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Extensive Analysis of Tetrabutylammonium Tribromide: A Preferential Safe Solid Brominating Reagent in Coumarin-Quinoxaline Hybrid Motifs

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Abstract

Owing to the elevation in the applications in potential pharmacopeia of Coumarin condensed/fused heterocyclics, there is a substantial attention for the fabrication of coumarin scaffolds in preference to the green protocols. In order to achieve this in one-pot synthesis, 3-acetyl-2*H*-chromen-2-ones blended with *O*-phenylenediamines and tetrabutylammonium tribromide (TBATB) in stochiometric ratio. To extend the applicability of solid brominating agent Tetrabutylammonium tribromide, it is intended to be subjected for advanced analysis pertaining, Scanning Electron Microscopy-Energy Dispersive X-Ray analysis, Powder XRD analysis, Thermogravimetric/ Deferential Thermal Analysis. Efficacy profiling of title compounds were carried by comparing the mode of synthesis namely one-pot three components and sequential step-wise synthesis in addition to the solvent optimization screening. Analysis reports confirmed the supremacy of one-pot-three component synthesis in green reaction conditions. Qualitative analysis of the target compounds perceived with ^{1}H-NMR, ^{13}C-NMR, HRMS and IR spectroscopy.

Keywords: Coumarin condensed heterocyclics; One-pot-three component reaction; Scanning Electron Microscopy-Energy Dispersive X-Ray analysis; Powder XRD analysis; Thermogravimetric/ Deferential Thermal Analysis; Green protocols

Introduction

As defined by the Wikipedia, Coumarin IUPAC pronunciation is 2*H*-chromen-2-one is the fused aromatic Organic Chemical compound pertaining the Chemical formula, $C_9H_6O_2$. Benzene condensed with lactone ring made bicyclic conjugated cyclic ester, member of Benzopyrone and Chemical class of Lactone. Coumarin is the colourless crystal with vanilla odour, and tastes as bitter. It is the defensive chemical in the plants against predators. The drug Warfarin inhibits the blood clots, vein thrombosis and pulmonary embolism.

Natural occurrence

Chemistry of the Coumarin is initiated from the plant origin and further explored in few animals, even in the laboratory scale and eventually industrial bulk drug formulations are possible now. In the year 1820 first Coumarin extract was achieved from the tonka beans by Scientist A. Vogel and French word Coumarou is the base for the Coumarin indeed means Fragrance (Figure 1.1).

The plants of high Coumarin economy are .....(Figure 1.2)

- Vanilla Grass (*Anthoxanthum Odoratum*)
- Sweet woodruff (*Galium Odoratum*)
- Sweet Grass (*Hierochloe Odorata*)
- Sweet Clover (*Genus Melilotus*)
- Tonka Bean (*Dipteryx Odorata*)
- Cinnamon (*Cinnamon Verum*)
- Deertongue (*Carphephorus Odoratissimus*)
- Tilo (*Justicia Pectoralis*)
- Mullein (*Genus Verbascum*)
- Many Cherry Blossom (*Genus Prunus*)
- *Genus Glycyrrhiza*

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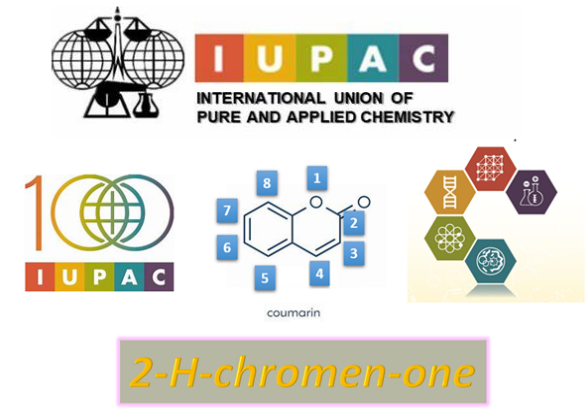

