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SYNTHESIS OF PENTACYCLIC HETEROAROMATIC SYSTEMS RELATED TO INDOLOCARBAZOLES ALKALOIDS.

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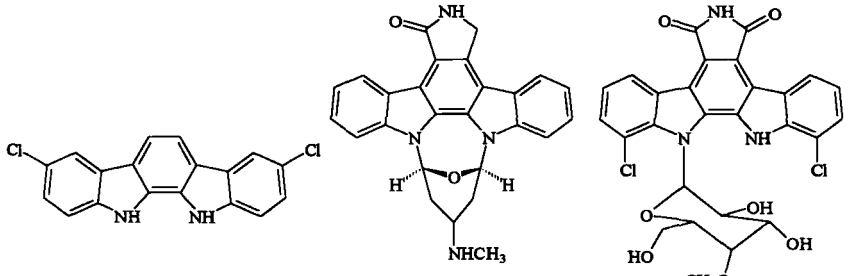
Abstract: A direct access to pentacyclic molecules via Fischer Indole Synthesis starting from tricyclic ketones is described.

Introduction: Indolocarbazole alkaloids are a structurally rare but biologically interesting class of natural compounds[1]. Tjipanazole **1**, Staurosporine **2**, Rebeccamycine **3** as well as Staurosporinone, Arcryaflavin and their derivatives have been extensively studied, due to a large variety of biological activities such as

antifungal[2], inhibition of protein kinase C[3] and platelet aggregation[4], antitumor[2] and antimicrobial[5].



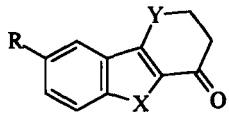
1 2 3

The indolo[2,3-a]carbazole system constitutes the framework of such natural products. Many studies toward the synthesis of indolocarbazole have been made in recent years[6-11]. No examples of replacement of one carbazole nitrogen in these compounds by other heteroatoms like sulfur or selenium have been described. As a part of our contribution to polycyclic polyheteroaromatic systems with potential biological activities[12-15], we describe here an easy access to pentacyclic molecules via Fischer Indole Synthesis starting from tricyclic ketones.

Starting ketones are derived from oxotetrahydrocarbazole, dibenzothiophene and dibenzoselenophene and have been prepared as described[16-19] previously.

Starting ketones used in this study are listed in table 1 and 2.

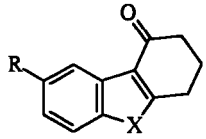
Table 1:



5

Compound	*X*	*R*	*Y*
5a	**NH**	**H**	**CH_2**
5b	**NH**	**OCH_3**	**CH_2**
5c	**NH**	**OCH_3**	**CH-CH_3**
5d	**NH**	**Br**	**CH_2**
5e	**S**	**H**	**CH_2**
5f	**O**	**OCH_3**	**S**

Table 2:

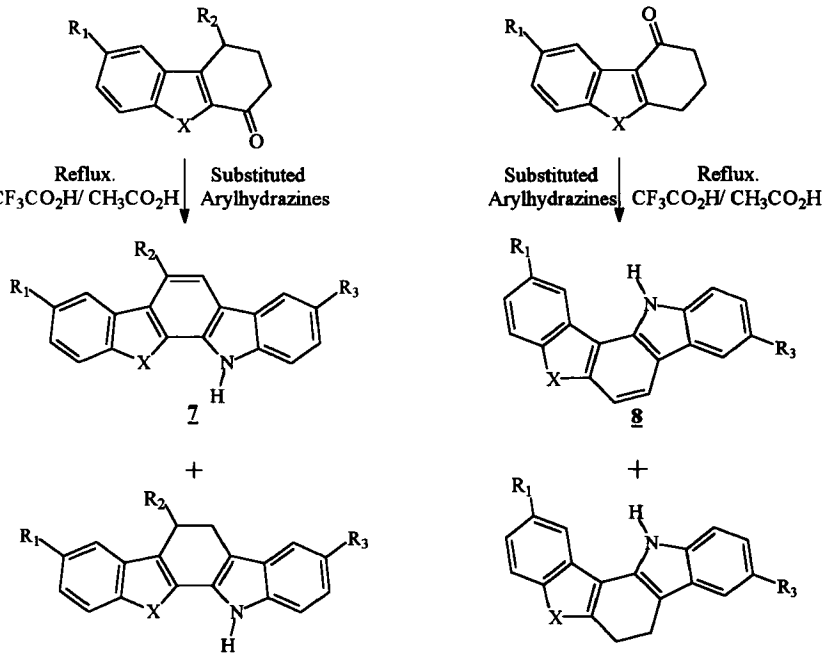


6

Compound	*X*	*R*
6a	**S**	**H**
6b	**Se**	**H**
6c	**N-CH_3**	**H**

Pentacyclic compounds 7 and 8 are obtained via Fischer Indole Synthesis (Scheme 1).

Scheme 1



R^1=H, OCH_3, Br. R^2=H, CH_3. R^3=H, Cl, OCH_3, CH_3, NO_2. X= S, Se, NH.

When Fischer Indole Synthesis was applied to ketone 5f pentacyclic structure 9 is obtained in a 65% yield (Scheme 2).

Fischer Indole Synthesis was carried out in an acidic media (CH_3CO_2H/ CF_3CO_2H 3:1) by reacting ketones 4 and 5 with substituted arylhydrazines. The

Scheme 2.

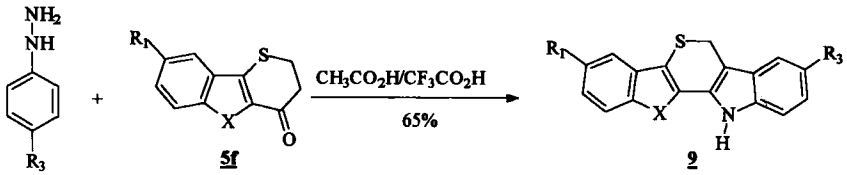


Table 3: *Chemical data on compounds 7 and 8.*

Compound	*X*	R^1	R^2	R^3	*Yield(%)*	*m.p.(°C)*
7a	NH	H	H	H	48	>300
7b	NH	OCH_3	H	H	24	>300
7c	NH	OCH_3	H	OCH_3	56	>300
7d	NH	H	H	CH_3	35	>300
7e	NH	H	H	Cl	29	>300
7f	NH	Br	H	H	54	>300
7g	NH	Br	H	Cl	24	>300
7h	NH	OCH_3	H	Cl	21	>300
7i	NH	OCH_3	CH_3	H	36	>300
7j	S	H	H	H	48	>300
7k	S	H	H	Cl	37	>300
7l	S	H	H	CH_3	30	>300
8a	S	H	H	H	22	185
8b	Se	H	H	H	41	188
8c	Se	H	H	Cl	33	210
8d	Se	H	H	NO_2	15	224
8e	N-CH_3	H	H	CH_3	14	175
9a	O	OCH_3	/	Cl	68	>300
9b	O	OCH_3	/	CH_3	66	>300

intermediate arylhydrazones were not isolated but directly cyclised. In all cases the Fischer Indole Synthesis resulted in a mixture of aromatic and dihydroaromatic compounds. However, extending the reaction time (12 hours at reflux instead of 2) allowed a direct access to compounds 7 and 8.

Experimental section.

^{1}H and ^{13}C NMR spectra were recorded using a 250 MHz Brucker spectrometer for solution in DMSO. δ values are given relatives to internal $CDCl_3$. Melting points were determined on a Kofler hot stage apparatus and are uncorrected. Elemental analyses were performed on a Carlo Erba elemental analyser. Infrared spectra (IR) were measured on a PERKIN-ELMER 881 spectrometer and are reported in wavenumbers (cm^{-1}). UV-vis absorbances were measured on a SHIMADZU UV 1205 apparatus and are reported in nanometers (nm). Elemental analyses were performed on a Carlo Erba elemental analyser.

