



# STUDY OF THE REACTION OF CHALCONE DIBROMIDE WITH PHENYLTHIOUREA IN ABSENCE AND PRESENCE OF CAUSTIC POTASH

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## ABSTRACT

The present paper deals with the effect of the absence and presence of caustic potash in different molar ratios in the reaction of chalcone dibromide with phenylthiourea. The presence of KOH in different molar ratios did not alter the course of reaction. The end products were found to be the same.

**Key words:** Chalcone dibromide, Phenylthiourea.

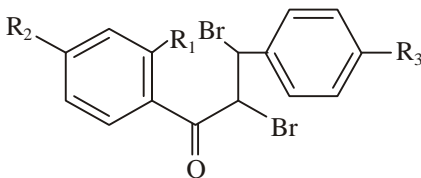
## INTRODUCTION

Sharma and Bokadia<sup>1</sup> reported the reaction of alcoholic thiourea with chalcone dibromide to yield free sulphur in addition to chalcones. Panda et al.<sup>2</sup> carried out the condensation of chalcone dibromide with thiourea in refluxing ethanolic sodium ethoxide to yield two compounds, only one of which is identified as chalcone. Harode and Sharma<sup>3</sup> studied the reaction between chalcone dibromide and thiourea in presence of KOH. The products were characterized as 4, 6-diaryl-2-imino-1,3-thiazines. In the absence of KOH, the corresponding thiazines could not be detected. The reaction of  $\alpha$ -bromo-chalcone with thiourea also resulted in the formation of corresponding thiazines.

In continuation of our previous work on chalcone dibromide<sup>4</sup>, here We report the synthesis of the following chalcone dibromides.

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- I. Chalcone dibromide ( $R_1 = R_2 = R_3 = H$ )
- II. 4-Methoxychalcone dibromide ( $R_1 = R_2 = H, R_3 = OCH_3$ )
- III. 2'-Hydroxy-4-chlorochalcone dibromide ( $R_1 = OH, R_2 = H, R_3 = Cl$ )

These chalcone dibromides were then refluxed with ethanolic phenylthiourea and the products obtained were found to be chalcone and sulphur. The presence of KOH in different molar ratio did not alter the reaction.

## EXPERIMENTAL

### Ia. Reaction of chalcone dibromide with ethanolic phenylthiourea

Chalcone dibromide (1.84 g.) and phenyl thiourea (0.8 g.) were dissolved in alcohol (50 mL) into a 250 mL round bottom flask. The reaction mixture was refluxed for 6 hours and then allowed to cool at room temperature. The crystals thus separated were filtered, washed with water and dried. Recrystallization from benzene gave yellow needles of free sulphur (m.p. 120°C). The filtrate was concentrated to afford colourless needles of chalcones (0.62 g; m.p. 56°C). Mixed melting point determination with an authentic sample of chalcone showed no depression.

IR: ( $\nu_{\max}$  KBr) 1660, 1590, 1570, 1440, 1330, 1290, 1230, 1040, 1020, 760, 690 and 560  $\text{cm}^{-1}$ .

### Ib. Reaction of chalcone dibromide with ethanolic phenylthiourea in presence of 1M, 2M and 3M KOH

Chalcone dibromide (1.84 g) and phenyl thiourea (0.8 g) were dissolved in alcohol (50 mL) into a 250 mL round bottom flask. To this, KOH solution (0.3 g, 0.6 g, and 0.9 g dissolved in 5 mL, 10 mL and 15 mL of water, respectively) was added. The reaction mixture was refluxed for 6 hours and then allowed to cool at room temperature. The crystals thus separated were filtered, washed with water and dried. Recrystallization from benzene gave yellow needles of free sulphur (m.p. 120°C). The filtrate was concentrated to afford colourless needles of chalcones (0.62 g; m.p. 56°C). Mixed melting point determination with an authentic sample of chalcone showed no depression.

IR: ( $\nu_{\max}$  KBr) 1660, 1590, 1570, 1440, 1330, 1290, 1230, 1040, 1020, 760, 690 and 560  $\text{cm}^{-1}$ .

