



Facile regiodivergent synthesis of spiro pyrrole-substituted pseudothiohydantoins and thiohydantoins via reaction of [e]-fused 1*H*-pyrrole-2,3-diones with thiourea

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Full Research Paper

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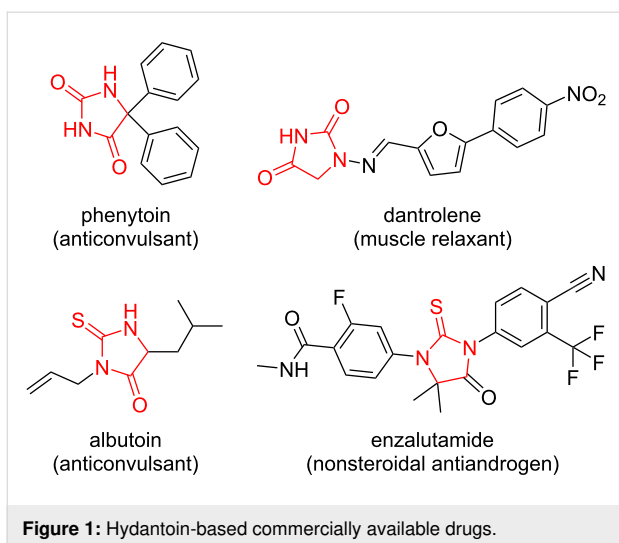
Abstract

A highly divergent synthesis of regioisomeric thiohydantoins and pseudothiohydantoins spiro-fused to a pharmacologically valuable pyrrole-2-one fragment involving the reaction of [e]-fused 1*H*-pyrrole-2,3-diones with thioureas was developed. The obtained spiro pseudothiohydantoin derivatives were found to undergo a pseudothiohydantoin–thiohydantoin rearrangement. The reactions were shown to proceed under catalyst-free conditions in good yields, and the products were isolated without applying preparative chromatography methods.

Introduction

Hydantoin (imidazolidine-2,4-dione) derivatives are omnipresent among biologically active compounds [1]. Many of them are commercially available drugs, for example, anticonvulsants phenytoin [2] and albutoin [3], the muscle relaxant dantrolene [4], or the nonsteroidal antiandrogen agent enzalutamide [5] (Figure 1).

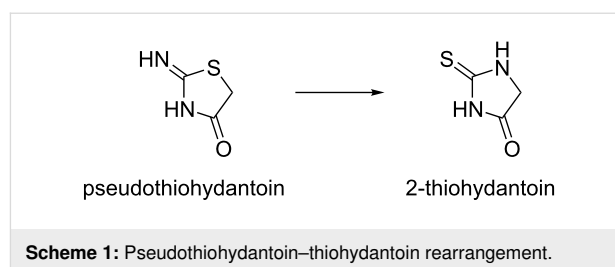
Despite this fact, hydantoin derivatives belong to a special group of scaffolds in medicinal chemistry. Indeed, they are structurally related to rhodanine (2-thioxothiazolidin-4-one), and sometimes are classified as pan-assay interference compounds (PAINS), which are abhorrent in high-throughput screenings [6]. To clarify whether such compounds are privi-



leged scaffolds or promiscuous binders, Mendgen and co-workers performed a systematic comparative study on rhodanines and related structures with respect to their medicinal chemistry properties [7]. As a result, it was shown that such structures could be suitable scaffolds for medicinal chemistry under proper conditions of biological evaluation, while their medicinal chemistry properties dramatically depend on the structure of the five-membered heterocyclic moiety and substituents in the C-5 position [7].

