

## CHEMICAL INVESTIGATION OF THE GENUS *RHEEDIA*. II.<sup>1</sup> PRENYLATED XANTHONES FROM *RHEEDIA GARDNERIANA*<sup>2</sup>

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ABSTRACT.—Two new prenylated xanthenes have been isolated from the root extract of *Rheedia gardneriana*, and their structures have been established as 4',5'-dihydro-1,5,6-trihydroxy-4',5',5'-trimethylfuran (2',3':3,2)-4 (1,1-dimethylprop-2-enyl)-xanthone (**5**) and 1,5-dihydroxy-6',6'-dimethylpyrano (2',3':6,7)-xanthone (**6**), respectively.

The genus *Rheedia* (tribe, Clusioideae; family, Guttiferae) has been, so far, scarcely investigated (1), in spite of its close relationship with a widely studied genus—*Garcinia*—which afforded xanthenes, polyprenylated benzophenones, and biflavonoids (2). Only recently, three new 1,3,5,6-tetraoxygenated xanthenes, namely rheedioxanthone A (**1**), B (**2**), and C (**3**) have been isolated, in addition to macluraxanthone (**4**), from the root-bark of *Rheedia benthamiana* Pl. and Tr. (3). As a part of a comparative study of the chemistry of a number of brazilian Guttiferae, we examined the roots of *Rheedia gardneriana* Pl. and Tr.

### RESULTS AND DISCUSSION

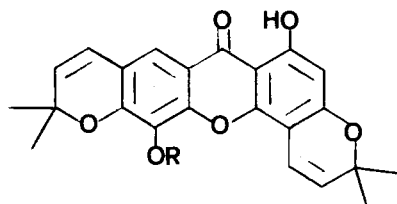
Rheedioxanthone A, B, C, and macluraxanthone (**1**, **2**, **3**, and **4**, respectively) were again isolated from the benzene extract of the plant and identified by comparison with authentic samples. Moreover, two new prenylated xanthenes were obtained and their structures determined. The former, **5**, C<sub>23</sub>H<sub>24</sub>O<sub>6</sub> (MW 396), is a tetraoxygenated xanthone (ir and uv evidence) with an  $\alpha,\alpha$ -dimethylallyl chain and an  $\alpha,\alpha,\beta$ -trimethyldihydrofuran ring (<sup>1</sup>H-nmr evidence), thus isomeric with rheedioxanthone B (**2**). The identity of the B rings of the two products is substantiated by the uv spectra, with additives, and by the comparable chemical shifts of the H-7 and H-8 protons in xanthenes **2-5**; it was confirmed by obtaining the two monoacetyl derivatives **5a** and **5b**, in whose <sup>1</sup>H-nmr spectra the H-7 and the H-8 protons are shifted downfield alternatively (the data are reported, in comparison with those of the compound **5** and its diacetyl derivative **5c**, in table 1).

TABLE 1. Ring B protons resonances ( $\delta$ , CDCl<sub>3</sub>) in compound **5** and its acetyl derivatives

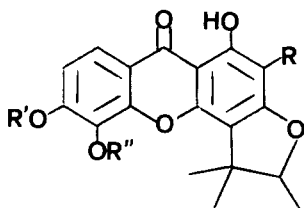
	H-7 (d, J=9)	H-8 (d, J=9)
<b>5</b> . . . . .	6.98	7.73
<b>5a</b> . . . . .	6.97	8.02
<b>5b</b> . . . . .	7.07	7.73
<b>5c</b> . . . . .	7.16	8.10

<sup>1</sup>Reference (3) can be considered as part I of the series.

