The Bioscan AN INTERNATIONAL QUARTERLY JOURNAL OF LIFE SCIENCES 21(2): S.I (2), 2003-2013, 2026 www.thebioscan.com

TETRABUTYLAMMONIUM BROMIDE-CESIUM CARBONATE: NEW REAGENT SYSTEM FOR THE ONE-POT SYNTHESIS OF OCTAHYDROQUINAZOLINONE DERIVATIVES

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DOI:https://doi.org/10.63001/tbs.2026.v21.i02.S.I(2).pp2003-2013

Keywords:
Cesium carbonate, Octahydroquinazolinone derivative, One pot Synthesis.
Received on:
20-05-2026
Accepted on:
07-06-2026
Published on:
17-06-2026

Abstract
Octahydroquinazolinone derivatives were synthesized in high yields by a simple one-pot, three-component condensation method. The reaction involved 5,5-dimethyl-1,3-cyclohexadione, urea or thiourea, and aromatic aldehydes as starting materials. The condensation was carried out in an eco-friendly poly(ethylene glycol)-water medium. A new reagent system catalyst consisting of Tetrabutylammonium bromide and Cesium carbonate was used. The TBAB-Cs2CO3 catalyst system efficiently promoted the reaction under mild conditions. This method provided the desired octahydroquinazolinone products in excellent yields. The procedure is simple, cost-effective, and operationally easy to handle. The use of PEG-water as solvent makes the process greener compared to conventional methods. Overall, this protocol offers an efficient and environmentally benign route for octahydroquinazolinone synthesis.

INTRODUCTION:

Octahydroquinazolinones have emerged as an important class of nitrogen heterocycles that have attracted significant attention because of their potent antibacterial activity against *Staphylococcus aureus, Escherichia Coli, Pseudomonas aeruginosa*[1] and calcium antagonist activity.[2] Various of methods have been developed for the synthesis of quinazolinone derivatives such as cyclodehydration of anthranilic acid by acetic anhydride;[3] reaction of anthranilic acid with acid chloride in pyridine;[4] reaction of aldehyde with $SOCl_2$ and pyridine and then with 2-aminobenzylamine in refluxing solvent such as benzene or xylene with azeotropic water removal[5] and treatment of 2-ureidobenzoates with concentrated sulfuric acid.[6] Recently, the octahydroquinazolinone derivatives were synthesized in absolute ethanol[7] with low

yields of products. More recently, H_2SO_4 in water[8] is reported for the synthesis of these compounds with improved yields.

In general, the reported methods for the synthesis of octahydroquinazolinone derivatives involve hazardous and expensive reagents or solvents and display moderate to low yields with low atom efficiency. Tetrabutylammonium bromide (TBAB) is a lo w cost, mild, water-tolerant catalyst and has found to be effective in various organic transformations such as Biginelli-type reaction,[9] synthesis of biscoumarine and 3,4-dihydropyrano[c]chromene derivatives,[10] and highly substituted imidazole derivatives.[11] In addition, TBAB which is one of the most common phase-transfer (PT) catalysts; is employed in numerous C–C, C–N, C–O, C–S, and C–P bond forming reactions performed under liquid– liquid and liquid– solid phase-transfer catalysis (PTC) conditions, as well as in halogenationand oxidation reactions.[12] Among these, many C–C, and C–N bond forming reactions have been carried out by using catalytic amount of TBAB and inorganic bases.[13-17] This has prompted us to explore TBAB as a catalyst for the synthesis of Synthesis of Octahydroquinazolinone Derivatives. Herein, we report a mild and practical method for the synthesis of Octahydroquinazolinone derivatives (4a- l) from the reaction of one-pot condensation of 5,5-dimethyl-1,3-cyclohexadione, urea or thiourea and aromatic aldehydes in the presence of tetrabutylammonium bromide (5 mol%) in combination with cesium carbonate (5 mol%) in poly(ethylene glycol)-water medium at room temperature (Scheme 1).

