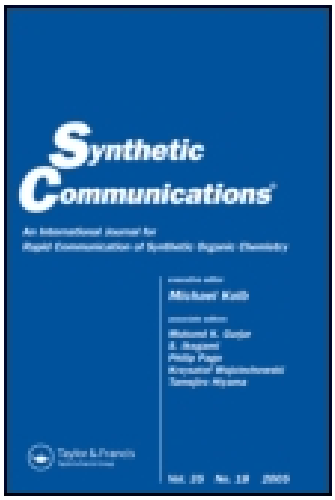

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A Facile Synthesis of 2-Aroylindoles by the Oxidation of 2-Arylmethylindoles Using Sarett Reagent

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ABSTRACT

A facile synthesis of 2-aroylindoles by the oxidation of 3-substituted 2-arylmethylindoles using Sarett reagent has been reported. The phenylthio group at 3-position has been cleaved by Raney nickel.

Key Words: 2-Arylmethylindoles; 2-Aroylindoles; Sarett reagent; Raney nickel; Chromium pyridine complex.

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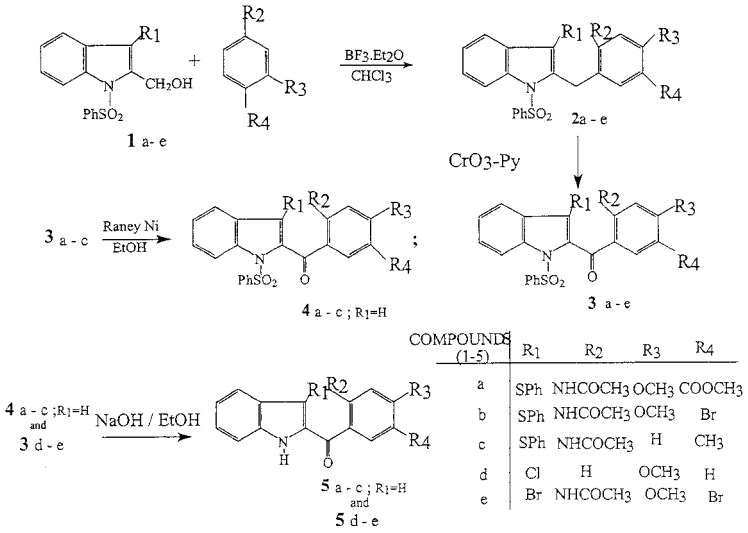
Recently antimitotic activity of some 2-aroylindoles has been reported.[1] 3-Iodo-2-aroylindole has been used as a precursor for the synthesis β- carbolines.[2] The synthesis of 2-aroylindoles from 2-Lithio-N-blocked indoles[3–5] and from 2-nitrobenzaldehydes[6] have been reported. In continuation of our studies towards the synthesis of fused heterocycles,[7] we were interested in developing a new synthesis of 2-aroylindoles. The oxidation of an active methylene group to the corresponding keto group by chromium trioxide has been reported.[8–10] So far there is no report on the oxidation of a 2-benzylindole to the corresponding ketone. So we report here the use of chromium trioxide-pyridine complex in pyridine [Sarett reagent][11] for the oxidation of 2-arylmethylindoles to 2-aroylindoles.

Treatment of 1-phenylsulfonyl-3-phenylthioindol-2-ylmethanol[12–13] **1a–c**, 3-chloro-1-phenylsulfonylindol-2-ylmethanol **1d** or 3-bromo-1-phenylsulfonylindol-2-ylmethanol **1e** with different arenes in chloroform or 1,2-dichloroethane in the presence of $BF_3{\cdot}OEt_2$ afforded the corresponding 2-arylmethylindoles **2a–e**. Compounds **2a–e** were oxidized to the corresponding 2-aroylindoles **3a–e** by chromium trioxide in pyridine(sarett reagent). Compounds **2a**, **2b**, **2d**, and **2e** required 5 equiv. of the oxidant. Compound **2c** required 10 equiv. for oxidation. The phenylthio group was removed by Raney nickel in boiling ethanol to afford compound **4a–c**; $\mathbf{R_1 = H}$. Treatment of **4a–c**; $\mathbf{R_1 = H}$ and **3d–e** with 10% NaOH in boiling alcohol afforded **5a–c**; $\mathbf{R_1 = H}$ and **5d–e**. In conclusion, a facile oxidation of 2-arylmethylindoles by chromium trioxide to 2-aroylindoles with various substituents has been accomplished.

