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Indirect spectrophotometric estimation of lidocaine using crystal violet

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Abstract. A simple, quick and perceptive method was developed to estimate Lidocaine in its pure and pharmaceutical forms spectrally using a spectrometer for ultraviolet visible rays, and the method was summarized: by oxidation of the drug using a mixture of bromate and potassium bromide KBr-KBrO3 with an acidic medium, where the remaining oxidizing agent reacts with the dye Crystal violet (CV) to be a dark blue product It gave peak absorption value at 612 nm, with Beer-lambert law (linear concentration) was between (15) and (100) µg/ml while the molar absorption coefficient was equal to (1945.022) L/mol/cm , limit of detection (0.36) µg/ml, while the quantity limit was (1.08) µg/ml. The method has been effictively applied to the measurement of Lidocaine in both its pure form and its pharmaceutical preparations.

1 Introduction

Lidocaine (Figure.1), The presence of 2-(diethylamino)-N-(2,6-dimethylphenyl) acetamide as a white powdered material and is soluble with ease in water [1]. It is used as a local anesthetic for tissues and as a treatment for ventricular tachycardia [2, 3]. It can also be used as an injection to block nerves. The effect of the drug usually starts within four minutes and lasts from half an hour to three hours [4]. Lidocaine can also be used as a direct anesthetic on the skin [3]. It is essential to use the drug with great caution due to the possibility of toxic side effects., which directly depends on the concentration of the drug in the human blood plasma [5]. Lidocaine was discovered in 1946 and sold in 1948 [6]. It is included as part of the World Health Organization for medications because it is considered effective and safe [7]. It is also not very expensive [8]. Lidocaine has been estimated in several ways to determine it, such as HPLC [9], UV- Vis Spectrophotometry [10] electrochemical detection [11] and gas chromatography [12].

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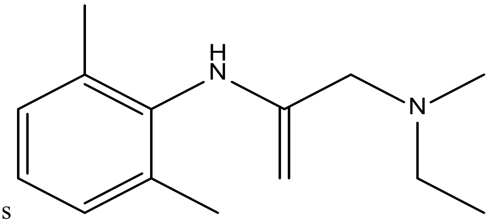

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Fig. 1. Lidocaine structure.

The research aims to find spectral analytical methods to estimate Lidocaine by oxidizing it in an acidic medium using potassium bromate and bromide and using Crystal violet dye (CV). This method was fast, simple and economical, and determination of Lidocaine in tablet form and pure.

2 Experimental section

2.1 Apparatus and materials

- UV-Vis. Spectrophotometer double beam. with 1cm glass cell's.
- Genesis UV10 Single-Beam UV-VIS Spectrometer.
- Lidocaine 99% / SDI; Samarra. Iraq.
- Crystal Violet 99% / Fluka.
- CH_3CH_2OH 99,9% / Merck.
- HCl 37% / Scharlan.
- KBr / Merck, and $KBrO_3$ / Fluka.

2.2 Solutions

- Lidocaine at concentration of 0.1 grams of LIDO in 100 milliliters of distilled water (1000 μg/ml). was dissolved.
- Crystal violet at concentration of 1×10^{-4} M: 0.002 gm was dissolved in fifty milliliters of purified water.
- HCl solution1.0 M, Distilled water is used to dilute 4.3 mL of concentrated acid (11.64 mol) to 50 mL.
- KBr-$KBrO_3$ (oxidizing agent): Take 0.5 grams of KBr and 0.0500 grams of $KBrO_3$, and dissolve this mixture in 50 ml of distilled water. Take 2.5 ml of this mixture and dilute it to 100 milliliters using pure water.

2.3 Process steps:

After preliminary tests to stabilize the optimal conditions for the reaction, they were achieved by adding 1.0 ml of Lidocaine solution at the concentration 500 μg/ml and In a volumetric flask with a capacity of 10 ml, followed by 1.0 ml of the mixture (KBr:$KBrO_3$, as moderate and acts as selective oxidant) to it. 5 minutes waiting, 0.5ml of HCl at a concentration of 1M, and 5 minutes waiting, 1 ml of tincture Crystal Violet was added Complete the volume to 10 ml using a solvent made entirely of ethanol. The blue output had a maximum absorption of 612 nm and a absorption of 0.576 in the room temperature, All experiments were made with $n \geq 3$. Figures (2) and (3).

