absorbance (for at least 24 h) when the extractions were carried out at pH in the range 6–9. The extraction of the aqueous phase was repeated till an organic extract that showed virtually no absorbance was obtained, which indicated complete and quantitative recovery of platinum from the aqueous phase.

The absorption spectrum of the Pt(IV)–INTC complex in chloroform shows absorption maxima at 369 and 435 nm. The reagent blank shows insignificant absorbances from 340 nm onwards. Hence the wavelength of 369 nm was selected for analytical measurements for higher absorbance.

It was noted that 0.5 ml of 0.4% ethanolic solution of INTC was the optimum reagent concentration to extract 100–200 μg of Pt(IV) in a single extraction. Higher reagent concentration was avoided, since in the presence of a higher concentration of INTC the absorption of the blank is high. Beer's law was obeyed over the concentration range 2–25 ppm of platinum (IV). The molar absorptivity and sensitivity index (on the basis of Pt content) of the colour system were found to be 0.82×10^4 l mol^{-1} cm^{-1} and 0.023 μg Pt/cm^2 respectively.

The precision and accuracy of the method were tested by analysing solutions containing a known amount of platinum. The average of six determinations (190 μg Pt) was found to be 191.33 μg, with a relative mean deviation of 1.91%.

A deviation of not more than ±3% from the recovery of platinum was taken as the standard tolerance limit in testing for interference due to other ions. In the estimation of 190 μg of Pt, the following ions did not interfere when present in the amounts (mg) shown in parentheses: Cd(II) (4.5), Ca(II) (5.2), Ce(III) (4), Cr(III) (4.5), Pb(II) (5.5), Mn(II) (3.2), Th(IV) (3), U(VI) (4), Zn(II) (5.5), Zr(IV) (4.5), Bi(III) (3), La(III) (4.5), Mg(II) (1), Al(III) (1), Mo(VI) (4.5). Palladium(II) (0.5), Cu(II) (0.5). These ions could be tolerated in the presence of thiosulphate and tartrate respectively. Interference due to Ni(II) (0.5) and Co(II) (0.5) was avoided by using EDTA as the masking agent. In the presence of silver(I), platinum(IV) developed no colour with INTC. High results are obtained in presence of rhodium(III).

Among the anions tested, 10 mg each of thioctanate, thiosulphate, iodide, EDTA, citrate, tartrate, oxalate and phosphate did not interfere. Less than 0.5 mg of fluoride could be tolerated. Ascorbate and thiourea interfered.

25 May 1988; Revised 13 December 1988

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CHEMICAL ANALYSIS OF *CALOTROPIS PROCERA* LATEX

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HESSE and his collaborators[1] detected, rather interestingly, α-lactucerol as the only constituent in the ethanolic extract of *Calotropis procera* latex. The latex also revealed the presence of a variety of cardioactive poisons, terpenoid alcohols, esters involving steam-volatile and other long-chain fatty acids[2,3], taraxasterol, calotropagenin and six of its glycosides[4–11], and three additional compounds[12]. All these glycosides contained a common aglycone[5] to which a structure[13–15] was assigned and subsequently established by its partial synthesis[16,17]. Table 1 lists the compounds hitherto identified and isolated in pure form from *Calotropis procera* latex, leaves and stem, with their molecular formulae and melting points, and the available relevant references.

Thin-layer chromatography of *Calotropis procera* latex extract revealed more than 22 spots on exposure of the plate to iodine vapour and sulphuric acid spray (60% v/v) and subsequent heating to 105°C for 5 min. This observation prompted the authors to make a systematic analysis of the latex and look for compounds yet undetected and/or unidentified.

