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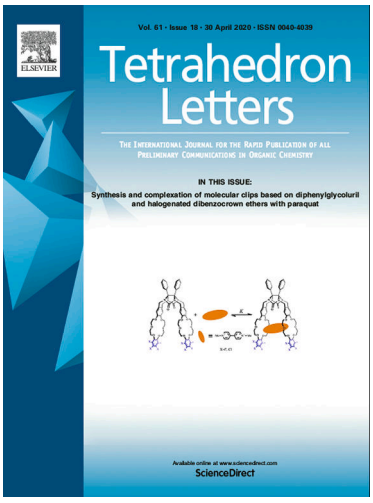

Stereoselective Synthesis of 9-Vinyl Substituted Unsymmetrical Xanthenes and Thioxanthenes

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Stereoselective Synthesis of 9-Vinyl Substituted Unsymmetrical Xanthenes and Thioxanthenes

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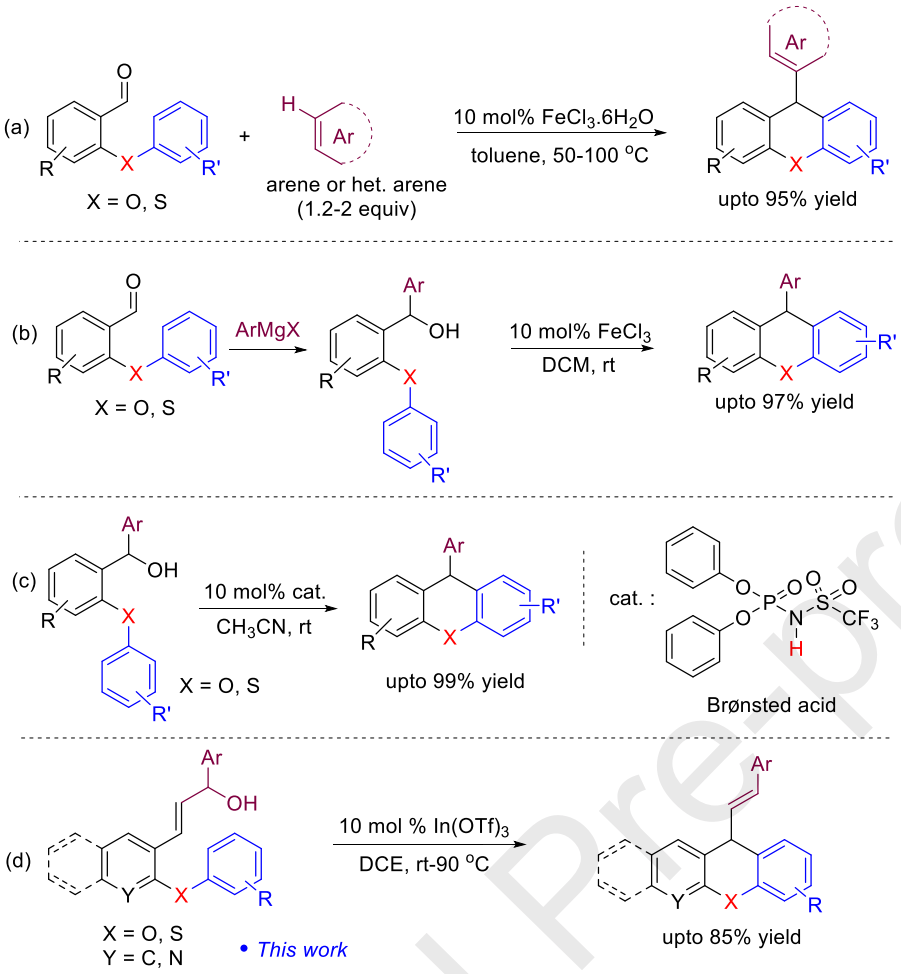
Abstract: Activated 2^{o}-allylic alcohols derived from 2-aryloxybenzaldehydes and 2-(arythio)benzaldehydes undergo intramolecular Friedel-Crafts alkylation reaction when heated with catalytic amount of a Lewis acid in 1,2-dichloroethane to provide highly *E*-selective 9-vinyl substituted unsymmetrical novel xanthenes and thioxanthenes in good yields.

