Supporting Information

Ruthenium(II)-Catalyzed *Ortho*-C–H Alkylation of Naphthylamines with Diazo Compounds for Synthesis of 2,2-Disubstituted π-Extended 3-Oxindoles in Water

Xiaogang Wang,^{a,+} Jin Zhang,^{a,*,+} Yuan He,^b Di Chen,^{a,c} Chao Wang,^a Fangzhou Yang,^a Weitao Wang,^a Yangmin Ma,^{a,*} and Michal Szostak^{a,d,*}

^a College of Chemistry and Chemical Engineering, Shaanxi Key Laboratory of Chemical Additives for Industry, Shaanxi University of Science and Technology, Xi'an 710021, China

^b Technology Center, China Tobacco Shaanxi Industrial Co., Ltd., Baoji 721013, Shaanxi, China

^c School of Food and Biological Engineering, Shaanxi University of Science and Technology, Xi'an 710021, China.

^d Department of Chemistry, Rutgers University, 73 Warren Street, Newark, New Jersey 07102, United States

⁺ These authors contributed equally to this work.

E-mail: zhangjin@sust.edu.cn, mym63@sina.com, michal.szostak@rutgers.edu

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I. General information

Unless otherwise noted, all reagents were obtained commercially and used without further purification. Unless otherwise specified, all other reagents were purchased from Aldrich, Fisher or TCI and used without further purification. The NMR spectra were recorded on a Bruker MERCURY plus-400 (400 MHz, ¹H; 101 MHz, ¹³C) spectrometer with chemical shifts reported in ppm relative to the residual deuterated solvent and the internal standard tetramethylsilane. Data for ¹H NMR are recorded as follows: chemical shift (δ , ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, br = broad singlet, coupling constant(s) in Hz, integration). Infrared spectra (IR) data were recorded on a TENSOR 27 FT-IR spectrometer and recorded in wave numbers (cm⁻¹). High resolution mass spectra (HRMS) were measured with the Bruker Daltonics solariX 7T Fourier transform ion cyclotron resonance mass spectra were collected on a Thermo Scientific Lumina fluorescence spectrometer. The absolute quantum yields of compounds were acquired on a ZEISS LSM 800 confocal microscope with a 63× lens.

II. 1.0 mmol scale preparation



To a mixture of naphthylamine **1a** (1.0 mmol) and α -diazo β -dicarbonyl **2a** (2.0 mmol, 2.0 eq) in H₂O (2.0 mL), EtOH (30.0 eq), CsOAc (47.7 mg, 25 mol%) and [RuCl₂[*p*-cymene]₂ (30.6 mg, 5 mol%) were added and the reaction was heated by oil bath at 65 °C for 16 h. The reaction mixture was poured into 50 mL of water and extracted with EtOAc (3 x 50 mL). The combined organic layers were dried over MgSO₄. Volatiles were removed under reduced pressure, and residue was purified by chromatography on silica gel to afford the desired compounds **3aa**. Yield 65% (175 mg). Yellow oil. Characterization data are included in the section below.

III. MTT assay and inhibitory activity

PC3 cells, A549 cells and MCF-7 cells were all cultured in Dulbecco's modified Eagle's medium (DMEM) supplemented with 10% fetal bovine serum (FBS). The cells were cultured in a humidified atmosphere of 5% CO₂/95% air at 37 °C. Cells were cultured in 35 mm glass bottom dishes ($\Phi = 20$ mm) for 24 h and used for fluorescent imaging.

After diluting to 5×10^4 cells mL⁻¹ with the complete medium, $100 \,\mu$ L of the cell suspension obtained was added to each well of 96-well culture plates. The subsequent incubation was performed at 37 °C, 5% CO₂ atmosphere for 24 h before the cytotoxicity assessments. Tested samples at preset concentrations were added to each well, and the cells were incubated for 48 h. The MTT solution (100 μ L, 0.5 mg/mL) was added to each well, and the cells were incubated for 48 h. The formazan crystals were dissolved in 150 μ L of DMSO. Cell viability was assessed by measuring the absorbance at a 490 nm wavelength using a Thermo Multiskan FC microplate photometer (Thermo Fisher Scientific). The inhibition was calculated with the formula:

Inhibition % = $(OD_c - OD_t)/OD_c \times 100\%$

where OD_c is the absorbance of negative control and OD_t is the absorbance of tested drug.

The results of inhibitory activity are summarized in Table S1. All cancer cells were exposed for 24 h to increasing concentrations of (**3aa-3ma**) and (**3ab-3ai**), and their survival was determined by the MTT (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide) assay. As shown, **3da**, **3ea**, **3fa** and **3ia** displayed promising growth inhibition in PC3, A549 and MCF-7 cells. In particular, **3ia** showed the most potent activity (IC₅₀ = 22.98 μ M against PC3 cells, IC₅₀ = 21.06 μ M against A549 cells, IC₅₀ = 21.29 μ M against MCF-7 cells). These results demonstrate that π -extended 3-oxo-indole derivatives might represent promising leads for the development of new cytotoxic agents.

