A RAPID AND EFFICIENT SYNTHESIS OF 3,3-DI(1*H*-INDOL-3-YL)INDOLIN-2-ONES AND 2,2-DI(1*H*-INDOL-3-YL)-2*H*-ACENAPHTHEN-1-ONES CATALYZED BY *p*-TSA

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Supporting Information

1 Experimental

All of the reagents were purchased from Aldrich-reagent and used without further purifications. ¹H NMR spectra and ¹³C NMR spectra were recorded on a Bruker Advance III 500 MHz spectrometer and using DMSO-*d*₆ (500 MHz for ¹H or 125 MHz for ¹³C, respectively) as solvent. Chemical shifts of ¹H and ¹³C NMR spectra were expressed in δ values (ppm) downfield from tetramethylsilane. Microanalyses were taken on a VARIOX Elemental Analyzer. Mass spectra were carried out on VARIAN1200. IR spectra were obtained on a TENSOR 27 spectrometer. Melting points were recorded on microscope (SGW X-4) melting point apparatus. The reaction was monitored by TLC.

2 Experimental details and characterization

General procedure for preparation of oxindole derivatives 3a-3p and 5a-5e

A mixture of the isatin **1** or acenaphthenequinone **4** (1 mmol), indole **2** (2 mmol), *p*-TSA (10 mol %), CH₂Cl₂ (10 mL) was added to 50 mL flask and stirred at room temperature until the completion of the reaction (**Scheme 1, 2**). Then the solid was filtered and washed with CH₂Cl₂ (5 mL) and ethanol (5 mL) in turns, affording the product in excellent yields, which was recrystallized from ethanol to produce pure crystalline products. The products were identified by elemental analysis, MS, ¹H NMR, ¹³C NMR, and IR spectra.

Scheme 1. Preparation of product **3a-3p**

Scheme 2. Preparation of product **5a-5e**

Spectroscopic and characterization data

3,3-di(1H-indol-3-yl)indolin-2-one (3a): Yield: 92%; white solid; m. p. 318-320 °C (lit.^[26] 312-314 °C); IR (KBr, cm^{-1}): 3428, 3324, 1707, 1613, 1468, 1106, 932, 736; ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 10.96 (s, 2H, NH), 10.60 (s, 1H, NH), 7.36 (d, $J = 8.1$ Hz, 2H), 7.24 (m, 4H), 7.01 (d, $J = 8.0$ Hz, 3H), 6.93 (t, $J = 7.5$ Hz, 1H), 6.86 (s, 2H), 6.80 (t, $J = 7.5$ Hz, 2H); ^{13}C NMR (125 MHz, $\text{DMSO}-d_6$): δ 178.7, 141.3, 136.9, 134.6, 127.8, 125.7, 124.9, 124.2, 121.4, 120.9, 120.7, 118.2, 114.3, 111.6, 109.5, 52.5; MS (EI, 70 ev): m/z 363 (M^+ , 91); Anal. calcd. for $\text{C}_{24}\text{H}_{17}\text{N}_3\text{O}$: C, 79.32; H, 4.72; N, 11.56; found: C, 79.40; H, 4.70; N, 11.60 %.

Scan 324 from d:\cz-01.xms

Spectrum from d:\cz-01.xms
 Scan No: 324, Time: 3.054 minutes
 3 points averaged. Not background corrected.
 Comment: 3.054 min. Scans: 323-325 50.0:450.0> Ion: NA RIC: 1.636e+8
 Pair Count: 77 MW: 0 Formula: None
 CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI
 Scan 1 Channel Description: 50.0:450.0> EI;Det 1000;WI Q1 Cal;ST 0.500
 Scan Information: qp = 760000.0 mTorr
 Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
76	701225	33	191	220050	10	293	220808	10
77	1.460e+6	69	216	289892	14	304	225083	11
78	635642	30	217	613742	29	305	678767	32
89	1.440e+6	68	218	2.491e+6	117	306	782692	37
90	1.329e+6	62	219	7.384e+6	347	307	401083	19
91	263092	12	220	1.307e+6	61	317	598575	28
103	502975	24	231	252258	12	318	417842	20
104	6.791e+6	319	233	267958	13	319	799567	38
105	517242	24	234	320742	15	320	279483	13
115	222242	10	242	347683	16	331	459133	22
116	808550	38	243	248908	12	332	1.370e+6	64
117	4.494e+6	211	245	645700	30	333	892408	42
118	654050	31	246	954017	45	334	2.082e+7	979
131	262475	12	247	2.616e+6	123	335	5.864e+6	276
132	1.126e+7	529	248	941617	44	336	661908	31
133	1.174e+6	55	249	749925	35	344	369342	17
139	231942	11	261	246775	12	345	374108	18
146	838058	39	262	283833	13	346	517692	24
147	2.125e+7	999	263	343592	16	347	220308	10
148	1.936e+6	91	275	224675	11	362	3.774e+6	177
152	240883	11	276	278783	13	363	1.927e+7	906
166	370200	17	277	631733	30	364	4.836e+6	227
167	536108	25	278	1.456e+6	68	365	645350	30
173	271492	13	279	334175	16	391	903483	42
182	539450	25	291	237467	11	392	302292	14
190	646983	30	292	322117	15

3,3-di(1H-indol-3-yl)-5-fluoroindolin-2-one (3b): Yield: 88 %; white solid, m. p. 232-234°C (lit.^[13] 245-247°C); IR (KBr, cm⁻¹): 3436, 3316, 1696, 1481, 1104, 861, 734; ¹H NMR (500 MHz, DMSO-*d*₆) δ 11.02 (s, 2H, NH), 10.65 (s, 1H, NH), 7.37 (d, *J* = 8.1 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.03 (m, 5H), 6.91 (s, 2H), 6.83 (t, *J* = 7.5 Hz, 2H); ¹³C NMR (125 MHz, DMSO-*d*₆): δ 178.6, 157.8 (*J* = 235 Hz), 137.5, 136.9, 136.3 (*J* = 7.8 Hz), 125.5, 124.4, 121.0, 120.6, 118.3, 114.2 (*J* = 23 Hz), 113.6, 112.4 (*J* = 24 Hz), 111.7, 110.3 (*J* = 7.9 Hz), 53.1; MS (EI, 70 ev): *m/z* 381 (M⁺, 7); Anal. calcd. for C₂₄H₁₆FN₃O: C, 75.58; H, 4.23; N, 11.02; found: C, 75.49; H, 4.25; N, 10.98%.

Scan 380 from d:\cz-02.xms

Spectrum from d:\cz-02.xms
 Scan No: 380, Time: 3.595 minutes
 3 points averaged. Not background corrected.
 Comment: 3.595 min. Scans: 379-381 50.0:450.0> Ion: NA RIC: 7.077e+6
 Pair Count: 87 MW: 0 Formula: None
 CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI
 Scan 1 Channel Description: 50.0:450.0> EI;Det 1000;WI Q1 Cal;ST 0.500
 Scan Information: cp = 760000.0 mTorr
 Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
59	14192	10	190	21258	15	306	20033	14
76	17267	12	203	14483	10	308	25858	18
77	36550	26	208	85142	60	323	16425	12
81	15108	11	209	14992	11	324	31717	22
84	34375	24	210	19317	14	326	15258	11
86	31275	22	222	31108	22	330	27508	20
89	60708	43	229	14858	11	331	43817	31
90	53683	38	232	31092	22	332	31283	22
92	21092	15	236	114683	81	334	24700	18
102	22525	16	237	325833	231	336	17917	13
104	26075	18	238	104050	74	337	33858	24
110	17717	13	243	17925	13	338	32733	23
112	21267	15	245	22425	16	349	29017	21
117	155242	110	247	59808	42	350	50442	36
118	15883	11	260	18792	13	351	35508	25
126	19008	13	261	14567	10	352	1.409e+6	999
128	15692	11	263	18158	13	353	373508	265
134	36158	26	264	69358	49	354	51925	37
140	26950	19	265	258875	184	361	26283	19
141	16075	11	266	163683	116	362	55375	39
151	27492	19	267	29317	21	364	25642	18
158	32692	23	276	20142	14	365	16858	12
161	19317	14	277	16233	12	381	649917	461
162	38200	27	278	28950	21	382	164700	117
168	19075	14	279	23183	16	383	21983	16
175	14500	10	280	17458	12	404	18292	13
179	14567	10	281	17367	12	406	14533	10
181	17492	12	290	20975	15	407	107708	76
183	37183	26	291	23458	17	408	22958	16

3,3-di(1*H*-indol-3-yl)-7-trifluoromethylindolin-2-one (3c): Yield: 85 %; white solid, m. p. 257°C; IR (KBr, cm⁻¹): 3417, 3249, 3057, 1730, 1458, 1104, 1060, 744; ¹H NMR (500 MHz, DMSO-*d*₆) δ 11.11 (s, 1H, NH), 11.05 (s, 2H, NH), 7.55 (d, *J* = 8.0 Hz, 1H), 7.50 (d, *J* = 7.4 Hz, 1H), 7.38 (d, *J* = 8.1 Hz, 2H), 7.23 (d, *J* = 8.1 Hz, 2H), 7.12 (t, *J* = 7.9 Hz, 1H), 7.04 (t, *J* = 7.4 Hz, 2H), 6.87 (s, 2H), 6.85-6.81 (m, 2H); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 179.0, 138.6, 136.9, 136.3, 128.9, 125.5, 123.8, 124.5, 124.4, 121.7, 121.1, 120.5, 118.5, 113.4, 111.8, 110 (q, *J* = 32.6 Hz), 51.7; MS (EI, 70 ev): *m/z* 431 (*M*⁺, 100); Anal. calcd. for C₂₅H₁₆F₃N₃O: C, 69.60; H, 3.74; N, 9.74; found: C, 69.71; H, 3.71; N, 9.81%.

Scan 319 from d:\cz-03.xms

Spectrum from d:\cz-03.xms

Scan No: 319, Time: 3.187 minutes

3 points averaged. Not background corrected.