### **IIa. Reaction of 4-methoxychalcone dibromide with ethanolic phenylthiourea in absence of KOH**

Chalcone dibromide (1.95 g) and phenyl thiourea (0.8 g) were dissolved in alcohol (50 mL) into a 250 mL round bottom flask. The reaction mixture was refluxed for 6 hours and then allowed to cool at room temperature. The crystals thus separated were filtered, washed with water and dried. Recrystallization from benzene gave yellow needles of free sulphur (m.p.120°C). The filtrate was concentrated to afford colourless needles of 4-methoxy chalcones (0.7 g; m.p. 78°C). Mixed melting point determination with an authentic sample of 4-methoxychalcone showed no depression.

IR: ( $\nu_{\max}$  KBr) 1650, 1600, 1570, 1500, 1440, 1340, 1270, 1220, 1180, 1020, 970, 820, 780, 760 and 660  $\text{cm}^{-1}$ .

### **IIb. Reaction of 4-methoxychalcone dibromide with ethanolic phenylthiourea in presence of 1M, 2M and 3M KOH**

Chalcone dibromide (1.95 g.) and phenyl thiourea (0.8 g.) were dissolved in alcohol (50 mL) into a 250 mL round bottom flask. To this, KOH solution (0.3 g., 0.6 g, and 0.9 g dissolved in 5 mL, 10 mL and 15 mL of water, respectively) was added. The reaction mixture was refluxed for 6 hours and then allowed to cool at room temperature. The crystals thus separated were filtered, washed with water and dried. Recrystallization from benzene gave yellow needles of free sulphur (m.p.120°C). The filtrate was concentrated to afford colourless needles of 4-methoxychalcones (0.7 g; m.p. 78°C). Mixed melting point determination with an authentic sample of 4-methoxychalcone showed no depression.

IR: ( $\nu_{\max}$  KBr) 1650, 1600, 1570, 1500, 1440, 1340, 1270, 1220, 1180, 1020, 970, 820, 780, 760 and 660  $\text{cm}^{-1}$ .

### **IIIa. Reaction of 2'-hydroxy-4-chlorochalcone dibromide with ethanolic phenylthiourea in absence of KOH**

2'-Hydroxy-4- chlorochalcone dibromide (2.1 g.) and phenyl thiourea (0.8 g.) were dissolved in alcohol (50 mL) into a 250 mL round bottom flask. The reaction mixture was refluxed for 6 hours on a water bath and then allowed to cool at room temperature. The crystals thus separated were filtered, washed with water and dried. Recrystallization from benzene gave yellow needles of free sulphur (m.p.120°C). The filtrate was concentrated to afford colourless needles of 2'-hydroxy-4-chlorochalcone (1.3 g; m.p. 150°C). Mixed

melting point determination with an authentic sample of 2'-hydroxy-4-chloroalcone showed no depression.

IR: ( $\nu_{\max}$  KBr) 3380, 1650, 1590, 1560, 1500, 1410, 1330, 1270, 1220, 1160, 1090, 1020, 980, 810, 760 and 670  $\text{cm}^{-1}$ .

### **IIIb. Reaction of 2'-hydroxy-4-chloroalcone dibromide with ethanolic phenylthiourea in presence of 1M, 2M and 3M KOH**

2'-Hydroxy-4-chloroalcone dibromide (2.1 g) and phenyl thiourea (0.8 g) were dissolved in alcohol (50 mL) into a 250 mL round bottom flask. To this, KOH solution (0.3 g, 0.6 g and 0.9 g dissolved in 5 mL, 10 mL and 15 mL of water, respectively) was added. The reaction mixture was refluxed for 6 hours on a water bath and then allowed to cool at room temperature. The crystals thus separated were filtered, washed with water and dried. Recrystallization from benzene gave yellow needles of free sulphur (m.p. 120°C). The filtrate was concentrated to afford colourless needles of 2'-hydroxy-4-chloroalcone (1.3 g; m.p. 150°C). Mixed melting point determination with an authentic sample of 2'-hydroxy-4-chloroalcone showed no depression.

IR: ( $\nu_{\max}$  KBr): 3380, 1650, 1590, 1560, 1500, 1410, 1330, 1270, 1220, 1160, 1090, 1020, 980, 810, 760 and 670  $\text{cm}^{-1}$ .

