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Tetrahedron Letters 47 (2006) 3693–3696
Tetrahedron Letters

Expedient synthesis of 1-vinylpyrrole-2-carbaldehydes

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Received 20 December 2005; revised 14 March 2006; accepted 22 March 2006
Available online 12 April 2006

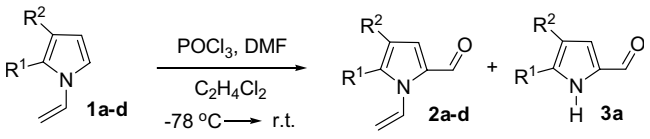
Abstract—1-Vinylpyrrole-2-carbaldehydes and 1-vinyl-4,5-dihydrobenzo[*g*]indole-2-carbaldehyde were synthesized in 56–91% yields from their 1-vinyl derivatives by a modified Vilsmeier–Haack reaction. A low temperature (−78 °C) is required to avoid removal of the *N*-vinyl group, whereas at elevated temperature (reflux in 1,2-dichloroethane) the latter process leads to direct conversion of 1-vinylpyrroles to pyrrole-2-carbaldehydes.

Functionalized 1-vinylpyrroles attract attention as powerful building blocks for the design of complex pyrrolic assemblies.[1,2] However, little is known about 1-vinylpyrroles bearing an aldehyde moiety. Meanwhile, formylpyrroles are very important intermediates and building blocks widely employed in the synthesis of diverse oligopyrrolic systems,[3] anion receptors in biomedical analysis,[4] porphyrins,[5] models for the investigation of multiple sclerosis[6] and expansion of the genetic alphabet,[7] ligands for metallocomplexes,[8] conjugated polymers[9] and others.

Thus, the development of a general approach to pyrroles, containing both *N*-vinyl and aldehyde functionalities represents a synthetic challenge. Herein, we report an approach based on the formylation of *N*-vinylpyrroles, readily available from ketoximes and acetylenes by the Trofimov reaction.[1] As far as we know, only one quite specific representative of this class of compounds has been synthesized from the corresponding *N*-vinyl derivative, namely, 5-benzyl-1-vinyl-4,5,6,7-tetrahydro-1*H*-pyrrolo[3,2-*c*]pyridine-2-carbaldehyde,[10] which among other pyrrolo[3,2-*c*]pyridines, inhibited aggregation of human platelet-rich plasma induced by adenosine 5′-diphosphate.

Formylpyrroles are usually synthesized by the Vilsmeier–Haack formylation using the $Me_2NCHO/POCl_3$ system.[11] *N*-Vinylpyrroles are highly reactive towards acids, which catalyze their dimerization, oligomerization or diverse Markovnikov additions to the double bond. Thus, due to the acidic nature of $POCl_3$, it may be unsuitable for such substrates. Probably this is why 1-vinylpyrrole-2-carbaldehyde was prepared indirectly by dehydrochlorination of 1-(2-chloroethyl)pyrrole-2-carbaldehyde.[12]

As expected, we observed that under the conditions previously and successfully employed for the formylation of pyrroles involving reflux of the reaction mixture in 1,2-dichloroethane and water, *N*-vinylpyrrole **1a** gave mixtures of the expected product **2a** and vinyl-free pyrrole-2-carbaldehyde **3a** in a ratio of up to 1:1 (Scheme 1).[13]



Pyrrole	R^1	R^2	Yield of **2**, %
a	$(CH_2)_4$		56
b	Ph	H	66
c	4-$MeOC_6H_4$	H	82
d	2-Thienyl	H	88

Scheme 1.

Keywords: 1-Vinylpyrrole-2-carbaldehydes; 1-Vinylpyrrole; Formylation; Vilsmeier–Haack reaction.
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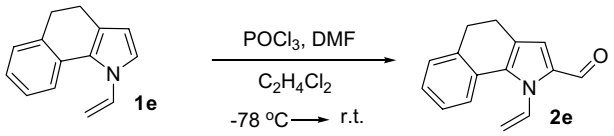
doi:10.1016/j.tetlet.2006.03.126



Scheme 2.

