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$Bi(OTf)_3$-catalysed domino Friedel–Crafts alkylation of arenes with aldehydes: an upgraded method for efficient synthesis of triarylmethanes and anthracene derivatives

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ABSTRACT

A variety of novel triarylmethanes including *bis*-(dihexyloxyphenyl)arylmethanes and diveratrylmethanes were prepared by the domino Friedel–Crafts alkylation of arenes with aldehydes catalysed by $Bi(OTf)_3$. The reaction of veratrole with aromatic dialdehydes afforded different results based on the molar ratio of reactants and the nature of aromatic dialdehydes. $Bi(OTf)_3/O_2$ is shown to be a highly efficient reagent system to promote the tandem three-step reaction of acylals with *bis*-(dihexyloxyphenyl)arylmethanes or diveratrylmethanes for the preparation of 9,10-disubstituted-2,3,6,7-tetraalkoxyanthracenes. The substrate scope, the simplicity of the reactions and work-up processes, besides a significant improvement in the yields and reaction times are valuable advantages of these methods, which represent a new and powerful route to multi-substituted anthracenes.

1. Introduction

Triarylmethanes (TRAMs) are of synthetic and pharmacological interest.[1–6] They are useful as dyes, building blocks in dendrimers as well as in electro-optic devices.[5,7–17] Several methods are available for the synthesis of TRAMs. The Friedel–Crafts alkylation of arenes in the presence of Lewis- or Brønsted acids,[7–11,18–20] C–C bond forming reactions catalysed by palladium derivatives,[21–24] Vicarious nucleophilic substitution of hydrogen,[25] Baeyer condensation of aldehydes and *N*,*N*-dimethylaniline by atomized sodium in THF,[26] and benzylation/[3+3] cyclocondensation in the presence of $FeCl_3$,[27] are among the most important of reported methodologies. Recently, some efforts have been also made to study the synthesis and reactions of diveratrylmethanes (DVMs), a new class of triarylmethanes.[28–32]

In recent years, Bi(III) salts have attracted the attention of synthetic organic chemists as efficient catalysts because of their low cost and toxicity, ease of handling and relative insensitivity to air and moisture. $Bi(OTf)_3$ is commercially available or can be easily prepared in laboratory, hence it has become a common place catalyst in organic synthesis.[33–46]

To overcome a part of these problems and in continuing our studies on bismuth-salts as catalyst,[47–52] we describe herein the ready synthesis of triarylmethanes and 9,10-disubstituted-2,3,6,7-tetraalkoxyanthracenes using catalytic amount of $Bi(OTf)_3$.

2. Results and discussion

With the aim of preparing TRAMs, the reaction of 1,2-dihexyloxybenzene **1a** with benzaldehyde **2a**, was chosen as a model and the reaction parameters such as solvent, temperature, amounts of substrates and catalyst were considered (Scheme 1). After careful examinations, the 3:1 molar ratio of 1,2-dihexyloxybenzene to aldehyde in the presence of 8 mol % $Bi(OTf)_3$ at 70 °C under solvent-free conditions was found to be the optimal conditions. Accordingly, treatment of 1,2-dihexyloxybenzene **1a** with benzaldehyde **2a** in the presence of 8 mol % $Bi(OTf)_3$ at 70 °C under solvent-free conditions, resulted in the formation of 4,4′-(phenylmethylene)bis(1,2-bis(hexyloxy)benzene) **3a** in 93% yield. The reaction went to near completion within 15 min, as indicated by TLC monitoring. Following this protocol, other derivatives of *bis*-

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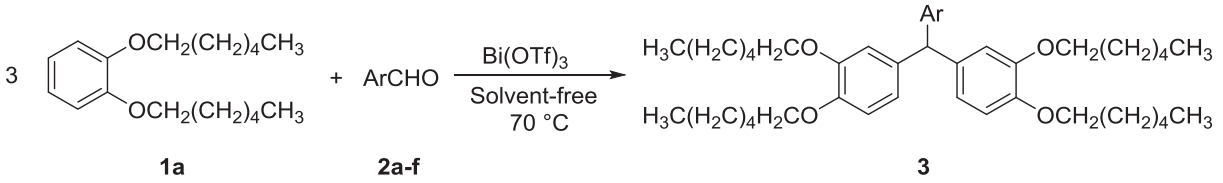
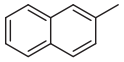