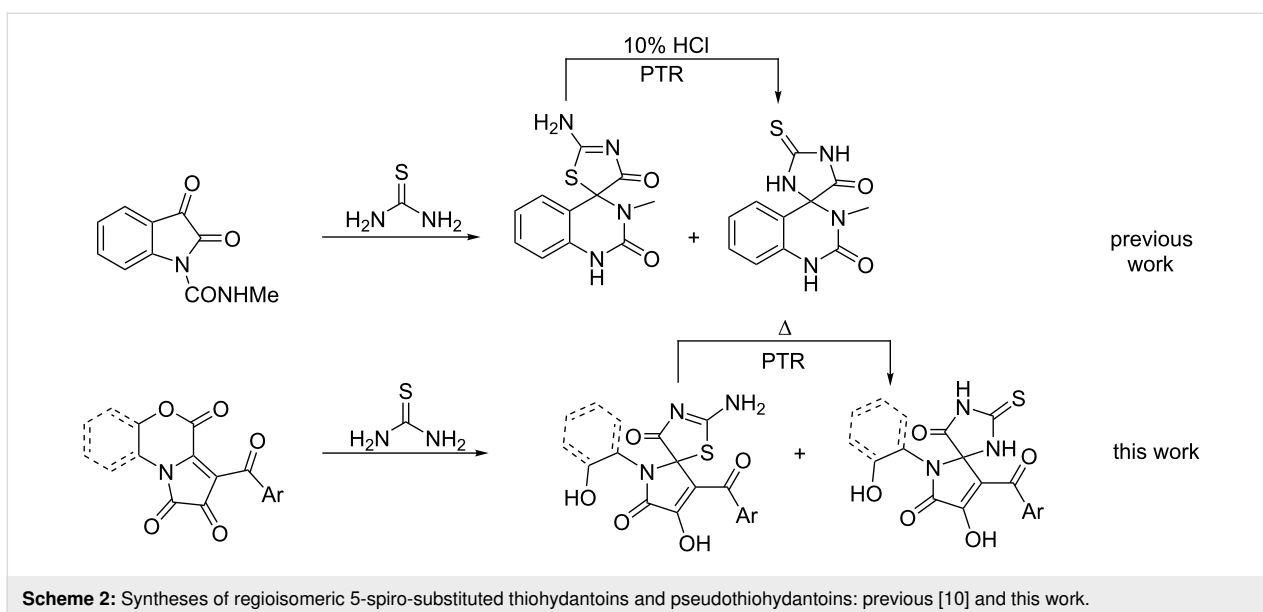
Some pseudothiohydantoins (2-iminothiazolidin-4-ones) are known to undergo a pseudothiohydantoin–thiohydantoin rearrangement (PTR, Scheme 1) [8–11], which is a special case of a quite poorly investigated iminothiolactone–thiolactam rearrangement [12–16]. This reaction offers attractive opportunities

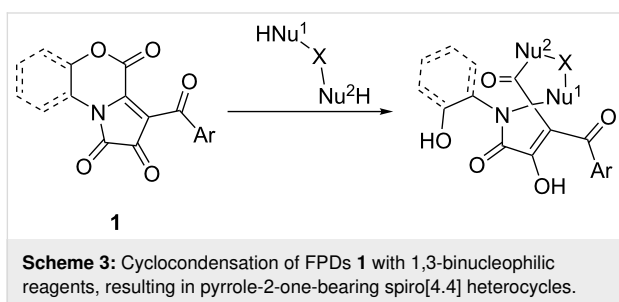
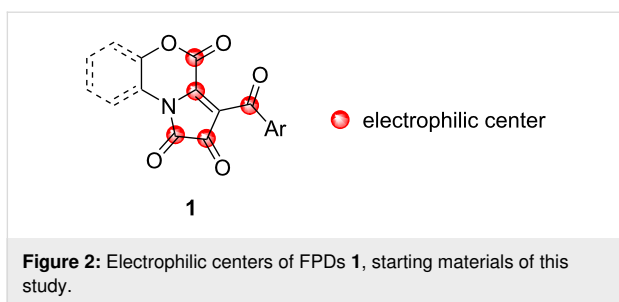
for the design of libraries of regioisomeric hydantoin-based compounds for drug discovery.



Spiro heterocycles are relatively new and insufficiently investigated structures in medicinal chemistry [17,18]. The introduction of spiro-fused cyclic moieties in small molecules increases the degree of their structural (shape) complexity, which may lead to the reduced binding promiscuity and improved clinical success [19]. Thus, recent development of 3D modelling methods facilitated investigations on the importance of 3D properties of small-molecular drug candidates [20–22] and inspired chemists to develop diversity-oriented methods for complex 3D molecules [23,24].

