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Acid-catalyzed reaction of 1-(2,2-dimethoxyethyl)ureas with phenols as an effective approach to diarylethanes and dibenzoxanthenes

A. V. Smolobochkin,[a] A. S. Gazizov,[a] J. K. Voronina, [b,c] T.S. Rizbayeva,[a] A. R. Burilov,[a] and M. A. Pudovik[a]

[a] *Arbuzov Institute of Organic and Physical Chemistry, FRC Kazan Scientific Center, Russian Academy of Sciences, Arbuzova str., 8, Kazan, Russian Federation*

[b] *N.S. Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences, 31 Leninsky Ave., Moscow, Russian Federation*

[c] *A. N. Nesmeyanov Institute of Organoelement Compounds of Russian Academy of Sciences, Vavilova Str. 28, 119991 Moscow, Russian Federation*

Email: smolobochkin@iopc.ru

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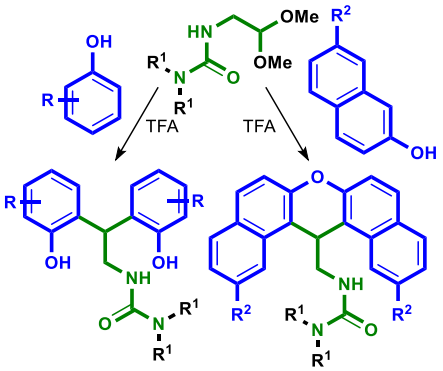
Abstract

A one-pot synthesis of urea-substituted diarylethanes and dibenzoxanthenes starting from 1-(2,2-dimethoxyethyl)ureas and phenols has been developed. The approach uses readily available reagents and catalysts, requires mild reaction conditions and provides the target compounds in good to high yields.



Keywords: Phenols, dibenzoxanthenes, diarylethanes, acetals

Introduction

Diarylethanes containing urea fragments are known for their pharmacological activity. According to the literature, these compounds exhibit antibacterial activity,[1–3] may act as inhibitors of lysine-specific histone demethylase 1 (LSD1)[4] and antagonists of muscarinic acetylcholine[5] and chemokine type 3 receptors (CCR3)[6]. Additionally, some of them can be used in malaria[7] and sclerosis[8] treatment (Figure 1, A). The main synthetic approach to urea-containing diarylethanes is based on the reaction of amines with isocyanates (Figure 1, B). While from a technical point of view the approach is simple, it suffers from hard to access starting amines and/or isocyanates. As a result, varying the target urea substituents is a complex and cumbersome task.