onter		$IC_{50} (\mu m)$)	optra		$IC_{50} (\mu m)$)
entry	PC3 A549	MCF-7	entry	PC3	A549	MCF-7	
3aa	158.3	152.1	192.1	3ma	>200	>200	>200
3ba	66.5	91.3	109.2	3ab	155.1	>200	>200
3ca	87.2	89.8	39.9	3ac	>200	>200	>200
3dc	36.2	26.7	33.0	3ad	>200	>200	>200
3ea	33.6	22.9	23.2	3ae	>200	>200	>200
3fa	26.5	25.0	31.8	3af	>200	>200	>200
3ga	104.8	133.8	>200	3ag	>200	159.9	>200
3ha	69.7	72.3	88.3	3ah	48.5	45.5	85.0
3ia	23.0	21.1	21.3	3ai	79.1	80.8	199.6
3ja	118.5	78.8	155.0	docorubicin	3.653	1.933	2.618
3ka	>200	>200	>200				

Table S1 Cytotoxic Activity of 3aa-3ai in Human Cancer Cell Lines

IV. Discussion of fluorescent properties

We observed that these C–H activation products show bright cyan fluorescence in aqueous solutions, which renders them attractive for fluorescent imaging in biological samples. First, the fluorescent characteristics of (**3aa**), (**3da**) and (**3ka**) were investigated in various protic and aprotic solvents (Fig. 3a-b, Fig. S1 and Table S2). The fluorescence spectra and images reveal that these compounds show strong fluorescence with a little red-shift in protic solvents, and weaker fluorescence with a little blue-shift in aprotic polar and nonpolar solvents.

These fluorescence properties are unique in comparison with the common fluorescent dyes suffering from solvatochromic quenching in aqueous solutions. For comparison, the C– H activation substrates (**1b**) and (**1d**) show strong fluorescence in aprotic solvents, but weak fluorescence in protic solvents (Fig. S2). This unusual strong fluorescence of the C–H functionalization products in protic solvents appears to be correlated with the hydrogen bonding ability. Fluorescence can be enhanced by hydrogen bonding between the protic solvent (HB donor) and the C3-carbonyl group of the products (HB acceptor).^{28b} The high quantum yield in PBS buffer (74.4%, **3aa**; 59.5%, **3da**; 48.7%, **3ka**) and high concentration quenching tolerance (Fig. S3) indicate that these compounds are well-suited for fluorescent imaging.



Figure S1. Spectral properties of compounds **3aa**, **3da** and **3ka**. Absorption and fluorescence spectra of **3aa** (a) and **3da** (b) in PBS buffer. Fluorescence spectra of **3aa** (c) and **3ka** (d) in different solvents.

	$\epsilon (M^{-1}cm^{-1})$	λ_{abs} (nm)	λ_{em} (nm)	$\Phi_{ m f}$	Stokes shift (nm)
3aa	4300	408	474	0.744	66
3da	5100	417	482	0.595	65
3ka	1300	408	475	0.487	67

Table S2. Spectral properties of compounds 3aa, 3da and 3ka



Figure S2. Fluorescence spectra of the synthetic substrates 1b (a) and 1d (b) in different solvents.



Figure S3. Fluorescent intensities of compounds 3aa (a), 3da (b) and 3ka (c) with different concentrations.



Figure S4. Fluorescent imaging of A549 cells. Compounds 3aa (a), 3ka (b) and blank control (c).

V. X-ray Crystallographic data of 3ai

Sample preparation and crystal measurement

Single crystals suitable for X-ray diffraction experiment were obtained by slow evaporation of EA/n-hexane (1:10, V/V) solution containing the corresponding compounds. The data was collected at 293(2) K with Rigaku XtaLAB Synergy four-circle diffractometer under Mo K α radiation (λ = 0.71073 Å) for **3ai**, with the CrysAlisPro software (version 1.171.39.34b) for data reduction and analysis. The structures were solved by direct methods and refined by full-matrix least-squares method on F2 using SHELX algorithms in Olex2. All non-hydrogen atoms in the structure were refined with anisotropic models. All hydrogen atoms were generated geometrically.

CCDC 1965995 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre.



Figure S5. Crystal structure of 3ai (50% ellipsoids).

Tab	le S3.	Crystal	Data a	and	Structure	Refinement	Summaries	for	3ai	i
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Compound	3ai	
Empirical formula	$C_{21}H_{17}NO_3$	
Formula weight	331.36	
Crystal size/mm ³	$0.15 \times 0.2 \times 0.25$	
Crystal system	monoclinic	
Space group	P 21/n	
a/Å	11.3288(5)	

b/Å	12.0111(6)
c/Å	12.6355(6)
a/°	90
β/°	101.536(3)
$\gamma/^{\circ}$	90
Volume/Å ³	1684.60(14)
Z	4
D_c/g cm ⁻³	1.306
μ/mm^{-1}	0.088
F(000)	696.0
R(int)	0.206
Temperature/K	273
Unique reflns	4165
$I > 2\sigma(I)$	1872
$R\left[I > 2\sigma(I)\right]$	$R_1 = 0.0907$
	$wR_2 = 0.2359$
Gof	1.045
CCDC number	1965995

VI. Synthesis of compounds 2 and characterization data

All compounds **2** have been previously described in literature and prepared by the method reported previously (Ye, T.; McKervey, M. A. *Chem. Rev.* **1994**, *94*, 1091; Ford, A.; Miel, H.; Ring, A.; Slattery, C. N.; Maguire, A. R.; McKervey, M. A. *Chem. Rev.* **2015**, *115*, 9981).