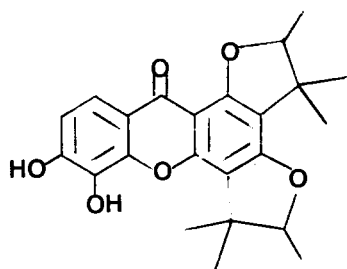
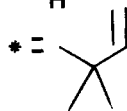
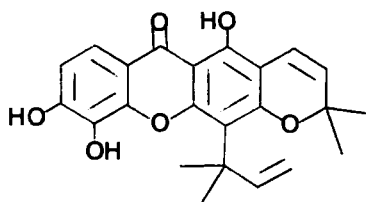
<sup>2</sup>A preliminary communication was presented at the First International Conference on the Chemistry and Biotechnology of Biologically Active Natural Products, Varna, Bulgaria, September 21-26, 1981.



	R
<b>1</b>	H
<b>1a</b>	COCH <sub>3</sub>



	R	R'	R''
<b>2</b>	*	H	H
<b>2a</b>	*	COCH <sub>3</sub>	COCH <sub>3</sub>
<b>2b</b>	*	CH <sub>3</sub>	H
<b>2c</b>	*	CH <sub>3</sub>	CH <sub>3</sub>
<b>2d</b>	H	H	H

**3****4**

Considering the close accord of the uv spectrum of **5** with that of **1-4** and other 1,3,5,6-tetraoxygenated xanthenes, the substitution pattern of the ring A can be only the one indicated in structure **5**. Notably, the propenyl *gem*-CH<sub>3</sub> resonances in CD<sub>3</sub>COCD<sub>3</sub> and C<sub>5</sub>D<sub>5</sub>N ( $\delta$  1.70 and 2.02) are coincident with those of macluraxanthone (**4**,  $\delta$  1.75 and 2.01), where the substituent is on C-4 position, and are largely different from those of rheediaxanthone B (**2**,  $\delta$  1.57 and 1.76, respectively), where the substituent is on C-2.

A chemical confirmation of the structure **5** was finally obtained when the xanthone **5** was boiled in HCOOH, yielding the product **5d**, C<sub>18</sub>H<sub>16</sub>O<sub>6</sub> (MW 328), by loss of the isoprenyl chain. The chemical shift of the generated aromatic proton (H-4) was unchanged in the <sup>1</sup>H-nmr spectrum when the solvent (CD<sub>3</sub>COCD<sub>3</sub>) was substituted by C<sub>5</sub>D<sub>5</sub>N (**4**), as contrasted by the behavior of the isomer **2d**, obtained from rheediaxanthone B (**2**) under the same conditions (**3**), where the new proton (H-2) is shifted downfield (0.28 ppm). Moreover, only the isomer **2d** showed an immediate bathochromic shift in the uv spectrum upon addition of AlCl<sub>3</sub> (**4**).

The structure of the latter new xanthone (**6**), C<sub>18</sub>H<sub>14</sub>O<sub>5</sub> (MW 310), follows from consideration of the spectroscopic data. The uv spectrum relates compound **6** to 1,5,6-trioxygenated xanthenes (**5**). The signals of a *gem*-dimethyl group and of two coupled ( $J = 10$  Hz) olefinic protons in the <sup>1</sup>H-nmr spectrum and the loss of 15 mass units from the molecular ion in the mass spectrum, to give the base peak, indicate the presence of a 2,2-dimethyl (2H) pyran ring. Further, the shoulder on the longer wave-length band, which is also present in the uv spectra of **1** and **4**, can be considered a typical feature of the chromenoxanthenes.

The <sup>1</sup>H-nmr spectrum is completed by the resonances of two hydroxyls, one of which is *peri* to the carbonyl group, and of four aromatic protons. Three of them show up as an AXY system with a coupling pattern very similar (table 2) to that of the ring A protons of buchanaxanthone (1,6-dihydroxy-5-methoxyxanthone) and related compounds (**5**).