Introduction: Xanthenes and thioxanthenes are important polycyclic motifs found in many biologically important compounds including natural products.[1] Xanthene and thioxanthene derivatives are known to exhibit antimicrobial,[2] antiparasitic,[3] anti-cancer,[4] antitumor,[5] anti-proliferic,[6] anti-inflamatory and antioxidant activities.[7] In addition, thioxanthenes are utilized for the development of neuroleptic and antidepressive drugs. Some thioxanthenes are used as antipsychotic drugs used in the treatment of schizophrenia and other psychotic disorders.[8] Besides these valuable biological properties, xanthene derivatives are also used in photodynamic therapy,[9] as chirooptical molecular switches[10] and as commercially available dyes[11] such as fluorescence, rhodamines, etc. Unsubstituted 9*H*-xanthenes and thioxanthenes are extremely useful advanced starting material for the development of novel C-H functionalization reactions,[12] organocatalytic asymmetric cross-dehydrogenative coupling reactions,[13] anodic oxidative cross-dehydrogenative reactions[14] and kinetic as well as mechanistic studies.[15] Due to these widespread importance, that range from medicinal chemistry to material science, synthesis of xanthene and thioxanthene derivatives have continued attracting research interest of synthetic chemists worldwide.[16]

2-Aryloxybenzaldehydes and 2-(arylthio)benzaldehydes are easily accessible from commercially available 2-flurobenzaldehydes and phenols/thiophenols via direct aromatic nucleophilic substitution reaction,[17] and thus have been extensively utilized for the synthesis of xanthene and thioxanthene derivatives, particularly 9-aryl/heteroaryl substituted xanthenes and thioxanthenes.[18] For instance, Li et al. reported a $FeCl_3$-catalyzed domino reaction wherein two equivalent of electron-rich (hetero)arenes undergo condensation with 2-aryloxybenzaldehydes/2-(arylthio)benzaldehydes to provide 9-(hetero)aryl xanthenes/thioxanthenes (Scheme 1a).[18a] Similar domino reaction catalyzed by $Sc(OTf)_3$ was disclosed by Panda et al.[18b] Addition of (hetero)aryl Grignard reagents to 2-aryloxybenzaldehydes and 2-(arylthio)benzaldehydes leads to π-activated 2^{o}-alcohols which were converted into 9-arylxanthenes/thioxanthenes via intramolecular Friedel-Crafts alkylation (FCA) reaction using catalytic amount of $FeCl_3$ (Scheme 1b)[17] as

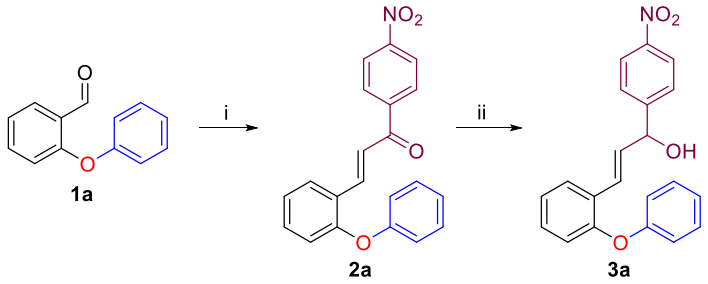


Scheme 1: Synthesis of 9-substituted xanthene and thioxanthenes from 2-aryloxybenzaldehydes and 2-(arylthio)benzaldehydes.

well as organic Brønsted super acid[18c,18d] (Scheme 1c). Notably, allylic alcohols, being another important class of π-activated alcohols,[19] have also been widely used in Brønsted/Lewis acid catalyzed inter- and intramolecular FCA reaction. For 2°-allylic alcohols, these reactions often lead to stereoselective formation of alkenes.[20] For example, Morita-Baylis-Hillman (MBH) adducts undergo intermolecular FCA reaction with arenes providing *E*- or Z-selective trisubstituted olefins depending on the electron-withdrawing group present in the MBH adducts.[21] Drawing inspiration from these literature reports, we devised a strategy to synthesize 9-vinyl xanthenes/thioxanthens via intramolecular FCA reaction on 2°-allylic alcohols derived from 2-aryloxybenzaldehydes and 2-(arylthio)benzaldehydes (Scheme 1d). In continuation with our ongoing effort to synthesize novel xanthene and thioxanthenes,[22] herein we report a $In(OTf)_3$-catalyzed intramolecular FCA reaction for the synthesis of *E*-selective 9-vinyl unsymmetrical novel xanthenes and thioxanthenes.

