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Nova sintetska metoda za dobivanje *N*-metil aromatičnih imina bez upotrebe rastvarača

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Sinteza *N*-metil aromatičnih imina bez prisustva rastvarača vršena je homogenizacijom aromatičnih aldehida, metilamin-hidrohlorida i baze, u tarioniku pistilom. Najbolji prinosi ostvareni su kada je reakcija izvođena u prisustvu viška $CH_3NH_2{\cdot}HCl$ i $NaHCO_3$ (najčešće u odnosu 1:5:5 = $ArCHO:CH_3NH_2{\cdot}HCl:NaHCO_3$), a reakciona smeša ostavljena nakon homogenizacije da stoji na sobnoj temperaturi od 1 h (u slučaju aldehida sa elektron-akceptorskim supstituentima) do 24 h (u slučaju aldehida sa elektron-donorskim supstituentima). Nakon jednostavne obrade reakcione smeše dobijeni proizvodi su bili uglavnom dovoljno čisti za spektralnu karakterizaciju. Na ovaj način sintetisan je 31 imin, od kojih osam predstavljaju potpuno nova jedinjenja, a njihove strukture su potpuno spektralno okarakterisane (^{1}H- i ^{13}C-NMR, IR, MS). U slučaju salicilaldehida i 4-hlorbenzaldehida izvršena je sinteza sa gramskim količinama reaktanata. Ovaj način sinteze *N*-metil aromatičnih imina ne samo da obezbeđuje dobre prinose, već i eliminiše nedostatke tradicionalne sinteze *N*-metil imina, kao što su: upotreba opasnih rastvarača, manje ili više skupih katalizatora i rad sa anhidrovanim gasovima pod pritiskom.

Simple and efficient one-pot solvent-free synthesis of *N*-methyl imines of aromatic aldehydes

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A one-pot solvent-free synthesis of *N*-methyl imines in good to excellent yields was performed by grinding together aromatic aldehydes and methylamine hydrochloride in the presence of a base. The best yields were achieved when an excess of $CH_3NH_2{\cdot}HCl$ and $NaHCO_3$ was used (usually in a molar ratio 1:5:5 = $ArCHO:CH_3NH_2{\cdot}HCl:NaHCO_3$), allowing the reaction to proceed for 1 h (in the case of aromatic aldehydes containing electron-withdrawing substituents) or overnight (in the case of electron-rich aldehydes). After a simple work-up the obtained products were mostly pure enough for spectral characterization. In this way, 31 *N*-methyl imines were prepared, among which 8 were synthesized for the first time. All synthesized products were fully spectrally (^{1}H- and ^{13}C-NMR, IR, MS) characterized. In the case of salicylaldehyde and 4-chlorobenzaldehyde the synthesis of the corresponding imines was also conducted on a gram-scale. This approach not only provides good to high yields, but also eliminates the disadvantages of the traditional synthesis of *N*-methyl imines, such as the use of hazardous solvents and more or less expensive catalysts and the necessity of work/handling with an anhydrous gas in pressurized containers.

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