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Synthesis and Spectral Characteristics of *N*-(1-(((2*E*,4*E*)-6-(2-Bromophenyl)-3-cyclohexyl-2-(cyclohexylimino)-2,3-dihydro-4*H*-1,3,5-oxadiazin-4-ylidene)amino)-2,2,2-trichloroethyl)acetamide †

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Abstract

This work presents a step-by-step synthesis of a new derivative of 1,3,5-oxadiazine. In the first step, by reacting *N*-(1-amino-2,2,2-trichloroethyl)acetamide with 2-bromobenzoyl isothiocyanate, a previously undescribed thiourea, *N*-((1-acetamido-2,2,2-trichloroethyl)carbamothioyl)-2-bromobenzamide, was obtained. In the next step, this compound was treated with dicyclohexylcarbodiimide (DCC), resulting in cyclization and the formation of a new derivative of 1,3,5-oxadiazine-*N*-(1-(((2*E*,4*E*)-6-(2-bromophenyl)-3-cyclohexyl-2-(cyclohexylimino)-2,3-dihydro-4*H*-1,3,5-oxadiazin-4-ylidene)amino)-2,2,2-trichloroethyl)acetamide. The reaction was carried out in acetonitrile at reflux for one hour. Presumably, in the first step of the transformation, under the action of DCC, the elimination of a hydrogen sulfide molecule occurs with the formation of an intermediate carbodiimide derivative-*N*-(((1-acetamido-2,2,2-trichloroethyl)imino)methylene)-2-bromobenzamide. This intermediate then undergoes a [4+2] cycloaddition reaction with another DCC molecule to form the desired 1,3,5-oxadiazine product in 82% yield. Its structure was confirmed by NMR spectroscopy (^{1}H and ^{13}C).

Keywords: 1,3,5-oxadiazine; thiourea; dicyclohexylcarbodiimide; [4+2] cycloaddition; heterocyclization; synthesis

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1. Introduction

In recent decades, 1,3,5-oxadiazine derivatives have attracted growing attention across various scientific fields [1–5]. Compounds containing the 1,3,5-oxadiazine ring are considered in pharmacy and medicine as potential chemotherapeutic agents [4], as well as antibacterial [7–11] and antifungal agents [10,11]. They are also important in agriculture as insecticides (e.g., thiamethoxam [13,14] and its analogues [15,16]), herbicides [17], and larvicides [18–20]. Beyond biological activity, these compounds serve as valuable synthons and catalysts in organic synthesis and have been applied in the production of ionic liquids [21], polymers [22], and explosives [23]. Cucurbit[n]uryls incorporating a

1,3,5-oxadiazine ring are of particular interest in supramolecular chemistry for constructing molecular clips [24–33].

Most 1,3,5-oxadiazines are synthetic; only two natural representatives are known [34,35]. Their main synthetic routes involve [4+2]-cycloaddition [8–11,17–19,36–40], intramolecular cyclization of diols [15,16,23,41–46], bisamidials [47], or substituted thioureas [48–50]. Recently, their preparation via transformation of other heterocyclic systems has also been reported [23,51]. This article describes the synthesis of a new 1,3,5-oxadiazine derivative: *N*-(1-(((2*E*,4*E*)-6-(2-bromophenyl)-3-cyclohexyl-2-(cyclohexylimino)-2,3-dihydro-4*H*-1,3,5-oxadiazin-4-ylidene)amino)-2,2,2-trichloroethyl)acetamide.

