Supporting Information

One-Pot Approach to Chiral Chromenes *via* Enantioselective Organocatalytic Domino Oxa-Michael-Aldol Reaction

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General Information: Commercial reagents were used as received, unless otherwise stated. Merck 60 silica gel was used for chromatography, and Whatman silica gel plates with fluorescence F_{254} were used for thin-layer chromatography (TLC) analysis. ^{1}H and ^{13}C NMR spectra were recorded on Broker Avance 500, and tetramethylsilane (TMS) was used as a reference. Data for ^{1}H are reported as follows: chemical shift (ppm), and multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet). Data for ^{13}C NMR are reported as ppm.

General Procedure for addition of 2-Hydroxy-benzaldehydes to unsaturated aldehydes (Table 2): To a solution of a *trans*-cinnamaldehyde (1a) (0.1 mmol) in the presence of catalyst III (30 mol %), benzoic acid (0.03 mmol) and 4Å MS (50 mg) in 1, 2-dichloroethane (0.5 mL) was added a 2-salicylaldehyde (2a) (1.0 mmol) and the resulting solution was stirred at specified temperature for a specified time. The reaction mixture was directly purified by silica gel chromatography and fractions were collected and concentrated *in vacuo* to give a pure product.

2-Phenyl-2*H***-chromene-3-carbaldehyde (Table 2, entry 1**): The title compound was prepared according the typical procedure, as described above in 87% yield. ¹H NMR (500 MHz, CDCl₃, TMS): δ 9.64 (s, 1H; CHO), 7.40 (s, 1H; CH), 7.34-7.36 (m, 2H; Ar), 7.24-7.30 (m, 5H; Ar), 6.94 (t, ²*J* (H,H) = 7.5 Hz, 1H; Ar), 6.87 (d, ²*J* (H,H) = 8.0 Hz, 1H; Ar), 6.34 (s, 1H; OCH); ¹³C NMR (125 MHz, CDCl₃, TMS): δ 190.0, 154.8, 140.8, 139.0, 133.7, 129.3, 128.6, 128.5, 126.7, 121.7, 119.9, 117.1, 74.2; $[\alpha]_D^{23} = -39.2$ (c = 1.0, MeOH); HPLC (Daicel CHIRALPAK AS-H, Hexane/*i*-PrOH = 90:10, flow rate 0.5 mL/min, λ = 254 nm): $t_{maior} = 25.54$ min, $t_{minor} = 35.53$ min, ee = 88%.

2-(4-Nitro-phenyl)-2*H***-chromene-3-carbaldehyde** (**Table 2, entry 2**): The title compound was prepared according the typical procedure, as described above in 96% yield. ¹H NMR (500 MHz, CDCl₃, TMS): δ 9.68 (s, 1H; CHO), 8.11 (d, ²*J* (H,H) = 8.5 Hz, 2H; Ar), 7.52 (d, ²*J* (H,H) = 8.5 Hz, 2H; Ar), 7.47 (s, 1H; CH), 7.35 (t, ²*J* (H,H) = 8.5 Hz, 1H; Ar), 7.27 (d, ²*J* (H,H) = 6.5 Hz, 1H; Ar), 7.00 (t, ²*J* (H,H) = 7.5 Hz, 1H; Ar), 6.93 (d, ²*J* (H,H) = 8.0 Hz, 1H; Ar), 6.41 (s, 1H; OCH); ¹³C NMR (125 MHz, CDCl₃, TMS): δ 189.8, 154.3, 147.8, 146.1, 141.4, 134.2, 132.6, 129.7, 127.5, 123.7, 122.4, 119.5, 117.0, 72.9; $[\alpha]_D^{23} = -61.0$ (c = 1.0, MeOH); HPLC (Daicel CHIRALPAK AS-H, Hexane/*i*-PrOH = 60:40, flow rate 0.6 mL/min, λ = 254 nm): $t_{major} = 37.40$ min, $t_{minor} = 53.40$ min, ee = 95%.