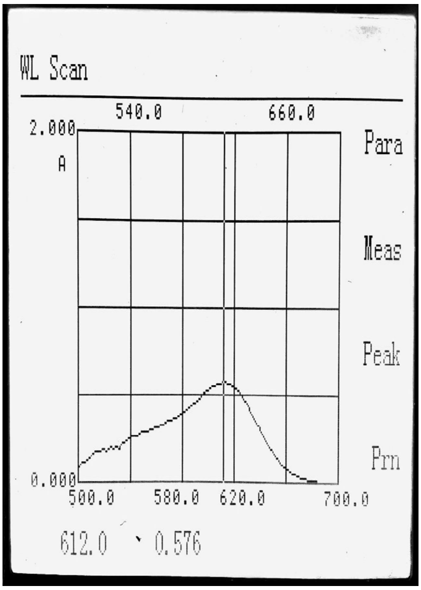

Fig. 2. Blank's absorption spectra in comparison to ethanol solvent.

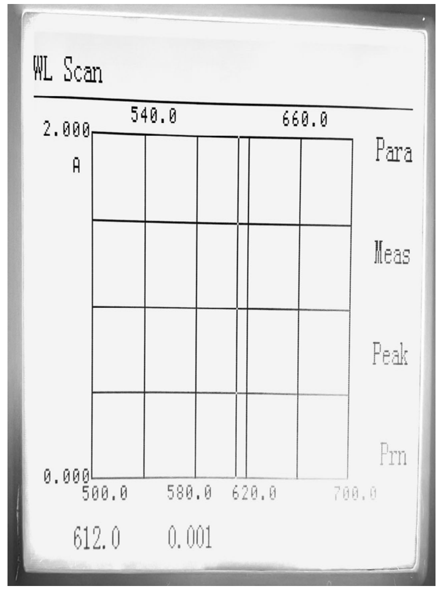

Fig. 3. Absorption spectrum of Lidocaine product against blank.

3 Results and discussion

3.1 Optimal conditions

3.1.1 Study of acid volume and its effect on product

The volume with optimal absorption was found after testing different volumes with a concentration of 1 M of hydrochloric acid, and the best production and absorption occurred as indicated in Figure (4), at a volume of 0.5 mL.

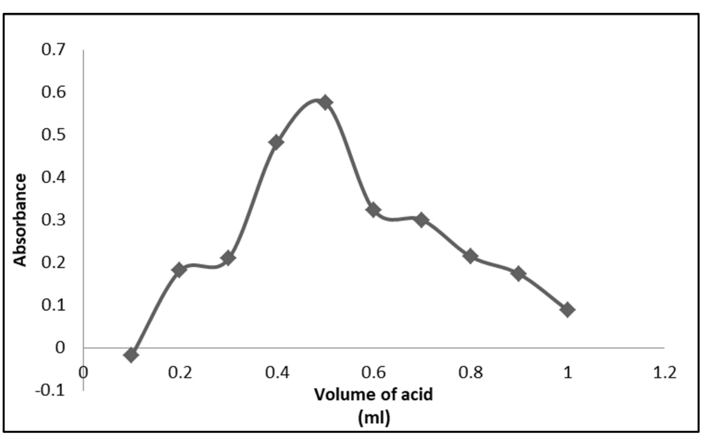

Fig. 4. Study of the impact volume of hydrochloric acid.

3.1.2 Study the effect of using different types of acids

A group of strong and weak acids (HCl, H2SO4, H1NO3, CH3COOH) were used, all of which were installed 1 molar and the Different types of acids were used to determine which acids The optimal absorption of the resultant substance was achieved.. The optimal absorption value was found when using HCl, according to table (1). In hydrochloric acid environments, a high concentration of chloride ions will cause coordination with the complex ion center, thus forming ionic pair reactions in the outer sphere, which enhances stability compared to other ions.

Table 1. Utilization of various acids and how they affect the product's absorption.

Acidic	Abs.
HCl	0.576
HNO_3	0.149
H_2SO_4	0.097
CH_3COOH	-0.182

3.1.3 Study the effect of the oxidizing agent on the resulting product:

The oxidizing agent consists of a mixture of bromate and potassium bromide, with various volumes added to find the best impact on the product's absorption values. Adding a volume of 1 ml resulted in good absorption, as demonstrated in the Figure 5 [5].

