TUTA Journal Vol 9/10 (1), 2021/2022, ISSN : 2350-8892

Oxidation of 2'-Hydroxy Chalcone Imines

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Abstract

2'-hydroxy chalcone imines (III_{A-T}) on refluxing in DMSO for 2 hours in presence of catalytic amount of iodine in presence of conc. H_2SO_4, afford the corresponding amino flavone (IV_{A-T}) in high yield. This method is quicker and appears to be of general applicability. The structures were established on the basis of spectral data (IR, NMR) and chemical reactions.

Kewords :- chalcone, refluxing, amino flavone, applicability

Articles information : Manuscript Receive : 20/07/2021, Review Date : 22/05/2022, Date of Acceptance : 15/10/2022, Publisher : Mahendra Morang Adarsh Multiple Campus, TUTA Unit, Biratnagar.
DOI : https://doi.org/10.3126/tutaj.v10i1.74327
Journal Homepage : https://nepjol.info/index.php/tutaj/index

Introduction

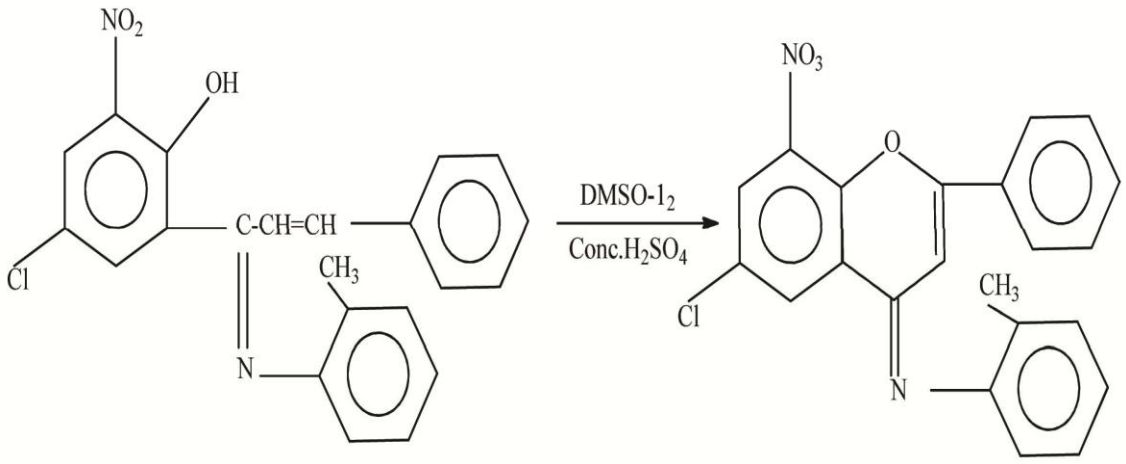
The conversion of 2'-hydroxy chalcones into flavones by prolonged refluxing with SeO_2 in isoamyl alcohol [1] is time consuming and is applicable in those chalcones which do not have free hydroxyl group other than at 2'-position. The Kostanecki [2] method of converting 2'-hydroxy chalcones into flavones by the action of ethanolic alkali on chalcone dibromide has limited applications. The use of dimethyl sulphoxide (DMSO) as an oxidizing agent for affecting this conversion has been reported by several workers [3-5]. However the reagent DMSO-I_2 for the oxidation of 2'-hydroxy chalcones to flavones has not been used so far, though recently iodine-DMSO-sulphuric acid system has been applied [6] for dehydration of flavonoids. Recently 2-hydroxy chalcone or flavanone when refluxed with DMSO [7] in presence of catalytic amount of I_2 gives corresponding flavone in more than 80% yield and flavanone was converted into flavone by the use of DDQ [8] in dioxane. Recently chalconeimine is converted into flavone imine by oxidative cyclisation in presence of DMSO-I_2 [9-10].

In this communication synthesis of flavoneimine by oxidative cyclisation of chalconeimines is performed in DMSO medium in presence of catalytic amount of iodine and 2-3 drops of conc. H_2SO_4.

Experimental

Synthesis of 6-chloro-8-nitro-4(0-methyl phenyl) imino flavone. 2'-hydroxy-3'- nitro-5'-chloro N-(0-methyl phenyl) chalcone imine (III_B) (0.01 mole) was dissolved in dimethyl

sulphoxide (DMSO) (40 ml) and 2-3 drops of conc. H_2SO_4 and catalytic amount of iodine was added to this mixture and was refluxed for 2-3' hours on water both, cooled and diluted with ice cold water. The resulting solid was treated with 10% sodium thiosulphate solution to remove unreacted iodine and finally with water and crystallized from ethanol to give yellow crystalline compound. m.p. 286°C, yield 81%.



