# Supplementary Information

# Two 5-methoxyindole carboxylic acid-derived hydrazones of neuropharmacological interest: synthesis, crystal structure, and chemiluminescent study of radical scavenging properties

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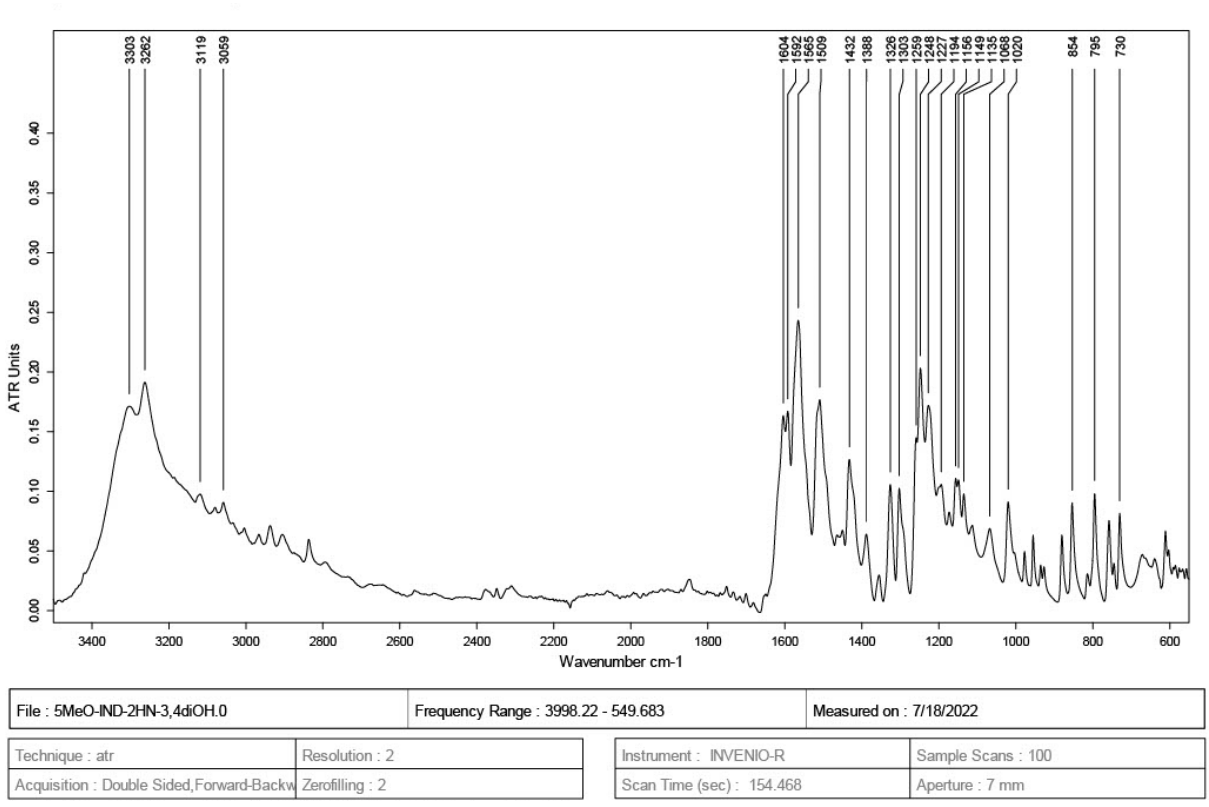