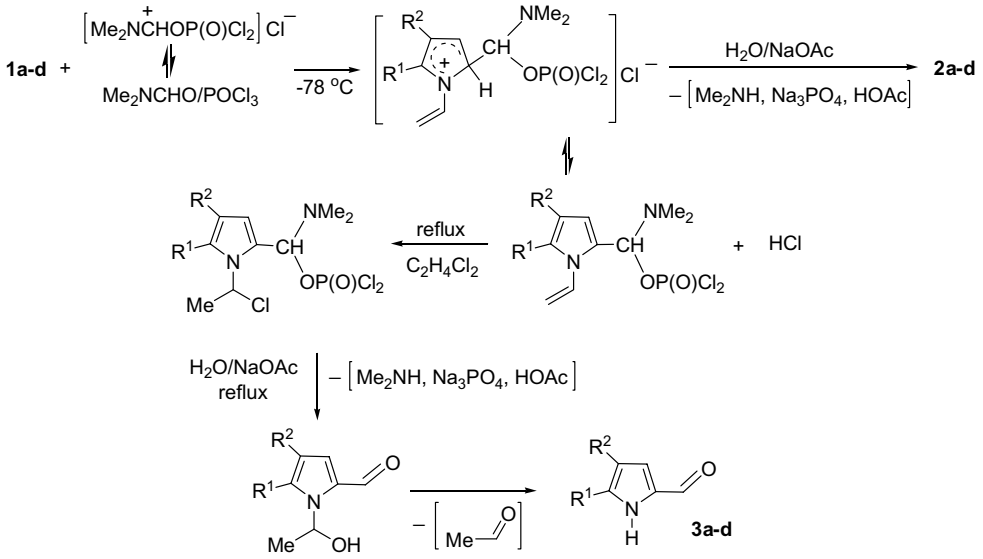
Decreasing the reaction mixing temperature to −78 °C and performing the other steps of the reaction at room temperature allowed removal of the *N*-vinyl group to be avoided. As a result, 1-vinylpyrrole-2-carbaldehydes **2a–d** were selectively synthesized in 56–88% yields[14] (unoptimized). The reaction was carried out in 1,2-dichloroethane, since, when dry diethyl ether, dichloromethane or chloroform were employed, mainly resinification was observed and 1-vinylpyrrole-2-carbaldehydes were minor products.

The protocol appeared to be excellently applicable to *N*-vinylpyrroles condensed with a dihydronaphthalene moiety, for example, 1-vinyl-4,5-dihydrobenzo[*g*]indole **1e**, which was transformed to the corresponding aldehyde **2e** almost quantitatively, (Scheme 2, 91% yield).

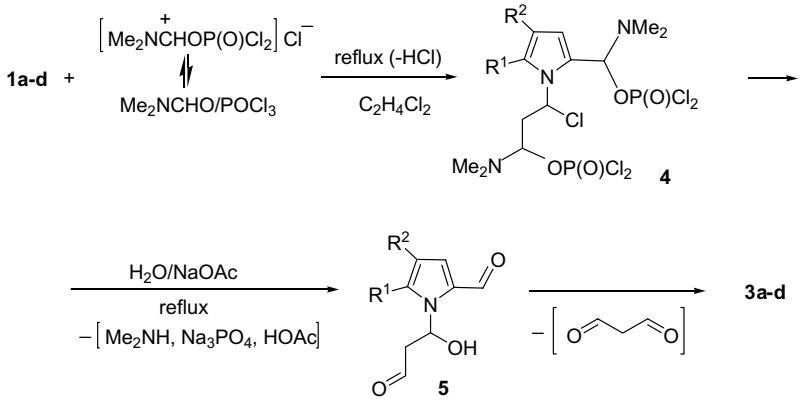
Additionally, the easy removal of the *N*-vinyl group from *N*-vinylpyrroles under conventional Vilsmeier–Haack reaction conditions may contribute to the existing quest for such a deprotection.[15]

Apparently, electrophilic attack on the pyrrole ring by the cationic complex $Me_2NCHO/POCl_3$ is accompanied by addition of the released HCl to the vinyl group (Scheme 3). As known from *N*-vinylpyrrole protonation studies,[16] at low temperature (−80 °C) electrophilic attack occurs on the pyrrole α-position, while at higher temperature (−40 °C) hydrogen halides add to the *N*-vinyl group.[16]

Thus, Scheme 3 rationalizes why removal of the *N*-vinyl group occurs at higher temperature (reflux in $C_2H_4Cl_2$), whereas for the synthesis of 1-vinylpyrrole-2-carbaldehydes **2a–d**, lower temperature (−78 °C → rt) is required.



Scheme 3.



Scheme 4.

Also, according to Ref. 16, at elevated temperature (reflux in $C_2H_4Cl_2$), electrophilic attack of the electron-rich *N*-vinyl group by the Vilsmeier–Haack cationic complex may form diadduct **4**, which upon boiling in H_2O/NaOAc gives the *N*-vinyl-free pyrrole-2-carbaldehydes **3a–d** and malonic aldehyde through unstable dialdehyde intermediate **5** (Scheme 4).

