127, 770, 697.

Compound **7** was obtained in 83% yield as a separable mixture of two regioisomers with ratio of 1:3 for **7a:7b**



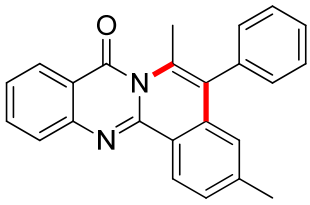

**5-(4-methoxyphenyl)-3-methyl-6-phenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (7a).** Compound was obtained as a white solid: mp 226-227 ℃; $^1$H NMR (400 MHz, $CDCl_3$): δ 8.97 (d, $J$ = 8.0 Hz, 1H), 8.16 (d, $J$ = 7.6 Hz, 1H), 7.86-7.79 (m, 2H), 7.41 (t, $J$ = 8.4 Hz, 2H), 7.31-7.24 (m, 3H), 7.10-7.04 (m, 2H), 6.97-6.95 (m, 3H), 6.65 (d, $J$ = 8.0 Hz, 2H), 3.72 (s, 3H), 2.36 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.6, 158.1, 147.8, 147.0, 142.7, 135.8, 135.1, 134.4, 134.0, 131.2, 129.7, 129.5, 128.1, 127.8, 127.1, 127.1, 126.7, 126.1, 125.3, 124.9, 120.2, 112.7, 55.0, 22.0; HRMS calcd. For $C_{30}H_{23}N_2O_2^+$ $[M + H]^+$ 443.1760, found: 443.1736; IR ($cm^{-1}$) v 2930, 1699, 1605, 1545, 1492, 1467, 1293, 1246, 832, 779, 696.




**6-(4-methoxyphenyl)-3-methyl-5-phenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (7b).**

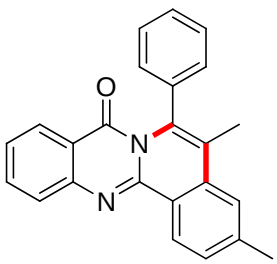
Compound was obtained as a white solid: mp 272-273 ℃; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.99 (d, *J* = 8.4 Hz, 1H), 8.16 (d, *J* = 8.0 Hz, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.80 (t, *J* = 7.6 Hz, 1H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.38 (t, *J* = 8.0 Hz, 1H), 7.15-7.13 (m, 3H), 7.07-7.04 (m, 2H), 6.98 (d, *J* = 8.8 Hz, 3H), 6.81 (d, *J* = 8.4 Hz, 2H), 3.80 (s, 3H), 2.38 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.4, 158.5, 147.7, 147.0, 142.7, 137.3, 135.5, 134.4, 134.3, 132.2, 131.0, 129.8, 128.9, 128.5, 127.6, 127.2, 127.1, 126.8, 126.7, 126.2, 125.3, 124.9, 120.1, 113.5, 55.2, 22.0; HRMS calcd. For $C_{30}H_{23}N_2O_2^+$ $[M + H]^+$ 443.1760, found: 443.1736; IR ($cm^{-1}$) v 2926, 1704, 1602, 1545, 1511, 1468, 1292, 1248, 828, 768, 694.

Compound **9** was obtained in 92% yield as a separable mixture of two regioisomers with ratio of 1:6 for **9a:9b**



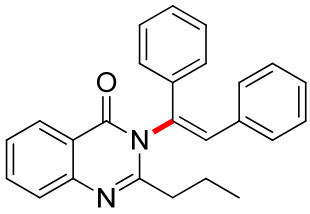

**3,6-dimethyl-5-phenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (9a).**

Compound was obtained as a white solid: mp 185-186 ℃; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.88 (d, *J* = 8.4 Hz, 1H), 8.32 (d, *J* = 8.0 Hz, 1H), 7.84 – 7.81 (m, 2H), 7.53 (t, *J* = 7.2 Hz, 2H), 7.48 (d, *J* = 7.2 Hz, 1H), 7.44 (t, *J* = 8.0 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.32 (d, *J* = 6.8 Hz, 2H), 6.80 (s, 1H), 2.50 (s, 3H), 2.35 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 162.7, 147.8, 147.1, 142.4, 137.2, 134.3, 134.2, 133.5, 130.7, 129.0, 128.9, 128.8, 128.0, 127.1, 126.9, 126.7, 125.5, 125.3, 124.6, 120.2, 21.9, 14.1; HRMS calcd. For $C_{24}H_{19}N_2O^+$ $[M + H]^+$ 351.1497, found: 351.1492; IR ($cm^{-1}$) v 2917, 1701, 1606, 1537, 1491, 1466, 1296, 828, 774, 697.



**3,5-dimethyl-6-phenyl-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (9b).**

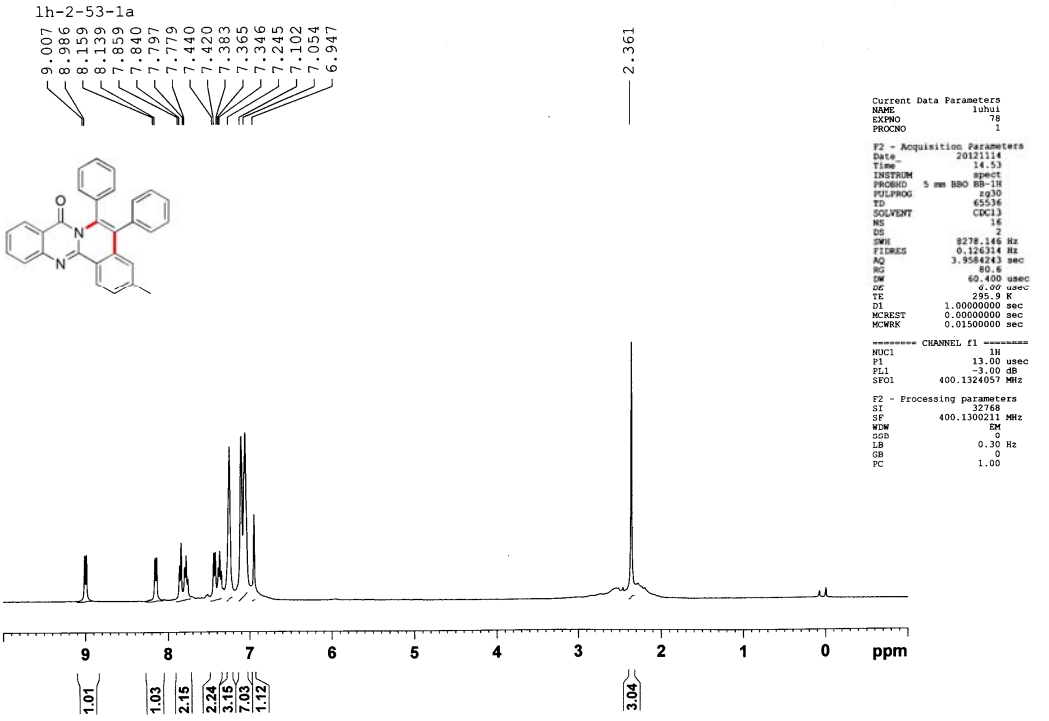
Compound was obtained as a white solid: mp 205-206 ℃; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.96 (d, J = 8.0 Hz, 1H), 8.12 (d, J = 8.0 Hz, 1H), 7.81 (d, J = 7.6 Hz, 1H), 7.76 (t, J = 7.6 Hz, 1H), 7.57 (s, 1H), 7.49-7.39 (m, 4H), 7.35 (t, J = 7.6 Hz, 1H), 7.31 (d, 8.0 Hz, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 161.0, 147.4, 147.0, 142.6, 138.0, 134.3, 134.1, 133.8, 129.5, 128.6, 127.8, 127.4, 127.3, 127.1, 126.6, 125.1, 125.0, 123.9, 120.2, 119.9, 22.1, 15.1; HRMS calcd. For $C_{24}H_{19}N_2O^+$ $[M + H]^+$ 351.1497, found: 351.1492; IR ($cm^{-1}$) v 2946, 1701, 1625, 1537, 1467, 1446, 1314, 828, 774, 642.

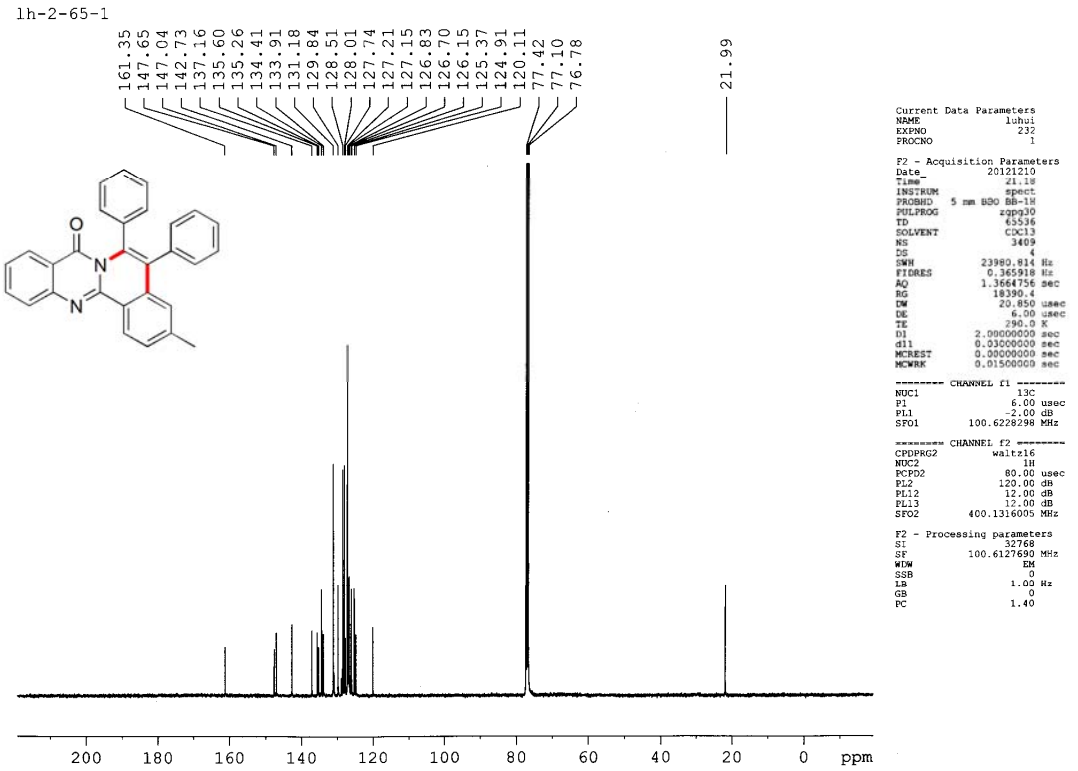


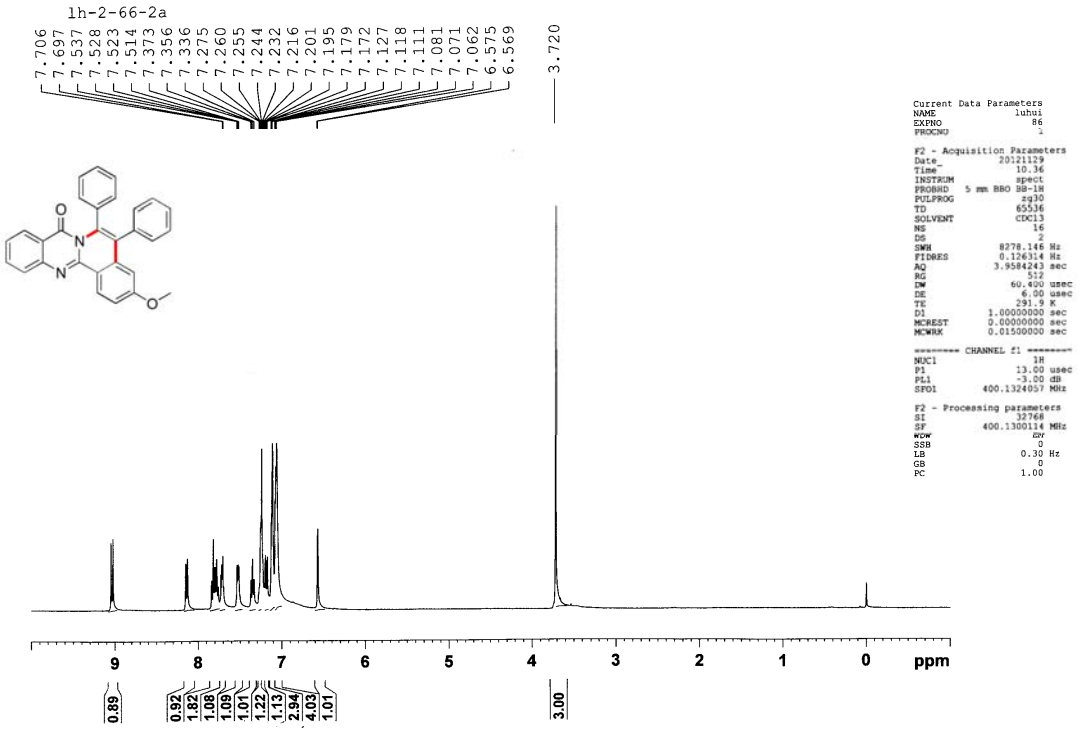

**(*E*)-3-(1,2-diphenylvinyl)-2-propylquinazolin-4(3*H*)-one (12).**

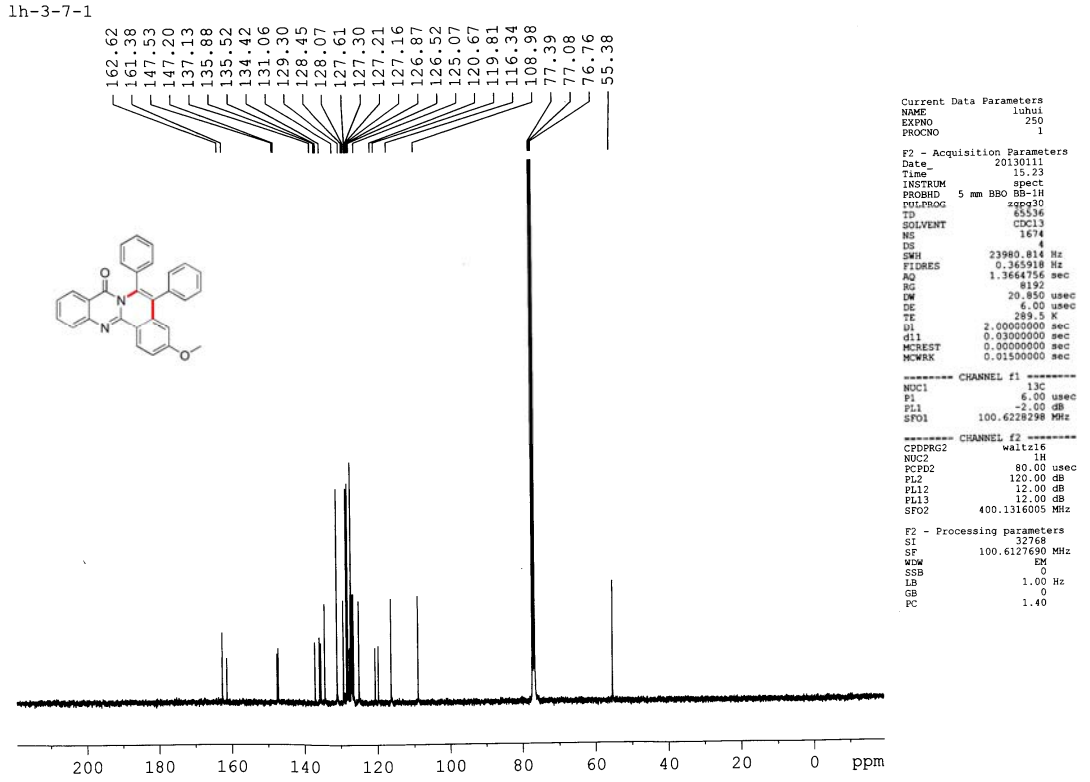
Compound was obtained as a white solid: mp 151-152 ℃; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.71 (t, J = 8.0 Hz, 1H), 7.63 (d, J = 8.0 Hz, 1H), 7.41 (d, J = 6.8 Hz, 3H), 7.37 (d, J = 8.0 Hz, 3H), 7.26 (t, J = 6.8 Hz, 3H), 7.23-7.18 (m, 3H), 6.84 (d, J = 7.2 Hz, 1H), 2.94 (t, J = 8.0 Hz, 2H), 1.94 (m, 2H), 1.05 (t, J = 7.6 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 169.8, 166.1, 152.2, 150.4, 135.6, 134.0, 133.3, 132.6, 130.5, 129.4, 129.3, 129.2, 128.3, 127.9, 122.8, 119.5, 117.5, 111.6, 42.1, 22.4, 14.1; HRMS calcd. For $C_{25}H_{19}N_2O^+$ $[M + H]^+$ 351.1497, found: 351.1492; IR ($cm^{-1}$) v 2927, 1642, 1580, 1466, 1349, 1214, 1137, 832, 774, 604.

