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# Chromatography-free multicomponent synthesis of thioureas with aqueous polysulfide solutions under batch and continuous flow conditions

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## Abstract

We have developed a batch and continuous flow (CF) process for the three-component chromatography-free synthesis of thioureas starting from isocyanides, amidines or amines, and elemental sulfur in water. First, screening a large pool of organic and inorganic bases, we realized the preparation of aqueous polysulfide solutions from elemental sulfur. These solutions facilitated the separation of the excess of sulfur from the crystallized products by a simple filtration, retaining sulfur in the mother liquid. This approach enabled us to tackle the otherwise mandatory chromatographic purification in sulfur-based reactions. Moreover, applying polysulfide solutions, we carried out the convenient CF application of sulfur. Presenting thioureas of a broad variety synthesized by this procedure, highlighting challenging *N, N'*-diaryl derivatives, confirms the reasonable substrate and functional group tolerance of the developed protocol.

**Keywords:** multicomponent reaction; sulfur; thiourea; chromatography-free, continuous flow

## Introduction and Objectives

Sulfur-containing compounds, such as thioureas, are widely known as biologically active molecules [1] and functional organic materials [2]. Notably, in the last two decades, thioureas were also applied as highly selective and efficient organocatalysts [3]. Given the wide utility of thioureas, their clean and efficient synthesis is of high interest. Elemental sulfur is a bench-stable, environmentally benign, inexpensive and nontoxic reagent offering an atom economical and safe alternative to incorporate the sulfur atom into products [4]. Certain nucleophiles, such as aliphatic amines, can generate open-chain ionic polysulfide anions from elemental sulfur [5], resulting in an efficient nucleophilic sulfurating reagent. In the last two decades, CF synthesis has become a powerful and versatile tool in synthetic organic chemistry. The main advantages of that approach are facile automation, reproducibility, safety, and process reliability, because of constant reaction parameters (temperature, time, concentrations etc.) can be assured [6]. The CF techniques are suitable for multistep [7] and automated synthetic processes, which is a burgeoning and exciting area of research [8]. In the past decade, a couple of innovative, multicomponent and one-pot procedures have been published for the use of elemental sulfur in synthetic procedures leading to the formation of thioureas [9]. However, the

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