Synthesis of 3-halochromones with simple KX halogen sources enabled by in situ halide oxidation

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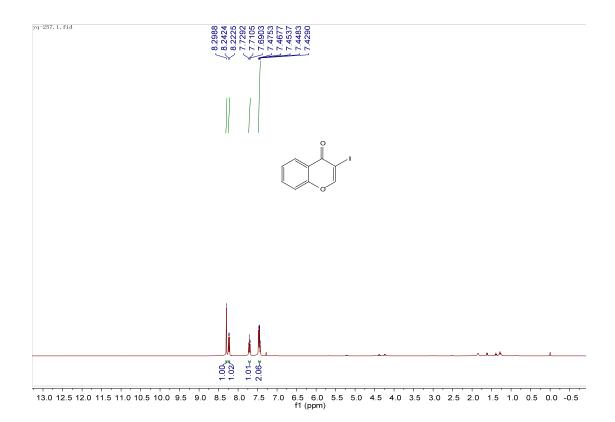
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General experimental information

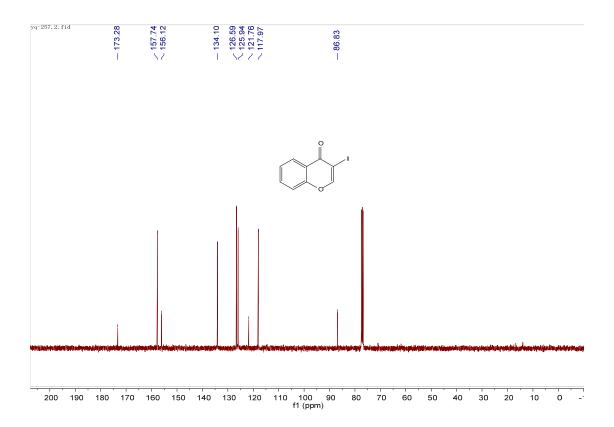
Enaminones 1 were prepared following known procedure. All other reagents were obtained from commercial sources and used directly without further purification. The organic solvents were treated following standard procedures before use. All reactions were performed in 25 mL round bottom flask. Silica gel plate GF254 was used for thin layer chromatography (TLC) by visualizing with ultraviolet light. Nuclear magnetic resonance (NMR) spectra are recorded in a 400 MHz apparatus in CDCl₃ or DMSO- d_6 with TMS internal standard. The frequencies for ¹H and ¹³C NMR test were 400 HMz and 100 MHz, respectively. The chemical shifts were reported in parts per million with reference to the TMS internal standard. HMRS data for new compounds were acquired in the mass spectrometer equipped with TOF analyzer. Melting points were tested in an X-4A apparatus without correcting temperature.

Reference

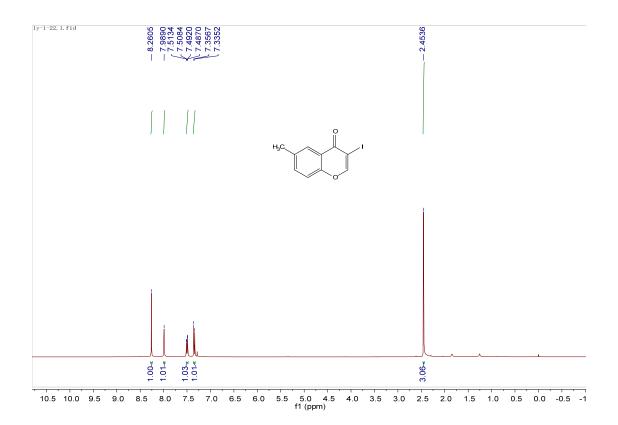
1. Y. Gao, Y. Liu and J.-P. Wan, J. Org. Chem., 2019, 84, 2243-2251.