**Figure S1.** ATR-FTIR spectrum of compound **3a**

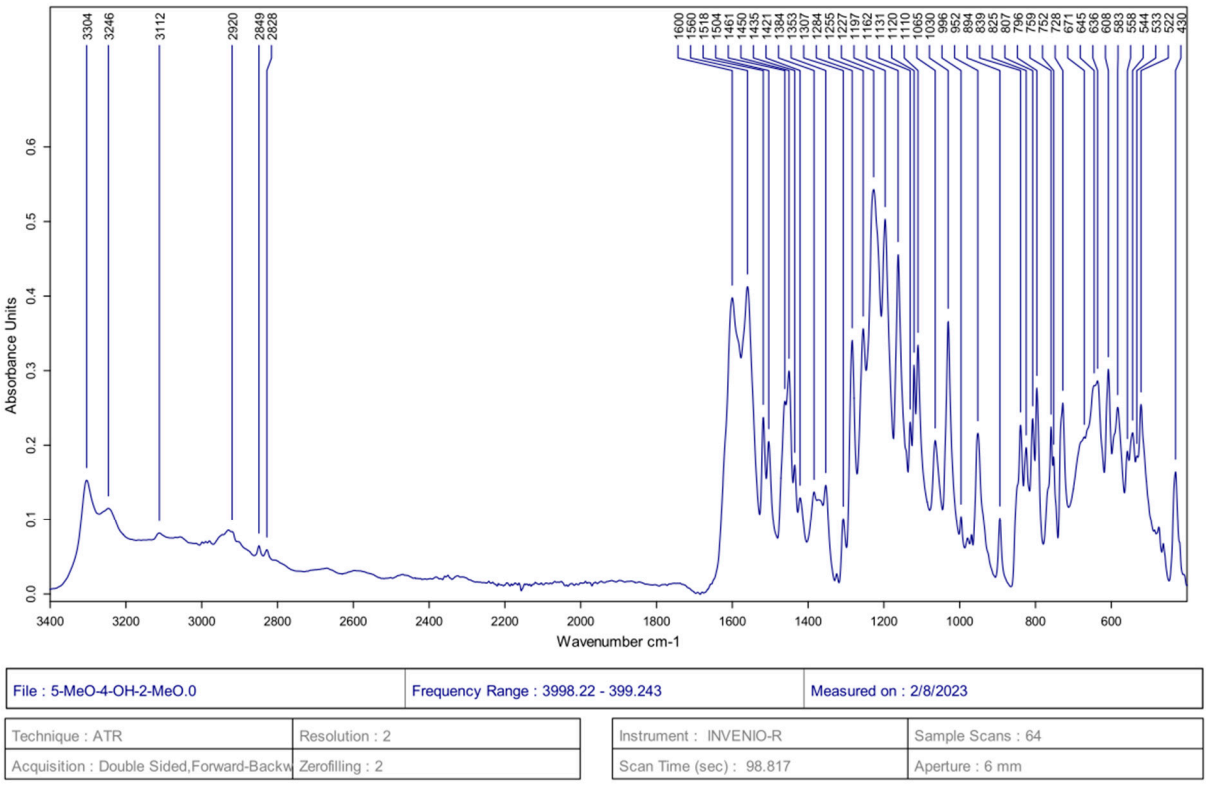


**Figure S2a.** ATR-FTIR spectrum of compound **3b**

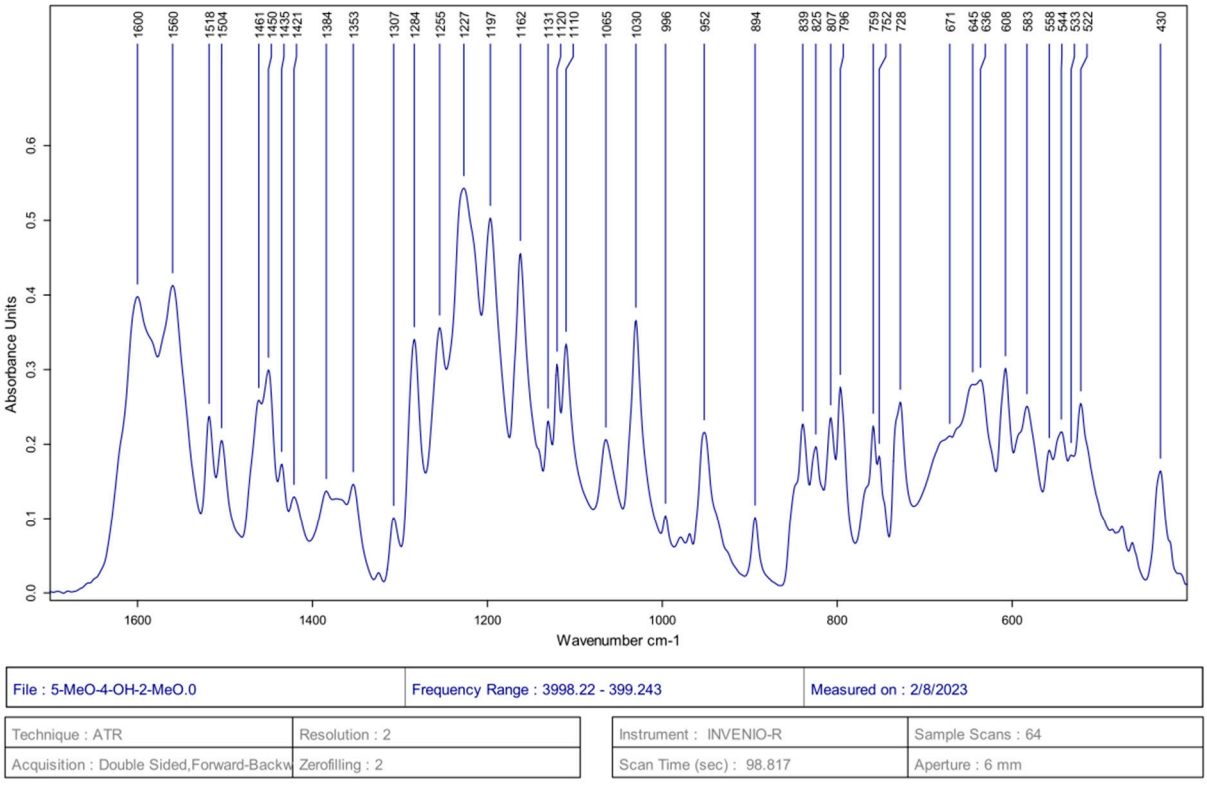


**Figure S2b.** ATR-FTIR spectrum of compound **3b**

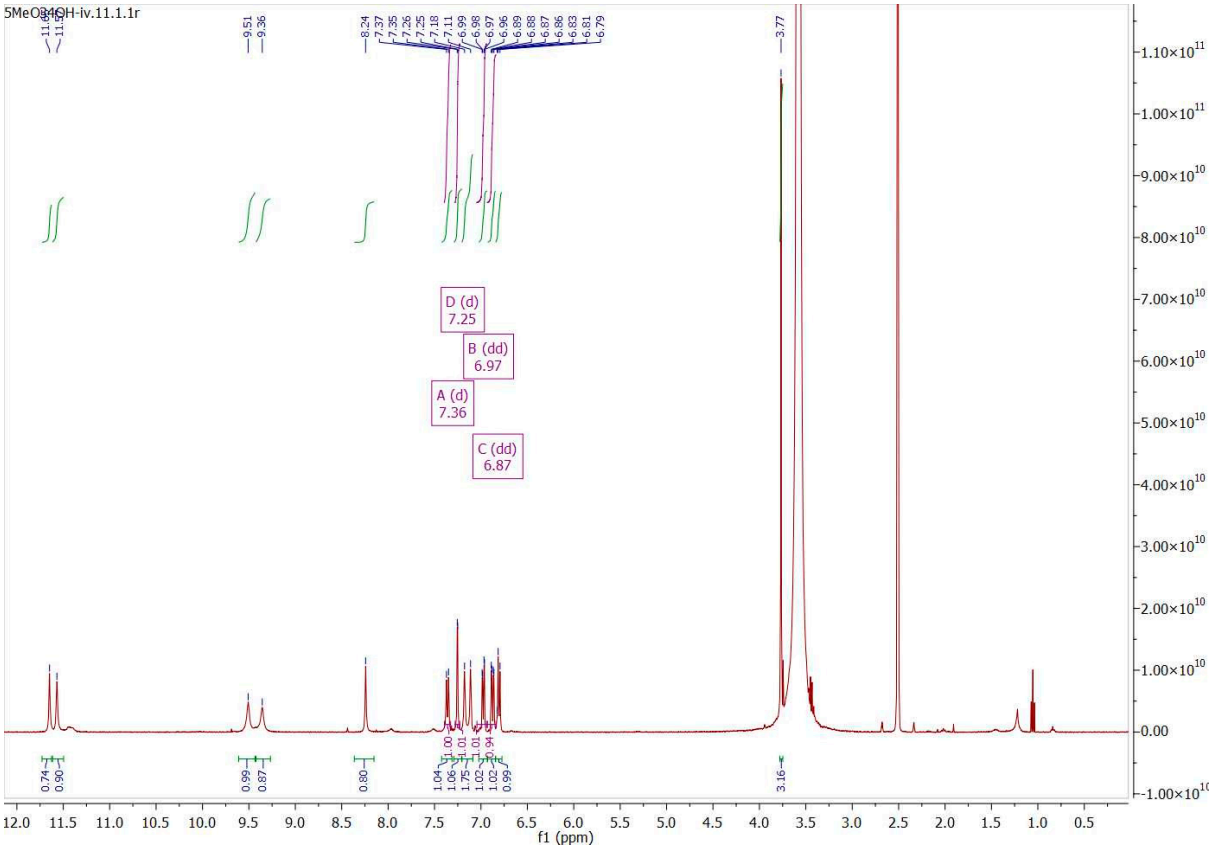


**Figure S3a**. [1]HNMR spectrum of compound **3a** in DMSO-$d_6$

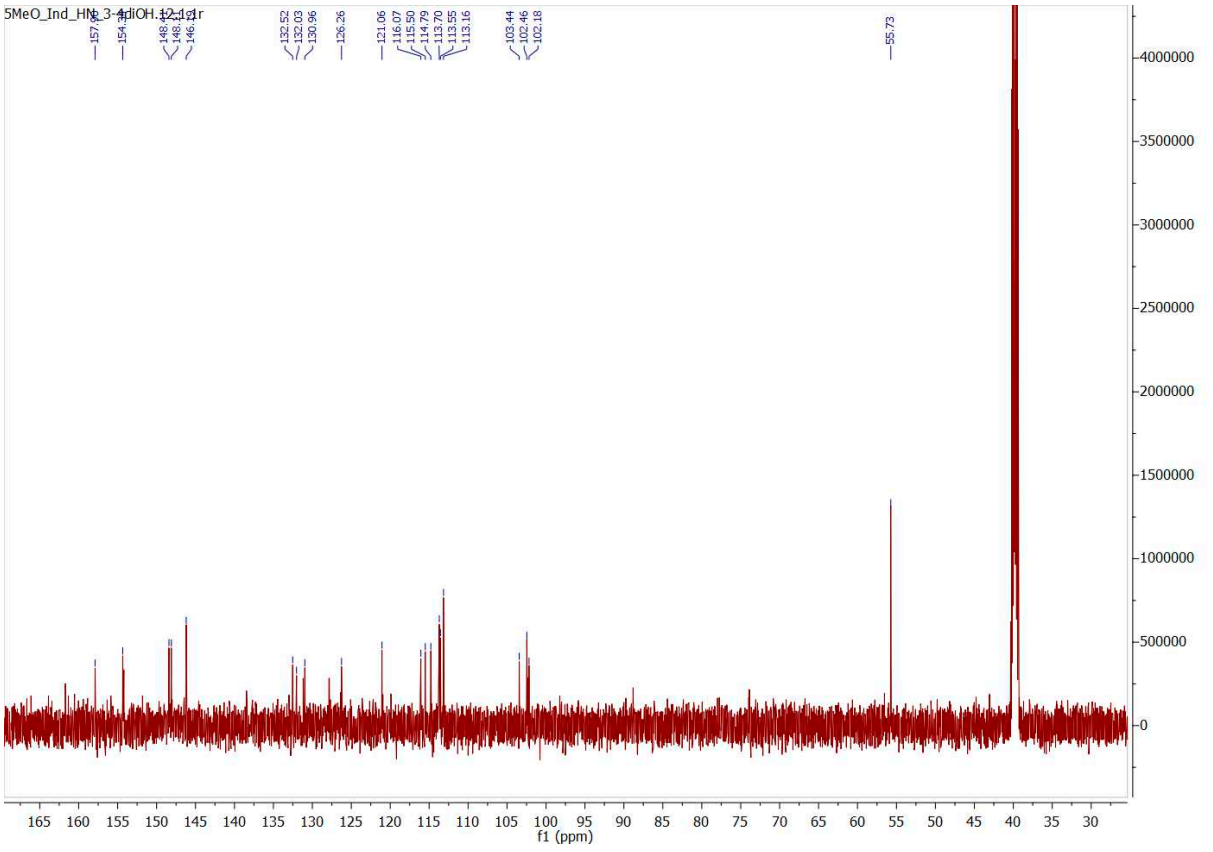


**Figure S3b**. [13]CNMR spectrum of compound **3a** in DMSO-$d_6$

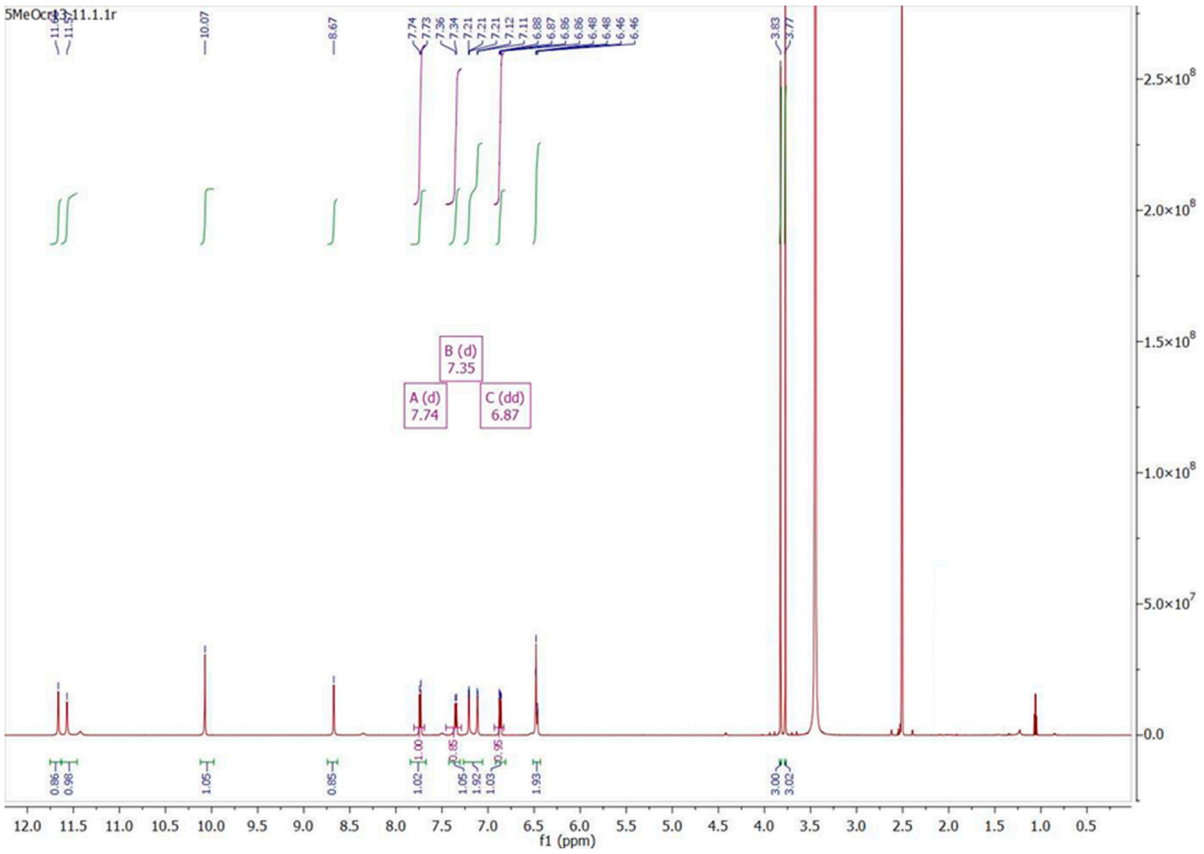


**Figure S4a.** [1]HNMR spectrum of compound **3b** in DMSO-$d_6$

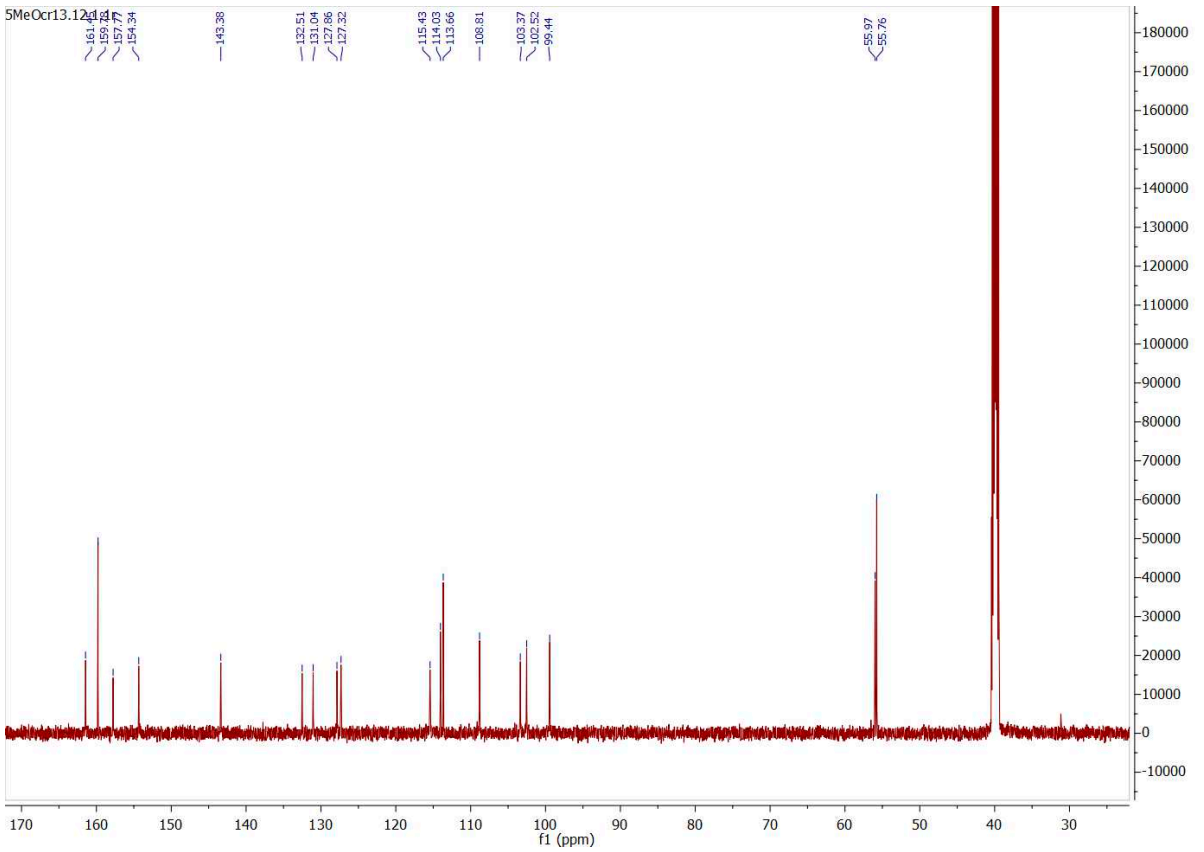


**Figure S4b.** [13]C-NMR spectrum of compound **3b** in DMSO-$d_6$

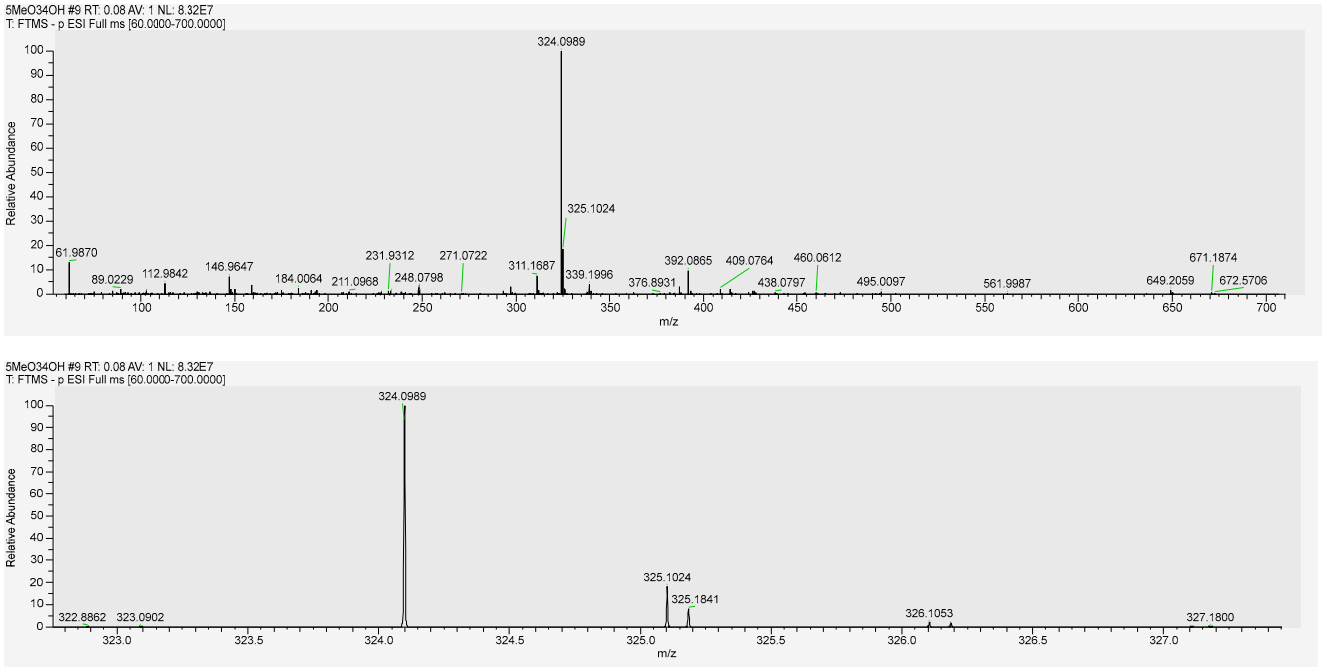


**Figure S5a.** HRMS spectrum of **3a**

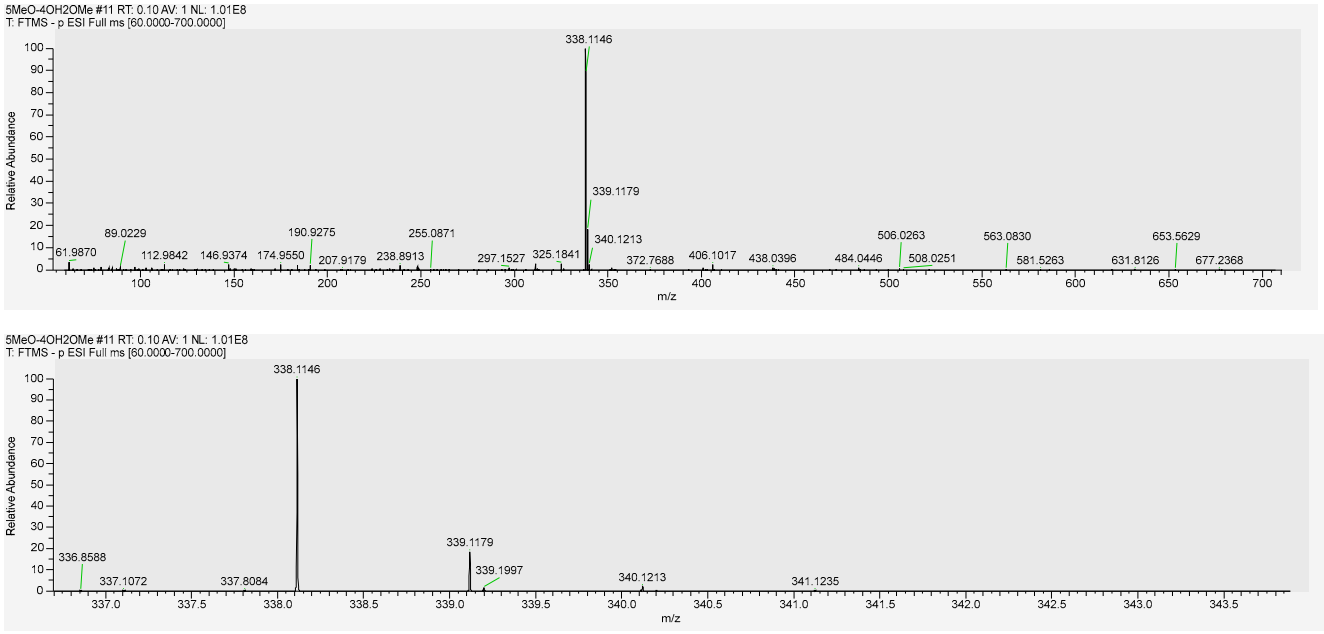


**Figure S5b.** HRMS spectrum of **3b**

**Table S1.** Most important crystallographic parameters for the crystal structures of **3a** and **3b**

| Compound | **3a** | **3b** |
|---|---|---|
| Empirical formula | $C_{17}H_{15}N_3O_4$ | $C_{18}H_{17}N_3O_4$ |
| Formula weight | 325.32 | 339.34 |
| Temperature/K | 290 | 290 |
| Crystal system | Monoclinic | Triclinic |