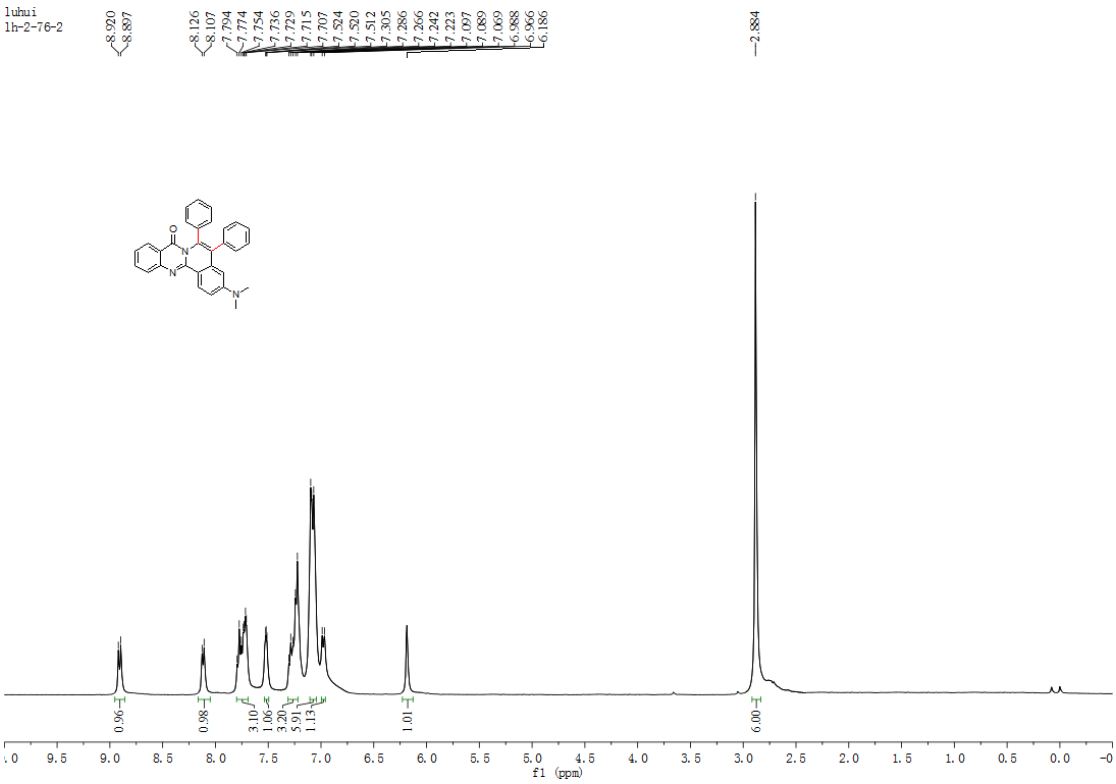
# $^{1}H$ and $^{13}C$ NMR spectra of the products

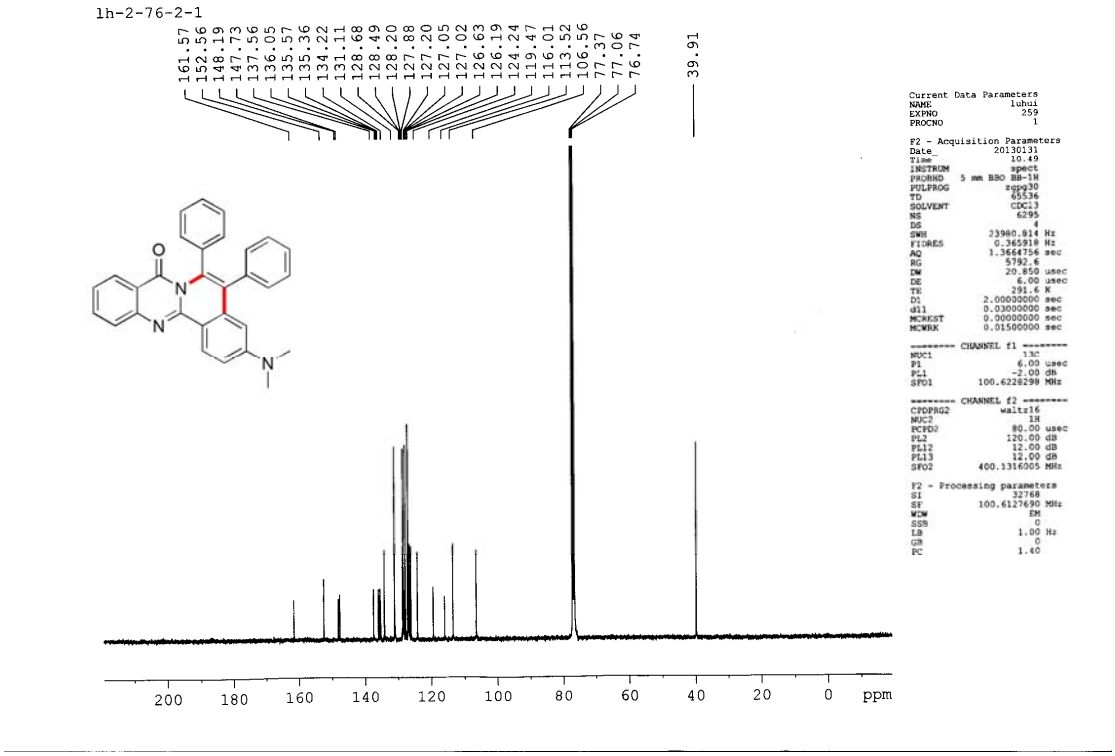


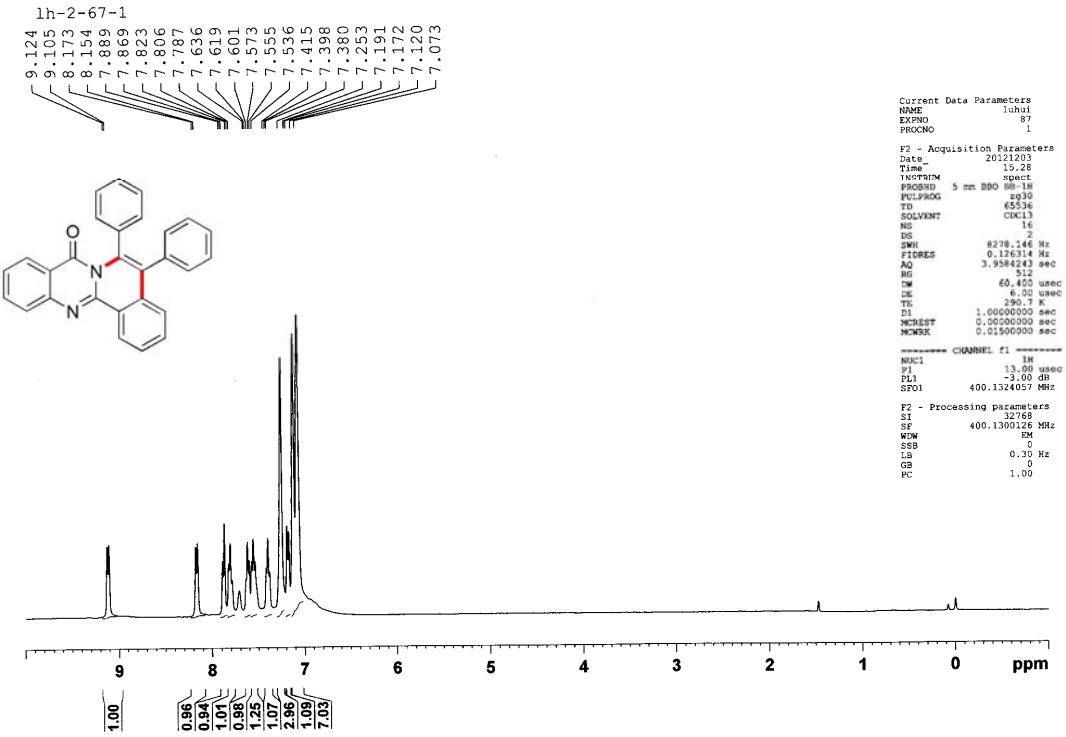



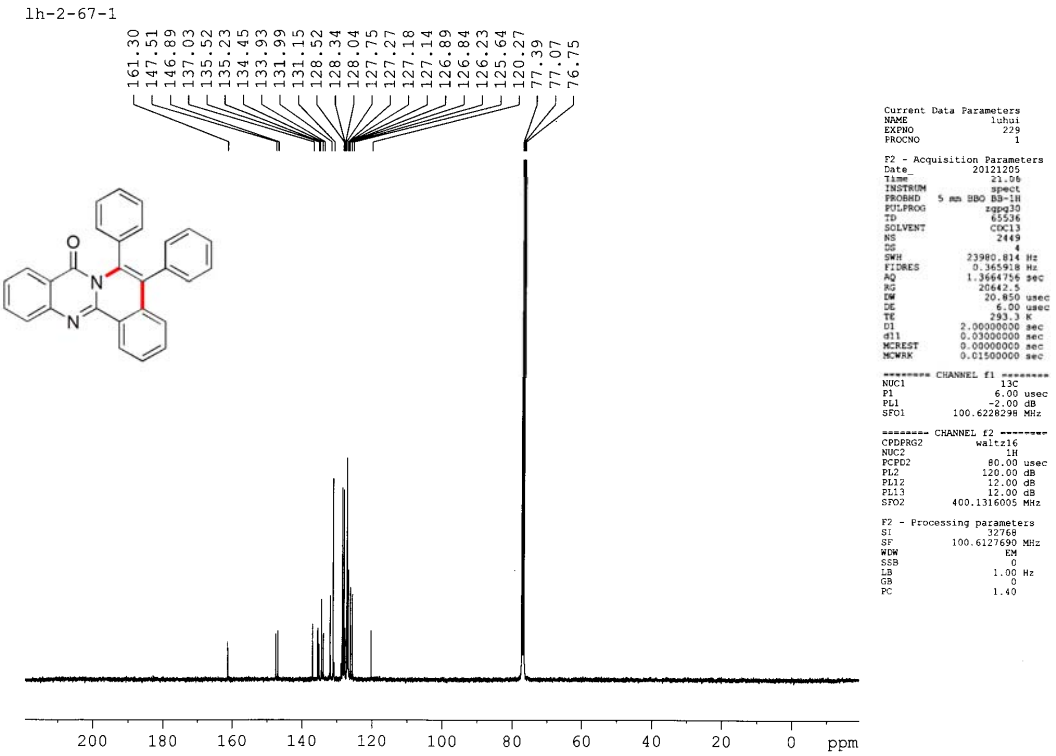



luhui
1h-2-76-2
8.920
8.897
8.126
8.107
7.794
7.774
7.754
7.736
7.729
7.715
7.707
7.524
7.520
7.512
7.305
7.286
7.266
7.242
7.223
7.097
7.089
7.069
6.988
6.966
6.186
2.884
0.96
0.98
3.10
1.06
3.20
5.91
1.13
1.01
6.00
f1 (ppm)

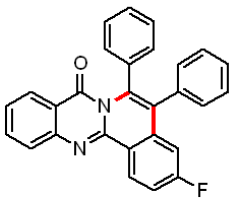

1h-2-76-2-1
161.57
152.56
148.19
147.73
137.56
136.05
135.57
135.36
134.22
131.11
128.68
128.49
128.20
127.88
127.20
127.05
127.02
126.63
126.19
124.24
119.47
116.01
113.52
106.56
77.37
77.06
76.74
39.91
Current Data Parameters
NAME luhui
EXPNO 259
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130131
Time 10.49
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6295
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 5792.6
DW 20.850 usec
DE 6.00 usec
TE 291.6 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.00 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 12.00 dB
PL13 12.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

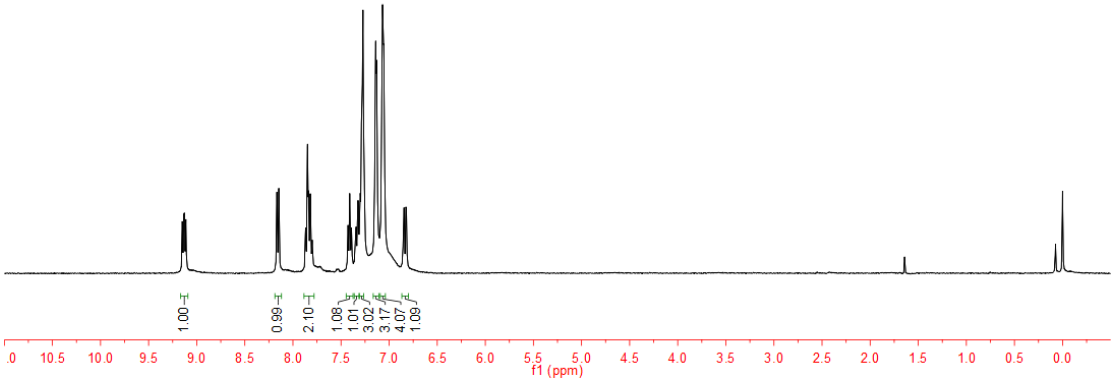


luhui
lh-2-88-1

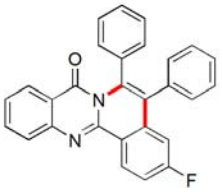
9.150 9.135 9.128 9.113 8.167 8.147 7.868 7.848 7.820 7.800 7.427 7.410 7.391 7.343 7.323 7.300 7.284 7.272 7.261 7.140 7.131 7.073 7.056 6.850 6.825




lh-3-1-2a

166.36 163.84 161.19 146.88 146.81 136.71 136.57 136.39 136.29 134.97 134.60 130.98 130.26 130.17 128.38 128.25 127.55 127.24 127.10 126.99 126.73 125.69 123.69 120.08 116.71 116.48 112.00 111.77 77.38 77.06 76.74







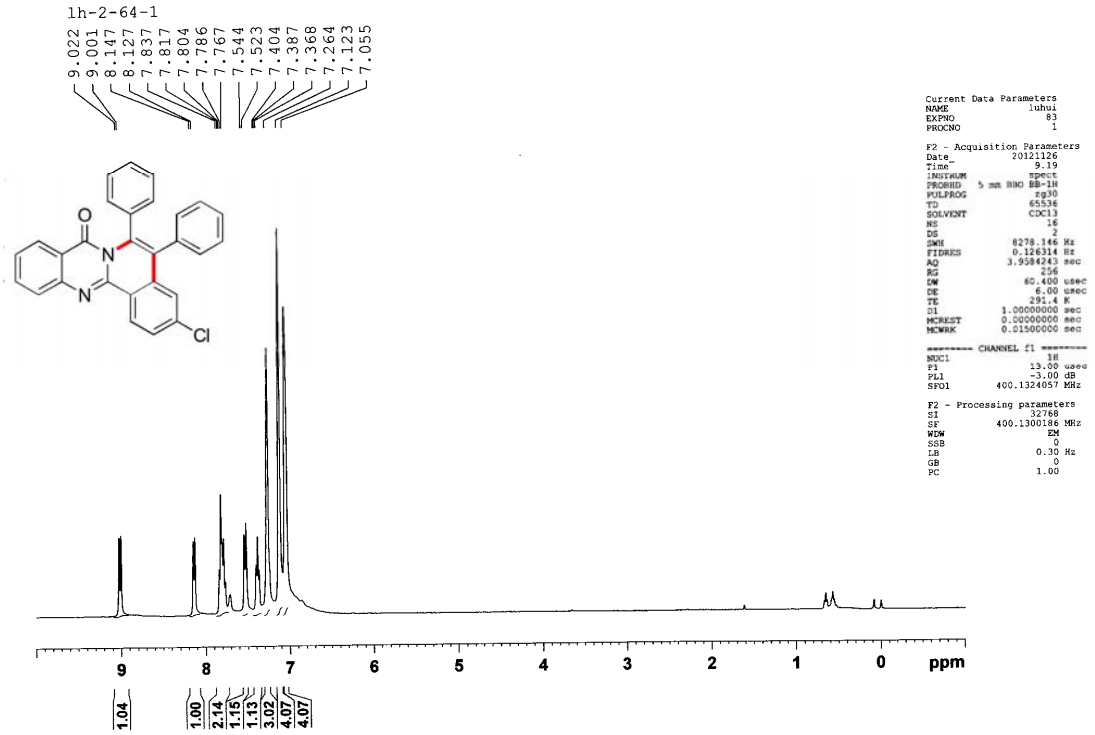
Current Data Parameters
NAME luhui
EXPNO 247
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130109
Time 9.52
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2253
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 6502
DW 20.850 usec
DE 6.00 usec
TE 290.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

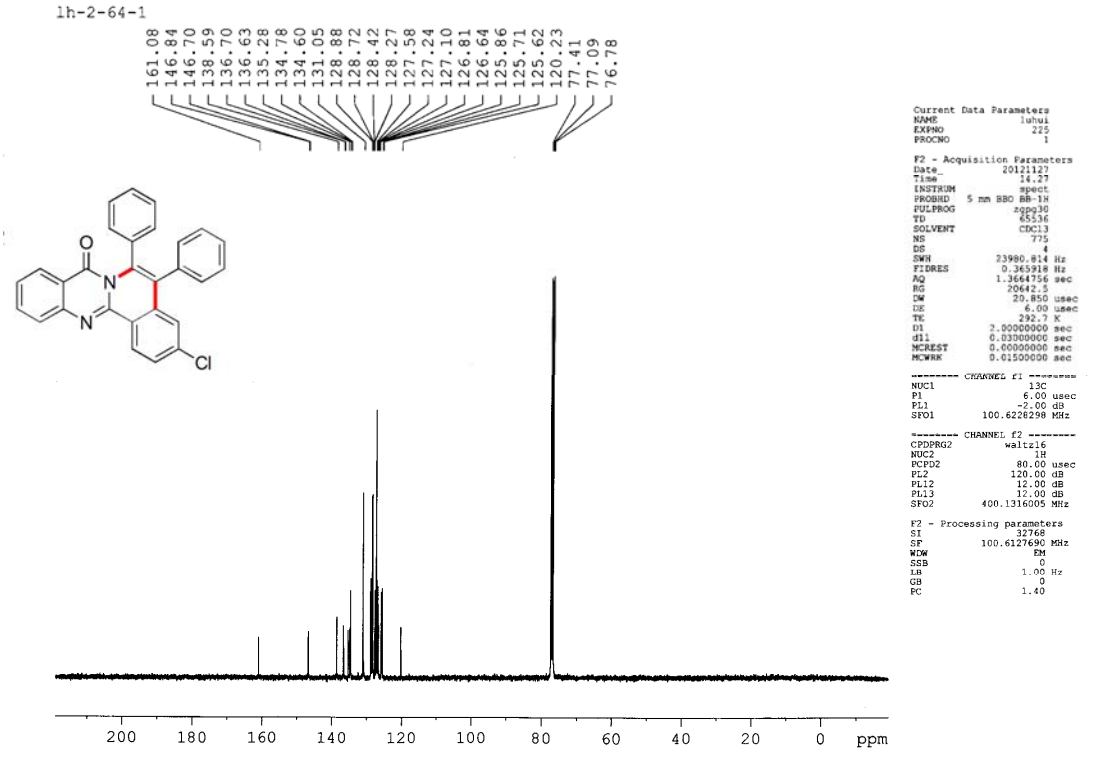
-------- CHANNEL f1 --------
NUC1 13C
P1 6.00 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 12.00 dB
PL13 12.00 dB
SFO2 400.1316005 MHz

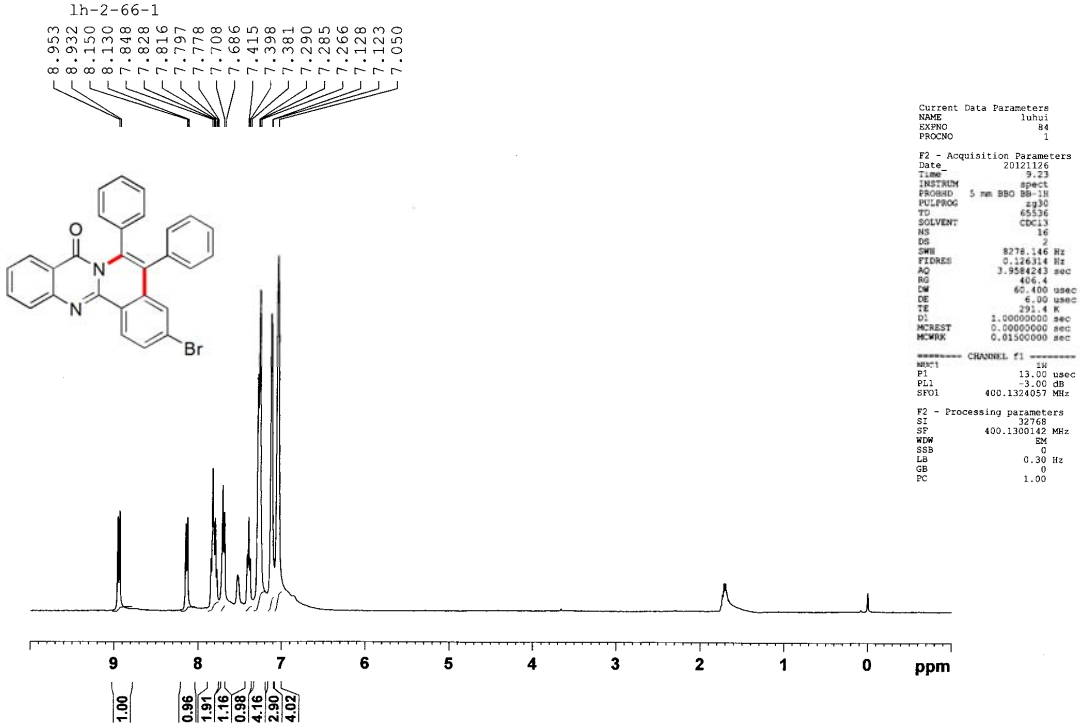
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

