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Quenching-Chemiluminescence New Method for Determination of Ethylmorphine by New Design Chemiluminescence Flow Injection System

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Abstract: A new flow-injection chemiluminescence (CL) method for the determination of ethylmorphine had been established in this study. Based on the chemiluminescence reaction ethylmorphine in hydrogen peroxide and oxidation with luminol in alkali condition, the CL intensity ofluminol decreased by adding ethylmorphine,the determination of ethylmorphine was developed using the quenching-chemi- luminescence by new design has been modulating of Spectroinc 20 to measure thechemiluminescence flow injection system .The proposed method was indirect, simple and rapid for determined the rang (1×10^{-1}-3.1×10^{-3}) M. The detection limit (3×noice) of 7.8×10^{-4} M,the R.s.d % for fife determination of 3.1×10^{-3} M ethylmorphinewas 0.64%. The applicability of the method was demonstrated by the determination ofethylmorphine in synthetic sample.

Keywords: Ethylmorphine, Chemiluminescence , Flow injection, Luminol, Hydrogen peroxide, pharmaceutical formulations.

Introduction

Chemiluminescence (CL) reactions are usually very rapid and therefore require rapid, reproducible mixing prior to detection. Flow injection analysis (FIA) can provide the necessary requirement of rapid, less contamination of samples, on-line elimination of interferences and possibility of using unstable reagents[1], high precision enhancement of on-line sensitivity, micro sample analysis, Low cost, high sample throughput , excellent accuracy [2,3], reproducible mixing for chemiluminescence. FIA also provides portable, low cost automated reactions, whilst CL provides rapid, sensitive and selective detection using simple instrumentation [4] . For CL the greatest sensitivity, the manifold and the flow cell should be configured to maximize the emission and detection of light when the reacting mixture passes through the flow cell [5]. It was observed that a chemical reaction produces a species in an electronically excited state that is produced during the course of a chemical reaction [6]. There are two forms of CL, direct CL where the species is promoted to an electronically excited state which emits light when it returns to the ground state and indirect CL, where the species transfers energy to another molecule which then re-emits light [7]. The analyte interacts with the CL reaction, usually as a reagent, a catalyst, a quencher, and even an enhancer[8, 9]. Ethylmorphine is one of the materials that have the ability to quenching the Chemiluminescence for luminol –H_2O_2 in aqueous alkaline conditions[10]. Ethylmorphineor ethylmorphiniumchloratum ($C_{19}H_{24}NO_3Cl$) figure 1, also known as dionine, ethomor-phine, codethyline or 3-o-ethylmorphine,hasthesystematicname7,8-didehydro-4,5a-epoxy-3-ethoxy-17-methylmorphinan-6a-ol-hydrochloride and has a molar weight of 313.40. Crystals can be isolated from ethanol. The published dissociation constant is pKa=8.08 at 15□C[11]. It is belong to a therapeutic category - analgesic, narcotic and antitussive which act on the central nervous system to relieve pain. When narcotics are used over a long period of time, the body becomes used to the narcotics and begins to require more of the drug to achieve the same relief. Homat- ropine belongs to anticholinergics and antispasmodics which are usually used to treat nausea, vomiting, abdominal cramps and stool motility problems. A knowledge of dissociation constant of these drugs is important[12]. Various methods

based on HPLC have been developed for determination of Ethylmorphine[R.W] in pharmaceutical, usedHPLC with post –column Chemiluminescence detection[13],used Ion-pair inpharmaceutical[14], determined by solid state for potentiometric[15].

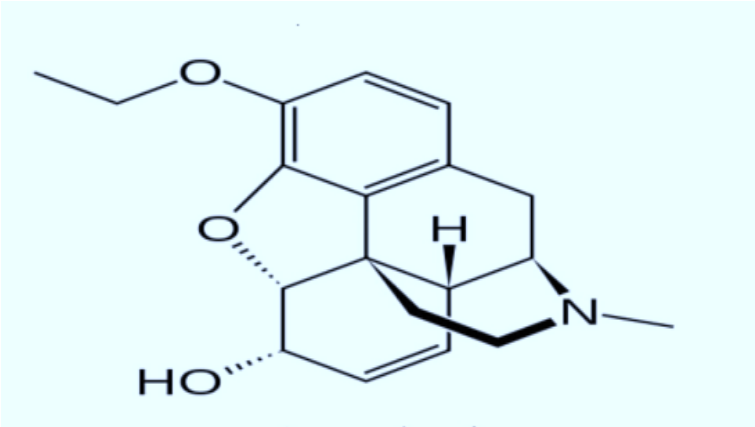


Fig1. *Chemical structure of Ethylmorphine*

The present study describes a sensitive and selective method for the determination of EthylmorphinebyChemiluminescence detection coupled with flow injection analysis system.