| Space group | $P2_1/c$ | $P$-1 |
| a/Å | 11.2319(3) | 6.6305(17) |
| b/Å | 8.6743(3) | 11.155(3) |
| c/Å | 15.4783(5) | 23.660(5) |
| $\alpha$/° | 90 | 83.663(8) |
| $\beta$/° | 96.478(2) | 82.762(8) |
| $\gamma$/° | 90 | 89.536(9) |
| Volume/Å$^3$ | 1498.40(8) | 1725.3(7) |
| Z | 4 | 4 |
| $\varrho_{calc}$g/cm$^3$ | 1.442 | 1.306 |
| $\mu$/mm$^{-1}$ | 0.105 | 0.094 |
| F(000) | 680.0 | 712.0 |
| Crystal size/mm$^3$ | 0.3×0.3×0.2 | 0.25×0.2×0.2 |
| Radiation | Mo$K\alpha$<br>$\lambda$ = 0.71073 | Mo$K\alpha$<br>$\lambda$ = 0.71073 |
| 2$\Theta$ range for data collection/° | 5.298 to 52.792 | 4.238 to 52.044 |
| Reflections collected/independent | 52591/3059 | 80072/6786 |
| $R_{int}/R_{sigma}$ | 0.1290/0.0323 | 0.0980/0.0410 |
| Data/restraints/parameters | 3059/0/235 | 6786/0/480 |
| Goodness-of-fit on $F^2$ | 1.036 | 1.041 |
| Final $R$ indexes [$I$>=2$\sigma$ ($I$)] | $R_1$ = 0.0446,<br>$wR_2$ = 0.1041 | $R_1$ = 0.0476,<br>$wR_2$ = 0.1049 |
| Final $R$ indexes [all data] | $R_1$ = 0.0669,<br>$wR_2$ = 0.1175 | $R_1$ = 0.0805,<br>$wR_2$ = 0.1219 |
| Largest diff. peak/hole / e Å$^{-3}$ | 0.23/-0.18 | 0.25/-0.19 |
| CCDC | 2342743 | 2342744 |

