

mol, 9.25 g) dissolved in acetone, and the temperature maintained at 0–5°. The mixture was adjusted to pH 6–7 and stirred for 2 h. The content was then poured on crushed ice, filtered, and the resulting solid was dried and crystallised from 95% ethanol, (80%), m.p. 350°.

2-[N⁴-{N¹-(2'-Thiazolyl)sulphanilamido}]-4-(4'-bromoanilino)-6-chloro-s-triazine: *p*-Bromoaniline (0.01 mol, 1.72 g) dissolved in acetone was added dropwise during 1.5 h to a mechanically stirred solution of 2-[N⁴-{N¹-(2'-thiazolyl)sulphanilamido}]-4,6-dichloro-s-triazine (0.01 mol, 4.03 g) in acetone at 35°. The temperature was gradually increased 45° during 2 h and pH maintained at 7 by aqueous sodium carbonate solution. The reaction mixture was then cooled to room temperature. It was poured in crushed ice and 2% HCl added. The resulting solid was filtered, dried and crystallised from 95% ethanol, (70%), m.p. > 350°.

General method for preparation of 2-[N⁴-{N¹-(2'-thiazolyl)sulphanilamido}]-4-(4'-bromoanilino)-6-(substituted-phenylthiourea)-s-triazine: 2-[N⁴-{N¹-(2'-Thiazolyl)sulphanilamido}]-4-(4'-bromoanilino)-6-chloro-s-triazine (0.01 mol, 5.4 g) in acetone and various phenylthiourea (0.01 mol) in acetone at pH 7 were refluxed for 3 h at 80–90° on a water-bath. The mixture was cooled to room temperature and then poured on crushed ice and filtered. The resulting solid was crystallised from 95% ethanol.

The yield varied from 50 to 65%; m.p. of the compounds: R=phenyl, 139°; *o*-tolyl, 198°; *m*-tolyl, 165°; *p*-tolyl, 166°; *o*-nitrophenyl, 120°; *m*-nitrophenyl, 158°; *p*-nitrophenyl, 190°; *o*-chlorophenyl, 218°; *p*-chlorophenyl, 173°.

Antibacterial activity: The compounds were screened for antibacterial activity using paper-disc or agar-diffusion technique² in acetone solution. The bacteria taken for the study were *Staphylococcus aureus* (gram-positive) and *Escherichia coli* (gram-negative).

In case of *E. coli*, the maximum zone of inhibition was found for 2-[N⁴-{N¹-(2'-thiazolyl)sulphanilamido}]-4-(4'-bromoanilino)-6-(2'-chlorophenylthiourea)-s-triazine (2.0 mm) and the minimum zone of inhibition for 2-[N⁴-{N¹-(2'-thiazolyl)sulphanilamido}]-4-(4'-bromoanilino)-6-(4'-methylphenylthiourea)-s-triazine (0.5 mm). In case of *S. aureus*, the maximum zone of inhibition was found for 2-[N⁴-{N¹-(2'-thiazolyl)sulphanilamido}]-4-(4'-bromoanilino)-6-(2'-chlorophenylthiourea)-s-triazine (1.5 mm) and the minimum zone of inhibition for 2-[N⁴-{N¹-(2'-thiazolyl)sulphanilamido}]-4-(4'-bromoanilino)-6-(4'-methylphenylthiourea)-s-triazine and 2-[N⁴-{N¹-(2'-thiazolyl)sulphanilamido}]-4-(4'-bromoanilino)-6-(4'-chlorophenylthiourea)-s-triazine (0.5 mm).

All the compounds in general recorded the following ir bands: 860–830 (C₃N₃), 1580–1570 (NH), 1160–1140 (C=S), 1560–1520 (NHCSNH) and 1130–1100 cm⁻¹ (SO₂NH).

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A New Aliphatic Constituent of *Melastoma malabathricum* Linn.†

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THE leaves and flowers of *Melastoma malabathricum* Linn.^{1,2} (Melastomaceae), a shrub of variable heights, are used in folk medicine.

The investigation on the leaves and flowers of *M. malabathricum* resulted in the isolation of a number of compounds. We report here the isolation of a new alcoholic constituent Mm₁, and its characterisation as 32-methyl-1-tritriacontanol along with some known compounds.

The petrol (b.p. 60–80°) extract of the air-dried finely powdered leaves (3 kg) on chromatography (silica gel, 60–120 mesh; eluant: petrol–benzene, 1:1) yielded β -sitosterol, the identity of which was confirmed by m.m.p. and superimposable ir spectra with an authentic sample.