Calotropis procera latex was collected as described earlier[18]. The dried solid was extracted exhaustively with ethanol and the pooled concentrate on filtration deposited white shining needles melting at 224–225°C. The pentacyclic triterpenol answered to the formula $C_{30}H_{50}O$ deduced from its elemental analysis and molecular weight read from its mass spectrum. The properties and melting point of the

Table 1 *Compounds isolated from Calotropis procera latex*

Compound	Molecular formula	Melting point (°C)	Reference
Calactin	$C_{29}H_{40}O_9$	265–268	12
Calotropagenin	$C_{23}H_{36}O_6$	—	12
Calotropin	$C_{29}H_{40}O_9$	181–186	i) Hesse, G., Fasola, H. and Geiger, W., *Liebigs Annln. Chem.*, 1959, **625**, 157. ii) Crout, D. H. G., Curtis, R. F., Hassall, C. H. and Jones, T. L., *J. Chem. Soc.*, 1964, 2187. iii) 12
Calotoxin	$C_{29}H_{40}O_{10}$	229–237	i) Hesse, G., Fasola, H. and Geiger, W., *Liebigs Annln. Chem.*, 1959, **625**, 157. ii) 12
α-Lactucerol	$C_{30}H_{50}O$	224–225	1,3
Procerocid	$C_{29}H_{40}O_{10}.H_2O$	222–223	12
Syriogenin	$C_{23}H_{34}O_5$	275–278	12
Taraxasterol	$C_{30}H_{50}O$	215–217	12
Uscharin	$C_{31}H_{41}NO_8S$	270–271	12
Uscharidin	$C_{29}H_{38}O_9$	298–299	12
Uzarigenin	$C_{23}H_{34}O_4$	249–250	12
Voruscharin	$C_{31}H_{43}NO_8S$	—	12
β-Amyrin	$C_{30}H_{50}O$	199–200	Present communication
α-Calotropeol	$C_{30}H_{50}O$	204–205	
β-Calotropeol	$C_{30}H_{50}O$	216–217	
3-Epimoretenol	$C_{30}H_{50}O$	223–224	
Lupeol	$C_{30}H_{50}O$	214–215	Pant and Chaturvedi, *Curr. Sci.*, 1989, **58**, 302.

compound and those of its acyl (m.p. 251–252°C) and benzoyl (m.p. 255–257°C) derivatives tallied with those of α-lactucerol and its derivatives[1]. The identity was finally established by its conversion to iso-lactucerol (m.p. 200–202°C), preparation of the latter's derivatives and comparison of their properties with those of the preparations of Hesse *et al*[1].

Curiously, however, repetition of the experiment with latex collected from different *Calotropis procera* plants from different localities in an identical manner but during different months of the year did not always yield exclusively α-lactucerol but afforded a mixture of several compounds with α-lactucerol as one of its components. This made us believe that the chemical composition of latex depends on seasonal variations, environment, soil and the maturity of the lactifer.

The saponifiable fraction of the latex obtained in the usual manner afforded a mixture of acetic, butyric and valeric acids, which were characterized[19] by TLC employing methyl acetate ammonia (95:5 v/v) as the developing solvent and alcoholic solution of methyl red as the spray reagent. The red spots appearing on an orange background, on heating the plate at 105°C for 2 min, were compared with those of reference pure known fatty acid samples.

The unsaponifiable fraction of the latex yielded a mixture of α and β calotropeols, β-amyrin, lupeol and a triterpene which showed a retention time and mass spectrum suggesting it to be 3-epimoretenol. NMR (4.6, 2H) and IR (1645 m, 980 m, 910 m) data were consistent with the presence of an isopropenyl group. The carbinol proton was 4.5, $J=6$ and $10H_3$. The NMR spectrum also revealed signals from 0.69 to 0.97 ppm (18H) and at 1.68 ppm (3H), corresponding to six angular methyl groups and one CH_3 being on a doubly bonded carbon atom respectively. The optical rotation $[\alpha]_D$ of the compound was determined to be $-2.5°$ and is in agreement with that found by Khastgir *et al*[20]. Based on the above observations the compound was confirmed as 3-epimoretenol.

Occurrence of 3-epimoretenol in *Calotropis procera* latex is being reported for the first time.

We are greatly indebted to Dr R. J. Highet, Laboratory of Chemistry, National Institutes of Health, Bethesda, USA, for the IR, NMR and mass spectra of the compounds isolated and for carrying out the GLC analysis.