Experiments have shown that removal of the *N*-vinyl group did not occur upon reflux of starting *N*-vinylpyrroles **1a–c** in the presence of the acetate buffer (NaOAc+HOAc).

Thus, despite the high sensitivity of *N*-vinyl pyrroles towards acidic species, they are selectively formylated at the α-position of the pyrrole ring by modified Vilsmeier–Haack reaction, which represents an expedient general synthesis of previously inaccessible 1-vinylpyrrole-2-carbaldehydes. Under conventional conditions, the approach affords 2-formylpyrroles without *N*-vinyl functionality, thus providing a direct transition from *N*-vinylpyrroles to pyrrole-2-carbaldehydes.

Acknowledgements

This work was supported by the Presidium of the Russian Academy of Sciences (Grants No. 10002-251/P-25/155-305/200404-072 and 10104-71/P-25/155-305/090605-003) and the Russian Science Support Foundation.

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13. To DMF (0.40 g, 5.5 mmol) at 0 °C, $POCl_3$ (0.84 g, 5.5 mmol) was added dropwise, and the mixture obtained was stirred for 15 min without cooling. Then 1,2-dichloroethane (1.5 mL) was added, and after cooling to 0 °C, a solution of *N*-vinylpyrrole **1a** (0.74 g, 5 mmol) in 1,2-dichloroethane (1.5 mL) was added dropwise over 10 min. The resulting mixture was refluxed for 15 min. After cooling to room temperature a solution of sodium acetate (2.26 g, 28 mmol) in water (10 mL) was added followed by refluxing of the mixture for 15 min. The reaction mixture was cooled and extracted with diethyl ether (5 × 5 mL). The combined organic phases were washed with saturated aqueous sodium hydrocarbonate solution (3 × 5 mL) and dried over magnesium sulfate. The residue obtained after evaporation of the ether was chromatographed on basic alumina (hexane) to give formylpyrroles **2a** (0.18 g, 21%) and **3a** (0.17 g, 23%).
4,5,6,7-Tetrahydroindole-2-carbaldehyde (**3a**). Yield 23%. Yellowish crystals (95% purity, mp. 27–29 °C). ^{1}H NMR (400.13 MHz, $CDCl_3$): δ 9.32 (s, 1H, CHO), 9.20 (br s, 1H, NH), 6.68 (s, 1H, H-3), 2.64 (t, 2H, $^3J_{6,7} = 5.9$ Hz, H-7), 2.52 (t, 2H, $^3J_{4,5} = 5.9$ Hz, H-4), 1.83 (m, 2H, H-6), 1.78 (m, 2H, H-5). ^{13}C NMR (101.61, $CDCl_3$): δ 178.0 (C=O), 139.0 (C-2), 131.3, 121.2 (C-3a, C-7a), 121.4 (C-3), 23.4, 23.1, 22.7 (C-4, C-5, C-6, C-7).
14. *General procedure*. To DMF (0.40 g, 5.5 mmol) at −78 °C (acetone-dry ice bath), $POCl_3$ (0.84 g, 5.5 mmol) was added dropwise, and the mixture obtained was stirred for 15 min without cooling. Then 1,2-dichloroethane (1.5 mL) was added, and after cooling to −78 °C, a solution of *N*-vinylpyrrole **1** (5 mmol) in 1,2-dichloroethane (1.5 mL) was added dropwise over 10 min. The resulting mixture was stirred for 3 h at ambient temperature. Next, a solution of sodium acetate (2.26 g, 28 mmol) in water (10 mL) was added and stirring was continued for 1 h at ambient temperature. The reaction mixture was extracted with diethyl ether (5 × 5 mL), the combined organic phases were washed with saturated aqueous sodium hydrocarbonate solution (3 × 5 mL) and dried over magnesium sulfate. The residue obtained after evaporation of the ether was purified on basic alumina (hexane/ether 3:1) to give formylpyrrole **2**.
4,5,6,7-Tetrahydro-1-vinylindole-2-carbaldehyde (**2a**). Yield 56%. Yellowish oil, n_D^{25} 1.5762. ^{1}H NMR (400.13 MHz, $CDCl_3$): δ 9.43 (s, 1H, H_{CHO}), 7.56 (dd, 1H, $^3J_{AX} = 9.1$ Hz, $^3J_{BX} = 16.1$ Hz, H_X), 6.74 (s, 1H, H-3), 5.11 (d, 1H, H_B, $^3J_{BX} = 16.1$ Hz), 5.07 (d, 1H, H_A, $^3J_{AX} = 9.1$ Hz), 2.70 (t, 2H, $^3J_{6,7} = 6.0$ Hz, H-7), 2.54 (t, 2H, $^3J_{4,5} = 6.0$ Hz, H-4), 1.79 (m, 2H, H-6), 1.72 (m, 2H, H-5). ^{13}C NMR (101.61, $CDCl_3$): δ 178.4 (C=O), 139.3