6-Methyl-2-(4-nitro-phenyl)-*2H***-chromene-3-carbaldehyde (Table 2, entry 3):** The title compound was prepared according the typical procedure, as described above in 98% yield. ¹H NMR (500 MHz, CDCl₃, TMS): δ 9.67 (s, 1H), 8.10 (d, 2J (H,H) = 9.0 Hz, 2H; Ar), 7.51 (d, 2J (H,H) = 8.5 Hz, 2H; Ar), 7.42 (s, 1H; CH), 7.15 (dd, 2J (H,H) = 8.5 Hz, 3J (H,H) = 1.5 Hz, 1H; Ar), 7.07, (s, 1H; Ar), 6.83 (d, 2J (H,H) = 8.5 Hz, 1H; Ar), 6.38 (s, 1H; OCH), 2.28 (s, 3H; CH₃); ¹³C NMR (125 MHz, CDCl₃, TMS): δ

189.9, 152.2, 147.7, 146.3, 141.7, 134.9, 132.7, 131.8, 129.8, 127.4, 123.6, 119.4, 116.8, 72.7, 20.3; $\left[\alpha\right]_{D}^{23} = -63.7 \ (c = 1.0, \text{ EtOAc}); \text{ HPLC (Daicel CHIRALPAK AS-H, Hexane/i-PrOH = 60:40, flow rate 0.6 mL/min, } \lambda = 254 \text{ nm}): t_{\text{major}} = 25.39 \text{ min, } t_{\text{minor}} = 37.72 \text{ min, } \text{ee} = 96\%.$

6-Chloro-2-(4-nitro-phenyl)-*2H***-chromene-3-carbaldehyde (Table 2, entry 4):** The title compound was prepared according the typical procedure, as described above in 96% yield. ¹H NMR (500 MHz, CDCl₃, TMS): δ 9.69 (s, 1H; CHO), 8.12 (d, ²*J* (H,H) = 9.0 Hz, 2H; Ar), 7.49 (d, ²*J* (H,H) = 8.5 Hz, 2H; Ar), 7.42 (s, 1H; CH), 7.26-7.29 (m, 2H; Ar), 6.87 (d, ²*J* (H,H) = 8.5 Hz, 1H; Ar), 6.40 (s, 1H; OCH); ¹³C NMR (125 MHz, CDCl₃, TMS): δ 189.6, 152.7, 147.9, 145.4, 139.8, 133.6, 133.5, 128.7, 127.5, 127.2, 123.8, 120.7, 118.4, 73.1; [α]_D²³ = -23.8 (*c* = 1.0, MeOH); HPLC (Daicel CHIRALPAK AS-H, Hexane/EtOH = 60:40, flow rate 0.5 mL/min, λ = 254 nm): t_{major} = 23.38 min, t_{minor} = 42.53 min. ee = 91%.

7-Methoxy-2-(4-nitro-phenyl)-*2H***-chromene-3-carbaldehyde** (**Table 2, entry 5**): The title compound was prepared according the typical procedure, as described above in 64% yield. ¹H NMR (500 MHz, CDCl₃, TMS): δ 9.61 (s, 1H; CHO), 8.13 (d, ²*J* (H,H) = 9.0 Hz, 2H; Ar), 7.53 (d, ²*J* (H,H) = 9.0 Hz, 2H; Ar), 7.42 (s, 1H; CH), 7.19 (d, ²*J* (H,H) = 8.5 Hz, 1H; Ar), 6.55 (dd, ²*J* (H,H) = 8.5 Hz, ³*J* (H,H) = 2.0 Hz, 1H; Ar), 6.47 (d, ³*J* (H,H) = 2.0 Hz, 1H; Ar), 6.41 (s, 1H; OCH), 3.82 (s, 3H; OCH₃); ¹³C NMR (125 MHz, CDCl₃, TMS): δ 189.5, 164.9, 156.3, 147.8, 146.4, 141.6, 131.0, 130.0, 127.5, 123.8, 112.9, 109.4, 102.0, 73.2, 55.6; $[\alpha]_D^{23} = -194.6$ (c = 1.0, MeOH); HPLC (Daicel CHIRALPAK AS-H, Hexane/EtOH = 60:40, flow rate 0.6 mL/min, λ = 254 nm): $t_{major} = 21.75$ min, $t_{minor} = 25.74$ min. ee = 86%.

