

## Mamegakinone: A New Narrow Range Acid-Base Indicator

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### Introduction

The precision of acid-base titrations is dependent on the pH range through which the colour of the indicator changes. The merits of reducing this range to a minimum value by using a narrow range pH indicator have been discussed in the previous paper, in which 7-methyl juglone was described as a new narrow range acid-base indicator [1]. Juglone and lawsone have been recently reported to be useful narrow range acid-base indicators with pH ranges 7.4-8.2 and 2.2-3.4 respectively [2]. In continuation to these preliminary findings on the possible use of naturally occurring hydroxy naphthoquinones as new narrow range indicators, mamegakinone (3,3'-bis-5-hydroxy-7-methyl-1,4-naphthoquinone) has been examined for this purpose. Mamegakinone occurs naturally in the roots of some *Dispyros* and *Euclea* species [3] of family Ebenaceae.

### Experimental

#### Reagents

All the reagents used were of the analytical grade. Mamegakinone was isolated from the root bark of *Euclea natalensis* by the method described earlier [4]. Mamegakinone ( $C_{22}H_{14}O_6$ ) is an orange crystalline solid with melting point of 235°C. It was identified by comparing the spectra (IR, UV, NMR, and MS) with the previously reported data [5].

A 0.05 w/v solution of the indicator in methyl alcohol was used throughout.

Sodium hydroxide (0.1N) and hydrochloric acid (0.1N) were prepared and standardised.

#### Apparatus

A Metrohm Hcrisou E520 and electrode was used for the pH and potential measurements. A PYE UNICAM SP 1800 spectrophotometer was used for determining the spectra of the acid (spectrum I) and base (spectrum II) forms of the indicator (Figure I).

#### Titration of various acids

To a standard solution of an acid (0.1 to 0.01N) in distilled water was added 2-4 drops of the indicator and then titrated against a standard sodium hydroxide solution. The colour changes were observed. Benzoic, anisic and hydrochloric acids were titrated in aqueous medium using this indicator. The recovery of the acid in each case was  $99.7 \pm 0.2\%$ , with a good end point observation.

#### pH Range Determination

The determination of the transition interval was done by the following simple method. A buffer series (Borax), according to the transition interval to be expected, was prepared in a number of test tubes of equal colour and diameter in such a way that the initial pH value of the series was 0.6 pH units less than the lowest limit, and the final pH value of the series was 0.6 pH units higher than the upper limit of the expected transition interval

of the indicator. The remaining test tubes were filled with buffer solutions of the pH values, lying between these two limits, in a succession of gradually increasing the pH by 0.1 units at a time. Mamegakinone had slight pale yellow colour in acidic solution, while its alkaline colour was deep violet and the transition interval was between pH 8.4 to 9.2.

The indicator solution (0.2 ml) was added to 25 ml of the buffer solution in each test tube proceeding from lower towards higher pH values. The colour was assessed by looking through the depth of the solution against a white background. The first violet colour appeared at pH 8.4 and the intensity of the colour gradually increased till pH 9.2. Whereas in the reverse direction, the decrease in intensity of the violet colour was first perceptible at pH 9.2 and reached its minimum at pH 8.4. These limits of transition intervals had also been examined with a pH meter by gradually increasing the pH of the buffer solution having the indicator from pH 7.0 to 10.0 and then reversing the process from pH 10.0 to 7.0. The addition of alternate drops of 0.1N HCl and 0.1N NaOH to 1.0 ml of the indicator solution showed that the colour change was reversible and the colour of the solution was permanent at all pH levels. One part of the indicator could be detected in  $2.5 \times 10^5$  parts of water.

## Results and Discussion

The preliminary work done with mamegakinone indicated that only two to three drops of the indicator were needed to give a good colour change (from yellow to violet) in a 50 ml solution. The number of drops of the indicator required depends on the strength of the acid titrated. In most cases due to the high optical density of the anion, no indicator blank is needed.