Figure 1.1: Chemical composition and IUPAC nomenclature of the Coumarin

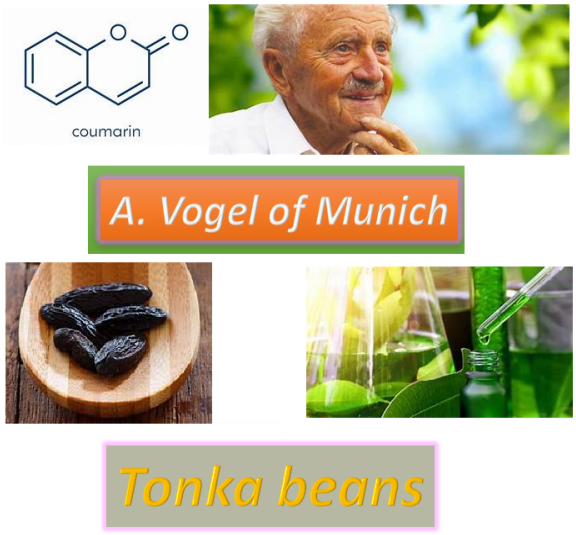

Figure 1.2: Inception of Coumarin research from the natural origin Tonka Beans

Step towards the Coumarin Laboratory synthesis

Due to its wide spread applications and amicable properties, there is a search for synthetic alternate for the Coumarin which made possible for the first time in the year 1868 by the **English Chemist William Henry Perkin** (Figure 1.3).

What makes Coumarin is the potential medicinal pharmacophore?

There are few snippets to choose Coumarin as the curative pharmacophore in the medicinal industry, they are:

- Simple methods of laboratory synthesis
- Being lactone is sensitive for the concentrated solutions accompanied by the mild reaction conditions
- Many more scientific reports on Green synthetic pathways
- Viable through multi-component-one-pot-tandem/cascade synthesis
- Active research all across the World
- Coumarin based heterocyclics are multifaceted therapeutics for instance:

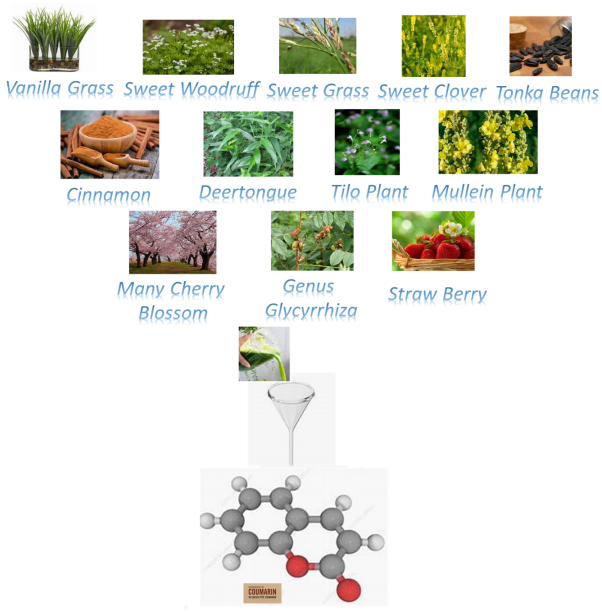

Figure 1.3: Plant Origin composed of the Coumarin

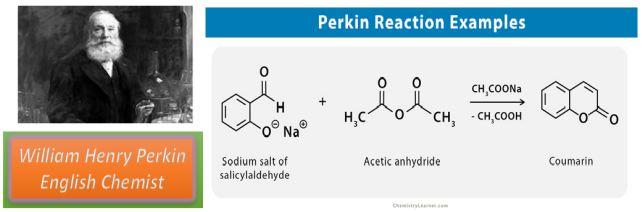

Figure 1.4: Laboratory synthetic design of the Coumarin starting from the Salicylaldehyde

Coumarin fused heterocyclics of five membered/six membered/condensed systems are efficient preventives/curatives with pathogenic resistance/static /cidal nature (Figure 1.4).

Coumarin and its extensions are active against Cancer tumours [1], Viruses [2], Anti-Bacterial [3], Anti-Fungal [4], Anti-Tubercular [5], Anti-Diabetic [6], Anti-Microbial [7], Anti-Convulsant [8], Anti-Prolifirative [9,] Anti-Alzheimer's [10], Anti-Protozoan [11] activity and Agro-Chemical applications including insect static and insect cidal activity [12].

