In pursuance of our research on the development of a simple method for the synthesis of various nitrogen-containing heterocycles, we became interested in the heteroarylation of arenes/heteroarenes through AlCl$_3$ induced C–C bond-forming reactions. We anticipated that such a protocol could be a useful alternative to the Suzuki coupling reactions (when applied to a similar type of C–C bond formation reaction) as preparations of required boronic acids are often cumbersome. This prompted us to develop a novel method for the synthesis of 4-(hetero)aryl substituted 1-chlorophthalazines and 6,7-disubstituted pyrrolo[1,2-b]pyridazines via AlCl$_3$ induced C–C bond-forming reactions. Herein we report on the application of that methodology in the synthesis and structure determination by X-ray crystallography of 2-(1-methyl-1H-indol-3-yl)nicotinonitrile. The chemical structure of the title compound is shown in Fig. 1.

A mixture of 3-cyano-2-chloropyridine (0.2 g, 1.43 mmol) and AlCl$_3$ (0.23 g, 1.72 mmol) in 1,2-dichloroethane (10 mL) was stirred at 25˚C for 10 min under an anhydrous atmosphere. To this was added N-methylindole (0.19 g, 1.43 mmol), and the mixture was stirred at 25˚C for 30 min and then at 80 – 90˚C for 27 h. The mixture was poured into ice (20 g) and extracted with CHCl$_3$ (2 × 25 mL). The organic layers were collected, combined-washed with brine (2 × 10 mL), dried over Na$_2$SO$_4$, and filtered and concentrated under vacuum. The thus obtained residue was purified by recrystallization from 5% EtOAc-petroleum ether to afford the desired product.

The crystal structure of 2-(1-methyl-1H-indol-3-yl)nicotinonitrile C$_{15}$H$_{11}$N$_3$ was determined. The compound crystallizes from a methanol and chloroform solvent mixture in the triclinic system, space group $P1$ with the following unit-cell parameters: $a = 7.018(1)$, $b = 8.399(1)$, $c = 20.362(3)$Å, $\alpha = 90.731(4)$˚, $\beta = 90.149(5)$˚, $\gamma = 103.467(6)$˚, $Z = 4$, $V = 1167.1(3)$Å$^3$. The crystal structure was solved by direct methods and refined by full-matrix least squares to a final $R$-value of 0.058 with 4338 unique reflections.

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The X-ray structure of the title compound, shown in Fig. 2, contains the nicotinonitrile and indoyl group, with all of the bond lengths and angles normal. The torsion angles N2A–C6A–C7A–C10A in molecule A and

Fig. 1 Chemical diagram of the title compound.

Fig. 2 Molecular structure of the title compound. Thermal ellipsoids are drawn at 50%.
N2B–C6B–C7B–C10B in molecule B are 12.6(3) and 6.4(3), respectively, which shows that they are independent molecules. The packing in 3-dimension is stabilized by four intramolecular and two weak intermolecular interactions. The two intramolecular interactions of A are [C8A –H4A · N1A: H4A · N1A = 2.540(3)Å, C8A –N1A = 3.331(3)Å, C8A–H4A · N1A = 141.36(18)˚; C11A –H5A · N2A: H5A · N2A = 2.555(3)Å, C11A –N2A = 3.051(3)Å, C11A–H5A · N2A = 112.76(17)˚; and the two intramolecular interactions of B are C8B–H4B · N1B: H4B · N1B = 2.530(3)Å, C8B –N1B = 3.345(3)Å, C8B–H4B · N1B = 143.94(17)˚; C11B–H5B · N2B: H5B · N2B = 2.477(2)Å, C11B–N2B = 3.002(2)Å, C11B–H5B · N2B = 114.81(17)˚. These intramolecular interactions are responsible for the observed conformation of molecules A and B. The intermolecular interactions between molecules A and B are through carbon atom C3 of A and the nitrogen atom N1 (–x + 1, –y, –z + 1) of B through H1A (C3A-H1A–N1B: H1A–N1B = 2.610(3)Å, C3A-N1B = 3.410(3)Å, C3A-H1A–N1B = 142.00(2)˚) and between the carbon atom C3 of B and the nitrogen atom N1 (–x + 1, –y + 1, –z + 1) of A through H1B (C3B-H1B–N1A: H1B–N1A = 2.662(3)Å, C3B-N1A = 3.415(3)Å, C3B-H1B–N1A = 137.37(2)˚). These intermolecular interactions form a continuous zig-zag chain along the b axis.

### References