2. Materials and Methods

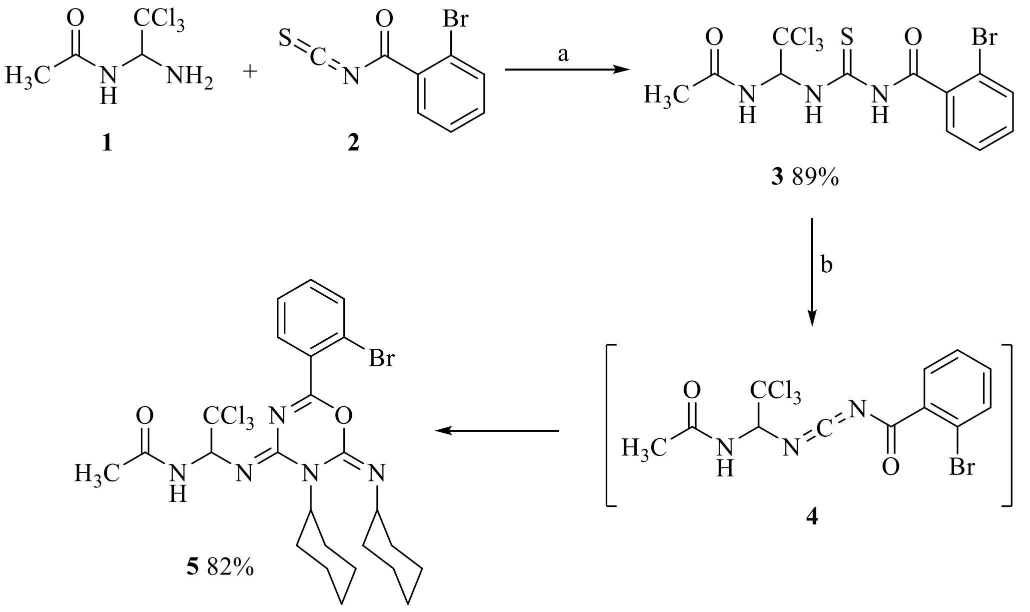
Spectral studies, including ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz), were conducted on DMSO-d_6 solutions using a Varian Agilent VNMRS 400 MHz spectrometer. Residual solvent signals served as internal standards. Elemental analysis was performed with a LECO CHNS-900 instrument. The reaction progress and purity of the synthesized compounds were monitored by thin-layer chromatography on Silufol UV-254 plates with a chloroform and acetone (3:1) eluent ratio. Melting points were determined in open capillaries.

Synthesis of *N*-((1-acetamido-2,2,2-trichloroethyl)carbamothioyl)-2-bromobenzamide (**3**). A solution of 10 mmol of 2-bromobenzoyl isothiocyanate (**2**) in 10 mL of acetonitrile was added to 10 mmol (2.05 g) of *N*-(1-amino-2,2,2-trichloroethyl)acetamide (**1**) [52] in 15 mL of MeCN. The mixture was brought to a boil and left at room temperature for 12 h. The resulting precipitate of thiourea (**3**) was filtered, washed with 10 mL of acetonitrile, and purified by recrystallization from methanol. Pale yellow crystals; yield 89% (3.98 g); mp 190–192 °C (MeOH); R_f = 0.74. ^{1}H NMR: δ 12.25 (s, 1H, NH), 11.54 (d, J = 9.3 Hz, 1H, NH), 9.25 (br. s, 1H, NH), 7.71 (d, J = 7.3 Hz, 1H, $H_{arom.}$), 7.57 (d, J = 7.3 Hz, 1H, $H_{arom.}$), 7.51–7.44 (m, 2H, $H_{arom.}$), 7.27 (dd, J = 8.8, 9.3 Hz, 1H, CH), 1.97 (s, 3H, CH3). Anal. Calcd (%) for $C_{12}H_{11}BrCl_3N_3O_2S$ (447.55): C, 32.20; H, 23.76; N, 9.39; S, 7.16. Found: C, 32.13; H, 23.71; N, 9.45; S, 7.22.