Search for the Solid Brominating Agents - To Obey the Green Protocols

Schemes were sketched for the synthesis of series of 3-(Quinoxalin-2-yl)-2*H*-chromen-2-ones [4], where the starting material 3-acetyl-2*H*-chromen-2-ones required to be brominated and further condensed with *O*-phenylene diamines. Here in, the precious intermediate 3-bromo-acetyl-Coumarin proven as pivotal synthon for the library of heterocyclic compounds and other transformations. Hence, in order to synthesis 3-bromo-acetyl-Coumarin, acetyl Coumarin is brominated as per the Chemical requirement. But in laboratory practice we faced the hurdle to carry the liquid bromination due to there are multiple complications incurred, for instance:

- Liquid bromine is highly corrosive and hard to handle in open laboratory. Throat, skin, eyes and nozzle tissue damage due to direct contact with bromine fumes.
- It is difficult maintain the stichometry being it is in liquid form.
- Preparation of Br_2/H_2O or Br_2/ Glacial Acetic Acid should

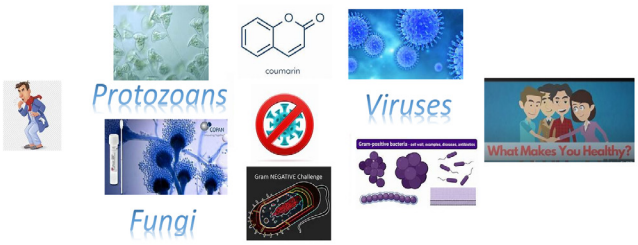

Figure 1.5: Coumarin in the treatment of the pathogens

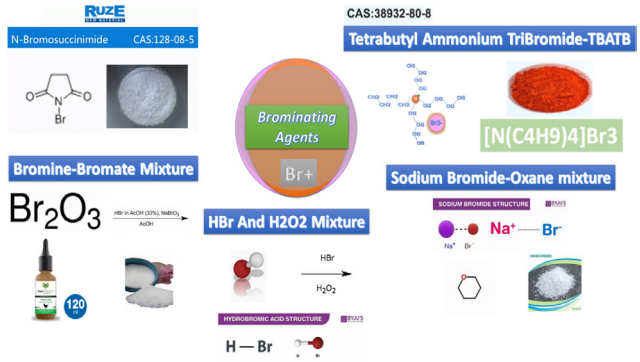

Figure 1.6: Compilation of Possible Solid Brominating Agents

strictly carry in the fuming hood with expert supervision.

- Product quality is not up to the mark with liquid bromination.
- % of the yield is not satisfactory.
- Difficult to wash out excessive or unreactive Bromine.
- Being it is fuming, not stable hence storage and transportation is difficult to achieve.

Hence there is a vigorous search for the mild, safe to handle and moreover overcomes all the practical problems faced with liquid Bromine (Figure 1.5).

Bromine-Bromate mixture to afford Hypo Bromous Acid [13], in-situ generation of the Bromine and HBr and H_2O_2 mixture [14], Oxidative Bromination [15], Sodium Bromide-Oxane [16], N-Bromosuccinmide-AIBN-CCl_4 [17], (Diacetoxyiodo)benzene and Lithium Bromide [18], Resin-BrO_3^- $MeNO_2$ [19], TBATB-TBAB [20] (Figure 1.6).

After the careful literature review on the safe Brominating agents TBATB was chosen as the efficient Brominating agent due to availability of the raw materials in the laboratory and ease of synthesis of solid Brominating agent at Room Temperature within 10-15 minutes accorded high yield of the product.

In order to explore the properties of the solid Brominating agent TBATB, we subjected it for various advanced analytical analysis. If the compound extensive properties are well known it is obvious to expand the applicability. That is why, SEM-EDX analysis performed for the screening of surface morphology, Powder XRD to understand its packing of atoms and TG/DTA analysis for the thermal stability.

Results and Discussion

In this research, 3-(quinoxaline-2-yl)-2*H*-chromen-2-one is planned for the synthesis starting from 3-acetyl-2*H*-chromen-2-one (**1**) Target compound is accorded in the multiple pathways namely conventional step-wise method and one-pot-multicomponent synthesis. In the earlier case, Coumarin-

Figure 2.1: Knoevenagel synthesis of 3-acetyl-2*H*-chromen-2-oneand further bromination

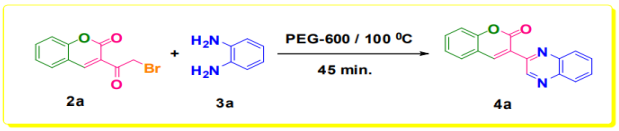

Figure 2.2: Condensation followed by cyclization of 2a with 3a to achieve Coumarin-Quinoxaline hybrid