1,3-dicarbonyl compounds (3.0 mmol) was dissolved in dichloromethane (10 mL) at room temperature followed by addition of trimethylamine (4.2 mmol, 1.4 equiv). the resulting solution of 4-methylbenzenesulfonyl azide (3.6 mmol, 1.2 equiv.) was added in one portion, and the suspension was stirred at room temperature for 4 h. Solvent was then removed under reduced pressure. Purification by column chromatography on silica gel gave diazo compounds 2a - 2i. ¹H NMR and ¹³C NMR data are given for all compounds 2 in the section below for characterization purposes.

Ethyl 2-diazo-3-oxobutanoate (2a). Yellow oil (407 mg, 87% yield, purified by silica gel chromatography using PE/EA 10:1). ¹H NMR (400 MHz, CDCl₃) δ = 4.31 (q, *J* = 7.1 Hz, 2H), 2.49 (s, 3H), 1.34 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ = 189.7, 160.9, 60.9, 27.7, 13.8.

Methyl 2-diazo-3-oxobutanoate (2b). Yellow oil (383 mg, 90% yield, purified by silica gel chromatography using PE/EA 10:1). ¹H NMR (400 MHz, CDCl₃) δ = 3.85 (s, 3H), 2.49 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ = 189.6, 161.3, 51.7, 27.7.

$$Me \underbrace{\bigcup_{N_2}^{O} CO_2 Bn}_{N_2}$$

Benzyl 2-diazo-3-oxobutanoate (2c). Yellow oil (582 mg, 89% yield, purified by silica gel chromatography using PE/EA 10:1). ¹H NMR (400 MHz, CDCl₃) δ = 7.41 – 7.37 (m, 5H), 5.29 (s, 2H), 2.50 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ = 189.5, 160.8, 134.7, 128.2, 128.2, 127.9, 75.9, 66.5, 27.8.

$$Me \underbrace{\bigcup_{N_2}^{O} CO_2 ^t Bu}_{N_2}$$

tert-Butyl 2-diazo-3-oxobutanoate (2d). Yellow oil (497 mg, 90% yield, purified by silica gel chromatography using PE/EA 10:1). ¹H NMR (400 MHz, CDCl₃) δ = 2.46 (s, 3H), 1.54 (s, 9H); ¹³C NMR (101 MHz, CDCl₃) δ = 190.1, 160.1, 82.7, 27.8, 27.7.

Ethyl 2-diazo-3-oxopentanoate (2e). Yellow oil (454 mg, 89% yield, purified by silica gel chromatography using PE/EA 10:1). ¹H NMR (400 MHz, CDCl₃) δ = 4.29 (q, *J* = 7.1 Hz, 2H), 2.86 (q, *J* = 7.4 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H), 1.13 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ = 193.0, 160.9, 60.8, 33.2, 13.8, 7.7.



Ethyl 2-diazo-3-oxohexanoate (2f). Yellow oil (497 mg, 90% yield, purified by silica gel chromatography using PE/EA 10:1). ¹H NMR (400 MHz, CDCl₃) δ = 4.30 (q, *J* = 7.1 Hz, 2H), 2.83 (t, *J* = 7.4 Hz, 2H), 1.67 (q, *J* = 7.4 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H), 0.97 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ = 192.5, 160.9, 60.9, 41.6, 17.4, 13.9, 13.3.

$$Pr$$
 N_2 O_2Et

Ethyl 2-diazo-4-methyl-3-oxopentanoate (2g). Yellow oil (502 mg, 91% yield, purified by silica gel chromatography using PE/EA 10:1). ¹H NMR (400 MHz, CDCl₃) δ = 4.31 (q, *J* = 7.1 Hz, 2H), 3.59 (hept, *J* = 6.8 Hz, 1H), 1.34 (t, *J* = 7.2 Hz, 3H), 1.13 (d, *J* = 7.0 Hz, 6H); ¹³C NMR (101 MHz, CDCl₃) δ = 196.1, 160.4, 74.4, 60.6, 36.1, 17.8, 13.6.

$$Bu$$
 V CO_2Et N_2

Ethyl 2-diazo-4,4-dimethyl-3-oxopentanoate (2h). Yellow oil (505 mg, 85% yield, purified by silica gel chromatography using PE/EA 10:1). ¹H NMR (400 MHz, CDCl₃) $\delta = \delta$ 4.27 (q, J = 7.1 Hz, 2H), 1.33 (t, J = 7.2 Hz, 3H), 1.30 (s, 9H); ¹³C NMR (101 MHz, CDCl₃) $\delta = 196.8$, 160.4, 60.8, 43.8, 25.3, 13.8.

$$Ph \longrightarrow CO_2Et$$

Ethyl 2-diazo-3-oxo-3-phenylpropanate (2i). Yellow oil (608 mg, 93% yield, purified by silica gel chromatography using PE/EA 10:1). ¹H NMR (400 MHz, CDCl₃) δ = 7.67 – 7.57 (m, 2H), 7.55 – 7.49 (m, 1H), 7.47 – 7.38 (m, 2H), 4.24 (q, *J* = 7.1 Hz, 2H), 1.25 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ = 186.4, 160.5, 136.6, 131.7, 127.8, 127.4, 61.1, 13.7.