Considering that to the best of our knowledge, only a sole report exists [10] that describes the synthesis of regioisomeric 5-spiro-substituted thiohydantoins and pseudothiohydantoins and their PTR (Scheme 2), we were encouraged to develop a divergent synthetic approach to access regioisomeric thiohydantoins and pseudothiohydantoins that are spiro-fused to a pharmacologically valuable pyrrole-2-one fragment and to investigate the scope and conditions of their PTR behavior (Scheme 2).





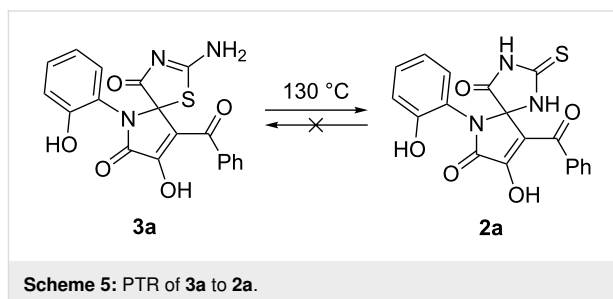
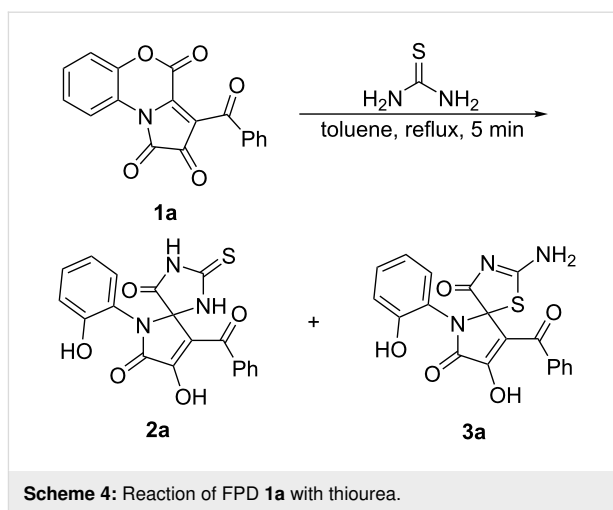
Results and Discussion

1*H*-Pyrrole-2,3-diones fused at [*e*]-side (FPDs **1**) can be viewed as a versatile polyelectrophilic synthetic platform (Figure 2), enabling facile preparation of libraries of heterocyclic molecules with an emphasis on skeletal diversity from a single set of reagents [25–27].

One of the most intriguing chemical properties of FPDs **1** is their ability to undergo a cyclocondensation with 1,3-binucleophilic reagents to form spiro[4.4] heterocycles bearing a pharmacophoric pyrrole-2-one moiety (Scheme 3) [28,29]. We employed this feature as a key step in the development of a straightforward and concise synthetic approach towards regioisomeric thiohydantoin and pseudothiohydantoin spiro-fused to a pyrrole-2-one fragment.

To test the preparation of the target spiro-fused pseudothiohydantoin and thiohydantoin, we examined the reaction of FPD **1a** with thiourea by heating equimolar amounts of the reagents in toluene for 5 min (until the disappearance of the dark violet color of FPD **1a**). The reaction mixture was examined by UPLC–MS. Two major products with $m/z = 396$ ($[M + H]^+$, ESI^+) were observed in a ratio of $\approx 1:1$, which corresponded to adducts of thiourea with FPD **1a**. The adducts were isolated, and their structures were elucidated as the desired spiro-fused thiohydantoin **2a** and pseudothiohydantoin **3a** (Scheme 4).

It should be pointed out that earlier, we have communicated initial data on this transformation [30,31]. However, taking into account more advanced structure elucidation methods employed in the present work (e.g., NMR, single crystal X-ray



diffraction), we concluded that the structures of the products were identified incorrectly, and they are revised herein (for a detailed revision see Supporting Information File 1).

Next, to find out whether compounds **2a** and **3a** were prone to undergo PTR, they were heated in open capillaries at 130 °C for 2 h, and the resulting mixtures were examined by UPLC–MS. As a result, we found that compound **2a** did not rearrange, and compound **3a** partially converted into its regioisomer **2a** due to irreversible PTR (Scheme 5).

