and standardised[6]. The dye CHDMAS was synthesised[7] from 5-sulphoanthranilic acid. The acid was diazotised[8] and immediately coupled with 2,4-dimethylphenol in alkaline medium. The product was isolated as its barium salt and converted into sodium salt by treating with a calculated amount of sodium sulphate. The sodium salt was recrystallised from aqueous ethanol. Finally, the dye was purified by making its free acid. The free acid was crystallised repeatedly from 50% ethanol. About 8.0×10^{-4} M solution of the dye was used for the spectrophotometric studies. The pH was adjusted with ammonium hydroxide (2 M) and ammonium chloride (2 M) solution.

Absorbance measurements were carried out on a Beckman DU-2 spectrophotometer with 10 mm quartz cells and pH measurements were made with a Elico LI-10 T pH meter.

Phosphate determination : An aliquot (2.0 ml) of 8.0×10^{-4} M Mg^{II} and 8.0×10^{-4} M CHDMAS (2.0 ml) and less than 50 μg phosphate solution in 10.0 ml was adjusted to pH 11.0 with the help of ammonium hydroxide and ammonium chloride buffer. The absorbance of the solution was measured at 510 nm against the reagent blank. The decrease in absorbance (*i.e.* the absorbance in presence and absence of phosphate) was noted. The quantity of phosphate present was computed from the calibration curve constructed by measuring absorbance using different known amount (0 − 60 μg) of phosphate.

Results and Discussion

The absorption spectra of Mg^{II}-CHDMAS-phosphate system at pH values 8.0 − 11.5, revealed the optimum pH for the work to be 11 0 − 11.5. The study of complex formation as a function of pH showed that the complex formed at pH 11.0 has a constant maximum absorbance. Equimolar amount of CHDMAS was sufficient for maximum absorption which was unchanged for 8 h. Beer's law was followed within the range 4 − 46 μg phosphate per ml. The decrease in molar absorptivity of the Mg^{II}-CHDMAS complex was 7.5×10^2, and the Sandell's sensitivity[9] 0.16 μg phosphate per cm^2. pH is adjusted after the addition of CHDMAS to the Mg^{II} solution to avoid precipitation. A hundred-fold presence of chloride, bromide, iodide, sulphite, sulphate and nitrate had no effect on the estimation of phosphate.

The absorbance obtained from eight determinations of 23 μg phosphate solution was 0.132 ±0.005. Relative deviation and standard deviation were found to be ± 2.3% and ± 1.30%, respectively. The total operation required only 30 min.

References

1. O. P. SHARMA and R. B. KHARAT, *Indian J. Chem.*, 1975, 13, 848.

2. O P. SHARMA and R. B. KHARAT, Ph.D. Thesis, Nagpur University, 1975.
3. O. P. SHARMA and R. B. KHARAT, unpublished work.
4. A. BOKARE, B. K. DESHMUKH and R. B. KHARAT, *Indian J. Chem., Sect. A*, 1976, 14, 541.
5. B. K. DESHMUKH and D. S. MAHESHWARI, *Chem. Anal. (Warsaw).*, 1981, 26.
6. A. I. VOGEL, "A Text Book of Quantitative Inorganic Analysis", ELBS, London, 1975.
7. W. F. HARRIS and T. R. SWEET, *J. Am. Chem. Soc.*, 1955, 77, 2893.
8. K. VENKATARAMAN, "The Chemistry of Synthetic Dyes", Academic, New York, 1952, p. 210.
9. E. B. SANDELL, "Colorimetric Determination of Traces of Metals", Interscience, New York, 1959.

Spectrophotometric Determination of Primary Aromatic Amines using Syringaldehyde

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ALDEHYDES such as *p*-dimethylaminobenzaldehyde[1], *p*-dimethylaminocinnamaldehyde[2], 2-hydroxynaphthaldehyde[3], vanillin[4], glutaconaldehyde[5] and furfural[6] have been utilised earlier for the spectrophotometric determination of primary aromatic amines based on the formation of coloured Schiff's bases. The stability and λ_{max} of such Schiff's bases depend upon the nature and positions of the other substituent groupings on the aromatic ring of the aldehyde moiety. In this paper, we give the results of our investigation on the utilisation of syringaldehyde (3,5-dimethoxy-4-hydroxybenzaldehyde), which has not been used earlier, for the spectrophotometric determination of primary aromatic amines including some sulpha drugs, dapsone and benzocaine.