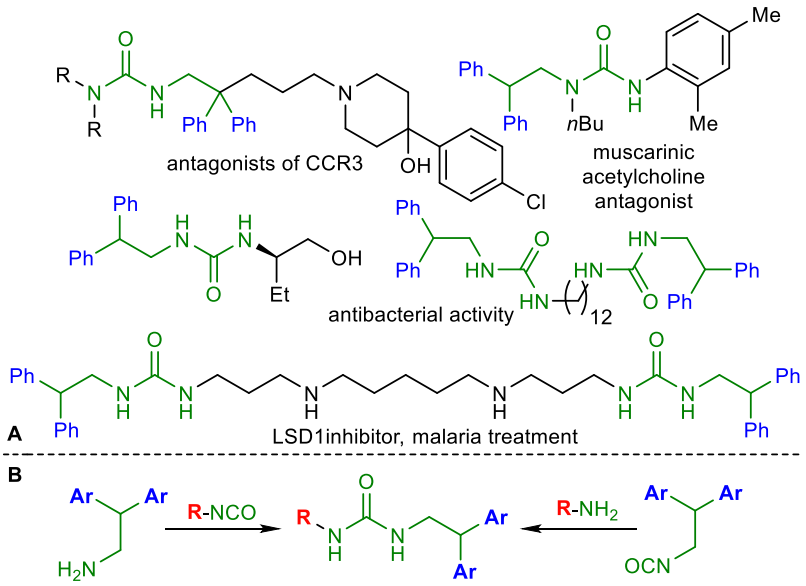


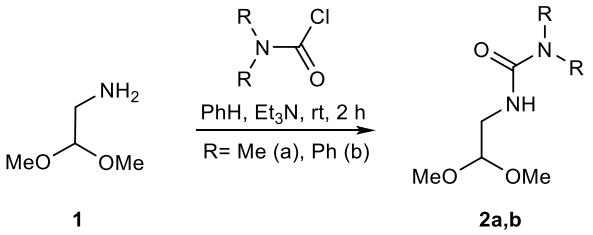
Figure 1. A) Diarylethane-based ureas of biological importance. B) Synthetic approach to urea-containing diarylethanes.

At the same time, the chemistry of nitrogen-containing acetals is gaining increased interest, as indicated by a number of recent review papers.[9–12] The main reason for this, is the presence of both nucleophilic nitrogen and electrophilic α-carbon atoms leading to a diverse array of potential reactions. Moreover, nitrogen-containing acetals serve as valuable reagents for the synthesis of various biologically active compounds, both natural and synthetic. For example, *Erythroxylon coca* alkaloids were obtained with high enantioselectivity via acid-catalyzed cyclization of 2,2'-{[1,9-di(1,3-dioxan-2-yl)nonane-3,7-diyl]bis(azanediyl)}bis(2-phenylethan-1-ol).[13] 1-[2-(1,3-Dioxolan-2-yl)ethyl]-6,7-dimethoxy-1,2,3,4-tetrahydro-isoquinoline was used as starting compound in the synthesis of *S*-(-)-crispin A.[14,15] Similarly, *N*-(2,2-methoxyethyl)-1*H*-indole-2-carboxamide was employed in the synthesis of carbolines.[16] Kuehne et al.[17] described a synthesis of 18-methoxycoronaridine from methyl 5-[1-(1,3-dioxolan-2-yl)-3-methoxypropyl]-1,2,3,4,5,6,7,8-octahydroazonino[5,4-*b*]indole-7-carboxylate. 2,2-Dimethoxyethan-1-amine is used in one stage of the synthesis of the anthelmintic drug praziquantel.[18] Recently, we successfully used N-substituted 4,4-diethoxybutane-1-amines to prepare pyrazolyl-substituted nicotine analogues.[19]

Herein, we describe results obtained during our ongoing research of nitrogen-containing acetal chemistry and report the employment of 2,2-dimethoxyethane-1-amines in the synthesis of urea-containing diarylethanes and dibenzoxanthenes.