8-Methoxy-2-(4-nitro-phenyl)-2*H***-chromene-3-carbaldehyde (Table 2, entry 6):** The title compound was prepared according the typical procedure, as described above in 98% yield. ¹H NMR (500 MHz, CDCl₃, TMS): δ 9.69 (s, 1H; CHO), 8.08 (d, ²*J* (H,H) = 9.0 Hz, 2H; Ar), 7.51 (d, ²*J* (H,H) = 8.5 Hz, 2H; Ar), 7.45 (s, 1H; CH), 6.93-6.99 (m, 2H; Ar), 6.87 (dd, ²*J* (H,H) = 7.5 Hz, ³*J* (H,H) = 1.5 Hz, 1H; Ar), 6.48 (s, 1H; OCH), 3.88 (s, 3H; OCH₃); ¹³C NMR (125 MHz, CDCl₃, TMS): δ 190.0, 148.3, 147.7, 146.1, 143.3, 141.5, 133.0, 127.1, 123.6, 122.2, 121.3, 120.3, 116.2, 72.8, 56.2; $[\alpha]_D^{23} = 105.9$ (c = 2.0, MeOH); HPLC (Daicel CHIRALPAK AS-H, Hexane/EtOH = 60:40, flow rate 0.6 mL/min, λ = 254 nm): t_{major} = 16.84 min, t_{minor} = 25.07 min. ee = 90%.

6-Methoxy-2-(4-nitro-phenyl)-*2H***-chromene-3-carbaldehyde** (**Table 2, entry 7**): The title compound was prepared according the typical procedure, as described above in 95% yield. ¹H NMR (500 MHz, CDCl₃, TMS): δ 9.69 (s, 1H, CHO), 8.10 (d, ²*J* (H,H) = 8.5 Hz, 2H; Ar), 7.50 (d, ²*J* (H,H) = 8.5 Hz, 2H; Ar), 7.44 (s, 1H; CH), 6.91 (dd, ²*J* (H,H) = 9.0 Hz, ³*J* (H,H) = 3.0 Hz, 1H; Ar), 6.87 (d, ²*J* (H,H) = 9.0 Hz, 1H; Ar), 6.78 (d, ³*J* (H,H) = 3.0 Hz, 1H; Ar), 6.36 (s, 1H; OCH), 3.78 (s, 3H; OCH₃); ¹³C NMR (125 MHz, CDCl₃, TMS): δ 189.9, 154.6, 148.2, 147.7, 146.1, 141.6, 133.3, 127.5, 123.6, 120.3, 120.0, 117.9, 113.1, 72.5, 55.7; $[α]_D^{23} = -11.0$ (c = 1.0, EtOAc); HPLC (Daicel CHIRALCEL OD-H, Hexane/EtOH = 60:40, flow rate 0.6 mL/min, λ = 254 nm): $t_{major} = 12.88$ min, $t_{minor} = 14.31$ min. ee = 94%.

6-Chloro-2-(2-nitro-phenyl)-*2H***-chromene-3-carbaldehyde (Table 2, entry 8):** The title compound was prepared according the typical procedure, as described above in 82% yield. ¹H NMR (500 MHz, DMSO-d⁶): δ 9.71 (s, 1H; CHO), 7.98 (s, 1H; CH), 7.94-7.96 (m, 1H; Ar), 7.58-7.61 (m, 3H; Ar), 7.32-7.35 (m, 2H; Ar), 6.80 (s, 1H; OCH), 6.74 (d, ²*J* (H,H) = 8.5 Hz, 1H; Ar); ¹³C NMR (125 MHz, DMSO-d⁶): δ 200.9, 161.7, 158.7, 150.8, 143.1, 143.1, 142.1, 140.7, 140.3, 138.9, 138.7, 136.1, 134.8, 131.5, 128.4, 78.6; $[\alpha]_D^{23} = +176.7$ (c = 1.0, EtOAc); HPLC (Daicel CHIRALCEL OD-H, Hexane/EtOH = 60:40, flow rate 0.6 mL/min, λ = 254 nm): t_{major} = 13.88 min, t_{major} = 24.77 min, ee = >99%.