General procedure for the Fischer indole synthesis:

A mixture of tricyclic ketone (5 mmol) and substituted arylhydrazine (5 mmol) in 15 ml CH_3CO_2H/ CF_3CO_2H 3:1 is refluxed for 12 h. The reaction flask is cooled to room temperature, water is added and the solid is collected by filtration. Pentacyclic compounds 7 and 8 are purified by recristallisation or chromatography on silica gel using generally CH_2Cl_2 as eluent.

Compound 7a: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 10.30(s, 2H, NH); 7.98(d, 2H); 7.76(s, 2H); 7.40(d, 2H); 7.26(m, 2H); 7.10(m, 2H). RMN ^{13}C $(CDCl_3/DMSO)$ δ(ppm) 137.1; 123.9; 122.5; 121.9; 118.2; 117.3; 116.9; 109.6; 109.5. IR(KBr): 3413. UV-Vis λ_{max} : 329. Anal. calcd for $C_{18}H_{12}N_2$: C, 84.37; H, 4.69; N, 10.94. Found: C, 84.47; H, 4.75; N, 10.78.

Compound 7b: RMN $^{1}H(CDCl_3$-DMSO) δ(ppm) 10.28(s, 1H); 10.10(s, 1H); 7.94(d, 1H); 7.69(m, 2H); 7.36(m, 2H); 7.23(m, 2H); 7.05(m, 1H); 6.89(dd, 1H); 3.79(s, 3H, OCH_3); RMN ^{13}C $(CDCl_3/DMSO)$ δ(ppm) 151.8; 137.2; 132.2; 124.9; 124.1; 122.7; 122.3; 118.5; 118.3; 117.9; 117.2; 112.1; 110.3; 109.9; 109.6; 109.5; 109.4; 100.5; 53.9. IR(KBr): 3401. UV-Vis λ_{max} : 359. Anal. calcd for $C_{19}H_{14}N_2O$: C, 79.72; H, 4.89; N, 9.79. Found: C, 79.66; H, 4.94; N, 9.64.

Compound 7c: RMN ^{1}H $(CDCl_3/DMSO)$ δ(ppm) 10.03(s, 2H); 7.62(s, 2H); 7.41(dd, 2H); 7.25(dd, 2H); 6.85(dd, 2H); 3.77(s, 6H, OCH_3); RMN ^{13}C $(CDCl_3/DMSO)$ δ(ppm) 152.0; 132.4; 125.1; 122.9; 118.6; 112.3; 110.8; 109.9; 100.9; 54.1. IR(KBr): 3412. UV-Vis λ_{max} : 356. Anal. calcd for $C_{20}H_{16}N_2O_2$: C, 75.95; H, 5.06; N,8.86. Found: C, 79.89; H, 5.02; N, 8.96

Compound 7d: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 10.23(s, 1H, NH); 10.10(s, 1H, NH); 7.82(d, 1H); 7.63(s, 1H); 7.57(s, 2H); 7.28(d, 1H); 7.12(m, 2H); 6.95(m, 2H); 2.28(s, 3H)-CH_3; RMN ^{13}C $(CDCl_3/DMSO)$ δ(ppm) 137.4; 135.7; 126.2(x2); 124.5; 124.3; 124.2; 122.8; 122.5; 122.4; 118.5; 118.0; 117.9; 117.3; 117.2;

109.9; 109.7; 109.4; 19.8. IR(KBr): 3403. UV-Vis λ_{max} : 325. Anal. calcd for $C_{19}H_{14}N_2$: C, 84.44; H, 5.19; N, 10.37. Found: C, 84.25; H, 5.28; N, 10.47.

Compound 7e: RMN ^{1}H($CDCl_3$/DMSO) δ(ppm) 10.39(s, 1H, NH); 10.27(s, 1H, NH); 7.70(d, 1H); 7.65(s, 1H); 7.45(m, 2H); 7.15(m, 2H); 7.00(t, 1H); 6.93(d, 1H); 6.83(t, 1H). RMN ^{13}C ($CDCl_3$/DMSO) δ(ppm) 138.1; 136.4; 125.8; 124.4; 124.2; 123.8; 123.4; 123.1(x2); 119.9; 118.9; 118.5; 118.3; 118.1; 111.3; 111.0; 110.6; 110.3. IR(KBr): 3379. UV-Vis λ_{max} : 327. Anal. calcd for $C_{18}H_{11}N_2Cl$: C, 74.35; H, 3.79; N, 9.64. Found: C, 74.44; H, 3.66; N, 9.58.

