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Note

A novel coupling reaction of 3-substituted 4-alkoxy and 4-aminocyclobutene-1,2-diones induced by $TiCl_4$–Zn

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Abstract

The coupling of 3-substituted 4-alkoxy or 4-aminocyclobutene-1,2-diones induced by $TiCl_4$–Zn was studied. It is interesting to find that the double bonds involved in the coupling reaction and the unsymmetrical coupling compounds were obtained as major or sole products. A possible mechanism mediated by titanium coordination intermediates is proposed. © 2001 Published by Elsevier Science B.V.

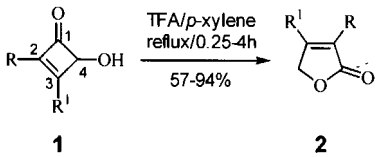
Keywords: Reductive coupling; $TiCl_4$–Zn reagent; 4-Substituted cyclobutene-1,2-diones

1. Introduction

In our previous research, a general and efficient route for the preparation of 3,4-disubstituted 2(5*H*)-furanone (**2**) was developed by a trifluoroacetic acid catalyzed ring transformation of 4-hydroxycyclobutenone (**1**) (Scheme 1) [1]. This result prompted us to explore the conversion of 4,4′-bi(2,3-disubstituted 4-hydroxycyclobutenone) (**4**) to the corresponding (2,2′-bifuran)-5,5′(2*H*,2*H*′)-dione (**5**) (Scheme 2). Since reductive coupling of carbonyl groups induced by low-valent titanium usually leads to 1,2-diols under mild conditions [2], it was employed in our strategy for the preparation of the key intermediate **4** from 3,4-disubstituted cyclobutene-1,2-dione (**3**).

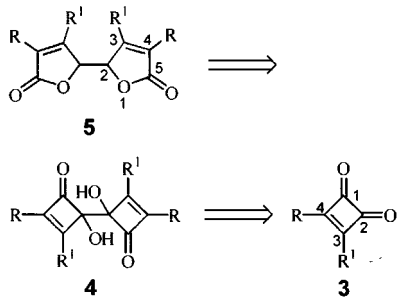
2. Results and discussion

Following the known procedure, 3-isopropoxy-4-phenylcyclobutene-1,2-dione (**3a**) was treated with $TiCl_4$–Zn reagent for 2 h at room temperature. After normal workup, two white crystalline products were separated by chromatography. Their MS spectra (FAB, $m/z = 434$) and elemental analyses are consistent with formulations as coupling products of **3a**. However, their ^{1}H-NMR spectra showed clearly different fea-



1 **2**

Scheme 1.



5 **4** **3**

Scheme 2.

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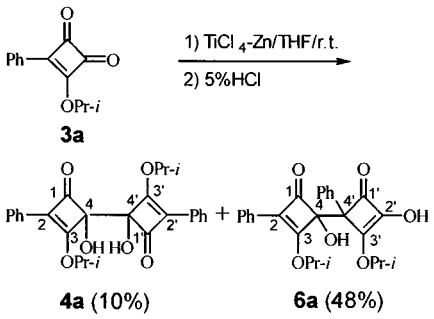


Scheme 3.

tures. The minor product (10%) was assigned as a symmetric coupling product **4a** with equivalent isopropyl and phenyl groups. The major product (48%) exhibited resonance for two isopropyl and two phenyl groups (Scheme 3). Both products were characterized by single-crystal X-ray diffraction analysis. Compound **4a** was confirmed to be the symmetric isomer shown in Fig. 1 and compound **6a** was confirmed to be its unsymmetric isomer shown in Fig. 2.

By altering the conditions, it was found that the yields of **4a** and **6a** were strongly influenced by the reaction temperature. As shown in Table 1, the best total yield (77%) was obtained for the reaction at -10°C. The unsymmetrical isomer **6a** is always the major product. When the ratio of **3a**/$TiCl_4$/Zn = 1:2:4 was increased to 1:4:8, there is no change in the yields or the ratio of **4a**/**6a**.