**Table S2.** Bond lengths for the crystal structure of **3a**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| O10 | C6 | 1.391(2) | C4 | C5 | 1.410(3) |
| O10 | C11 | 1.390(3) | C4 | C9 | 1.408(3) |
| O13 | C12 | 1.243(2) | C5 | C6 | 1.370(3) |
| O23 | C20 | 1.363(2) | C6 | C7 | 1.402(3) |
| O24 | C19 | 1.366(2) | C7 | C8 | 1.372(3) |
| N1 | C2 | 1.371(2) | C8 | C9 | 1.388(3) |
| N1 | C9 | 1.371(2) | C16 | C17 | 1.452(3) |
| N14 | N15 | 1.380(2) | C17 | C18 | 1.392(3) |
| N14 | C12 | 1.340(3) | C17 | C22 | 1.390(3) |
| N15 | C16 | 1.278(3) | C18 | C19 | 1.374(3) |
| C2 | C3 | 1.365(3) | C19 | C20 | 1.396(3) |
| C2 | C12 | 1.462(3) | C20 | C21 | 1.381(3) |
| C3 | C4 | 1.416(3) | C21 | C22 | 1.384(3) |

**Table S3.** Bond angles for the crystal structure of **3a**

| Atom | Atom | Atom | Angle/˚ | Atom | Atom | Atom | Angle/˚ |
|---|---|---|---|---|---|---|---|
| C11 | O10 | C6 | 117.21(18) | N1 | C9 | C8 | 130.68(18) |
| C9 | N1 | C2 | 108.69(16) | C8 | C9 | C4 | 121.77(17) |
| C12 | N14 | N15 | 119.05(16) | O13 | C12 | N14 | 122.93(17) |
| C16 | N15 | N14 | 115.57(16) | O13 | C12 | C2 | 120.46(18) |
| N1 | C2 | C12 | 117.12(16) | N14 | C12 | C2 | 116.61(16) |
