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Facile construction of 3-indolochromenes and 3-indoloxanthenes *via* EDDF catalyzed one-pot three component reactions

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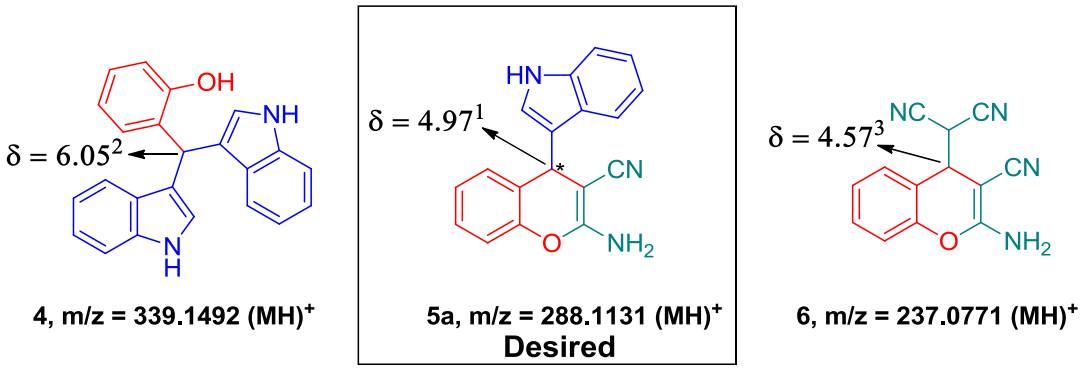
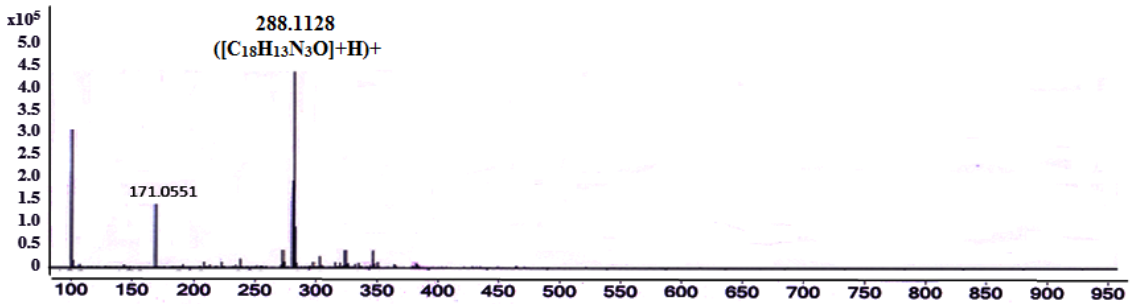
Supplementary Information

Spectroscopic analysis of reaction-crude (5a) for identification of possible side products



4, m/z = 339.1492 (MH)⁺ | **5a, m/z = 288.1131 (MH)⁺ Desired** | **6, m/z = 237.0771 (MH)⁺**



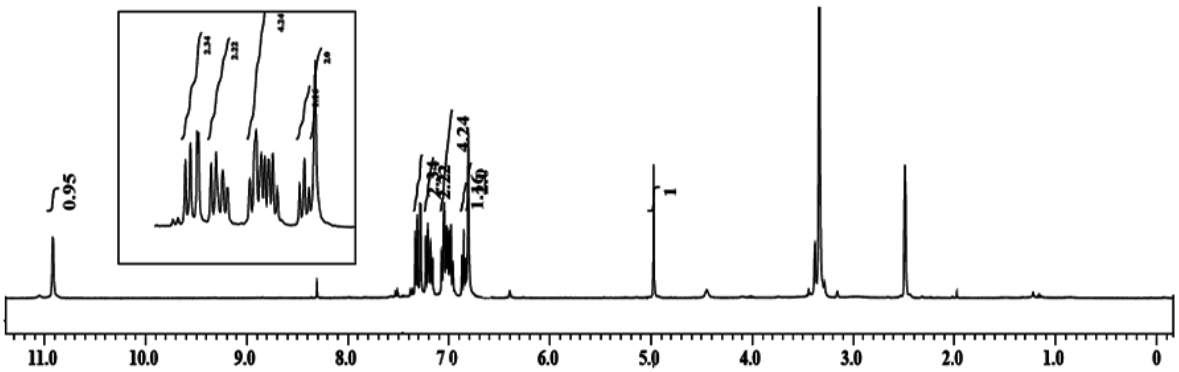


Figure 1: ESI-HRMS and ^{1}H NMR spectra of the crude for the synthesis of 3-indolochromene (**5a**) showing no traces of side products

The reaction of salicylaldehyde (**1a**), malononitrile (**2**) and indole (**3a**) could lead to possible formation of side-products **4** and **6** besides the desired 3-indolochromene **5a** (see Scheme 1 in manuscript). The reaction crude was taken and analysed spectroscopically (^{1}HNMR and ESI-HRMS) and the data is shown in Figure 1.