Our investigation on the intramolecular FCA reaction commenced with (*E*)-1-(4-nitrophenyl)-3-(2-phenoxyphenyl)prop-2-en-1-ol **3a** which was obtained in two steps from 2-

phenoxybenzaldehyde **1a** following reported procedures (Scheme 2).[23] Knoevenagel type condensation of 2-phenoxybenzaldehyde **1a** with 4′-nitroacetophenone in the presence of 20 mol% piperidine and 40 mol% AcOH in refluxing benzene provided (*E*)-1-(4-nitrophenyl)-3-(2-phenoxyphenyl)prop-2-en-1-one **2a** in 78% yield. Chemoselective reduction of enone **2a** under Luche conditions[24] provided allylic alcohol **3a** in nearly quantitative yield (98%). With the allylic alcohol **3a** in hand, we sought to find conditions for the envisaged intramolecular FCA reaction.



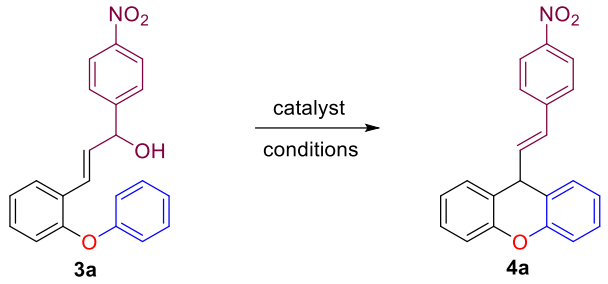
Scheme 2: (i) 4′-nitroacetophenone, piperidine, AcOH, benzene, reflux, 4 h, 78%; (ii) $CeCl_3.7H_2O$, $NaBH_4$, MeOH-DCM, 0 ^oC, 15 min, 98%.

Reaction of **3a** in the presence of 10 mol% $In(OTf)_3$ in DCM at rt (35 ^oC) was slow and underwent approximately 50% conversion (40% yield) to the xanthene **4a** after 24 h (entry a, Table 1). Compound **4a** was characterized unambiguously by ^1H & ^{13}C NMR and HRMS data. Notably, formation of only the *E*-isomer was observed to the limits of detection by ^1H NMR of the crude reaction mixture. Carrying out the reaction at 50 ^oC in 1,2-dichloroethane (DCE), better conversion (80%) and yield (65%) was observed after 4 h (entry b, Table 1). However, to our delight, complete conversion and satisfactory yield (78%) was obtained within 15 min when the reaction was carried out at 70 ^oC. The $In(OTf)_3$-catalyzed cyclization reaction in CH_3CN and THF as solvent provided slightly lower yield of xanthene **4a** (entries d-e, Table 1). When 10 mol% $Sc(OTf)_3$ was used as a catalyst, the reaction provided xanthene **4**a in 72% yield after 30 min (entry f, Table 1). $Yb(OTf)_3$ was found to be less effective when compared to $In(OTf)_3$ for the intramolecular FCA reaction. $InCl_3$ provided 75% yield of xanthene **4a** whereas $FeCl_3$ provided 65% yield of xanthene **4a** (entries h-i, Table 1). It is worth mentioning that the intramolecular FCA reaction of **3a** gave only a small amount of xanthene **4a** and a mixture of unidentified products (entry j, Table 1) under solvent free conditions at 50 ^oC.

With the optimized conditions in hand, we sought to evaluate the scope of the intramolecular FCA reaction. As shown in Scheme 3, the $In(OTf)_3$-catalyzed intramolecular FCA reactions were successful on a number of 2^o-allylic alcohols synthesized in two steps (as shown in Scheme 2) from a variety of 2-aryloxybenzaldehydes and 2-(arythio)benzaldehydes. In all cases, acetopheneones bearing an electron-withdrawing group (NO_2, CN, Cl, Br) at p- or m-position were utilized for the Knoevenagel type condensation reaction. Interestingly, 2-aryloxyquinoline 3-carboxyaldehyde could also be employed for the preparation of 2^o-allylic alcohols. As evident from the experimental results presented in Scheme 3, the electron density of the aryl ring B as

well as the nature of substituent on the aryl rings A & C have significant effect on the reactivity of the allylic alcohols towards the intramolecular FCA reaction. For example, the cyclization reaction occurred at lower temperature when a strong electron-withdrawing NO_2-group is

Table 1: Optimization of reaction conditions[a]