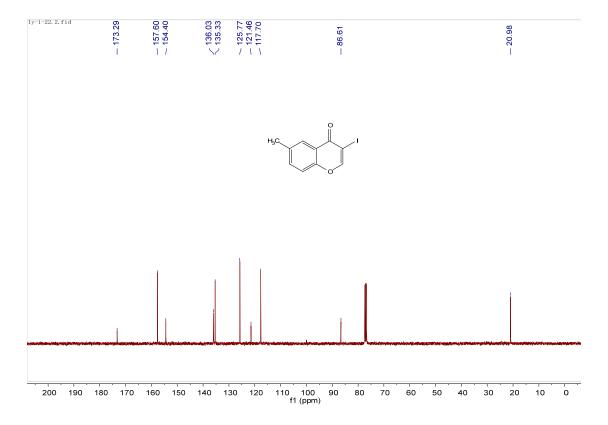
¹H NMR spectrum of 2a



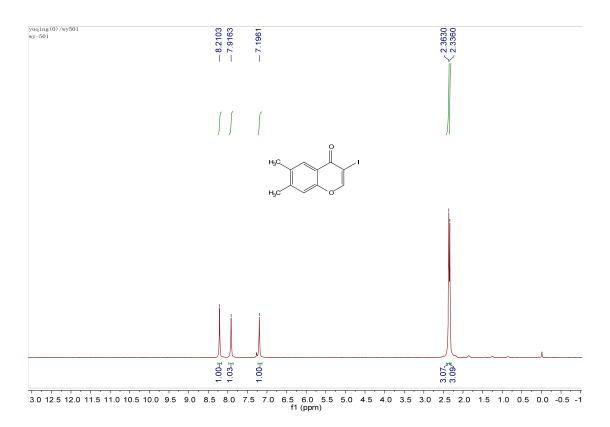
¹³C NMR spectrum of 2a



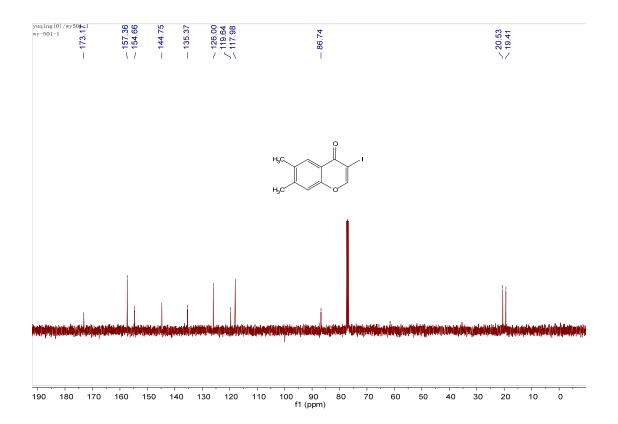
¹H NMR spectrum of 2b



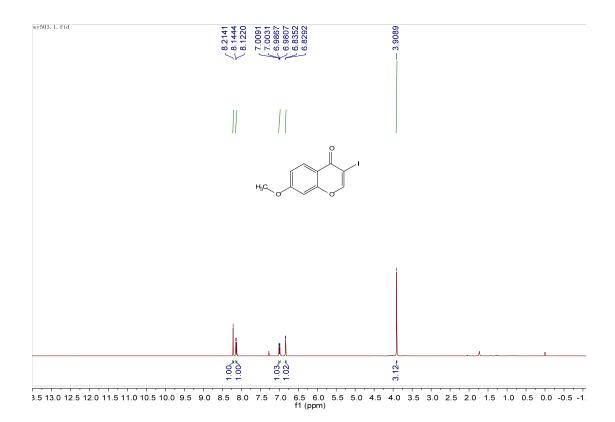
¹³C NMR spectrum of 2b



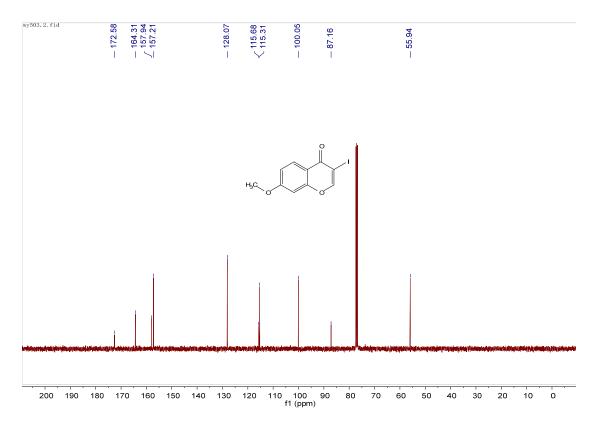
¹H NMR spectrum of 2c



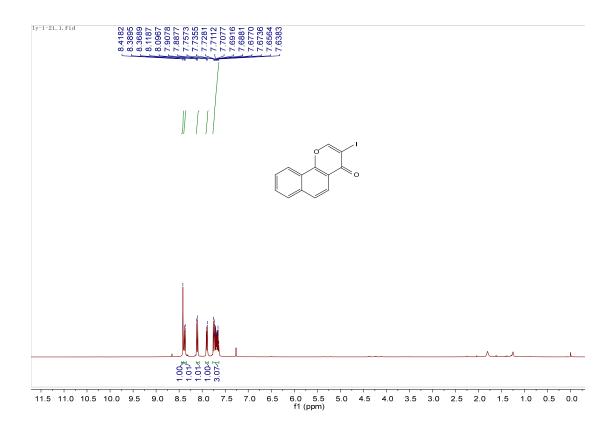
