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Determination and validation of some important drugs by some molecular spectrometric analysis.

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LIST OF ABBREVIATIONS AND SYMBOLS

AOAC: Association of analytical communities
Abs: Absorbance
ACN: Acetonitrile
AZ: Azithromycin
A^{O}: Angstrom
API: Active pharmaceutical ingredients
CTC: Charge transfer complex
CAA: Chloranilic acid
CHL: Chloranil
CNDO/2: Complete neglect of differential overlap/2
CGMP: Current good manifacturing practices
DCM: Dichloromethane
DVD: Diaveridine
DDQ: 2,3-dichloro-5.6-dicyano-1,4- benzoquinone
EDA: Electron donor-acceptor
EPA: Environmental Protection Agency
ESR: Electron Spin Resonance
FDA: Food and drug administration
EV: electron volt
ETOH: Ethanol
FD : Forced degradation
HPLC: High performance liquid chromatography
ICH: International Conference on Harmonization
IR: Infrared
IP: Ionization potential
ISO/IEC: International organization of standardization/International electro technical commission
I_2: Iodine
K_f : Equilibrium constant
LC: Liquid chromatography
LOD: Limit of detection
LOQ: Limit of quantitation
mm: Millimeter
mAU: Milli absorbance unit
MW: Molecular weight
nm: Nanometer
NMR: Nuclear magnetic resonance

ODS: Octadeceylsilane
PA: Picric acid
Pm: Picometer
QC: Quality control
RPG: Repaglinide
RSD: Relative standard deviation
RCRA: Resource conservation and recovery act
SDI: Sulphadimidine
TCNE: Tetracyanoethylene
TCNQ : Tricyanoquinodimethane
UV-VIS: Ultraviolet visible
UPLC: Ultra Performance Liquid Chromatography
USP: United state pharmacopeia
WHO: World Health Organization
W-h/m2: Watt- hour per square meter
ε: Molar absorptivity

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Aim of the work

The present thesis aimed to study new spectroscopic and analytical methods for the following determination of some pharmaceutical compounds:

(1) Diaveridine (DVD), it is antibacterial and antiprotazoal drug.

(2) Sulphadimidine (SDI), it is an antibacterial intestinal drug.

(3) Azithromycin (AZ), it is used to treat bacterial infections in many different parts of the body.

(4) Repaglinide (RPG), it is antidiabetic drug.

(5) Granisteron hydrochloride (GRAN), it is antiemetic drug, **using the following techniques:**

UV-Vis, IR, Raman spectroscopy, NMR, elemental analysis and HPLC

It is aimed also to describe some validated procedures for the trace quantification of these drugs in pharmaceutical preparations without the necessity for samples pretreatment or time consuming extraction or evaporation steps prior to analysis of the drugs.

Abstract

Simple, rapid and sensitive spectrophotometric and chromatographic procedures were developed for the analysis of diaveridine, sulphadimidine, azithromycin, repaglinide and granisteron hydrochloride in pure form as well as in their pharmaceutical formulations using charge transfer (CT) complexes formed between the drugs and iodine (I_2) as σ- acceptor and picric acid (PA), tetracyanoethylene (TCNE) as π-acceptors in dichloromethane.

Job's method of continuous variation was used to detect the stoichiometric ratio of the formed complexes. The formation constant and molar absorptivity were determined by Benesi-Hildebrand equation.

The validation of analytical procedures like accuracy, precision, LOD, LOQ, linearity, repeatability and Robustness of drugs were determined.

The formed solid charge transfer complexes between the two drugs and iodine were prepared and their structures were investigated through elemental analysis, IR, Raman spectroscopy and NMR spectra.

Also HPLC methods were developed to determine the concentrations of these drugs in bulk and pharmaceutical formulation forms.

Key words: Diaveridine, Sulphadimidine, Azithromycin, Repaglinide and Granisteron hydrochloride, charge transfer complexes, pharmaceutical analysis, spectrophotometry, HPLC.