By looking at the mass spectrum, the formation of **4** and **6** can be ruled out as there are no peaks at m/z 339.1492 (calcd. MH^+ for **4**) and m/z 237.0771 (calcd. MH^+ for **6**). Also, we can confirm the formation of desired product (**5a**) over side products **4** and **6** by the presence of a characteristic singlet at δ 4.97,[1] with almost no traces of a singlet at δ 6.05 or a doublet at δ 4.57 which are the characteristic peaks of the possible side-products **4**[2] and **6**[3], respectively, in the ^{1}H NMR of crude product **5a.**

Spectroscopic analysis of reaction-crude (8a) for identification of possible side products

Following similar line of thought, ^{1}H NMR and mass spectrum (HRMS) of the reaction crude (**8a**, figure 2) was carefully scrutinized for any possible side-products, which confirmed that only formation of the desired 3-indoloxanthene **8a** took place in the reaction between salicylaldehyde (**1a**), 1,3-cyclohexadione (**7**) and indole (**3a**) (refer scheme 3 in the manuscript). The characteristic peak of the desired product (**8a**, δ = 5.34) was observed in the ^{1}HNMR spectrum (matched with literature data)[4] while no peak at δ 6.05 for **4** or at δ 4.64 for formation of **9**[5] was observed. Further, the HRMS spectra exhibited MH^+ peak of only the product **8a** (m/z = 316.1332; MNa^+ and MK^+ values were also detected) which established the exclusive formation of the desired indolo-xanthene (**8a**).

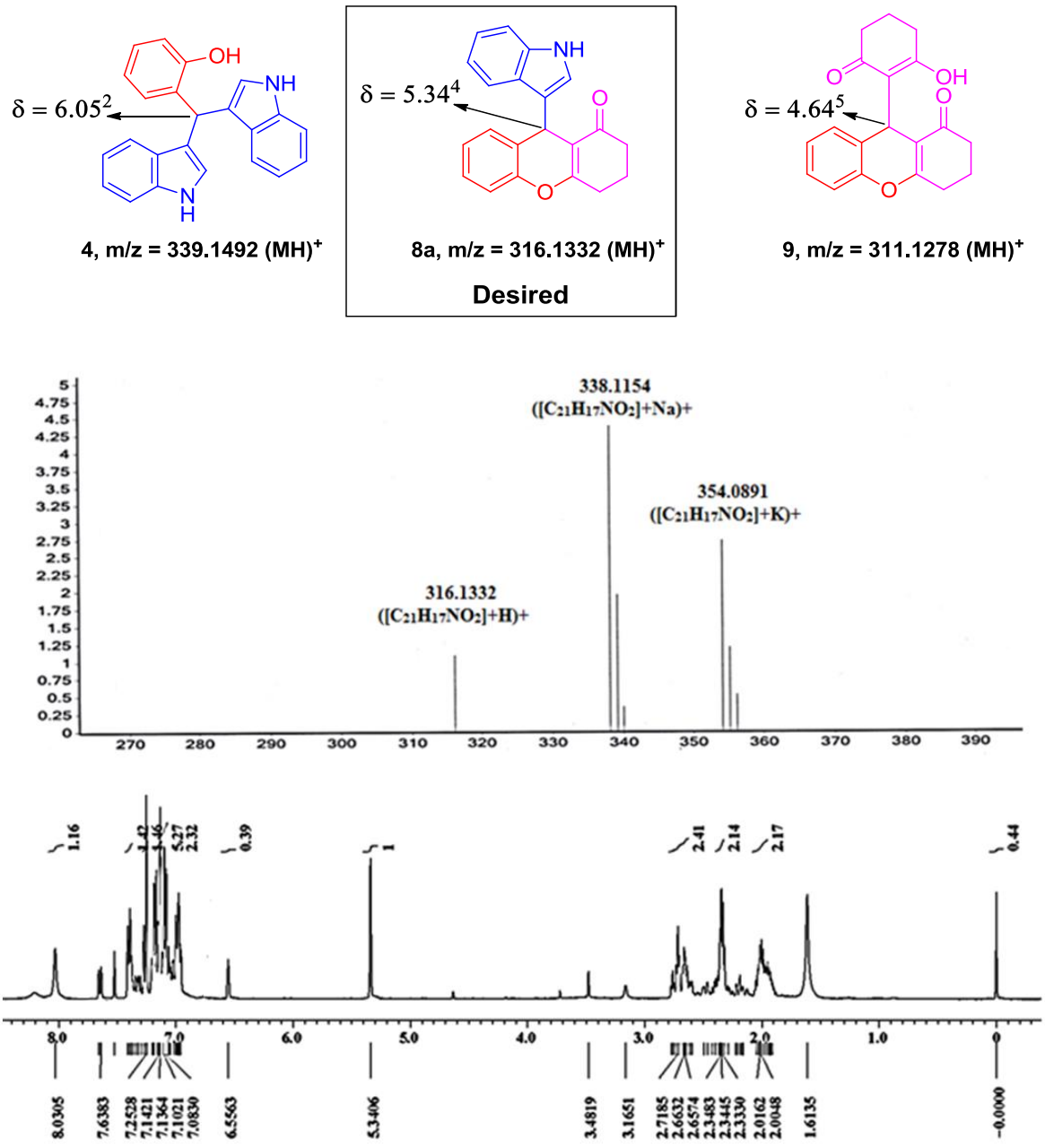


Figure 2: HRMS and ^{1}H NMR spectra of the crude product for the synthesis of 3-indolochromene (**8a**) showing no traces of side products.

References

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