Experimental

A 2.5% (w/v) solution of syringaldehyde (Fluka) was prepared in alcohol. Standard solutions of some sulpha drugs (sulphathiazole, sulphamethoxy pyridazine, sulphamethizole, sulphaguanidine, sulphadiazine, sulphapyridine and sulphafurazole), dapsone, benzocaine and aniline were prepared by dissolving of each compound (I.P. or B.P. grade) (50 mg) in methanol (500 ml). Tablet powder or suspension equivalent to 50 mg of each drug was extracted with methanol (3 × 50 ml), and the combined methanolic extract was filtered and the volume made upto 500 ml with methanol.

All uv absorbance measurements were made on a Shimadju 140 double beam spectrophotometer.

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TABLE 1—OPTICAL CHARACTERISTICS AND PRECISION

Sl. no.	Primary aromatic amine	Beer's law limits μg/25 ml	Molar absorptivity × 10^3 dm^3 mol^{-1} cm^{-1}	Sandell sensitivity μg cm^{-2}/0.001 Abs. unit	Standard deviation
1.	Aniline	40−350	1.25	0.18	0.014
2.	Benzocaine	25−300	2.58	0.15	0.011
3.	Dapsone	25−200	6.36	0.10	0.007
4.	Sulphathiazole	30−175	3.09	0.21	0.010
5.	Sulphamethoxypyridiazine	90−250	3.07	0.23	0.006
6.	Sulphamethizole	60−350	2.98	0.23	0.004
7.	Sulphaguanidine	40−400	2.67	0.20	0.007
8.	Sulphadiazine	40−350	2.85	0.22	0.006
9.	Sulphapyridine	25−250	2.90	0.21	0.012
10.	Sulphafurazole	40−300	2.70	0.25	0.014

TABLE 2—RESULTS OF ESTIMATION AND RECOVERY EXPERIMENTS OF PRIMARY AROMATIC AMINES IN DOSAGE FORMS

Sl. no.	Tablet/Sample (Labelled amount in mg)	Labelled amount found by the method Proposed	Labelled amount found by the method Official	Recovery %
1.	Sulphadiazine (500)	491	494	100.0
2.	Sulphapyridine (500)	493	496	99.6
3.	Sulphamethizole (100)	98.7	99.3	99.2
4.	Sulphafurazole (400)	395	397	99.8
5.	Sulphamethoxypyridiazine (500)	494	498	100.3
6.	Sulphathiazole (200)	197.1	197.7	100.1
7.	Sulphaguanidine (500)	487	491	99 0
8.	Sulphaguanidine (292.5) Quinodochlor (975) Papain (65) Diastase (12.5 ml) Ext. Ba cl (97.5)	285	290	98.9
9.	Dapsone (50)	49.36	49.8	98.5
10.	Sulphadiazine (1 000)	994	998	100.3
11.	Sulphamethoxypyridiazine (250)	246	248	99.2
12.	Sulphaguanidine (1 000) Tincture carminative (0.25 ml) Light kaolin (3 000) Pectin (130) Saccharin Na (0.6) Syrup (2 ml)	992	998	99.4

Each value represents mean of two readings.

Procedure : Aliquots (1−4 ml) of standard solution were taken into different 10 ml volumetric flasks, syringaldehyde solution (1 ml) and concentrated hydrochloric acid (5 ml) were added to each flask. The volume in each flask was adjusted to 10 ml with methanol. The absorbance was measured at 400 nm against a reagent blank. The sample solutions were also treated in a similar manner and the amount of drug in the sample was deduced from the standard graph.

Results and Discussion

The Beer's law limits, molar absorptivity, Sandell's sensitivity and standard deviation for each compound are presented in Table 1. The results of analysis of pharmaceutical preparations of the sulpha drugs, dapsone and benzocaine (Table 2) by the proposed and reported[7] methods were found to be in good agreement (within 1%). Recovery of added drugs to the formulations by the proposed method was found to be 98.5−100.3%. The usual excipients present in the dosage forms did not interfere in the determination of the specified drugs by the proposed procedure. The method offers a more simple, sensitive and accurate procedure for the estimation of important primary aromatic amines compared to many of the similar methods reported earlier.

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References

1. M. B. AMER, *J. Clin. Pathol.*, 1956, 26, 552.
2. M. QURESHI and I. A. KHAN, *Anal. Chim. Acta*, 1976, 86, 309.
3. F. NIMIGOHR and J. KHORRANI, *Trav. Soc. Pharm. Mont. Pellier.*, 1977, 37, 141.
4. E. N. DEEB, *Drug Standards*, 1958, 26, 179.
5. G. KUNOVITS, *Mikrochim. Acta*, 1974, 4, 717.
6. H. A. EI-OBEID and A. I. JADO, *Anal. Lett.*, 1981, 14, 129.
7. A. C. BRATTON and E. K. MARSHALL, *J. Biol. Chem.*, 1939, 128, 537.