When the 3-substituted 4-alkoxycyclobutene-1,2-diones **3b–d** were employed, the corresponding products **4b** and **c** and **6b–d** were obtained. The yields and the ratio of products **4** and **6** depend on the substituents on C4 (Scheme 4, Table 2).

To explore the scope of this coupling reaction further, the series of 3-substituted 4-aminocyclobutene-1,2-dione **3e–i** was subjected to treatment with $TiCl_4$–Zn under the same conditions. In this case, the unsymmetrical products **6e–i** were obtained in 38–53% yields as the only products.

In most cases for α,β-conjugated carbonyl compounds, reductive coupling reactions induced by $TiCl_4$–Zn usually give only carbonyl coupling product [2d–g,3a–e]. However, a few reports of 'abnormal' coupling are also available [3c,4]. The result herein is a new example of 'abnormal' coupling of α,β-conjugated carbonyl compounds. To explain the regiochemistry of products **4** and **6**, a possible mechanism mediated by titanium coordination intermediates is proposed (as shown in Scheme 5). In the first step, an electron is transferred from titanium to the carbonyl group of compound **3** generating a radical anion **7**. It dimerizes to yield **8** and the latter is then hydrolyzed to the

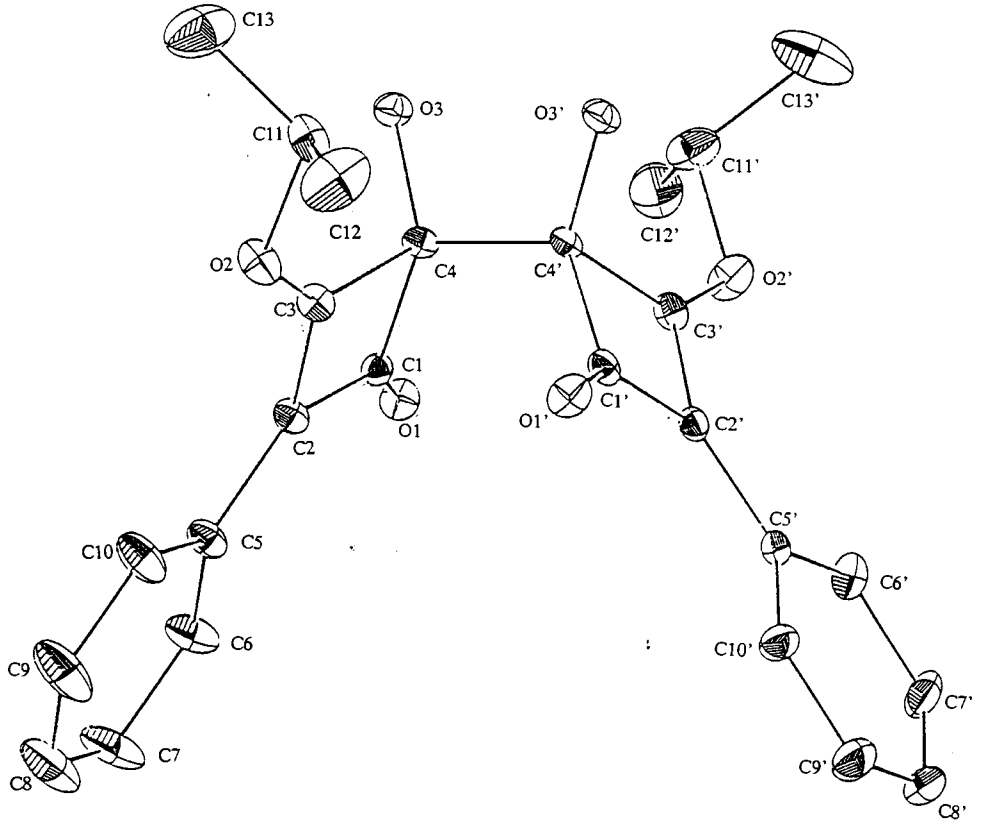


Fig. 1. Structure of 2,2′-diphenyl-3,3′-diisopropoxy-4,4′-dihydroxy-4,4′-bicyclobutenone (**4a**).

