nickel and palladium, for the amperometric determination of copper[4] and nickel[5] and for the spectrophotometric determination of uranium[6] and vanadium[7]. A survey of literature shows that no extraction studies are carried out with this reagent. The present paper communicates the results obtained, in the rapid extraction spectrophotometric determination of copper(II) with OHAPO into methyl iso-butyl ketone (MIBK). The proposed method is simple and rapid. The high tolerance limit of zinc makes the method useful for the determination of copper in brass.

2-Hydroxyacetophenoneoxime was prepared from 2-hydroxyacetophenone by the literature method[8]. A $1 \cdot 5\%$ solution of the reagent in 50% aqueous methanol was used for the studies. All the other chemicals used were of AnalaR grade.

Recommended Procedure

The extractions are carried out by shaking 15 ml of the aqueous solution containing 1 ml of the oxime solution, 7 ml of sodium acetate-acetic acid buffer (pH 5·5) and the metal ion solution (10–60 μg) with 10 ml of MIBK for two minutes in a 25 ml separatory funnel. The organic layer is separated, dried over anhydrous sodium sulphate and the absorbance measured at 355 nm against the reagent blank.

Results

The absorbance of the coloured complex formed between copper(II) and OHAPO is stable for 16 hrs. Beer's law is obeyed in the range 10–60 μg of copper per 10 ml of the organic layer. The composition of the complex is ascertained to be $Cu(C_8H_8O_2N)_2$. The complex, extracted completely over the pH range 5–8, has an absorbance maximum at 355 nm; pH 5·5 is chosen for the present study since the minimum interference by diverse metal ions is observed at this pH. The molar absorption coefficient and the Sandell sensitivity are $3 \cdot 4 \times 10^3$ lit. mol^{-1} cm^{-1} and $1 \cdot 87 \times 10^{-2}$ μg/cm^2 respectively. Among various solvents tried (benzene, toluene, chloroform, carbontetrachloride, 1,2-dichloroethane, n-butanol, iso-amylalcohol, cyclohexanone and MIBK) only MIBK has been found suitable. A ten-fold excess of the reagent was found to be adequate. The reproducibility of the determination is about $\pm 1 \cdot 5\%$. The relative mean deviation is 1%. The tolerance limit of various foreign ions is given in Table I. The tolerance was set at the amount needed to cause $\pm 2\%$ error in the recovery of copper. Ni^{2+} and Fe^{3+}, Zr^{4+} interfere strongly. However Fe^3 and Zr^{4+} can be masked by the addition of the fluoride.

TABLE I

Effect of diverse ions

Cu $= 35$ μg
pH $= 5 \cdot 5$

Tolerance limit μg	Ion present
$1 \cdot 0 \times 10^4$	Na^+, K^+, Ca^{2+}, Ba^{2+}, Sr^{2+}, Zn^{2+}, NO_3^-, Cl^-, F^-, CH_3COO^-, SO_4^{2-}, ClO_4^-
$5 \cdot 0 \times 10^3$	Cr^{6+}, Mo^{6+}, UO_2^{2+}, W^{6+}, Pb^{2+}, Mn^{2+}, Mg^{2+}, Cd^{2+} PO_4^{3-}, SCN^-, Tartrate.
$1 \cdot 0 \times 10^3$	Ce^{4+}, Th^{4+}, V^{5+}, Cr^{3+}, I^-, NO_2^-
$5 \cdot 0 \times 10^2$	Co^{2+}, Al^{3+}, Bi^{3+}, $S_2O_3^{2-}$, Citrate
$1 \cdot 0 \times 10^2$	EDTA, $C_2O_4^{2-}$

Department of Chemistry,   T. SREENIVASULU REDDY.
Autonomous P–G   S. BRAHMAJI RAO.
   Centre,
Anantapur 515 003,   South India,
September 18, 1978.

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POLYPHENOLIC COMPONENTS OF CLERODENDRUM SERRATUM

As part of our continued interest in the study of *Clerodendrum* species[1-3] and in view of the varying reports on the constituents of *Clerodendrum serratum*[4-6], a detailed investigation of this plant was undertaken and the results are recorded here.