Comment: 3.187 min. Scans: 318-320 50.0:450.0> Ion: NA RIC: 2.023e+9

Pair Count: 92 MW: 0 Formula: None

CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:450.0> EI;Det 1000;WI Q1 Cal;ST 0.500

Scan Information: cp = 760000.0 mTorr

Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
63	3.592e+6	11	218	5.342e+6	17	347	3.374e+6	11
77	3.559e+6	11	219	4.768e+6	15	353	4.241e+6	13
89	1.161e+7	37	243	5.244e+6	17	354	1.281e+7	41
90	6.863e+6	22	245	1.106e+7	35	355	7.080e+6	23
116	7.054e+6	22	246	3.372e+6	11	360	4.874e+6	16
117	7.382e+7	235	247	1.140e+7	36	361	5.958e+6	19
118	8.114e+6	26	248	3.170e+6	10	362	1.150e+7	37
138	3.943e+6	13	265	5.234e+6	17	363	3.962e+6	13
139	3.895e+6	12	266	1.130e+7	36	364	4.440e+6	14
140	3.283e+6	10	267	9.149e+7	291	365	8.383e+6	27
152	5.476e+6	17	268	1.768e+7	56	366	3.681e+6	12
153	3.786e+6	12	278	5.379e+6	17	367	7.837e+6	25
158	3.442e+6	11	279	3.875e+6	12	380	1.902e+7	61
159	4.074e+6	13	286	1.470e+7	47	381	1.469e+7	47
163	3.232e+6	10	287	5.617e+7	179	382	1.143e+8	364
164	4.043e+6	13	288	1.209e+7	38	383	3.233e+7	103
166	5.715e+6	18	295	5.825e+6	19	384	7.885e+6	25
167	3.285e+6	10	303	5.621e+6	18	387	4.303e+6	14
174	3.892e+6	12	304	5.699e+6	18	393	6.636e+6	21
176	3.243e+6	10	305	4.321e+6	14	394	4.272e+6	14
177	4.656e+6	15	314	7.132e+6	23	402	1.907e+8	607
183	5.631e+6	18	315	4.532e+7	144	403	5.470e+7	174
184	4.675e+6	15	316	1.635e+7	52	404	8.375e+6	27
189	3.481e+6	11	317	4.789e+6	15	410	8.089e+6	26
190	1.027e+7	33	330	4.396e+6	14	412	5.451e+6	17
191	1.422e+7	45	331	1.640e+7	52	414	9.907e+6	32
192	1.208e+7	38	332	1.041e+7	33	430	4.025e+7	128
192	3.283e+6	10	333	5.198e+6	17	431	3.140e+8	999
206	2.269e+7	72	334	6.006e+6	19	432	8.471e+7	270
216	9.985e+6	32	335	3.871e+6	12	433	1.239e+7	39
217	5.079e+6	16	342	4.122e+6	13

3,3-di(1*H*-indol-3-yl)-6-chloroindolin-2-one (3d): Yield: 82 %; white solid, m. p. 244°C; IR (KBr, cm⁻¹): 3421, 3221, 1724, 1610, 1439, 1099, 922, 742; ¹H NMR (500 MHz, DMSO-*d*₆) δ 11.01 (s, 2H, NH), 10.76 (s, 1H, NH), 7.37 (d, *J* = 8.1 Hz, 2H), 7.22 (dd, *J* = 8.0, 4.7 Hz, 3H), 7.05-7.01 (m, 3H), 6.99 (dd, *J* = 8.0, 4.5 Hz, 1H), 6.87 (s, 2H), 6.82 (t, *J* = 8.1 Hz, 2H); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 178.6, 142.8, 136.9, 133.4, 131.9, 126.3, 125.5, 124.3, 125.5, 124.3, 121.2, 121.0, 120.6, 118.4, 113.7, 111.7, 109.6, 52.3; MS (EI, 70 ev): *m/z* 397 (*M*⁺, 100), 399 (*M*+2, 36); Anal. calcd. for C₂₄H₁₆ClN₃O: C, 72.45; H, 4.05; N, 10.56; found: C, 72.55; H, 4.08; N, 10.52%.

Scan 433 from d:\cz-04.xms

Spectrum from d:\cz-04.xms

Scan No: 433, Time: 4.064 minutes

3 points averaged. Not background corrected.

Comment: 4.064 min. Scans: 432-434 50.0:450.0> Ion: NA RIC: 1.963e+7

Pair Count: 70 MW: 0 Formula: None

CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:450.0> EI;Det 1000;WI Q1 Cal;ST 0.500

Scan Information: cp = 760000.0 mTorr

Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
89	47608	15	253	785508	246	339	43025	13
90	66275	21	254	291750	92	340	42525	13
116	53442	17	255	262942	82	341	38408	12
117	396475	124	256	62617	20	343	34042	11
118	32600	10	277	40800	13	353	50467	16
132	48708	15	278	95683	30	354	36733	12
147	110308	35	280	66433	21	362	41742	13
152	38575	12	281	111417	35	363	166175	52
153	33950	11	282	102642	32	366	75808	24
159	51667	16	283	92783	29	368	3.145e+6	987
163	35333	11	284	42467	13	369	1.027e+6	322
166	69450	22	304	91367	29	370	1.170e+6	367
167	36908	12	305	101425	32	371	290083	91
172	32567	10	306	49117	15	379	37083	12
185	37800	12	317	43025	13	380	56242	18
189	47967	15	318	34608	11	391	35108	11
190	48917	15	328	41683	13	393	35150	11
199	56108	18	330	67258	21	394	62033	19
218	142783	45	331	237550	75	397	3.184e+6	999
219	78658	25	332	336483	106	398	1.089e+6	342
245	75367	24	333	185100	58	399	1.156e+6	363
249	54550	17	334	108358	34	400	353000	111
251	43792	14	335	34350	11	401	43375	14
252	142158	45

3,3-di(1*H*-indol-3-yl)-5-methoxylindolin-2-one (3e): Yield: 90 %; white solid, m. p. 290°C; IR (KBr, cm⁻¹): 3364, 3048, 1689, 1487, 1148, 1015, 858, 742; ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.96 (s, 2H, NH), 10.44 (s, 1H, NH), 7.36 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.1 Hz, 2H), 7.04-7.01 (m, 2H), 6.92 (m, 1H), 6.88 (s, 2H), 6.83-6.80 (m, 4H), 3.62 (s, 3H, O-CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 178.6, 154.6, 136.9, 135.9, 134.7, 125.7, 124.3, 120.9, 120.7, 118.2, 114.3, 112.2, 112.0, 115.6, 109.8, 55.3, 53.0; MS (EI, 70 ev): *m/z* 393 (M⁺, 34); Anal. calcd. for C₂₅H₁₉N₃O₂: C, 76.32; H, 4.87; N, 10.68; found: C, 76.22; H, 4.90; N, 10.64%.

Scan 327 from d:\cz-06.xms

Spectrum from d:\cz-06.xms
 Scan No: 327, Time: 3.313 minutes
 3 points averaged. Not background corrected.
 Comment: 3.313 min. Scans: 326-328 50.0:450.0> Ion: NA RIC: 4.016e+8
 Pair Count: 122 MW: 0 Formula: None
 CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:450.0> EI;Det 1000;WI Q1 Cal;ST 0.500
 Scan Information: cp = 760000.0 mTorr
 Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
55	766725	13	192	890825	16	277	3.701e+6	65
63	996000	17	193	1.903e+6	33	278	2.905e+6	51
75	704342	12	194	1.951e+6	34	279	2.487e+7	434
76	683875	12	195	3.981e+6	69	280	5.032e+6	88
77	1.262e+6	22	196	2.575e+6	45	281	1.445e+6	25
89	1.678e+6	29	197	1.146e+6	20	293	692725	12
90	1.709e+6	30	204	711158	12	295	732650	13
91	593067	10	205	2.240e+6	39	296	5.727e+7	999
102	678450	12	206	3.686e+6	64	297	1.110e+7	194
103	691800	12	207	1.234e+6	22	298	1.444e+6	25
114	761950	13	208	835950	15	302	1.101e+6	19
115	1.048e+6	18	219	856567	15	304	1.368e+6	24
116	957492	17	220	856558	15	305	743475	13
117	4.928e+6	86	221	1.775e+6	31	307	2.191e+6	38
118	885475	15	222	1.201e+6	21	308	1.027e+6	18
125	917425	16	223	2.586e+6	45	309	1.420e+6	25
126	1.135e+6	20	224	1.113e+6	19	310	1.138e+6	20
127	1.290e+6	23	232	634000	11	319	771667	13
128	880008	15	233	1.736e+6	30	320	2.650e+6	46
130	622983	11	234	2.456e+6	43	321	1.594e+6	28
139	1.650e+6	29	235	1.255e+6	22	323	3.602e+6	63
140	1.494e+6	26	236	723817	13	324	2.777e+6	48
141	862367	15	239	924958	16	325	700442	12
151	1.041e+6	18	240	9.022e+6	157	332	634975	11
152	2.219e+6	39	241	1.688e+6	29	347	761300	13
153	1.591e+6	28	243	579700	10	348	1.470e+6	26
154	1.649e+6	29	245	790658	14	349	1.383e+6	24
155	676150	12	248	2.051e+6	36	350	906100	16
161	793292	14	249	1.063e+7	185	352	5.253e+6	92
165	807417	14	250	3.590e+6	63	353	1.166e+6	20
166	1.879e+6	33	251	1.071e+7	187	364	2.039e+7	356
167	2.697e+6	47	252	2.245e+6	39	365	5.769e+6	101
168	1.839e+6	32	253	687067	12	366	899100	16

Scan 327 from d:\cz-06.xms - Page 2

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
175	596767	10	261	2.752e+6	48	373	684592	12
177	728983	13	262	786733	14	375	657725	11
178	966492	17	264	807667	14	392	2.908e+6	51
179	1.299e+6	23	267	618350	11	393	1.958e+7	341
180	1.404e+6	24	268	9.350e+6	163	394	5.315e+6	93
181	1.356e+6	24	269	2.006e+6	35	395	877458	15
182	913958	16	275	686367	12	407	639050	11
191	619992	11	276	3.178e+6	55

3,3-di(1*H*-indol-3-yl)-5-nitroindolin-2-one (3f): Yield: 84 %; yellow solid, m. p. 281-282 °C (lit.^[26] 297-298 °C); IR (KBr, cm⁻¹): 3363, 3054, 1714, 1611, 1479, 1080, 941, 749; ¹H NMR (500 MHz, DMSO-*d*₆) δ 11.34 (s, 1H, NH), 11.10 (s, 2H, NH), 8.25 (dd, *J* = 8.5, 2.3 Hz, 1H), 7.98 (d, *J* = 2.3 Hz, 1H), 7.39 (d, *J* = 8.2 Hz, 2H), 7.22 (m, 3H), 7.07-7.03 (m, 2H), 6.97 (s, 2H), 6.86-6.83 (m, 2H); ¹³C NMR (125 MHz, DMSO-*d*₆): δ 178.9, 147.8, 142.2, 137.0, 135.3, 125.5, 125.4, 124.6, 121.2, 120.3, 120.1, 118.6, 112.8, 111.8, 109.9, 52.5; MS (EI, 70 ev): *m/z* 408 (M⁺, 37); Anal. calcd. for C₂₄H₁₆N₄O₃: C, 70.58; H, 3.95; N, 13.72; found: C, 70.66; H, 3.92; N, 13.69%

Scan 401 from d:\cz-07.xms

Spectrum from d:\cz-07.xms

Scan No: 401, Time: 3.937 minutes

3 points averaged. Not background corrected.