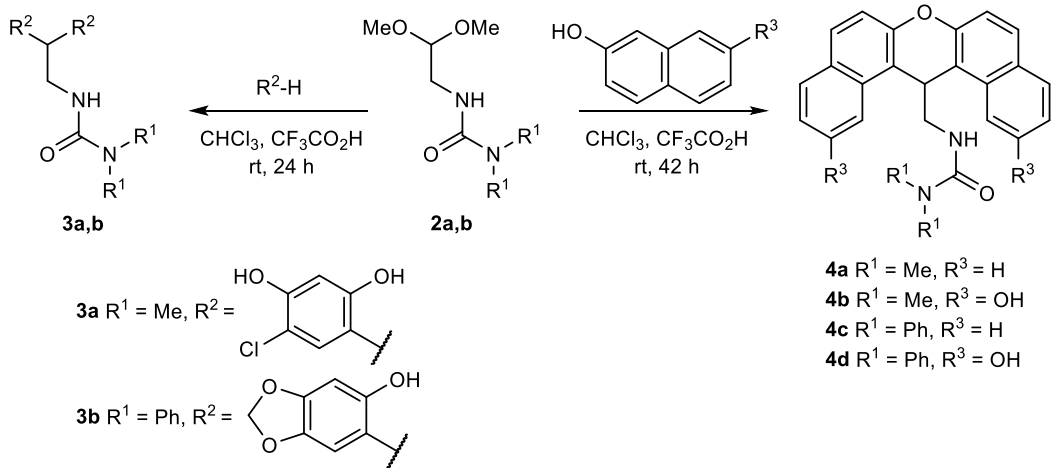
Results and Discussion

Previously, we reported a synthesis of imidazolidin-2-ones via acid-catalyzed reaction of 1-(2,2-dimethoxyethyl)-3-arylurea with resorcinol and its derivatives.[20–22] According to the proposed mechanism, the key factor affecting a new C-N bond formation and intramolecular heterocyclic ring closure is a presence of the NH group in the molecule of the starting urea. We speculated that a presence of a second substituent at the nitrogen atom will prevent intramolecular cyclization of 1-(2,2-dimethoxyethyl)ureas and a reaction will result in a formation of acyclic diarylethanes. Not only would this allow a one-step synthesis of target compounds, but also the easy availability of starting compounds would be advantageous. With this in mind, we obtained starting acetals **2** via the reaction of 2,2-dimethoxyethane-1-amine **1** with 1,1-disubstituted carbamoyl chlorides (Scheme 1). Both *N*,*N*-dialkyl- and *N*,*N*-diaryl-substituted ureas were obtained to check whether the nature of a substituent affects a reaction coarse.



Scheme 1. Synthesis of acetals **2a,b**.

Next, we carried out the reaction of the synthesized acetals **2a,b** with 4-chlororesorcinol and sesamol. The reaction proceeded smoothly at room temperature in the presence of trifluoroacetic acid and resulted in the diarylethanes **3a,b** with fairly good yield (Table 1, entries 1 and 2). Earlier, we showed that reactions of nitrogen-containing acetals can also be used to prepare dibenzoxanthenes.[23–25] Thus, 2-naphthol and 2,7-naphthalenediol were used in the reaction with acetals **2a,b** in the presence of an excess of trifluoroacetic acid (Scheme 2). As a result, dibenzoxanthenes **4a-d** were obtained in good yields (Table 1, entries 3-6). Notably, no influence of substituents at the nitrogen atom on target compound yields was detected (cf. Table 1, entries 3,5 and 4,6).



Scheme 2. Synthesis of diarylethanes **3** and dibenzoxanthenes **4**.

Table 1. Synthesis of diarylethanes **3a,b** and dibenzoxanthenes **4a-d**

№	Acetal	R^1	R^2	R^3	Product	Yield (%)[a]
1	**2a**	Me	5-chloro-2,4-dihydroxyphenyl	-	**3a**	66
2	**2b**	Ph	6-hydroxybenzo[*d*][1,3]dioxol-5-yl	-	**3b**	88
3	**2a**	Me	-	H	**4a**	87
4	**2a**	Me	-	OH	**4b**	70
5	**2b**	Ph	-	H	**4c**	79
6	**2b**	Ph	-	OH	**4d**	68

[a] Isolated yield.

The bond lengths, valence and torsion angles taken from a single crystal x-ray structure of compound **4c** were in the ranges typical for every bond type as proved by X-ray analysis. The conformation of molecule **4c** is folded and stabilized by CH(Ph)…π and π…π interactions (Figure 1).

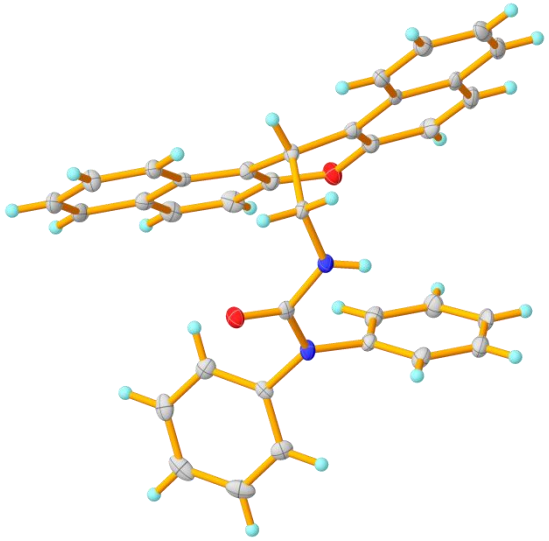


Figure 1 Molecular structure of compound **4c** in the crystalline state. Ellipsoids are shown with 50% probability.