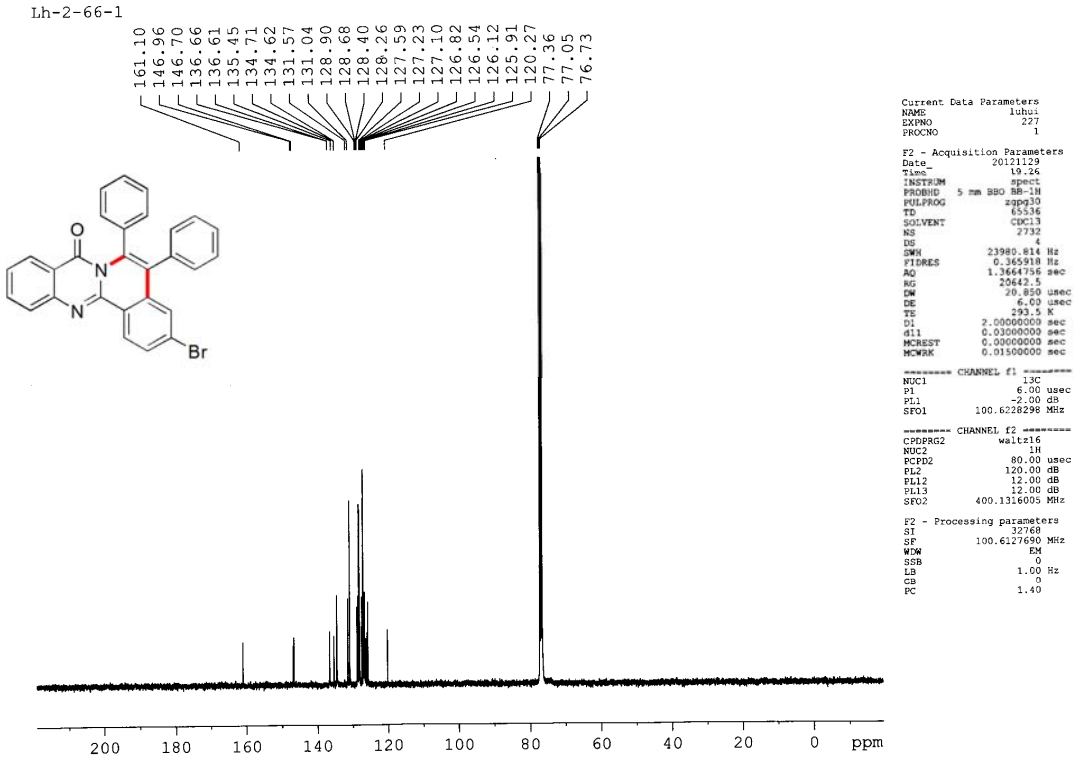


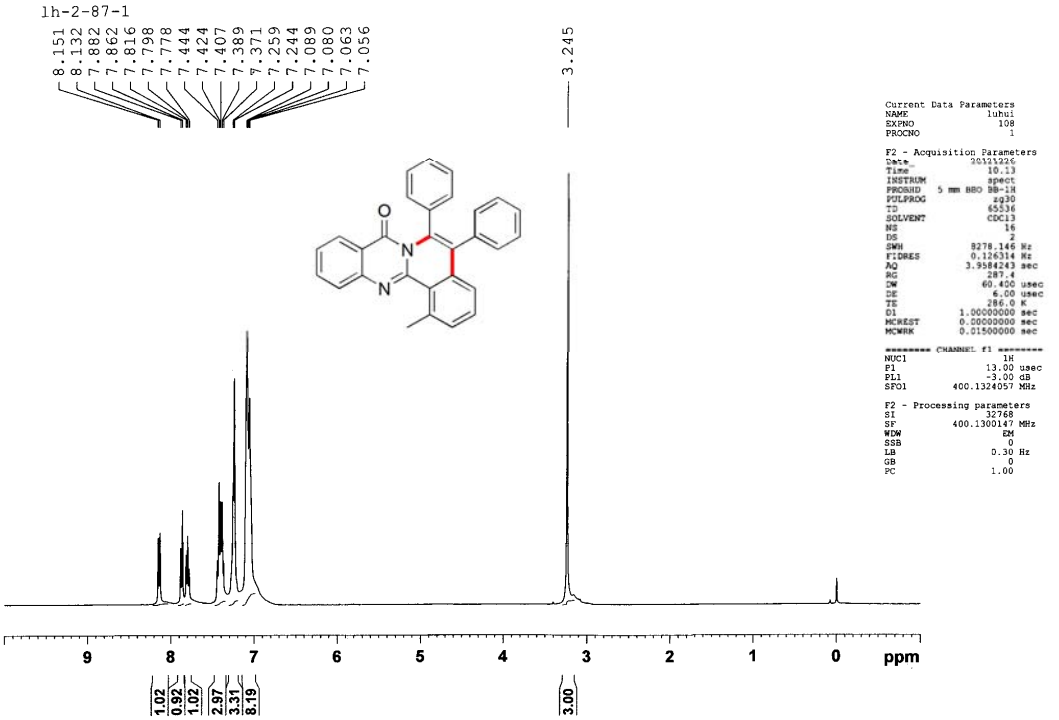

```
Current Data Parameters
NAME                  luhui
EXPNO                    83
PROCNO                    1

F2 - Acquisition Parameters
Date_              20121126
Time                   9.19
INSTRUM               spect
PROBHD  5 mm BBO BB-1H
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                      256
DW                   60.400 usec
DE                    6.00 usec
TE                   291.4 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    13.00 usec
PL1                   -3.00 dB
SFO1          400.1324057 MHz

F2 - Processing parameters
SI                       32768
SF                400.1300186 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                    1.00
```

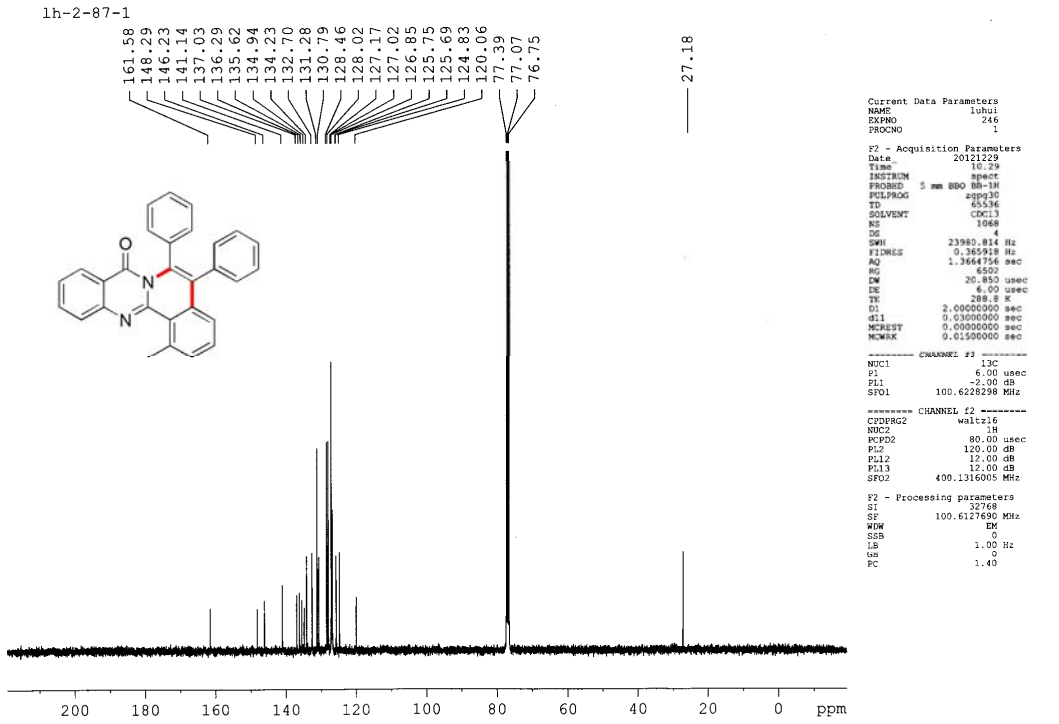


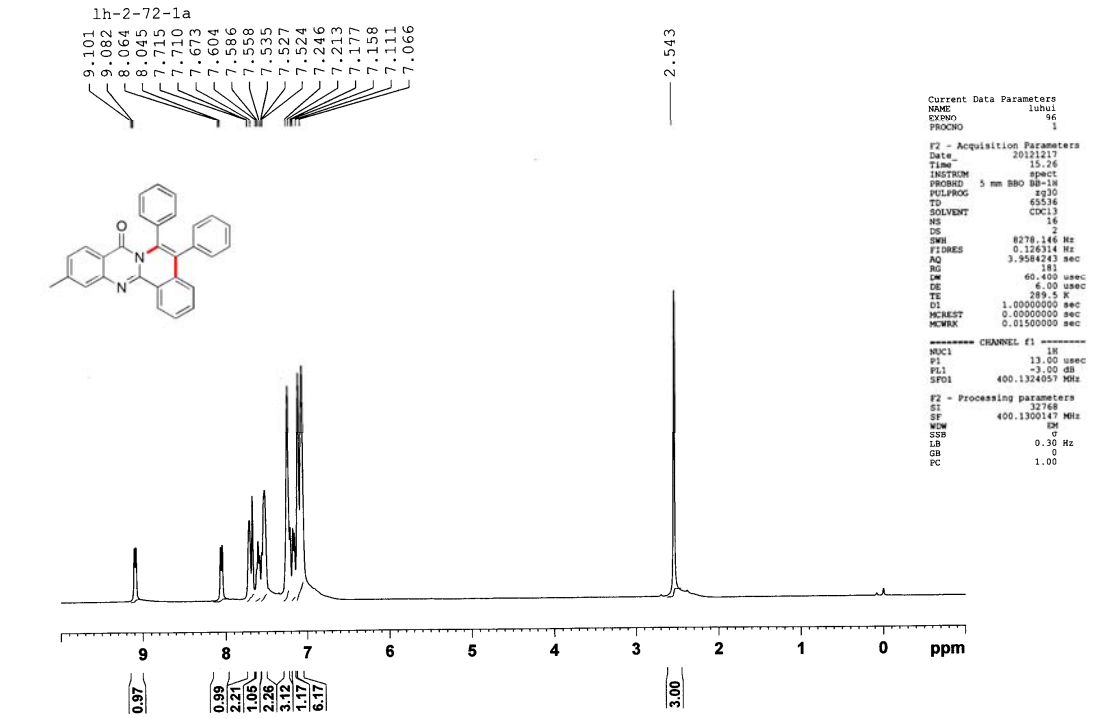

```
Current Data Parameters
NAME                  luhui
EXPNO                   225
PROCNO                    1

F2 - Acquisition Parameters
Date_              20121127
Time                  14.27
INSTRUM               spect
PROBHD  5 mm BBO BB-1H
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      775
DS                        4
SWH               23980.814 Hz
FIDRES            0.365918 Hz
AQ                1.3664756 sec
RG                    20642.5
DW                   20.850 usec
DE                    6.00 usec
TE                   292.7 K
D1                2.00000000 sec
d11               0.03000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    6.00 usec
PL1                  -2.00 dB
SFO1          100.6226298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                80.00 usec
PL2                  120.00 dB
PL12                  12.00 dB
PL13                  12.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF                100.6127690 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                    1.40
```

