

## NOTE

**Cyclization of 2'-Hydroxychalcones in Dimethyl Sulphoxide**

SURENDER KUMAR and J.K. MAKRANDI\*

*Department of Chemistry*  
*M.D. University, Rohtak-124 001, India*  
*E-mail: s\_kaushik4@rediffmail.com*

An attempt to cyclize 2-Hydroxy-chalcones in dimethyl sulphoxide (DMSO) has been made.

**Key Words:** Cyclization, 2'-Hydroxychalcones, DMSO.

One of the methods for the synthesis of flavones, a well known class of naturally occurring compounds, involves the oxidative cyclization of 2'-hydroxychalcones<sup>1</sup>. Various oxidizing agents being used include  $\text{SeO}_2$ , DDQ<sup>3</sup>,  $\text{I}_2$ <sup>4</sup>,  $\text{HIO}_4$ <sup>5</sup>. During an attempt to cyclize 2'-hydroxychalcones using  $\text{SeO}_2$  in DMSO<sup>6</sup>, the reaction time was found to improve tremendously (from 24 to 1.5 h).

Earlier conversion of 2'-hydroxychalcones into flavanones has been reported using acid<sup>7</sup>, base<sup>8</sup> or metal ions<sup>9</sup>. The yields are generally poor due to reversible nature of the reaction.

It is the first report that 2'-hydroxychalcones is being cyclized in aprotic polar solvent alone. Using the above conditions, differently substituted flavanones were obtained.

In order to study the role of DMSO in the reaction, 2'-hydroxychalcone was heated with DMSO alone. The progress of the reaction was checked on TLC after 30 min, formation of a compound was observed which was different from flavone. The reaction was worked up after 4 h, when a colourless compound, m.p. 76–77°C separated out. Its <sup>1</sup>H NMR showed a multiplet at  $\delta(2.80\text{--}3.00)$  for two protons ( $\text{CH}_2$  at position 3), a double doublet at  $\delta(5.20)$  for one proton (H-2), a doublet at  $\delta(7.60)$  for one proton (H-5) along with a multiplet at  $\delta(6.70\text{--}7.20)$  for eight aromatic protons ( $\text{C}_6\text{H}_5$ , H-6, H-7 and H-8).

Based on the above data, the compound was assigned the structure dihydroflavone (flavanone). The structure was finally confirmed by its comparison with authentic sample<sup>9</sup>.

TABLE

Compound	R <sup>1</sup>	R <sup>2</sup>	R <sup>3</sup>	m.pt. (°C)	Lit. m.pt. (°C)	Yield (%)
1a	H	H	H	76–77	78 <sup>4</sup>	60
1b	H	H	OCH <sub>3</sub>	96–98	98 <sup>4</sup>	55
1c	OCH <sub>3</sub>	H	H	90	91 <sup>3</sup>	50
1d	OCH <sub>3</sub>	H	OCH <sub>3</sub>	92–93	94–95 <sup>3</sup>	53
1e	H	CH <sub>3</sub>	H	104–105	105 <sup>4</sup>	57
1f	H	CH <sub>3</sub>	OCH <sub>3</sub>	109–110	110 <sup>4</sup>	60
1g	H	Br	H	113–115	115 <sup>9</sup>	55
1h	H	Br	OCH <sub>3</sub>	108–109	110 <sup>9</sup>	58

### General procedure (flavanone)

A solution of 2'-hydroxychalcone (0.5 g) in dry dimethyl sulphoxide was heated under anhydrous condition on an oil bath at 120–130°C for 4 h. Completion of the reaction was checked by TLC. Ice-cold water was added to the reaction mixture after cooling. The solid that separated out was filtered, washed with water and recrystallized from petroleum ether to give flavanone.

### REFERENCES

1. D.N. Dhar, *The Chemistry of Chalcones and Related Compound*, John Wiley Sons, p. 46 (1981).
2. H.H. Lee and C.H. Tan, *J. Chem. Soc.*, 2743 (1965).
3. K. Imafuku, M. Honda and J.F.W. Mcomie, *Synthesis*, 199 (1987).
4. A.G. Dosh, P.A. Soni and B.J. Ghiye, *Indian J. Chem.*, **25B**, 759 (1986).
5. N. Hans and S.K. Grover, *Syn., Commun.*, **23**, 1021 (1993).
6. J.K. Makrandi and Seema, *Chem. Ind.*, 607 (1988).
7. R. Ludwig, B. Walter, *Chem. Ber.*, **37**, 2634 (1904).
8. T.A. Geismann and R.O. Clinton, *J. Am. Chem. Soc.*, **68**, 697 (1946).
9. J.K. Makrandi and S. Bala, *Syn. Commun.*, **39**, 3555 (2000).

(Received: 3 December 2003; Accepted: 11 October 2004)

AJC-4084