Synthesis of *N*-(1-(((2*E*,4*E*)-6-(2-bromophenyl)-3-cyclohexyl-2-(cyclohexylimino)-2,3-dihydro-4*H*-1,3,5-oxadiazin-4-ylidene)amino)-2,2,2-trichloroethyl)acetamide (**5**). A mixture of 10 mmol of thiourea 3 and 20 mmol (4.13 g) of dicyclohexylcarbodiimide was added to 40 mL of dry acetonitrile and refluxed for 40–50 min. The reaction mixture was cooled, and the precipitated crystals were filtered and washed with 10 mL of MeCN. The final product was purified by recrystallization from methanol. White crystals; yield 82% (5.08 g); mp 169–171 °C (MeOH); R_f = 0.75. ^{1}H NMR: δ 8.50 (br. s, 1H, NH), 7.91 (d, J = 7.0 Hz, 1H, $H_{arom.}$), 7.84 (d, J = 7.0 Hz, 1H, $H_{arom.}$), 7.66–7.58 (m, 2H, $H_{arom.}$), 6.78 (d, J = 8.8 Hz, 1H, CH), 4.81–4.75 (m, 1H, cyclohexyl), 3.79 (br. s, 1H, cyclohexyl), 2.75–2.59 (m, 2H, cyclohexyl), 1.91 (s, 3H, CH_3), 1.82–1.12 (m, 18H, cyclohexyl). ^{13}C NMR: δ 168.7 (C=O), 157.4 (C=N), 147.5 (C=N), 135.1 (C=N), 134.3, 134.0, 132.4, 129.9, 128.0, 120.7 ($C_{arom.}$), 103.8 (CCl_3), 72.4 (CH), 56.0, 52.5, 33.8, 33.6, 26.9, 26.1, 26.0, 25.5, 25.2, 23.7, 22.7 (cyclohexyl). Anal. Calcd (%) for $C_{25}H_{31}BrCl_3N_5O_2$ (619.81): C, 48.45; H, 5.04; N, 11.30. Found: C, 48.39; H, 4.98; N, 11.38.

3. Results and Discussion

The starting compound, *N*-(1-amino-2,2,2-trichloroethyl)acetamide (**1**), was synthesized according to a previously reported procedure [52]. It readily reacted with 2-bromobenzoyl isothiocyanate (**2**), yielding *N*-((1-acetamido-2,2,2-trichloroethyl)carbamothioyl)-2-bromobenzamide (**3**) (Scheme 1). The product was obtained in almost quantitative yield, reaching approximately 89% after recrystallization. Subsequently, thiourea **3** was treated with dicyclohexylcarbodiimide (DCC). Under the influence of DCC, hydrogen sulfide was eliminated from thiourea 3, most likely leading to the formation of carbodiimide intermediate **4**. This intermediate then participated in a [4+2] cycloaddition with an additional DCC molecule, affording the target oxadiazine **5**.



Scheme 1. Synthesis of *N*-((1-acetamido-2,2,2-trichloroethyl)carbamothioyl)-2-bromobenzamide (**3**) and its cyclisation into 1,3,5-oxadiazine (**5**). Reagents and conditions: (a) MeCN, reflux 1–2 min, r.t. 12 h; (b) 2 DCC, MeCN, reflux 40–50 min, r.t. 1–2 h.

The structures of the synthesized compounds were confirmed by spectral analysis. In the ^{1}H NMR spectrum of compound **3**, signals corresponding to three NH protons were detected, along with a characteristic doublet–doublet signal from the CH proton of the alkylamide fragment. In contrast, the spectrum of oxadiazine 5 displayed a single NH proton signal and a doublet from the CH group. Additionally, the ^{1}H NMR spectrum of compound **5** contained signals corresponding to 22 protons of two cyclohexyl rings, which is consistent with the [4+2] cycloaddition process. The ^{13}C NMR data further corroborated the formation of oxadiazine 5, revealing the absence of the C=S signal, the presence of a single C=O signal at 168.7 ppm, and three C=N signals in the 157.4–135.1 ppm range.

4. Conclusions

In summary, we have developed a convenient and efficient method for the synthesis of a new 1,3,5-oxadiazine derivative. Our synthetic approach is based on the reaction of *N*-(1-amino-2,2,2-trichloroethyl)acetamide with 2-bromobenzoyl isothiocyanate, followed by DCC-mediated cyclization through a [4+2] cycloaddition mechanism. The target compound was obtained in high yield, demonstrating the effectiveness of the developed method. The structures of the intermediates and final product were confirmed by ^{1}H and ^{13}C NMR spectroscopy, which clearly indicated the transformation of thiourea into the oxadiazine core. The absence of the C=S signal and the presence of characteristic C=N signals supported the successful ring formation. This synthetic strategy provides a practical pathway for the preparation of structurally diverse oxadiazines. The results of our work may serve as a foundation for the further design of heterocyclic systems with potential biological activity.

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