SUPPLEMENTARY MATERIAL

Cytotoxicity of an Unprecedented Brominated Oleanolide and a New Furoceramide from the Cameroonian Spice, *Echinops giganteus*.

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Abstract

A preliminary study on *Echinops giganteus* (Asteraceae) showed that the methanolic extract has interesting cytotoxicities against a panel of cancer cell lines. From this extract, a lignan, a flavonoid and a polyacetylenic thiophene identified were 3 folds less cytotoxic than the extract. In the search of the metabolites responsible for the bioactivity, a new harvested *E. giganteus* was subjected to a phytochemical study using chromatographic methods. In the course of the work, two new compounds: a brominated oleanolide (1) and a tetrahydrofurano-ceramide (2) were obtained along with \(\beta\)-amyrin acetate (3), 2-(penta-1,3-diynyl)-5-(4-hydroxybut-1-ynyl)-thiophene (4), 2-(penta-1,3-diynyl)-5-(3,4-dihydroxybut-1-ynyl)-thiophene (5) and 4-hydroxy-2,6-di-(3′,4′-dimethoxyphenyl)-3,7-dioxabicyclo-(3.3.0)octane (6). Their structures were determined on the basis of NMR spectroscopy and mass spectrometry data in conjunction with those reported in the literature. The cytotoxicity of 1, 2, and 5 was evaluated by employing resazurin assay against a panel of cancer cell lines with \(IC_{50}\) values in range 6.12±0.46 - 46.96±3.61 \(\mu\)M.

\textbf{Keywords:} Asteraceae; *Echinops giganteus*; brominated triterpene; furoceramide; cytotoxicity
Figure S1 Mass spectrum of compound 1

Figure S2 $^1$H NMR spectrum of compound 1
Figure S3 $^{13}$C NMR spectrum of compound 1

Figure S4 COSY spectrum of compound 1
Figure S5 HSQC spectrum of compound 1

Figure S6 HMBC spectrum of compound 1
Figure S7 NOESY spectrum of compound 1
Figure S8 $^1$H NMR spectrum of compound 2

Figure S9 $^{13}$C NMR spectrum of compound 2
Figure S10 COSY spectrum of compound 2

Figure S11 HSQC spectrum of compound 2
Figure S12 HMBC spectrum of compound 2

Figure S13 NOESY spectrum of compound 2
Figure S14 COSY, HMBC and NOESY correlations of compound 1

Figure S15 COSY, HMBC and NOESY correlations of compound 2

Figure S16 Position of the double bond in compound 2
Figure S17 Proposal of the biosynthetic pathway of compound 1 (Thimmappa et al., 2014)
Figure S18 ESI Tandem MS spectrum of compound 2, m/z 678.6 [M+H]^+
Figure S19 Fragmentation pattern of compound 2 (part 1)
Figure S20 Fragmentation pattern of compound 2 (part 2)
Reference

Thimmappa R, Geisler K, Louveau T, O’Maille P, Osbourn A. 2014. Triterpene Biosynthesis in Plants. Annu. Rev. Plant Biol. 65:225–257