Compound 7f: RMN ^{1}H($CDCl_3$/DMSO) δ(ppm) 10.40(s, 1H, NH); 10.26(s, 1H, NH); 7.84(s, 1H); 7.73(d, 1H); 7.48(m, 2H); 7.21(d, 1H); 7.08(m, 3H); 6.83(t, 1H). RMN ^{13}C ($CDCl_3$/DMSO) δ(ppm) 137.3; 135.9; 125.1; 124.7; 124.1; 123.9; 122.9; 122.0; 120.4; 118.9; 118.0; 117.5; 117.3; 111.4; 110.3; 109.9; 109.7; 109.5. IR(KBr): 3404. UV-Vis λ_{max} : 327. Anal. calcd for $C_{18}H_{11}N_2Br$: C, 64.48; H, 3.28; N, 8.36. Found: C, 64.58; H, 3.21; N, 8.30.

Compound 7g: RMN ^{1}H($CDCl_3$/DMSO) δ(ppm) 10,38(s, 1H, NH); 9,48(s, 1H)-NH; 7,54(d, 2H); 7.18 (s, 1H); 7,03(s, 1H); 6,88(d, 2H); 6,77(d, 1H); 6,65(d, 1H). RMN ^{13}C ($CDCl_3$/DMSO) δ(ppm) 137.5; 135.7; 125.6; 124.9; 124.2; 123.8; 122.9; 122.2; 121.0; 118.9; 118.5; 117.9; 117.3; 111.2; 110.7; 109.9; 109.4; 109.2. IR(KBr): 3414. UV-Vis λ_{max} : 329. Anal. calcd for $C_{18}H_{10}N_2ClBr$: C, 58.46; H, 2.71; N, 7.58. Found: C, 58.52; H, 2.81; N, 7.48.

Compound 7h: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 10.37(s, 1H, NH); 10.07(s,1H, NH); 7.53(s, 1H); 7.28(m, 2H); 7.00(m,2H); 6.80(d, 1H); 6.51(d, 1H); 3.41 (s,3H, OCH_3); RMN ^{13}C $(CDCl_3/DMSO)$ δ(ppm) 151.7; 135.5; 132.1; 124.8; 124.5; 123.4; 122.4; 122.3; 121.8; 118.9; 117.3; 117.2; 112.3; 110.9; 110.4; 110.2; 109.4; 100.4. IR(KBr): 3409. UV-Vis λ_{max} : 333. Anal. calcd for $C_{19}H_{13}N_2OCl$: C, 71.14; H, 4.06; N,8.74. Found: C, 71.11; H, 4.04; N, 8.79.

Compound 7i: RMN $^{1}H(CDCl_3$-DMSO) δ(ppm) 10.28(s, 1H); 10.26(s, 1H); 8.05(d, 1H); 7.72(s, 1H); 7.58(s, 1H); 7.35(m, 3H); 7.17(t, 1H); 7.03(d, 1H); 3.85(s, 3H, OCH_3); 2.97(s, 3H, CH_3). RMN ^{13}C $(CDCl_3/DMSO)$ δ(ppm) 151.5; 138.4; 133.5; 125.2; 124.5; 123.1; 122.2; 118.9; 118.4; 117.8; 117.1; 111.8; 110.1; 109.6; 109.5; 109.4; 109.2; 100.5; 53.9; 21.4. IR(KBr): 3405. UV-Vis λ_{max}: 357. Anal. calcd for $C_{20}H_{16}N_2O$: C, 80.00; H, 5.33; N, 9.33. Found: C, 79.95; H, 5.31; N, 9.40.