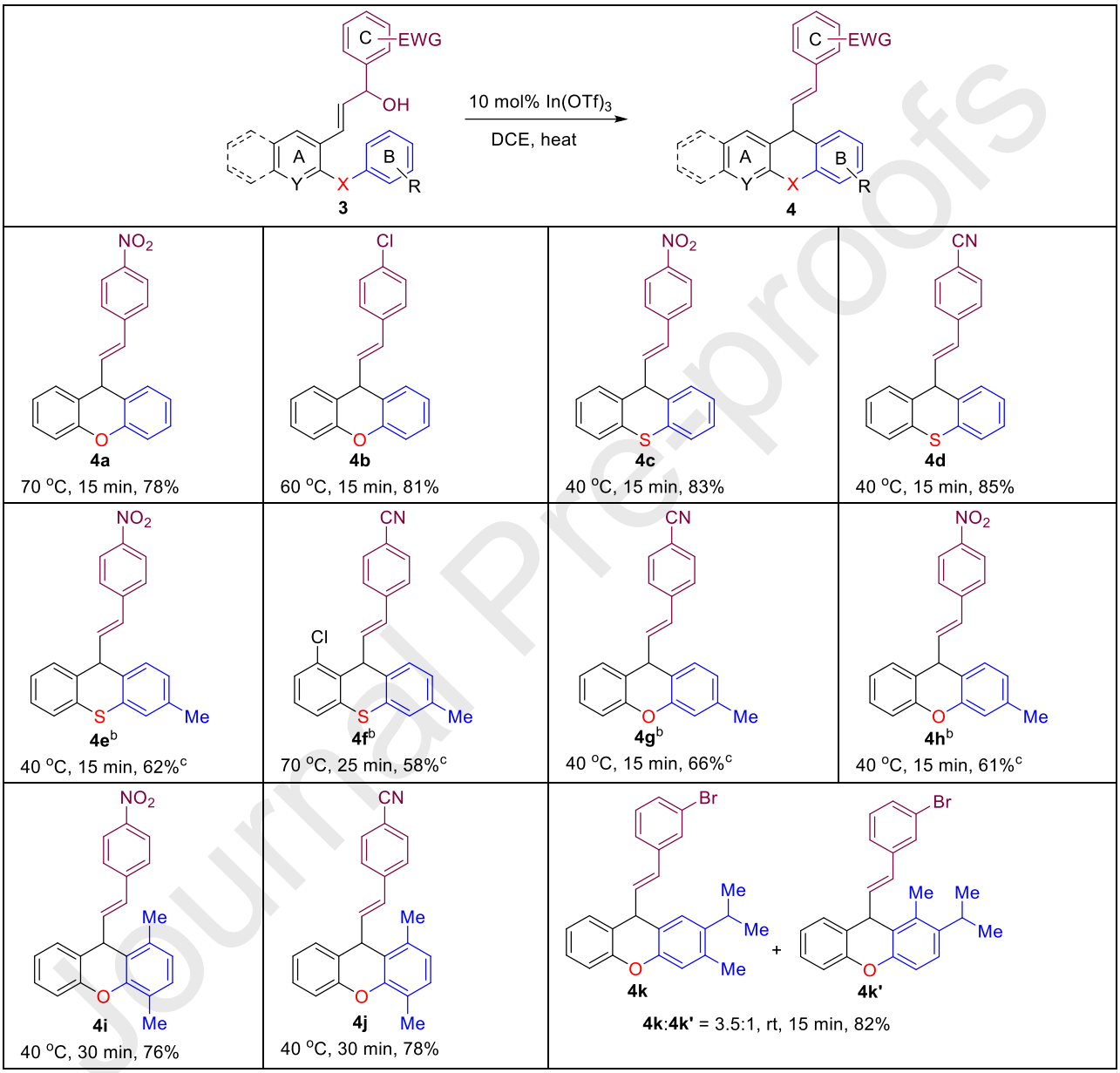
entry	catalyst	conditions	yield (%)
a	$In(OTf)_3$	DCM, rt, 24 h	40[b]
b	$In(OTf)_3$	DCE, 50 °C, 4 h	65
c	**$In(OTf)_3$**	**DCE, 70 °C, 15 min**	**78**
d	$In(OTf)_3$	CH_3CN, 70 °C, 15 min	72
e	$In(OTf)_3$	THF, 70 °C, 30 min	70
f	$Sc(OTf)_3$	DCE, 70 °C, 30 min	72
g	$Yb(OTf)_3$	DCE, 70 °C, 30 min	68
h	$InCl_3$	DCE, 70 °C, 15 min	75
i	$FeCl_3$	DCE, 70 °C, 20 min	64
j	$In(OTf)_3$	neat, 50 °C, 2 h	ND