(C-2), 131.6 (C_α), 130.8 (C-7a), 124.2 (C-3), 121.9 (C-3a), 106.7 (C_β), 24.7 (C-4), 23.0 (C-7), 22.7 (C-5, C-6). IR (thin film, KBr plate): 3125, 3088, 2933, 2854, 2784, 2716, 1655, 1638, 1565, 1481, 1462, 1440, 1424, 1394, 1372, 1329, 1288, 1257, 1241, 1212, 1160, 1132, 1105, 1083, 1059, 970, 945, 869, 833, 815, 743, 705, 686, 636, 551, 486 cm^{-1}. Anal. Calcd for $C_{11}H_{13}NO$ (175.23): C, 75.40; H, 7.48; N 7.99. Found: C, 75.21; H, 7.62; N, 8.03.

5-Phenyl-1-vinylpyrrole-2-carbaldehyde (**2b**). Yield 66%. Yellowish oil, n_D^{23} 1.6550. ^1H NMR (400.13 MHz, $CDCl_3$): δ 9.63 (s, 1H, H_{CHO}), 7.42 (m, 5H, Ph) 7.40 (dd, 1H, H_X, $^3J_{B-X}$ 15.9 Hz, $^3J_{A-X}$ 8.7 Hz), 7.07 (d, 1H, H-3, $^3J_{3-4}$ 3.9 Hz), 6.34 (d, 1H, H-4, $^3J_{3-4}$ 3.9 Hz), 5.10 (d, 1H, H_A, $^3J_{A-X}$ 8.7 Hz), 4.87 (d, 1H, H_B, $^3J_{B-X}$ 15.9 Hz). ^{13}C NMR (101.6, $CDCl_3$): δ 179.5 (C=O), 142.3 (C-2), 133.3 (C_i), 131.5 (C-5), 131.2 (C_p), 129.3 (C_m), 128.5 (C_o), 128.5 (C-3), 124.4 (C-4), 112.7 (C_α), 112.6 (C_β). IR (thin film, KBr plate): 3121, 3065, 3027, 2985, 2928, 2803, 2721, 1655, 1636, 1595, 1564, 1530, 1497, 1453, 1440, 1421, 1350, 1328, 1288, 1223, 1065, 1033, 949, 919, 895, 869, 837, 788, 746, 686, 663, 606, 531, 462 cm^{-1}. Anal. Calcd for $C_{13}H_{11}NO$ (197.23): C, 79.16; H, 5.62; N 7.10. Found: C, 79.05; H, 5.92; N, 7.42.

5-(4-Methoxyphenyl)-1-vinylpyrrole-2-carbaldehyde (**2c**). Yield 82%. Yellowish crystals, mp 74–76 °C. ^1H NMR (400.13 MHz, $CDCl_3$): δ 9.55 (s, 1H, H_{CHO}), 7.40 (dd, 1H, H_X, $^3J_{B-X}$ 15.9 Hz, $^3J_{A-X}$ 8.7 Hz), 7.30 (d, 2H, H_o, $^3J_{o-m}$ 9.0 Hz), 6.96 (d, 1H, H-3, $^3J_{3-4}$ 3.9 Hz), 6.86 (d, 2H, H_m, $^3J_{o-m}$ 9.0 Hz), 6.24 (d, 1H, H-4, $^3J_{3-4}$ 3.9 Hz), 5.05 (d, 1H, H_A, $^3J_{A-X}$ 8.7 Hz), 4.85 (d, 1H, H_B, $^3J_{B-X}$ 15.9 Hz), 3.81 (s, 3H, Me). ^{13}C NMR (101.6 MHz, $CDCl_3$): δ 179.2 (C=O), 159.9 (C_p), 142.4 (C-2), 133.2 (C_i), 131.3 (C_α), 130.6 (C_o), 124.5 (C-3), 123.9 (C-5), 114.0 (C_m), 112.2 (C_β, C-4), 55.4 (Me). IR (KBr): 2967, 2924, 2828, 1638, 1597, 1565, 1523, 1497, 1440, 1421, 1375, 1333, 1308, 1288, 1223, 1165, 1099, 1023, 1004, 944, 910, 817, 786, 757, 730, 709, 676, 643, 586, 521, 434 cm^{-1}. Anal. Calcd for $C_{14}H_{13}NO_2$ (227.26): C, 73.99; H, 5.77; N 6.16. Found: C, 73.87; H, 5.81; N, 6.23.