¹³C NMR spectrum of 2c



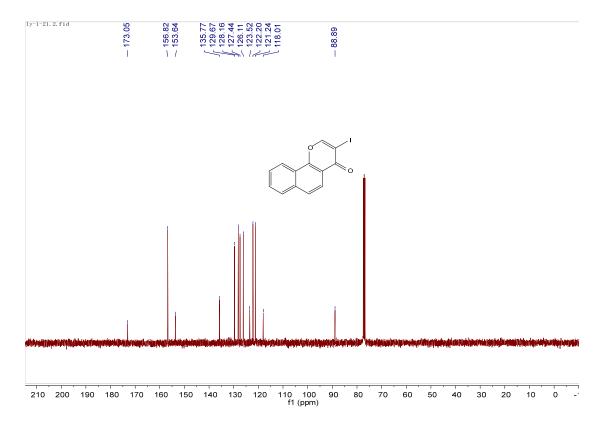
¹H NMR spectrum of 2d



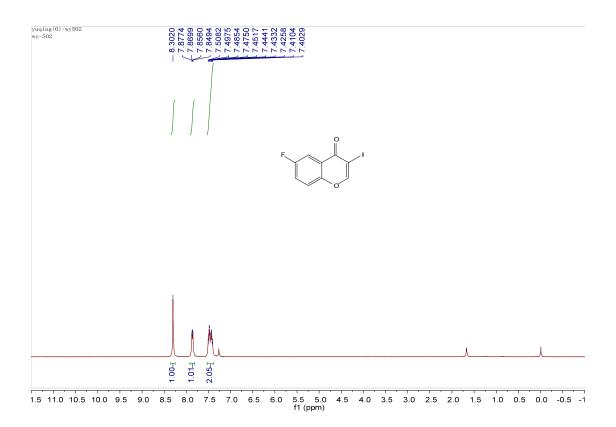
¹³C NMR spectrum of 2d



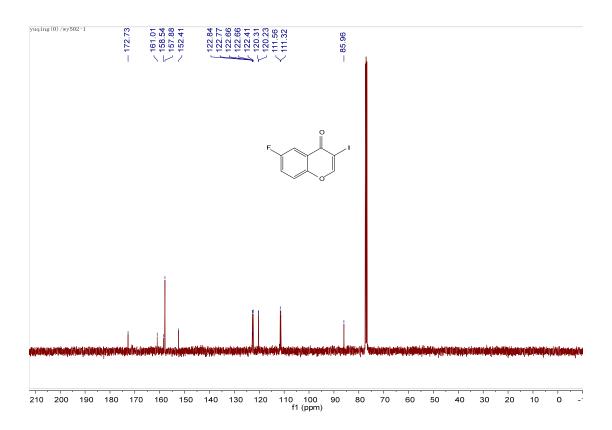
¹H NMR spectrum of 2e



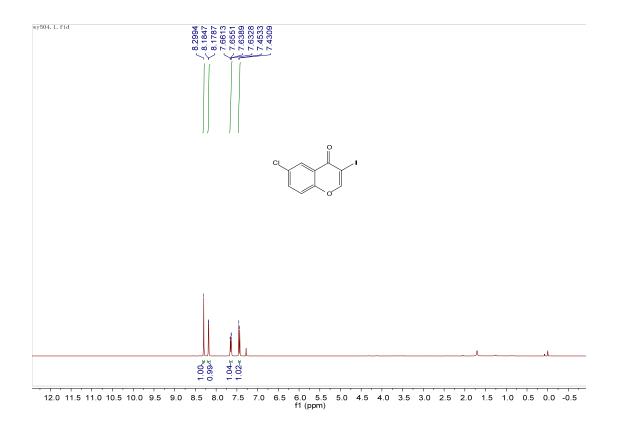
¹³C NMR spectrum of 2e



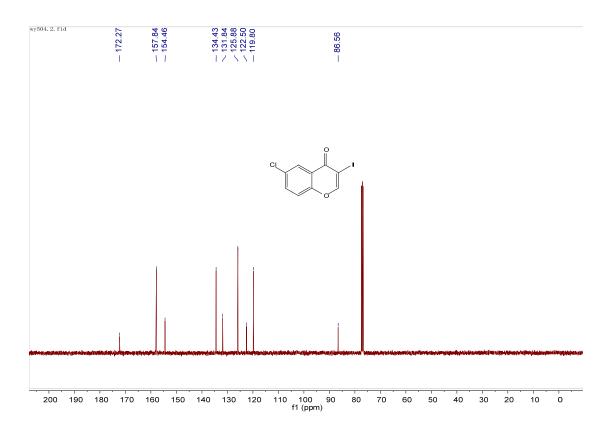
¹H NMR spectrum of 2f



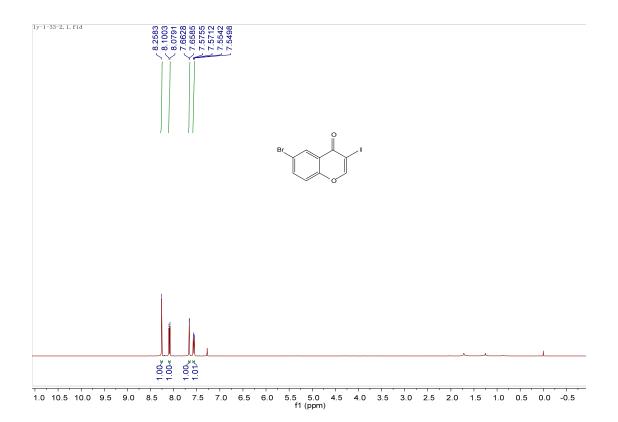