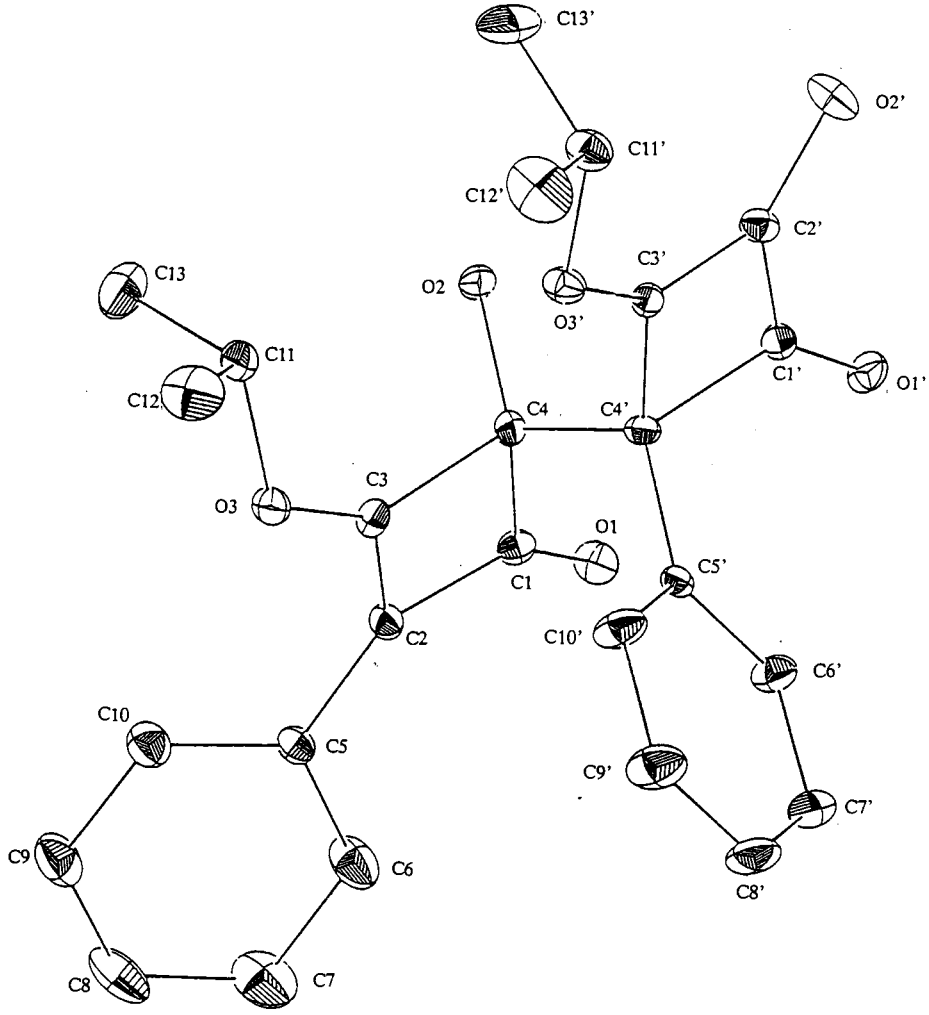


Fig. 2. Structure of 2,4′-diphenyl-4,2′-dihydroxy-3,3′-diisopropoxy-4,4′-bicyclobutenone (**6a**).

syn-diol **4**. However, when radical anion **7** attacks the double bond of compound **3**, a new radical **9** is generated. This accepts another electron from titanium to form a stable intermediate **10**, which is then hydrolyzed to give the unsymmetrical coupling product **6**.