All melting points are uncorrected. Merck silica gel 60 GF 254 thin-layer plates were employed for TLC. Merck silica gel (finer than 350 mesh) was used for flash column chromatography. IR spectra were recorded on Perkin-Elmer FT-paragon 1000 and Shimadzu-8300 FT instruments. ^{1}H-NMR spectra were recorded on a Brucker 304 MHz instrument. Mass spectra were recorded on Finningan MAT 8230 instrument. Chromium trioxide was purchased from Merck-India.

Compound 1d. 3-Chloro-2-methylindole was prepared according to the Lit. procedure[14] from 2-methylindole.

3-Chloro-2-methyl-1-phenylsulfonylindole. To a solution of 3-chloro-2-methylindole (1.7 g; 10.3 mmol) in benzene (30 mL) was added 50% sodium hydroxide solution (3 mL) followed by cetyltrimethylammonium-bromide (5 mg). phenylsulfonylchloride (1.5 mL) was added in one lot at RT and stirred for 24 h. The completion of the reaction was monitored by TLC. Water (50 mL) was added and the benzene layer was separated and distilled off. The residue was digested with chloroform and filtered. The



Scheme 1.

filtrate was concentrated and the pasty mass was crystallized from methanol. (82%) m.p. 110–112°C. IR (KBr): 1446, 1374, 1236, 1183, 1097, 751, 793, 587, 565 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.91 (s, 3H), 6.66–7.85 (m, 9H). (Calcd. For $C_{15}H_{12}ClNO_2S$: C, 58.92; H, 3.96; N, 4.58. Found: C, 58.82; H, 3.70; N, 4.86%).

3-Chloro-2-hydroxymethyl-1-phenylsulfonylindole. To a solution of 3-Chloro-1-phenylsulfonyl-2-methylindole (1 g, 3.2 mmol) in Carbontetrachloride (25 mL), N-bromosuccinimide (0.7 g), and dibenzoylperoxide (5 mg) were added. The solution was refluxed for 4 h. The succinimide was filtered and carbontetrachloride (20 mL) was distilled out. The residue was dissolved in acetonitrile (20 mL). To this a solution of potassium carbonate (1 g) in water (2 mL) was added. It was refluxed for 3 h. The lower layer was discarded. Acetonitrile was distilled out completely. The residue was dissolved in chloroform (25 mL) and filtered. Chloroform was distilled out. The pasty mass was taken for next step after IR and ^1H NMR analysis. (80%), IR (KBr): 3451, 1591, 1480, 1183, 793, 587 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.31 (s, 1H), 4.82 (s, 2H), 6.91–7.98 (m, 9H).

Compound 1e. 3-Bromo-2-bromomethyl-1-phenylsulfonylindole was prepared according to the Lit. procedure.[15] To a solution of 3-bromo-2-bromomethyl-l-phenylsulfonylindole (27.9 mmol) in acetonitrile (200 mL), a solution of potassium carbonate (10 g) in water (20 mL) was added. It was refluxed for 5 h. The bottom layer was discarded.

Acetonitrile was distilled out completely. The residue was dissolved in chloroform (100 mL) and filtered. The chloroform was distilled off completely. The residue was crystallized from methanol. (78.4%), m.p.: 128–130°C (MeOH). IR (KBr): 3557, 3435, 1448, 1315, 896, 764, 471 cm^{-1}. ^1H NMR ($CDCl_3$): 2.31 (s, 1H), 4.88 (s, 3H), 6.8–7.99 (m, 9H). (Calcd. for $C_{15}H_{12}BrNO_3S$: C, 49.19; H, 3.30; N, 3.82. Found: C, 49.29; H, 2.88; N, 3.44%).

Compound 2: General Procedure

1-Phenylsulfonyl-2-bromomethyl-3-phenylthioindole[11] was converted by partial hydrolysis (CH_3CN-H_2O, K_2CO_3, Δ, 3h) into 1-phenylsulfonyl-3-phenylthioindol-2-ylmethanol by the published procedure.[12] To a solution of 1-phenylsulfonyl-3-phenylthioindol-2-ylmethanol or 3-chloro-l-phenylsulfonylindol-2-ylmethanol **1d** or 3-bromo-1-phenylsulfonylindol-2-ylmethanol **1e** (25 mmol) in chloroform (400 mL), a solution of arene (25 mmol) in the same solvent (25 mL) was added followed by anhydrous magnesium sulfate (10 g) and borontrifluoride etherate (2.0 mL). [For **2c**, 1,2-dichloroethane was used as solvent]. The resulting solution was refluxed for 3 h. Then water (100 mL) was added and the organic layer was separated. The organic layer was washed with 20% hydrochloric acid (1 × 50 mL) followed by water and saturated bicarbonate solution. The solvent was removed by distillation after drying over anhydrous sodium sulfate. The residue was chromatographed on a silica gel column (350 mesh) and eluted successively with 20% ethylacetate in hexane followed by 25% and then finally with 30%. Thirty percent ethylacetate in hexane eluent gave 2-arylmethylindole which was then crystallized.

