

Figure 5: Gas filled photo tube Fig.6 A circuit of an operational amplifier



Figure 7a, b: The PMT was attached directly to the home-made glass flow coil, c glass flow, d covered with sheet of aluminum foil and black chamber of PMT

2.3 Procedure

The carrier stream was 1×10^{-3} M Luminol solution run into the manifold (Fig.2) at flow rate 3 ml min^{-1} . A $60 \mu\text{L}$ as a sample volume of hydrogen peroxide was injected manually through the injection valve into carrier stream. Reaction coil

of 5 cm length was inserting before the detector and the chemiluminescence of the hydrogen peroxide sample was measured by the home-made system. The recorded peak height can be related to the concentration of injected sample

3. Result and discussion

3.1 Optimization of Manifold Parameters and Reagent Concentrations

The FI manifold is optimized for rapid determinations of hydrogen peroxide by conducting a series of experiments. When the chemiluminescence FI system in (Fig 2) was used, the flow rate increased almost parabolically with increasing the flow rate (Fig 8), 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 injected volume (Fig 9) between 10 -150 μL . The maximum peak height was obtained when 120 μL was injected, but the peak shape was smooth distorted, so 60 μL was injected in subsequent experiment (Fig 10) indicated that the peak height increased with increasing the luminol concentration in the range 3×10^{-4} - $5.0 \times 10^{-3} \text{ M}$. It is well known that increasing in luminol concentration leading to more intensity of chemiluminescence. So $1.0 \times 10^{-3} \text{ M}$ was selected as an optimum concentration¹⁹.