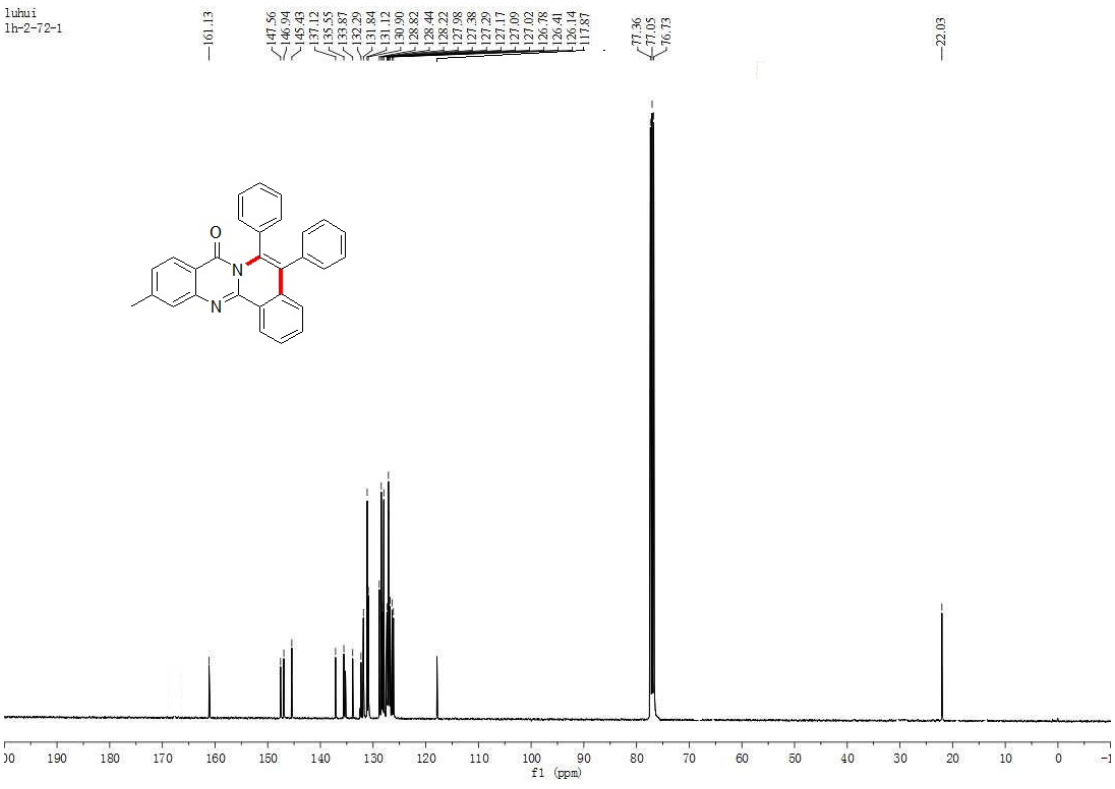


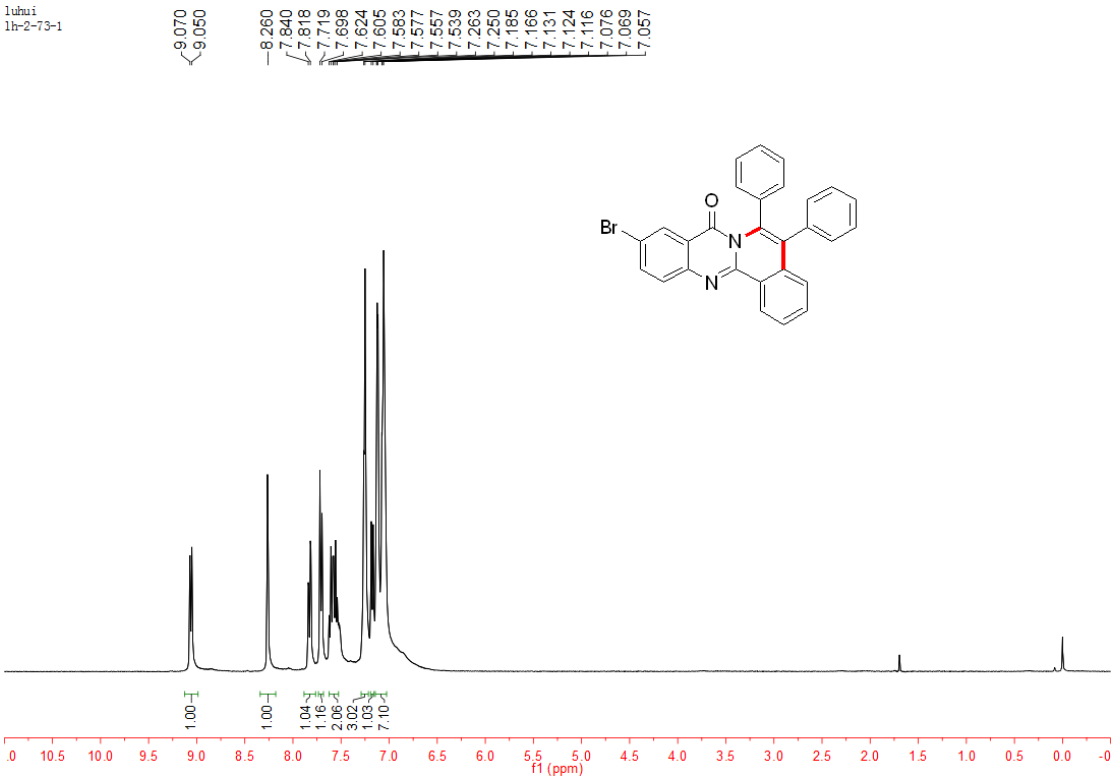



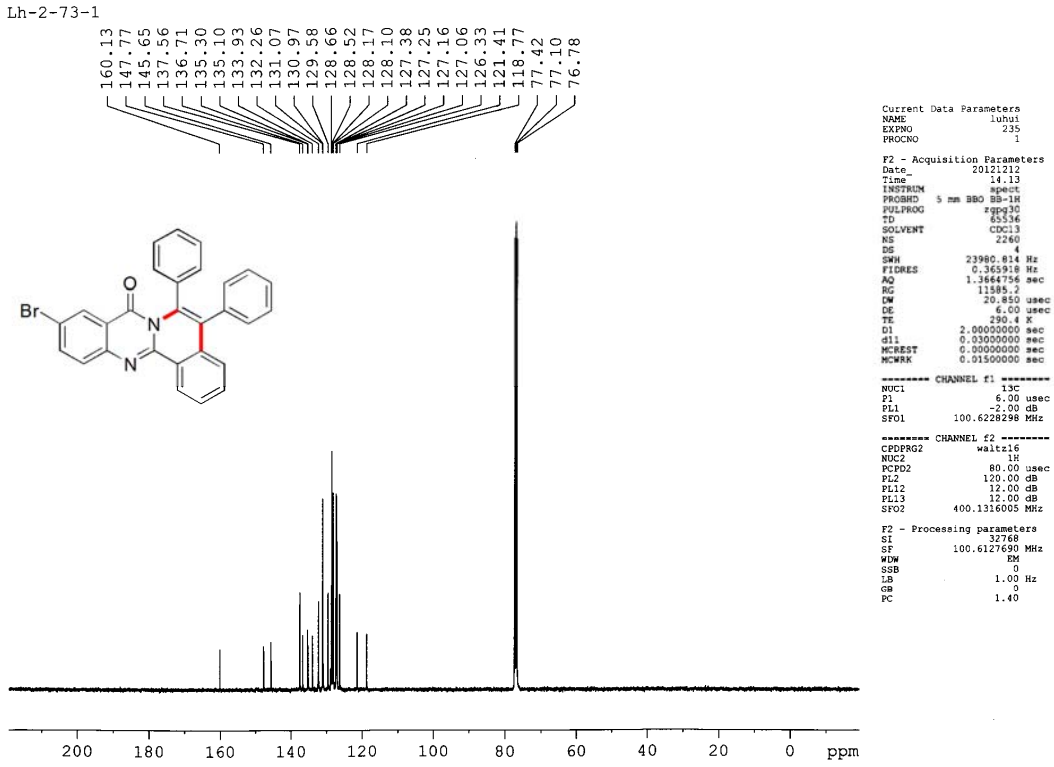






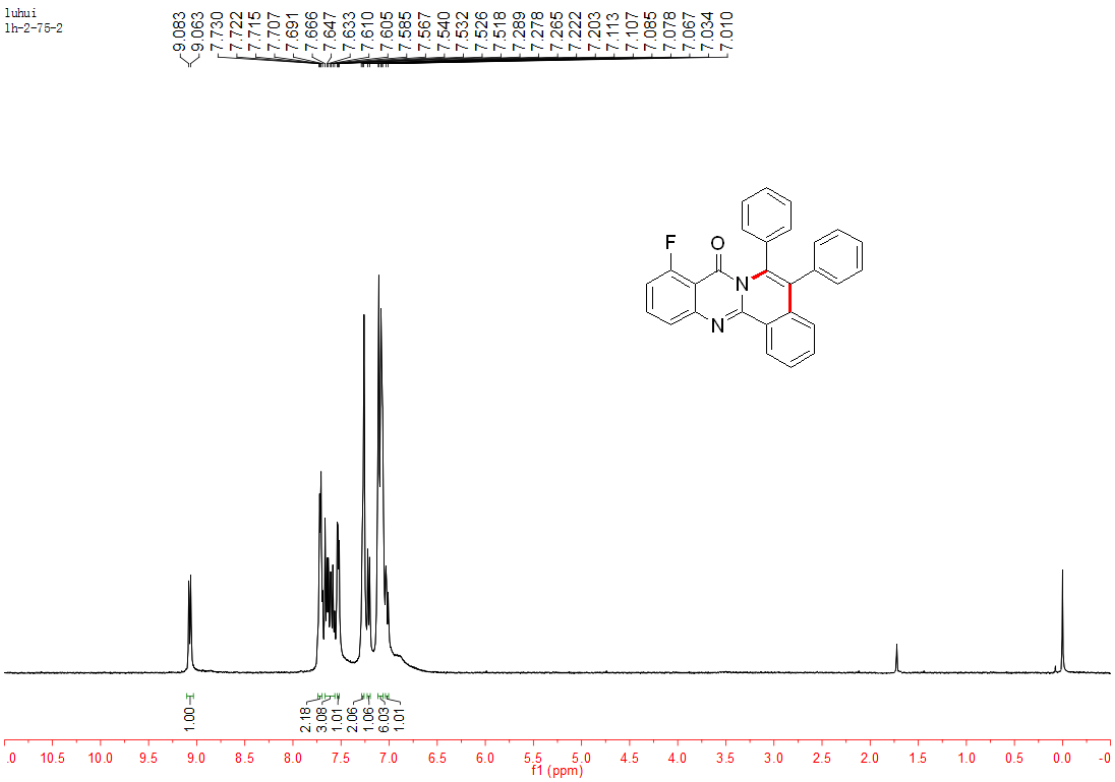

Current Data Parameters
NAME luhui
EXPNO 235
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121212
Time 14.13
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2260
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 11585.2
DW 20.850 usec
DE 6.00 usec
TE 290.4 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 12.00 dB
PL13 12.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


luhui
lh-2-75-2
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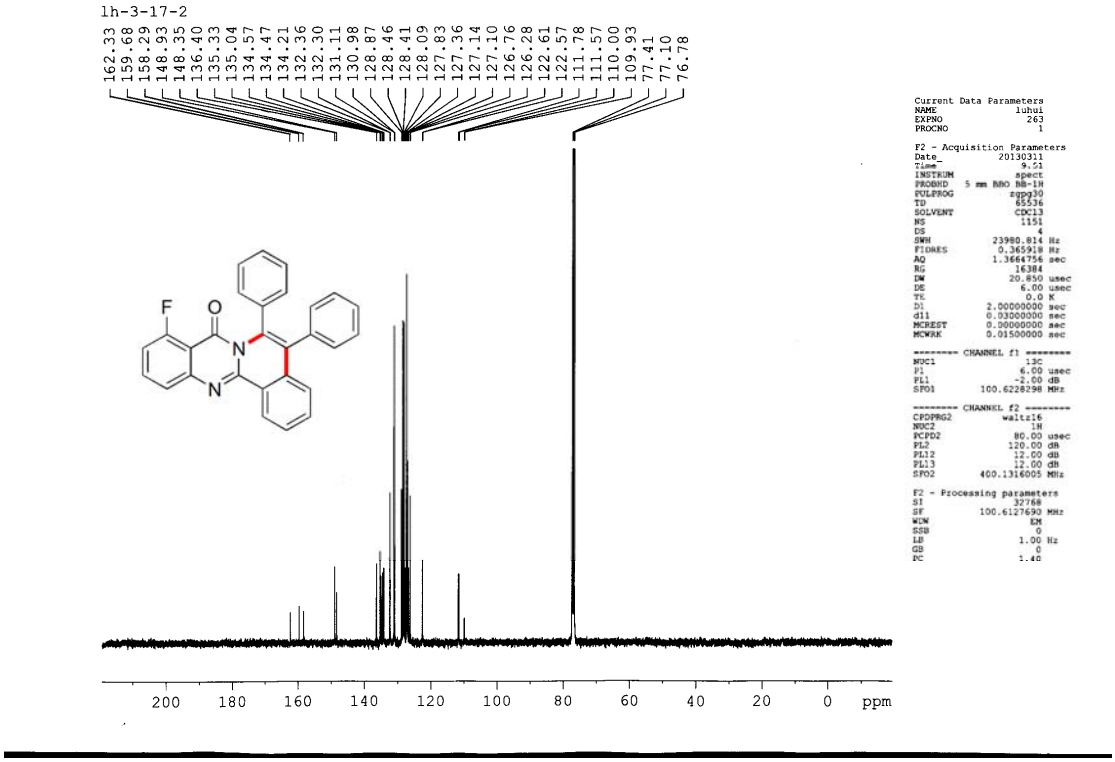
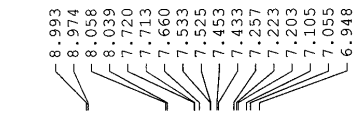
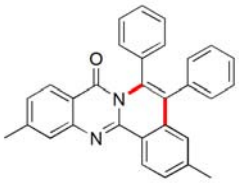
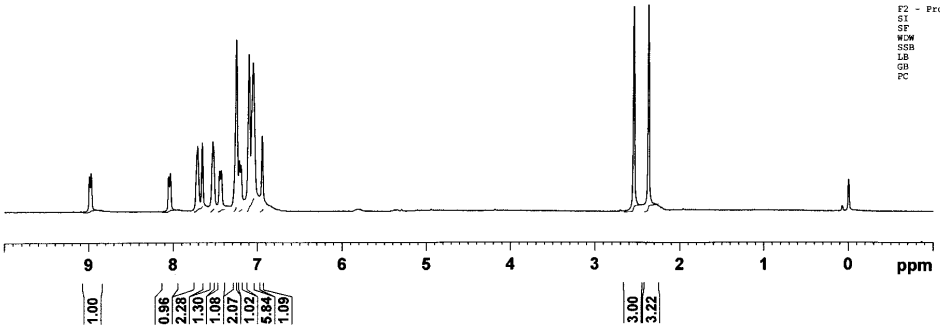
9.063
7.730
7.722
7.715
7.707
7.691
7.666
7.647
7.633
7.610
7.605
7.585
7.567
7.540
7.532
7.526
7.518
7.289
7.278
7.265
7.222
7.203
7.113
7.107
7.085
7.078
7.067
7.034
7.010
F
O
N
N
1.00
2.18
3.08
1.01
2.06
1.06
6.03
1.01
f1 (ppm)


lh-3-17-2
162.33
159.68
158.29
148.93
148.35
136.40
135.33
135.04
134.57
134.47
134.21
132.36
132.30
131.11
130.98
128.87
128.46
128.41
128.09
127.83
127.36
127.14
127.10
126.76
126.28
122.61
122.57
111.78
111.57
110.00
109.93
77.41
77.10
76.78
F
O
N
N
ppm
Current Data Parameters
NAME luhui
EXPNO 263
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130311
Time_ 9.51
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1151
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.00 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 12.00 dB
PL13 12.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

lh-2-83-2a

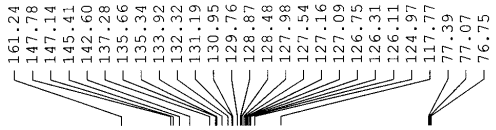


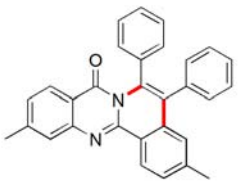



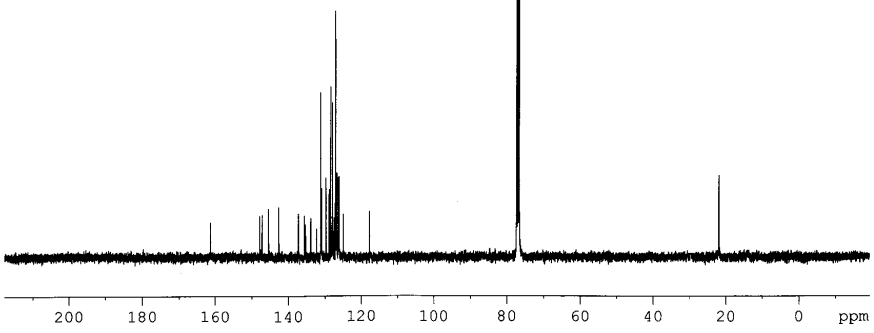




```
Current Data Parameters
NAME                  luhui
EXPNO                   100
PROCNO                    1

F2 - Acquisition Parameters
Date_              20121220
Time               17.09
INSTRUM            spect
PROBHD    5 mm BBO BB-1H
PULPROG            zg30
TD                 65536
SOLVENT            CDCl3
NS                 16
DS                 2
SWH                8278.146 Hz
FIDRES             0.126314 Hz
AQ                 3.9584243 sec
RG                 256
DW                 60.400 usec
DE                 6.00 usec
TE                 289.6 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.00 usec
PL1                -3.00 dB
SFO1          400.1324057 MHz

F2 - Processing parameters
SI                 32768
SF            400.1300106 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

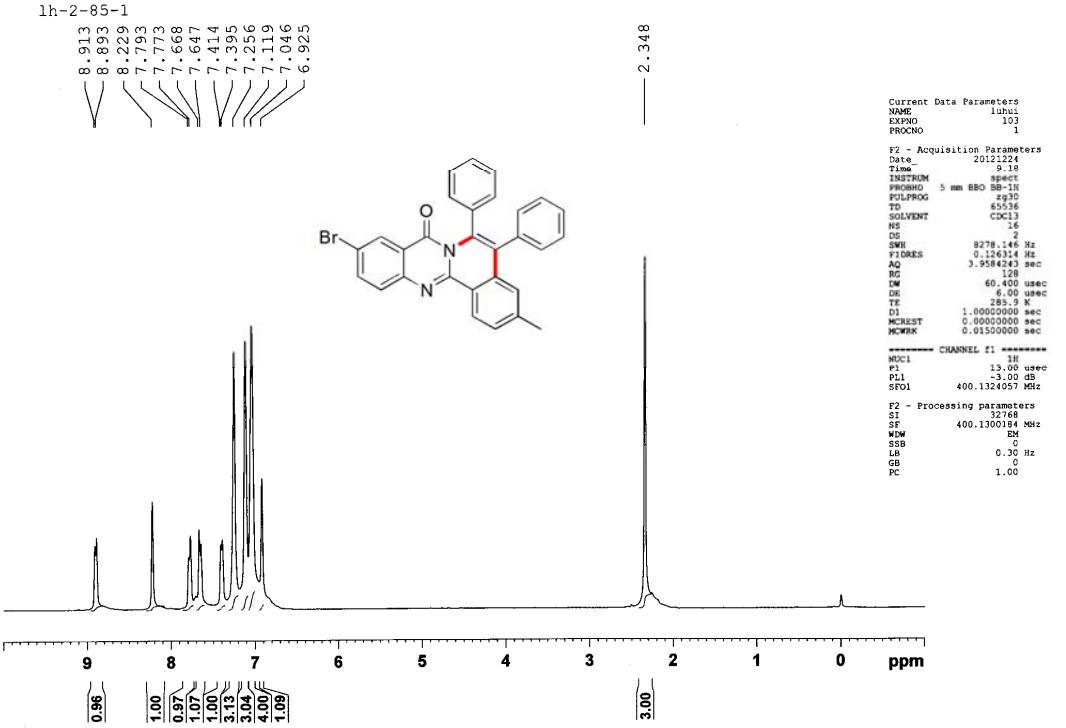
lh-3-4-1




```
Current Data Parameters
NAME                  luhui
EXPNO                   248
PROCNO                    1

F2 - Acquisition Parameters
Date_              20130109
Time               10.22
INSTRUM            spect
PROBHD    5 mm BBO BB-1H
PULPROG            zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 759
DS                 4
SWH                23980.814 Hz
FIDRES             0.365918 Hz
AQ                 1.3664756 sec
RG                 11585.2
DW                 20.850 usec
DE                 6.00 usec
TE                 290.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 6.00 usec
PL1                -2.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2               120.00 dB
PL12               12.00 dB
PL13               12.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127690 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

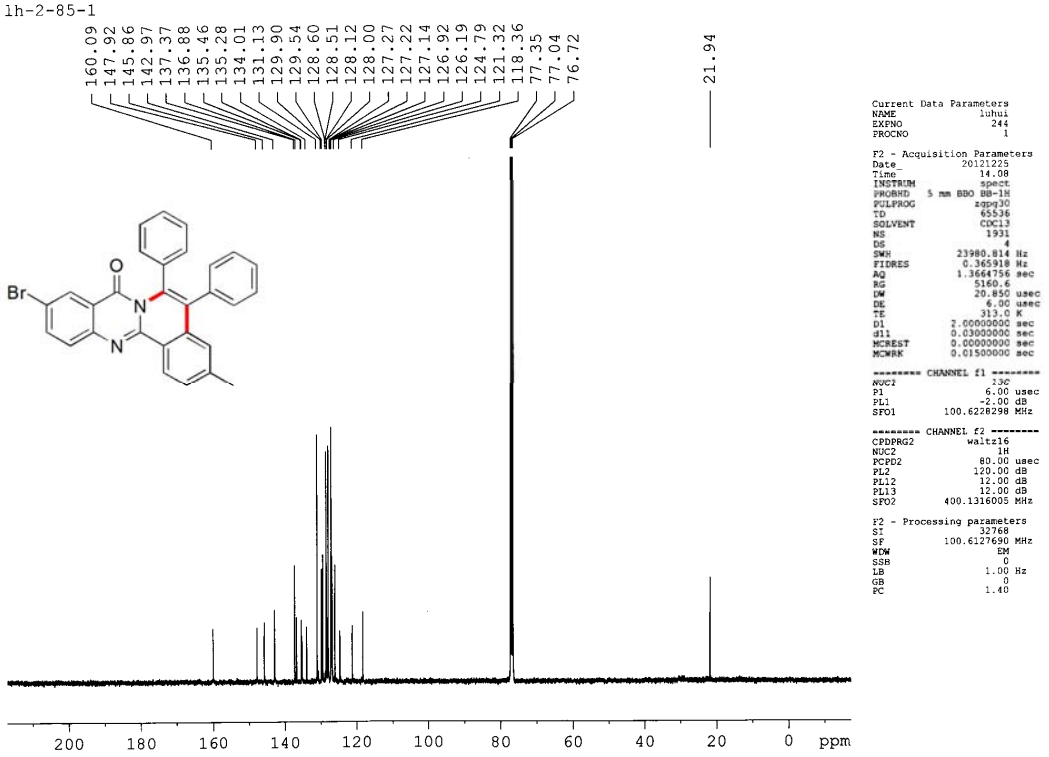



Current Data Parameters
NAME luhui
EXPNO 103
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121224
Time 9.18
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 285.9 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 -3.00 dB
SFO1 400.1324057 MHz

F2 - Processing parameters
SI 32768
SF 400.1300184 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



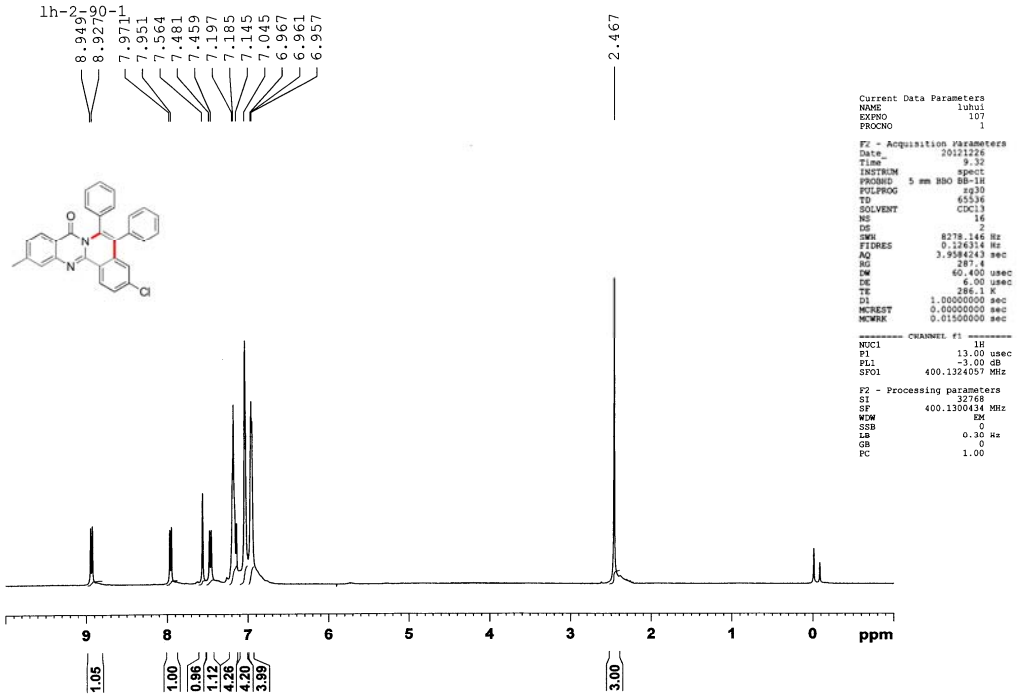

Current Data Parameters
NAME luhui
EXPNO 244
PROCNO 1

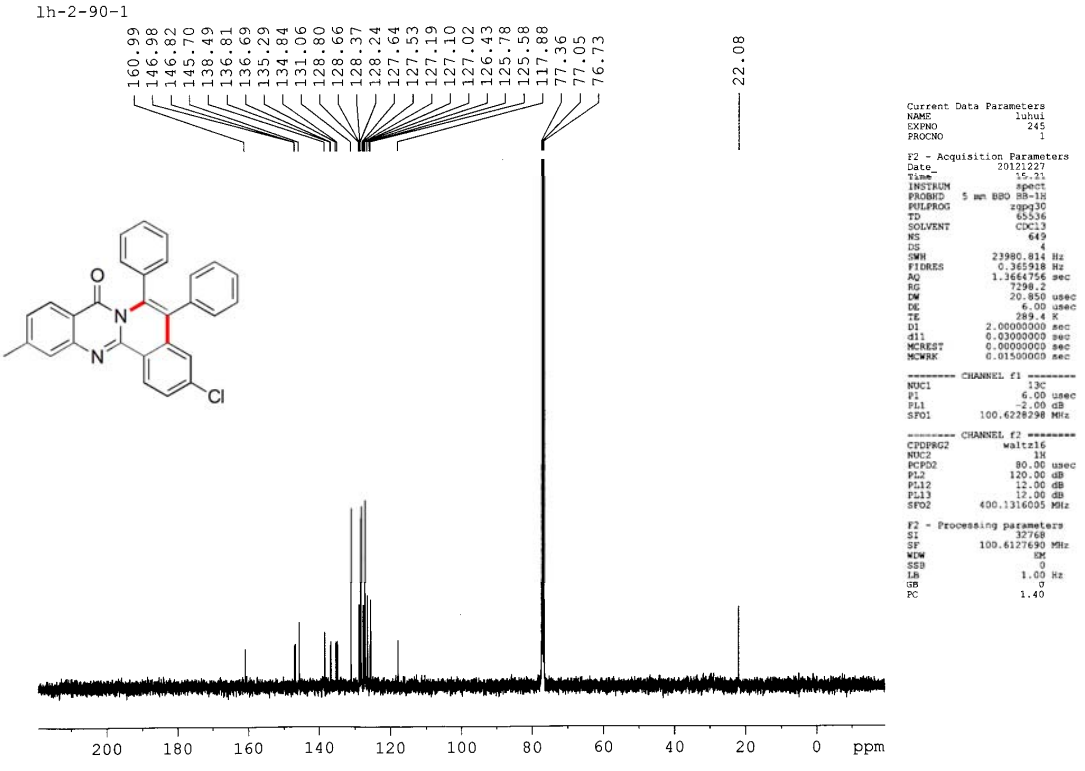
F2 - Acquisition Parameters
Date_ 20121225
Time 14.08
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1931
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 5160.6
DW 20.850 usec
DE 6.00 usec
TE 313.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