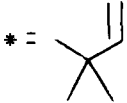
TABLE 2. Ring A protons resonances<sup>a</sup> ( $\delta$ , CDCl<sub>3</sub>) of 1-hydroxy-xanthenes

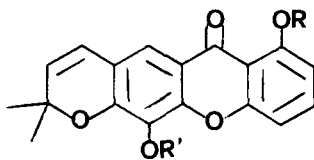
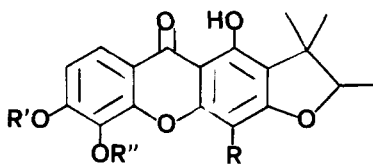
	H-2	H-3	H-4
Buchanaxanthone <sup>b</sup> . . . . .	6.67	7.67	7.01
1-Hydroxy-5,6-dimethoxyxanthone . . . . .	6.80	7.60	7.01
1-Hydroxy-5-methoxyxanthone . . . . .	6.84	7.65	7.06
1-Hydroxy-5-methoxy-6-propylxanthone . . . . .	6.74	7.56	6.95
Rheediachromenoxanthone . . . . .	6.80	7.62	6.90

<sup>a</sup>H-3 signal is a triplet. H-2 and H-4 show up as *ortho* split doublets with additional *meta* splitting.

<sup>b</sup>In DMSO.

The C<sub>5</sub>D<sub>5</sub>N influence in the <sup>1</sup>H-nmr spectrum confirms this assignment, with only the H-2 signal being shifted to lower field. Comparison of the resonance of the protons H-8, H-4', and H-5' of **6** and its acetyl derivative **6a** with those of **1** and its acetyl derivative **1a** established the substitution pattern of the ring B as indicated in the final structure **6**. The compound **6**, for which we propose the name of rheediachromenoxanthone, is the first example of prenylated xanthone with 1,5,6 oxygenation pattern.

	R	R'	R''
<b>5</b>	*	H	H
<b>5a</b>	*	COCH <sub>3</sub>	H
<b>5b</b>	*	H	COCH <sub>3</sub>
<b>5c</b>	*	COCH <sub>3</sub>	COCH <sub>3</sub>
<b>5d</b>	H	H	H
	* = 		
<b>6</b>	R	R'	
<b>6a</b>	H	H	
<b>6b</b>	COCH <sub>3</sub>	COCH <sub>3</sub>	
<b>6c</b>	H	CH <sub>3</sub>	
<b>6c</b>	CH <sub>3</sub>	CH <sub>3</sub>	



## EXPERIMENTAL

GENERAL EXPERIMENTAL PROCEDURES.—Elemental analyses were in agreement with molecular formulas. Each mp was determined by means of a Kofler hot plate and is uncorrected. Spectra were recorded with the following instruments: uv, Beckmann Acta III; <sup>1</sup>H-nmr, Varian EM 360; ms, AEI 12; ir, Perkin Elmer 247. Adsorbants used were from E. Merck (plc and tlc) and Macharey-Nagel (cc).

PLANT MATERIAL.—Roots of *Rheedia gardneriana* Pl. and Tr. were collected in northeastern Brazil (Pacatuba, Fortaleza) and identified by José Elias de Paula (Universidade Federal de Brasília).

EXTRACTION AND SEPARATION OF THE XANTHONES.—Bark (500g) and wood (500g) of the roots gave, by extraction with hot benzene, the same qualitative and quantitative results (7 g each). The crude extract was fractionated on a silica gel column with benzene-ethyl acetate mixtures. Extended cc and plc gave the pure products.

*Rheediaxanthone A*, (**1**). 1%, C<sub>23</sub>H<sub>20</sub>O<sub>6</sub> (MW 392), mp 258-60° (ether-hexane); *acetyl derivative*, (**1a**): mp 207-9° (CH<sub>2</sub>Cl<sub>2</sub>-heptane). The identification of rheediaxanthone A (**1**) and its acetyl derivative (**1a**) was confirmed by comparison with authentic samples from *Rheedia benthamiana* (**3**).