¹³C NMR spectrum of 2f



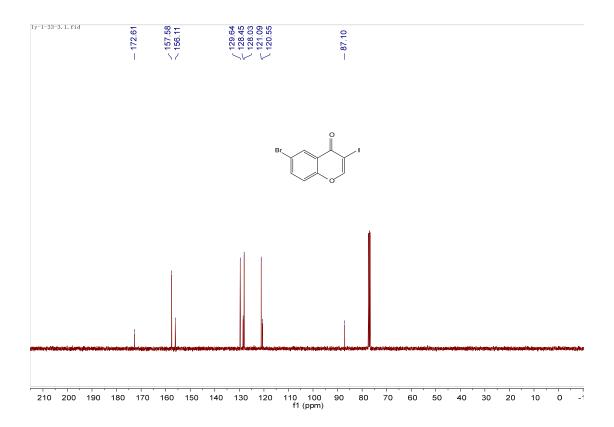
¹H NMR spectrum of 2g



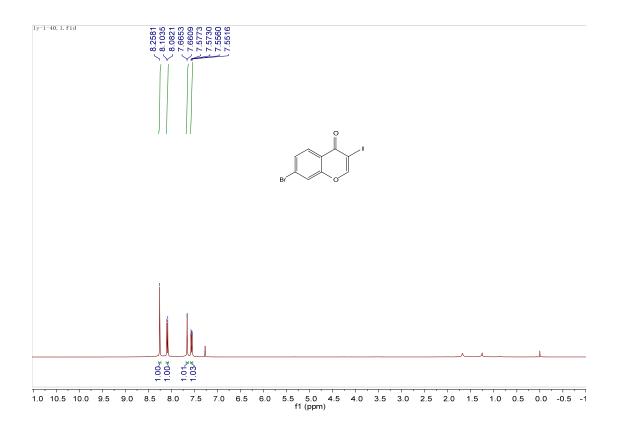
¹³C NMR spectrum of 2g



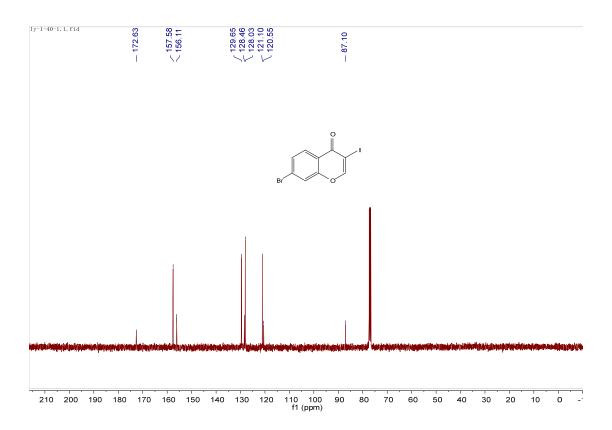
¹H NMR spectrum of 2h



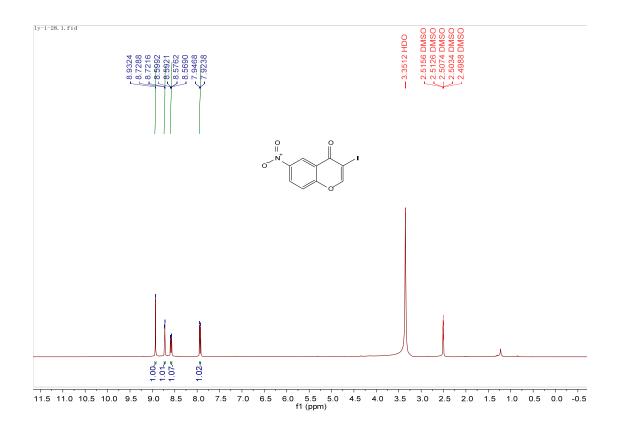
¹³C NMR spectrum of 2h



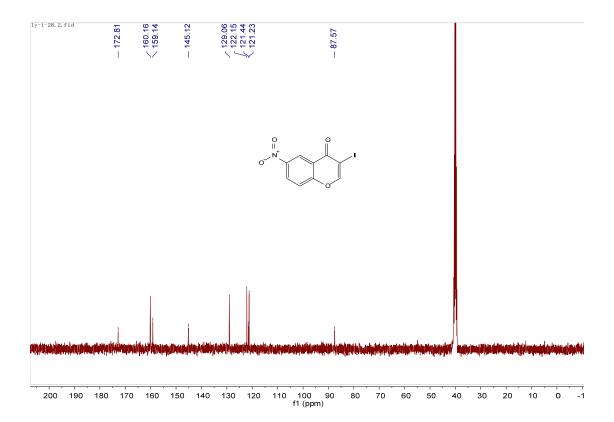
¹H NMR spectrum of 2i



