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NOTE

Triterpenes and Sterol from *Tabernaemontana pandacaqui* Poir.

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Chemical investigation of the dichloromethane extract of *Tabernaemontana pandacaqui* Poir. flowers has led to the isolation of α-amyrin acetate (**1**), a mixture of α-amyrin acetate (**1**), lupeol acetate (**2**) and β-amyrin acetate (**3**) in about 4:2:1 ratio, α-amyrin fatty acid ester (**4**), a mixture of ursolic acid (**5**) and oleanolic acid (**6**) in about 1:1 ratio, and β-sitosterol (**7**). The structures of compounds **1-7** were identified by comparison of their NMR data with those reported in the literature.

Keywords: *Tabernaemontana pandacaqui* Poir., Apocynaceae, α-Amyrin acetate, Lupeol acetate, β-Amyrin acetate, Ursolic acid.

Tabernaemontana pandacaqui Poir., commonly known as banana bush and locally known as pandakaking-puti and kampupot, is found in thickets at low altitudes throughout the Philippines [1]. An earlier study [2] reported that the crude alcoholic extract from the dried leaves of *T. pandacaqui* afforded α-amyrin acetate. Another study [3] reported that the stems of *T. pandacaqui* showed significant anti-inflammatory, antipyretic and antinociceptive properties which were attributed to the alkaloids found in the stems. Voacangine, a major component of *T. pandacaqui* possessed analgesic and hypothermic activities and revealed surface anesthesia [4] in mice. The stems of *T. pandacaqui* crude alkaloidal fraction exerted a hypotensive activity and bradycardiac response [5], and possessed a CNS depressant activity [6] in rats. Another study reported the isolation of coronaridine from *T. pandacaqui* [7].

In this study, the flowers of *T. pandacaqui* afforded α-amyrin acetate (**1**), a mixture of α-amyrin acetate (**1**), lupeol acetate (**2**) and β-amyrin acetate (**3**), α-amyrin fatty acid ester (**4**), a mixture of ursolic acid (**5**) and oleanolic acid (**6**), and β-sitosterol (**7**). To the best of our knowledge, this is the first report on the isolation of compounds **2-7** from *T. pandacaqui*.

NMR spectra were recorded on a Varian VNMRS spectrometer in $CDCl_3$ at 600 MHz for ^1H NMR and 150 MHz for ^{13}C NMR spectra. Column chromatography was performed, with silica gel 60 (70-230 mesh). Thin layer chromatography was performed with plastic backed plates coated with silica gel F_{254} and the plates were visualized by spraying with vanillin/H_2SO_4 solution followed by warming.

Sample collection: The flowers of *T. pandacaqui* were collected from Pampanga, Philippines in March 2016. It was identified as *Tabernaemontana pandacaqui* Poir. at the Botany Division, Philippine National Museum.

General isolation procedure: A glass column 12 inches in height and 0.5 inch internal diameter was packed with silica gel. The crude extracts were fractionated by silica gel chromatography using increasing proportions of ethyl acetate in petroleum ether (2.5 % increment) as eluents. Five milliliters fractions were collected. Fractions with spots of same R_f values were combined and rechromatographed in appropriate solvent systems until TLC pure isolates were obtained. All fractions were monitored by thin layer chromatography. Rechromatography and final purifications were conducted using Pasteur pipettes as columns. Two milliliter fractions were collected.

Isolation of chemical constituents of *T. pandacaqui* flowers: The air-dried flowers (66.00 g) were ground in a blender, soaked in CH_2Cl_2 for three days and then filtered. The filtrate was concentrated under vacuum to afford a crude extract (1.66 g) which was chromatographed by gradient elution with petroleum ether, followed by increasing amounts of ethyl acetate at 2.5 % increments by volume. The 2.5 % ethyl acetate in petroleum ether fraction was rechromatographed using 2.5 % EtOAc in petroleum ether to afford a mixture of compounds **1-3** (3.7 mg) after washing with petroleum ether. The 5 % EtOAc in

petroleum ether fraction was rechromatographed (2 ×) using 2.5 % EtOAc in petroleum ether to afford compound **1** (1.3 mg) after washing with petroleum ether. The 7.5 % EtOAc in petroleum ether fraction was rechromatographed (2 ×) using 5 % EtOAc in petroleum ether to afford compound **4** (12 mg) after washing with petroleum ether. The 12.5 % EtOAc in petroleum ether fraction was rechromatographed (2 ×) using 15 % EtOAc in petroleum ether to afford compound **7** (4.3 mg) after washing with petroleum ether. The 20 % EtOAc in petroleum ether fraction was rechromatographed (3 ×) using 20 % EtOAc in petroleum ether to afford a mixture of compounds **5-6** (2.8 mg) after washing with petroleum ether.

Silica gel chromatography of the dichloromethane extracts of the flowers of *T. pandacaqui* yielded compounds **1-7**. The NMR spectra of compound **1** are in accordance with data reported in the literature for α-amyrin acetate [8]; compound **2** for lupeol acetate [9]; compound **3** for β-amyrin acetate [8]; compound **4** for α-amyrin fatty acid ester [10]; compound **5** for ursolic acid [11] and compound **6** for oleanolic acid [11], and compound **7** for β-sitosterol [12]. The 4:2:1 ratio of the mixture of α-amyrin acetate (**1**), lupeol acetate (**2**) and β-amyrin acetate (**3**) was deduced from the intensities and integrations of the ^{1}H NMR resonances for olefinic protons of compound **1** at δ 5.11 (t, J = 3.6 Hz, H-12) [11,13], δ 5.16 (t, J = 3.6 Hz, H-12) of compound **3** [11,13], and δ 4.66 (d, J = 2.4 Hz, H_a-29), and 4.55 (d, J = 1.2 Hz, H_b-29) of compound **2** [13]. The 1:1 ratio of the mixture of ursolic acid and oleanolic acid was deduced from the integrations of the ^{1}H NMR resonances for olefinic protons of **6** at δ 5.26 (t, J = 3.6 Hz, H-12) and compound **5** at δ 5.23 (t, J = 3.6 Hz, H-12) and the H-18 proton of compound **6** at δ 2.81 [14].

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