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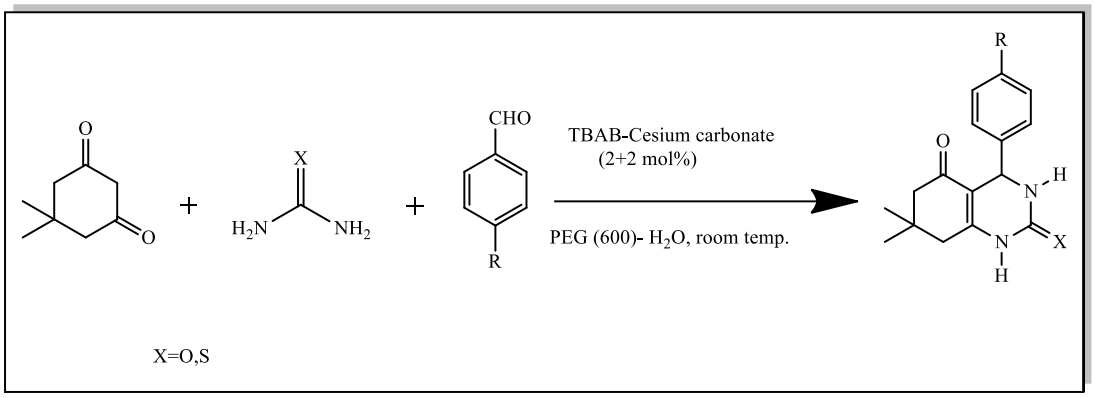


Scheme 1

EXPERIMENTAL:

General Experimental Procedure

A mixture of dimedone (1 mmol), urea or thiourea (1.5 mmol) and aldehyde (1 mmol) in PEG (600)-water (4 mL) using tetrabutylammonium bromide (5 mol%) in combination with cesium carbonate (5 mol%) using as catalyst was stirred at room temperature for the specified time (see Table 2). After completion of the reaction (TLC), the reaction mixture was cooled at room temperature and then poured onto crushed ice (20 g). The solid precipitate thus obtained was filtered and washed with cold water. The crude solid was further purified by recrystallization from 50% ethanol to get practically pure products

Spectral And Analytical Data

4a:4-Phenyl-7,7-dimethyl-1,2,3,4,5,6,7,8-octahydroquinazoline-2,5-dione;Solid: mp 180-182^{0}C; IR (KBr): 3444, 2966, 1680, 1580, 1243, 840, 668 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.08 (s, 3H, C-CH_3), 1.20 (s, 3H, C-CH_3), 2.24-2.41 (m, 4H, 2 x CH_2), 5.44 (s, 1H, C-H), 6.49-6.68 (m, 3H, Ar-H), 6.94-7.05 (m, 3H, Ar-H), 11.88 (brs, 2H, NH); Anal. calcd for: $C_{16}H_{18}O_2N_2$: C, 71.08; H, 6.73; N, 10.36; Found: C, 71.12; H, 6.69; N, 10.32.

4b:4-Phenyl-7,7-dimethyl-5-oxo-1,2,3,4,5,6,7,8-octahydroquinazoline-2-

thione; Solid: mp 160-162^{0}C; IR (KBr): 3442, 2959, 1677, 1595, 1245, 868, 664 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.09 (s, 3H, C-CH_3), 1.22 (s, 3H, C-CH_3), 2.24-2.44 (m, 4H, 2 x CH_2), 5.50 (s, 1H, C-H), 6.78-6.6.95 (m, 3H, Ar-H), 7.05-7.30 (m, 3H, Ar-H), 11.87 (brs, 2H, NH); Anal. calcd for: $C_{16}H_{18}O_2N_2$ S : C, 67.19; H, 6.34; N, 9.79; S, 11.10; Found : C, 67.17; H, 6.33; N, 9.77; S, 11.17.

4c: 4-[4-(Dimethylamino)phenyl]-7,7-dimethyl-1,2,3,4,5,6,7,8-octahydroquinazoline-2,5- dione; Solid: mp 247^{0}C; IR (KBr): 3436, 2958, 1632, 1580, 1219,1140, 822 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.08 (s, 3H, C-CH_3), 1.22 (s, 3H, C-CH_3), 2.28-2.50 (m, 4H, 2 x CH_2), 2.98 (s, 6H, 2xNCH_3), 5.38 (s, 1H, C-H), 6.81 (d, 2H, *J* = 8.6 Hz, Ar-H), 7.02 (d, 2H, *J* = 8.2 Hz, Ar-H), 11.86 (brs, 2H, NH); Anal. calcd for: $C_{18}H_{23}O_2N_3$: C, 69.07; H, 6.7.40; N, 13.42; Found: C, 69.02; H, 6.7.43; N, 13.40

4d:4-[4-(Dimethylamino)phenyl]-7,7-dimethyl-5-oxo-1,2,3,4,5,6,7,8-octahydroquinazoline-2-thione; Solid: mp 209-211^{0}C; IR (KBr): 3440, 2958, 1668, 1610, 1194,1061, 817 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.07 (s, 3H, C-CH_3), 1.26 (s, 3H, C-CH_3), 2.26-2.48 (m, 4H, 2 x CH_2), 2.96 (s, 6H, 2xNCH_3), 5.48 (s, 1H, C-H), 6.96 (d, 2H, *J* = 8.6 Hz, Ar-H), 7.09 (d, 2H, *J* = 8.2 Hz, Ar-H), 11.86 (br, s, 2H, NH). Anal. calcd for: $C_{18}H_{23}ON_3S$: C, 65.71; H, 7.04; N, 12.77; Found: C, 65.69; H, 7.02; N, 12.73.

