NEW ION SELECTIVE ELECTRODES FOR THE DETERMINATION OF PARASYMPATHOMIMETICS PHARMACEUTICAL COMPOUND DISTIGMINE BROMIDE

A Thesis Presented

To

Faculty of Science
Cairo University

By

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B. Sc. Chemistry (1987)
M. Sc. Analytical Chemistry (2006)

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فإن التقوى يحصل بها زيادة الهدى والعلم والحفظ حيث

يفتح الله على الإنسان من العلوم مالا يفتحها لغيرة

وكذلك اليسر والسهولة في أموره

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Title of Ph. D. thesis:

New ion selective electrodes for the determination of parasympathomimetics pharmaceutical compound distigmine bromide

Name of the Candidate: Amal Mohamed Fouad Abd El Haleim Khorshid

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ABSTRACT

Title of the thesis:

New ion selective electrodes for the determination of parasympathomimetics pharmaceutical compound distigmine bromide

Name of the candidate: Amal Mohamed Fouad Abd El Haleim Khorshid.

Degree: Ph. D., Faculty of Science, Cairo University.

This work has been carried out to present and characterize new plastic membrane, coated wire and chemically modified carbon paste electrodes for the determination of parasympathomimetics pharmaceutical compound; distigmine bromide. The electrodes are based on individual, mixed and or additives with ion-exchangers formed by the ion-associations of the drug cation with the anions phosphomolybdate, phosphotungstate, silicomolybdate, silicotungstate, tetraphenylborate, reineckate and or picrate. The electrodes were constructed and fully characterized in terms of composition, life span, response time, usable pH range, working concentration range and temperature according to the IUPAC recommendation.

Each electrode was applied to the potentiometric determination of distigmine cation in pure solution, pharmaceutical preparation, dissolution profile or in urine in batch or in flow injection (FI) conditions.

Keywords: (1) parasympathomimetics drug, (2) distigmine bromide, (3) ion-exchanger, (4) potentiometry, (5) PVC-electrode, (6) coated wire electrode, (7) chemically modified carbon paste electrode, (8) dissolution, (9) flow injection analysis (FIA).

Supervisors: Prof. Dr. Y. M. Issa, Prof. Dr. Hosny Ibrahim, and Prof. Dr. M. A. El Ries

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Amal M. Fouad Abd El Haleim Khorshid

Dedication

To my lovely family

My beloved husband

My lovely children

Ahmed and Menna

LIST OF ABBREVIATIONS

$DsBr_2$	Distigmine bromide
PM	Phosphomolybdic
PT	Phosphotungstic
SM	Silicomolybolic
ST	Silicotungstic
TPB	Tetraphenylborate
Rein	Reineckate
$(Ds)_3(PM)_2$ or Ds_3PM_2	Distigmine phosphomolybdate
$(Ds)_3(PT)_2$ or Ds_3PT_2	Distigmine phosphotungstate
$(Ds)_2(SM)$ or Ds_2SM	Distigmine silicomolybdate
$(Ds)_2(ST)$ or Ds_2ST	Distigmine silicotungstate
$Ds(TPB)_2$ or $Ds\text{-}TPB_2$	Distigmine tetraphenylborate
$Ds(Rein)_2$ or $Ds\text{-}Rein_2$	Distigmine reineckate
$Ds(Pi)_2$ or $Ds\text{-}Pi_2$	Distigmine picrate
PMA	Phosphomolybdic acid
PTA	Phosphotungstic acid
SMA	Silicomolybolic acid
STA	Silicotungstic acid
NaTPB	Sodium tetraphenylborate
AmmRein	Ammonium reineckate
PiH	Picric acid
KTPB	Potassium tetraphenylborate
β-CD	β-Cyclodexterine
DBP	Dibutylphthalate
DOP	Dioctylphthalate
TBP	Tributylphosphate
TCP	Tricresylphosphate
PVC	Polyvinylchloride
THF	Tetrahydrofuran
CWE	Coated wire electrode
SCE	Saturated calomel electrode

CPE	Carbon paste electrode
CMCPE	Chemically modified carbon paste electrode
FIA	Flow injection analysis
ISE	Ion selective electrode
USP	United States Pharmacopeia
FIP	First Industrial Pharmacists
C	Carbon
G	Graphite
Ds-PM	Distigmine PVC electrode based on distigmine phosphomolybdate
Ds-PT	Distigmine PVC electrode based on distigmine phosphotungstate
Ds-SM	Distigmine PVC electrode based on distigmine silicomolybdate
Ds-ST	Distigmine PVC electrode based on distigmine silicotungstate
Ds-TPB	Distigmine PVC electrode based on distigmine tetraphenylborate
Ds-Rein	Distigmine PVC electrode based on distigmine reineckate
Ds-Pi	Distigmine PVC electrode based on distigmine picrate
Ds-(PM+TPB)	Distigmine PVC electrode based on distigmine phosphomolybdate mixed with distigmine tetraphenylborate
Ds-(ST+TPB)	Distigmine PVC electrode based on distigmine silicotungstate mixed with distigmine tetraphenylborate
Ds-ST+NaTPB	Distigmine PVC electrode based on distigmine silicotungstate mixed with sodium tetraphenylborate
Ds-PT+KTPB	Distigmine PVC electrode based on distigmine phosphotungstate mixed with potassium tetraphenylborate
Ds-PT+β-CD	Distigmine PVC electrode based on distigmine phosphotungstate mixed with β-Cyclodexterine