Comment: 3.937 min. Scans: 400-402 50.0:450.0> Ion: NA RIC: 2.621e+7

Pair Count: 105 MW: 0 Formula: None

CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:450.0> EI;Det 1000;VI Q1 Cal;ST 0.500

Scan Information: cp = 760000.0 mTorr

Precursor Mass Range: 50 - 450 m/z

Ion			Ion			Ion		
Int	Norm		Int	Norm		Int	Norm	
50	36311	10	217	247742	68	316	39000	11
58	73125	20	218	580917	160	319	59917	17
62	45608	13	219	148858	41	320	41475	11
63	166108	46	230	112192	31	328	36508	10
64	55383	15	231	326633	90	333	338625	93
65	94725	26	232	770725	213	334	169283	47
77	37767	10	233	123858	34	335	58142	16
78	47608	13	235	40875	11	344	50117	14
88	37525	10	236	72208	20	345	83017	23
89	781642	216	237	180358	50	346	68042	19
90	896483	247	245	98742	27	347	49008	14
91	121383	34	246	152417	42	350	36675	10
104	62542	17	247	339650	94	351	37883	10
114	64383	18	248	84908	23	352	640067	177
115	152475	42	249	38083	11	353	202492	56
116	155675	43	251	37067	10	359	74958	21
117	3.619e+6	999	253	67992	19	361	111183	31
118	228358	63	254	46508	13	362	560575	155
130	78567	22	263	74492	21	363	280825	78
132	59058	16	264	725000	200	364	82583	23
139	47258	13	265	187300	52	368	80950	22
145	51892	14	266	119300	33	374	46717	13
147	50508	14	275	52767	15	378	106500	29
164	61033	17	276	96325	27	379	1.279e+6	353
165	54475	15	278	54533	15	380	341775	94
167	55000	15	279	49408	14	381	539550	149
176	36733	10	282	42242	12	382	161950	45
178	69558	19	291	129558	36	390	116000	32
180	47692	13	292	440908	122	391	596350	165
188	42392	12	293	1.057e+6	292	392	210875	58
190	66375	18	294	230525	64	397	38583	11
204	73800	20	302	51175	14	405	87342	24
205	44033	12	303	48333	13	408	1.345e+6	371

3,3-di(1*H*-indol-3-yl)-5-chloroindolin-2-one (3g): Yield: 86 %; white solid, m. p. 289-291 °C (lit.^[27] 295-297 °C); IR (KBr, cm⁻¹): 3444, 3362, 3038, 1698, 1475, 1104, 815, 735; ¹H NMR (500 MHz, DMSO-*d*₆) δ 11.03 (s, 2H, NH), 10.77 (s, 1H, NH), 7.38 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.3 Hz, 1H), 7.20 (m, 3H), 7.03 (m, 3H), 6.90 (s, 2H), 6.83 (t, *J* = 7.5 Hz, 2H); ¹³C NMR (125 MHz, DMSO-*d*₆): δ 178.3, 140.3, 136.9, 136.6, 127.8, 125.5, 125.4, 124.6, 124.4, 121.0, 120.5, 118.4, 113.5, 111.7, 111.1, 52.8; MS (EI, 70 ev): *m/z* 397 (*M*⁺, 52), 399 (*M*+2, 15); Anal. calcd. for C₂₄H₁₆ClN₃O: C, 72.45; H, 4.05; N, 10.56; found: C, 72.38; H, 4.02; N, 10.60%.

Scan 439 from d:\cz-08.xms

Spectrum from d:\cz-08.xms

Scan No: 439, Time: 4.148 minutes

3 points averaged. Not background corrected.

Comment: 4.148 min. Scans: 438-440 50.0:450.0> Ion: NA RIC: 1.882e+7

Pair Count: 113 MW: 0 Formula: None

CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:450.0> EI;Det 1000;WI Q1 Cal;ST 0.500

Scan Information: cp = 760000.0 mTorr

Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
90	50542	22	254	89717	38	344	43800	19
116	40900	18	255	106525	46	345	141967	61
117	147008	63	261	57800	25	346	26625	11
118	32125	14	264	57517	25	348	30483	13
130	51067	22	265	200150	86	349	36850	16
131	34450	15	266	55267	24	351	61650	26
132	42975	18	267	37908	16	352	1.733e+6	743
133	33475	14	268	37750	16	353	611508	262
145	32250	14	275	24908	11	354	98250	42
147	59492	26	276	55567	24	361	30225	13
152	54008	23	278	78533	34	362	108808	47
158	36625	16	280	38975	17	364	59383	25
162	29875	13	281	154883	66	366	47392	20
166	43842	19	282	102250	44	367	53392	23
167	66600	29	283	59092	25	368	2.329e+6	999
170	26775	11	284	50667	22	369	673692	289
183	31192	13	292	66083	28	370	758675	325
190	69900	30	292	34600	15	371	233467	100
191	26875	12	294	25108	11	379	47892	21
201	37625	16	296	26117	11	380	586583	252
204	24542	11	302	29608	13	381	895000	384
208	32958	14	303	44767	19	382	367358	158
215	23733	10	304	35708	15	383	35117	15
217	34242	15	306	50975	22	384	65892	28
218	46433	20	315	24842	11	394	28858	12
219	36083	15	316	45375	19	395	38625	17
229	25633	11	317	39483	17	396	229300	98
231	33083	14	323	49283	21	397	1.215e+6	521
232	24583	11	330	34242	15	398	328242	141
236	179883	77	331	160108	69	399	346117	148
237	441167	189	332	301775	129	400	80775	35
238	125367	54	333	99558	43	404	33092	14
242	23883	10	334	111925	48	407	23625	10

3,3-di(1*H*-indol-3-yl)-5-methylindolin-2-one(3h): Yield: 93 %; white solid, m. p. 305-307°C (lit.^[16] 321-322°C); IR (KBr, cm⁻¹): 3379, 3320, 3042, 1706, 1488, 1101, 811, 737; ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.95 (s, 2H, NH), 10.49 (s, 1H, NH), 7.36 (d, *J* = 8.1 Hz, 2H), 7.25 (d, *J* = 7.9 Hz, 2H), 7.05-7.01 (m, 4H), 6.89 (m, 1H), 6.85 (s, 2H), 6.83-6.79 (m, 2H), 2.19 (s, 3H, CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆): δ 178.7, 138.9, 136.9, 134.6, 130.1, 128.1, 125.7, 125.4, 124.3, 120.9, 120.8, 118.2, 114.4, 111.5, 109.3, 52.6, 20.8; MS (EI, 70 ev): *m/z* 377 (*M*⁺, 50); Anal. calcd. for C₂₅H₁₉N₃O: C, 79.55; H, 5.07; N, 11.13; found: C, 79.46; H, 5.03; N, 11.10%.

Scan 350 from d:\cz-09.xms

Spectrum from d:\cz-09.xms
 Scan No: 350, Time: 3.530 minutes
 3 points averaged. Not background corrected.
 Comment: 3.530 min. Scans: 349-351 50.0:450.0> Ion: NA RIC: 7.952e+6
 Pair Count: 159 MW: 0 Formula: None
 CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:450.0> EI;Det 1000;WI Q1 Cal;ST 0.500
 Scan Information: cp = 760000.0 mTorr
 Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
50	38233	36	179	20267	19	296	25233	24
62	21000	20	189	14100	13	304	19083	18
63	11792	11	190	18850	18	306	16308	15
64	11550	11	191	24592	23	307	17442	16
76	24583	23	192	12083	11	309	12850	12
77	14092	13	193	14283	13	310	17067	16
78	16250	15	203	21050	20	315	11000	10
81	10758	10	204	56200	52	317	31242	29
84	17192	16	206	17858	17	318	20567	19
86	17317	16	207	14542	14	320	33250	31
89	43917	41	210	13542	13	320	21325	20
90	41475	39	211	17583	16	321	37108	35
91	12433	12	214	14450	13	327	14775	14
95	18683	17	218	64700	60	330	15567	15
99	17258	16	220	23192	22	332	44575	42
100	13742	13	222	13275	12	333	30258	28
102	17642	16	230	11842	11	334	41967	39
103	10750	10	231	50267	47	335	36917	34
104	77058	72	232	94175	88	336	19650	18
105	15117	14	233	397600	371	343	25675	24
112	14258	13	234	42517	40	344	37292	35
114	17425	16	236	21442	20	347	72733	68
115	41908	39	237	23267	22	348	1.071e+6	999
117	134608	126	238	26792	25	349	350767	327
118	28708	27	243	25717	24	350	24250	23
119	35067	33	244	27258	25	352	22867	21
121	15600	15	245	25892	24	360	34242	32
123	25008	23	247	26408	25	362	11217	10
126	14333	13	249	24183	23	364	16100	15
130	18117	17	250	19050	18	373	18425	17
132	80000	75	252	24208	23	375	12533	12
133	34950	33	254	11767	11	376	172325	161
137	22308	21	255	26683	25	377	533583	498

Scan 350 from d:\cz-09.xms - Page 2

Ion			Ion			Ion		
Int	Norm		Int	Norm		Int	Norm	
138	16617	15	259	12975	12	378	195492	182
139	13167	12	261	224708	210	380	21650	20
145	34433	32	262	126408	118	381	23458	22
146	17000	16	263	23567	22	383	15675	15
147	106925	100	265	10775	10	388	18150	17
148	14075	13	266	19383	18	389	23000	21
150	16817	16	268	13033	12	393	27167	25
152	15008	14	269	15908	15	394	28708	27
153	11483	11	270	21350	20	394	16925	16
155	25475	24	275	22150	21	395	11483	11
157	10883	10	276	27558	26	396	13908	13
158	21508	20	277	62492	58	404	42633	40
159	22517	21	278	48308	45	407	56742	53
163	15750	15	280	17042	16	407	32233	30
166	29617	28	281	28308	26	409	33000	31
167	16867	16	286	15608	15	422	12525	12
172	17808	17	287	11817	11	423	56792	53
176	23525	22	288	14283	13	433	11100	10
177	20867	19	292	22842	21	434	12350	12
178	13875	13	293	22250	21	440	10958	10

3,3-di(1*H*-indol-3-yl)-5,7-dimethylindolin-2-one (3i): Yield: 95 %; white solid, m. p. 297 °C; IR (KBr, cm⁻¹): 3457, 3337, 1680, 1456, 1101, 850, 743; ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.93 (s, 2H, NH), 10.53 (s, 1H, NH), 7.36 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.04-7.00 (m, 2H), 6.86-6.82 (m, 6H), 2.29 (s, 3H, CH₃), 2.15 (s, 3H, CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 179.2, 137.4, 136.9, 134.3, 129.9, 129.4, 125.7, 124.2, 122.7, 120.9, 120.8, 118.4, 118.1, 114.6, 111.5, 52.8, 20.7, 18.5; MS (EI, 70 ev): *m/z* 391 (*M*⁺, 23); Anal. calcd. for C₂₆H₂₁N₃O: C, 79.77; H, 5.41; N, 10.73; found: C, 79.72; H, 5.44; N, 10.69%.