4e:4-(4-Chlorophenyl)-7,7-dimethyl-1,2,3,4,5,6,7,8-octahydroquinazoline-2,5-dione; 1.06 (s, 3H, C-CH_3), 1.24 (s, 3H, C-CH_3), 2.30-2.44 (m, 4H, 2 x CH_2), 5.50 (s, 1H, C-H), 7.04 (d, 2H, *J* = 9.6 Hz, Ar-H), 7.21 (d, 2H, *J* = 9.4 Hz, Ar-H), 11.82 (brs, 2H, NH). Anal. calcd for: $C_{16}H_{17}ON_2Cl$: C, 63.21; H, 5.36; N, 9.22; Found: C, 63.24; H, 5.38; N, 9.18.

4f:4-(4-Chlorophenyl)-7,7-dimethyl-5-oxo-1,2,3,4,5,6,7,8-octahydroquinazoline-2-thione; Solid: mp 218-220^{0}C; IR (KBr): 3360, 2959, 1670, 889,587 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.05 (s, 3H, C-CH_3), 1.26 (s, 3H, C-CH_3), 2.33-2.46 (m, 4H, 2 x CH_2), 5.47 (s, 1H, C-H), 7.02 (d, 2H, *J* = 9.6 Hz, Ar-H), 7.21 (d, 2H, *J* = 9.4 Hz, Ar-H), 11.87 (brs, 2H, NH); Anal. calcd for: $C_{16}H_{17}ON_2ClS$: C, 60.05; H, 5.35; N, 8.75; S, 10.01; Found:

C, 60.01; H,

5.38; N, 8.73; S,9.90.

4g:4-(3-Nitrophenyl)-7,7-dimethyl-

1,2,3,4,5,6,7,8-octahydroquinazoline-2,5-

dione;

= 1.18 (s, 3H, C-CH_3), 1.30 (s, 3H, C-CH_3), 2.36-2.50 (m, 4H, 2 x CH_2), 5.40 (s, 1H, C-H),

7.01-7.36 (m, 4H, Ar-H), 11.89 (brs, 2H, NH); Anal. calcd for: $C_{16}H_{17}O_4N_3$: C, 61.00; H,

5.43; N, 13.34; Found: C, 61.03; H, 5.40; N, 13.31.

4h: 4-(3-Nitrophenyl)-7,7-dimethyl-5-oxo-

1,2,3,4,5,6,7,8-octahydroquinazoline-2-

thione Mp. 207^{0}C; IR (KBr): 3340, 2962, 1667, 1230, 770 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.20 (s, 3H, C-CH_3), 1.28 (s, 3H, C-CH_3), 2.42-2.48 (m, 4H, 2 x CH_2), 5.54 (s, 1H, C-H), 7.08-

7.42 (m, 4H, Ar-H), 11.88 (brs, 2H, NH); Anal. calcd for: $C_{16}H_{17}O_3N_3S$: C, 58.05; H, 5.17; N, 12.69; S, 9.66; Found : C, 58.03; H, 5.18; N, 12.67; S,9.63

4i:4-(4-Methylphenyl)-7,7-dimethyl-

1,2,3,4,5,6,7,8-octahydroquinazoline-2,5-

dione; Solid: mp 257-259^{0}C; IR (KBr): 3350, 2954, 1645,1559, 824,670 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.08 (s, 3H, C-CH_3), 1.24 (s, 3H, C-CH_3), 2.28 (s, 3H, Ar-CH_3), 2.34-2.51 (m,

4H, 2 x CH_2), 5.47 (s, 1H, C-H), 7.02 (d, 2H, J = 8.2 Hz, Ar-H), 7.09 (d, 2H, J =8.0 Hz, Ar- H), 11.89 (brs, 2H, NH); Anal. calcd for: $C_{17}H_{20}O_2N_2$: C,71.89; H,7.09; N, 9.86; Found: C,71.87; H,7.08; N, 9.88.