Ag/Ds-PM	Distigmine coated silver electrode based on distigmine phosphomolybdate
Ag/Ds-PT	Distigmine coated silver electrode based on distigmine phosphotungstate
Ag/Ds-SM	Distigmine coated silver electrode based on distigmine silicomolybdate
Ag/Ds-ST	Distigmine coated silver electrode based on distigmine silicotungstate
Ag/Ds-TPB	Distigmine coated silver electrode based on distigmine tetraphenylborate
Ag/Ds-Rein	Distigmine coated silver electrode based on distigmine reineckate
Ag/Ds-Pi	Distigmine coated silver electrode based on distigmine picrate
Ag/Ds-(PM+TPB)	Distigmine coated silver electrode based on distigmine phosphomolybdate mixed with distigmine tetraphenylborate
Ag/Ds-(ST+TPB)	Distigmine coated silver electrode based on distigmine silicotungstate mixed with distigmine tetraphenylborate
Ag/Ds-ST+NaTPB	Distigmine coated silver electrode based on distigmine silicotungstate mixed with sodium tetraphenylborate
Ag/Ds-PT+KTPB	Distigmine coated silver electrode based on distigmine phosphotungstate mixed with potassium tetraphenylborate
Ag/Ds-PT+β- CD	Distigmine coated silver electrode based on distigmine phosphotungstate mixed with β-Cyclodexterine
Ag-AgCl/Ds-PM	Distigmine coated silver-silver chloride electrode based on distigmine phosphomolybdate
Ag-AgCl/Ds-PT	Distigmine coated silver-silver chloride electrode based on distigmine phosphotungstate
Ag-AgCl/Ds-SM	Distigmine coated silver-silver chloride electrode based on distigmine silicomolybdate
Ag-AgCl/Ds-ST	Distigmine coated silver-silver chloride electrode based on distigmine silicotungstate
Ag-AgCl/Ds-TPB	Distigmine coated silver-silver chloride electrode based on

	distigmine tetraphenylborate
Ag-AgCl/Ds-Rein	Distigmine coated silver-silver chloride electrode based on distigmine reineckate
Ag-AgCl/Ds-Pi	Distigmine coated silver-silver chloride electrode based on distigmine picrate
Ag-AgCl/Ds-(PM+TPB)	Distigmine coated silver-silver chloride electrode based on distigmine phosphomolybdate mixed with distigmine tetraphenylborate
Ag-AgCl/Ds-(ST+TPB)	Distigmine coated silver-silver chloride electrode based on distigmine silicotungstate mixed with distigmine tetraphenylborate
Ag-AgCl/Ds-ST+NaTPB	Distigmine coated silver-silver chloride electrode based on distigmine silicotungstate mixed with sodium tetraphenylborate
Ag-AgCl/Ds-PT+KTPB	Distigmine coated silver-silver chloride electrode based on distigmine phosphotungstate mixed with potassium tetraphenylborate
Ag-AgCl/Ds-PT+β- CD	Distigmine coated silver-silver chloride electrode based on distigmine phosphotungstate mixed with β-Cyclodexterine
Cu/Ds-PM	Distigmine coated copper electrode based on distigmine phosphomolybdate
Cu/Ds-PT	Distigmine coated copper electrode based on distigmine phosphotungstate
Cu/Ds-SM	Distigmine coated copper electrode based on distigmine silicomolybdate
Cu/Ds-ST	Distigmine coated silver electrode based on distigmine silicotungstate
Cu/Ds-TPB	Distigmine coated copper electrode based on distigmine tetraphenylborate
Cu/Ds-Rein	Distigmine coated copper electrode based on distigmine reineckate
Cu/Ds-Pi	Distigmine coated copper electrode based on distigmine picrate