1a **2a-f** **3**

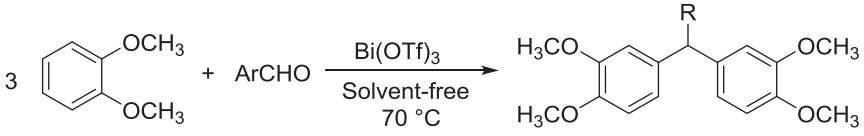
3	Ar	Time (min)	Isolated yield (%)
a	Ph	15	93
b	3-$NO_2C_6H_4$	8	96
c	4-ClC_6H_4	10	95
d	4-BrC_6H_4	10	94
e	4-NCC_6H_4	12	95
f		15	93

Scheme 1. Solvent-free synthesis of *bis*-(dihexyloxyphenyl)arylmethanes.

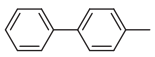
(dihexyloxyphenyl)arylmethanes **3b–f** were synthesized via the domino Friedel–Crafts alkylation of 1,2-dihexyloxybenzene with various aldehydes **2b–f** in excellent yields within 8–15 min (Scheme 1). To the best of our knowledge, there are no reports on the preparation of *bis*-(dihexyloxyphenyl)arylmethanes.

Like 1,2-dihexyloxybenzene, veratrole **1b** also reacted readily with aldehydes **2** in the presence of catalytic amounts of $Bi(OTf)_3$ (8 mol%) to afford the corresponding diveratrylmethanes **3g–f** in 94–96% yields within 10–15 min. Of course, acetaldehyde **2f** was treated to **1b** at room temperature due to its volatility (Scheme 2).

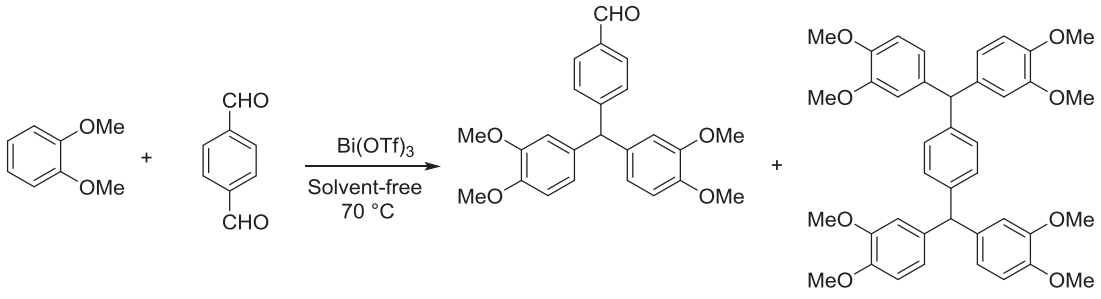
G. A. Olah et al. have shown that alkylation reaction of arenes with terephthalaldehyde **2k** and isophthalaldehyde **2l** in the presence of $BF_3 \cdot H_2O$ gives the corresponding triarylmethanes, while the reaction of arenes by phthalaldehyde **2m** constructs various anthracene derivatives.[53] We were interested in determining whether expanding our protocol on the reaction of aromatic dialdehydes with veratrole obtains the same results. First, terephthalaldehyde was treated with different amounts of veratrole in the presence of 8 mol% $Bi(OTf)_3$ at 70 °C under solvent-free conditions. As illustrated in Scheme 3, when **2k** was reacted with



1b **2c** and **2g-j** **3**

3	R	Time (min)	Isolated yield(%)
g	3-ClC_6H_4	10	95
h	4-ClC_6H_4	10	96
i	3-BrC_6H_4	10	96
j		15	95
k	CH_3	15	$94^{R.T.}$

Scheme 2. Solvent-free synthesis of diveratrylmethanes.



1b (3.0 mmol) **2k** (1 mmol) **Cat.** (8 mol%), 10 min **3l** (93%) **4** (0%)

1b (5.0 mmol) **2k** (1 mmol) **Cat.** (10 mol%), 25 min **3l** (0%) **4** (96%)

Scheme 3. Chemoselectivity in the reaction of veratrole **1b** with terephthalaldehyde **2k** under solvent-free conditions.

