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Spectrophometric Determination Of Eugenol*

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A rapid and simple method for the spectrophotometric determination of eugenol has been developed by using the reaction between eugenol and 1-nitroso-2-naphthol.

A study of variables that may interfere in the reaction is presented and the optimum conditions for performing the quantitative determination are established.

INTRODUCTION

The colour reaction between 1-nitroso-2-naphthol and para-substituted free phenols has been used by many authors for the quantitative determination of different materials [1,2,3].

Anger and Ofri [4] have been reported that not only para-substituted free phenols but ortho- and para- substituted free phenols also give a colour reaction with 1-nitroso-2-naphthol. This reaction has been used by these authors for spot tests analyses.

In the present paper a simple and sensitive method for the spectrophotometric determination of eugenol is described. The quantitative determination is accomplished by the colour reaction between 1-nitroso-2-naphthol and eugenol. When a solution of eugenol and 1-nitroso-2-naphthol is treated with hydrochloric acid and nitric acid and then heated an orange-red colour is obtained.

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By a systematic study of the different variables that may interfere in the reaction, it was possible to find optimum conditions for performing the quantitative determination.

The method also seems to offer good possibilities for the quantitative determination of para - and ortho - substituted free phenol groups.

APPARATUS AND MATERIALS

UV absorption curves were determined with a Beckmann quartz spectrophotometer Model DU II, using matched 1.000 cm quartz absorption cells.

1-Nitroso-2-naphthol was recrystallized from ethanol and dissolved in 96 % ethanol to give 0.1 % solution.

Nitric acid solution was prepared by diluting concentrated chemically pure nitric acid (sp.gr. 1.42) with distilled water to 2.5 N.

Concentrated chemically pure hydrochloric acid (sp.gr. 1.19) was used.

Eugenol was purified by distillation two times in vacuo under a nitrogen atmosphere and the purity was checked by paper chromatography. For colour reaction 96 % ethyl alcoholic solutions were made at concentrations ranging between 10–100 μg/ml.

PROCEDURE

To one of Pyrex test tube 5 ml samples of solution containing eugenol and to the other 5 ml of ethyl alcohol were placed. To each tube 1 ml of 1-nitroso-2-naphthol solution, 0.25 ml of nitric acid solution and 1.5 ml of hydrochloric acid were added. The contents of the tubes are well mixed by shaking and heated in a water bath maintained at 85 ± 0.5°C. After the beginning of the development of the orange-red colour, the tubes were kept in the bath for 30 second more. After which they are kept at room temperature for 30 seconds then cooled in a ice-water bath.

RESULTS AND DISCUSSIONS

The orange-red colour developed was read on the spectrop-

hotometer against the blanck treated in an identical manner. The absorption curve (Fig. 1) showed a peak with a maximum at 505 mμ and a minimum at 450 mμ. For concentrations of eugenol ranging between 10 and 100 μg/ml there is a linear relationship between quantity and colour yield (Fig. 2), and there is a good reproducibility. The value of $E^{1\%}_{1cm}$ was 102.

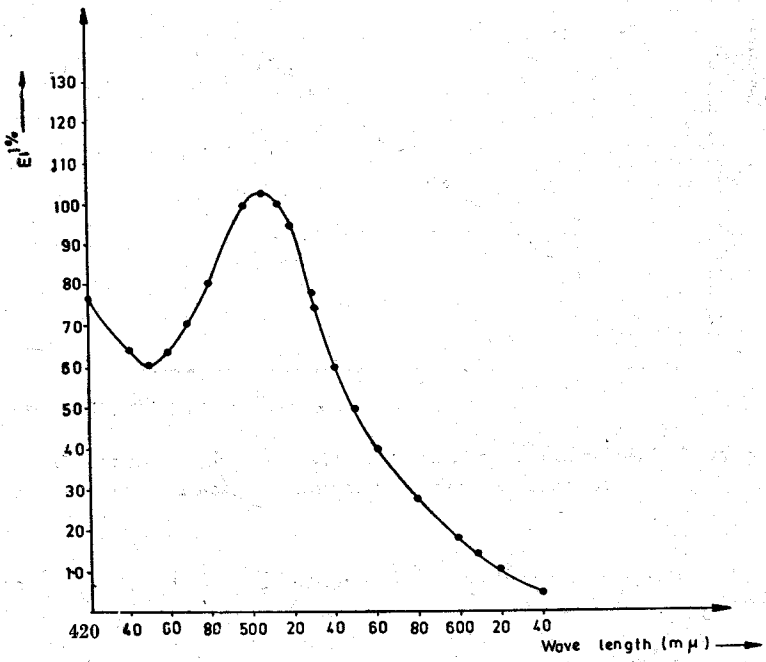


Figure 1. Ultraviolet absorption spectrum of the red-orange colour formed between the reaction eugenol and 1-nitroso-2-naphthol.

