mg/ml (regression coefficient r = 0.9896) for atenolol and 0.02 - 0.17 mg/ml (r=0.9998) for timolol.

The accuracy of the method was tested by recovery experiments on known amounts of bulk drug. The proposed method gave recoveries of 99.0±0.9 and 99.54±0.43 for atenolol and timolol respectively which were comparable to those obtained by the official method (99.10±0.69) for atenolol and 99.87±0.78 for timolol).

Various dosage forms of the two drugs were assayed by the proposed as well as by the official method (Table 1). The recovery values are based on the amount found and those calculated to be present according to the nominal concentration of the product. Pharmaceutical excipients likely to be present in the dosage forms e.g. starch, lactose, talc, magnesium stearate in tablets and buffers, preservatives in eye-drops exhibited no interference during the assay procedure. The proposed method us useful in routine analysis and quality control of these drugs.

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Spectrophotometric determination of Pentazocine in pharmaceutical formulations

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Four simple and sensitive visible spectrophotometric methods have been described for the determination of pentazocine, based either on the formation of chloroform soluble, coloured ion-association complex between pentazocine and the dye [Alizarine Red S, λ_{max} : 435 nm or Tropaeolin 000, λ_{max} : 490 nm] or by finding the oxidant (N-bromosuccinimide or potassium permanganate) reacted with pentazocine using a dye [Celestine Blue λ_{max} : 540 nm or Fast Green FCF, λ_{max} : 625 nm].

A survey of literature revealed a few reported visible spectrophotometric methods[1-13] for the estimation of pentazocine (PZ). This paper describes the development of four more visible spectrophotometric methods for its determination by exploiting its basic

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behaviour and reducing characteristics. Methods A and B are based on the formation of chloroform soluble ion-association complex between the drug and the dye [Alizarine Red S (ARS; method A) or Tropaeolin 000 (TP 000, method B)]. Methods C and D are indirect ones, Method C, involving the addition of excess

Table : 1 Optical characteristics, precision and accuracy

Parameter	Proposed Methods			
	A	B	C	D
λ_{max} (nm)	435	490	540	625
Beer's law limits (μg.ml^{-1})	1.0-5.0	2.5-15.0	0.4-5.0	0.8-4.0
Molar absorptivity (lit. mole^{-1} cm^{-1})	5.319 x 10^4	1.798 x 10^4	3.15 x 10^4	2.52 x 10^4
Sandell's sensitivity				
(μg cm^{-2}/0.001 absorbance unit)	5.36 x 10^{-3}	1.587 x 10^{-2}	9.04 x 10^{-3}	1.12 x 10^{-2}
Regression equation (Y)**				
Slope (b)	1.93 x 10^{-1}	6.521 x 10^{-2}	1.28 x 10^{-1}	8.86 x 10^{-2}
Intercept (a)	-3.1 x 10^{-2}	-1.67 x 10^{-2}	-7.44 x 10^{-2}	1.5 x 10^{-3}
Standard error of estimation (Se)	0.35	0.36	0.18	0.13
Correlation coefficient (γ)+	0.9997	0.9995	0.9995	0.9993
Relative standard deviation (%)*	0.34	0.47	0.47	0.83
Range of error* (%, 95% confidence limit)	0.36	0.49	0.49	0.87

** $Y = a + bc$ where C is concentration in μg/ml and Y in absorbance units
* Six replicate samples.
+ 9 points

N-bromosuccinimide (NBS) and determining the consumed NBS with decrease in colour intensity of the dye, Celestine Blue (CB) and Method D is based on the addition of excess potassium permanganate (KMnO$_4$) and determining the unconsumed Permanganate using the dye, Fast Green FCF (FG FCF).

A Milton roy spectronic 1201 UV-Visible spectrophotometer and a systronics 106 digital spectrophotometer with 1 cm matched quartz cells were used for all absorbance measurements. An Elico L1 120 digital pH meter was used for pH measurements. Aqueous solutions of ARS (0.2 %), TP 000 (0.2%), HCl (0.1 M or 5 M), CB (2.75 x 10^{-4} M), KMnO$_4$ (2.0 x 10^{-3} M) in 2.0 M H$_2$SO$_4$, FG FCF (1.23 x 10^{-4} M) and sodium sulphate (1.0 M) were prepared. One mg/ml stock solution of PZ was prepared by dissolving 100 mg of the drug in 100 ml of either chloroform (for methods A and B) or distilled water (for methods C and D). Working standard solutions were obtained by appropriate dilution of stock solution with the same solvent used for stock preparation (100 μg/ml, methods A and B; 20 μg/ml, methods C and D).