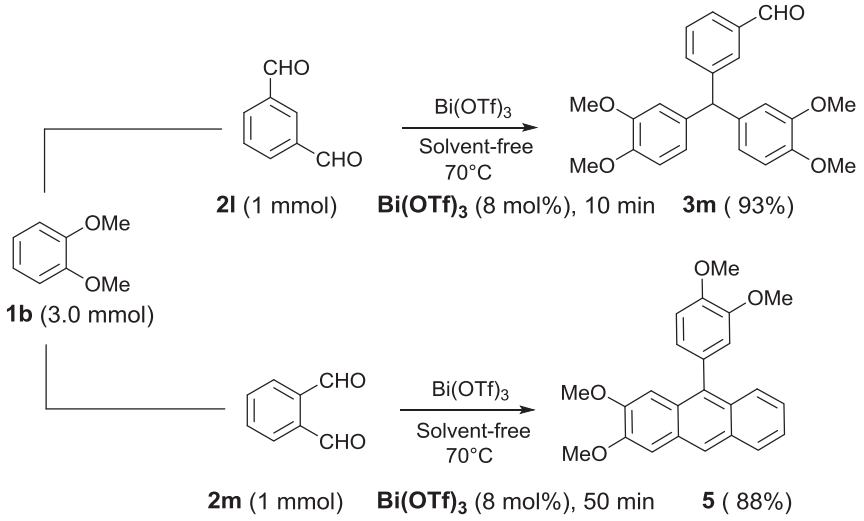
veratrole in a 1:3 molar ratio, only one formyl group selectively contributed and the desired DVM **3l**[28,30–32] was isolated in 93% yield. Using a 1:5 molar ratio of **2k** to veratrole, both formyl groups reacted and adduct **4**[28–32] was produced in 96% yield.

The reaction of isophthalaldehyde **2l** with veratrole in different molar ratios gave diveratrylmethane **3m** as exclusive product. Under optimized conditions compound **3m** obtained in 93% yield within 10 min. Interestingly, the anthracene derivative **5** was isolated as major product via the reaction of phthalaldehyde **2m** with veratrole in the presence of 8 mol % $Bi(OTf)_3$ at 70 °C under solvent-free conditions (Scheme 4). Thus, not only the nature of aromatic dialdehyde but also the molar ratio of aldehyde to veratrole affects on the yield, reaction times and the product of reaction.

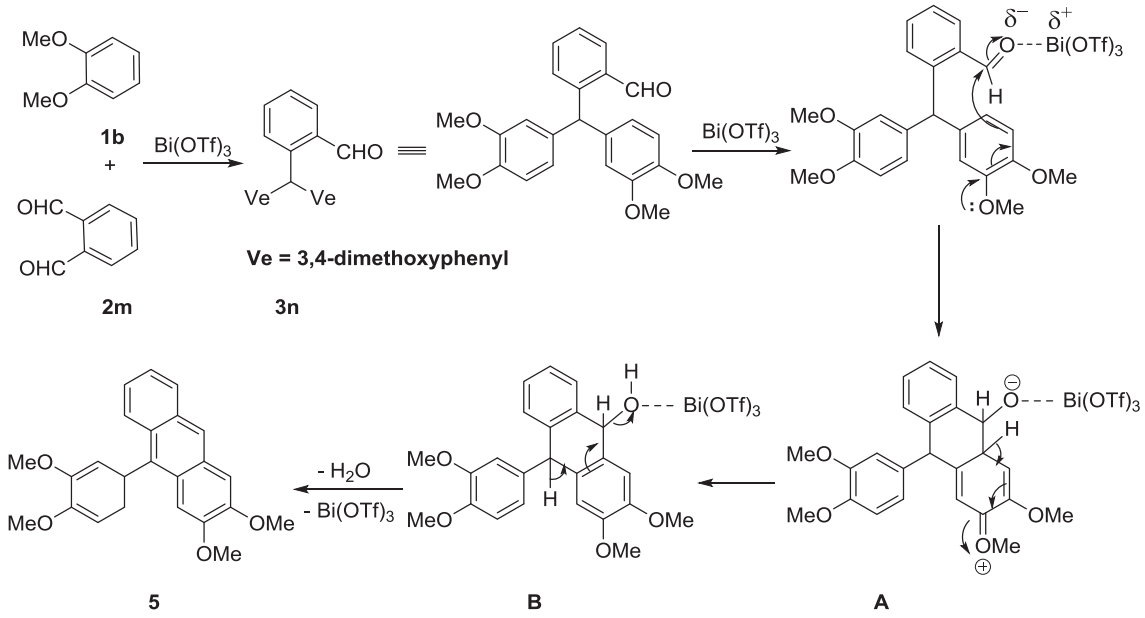
On the basis of the above results, a plausible pathway for the formation of anthracene **5** is proposed (Scheme 5). Initially, phthalaldehyde **2m** reacts with veratrole **1b** in the presence of $Bi(OTf)_3$ to give in situ DVM **3n**. Activation of carbonyl group in **3n** by $Bi(OTf)_3$ followed by intramolecular nucleophilic attack cause to intermediate **A**. The intermediate **A** is then converted to **B** by a prototropic shift. Finally, desired product **5** is produced by dehydration of **B**.

