POLYPHENOLS OF THE LEAVES OF
OCHNA JAPOTAPITA

Ochna japonapita Linn. 1 (Syn. O. squarrosa Linn., fam. Ochnaceae) is a small tree whose different parts are useful in the indigenous system of medicine 2-3. Recently Okigawa et al. 4 reported the isolation of a biflavone, ochnaflavone from the acetone extract of the leaves. In continuation of our work on the leaf flavonoids of Indian Medicinal Plants, we have systematically examined the leaves of O. japonapita and the results are recorded here.

Shade dried leaves of O. japonapita collected from the Annamalai University Campus, Chidambaram, were extracted with hot acetone followed by rectified spirit, and the concentrates were worked up separately. The alcohol concentrate was divided into three fractions by partition using ether, ethyl acetate and ethyl methyl ketone. The ether fraction showed the presence of minute quantities of apigenin and luteolin. The ethyl acetate and ethyl methyl ketone extracts contained the same components, and hence they were mixed and concentrated to yield a cream coloured solid. This was found to be a mixture of two substances, which were separated into acetone solubles and acetone insolubles. The acetone soluble component was thrice recrystallised from acetone-chloroform to yield a buff coloured powder, blackening above 220°. [α]28 + 32.7° (acetone). It had λmax. 278 nm and νmax, 3380 (broad), 1620, 1540, 1460, 1380, 1300, 1290 and 1110 cm⁻¹ characteristic of proanthocyanidins. The NMR spectrum of the compound and the NMR and IR spectra of its acetate further confirmed its proanthocyanidin nature. On treatment with 5 N CH₃I in ethanol, it yielded cyanidin (identified by λmax. and co-chromatography with an authentic sample). Thus, the original compound was identified as a procyanidin. Fuller characterisation is in progress.

The acetone insoluble fraction was recrystallised from aqueous methanol to yield a light yellow solid, answering all the tests for flavonoids. It was found to be a mixture of three closely related glycosflavones by paper chromatography. They were separated into pure components by preparative PC using n-butanol : 27% acetic acid (1 : 1) mixture as developing solvent. Each component was identified by λmax., preparation of its acetate, resistance to hydrolysis with 7% H₂SO₄, treatment with HI in phenol, Rf and co-chromatography with authentic samples. Vitexin (apigenin-8-C-glucoside), orientin (luteolin-8-C-glucoside) and isoorientin (luteolin-6-C-glucoside) were thus isolated and identified, in almost equal proportion.

The acetone extract of the leaves contained larger quantities of the procyanidin and small quantities of the flavonoids. No biflavone could be detected by us.

This is the first record of occurrence of glycosflavones in the Ochnaceae. Our isolation of procyanidin and glycosflavones from the leaves of O. japonapita collected in South India is in contrast with the isolation of ochnaflavone (a biflavonoid) from the North Indian sample. The formation of procyanidin (flavan condensation) or ochnaflavone (flavone condensation) appears to be a favourable polyphenolic condensation process taking place in O. japonapita. Very recently, the leaves of O. squarrosa have been reported to contain only sitosterol4; no flavonoid has been isolated5.

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A NEW PHOTOMETRIC METHOD FOR THE DETERMINATION OF TERTVALENT GOLD WITH NICOTINIC AND BENZOIC ACID HYDRAZIDES

BEAMISH1 AND BOLTZ2 have critically reviewed the spectrophotometric methods for the determination of tervalent gold. A perusal of these reviews reveals that the reported spectrophotometric methods are based on the formation of coloured products or colloidal gold by reaction with a variety of ligands. The present investigation is based on the formation of a water soluble pink coloured product when tervalent gold is treated with an excess of either nicotinic acid hydrazide (NAH) or benzoic acid hydrazide (BAH) in an alkaline solution. The colour system obeys Beer's law at 520-530 nm in the range 4.0-36.0 μg per ml of gold. The new method has advantages in that (1) as low as 4.0 μg gold per ml can be determined with a coefficient of variance of 0.185 with NAH and 0.22 with
BAH. (2) The colour formation is quite rapid and is stable over a period of 20 hours.