The crystal packing of compound **4c** involves a large number of non-covalent interactions. Infinite chains are formed by the hydrogen bonds with additional stabilization of Lp…π interactions. Folded layers are formed perpendicular to them due to stacking and CH…π interactions (Figure 2).

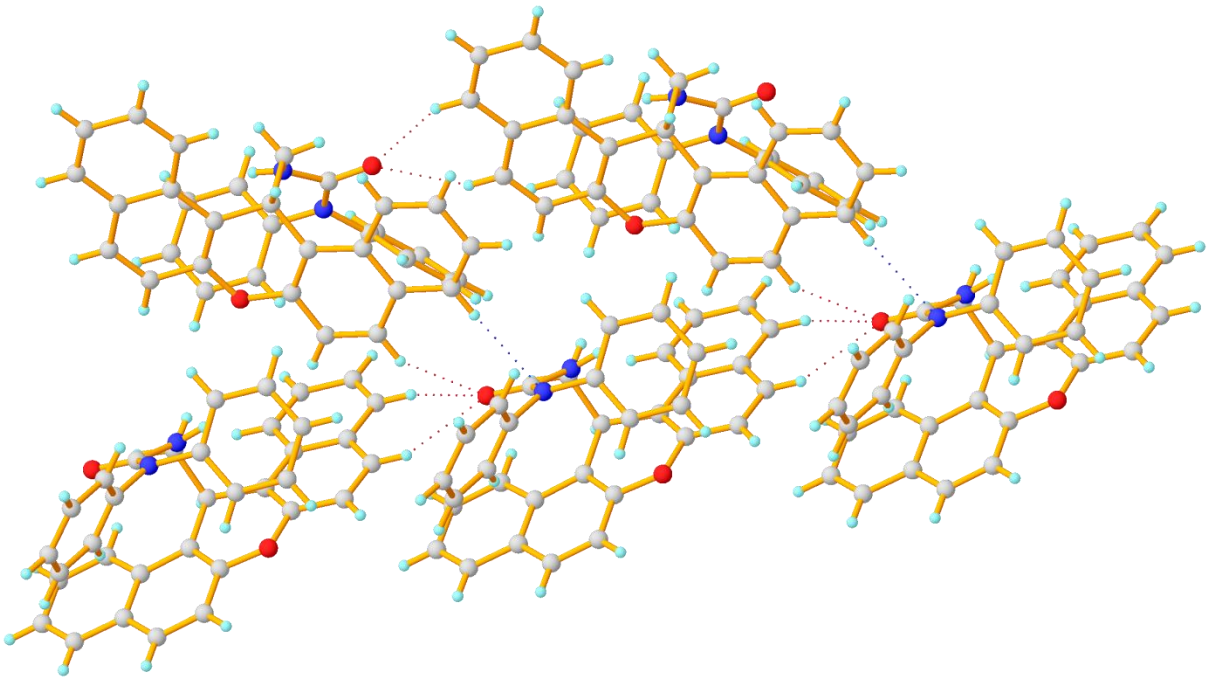
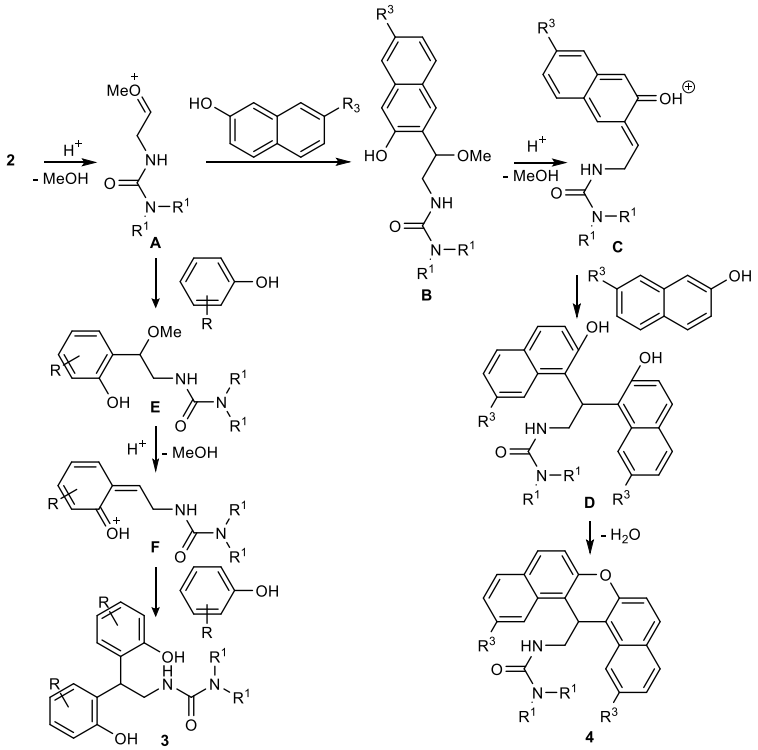


