

Notes

Chemical Investigation of the Bark of *Callicarpa arborea* (Verbenaceae)

(MISS) MANJU SEN & BIKAS CHANDRA PAL

Department of Chemistry, University of Kalyani, Nadia,
West Bengal

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THE bark is aromatic, bitter and is applied¹ in decoction to cutaneous diseases. Since other species of *Callicarpa* are known to yield diterpenoids², we became interested to undertake a chemical investigation of the bark of the plant.

Dried and powdered bark of *C. arborea* was extracted with benzene for 10 hrs. The extract was separated into neutral and acidic fractions. The acidic fraction was esterified with diazomethane and the crude methyl ester was chromatographed over deactivated alumina. Elution with a mixture of petroleum ether and benzene (3:2) provided a crystalline solid which on crystallisation from methanol yielded methyl betulinate, m.p. 218°–220° (Lit.³ m.p. 220°–224°), identical, (mixed m.p. and TLC) with an authentic specimen (Found: C, 78.63; H, 10.49. Calc. for C₃₁H₅₀O₃; C, 79.10; H, 10.71%). On acetylation it furnished methyl betulinate acetate, m.p. 200°–203°, (α)_D +15.3° (Lit.³ m.p. 200°–203°, (α)_D +18°), identical (mixed m.p. and TLC) with an authentic specimen (Found: C, 77.17; H 10.06. Calc. for C₃₃H₅₂O₄; C, 77.29; H, 10.72%).

The neutral fraction was chromatographed over silica gel. Elution with a mixture of pet ether and benzene (2:3) afforded a gummy solid, m.p. 130°–132° which on acetylation and crystallisation from acetone furnished β -sitosteryl acetate, m.p. 126°–128°, (α)_D +27° (Lit.⁴ m.p. 127°–128°), identical (mixed m.p. and TLC) with authentic specimen.

Further elution of the column with benzene and ether afforded a gum, which was saponified. The non-saponifiable fraction upon chromatography on silica gel gave a solid, which on crystallization from acetone furnished baurenol, m.p. 200°–203° (Lit.⁵ m.p. 206°–208°), identical (mixed m.p. and TLC) with authentic sample. (Found: C, 84.48; H, 11.57; Calc. for C₃₀H₅₀O; C, 81.99; H, 11.18%).

Investigations of the other parts of the plant are in progress.

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N-Benzoyl-o-tolylhydroxylamine as a Gravimetric Reagent for Bismuth (III)

S. LAHIRI

Department of Chemistry, Jadavpur University, Calcutta-32

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N-BENZOYL-o-tolylhydroxylamine has been used for gravimetric determination of bismuth (III) and its separation from a large number of other ions. The precipitation of bismuth as its complex starts at pH 5.1 and is quantitative in the pH range 6.0–8.0. The creamy white precipitate can be weighed as such after drying at 110°–120° and its composition corresponds to the formula Bi(C₁₄H₁₂O₂N)₃. The interference due to Mg²⁺, As³⁺, Sb³⁺, MoO₄²⁻, WO₄²⁻, Be²⁺, Pb²⁺, La³⁺, Th⁴⁺, UO₂²⁺, Mn²⁺, Co²⁺, Ni²⁺, Cu²⁺, Zn²⁺, Cd²⁺, Hg²⁺ and Pd²⁺ has been avoided by working at controlled pH using suitable masking agents. The average error is generally $\pm 0.5\%$.

N-Benzoyl-o-tolylhydroxylamine has been reported as a reagent for the separation of niobium and tantalum¹, spectrophotometric determination of vanadium² and gravimetric determination of uranium³. Like N-benzoyl-phenylhydroxylamine⁴, this reagent reacts with bismuth (III) giving precipitate of definite composition.

N-Benzoyl-o-tolylhydroxylamine: The reagent was prepared by the method described earlier¹. Reagent solutions were made by dissolving it in 10–20 ml of 90% ethanol before use.

Bismuth (III) solution: A stock solution was prepared by dissolving bismuth (III) subnitrate in about 0.2M nitric acid. The metal content was determined gravimetrically⁵.