## Electronic Supplementary Information for

## Synthesis of azepino[4,5-*b*]indolones via an intermolecular radical oxidative substitution of *N*-Boc tryptamine

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## General

<sup>1</sup>H NMR spectra were recorded on Varian Gemini-200 MHz and Eclipse 300 MHz JEOL spectrometers in deuterated chloroform (CDCl<sub>3</sub>) solutions with internal standard TMS (0 ppm) or in deuterated dimethyl sulfoxide (DMSO-d<sub>6</sub>), and the chemical shifts were reported in parts per million (δ/ppm). <sup>13</sup>C NMR spectra were recorded at 50 MHz and 75 MHz on the same instruments. Assignments of <sup>13</sup>C spectra were performed by DEPT experiments. IR spectra were collected on a FT-IR Tensor 27 Bruker spectrometer. Mass spectra were recorded on a JEOL JEM-AX505HA spectrometer by electronic impact (EI) of lower resolution at 70 eV. Elemental analyses were determinated on a CE-440 Elemental analyzer (Exeter analytical, INC). The X-ray crystallography was carried out on a Bruker Smart Apex CCD diffractometer. Flash column chromatography was carried out with silica gel 60 (230-400 mesh ASTM) from Macherey-Nagel GmbH & Co.



<sup>1</sup>H and <sup>13</sup>C NMR Spectrum of **5** (300 and 75 MHz, CDCl<sub>3</sub>)  $\delta$ (ppm)







<sup>1</sup>H and <sup>13</sup>C NMR Spectrum of **16** (300 and 75 MHz, CDCl<sub>3</sub>)  $\delta$ (ppm)



<sup>1</sup>H and <sup>13</sup>C NMR Spectrum of **17**, mixture of diastereomers (300 and 75 MHz, CDCl<sub>3</sub>)  $\delta$ (ppm)



























<sup>1</sup>H and <sup>13</sup>C NMR Spectrum of **26**, mixture of diastereomers (300 and 75 MHz, DMSO-d<sub>6</sub>)  $\delta$ (ppm)































S23