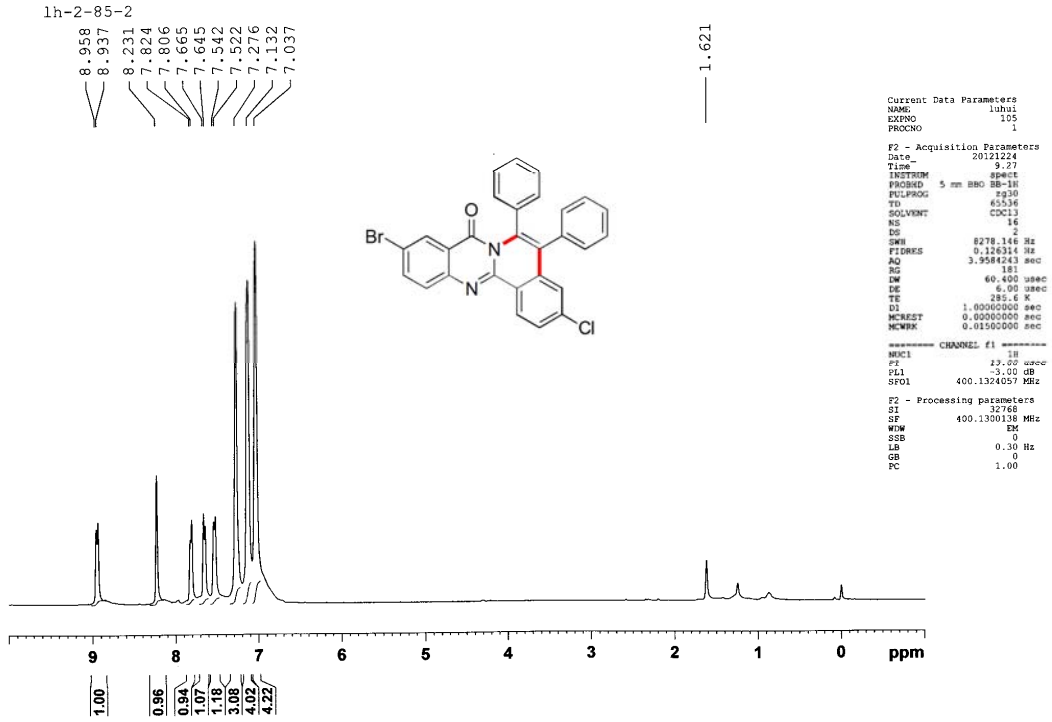
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 12.00 dB
PL13 12.00 dB
SFO2 400.1316005 MHz

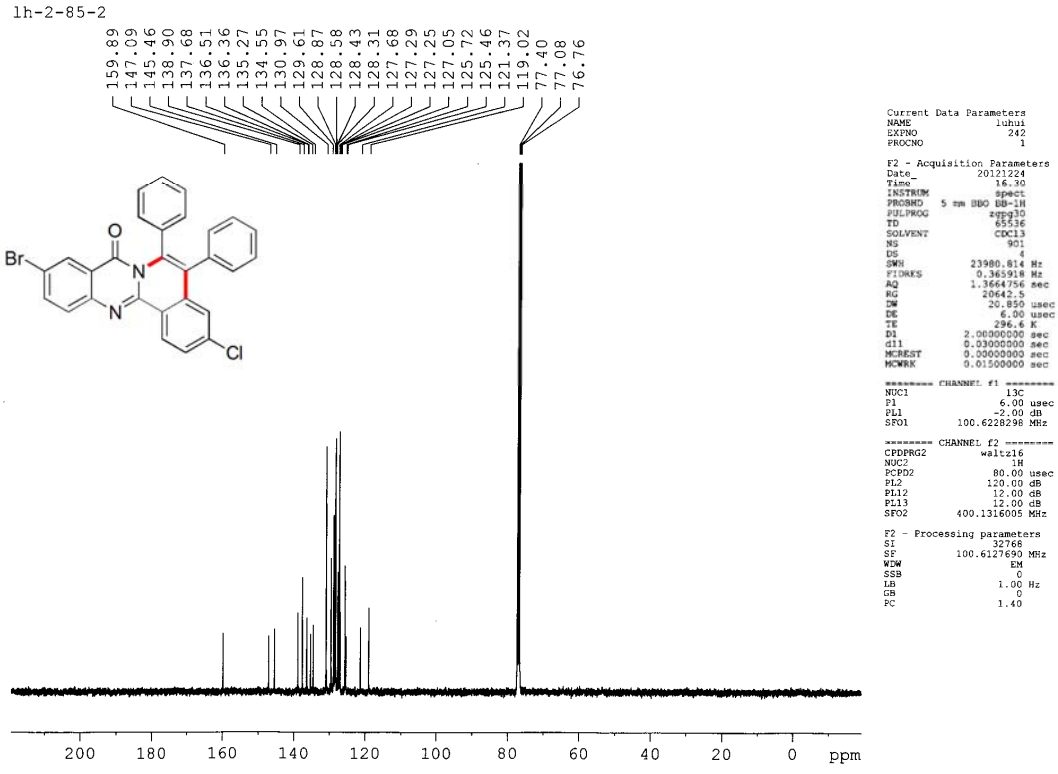
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



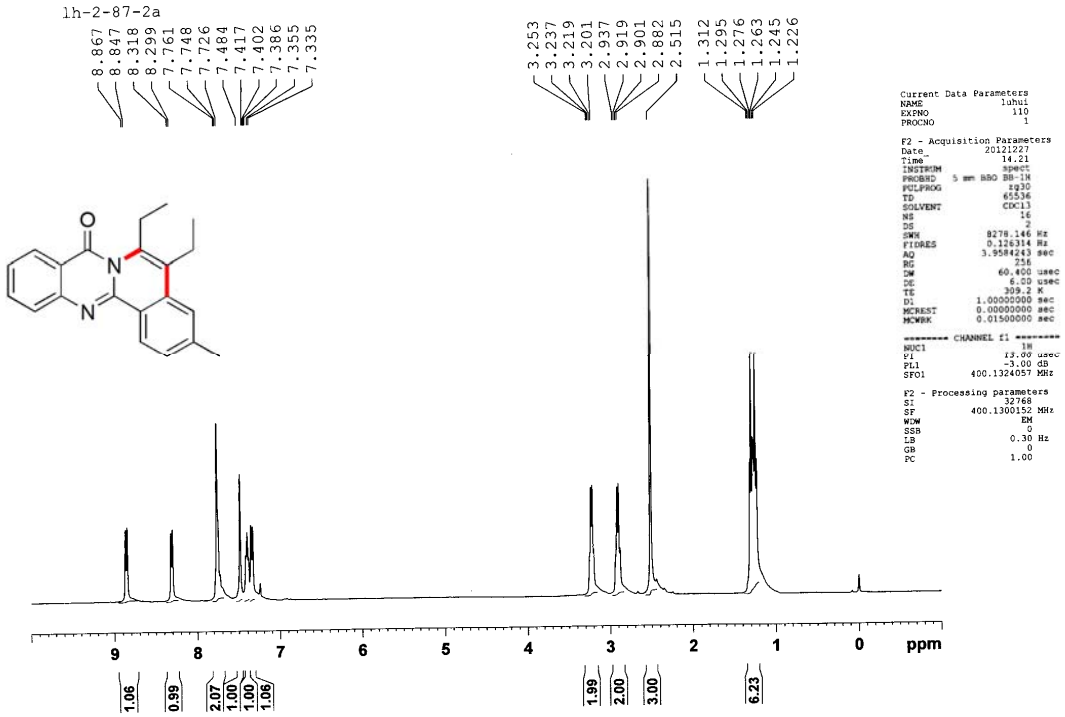



```
Current Data Parameters
NAME                  luhui
EXPNO                   105
PROCNO                    1

F2 - Acquisition Parameters
Date_              20121224
Time                   9.27
INSTRUM               spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                    181
DW                 60.400 usec
DE                  6.00 usec
TE                 285.6 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.00 usec
PL1                -3.00 dB
SFO1         400.1324057 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300138 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



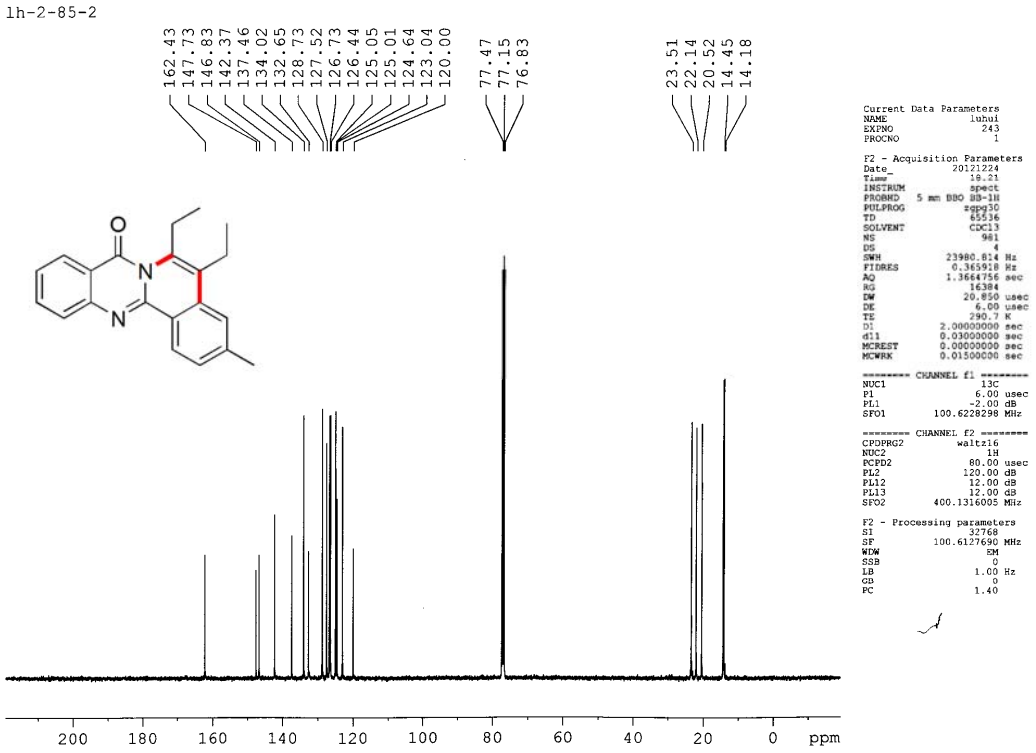

```
Current Data Parameters
NAME                  luhui
EXPNO                   242
PROCNO                    1

F2 - Acquisition Parameters
Date_              20121224
Time                  16.30
INSTRUM               spect
PROBHD   5 mm BBO BB-1H
PULPROG             zgpg30
TD                 65536
SOLVENT             CDCl3
NS                    901
DS                      4
SWH              23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664756 sec
RG                  20642.5
DW                 20.850 usec
DE                  6.00 usec
TE                 296.6 K
D1              2.00000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  6.00 usec
PL1                -2.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                120.00 dB
PL12                12.00 dB
PL13                12.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

lh-2-87-2a

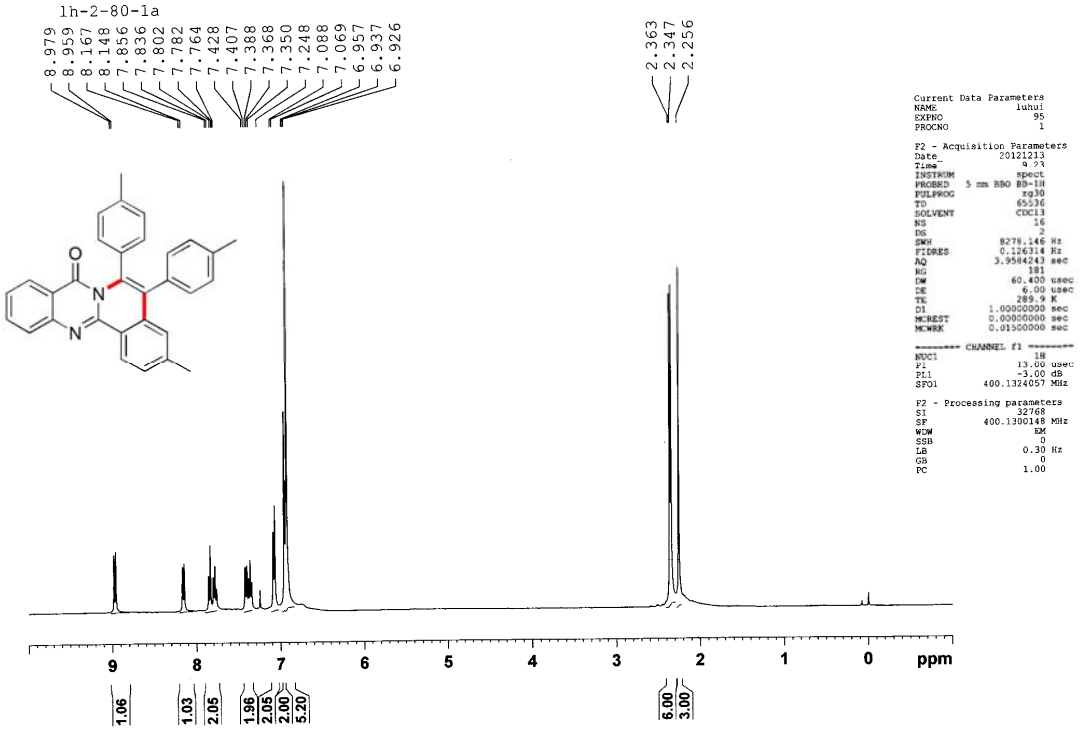


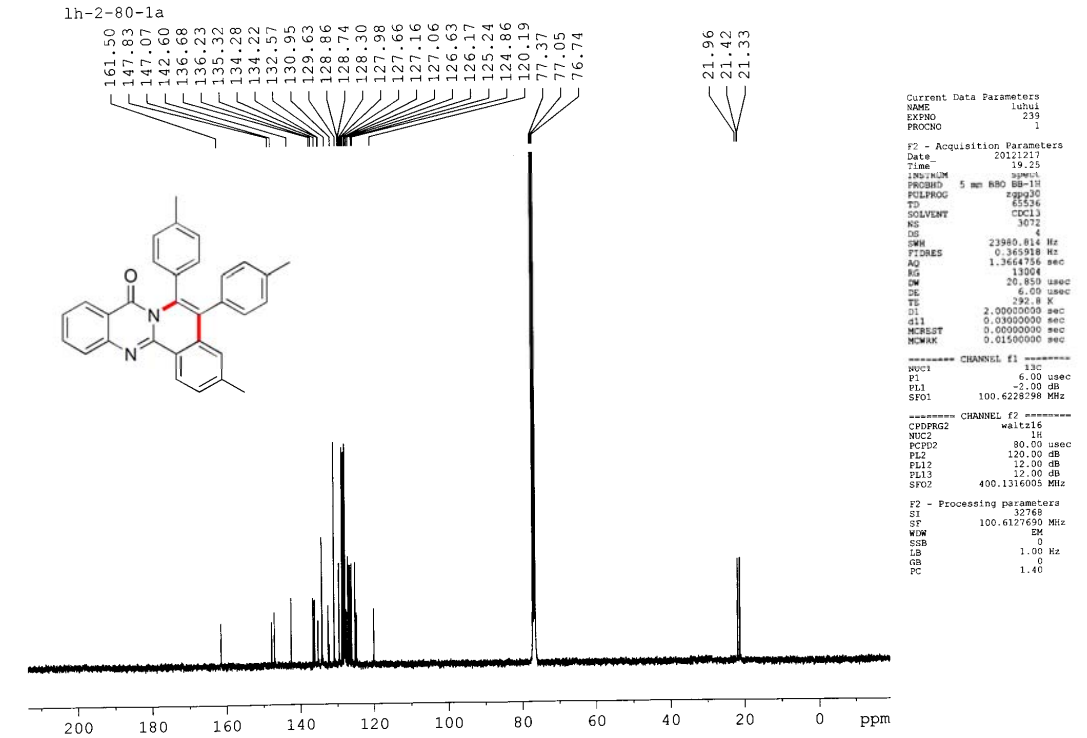

```
Current Data Parameters
NAME                  luhui
EXPNO                   110
PROCNO                    1

F2 - Acquisition Parameters
Date_              20121227
Time                  14.21
INSTRUM               spect
PROBHD  5 mm BBO BB-1H
PULPROG               zg30
TD                   65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8278.146 Hz
FIDRES             0.126314 Hz
AQ                 3.9584243 sec
RG                      256
DW                   60.400 usec
DE                    6.00 usec
TE                   309.2 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    13.00 usec
PL1                   -3.00 dB
SFO1           400.1324057 MHz

F2 - Processing parameters
SI                       32768
SF                 400.1300152 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                        1.00
```