| C3 | C2 | N1 | 109.71(16) | N15 | C16 | C17 | 121.72(18) |
| C3 | C2 | C12 | 133.15(18) | C18 | C17 | C16 | 121.61(18) |
| C2 | C3 | C4 | 106.99(17) | C22 | C17 | C16 | 120.04(17) |
| C5 | C4 | C3 | 133.87(19) | C22 | C17 | C18 | 118.35(17) |
| C9 | C4 | C3 | 107.05(16) | C19 | C18 | C17 | 121.11(18) |
| C9 | C4 | C5 | 119.07(18) | O24 | C19 | C18 | 117.45(17) |
| C6 | C5 | C4 | 118.55(19) | O24 | C19 | C20 | 122.45(17) |
| O10 | C6 | C7 | 121.73(19) | C18 | C19 | C20 | 120.10(17) |
| C5 | C6 | O10 | 116.85(19) | O23 | C20 | C19 | 123.36(17) |
| C5 | C6 | C7 | 121.41(18) | O23 | C20 | C21 | 117.43(18) |
| C8 | C7 | C6 | 121.07(19) | C21 | C20 | C19 | 119.20(17) |
| C7 | C8 | C9 | 118.1(2) | C20 | C21 | C22 | 120.40(19) |

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| N1 | C9 | C4 | 107.55(16) | C21 | C22 | C17 | 120.76(18) |

**Table S4.** Bond lengths for the crystal structure of **3b**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| O131 | C121 | 1.242(2) | C171 | C181 | 1.390(3) |
| O132 | C122 | 1.248(2) | C201 | C211 | 1.383(3) |
| O231 | C201 | 1.370(2) | C201 | C191 | 1.383(3) |
| O232 | C202 | 1.367(2) | C211 | C221 | 1.382(3) |