Possibly, when carrying out the reaction of FPD **1a** with thiourea by refluxing the reaction mixture in toluene, part of the product **3a** was converted into **2a** because of PTR. We assumed that at lower temperatures, the main product of the reaction could be pseudothiohydantoin **3a**. To prove this assumption, we examined the reaction of FPD **1a** with thiourea at room temperature in various solvents (Table 1).

Obviously, the ratio of products **2a** and **3a** was dependent not only on the temperature of the reaction, but also on the polarity of the solvent. We found that optimal conditions for dominant formation of pseudothiohydantoin **3a** were applied by stirring the reaction mixture at room temperature in ethyl or butyl acetate (Table 1, entries 2 and 6).

Table 1: Reaction of FPD **1a** with thiourea at room temperature in various solvents.^a

Entry	Solvent	2a:3a ^b
1	acetone	30:70
2	ethyl acetate	5:95
3	1,4-dioxane	50:50
4	acetic acid	30:70
5	acetonitrile	50:50
6	butyl acetate	5:95
7	toluene	traces ^c

^aReaction mixtures were stirred until the disappearance of the dark violet color of FPD **1a** (about 2–4 h). ^bAccording to UPLC–MS data. ^cThe reaction proceeded too slowly, and FPD **1a** underwent hydrolysis faster than reaction with thiourea (the reaction vessel was contaminated with water during samplings for analyses).

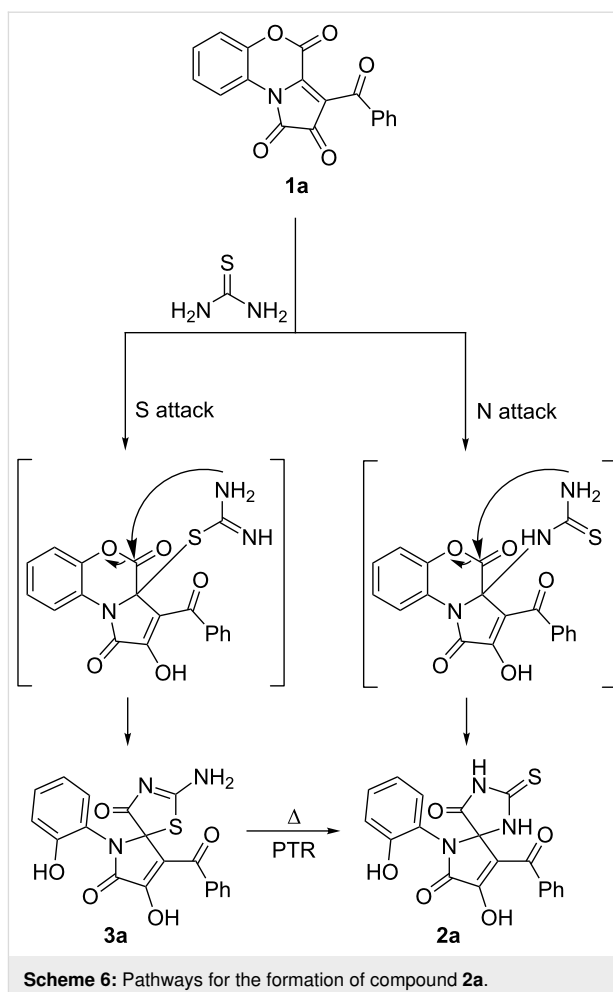
For the development of a convenient procedure for thiohydantoin **2a** without isolation of its regioisomer **3a** being required, we carried out the reaction of FPD **1a** with thiourea in various solvents under reflux (Table 2).

Table 2: Reaction of FPD **1a** with thiourea at reflux in various solvents.^a

Entry	Solvent	2a:3a ^b
1	acetone	30:70 ^c
2	ethyl acetate	22:78 ^c
3	1,4-dioxane	90:10 ^c
4	1,4-dioxane	99:1 ^d
5	acetic acid	30:70 ^c
6	acetonitrile	25:75 ^c
7	butyl acetate	95:5 ^c
8	toluene	50:50 ^c

^aReaction mixtures were refluxed until the disappearance of the dark violet color of FPD **1a** (about 5–15 min). ^bAccording to UPLC–MS data. ^cThe reagents were mixed prior to the heating. ^dThiourea was added to a boiling solution of FPD **1a**.