The colour formation was found to be dependent upon the reaction time and the temperature.

Effect of temperature of the water bath: the experiments at the various water bath temperatures were indicated that the optimum temperature for the procedure was 85°C. At lower temperatures the time for the development of the colour was longer than 15 minutes. At higher temperatures, appearance of the colour was

so instantaneous that it was not possible to keep the heating time constant. At 85°C the time required for the completion of the reaction was about 2 minutes and then the colour decreases slowly.

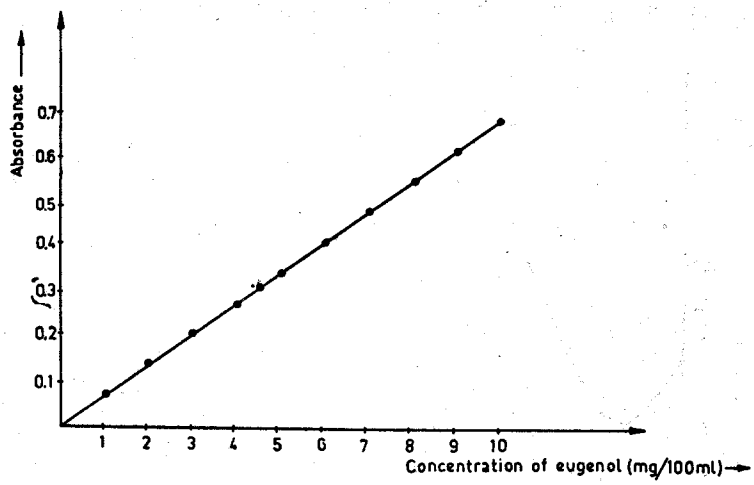


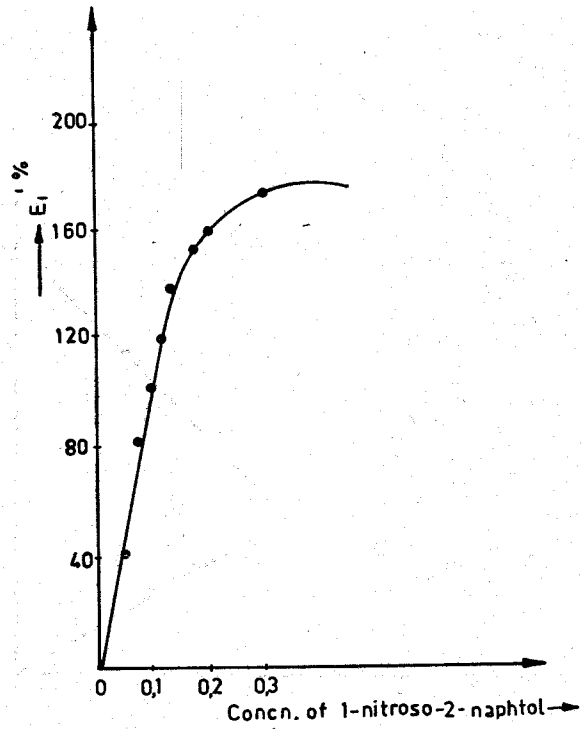
Figure 2. Relation between the quantity of eugenol and the absorbance of the colour formed during the reaction with 1-nitroso-2-naphthol.

The greatest advantage of the method was the stability of the colour.

The colour was showed no significant change for relatively long time when the reaction mixture was kept at 0°C.

After three hours at room temperature it showed only 3 % decrease in absorbance.

Influence of 1-nitroso-2-naphthol concentration: the above described reaction is carried out with 1-nitroso-2-naphthol solutions at concentrations ranging from 0.05 % to 0.3 %. At lower concentrations, a linear increase in absorbance of the orange - red colour was observed with increasing 1-nitroso-2-naphthol concentration (Fig. 3). At concentrations higher than 0.1 % very little increase in absorbance was observed.



Fifure 3. Effect of the concentration of 1-nitroso-2-naphtol on the absorbance of the orange-red colour formed during the reaction of eugenol with 1-niroso-2-naphthol.

The effect of nitric acid and of hydrochloric acid was determined by measuring absorbance of colour formed from the reaction of eugenol and 1-nitroso-2-naphthol solutions with varying quantities of acides. The colour showed concederable decrease in absorbance as the quantity of hydrochloric acid and nitric acid were increased.