| N141 | N151 | 1.385(2) | C21 | C31 | 1.370(3) |
| N141 | C121 | 1.342(2) | C22 | C122 | 1.459(3) |
| N11 | C91 | 1.372(2) | C22 | C32 | 1.380(3) |
| N11 | C21 | 1.374(2) | C41 | C31 | 1.417(3) |
| O241 | C221 | 1.365(2) | C41 | C51 | 1.400(3) |
| O241 | C251 | 1.410(3) | C32 | C42 | 1.417(3) |
| N151 | C161 | 1.283(2) | C51 | C61 | 1.367(3) |
| N142 | N152 | 1.384(2) | C202 | C212 | 1.378(3) |
| N142 | C122 | 1.339(2) | C202 | C192 | 1.378(3) |
| N12 | C22 | 1.375(2) | C92 | C42 | 1.400(3) |
| N12 | C92 | 1.371(3) | C92 | C82 | 1.396(3) |
| O102 | C62 | 1.381(3) | C81 | C71 | 1.380(3) |
| O102 | C112 | 1.408(3) | C222 | C172 | 1.395(3) |
| N152 | C162 | 1.276(2) | C222 | C212 | 1.384(3) |
| O242 | C222 | 1.368(2) | C172 | C162 | 1.449(3) |
| O242 | C252 | 1.419(3) | C172 | C182 | 1.390(3) |
| C121 | C21 | 1.460(3) | C42 | C52 | 1.413(3) |
| O101 | C61 | 1.373(3) | C181 | C191 | 1.376(3) |
| O101 | C111 | 1.401(3) | C61 | C71 | 1.405(3) |
| C91 | C41 | 1.408(3) | C52 | C62 | 1.367(3) |
| C91 | C81 | 1.389(3) | C82 | C72 | 1.366(3) |
| C171 | C161 | 1.456(3) | C182 | C192 | 1.375(3) |
| C171 | C221 | 1.401(3) | C62 | C72 | 1.407(3) |