Shade-dried leaves of *C. serratum* (L.) Moon (Family: Verbenaceae) were exhaustively extracted with 80% ethanol. The extract after concentration was partitioned using light petroleum, ether and ethyl acetate. The light petroleum soluble portion yielded α-spinasterol as the only crystalline component as reported earlier[6]. No crystalline compound could be isolated from the ether extract; the EtOAc fraction gave a minute quantity of a flavone glycoside which on acid hydrolysis yielded 6-hydroxy luteolin and glucose. This glucoside could not be fully characterised owing to the paucity of the material.

The aqueous portion left after the EtOAc extraction was hydrolysed with 2N HCl for 2 hrs. The residue from the ether extract of the hydrolysate was found to be a mixture of flavones and phenolic acids (PC) which was separated by preparative PC using first 50% and then 15% HOAc as the developing solvents. The individual components were then subjected to colour reactions, UV fluorescence, Rf determination and UV absorption maximum. The flavones were identified as apigenin and luteolin (common flavones), baicalein, scutellarein and 6-hydroxy luteolin (uncommon 6-oxygenated flavones), while the phenolic acids were characterised as caffeic and ferulic acids. The identity of all the compounds was finally confirmed by co-PC with authentic samples using a number of developing solvents. The sugar was found to be a mixture of glucose, arabinose and glucuronic acid and hence no characterisation of the individual glycoside could be made.

Our identification of the flavones and phenolic acids from *C. serratum* is in general agreement with the flavonoid pattern of the Verbenaceae and *Clerodendrum* in particular. However, the sterol of molecular taxonomic significance (24S-ethyl cholesta-5, 22, 25-trien-3β-ol) of this genus identified in all other species examined earlier[-3,7] was found to be absent in *C. serratum* casting a doubt on the reliability of this compound as a dependable chemotaxonomic marker. Recent anatomical studies on this plant by one of us[8] revealed that *C. serratum* differs markedly from the other members of the genus in the histology of root and leaf suggesting placement of this plant out of the genus *Clerodendrum* thus retaining the importance of the sterol as the taxonomic marker.

Jawaharlal Institute,   A. G. RAMACHANDRAN NAIR.
Pondicherry 605 006,   T. N. C. VEDANTHAM.
January, 22, 1979.   B. KANNABIRAN.

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N_2O DECOMPOSITION ON La_2TiMO_6 (M = Ni, Cu AND Zn)

THE compounds La_2TiMO_6 where M = any one of Ni, Cu, Zn and Co have been shown to possess interesting physico-chemical properties by Ramadass et al.[1]. The catalytic activity of the first three compounds of the above series has been determined with N_2O decomposition as the test reaction. The experimental procedures for the kinetic study were the same as those used by Vijayakumar and Swamy[2] excepting that the temperature of activation was 500° C. The catalysts were pretreated with oxygen before any reaction in order to avoid correction for dead time.

The reaction has been studied in the temperature range 330–490° C. The effect of initial pressure on the kinetics of N_2O decomposition has been very well portrayed on La_2TiCuO_6. At 50 torr initial pressure, the rate of decomposition is first order with respect to the partial pressure of N_2O whereas at 200 torr, the reaction is strongly inhibited by product oxygen. The kinetic parameters have been evaluated using the equations suggested by Cimino et al[3]. Inhibition by oxygen has been confirmed[4] by conducting reactions with $N_2O + O_2$ mixture instead of N_2O alone when the rate of the reaction decreased accompanied by an increase in the energy of activation.

On La_2TiNiO_6, strong inhibition by oxygen is observed even at 100 torr initial pressure of N_2O. Equilibrium adsorption measurements indicated that oxygen is adsorbed in an activated form. In the absence of compensation effect, the activity order has been found to be $La_2TiNiO_6 > La_2TiCuO_6 > La_2TiZnO_6$ as seen from the energy of activation values (E_a) for 200 torr reactions.

Inverse linear relationship has been found between E_a for 200 torr reactions and (i) E_a for electrical conduction and (ii) magnetic moment. The first of these indicates that the rate determining step of these indicates that the rate determining step (oxygen desorption in this case) involves charge