Influence of nitric acid: To eugenol solutions, 1 ml of 1-nitroso-2-naphthol and 1.5 ml of concentrated hydrochloric acid and various amounts of 2.5 N HNO_3 were added (0.0–3.0 ml) and the reaction was carried out as usual. With increasing amounts of nitric acid, a decrease in absorbance was observed.

With 0.25 ml of 2.5 N nitric acid a maximum absorbance was obtained (Fig. 4 A).

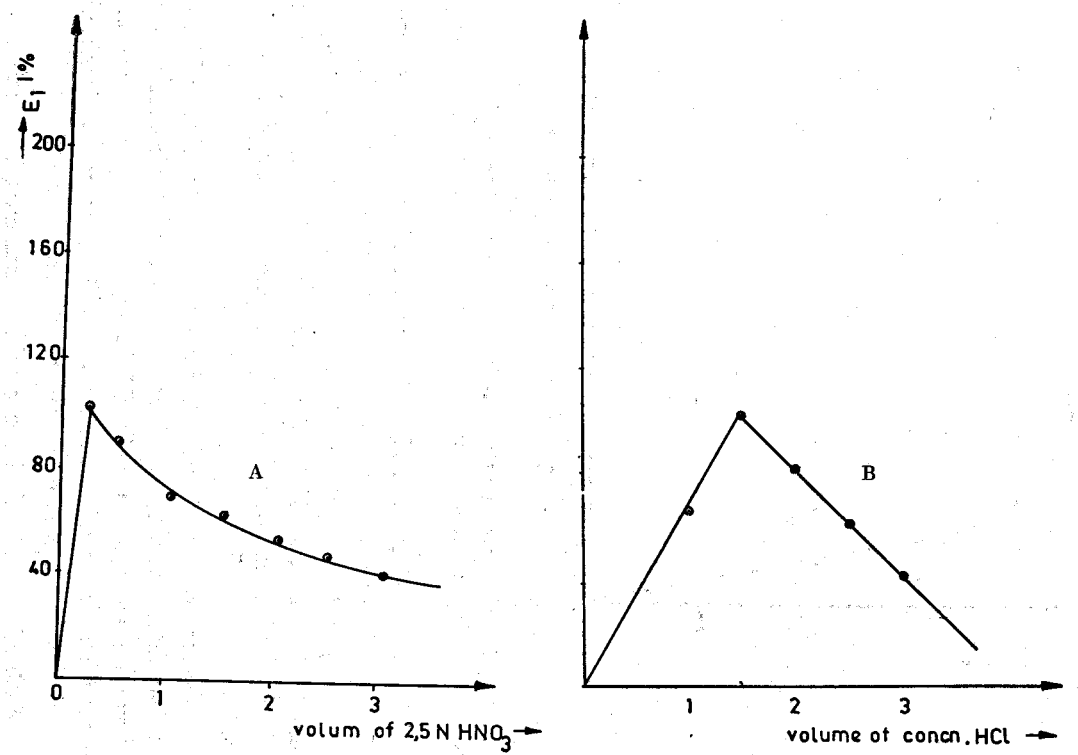


Figure 4. Influence of the amount of 2.5 N nitric acid (A) and the amount of concentrated hydrochloric acid (B) on the colour yield in the reaction of eugenol with 1-nitroso-2-naphthol.

Influence of hydrochloric acid: the same procedure was followed, with the difference that the amount of nitric acid remained constant and that of hydrochloric acid was varied (0.0–3.0 ml). In this case, maximum absorbance was observed when 1.5 ml of concentrated hydrochloric acid for the reaction was used (Fig. 4 B).

When hydrochloric acid was not used, the orange-red colour did not developed. This was the case when nitric acid was not added.

These observations are indicating the similarity of the reaction mechanism proposed for the reaction between p-cresol and 1-nitroso-2-naphthol [5]. According to this suggestion, the presence of para-and orto-substituted free phenol groups for the colour reaction is essential. Therefore the method can also be used for the quantitative determination of ortho - and para - substituted free phenol groups.

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ÖZET

Eugenolün kantitatif olarak çabuk tayini için, eugenol ile 1-nitroso-2-naftol arasındaki renk reaksiyonundan istifade edilerek basit ve hassas bir metot geliştirilmiştir.

Reaksiyona tesir eden muhtelif faktörler tesbit edilerek reaksiyon üzerine etkileri incelenmiş ve kantitatif tayin için optimum şartlar tesbit edilmiştir.

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