3. Experimental

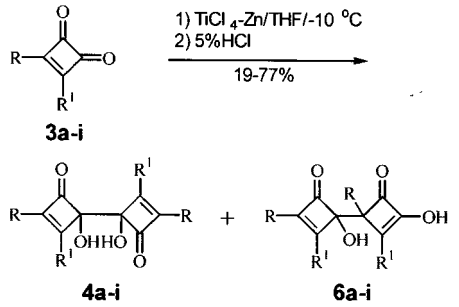
All melting points were determined on a Yanaco melting point apparatus and are uncorrected. IR spectra were recorded on a Nicolet FTIR 5DX spectrometer with KBr pellets. ^{1}H-NMR spectra were recorded on a Bruker MD500 spectrometer in $CDCl_3$ with Me_4Si as internal reference. The *J* values are given in Hz. MS spectra were obtained on a FAB-HS mass spectrometer at 70 eV. Elemental analyses were performed on a Perkin–Elmer 240C instrument. 3-Sustituted 4-alkoxycyclobutene-1,2-dione (**3a**–**d**) and 3-substituted 4-aminocyclobutene-1,2-dione (**3e**–**i**) were prepared by known procedures [1]. PE is petroleum ether (60–90°C).

3.1. A general procedure for the reductive coupling of 3,4-disubstituted cyclobuten-1,2-ones (3)

$TiCl_4$ (2.2 ml, 20 mmol) was added slowly by using a syringe (under nitrogen atmosphere) to a stirred suspension of zinc powder (2.6 g, 40 mmol) in anhydrous THF (30 ml). The resultant mixture was then refluxed for 2 h. After cooling to −10°C, a solution of **3** (10 mmol) in THF (15 ml) was added slowly using a syringe. The solution was then stirred for 0.5–2.5 h at the same temperature (monitored by TLC). The reaction was quenched by the addition of 5% aq. HCl. The

Table 1
Effect of temperature on the yields of **4a** and **6a**

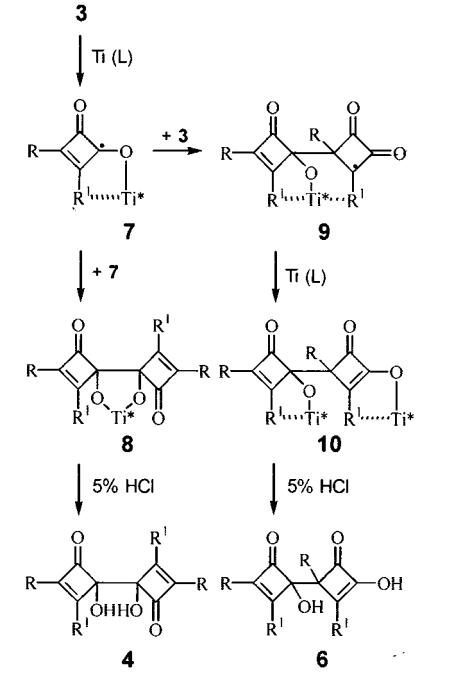
Temp (°C)	65	25	−10	−45
4a (%)	0	10	15	NR
6a (%)	23	48	62	NR



Scheme 4.

Table 2
Compounds **4a–c** and **6a–i** prepared

3–6	R	R^1	Time (h)	Yield (%)	
				4	6
a	Ph	*i*-PrO–	2.0	15	62
b	Ph	EtO–	1.0	12	52
c	*n*-Bu	*i*-PrO–	0.5	10	9
d	H	*i*-PrO–	0.5	0	28
e	Ph	Pyrrolidino–	2.0	0	51
f	Ph	3-$CH_3C_6H_4CH_2NH$-	2.5	0	53
g	*n*-Bu	Pyrrolidino–	2.0	0	38
h	*n*-Bu	3-$CH_3C_6H_4CH_2NH$–	2.5	0	51
I	H	3-$CH_3C_6H_4CH_2NH$–	1.5	0	28



Ti* = Ti (L+1);
R = H, *n*-Bu, Ph;
R^1 = *i*-PrO, EtO, pyrrolidino-, 3-$CH_3C_6H_4CH_2NH$

Scheme 5.

mixture was then extracted with EtOAc. The combined organic layers were washed with water and dried over $MgSO_4$. The solvent was removed to give a solid, which was separated or purified by chromatography (silica gel, EtOAc–PE–MeOH = 5:5:1) to give compounds **4** and/or **6**.