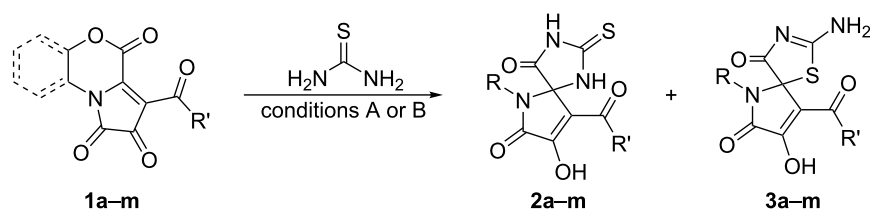
As a result of this optimization, we established that thiohydantoin **2a** was formed as major product upon refluxing the reaction mixture in butyl acetate or 1,4-dioxane (Table 2, entries 3, 4, and 7). Interestingly, at different reagent ratios, the yield of compound **2a** was affected as well (Table 2, entries 3 and 4). When the reagents were mixed prior to heating (Table 2, entry 3), the yield of compound **2a** was lower than when the reagents were mixed in the boiling solvent (Table 2, entry 4). Probably, compound **2a** was formed not only as a result of the corresponding PTR, but as a result of a concurrent attack of the amino group of thiourea on the C-3a atom of FPD **1a** (Scheme 6).



Using the optimization data, we obtained a series of spiro-fused thiohydantoin **2a–m** and pseudothiohydantoin **3a–m** by utilizing various FPDs **1a–n** (Table 3). The aryl group-bearing FPDs **1a–m** reacted readily to yield the desired compounds **2** and **3**, but ethoxycarbonyl group-bearing FPD **1n** reacted unselectively, giving a complex mixture of hardly identifiable products due to the occurrence of multiple side reactions involving the ethoxycarbonyl group.

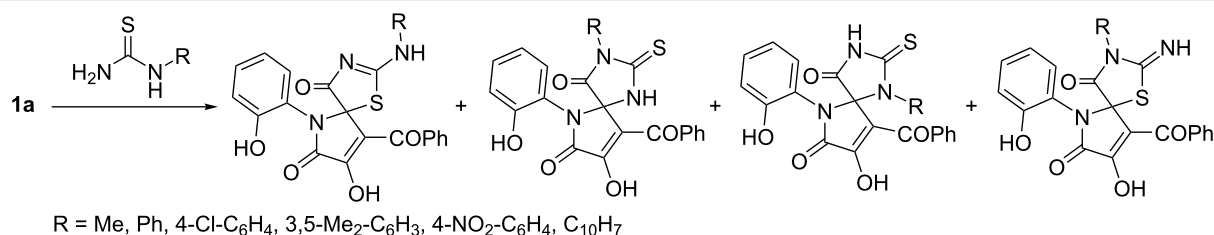
Next, we examined the influence of substituents in the thiourea motif on its reaction with FPDs **1**. It was found that monosubstituted thioureas (*N*-methylthiourea, *N*-phenylthiourea, *N*-(4-chlorophenyl)thiourea, *N*-(3,5-dimethylphenyl)thiourea, *N*-(4-nitrophenyl)thiourea, and *N*-1-naphthylthiourea) reacted with FPD **1a** unselectively, forming a mixture of four inseparable adducts (the reaction mixtures were analyzed by UPLC–MS) (Scheme 7). Unfortunately, we did not succeed to find any conditions for a selective formation of either of them.

Unexpectedly, the implementation of *N*-acetylthiourea in the reaction with FPDs **1**, both under conditions A and B (see Ta-

Table 3: Series of spiro-fused thiohydantoin **2a–m** and pseudothiohydantoin **3a–m** from various FPDs **1a–n**.