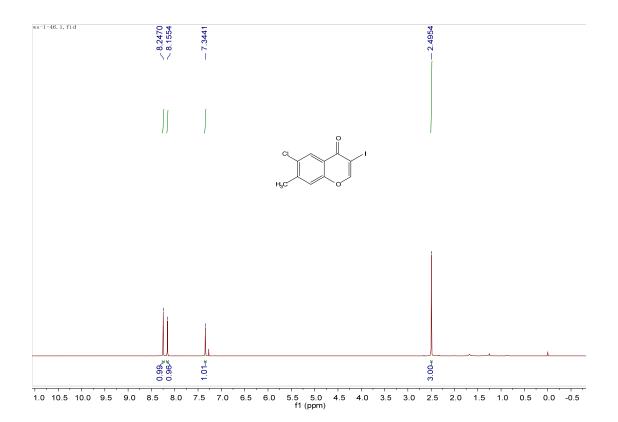
¹³C NMR spectrum of 2i



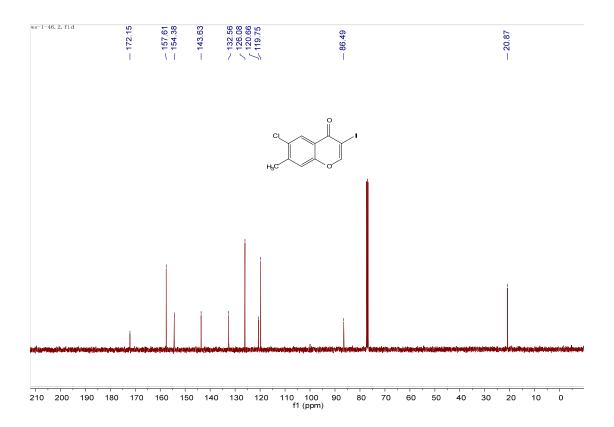
¹H NMR spectrum of 2j



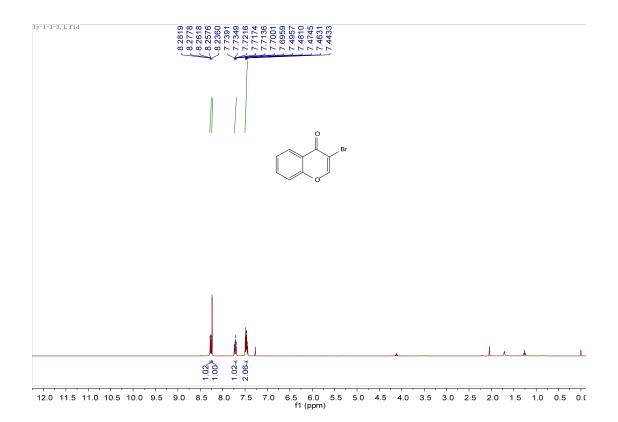
¹³C NMR spectrum of 2j



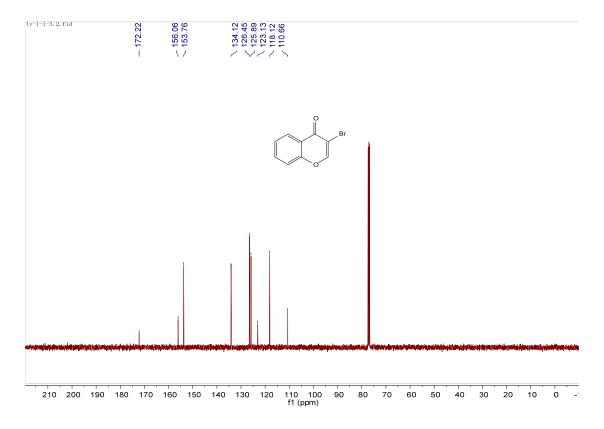
¹H NMR spectrum of 2k



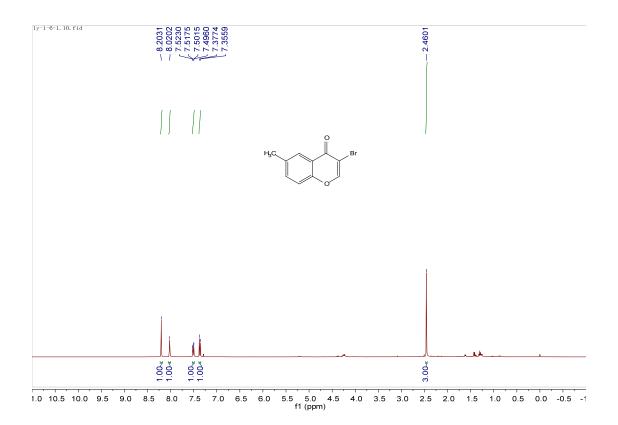
¹³C NMR spectrum of 2k



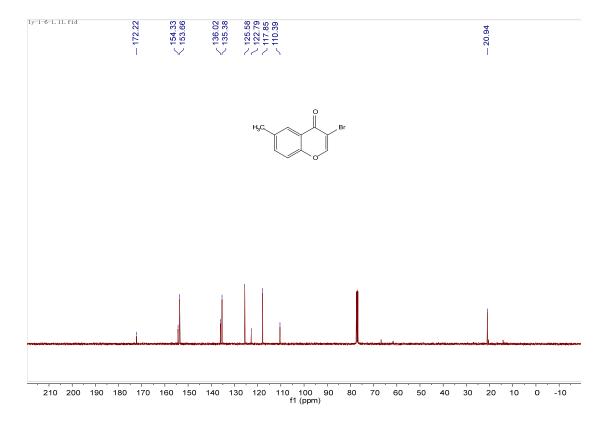
¹H NMR spectrum of 21