Figure 2 Fragment of H-bonded chain in crystal packing of compound **4c**.

The proposed reaction mechanism is similar to that previously described for formation of polyphenols from *N*-(4,4-diethoxybutyl)sulfonylamides (Scheme 3).[24] The key step of the reaction is the elimination of methanol from acetal **2**, which affords oxonium ion **A**. The next stage is the interaction of this species with phenolic nucleophile. Depending on the phenol used, the reaction can lead either to dibenzoxanthenes **4** or diarylethanes **3**. The use of naphthol leads to an intermediate compound **B**; and subsequent stages result in dibenzoxanthenes **4** according to the previously described mechanism.[24] When 4-bromoresorcinol or sesamol are used as nucleophiles, diarylethanes **3** are formed through the intermediate **E** and benzylic cation **F**.



Scheme 3. Proposed mechanism for the substituted ureas **3,4** formation.

Conclusions

In conclusion, we have developed a convenient one-pot protocol for the synthesis of otherwise hard to access diarylethanes and dibenzoxanthenes via metal-free acid-catalyzed reaction of phenols with 1-(2,2-dimethoxyethyl)ureas.

Experimental Section

General. ^{1}H NMR spectra were recorded on Bruker MSL 400 spectrometer (working frequency 400.13 MHz) in $CDCl_3$ and $(CD_3)_2SO$ relative to the residual solvent protons. ^{13}C NMR spectra were recorded on Bruker Avance 600 spectrometer (working frequency 150.90 MHz). The MALDI-TOF mass spectra were recorded on a Bruker ULTRAFLEX III TOF/TOF instrument (with 2,5-dihydroxybenzoic acid matrix). IR spectra were obtained with a Bruker Vector 22 spectrometer. Elemental analysis was performed on Carlo Erba EA 1108 instrument. Schöniger method was applied for chlorine determination.[28] Melting points were determined in glass capillaries with a Stuart SMP 10 apparatus. Single-crystal X-ray diffraction analysis was performed at 295(2) K on Smart Apex II automatic diffractometer using graphite monochromated radiation. All solvents were purified and dried according to standard procedures.

The X-ray diffraction data for the crystal of **4c** were collected on a Smart Apex II automatic diffractometer using graphite monochromated radiation. The structures were solved by direct methods and refined by full-matrix least-squares using the SHELXL97[26] program. All the non-hydrogen atoms were refined with anisotropic atomic displacement parameters. All figures were made using the program OLEX2[27]. Crystallographic data for

the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Center (deposit number is 1950456).

