

Structure Elucidation of Two Physalins Isolated from *Physalis angulata* L. (Solanaceae).

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The identification and the crystal structure determination of two steroids extracted from a medicinal plant named *Physalis angulata* L., collected in the D.R. Congo and whose antimalarial activity is known [1-3], have been fully realized by the X-ray diffraction analysis. Whole plants were then dried at room temperature in the laboratory protected from sunlight. Crude extract, obtained by maceration of the powdered plants in CH₂Cl₂ at room temperature, was filtered and evaporated to dryness under reduced pressure at 40 °C. Two products, X and Y, were isolated by bio-guided fractionation of the extract using Si-60 open chromatography followed by preparative HPLC. The powders of both samples were separately dissolved in an acetone:DMSO mixture. Plate-like crystals, suitable for X-ray diffraction measurement, were obtained in DMSO after the slow evaporation. For the compound X, diffraction data were collected on a diffractometer equipped with a Bruker-AXS SMART 6000 CCD detector and integrated by the program SAINT. A multi-scan absorption was performed by the program SADABS. For the compound Y, diffraction data collected on a Supernova diffractometer were analyzed with CrysAlisPRO software, and an empirical absorption correction based on the crystal morphology was performed. The structures were, in both cases, solved by direct methods using the SHELXS program and refined according to the least-squares methods to R-values of 0.0490 for X and 0.0563 for Y. Both crystals belong to the monoclinic space group *P2₁* and show disorder. The asymmetric unit of the compound X comprises two physalin B molecules, one acetone molecule, and one water molecule with occupancy 0.22. The asymmetric unit of Y consists of one acetone molecule and two disordered physalin molecules, each corresponding to co-physalin B–5β,6β-epoxyphysalin B with occupancies of 0.76 and 0.57 for physalin B and 0.24 and 0.43 for 5β,6β-epoxyphysalin B. In both crystal structures, the physalin molecules in the asymmetric unit are strongly hydrogen-bonded through two O–H...O contacts, leading to dimers, which are used as building blocks during the crystallization of both compounds.

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