Scan 365 from d:\cz-10.xms

Spectrum from d:\cz-10.xms

Scan No: 365, Time: 3.495 minutes

3 points averaged. Not background corrected.

Comment: 3.495 min. Scans: 364-366 50.0:450.0> Ion: NA RIC: 1.665e+7

Pair Count: 38 MW: 0 Formula: None

CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:450.0> EI;Det 1000;WI Q1 Cal;ST 0.500

Scan Information: cp = 760000.0 mTorr

Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
117	102425	21	248	149025	30	362	4.949e+6	999
132	57383	12	262	59100	12	363	1.396e+6	282
147	96158	19	273	104875	21	364	200858	41
152	58125	12	275	295625	60	374	96350	19
158	60267	12	276	116283	23	375	56725	11
204	52408	11	331	106933	22	376	129058	26
217	63150	13	332	105092	21	377	64375	13
231	164375	33	333	49625	10	378	120392	24
232	188858	38	334	50467	10	390	833592	168
243	57058	12	345	71508	14	391	1.160e+6	234
245	233325	47	346	138958	28	392	466400	94
246	231950	47	347	237925	48	393	81608	16
247	830350	168	348	122608	25

3,3-di(5-methoxy-1*H*-indol-3-yl)indolin-2-one (3j): Yield: 87 %; white solid, m. p. 286-287°C (lit.^[13] 292-294°C); IR (KBr, cm⁻¹): 3388, 3317, 3120, 1685, 1478, 1019, 759; ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.81 (s, 2H, NH), 10.60 (s, 1H, NH), 7.27-7.20 (m, 4H), 7.01 (d, *J* = 7.7 Hz, 1H), 6.95 (d, *J* = 7.5 Hz, 1H), 6.86 (s, 2H), 6.70 (m, 4H), 3.53 (s, 6H, O-CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 178.8, 152.4, 141.4, 134.6, 132.2, 127.8, 126.1, 125.2, 124.9, 121.5, 113.6, 112.1, 110.4, 109.4, 103.4, 55.1, 52.5; MS (EI, 70 ev): *m/z* 423 (*M*⁺, 35); Anal. calcd. for C₂₆H₂₁N₃O₃: C, 73.74; H, 5.00; N, 9.92, found: C, 73.80; H, 5.04; N, 9.96%.

Scan 352 from d:\cz-11.xms

Spectrum from d:\cz-11.xms
 Scan No: 352, Time: 3.361 minutes
 3 points averaged. Not background corrected.
 Comment: 3.361 min. Scans: 351-353 50.0:450.0> Ion: NA RIC: 1.879e+7
 Pair Count: 140 MW: 0 Formula: None
 CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI
 Scan 1 Channel Description: 50.0:450.0> EI; Det 1000; W1 Q1 Cal; ST 0.500
 Scan Information: ϕ = 760000.0 mTorr
 Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
63	38258	20	204	29442	15	302	21883	11
75	22475	12	205	189667	99	304	20658	11
76	25367	13	206	261392	136	305	54508	28
77	149033	77	207	25650	13	307	31708	16
78	47683	25	208	19775	10	308	47500	25
89	57975	30	212	19592	10	309	20842	11
90	36133	19	216	20050	10	320	25867	13
102	32733	17	218	23792	12	322	21850	11
104	724775	377	219	37392	19	323	22383	12
105	19433	10	220	25458	13	326	20050	10
115	27517	14	221	28058	15	332	20667	11
116	64667	34	223	28233	15	335	25775	13
117	34650	18	233	108858	57	336	74325	39
126	30683	16	234	330833	172	337	34933	18
128	36917	19	235	83242	43	345	33967	18
131	24008	12	242	20933	11	346	36283	19
132	842667	438	246	42625	22	347	36783	19
133	123067	64	247	87250	45	348	48342	25
138	42217	22	248	94875	49	350	55742	29
140	21558	11	249	686925	357	351	41008	21
142	22075	11	250	178583	93	361	19683	10
145	47917	25	251	22367	12	361	25450	13
146	113225	59	254	27542	14	362	37700	20
147	1.311e+6	681	255	31483	16	364	40767	21
148	129567	67	258	29475	15	365	57875	30
150	20758	11	260	24442	13	366	29825	15
151	33733	18	261	247517	129	378	284433	148
152	36450	19	262	53858	28	379	232792	121
154	19700	10	263	185750	96	380	46817	24
156	22392	12	264	62550	32	381	44217	23
160	35067	18	265	25708	13	390	28042	15
166	47200	25	267	24475	13	392	83317	43
168	30258	16	268	23167	12	394	1.923e+6	999

Scan 352 from d:\cz-11.xms - Page 2

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
169	19283	10	269	19992	10	395	669808	348
174	22792	12	274	32825	17	396	70367	37
175	45467	24	276	264300	137	404	34700	18
178	45367	24	277	765100	397	406	111183	58
179	37892	20	278	700800	364	407	74700	39
180	41758	22	279	144650	75	408	88958	46
181	21125	11	280	23142	12	409	51417	27
182	41792	22	281	48917	25	410	31642	16
188	76908	40	282	39775	21	421	24092	13
190	55883	29	290	20942	11	422	549433	285
191	23908	12	292	189467	98	423	801049	416
195	34067	18	293	60558	31	424	632217	328
198	21400	11	294	31700	16	425	104742	54
203	36883	19	301	26558	14

3,3-di(5-methoxyl-1*H*-indol-3-yl)-5-methylindolin-2-one (3k): Yield: 94 %; white solid, m. p. 284 °C; (KBr, cm⁻¹): 3180, 1708, 1483, 1173, 806; ¹H NMR (500 MHz, DMSO) δ 10. (s, 2H, NH), 10.50 (s, 1H, NH), 7.28-7.25 (m, 2H), 7.05 (d, *J* = 7.8 Hz, 1H), 7.00 (s, 1H), 6.90 (d, *J* = 7.6 Hz, 1H), 6.85 (s, 2H), 6.72-6.69 (m, 4H), 3.54 (s, 6H, O-CH₃), 2.20 (s, 3H, CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 178.7, 152.4, 139.0, 134.5, 132.2, 130.2, 128.1, 126.1, 125.5, 125.2, 113.7, 112.0, 110.4, 109.2, 103.4, 55.1, 52.6, 20.8; MS (EI, 70 ev): *m/z* 437 (*M*⁺, 100); Anal. calcd. for C₂₇H₂₃N₃O₃: C, 74.12; H, 5.30; N, 9.60; found: C, 74.18; H, 5.34; N, 9.56%.

Scan 414 from d:\cz-13.xms

Spectrum from d:\cz-13.xms
Scan No: 414, Time: 4.090 minutes
3 points averaged. Not background corrected.
Comment: 4.090 min. Scans: 413-415 50.0:450.0> Ion: NA RIC: 2.881e+8
Pair Count: 204 MW: 0 Formula: None
CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI
Scan 1 Channel Description: 50.0:450.0> EI;Det 1000;WI Q1 Cal;ST 0.500
Scan Information: qp = 760000.0 mTorr
Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
51	475933	20	193	397250	17	294	366325	15
52	273617	11	194	379575	16	296	391775	16
63	581025	24	195	877000	37	299	330825	14
65	277517	12	197	455408	19	302	496458	21
66	274675	12	201	252842	11	304	533233	22
76	593158	25	203	1.352e+6	57	305	836100	35
77	1.837e+6	77	204	967191	41	306	666858	28
78	538908	23	205	877241	37	307	1.698e+6	71
87	262342	11	206	455308	19	308	546433	23
88	250150	11	207	582050	24	319	319783	13
89	641933	27	214	330467	14	320	302633	13
90	366217	15	216	397917	17	321	252900	11
91	424317	18	217	245025	10	322	498233	21
101	295100	12	218	1.696e+6	71	323	359142	15
102	626483	26	219	2.692e+6	113	325	330108	14
103	927950	39	220	2.379e+6	100	326	621858	26
104	3.183e+6	134	221	683425	29	327	260925	11
105	401158	17	222	1.044e+6	44	328	313892	13
109	240758	10	223	443225	19	330	385408	16
110	268542	11	226	384908	16	331	283883	12
113	245692	10	228	244717	10	332	451750	19
114	307508	13	229	370233	16	333	379075	16
115	477267	20	230	373525	16	334	559808	24
116	613633	26	231	664392	28	335	543683	23
117	525200	22	232	1.083e+6	46	336	242683	10
118	351958	15	233	1.350e+6	57	337	269908	11
119	296425	12	234	590008	25	344	447433	19
124	309783	13	235	286875	12	345	845192	36
125	269667	11	239	251333	11	346	605400	25
126	254358	11	241	384000	16	347	867542	36
127	325750	14	242	509217	21	348	820633	34
128	504833	21	243	338717	14	349	533433	22
129	366258	15	245	480058	20	350	1.080e+6	45