To establish the usefulness of mamegakinone as an indicator, benzoic, anisic and hydrochloric acids were titrated in aqueous solutions. In most of the cases 0.1 to 0.001N solutions were used, the concentration of the acids seemed to have little effect on the visual end point of the indicator. All the titrations were carried out in triplicate. These titrations were simultaneously followed by potentiometric titrations to determine at what point on the titration curve does the indicator changes its colour. Taking a typical example of the titration of a strong acid (0.01M) with a strong base (0.1M).NaOH, a neutralization of 99.9% corresponds to pH 5; while 0.1% over titration will give pH 9. For the indication of the end point the indicator with a narrow range falling between pH 5 to 9 will be more accurate specially when the solutions are further diluted. Mamegakinone with pH transition between pH 8.4-9.2 can safely be used as a visual indicator. In the titrations of weak acids (organic acids) with strong bases, indicators with pH transition on the basic side will be more accurate when the solutions are dilute. Mamegakinone falls into that category. The pH transition of mamegakinone (pH 8.4-9.2) is quite narrow as compared to most of the usual indicators, making it superior especially where dilute acid solutions are titrated. The colour change from pale yellow in acid medium to violet in alkalies may be attributed to the following resonating structures (Figure 1). The pH transition interval of mamegakinone (pH 8.4-9.2) is on the basic side, which could be attributed to the intramolecular hydrogen bonding forming six membered ring which suppresses the ionization of the phenolic hydrogens as compared to the pH intervals of lapachol (pH 4.8-5.8)[6], hydrolapachol (5.0-6.0)[7] and lawson (pH 2.3-3.3), where only five membered hydrogen bonding is possible. Similar observation for juglone (I) and lawsone (II) had been reported earlier [2].

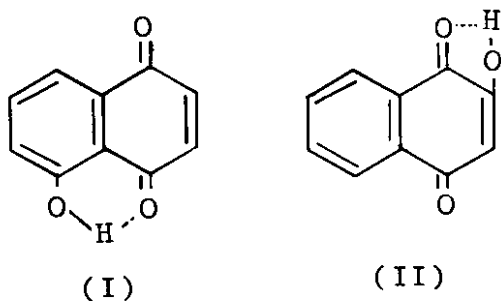
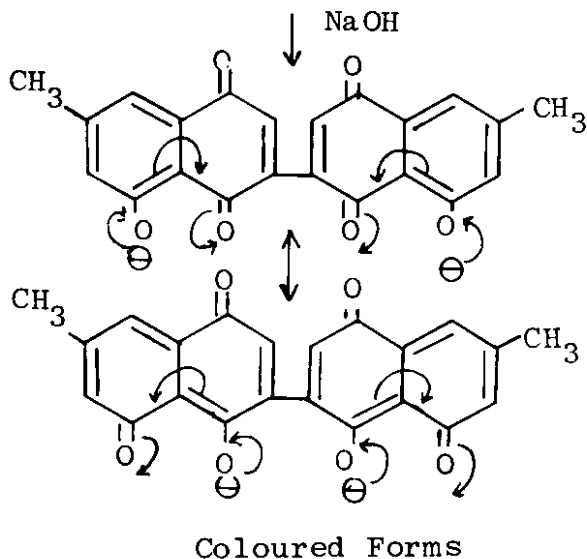
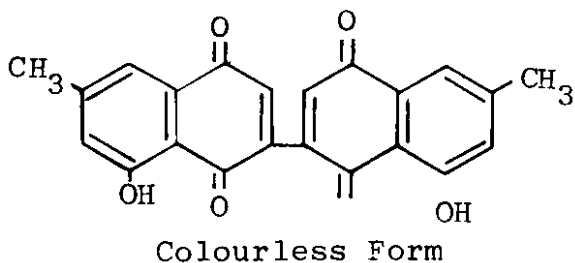


Fig.1

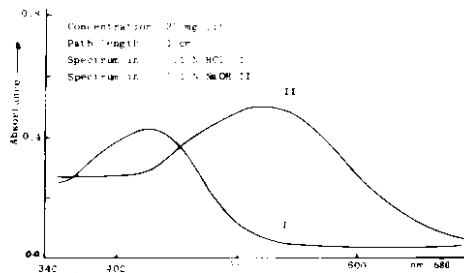


Fig.2: The Absorption Spectra of Mamegakinone in Acidic and Basic Medium.

The presence of the phenolic hydroxyl groups seems to be essential for the indicator properties of mamegakinone (Figure II) as the colour of its methyl ether was independent of the pH of the solution.

### References

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