SYNTHESIS AND CHARACTERIZATION OF *N*-ACETYLACETAMIDO-1*H*-1,2,3-TRIAZOLE DERIVATIVES

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Introduction

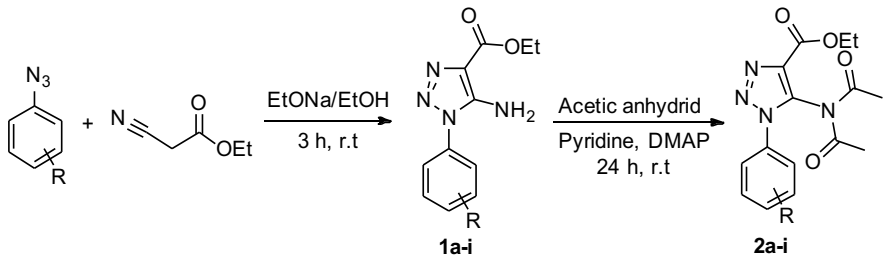
Diacetyl compounds have recently attracted significant interest due to their broad spectrum of biological activities such as anticancer (El Massry et al., 2012), herbicide (Shasheva et al., 2017), antifungal (Zhou et al., 2010), anti-ulcer and anti-secretory (Beatie et al., 1979). This work describes the synthesis of diacetyl-triazoles via the diacetylation of amino-triazole derivatives.

Method

The methods for the preparation of diacetylamines, in particular involve the reaction of amines with acetylating agents, such as acetyl chloride, acetic anhydride, and ketene and enol esters in the presence of catalysts, such as pyridine, sulphuric acid, and sodium acetate (Avyangar et al., 1984).

Results / Discussion

In this work, ethyl 5-amino-1*H*-triazole-4-carboxylate derivatives **(1a-i)** were obtained from the reaction between phenylazides and ethyl cyanoacetate in the presence of sodium ethoxide at room temperature for 3 hours (Hoover et al., 1956). Then, the derivatives **(1a-i)** were reacted with acetic anhydride, pyridine and DMAP as the catalyst at room temperature for 24 hours to form nine new *N*-diacetyl derivatives **(2a-i)**. All synthesized compounds were characterized using H^1- and C^{13}-NMR, as well as having their structure being determined by X-ray crystallography.



1a R = H 62%	**2a** R = H 64%
1b R = 4-CH_3 65%	**2b** R = 4-CH_3 70%
1c R = 4-OCH_3 67%	**2c** R = 4-OCH_3 86%
1d R = 4-Cl 60%	**2d** R = 4-Cl 57%
1e R = 4-F 60%	**2e** R = 4-F 50%
1f R = 4-Br 93%	**2f** R = 4-Br 61%
1g R = 2-Cl 56%	**2g** R = 2-Cl 62%
1h R = 2,5-Cl 65%	**2h** R = 2,5-Cl 64%
1i R = 4-NO_2 87%	**2i** R = 4-NO_2 74%

Conclusion

In this work, eighteen 1,2,3-triazole derivatives were synthesized, among which nine are unpublished in the literature. Trough the monocrystal X-ray crystallography technique it was possible to elucidate the chemical structures of derivatives obtained from *N*-acetylation reactions.

Acknowledgments

Fellowships granted by the Brazilian agencies CNPq, CAPES and FAPERJ are gratefully acknowledged. FAPERJ (Project E - 26/200.930/2017) and CAPES (Finance Code 001). The authors thank LDRX-UFF (www.ldrx.uff.br/) for the X-ray diffraction analysis.

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