Scan 414 from d:\cz-13.xms - Page 2

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
130	8.804e+6	370	246	781300	33	351	536025	23
131	4.843e+6	203	247	3.019e+6	127	360	484508	20
132	5.081e+6	213	248	2.982e+6	125	361	288083	12
133	699692	29	249	1.190e+6	50	362	2.493e+6	105
134	375475	16	250	469575	20	363	1.067e+6	45
138	306433	13	251	329400	14	364	447033	19
139	677233	28	252	409450	17	365	372358	16
140	584575	25	253	339058	14	373	329042	14
141	431242	18	257	320717	13	376	625583	26
144	297992	13	258	465917	20	377	358025	15
146	895217	38	259	1.037e+6	44	378	835450	35
147	9.968e+6	419	260	528392	22	379	505042	21
148	1.252e+6	53	261	1.650e+6	69	388	259700	11
151	436808	18	262	2.674e+6	112	390	1.598e+6	67
152	818500	34	263	1.171e+7	492	391	678033	28
153	888733	37	264	3.525e+6	148	392	1.840e+6	77
159	350892	15	265	588992	25	393	1.910e+6	80
160	356467	15	268	459333	19	394	1.734e+6	73
161	311167	13	269	501950	21	395	477967	20
163	379483	16	273	1.252e+6	53	404	1.042e+6	44
164	522308	22	274	1.224e+6	51	405	2.952e+6	124
165	711917	30	275	4.253e+6	179	407	2.451e+6	103
166	635583	27	276	3.125e+6	131	408	1.767e+7	742
167	572750	24	277	1.576e+6	66	409	6.025e+6	253
168	583367	25	278	4.584e+6	193	410	1.092e+6	46
176	465000	20	279	3.020e+6	127	420	709750	30
177	509858	21	280	758692	32	421	337317	14
178	443375	19	281	1.493e+6	63	422	563858	24
179	353008	15	282	457200	19	433	408275	17
180	729592	31	288	367017	15	434	277425	12
181	761092	32	289	652083	27	435	365458	15
182	421942	18	290	7.486e+6	314	436	4.158e+6	175
190	800908	34	291	7.418e+6	312	437	2.378e+7	999
191	546717	23	292	6.073e+6	255	438	6.836e+6	287
192	503175	21	293	1.260e+6	53	439	1.217e+6	51

3,3-di(2-methyl-1*H*-indol-3-yl)-1-methylindolin-2-one (3l): Yield: 95 %; white solid, m. p. 291-293 °C (lit.^[26] 282-284 °C); IR (KBr, cm⁻¹): 3406, 3280, 3051, 1697, 1459, 1127, 1018, 742; ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.89 (s, 2H, NH), 7.34 (d, *J* = 7.7 Hz, 1H), 7.21 (m, 3H), 7.14 (d, *J* = 7.5 Hz, 1H), 6.96 (d, *J* = 8.0 Hz, 1H), 6.92-6.86 (m, 2H), 6.67-6.59 (m, 3H), 6.37 (d, *J* = 8.1 Hz, 1H), 3.24 (s, 3H, N-CH₃), 2.01 (s, 3H, CH₃), 1.93 (s, 3H, CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆): δ 177.3, 142.6, 134.9, 134.8, 134.5, 134.1, 131.8, 127.9, 125.1, 121.9, 119.8, 119.6, 119.2, 118.0, 117.9, 110.3, 110.2, 108.9, 108.5, 51.9, 26.2, 13.1, 12.9; MS (EI, 70 ev): *m/z* 405 (M⁺, 100); Anal. calcd. for C₂₇H₂₃N₃O: C, 79.97; H, 5.72; N, 10.36; found: C, 79.93; H, 5.75; N, 10.40%.

Scan 264 from d:\cz-16.xms

Spectrum from d:\cz-16.xms

Scan No: 264, Time: 2.692 minutes

3 points averaged. Not background corrected.

Comment: 2.692 min. Scans: 263-265 50.0:450.0> Ion: NA RIC: 1.397e+8

Pair Count: 160 MW: 0 Formula: None

CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:450.0> EI; Det 1000; WI Q1 Cal; ST 0.500

Scan Information: cp = 760000.0 mTorr

Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
51	412908	34	160	632408	52	263	129683	11
52	216508	18	165	657025	54	264	296700	24
53	128383	10	166	634133	52	269	197350	16
55	274067	22	167	217517	18	271	129350	11
57	413783	34	171	163692	13	273	4.864e+6	398
60	468892	38	171	357458	29	274	7.368e+6	602
61	144225	12	173	819683	67	275	2.661e+6	217
63	307825	25	174	717217	59	276	823950	67
64	158550	13	175	171708	14	277	191925	16
65	196208	16	176	238650	20	278	1.088e+6	89
66	143700	12	177	143992	12	279	712958	58
69	172925	14	178	179808	15	280	151275	12
71	130992	11	181	665858	54	281	338042	28
73	459708	38	182	220867	18	288	170092	14
75	205525	17	188	127133	10	291	163425	13
76	252158	21	189	182442	15	292	143042	12
77	993900	81	190	252475	21	296	199383	16
78	205733	17	195	139675	11	303	173742	14
81	147017	12	203	1.462e+6	119	304	276233	23
83	126367	10	204	684525	56	305	476867	39
84	138733	11	205	252450	21	306	266167	22
87	146483	12	209	298033	24	307	326108	27
88	137317	11	213	185325	15	317	143392	12
89	289792	24	215	148083	12	319	309458	25
90	231783	19	216	429983	35	320	204508	17
91	214875	18	217	882200	72	329	135142	11
97	257050	21	218	692650	57	330	333283	27
98	284150	23	219	302592	25	331	623425	51
101	235092	19	222	168133	14	332	728508	60
102	443917	36	229	229792	19	333	316533	26
103	682908	56	230	216100	18	334	136708	11
104	281092	23	231	658758	54	344	641167	52
113	139833	11	232	903417	74	345	994974	81

Scan 264 from d:\cz-16.xms - Page 2

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
114	152575	12	233	489742	40	346	1.940e+6	159
115	165025	13	234	237783	19	347	1.901e+6	155
117	201850	16	241	154942	13	348	737350	60
125	143442	12	242	141058	12	349	253267	21
126	179167	15	243	357625	29	360	317225	26
127	164775	13	244	348425	28	361	342825	28
128	355075	29	245	1.412e+6	115	362	1.217e+7	994
129	402400	33	246	2.143e+6	175	363	3.470e+6	284
130	5.731e+6	468	247	1.056e+6	86	364	672575	55
131	4.149e+6	339	248	370300	30	374	304117	25
132	609442	50	249	139808	11	376	1.520e+6	124
139	317700	26	251	147233	12	377	432983	35
140	242508	20	255	286200	23	390	1.013e+7	828
143	197508	16	256	231158	19	391	3.253e+6	266
144	471992	39	257	580492	47	392	567392	46
145	229108	19	258	507642	41	404	858233	70
152	280683	23	259	737850	60	405	1.222e+7	999
153	236092	19	260	207508	17	406	3.755e+6	307
154	164683	13	261	329567	27	407	898217	73
158	305317	25	262	204550	17	408	134867	11
159	331933	27

3,3-di(5-chloro-1H-indol-3-yl)indolin-2-one (3m): Yield: 89 %; white solid, m. p. 303°C; IR (KBr, cm⁻¹): 3421, 3258, 1708, 1465, 1100, 922, 750; ¹H NMR (500 MHz, DMSO-*d*₆) δ 11.23 (s, 2H, NH), 10.74 (s, 1H, NH), 7.41 (d, *J* = 8.0 Hz, 2H), 7.28 (m, 1H), 7.20 (d, *J* = 7.8 Hz, 3H), 7.06-6.98 (m, 4H), 6.94 (s, 2H); MS (EI, 70 ev): *m/z* 431 (*M*⁺, 87), 433 (*M*+2, 56), 435 (*M*+4, 11); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 178.4, 141.2, 135.4, 133.6, 128.2, 126.6, 126.1, 124.8, 122.9, 121.8, 121.1, 119.6, 113.9, 113.3, 109.8, 52.1; Anal. calcd. for C₂₄H₁₅Cl₂N₃O: C, 66.68; H, 3.50; N, 16.40; found: C, 66.63; H, 3.53; N, 16.45%.

Scan 369 from d:\cz-17.xms

Spectrum from d:\cz-17.xms
 Scan No: 369, Time: 3.635 minutes
 3 points averaged. Not background corrected.
 Comment: 3.635 min. Scans: 368-370 50.0:450.0> Ion: NA RIC: 1.765e+8
 Pair Count: 184 MW: 0 Formula: None
 CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI
 Scan 1 Channel Description: 50.0:450.0> EI; Det 1000; WI Q1 Cal; ST 0.500
 Scan Information: cp = 760000.0 mTorr
 Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
63	117733	10	229	230150	20	335	3.150e+6	268
77	125008	11	231	267283	23	336	452467	39
84	763033	65	232	372525	32	339	236500	20
86	451508	38	233	145792	12	340	151525	13
88	185250	16	237	215108	18	341	155092	13
89	717617	61	238	129017	11	342	176250	15
90	692367	59	242	296850	25	344	379442	32
101	118142	10	243	192758	16	345	335083	29
116	375675	32	245	910833	78	346	420167	36
117	2.538e+6	216	246	641125	55	347	397167	34
118	316042	27	247	2.342e+6	199	348	239908	20
123	213350	18	248	590483	50	350	161383	14
124	153600	13	249	179850	15	351	184492	16
125	175442	15	250	160817	14	352	708267	60
126	167283	14	251	266183	23	353	319508	27
130	231067	20	252	1.186e+6	101	354	186675	16
131	221008	19	253	6.511e+6	554	360	257742	22
132	152183	13	254	1.454e+6	124	361	166558	14
138	204242	17	255	2.021e+6	172	362	3.947e+6	336
139	309375	26	256	435967	37	363	1.112e+7	947
145	277742	24	263	204183	17	364	3.134e+6	267
146	188542	16	265	256825	22	365	750383	64
147	145575	12	266	144758	12	366	1.082e+6	92
150	169608	14	274	191600	16	367	908392	77
151	1.444e+6	123	275	494217	42	368	765275	65
152	779600	66	276	234017	20	369	418333	36
153	789750	67	277	360642	31	370	170775	15
154	161108	14	278	537208	46	373	279575	24
159	769675	66	279	334975	29	374	171167	15
160	161217	14	280	434692	37	375	224917	19
163	191608	16	281	2.082e+6	177	376	167425	14
164	132467	11	282	879942	75	379	1.029e+6	88
165	435708	37	283	710800	60	381	355958	30