Compound 7j: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 10.92(s, 1H, NH); 7.97(dd, 1H); 7.86(m, 2H); 7.73(d, 1H); 7.67(dd, 1H); 7.32(d, 1H); 7.21(m, 2H); 7.15(m, 1H); 6.98(m, 1H). RMN ^{13}C $(CDCl_3/DMSO)$ δ(ppm) 138.3; 136.9; 134.8; 132.8; 131.8; 124.5; 123.7; 123.1; 121.5; 121.4; 120.0; 119.6; 119.2; 118.4; 117.6; 115.6; 111.0; 109.8. IR(KBr): 3413. UV-Vis λ_{max} : 352. Anal. calcd for $C_{18}H_{11}NS$: C, 79.12; H, 4.03; N, 5.13. Found: C, 79.06; H, 4.10; N,5.07.

Compound 7k: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 11.23(s, 1H, NH); 7.79(s, 1H); 7.62(m, 2H);7.53(m, 2H); 7.08(m, 2H); 6.89(m, 2H). RMN ^{13}C $(CDCl_3/DMSO)$

δ(ppm) 137.5; 136.7; 134.7; 132.9; 132.0; 124.9; 123.8; 123.2; 121.7; 121.4; 120.0; 119.4; 119.2; 118.7; 117.6; 115.8; 110.8; 109.5. IR(KBr): 3417. UV-Vis λ_{max} : 355. Anal. calcd for $C_{18}H_{10}NSCl$: C, 70.24; H, 3.25; N, 4.55. Found: C, 70.21; H, 3.21; N, 4.62.

Compound 7l: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 10.89(s, 1H, NH); 7.61(d, 1H); 7.47(d, 1H); 7.35(m,3H); 6.84(m, 3H); 6.60(d, 1H); 1.92 (s, 3H)-CH_3. RMN ^{13}C (CDCl3-DMSO-250 MHz) δ(ppm) 136.6; 136.3; 134.6; 132.7; 131.1; 126,2; 125.7; 125.4; 124.8; 121.4; 121.3; 119.2; 118.9; 118.8; 118.2; 114.6; 110.9; 109.3; 21.2. IR(KBr): 3416. UV-Vis λ_{max} : 385. Anal. calcd for $C_{19}H_{13}NS$: C, 79.44; H, 4.53; N, 4.88. Found: C, 79.49; H, 4.50; N, 4.91.

Compound 8a: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 8.78(s, 1H)-NH; 8.25(d, 1H); 8.15(t, 2H); 7.97(d, 1H); 7.72(d, 1H); 7.54(m, 4H); 7.34(t, 1H). RMN ^{13}C ($CDCl_3$/DMSO) δ(ppm) 139.3; 138.9; 137.8; 135.0; 134.6; 125.5; 125.4; 124.5; 123.4; 123.1; 122.4; 120.5; 120.3; 119.8; 119.5; 119.2; 114.3; 111.1. IR(KBr): 3408, 2935. UV-Vis λ_{max} : 355. Anal. calcd for $C_{18}H_{11}NS$: C, 79.12; H, 4.03; N, 5.13. Found: C, 79.01; H, 3.98; N, 5.21.

Compound 8b: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 8.82(s, 1H, NH); 8.26(d, 1H); 8.15(m, 2H); 8.01(d, 1H); 7.77(d, 1H); 7.62(m, 2H); 7.46(m, 2H); 7.33(m, 1H); RMN ^{13}C ($CDCl_3$/DMSO) δ(ppm) 137.9; 136.9; 135.7; 135.5; 134.0; 124.4; 123.9; 123.5; 123.3(x2); 120.8; 120.2; 119.1; 117.8; 117.7; 117.4; 114.7; 110.1.

IR(KBr): 3435. UV-Vis λ_{max} : 358. Anal. calcd for $C_{18}H_{11}NSe$: C, 67.5; H, 3.44; N, 4.38. Found: C, 67.42; H, 3.35; N, 4.46.