The petrol extract afforded the new aliphatic alcohol, Mm₁ (eluant: benzene–ethyl acetate, 4:1). Compound Mm₁ (0.002%), C₃₄H₇₀O (*M*⁺494), white crystals (petrol–chloroform), m.p. 76–8°, was neutral to litmus, soluble in chloroform, benzene and sparingly soluble in petroleum ether. The compound did not show any high intensity absorption maxima in the uv region, but showed ir absorption maxima characteristic of an aliphatic primary alcohol at $\nu_{\max}$ (KBr) 3445 (m, primary OH), 2920, 2840 (s, alkane CH₂ stretch), 1385 (s), 1360 (m, gem-dimethyl), 1050 (br, m), 720 and 710 cm⁻¹. ¹H nmr spectrum (200 MHz) showed the signals in conformity with the proposed structure of an aliphatic alcohol. The ¹H nmr signals were observed at δ (CDCl₃) 0.91 (3H, s, CH₃), 0.93 (3H, s,

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CH₃), 1.30 (6H, br, s, CH₂), 1.99 (1H, s, D₂O exchangeable OH) and 3.61 (2H, t, J₁, J₂ 6 Hz, CH₂OH). The two singlets at δ 0.91 and 0.93 (3H each) are due to the *gem*-dimethyl protons which are also apparent from the characteristic ir bands at 1385 and 1360 cm⁻¹. The singlet at δ 1.99 (1H exchangeable with D₂O) and a triplet at δ 3.61 (2H, J₁, J₂ 6 Hz) suggest the primary nature of the hydroxyl group⁸. The broad singlet at δ 1.30 suggest the presence of long carbon chain in Mm₁ (integrating for 61 protons). The ¹³C nmr spectra (80 MHz, noise-decoupled and SFORD spectra) of Mm₁ provided an unambiguous proof of the structure. The signals confirmed the presence of a *gem*-dimethyl group and a primary alcoholic group in the compound Mm₁. The signals were obtained at δ (CDCl₃) 62.85 (t, C-1), 32.66 (t, C-2), 25.55 (t, C-3), 30.47 (t, C-4), 31.70 (t, C-31), 25.04 (d, C-32), 22.44 (q, C-33, C-33') and 29.22–29.64 (t, other CH₂ carbons). The downfield signal at 62.85 ppm(t) was assigned for the carbon bearing the hydroxyl group⁴. The *gem*-dimethyl carbons, at C-33 and C-33' appeared at 22.44 ppm(q) and the C-32 signal appeared at 25.04 ppm(d). The mass-fragmentation pattern was in conformity with the structure, (CH₃)₂CH.(CH₂)₃₀CH₂OH, the significant ion-peaks appearing at *m/z* 494 (*M*⁺, 0.70), 476 (0.74), 462 (0.42), 451 (4.20), 448 (2.20), 406 (1.74), 392 (2.76), 97 (62.41), 83 (62.60), 57 (100) and 43 (92.65). The loss of 18 mu (*m/z* 476) and 43 mu (*m/z* 451) from molecular ion peak postulated the presence of the hydroxyl group at one end and the *gem*-dimethyl group at the other⁸.

The leaves also afforded ursolic acid⁶, C₃₀H₄₈O₈ (*M*⁺ 456), and the structure was confirmed by m.p., m.m.p. and superimposable ir spectra with an authentic sample. The extract also yielded another triterpenoid, C₃₂H₄₈O₄ (*M*⁺ 496), m.p. 314–17°d, [α]_D²⁵ +39 (chloroform) but no further work was possible on this compound due to paucity of the material.

The de-fatted ethyl acetate extract of the flower of this plant afforded *p*-hydroxybenzoic acid, gallic acid and kaempferol⁷.

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Solvent Extraction and Spectrophotometric Determination of Copper using Mixed Ligand Complex Formation with Pyridine, α -Picoline, β -Picoline, γ -Picoline or 2,4,6-Collidine and Bromide/Iodide

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REFERENCE to pyridine as an auxiliary ligand in the solvent extraction of metals is not new in the literature. Use of pyridine–thiocyanate for extractive-photometric determination of various metals is well known^{1–6}. It has been noted that copper(II) forms complexes with pyridine bases in presence of potassium bromide or potassium iodide. The mixed ligand complexes are extractable into chloroform. These properties of the copper complexes suggested that further studies of the systems might lead to the development of a simple spectrophotometric method for the determination of copper. Apart from pyridine, some of its methyl-substituted derivatives used are α -picoline, β -picoline, γ -picoline and 2,4,6-collidine.

Experimental

Spectral curves and analytical measurements were made with a Shimadzu PR1 spectrophotometer. Stoppered quartz cells of 10 mm optical path length were used for absorbance measurements.

A stock solution of copper(II) was prepared by dissolving CuSO₄.5H₂O in distilled water followed by its standardisation as benzoin- α -oximate⁷. A working solution of Cu^{II} (120 μ g ml⁻¹) was prepared by dilution. Chloroform (E. Merck), pyridine (B.D.H.), α -picoline (Riedel), β -picoline (B.D.H.), γ -picoline (Fluka) and 2,4,6-collidine (B.D.H.) were