Experimental

All chemicals used were of analytical grade reagents. Deionized water was used, with conductivity less than 0.2 µS and all measurements were done at 25C°throughout this work.

1- 0.1M Na2 CO3 (Merck) buffer solution: was prepared by dissolving 10.50 g of Na2 CO3 and 0.5M NaOH(20 g) to exactly 1liter [16]

2- 0.02 M Luminol solution: (5-amino-2,3-dihydro-1,4_phthalazinedione) (Sigma-Aldrich , St. Louis, MO, USA) was prepared by dissolving 0.3544 g of luminol (97%) in 100 ml of 0.1M Na2 CO3 solution the buffer(pH 10) [17].

3- 0. 5M Hydrogen peroxide was prepared by diluting 14 ml of *ca*30% (d=1.13) hydrogen peroxide (Sigma, St. Louis, MO, USA) in 250 ml(138) in deionized water .

4- 0.1M Ethylmorphine hydrochloride two hydrate (Merck) solution was prepared by dissolving 3.7 g Ethylmorphine in 100 ml of 0.02 Hydrogen peroxide solution.

Instruments and flow system

Flow injection Chemiluminescence system (FI-CI)

Figure 2 shows the home –made FI-CL system which was used in this work previously described[18] Bidirectional pump (ISMATEC ,REGLO, ISM796,Switzerland) and 1.00 mm (i.d) standard pump tubing was used for the carrier stream. The sample was Injected by valve (V-7 Sweden) equipped with variable sample loop, Spectroinc20 (Sp20 Diagram courtesy of Bausch and Lomb, Rochester ,NY.) equipped with PMT as gas filled photo tube (CETRON CEA95) and flow cell was adapted to be CL detector. The measured CL signal was recorded as the peak height by (Siemens Kompenso graph model 7KC 1032-8BC). A home-made flow cell which is design in laboratory and teflon tube (0.5 mm i.d.) was used for the rest of the flow system.