Entry	R	R'	Yield of 2 (%) ^{a,b}	Yield of 3 (%) ^{a,c}
a	2-OH-C ₆ H ₄	Ph	97 (CCDC 1952743)	79
b	5-Cl-2-OH-C ₆ H ₃	Ph	78	87
c	2-OH-C ₆ H ₄	4-OMe-C ₆ H ₄	79	91
d	2-OH-C ₆ H ₄	4-OEt-C ₆ H ₄	90	79
e	2-OH-C ₆ H ₄	4-Cl-C ₆ H ₄	78	61 (CCDC 1952745)
f	2-OH-C ₆ H ₄	4-Br-C ₆ H ₄	87	61
g	2-OH-C ₆ H ₄	4-Me-C ₆ H ₄	89	89
h	5-NO ₂ -2-OH-C ₆ H ₃	Ph	95	58
i	2-OH-C ₆ H ₄	4-NO ₂ -C ₆ H ₄	61	53
j	5-Br-2-OH-C ₆ H ₃	Ph	65	59
k	2-OH-C ₆ H ₄	4-F-C ₆ H ₄	81	82
l	CH ₂ CH ₂ OH	4-Cl-C ₆ H ₄	90 (CCDC 1952798)	97
m	CH ₂ CH ₂ OH	4-Me-C ₆ H ₄	98	75
n	2-OH-C ₆ H ₄	OEt	–	–

^aIsolated yields. ^bConditions A: a mixture of FPD **1a–n** (1 mmol) and thiourea (1 mmol) was refluxed in 1,4-dioxane (5 mL) for 4 h. ^cConditions B: a mixture of FPD **1a–n** (1 mmol) and thiourea (1 mmol) was stirred in ethyl acetate (5 mL) for 12 h at room temperature.

**Scheme 7:** Reaction of FPD **1a** with monosubstituted thioureas.

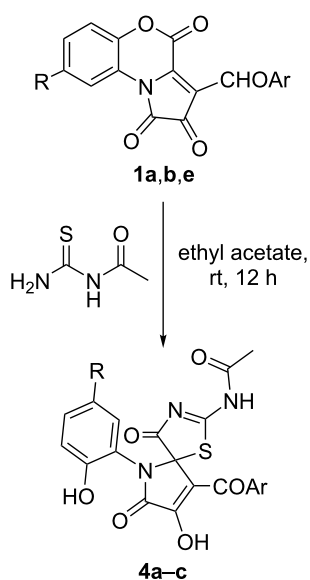
ble 3), afforded only one type of adducts, pseudothiohydantoin **4** (Table 4). Boiling compounds **4** in various solvents and heating them in open capillaries at 130 °C for 12 h did not provoke PTR, and compounds **4** remained unconverted. This phenomenon could be explained by the influence of the electron-withdrawing effect of the acetyl group, which reduced the nucleophilic properties of the acetyl-substituted nitrogen atom in *N*-acetylthiourea and decreased its reactivity, preventing the formation of regioisomeric compounds.

1,3-Dibutylthiourea reacted smoothly at room temperature in ethyl acetate with FPDs **1** to form mixtures of products **5** and **6**

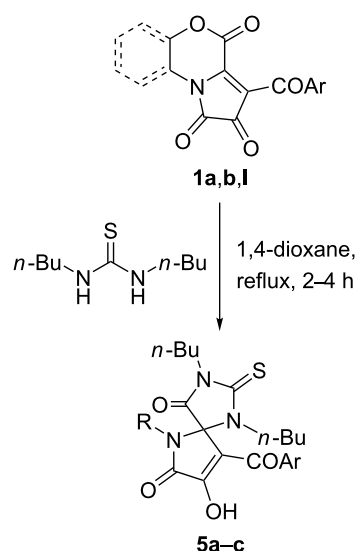
(Scheme 8). Moreover, it was observed that the percentage of compounds **5** in reaction mixtures increased over time upon storage of the solutions. As such, **6** readily underwent PTR, affording the corresponding compounds **5**, but unfortunately, we did not succeed to isolate pseudothiohydantoin **6**.

The reaction of FPDs **1** with 1,3-dibutylthiourea in 1,4-dioxane resulted in exclusive formation of thiohydantoin **5** upon heating at reflux for 2–4 h (Table 5).

