Utility of molecular complex formation and redox reaction for micro determination of some alkaloids

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Contains the introduction which includes two parts : The first part gives idea about the definitions, action, metabolism, chemical structures, chemical names, and character of the studied drugs (Codeine, Caffeine, Nalbuphine, Heroin, Strychnine and Quinine).The second part gives a literature survey of the previous studies for the analysis of the studied drugs including Spectrophotometeric , Spectrofluorimeteric, Ultra-violt spectrophotometeric, Electro analysis and Chromatographic methods.Chapter.(2)Contains the experimental part which includes apparatus used for measurement and procedures for the preparation of the drugs and reagents. It also contains the proposed spectrophotometeric methods for determination the studied drugs in pure form, in dosage form, and in postmortem cases in the presence of their oxidative degredates, also it contains pharmacopeias and official methods for analysis of the studied drugs.Chapter.(3)Contains the results and discussion which includes 6 parts.The first part includes -spectrophotometeric procedure for determination of the studied drugs using N bromosuccinimide(NBS)The proposed methods are based on oxidation of the studied drugs by NBS and determination of the unreacted NBS by measuring the decrease in absorbance of amaranth and acid red73 dyes . the following experimental variables are investigated1- Effect of acid concentration2-Effect of the time and temperature3-Effect of the sequence of addition4-Effect of KBr concentration5- Effect of dye concentrationBeer’s law is obeyed in the concentration range 0.4 – 3.5, 0.2 – 3.0, 0.4 – 6.0, 0.5 – 5.0, 1.5 – 10.0, 0.5 – 4.5 µg ml-1 for Cod, Caff, Nalb, Her, Stry and Quin respectively in case of amaranth dyes but in case of Acid Red 73 dye, Beer’s law is obeyed in the concentration range 0.6 – 2.5, 0.5 – 2.5, 0.5 – 3.0, 0.5 – 4.0, 1.1 – 8.0, 1.0 – 5.0 µg ml-1 for the studied drugs in the same above sequence.The stoichemetric ratio of the studied drugs with NBS are established using the molar ratio method and found to be 1 : 3, 1 : 5, 2 : 5, 1 : 2, 1 : 1, 2 : 3, for Cod, Caff, Nalb, Her, Stry and Quin to NBS in case of amaranth dye.But in case of acid Red 73 dye. The molar ratio method are found to be 1 : 6, 1 : 5, 1 : 3, 1 : 3, 2 : 3, 1 : 2 for the studied drugs to NBS in the above sequence.The second part include spectrophotometeric procedures for determination of the studied drugs using Cerric sulphate Ce(SO4)2. The proposed methods are based on oxidation of the studied drugs by Ce(SO4)2 and determination of the unreacted Ce(SO4)2 by measuring the decrease in absorbance of Chromotrope 2R the

following experimental variable are investigated.1-Effect of acid concentration2-Effect of temperature3-Effect of time4-Effect of sequence of addition5-Effect of dye concentrationBeer's law is obeyed in the concentration ranges 0.1 – 1.4, 0.1 – 1.6, 0.4 – 1.5, 0.2 – 1.8, 1.0 – 10.0 µg ml-1 for Cod, Caff, Nalb, Her, Stry and Quin respectively detection and quantitation limits are calculated the stoichemetric ratio of the studied drugs with Ce(SO4)2 are established using the molar ratio method found to be 1 : 23, 1 : 25, 1 : 22, 1 : 22, 1 : 19, 2 : 1 for Cod, Caff, Nalb, Her, Stry and Quin to Ce(SO4)2 respectively.The third part includes spectrophotometeric procedures for determination of the studied drugs using KMnO4. The proposed method are based on oxidation of the studied drugs by KMnO4 and determination of the unreacted KMnO4 by measuring the decrease in absorbance MB dye.The experimental variable are investigated1-Effect of potassium permanganate concentration2-Effect of sulphuric acid concentration3-Effect of time and temperature4-Effect of sequenced of addition5-Effect of dye concentrationThe forth part include spectrophotometric procedures for determination of the studied drugs (Cod, Nalb, Her) using tris 1.10 phenanthroline iron(III) ion complex the proposed methods are based on oxidation of the studied drugs and formation of tris 1.10 phenanthroline iron.(II) ion complex which had red color. The intensity of the color were measured spectrophotometrically, the following experimental variable are investigated.1-Effect of PH of the solution .2-Effect of the volume of Reagent3-Effect of temperature and heating timeBeer's law is obeyed in the concentration range 0.5 – 5.0, 0.4 – 4.5, 0.6 – 5.5 for the drugs Cod, Nalb, Her respectively.The fifth part include spectrophotometric procedures for determination of the studied drugs (Cod, Nalb, Her) using, tris 2.2- bipryrdyl iron(III) ion complex. The proposed methods are based on oxidation of the studied drugs and formation of tris 2.2- bipryrdyl iron(II) ion complex which had red color. The intensity of the color where measured spectrophotometrically, the following experimental variable are investigated.1-Effect of PH of the solution2-Effect of the volume of Reagent3-Effect of temperature and heating timeBeer's law is obeyed in the concentration range 1.0-6.0, 1.0-10.0, 0.9-8.0 for the drugs Cod, Nalb, Her respectively.For more accurate results Ring boom, optimum concentration ranges are determined, molar absorptivity, sandal sensitivity, detection and quantation limit are calculated.In order to determine the precision of the proposed methods, solutions containing three different concentrations of the studied drugs are prepared and analyzed in six replicates, the recovery, the relative standard deviation, the relative error and the confidence limits are calculated. The proposed methods are successfully applied to determine the studied drugs in the pure form and in their dosage form, the results obtained are compared. Statistically by student's t-lest (for accuracy) and variance ratio F test (for precision) with the official method at 95% confidence level. The results showed that the t-and F-values are less than the critical tabulated value indicating that there is no significant difference between the proposed and official methods. Thus the proposed spectrophotometric methods can applied in routine analysis for determination of the studied drugs in pure and doge form and in postmortem cases.The sixth part included GC chromatography using specific thermal programming which capable of separating the studied drugs in different retention times (Cod, Caff, Nalb, Her, Stry, Quin and Mor) at 9.709, 7.061, 12.946,

9.421, 15.967, 8.219 and 10.012 respectively the studied drugs were injected into a capillary column Hp5 , Helium as a carrier gas by the ratio 0.5 ml/min using FID detectorThe developed method revealed high linearity with correlation coefficient more than 0.999.In order to determine the accuracy and the precision of the proposed method three different concentration of the pure drug were analyzed six replicate. The accuracy were (98.5 – 102.85) with SD of not more than 87.0%.The applied work which include giving a specific dose from each drug to albino rates either by injection or oral. After (3-4) hours the animals sacrificed and the internal organs (brain, Liver, Kidney, hart and blood, were taken and the drugs were extracted from them by the ammonium sulphate method and organic solvents using (liquid/liquid) extraction Then determination of each drugs in each organs by the proposed spectrophotometic and the proposed gas chromatographic method.