Compound 2a. (76%), m.p.: 154–156°C. (benzene–hexane 1:1). IR (KBr): 3425, 1727, 1666, 1373, 1172 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.25 (s, 3H), 3.67 (s, 3H), 3.84 (s, 3H), 4.52 (s, 2H), 6.86–8.25 (m, 17H, NH+arom). MS: *m*/*z* (%) 481(100), 480 (30), 479 (50), 328 (38), 77 (18). (Calcd. for $C_{32}H_{28}N_2O_6S_2$: C, 64.04; H, 4.70; N, 4.66. Found: C, 64.02; H, 4.45; N, 4.35%).

Compound 2b. (85%), m.p.: 182–184°C. (benzene). IR(KBr): 3409, 1689, 1365, 1172 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.22 (s, 3H), 3.81 (s, 3H), 4.50 (s, 2H), 6.81–8.19 (m, 17H, NH+arom). MS: *m*/*z* (%) [Br^{81} and Br^{79}] 481 (38), 479 (40), 372 (2.5), 370 (2.5), 329 (54), 327 (52), 248 (28), 236 (40), 205 (30), 77 (100). (Calcd. for $C_{30}H_{25}N_2O_4S_2Br$: C, 58.11; H, 4.06; N, 4.51. Found: C, 55.08; H, 3.75; N, 3.18%).

Compound 2c. (78%), m.p.: 178–180°C. (benzene). IR (KBr): 3394, 1662, 1371, 1172 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 1.93 (s, 3H), 2.24 (s, 3H),

4.59 (s, 2H), 6.47–8.18 (m, 18H, NH+arom). MS: *m*/*z* (%) 526 (M^+, 2), 385 (38), 233 (100), 77 (48). (Calcd. for $C_{30}H_{26}N_2O_3S_2$: C, 68.48; N, 4.98, H, 5.32. Found: C, 68.64, H, 4.73, N 5.01%).

Compound 2d. (85%) m.p.: 116–118°C. (benzene–hexane 1:1). IR (KBr): 1183, 1371 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 3.75 (s, 3H), 4.46 (s, 2H), 6.7–8.1 (m, 13H). MS: *m*/*z* (%) 380 (1), 378 (2), 287 (2), 285 (6), 272 (48), 270 (52), 237 (36), 235 (100). (Calcd. for $C_{22}H_{18}ClNO_3S$: C, 64.15; N, 3.40; H, 4.40. Found: C, 64.38; H, 4.19; N, 3.13%).

Compound 2e. (76%), m.p.: 202–204°C. (benzene). IR(KBr): 3217, 1660 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.27 (s, 3H), 3.88 (s, 3H), 4.38 (s, 2H), 7.21–8.15 (m, 12H, NH+arom). MS: *m*/*z* (%) [Br^{81} and Br^{79}] 594 (M^+,3.3), 592 (4.7), 590 (3.5), 454 (12.3), 453 (50.2), 452 (22.6), 451 (100), 450 (13.4), 208 (3.4), 77 (37.2). (Calcd. for $C_{24}H_{20}Br_2N_2O_4S$: C; 48.67; H, 3.4; N, 4.73. Found: C, 48.85; H, 3.18; N, 4.98%).

Oxidation of 2a–e: General Procedure

Chromium trioxide (60 g) was slowly added to dry pyridine (600 mL) at 0–5°C over a period of 1 h. After the addition of 50% of chromium (VI) oxide, a color change from dark brown to an orange red indicated the formation dipyridine chromium (VI) oxide complex. Then the temperature was allowed to come to RT. A solution of 2-arylmethylindole (12 g, 20 mmol) in pyridine (30 mL) was added in one portion to the dipyridine chromium (VI) oxide complex and the temperature of the reaction mixture was maintained at 50–60°C with vigorous stirring for 48 h. [For compound 2C, 105 g of CrO_3 and 1000 mL of pyridine was used and the temperature was maintained for 60–70°C]. Complete disappearance of the starting material was ascertained by periodic TLC analysis. Then the excess pyridine was distilled off under reduced pressure and the residue was poured over crushed ice (500 g). The pH was adjusted to 2 by the addition of concentrated hydrochloric acid. The precipitated solid was filtered, washed with ice-cold water, pressed dry and desiccated over anhydrous calcium chloride. Then it was extracted on a soxhlet apparatus with chloroform. Removal of chloroform by distillation gave the crude product which was then purified by crystallization.