<table>
<thead>
<tr>
<th>NAH</th>
<th>BAH</th>
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<tbody>
<tr>
<td>Beer's law limits</td>
<td>4·0 - 36·0 µg/ml</td>
</tr>
<tr>
<td>Effective photometric range</td>
<td>21·0 - 36·0 µg/ml</td>
</tr>
<tr>
<td>Molar absorptivity</td>
<td>3515 ± 15 lit. mole⁻¹ cm⁻¹</td>
</tr>
<tr>
<td>Sandell sensitivity</td>
<td>0·056 µg/ml</td>
</tr>
</tbody>
</table>

Reagents.—A 1·6% gold (III) chloride (Johnson Mathey Co., London), in 1·0 M hydrochloric acid is prepared and standardized iodometrically. 0·05 aqueous solutions of nicotine and benzoic acid hydrazides are prepared from Fluka’s pure samples. These solutions are rendered alkaline just before mixing with the gold (III) solution, such that the net alkalinity of the reagent solution is 0·25 M.

All other reagents are of analytical reagent quality.

Apparatus.—A Hilger Spekker absorptometer, model H-760 with filter No. 4 (maximum transmission 520 nm) and optically matched glass cuvettes is used for quantitative absorption data. Micropipettes are used for measuring the fractional volumes of solutions.

Procedure for detection of gold (III).—To a mixture of 0·2 ml of the reagent solution and 0·1 ml of 0·1 M sodium hydroxide taken in the cavity of a spot plate, 0·05 ml of gold (III) solution is added and the total volume made up to 0·5 ml with deionised water. A pink colour which develops on mixing the reagents in the cavity of the spot plate confirms the presence of gold. The identification and dilution limits are found to be as follows:

<table>
<thead>
<tr>
<th>Using NAH</th>
<th>Using BAH</th>
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<tbody>
<tr>
<td>Identification limit:</td>
<td>1·0 µg in 0·5 ml</td>
</tr>
<tr>
<td>Dilution limit:</td>
<td>1 : 5 × 10⁴</td>
</tr>
</tbody>
</table>

Procedure for the photometric determination of gold (III).—To an aliquot of the gold (III) solution (containing 4·0 - 36·0 µg/ml) taken in a 25 ml volumetric flask, 2·0 - 6·0 ml of freshly mixed alkaline reagent solution (either NAH or BAH) is added and the mixture is diluted to 25 ml with deionised water. The absorbance of the resulting pink coloured solution is measured against a water blank in an Hilger Spekker absorptometer using Filter No. 4 and referred to a standard calibration curve.

For accurate results, it was found necessary to follow the above order of mixing the reagents as otherwise, addition of alkali to a mixture of gold (III) and hydrazide results in the formation of colloidal gold, thus leading to erroneous results. A 20-fold excess of the reagent is found necessary for the rapid development of colour. The pink coloured product obtained with NAH and BAH has the following absorption characteristics:

Interferences.—1000-fold excess of Al³⁺, 500-fold excess of Cd²⁺, Cr³⁺, 200-fold excess of Cu²⁺, V⁵⁺, UO²⁺, 100-fold excess of Ni²⁺, 50-fold excess of Th⁴⁺, Ru³⁺, Cr(VI) and 10-fold excess of Fe²⁺, Ti⁴⁺, Pt⁶⁺, Pd⁴⁺, Pt(IV) Os(VIII) do not interfere. Cu²⁺ interferes in all proportions and should be eliminated prior to the determination of gold.

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ON THE BOEHM LAMELLAE

The deformational features associated with the quartz grains are the deformation lamellae, called the Boemh lamellae, and the undulatory extinction bands. The author recognised both these features in the quartzites of Satnur, Bangalore District, India. The Boemh Lamellae were observed in only a few of the several grains selected for studying the quartz-axes orientation. The grains were tilted on the horizontal axes of the Universal Stage in their various positions to recognise the presence of the lamellae. It is to be noted that, while undulatory bands are present in all the quartz grains, the Boemh lamellae are observed only in a few grains. When mutually associated, the boundary surfaces of the undulatory bands lie close to [0001] and the Boemh lamellae consistently intersect [0001] at high angles. The angle between the quartz axis and the lamellae poles, in several grains in Satnur quartzites, was found to have a narrow range of 28° to 36°. Sander (1948 and 1950, p. 362) and Fairbairn (1949, p. 14) give angles varying from 7° to 36°. Fairbairn refers to the irrational crystallographic