VII. Synthesis of compounds 3 and characterization data

To a mixture of naphthylamine (0.10 mmol) and α -diazo β -dicarbonyl (0.20 mmol, 2.0 eq) in H₂O (2.0 mL), EtOH (30.0 eq), CsOAc (4.8 mg, 25 mol%) and [RuCl₂[*p*-cymene]₂ (3.0 mg, 5 mol%) were added and the reaction was heated by oil bath at 65 °C for 16 h. The reaction mixture was poured into 5 mL of water and extracted with EtOAc (3 x 5 mL). The combined organic layers were dried over MgSO₄. Volatiles were removed under reduced pressure, and residue was purified by chromatography on silica gel to afford the desired compounds **3**.



Ethyl 2-methyl-3-oxo-2,3-dihydro-1H-benzo[*g*]**indole-2-carboxylate** (3aa). Yellow oil (20.4 mg, 76% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO-*d*₆) δ = 8.91 (s, 1H), 8.24 (d, *J* = 8.2 Hz, 1H), 7.92 (d, *J* = 8.1 Hz, 1H), 7.73 (t, *J* = 7.5 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.37 (d, *J* = 8.6 Hz, 1H), 7.21 (d, *J* = 8.6 Hz, 1H), 4.10 (qq, *J* = 7.1, 3.8 Hz, 2H), 1.55 (s, 3H), 1.11 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 195.9, 168.6, 162.5, 137.9, 130.6, 128.6, 126.1, 123.5, 120.6, 120.0, 118.4, 111.3, 70.1, 61.5, 19.9, 13.9; IR (KBr): 2960, 2926, 2854, 1741, 1672, 1624, 1533, 1412, 1250, 1089, 1022, 965, 798, 758 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₆H₁₅NNaO₃⁺ 292.0944; Found 292.0945.



Ethyl 5-bromo-2-methyl-3-oxo-2,3-dihydro-1*H*-benzo[*g*]indole-2-carboxylate (3ba). Yellow oil (20.5 mg, 59% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO-*d*₆) δ = 9.20 (s, 1H), 8.32 (d, *J* = 7.7 Hz, 1H), 8.15 (d, *J* = 8.3 Hz, 1H), 7.92 (t, *J* = 8.3 Hz, 1H), 7.75 (t, *J* = 8.1 Hz, 1H), 7.69 (s, 1H), 4.11 (qq, *J* = 7.2, 3.8 Hz, 2H), 1.56 (s, 3H), 1.11 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 194.5, 168.1, 161.8, 134.9, 132.2, 127.5, 127.2, 124.1, 123.5, 121.6, 111.8, 110.3, 70.3, 61.6, 19.8, 13.9; IR (KBr): 2956, 2924, 2854, 1746, 1637, 1460, 1378, 1260, 1087, 1046, 800, 749 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₆H₁₄BrNNaO₃⁺ 370.0049; Found 370.0051.



Ethyl 5-amino-2-methyl-3-oxo-2,3-dihydro-1*H*-benzo[g]indole-2-carboxylate (3ca). Yellow oil (13.4 mg, 47% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H

NMR (400 MHz, DMSO- d_6) δ = 8.62 (s, 1H), 7.37 (dd, *J* = 8.3, 5.5 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 1H), 7.20 (s, 1H), 6.91 – 6.88 (m, 1H), 5.85 (s, 2H), 4.08 (qt, *J* = 7.1, 3.8 Hz, 2H), 1.51 (s, 3H), 1.10 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO- d_6) δ = 195.2, 169.5, 158.4, 140.9, 124.1, 116.9, 112.2, 108.1, 104.7, 101.1, 69.6, 61.1, 20.4, 13.9; IR (KBr): 2958, 2921, 2852, 1703, 1621, 1491, 1264, 1076, 1049, 957, 745 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺Calcd for C₁₆H₁₆N₂NaO₃⁺ 307.1053; Found 307.1055.



Ethyl 2-methyl-3-oxo-5-phenyl-2,3-dihydro-1*H*-benzo[*g*]indole-2-carboxylate (3da). Yellow oil (20.0 mg, 58% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO-*d*₆) δ = 9.04 (s, 1H), 8.34 (d, *J* = 7.8 Hz, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.75 – 7.70 (m, 1H), 7.70 – 7.65 (m, 1H), 7.53 – 7.48 (m, 2H), 7.44 (d, *J* = 6.7 Hz, 3H), 7.23 (s, 1H), 4.13 (q, *J* = 7.0 Hz, 2H), 1.59 (s, 3H), 1.14 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 195.9, 168.6, 161.9, 139.7, 136.1, 130.9, 130.3, 129.8, 128.5, 127.2, 126.5, 126.2, 123.9, 120.8, 120.1, 110.8, 70.2, 61.5, 20.0, 13.9; IR (KBr): 2956, 2923, 2853, 1741, 1461, 1376, 1260, 1093, 1020, 800 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₂H₁₉NNaO₃⁺ 368.1257; Found 368.1260.



Ethyl 5-(4-methoxyphenyl)-2-methyl-3-oxo-2,3-dihydro-1*H*-benzo[g]indole-2carboxylate (3ea). Yellow oil (24.0 mg, 64%, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO- d_6) $\delta = 8.99$ (s, 1H), 8.33 (d, J = 7.9 Hz, 1H), 7.78 (d, J = 8.1 Hz, 1H), 7.74 – 7.69 (m, 1H), 7.68 – 7.63 (m, 1H), 7.36 (d, J = 8.6 Hz, 2H), 7.19 (s, 1H), 7.06 (d, J = 8.6 Hz, 2H), 4.12 (q, J = 7.0 Hz, 2H), 3.83 (s, 3H), 1.58 (s, 3H), 1.13 (t, J = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO- d_6) $\delta = 196.0$, 168.6, 161.8, 158.5, 136.4, 131.9, 130.8, 130.8, 130.0, 126.6, 126.1, 123.9, 120.9, 119.9, 114.0, 110.9, 70.1, 61.5, 55.1, 20.0, 13.9; IR (KBr): 2958, 2925, 2854, 1740, 1676, 1617, 1470, 1246, 1179, 1032, 967, 835, 769 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺Calcd for C₂₃H₂₁NNaO₄⁺ 398.1363; Found 398.1369.