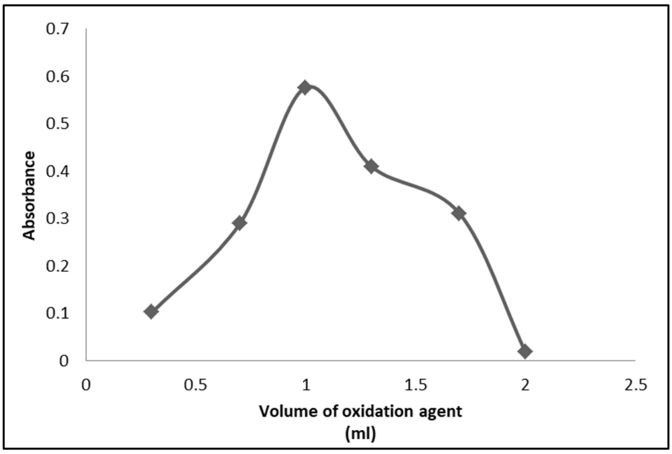

Fig. 5. Volume of oxidation agent.

3.2 Effect of (CV) dye volume :

The impact of dye volume on the final product's absorption yield was investigated; adding multiple volumes of the dye produced the best absorption, as seen in the Figure 6.

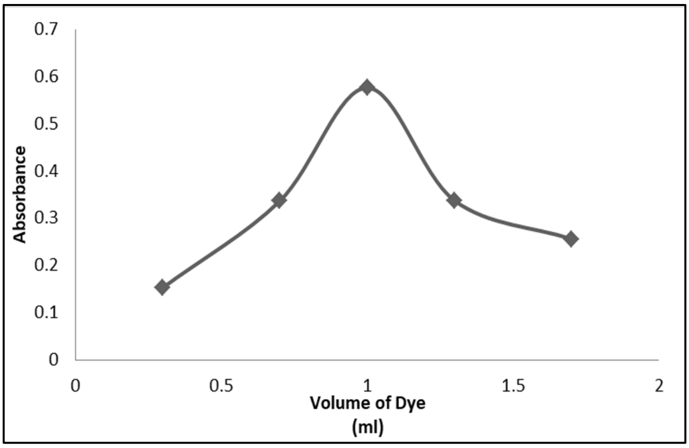

Fig. 6. Volume of dye (CV).

3.3 Effect of time

Through tests conducted found that absorption is stable of the product formed for a period of more than 24 hours approximately and at the same length is sufficient to make the measurements required for this study.

3.4 Calibration curve

In the pure form of the drug with Crystal Violet dye, linear titration curve appeared in a range of concentrations (15-135) µg/ml, Figure (7) it shows that.

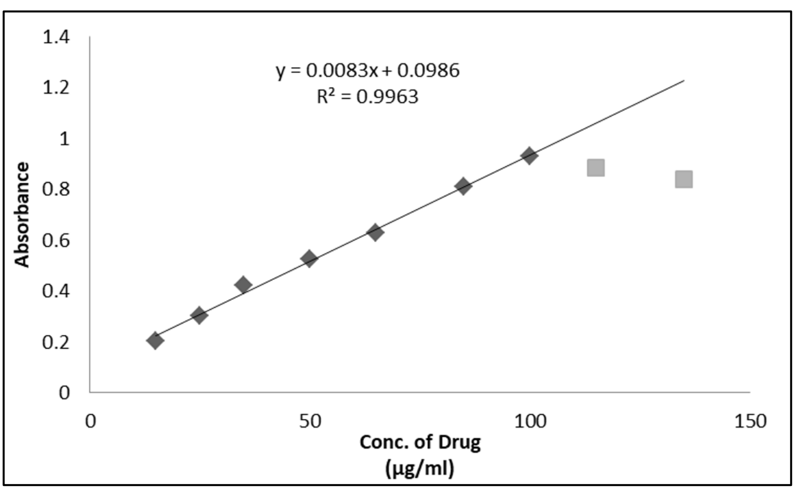

Fig. 7. Standard curve of the resulting product.

3.5 Possibility of applying the studied method:

Determination of Lidocaine in its medicinal formulations (as tablets). Table (2).

Table 2. Lido determination (as tablet).