Cu/Ds-(PM+TPB)	Distigmine coated copper electrode based on distigmine phosphomolybdate mixed with distigmine tetraphenylborate
Cu/Ds-(ST+TPB)	Distigmine coated copper electrode based on distigmine silicotungstate mixed with distigmine tetraphenylborate
Cu/Ds-ST+NaTPB	Distigmine coated copper electrode based on distigmine silicotungstate mixed with sodium tetraphenylborate
Cu/Ds-PT+KTPB	Distigmine coated silver electrode based on distigmine phosphotungstate mixed with potassium tetraphenylborate
Cu/Ds-PT+β- CD	Distigmine coated copper electrode based on distigmine phosphotungstate mixed with β-Cyclodexterine
Cu-CuS/Ds-(PM+TPB)	Distigmine coated copper-copper sulfide electrode based on distigmine phosphomolybdate mixed with distigmine tetraphenylborate
G(GH2B, G2B, GB, GHB)/Ds-PM	Distigmine coated graphite electrode based on distigmine phosphomolybdate (four pencil rods)
G(GH2B, G2B, GB, GHB)/Ds-PT	Distigmine coated graphite electrode based on distigmine phosphotungstate (four pencil rods)
G(GH2B, G2B, GB, GHB)/Ds-SM	Distigmine coated graphite electrode based on distigmine silicomolybdate (four pencil rods)
G(GH2B, G2B, GB, GHB)/Ds-ST	Distigmine coated graphite electrode based on distigmine silicotungstate (four pencil rods)
G(GH2B, G2B, GB, GHB)/Ds-TPB	Distigmine coated graphite electrode based on distigmine tetraphenylborate (four pencil rods)
G(GH2B, G2B, GB, GHB)/Ds-Rein	Distigmine coated graphite electrode based on distigmine reineckate (four pencil rods)
G(GH2B, G2B, GB, GHB)/Ds-Pi	Distigmine coated graphite electrode based on distigmine picrate (four pencil rods)
G(GH2B, G2B, GB, GHB)/Ds-(PM+TPB)	Distigmine coated graphite electrode based on distigmine phosphomolybdate mixed with distigmine tetraphenylborate (four pencil rods)
G(GH2B, G2B, GB, GHB)/Ds-(ST+TPB)	Distigmine coated graphite electrode based on distigmine silicotungstate mixed with distigmine tetraphenylborate

	(four pencil rods)
G(GH2B, G2B, GB, GHB)/Ds-ST+NaTPB	Distigmine coated graphite electrode based on distigmine silicotungstate mixed with sodium tetraphenylborate (four pencil rods)
G(GH2B, G2B, GB, GHB)/Ds-PT+KTPB	Distigmine coated graphite electrode based on distigmine phosphotungstate mixed with potassium tetraphenylborate (four pencil rods)
G(GH2B, G2B, GB, GHB)/Ds-PT+β-CD	Distigmine coated graphite electrode based on distigmine phosphotungstate mixed with β-cyclodextrine (four pencil rods)
GC/Ds-PM	Distigmine coated glassy carbon electrode based on distigmine phosphomolybdate
GC/Ds-PT	Distigmine coated glassy carbon electrode based on distigmine phosphotungstate
GC/Ds-SM	Distigmine coated glassy carbon electrode based on distigmine silicomolybdate
GC/Ds-ST	Distigmine coated glassy carbon electrode based on distigmine silicotungstate
GC/Ds-TPB	Distigmine coated glassy carbon electrode based on distigmine tetraphenylborate
GC/Ds-Rein	Distigmine coated glassy carbon electrode based on distigmine reineckate
GC/Ds-Pi	Distigmine coated glassy carbon electrode based on distigmine picrate
GC/Ds-(PM+TPB)	Distigmine coated glassy carbon electrode based on distigmine phosphomolybdate mixed with distigmine tetraphenylborate
GC/Ds-(ST+TPB)	Distigmine coated glassy carbon electrode based on distigmine silicotungstate mixed with distigmine tetraphenylborate
GC/Ds-ST+NaTPB	Distigmine coated glassy carbon electrode based on distigmine silicotungstate mixed with sodium tetraphenylborate

GC/Ds-PT+KTPB	Distigmine coated glassy carbon electrode based on distigmine phosphotungstate mixed with potassium tetraphenylborate
GC/Ds-PT+β- CD	Distigmine coated glassy carbon electrode based on distigmine phosphotungstate mixed with β-Cyclodexterine
Ds-CMCPE	Distigmine chemically modified carbon paste electrode
emf	Electromotive force
LOD	Limit of detection
MPM	Matched potential method
SSM	Separate solution method
r	Correlation coefficient
$\mathbf{t_{resp}}$	Response time
$\mathbf{(dE^{o}/dt)}$	Thermal coefficient
τ	Life time
$\mathbf{V_{inj}}$	Injection volume
$\mathbf{F_m}$	Flow rate
$\mathbf{a_J}$	The activity of the added interferent
S	Solubility (mol/l)
RSD	The relative standard deviation
D	The dispersion coefficient
s	Response time
S	Standard deviation
$\mathbf{K_{SP}}$	Solubility product constant
RPM	Revolutions per minute
$\mathbf{D^{2+}}$	The drug cation
$\mathbf{A^{n-}}$	The counter anion
$\mathbf{S_p}$	The pooled standard deviation
μ Ω cm	Resistivity
$\mathbf{Sm^{-1}}$	Conductivity, where 1 Siemens, $S = \Omega^{-1}$ = mho = 1/ohm

CONTENTS