*Rheediaxanthone B*, (**2**). 15%, C<sub>23</sub>H<sub>24</sub>O<sub>6</sub> (MW 396), mp 208-10° (benzene-ethyl acetate, [α]<sup>22</sup><sub>D</sub> = -25 (c 1.2, acetone); *diacetyl derivative*, (**2a**): mp 168-9° (CH<sub>2</sub>Cl<sub>2</sub>-heptane). The identity with rheediaxanthone B (**2**) and its diacetyl derivative (**2a**) was confirmed by comparison with authentic samples from *Rheedia benthamiana*. *Methyl derivatives*. Rheediaxanthone B (**2**) gave, with CH<sub>3</sub>N<sub>2</sub>, two products, separated on silica gel with benzene: *monomethyl derivative*, (**2b**): C<sub>24</sub>H<sub>26</sub>O<sub>6</sub>, mp 198-200° (benzene-hexane); δ (CD<sub>3</sub>COCD<sub>3</sub>): 14.26 (1H, s, ex. D<sub>2</sub>O, 1-OH), 7.69 (1H, d, J=9, H-8), 7.10 (1H, d, J=9, H-7), 6.57-6.11 (1H, A part of AXY), 5.07-4.73 (2H, XY part of AXY), 4.50 (1H, q, J=7, H-4'), 4.02 (3H, s, OCH<sub>3</sub>), 1.60 (12H, large s, 4 x Me), 1.40 (3H, d, J=7, 4'-Me); λ max (MeOH and AcONa): 259, 286sh, 332; (AlCl<sub>3</sub> after 30' and AlCl<sub>3</sub>/HCl): 274, 359, 420sh. *dimethyl derivative*, (**2c**): C<sub>25</sub>H<sub>28</sub>O<sub>6</sub>, mp 143-5° (ether-heptane); δ (CCl<sub>4</sub>): 13.78 (1H, s, ex. D<sub>2</sub>O, 1-OH), 7.75 (1H, d, J=9, H-8), 6.90 (1H, d, J=9, H-7), 6.53-6.08 (1H, A part of AXY), 5.03-4.73 (2H, XY part of AXY), 4.41 (1H, q, J=7, H-4'), 4.01+3.94 (3H+3H, s+s, 5-OCH<sub>3</sub>+6-OCH<sub>3</sub>), 1.60 (12H, large s, 4 x Me), 1.42 (3H, d, J=7, 4'-Me); λ max (MeOH and AcONa): 244, 324; (AlCl<sub>3</sub> after 30' and AlCl<sub>3</sub>/HCl): 246, 274sh, 350.

*Rheediaxanthone C*, (**3**). 1%, C<sub>23</sub>H<sub>24</sub>O<sub>6</sub> (MW 396), mp 262-4° (ether-heptane), [α]<sup>24</sup><sub>D</sub> = -22 (c 0.5, acetone). It was identified as rheediaxanthone C (**3**) by comparison with an authentic specimen from *Rheedia benthamiana* (**3**).

*Macluraxanthone*, (**4**). semimicro amount, C<sub>23</sub>H<sub>22</sub>O<sub>6</sub> (MW 394), mp 180-2° and 204-6° (benzene-ethyl acetate). It was identified as macluraxanthone (**4**) by comparison with an authentic specimen from *Rheedia benthamiana* (**3**). Δδ = δ (C<sub>5</sub>D<sub>5</sub>N)-δ (CD<sub>3</sub>COCD<sub>3</sub>): H-8 (+0.35), H-7 (+0.23), H-4' (+0.28), H-5' (-0.08), 6'-Me<sub>2</sub> (-0.07), propenyl-Me<sub>2</sub> (+0.28).