Submission of electron rich arenes **1c** (Scheme 6) and **1d** (Scheme 7) respectively to the reaction with 3-chlorobenzaldehyde **2g** and 3-boromobenzaldehyde **2h** revealed further the generality of this protocol for preparation of TRAMs in excellent yields.

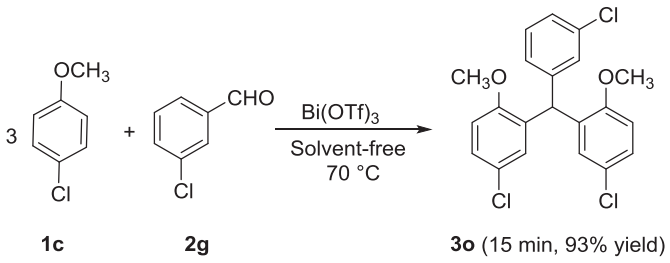
It is noteworthy that these reactions are carried out efficiently without an inert atmosphere. The use of $Bi(OTf)_3$ as catalyst in this protocol is recognized as an advantage, since after completion of reaction it can be recovered quantitatively and then reused in subsequent experiments. For example, the recovered catalyst in the reaction of 1,2-dihexyloxybenzen **1a** with 3-nitrobenzaldeyde **2b** (Scheme 1) was used for further runs and the yields was comparable to that of the first run even after three runs. However, advantages such as simplicity and cleanness, high selectivity,



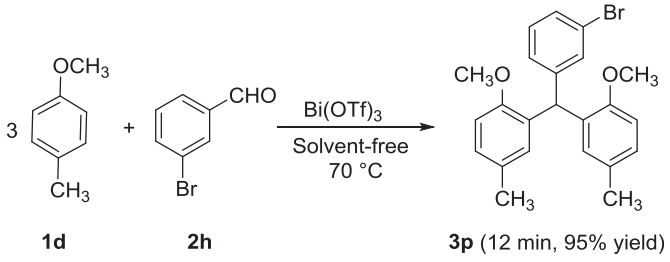
Scheme 4. Chemoselectivity in the reaction of veratrole with isophthalaldehyde **2k** and phthalaldehyde **2l** under solvent-free conditions.



Scheme 5. The plausible pathway for the formation of anthracene **5** through the reaction of phthalaldehyde **2m** with veratrole **1b** catalysed by $Bi(OTf)_3$.



Scheme 6. Synthesis of triarylmethane **3o**.

