Sample No.	Amount of tobacco taken (g)	Amount of arsenic taken (µg)	Amount of arsenic found (µg)	Error (%)
2000	<u></u>	10 15 20	10·3 15·4 20·7	+3.0 + 2.7 + 3.5
A B	57	50	49·1 4·5 6·3	-1.8
C D E	9 6 10	10.30000	3·5 5·8 4·7	

tobacco sample (1 g) and Na_2O_2 (1.5 g) were added on to the solidified mass. It was heated gently for 10 min while adding Na_2O_2 (2×1 g) intermittently until there was a complete decomposition of the organic matter.

The above procedure was repeated with more of tobacco and Na_2O_2 till 5 g of tobacco sample had been decomposed. The crucible was finally heated for 15 min, cooled and water (15 ml) added. The contents of the crucible were warmed on a steambath until the melt was completely disintegrated. The solution was centrifuged and the residue washed with water. The centrifugate and washings were collected in a 100 ml conical flask, acidified with HCl (6M) and boiled gently to decompose peroxide. The solution was cooled and conc. HCl (15 ml) added followed by aq. KI (2 ml, 15%) and SnCl₂ solution (1 ml, 40%) in conc. HCl. The contents were kept for 30 min at room temperature. Zinc metal (3-4 g) was added and the evolved arsine (AsH₃) decomposed to metallic arsenic by heating the tube. Metallic arsenic was dissolved in a minimum amount of hot HNO₃ and the resultant solution neutralized with dil. NaOH. It was reacidified with AcOH (2N) and the final volume made up to 2 ml by evaporation and dilution. An aliquot of the solution was subjected to the ring oven method as described above whereby the arsenic content of tobacco was determined and the results are given in Table 2. The efficacy of the above alkaline peroxide fusion method was checked separately by ring-colorimetric and volumetric (potassium bromate)⁸ methods. In both the methods known amounts of arsenic as arsenic trioxide were taken. A few representative ring colorimetric results have also been incroporated in Table 2.

Uranium forms a red insoluble chelate with the reagent under the specified conditions. The interference of various foreign ions was studied by developing rings for UO₂(II) ions with and without the foreign ions taken in varying amounts up to 25 µg. The ions which did not interfere in the procedure are: NH₄, Li⁺, Na⁺, K⁺, Rb⁺, Cs⁺, Ag⁺, Cu(II), Hg(II), Cd(II), Sn(II), Pb(II), Bi(II), Be(II), Mg(II), Ca(II), Ba(II), Sr(II), Al(III), Mn(II), Cr(III), As(III), Sb(III, V), Ni(II), Co(II), Zn(II), Ga(III), In(III), Tl(I), Se(IV, VI), V(V), Au(III), Pt(IV), Pd(II), Ru(III), Rh(III), Os(VIII), Ir(IV), La(III), Nd(III), Pr(III), Gd(III), Sm(III), Y(III),

Hf(IV), Zr(IV), Re(VII), W(VI), NO₂, NO₃, CO₃²⁻, Cl⁻, Br⁻, I⁻, SCN⁻, SO²₄⁻, SO²₃⁻, S₂O²₅⁻, S₂O²₈⁻, CrO²₄⁻, Cr₂O³₇⁻, ClO³₃, ClO³₄, BrO³₃, IO³₃, Fe(CN)³₆⁻ and Fe(CN)⁴₆⁻. Th(IV), Ce(III, IV), V(IV), Fe(II, III), S²⁻ and PO³₄⁻ ions, however, interfere.

The serious interference caused by fluoride, oxalate, citrate, tartrate, and EDTA is obviated by boiling the test sample three to four times with HNO₃ to almost dryness followed by evaporation with water. Interference due to fluoride and EDTA has also been overcome by treating the sample solution with a mixture of aluminium and nickel ions in the presence of ammonium acetate and AcOH. During this operation aluminium ions form more stable complex with fluoride ions than uranium ions whereas nickel ions mask EDTA. The high selectivity of the determination of uranyl ions has been achieved by incorporating arsenate ions as the fixing agent for uranyl ions under the specified conditions.