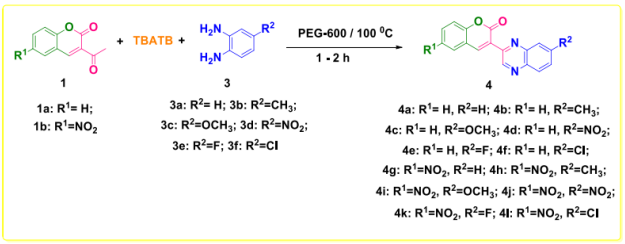

Figure 2.3: Greener One-Pot-Three-Compound Synthesis of 3-(quinoxalin-2-yl)-2*H*-chromen-2-one (**4a-l**)

Quinoxaline hybrids are achieved by the key intermediate i.e., 3-(2-bromoacetyl)-2*H*-chromen-2-one (**2**) which is formed by the stochiometric Bromination of 3-acetyl-2*H*-chromen-2-one (**1**) with solid TBATB in the presence of TBAB as the phase transfer catalyst. Synthesis of 3-acetyl-2*H*-chromen-2-one (**1**) is achieved by the Knoevenagel condensation between Salicylaldehyde with Ethyl Aceto Acetate (EAA) in equimolar ratio (Figure 2.1).

3-(2-bromoacetyl)-2*H*-chromen-2-one (**2a**) further participate in the condensation cyclization with benzene-1,2-diaminein (**3a**) the presence of green reaction conditions afforded the expected compound 3-(quinoxalin-2-yl)-2*H*-chromen-2-one (**4a**) (Figure 2.2).

In the alternative methodology, the target compound is achieved in a one-pot-three-component methodology by blending 3-acetyl-2*H*-chromen-2-one (**1**), TBATB in the presence of TBAB, benzene-1,2-diaminein (**3a**) in the molar ratio gave 3-(quinoxalin-2-yl)-2*H*-chromen-2-one (**4**).

Further similar synthetic strategy is extended to the other derivatives of compound (**1**) and (**3**) afforded series of **4(a-l)** extensions as mentioned in **scheme-3** (Figure 2.3).

Based on the importance of alternative in specific efficient Brominating agent, Tetrabutyl Ammonium Tribromide (TBATB) synthesis conditions are optimized as follows (Figure 2.4).

Synthesis of the Tetrabutyl Ammonium Tribromide (Tbatb)

Tetrabutyl Butyl Ammonium Bromide (**TBAB**) (120 mmol), Sodium Bromate (**$NaoBr_3$**) (40 mmol) and Water (300 mL) was mixed and stirred on magnetic plate at room temperature for 10 min. Add 48% Aq. HBr drop wise while stirring until the complete bromination is carried

(Approx. 28 ml) and further stirred more 30'. An Orange colour solid precipitated is filtered, washed with water and air-dried accorded 55.36 g. (96%) yield. and M.P: 75° C.

Charectarization of the Solid TBATB

TBATB is the well-established efficient indeed environmental-benign solid brominating reagent accord the Bromination within minutes at simple room temperature stirring. Researchers and even at the academic laboratory practical experimentation TBATB find general applications. Hence, further unfolding the extensive properties of the potential compound will assort the additional applications with supremacy in the quality. In this context, TBATB is disclosed for the analytical testing as follows (Figure 3.1).

Surface Morphology Evolution by Scanning Electron Microscopy (SEM)

As per the Geochemical Instrumentation and Analysis, the Scanning Electron Microscope (SEM) is equipped with concentrated beam of high-energy electrons on interaction with solid specimens captures their surface signals. The way of Electron-Sample interaction rolls out the external morphology called texture, Chemical composition, and Crystalline arrangement of atoms. In addition, SEM analysis serves at selected point locations, which intern anticipates the qualitative and semi-qualitative Chemical composition. Advanced, all-inclusive, high-end, Windows-10 version software, suite Iridium-Ultra features is operated for the mapping, imaging, and for fore fronted automation and analysis tools [21].