Note: The chromium trioxide was made into granules in such a way that it should sink into the cold pyridine solution immediately after the addition. If it floats on the pyridine surface it becomes pyrophoric.

Compound 3a. (60%), m.p.: 190–191°C. (DME). IR (KBr): 3249, 1730, 1698, 1631 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.33 (s, 3H), 3.71 (s, 3H), 4.00 (s, 3H), 7.03–8.63 (m, 16Harom) 11.85 (s, 1H). MS: *m*/*z* (%)

614 (M^+, 20), 613 (16), 431 (14), 323 (22), 322 (100). The structure of acid obtained by hydrolysis of ester moiety in **3a** has been determined by single crystal X-ray analysis. (Calcd. for $C_{32}H_{26}N_2O_7S_2$: C, 62.52; H, 4.26; N, 4.56. Found: C, 62.80; H, 4.18; N, 4.39%).

Compound 3b. (50%), m.p.: 226–228°C. (Benzene–hexane 1:1). IR (KBr): 3259, 1688, 1622 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.16 (s, 3H), 3.89 (s, 3H), 6.95 – 8.08 (m, 16H_{arom}), 11.82 (s, 1H). MS: *m*/*z* (%) 626 (6), 624 (6). 481 (6), 479 (3), 453 (2), 451 (2), 344 (28), 342 (25), 78 (100). (Calcd. for $C_{30}H_{23}N_2O_4S_2Br$: C, 56.78; H, 3.65; N, 4.41. Found: C 56.68; H, 3.53; N, 4.56%)

Compound 3c. (65%), m.p.: 180-–182°C. (Benzene). IR (KBr): 3301, 1698, 1641 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 1.98 (s, 3H), 2.22 (s, 3H), 7.01–8.70 (m, I7H_{arom}), 11.26 (s, 1H). MS: *m*/*z* (%) 540 (M^+, 8), 385 (2), 357 (6), 248 (100), 247 (16), 77 (54). (Calcd. for $C_{30}H_{24}N_2O_4S_2$: C, 66.65; H, 4.47; N, 5.18. Found: C, 66.67; H, 4.29: N; 4.97%).

Compound 3d. (56%), m.p.: 140–141°C (Benzene–hexane 1:1). IR (KBr): 1688, cm^{-1}. ^1H NMR. ($CDCl_3$/TMS): 3.82 (s, 3H), 6.95–8.30 (m, 13H_{arom}), MS: *m*/*z* (%) 362 (2), 360 (6), 286 (36), 256 (22), 179 (16), 177 (16), 177 (38), 77 (100), (Calcd. for $C_{22}H_{16}CINO_4S$: C, 62.05; H, 3.79; N, 3.29. Found: C, 62.18; H, 3.83; N, 3.91%).

Compound 3e. (61%), m.p.: 244–246 °C (DME). IR (KBr): 3257, 1708, 1623, 728 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.35 (s, 3H), 4.03 (s, 3H), 7.26–8.67 (m, 11H_{arom}), 11.76 (s, 1H). MS: *m*/z (%) 608 (M^+,4.8), 606 (9.9), 485 (16.8), 483 (4), 344 (34), 342 (32), 77 (100). (Calcd. for $C_{24}H_{18}$ Br_2 N_2O_5S: C, 47.55; H, 2.99; N, 4.62. Found: C, 47.88; H, 2.89; N, 4.91%).

Desulfurization of 3a–c: General Procedure

A solution of compound **3** (15 mmol) in ethanol (300 mL) containing Raney nickel (50 g) was refluxed for 1 h. The catalyst was filtered off and washed with hot ethanol (3 × 20 mL). The combined filtrates were concentrated and the residue was crystallized . The products were directly used for next step after IR and ^1H NMR analysis.

Compound 4a. (87%), m.p.: 212–214°C. (Acetone—hexane). IR (KBr): 3426, 1727, 1700, 1626, 1371, 1172 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.32 (s, 311), 3.78 (s, 3H), 4.04 (s, 3H), 6.84 (s, 1H, indole-3H), 7.25–8.63 (m, 11Harom) 11.81 (s, 1H).