4j:4-(4-Methylphenyl)-7,7-dimethyl-5-oxo- Solid: mp 188-190^{0}C; 1,2,3,4,5,6,7,8-octahydroquinazoline-2-

thione; Solid: mp 123-125^{0}C; IR (KBr): 3358, 2959, 1671,1585, 826,664 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.09 (s, 3H, C-CH_3), 1.24 (s, 3H, C-CH_3),2.28 (s, 3H, Ar-CH_3), 2.32-2.48 (m,

4H, 2 x CH_2), 5.47 (s, 1H, C-H), 6.96 (d, 2H, J = 8.1 Hz, Ar-H), 7.05 (d, 2H, J = 7.8 Hz, Ar-

H), 11.86 (brs, 2H, NH); Anal. alcd for: $C_{17}H_{20}ON_2$ S : C,68.06; H,6.72; N, 9.33;S,10.68; Found: C,68.09; H,6.73; N, 9.30;S,10.65.

4k:4-(4-Methoxyphenyl)-7,7-dimethyl-

1,2,3,4,5,6,7,8-octahydroquinazoline-2,5-

dione; Solid: mp 117-119^{0}C; IR (KBr): 3345, 2959, 1656,1586, 825,666 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.07 (s, 3H, C-CH_3), 1.19 (s, 3H, C-CH_3), 2.21-2.40 (m, 4H, 2 x CH_2), 3.74 (

s, 3H, OCH_3), 5.50 (s, 1H, C-H), 6.82 (d, 2H, J = 8.6 Hz, Ar-H), 6.98 (d, 2H, J = 8.2 Hz, Ar- H), 11.89 (brs, 2H, NH); Anal. calcd for: $C_{17}H_{20}O_3N_2$: C, 68.06; H, 6.72; N, 9.33; Found: C, 68.09; H,6.73; N, 9.31.

4l:4-(4-Methoxyphenyl)-7,7-dimethyl-5-
oxo-1,2,3,4,5,6,7,8-octahydroquinazoline-

2-thione; Solid: mp 130^{0}C; IR (KBr): 3445, 2959, 1636,1595, 923,823 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.07 (s, 3H, C-CH_3), 1.24 (s, 3H, C-CH_3), 2.28-2.47 (m, 4H, 2 x CH_2), 3.75 (s, 3H, OCH_3), 5.48 (s, 1H, C-H), 6.78 (d, 2H, *J* = 8.7 Hz, Ar-H), 6.97 (d, 2H, *J* = 8.1 Hz, Ar-H), 11.88 (brs, 2H, NH); Anal. calcd for: $C_{17}H_{20}O_2N_2$ S : C,64.61; H,6.37; N, 8.86;S,10.14; Found: C,64.64; H,6.39; N, 8.83;S,10.11.

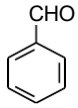
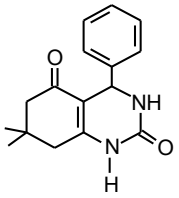
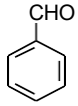
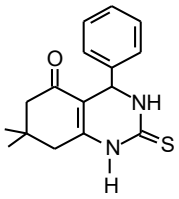
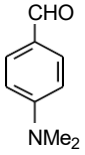
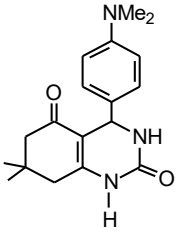
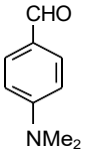
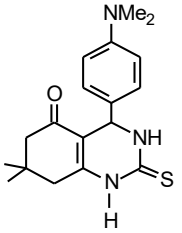
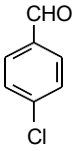
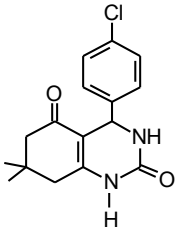
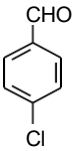
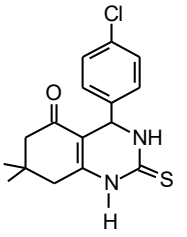
TABLE 1. EFFECT OF SOLVENT ON REACTION[a]

Entry	Solvent	Ratio (v/v)	Time (h)	Yield[b] (%)
1	H_2O	04	10	Trace
2	PEG(300)	04	02	---
3	PEG(400)	04	02	---
4	PEG(600)	04	02	20
5	Ethylene glycol	04	02	---
6	PEG(600)-H_2O	1:3	03	93
7	PEG(600)-H_2O	2:2	03	72
8	PEG-(600)-H_2O	3:1	03	04
9	PEG-(300)-H_2O	1:3	03	40
10	PEG-(400)-H_2O	1:3	03	65
11	Ethylene glycol-H_2O	1:3	03	35