*3.1.1. 2,2′-Diphenyl-3,3′-diisopropoxy-4,4′-dihydroxy-4,4′-bicyclobutenone (**4a**) and 2,4′-diphenyl-4,2′-dihydroxy-3,3′-diisopropoxy-4,4′-bicyclobutenone (**6a**)*

Compound **4a** was obtained as white crystals: m.p. 168–170°C (EtOAc). IR (cm^{-1}): 3451, 3313, 2989, 1751, 1746, 1618, 1588, 1494, 1406, 1333. ^{1}H-NMR ($CDCl_3$): $\delta = 7.60$ (d, 4H, $J = 7.6$ Hz), 7.26 (t, 4H, $J = 8.2$ Hz), 7.20 (t, 2H, $J = 7.2$ Hz), 5.16 (hept, 2H, $J = 5.9$ Hz), 5.04 (s, 2H), 1.48 (d, 6H, $J = 5.9$ Hz), 1.39 (d, 6H, $J = 5.9$ Hz). MS; *m*/*z* (%): 434 (M^+, 1), 432 (4), 416 (M − 18, 7), 390 (M − 44, 4), 374 (26), 348 (29.8), 332 (44), 276 (8), 214 (27), 195 (76), 167 (22), 145 (51), 118 (100), 89 (29), 46 (54). Anal. Found: C, 71.92; H, 6.09. Calc. for $C_{26}H_{26}O_6$: C, 71.87; H, 6.03%.

Compound **6a** was obtained as white crystals: m.p. 214–216°C (MeOH). IR (cm^{-1}): 3273, 3064, 2983, 1774, 1747, 1631, 1493, 1407, 1327. ^{1}H-NMR (Me_2SO-d_6): $\delta = 9.97$ (s, 1H), 7.50 (d, 2H, $J = 7.7$ Hz), 7.42 (d, 2H, $J = 7.5$ Hz), 7.33 (t, 2H, $J = 7.5$ Hz), 7.23 (t, 3H, $J = 7.5$ Hz), 7.16 (t, 1H, $J = 7.2$ Hz), 6.95 (s, 1H), 5.24 (hept, 1H, $J = 5.9$ Hz), 4.91 (hept, 1H, $J = 5.9$ Hz), 1.39 (d, 3H, $J = 5.9$ Hz), 1.36 (d, 3H, $J = 5.9$ Hz), 1.31 (d, 3H, $J = 5.9$ Hz), 1.22 (d, 3H, $J = 5.9$ Hz). MS; *m*/*z* (%): 434 (M^+, 2), 416 (M − 18, 1), 392 (M − 42, 3), 350 (9), 332 (14), 294 (20), 277 (29), 175 (10), 145 (80), 118 (65), 89 (100), 63 (20). Anal. Found: C, 71.77; H, 6.18. Calc. for $C_{26}H_{26}O_6$: C, 71.87; H, 6.03%.

*3.1.2. 2,2′-Diphenyl-3,3-diethoxy-4,4′-dihydroxy-4,4′-bicyclobutenone (**4b**) and 2,4′-diphenyl-4,2′-dihydroxy-3,3′-diethoxy-4,4′-bicyclobutenone (**6b**)*

Compound **4b** was obtained as white crystals: m.p. 165–166.5°C (EtOAc). IR (cm^{-1}): 3270, 1738, 1610, 1582, 1485, 1405, 1375, 1342. ^{1}H-NMR (Me_2SO-d_6): $\delta = 7.56$ (d, 4H, $J = 7.8$ Hz), 7.37 (t, 4H, $J = 7.6$ Hz), 7.27 (d, 2H, $J = 7.5$ Hz), 4.79 (q, 4H, $J = 7.1$ Hz), 1.49 (t, 6H, $J = 7.1$ Hz). MS; *m*/*z* (%): 406 (M^+, 71), 360 (M − 46, 33), 332 (15), 259 (15), 231 (16), 203 (36), 188 (32), 145 (77), 131 (29), 118 (68), 91 (81), 89 (100), 77 (24). Anal. Found: C, 70.85; H, 5.32. Calc. for $C_{24}H_{22}O_6$: C, 70.93; H, 5.46%.