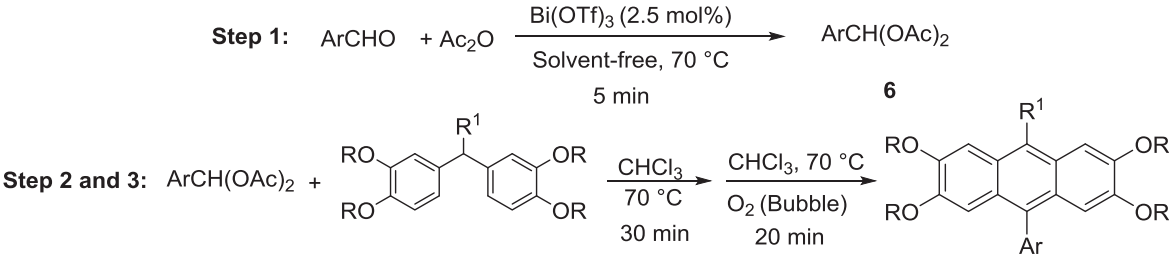


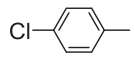
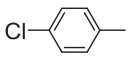
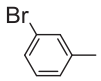
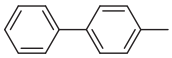
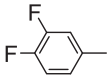
Scheme 7. Synthesis of triarylmethane **3p**.

excellent yields, short reaction times, and extensive substrate scope, nominate this protocol as a powerful and general method for the combinatorial synthesis of triarylmethanes.

Organic compounds including an anthracene unit are amongst polyaromatic hydrocarbons. Since anthracenes reveal unique electronic and photonic properties, they are widely used in material science and synthesis.[54–65] Short-coming such as low yields and limited scope are suggested to circumvent by the use of new synthetic methods.[66–74] DVMs have been identified as synthon for the synthesis of 9,10-diaryl-2,3,6,7-tetramethoxyanthracenes.[28,29] We have also reported a consecutive strategy for the synthesis of various 9,10-diaryl-2,3,6,7-tetramethoxyanthracenes catalysed by $H_3PW_{12}O_{40}$. Despite efficiency of this method in preparation of anthracene derivatives, it is accompany with some crucial challenges such as limitation in substrate scope, low yields, prolonged reaction times, and especially harsh reaction conditions. For example, The use of AcOH (glc.) as solvent, is so problematic because of its hygroscopicity, strongly irritating and corrosive properties.[75] Finding a suitable alternative in place of that could take away a large part of method's complexity. During development of our protocol, we noted that presence of O_2 has effective influence in oxidation/aromatization step of synthesis of anthracenes.[30] In the literature, Bi(III)-salts have proven to be useful catalysts and/or oxidants in synthetic transformations.[76–78] These facts gave us good reasons to believe that $Bi(OTf)_3/O_2$ reagent system would facilitate the synthesis of 9,10-disubstituted-anthracenes. Towards above objectives, we have developed a tandem-three-step procedure for the synthesis of 9,10-disubstituted-anthracenes starting from aldehyde and TRAM promoted by $Bi(OTf)_3/O_2$ system.

Since the efficiency of $Bi(OTf)_3$ as catalyst in the synthesis of acylals has been reported previously,[79] the reactions were performed by first mixing 2 mmol aldehyde **1** and 6 mmol Ac_2O in the presence of 2.5 mol % $Bi(OTf)_3$ at 70 °C to achieve desired acylal **6**. Next, 1 mmol TRAM **3** and 3 mL $CHCl_3$ (as the best choice of solvent) were added immediately and the resulting mixture was stirred at 70 °C for 30 min under ambient air. The reaction mixture was then allowed to further stir for 20 min at 70 °C under oxygen atmosphere (Scheme 8).



7	R	R^1	Ar	Isolated yield (%)
a	$CH_3(CH_2)_4CH_2$	Ph	Ph	79
b	$CH_3(CH_2)_4CH_2$		Ph	58
b	$CH_3(CH_2)_4CH_2$	Ph		71
c	CH_3	CH_3	Ph	87
d	CH_3		Ph	74
e	CH_3		Ph	81
f	CH_3		Ph	69

Scheme 8. General plan for the synthesis of 9,10-disubstituted anthracenes.