*Isorheediaxanthone B*, (**5**). 4',5'-dihydro-1,5,6-trihydroxy-4',5',5'-trimethylfurano (2',3':3,2)-4 (1,1-dimethylprop-2-enyl)-xanthone: 2%, C<sub>23</sub>H<sub>24</sub>O<sub>6</sub> (MW 396), mp 212-3° (benzene-hexane), [α]<sup>27</sup><sub>D</sub> = +16 (c 0.6, acetone); δ (CD<sub>3</sub>COCD<sub>3</sub>): 13.66 (1H, s, ex. D<sub>2</sub>O, 1-OH), 7.52 (1H, d, J=9, H-8), 6.92 (1H, d, J=9, H-7), 6.73-6.23 (1H, A part of AXY), 5.36-4.72 (2H, XY part of AXY), 4.43 (1H, q, J=7, H-4'), 1.70 (6H, s, propenyl-Me<sub>2</sub>), 1.46+1.23 (3H+3H, s+s, 5'-Me<sub>2</sub>), 1.37 (3H, d, J=7, 4'-Me); Δδ = δ (C<sub>5</sub>D<sub>5</sub>N)-δ (CD<sub>3</sub>COCD<sub>3</sub>): H-8 (+0.41), H-7 (+0.30), 5'-Me<sub>2</sub> (+0.04 and +0.07), propenyl-Me<sub>2</sub> (+0.32); ν max (CHCl<sub>3</sub>): 3545, 3440, 1657, 1650, 1628, 1589, 1386-1382, 1262, 1188, 1114, 1093, 1070, 954, 912, 884; λ max (MeOH): 256 (log ε 4.68), 288 (4.25), 332 (4.43); (AcONa): 258, 289, 360; (MeONa): 260, 294, 363; (AcONa/H<sub>3</sub>BO<sub>3</sub>): 264, 291, 348; (AlCl<sub>3</sub> after 30'): 276, 298, 391; (AlCl<sub>3</sub>/HCl): 258, 298, 358; m/z (rel. int.): 396 (M<sup>+</sup>, 44), 395 (3), 381 (100), 379 (9), 369 (3), 367 (1), 365 (2), 355 (3), 351 (3), 339 (5), 327 (1), 325 (8), 323 (2), 313 (7), 311 (3), 299 (2), 297 (2), 295 (1), 285 (2), 281 (1), 269 (1), 198 (1), 183 (3), 69 (4), 41 (5); m\*: 366.6 (396→381), 318.2 (396→355), 277.2 (381→325), 257.1 (381→313), 24.4 (69→41).

*Acetyl derivatives*: Isorheediaxanthone B (**5**) with Ac<sub>2</sub>O in pyridine gave three products: the first one was obtained by cc (on silica gel with benzene-ethyl acetate 9:1), whereas the latter two were separated by crystallization and plc.

5,6-Diacetyl derivative, (**5c**): C<sub>27</sub>H<sub>28</sub>O<sub>8</sub>, mp 188° (heptane); δ (CDCl<sub>3</sub>): 12.94 (1H, s, ex. D<sub>2</sub>O, 1-OH), 8.12 (1H, d, J=9, H-8), 7.18 (1H, d, J=9, H-7), 6.48-5.98 (1H, a part of AXY), 5.00-4.64 (2H, XY part of AXY), 4.36 (1H, q, J=7, H-4'), 2.40+2.35 (3H+3H, s+s, 2 x COCH<sub>3</sub>), 1.62 (6H, s, propenyl-Me<sub>2</sub>), 1.48+1.24 (3H+3H, s+s, 5'-Me<sub>2</sub>), 1.35 (3H, d, J=7, 4'-Me<sub>2</sub>).

5-Monoacetyl derivative, (**5b**): C<sub>25</sub>H<sub>26</sub>O<sub>7</sub>, mp 203-5° (ether-hexane), green spot with FeCl<sub>3</sub>; δ (CDCl<sub>3</sub>):

13.20 (1H, s, ex. D<sub>2</sub>O, 1-OH), 8.02 (1H, d, *J*=9, H-8), 6.97 (1H, d, *J*=9, H-7), 6.58-6.06 (1H, A part of AX<sub>2</sub>Y), 5.24-4.70 (2H, XY part of AX<sub>2</sub>Y), 4.42 (1H, q, *J*=7, H-4'), 2.42 (3H, s, COCH<sub>3</sub>), 1.65 (6H, s, propenyl-Me<sub>2</sub>), 1.43+1.26 (3H+3H, s+s, 5'-Me<sub>2</sub>), 1.35 (3H, d, *J*=7, 4'-Me).