Ethyl 2-methyl-3-oxo-5-(*p*-tolyl)-2,3-dihydro-1*H*-benzo[*g*]indole-2-carboxylate (3fa). Yellow oil (21.9 mg, 61%, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO- d_6) δ = 8.99 (s, 1H), 8.33 (d, *J* = 7.8 Hz, 1H), 7.78 (d, *J* = 7.9 Hz, 1H), 7.74 – 7.63 (m, 2H), 7.32 (s, 4H), 7.20 (s, 1H), 4.12 (qd, *J* = 7.1, 1.2 Hz, 2H), 2.40 (s, 3H), 1.58 (s, 3H), 1.13 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO- d_6) δ = 195.9, 168.6, 161.9, 136.8, 136.4, 136.2, 130.8, 130.3, 129.6, 129.1, 126.5, 126.1, 123.9, 120.8, 119.9, 110.9, 70.1, 61.5, 20.8, 20.0, 13.9; IR (KBr): 2957, 2923, 2853, 1740, 1678, 1461, 1261, 1095, 1021, 799 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₃H₂₁NNaO₃⁺ 382.1414; Found 382.1418.



Ethyl 5-(4-isopropylphenyl)-2-methyl-3-oxo-2,3-dihydro-1*H*-benzo[g]indole-2carboxylate (3ga). Yellow oil (25.2 mg, 65% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO- d_6) δ = 9.00 (s, 1H), 8.33 (d, *J* = 7.5 Hz, 1H), 7.80 (d, *J* = 8.2 Hz, 1H), 7.75 – 7.70 (m, 1H), 7.69 – 7.64 (m, 1H), 7.37 (d, *J* = 1.7 Hz, 4H), 7.21 (s, 1H), 4.13 (q, *J* = 6.9 Hz, 2H), 2.98 (p, *J* = 6.9 Hz, 1H), 1.58 (s, 3H), 1.28 (d, *J* = 6.9 Hz, 6H), 1.13 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO- d_6) δ = 195.9, 168.6, 161.9, 147.2, 137.1, 136.1, 130.8, 130.2, 129.7, 126.6, 126.4, 126.1, 123.9, 120.8, 119.9, 110.8, 70.1, 61.5, 33.1, 23.9, 20.0, 13.9; IR (KBr): 2956, 2924, 2854, 1745, 1637, 1460, 1377, 1260, 1093, 1019, 799 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₅H₂₅NNaO₃⁺ 410.1727; Found 410.1730.





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carboxylate (**3ha**). Yellow oil (24.5 mg, 61% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO- d_6) δ = 9.03 (s, 1H), 8.33 (d, *J* = 8.0 Hz, 1H), 7.80 (d, *J* = 8.4 Hz, 1H), 7.76 – 7.70 (m, 1H), 7.69 – 7.64 (m, 1H), 7.53 (d, *J* = 8.3 Hz, 2H), 7.38 (d, *J* = 8.2 Hz, 2H), 7.21 (s, 1H), 4.12 (q, *J* = 7.0 Hz, 2H), 1.58 (s, 3H), 1.36 (s, 9H), 1.13 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO- d_6) δ = 195.9, 168.6, 161.9, 149.5, 136.7, 136.2, 130.8, 130.2, 129.4, 126.6, 126.1, 125.3, 123.9, 120.8, 119.9, 110.9, 70.1, 61.5, 34.3, 31.2, 20.0, 13.9; IR (KBr): 2956, 2923, 2853, 1742, 1662, 1526, 1375, 1261, 1093, 1021, 800 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₆H₂₇NNaO₃⁺ 424.1883; Found 424.1889.



Ethyl 5-(3,5-dimethylphenyl)-2-methyl-3-oxo-2,3-dihydro-1*H*-benzo[*g*]indole-2carboxylate (3ia). Yellow oil (23.9 mg, 64% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO-*d*₆) δ = 8.99 (s, 1H), 8.33 (d, *J* = 7.9 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.74 – 7.69 (m, 1H), 7.68 – 7.63 (m, 1H), 7.19 (s, 1H), 7.07 – 7.02 (m, 3H), 4.13 (qd, *J* = 7.0, 2.0 Hz, 2H), 2.34 (s, 6H), 1.58 (s, 3H), 1.13 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 195.9, 168.6, 161.9, 139.6, 137.5, 136.1, 130.8, 130.5, 128.6, 127.5, 126.6, 126.1, 123.9, 120.8, 119.9, 110.8, 70.1, 61.5, 20.9, 20.0, 13.9; IR (KBr): 2956, 2923, 2853, 1740, 1685, 1461, 1375, 1260, 1095, 1022, 800, 749 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₄H₂₃NNaO₃⁺ 396.1570; Found 396.1575.