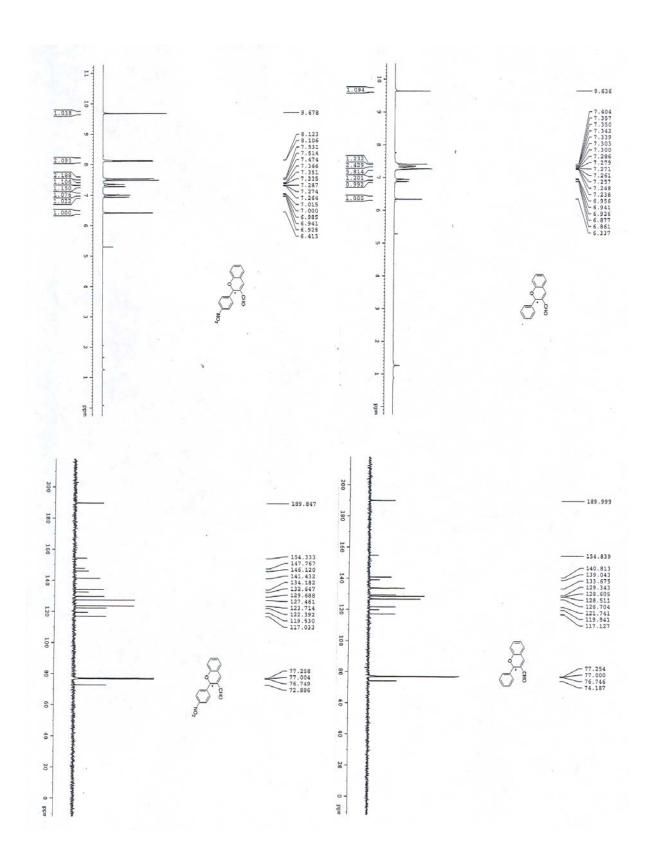
6-Methoxy-2-phenyl-*2H***-chromene-3-carbaldehyde** (**Table 2, entry 9**): The title compound was prepared according the typical procedure, as described above in 97% yield. ¹H NMR (500 MHz, CDCl₃, TMS): δ 9.65 (s, 1H; CHO), 7.36 (s, 1H; CH), 7.33 (m, 2H; Ar), 7.25 (m, 3H; Ar), 6.86 (d, ²*J* (H,H) = 8.5 Hz, 1H; Ar), 6.80 (d, ²*J* (H,H) = 8.5 Hz, 1H; Ar), 6.76 (s, 1H; Ar), 6.29 (s, 1H; OCH), 3.76 (s, 3H; OCH₃); ¹³C NMR (125 MHz, CDCl₃, TMS): δ 190.0, 154.2, 148.7, 140.9, 138.8, 134.3, 128.5, 128.4, 126.7, 120.3, 119.8, 117.9, 112.9, 73.8, 55.7; $[\alpha]_D^{23} = +17.0$ (c = 1.0, CHCl₃); HPLC (Daicel CHIRALCEL OD-H, Hexane/*i*-PrOH = 60:40, flow rate 0.5 mL/min, $\lambda = 254$ nm): t_{major} = 13.32 min, t_{minor} = 17.34 min, ee = 87%.

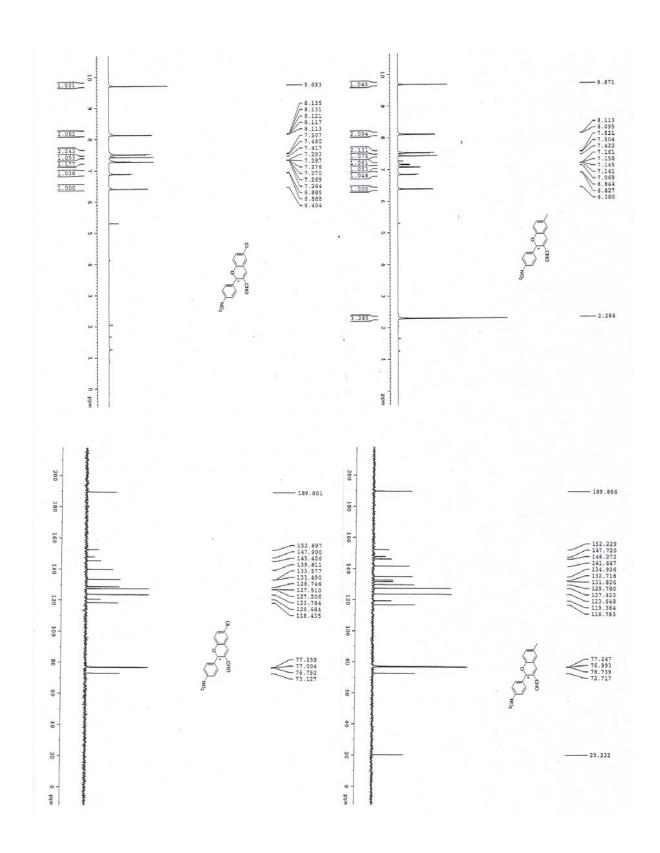
6-Chloro-2-(4-methoxy-phenyl)-2*H***-chromene-3-carbaldehyde** (**Table 2, entry 10**): The title compound was prepared according the typical procedure, as described above in 53% yield. ¹H NMR (500 MHz, CDCl₃, TMS): δ 9.63 (s, 1H; CHO), 7.33 (s, 1H; CH), 7.19-7.25 (m, 3H; Ar), 6.76-6.81 (m,