Scan 369 from d:\cz-17.xms - Page 2

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
166	1.144e+6	97	284	231667	20	382	184292	16
167	335333	29	289	149633	13	387	179358	15
172	186325	16	290	167775	14	389	226450	19
173	196883	17	291	245192	21	390	400400	34
175	118358	10	292	149083	13	391	2.543e+6	216
176	199625	17	293	139417	12	392	806875	69
177	159442	14	302	300408	26	394	275567	23
178	127183	11	303	610900	52	395	142083	12
180	356108	30	304	538667	46	396	509867	43
181	298567	25	305	658033	56	397	183458	16
184	282467	24	306	484467	41	398	175133	15
187	167017	14	307	321475	27	400	136825	12
188	151817	13	313	312233	27	402	1.058e+7	900
189	324575	28	314	140642	12	403	2.995e+6	255
190	800208	68	315	266058	23	404	7.347e+6	625
191	238733	20	316	407475	35	405	2.059e+6	175
198	187383	16	317	503825	43	406	1.558e+6	133
199	128867	11	318	467825	40	407	1.030e+6	88
201	132892	11	319	521442	44	408	988042	84
203	134250	11	325	121958	10	409	440192	37
204	197042	17	326	145633	12	414	169808	14
216	722033	61	328	179767	15	430	1.366e+6	116
217	664383	57	329	220758	19	431	1.018e+7	867
218	2.414e+6	205	330	571442	49	432	3.603e+6	307
219	3.658e+6	311	331	1.580e+6	134	433	6.583e+6	560
220	691408	59	332	1.635e+6	139	434	1.890e+6	161
224	155792	13	333	1.091e+6	93	435	1.293e+6	110
225	121717	10	334	1.174e+7	999	436	349050	30
228	128217	11

3,3-di(1-methyl-1*H*-indol-3-yl)-5-iodine-indolin-2-one (3n): Yield: 86 %; white solid, m. p. 252°C; IR (KBr, cm⁻¹): 3222, 1715, 1470, 1157, 862, 733; ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.77 (s, 1H, NH), 7.59 (d, *J* = 8.0 Hz, 1H), 7.46 (m, 1H), 7.40 (d, *J* = 8.2 Hz, 2H), 7.21 (m, 2H), 7.12-7.09 (m, 2H), 6.93 (s, 2H), 6.88-6.85 (m, 3H), 3.73 (s, 6H, N-CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 177.8, 141.0, 137.3, 137.0, 136.5, 132.8, 128.5, 125.7, 121.2, 120.7, 118.5, 112.7, 112.2, 109.9, 84.4, 52.4, 32.3; MS (EI, 70 ev): *m/z* 517 (M⁺, 100); Anal. calcd. for C₂₆H₂₀IN₃O: C, 60.36; H, 3.90; N, 8.12; found: C, 60.42; H, 3.94; N, 8.16%.

Scan 490 from d:\cz-18.xms

Spectrum from d:\cz-18.xms

Scan No: 490, Time: 3.882 minutes

3 points averaged. Not background corrected.

Comment: 3.882 min. Scans: 489-491 50.0:550.0> Ion: NA RIC: 4.758e+7

Pair Count: 93 MW: 0 Formula: None

CAS No: None Acquired Range: 50 - 550 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:550.0> EI;Det 1000;WI Q1 Cal;ST 0.500

Scan Information: cp = 760000.0 mTorr

Precursor Mass Range: 50 - 550 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
131	102027	14	312	122700	16	391	193513	26
147	111733	15	313	110133	15	396	257567	34
151	117587	16	316	204953	27	397	506067	68
158	86100	12	317	195893	26	398	183200	25
166	336313	45	325	83880	11	399	234800	31
171	131240	18	329	157027	21	400	133213	18
172	168207	23	330	221367	30	407	91327	12
173	261267	35	331	194220	26	408	80227	11
174	112713	15	332	84460	11	425	102307	14
181	79680	11	333	102013	14	426	117013	16
184	127607	17	334	504060	67	429	191007	26
187	94407	13	335	136067	18	437	106893	14
190	100073	13	344	222900	30	438	92507	12
195	136520	18	345	492033	66	439	91200	12
204	91733	12	346	312747	42	457	167047	22
215	103887	14	347	95687	13	458	808727	108
216	89807	12	358	313693	42	459	254320	34
217	94907	13	359	1.071e+6	143	466	309580	41
219	94520	13	360	660333	88	467	106933	14
231	242507	32	361	1.537e+6	206	468	143180	19
232	488947	65	362	733060	98	472	92653	12
233	91593	12	363	610513	82	473	177100	24
247	120667	16	364	152820	20	474	179473	24
250	158413	21	368	376373	50	487	88047	12
251	130213	17	370	150347	20	488	6.730e+6	901
253	79907	11	372	86053	12	489	2.129e+6	285
259	305480	41	386	134000	18	490	334533	45
259	252933	34	387	830607	111	516	372647	50
260	201487	27	388	299667	40	517	7.461e+6	999
296	76547	10	389	84213	11	518	2.176e+6	291
305	85300	11	390	409847	55	519	286253	38

3,3-di(5-methoxyl-1H-indol-3-yl)-5-methoxylindolin-2-one (3o): Yield: 94 %; white solid, m. p. 261 °C; IR (KBr, cm⁻¹): 3398, 3313, 1705, 1483, 1193, 1024, 774; ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.81 (s, 2H, NH), 10.45 (s, 1H, NH), 7.27 (d, *J* = 8.5 Hz, 2H), 6.93 (d, *J* = 8.4 Hz, 1H), 6.88 (s, 2H), 6.83 (m, 1H), 6.77 (d, *J* = 2.5 Hz, 1H), 6.72 (d, *J* = 2.3 Hz, 1H), 6.70 (s, 3H), 3.63 (s, 3H, O-CH₃), 3.54 (s, 6H, O-CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 178.6, 154.6, 152.4, 135.8, 134.8, 132.1, 126.0, 125.2, 113.6, 112.2, 112.1, 112.0, 110.4, 109.7, 103.3, 55.3, 55.1, 52.9; MS (EI, 70 ev): *m/z* 453 (*M*⁺, 2); Anal. calcd. for C₂₇H₂₃N₃O₄: C, 71.51; H, 5.11; N, 9.27; found: C, 71.60; H, 5.08; N, 9.23%.

Scan 514 from d:\cz-23.xms

Spectrum from d:\cz-23.xms

Scan No: 514, Time: 3.534 minutes

3 points averaged. Not background corrected.

Comment: 3.534 min. Scans: 513-515 50.0:500.0> Ion: NA RIC: 1.409e+8

Pair Count: 44 MW: 0 Formula: None

CAS No: None Acquired Range: 50 - 500 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:500.0> EI;Det 1000;WI Q1 Cal;ST 0.500

Scan Information: qp = 760000.0 mTorr

Precursor Mass Range: 50 - 500 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
51	366033	10	116	815547	23	277	514273	15
63	645687	18	117	905253	26	279	3.721e+6	106
74	379193	11	118	593593	17	280	596680	17
75	452180	13	131	595780	17	292	809020	23
76	1.212e+6	34	132	1.980e+7	562	293	971407	28
77	2.693e+6	76	133	2.126e+6	60	306	799513	23
78	1.360e+6	39	146	1.393e+6	40	307	2.753e+6	78
84	1.279e+6	36	147	3.522e+7	999	308	8.932e+6	253
86	1.004e+6	28	148	3.739e+6	106	309	1.664e+6	47
89	1.148e+6	33	193	413660	12	310	1.172e+6	33
90	408280	12	236	661840	19	311	886153	25
91	373273	11	249	481767	14	334	406013	12
103	1.293e+6	37	263	429673	12	363	382293	11
104	1.362e+7	386	264	914107	26	453	492240	14
105	1.140e+6	32	265	659080	19

3,3-di(2-methyl-1H-indol-3-yl)-5-methyl-indolin-2-one (3p): Yield: 88 %; white solid, m. p. 251-252 °C (lit.^[16] 239-240 °C); IR (KBr, cm⁻¹): 3408, 3388, 1711, 1456, 1019, 749; ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.85 (s, 2H, NH), 10.42 (s, 1H, NH), 7.22 (d, *J* = 8.1 Hz, 2H), 7.03 (d, *J* = 7.9 Hz, 1H), 6.95-6.90 (m, 2H), 6.89-6.87 (m, 1H), 6.84 (d, *J* = 7.9 Hz, 1H), 6.70 (d, *J* = 8.0 Hz, 1H), 6.63 (m, 2H), 6.48 (d, *J* = 8.1 Hz, 1H), 2.13 (s, 3H, CH₃), 2.06 (s, 3H, CH₃), 1.93 (s, 3H, CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 179.2, 138.8, 135.6, 134.9, 134.8, 133.8, 132.0, 129.8, 127.9, 127.7, 127.1, 126.0, 119.7, 117.8, 119.5, 119.3, 117.9, 117.8, 110.5, 110.3, 109.5, 109.1, 52.4, 20.8, 13.1, 13.0; MS (EI, 70 ev): *m/z* 405 (M⁺, 39); Anal. calcd. for C₂₇H₂₃N₃O: C, 79.97; H, 5.72; N, 10.36; found: C, 79.89; H, 5.69; N, 10.32%.

Scan 684 from d:\cz-04.xms

Spectrum from d:\cz-04.xms
Scan No: 684, Time: 4.780 minutes
3 points averaged. Not background corrected.
Comment: 4.780 min. Scans: 683-685 50.0:500.0> Ion: NA RIC: 5.103e+7
Pair Count: 79 MW: 0 Formula: None
CAS No: None Acquired Range: 50 - 500 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:500.0> EI; Det 1000; WI Q1 Cal; ST 0.500

Scan Information: cp = 760000.0 mTorr

Precursor Mass Range: 50 - 500 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
64	90727	13	217	196940	28	289	88113	13
77	248560	35	218	315420	45	305	87480	12
89	160720	23	219	99400	14	330	85680	12
98	138180	20	229	136000	19	332	172653	25
100	71613	10	230	247667	35	333	139967	20
102	108660	16	231	447173	64	334	83813	12
103	304713	43	233	1.218e+6	174	344	233787	33
104	99247	14	234	254253	36	345	108433	15
116	79587	11	242	80067	11	346	302347	43
127	75593	11	243	171840	25	347	177933	25
128	199133	28	244	89493	13	359	101033	14
130	3.274e+6	467	245	216367	31	360	1.459e+6	208
131	1.965e+6	280	247	746080	106	361	874013	125
132	168493	24	248	286747	41	362	7.001e+6	999
136	78427	11	252	76727	11	363	2.161e+6	308
144	108433	15	255	74620	11	364	282200	40
151	123960	18	256	71633	10	374	80953	12
158	154047	22	259	129093	18	376	347133	50
165	222560	32	260	117220	17	377	151773	22
166	118933	17	261	1.107e+6	158	389	167207	24
171	110633	16	262	227427	32	390	5.658e+6	807
173	304080	43	272	133460	19	391	1.557e+6	222
173	105513	15	274	365253	52	392	289607	41
181	138827	20	275	1.044e+6	149	404	329160	47
203	95040	14	276	1.724e+6	246	405	2.721e+6	388
204	123647	18	277	423053	60	406	789907	113
216	111780	16

2,2-di(1*H*-indol-3-yl)-2*H*-acenaphthylen-1-one (5a): Yield: 89 %; yellow solid, m. p. 282°C (lit.^[31] 289-290°C); IR (KBr, cm⁻¹): 3415, 3357, 3120, 1683, 1414, 1101, 751; ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.99 (s, 2H, NH), 8.36 (d, *J* = 8.1 Hz, 1H), 8.01 (d, *J* = 8.0 Hz, 2H), 7.91 (t, *J* = 7.5 Hz, 1H), 7.69-7.55 (m, 2H), 7.35 (d, *J* = 7.3 Hz, 2H), 7.01 (m, 4H), 6.85 (s, 2H), 6.75 (m, 2H); ¹³C NMR (125 MHz, DMSO-*d*₆): δ 202.6, 143.9, 139.6, 137.0, 132.0, 131.5, 130.5, 129.0, 128.8, 125.8, 124.6, 124.1, 122.2, 121.7, 121.0, 120.6, 118.3, 114.9, 111.7, 57.7; MS (EI, 70 ev): *m/z* 398 (M⁺, 98); Anal. calcd. for C₂₈H₁₈N₂O: C, 84.40; H, 4.55; N, 7.03; found: C, 84.49; H, 4.58; N, 7.00%.