Ethyl 2-methyl-5-(naphthalen-2-yl)-3-oxo-2,3-dihydro-1*H*-benzo[g]indole-2carboxylate (3ja). Yellow oil (15.4 mg, 39%, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO- d_6) $\delta = 9.08$ (s, 1H), 8.39 – 8.35 (m, 1H), 8.06 – 7.97 (m, 4H), 7.86 – 7.82 (m, 1H), 7.71 (dddd, *J* = 16.6, 8.3, 7.0, 1.5 Hz, 2H), 7.62 – 7.55 (m, 3H), 7.36 (s, 1H), 4.14 (qd, *J* = 7.0, 1.6 Hz, 2H), 1.60 (s, 3H), 1.14 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO- d_6) $\delta = 195.9$, 168.6, 162.0, 137.3, 136.2, 133.2, 132.0, 131.0, 130.1, 128.3, 128.2, 127.9, 127.7, 127.5, 126.6, 126.3, 126.2, 126.1, 124.0, 120.8, 120.6, 110.9, 99.5, 70.2, 61.55, 20.0, 13.9; IR (KBr): 2958, 2923, 2853, 1742, 1461, 1376, 1261, 1091, 1020, 799 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₆H₂₁NNaO₃⁺ 418.1414; Found 418.1418.



Ethyl 1,2-dimethyl-3-oxo-2,3-dihydro-1*H*-benzo[*g*]indole-2-carboxylate (3ka). Yellow oil (15.9 mg, 56% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO- d_6) δ = 8.63 (dd, *J* = 8.6, 1.0 Hz, 1H), 7.92 (d, *J* = 7.9 Hz, 1H), 7.75 – 7.67 (m, 1H), 7.56 (ddd, *J* = 8.4, 7.0, 1.3 Hz, 1H), 7.37 (d, *J* = 8.5 Hz, 1H), 7.20 (d, *J* = 8.5 Hz, 1H), 4.22 – 4.02 (m, 2H), 3.55 (s, 3H), 1.57 (s, 3H), 1.12 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO- d_6) δ = 194.1, 167.6, 161.3, 140.1, 130.6, 129.6, 126.5, 126.3, 122.0, 120.4, 119.1, 113.5, 76.5, 62.3, 34.6, 18.3, 14.5; IR (KBr): 2956, 2924, 2853, 1742, 1688, 1461, 1404, 1261, 1093, 1021, 800 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₇H₁₇NNaO₃⁺ 306.1101; Found 306.1103.



Ethyl 2-methyl-3-oxo-1,2,3,4-tetrahydropyrrolo[**3,2-***b*]**indole-2-carboxylate** (**3ma**). Yellow oil (19.1 mg, 74% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.61 (s, 1H), 8.18 (s, 1H), 7.32 – 7.27 (m, 1H), 7.08 (d, *J* = 8.6 Hz, 1H), 6.83 (d, *J* = 8.6 Hz, 1H), 6.66 – 6.61 (m, 1H), 4.07 (qq, *J* = 6.9, 3.8 Hz, 2H), 1.47 (s, 3H), 1.11 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 195.2, 169.5, 158.4, 140.9, 124.1, 116.9, 112.2, 108.1, 104.7, 101.1, 69.6, 61.1, 20.4, 13.9; IR (KBr): 2955, 2924, 2853, 1740, 1661, 1461, 1376, 1261, 1092, 1021, 801 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₄H₁₄N₂NaO₃⁺281.0897; Found 281.0898.



Methyl 2-methyl-3-oxo-2,3-dihydro-1*H***-benzo**[*g*]**indole-2-carboxylate (3ab)**. Yellow oil (20.2 mg, 79% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO-*d*₆) δ = 8.92 (s, 1H), 8.23 (d, *J* = 8.2 Hz, 1H), 7.92 (d, *J* = 8.1 Hz, 1H), 7.77 – 7.70 (m, 1H), 7.65 – 7.59 (m, 1H), 7.37 (d, *J* = 8.6 Hz, 1H), 7.22 (d, *J* = 8.6 Hz, 1H), 3.63 (s, 3H), 1.56 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 195.8, 169.1, 162.6, 137.9, 130.7, 128.6, 126.1, 123.5, 120.58, 119.9, 118.5, 111.4, 70.0, 52.8, 19.9; IR (KBr): 2958, 2923, 2853, 1744, 1684, 1460, 1376, 1260, 1090, 1020, 799 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₅H₁₃NNaO₃⁺ 278.0788; Found 278.0789.



Benzyl 2-methyl-3-oxo-2,3-dihydro-1H-benzo[g]indole-2-carboxylate (3ac). Yellow oil

(24.5 mg, 74% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO-*d*₆) δ = 8.96 (s, 1H), 8.24 (d, *J* = 8.1 Hz, 1H), 7.92 (d, *J* = 8.1 Hz, 1H), 7.75 – 7.70 (m, 1H), 7.64 – 7.59 (m, 1H), 7.37 (d, *J* = 8.6 Hz, 1H), 7.34 – 7.23 (m, 5H), 7.21 (d, *J* = 8.6 Hz, 1H), 5.18 (dd, *J* = 18.2, 5.3 Hz, 2H), 1.59 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 195.7, 168.5, 162.6, 137.9, 135.8, 130.6, 128.6, 128.3, 127.9, 127.2, 126.1, 123.5, 120.6, 119.9, 118.5, 111.4, 70.1, 66.4, 19.9; IR (KBr): 2956, 2924, 2853, 1742, 1685, 1461, 1376, 1261, 1093, 1022, 800, 749 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₁H₁₇NNaO₃⁺ 354.1101; Found 354.1103.

