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DIBUTYL PHTHALATE, A SECONDARY METABOLITE FROM *Mimusops elengi*

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Mimusops elengi, commonly called Bakul, is a medicinal plant belonging to the family Sapotaceae. It is a small to large evergreen tree up to 15 m in height. All parts of the tree have medicinal properties. The bark, flowers, and fruits are acrid, astringent, cooling, and anthelmintic [1]. The bark is used as a tonic [1–4], febrifuge, and as a gargle for odontopathy, inflammation, and bleeding of gums [1]. The powder of dried flower is a brain tonic and is useful as snuff to relieve cephalalgia. Young twigs are used for cleaning teeth [2]. It is antipyretic and increases fertility in women [1, 3]. It is also useful in urethrorrhea, cystorrhea, diarrhea, and dysentery. Flowers are used for preparing lotion for wounds and ulcers [3]. The unripe fruit is used as a masticatory and helps to fix loose teeth. Seeds are used for preparing suppositories to treat constipation, especially in children [2–4]. Ripe fruit pulp is useful in chronic dysentery [3, 4]. Leaves are used in snake bite [3, 4].

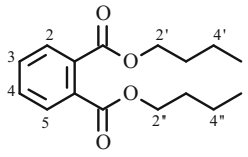
In our continuing research on bioactive metabolites of plant origin, we have isolated dibutyl phthalate (**1**) for the first time from the bark of *Mimusops elengi*. It has been also isolated from other plants, marine algae, bacteria, and fungi [5–8]. The cambridge Crystallographic Database has been searched for single-crystal analysis. So far, the molecular structure for compound **1** has not been determined; however, the manganese complex of the same has been studied by D. Moon and M. SooLah [9].

General Comments. Instrument: HPLC (Shimadzu 2010, LC Solution); LC-MS (Shimadzu 2010, LC-MS).

Solution. Instrumental conditions: HPLC column YMC ODC – A (5 cm × 4.5 cm × 5μ), A = 0.1% TFA in H_2O, B = 0.1% TFA in acetonitrile, gradient = 5% B to 90% B in 8 min hold for 2 min, injection volume 3 μL; column oven temperature 30°C; flow rate = 1.0 mL/min.

LC-MS conditions: scan mode = positive; CDC temperature 250°C; heater block 200°C, detector voltage 1.5 kV; scan speed 1000 amu/sec; nebulizing gas flow 1.5 L/min; ionization mode ESI (atmospheric pressure ionization).

Extraction and Isolation. Plant material used in this study was purchased from the market. It was authenticated at the Agharkar Research Institute, Pune, India. Its authentication number is AHMA S/B-065. Air shade dried powdered bark material (300 g) was extracted with a Soxhlet extractor using different solvents such as hexane, chloroform, ethanol, and methanol for 18 hours. The solvents were removed under reduced pressure to get the respective extracts. The chloroform extract (1.33%, 4.018 g) was further purified. Broad fractionation of the crude chloroform extract (4.0 g) was carried out using gradient polarity solvents on silica gel (60–120 mesh, 160 g) to get ten fractions. Fractions were monitored by thin layer chromatography. Fractions 3 and 4 (toluene, 100%, toluene–ethyl acetate, 75:25, 1, 2 g) were mixed together for repeated column chromatography using gradient polarity solvents on silica gel (60–120 mesh, 60 g), and a total of nine fractions was collected. The fractions were monitored by thin-layer chromatography. The toluene–ethyl acetate fraction (90:10, 180 mg) was further fractionated using hexane–ethyl acetate with increasing percentage of ethyl acetate over silica gel (60–120 mesh, 50 g) to obtain an impure compound **1**, purified by preparative TLC.



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The compound **1** isolated is a colorless transparent liquid. LC-MS of the compound exhibited a molecular ion peak at *m/z* 279 on positive mode, which matches the molecular formula $C_{16}H_{22}O_4$.

IR (Nujol, ν, cm^{-1}): 1728 (ester carbonyl), 1600, 1579 (Ar). ^1H NMR (500 MHz, $CDCl_3$, δ, ppm, J/Hz): 0.98 (6H, t, J = 5, CH_3-5′ and CH_3-5″), 1.47 (4H, m, CH_2-4′ and CH_2-4″), 1.74 (4H, m, CH_2-3′ and CH_2-3″), 4.33 (4H, t, J = 5, CH_2-2′ and CH_2-2″), 7.73 (2H, dd, J = 10 and 5, H-2 and H-5), 7.56 (2H, dd, J = 10 and 5, H-3 and H-4).

^{13}C NMR (125 MHz, $CDCl_3$, δ, ppm): 13.70 (q, C-5′ and C-5″), 19.18 (t, C-4′ and C-4″), 30.59 (t, C-3′ and C-3″), 65.55 (t, C-2′ and C-2″), 128.84 (d, C-2 and C-5), 130.89 (d, C-3 and C-4), 132.35 (s, C-1 and C-6), 167.69 (s, C-1′ and C-1″).

Saponification of the isolate led to the emergence and increase of new peaks for the hydrolytic products and decrease of the molecular ion peak for the compound. MS chromatograms for the hydrolytic products confirm the isolated compound as dibutyl phthalate.

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