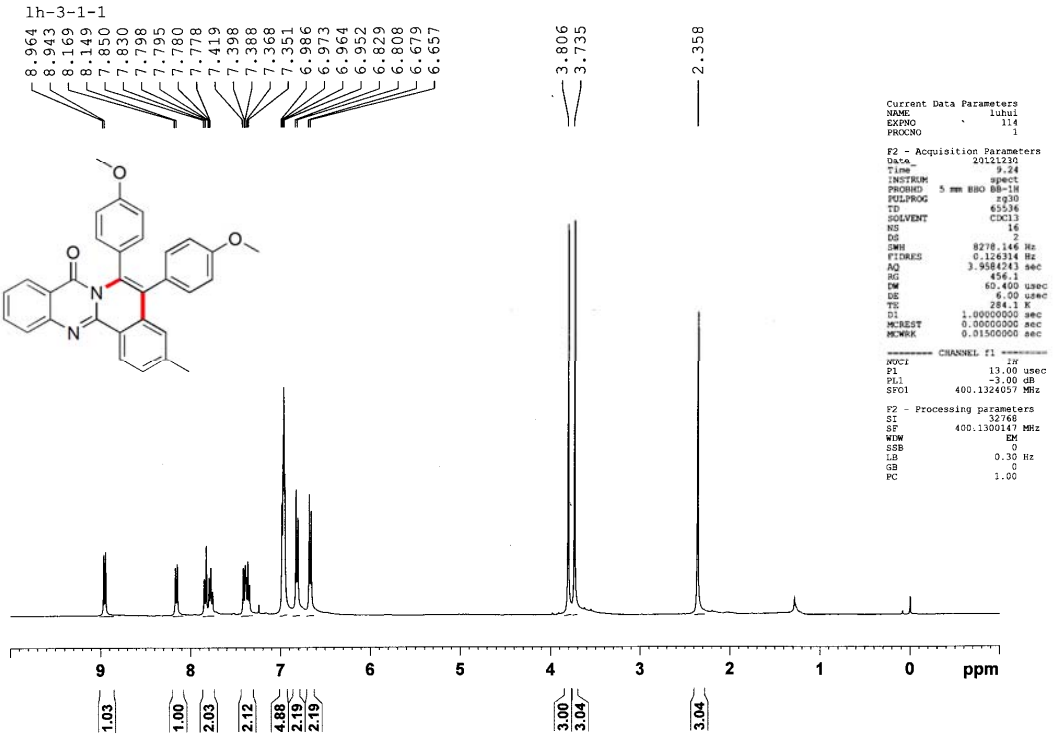
lh-2-85-2

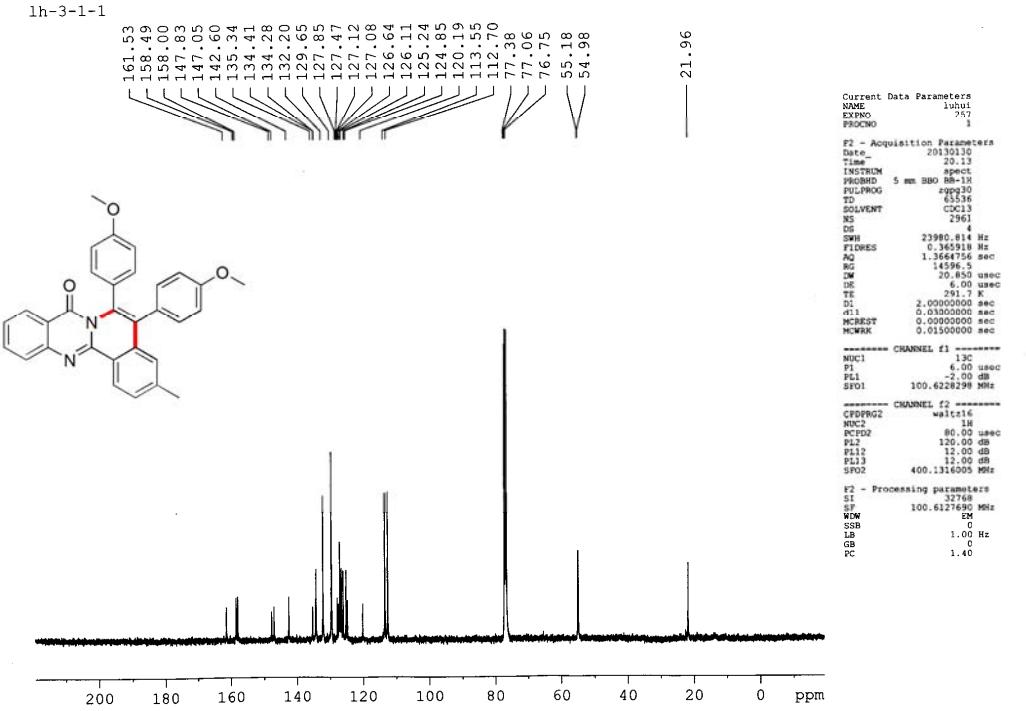


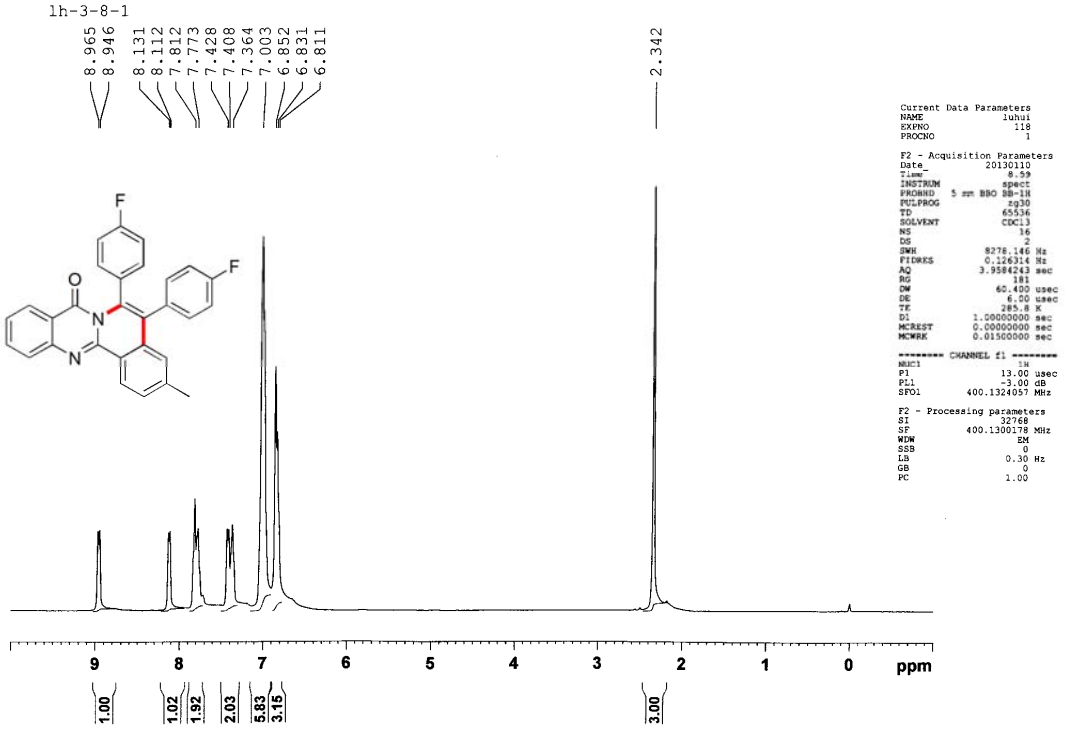

```
Current Data Parameters
NAME                  luhui
EXPNO                   243
PROCNO                    1

F2 - Acquisition Parameters
Date_              20121224
Time                  18.21
INSTRUM               spect
PROBHD  5 mm BBO BB-1H
PULPROG             zgpg30
TD                   65536
SOLVENT               CDCl3
NS                      981
DS                        4
SWH                23980.814 Hz
FIDRES             0.365918 Hz
AQ                 1.3664756 sec
RG                    16384
DW                   20.850 usec
DE                    6.00 usec
TE                   290.7 K
D1                 2.00000000 sec
d11                0.03000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                    6.00 usec
PL1                   -2.00 dB
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                80.00 usec
PL2                  120.00 dB
PL12                  12.00 dB
PL13                  12.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF                 100.6127690 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                        1.40
```