General method for the synthesis of ureas 2a,b. To a solution of 2,2-dimethoxyethan-1-amine (0.90 g, 8.60 mmol) and triethylamine (1.74 g, 17.21 mmol) in benzene (15 mL) was added dropwise with cooling (5-7 °C), carbamoyl chloride (8.60 mmol). The reaction mixture was stirred under cooling for 2 hours. The precipitate was filtered off, the filtrate was evaporated in vacuum to afford the oily product.

3-(2,2-Dimethoxyethyl)-1,1-dimethylurea (2a). Yellow oil, yield 1.35 g, 89%. ^{1}H NMR ($CDCl_3$): δ 2.78 (s, 3H, CH_3), 3.22 (t, *J* 5.5 Hz, 2H, CH_2), 3.27 (s, 6H, CH_3), 4.27 (t, *J* 5.3 Hz, 1H, CH), 4.69 (s, 1H, NH). ^{13}C NMR ($CDCl_3$): δ 36.1, 42.4, 54.3, 103.4, 158.3. MALDI-TOF: *m/z* 199 $[M+Na]^+$.

3-(2,2-Dimethoxyethyl)-1,1-diphenylurea (2b). Yellow oil, yield 2.35 g, 91%. ^{1}H NMR ($CDCl_3$): δ 3.39 (s, 6H, CH_3), 4.43 (t, *J* 5.4 Hz, 1H, CH), 4.81 (s, 1H, NH), 7.25–7.21 (m, 2H, ArH), 7.39–7.27 (m, 4H, ArH), 7.35-7.37 (m, 4H, ArH). ^{13}C NMR ($CDCl_3$): δ 42.4, 54.6, 103.2, 126.2, 127.4, 129.4, 142.8, 156.2. MALDI-TOF: *m/z* 323 $[M+Na]^+$.

General method for the synthesis of diaryletanes 3a,b. To a mixture of phenol (6.94 mmol), chloroform (10 mL), and acetal **2** (3.47 mmol), was added trifluoroacetic acid (2 mL). The reaction mixture was stirred for 24 hours at room temperature, the solvent was removed in vacuum, the residue was washed with diethyl ether and dried in vacuum.

3-[2,2-Bis(5-chloro-2,4-dihydroxyphenyl)ethyl]-1,1-dimethylurea (3a). Colorless solid. Yield 0.92 g, 66%, m.p. 157-158 °C. IR (KBr, □/cm^{-1}): 1596, 1627, 2967, 3089, 3066, 3283. ^{1}H NMR (DMSO-d_6): δ 2.70 (s, 6H, CH_3), 3.41-3.52 (m, 2H, CH_2), 4.48 (t, *J* 7.5 Hz, 1H, CH), 6.19 (s, 1H, NH), 6.45 (s, 2H, ArH), 6.87 (s, 2H, ArH), 9.49 (s, 2H, OH), 9.72 (s, 2H, OH). ^{13}C NMR (DMSO-d_6): δ 15.6, 36.2, 65.3, 104.3, 109.4, 121.54, 129.1, 152.0, 155.2, 159.0. Found (%): C, 51.02; H, 4.75; Cl, 17.59; N, 7.09. Calc. for $C_{17}H_{18}Cl_2N_2O_5$ (%): C, 50.89; H, 4.52; Cl, 17.47; N, 6.98. MALDI-TOF: *m/z* 423 $[M+Na]^+$.