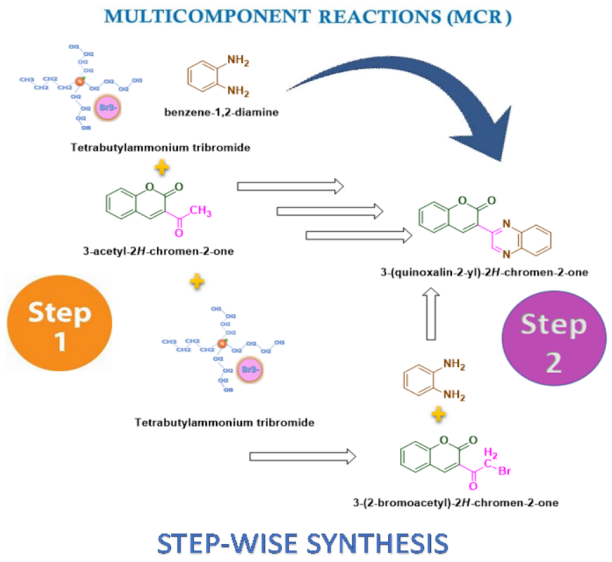

Figure 2.4: Comprehensive Chemical synthesis chart of the3-(quinoxalin-2-yl)-2*H*-chromen-2-one

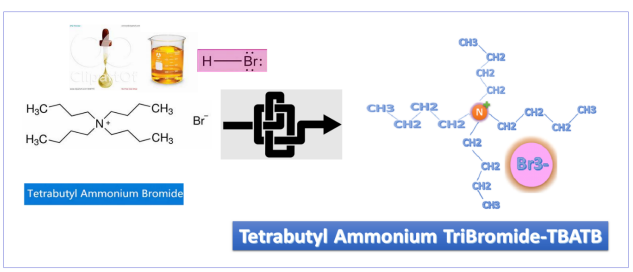

Figure 3.1: Synthetic analogy of the TBATB starting from TBAB

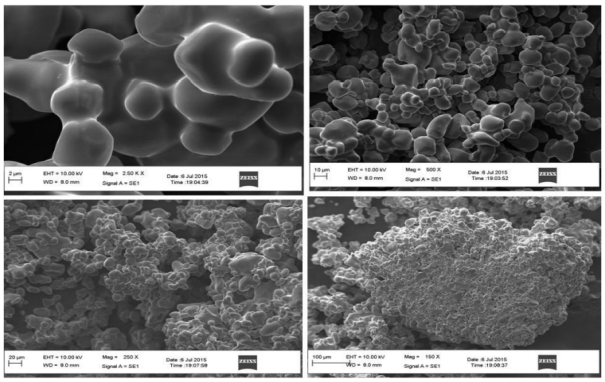

Figure 3.2: SEM surface morphology micrographs of TBATB in different dimensions and magnifications

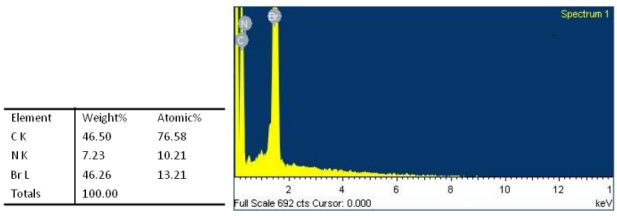

Figure 3.3: EDX-Chemical spectral graph of the TBATB, tri-Peak confirms the presence of Tri elements in the sample.

TBATB surface morphology SEM Micrographs predicted that proper alignment and regularity even in the different dimensions and magnifications assigns crystallinity Characterization of TBATB solid (Figure 3.2).

Elemental Composition of TBATB by EDX analysis

As quoted by the intertech, Energy Dispersive X-Ray Analysis is the X-ray tool for the identification of the elemental composition of the materials. EDX is either connected with SEM or TEM where the specimen images captured, spectral peaks correspond to the elements that make true composition of the sample under analysis. This technique is applicable for the mapping of the sample by identifying near-surface elemental composition at different dimensions. It is executed that, EDX in conjunction with SEM gives the elemental composition. A beam of the electrons associated with 10-20 keV strikes on the sample surface, emits the X-ray from the material and energy emission is sample dependent [22].

EDX verification of the TBATB accorded tri peak in the graphs pertaining Carbon, Nitrogen and Bromine elements respectively at its core. The analysis is quantitative in terms of weight and atomic % composition of the individual elements (Figure 3.3).

Crystallinity Screening By X-Ray Powder Diffraction: A 3-D Analysis

As manifested by the Geochemical Instrumentation and Analysis, X-Ray Powder Diffraction (XRD) is the powerful analytic tool for the evolution of the Unit cell dimensions in the crystalline substances, Crystal structures and atom spacing as well. Sample of analysis must be grounded to fine powder, homogenized and tested for the average bulk composition.