4 June 1988; Revised 7 November 1988

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GARNET CONTENT IN THE BEACH SANDS OF GOPALPUR (BAY OF BENGAL)

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GARNET an industrially important heavy mineral, has been found to be enriched in the black sand and sand dunes of Gopalpur coast, Bay of Bengal. The average width of the beach is 45 m. The beach runs along the north–south direction and is fringed with ephemeral streams and riverlets. Dunes of various sizes occur all along the backshore region. Black sand concentrations are not uncommon on Gopalpur beach and its occurrence along the East coast of India is well known[1–6] as it is a potential source of rare-earth minerals. In the present investigation the distribution of garnet and its concentration, in relation to the prevailing hydrodynamical conditions and topography of the area along the beach, have been studied.

Sand samples were collected from three stations (Gopalpur, Arzipalleam, and mouth of Rushikulya river) along a 15 km length at 30 m intervals representing the backshore and foreshore zones of the beach (figure 1). Beach profiles were studied during the pre-monsoon (May 1987) and post-monsoon periods (December 1987). The samples were washed, dried and sieved by using ASTM sieves of 18, 60 and 230 mesh. Garnet minerals were examined using a petrological microscope (MEOPTA)[7] and the percentage number of the mineral was counted for each fraction. The size analysis of the bulk samples was made following the Folk and Ward Method[8] on $\frac{1}{2}\phi$ scale and the statistical parameters were calculated in relation to the distribution of the garnet sands.

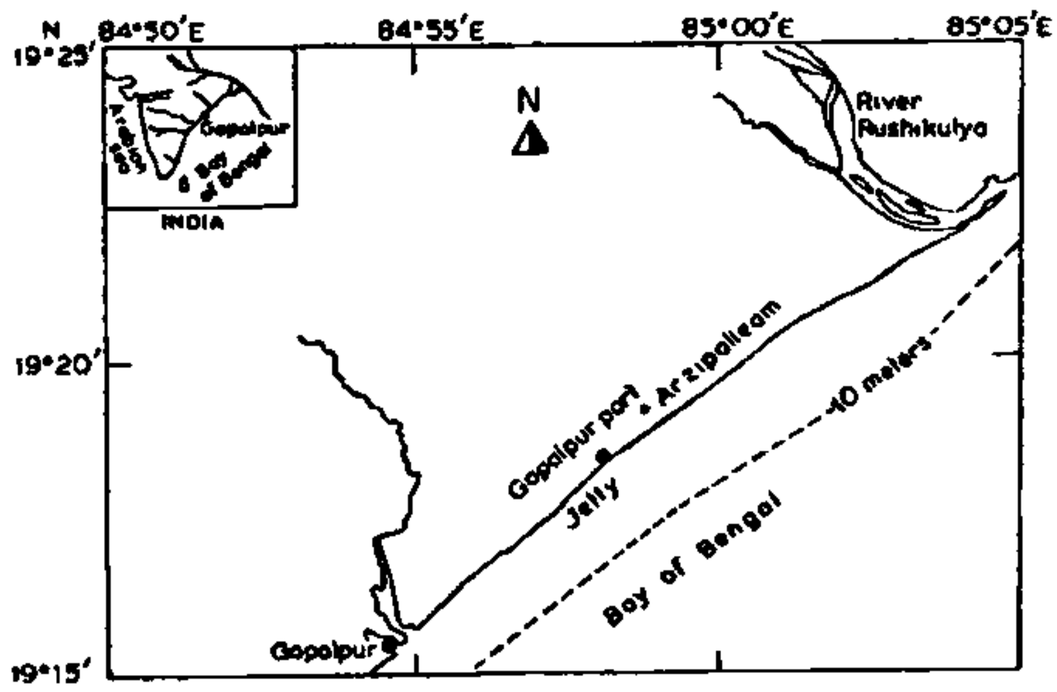

Figure 1. Location map.