3-[2,2-Bis(6-hydroxybenzo[*d*][1,3]dioxol-5-yl)ethyl]-1,1-diphenylurea (3b). Colorless solid. Yield 1.56 g, 88%, m.p. 229-230 °C. IR (KBr, □/cm^{-1}): 1596, 1626, 2975, 3058, 3251. ^{1}H NMR (DMSO-d_6): δ 3.43-3.57 (m, 2H, CH_2), 4.68 (t, *J* 8.1 Hz, 1H, CH), 5.42 (t, *J* 5.5 Hz, 1H, NH), 5.87 (d, *J* 10.2 Hz, 4H, CH_2), 6.44 (s, 2H, ArH), 6.67 (s, 2H, ArH), 7.00 (d, *J* 7.9 Hz, 4H, ArH), 7.15 (t, *J* 7.5 Hz, 2H, ArH), 7.26 (t, *J* 7.7 Hz, 4H, ArH), 9.01 (s, 1H, OH). ^{13}C NMR (DMSO-d_6): δ 37.5, 43.5, 98.1, 100.9, 108.6, 120.7, 125.9, 127.5, 129.5, 140.1, 143.5, 146.0, 150.1, 156.0. Found (%): C, 68.15; H, 4.90; N, 5.21. Calc. for $C_{29}H_{24}N_2O_7$ (%): C, 67.96; H, 4.72; N, 5.47. MALDI-TOF: *m/z* 513 $[M+H]^+$, 535 $[M+Na]^+$, 551 $[M+K]^+$.

General method for the synthesis of dibenzoxantenes 4a-c. To a mixture of naphthol (2.95 mmol), chloroform (5 mL), and acetal **2** (1.47 mmol), was added trifluoroacetic acid (2 mL). The reaction mixture was stirred for 24 hours at room temperature, the solvent was removed in vacuum, the residue was washed with diethyl ether and dried in vacuum.

3-[(14*H*-dibenzo[*a,j*]xanthen-14-yl)methyl]-1,1-dimethylurea (4a). Colorless solid. Yield 0.49 g, 87%, m.p. 250-251 °C. IR (KBr, □/cm^{-1}): 1593, 1676, 2975, 3013, 3374. ^{1}H NMR (DMSO-d_6): δ 2.57 (s, 6H, CH_3), 3.27 (t, *J* 6.2 Hz, 2H, CH_2), 5.79 (t, *J* 6.8 Hz, 1H, CH), 6.28 (t, *J* 5.5 Hz, 1H, NH), 7.47 (d, *J* 8.9 Hz, 2H, ArH), 7.49 (t, *J* 7.0 Hz, 2H, ArH), 7.61 (t, *J* 7.1 Hz, 2H, ArH), 7.91 (d, *J* 8.9 Hz, 2H, ArH), 7.94 (d, *J* 7.9 Hz, 2H, ArH), 8.51 (d, *J* 8.5 Hz, 2H, ArH). ^{13}C NMR (DMSO-d_6): δ 30.9, 36.2, 47.6, 117.2, 117.7, 123.5, 124.8, 127.0, 128.8, 129.0, 131.0, 132.5, 150.1, 158.9. Found (%): C, 78.63; H, 5.94; N, 7.50. Calc. for $C_{25}H_{22}N_2O_2$ (%): C, 78.51; H, 5.80; N, 7.32. MALDI-TOF: *m/z* 382 $[M+H]^+$.

3-[(2,12-Dihydroxy-14*H*-dibenzo[*a,j*]xanthen-14-yl)methyl]-1,1-dimethylurea (4b). Colorless solid. Yield 0.43 g, 70%, m.p. >250 °C. IR (KBr, ν/cm^{-1}): 1595, 1680, 2875, 3085, 3319. ^{1}H NMR (DMSO-d_6): δ 2.58 (s, 6H, CH_3), 3.23 (t, *J* 6.1 Hz, 2H, CH_2), 5.40 (t, *J* 6.7 Hz, 1H, CH), 6.13 (t, *J* 5.6 Hz, 1H, NH), 7.07 (dd, *J* 8.7, 2.2 Hz, 2H, ArH), 7.18 (d, *J* 8.8 Hz, 2H, ArH), 7.69 (d, *J* 1.9 Hz, 2H, ArH), 7.73 (d, *J* 8.9 Hz, 2H, ArH), 7.76 (d, *J* 8.9 Hz, 2H, ArH). ^{13}C NMR (DMSO-d_6): δ 31.3, 36.2, 46.8, 105.7, 114.3, 115.9, 117.0, 125.5, 128.6, 130.3, 134.4, 150.6, 156.8, 158.9. Found (%): C, 72.57; H, 5.49; N, 6.52. Calc. for $C_{25}H_{22}N_2O_4$ (%): C, 72.45; H, 5.35; N, 6.76. MALDI-TOF: *m/z* 415 $[M+H]^+$, 437 $[M+Na]^+$.