Properties and constitution of the compound (IV_B)

1. It is brown yellow crystalline solid m.p. 287°C.
2. From analytical data, compound (IVB) agreed with molecular formula $C_{22}H_{15}O_3N_2Cl$.
3. Elemental analysis of compound(IV_B)

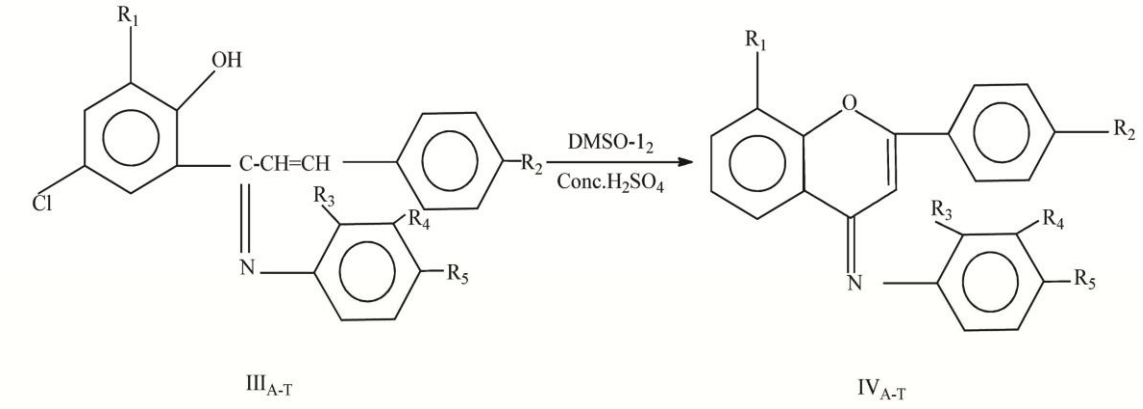
Analysis	% C	% H	% N
Found	65.23	3.10	7.12
Calculated	65.93	3.57	7.69

Spectral data of the compound (IV_B) is as follows:

i. **IR (KBr) :-** 3072 cm^{-1} (CH aromatic stretching), 2922 cm^{-1} (CH aliphatic stretching), 1651 cm^{-1} (C=N stretching), 1612 cm^{-1} (C=C aromatic stretching), 1533 cm^{-1} (C-NO_2 asymmetric stretching), 1354 cm^{-1} (C-NO_2 symmetric stretching), 1252 cm^{-1} (C-O stretching), 886 cm^{-1} (tri substituted), 774 cm^{-1} (C—Cl stretching).

ii. **NMR (ð) :-** 1.25 (d, 3-H, -CH, -CH_3) 2.63 (S, 1H, C=CH) 6.94-8.47 (m, 11H, Ar-

H). The other flavoneimines were prepared by the same method and are listed inTable I.



S.No.	Compound	R_1	R_2	R_3	R_4	R_5	M.F.	M.W.	mp^{O}C	%yield
1	IV_A	NO_2	H	H	H	H	C21H13O3N2Cl	376.5	107	82
2	IV_B	NO_2	H	CH_3	H	H	C22H1503N2C1	390.5	287	82
3	IV_C	NO_2	H	H	H	CH_3	C22H1503N2C1	390.5	200	88
4	IV_D	NO_2	H	NO_2	H	H	C21H12O5N3C1	421.5	75	84
5	IV_E	NO_2	H	H	NO_2	H	C21H12O5N3Cl	421.5	74	85
6	IV_F	NO_2	H	H	H	NO_2	C21 H12 O5 N3C1	421.	116	85
7	IV_G	NO_2	H	Cl	H	H	C21H12O3N2C12	412	118	88
8	IV_H	NO_2	H	H	H	OH	C21H13O3N2C1	376.	220	86
9	IV_I	NO_2	H	OH	H	H	C21H13O3N2C1	376.	212	87
10	IV_J	NO_2	H	H	H	COOH	C22H13O5N2C1	420.	120	82
II	IV_K	NO_2	OCH_3	H	H	H	C22H15 O 4N2C1	406.	102	84
12	IV_L	NO_2	OCH_3	CH_3	H	H	C23H17	420.	106	86

							O4N2C1			
13	IV_M	NO_2	OCH_3	H	H	CH_3	C23H17 O4N2C1	420.5	112	86
14	IV_N	NO_2	OCH_3	NO_2	H	H	C22H14 O6N3C1	451.	118	87
I5	IV_O	NO_2	OCH_3	H	NO_2	H	C22H14 O6N3C1	451.	122	84
16	IV_P	NO_2	OCH_3	H	H	NO_2	C22H14 O6N3C1	451.	98	87
17	IV_Q	NO_2	OCH_3	Cl	H	H	C22H14 O4N2C12	441	92	84
18	IV_R	NO_2	OCH_3	H	H	OH	C22H15O4N2 C1	406.	110	83
19	IV_S	NO_2	OCH_3	OH	H	H	C22H15O4N2 C1	406.	110	87
20	IV_T	NO_2	OCH_3	H	H	COOH	C23H15O6N2 C1	450.	92	82

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