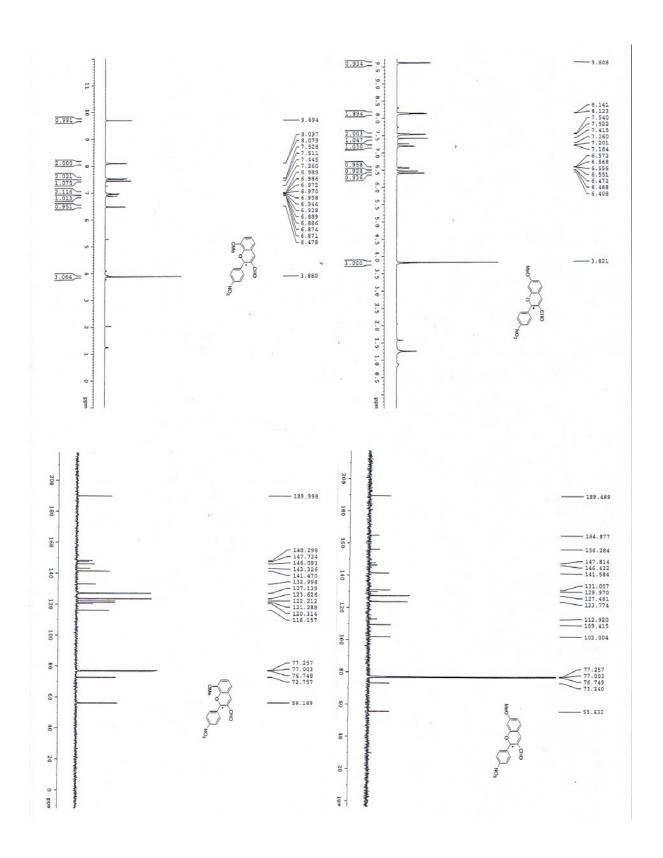
3H; Ar), 6.26 (s, 1H; OCH), 3.74 (s, 3H; OCH₃); ¹³C NMR (125 MHz, CDCl₃): δ 189.7, 160.0, 153.1, 139.0, 134.6, 133.0, 130.5, 128.3, 126.4, 121.2, 118.6, 114.0, 74.2, 55.2; $\left[\alpha\right]_D^{23} = +$ 27.7 (c = 1.0, MeOH); HPLC (Daicel CHIRALPAK AS-H, Hexane/EtOH = 60:40, flow rate 0.5 mL/min, $\lambda = 254$ nm): $t_{major} = 18.58$ min, $t_{minor} = 24.78$ min, ee = 75%.