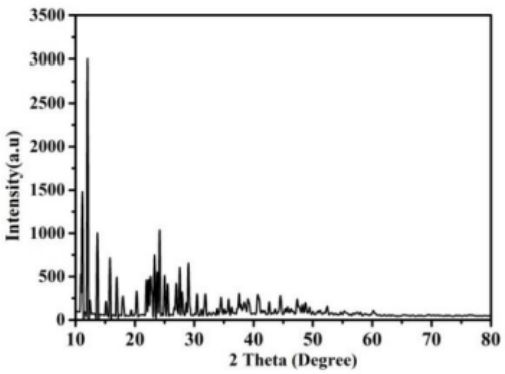

Figure 3.4: Presence of strong signals at 2 Theta degree angle is the authorization for the crystallinity of TBATB

Since the crystals are 3-Dimensional diffraction gratings they are susceptible to obey Brag's equation (nλ = 2dsinθ). X-Ray Powder Diffraction (XRD) is discovered by Max Von Laue in the year 1912 which applied for the nature of

Crystalline substances i.e.: 3-Dimensional Diffraction Grating for X-ray wavelengths which in line with the spacing of the planes of the crystalline lattice. This technique is amicable in the determination of the crystal dimensions and atomic spacing [23].

TBATB under the XRD screening received the reports as: 2 θ = 10-80° range with wide peak centred at 2 θ angle inclination mentions the strong crystallinity. Crystalline peaks in the range of 10-60° further confirm the regular 3-Dimensional crystallinity (Figure 3.4).

Thermal Stability and/or Degradation Screening Of TBATB by TG/DTA Approach

In access to the IIT Bombay, TG/DTA analysis is the combined feature of Thermo Gravimetry and Differential Thermal Analysis ensures high flexibility of analysis. Sample kept for the observation, solicited for the identical thermal interaction and environment throughout the analysis. This analysis provides whether an endothermic or

exothermic transition which is associated with weight loss in contrary to the physical transformations such as melting or crystallization [24].

TG/DTA analysis encapsulates the following applications:

- Decomposition and transition temperatures
- Filler content
- Heat of Transition
- Volatile nature
- Oxidative and Thermal stability supervision

TBATB on secured thermal analysis with TG/DTA approach seized the melting endotherm on thermal treatment, with Enthalpy value 278.6 J/g. Differential Thermal Analysis (DTA) and Thermo Gravimetric Analysis (TGA) of the sample TBATB are observed under standard set of conditions. Thermal heating of sample is accompanied by inert Nitrogen atmosphere with exponential rise in temperature 5^0 C / min. Thermo Gravimetric Endo-thermic peak is emerged at 75^0 C denotes its Melting Point of TBATB. This data is further supported by its DSC curve. Another, far-reaching fetching of the analysis is the sample retains its morphology and not incurred with phase transition as the temperature gradation is possible is the confirmation for its thermal stability and even higher temperature resistance.

Experimental Section

Structural prediction is accomplished by general physical and analytical screening. Melting point of the final compounds is carried by open capillary tubes in the Sulphuric Acid bath. Preliminary identification is carried by the TLC analysis, silica gel-G coated sheets marketed by the MERCK Company and compound is visualized under UV lamp and Pulverized Iodine. [1] HNMR spectrums were recorded in Deuterated DMSO-d6 solvent media using TMS as the internal standard using 400 MHz spectrometer. [13] CNMR spectrums were recorded in Deuterated DMSO-d6 solvent media using TMS as the internal standard using 100 MHz spectrometer. Agilent-LCMS instrument is used for HRMS spectrums recorded in NIPER. Perkin Elmer IR Spectrophotometer is used for the IR analysis. Chemical reagents, starting materials purchased from commercial sources and are laboratory grade of purity.

Step-wise synthesis of 3-(Quinoxalin-2-yl)-2*H*-chromen-2-one [4]

Step-1: General procedure for the synthesis of 3-(2-bromoacetyl)-2*H*-chromen-2-one [2] from 3-acetyl-2*H*-chromen-2-one [1]

20 mmol of the compound [1] is added to the TBATB (20 mmol) in PEG-600 solvent media (30 mL). The reaction mixture is stirred on the mechanical stirrer at room temperature for about 1-1 ½ hr. Completion of the reaction confirmed by the TLC and water work up is performed with ice cold water. Separated solid filtered under suction, water washed and air dried. The crude product is recrystallized in activated charcoal gave the pure 3-Bromoacetyl Coumarin.