Compound 8c: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 10.95(s, 1H, NH); 8.03(d, 1H); 7.40(m, 2H); 7.31(d, 1H); 7.00(m, 2H); 6.88(t, 1H); 6.69(m, 2H); RMN ^{13}C ($CDCl_3$/DMSO) δ(ppm) 136.9; 136.3; 136.2; 135.3; 134.5; 124.4; 123.9; 123.2; 123.1; 122.4; 122.0; 120.1; 118.1; 117.6; 117.3; 115.0; 111.3. IR(KBr): 3411. UV-Vis λ_{max} : 359. Anal. calcd for $C_{18}H_{10}NSeCl$: C,60.93; H, 2.82; N, 3.95. Found: C, 61.01; H, 2.86; N, 3.90.

Compound 8d: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 11.62(s, 1H, NH); 8.84(s, 1H); 8.56(d, 1H); 8.15(d, 1H); 7.97(d, 1H); 7.82(d, 1H); 7.62(d, 1H); 7.43(t, 1H); 7.28(t, 1H). RMN ^{13}C ($CDCl_3$/DMSO) δ(ppm) 135.9; 135.5; 135.2; 135.1; 134.2; 124.1; 123.8; 123.1; 122.9; 122.4; 122,0; 120,1; 117.9; 117.6; 117.1; 114.8; 111.2. IR(KBr): 3417. UV-Vis λ_{max} : 357. Anal. calcd for $C_{18}H_{10}N_2SeO_2$: C, 59.18; H, 2.74; N, 7.67. Found: C,59.08; H, 2.81; N,7.65.

Compound 8e: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 8.51 (s, 1H, NH); 8.16(d, 1H); 8.12(d, 1H); 7.91(s, 1H); 7.49(m, 3H); 7.36(m, 1H); 7.30(m, 2H); 3.92(s, 3H, N-CH_3); 2.58(s, 3H, CH_3). RMN ^{13}C ($CDCl_3$/DMSO) δ(ppm) 140.5; 140.0; 137.2; 134.7; 129.1; 125.2; 124.4; 124.3; 121.4; 120.5; 119.2; 119.0; 118.3; 115.7; 110.4; 108.6; 106.3; 101.5; 29.5; 21.5. IR(KBr): 3398. UV-Vis λ_{max} : 357. Anal. calcd for $C_{20}H_{14}N_2$: C, 85.11; H, 4.96; N, 9.93. Found: C, 85.08; H, 5.01; N, 9.88.

Compound 9a: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 11.66(s, 1H, NH); 9.70(s, 1H); 9.03(m, 2H); 8.84(m, 2H); 8.46(s, 1H); 4.95(s, 2H); 3.56(s, 3H)-OCH_3. RMN ^{13}C $(CDCl_3/DMSO)$ δ(ppm) 148.8; 141.5; 136.6; 129.4; 128.5; 127.9; 122.2; 121.8; 121.1; 120.3; 116.9; 114.8; 112.7; 112.3; 107.7; 102.4; 54.9; 44.3. IR(KBr): 3399. Anal. calcd for $C_{18}H_{12}NO_2SCl$: C, 63.25; H, 3.51; N, 4.10. Found: C, 63.29; H, 3.58; N, 4.02.

Compound 9b: RMN $^{1}H(CDCl_3/DMSO)$ δ(ppm) 11.56(s, 1H, NH); 9.31(s, 1H); 9.02(d, 2H); 8.75(d, 2H); 8.63(d, 2H); 8.46(d, 2H); 4.95(s, 2H); 3.56(s, 3H)-OCH_3; 2.21(s, 3H)-CH_3. RMN ^{13}C $(CDCl_3/DMSO)$ δ(ppm) 148.7; 141.2; 138.1; 135.4; 133.5; 130.9; 129.0; 128.8; 122.2; 120.7; 120.3; 120.2; 116.8; 112.6; 112.4; 102.4; 54.9; 44.4; 19.9. IR(KBr): 3408. Anal. calcd for $C_{19}H_{15}NO_2S$: C, 71.03; H, 4.67; N, 4.36. Found: C, 70.94; H, 4.72; N, 4.40.

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