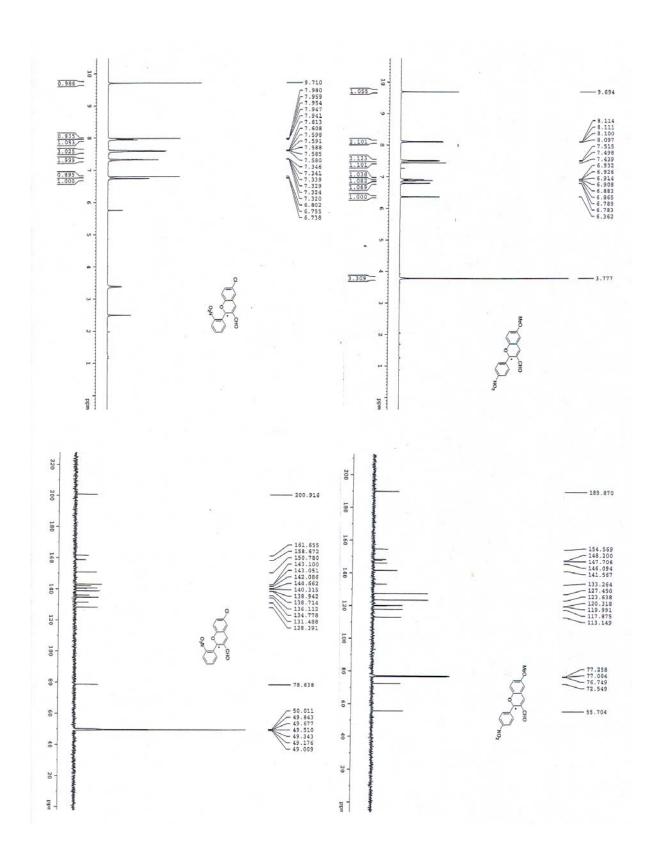
6-Chloro-2-methyl-*2H***-chromene-3-carbaldehyde** (**Table 2, entry 11**): The title compound was prepared according the typical procedure, as described above in 67% yield. ¹H NMR (500 MHz, CDCl₃, TMS): δ 9.56 (s, 1H; CHO), 7.25 (dd, ²*J* (H,H) = 8.5 Hz, ³*J* (H,H) = 2.5 Hz, 1H; Ar), 7.20 (d, ³*J* (H,H) = 2.5 Hz, 1H; Ar), 7.12 (s, 1H; CH), 6.82 (d, ²*J* (H,H) = 8.5 Hz, 1H; Ar), 5.41 (q, ²*J* (H,H) = 6.5 Hz, 1H; OCH), 1.35 (d, ²*J* (H,H) = 6.5 Hz, 3H; CH₃); ¹³C NMR (125 MHz, CDCl₃, TMS): δ 189.8, 152.8, 138.6, 137.0, 132.8, 128.3, 126.4, 121.0, 118.7, 70.0, 19.8; $[\alpha]_D^{23} = -27.4$ (c = 1.0, MeOH); HPLC (Daicel CHIRALPAK AS-H, Hexane/*i*-PrOH = 90:10, flow rate 0.5 mL/min, λ = 254 nm): t_{major} = 12.31 min, t_{minor} = 16.47 min, ee = 82%.

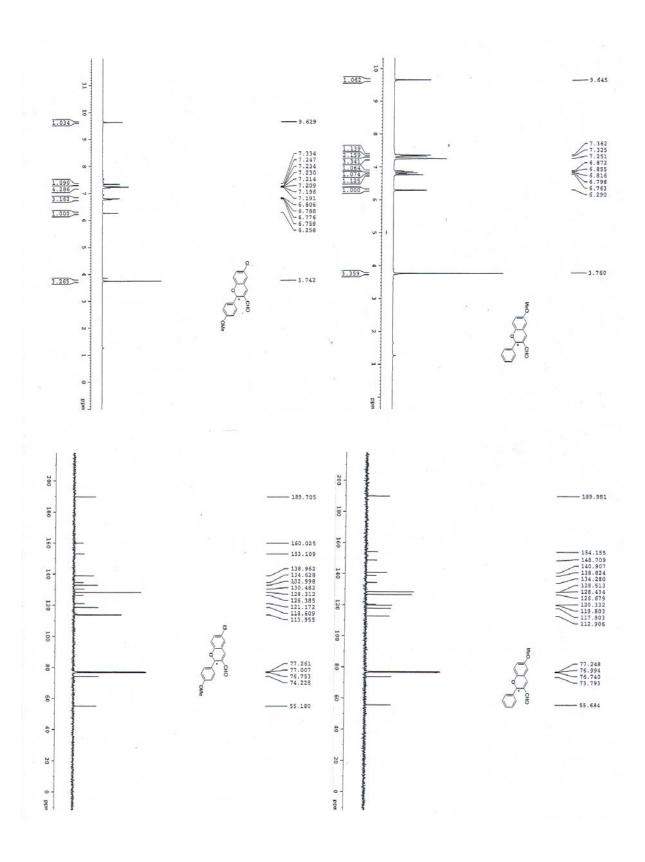
6-Methoxy-2-methyl-*2H***-chromene-3-carbaldehyde** (**Table 2, entry 12**): The title compound was prepared according the typical procedure, as described above in 84% yield. ¹H NMR (500 MHz, CDCl₃, TMS): δ 9.54 (s, 1H; CHO), 7.26 (s, 1H; CH), 6.88 (dd, ²*J* (H,H) = 9.0 Hz, ³*J* (H,H) = 3.0 Hz, 1H; Ar), 6.81 (d, ²*J* (H,H) = 8.5 Hz, 1H; Ar), 6.73 (d, ³*J* (H,H) = 3.0 Hz, 1H; Ar), 5.36 (q, ²*J* (H,H) = 6.5 Hz, 1H; OCH), 3.78 (s, 3H; OCH₃), 1.32 (d, ²*J* (H,H) = 6.5 Hz, 3H; CH₃); ¹³C NMR (125 MHz, CDCl₃, TMS): δ 189.9, 154.1, 148.2, 140.1, 136.8, 120.2, 119.5, 118.0, 112.8, 69.5, 55.8, 19.4; [α]_D²³ = + 10.7 (c = 1.0, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/i-PrOH = 70:30, flow rate 0.5 mL/min, λ = 254 nm): t_{major} = 16.26 min, t_{minor} = 17.61 min, ee = 85%.