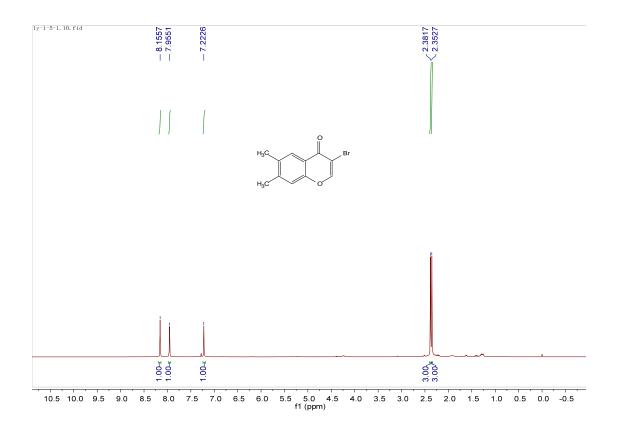
¹³C NMR spectrum of 21



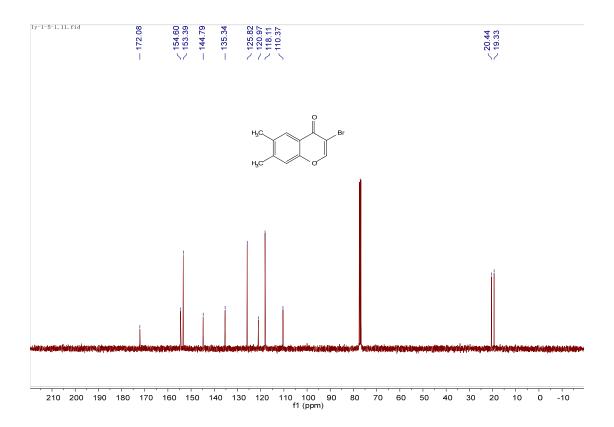
¹H NMR spectrum of 2m



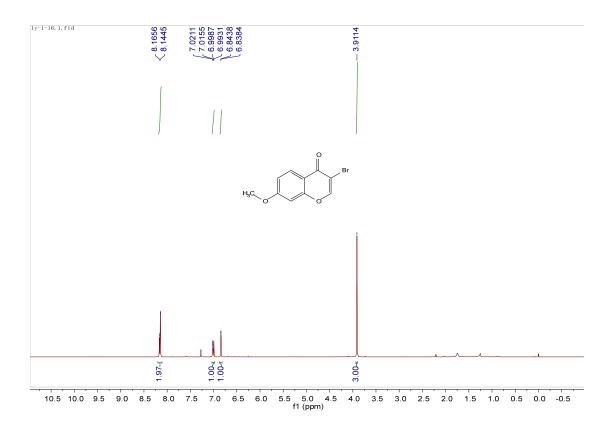
¹³C NMR spectrum of 2m



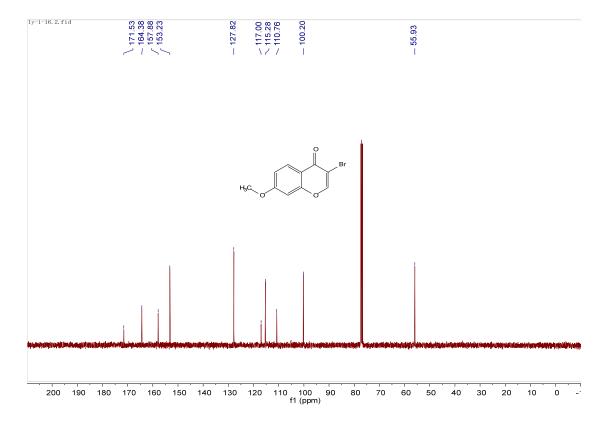
¹H NMR spectrum of 2n



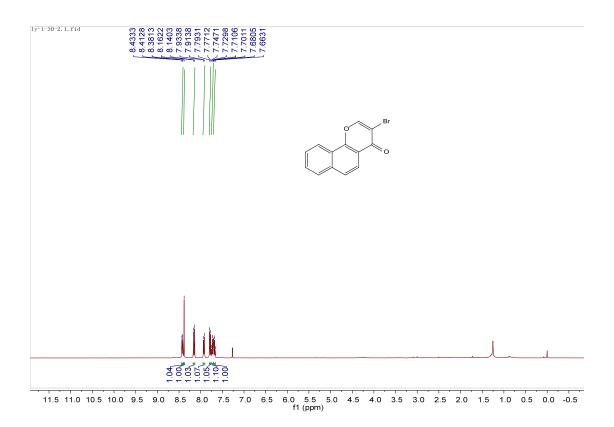
¹³C NMR spectrum of 2n



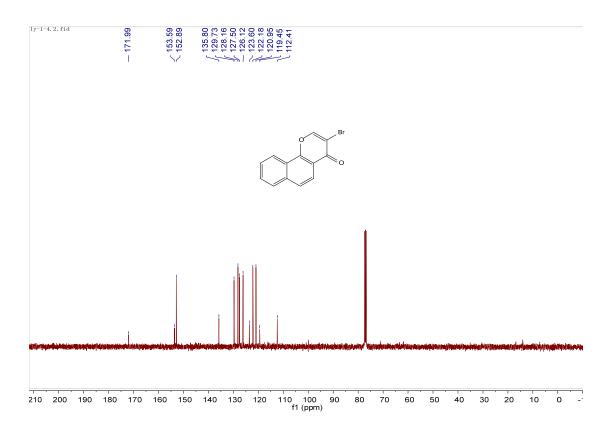
