Since MPH and HDH ionise in aqueous solution into protonated drug moiety and chloride ions, both drugs could be titrated conveniently with $AgNO_3$. The results in Table 1 indicate that MPH reacts with $AgNO_3$ in 1:1 molar ratio, whereas HDH does so in 1:2 ratio. The titration methods were applied to the analysis of pharmaceutical preparations containing MPH and HDH. The percent recoveries with RSD values are shown in Table 2. The potentiometric method was found to be more precise and accurate compared to visual and conductometric end-point detection methods. Excipients such as lactose, starch, magnesium stearate and talc were found not to interfere in the determination. From the results compiled in Table 2, it can be concluded that the titrimetric methods described in this paper are suitable for the determination of MPH and HDH in various pharmaceutical preparations through the titration of chloride content. The analysis can be carried out within a few minutes and therefore be used for quick routine analysis.

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Spectrophotometric Determination of Sildenafil Citrate in Pharmaceutical Dosage Forms

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Two simple extractive spectrophotometric methods have been developed for the estimation of sildenafil citrate in both pure and pharmaceutical dosage forms. These methods are based on the formation of ion association-pair complexes of the drug with two acid dyes namely orange-II and erichrome black-T in acidic medium followed by their extraction in to chloroform. The absorbance of the chloroform layers was measured at their respective wavelength of maximum absorbance against the corresponding reagent blank. The method has been statistically evaluated and is found to be precise and accurate.

Sildenafil citrate (SLD) designated chemically as 1-[[3-(6,7-dihydro-1-methyl-7-oxo-3-propyl-1H-pyrazolo-[4,3-d]-pyrimidin-5-yl)-4-ethoxyphenyl]sulfonyl]-4-methylpiperazine citrate, which is a selective inhibitor of cyclic guanosine

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monophosphate (cGMP) specific phosphodiesterase[1] (type V) and now it is being extensively used for curing impotence in the case of penile dysfunction[2]. The drug is not official in any pharmacopoeia. Literature survey reveals the presence of only a single RP- HPLC[3] method for the determination of SLD in the presence of its oxidative degradation products and two RP-HPLC[4,5] methods in human plasma.

Although spectrophotometric methods are the instrumental method of choice commonly used in industrial laboratories, no colourimetric method has been reported so far for the determination of SLD. Therefore the need for a fast, low cost and selective method is obvious, especially for routine quality control analysis of pharmaceutical formulations, containing SLD. The proposed extractive spectrophotometric methods are based on the formation of ion-pair complexes with orange-II and erichrome black-T.

A Systronics UV/vis spectrophotometer, model 117 with 10 mm matched quartz cells was used for all spectral measurements. A Systronics model-361 pH meter was used for all pH measurements. All chemicals used were of analytical grade. Orange II (O-II, 1.41×10^{-3}M) was prepared by dissolving 500 mg of O-II in 100 ml of distilled water and erichrome Black-T (EBT, 2.17×10^{-2} M) was prepared by dissolving 100 mg of EBT in 100 ml of distilled water. These solutions were treated with chloroform to remove any chloroform soluble impurities if present.

For the preparation of standard drug solution, about 100 mg of the SLD was accurately weighed and dissolved in 100 ml of distilled water in a standard volumetric flask to obtain a stock solution of 1 mg/ml. This solution was further diluted with the distilled water to get working standard solutions of 100 µg/ml (O-II) and 250 µg/ml (EBT).

Volumes of standard SLD solution ranging from 0.5-3.0 ml (1 ml=100 µg for O-II and 1 ml=250 µg for EBT) were transferred into a series of 125 ml separating funnels. To that 0.5 ml of HCl (0.1 N) and 0.5 ml of O-II dye (1.41×10^{-3} M) or 3.0 ml of HCl (0.1 N) and 3.0 ml of EBT dye (2.17×10^{-2} M) were added to each separating funnel and the total volume of the aqueous phase was made up to 10 ml with distilled water. Chloroform (10 ml) was added to each funnel and the contents were shaken for 2 min. The two phases were allowed to separate and the absorbance of chloroform

TABLE 1: OPTICAL CHARACTERISTICS AND PRECISION DATA.

Parameters	Orange-II	EBT
Beer's law limit (µg/ml)	5.0-40.0	12.5-75.0
Molar absorptivity (l/mol.cm)	1.4622×10^4	0.756×10^4
Sandell's sensitivity (µg/cm² absorbance unit/0.01)	0.04559	0.08818
Regression equation (y)*		
Slope (b)	0.0221	0.0110
Standard deviation on slope (S_b)	2.4816×10^{-4}	1.0998×10^{-4}
Intercept (a)	-0.00003	0.0148
Standard deviation on Intercept (S_a)	4.5080×10^{-3}	5.35×10^{-3}
Standard error of estimation (Sc)	4.7735×10^{-3}	5.75×10^{-3}
Correlation coefficient (r)	0.9998	0.9998
Relative standard deviation (%)[b]	0.8720	0.4514
% Range of error[b]		
(95% confidence limit)	0.7291	0.3774
(99% confidence limit)	1.0787	0.5584
Detection limit (µg/ml)	0.6109	0.4832

* With respect to Y= a+bC, where C is concentration (µg/ml) and Y is absorbance, [b] Eight replicate samples.

layer was measured at 495 nm for O-II and 520 nm for EBT against their respective reagent blanks. The amount of SLD present in the sample solution was computed from calibration curve.