Scan 417 from d:\hjs-01.xms

Spectrum from d:\hjs-01.xms
 Scan No: 417, Time: 3.893 minutes
 3 points averaged. Not background corrected.
 Comment: 3.893 min. Scans: 416-418 50.0:450.0> Ion: NA RIC: 9.657e+6
 Pair Count: 77 MW: 0 Formula: None
 CAS No: None Acquired Range: 50 - 450 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:450.0> EI;Det 1000;WI Q1 Cal;ST 0.500
 Scan Information: cp = 760000.0 mTorr
 Precursor Mass Range: 50 - 450 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
108	15583	10	255	49492	32	352	115208	74
117	103442	67	265	22208	14	353	64583	42
154	16392	11	268	15767	10	354	51458	33
157	24767	16	278	46042	30	362	43292	28
163	24858	16	281	93333	60	363	120242	78
169	29850	19	282	361817	233	365	42792	28
170	24308	16	283	62192	40	366	101900	66
181	21967	14	292	20517	13	367	375342	242
182	16658	11	293	22292	14	368	115850	75
184	69867	45	302	18825	12	369	1.549e+6	999
185	49308	32	313	20108	13	370	813467	525
190	43617	28	314	22383	14	371	162575	105
199	39842	26	316	23350	15	372	20392	13
199	27450	18	325	18858	12	379	33400	22
218	41408	27	326	17200	11	380	61733	40
219	27800	18	329	17667	11	381	67433	43
224	17175	11	332	23992	15	382	26800	17
226	42092	27	334	49192	32	382	17050	11
231	16675	11	335	31625	20	384	15675	10
237	18475	12	338	45325	29	395	29942	19
240	15533	10	339	39692	26	398	1.512e+6	975
242	18150	12	341	151642	98	399	496033	320
246	18425	12	342	63158	41	400	52908	34
249	17500	11	343	25975	17	404	18617	12
251	35642	23	350	16083	10	409	15983	10
254	211175	136	351	39908	26

2,2-di(5-chloro-1H-indol-3-yl)-2H-acenaphthylen-1-one (5b): Yield: 86 %; yellow solid, m. p. 315°C; IR (KBr, cm⁻¹): 3431, 3314, 1707, 1460, 1096, 765; ¹H NMR (500 MHz, DMSO-*d*₆) δ 11.26 (s, 2H, NH), 8.40 (d, *J* = 8.0 Hz, 1H), 8.05 (m, 2H), 7.94-7.90 (m, 1H), 7.74 (m, 1H), 7.55 (d, *J* = 8.0 Hz, 1H), 7.40 (d, *J* = 8.6 Hz, 2H), 7.03 (dd, *J* = 8.6, 2.1 Hz, 2H), 6.98 (d, *J* = 2.0 Hz, 2H), 6.95 (s, 2H); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 202.3, 142.9, 139.5, 135.0, 132.4, 131.0, 130.5, 129.1, 129.0, 126.7, 126.4, 124.4, 123.0, 122.6, 121.7, 121.2, 119.4, 114.4, 113.4, 57.2; MS (EI, 70 ev): *m/z* 466 (*M*⁺, 38), 468 (*M*+2, 25); 470 (*M*+4, 3); Anal. calcd. for C₂₈H₁₆Cl₂N₂O: C, 71.96; H, 3.45; N, 5.99; found: C, 72.02; H, 3.49; N, 6.03%.

Scan 639 from d:\hjs-03.xms

Spectrum from d:\hjs-03.xms
 Scan No: 639, Time: 4.932 minutes
 3 points averaged. Not background corrected.
 Comment: 4.932 min. Scans: 638-640 50.0:550.0> Ion: NA RIC: 1.333e+8
 Pair Count: 155 MW: 0 Formula: None
 CAS No: None Acquired Range: 50 - 550 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:550.0> EI; Det 1000; W1 Q1 Cal; ST 0.500
 Scan Information: cp = 760000.0 mTorr
 Precursor Mass Range: 50 - 550 m/z

Ion			Ion			Ion		
Int	Norm		Int	Norm		Int	Norm	
75	266940	23	191	220380	19	296	1.590e+6	135
76	223933	19	194	393727	33	297	3.021e+6	256
77	189553	16	195	692160	59	298	638427	54
85	149753	13	196	193947	16	299	647207	55
86	244907	21	197	162107	14	309	130507	11
87	192413	16	198	126247	11	312	136000	12
88	387547	33	199	128447	11	315	312220	26
89	430707	37	201	204360	17	316	1.659e+6	141
90	303113	26	202	134700	11	317	803660	68
100	172027	15	203	137167	12	318	570167	48
101	208760	18	207	148027	13	319	234687	20
102	287260	24	208	272800	23	324	593653	50
104	122600	10	209	2.227e+6	189	325	1.177e+7	999
113	172320	15	210	2.452e+6	208	326	2.283e+6	194
114	297400	25	211	2.262e+6	192	327	824220	70
115	236387	20	212	524640	45	334	791447	67
116	1.567e+6	133	215	120893	10	335	184693	16
117	771960	66	216	120213	10	338	257687	22
118	456300	39	219	393813	33	362	262793	22
126	374633	32	223	981147	83	363	1.156e+6	98
127	465647	40	224	427447	36	364	261880	22
128	572113	49	225	269460	23	365	128260	11
129	283913	24	226	354400	30	366	472460	40
130	638087	54	227	132773	11	367	524880	45
131	217427	18	237	708960	60	368	636953	54
134	137400	12	238	4.868e+6	413	370	121520	10
139	192400	16	239	955867	81	391	118880	10
140	345213	29	240	288967	25	397	762373	65
141	288960	25	245	159760	14	398	334000	28
142	344633	29	250	148760	13	399	315067	27
143	983173	83	251	599587	51	400	152893	13
144	471020	40	252	357213	30	401	764900	65
146	271240	23	253	1.028e+6	87	402	385593	33

Scan 639 from d:\hjs-03.xms - Page 2

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
147	124140	11	254	1.074e+6	91	403	1.965e+6	167
151	567640	48	255	386627	33	404	610727	52
152	193887	16	259	8.185e+6	695	405	593493	50
153	895080	76	260	1.263e+6	107	406	289127	25
154	867040	74	261	475647	40	407	131847	11
155	1.476e+6	125	262	124887	11	426	209320	18
156	222393	19	265	118527	10	437	2.329e+6	198
158	393473	33	266	2.794e+6	237	438	1.277e+6	108
166	172113	15	267	723693	61	439	1.703e+6	145
167	260847	22	268	1.329e+6	113	440	760573	65
168	339020	29	269	328467	28	441	254993	22
169	327567	28	280	153713	13	449	127387	11
171	287260	24	282	1.258e+6	107	465	921173	78
172	2.950e+6	250	283	334160	28	466	4.512e+6	383
173	467593	40	287	506693	43	467	1.376e+6	117
180	393893	33	288	779353	66	468	2.996e+6	254
183	439447	37	289	298307	25	469	930920	79
184	2.059e+6	175	290	335620	28	470	366240	31
185	291167	25	292	135820	12

2,2-di(5-methoxyl-1H-indol-3-yl)-2H-acenaphthylen-1-one (5c): Yield: 92 %; yellow solid, m. p. 302-304°C (lit.^[31] >300°C); IR (KBr, cm⁻¹): 3396, 1698, 1483, 1174, 1027, 780; ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.85 (s, 2H, NH), 8.37 (d, *J* = 7.5 Hz, 1H), 8.02-7.90 (m, 3H), 7.71 (m, 1H), 7.55 (d, *J* = 7.4 Hz, 1H), 7.25 (d, *J* = 8.6 Hz, 2H), 6.87 (s, 2H), 6.68 (dd, *J* = 8.8, 2.5 Hz, 2H), 6.42 (d, *J* = 2.2 Hz, 2H), 3.40 (s, 6H, O-CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆): δ 202.8, 152.5, 147.5, 143.8, 139.7, 137.5, 132.2, 131.7, 129.0, 128.8, 126.2, 125.4, 122.0, 121.7, 114.2, 112.1, 110.4, 103.2, 57.6, 54.9; MS (EI, 70 ev): *m/z* 458 (*M*⁺, 100); Anal. calcd. for C₃₀H₂₂N₂O₃: C, 78.59; H, 4.84; N, 6.11; found: C, 78.65; H, 4.88; N, 6.07%.