¹H NMR spectrum of 20



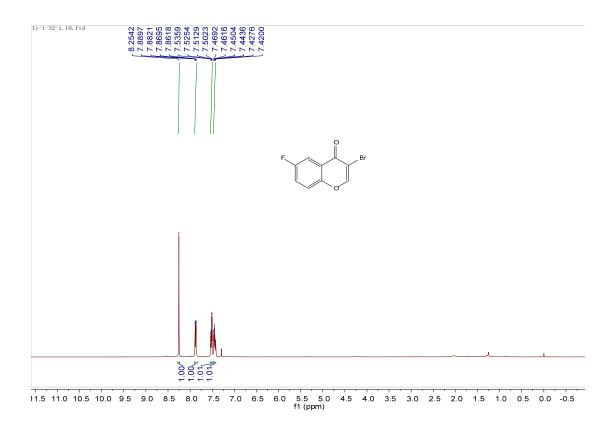
¹³C NMR spectrum of 20



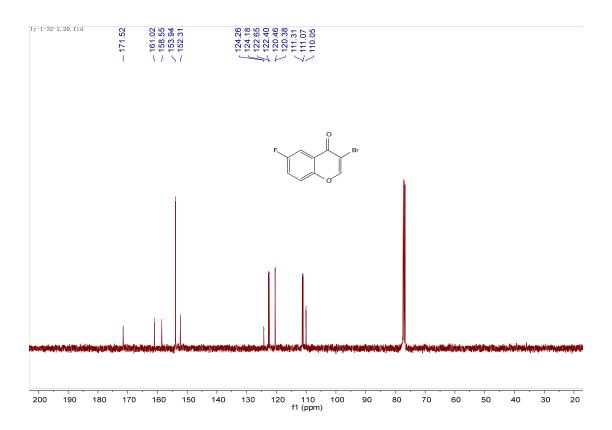
¹H NMR spectrum of 2p



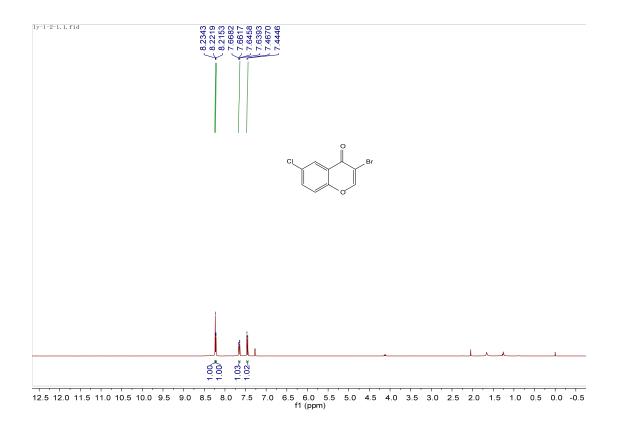
¹³C NMR spectrum of 2p



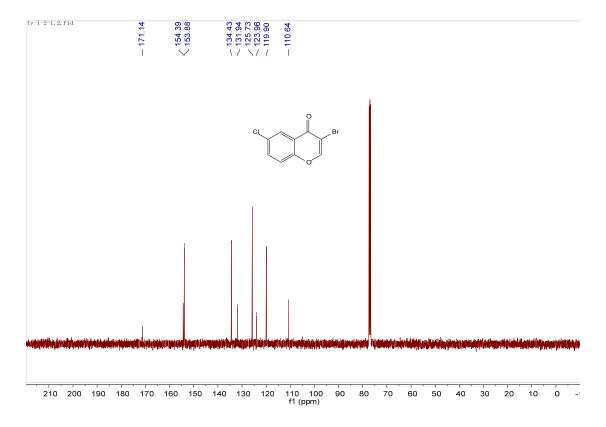
¹H NMR spectrum of 2q



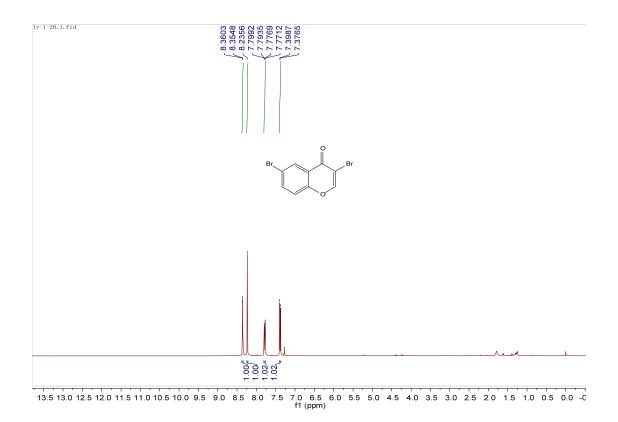
¹³C NMR spectrum of 2q



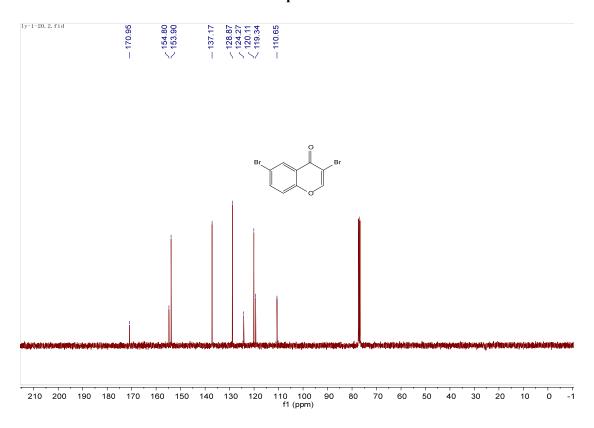
