

Comprehensive Organic Chemistry Experiments for the Laboratory Classroom

Edited by Carlos A M Afonso, Nuno R Candeias,
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12.2.5. An “Out-of-the-Box” Example in Heterocyclic Chemistry: Synthesis of 3,6-Bis-(3,5-dimethyl-1*H*-pyrazol-1-yl)-1,2,4,5-tetrazine

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<p>Preparation of the starting material:</p> <p style="text-align: right;">(1)</p>			
<p>Synthesis:</p> <p style="text-align: right;">(2)</p> <p style="text-align: right;">(3)</p>			
Number of sessions (duration of each session)	Hazard level	Difficulty level	Level of study
3 (3 h + 6 h + 6 h)	High	Medium	Intermediate
Class names Amines, 2,4-diketones, imines, nitrites, tetrazines			
Concepts involved This procedure is simple and provides the students with an “out-of-the-box” example of how to synthesize heterocyclic aromatic compounds bearing more than three nitrogen atoms that does not follow the usual Pinner method. The experiment involves reaction routes widely used in heterocyclic ring synthesis: C–heteroatom bond formation through C=O nucleophilic addition to 2,4-dicarbonyl compounds, intramolecular cyclization and an oxidation reaction			
Chemicals needed Guanidine hydrochloride, hydrazine hydrate (80% solution), 1,4-dioxane, acetylacetone (2,4-pentanedione), isoamyl nitrite, dichloromethane, anhydrous magnesium sulfate, silica gel, deuterated chloroform			
Equipment and experimental techniques involved Vacuum filtration apparatus, liquid–liquid extraction, rotatory evaporation apparatus, addition funnel, chromatography column, laboratory weighing equipment, heating plate with magnetic stirring, reflux apparatus			
Keywords Column chromatography, FTIR spectroscopy, heterocyclic ring formation, NMR spectroscopy, oxidation, tetrazines, vacuum filtration			

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