**Table S5.** Bond angles for the crystal structure of **3b**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| C121 | N141 | N151 | 119.74(17) | C22 | C32 | C42 | 107.15(17) |
| C91 | N11 | C21 | 108.68(17) | C61 | C51 | C41 | 119.0(2) |
| C221 | O241 | C251 | 119.70(17) | O241 | C221 | C171 | 114.46(17) |
| C161 | N151 | N141 | 113.42(16) | O241 | C221 | C211 | 124.35(18) |
| C122 | N142 | N152 | 119.03(17) | C211 | C221 | C171 | 121.18(18) |
| C92 | N12 | C22 | 108.72(17) | O232 | C202 | C212 | 121.21(18) |
| C62 | O102 | C112 | 118.2(2) | O232 | C202 | C192 | 118.36(18) |
| C162 | N152 | N142 | 114.73(17) | C212 | C202 | C192 | 120.42(19) |
| C222 | O242 | C252 | 118.21(18) | N12 | C92 | C42 | 108.22(17) |
| O131 | C121 | N141 | 122.26(18) | N12 | C92 | C82 | 129.8(2) |
| O131 | C121 | C21 | 121.82(17) | C82 | C92 | C42 | 121.9(2) |
| N141 | C121 | C21 | 115.85(17) | C71 | C81 | C91 | 117.7(2) |
| C61 | O101 | C111 | 119.3(2) | O242 | C222 | C172 | 115.66(18) |
| N11 | C91 | C41 | 108.03(16) | O242 | C222 | C212 | 123.44(18) |
| N11 | C91 | C81 | 130.12(19) | C212 | C222 | C172 | 120.88(19) |
| C81 | C91 | C41 | 121.83(19) | C222 | C172 | C162 | 120.05(19) |
| C221 | C171 | C161 | 118.78(18) | C182 | C172 | C222 | 117.46(18) |
| C181 | C171 | C161 | 122.81(18) | C182 | C172 | C162 | 122.48(18) |
| C181 | C171 | C221 | 118.08(18) | C92 | C42 | C32 | 106.93(18) |
| N151 | C161 | C171 | 122.06(18) | C92 | C42 | C52 | 119.50(19) |
| O231 | C201 | C211 | 121.70(18) | C52 | C42 | C32 | 133.56(19) |
| O231 | C201 | C191 | 117.25(18) | C191 | C181 | C171 | 121.32(19) |
| C191 | C201 | C211 | 121.05(18) | C202 | C212 | C222 | 119.91(19) |
| C221 | C211 | C201 | 118.99(18) | O101 | C61 | C71 | 123.5(2) |
| N11 | C21 | C121 | 118.69(17) | C51 | C61 | O101 | 115.4(2) |
| C31 | C21 | N11 | 109.20(17) | C51 | C61 | C71 | 121.1(2) |
| C31 | C21 | C121 | 131.95(17) | N152 | C162 | C172 | 120.83(19) |
| N12 | C22 | C122 | 117.39(17) | C181 | C191 | C201 | 119.36(19) |
| N12 | C22 | C32 | 108.97(17) | C81 | C71 | C61 | 121.1(2) |
| C32 | C22 | C122 | 133.58(18) | C62 | C52 | C42 | 118.3(2) |
| C91 | C41 | C31 | 106.56(17) | C72 | C82 | C92 | 117.2(2) |
| C51 | C41 | C91 | 119.23(18) | C192 | C182 | C172 | 122.2(2) |
| C51 | C41 | C31 | 134.19(19) | C182 | C192 | C202 | 119.2(2) |

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| O132 | C122 | N142 | 121.85(18) | O102 | C62 | C72 | 113.9(2) |
| O132 | C122 | C22 | 121.12(17) | C52 | C62 | O102 | 125.0(2) |
| N142 | C122 | C22 | 117.01(17) | C52 | C62 | C72 | 121.0(2) |
| C21 | C31 | C41 | 107.50(17) | C82 | C72 | C62 | 122.0(2) |

**Table S6**. Interaction energies (kJ/mol) of molecule **3a** computed using single-crystal geometries. R is the distance between molecular centroids (mean atomic position) in Å.

| | N | **Symmetry operation** | R | $E_{ele}$ | $E_{pol}$ | $E_{dis}$ | $E_{rep}$ | $E_{tot}$ |
|---|---|---|---|---|---|---|---|---|
| | 0 | -x, y+1/2, -z+1/2 | 10.73 | -103.0 | -27.7 | -23.8 | 114.4 | -79.5 |
| | 0 | x, -y+1/2, z+1/2 | 8.12 | -25.8 | -6.4 | -21.6 | 37.5 | -27.6 |
| | 0 | x, -y+1/2, z+1/2 | 9.92 | -16.2 | -6.4 | -18.3 | 26.7 | -21.4 |
| | 1 | -x, -y, -z | 4.04 | -31.1 | -6.2 | -98.6 | 58.7 | -87.1 |

| D | H | A | R | Electron Density | Interaction Energies (kJ/mol) | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | $E_{ele}$ | $E_{pol}$ | $E_{dis}$ | $E_{rep}$ | $E_{tot}$ |
| N14 | H14 | O24[1] | 8.12 | B3LYP/6-31G(d,p) | -27.3 | -4.7 | -18.8 | 23.2 | -27.6 |
| N1 | H1 | O10[2] | 9.92 | B3LYP/6-31G(d,p) | -17.1 | -4.7 | -15.9 | 16.5 | -21.3 |
| O23 | H23 | O13[3] | 10.73 | B3LYP/6-31G(d,p) | -54.4 | -10.2 | -10.4 | 35.3 | -39.7 |
| O24 | H24 | O13[3] | 10.73 | B3LYP/6-31G(d,p) | -54.4 | -10.2 | -10.4 | 35.3 | -39.7 |
| π | ⋯ | π | 4.04 | B3LYP/6-31G(d,p) | -33.0 | -4.6 | -85.9 | 35.35 | -87.1 |