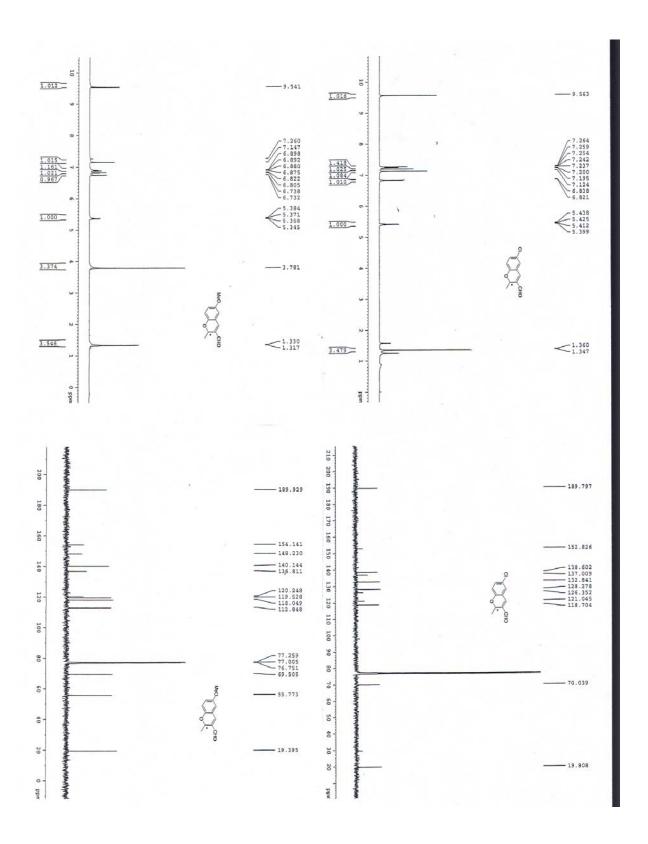




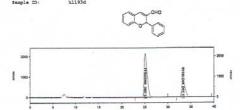








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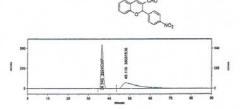


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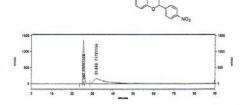
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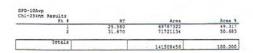


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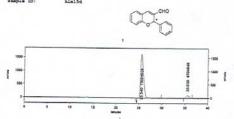
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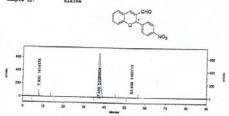
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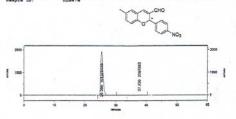
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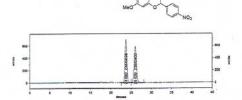
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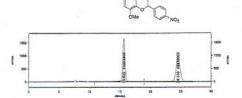
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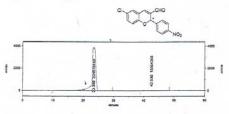
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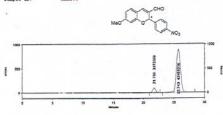
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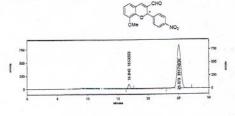
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Sample ID: himsto



