



**Figure S1.** LC-MS/MS chromatograms of purified CTX3C (a) and the product (b) of acid-catalyzed epimerization reaction described in the section 2.2. *Reference Toxins*.

**Table S1.** The specimens used for this study.

Specimen no.	Collection site	Species	SL <sup>1</sup> (cm)	BW <sup>2</sup> (g)
151001	Philippines	<i>Sphyræna</i> sp.	33	281
151002	Philippines	<i>Lethrinus lentjan</i>	22	274
151003	Philippines	<i>Scarus rivulatus</i>	21	290
151004	Philippines	<i>Siganus virgatus</i>	16	130
151005	Philippines	<i>Siganus virgatus</i>	13	86
151006	Philippines	<i>Epinephelus megachir</i>	20	201
151007	Philippines	<i>Variola albimarginata</i>	21	217
151012	Taiwan	<i>Variola louti</i>	37	1475
151013	Taiwan	<i>Variola louti</i>	31	760
151014	Taiwan	<i>Variola albimarginata</i>	24	430
151015	Taiwan	<i>Variola albimarginata</i>	26	455
151016	Taiwan	<i>Epinephelus fuscoguttatus</i>	27	800
151027	Taiwan	<i>Lutjanus</i> sp.	25	680
151017	Thailand	<i>Lutjanus russellii</i>	23	387
151018	Thailand	<i>Lutjanus russellii</i>	23	350
151022	Thailand	<i>Lutjanus johnii</i>	44	1927
151026	Thailand	<i>Lutjanus johnii</i>	-	
151020	Thailand	<i>Epinephelus areolatus</i>	21	253
151019	Thailand	<i>Epinephelus sexfasciatus</i>	23	411
151021	Thailand	<i>Epinephelus sexfasciatus</i>	25	388
151023	Thailand	<i>Epinephelus</i> sp.	39	1374
151024	Thailand	<i>Scomberomorus commerson</i>	-	
151025	Thailand	<i>Scomberomorus commerson</i>	-	
151028	Fiji	<i>Lutjanus argentimaculatus</i>	41	1,295
151029	Fiji	<i>Lutjanus monostigma</i>	27	640
151041	Fiji	<i>Lutjanus fulviflamma</i>	17	120
151042	Fiji	<i>Lutjanus fulviflamma</i>	21	205
151043	Fiji	<i>Lutjanus fulviflamma</i>	19	195
151044	Fiji	<i>Lutjanus bohar</i>	38	2,150
151033	Fiji	<i>Lutjanus</i> sp.	21	635
151045	Fiji	<i>Epinephelus fuscoguttatus</i>	-	1,515
151030	Fiji	<i>Plectropomus leopardus</i>	31	725
151031	Fiji	<i>Plectropomus leopardus</i>	27	455
151032	Fiji	<i>Plectropomus leopardus</i>	22	210
151038	Fiji	<i>Plectropomus leopardus</i>	33	750
151039	Fiji	<i>Plectropomus leopardus</i>	-	1,575
151034	Fiji	<i>Anyperodon leucogrammicus</i>	-	515
151035	Fiji	<i>Variola louti</i>	-	1,780
151040	Fiji	<i>Variola louti</i>	37	1,465
151036	Fiji	<i>Upeneus</i> sp.	-	360
151037	Fiji	<i>Plectorhynchus chaetodontoides</i>	22	395
151046	Fiji	<i>Gymnothorax javanicus</i>	86	1,845

<sup>1</sup> standard length, <sup>2</sup> body weight

**Table S2.** CTX analogs and monitor ions on LC-MS/MS analysis.

Analog	m/z
4-hydroxy-7-oxo-CTX1B*	1167.6
7-oxo-CTX1B*	1149.6
7-hydroxy-CTX1B*	1151.6

CTX1B	1133.6
M-seco-CTX4A/4B*	1101.6
52- <i>epi</i> -54-deoxyCTX1B	1117.6
54-deoxyCTX1B	1117.6
CTX4A	1083.6
CTX4B	1083.6
2,3,51-trihydroxy-CTX3C*	1095.6
A-seco-51-hydroxy-CTX3C*	1081.6
M-seco-CTX3C*	1063.6
2,3-dihydroxyCTX3C*	1079.6
51-hydroxyCTX3C	1061.6
2-hydroxyCTX3C*	1063.6
2-oxo-CTX3C*	1063.6
49- <i>epi</i> CTX3C	1045.6
CTX3C	1045.6

\* no reference was present and deduced from relative retention times described in Yasumoto et al. (2000).