

First Total Synthesis of Protoapigenone and its Analogs as Potent Cytotoxic Agents

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Supporting Information

Contents:

High resolution mass spectral data for target compounds.

HPLC analysis of the target compounds.

High resolution mass spectral data for target compounds

Compound	High resolution mass spectra		
	Chemical formula	Calculated	Measured
1	$C_{15}H_{10}O_6 + H$	287.0556	287.0556
14	$C_{17}H_{14}O_7 + H$	331.0818	331.0812
15	$C_{17}H_{14}O_6 + H$	315.0869	315.0858
16	$C_{18}H_{16}O_6 + H$	329.1025	329.1011
20	$C_{15}H_{10}O_4 + H$	255.0657	255.0650
21	$C_{16}H_{12}O_4 + H$	269.0814	269.0811
22	$C_{15}H_{10}O_5 + H$	271.0606	271.0590
23	$C_{16}H_{12}O_5 + H$	285.0763	285.0758
24	$C_{16}H_{12}O_6 + H$	301.0712	301.0712
26	$C_{19}H_{12}O_4 + H$	305.0814	305.0806
27	$C_{20}H_{14}O_4 + H$	319.0970	319.0967
29	$C_{20}H_{14}O_4 + H$	319.0970	319.0974

HPLC analysis of the final compounds.

Compound purities were determined by two diverse HPLC conditions.

HPLC conditions No.1:

System: Shimadzu SLC 10A system controller,

Shimadzu LC-20A performance liquid chromatography

Detector: Shimadzu SPD-M10A at 254 nm

Column: Alltech 4.6 mm x 250 mm C-18

Solvents:

A: [MeCN/H₂O 70/30] 90%

B: [H₂O] 10%

Mode: Isocratic system

Flow rate: 1 mL / min

HPLC conditions No.2:

System: Shimadzu SLC 10A system controller,

Shimadzu LC-20A performance liquid chromatography

Detector: Shimadzu SPD-M10A at 254 nm

Column: Alltech 4.6 mm x 250 mm C-18

Solvents:

A: [MeOH] 70%

B: [H₂O] 30%

Mode: Isocratic system

Flow rate: 1 mL / min

HPLC analysis of the final compounds

Compound	HPLC condition No.1		HPLC condition No.2	
	Retention time (min)	Purity (area %)	Retention time (min)	Purity (area %)
1	3.40	100.0	5.00	99.3
14	4.57	99.2	8.82	98.4
15	2.92	98.2	4.83	98.1
16	4.28	98.7	6.21	99.3
20	3.40	98.9	4.69	98.3
21	4.91	98.8	5.97	98.6
22	4.16	98.5	6.48	98.0
23	7.21	100.0	9.45	100.0
24	4.30	99.4	7.73	100.0
26	5.14	99.5	9.97	98.3
27	15.2	98.4	15.21	98.4
29	8.26	98.8	13.93	98.2