SPD-10Avp Ch1-25¢nm Kesults Pk #	87	Area	Area 5
1 2	21.750 25.740	3173399 42491016	93.049
Totals	8	45654415	100.000

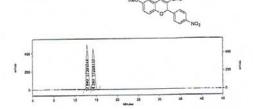
University Of New Mexico Department of Chemistry

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Method Name:
Data File:
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4/9/2006 12:00:59 PM Data Printed: 05/03/2006 10:19:20 AM
Shaple 2D:
AhaEE

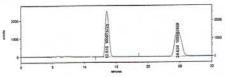


SPD-10Avp Ch1-254nm Results PK #	2.7	Area	Area 1
1 2	12.840	17301244	50.018 49.982
Totals		34590364	100.000

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Method Name: C:\EIStart\Projecta\Default\Method\Naclio2.met
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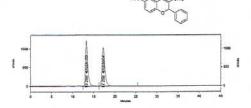




SPD-10Avp Ch1-254nm Results 2k #	RT	Area	Aces %
1	13.510	100915725	49.774
2	24.630	105987869	51.226
Totals		206903594	100.000

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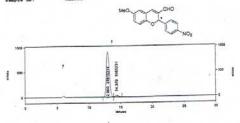
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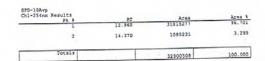


SPS-10Avp Ch1-254nm Results Pk #	RT	Area	Area N
1 2	13.260 17.250	47029150 47058604	49.984 50.016
Totals		94086754	100.000

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Method Mane: C:\Esstart\Projecta\Default\Method\Racidi.met
C:\Esstart\Projecta\Method\Racidi.met
C:\Esstart\Projecta\Method\Racidi.det
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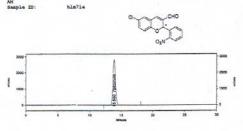


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Mathod Name: C:\EIStart\Projects\Default\Method\NaoLi09.met

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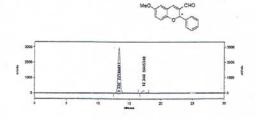
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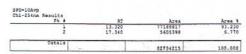


SPD-10Avp Ch1-254nm Results 9k #	87	Area	Area 1
1	13.880	79859578	100.000
Totals		79859578	100.000

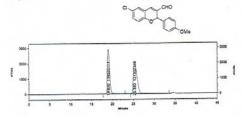
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AM
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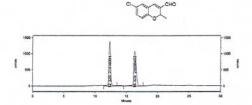
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Philb?**



SPD-10Avp Ch1-254nm Results Pk #	75	Area	Area
1 2	19.020 25.320	114520111 131337346	46.58 53.42
Totals		245857457	. 100.00

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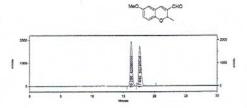
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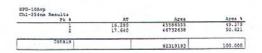


SPO-10Avp Ch1-254nm Results Pk #	RT	Area	Area 1
1 2	12.320 16.320	27610314 29656402	49.214 51.786
Totals		57266716	100.000

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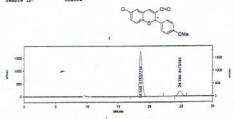
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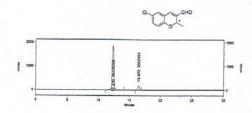
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AM Sample DD: Masie



SPD-10Avp Chi-254nm Results Pk *	27	Area	Area 1
1 2	18.580	37537718 8479181	87.156 12.844
Totals	10.00	66016899	100.000

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Method Name: C:\EIStart\Projecta\Default\Method\NaoLiO2.met
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Bample ID: hlm59e



SPD-10Avp Ch1-254nm Results Pk #	RT	Area	Area 1
1 2	12.310 16.470	36229268 3562154	91.048 8.952
Totals		39791422	100.000

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Method Name: C:\EIStart\Projects\Default\Sethod\RaoLiO9.met Data File: C:\EIStart\Projects\Methang\hisfol.dat Date Acquired: 4/19/2006 12:48:17 FM Date Printed: 05/03/2006 10:29:41

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