

injections the average content of cefazolin was 249.60 mg/inj (labelled amount 250 mg/inj) and relative standard deviation was  $\pm 0.147\%$  respectively. The average content of cefotaxime was 249.68 mg/inj (labelled amount 250 mg/inj) and relative standard deviation was  $\pm 0.191\%$  respectively.

The results of the recovery analysis (standard addition) for cefazolin and cefotaxime are tabulated in table 1. From the results it can be revealed that there is a good correlation between amount of standards added and total amount of drugs found at all concentration levels and hence it shows that there is no interference from the excipients used in the formulation. The mean recovery obtained for cefazolin and cefotaxime were between 99.93-100.95% and 98.46-99.29% respectively.

The proposed method is simple, precise and sensitive for simultaneous determination of cefazolin and cefotaxime from injections. The proposed method gives a good resolution between cefazolin and cefotaxime within a short analyses time (< 10 min) and is nowhere involves use of complex instruments or cumbersome sample preparation.

The proposed method is less expensive and takes much less time for the equilibration of the system as compared with the reported methods, therefore, it can be easily used for the simultaneous determination of these drugs from their injections.

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## Pulse Polarographic determination of Dapsone in Tablets

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The sulphone group present in dapsone is smoothly and quantitatively reduced at dropping mercury electrode in dimethylformamide medium in the presence of pyridinium perchlorate, yielding a diffusion controlled wave at -1.50 V vs SCE, has been made the basis of present method. The determination has been made by normal pulse and differential pulse polarography by linear calibration plots.

**D**APSONE, 4, 4-diaminodiphenyl sulphone is a drug used in the treatment of all forms of leprosy and dermatitis herpetiformis. It is also a bacteriostatic against a wide range of bacteria, but mainly used against *Mycobacterium leprae*. The official (IP) method<sup>1</sup>, involving nitrite titrations is not only semimicro but also nonspecific

and is also used for the assay of other sulphur drugs. Other methods include HPLC<sup>2,3</sup>, reversed phase HPLC<sup>4</sup> and bioassay using *Bacillus subtilis* BGA spores<sup>5</sup>, but these methods have primarily been developed for the determination of residues of dapsone in foodstuffs such as milk, but no efforts have been made to analyse

Table 1: Precision and recovery test for Dapsone tablets containing 100 mg per tablet\* active ingredient

S.No.	Amount taken $\mu\text{g l}^{-1}$	Dapsone Added and Found* in tablets				Average Recovery*, % age			
		Amount added $\mu\text{g l}^{-1}$	Expected ( $\mu\text{g l}^{-1}$ )		Found* ( $\mu\text{g l}^{-1}$ )		Average Recovery*, % age		
			NPP	DPP	NPP	DPP	NPP	DPP	
1.	10.0	-	10.0	10.0	9.96	9.97	99.6 $\pm$ 0.3	99.7 $\pm$ 0.3	
2.	80.0	-	80.0	80.0	79.98	80.02	100.0 $\pm$ 0.2	100.0 $\pm$ 0.4	
3.	15.0	25.0	40.0	40.0	39.74	39.88	99.4 $\pm$ 0.3	99.7 $\pm$ 0.4	
4.	20.0	30.0	50.0	50.0	49.93	50.02	99.9 $\pm$ 0.5	100.1 $\pm$ 0.2	
5.	20.0	40.0	60.0	60.0	59.94	59.99	99.9 $\pm$ 0.4	100.0 $\pm$ 0.3	

+ Maker's specification established separately by IP method<sup>1</sup>

\* Values are mean of three determinations with standard deviation( $\pm$ )

commercial drug formulations. Though, sulphone group in drug is susceptible to polarographic reduction<sup>6,9</sup>, but cannot be determined accurately by conventional aqueous polarography owing to the insolubility of drug in water. Thus, its determination by non-aqueous pulse polarography is advantageous. Of the various organic solvents and supporting electrolytes investigated for studying polarographic reduction of dapsone, dimethylformamide (DMF)-pyridinium perchlorate (Py ClO<sub>4</sub>) has been found to be the most suitable system, as in this system a linear base line having plateau parallel to potential axis is obtained over a wide range of potentials. The determination has been made by normal pulse (NPP) and differential pulse (DPP) polarography, by linear calibration plots.

Polarograms were recorded with Elico Pulse Polarograph Model CL-90 coupled with a X-Y polarocord Model LR-108. The electrode system consists of dropping mercury electrode (DME) as working electrode, modified calomel electrode (methanolic potassium chloride solution used instead of aqueous) as a reference electrode and a coiled platinum wire as an auxiliary electrode.

Calibration graphs have been prepared for pure compound using a stock standard solution (10<sup>-5</sup> M) prepared in DMF. Aliquots (0.1-1.0 ml) of stock solution were added to polarographic cell containing 90 ml Py.C1O<sub>4</sub> (0.01 M) and final volume made to 100 with DMF. Nitrogen gas was bubbled through each solution for 5 min. The

Polarogram of each solution was then recorded with the instrumental parameters as: initial applied voltage = 0; IR compensation = 0; height of mercury pool - 3.5 cm; pulse amplitude = 50 mV; droptime = 1. sec; scan rate = NOR, acquisition = fast; O/P zero = 500 (for NPP) and 485 (for DPP); time constant = 20 m sec.; temperature = 20  $\pm$  1°, and on polarocord: X-axis = 200 mV/cm and Y-axis = 200 mV/cm. A blank Polarogram was also recorded. All chemicals used were of AnalaR grade. In the analysis of dapsone tablets, a known number of tablets (say 20) were weighed and finely ground. Stock standard solution was prepared by dissolving accurately weighed amount equivalent to 2.5 mg of dapsone in 25 ml DMF. Working standard solution was prepared by diluting 0.1 ml of stock solution to 10 ml with the same solvent. Aliquots of this solution were taken in polarographic cells and processed as described above. In order to check the accuracy of the method, known amounts of pure dapsone were also added to the weighed formulation of drug and processed similarly. Results are given in Table 1.

The proposed method for the determination of dapsone is quite sensitive and can be employed for the determination of as little as 0.02  $\mu\text{g l}^{-1}$  of dapsone. Current-concentration relationship for both NPP and DPP are found to be linear upto 100  $\mu\text{g l}^{-1}$  and 2  $\mu\text{g l}^{-1}$  respectively. Dapsone in the range 10-80  $\mu\text{g}$  could be determined both by NPP and

DPP with maximum relative standard deviations of 0.8 and 0.6% respectively. When applied to the assay of dapsone tablets, the recoveries were in the range 99.4-100.1% of the nominal content with RSD's in the range 0.2-0.5% (Table 1).

The proposed method is advantageous over the official method in that, it is fairly selective towards the presence of sulphonamides and sulpha drugs, which otherwise interfere in the IP method<sup>1</sup>. The other advantage which the present method offers is that it can be applied to a wide range of concentrations in the same sample solution simply by altering the sensitivity control.

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