

A new method to prepare tricyclo quinazalone

Dilip Kumar

Chemical Research Laboratory, P.G. Department of Chemistry, Gaya College, Gaya,

Email : drdilipkumar76@yahoo.com

Manuscript received online 20 April 2020, accepted on 25 June 2020

Abstract : It has been found that 8-alkyl alkoxy substituted benzoxazones have been used as precursors for the synthesis of many alkoxy amino quinazolones. Reactivity of both the methyl or methylene carbon in the quinazolones and nitrogen of acyl amino group in enolic form on dehydration yields a new type of pyrazolo-quinazalone. The properties as described in the above compound have been utilised to enate a new attached ring producing a new compound. It has also been found that quinazoline derivatives with butyl amino group along with chloride at different positions are having antimalarial activity and hence several such compounds are being prepared and their activity on some of diseases are being tested quinethazone, a quinazoline derivative is an efficient diuretic. So the derivatives of quinazolones are of immense use and requires further work.

(Keywords : Quinazoline, Pyrazoloquinazalone, Anthranil, pyradazino ring).

Introduction

It has been found that N_3-NH_2 group and a methyl or methylene group at C_2 in position 2-in3-amino-2alkyl/aryl-4 oxoquinazolines are reactive. Former can be reacted with esters or β -keto esters while a methyl or methylene group at C_2 or C_4 even in simple quinazoline condensor early with esters like diethyl oxalated. It is acidic enough to react with pyridine to form pyridinium salt. The reactivity of C_2 in 4-oxoquinazolines is found to be even greater. This was supported by its condensation with propiophenone, when heated

at 1500-1800C for 1-2 hours to give 1-2-methyl derivative.

Taking these facts in to account efforts were made to prepare tricyclo quinazalone.

Experimental

3-Amino-2-benzyl-8-methyl-4-oxoquinazoline was prepared for this 3-methyl anthranilic acid (10gm) was taken in a mixture of acetone (25ml) and 6ml of dry pyridine in a 500ml conical flask fitted i.e with rubber cork then added 10ml of previously prepared phenyl acetyl chloride dropwise. It was left overnight. Next day acetone was distilled off. Ice cold water was added to reaction mixture when a solid product separated which was filtered off at the pump. It was crystalised with acetic acid. It was brown in colour and having mp 1850 yield 7 gm. 2nd step was cyclisation of acitide A to anthranil that was carried out in the following way In a 250ml RB flask (B-19) 10 gm of anilide prepared as above was taken in the above flask and 50 ml of dried acetic anhydride was poured in it. The mixture in R.B. flask was refluxed for five hours having a $CaCl_2$ tube filtered in the condenser. The excess of acetic anhydrate was distilled of under vacuum when a brown solid was left at the bottom as anthranils. 3rd step was cyclisation of anthranil to oxoquinazoline. The above prepared anthranil was taken in a 100ml quick fit R.B. flask fitted with condenser and 20ml of 99% hydrazine hydrate and dry methanol were prepared into the R. B. flask and was refluxed for 2.5 hrs. It was filtered while hot and cooled when a grey coloured

precipitate was obtained. It was recrystallised in ethanol. Its mp was 120°C and yield was 6.8gm. On elemental analysis it gave the following results.

C	72.32%	72.45%
H	5.4%	5.66%
N	15.90%	15.85%

IR values of the

ν_{KBr} 1660 cm^{-1} , 3325 cm^{-1} , 3230 cm^{-1} 1660 cm^{-1} for quinazolone carbonyl and rest two for N-H stretching frequencies.

Results and Discussion

The oxoquinazoline formed above was converted into the 3-amino-2-benzyl-8-methyl-4-oxoquinazoline (2.5gm) was converted into automethyl derivatives by refluxing it with ethyl auto actate (3ml) n-butanol (25ml) for 6 hours. Exam of butanol distilled off. On cooling a solid product appeared which was filtered off and recrystallised with ethyl acetate

when yellow crystal were obtained. Its mp was 1880C and yield (1.7gm 65%) on analysis it gave the following result.

Nitrogen - 12.27% (Experimental) while calculated value ($\text{C}_{20}\text{H}_{19}\text{N}_3\text{O}_3$) was 12.03% Now the cyclodehydration has done in the following way. (165gm) of 3-auto acetyl amino-2-benzyl-8-methyl-4-oxoquinazoline was heated under reflux in dry toluene (40ml) containing catatytic amount of p-toluene sulphonic acid for six hours. The bulk of toluene was azeotroped away using a Bean and stark appratus. The residual liquid was cooled and light petroleum added when a brown solid precipitated. This was filtered off at the pump and recrystallised in ethyl acetate when light brown crystals were obtained. Its mp was 154°C and yield was (0.75gm 52%)

On analysis it gave the percentage of nitrogen as 12.85% and calculated value for $\text{C}_{20}\text{H}_{17}\text{N}_3\text{O}_2$ was 12.69%

I.R. Values $\nu_{\text{Max}}^{\text{KBr}}$ 1660 cm^{-1} , and 1730 cm^{-1} for quinazoline carbonyl and open chain carbonyl respectively.

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