Synthesis and identification by GC-MS of 3-(pyridin-2-yl)-thiazolidinethiones

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INTRODUCTION

Thiazolidinones are an important group of heterocyclic compounds that has attracted considerable attention due facile synthesis and wide range of pharmaceutical activities.[1]

Transformation of a carbonyl functional group into thiocarbonyl has been an important interest to synthetic organic chemists for many years. Lawesson's Reagent is the most widely used agent for such a transformation.[2]

Besides, the bioisosteric replacement of carbonyl group by thiocarbonyl may increase biological activity such as in thionation of 4-thioxo-thiazolidin-2-ones.[3]

With this, the purpose of this study was to synthesize new compounds thiocarbonyl derivatives (thiazolidinethiones) arising from 3-(pyridin-2-yl)-thiazolidinones using Lawesson's Reagent as thionating agent and identified by GC-MS.

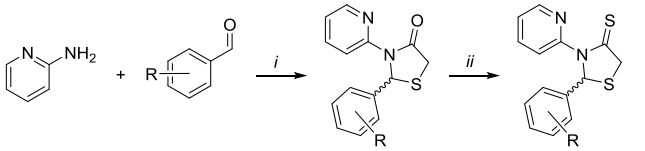
RESULTS AND DISCUSSION

For the synthesis of thiazolidinones **3a-h** was utilized ultrasonic irradiation methodology according Gouvea et al.[1]

The proposed substances **4a-h** were obtained with considerable yield in two steps as shown in **Squeme 1** through addition 0,5 mmol **3a-h**, 1 mmol Lawesson's reagent in dry toluene (25 mL), and the reaction mixture was refluxed with stirring for 12 h (monitored by TLC).

The characterization of **3a-h** and **4a-h** was performed by GC-MS, considering that the confirmation by ^{1}H and ^{13}C NMR has only been done with compounds **3a-h**.

Squeme 1.



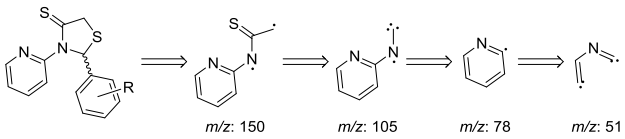
i: toluene, BF_3:MeOH, ultrasonic irradiation, 10 min.; $HSCH_2COOH$, ultrasonic irradiation, 25 min.
ii: toluene, Lawesson's Reagent, reflux, 12 hours.

Table 1. Mass spectroscopy data of compounds **3a-h** and **4a-h**.

R	M.W.[a]		GC-MS: *m/z*(%)[b]	
	3	**4**	**3**	**4**
a	290	306	290[M·]$^+$(13)	306[M·]$^+$(3)
b	274	290	274[M·]$^+$(16)	290[M·]$^+$(4)
c	286	302	286[M·]$^+$(13)	302[M·]$^+$(4)
d	301	317	255[M·-46]$^+$(3)	317[M·]$^+$(6)
e	290	306	290[M·]$^+$(18)	306[M·]$^+$(2)
f	274	290	274[M·]$^+$(19)	290[M·]$^+$(4)
g	286	302	286[M·]$^+$(37)	302[M·]$^+$(5)
h	301	317	301[M·]$^+$(14)	317[M·]$^+$(4)

[a]M.W. – Molecular Weight. [b]Only shown the molecular ion [M·]$^+$ in mass-to-charge ratio *m/z*.

Squeme 2. Common fragmentation of the compounds **4a-h**.



The results shown in **Table 1** identifies the thionation by GC-MS, either by increasing 16 *m/z* units of molecular íon [M·]$^+$ as own fragmentation pattern in relation to thiazolidinones **Squeme 2**.

CONCLUSION

In summary, thiazolidinethiones **4a-h** were obtained in good yields using an easy synthesis. Our preliminary results, guides us for further investigations of this compound class regarding biological activities like antimicrobial and antioxidant.

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