**Symmetry operations:** [1]+X,1/2-Y,-1/2+Z; [2]+X,3/2-Y,1/2+Z; [3]1-X,-1/2+Y,3/2-Z

**Table S7**. Interaction energies (kJ/mol) of molecules from **3b** computed using single-crystal geometries. R is the distance between molecular centroids (mean atomic position) in Å.

| | N | Symop | R | Electron Density | $E_{ele}$ | $E_{pol}$ | $E_{dis}$ | $E_{rep}$ | $E_{tot}$ |
|---|---|---|---|---|---|---|---|---|---|
| | 1 | - | 9.00 | B3LYP/6-31G(d,p) | -61.2 | -15.0 | -34.3 | 87.2 | -51.8 |
| | 1 | - | 8.79 | B3LYP/6-31G(d,p) | -22.3 | -4.9 | -27.5 | 38.0 | -27.8 |
| | 1 | - | 9.44 | B3LYP/6-31G(d,p) | -56.9 | -14.8 | -34.1 | 64.8 | -60.7 |
| | 1 | -x, -y, -z | 7.90 | B3LYP/6-31G(d,p) | -56.7 | -16.6 | -19.9 | 53.4 | -56.5 |
| | 0 | x, y, z | 6.63 | B3LYP/6-31G(d,p) | -10.1 | -2.3 | -22.5 | 9.2 | -26.3 |
| | 0 | - | 9.70 | B3LYP/6-31G(d,p) | -29.2 | -6.5 | -29.2 | 49.8 | -30.4 |

| D | H | A | R | Electron Density | Interaction Energies (kJ/mol) | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | $E_{ele}$ | $E_{pol}$ | $E_{dis}$ | $E_{rep}$ | $E_{tot}$ |
| O231 | H231 | O132 | 9.00 | B3LYP/6-31G(d,p) | -61.2 | -15.0 | -34.3 | 87.2 | -51.8 |
| N141 | H141 | O232[1] | 8.79 | B3LYP/6-31G(d,p) | -22.3 | -4.9 | -27.5 | 38.0 | -27.8 |
| O232 | H232 | O131[3] | 9.44 | B3LYP/6-31G(d,p) | -56.9 | -14.8 | -34.1 | 64.8 | -60.7 |
| N11 | H11 | O131[6] | 7.90 | B3LYP/6-31G(d,p) | -56.7 | -16.6 | -19.9 | 53.4 | -56.5 |
| N142 | H142 | O231[2] | 6.63 | B3LYP/6-31G(d,p) | -10.1 | -2.3 | -22.5 | 9.2 | -26.3 |
| N12 | H12 | O132[5] | 9.70 | B3LYP/6-31G(d,p) | -29.2 | -6.5 | -29.2 | 49.8 | -30.4 |

**Symmetry operations**: [1]+X,1+Y,+Z; [2]1+X,+Y,+Z; [3]1+X,-1+Y,+Z; [4]1-X,2-Y,1-Z; [5]1-X,1-Y,1-Z; [6]-X,2-Y,2-Z

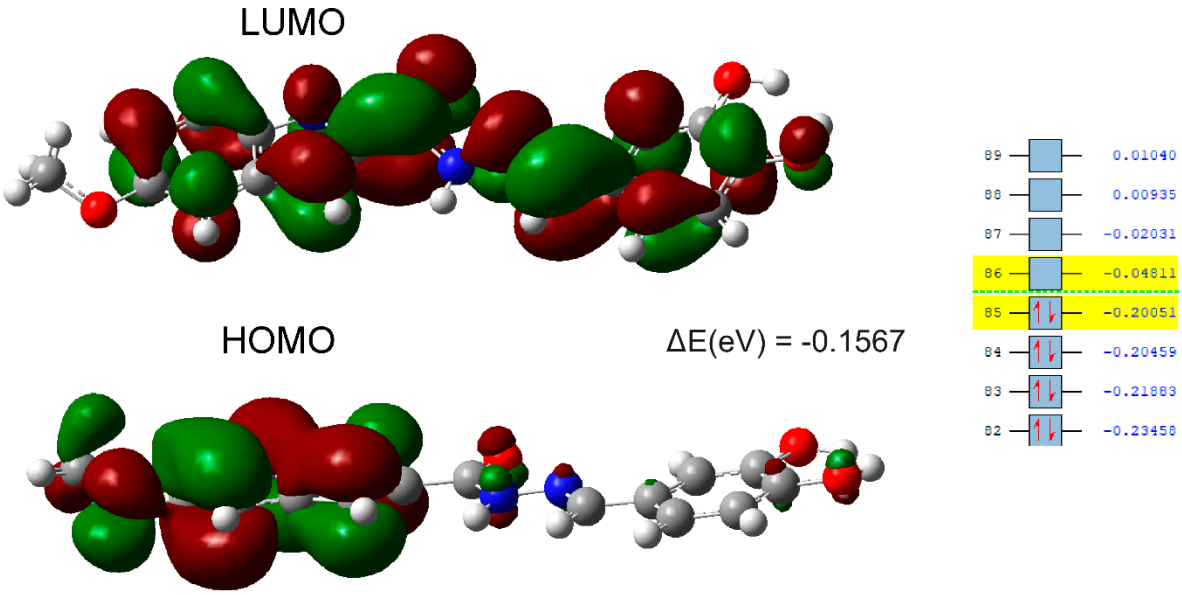


**Figure S6.** HOMO/LUMO representation for **3a**.