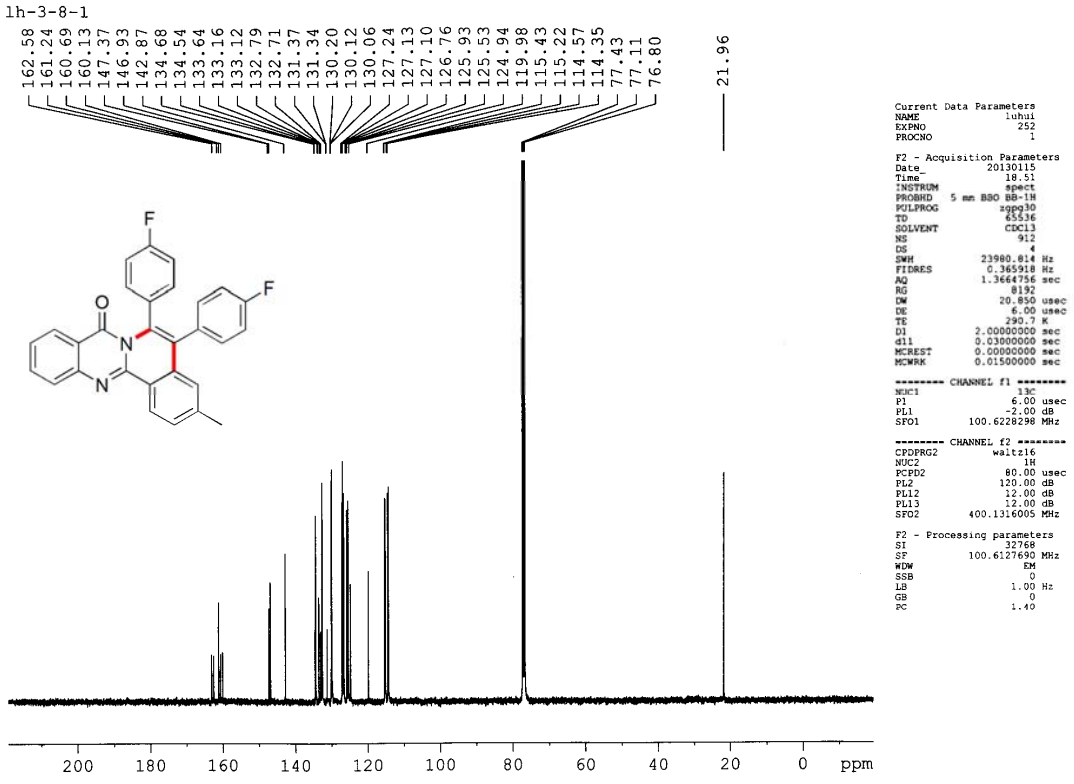


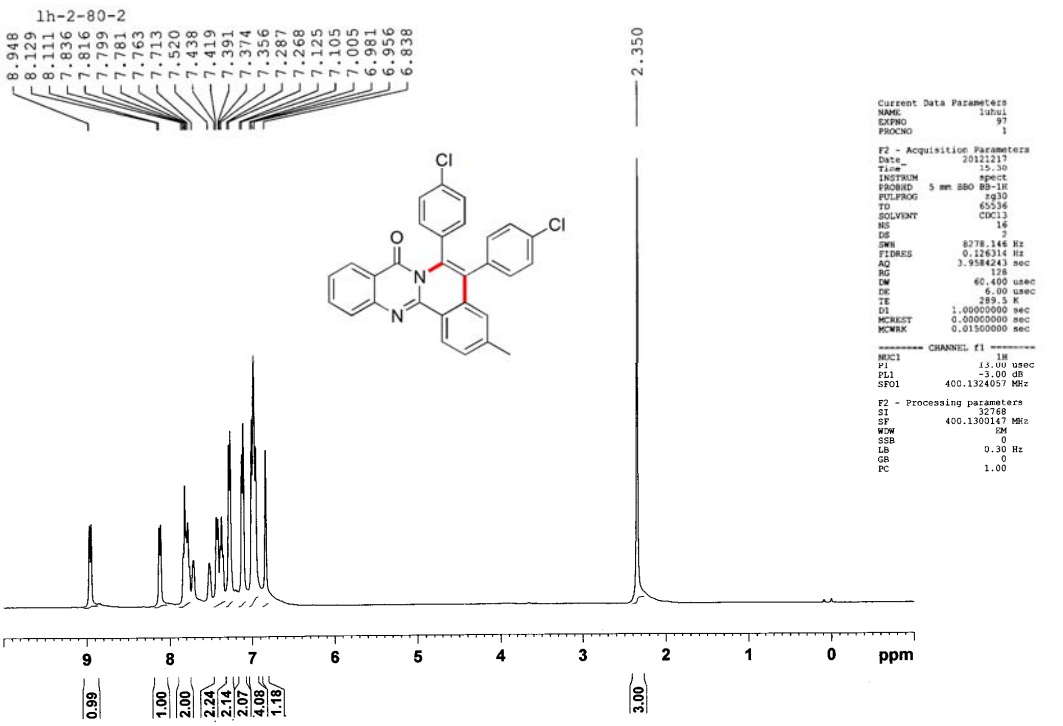



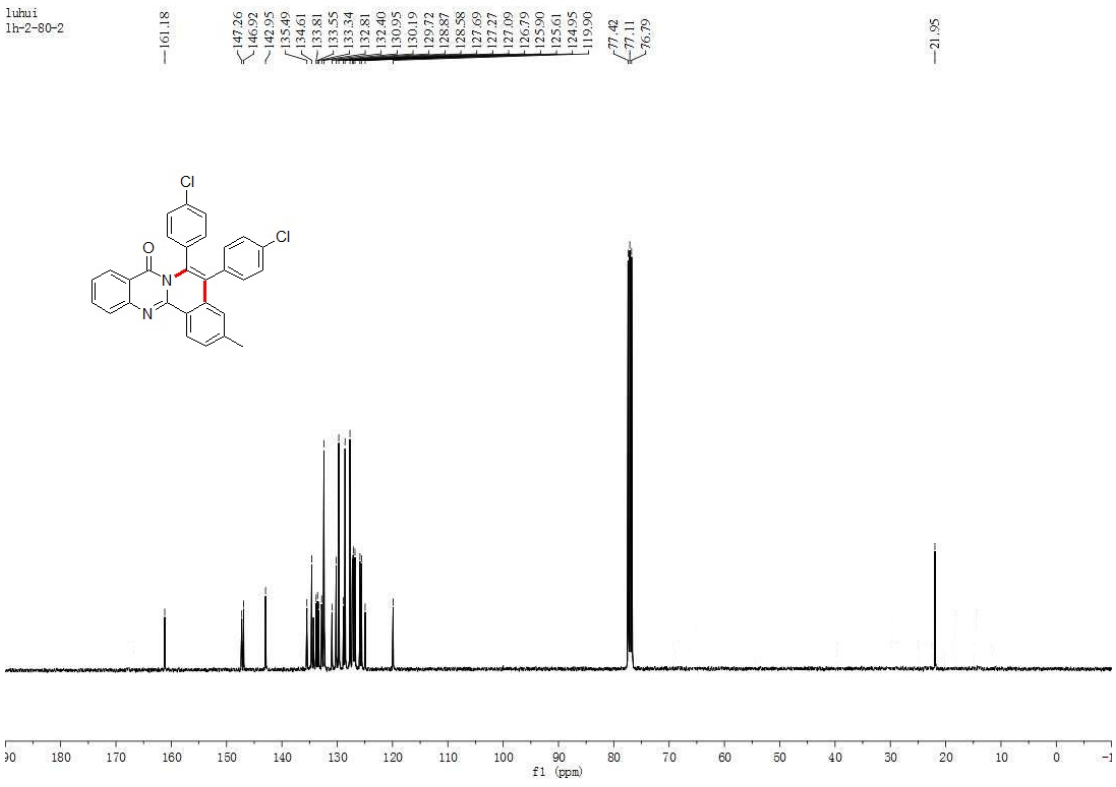


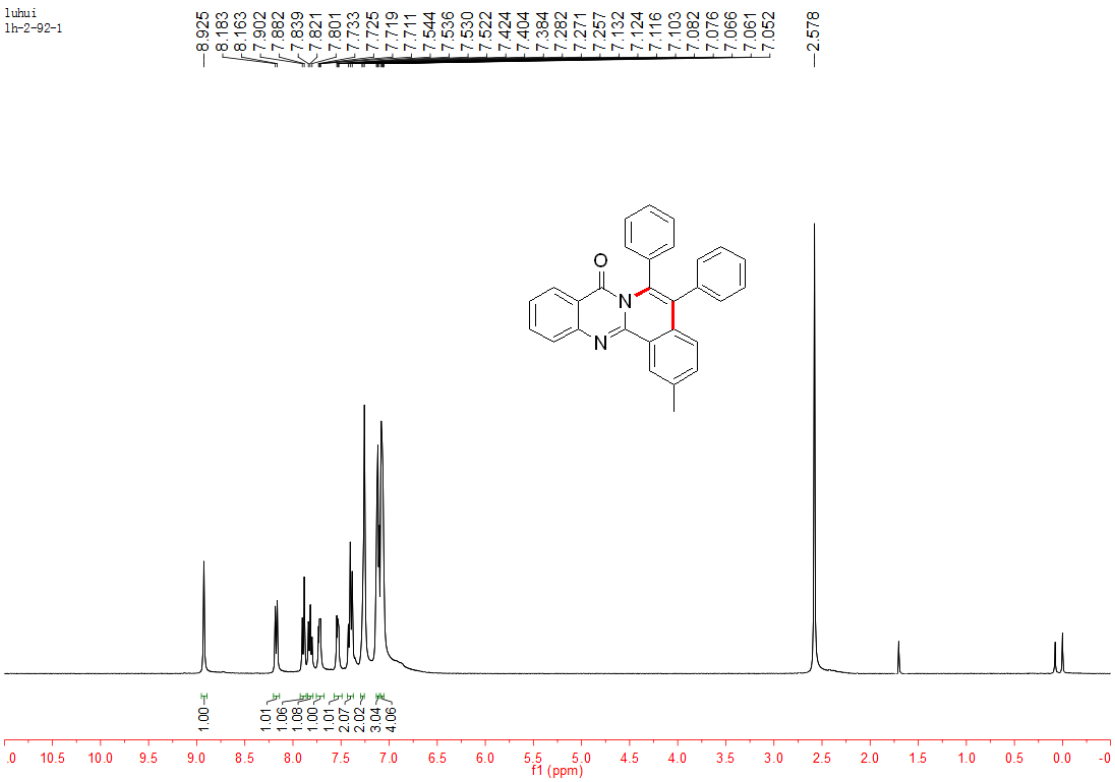



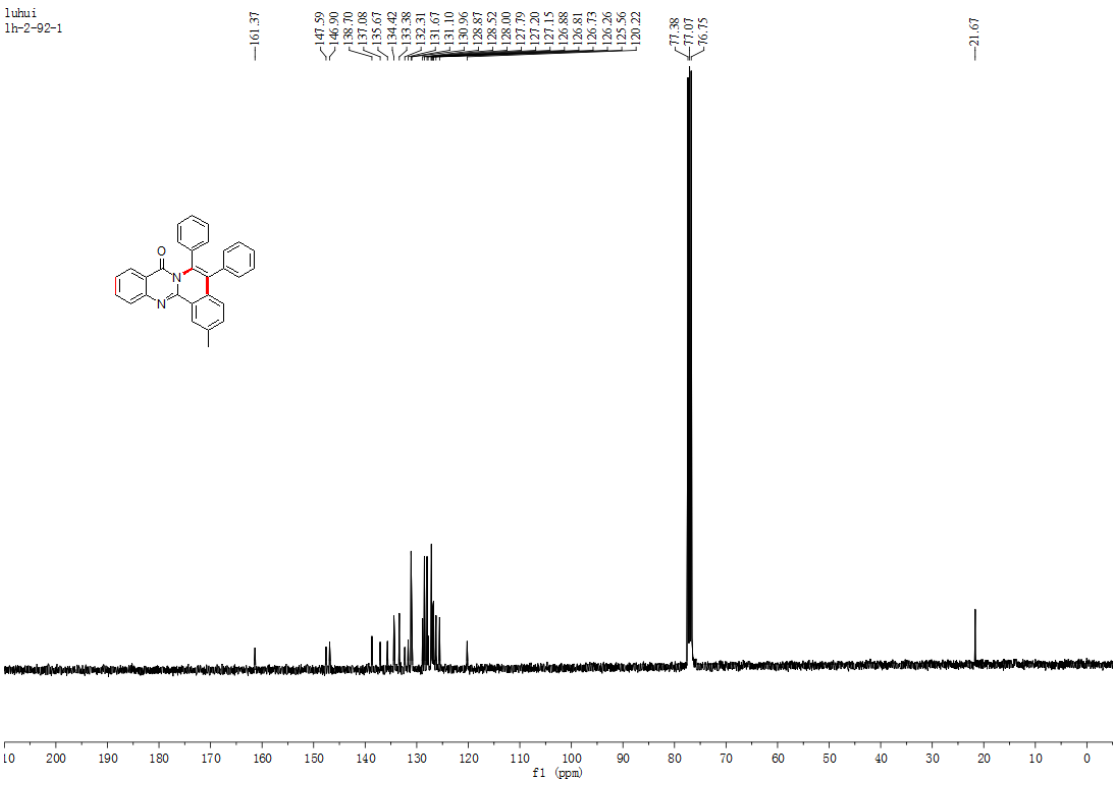


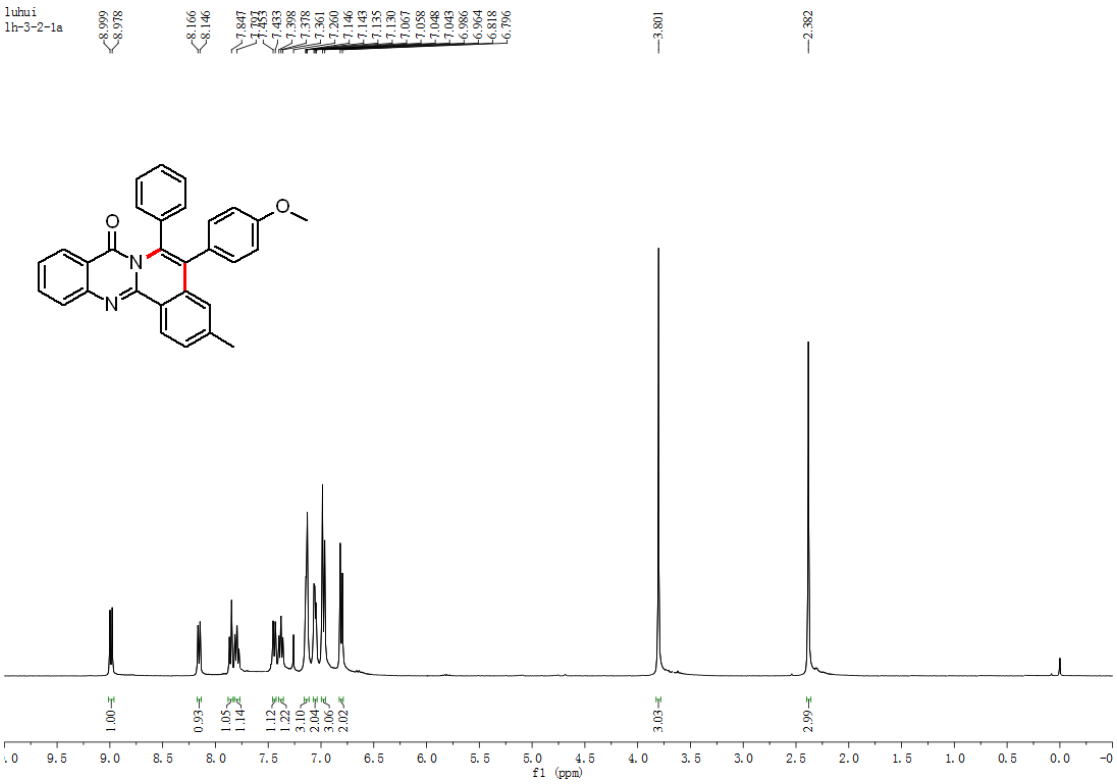



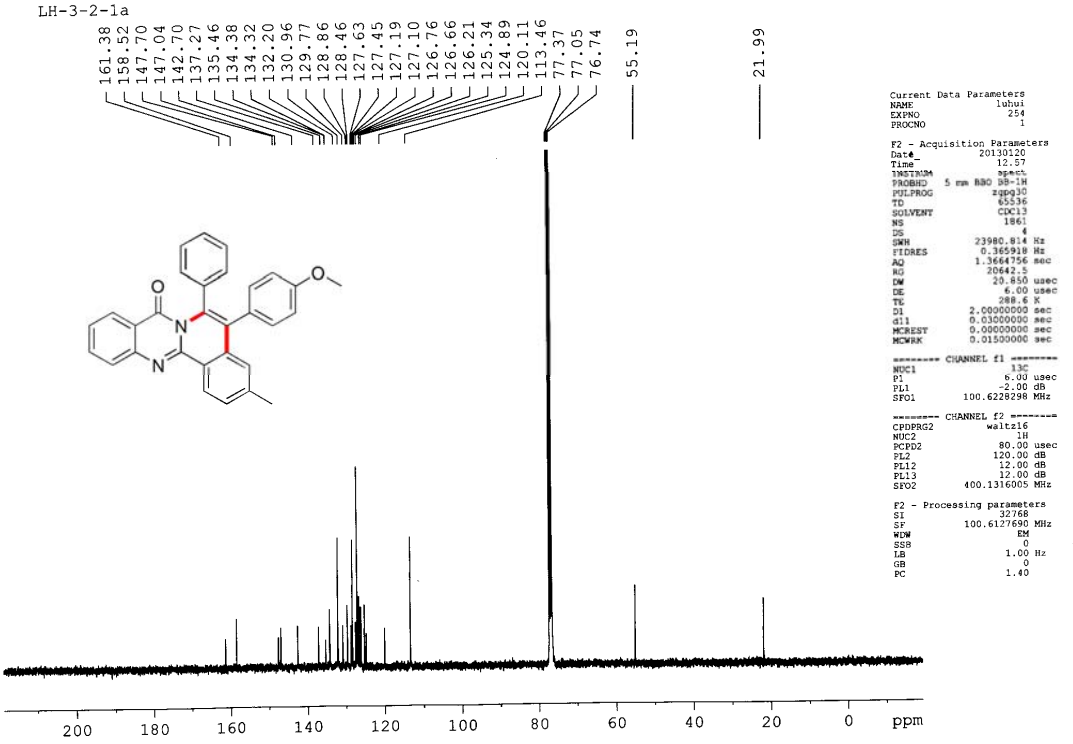






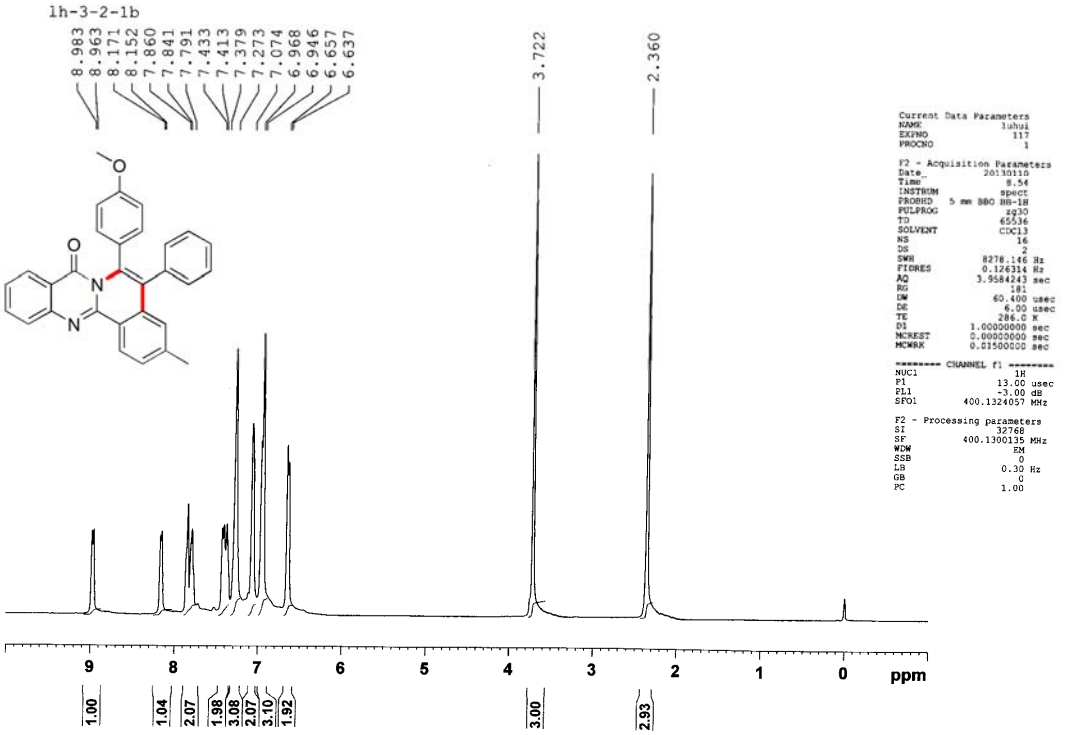

Current Data Parameters
NAME luhui
EXPNO 254
PROCNO 1

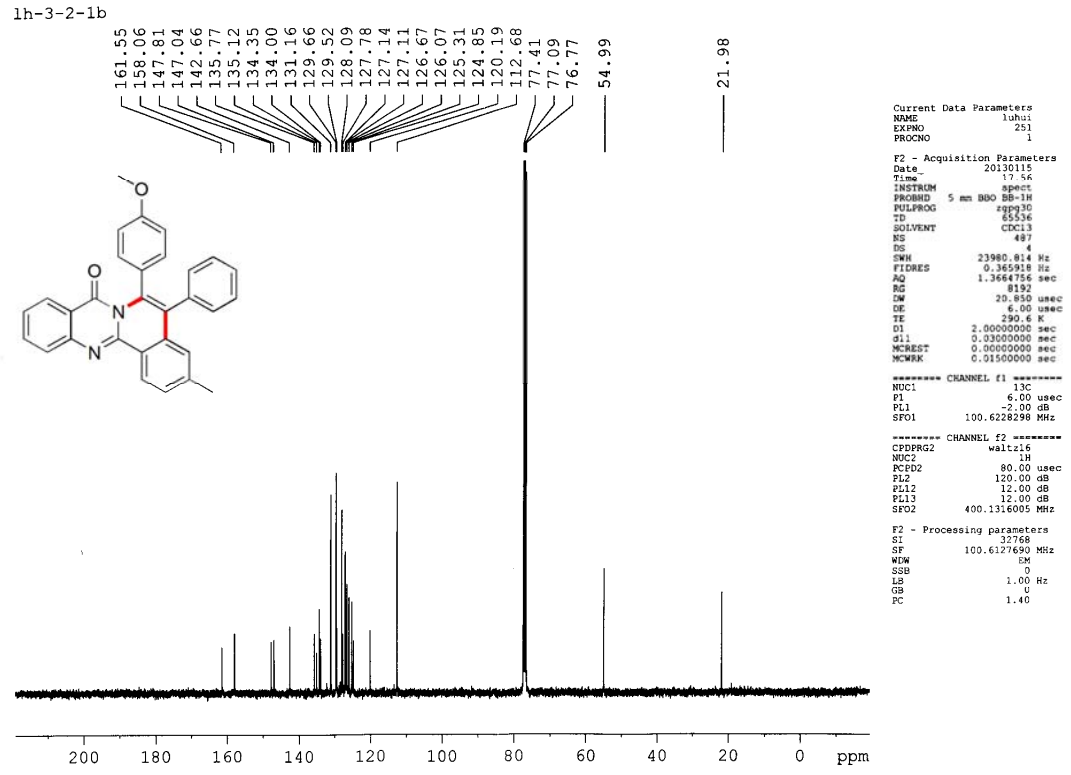
F2 - Acquisition Parameters
Date_ 20130120
Time 12.57
INSTRM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1861
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 20642.5
DW 20.850 usec
DE 6.00 usec
TE 288.6 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

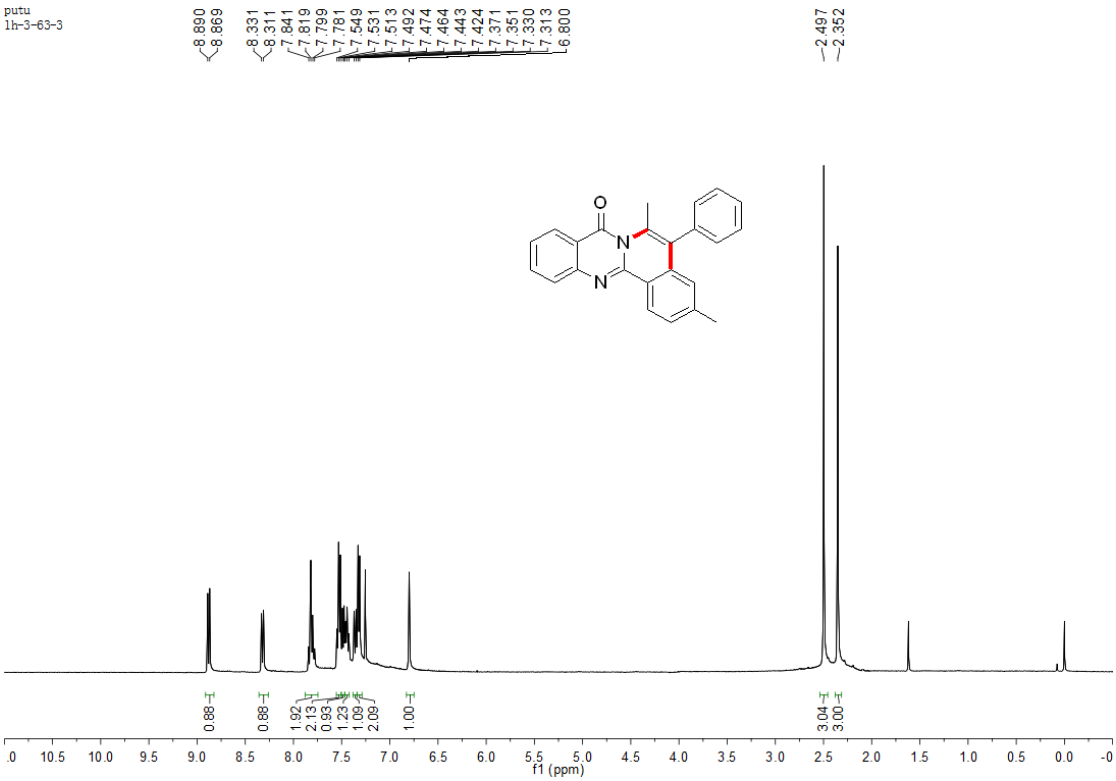
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

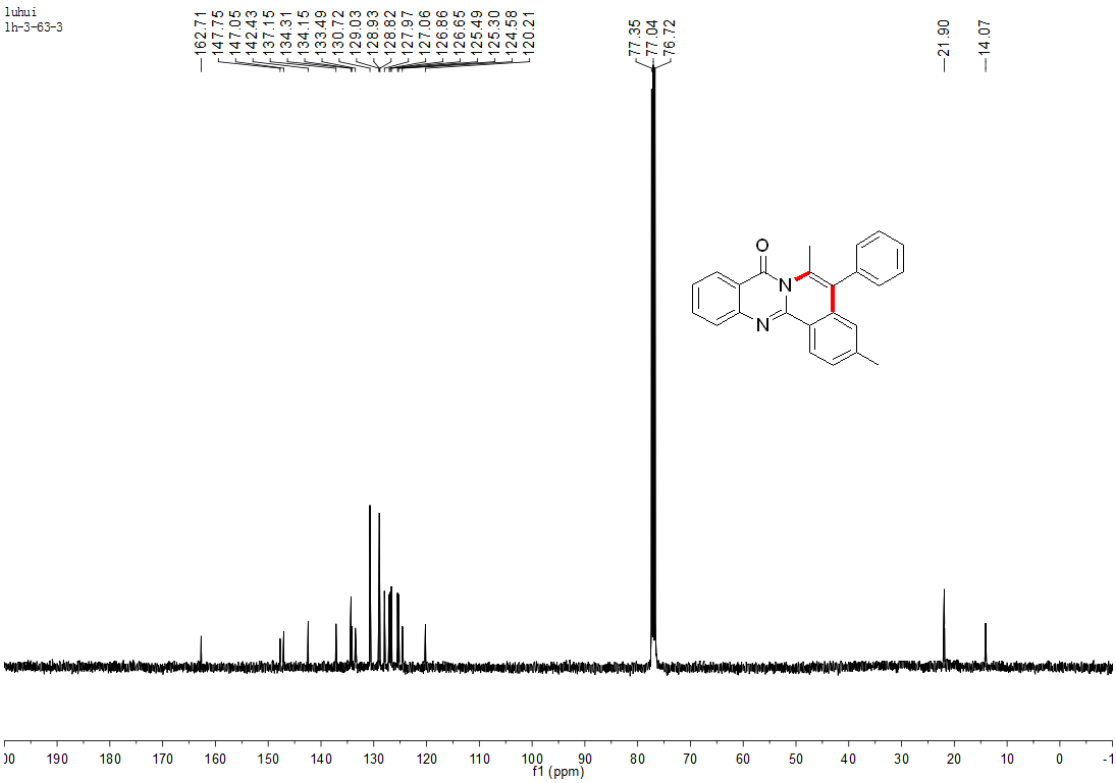
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 12.00 dB
PL13 12.00 dB
SFO2 400.1316005 MHz

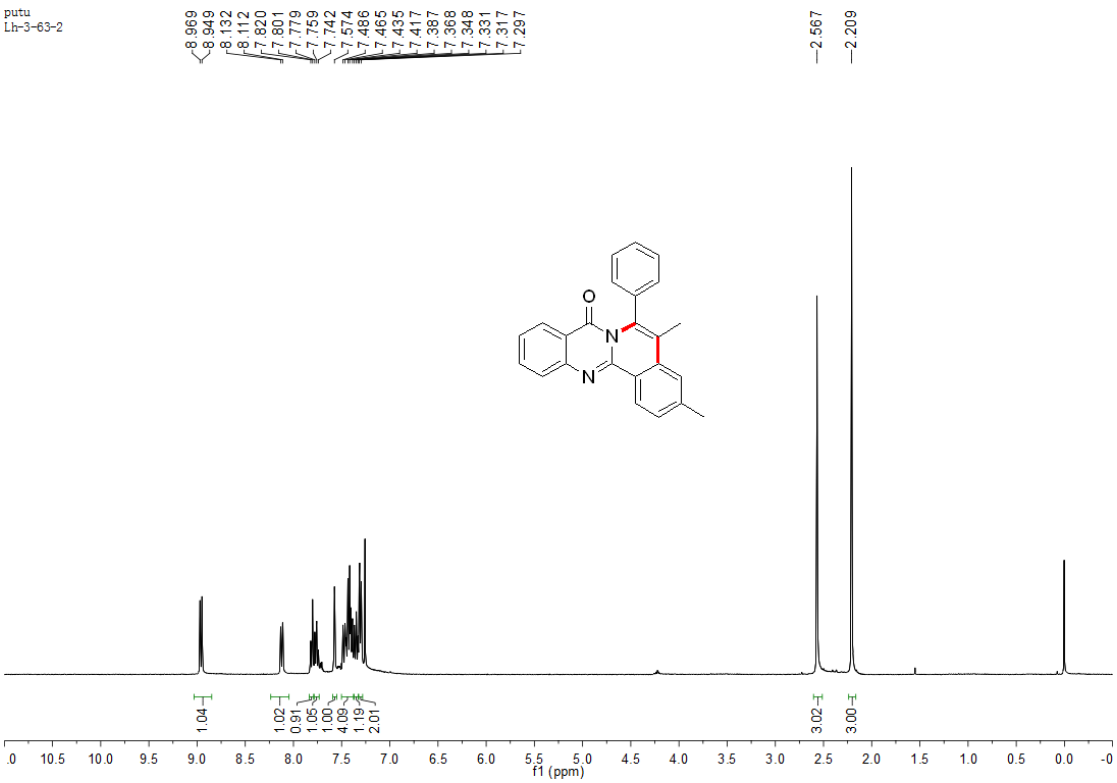
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