As depicted in Scheme 8, a series of novel 9,10-disubstituted anthracenes was produced in 58–87% yields in a single reaction vessel. *Bis*-(dihexyloxyphenyl)arylmethane **3a** in the reaction with acylal **6a** generated 9,10-diaryl-2,3,6,7-tetrahexyloxyanthracene **7a** in 79% yield. In addition, 9-(4-chlorophenyl)-2,3,6,7-tetrakis(hexyloxy)-10-phenylanthracene **7b** was prepared through two different pathways. Although, the reaction of *bis*-(dihexyloxyphenyl)arylmethane **3a** with acylal **6b** proceeded smoothly to furnish anthracene **7b** in 71% yield, this compound was obtained in 58% yield using *bis*-(dihexyloxyphenyl)arylmethane **3c** with acylal **6a** as starting material. These facts confirm that the reactivity of TRAM has a determining role on the progress and the yield of reaction. However, *bis*-(dihexyloxyphenyl)arylmethanes are suitable precursors for this protocol and it discloses truly the route to synthesis a novel class of multi-substituted anthracene derivatives. Yet, to the best of our knowledge, the present protocol to prepare of 9,10-diaryl-2,3,6,7-tetrahexyloxyanthracenes have never been attempted.

To estimate the generality of this procedure, a series of aldehydes **2** and diveratrylmethanes **3** were also submitted to the reaction to prepare other derivatives of anthracene. Once DVM **3k** was treated with acylal **6a**, anthracene **7c** was isolated in 87% yield (Scheme 8). This is the first report of contribution of DVMs bearing an aliphatic group in the synthesis of 9,10-disubstituted-2,3,6,7-tetramethoxyanthracenes. Interestingly, DVMs bearing an aryl group involving 3-Bromophenyl **3i**, biphenyl **3j**, and 3,4-difluorophenyl **3q**, in the reaction with favorite acylals gave the corresponding 9,10-diaryl-2,3,6,7-tetramethoxyanthracenes (**7d**–**f**) in high isolated yields.

Nevertheless, the present method was not compatible with *bis*-(dihexyloxyphenyl)arylmethane derivatives bearing Nitro- or Cyano-groups. Treatment of *bis*-(dihexyloxyphenyl)arylmethanes **3b** with acylal **6a**, whereby the reaction was worked-up after one hour, yielded starting material **3b**. The same experiment with *bis*-(dihexyloxyphenyl)arylmethane **3e** made available a complicated mixture of reaction (Scheme 8).

Of these results, it can be hypothesized that sufficiently stable and reactive *bis*-(dihexyloxyphenyl)arylmethanes or DVMs can be reacted with acylals to obtain the corresponding 9,10-disubstituted-2,3,6,7-tetraalkoxyanthracene derivatives.

The use of $Bi(OTf)_3/O_2$ reagent system in the place of $H_3PW_{12}O_{40}$,[30] to promote the reactions of anthracene synthesis benefits of promising features such as shorter reaction times, higher yields, milder reaction conditions, cleaner reactions, easier work-up, and wider substrate scope.

The structures of products were deduced from their elemental analyses and by IR, mass, ^1H NMR and ^{13}C NMR spectra. Formation of known compounds was confirmed by matching their resulting spectra with cited references. [28–32] Furthermore, the structure of **3h** was confirmed by X-ray crystallographic analysis (CCDC 833544, Fig. 1).

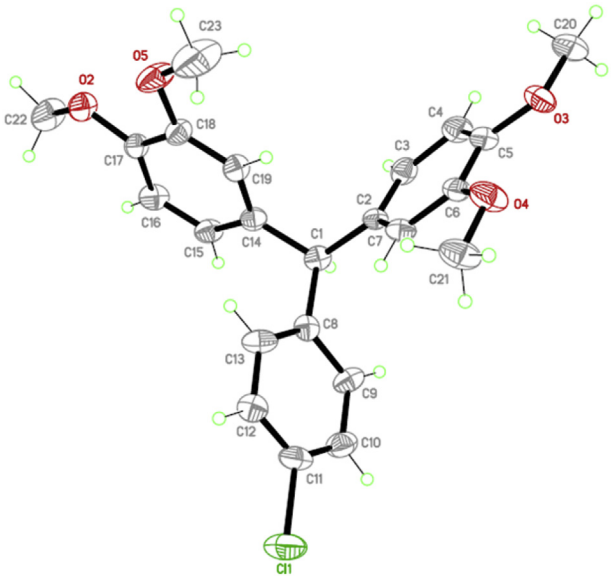


Fig. 1. X-ray crystal structure of **3h**.