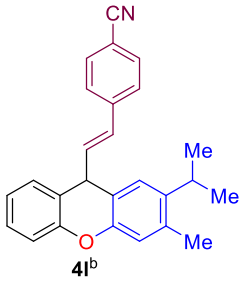
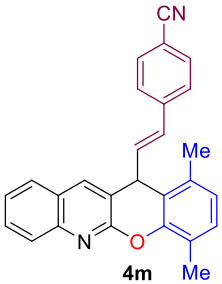
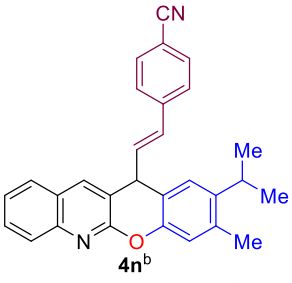
[a]All reactions were carried out in 0.20 mmol scale using 10 mol% catalyst in appropriate solvent (0.2M concentration); [b]50% conversion; ND = yield not determined.

replaced by a Cl-atom in the aryl ring C (e.g., **4a** vs **4b**). Due to the positive inductive effect, alkyl groups present in the aryl ring B increased the reactivity significantly (for comparison, **4a** vs **4h** vs **4l**). Presence of the electronegative Cl-atom in the aryl ring A had a negative impact on the reactivity (**4e** vs **4f**). Allyl alcohols derived from 2-(arylthio)benzaldehydes were, in general, more reactive towards the intramolecular FCA reaction due to the lower electronegativity of S-atom. Allyl alcohols having unsymmetrically substituted aryl ring B provided xanthenes/thioxanthenes as a variable mixture of regioisomers which were separated by column chromatoghaphy. Isolated yield for the major regioisomers (**4e-4h**, **4l**, **4n**) of the cyclized products are shown in Scheme 3. However, unfortunately, regioisomeric mixture of xanthenes (**4l**, **4l´**) could not be separated by column chromatography and were obtained as a mixture in 3.5:1 ratio in 82% yield. In general, the p-position to an alkyl (methyl) group is preferred over the o-position in the FCA process to minimize the steric effect. It is worth noting that the cyclization also occurs at the o-position to an alkyl (methyl) group at moderate temperature to provide xanthenes in good yields (**4i-4j**, Scheme 3). Due to the presence of an additional

electronegative atom (N-atom), the 2°-allylic alcohols bearing quinoline moiety as the aryl ring A, exhibited low reactivity at lower temperature. However, gratifyingly, complete conversion occurred at higher temperature using 20 mol% of $In(OTf)_3$ to furnish the desired novel quinoline

Scheme 3: Synthesis of 9-vinyl xanthene and thioxathene[a]



 **4l**[b] rt, 10 min, 64%[c,d]	 **4m** 90 °C, 90 min, 72%[d]	 **4n**[b] 70 °C, 50 min, 62%[c,d]	

[a]Reactions were carried out in 0.20 mmol scale using 10 mol% $In(OTf)_3$ in DCE (0.2M); [b]major isomer; [c]yield of major isomer; [d]reaction was carried out using 20 mol% $In(OTf)_3$.

fused xanthenes **4m** and **4n** in good yields. In all cases, the stereochemistry of the olefin in the synthesized 9-vinyl xanthenes and thioxanthenes was invariably found to be the thermodynamically stable *E*-configuration possessing a high coupling constant, J = 15.6 Hz. Interestingly, adding to our advantage, a solution of compound **4l** in hexanes upon storage in a freezer yielded single crystals suitable for X-ray diffraction studies. As shown in Figure 1, the ORTEP diagram further confirms the *E*-geometry of the disubstituted olefin as well as the orientation of alkyl groups in the aryl ring B. The vinyl side chain in xanthene **4l** orients in a linear fashion perpendicular to the plan containing the xanthene nucleus.