tert-Butyl 2-methyl-3-oxo-2,3-dihydro-1*H*-benzo[*g*]indole-2-carboxylate (3ad). Yellow oil (20.2 mg, 68% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO-*d*₆) δ = 8.87 (s, 1H), 8.26 (d, *J* = 8.2 Hz, 1H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.75 – 7.69 (m, 1H), 7.64 – 7.58 (m, 1H), 7.36 (d, *J* = 8.6 Hz, 1H), 7.19 (d, *J* = 8.5 Hz, 1H), 1.50 (s, 3H), 1.34 (s, 9H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 196.1, 167.5, 162.5, 137.8, 130.5, 128.5, 125.9, 123.5, 120.6, 120.0, 118.2, 111.4, 81.5, 70.9, 27.4, 19.9; IR (KBr): 2956, 2923, 2853, 1733, 1684, 1460, 1373, 1260, 1091, 1022, 799 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₈H₁₉NNaO₃⁺ 320.1257; Found 320.1259.



Methyl 2-ethyl-3-oxo-2,3-dihydro-1*H*-benzo[*g*]indole-2-carboxylate (3ae). Yellow oil (21.0 mg, 74% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO-*d*₆) δ = 8.87 (s, 1H), 8.37 (d, *J* = 8.2 Hz, 1H), 7.91 (d, *J* = 8.2 Hz, 1H), 7.73 (t, *J* = 7.5 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 8.5 Hz, 1H), 7.19 (d, *J* = 8.6 Hz, 1H), 4.12 (p, *J* = 6.9 Hz, 2H), 2.16 (dq, *J* = 14.7, 7.4 Hz, 1H), 1.96 (dt, *J* = 13.9, 7.1 Hz, 1H), 1.13 (t, *J* = 7.1 Hz, 3H), 0.83 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 194.6, 167.9, 163.1, 137.9, 130.6, 128.5, 126.0, 123.7, 120.5, 119.7, 118.2, 112.1, 74.6, 61.4, 27.4, 13.9, 7.8; IR (KBr): 2958, 2923, 2853, 1737, 1680, 1459, 1378, 1261, 1090, 1021, 800, 750 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₇H₁₇NNaO₃⁺ 306.1101; Found 306.1103.



Methyl 3-oxo-2-propyl-2,3-dihydro-1*H***-benzo**[*g*]**indole-2-carboxylate** (**3af**). Yellow oil (20.5 mg, 69% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO-*d*₆) δ = 8.90 (s, 1H), 8.36 (d, *J* = 8.2 Hz, 1H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.76 – 7.71 (m, 1H), 7.65 – 7.59 (m, 1H), 7.34 (d, *J* = 8.6 Hz, 1H), 7.18 (d, *J* = 8.6 Hz, 1H), 4.16 – 4.07 (m, 2H), 2.15 – 1.84 (m, 3H), 1.12 (t, *J* = 7.1 Hz, 3H), 0.86 (t, *J* = 7.3 Hz, 4H); ¹³C NMR

 $(101 \text{ MHz}, \text{DMSO-}d_6) \delta = 194.6, 167.9, 163.0, 137.9, 130.6, 128.5, 126.0, 123.7, 120.5, 119.8, 118.2, 112.0, 74.2, 61.4, 36.4, 16.5, 13.9; IR (KBr): 2956, 2923, 2853, 1740, 1629, 1461, 1370, 1261, 1092, 1021, 800, 750 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₈H₁₉NNaO₃⁺ 320.1257; Found 320.1260.$



Methyl 2-isopropyl-3-oxo-2,3-dihydro-1*H***-benzo**[*g*]**indole-2-carboxylate** (**3ag**). Yellow oil (19.3 mg, 65% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO-*d*₆) δ = 8.75 (s, 1H), 8.54 (d, *J* = 8.1 Hz, 1H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.76 – 7.70 (m, 1H), 7.65 – 7.60 (m, 1H), 7.32 (d, *J* = 8.6 Hz, 1H), 7.17 (d, *J* = 8.6 Hz, 1H), 4.20 – 4.09 (m, 2H), 2.72 (p, *J* = 6.8 Hz, 1H), 1.16 (t, *J* = 7.1 Hz, 3H), 1.09 (d, *J* = 6.8 Hz, 3H), 0.63 (d, *J* = 6.7 Hz, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 193.6, 167.4, 163.7, 137.9, 130.6, 128.5, 125.9, 124.1, 120.6, 119.6, 118.2, 112.4, 78.8, 61.5, 33.5, 18.0, 15.4, 14.0; IR (KBr): 2957, 2924, 2853, 1736, 1680, 1461, 1376, 1261, 1092, 1021, 800, 750 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₈H₁₉NNaO₃⁺ 320.1257; Found 320.1258.