¹H NMR spectrum of 2r



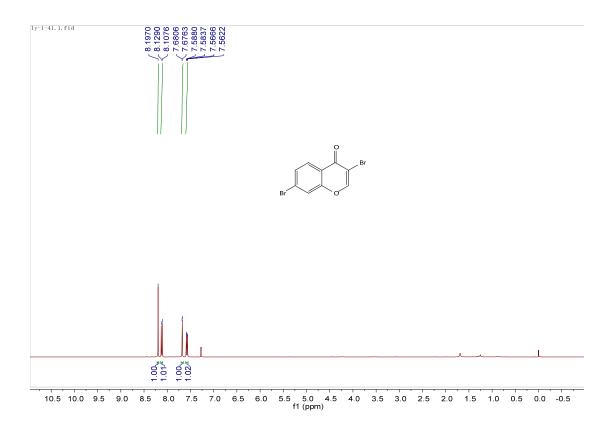
¹³C NMR spectrum of 2r



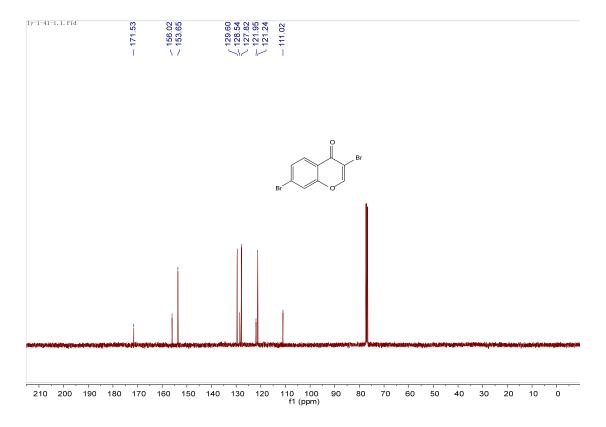
¹H NMR spectrum of 2s



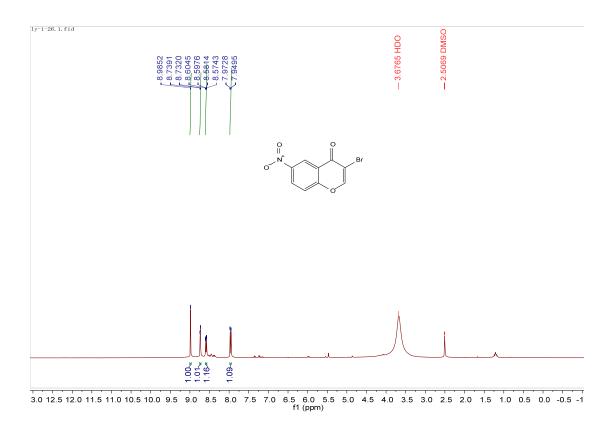
¹³C NMR spectrum of 2s



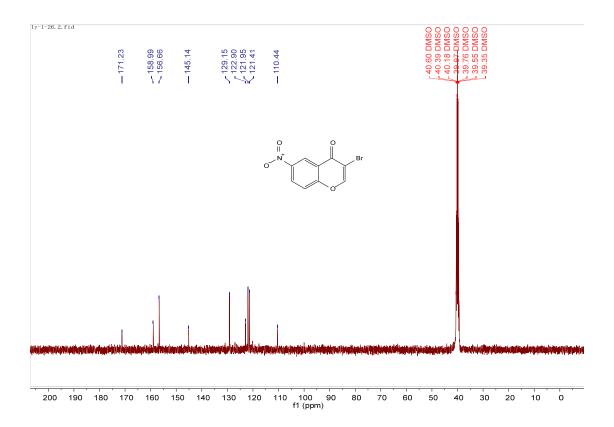
¹H NMR spectrum of 2t



¹³C NMR spectrum of 2t



¹H NMR spectrum of 2u



¹³C NMR spectrum of 2u