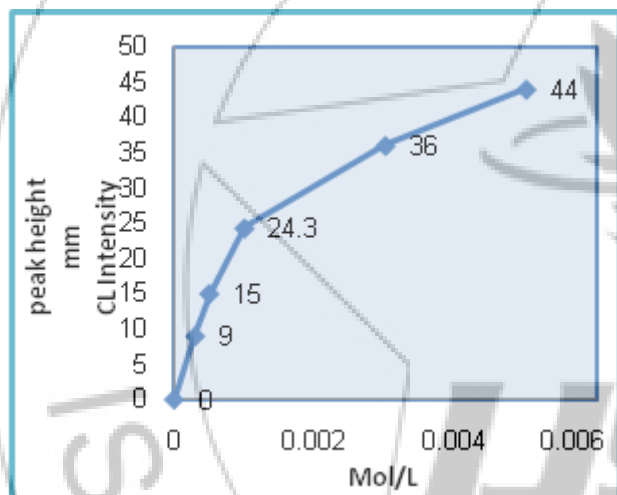


Figure 10: Effect of luminol Conc. on peak height

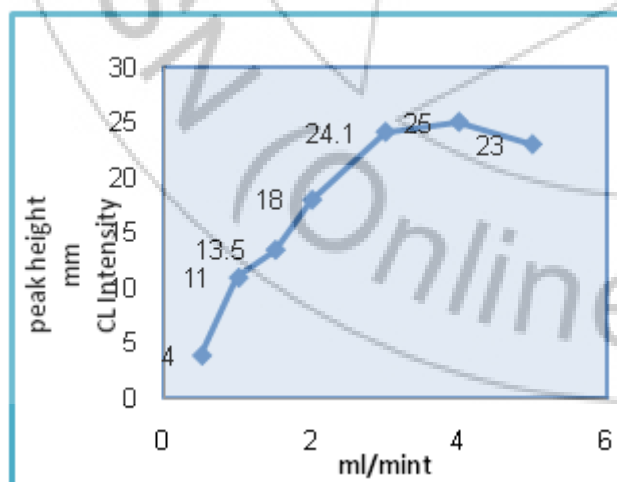


Figure 8: Effect of Flow rate on peak height of H_2O_2

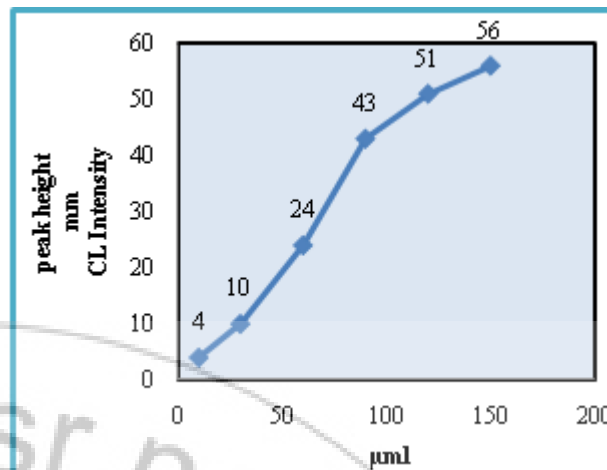


Figure 9: Effect of sample volume on peak height of H_2O_2

3.2. Calibration Characteristics

Under the optimum operating conditions, analytical characteristics²⁰ such as reproducibility and detection limit were investigated for hydrogen peroxide. A calibration graph of hydrogen peroxide was constructed between the CL intensity (peak height, Y, mm) and the concentration (X) range ($3.3 \mu\text{gml}^{-1}$ - $106 \mu\text{gml}^{-1}$) in which graph was linear. This linear range allows the determination of $4 \mu\text{gml}^{-1}$ of hydrogen peroxide. The regression line of emission intensity (I, peak height) on hydrogen peroxide concentration (C) was $I = 2.0061 + 0.4101C$ ($r^2 = 0.9943$ and $r = 0.9987$, for 6 points) with detection limit ($3 \times \text{noise}$) of $1.6 \mu\text{gml}^{-1}$. The r.s.d % for five determinations of $26.5 \mu\text{gml}^{-1}$ hydrogen peroxide was 0.35%. The sample throughput was $110 \text{ samples h}^{-1}$. The method was tested for several of synthetic samples of hydrogen peroxide. Some of the analytical results are given in the Table(1)

Table 1: Determination of H_2O_2 in synthetic sample

sample	Conc. Of H_2O_2 , μgml^{-1}		Error%	RSD%
	Taken	Found*		
1	10.0	9.85	-1.5	0.57
2	85.0	85.11	+0.13	0.57

* Average of five replicates.

The accuracy and precision of the synthetic sample was tested by analyzing in two different concentrations of H_2O_2 for five replicates. The values of the percentage errors (E%) and percentage relative standard deviation (RSD%) are summarized in Table (1). These values indicate the high accuracy and precision of the synthetic sample.

3.3. Application of the method

In order to demonstrate the applicability of the proposed method for the determination of H_2O_2 , the method was successfully applied to the analysis of H_2O_2 in pharmaceutical formulation, as shown in Table(2). The assay results of proposed method were in good agreement with the declared contents. In (EAU OXYGÉNÉE, Gifrer) quantitative recoveries between 98 and 98.62% were obtained; but presenting of recovery was low obtained, because this formulation is considered combined phosphoric acid, sodium salicylate and sodium stannate as well as

compounds which greatly affect the CL intensity²² due to the formation of stable complexes or insoluble compounds,

Table 2: Application of the proposed method for determination of H₂O₂ in pharmaceutical solution

pharmaceutical Solution	Conc. Of H ₂ O ₂ , µgml ⁻¹		Error %	Recovery %	RSD %
	present	Found*			
EAU OXYGÉNÉE, Gifrer (stabilisé 20 volumes) 6%	40	39.2	-1.93	98	0.69
	85	83.8	-1.35	98.62	0.4

*Average of five replicates.

3.4 Evaluation of the Proposed Method

For evaluating the competence and the success of the proposed method in the analysis of Hydrogen peroxide in pharmaceutical formulation, the results obtained were compared with those obtained by chemiluminescence flow injection method²¹. In which a quantity of solution there was good agreement between the methods.

4. Conclusion

In this paper, the design has been constructing and modulating of Spectroinc 20 to measure the home-made semi-automated chemiluminescence flow injection system so results were very good, high sensitivity and reproducibility when it was applied to determination some pharmaceuticals that contain hydrogen peroxide.

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