3. Conclusion

In summary, a versatile and highly efficient protocol for the synthesis of triarylmethanes including *bis*-(dihexyloxyphenyl) arylmethanes, diveratrylmethanes, etc. was described via the reaction of arenes with aldehydes catalysed by $Bi(OTf)_3$ under solvent-free conditions. Synthesis of *bis*-(dihexyloxyphenyl)arylmethanes is detailed for the first time. The reaction of veratrole with aromatic dialdehydes gave different results based on the molar ratio of reactants and the nature of aromatic dialdehyde. In addition, tandem three-step reaction of acylals with *bis*-(dihexyloxyphenyl)arylmethanes or diveratrylmethanes promoted by $Bi(OTf)_3/O_2$ reagent system was utilized for the synthesis of 9,10-disubstituted-2,3,6,7-tetraalkoxyanthracenes. The present protocols offer advantages such as short reaction times, high yields, clean procedures, simple work-up processes, milder reaction conditions, and broad substrate scope.

4. Experimental part

4.1. General considerations

$Bi(OTf)_3$ was prepared using reported procedures.[80,81] Other chemicals were purchased from commercial sources and used as received. All reactions were magnetically stirred and their progress was monitored by TLC using aluminum sheets precoated with silica gel 60, F 252. Chromatography columns were performed on silica gel 60 (230–400 mesh) using *n*-hexane/ethyl acetate as eluent. ^1H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectra were recorded on a Bruker-AC 500 MHz spectrometer in $CDCl_3$ at 25 °C, using TMS as an internal standard. Melting points are uncorrected and were determined on a Stuart Scientific SMP2 apparatus. IR spectra of products were measure on Nicolet-Impact 400D instrument by transmittance using KBr optics. Micromass Platform II spectrometer was used to record Mass spectra of products. X-ray diffraction data of compound **3h** was recorded on a Rigaku Mercury CCD area detector with graphite monochromated Mo-Ka radiation.

4.2. General procedures

4.2.1. General procedure for solvent-free synthesis of triarylmethanes (Schemes 1–7). To a magnetically stirred mixture of arene **1** (3 mmol) and the corresponding aldehyde **2** (1 mmol) was added $Bi(OTf)_3$ (8 mol %). The resulting mixture was stirred at 70 °C for the appropriate time (Schemes 1–7), and then was allowed to cool to rt. The crude product was diluted twice by addition CH_2Cl_2 (5 mL) and catalyst was separated by simple filtration. The solvent of combined organic layers was evaporated in vacuo and the pure product **3** was obtained by recrystallization from EtOH or $EtOH/H_2O$ (10:2) or by silica-gel column chromatography using *n*-hexane/ethyl acetate (5:1) as eluent. The same procedure were used for synthesis of *tetrakis*(veratryl) adduct **4**.

The recovered catalyst, after washing with CH_2Cl_2, dried at 50 °C and reused successfully at least three successive runs.

*4.2.2. Elemental analyses and spectral data for 1-(bis(3,4-bis(hexyloxy)phenyl)methyl)benzene (**3a**).* Mp 127–128 °C; R_f=0.63 in *n*-hexane/ethyl acetate 5:1; FTIR (KBr): υ_{max}=2930, 2862, 1593, 1509, 1468, 1263, 1134, 1022 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ=7.29 (2H, t, *J*=7.4 Hz, 3,5-H phenyl), 7.02 (1H, t, *J*=7.0 Hz, 4-H phenyl), 7.12 (2H, d, *J*=7.4 Hz, 2,6-H phenyl), 6.79 (2H, d, *J*=8.1 Hz, 6-H 3,4-dihexyloxyphenyl), 6.67 (2H, s, 2-H 3,4-dihexyloxyphenyl), 6.59 (2H, d, *J*=7.6 Hz, 5-H 3,4-dihexyloxyphenyl), 5.41 (1H, s, Ar_3CH), 3.97 (4H, t, *J*=9.5 Hz, OCH_2^1), 3.89 (4H, t, *J*=6.5 Hz, $OCH_2^{1'}$), 1.82 (4H, Q, *J*=7.1 Hz, CH_2^2 of hexyl), 1.75 (4H, Q, *J*=7.1 Hz, $CH_2^{2'}$ of hexyl), 1.46–1.49 (4H, m, CH_2^5 of hexyl), 1.41–1.44 (4H, m, $CH_2^{5'}$ of hexyl), 1.32–1.39 (16H, m, $CH_2^{3,3',4,4'}$ of hexyl), 0.70–1.10 (12H, m, CH_3) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ=148.9, 147.7, 144.6, 137.0, 129.3, 128.2, 126.1, 121.8, 121.1, 115.7, 114.3, 113.7, 69.4, 69.3, 55.9, 31.7, 29.4, 29.3, 25.8, 25.7, 22.6, 22.5, 14.0 ppm; Anal. Calcd for $C_{43}H_{64}O_4$: C, 80.08; H, 10.00. found: C, 80.23; H, 10.01.