## **RESULTS AND DISCUSSION**

The probable course of reaction is due to the fact that  $\alpha$ -carbon is more prone to nucleophilic attack.

The identity of these compounds has been confirmed by their IR spectra, melting points and mixed melting points.

IR spectra of chalcones have shown absorption bands at the following wave numbers.

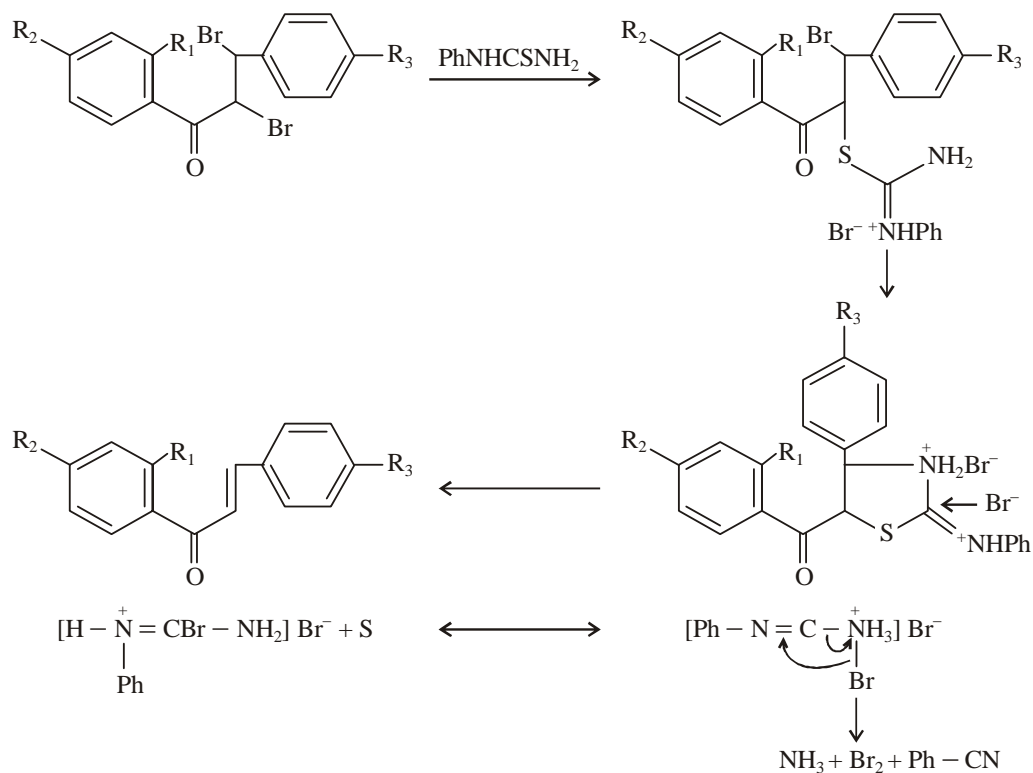
### **Absorption due to –OH group**

Absorption band due to –OH stretching has been observed at 3400  $\text{cm}^{-1}$ . This shift to longer wave length from normal position 3650-3590  $\text{cm}^{-1}$  indicates intramolecular hydrogen bonding.

### **Absorption due to $\alpha,\beta$ -unsaturated ketonic group**

The absorption band due to  $> \text{C} = \text{O}$  stretching vibrations has been observed in the

region 1640-1660  $\text{cm}^{-1}$ . This lowering may be attributed to the presence of conjugation and hydrogen bonding.



### Absorption due to aromatic nucleus

Four bands have been observed in the region 1600-1450  $\text{cm}^{-1}$  and are caused by  $\text{C}=\text{C}$  skeleton in plane vibrations. These bands are particularly diagnostic of aromatic structure.

### REFERENCES

1. T. C. Sharma and M. M. Bokadia, Indian J. Chem., **14B**, 65 (1976).
2. C. S. Panda, F. Khalldora and B. Dash, J. Hetrocycle Chem., **20**, 691 (1983).
3. R. Harode and T. C. Sharma, Indian J. Chem., **27B**, 1144 (1988).
4. Bharat Kumar, Vishal Pathak, Sushma Rani and I. C. Tiwari, Int. J. Microbiol. Research, **1(2)**, 20 (2009).

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