It is white colour solid afforded yield is 5.01 (95%), MP: 165-167 °C, IR(KBr) V*max*/Cm$^-$: 1717 Cm^{-1} for Coumarin carbonyl group (-CO-), 1683 for acetyl -CO- group (-CO-CH_3), [1] H-NMR (400 MHz, DMSO-d6/TMS): δ 4.64 (s, 2H, -CH_2), 7.36-8.01 (Complex, multiplet, 4H, Ar-H), 8.67 (s, 1H, Ar-H); [13] C-NMR (100 MHz, DMSO-d6): 36.4, 116.7, 118.3, 122.5, 125-1, 131.0, 135.2, 148.5, 152.3, 163.7, 190.4. HRMS calculated for $C_{11}H_7BrO_3$ $[M+H]^+$: 266.9657 and observed value is 265.957856 u.

Step-2: General procedure for the synthesis of 3-(Quinoxalin-2-yl)-2*H*-chromen-2-one [4] by reacting 3-(2-bromoacetyl)-2*H*-chromen-2-one (2) with TBATB [3]

A mixture of compound [2] (10 mmol) with compound 3 (10 mmol) in Poly Ethylene Glycol-600 (20 mL) heated at 100 °C for the time duration of the 30 min. about 1 hr. Solid product obtained filtered, washed with water and dried at room temperature. The crude product is recrystallized from the activated charcoal accorded pure 3-(Quinoxalin-2-yl)-2*H*-chromen-2-one [4].

It is the brown colour solid. Yield: 2.41 g (88%), MP: 198-200 °C (Methanol), IR (KBr) V *max*/ Cm^{-1}: 1715 Cm^{-1} for Coumarin Carbonyl (Strong, sharp, -CO-), [1] H-NMR (400 MHz, DMSO-d6/TMS): δ 7.33-8.35 (multiplet, 8H, Ar-H), 8.86 (s, 1H, Ar-H), 9.57 (s, 1H, Ar-H); [13] C-NMR (100 MHz, DMSO-d6): 116.3, 119.2, 123.6, 124.6, 128.7, 129.1, 129.6, 130.2, 130.5, 133.1, 141.3, 144.6, 145.2, 147.3, 153.5, 159.3, HRMS calculated as $C_{17}H_{10}N_2O_2$ $[M+H]^+$: 275.08205 u, found 275. 08276.

Multi-Component-One-Pot-Synthesis of 3-(Quinoxalin-2-yl)-2*H*-chromen-2-one [4] from compound [1], Compound [3] and TBATB

Compound [1] (10 mmol), TBATB (10 mmol), compound [3] (10 mmol) dissolved in Poly Ethylene Glycol-600 (PEG-600) in 20 mL mixture is heated at 100 °C for 1-2 hrs. Reaction progress is monitored with TLC. Completed reaction is poured in to ice-cold water approximately 60 mL. Solid is filtered, washed with water and air-dried. Crude product is recrystallized with activated Charcoal to accord the pure 3-(Quinolin-2-yl)-2*H*-chromen-2-one [4].

Conclusions

In summary,3-(Quinoxalin-2-yl)-2*H*-chromen-2-ones [4] is the expected research output was conveniently synthesized by chemical transformation of the 3-acetyl-2*H*-chromen-2-one [1]. In initial attempts, 3-acetyl-2*H*-chromen-2-one [**1**] is reacted in the both step-wise and one-pot three components Green Methodology. In the step wise design approach, the starting material is required to be brominated hence found benign alternative for liquid bromine that is TBATB based on the availability and ease of synthesis. The results achieved are high grade in terms of quality and quantity. Hence, further extensive analytical screening of the TBATB was accomplished with SEM-EDX, XRD studies and TG/DTA approaches to evaluate Surface Morphology-Atomic Spacing-Element composition, Crystalline dimensions-structure and Thermal stability respectively. Based on the all above predictions it is anticipated that, TBATB exist in the regular 3-Dimensional crystalline structure which is the foundation for the explorable applications of TBATB in the near future. Finally, 3-(2-bromoacetyl)-2*H*-chromen-2-one [2] is condensed with benzene-1,2-diaminein [**3**] accorded the title compound [4].

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