4.2.3. General procedure for tandem three-step synthesis of 9,10-disubstituted-2,3,6,7-tetraalkoxyanthracenes (Scheme 8). **Step 1:** To a 50-mL round-bottom flask equipped with a condenser and stirrer bar was added aldehyde **2** (2 mmol), Ac_2O (6 mmol) and $Bi(OTf)_3$ (2.5 mol %). The mixture was reacted at 70 °C for 5 min to obtain the corresponding acylal **6**.

Step 2 and 3: Substrate **3** (1 mmol) and $CHCl_3$ (3 mL) were added, at once. The resulting mixture was stirred at 70 °C for 30 min, and then allowed to further stir for 20 min while O_2 was bubbled in mixture. The progress of reaction was checked by TLC (*n*-Hexane-EtOAc 4:1). The reaction was quenched by saturated $NaHCO_3$ (aq). After extraction by CH_2Cl_2 (3×5 mL), the combined organic layers were dried over Na_2SO_4, concentrated in vacuo and purified by flash chromatography on silica-gel (*n*-hexane/ethyl acetate 10:4) to afford the corresponding 9,10-disubstituted-2,3,6,7-tetralkoxyanthracenes **7**.

*4.2.4. Elemental analyses and spectral data for 9-(4-chlorophenyl)-2,3,6,7-tetrakis(hexyloxy)-10-phenylanthracene (**7b**).* R_f=0.28 in *n*-hexane/ethyl acetate 4:1; FTIR (KBr): υ_{max}=3001, 2954, 1635, 1606, 1529, 1433, 1204, 1111, 752, 700 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ=7.46–7.61 (5H, m, phenyl), 7.44 (2H, d, *J*=11.8 Hz, 2,6-H 4-chlorophenyl), 7.41 (2H, d, *J*=8.2 Hz, 3,5-H 4-chlorophenyl), 6.75 (2H, s, 1,8-H anthracene), 6.80 (2H, s, 4,5-H anthracene), 3.77–3.95 (8H, m, OCH_2 of Hexyl), 1.74 (8H, m, CH_2^2 of Hexyl), 1.39 (8H, Q, *J*=7.2 Hz, CH_2^5 of Hexyl), 1.30 (16H, m, $CH_2^{3,4}$ of Hexyl), 0.89 (12H, t, *J*=6.2 Hz, CH_3 of Hexyl) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ=149.0, 148.7, 133.2, 132.6, 131.1, 129.0, 128.9, 128.7, 128.6, 128.5, 128.3, 127.7, 127.6, 127.3, 126.7, 126.3, 125.8, 123.9, 105.6, 105.1, 68.6, 68.5, 31.7, 31.6, 31.6, 30.4, 29.7, 29.4, 29.3, 29.2, 28.8, 28.8, 25.8, 25.7, 25.7, 22.6, 14.0 ppm; MS (70 eV, EI): *m*/*z* (%)=524.18 ($[M+2]^+$, 13.79), 523.18 ($[M+1]^+$, 62.56), 522.16 ($[M]^+$, 100), 476.09 (37.93); Anal. Calcd for $C_{50}H_{65}ClO_4$: C, 78.45; H, 8.56. found: C, 78.47; H, 8.58.

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Supplementary data

Supplementary data (General experimental procedures, characterization data, and NMR spectra of new compounds along with X-ray crystal structure of **3h** can be found at http://www.sciencedirect.com.) associated with this article can be found in the online version, at http://dx.doi.org/10.1016/j.tet.2016.01.041.

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