The amount of SLD present in pharmaceutical formulations was determined by taking an amount of tablet powder equivalent to 100 mg of the SLD into a 100 ml volumetric flask and the volume was made up with distilled water and filtered. Appropriate aliquots of drug solution were taken and the assay procedure was followed for analysis of drug content. The results of analysis are given in Table 2. The amount of SLD per tablet was calculated by comparing the standard and sample at wavelength of maximum absorption by using the following formula.

Amount of SLD (mg)/tablet = $(A_2 x D_1 x W/A_1 x D_2 x S) \times M$ where, A_1 is the absorbance of the standard solution, A_2 is the absorbance of the sample solution, D_1 is the dilution factor for the standard, D_2 is the dilution factor for the sample, W is the weight of the standard taken, S is the weight of the sample taken and M is the average weight of the tablet.

To study the accuracy and precision of the method recovery experiments were carried out using standard addition method. The recovery of the added standard was studied at three levels i.e. 10%, 20% and 30% of the labelled claim of the tablet. Each level was analyzed in a similar manner as described earlier. Each set of addition was repeated six times and the percent recovery for the added standard was calculated by using following formula. Percentage recovery $= N [(\Sigma XY)-(\Sigma X)(\Sigma Y)/N (\Sigma X^2)-(\Sigma X)] \times 100$ where, N is the number of observations, X is the amount of the standard drug added per tablet and Y is the amount of the standard drug found per tablet.

The optical characteristics such as Beer's law limits, Sandell's sensitivity, molar extinction coefficient, percent relative standard deviation, (calculated from the eight measurements containing 3/4th of the amount of the upper Beer's

TABLE 2: DETERMINATION OF SILDENAFIL CITRATE IN COMMERCIAL PHARMACEUTICAL FORMULATIONS.

Tablets	Reagent	Label claim (mg)	Amount found mg		
			Proposed method	Reference method*	% Recovery **
EDEGRA	O-II	25	24.92	24.93	99.68
	EBT		24.90	24.87	99.60
SILAGRA	O-II	25	24.95	24.92	99.80
	EBT		25.09	25.11	100.36
CAVERTA	O-II	25	24.97	24.95	99.88
	EBT		25.02	24.82	100.08
EDEGRA	O-II	50	49.99	49.96	97.98
	EBT		49.89	50.02	99.78
SILAGRA	O-II	50	50.01	49.67	100.02
	EBT		49.99	50.06	99.98
CAVERTA	O-II	50	49.91	49.82	99.82
	EBT		49.79	49.93	99.58
PENEGRA	O-II	100	100.1	99.68	100.1
	EBT		99.81	99.95	99.81

* UV method developed in our laboratory, ** Average of six determinations.

law limits), % range of error (0.05 to 0.01 confidence limits) were calculated for all the methods and the results are summarized in Table 1. The values obtained for the determination of SLD in several pharmaceutical formulations (tablets) by the proposed and reported methods are compared in Table 2. Interference studies revealed that the common excipients and other additives usually present in the dosage form did not interfere in the proposed methods. In conclusion the proposed extractive spectrophotometric methods for the estimation of SLD are simple, sensitive, cheap, accurate and may found useful in the routine quality control analysis and quantitative determination of sildenafil from its pharmaceutical preparations.

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Antimicrobial Activity of Newly Synthesized Organic Complexes

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Halogenated organic complexes containing chromium as metal chelate, prepared by solvent extraction method were studied for antimicrobial activity. Solutions of the complexes were prepared with alcohol, acetone and chloroform and used in the study. Preliminary screening was done by disc diffusion method. Minimum inhibitory concentration and phenol co-efficient of the complexes was studied by tube dilution method. Of the 15 complexes studied (5 from each of Cl, Br and F), tetraphenyl phosphonium halochromate and tetraphenyl arsonium halochromate were found to be effective against 9 pathogenic bacterial strains, that include *Escherichia coli, Shigella sonnei, Shigella dysenteriae, Shigella flexnerae, Shigella boyedii, Salmonella typhimurium, Klebsiella sp., Pseudomonas aeruginosa and Vibrio cholerae.* The fluoro substituted complexes of chromium and arsenic were found to be most effective among the three halogenated organic complexes. The phenol co-efficient of the above two complexes was determined to be 1.60.

Laboratory-synthesized chemicals have long been used as antimicrobial agents, as antiseptics and disinfectants[1-3]. The usefulness of most of these agents became limited due to the development of resistance by various microorganisms[4-6]. Therefore, the present study has been taken up to investigate the antimicrobial efficiency of some newly synthesized organic complexes. The complexes studied contain cyclic components such as phenol, pyridine, quinoline and tetrazole to which halogens and metal ions have been attached as activity enhancing functional groups[7]. Minimum inhibitory concentrations (MIC), nature of toxicity and phenol co-efficient of the complexes are determined in this

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