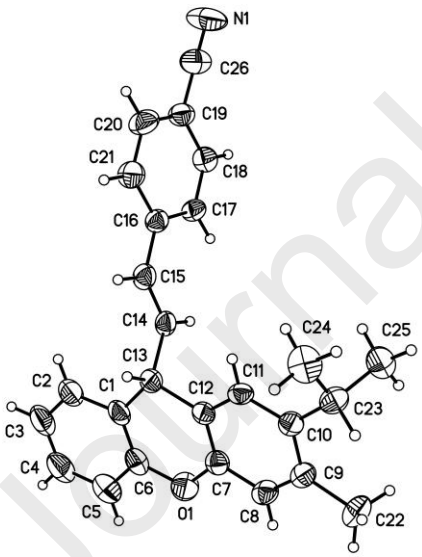
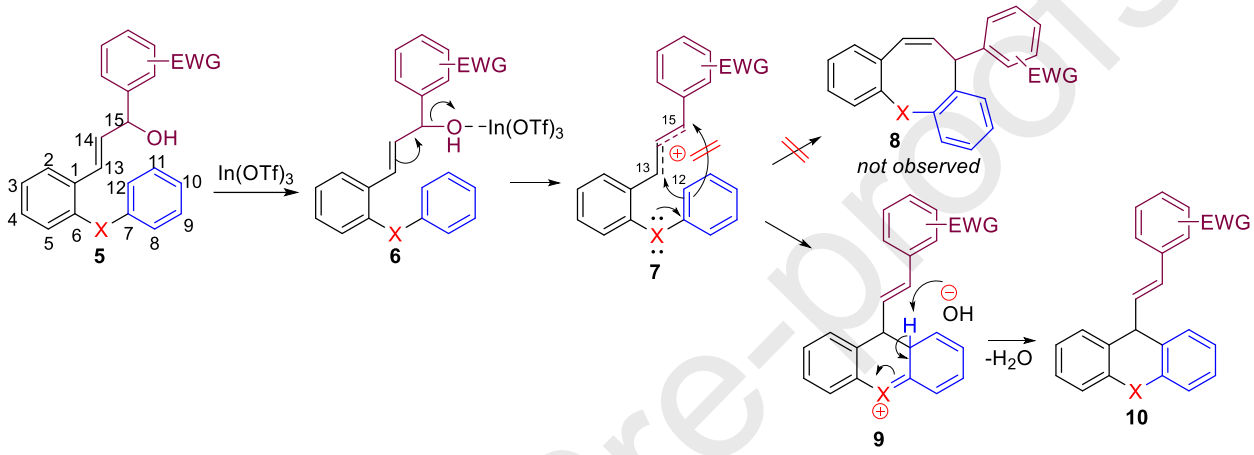


Figure 1: ORTEP diagram of compound **4l** (CCDC No. 2010351)

The tentative mechanism of the xanthene synthesis is outlined in Scheme 4. $In(OTf)_3$ promotes the formation of resonance stabilized allylic carbocation **7** at the benzylic position (C15). Intramulecular FCA reaction forming C12-C13 bond provides xanthene **10** with the loss of a neutral water molecule from intermediate **9**. Intramolecular FCA forming C12-C15 bond would lead to a strained 8-membered ring that incorporates an alkene making this pathway for

cyclization thermodynamically unfavorable. Although generation of a carbocation at the benzylic position of the aryl ring C that carries a strong electron-withdrawing group at the para position is of formidable challenge, the overall success of the reaction is attributed to the resonance stabilization of the resulting benzylic/allylic carbocation. Notably, the water molecule released in the intramolecular FCA reaction did not participate in hydration of the olefin present in the xanthenes/thioxanthenes under the reaction conditions. This could be attributed to the partial electron-deficient nature of the resulting olefin due to the electron-withdrawing group present in the aryl ring C and the use of a Lewis acid for the intramolecular FCA reaction.



Scheme 4: Plausible mechanism of the intramolecular FCA reaction

In summary, a novel method for highly stereoselective synthesis of 9-vinyl xanthenes and thioxanthenes has been developed. The syntheses rely upon a $In(OTf)_3$-catalyzed regio- and stereoselective intramolecular Friedel-Crafts alkylation reaction of easily accessible π-activated 2^o-allylic alcohols derived from a variety of 2-aryloxybenzaldehydes/2-(arylthio)benzaldehydes and commercially available acetophenones. Remarkably, the mild reaction conditions are also suitable for efficient synthesis of quinoline fused xanthenes. As the electronegativity of N-atom is less than O-atom, we anticipate that the aza-analogues of xanathenes and thioxanthenes could also be synthesized by this methodology. The presence of a versatile vinyl functionality in the products suggests that further annulation with appropriately substituted aryl ring B might be possible to generate novel polycyclic compounds. We will explore this in our ongoing study of the chemistry of xanthenes and thioxanthenes.

Notes: Authors declare no competing financial interest

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Associated content:

The Supplementary Information is available (Experimental procedures, analytical data and copies of ^{1}H & ^{13}C NMR spectra).

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- *E*-selective 9-vinyl novel unsymmetrical xanthene/thioxanthene synthesis
- Novel quinoline fused xanthene synthesis
- Lewis acid catalyzed cyclization of π-activated allylic alcohols
- High yielding reactions

Graphical Abstract

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