Methyl 2-(*tert*-butyl)-3-oxo-2,3-dihydro-1*H*-benzo[g]indole-2-carboxylate (3ah). Yellow oil (14.3 mg, 46% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO- d_6) δ = 8.65 (s, 1H), 8.62 (d, *J* = 7.7 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.74 – 7.67 (m, 1H), 7.59 (td, *J* = 7.6, 7.0, 1.2 Hz, 1H), 7.32 (d, *J* = 8.5 Hz, 1H), 7.15 (d, *J* = 8.5 Hz, 1H), 4.18 – 4.08 (m, 2H), 1.15 (t, *J* = 7.1 Hz, 3H), 1.11 (s, 9H); ¹³C NMR (101 MHz, DMSO- d_6) δ = 194.3, 167.5, 162.4, 138.3, 130.9, 128.9, 126.3, 124.7, 120.9, 120.0, 118.5, 113.7, 78.3, 61.7, 40.6, 27.9, 25.6, 14.5; IR (KBr): 2957, 2923, 2853, 1629, 1461, 1260, 1092, 1021, 800 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₉H₂₁NNaO₃⁺ 334.1414; Found 334.1416.



Methyl 3-oxo-2-phenyl-2,3-dihydro-1*H***-benzo**[*g*]**indole-2-carboxylate** (**3ai**). Yellow solid (27.8 mg, 84% yield, purified by silica gel chromatography using PE/EA 10:2). ¹H NMR (400 MHz, DMSO-*d*₆) δ = 9.55 (s, 1H), 8.53 (d, *J* = 8.1 Hz, 1H), 7.93 (d, *J* = 8.1 Hz, 1H), 7.78 – 7.73 (m, 1H), 7.70 – 7.63 (m, 3H), 7.44 – 7.33 (m, 4H), 7.22 (d, *J* = 8.6 Hz, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 1.14 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 192.6, 167.5, 162.3, 138.0, 135.3, 130.8, 128.6, 128.2, 126.5, 126.2, 123.9, 120.4, 120.0, 118.7, 110.8, 75.2, 62.0, 13.9; IR (KBr): 2981, 2923, 2851, 1741, 1679, 1625, 1532, 1472, 1411, 1236, 811, 755, 697 cm⁻¹. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₁H₁₇NNaO₃⁺ 354.1101; Found 354.1104.

VIII. ¹H and ¹³C NMR spectra

Ethyl 2-diazo-3-oxobutanoate (2a)



¹³C NMR (101 MHz, CDCl₃) Spectrum of Compound 2a

Methyl 2-diazo-3-oxobutanoate (2b)



¹³C NMR (101 MHz, CDCl₃) Spectrum of Compound **2b**

Benzyl 2-diazo-3-oxobutanoate (2c)



 ^{13}C NMR (101 MHz, CDCl₃) Spectrum of Compound 2c



 ^{13}C NMR (101 MHz, CDCl₃) Spectrum of Compound 2d



¹³C NMR (101 MHz, CDCl₃) Spectrum of Compound 2e



¹³C NMR (101 MHz, CDCl₃) Spectrum of Compound 2f

Ethyl 2-diazo-4-methyl-3-oxopentanoate (2g)



¹³C NMR (101 MHz, CDCl₃) Spectrum of Compound **2g**



¹³C NMR (101 MHz, CDCl₃) Spectrum of Compound **2h**

Ethyl 2-diazo-3-oxo-3-phenylpropanate (2i)



¹³C NMR (101 MHz, CDCl₃) Spectrum of Compound 2i



Ethyl 2-methyl-3-oxo-2,3-dihydro-1*H*-benzo[g]indole-2-carboxylate (3aa)

¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3aa**





¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ba**



Ethyl 5-amino-2-methyl-3-oxo-2,3-dihydro-1*H*-benzo[*g*]indole-2-carboxylate (3ca)

¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ca**



Ethyl 2-methyl-3-oxo-5-phenyl-2,3-dihydro-1*H*-benzo[*g*]indole-2-carboxylate (3da)

¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3da**





¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ea**

Ethyl 2-methyl-3-oxo-5-(*p*-tolyl)-2,3-dihydro-1*H*-benzo[*g*]indole-2-carboxylate (3fa)



¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3fa**

Ethyl 5-(4-isopropylphenyl)-2-methyl-3-oxo-2,3-dihydro-1*H*-benzo[g]indole-2-carboxylate (3ga)



¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ga**

Ethyl 5-(4-(*tert*-butyl)phenyl)-2-methyl-3-oxo-2,3-dihydro-1*H*-benzo[g]indole-2-carboxylate (3ha)



¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ha**

Ethyl 5-(3,5-dimethylphenyl)-2-methyl-3-oxo-2,3-dihydro-1H-benzo[g]indole-2-carboxylate (3ia)



¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ia**

Ethyl 2-methyl-5-(naphthalen-2-yl)-3-oxo-2,3-dihydro-1*H*-benzo[g]indole-2-carboxylate (3ja)



Ethyl 1,2-dimethyl-3-oxo-2,3-dihydro-1*H*-benzo[g]indole-2-carboxylate (3ka)



¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ka**



¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ma**

Methyl 2-methyl-3-oxo-2,3-dihydro-1*H*-benzo[*g*]indole-2-carboxylate (3ab)



¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ab**





¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ac**

tert-Butyl 2-methyl-3-oxo-2,3-dihydro-1*H*-benzo[g]indole-2-carboxylate (3ad)



¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ad**



¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ae**

Methyl 3-oxo-2-propyl-2,3-dihydro-1*H*-benzo[g]indole-2-carboxylate (3af)



¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3af**

Methyl 2-isopropyl-3-oxo-2,3-dihydro-1*H*-benzo[g]indole-2-carboxylate (3ag)



¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ag**





¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ah**

Methyl 3-oxo-2-phenyl-2,3-dihydro-1*H*-benzo[g]indole-2-carboxylate (3ai)



¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of Compound **3ai**