3-[(14*H*-Dibenzo[*a,j*]xanthen-14-yl)methyl]-1,1-diphenylurea (4c). Colorless solid. Yield 0.59 g, 79%, m.p. >250 °C. IR (KBr, ν/cm^{-1}): 1591, 1676, 2921, 3059, 3414. ^{1}H NMR (DMSO-d_6): δ 3.40 (t, *J* 6.0 Hz, 2H, CH_2), 5.74 (t, *J* 5.6 Hz, 1H, CH), 5.81 (t, *J* 6.4 Hz, 1H, NH), 6.79 (d, *J* 7.5 Hz, 4H, ArH), 7.13 (t, *J* 7.4 Hz, 2H, ArH), 7.23 (t, *J* 7.8 Hz, 4H, ArH), 7.44 (d, *J* 8.8 Hz, 2H, ArH), 7.51 (t, *J* 7.3 Hz, 2H, ArH), 7.66 (t, *J* 7.2 Hz, 2H, ArH), 7.90 (d, *J* 8.8 Hz, 2H, ArH), 7.97 (d, *J* 8.0 Hz, 2H, ArH), 8.57 (d, *J* 8.5 Hz, 2H, ArH). ^{13}C NMR (DMSO-d_6): δ 46.6, 116.6, 117.8, 123.5, 124.9, 126.2, 127.3, 128.0, 129.0, 129.1, 129.5, 131.0, 132.3, 143.2, 150.2, 156.1. Found (%): C, 83.11; H, 4.99; N, 5.74. Calc. for $C_{35}H_{26}N_2O_2$ (%): C, 82.98; H, 5.17; N, 5.53. MALDI-TOF: *m/z* 507 $[M+H]^+$, 529 $[M+Na]^+$, 545 $[M+K]^+$.

3-[(2,12-Dihydroxy-14*H*-dibenzo[*a,j*]xanthen-14-yl)methyl]-1,1-diphenylurea (4d). Colorless solid. Yield 0.54 g, 68%, m.p. 222 °C. IR (KBr, ν/cm^{-1}): 1593, 1675, 2986, 3078, 3446. ^{1}H NMR (DMSO-d_6): δ 3.21–3.35 (m, 2H, CH_2), 5.42 (t, *J* 6.0 Hz, 1H, CH), 5.49 (t, *J* 5.7 Hz, 1H, NH), 6.94 (d, *J* 7.3 Hz, 4H, ArH), 7.13–7.08 (m, 4H, ArH), 7.16 (d, *J* 9.0 Hz, 2H, ArH), 7.21 (t, *J* 7.5 Hz, 4H, ArH), 7.76–7.70 (m, 4H, ArH), 7.80 (d, *J* 8.8 Hz, 2H, ArH). ^{13}C NMR (DMSO-d_6): δ 31.4, 45.9, 105.6, 114.3, 115.1, 117.1, 125.5, 126.3, 128.3, 128.7, 129.4, 130.5, 134.0, 143.3, 150.6, 156.2, 157.0. Found (%): C, 77.84; H, 4.99; N, 5.34. Calc. for $C_{35}H_{26}N_2O_4$ (%): C, 78.05; H, 4.87; N, 5.20. MALDI-TOF: *m/z* 539 $[M+H]^+$, 561 $[M+Na]^+$, 577 $[M+K]^+$.

Acknowledgements

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Supplementary Material

Copies of ^{1}H and ^{13}C NMR spectra of all products and details of the crystallographic studies can be found at Supporting Information.

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