For pharmaceutical preparations, an amount of tablet powder or a volume of injection equivalent to 100 mg of active ingredient was extracted with 4 x 15 ml of chloroform (methods A and B) or distilled water (methods C and D), filtered and made upto 100 ml with the same solvent to give the concentration of 1 mg/ml. The stock solutions were further diluted to get working standard solutions and were analysed as described under the procedure for bulk samples.

To a series of 125 ml separating funnels containing aliquots (1.0-5.0 ml, method A; 0.25 - 1.5 ml, method B) of PZ (100 μg/ml), 0.1 M HCl solution (2.0 ml for method A, 6.0 ml for method B) and dye solution (2.0 ml of ARS for method A; 1.0 ml of TP 000 for method B) were successively added to each and the total volumes of aqueous and chloroform layers were adjusted to 15 ml and 10 ml respectively. The two phases were allowed to separate and the separated chloroform layer was dried over sodium sulphate and the absorbances were measured at appropriate λ_{max} (435 nm : method A or 490 nm: method B) against the corresponding reagent blank within the

Pharmaceutical Formulations	Labelled amount (mg)	Amount Found* (mg)					% Recovery by #proposed methods			
		Proposed methods				Reference[7] method				
		A	B	C	D		A	B	C	D
Tablets I	25	24.71±0.36	24.83±0.27	24.92±0.34	29.83±0.30	24.97±0.43	98.5±0.35	98.7±0.61	100.2±0.64	99.2±0.39
		F = 1.42	F = 2.5	F = 1.59	F = 2.05					
		t = 0.18	t = 0.25	t = 0.39	t = 0.15					
II	25	25.10±0.21	24.82±0.39	24.96±0.24	24.92±0.33	25.13±0.36	100.2±0.72	99.7±0.29	99.8±0.12	99.9±0.53
		F = 2.9	F = 1.17	F = 2.25	F = 1.19					
		t = 1.05	t = 0.92	t = 0.71	t = 0.29					
Injections I	30	29.81±0.19	29.75±0.24	30.21±0.18	29.84±0.16	30.12±0.26	99.1±0.36	99.6±0.55	100.2±0.53	99.8±0.49
		F = 1.8	F = 1.17	F = 2.08	F = 2.64					
		t = 0.65	t = 0.21	t = 1.01	t = 0.62					
II	30	29.91±0.31	30.3±0.29	30.1±0.39	29.90±0.36	30.0±0.35	99.1±0.45	99.8±0.32	100.6±0.51	99.8±0.55
		F = 1.27	F = 1.45	F = 1.24	F = 1.05					
		t = 0.21	t = 0.39	t = 0.81	t = 0.13					

* Average ± standard deviation of six determinations, the t- and F- values refer to compression of the proposed method with the reference method.

Theoretical value at 95% confidence limit : t = 2.27 and F = 5.05

Recovery of 10 mg added to the pharmaceutical preparation (average of six determinations)

stability period (1 min - 3 hrs; Method A or 1 min-2 hrs; method B). The amount of PZ was computed from the appropriate calibration curve.

To different aliquots of PZ solution (1.0 - 5.0 ml; 20 µg/ml) taken in a series of 25 ml calibrated flasks, 1.25 ml of 5M HCl and 2.5 ml of NBS were added and the volume was made upto 10.0 ml with distilled water in each flask. After 20 min, 15 ml of CB was added and mixed thoroughly and the absorbances were measured after 5 min at 540 nm against distilled water. Blanks were prepared appropriately. The decrease in absorbance corresponding to consumed NBS and, in turn, to drug concentration, was obtained by subtracting the absorbance of the blank solution from that of the test solution. The calibration graph was drawn by plotting the decrease in the absorbance of the dye (CB) against the amount of drug. The drug concentration in any sample was read out from the calibration graph.

Aliquots of standard PZ solution (1.0-5.0 ml; 20 µg/ml) were transferred into a series of 25 ml calibrated flasks. To each flask 0.6 ml of $KMnO_4$ solution was added and the volume was made upto 10 ml with distilled water and kept aside for 10 min. Then 4.0 ml of FG FCF solution was added, kept for 2 min and then 4.0 ml of 1 M sodium sulphate solution was added and made upto the mark with distilled water and the absorbance was measured against distilled water after 15 min at 625 nm. A blank experiment was carried out in a similar manner omitting PZ. The decrease in absorbance corresponding to PZ was obtained by subtracting the absorbance of the blank from that of the test solution. The amount of PZ present was calculated from its calibration graph.

Beer's law limits, molar extinction coefficient, Sandell's sensitivity and regression characteristics of proposed methods are presented in Table 1. The relative standard deviation and % range of error at 95% confidence level are also given in Table 1. The values obtained by the proposed and reference[7] methods for the estimation of PZ in pharmaceutical formulations are compared in Table 2 and are in good agreement. These methods can be used for the routine determination of PZ in pharmaceutical formulations depending upon the situation and availability of chemicals.

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