Scan 502 from d:\hjs-04.xms

Spectrum from d:\hjs-04.xms
Scan No: 502, Time: 3.952 minutes
3 points averaged. Not background corrected.
Comment: 3.952 min. Scans: 501-503 50.0:550.0> Ion: NA RIC: 2.175e+7
Pair Count: 211 MW: 0 Formula: None
CAS No: None Acquired Range: 50 - 550 m/z

Method Description: EI
Scan 1 Channel Description: 50.0:550.0> EI; Det 1000; WI Q1 Cal; ST 0.500
Scan Information: cp = 760000.0 mTorr
Precursor Mass Range: 50 - 550 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
70	24820	15	212	31913	19	337	47020	28
77	55800	33	213	177273	106	339	101107	61
78	39553	24	214	41467	25	340	61000	37
84	25593	15	215	42073	25	342	70600	42
89	28813	17	217	29860	18	343	46887	28
92	21613	13	218	33373	20	345	20180	12
100	20740	12	219	41153	25	347	35420	21
101	25993	16	220	37780	23	350	31593	19
104	38500	23	221	25153	15	352	56300	34
107	21493	13	222	24673	15	353	83967	50
112	51380	31	223	43460	26	354	46620	28
114	19660	12	226	38093	23	355	59960	36
117	58827	35	235	18940	11	356	32427	19
118	31960	19	237	67207	40	360	25500	15
119	21300	13	238	52293	31	361	19460	12
124	52547	32	240	85987	52	362	75107	45
126	26400	16	241	76567	46	363	239913	144
128	53393	32	244	21193	13	364	106220	64
130	41853	25	245	32540	20	365	33947	20
131	41593	25	247	29213	18	366	72387	43
133	26880	16	248	30940	19	368	235907	142
138	21447	13	250	95173	57	370	58620	35
140	18727	11	251	91407	55	370	74007	44
142	22547	14	252	101713	61	371	47940	29
143	30527	18	253	186193	112	376	29907	18
144	68233	41	254	25787	15	377	17887	11
145	39800	24	255	61740	37	379	47673	29
146	32073	19	256	24467	15	380	41427	25
147	212040	127	261	41253	25	381	229580	138
151	32100	19	265	24867	15	383	306473	184
153	22333	13	266	96060	58	384	148893	89
155	21360	13	267	177187	106	385	52840	32
156	45260	27	268	402940	242	386	20627	12

Scan 502 from d:\hjs-04.xms - Page 2

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
158	98093	59	269	80727	48	387	24933	15
162	25247	15	270	24700	15	391	43487	26
163	23107	14	274	32173	19	392	48433	29
164	48653	29	275	43087	26	393	19613	12
165	26073	16	276	19640	12	395	44327	27
166	36887	22	277	24933	15	396	57247	34
168	19080	11	278	17420	10	397	1.315e+6	789
170	58013	35	280	34120	20	398	669500	402
171	44927	27	281	49687	30	399	513300	308
172	63027	38	282	28187	17	400	153693	92
174	46233	28	284	30020	18	401	39773	24
175	22707	14	284	51213	31	404	30133	18
175	51500	31	290	33633	20	405	35767	21
176	88107	53	292	59767	36	407	58920	35
177	86620	52	295	42240	25	408	28340	17
180	31627	19	296	437227	262	409	27253	16
182	58247	35	297	134107	80	411	110380	66
183	52440	31	299	25633	15	413	23680	14
185	39000	23	301	21080	13	414	47447	28
186	21267	13	302	30293	18	415	157667	95
187	27380	16	304	21993	13	416	39693	24
188	27053	16	306	45900	28	425	219933	132
189	24260	15	308	19273	12	426	1.462e+6	877
190	16687	10	311	112007	67	427	648207	389
191	101033	61	312	377827	227	429	409567	246
192	27347	16	313	134753	81	430	295993	178
194	25300	15	314	18320	11	431	85173	51
196	49633	30	315	74340	45	433	27873	17
197	43207	26	319	21913	13	441	43047	26
198	79087	47	324	32673	20	442	33547	20
199	56793	34	325	254700	153	442	31720	19
201	27533	17	327	44247	27	443	37093	22
202	20627	12	329	43460	26	456	21020	13
204	20147	12	331	62760	38	457	366513	220
205	20373	12	334	117087	70	458	1.665e+6	999
207	80027	48	335	90720	54	459	539800	324
210	77700	47	336	22340	13	460	79253	48
211	24980	15

2,2-di(2-methyl-1H-indol-3-yl)-2H-acenaphthylen-1-one (5d): Yield: 90 %; brown solid, m. p. 285- 287°C (lit.^[31] 292-293 °C); IR (KBr, cm⁻¹): 3392, 3048, 1706, 1490, 1012, 740; ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.91 (s, 2H, NH), 8.34 (d, *J* = 8.1 Hz, 1H), 8.02 (m, 2H), 7.87-7.65 (m, 2H), 7.43 (m, 1H), 7.23-7.19 (m, 2H), 6.90-6.84 (m, 2H), 6.61-6.51 (m, 3H), 6.33 (d, *J* = 8.0 Hz, 1H), 1.84 (s, 3H, CH₃), 1.79 (s, 3H, CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆): δ 202.0, 144.0, 140.0, 135.0, 134.9, 133.6, 133.3, 132.7, 131.6, 130.3, 128.9, 128.7, 127.6, 127.3, 124.0, 122.2, 121.8, 119.7, 119.6, 119.5, 119.4, 118.0, 117.9, 110.9, 110.3, 110.1, 57.5, 13.4, 13.3; MS (EI, 70 ev): *m/z* 426 (M⁺, 37); Anal. calcd. for C₃₀H₂₂N₂O: C, 84.48; H, 5.20; N, 6.57; found: C, 84.54; H, 5.18; N, 6.61%.

Scan 436 from d:\hjs-05.xms

Spectrum from d:\hjs-05.xms
Scan No: 436, Time: 3.323 minutes
3 points averaged. Not background corrected.
Comment: 3.323 min. Scans: 435-437 50.0:550.0> Ion: NA RIC: 3.039e+7
Pair Count: 130 MW: 0 Formula: None
CAS No: None Acquired Range: 50 - 550 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:550.0> EI;Det 1000;WI Q1 Cal;ST 0.500
Scan Information: cp = 760000.0 mTorr
Precursor Mass Range: 50 - 550 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
64	33240	11	191	69980	23	298	141580	46
76	38287	12	193	61933	20	299	40953	13
77	165307	54	195	72347	23	315	35460	11
78	34000	11	196	75667	24	323	40240	13
86	104360	34	197	46280	15	325	612053	198
89	62207	20	200	44060	14	326	128987	42
103	89967	29	205	54107	18	332	34867	11
104	39347	13	209	43260	14	334	187580	61
113	60693	20	210	92733	30	335	62173	20
115	35927	12	211	116533	38	338	42433	14
116	66353	21	213	59167	19	339	56160	18
117	51540	17	216	35553	12	341	220420	71
118	36847	12	219	37707	12	342	42367	14
121	31160	10	224	71800	23	353	33927	11
126	113133	37	225	63320	20	356	40273	13
127	69120	22	226	164900	53	362	70400	23
128	47660	15	227	42840	14	363	157760	51
129	127360	41	238	255113	83	364	84447	27
130	3.087e+6	999	239	95567	31	365	147100	48
131	2.726e+6	882	251	252927	82	368	281767	91
132	288527	93	252	84287	27	369	117147	38
133	104513	34	253	211560	68	370	99473	32
140	70880	23	254	382767	124	371	32460	11
143	46900	15	255	91760	30	378	55527	18
144	54947	18	257	62827	20	380	189193	61
145	58173	19	259	81273	26	381	186200	60
152	67780	22	263	34687	11	383	2.047e+6	663
153	64300	21	264	67533	22	384	527440	171
154	236087	76	266	115480	37	385	143113	46
155	64440	21	267	283160	92	395	50347	16
156	39780	13	268	408627	132	396	48760	16
158	77973	25	269	138820	45	397	285747	92
162	46073	15	270	51960	17	398	51520	17

Scan 436 from d:\hjs-05.xms - Page 2

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
163	44800	14	274	53873	17	399	37387	12
168	43887	14	278	50507	16	407	90193	29
169	59820	19	279	53973	17	409	128840	42
170	45360	15	280	76640	25	410	63160	20
176	71760	23	282	439020	142	411	2.769e+6	896
177	50680	16	283	115087	37	412	844733	273
180	32380	10	290	36973	12	413	161413	52
182	87453	28	294	82013	27	426	1.128e+6	365
184	210500	68	296	700960	227	427	607500	197
189	51447	17	297	954607	309	428	91193	30
190	121100	39

2,2-di(1-methyl-1H-indol-3-yl)-2H-acenaphthylen-1-one (5e): Yield: 83 %; brown yellow solid, m. p. 297-298°C (lit.^[31] >300°C); IR (KBr, cm⁻¹): 3417, 3041, 1721, 1464, 1074, 740; ¹H NMR (500 MHz, DMSO-*d*₆) δ 8.37 (d, *J* = 8.1 Hz, 1H), 8.01-7.90 (m, 3H), 7.70 (m, 1H), 7.56 (d, *J* = 6.9 Hz, 1H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.09-7.02 (m, 4H), 6.88 (s, 2H), 6.79 (t, *J* = 7.0 Hz, 2H), 3.68 (s, 6H, N-CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆): δ 202.4, 143.7, 137.4, 132.1, 131.4, 130.5, 129.0, 128.8, 128.7, 126.1, 124.1, 122.3, 121.7, 121.1, 120.7, 118.5, 113.9, 109.8, 57.4, 32.3; MS (EI, 70 ev): *m/z* 426 (M⁺, 100); Anal. calcd. for C₃₀H₂₂N₂O: C, 84.48; H, 5.20; N, 6.57; found: C, 84.41; H, 5.23; N, 6.61%.

Scan 412 from d:\hjs-06.xms

Spectrum from d:\hjs-06.xms
 Scan No: 412, Time: 3.276 minutes
 3 points averaged. Not background corrected.
 Comment: 3.276 min. Scans: 411-413 50.0:550.0> Ion: NA RIC: 3.704e+7
 Pair Count: 59 MW: 0 Formula: None
 CAS No: None Acquired Range: 50 - 550 m/z

Method Description: EI

Scan 1 Channel Description: 50.0:550.0> EI;Det 1000;WI Q1 Cal;ST 0.500
 Scan Information: cp = 760000.0 mTorr
 Precursor Mass Range: 50 - 550 m/z

Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
77	137207	22	266	225387	36	380	120773	19
90	77767	13	267	664807	107	381	892773	144
130	315420	51	268	1.181e+6	190	382	440660	71
131	231873	37	269	255287	41	383	1.121e+6	180
169	117933	19	292	106447	17	384	440513	71
177	63080	10	295	147433	24	395	257553	41
183	119547	19	296	1.560e+6	251	396	365393	59
184	156453	25	297	567293	91	397	4.810e+6	773
190	105980	17	298	112233	18	398	2.189e+6	352
191	122233	20	338	81847	13	399	582873	94
198	92900	15	341	75587	12	400	78267	13
213	180600	29	351	76167	12	411	424980	68
240	62513	10	354	87040	14	412	134180	22
250	83000	13	363	105987	17	423	62420	10
251	145647	23	365	149400	24	424	240413	39
252	163687	26	366	597913	96	425	1.016e+6	163
253	640340	103	367	229680	37	426	6.214e+6	999
254	108767	17	368	131427	21	427	2.019e+6	325
255	93153	15	369	79247	13	428	330860	53
265	75347	12	378	62333	10

D:\axx52\axx.25 HJS-06 sample form

2013/05/28