Compound **6b** was obtained as white crystals: m.p. 193–195°C (MeOH). IR (cm^{-1}): 3185, 1775, 1738, 1625, 1585, 1483, 1376, 1325. ^{1}H-NMR (Me_2SO-d_6): $\delta = 9.91$ (s, br., 1H), 7.61–7.15 (m, 10H), 6.93 (s, br., 1H), 4.55 (q, 2H, $J = 7.0$ Hz), 4.47 (q, 2H, $J = 7.0$ Hz), 1.36 (t, 3H, $J = 7.0$ Hz), 1.26 (t, 3H, $J = 7.0$ Hz). MS; *m*/*z* (%): 406 (M^+, 5), 360 (M − 46, 7), 332 (22), 303 (20), 275 (36), 202 (31), 175 (23), 145 (100), 117 (35), 89

(84). Anal. Found: C, 70.91; H, 5.38. Calc. for $C_{24}H_{22}O_6$: C, 70.93; H, 5.46%.

3.1.3. 2,2′-Di(n-butyl)-3,3′-diisopropoxy-4,4′-dihydroxy-4,4′-bicyclobutenone (**4c**) and 2,4′-di(n-butyl)-4,2′-dihydroxy-3,3-diisopropoxy-4,4′-bicyclobutenone (**6c**)

Compound **4c** was obtained as white crystals: m.p. 133–135°C (EtOAc–PE). IR (cm^{-1}): 3314, 3180, 2959, 2932, 2862, 1774, 1742, 1633, 1397, 1320. ^{1}H-NMR (Me_2SO-d_6): $\delta = 5.24$ (hept, 2H, $J = 6.0$ Hz), 4.89 (s, 2H), 2.59 (m, 4H), 2.19 (m, 4H), 1.64 (m, 4H), 1.48 (d, 6H, $J = 6.0$ Hz), 1.42 (d, 6H, $J = 6.0$ Hz), 0.87 (t, 6H, $J = 7.0$ Hz). MS; m/z (%): 394 (M^+, 1), 350 (16), 332 (16), 293 (21), 234 (22), 207 (18), 85 (22), 56 (50), 45 (100). Anal. Found: C, 66.76; H, 8.75. Calc. for $C_{22}H_{34}O_6$: C, 66.98; H, 8.69%.

Compound **6c** was obtained as white crystals: m.p. 148–150°C (EtOAc–PE). IR (cm^{-1}): 3308, 3223, 2933, 1773, 1740, 1623, 1403, 1323. ^{1}H-NMR ($CDCl_3$): $\delta = 9.10$ (s, br., 1H), 5.23 (hept, 1H, $J = 6.0$ Hz), 5.09 (s, br., 1H), 4.96 (hept, 1H, $J = 6.0$ Hz), 2.09 (m, 2H), 1.47 (m, 2H), 1.41 (m, 13H), 1.31 (m, 3H), 1.21 (m, 3H), 1.11 (m, 1H), 0.87 (t, 3H, $J = 7.3$ Hz), 0.81 (t, 3H, $J = 6.9$ Hz). MS; m/z (%): 394 (M^+, 6), 309 (32), 266 (56), 249 (57), 125 (65), 58 (63), 45 (100). Anal. Found: C, 67.22; H, 8.64. Calc. for $C_{22}H_{34}O_6$: C, 66.98; H, 8.69%.

3.1.4. 4,2′-Dihydroxy-3,3′-diisopropoxy-4,4′-bicyclobutenone (**6d**)

Compound **6d** was obtained as white crystals: m.p. 153–155°C (EtOAc–PE). IR (cm^{-1}): 3245, 3087, 2984, 1779, 1741, 1624, 1585, 1407, 1323. ^{1}H-NMR (Me_2SO-d_6): $\delta = 9.89$ (s, 1H), 6.21 (s, 1H), 5.41 (s, 1H), 4.79 (hept, 1H, $J = 6.1$ Hz), 4.54 (hept, 1H, $J = 6.1$ Hz), 3.19 (s, 1H), 1.29 (m, 12H). MS; m/z (%): 282 (M^+, 3), 240 (M − 42, 6), 226 (11), 198 (15), 180 (11), 169 (18), 152 (29), 142 (74), 69 (24), 43 (100). Anal. Found: C, 59.37; H, 6.53. Calc. for $C_{14}H_{18}O_6$: C, 59.57; H, 6.43%.