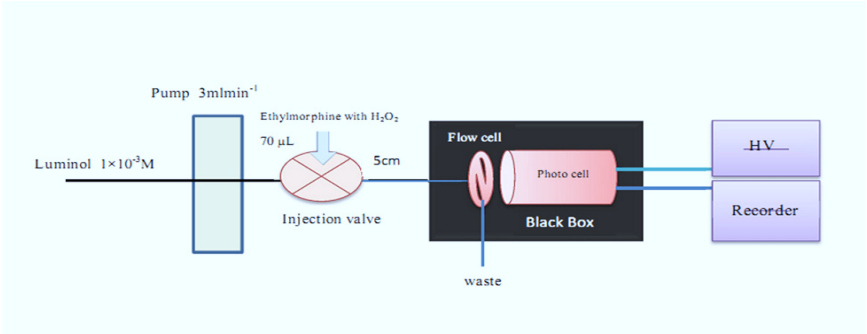


Fig2. *The home-made semi-automated FI CL system*

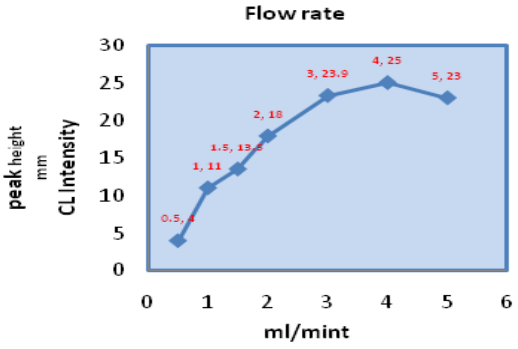
Procedure

In this technique 1× 10^{-3} M Luminol solution was the carrier stream and run into the manifold at flow rate (3 ml min-1). A (70 µL) as a sample volume of Ethylmorphine was injected manually through the injection valve into carrier stream. Reaction coil of 5 cm length was inserted before the detector. The recorded peak height is related to the concentration of injected sample. When preparing Ethylmorphine diluting a solution of 1 × 10^{-2} hydrogen peroxide interaction between them will happen[19], Ethylmorphine prevents hydrogen peroxide attack on Ethylmorphine to give less luster compared to hydrogen peroxide alone. Quenching-Chemiluminescence of the light was operation.

Result and Discussion

Optimization of manifold parameters and reagent concentrations

The FI manifold is optimized for rapid determinations of Ethylmorphine by conducting a series of experiments When the FI-CI system in Figure 2 was used the flow rate increased almost parabolically with increasing the flow rate (Figure 3), so 3 min h^{-1} was selected for subsequent work due to smooth and good reproducibility of the obtained peaks. The peak height increased almost parabolically with increasing of the in injected volume(Figure 4) between 10 -120 µL. The maximum peak height was obtained when 120µL was injected, but the peak shape was smooth distorted, so 70 µL was injected in subsequent experiment Figure 5 indicated that the peak height increased with increasing the hydrogen peroxide concentration in the rang 1×10^{-3}-1.0× 10^{-1} M. It is well know that increasing in hydrogen peroxide concentration leading to more intensity of chemiluminescence, so1.0×10^{-2}M was selected as an optimum concentration Figure6 indicated that the peak height creased with increasing of the luminol concentration in the rang 3×10^{-4}-5.0×10^{-3}M. It is well know that increasing in luminol concentration leading to low intensity of chemiluminescence because quenching-chemiluminescence of luminol, So1.0×10^{-3}M was selected as an optimum concentration[20].



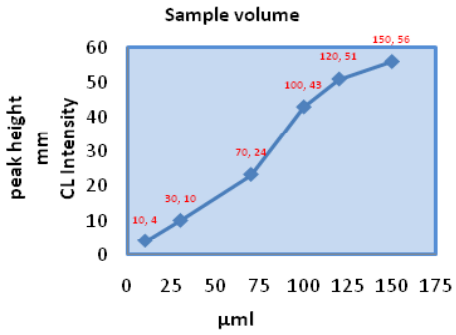
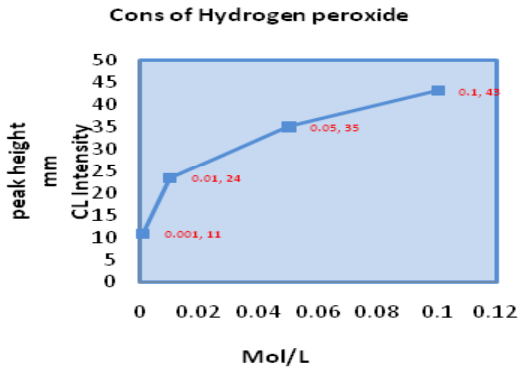


Fig3. *Effect of Flow rate on peak height*

Fig 4. *Effect of sample volume on peak height*



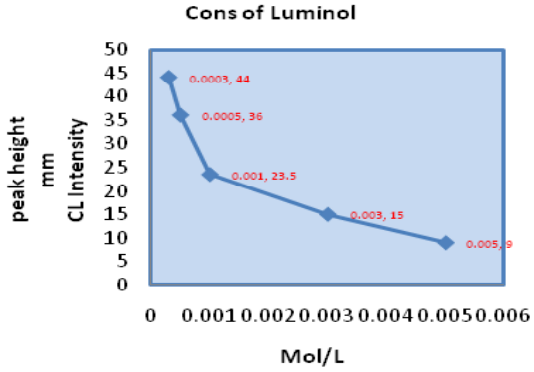


Fig5. *Effect of Conc.hydrogen peroxide on peak height*

Fig6. *Effect of luminal Conc. on peak height*

3.2. Calibration characteristics

Under the optimum operating conditions, analytical characteristics[21], a calibration graph(Figure 7) of Ethylmorphinewas constructed between the CL intensity (peak height, Y, mm)and the concentration(X) range (1×10^{-1} - M-3.1×10^{-3}M)in which graph was linear. The regression line of emission intensity (*I*, peak height)on Ethylmorphineconcentration (C)was *I* =38.51+19.87lnC (r^2=0.987 for 6 points) with detection limit of7.8×10^{-4}M. The r.s.d % for fife determination of 3.1×10^{-3} MEthylmorphinewas 0.64%. The sample throughput was 96 samples h^{-1}.