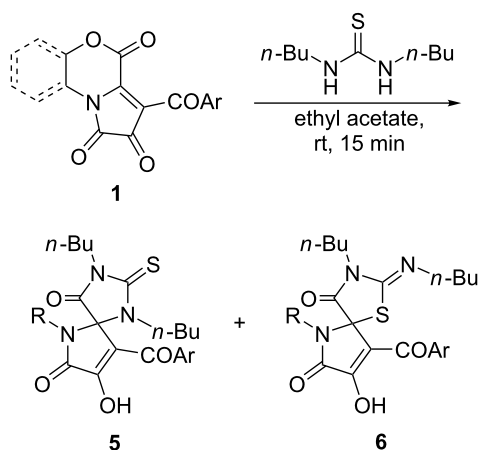
The facility of thiohydantoin **5** formation could be explained by the electron-donating effect of the butyl substituents, which

Table 4: ^bReaction of FPDs **1** with *N*-acetylthiourea.

Product	Ar	R	Yield of 4 (%) ^a
4a^b	Ph	H	80
4b	4-Cl-C ₆ H ₄	H	75
4c	Ph	Cl	79

^aIsolated yields. ^bCCDC 1952746.**Table 5:** Reaction of FPDs **1** with 1,3-dibutylthiourea at reflux.

Product	Ar	R	Yield of 5 (%) ^a
5a	Ph	2-OH-C ₆ H ₄	75
5b^b	Ph	5-Cl-2-OH-C ₆ H ₃	76
5c	4-Cl-C ₆ H ₄	CH ₂ CH ₂ OH	76

^aIsolated yields. ^bCCDC 1952744.**Scheme 8:** Reaction of FPDs **1** with 1,3-dibutylthiourea at room temperature.

increased the nucleophilicity of the butyl-substituted nitrogen atoms and promoted its attack on the electrophilic center C-3a of FPDs **1**.

The reaction of FPDs **1** with 1,3-diphenylthiourea proceeded in a similar manner. Thiohydantoin **7** were predominantly formed when the reaction was conducted at reflux in 1,4-dioxane, and

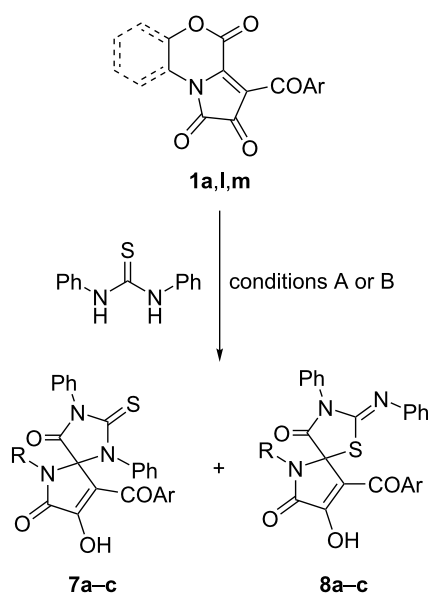
pseudothiohydantoin **8** were formed as main products at room temperature (Table 6).

Notably, the formation of thiohydantoin **7** required longer time in comparison with unsubstituted thiourea and 1,3-dibutylthiourea. Possible reasons for this are the influence of the steric hindrance induced by the phenyl substituents and weakening of the nucleophilic properties of the phenyl-substituted nitrogen atom, which prevented a nucleophilic attack of 1,3-diphenylthiourea on the electrophilic center C-3a of FPDs **1**.

The observed PTRs could have proceeded through two alternative pathways (Scheme 9), with the first stage being dissociation of the C_{spiro}-S bond [32] in pseudothiohydantoin **PThH**, affording intermediate **A**. Then, **A** could have further undergone either an intramolecular cyclization by NH attack, forming thiohydantoin **ThH** (path A), or further dissociation to FPD **1** and thiourea. The latter would subsequently attack FPD **1** with both nucleophilic NH centers, forming thiohydantoin **ThH** (path B).