Compound 4b. (85%), m.p.: 144–146°C. (Benzene–hexane). IR (KBr): 1688, 1622, 1371, 1172 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.21 (s, 3H), 3.85 (s, 3H), 6.15 (s, 1$H_{indole\text{-}3H}$), 6.82–8.52 (m, 11H_{arom}), 11.81 (s, 1H).

Compound 4c. (88%), m.p.: 158–160°C. (Methanol). IR (KBr): 3347, 1686, 1639 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 1.93 (s, 3H), 2.24 (s, 3H), 6.47 (s, $1H_{indole\text{-}3H}$), 6.98–8.18 (m, $12H_{arom}$), 11.00 (s, 1H).

Hydrolysis of 4a–c; $R_1 = H$ and 3d–e: General Procedure

A solution of compound (15 mmol) in alcohol (300 mL) was refluxed with 10% sodium hydroxide solution (50 mL) for 30 min. The solution was poured over ice and the precipitated solid was filtered, washed with water, dried over anhydrous calcium chloride and purified by crystallization .

Compound 5a. (80%), m.p.: 179–181°C (MeOH). IR (KBr): 3477, 3336, 1693, 1625, 1612 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.29 (s, 3H), 3.76 (s, 3H); 4.02 (s, 3H), 6.84 (s, $1H_{indole\text{-}3H}$), 7.23–8.61 (m, 7H, NH+arom), 11.79 (s, 1H). MS: *m*/*z* (%) 324 (30), 323 (100), 250 (12), 236 (12), 205 (10), 77 (18). (Calcd. for $C_{20}H_{18}N_2O_5$: C, 65.57: H, 4.91; N, 7.65. Found: C, 65.68; H, 4.62; N, 7.32%).

Compound 5b. (85%), m.p.: 190–92°C (MeOH). IR (KBr): 3365, 3305, 1607, 1586 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.16 (s, 3H). 3.89 (s, 3H), 6.95 (s, $1H_{indole\text{-}3H}$), 6.98–8.08 (m, 7H. NH+arom). 11.81 (s, 1H). MS: *m*/*z* (%) 266 (100), 265 (90). 250 (8), 249 (20), 238 (9), 237 (28), 206 (8), 150 (22), 89 (54). (Calcd. for $C_{18}H_{15}BrN_2O_3$: C, 55.81; H, 3.87; N, 7.23. Found: C, 55.82; H; 3.66; N, 6.92%).

Compound 5c. (80%), m.p.: 290–292°C (MeOH). IR (KBr): 3224, 1685, 1621 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 1.90 (s, 3H), 2.22 (s, 3H), 6.45 (s, $1H_{indole\text{-}3H}$), 6.97–8.25 (m, 8H, NH+arom), 11.81 (s, 1H). MS: *m*/*z* (%) 286 (16), 285 (24), 284 (43), 256 (12), 178 (16), 177 (36), 135 (48), 392 (26), 77 (100). (Calcd. for $C_{18}H_{16}N_2O_2$: C, 73.97; H, 5.47; N, 9.58. Found : C, 74.10: H, 5.26; N, 9.35%).

Compound 5d. (82%), m.p.: 168–170°. (MeOH); IR (KBr): 3293, 1619, 1594 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 3.87 (s, 3H), 6.95–7.91 (m, 9H, NH+arom). MS: *m*/*z* (%) 236 (12), 234 (24), 223 (100), 222 (34), 207 (20), 205 (12), 134 (10), 133 (10), 117 (22). (Calcd. for $C_{16}H_{12}ClN_2O_2$: C, 67.26; H, 4.23; N, 4.91. Found: C, 67. 18; H, 4.61; N, 4.81%).

Compound 5e. (68%), m.p.: 292–294°C Dec. (MeOH). IR (KBr): 3614, 3543, 3428, 1669, 1615 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): 2.38 (s, 3H), 3.89 (s, 3H), 7.16–8.92 (m, 7H, NH+arom), 11.81 (s, 1H). MS: *m*/*z* (%) 468 (M^+, 4.8), 466 (10.16), 485 (16.7), 483 (3), 343 (34), 341 (35), 77 (100). (Calcd. for $C_{18}H_{14}Br_2N_2O_3$: C, 46.38: H, 3.03; N, 6.01. Found: C 46.19; H, 2.91; N, 5.90%).

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