*6-Monoacetyl derivative*. (**5a**): C<sub>25</sub>H<sub>26</sub>O<sub>7</sub>, mp 165° (ether-hexane), violet spot with FeCl<sub>3</sub>; δ (CDCl<sub>3</sub>): 13.00 (1H, s, ex. D<sub>2</sub>O, 1-OH), 7.73 (1H, d, *J*=9, H-8), 7.07 (1H, d, *J*=9, H-7), 6.30-5.95 (1H, A part of AX<sub>2</sub>Y), 5.45-4.95 (2H, XY part of AX<sub>2</sub>Y), 4.23 (1H, q, *J*=7, H-4'), 2.38 (3H, s, COCH<sub>3</sub>), 1.59 (6H, s, propenyl-Me<sub>2</sub>), 1.50+1.24 (3H+3H, s+s, 5'-Me<sub>2</sub>), 1.30 (3H, d, *J*=7, 4'-Me).

*4',5'-Dihydro-1,5,6-trihydroxy-4',5',5'-trimethylfurano 2',3':3,2-xanthone*. (**5d**): Isorheediachromenoxanthone B (**5**, 30 mg) was refluxed 30 min in HCOOH. After cc purification (silica gel, CH<sub>2</sub>Cl<sub>2</sub>), 4',5'-dihydro-1,5,6-trihydroxy-4',5',5'-trimethyl furano (2',3':3,2)-xanthone (**5d**, 21 mg) was obtained: C<sub>18</sub>H<sub>16</sub>O<sub>6</sub> (MW 328), mp 246-8° (ether-hexane), [α]<sub>D</sub><sup>26</sup>+45 (c 0.2, MeOH); δ (CD<sub>3</sub>COCD<sub>3</sub>): 12.90 (1H, s, ex. D<sub>2</sub>O, 1-OH), 7.50 (1H, d, *J*=8.5, H-8), 6.87 (1H, d, *J*=8.5, H-7), 6.22 (1H, s, H-4), 4.43 (1H, q, *J*=6.5, H-4'), 1.47+1.22 (3H+3H, s+s, 5'-Me<sub>2</sub>), 1.37 (3H, d, *J*=6.5, 4'-Me); Δδ=δ (C<sub>5</sub>D<sub>5</sub>N)-δ (CD<sub>3</sub>COCD<sub>3</sub>): H-8 (+0.46), H-7 (+0.35), H-4 (0); λ max (MeOH): 250, 285sh, 328; (AcONa): 258, 280, 356; (AcONa/H<sub>3</sub>BO<sub>3</sub>): 258, 280sh, 342; (AlCl<sub>3</sub> after 30'): 266, 381; (AlCl<sub>3</sub>/HCl): 254, 348; *m/z* (rel. int.): 328 (M<sup>+</sup>, 25), 313 (M-Me, 100), 298 (9), 295 (7), 285 (16), 269 (6), 257 (6), 149 (33).