Conclusion

In conclusion, we have developed a divergent approach to pharmaceutically interesting regiomeric thiohydantoin and

Table 6: Reaction of FPDs **1** with 1,3-diphenylthiourea.

Entry	Ar	R	Yield of 7 (%) ^{a,b}	Yield of 8 (%) ^{a,c}
a	Ph	2-OH-C ₆ H ₄	71	78
b	4-Cl-C ₆ H ₄	CH ₂ CH ₂ OH	63	82
c	4-Me-C ₆ H ₄	CH ₂ CH ₂ OH	72	76

^aIsolated yields. ^bConditions A: a mixture FPD **1** (1 mmol) and 1,3-diphenylthiourea (1 mmol) was refluxed in 1,4-dioxane (5 mL) for 8–12 h. ^cConditions B: a mixture FPD **1** (1 mmol) and 1,3-diphenylthiourea (1 mmol) was stirred in ethyl acetate (5 mL) for 12 h at room temperature.

pseudothiohydantoin spiro-fused to a pyrrole-2-one fragment via the reaction of [*e*]-fused 1*H*-pyrrole-2,3-diones with thioureas. The obtained pseudothiohydantoin were found to be prone to undergo a pseudothiohydantoin–thiohydantoin rearrangement. Therein, the substituents of the thiourea moiety were found to be crucial for tuning the conditions of this rearrangement. Notably, the reactions proceeded under catalyst-free conditions with good yields.

Supporting Information

Supporting Information File 1

Experimental details, copies of ¹H and ¹³C NMR spectra, X-ray crystallographic details, references to antimicrobial assay results, and a detailed revision of previously published structures.

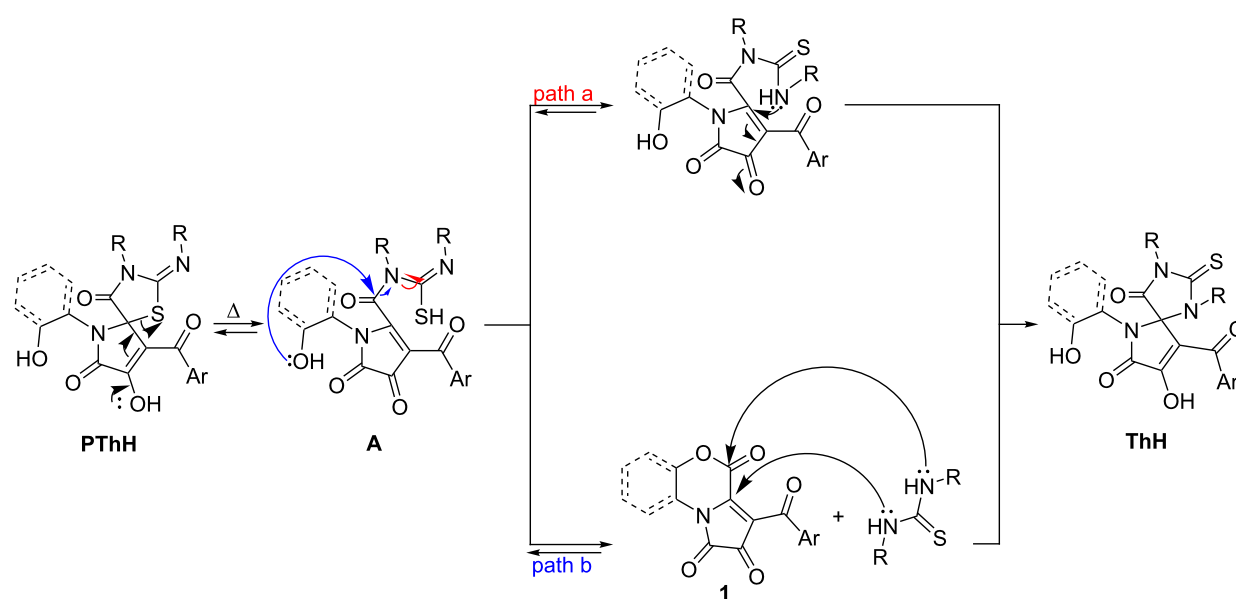
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**Scheme 9:** Plausible PTR pathways.

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