3.1.5. 2,4′-Diphenyl-4,2′-dihydroxy-3,3′-dipyrrolidino-4,4′-bicyclobutenone (**6e**)

Compound **6e** was obtained as white crystals: m.p. (dec.) 250–252°C (HOAc–H_2O). IR (cm^{-1}): 3251, 1708, 1577, 1442. ^{1}H-NMR (Me_2SO-d_6): $\delta = 9.83$ (s, 1H), 7.42 (d, 4H, $J = 7.7$ Hz), 7.32 (t, 4H, $J = 7.5$ Hz), 7.16 (t, 2H, $J = 7.4$ Hz), 6.33 (s, 1H), 3.77 (m, 4H), 3.58 (m, 2H), 3.07 (m, 2H), 1.89 (m, 2H), 1.80 (m, 6H). MS; m/z (%): 438 (M − 18, 0.02), 412 (M − 44, 10), 368 (5), 249 (6), 229 (10), 171 (100), 128 (13), 115 (37), 70 (23), 43 (46). Anal. Found: C, 73.50; H, 6.38; N, 6.27. Calc. for $C_{28}H_{28}O_4N_2$: C, 73.66; H, 6.18; N, 6.14%.

3.1.6. 2,4′-Diphenyl-4,2′-dihydroxy-3,3′-di(3-methylbenzylamino)-4,4′-bicyclobutenone (**6f**)

Compound **6f** was obtained as white crystals: m.p. (dec.) 234–235°C (acetone–PE). IR (cm^{-1}): 3380, 3272, 1729, 1607, 1580, 1335. ^{1}H-NMR (Me_2SO-d_6): $\delta = 9.82$ (s, 1H), 7.74 (m, 2H), 7.38–7.08 (m, 16H), 6.70 (s, 1H), 6.52 (s, 1H), 5.16 (s, 2H), 4.93 (s, 1H), 4.67 (s, 2H), 2.30 (s, 3H), 2.28 (s, 3H). MS; m/z (%): 556 (0.6), 538 (M − 18, 7), 512 (M − 44, 6), 407 (8), 390 (1), 290 (2), 248 (3), 132 (4), 105 (100), 91 (12), 77 (12), 44 (9). Anal. Found: C, 77.54; H, 5.82; N, 4.97. Calc. for $C_{36}H_{32}O_4N_2$: C, 77.68; H, 5.79; N, 5.03%.

3.1.7. 2,4′-Di(n-butyl)-4,2′-dihydroxy-3,3-dipyrrolidino-4,4′-bicyclobutenone (**6g**)

Compound **6g** was obtained as white crystals: m.p. 190–191°C (C_6H_6–PE). IR (cm^{-1}): 3115, 2949, 1730, 1563, 1444, 1354. ^{1}H-NMR ($CDCl_3$): $\delta = 10.33$ (s, 1H), 6.35 (s, br., 1H), 3.87 (m, 2H), 3.50 (m, 6H), 2.21 (m, 4H), 2.05 (m, 4H), 1.89 (m, 5H), 1.42 (m, 4H), 1.31 (m, 3H), 0.88 (t, 6H, $J = 7.3$ Hz). MS; m/z (%): 416 (M^+, 0.1), 398 (M − 18, 0.1), 372 (M − 44, 14), 345 (5), 329 (37), 302 (13), 258 (13), 179 (22), 150 (100), 136 (37), 108 (69), 95 (23), 80 (33), 70 (63), 55 (63), 43 (49). Anal. Found: C, 69.17; H, 8.84; N, 6.81. Calc. for $C_{24}H_{36}O_4N_2$: C, 69.20; H, 8.71; N, 6.73%.