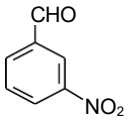
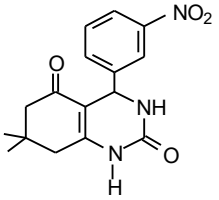
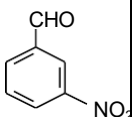
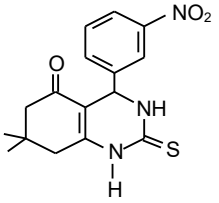
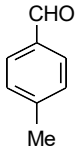
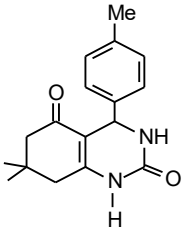
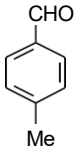
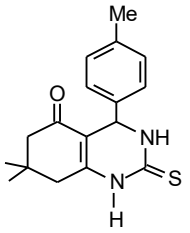
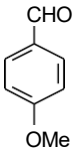
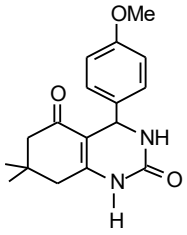
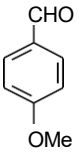
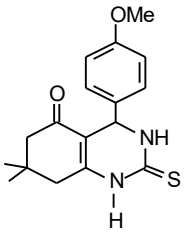
[a]Dimedone (1 mmol), urea (1.5 mmol), 4-methoxy benzaldehyde (1 mmol), solvent (4 mL)
[b]Isolated yields.

TABLE 2: REACTION OF DIMEDONE WITH UREA OR THIOUREA ANDALDEHYDES IN PEG(600) WATER MEDIUM.

Entry	Aldehyde	Product	Time (h)	Yield (%)	M.p. (°C)

a			2.00	87	180-182
b			2.45	89	165
c			2.45	87	247
d			3.00	91	209-211
e			2.15	84	262-264
F			2.30	86	218-220

Entry	A ldehy de	Product	Time (h)	Y ield (%)	M.p. (°C)

g			2.20	82	188-190
h			2.40	84	207
i			2.30	86	257-259
j			2.50	92	123 -125
k			2.30	89	117 -119
l			2.50	93	130

[a] All products were characterized by IR, [1]HNR and elemental analysis.
[b] Yield referes to pure isolated products
[c] Melting points were not corrected.

RESULT AND DISCUSSION:

To determine the most appropriate solvent combination, we attempted the

reaction of dimedone (1 mmol), urea (1.5 mmol) and 4- methoxy benzaldehyde (1mmol) in PEG (300), PEG (400), PEG (600), ethylene glycol, water and PEG-water (4mL, v/v ratio) without any added catalyst. The maximum yield of the desired product was obtained when the reaction was performed in PEG (600)-water v/v ratio 1:3 (Table 1 entry 6). The black oily mass was recovered when the reaction mixture was refluxed in pure PEG (300), PEG (400) and ethylene glycol as a solvent (Table 1, entries 2, 3, 5), possibly due to decomposition of starting materials. When the volume of PEG (600) was increased the decrease in yields of the products was observed (Table 1, entries 7 and 8). And when the reaction mixture was refluxed in water alone, the maximum amounts of reactants were recovered.

To generalize our solvent system, we synthesized several octahydroquinazolinone derivatives under the optimized reaction conditions and the results were summarized in Table 2. Various aromatic aldehydes underwent smooth reaction with dimedone and urea or thiourea in PEG (600)-water v/v ratio 1:3 to furnish excellent yields of products. The reactions were remarkably clean and no chromatographic separation was necessary to get pure products. In general, the aldehydes with electron-donating substituents on aromatic ring were provided slightly higher yields (Table 2, entries 4d, 4j, 4l) than the electron-withdrawing substituents (Table 2, entries 4f, 4h). Similarly, the reaction of dimedone and aldehyde with thiourea provided slightly higher yields than the reaction with urea. It is also important to note that the reaction proceeded smoothly in PEG (600)-water solvent system with tetrabutylammonium bromide (5 mol%) in combination with cesium carbonate (5 mol%) catalyst and products were obtained in excellent yields.

CONCLUSION:

In summary, we have devised a green solvent system for the three-component cyclisation of dimedone urea or thiourea and aromatic aldehydes using tetrabutylammonium bromide (5 mol%) in combination with cesium carbonate (5 mol%) at room temperature to provide octahydroquinazolinone derivatives with excellent yields. The simple work-up and isolation of pure products by recrystallization only are the important features of this protocol.

ACKNOWLEDGMENT:

The authors are grateful to Dr. Vijay Bhosale, Principal, Shivaji College Kannad, Aurangabad, MS, India for providing necessary research facilities to carry out Ph.D. work.

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