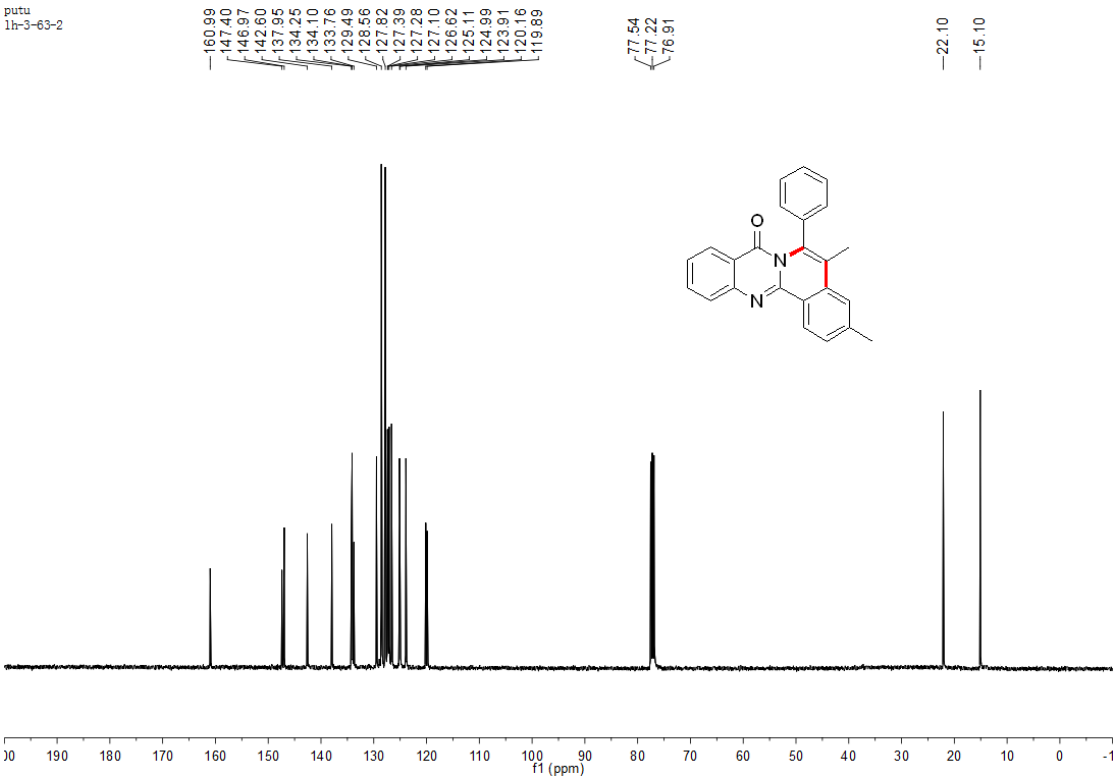


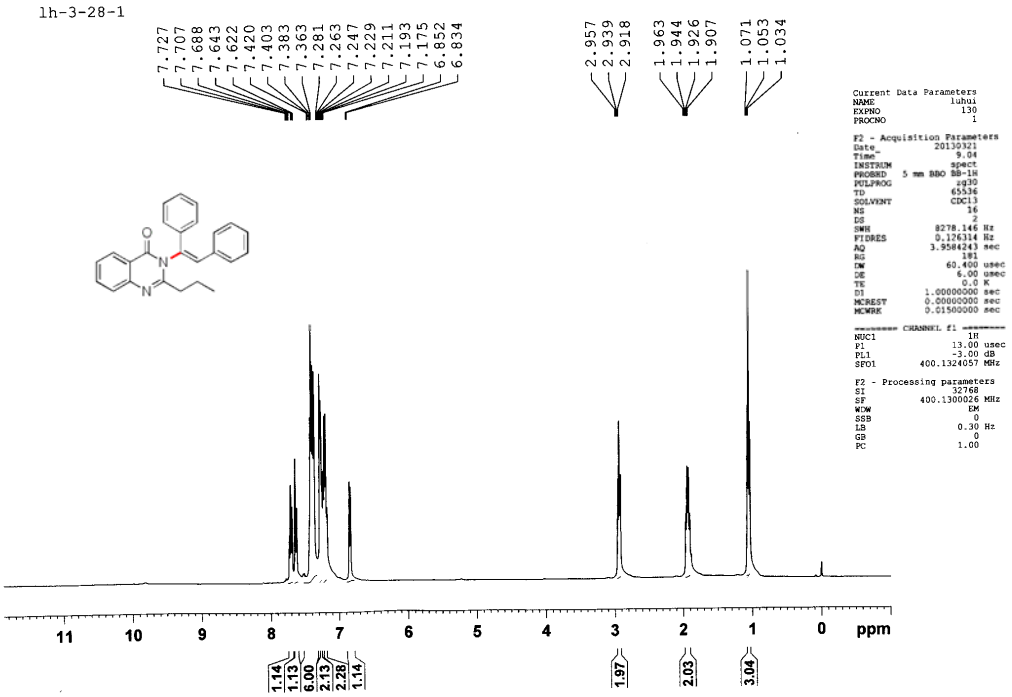



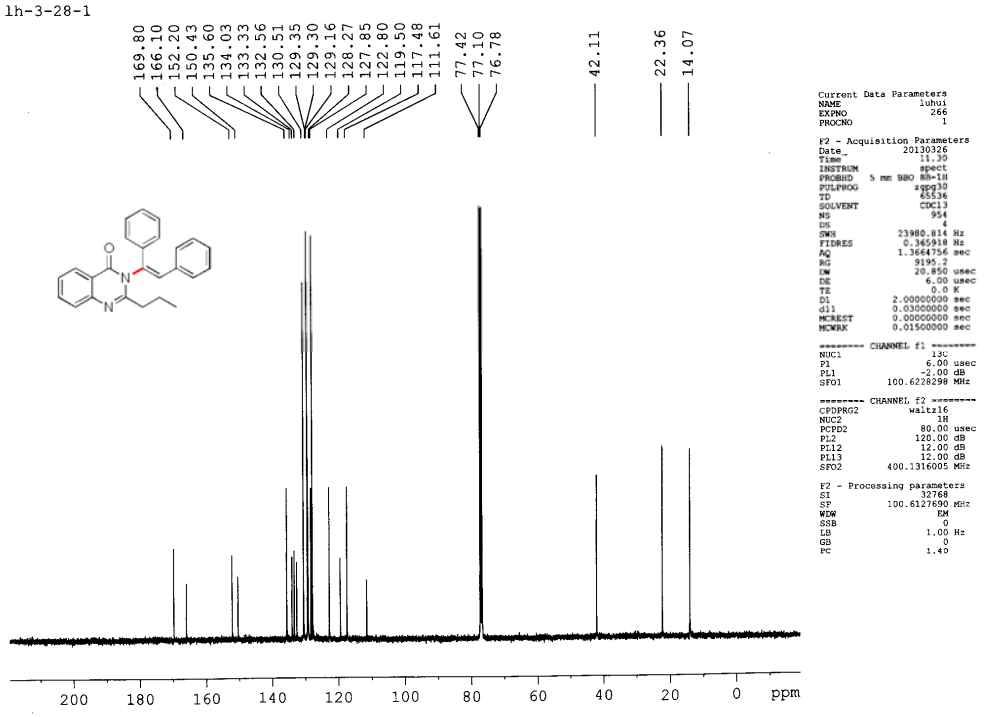


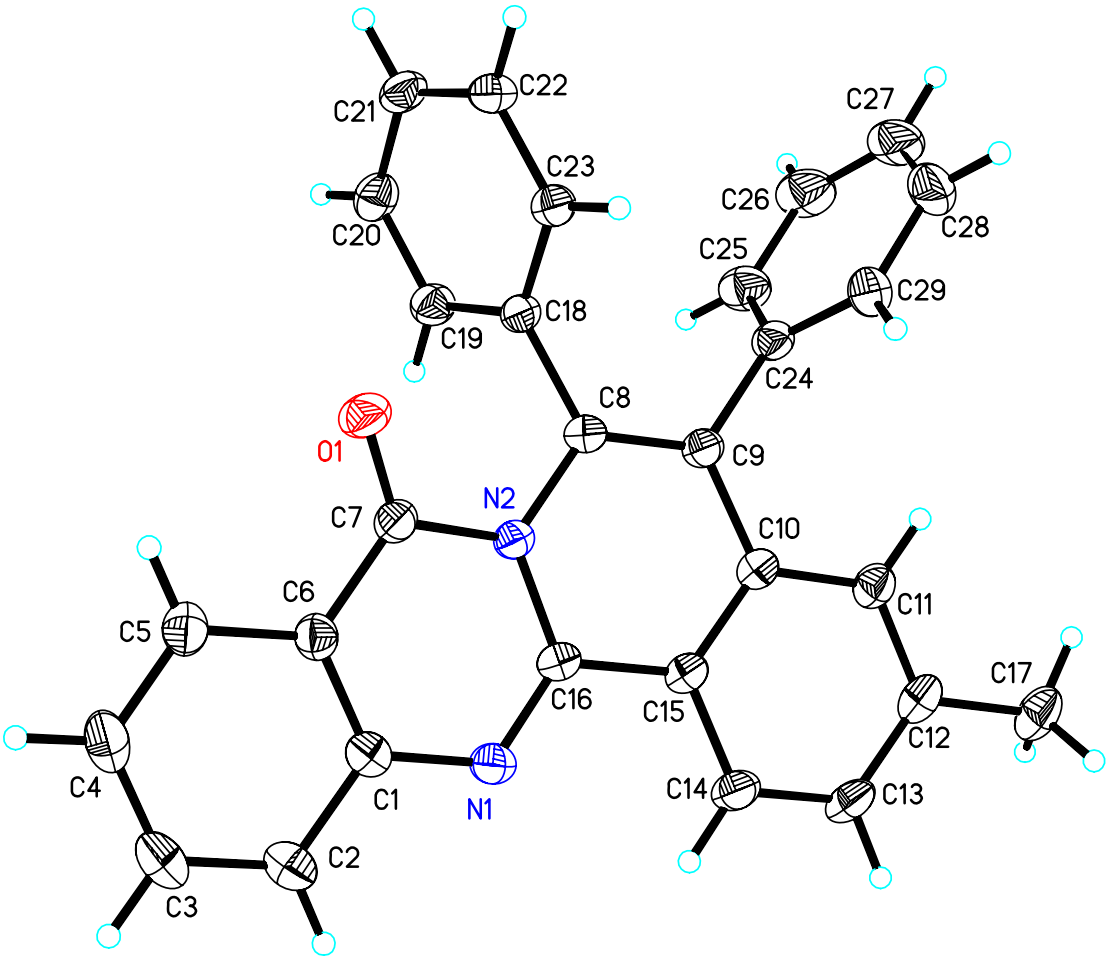



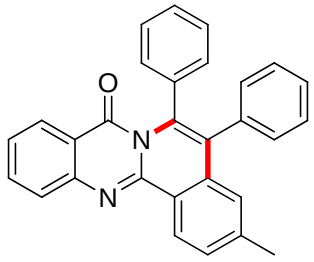


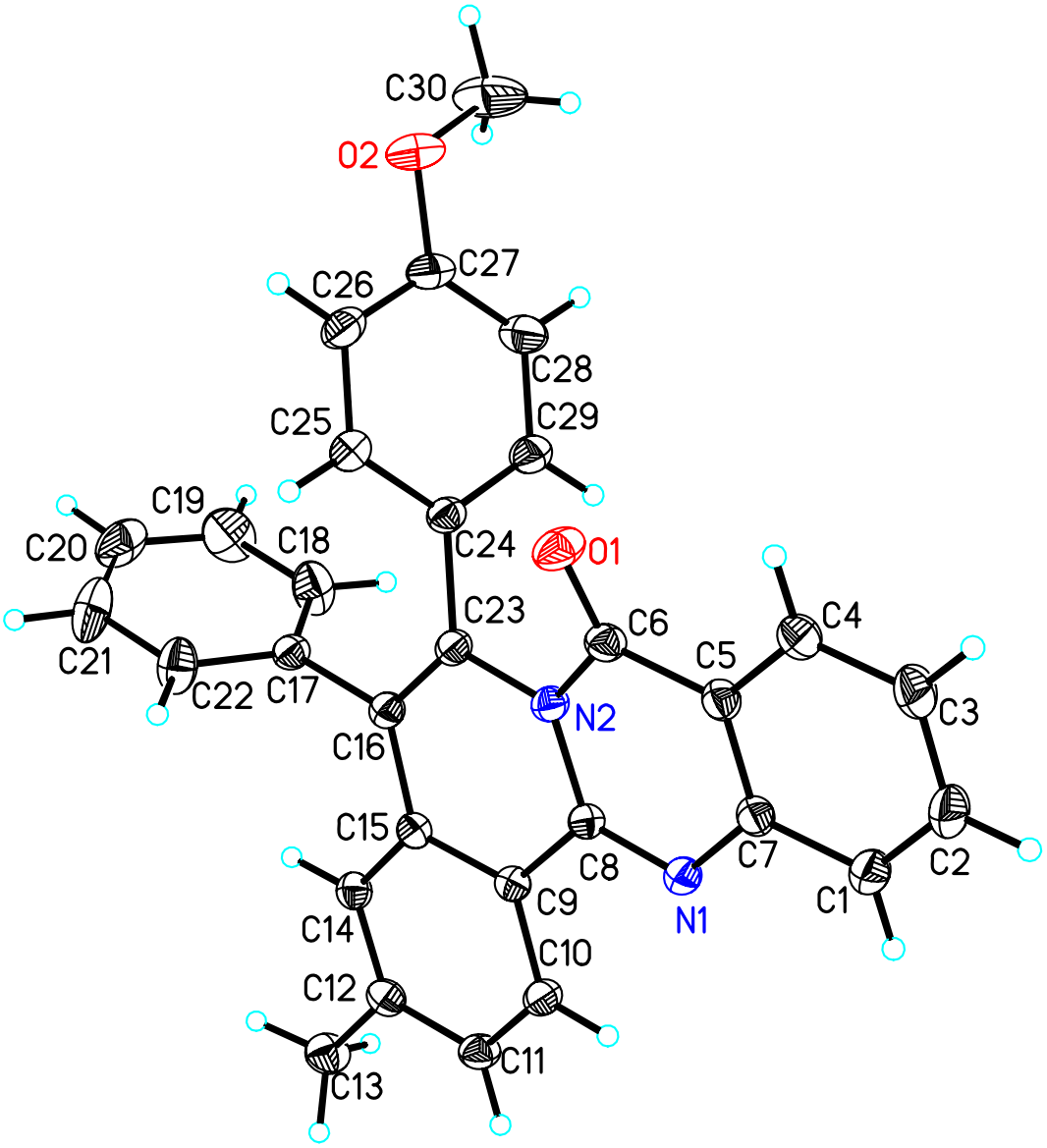


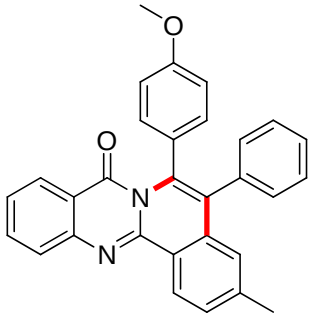





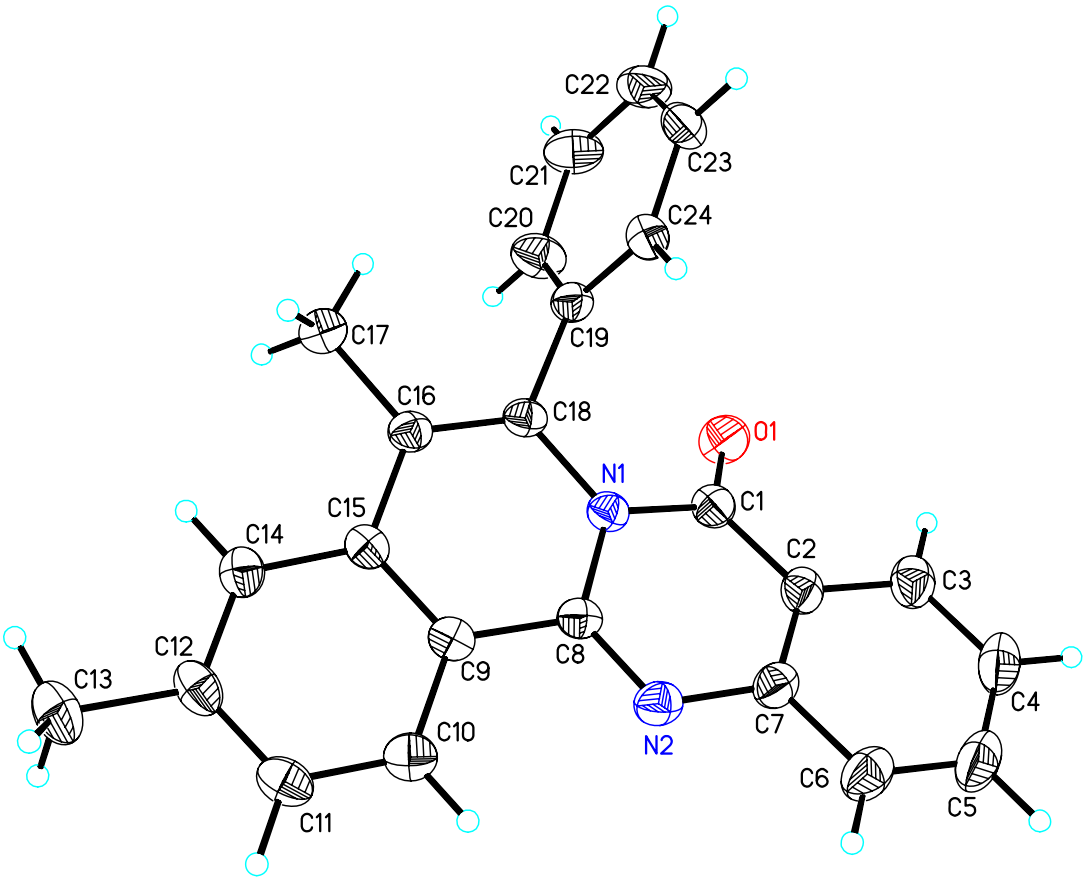



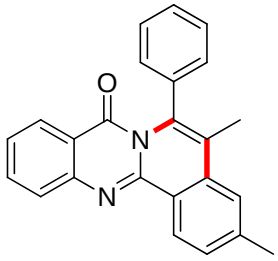

3a




**7b**




**9b**