*Rheediachromenoxanthone*. (**6**). 1,5-dihydroxy-6',6'-dimethylpyrano (2',3':6,7)-xanthone. 1%, C<sub>18</sub>H<sub>14</sub>O<sub>5</sub> (MW 310), mp 223-4° (ether-hexane); δ (CD<sub>3</sub>COCD<sub>3</sub>): 12.90 (1H, s, ex. D<sub>2</sub>O, 1-OH), 8.40 (1H, s, ex. D<sub>2</sub>O, 5-OH), 7.62 (1H, t, *J*=8.5, H-3), 7.43 (1H, s, H-8), 6.94 (1H, dd, *J*=8.5 and 1.5, H-4), 6.70 (1H, dd, *J*=8.5 and 1.5, H-2), 6.55 (1H, d, *J*=10, H-4'), 5.87 (1H, d, *J*=10, H-5'), 1.50 (6H, s, 6'-Me<sub>2</sub>); Δδ=δ (C<sub>5</sub>D<sub>5</sub>N)-δ (CD<sub>3</sub>COCD<sub>3</sub>): H-8 (+0.30), H-3 (-0.09), H-4 (-0.04), H-2 (+0.22), H-4' (-0.05), H-5' (-0.12), 6'-Me<sub>2</sub> (-0.17); ν (CHCl<sub>3</sub>): 3540, 1642, 1605sh, 1600sh, 1588, 1393, 1384, 1133, 1072, 884, 814; λ max (MeOH and AcONa): 262 (log ε 4.57), 282 (4.59), 312sh, (4.08), 338sh (4.04), 372sh (3.96); (AlCl<sub>3</sub> and AlCl<sub>3</sub>/HCl): 272, 294, 370, 428; *m/z* (rel. int.): 310 (M<sup>+</sup>, 29), 309 (10), 295 (M-Me, 100), 266 (1), 181 (1), 165 (M/2, 2), 147.5 (M-Me/2, 11), 133.5 (M-Me-CO/2, 2.5); m\*: 280.7 (310→295).

*Diacetyl derivative*, (**6a**): Rheediachromenoxanthone (**6**) with Ac<sub>2</sub>O in pyridine overnight gave a diacetyl derivative (**6a**), purified on silica gel (benzene-CH<sub>2</sub>Cl<sub>2</sub> mixtures): C<sub>22</sub>H<sub>18</sub>O<sub>7</sub>, mp 183-5°, H-2 (ether-hexane); δ (CDCl<sub>3</sub>): 7.69, H-4 (1H, s, H-8), 7.52 (1H, t, *J*=8.5, H-3), 7.28 (1H, dd, *J*=8.5 and 1.5), 6.92 (1H, dd, *J*=8.5 and 1.5), 6.36 (1H, d, *J*=10, H-4'), 5.70 (1H, d, *J*=10, H-5'), 2.47 (6H, s, 2 x COCH<sub>3</sub>), 1.47 (6H, s, 6'-Me<sub>2</sub>).

*Methyl derivatives*: Rheediachromenoxanthone (**6**) with CH<sub>2</sub>N<sub>2</sub> afforded a monomethyl-(**6b**) and a dimethyl derivative-(**6c**), separated on silica gel with hexane-ethyl acetate mixtures; *monomethyl derivative*: C<sub>19</sub>H<sub>16</sub>O<sub>5</sub>, mp 122-3° (ether); δ (CDCl<sub>3</sub>): 12.70 (1H, s, ex. D<sub>2</sub>O, 1-OH), 7.79 (1H, s, H-8), 7.73 (1H, t, *J*=8.5, H-3), 6.90 (1H, dd, *J*=8.5 and 2, H-4), 6.70 (1H, dd, *J*=8.5 and 2, H-2), 6.35 (1H, d, *J*=10, H-4'), 5.68 (1H, d, *J*=10, H-5'), 3.97 (3H, s, OCH<sub>3</sub>), 1.48 (6H, s, 6'-Me<sub>2</sub>); *dimethyl derivative*: C<sub>20</sub>H<sub>18</sub>O<sub>5</sub>, mp 123-4° (ether-hexane); δ (CDCl<sub>3</sub>): 7.59 (1H, s, H-8), 7.49 (1H, t, *J*=8.5, H-3), 7.02 (1H, dd, *J*=8.5 and 2, H-4), 6.72 (1H, dd, *J*=8.5 and 2, H-2), 6.37 (1H, d, *J*=10, H-4'), 5.63 (1H, d, *J*=10, H-5'), 3.97 (6H, s, 2 x OCH<sub>3</sub>), 1.52 (6H, s, 6'-Me<sub>2</sub>).

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