During the determination of arsenic in tobacco, alkaline peroxide method has been preferred over the conventional wet acid digestion process. Since the organic materials (pyridine and other nitrogen ring compounds) present in tobacco are hard to decompose, wet oxidation takes very long time with a large consumption of acids. This difficulty has been obviated in the present alkaline peroxide method for the decomposition of organic matter. The method has been found highly suitable in the case of tobacco.

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5-Hydroxy-1,4-naphthaquinone (Juglone), a New Indicator in Acidimetry & Alkalimetry

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5-Hydroxy-1,4-naphthaquinone has been successfully used in acidimetry and alkalimetry. With juglone the pH range for colour change (yellow \rightarrow violet) is 7.8-9.9, thereby rendering it more suitable for acid-base titrations as compared to phenolphthalein.

NAPHTHAQUINONE and its derivatives have been used in industry¹, medicine² and for qualitative and quantitative estimation of metal

ions³⁻⁵. However, the effect of $[H^+]$ on these compounds including the title compound has, so far, not been made. The present note deals with the systematic and comprehensive study in this direction.

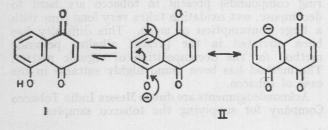
All the reagents employed were of analytical grade. Juglone was procured from Fluka AG Buchs.

The absorption spectra of juglone at different [H⁺] was recorded within 30 min of the preparation of the solutions, each set containing 1 ml (0.002M)juglone +5 ml buffer solution +10 ml doubly distilled water.

Indicator was prepared by dissolving 0.348 g/litre in EtOH.

Volumetric experiments were done by the usual process using 2 ml of indicator (juglone).

It is observed that with increasing pH, the absorbance at 520 nm increases while it decreases at 420 nm. The isobestic point appeared at 460 nm, confirming the presence of a system consisting of two chromophors. Hence the deepening of colour from yellow \rightarrow violet is due to the structural change $(I \rightleftharpoons II)$ and not because of any physical interaction.



Juglone with sufficiently wide pH range (7.8-9.9) for colour change could be employed with encouraging results, using normal (effective pH range⁶ 3-10.5) and decinormal (effective pH range⁶ 4.5-9.5), in the titration of strong acid with strong base and vice versa. For a comparative study, the titrations using phenolphthalein were also carried out. The results are quantitative in nature, except in acidimetry. The relative error with phenolphthalein and juglone are 0.75 and 0.02% respectively. The relative standard deviation as low as 0.02% can be achieved.

In pH-metric titration of decinormal sodium carbonate with decinormal hydrochloric acid in the

Mean titre (ml)		Relative error (%)		SD
Phen	Juglone	Phen	Juglone	(Juglone)
TITRAT	TION OF HCl (1	0 ml, $0.1N$) with $0.1N$	NaOH
9.998	9.997	0.02	0.03	0.002
TITRAT	ION OF NO2CO	3 (10 ml, 0	1N) WITH 0	1N HCl
4.850*	4.996*	3.00	0.06	0.002
TITRAT	ION OF NaOH	(10 ml, 0.1	N with 0.1	IN HCl
9.925	9.998	0.75	0.02	0.003

TABLE 1 - RESULTS FOR VOLUMETRIC TITRATIONS USING

JUGLONE AS INDICATOR

presence of indicator (juglone), the equivalence point and colour change (violet \rightarrow yellow) coincided at the primary stage of ionization of carbonic acid (effective pH 8.3 at the conversion of carbonate into bicarbonate stage⁷). Under these conditions, juglone with pH range 7.8-9.9, is most suitable indicator for the titration of carbonate to bicarbonate stage. The relative error with phenolphthalein and juglone are 3 and 0.06% respectively. The relative standard deviation in this case is 0.04%.

Table 1 incorporates the results for volumetric titrations.

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