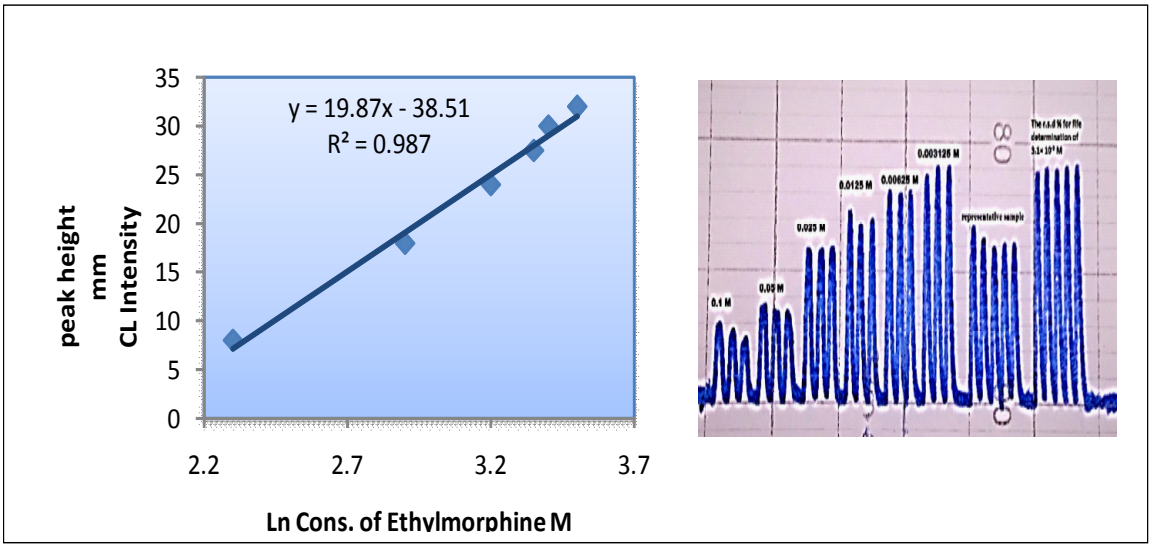


Fig7. *The corresponding calibration graph of Ethyl morphine*

Application of the method

In order to demonstrate the applicability of the proposed method for the determination of Ethylmorphine, the method was successfully applied to the analysis of Ethylmorphine in synthetic samples, as shown in Table(1), the assay results of proposed method were in good agreements with the declared contents, lack of analysis of some of the applications of the compound ethylmorphine because of the difficulty in obtaining blood or urine samples from some of the people who use this narcotic substance banned in our country, which only can be used for laboratory and special committees and approved by the university and the government exclusively.

Table1. *Determination of Ethylmorphine in synthetic sample*

sample	Conc. Of EthylmorphineM		Error%	Recovery % RSD%
	Taken	Found*		
1	5×10^{-2}	4.91×10^{-2}	-1.8	98 ± 1,24
2	2.5×10^{-2}	2.47×10^{-2}	-1.2	99 ± 1

*Average of fife determinations per sample.

Accuracy

In order to established the accuracy and validity of the home –made FI CL system used to determine a several representative samples were examined analysis by using standard additions method[22]. This method was used to avoid all possible interferences (Table 1). The average recoveries were in the range (98.00 - 99.00 %) to average five measurements. When compare this work withchromatographic method[23] find a good agreements between the results was obtained.

Conclusion

A new flow-injection chemiluminescence method for the determination of ethylmorphine had been established in this paper , ethylmorphine determination of was developed using the quenching-chemi- luminescence, the result were very good, high sensitivity and reproducibility when it was applied to determination some representative samples of ethylmorphine.

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