5-(2-Thienyl)-1-vinylpyrrole-2-carbaldehyde (**2d**). Yield 88%. Cherry coloured crystals, mp. 34–35 °C. ^1H NMR (400.13 MHz, $CDCl_3$): δ 9.61 (s, 1H, H_{CHO}), 7.36 (dd, 1H, H-3′, $^3J_{3-4}$, 5.1 Hz, $^3J_{3-5}$, 1.1 Hz), 7.25 (dd, 1H, H_X, $^3J_{B-X}$ 15.7 Hz, $^3J_{A-X}$ 8.4 Hz), 7.20 (dd, 1H, H-4′, $^3J_{3-4}$, 5.1 Hz, $^3J_{4-5}$ 3.6 Hz), 7.06 (dd, 1 H,$H_{5'}$, $^3J_{4-5}$ 3.6 Hz, $^3J_{3-5}$, 1.1 Hz), 7.04 (d, 1H, H-3, $^3J_{3-4}$ 4.0 Hz), 6.45 (d, 1H, H-4, $^3J_{3-4}$ 4.0 Hz), 5.40 (d, 1H, H_A, $^3J_{A-X}$ 8.4 Hz), 5.24 (d, 1H, H_B, $^3J_{B-X}$ 15.7 Hz). ^{13}C NMR (101.61, $CDCl_3$): δ 179.20 (C=O), 135.15 (C-2′), 133.53 (C-2), 132.43 (C-5′), 130.87 (C_α), 127.91 (C-5), 127.55 (C-4), 127.01 (C-3′), 123.28 (C-3), 114.66 (C_β), 112.80 (C-4). IR (thin layer): 3106, 3077, 2992, 2846, 2817, 2791, 2726, 1669, 1655, 1640, 1592, 1563, 1511, 1470, 1435, 1411, 1362, 1330, 1113, 1292, 1230, 1199, 1094, 1076, 1038, 1006, 958, 906, 847, 786, 747, 704, 670, 634, 616, 581, 501, 461 cm^{-1}. Anal. Calcd for $C_{11}H_9NOS$ (203.26): C, 65.00; H, 4.46; N 6.89, S 15.77. Found: C, 65.11; H, 4.34; N 6.68, S 15.91.

1-Vinyl-4,5-dihydrobenzo[g]indole-2-carbaldehyde (**2e**). Yield 91%. Beige crystals, mp. 133–135 °C. ^1H NMR (400.13 MHz, $CDCl_3$): δ 9.54 (s, 1H, H_{CHO}), 7.70 (d, 1H, H-6, $^3J_{6-7}$ 8.1 Hz), 7.50 (dd, 1H, H_X, $^3J_{B-X}$ 15.7 Hz, $^3J_{A-X}$ 8.3 Hz), 7.23 (d, 1H, H-9, $^3J_{8-9}$ 9.1 Hz), 7.14 (m, 2H, H-7, H-8), 6.84 (s, 1H, H-3), 5.37 (d, 1H, H_A, $^3J_{A-X}$ 8.3 Hz), 5.36 (d, 1H, H_B, $^3J_{B-X}$ 15.7 Hz), 2.88 (t, 2H, H-4, $^3J_{4-5}$ 7.2 Hz), 2.66 (t, 2H, H-5, $^3J_{4-5}$ 7.2 Hz). ^{13}C NMR (101.61, $CDCl_3$): δ 178.5 (C=O), 138.3 (C-5a), 136.2 (C-9b), 133.6 (C-2), 132.5 (C_α), 128.7 (C-7), 127.8 (C-9a), 127.6 (C-6), 126.4 (C-8), 125.1 (C-3a), 124.2 (C-9), 121.2 (C-3), 113.7 (C_β), 30.6 (C-5), 22.1 (C-4). IR (KBr): 3098, 3028, 2982, 2961, 2894, 2834, 1640, 1606, 1535, 1504, 1467, 1436, 1416, 1373, 1335, 1297, 1278, 1236, 1185, 1158, 1136, 1094, 1045, 1027, 982, 943, 926, 913, 867, 839, 778, 762, 738, 716, 683, 664, 637, 599, 574, 549, 522, 471, 437 cm^{-1}. Anal. Calcd for $C_{15}H_{13}NO$ (223.27): C, 80.69; H, 5.87; N 6.27. Found: C, 80.81; H, 6.14; N 6.07.

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