Study of N^1-alkylation of indoles from the reaction of 2(or 3)-aminoindole-3-(or 2)carbonitriles with DMF-dialkylacetals.†

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Supporting Information

General Information

All reactions were monitored by thin-layer chromatography with silica gel 60 F254 pre-coated aluminium plates (0,25mm). Melting points of solid compounds were measured on an Electrothermal IA 9000 melting point apparatus (compounds **3** and **4**) or a STUART apparatus advanced SMP3 with a precision of ±0.5°C (compounds **1**, **2**, **5**-**9**) and are uncorrected. IR spectra were recorded on a PerkinElmer Spectrum 100 Series FT-IR spectrometer. Liquids and solids were applied on the Single Reflection Attenuated Total Reflectance (ATR) Accessories. Absorption bands are given in cm^{-1}.

^1H, ^{13}C and ^{19}F NMR spectra were recorded on an AVANCE 400 MHz spectrometer (compounds **3** and **4**) or on a Bruker DXP 300 spectrometer at 300, 75 and 282 MHz respectively (compounds **1**, **2**, **5**-**9**). Abbreviations used for peak multiplicities are s: singlet, d: doublet, t: triplet, q: quadruplet, m: multiplet and *br* (broad). Coupling constants J are given in Hertz (Hz) and chemical shifts are given in parts per million (δ) and calibrated with DMSO-d_6 or D_2O (residual solvent signals). Mass spectra analysis was performed by the Mass Spectrometry Laboratory of the University of Rouen. Mass spectra (EI) were recorded with a Waters ZQ 2000 and a Waters LCP 1er XR spectrometers.

Microwave experiments were conducted in two commercial microwave reactors especially designed for synthetic chemistry. Start SYNTH^{TM} (Milestone S.r.l. Italy) is a multi-mode cavity with a microwave power delivery system ranging from 0 to 1200 W. Open vessel experiments were carried out in a 250 mL round bottom flask fitted with a reflux condenser. The temperature was monitored via a fibre-optic contact thermometer protected in a Teflon coated ceramic well inserted directly in the reaction mixture or via contact-less infrared pyrometer. The vessel contents were stirred by means of an adjustable rotating magnetic plate located below the floor of the microwave cavity and a Teflon-coated magnetic stir bar inside the vessel. Temperature, pressure and power profiles were monitored in both cases through the EASY-Control software provided by the manufacturer.

Multi SYNTH^{TM} (Milestone S.r.l. Italy) is a novel dedicated microwave system for synthetic applications. It allows a fast reaction optimization providing high energy density in a single-mode like configuration and an efficient scale-up (maximum working volume 300 mL) through parallel synthesis in a multi-mode configuration. The instrument features a special shaking system that ensures high homogeneity of the reaction mixtures. It is equipped with an indirect pressure-control through pre-calibrated springs at the bottom of the vessel shields and with both, contact-less infrared pyrometer (IRT) and fibre-optic contact thermometer (FO) for accurate temperature measurement. It is noteworthy that the IRT can be calibrated directly on the temperature read by the FO to ensure the highest accuracy and reproducibility.