3.1.8. 2,4′-Di(n-butyl)-4,2′-dihydroxy-3,3′-di(3-methylbenzylamino)-4,4′-bicyclobutenone (**6h**)

Compound **6h** was obtained as white crystals: m.p. (dec.) 218–220°C (MeOH). IR (cm^{-1}): 3372, 2955, 1736, 1595, 1577, 1527. ^{1}H-NMR (Me_2SO-d_6): $\delta = 9.27$ (s, 1H), 7.21 (m, 6H), 7.08 (m, 2H), 6.01 (s, 1H), 5.80 (s, 1H), 4.85 (s, 2H), 4.64 (s, 1H), 4.51 (s, 2H), 2.30 (s, 6H), 1.94 (t, 2H, $J = 7.0$ Hz), 1.86 (t, 2H, $J = 7.0$ Hz), 1.38 (m, 2H), 1.31 (m, 2H), 1.23 (m, 2H), 1.18 (m, 2H), 0.84 (t, 3H, $J = 7.0$ Hz), 0.76 (t, 3H, $J = 6.6$ Hz). MS; m/z (%): 516 (M^+, 0.2), 498 (M − 18, 13), 472 (M − 44, 2), 455 (17), 411 (32), 393 (38), 379 (7), 365 (5), 105 (100). Anal. Found: C, 74.26; H, 7.68; N, 5.51. Calc. for $C_{32}H_{40}O_4N_2$: C, 74.39; H, 7.80; N, 5.42%.

3.1.9. 4,2′-Dihydroxy-3,3′-(3-methylbenzylamino)-4,4′-bicyclobutenone (**6i**)

Compound **6i** was obtained as white crystals: m.p. (dec.) 222–223°C (HOAc–H_2O). IR (cm^{-1}): 3313, 1721, 1604, 1348. ^{1}H-NMR (Me_2SO-d_6): $\delta = 9.56$ (s, 1H), 7.33–7.04 (m, 8H), 6.32 (s, 1H), 6.17 (s, 1H), 6.08 (s, 1H), 4.98 (s, 1H), 4.37 (s, 4H), 2.29 (s, 6H). MS; m/z (%): 404 (M^+, 0.1), 402 (M − 2, 0.5), 360 (M − 44, 9), 255 (16), 214 (5), 158 (4), 120 (6), 105 (100), 91 (10), 77 (18), 44 (6). Anal. Found: C, 71.35; H, 5.90; N, 6.91. Calc. for $C_{24}H_{24}O_4N_2$: C, 71.27; H, 5.98; N, 6.93%.

Table 3
Crystallographic data for compounds **4a** and **6a**

Compound	**4a**	**6a**
Empirical formula	$C_{26}H_{26}O_6$	$C_{26}H_{26}O_6$
Formula weight	434.49	434.49
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$ (no. 14)	$P2_1/c$ (no. 14)
Unit cell dimensions		
a (Å)	11.806 (3)	13.034 (3)
b (Å)	19.14 (2)	9.804 (2)
c (Å)	11.832 (4)	19.029 (4)
β (°)	116.33 (2)	103.128 (4)
V (Å^3)	2395.8301	2369.0701
Z	4	4
D_{calc} (g cm^{-3})	1.204	1.219
$F(000)$	920.00	920.00
μ (Mo–Kα) (cm^{-1})	0.85	0.86
Reflections observed $[I>3\sigma(I)]$	1636	1547
Number of variables	290	290
Goodness-of-fit	1.21	1.07
Max. shift in cycle	0.00	0.00
Residuals: R; wR	0.056; 0.076	0.050; 0.069
Max/min transmission	7.22510×10^{-7}	$6.73070\times^{-7}$
Largest peak − final difference map (e Å^{-3})	0.24	0.24

*3.2. Crystallographic data collections and structure determination of **4a** and **6a***

The single crystals suitable for X-ray measurements was obtained by recrystallization of **4a** and **6a** from EtOAc having approximate dimensions of $0.40\times0.30\times0.20$ and $0.40\times0.30\times0.30$ mm^3, respectively. All measurements were made on a Rigaku RAXIS-IV imaging plate area detector with graphite monochromated Mo–Kα radiation at 18 ± 1°C. Structure solutions were performed by direct methods. Crystal data and details about data collection and structure refinement are given in Table 3.

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC nos. 160543 and 160544 for compounds **4a** and **6a**, respectively. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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