Making pharmaceuticals	Content declared (µg)	discovered (µg) using the suggested approach	%Recovery
LIDO Injection	35	34.98	99.94
	50	49.81	99.62
	100	99.88	99.88

3.6 Product equivalence study

The "product equivalence" of the reaction of Lidocaine with Crystal Violet dye was studied under the optimum conditions. Figures (8) and (9) demonstrate the 1:1 equivalency ratio between lidocaine and the reagent.

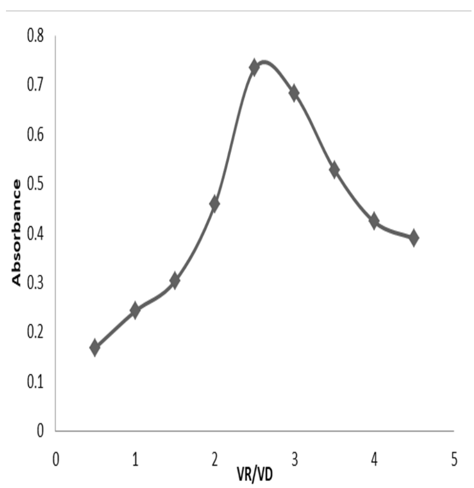

Fig. 9. Continuous variation method of Lido.

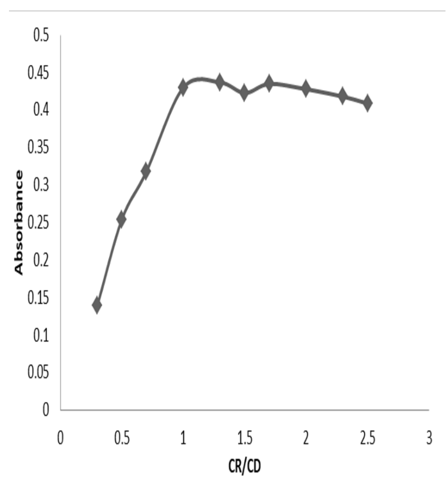

Fig. 8. The mole-ratio technique of Lido.

3.7 Proposed reaction equation

Suggested reaction according to how Lidocaine is oxidized by a mixture of potassium bromate and bromide ($KBr:KBrO_3$) [13] and the oxidation of the dye is completed with the remaining oxidizing agent , as shown in Figure 10. The detection limit for this study was equal to 0.36 µg/ml while the quantification limit was equal to 1.08 µg/ml. See Table 3.

$$BrO3- +5Br- +6H+ \longrightarrow 3Br- + 3H2O$$

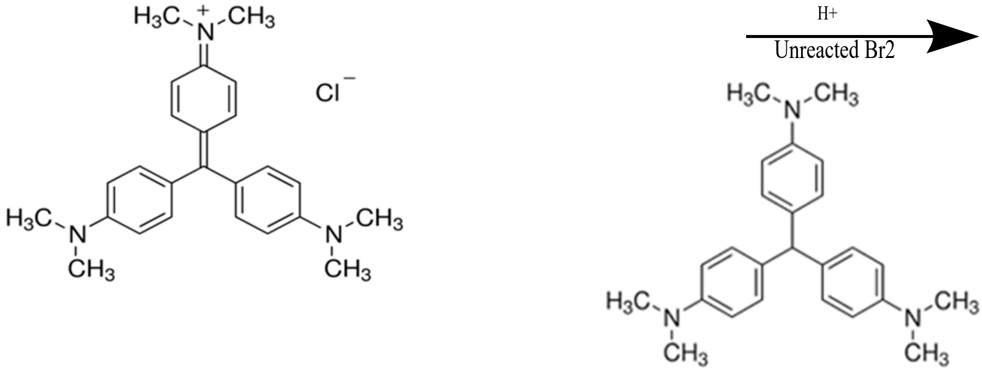

Fig. 10. Proposed mechanism for the decrease in (CV) dye.

Table 3. Features of the product that results from the reaction.

Parameters	Worth (crystal violat)
λmax(nm)	612
(L/mol.cm) Molar absorptivity	1945.022
coefficient of correlation (r)	0.9981
Detection Limit (µg/ml)	0.36
Quantification limit (µg/ml)	1.08
Slope	0.0083
%RSD	0.196

4 Conclusion

The proposed method for estimating lidocaine was quick, simple, economically inexpensive and also straightforward where working conditions were uncomplicated. It showed rapid results compared to other methods and therefore, It can be applied to the Determination of lidocaine in its Its pure shape and preparation.

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