İstanbul Ecz. Fak. Mec.
4, 28 (1968)

J. Fac. Pharm. İstanbul
4, 28 (1968)

Reaction of Sulfadiazine with Sodium 1,2-Naphtoquinone-4-sulfonate

Sülfadiazinin Sodium 1,2-Naftokinon-4-sülfonat Türevi

Hayriye AMAL and Serpil DEMİR *

Sodium 1,2-naphtoquinone-4-sulfonate is used to detect substances containing two removable hydrogen atoms attached to carbon or nitrogen, and paraquinoid condensation products are formed in this reaction. Schmidt(1) determined colorometrically the sulfonamides in blood serum using a solution of sodium 1,2-naphtoquinone-4-sulfonate at pH 4-5. Tulus and Güran(2) have developped a photometric method for determination of sulfamerazine, sulfametazine, sulfadiazine, sulfathiazole, sulfaguanidine, sulfapyridine and sulfacetamide, and isolated the reaction product of sulfacetamide with naphtoquinonesulfonate.

Pilsbury and Jackson(3) used an alkaline solution of sodium 1,2-naphtoquinone-4-sulfonate as spray reagent for detection of a serie of thiazides on chromatogram. The condensation products of sodium 1,2-naphtoquinone-4-sulfonate (NQS) with aniline and some aminoacides were prepared by Erlich at all(4) and Fruman at all(5) respectively.

Sulfadiazine reacts with (NQS) in neutral solution, gives a red coloured reaction product which is separeted after precipitation and prufied by crystallization.

The spectrophotometric determination of sulfadiazine is performed in aqueous solution at 480 mμ and a typical curve is shown in Fig. I. The reproducibility is satisfactory.

EXPERIMENTAL

Preparation of NQS derivative of Sulfadiazine: 1.25 g (0.005 mol) of sulfadiazine was dissolved in 200 to 250 ml of hot water, a hot solution of 1.3 g (0.005 mol) of NQS was added and the mixture was

* Department of Pharmaceutical Chemistry, Faculty of Pharmacy, University of Istanbul.

heated on the steam bath for 10 min. A red coloured substance was precipitated on cooling which was crystallized from water. The yield was 0.23 g. (14.4 %). mp. 197°C.

The thin layer chromatographic examination of the substance was performed on silica gel G plates (activated at 110°C for 20 min.) and chloroform-methanol (80:20) was used as developping system. The substance appears as a red spot on white background. The sulfadiazine and NQS was used as reference spots, sulfadiazine was located with NQS spray reagent, the NQS appears as yellow spot on silica Gel plates.

The UV spectrophotometric examination of the condensation product of sulfadiazine with NQS, was made in a VSU 1 model Zeiss spectrophotometer. $\lambda_{max.}^{EtOH}$ 247 mμ, ε max 612.

Anal. Calcd. for $C_{20}H_{14}N_4O_4S$: C, 58.15; H, 3.99; N, 12.90; S, 7.88. Found; C, 59,06; H, 3.99 N, 13.87; S, 7.88.

The spectrophotometric determination of sulfadiazine was performed in neutral medium. To the solutions containing 90-200 μg sulfadiazine in water, 15 ml of an aqueous solutions of NQS 0.0036 % was added and mixture was heated on a water bath for 10 min, cooled and reheated for 5 min.

Full colour development is attained when the solutions was heated as described abow. The absorbance of each solution (6 solutions) is measured with a VSU 1 model Zeiss spectrophotometer at a wavelength 480 mμ. Fig. I shows the relations of absorbance at the appropriat concentrations.

SUMMARY

A red coloured condensation product of sulfadiazine with sodium 1,2-naphtoquinone-4-sulfonate is prepared by heating the equimolecular ammounts of each componant in aqueous solution.

A spectrophotometric procedure for the determination of sulfadiazine is presented. The method is suitable as a rapid control method in neutrol medium.

ÖZET

Sülfadiazinin sodium 1,2-naftokinon-4-sülfonat ile kondansasyon mahsulü hazırlanmış ve bu reaksiyonda kırmızı renkli bir madde hu-

sule gelmesinden faydalanılarak sülfadiazinin nötr vasatlarda miktar tayini yapılmıştır. Bu metotla 90 μg'a kadar madde tayini mümkün olmuştur.

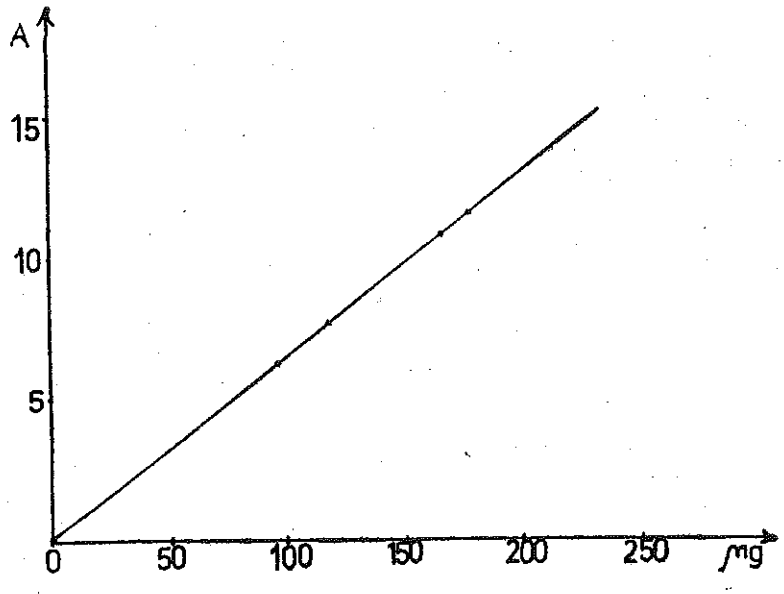

Fig 1

REFERENCES

1. Schimidt, E. G., *J. Biol. Chem.* **122**, 757 (1938).
2. Tulus, R., Güran, A., *J. Fac. Science Univ. İstanbul*, Serie C **28**, 108 (1963).
3. Pilsbury, V. B., Jackson, J. V., *J. Pharm. Pharmacol.*, **18**, 713 (1966).
4. Ehrlich, P., Herter, C. A. and Sylers, H., *Z. f. Physiol. Chem.* **41**, 329 (1904).
5. Fruman, N. H., Morrison, G. H. and Wagner, A. F., *Anal. Chem.* **22**, 1561 (1950).

(Received January 4, 1968)