Journal of Medicinal and Chemical Sciences 2018 (1) 26-27

J. Med. Chem. Sci.

K_2CO_3/CS_2: A mild and effiecint system for the prapartion of trithiocarbonates

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ARTICLE INFO

Article history:
Received 14 May 2018
Revised 26 May 2018
Accepted 27 May 2018
Available online 28 May 2018

Keywords:
Trithiocarbonates
Potassium carbonate
Carbon disulfide
One-pot

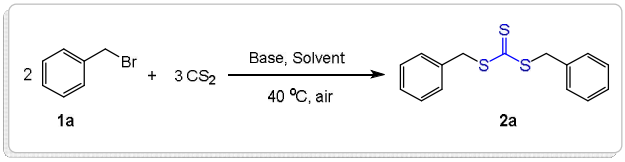
ABSTRACT

A mild and efficient one-pot protocol for the preparation of symmetrical trithiocarbonates using potassium carbonate and carbon disulfide in DMF has been developed. This protocol is mild and efficient compared to other reported methods.

1. Introduction

Organic trithiocarbonates (an important class of compounds containing C–S bonds) are key and effective intermediates in biological processes, agriculture, industry, biochemistry, organic synthesis, and materials science.[1-3] They have been used extensively as pharmaceuticals, agrochemicals, intermediates in organic synthesis, for protection of thiol functionality, in free radical polymerization reactions, as lubricating additives, in material science, in froth flotation for the recovery of minerals from their ores and for their absorption properties of the metals.[4-7] The reactions of trithiocarbonate anion (CS_3^{2-}) with alkyl halides, or thiols with carbon disulfide (CS_2) and alkyl halides is the most common protocol for the preparation of trithiocarbonate derivatives.[1]

In this paper, we report an efficient protocol for the preparation of symmetrical trithiocarbonates via the reaction alkyl halides with CS_2 in the presence of potassium carbonate at 40 °C in an aerial atmosphere.



Scheme 1. Model reaction for the synthesis of the product **2a**.

2. Result and Discussion

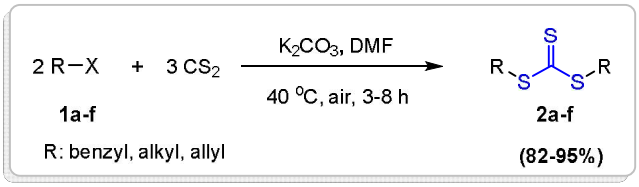
In order to optimize the reaction conditions with respect to base and solvent type, we studied the reaction of benzyl bromide with carbon disulfide as a model reaction (**Scheme 1**). The results are listed in **Table 1**. As shown in **Table 1**, the best results are obtained in the presence of potassium carbonate in DMF. To access the generality of our developed method for the synthesis of trithiocarbonates, a series of halides were screened via **Scheme 2** and the results are summarized in **Table 2**.

The results summarized in **Table 2** clearly reflects which the trithiocarbonates were obtained in good to excellent yields.

Table 1 Optimization of reaction condition for **Scheme 1** [a]

Entry	Base	Solvent	Time (h)	Yield (%)[b]
1	Li_2CO_3	CH_3CN	6	10
2	Na_2CO_3	CH_3CN	6	8
3	Cs_2CO_3	CH_3CN	6	74
4	K_3PO_4	CH_3CN	6	59
5	K_2CO_3	CH_3CN	6	85
6	K_2CO_3	EtOH	6	34
7	K_2CO_3	DMF	6	95
8	K_2CO_3	H_2O	6	41
9	K_2CO_3	Toluene	6	27

[a] Reaction conditions: 3 mmol of benzyl bromide, 4 mmol of CS_2 , 5 mmol of base, 5 mL of Solvent, 40 °C in air. [b] Isolated yields.



Scheme 2. Synthesis of the *trithiocarbonates*.

2. Conclusion

In summary, we developed an efficient protocol for the preparation of symmetrical trithiocarbonates via the reaction alkyl halides with CS_2 in the presence of potassium carbonate at 40 °C in an aerial atmosphere. The protocol is operationally simple, and gives high yields of products in suitable reaction times.

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Table 2 Synthesis of trithiocarbonate derivatives (according to **Scheme 2**) [a]

Entry	RX	Product	Time (h)	Yield (%)[b]
1	BnBr	**2a**	6	95
2	4-$NO_2C_6H_4Br$	**2b**	6	trace
3	4-MeC_6H_4Br	**2c**	7	90
4	4-ClC_6H_4Br	**2d**	5	93
5	n-BuBr	**2e**	3	91
6	CH_2=$CHCH_2Br$	**2f**	8	82

[a] Reaction conditions: 3 mmol of halide, 4 mmol of CS_2 , 4 mmol of K_2CO_3, 5 mL of DMF, 40 °C in air. [b] Isolated yields.

4. Experimental

All chemicals were purchased from Merck. Yields refer to isolated yields. All products were characterized by comparison of their spectral and physical data with those of known samples.

4.1. General procedure for the preparation of trithiocarbonates:

A mixture of K_2CO_3 (5 mmol) and carbon disulfide (4 mmol) in DMF (5 mL) was vigorously stirred at 40 °C for 20 min; then, alkyl halide (3 mmol) was added to the red blood mixture. The color of the mixture immediately changed from red to yellow. The reaction was continued until completion (cf. **Table 2**) as confirmed by TLC. The reaction mixture was then poured into distilled water (50 ml) and extracted with ethyl acetate thrice. The combined organic layer dried over anhydrous sodium sulfate and then concentrated to afford the desired trithiocarbonates.

Acknowledgments

I gratefully acknowledge financial support of this work by Islamic Azad University, Ilam branch.

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How to cite this paper: I. Amini, S. Mohammadi-Aghdam, F. Divsar, K_2CO_3/CS_2: A mild and effiecint system for the prapartion of trithiocarbonates.

J. Med. Chem. Sci. **2018**, 1, 26-27.

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