Chem. Anal. (Warsaw), **38**, 365 (1993)

Differentiating Spectrophotometric Titration of Binary Basic Mixtures Containing Aminophenazone in Non-Aqueous Media

by E. Sell

Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Medical Academy, 80-416 Gdańsk, Poland

Key words: aminophenazone, phenazone, caffeine, phenacetin, mixed solvents, non-aqueous media, spectrophotometric titration

A method of determination of the mixtures of aminophenazone with phenazone, caffeine or phenacetin has been elaborated, based on spectrophotometric titration in non-aqueous media. Titrations were performed with 5×10^{-3} mol l^{-1} $HClO_4$ in dioxan, using solvents differentiating the strength of the estimated bases. The results of the determination of the mixtures of aminophenazone–phenazone in 100 % acetic acid, 100 % acetic acid+acetic anhydride (5+1), benzene+dioxan (1+1) medium, that of aminophenazone–caffeine in benzene+dioxan (1+1) medium and that of aminophenazone–phenacetin in benzene medium can be regarded as satisfactory. The method makes possible to analyse directly the components of the mixtures, without the necessity of separating them.

Opracowano metodę oznaczania mieszanin aminofenazonu z fenazonem, kofeiną lub fenacetyną, polegającą na spektrofotometrycznym miareczkowaniu w środowiskach niewodnych. Miareczkowano przy użyciu 5×10^{-3} mol l^{-1} roztworu $HClO_4$ w dioksanie, stosując rozpuszczalniki różnicujące moc oznaczanych zasad. Pozytywne wyniki oznaczeń uzyskano w przypadku mieszaniny aminofenazonu z fenazonem w środowisku 100 % kwasu octowego, 100 % kwasu octowego z bezwodnikiem octowym (5+1) oraz benzenu z dioksanem (1+1), w przypadku mieszaniny aminofenazonu z kofeiną w środowisku benzenu z dioksanem (1+1), a mieszaniny aminofenazonu z fenacetyną w benzenie. Opracowana metoda pozwala bezpośrednio oznaczyć składniki badanych mieszanin bez konieczności uprzedniego ich rozdziału.

This paper is a continuation of studies on application of differentiating spectrophotometric titration for determination of mixtures containing compounds with the similar acidic or basic properties, respectively [1–3]. The aim of the present work

RESULTS AND DISCUSSION

The differentiating spectrophotometric titrations of the mixtures of aminophenazone with other very weak bases such as phenazone, caffeine and phenacetin were investigated.

In the preliminary studies the titrations of a single standard compound in some solvents at a few wavelengths were carried out to choose the appropriate medium of neutralization and determine proper wavelength. Although a large number of solvents systems investigated proved to give satisfactory results in analysis of individual bases only a few of them were suitable for a direct determination of their mixtures. Considering differentiating properties of the solvents and their influence on shifts of the absorption band for protonated and nonprotonated forms, 100 % acetic acid, 100 % acetic acid with acetic anhydride, benzene and benzene with dioxan were selected for further experiments. In these solvents all the compounds exhibited significant enough shifts of the absorption band to be spectrophotometrically titrated, but especially great shifts were in the case of phenazone.

The titrations of the mixture of aminophenazone and phenazone were performed in 100 % acetic acid, 100 % acetic acid with acetic anhydride (5+1) and in benzene with dioxan (1+1). In these media, except 100 % acetic acid, the spectrophotometric titration curves showed as much as three sharp breaks at the equivalence points, as aminophenazone behaved as a diprotic base. The first break in the curve, therefore, represented the neutralization of amino group of aminophenazone ($pK_B = 9.16$), the second one indicated the equivalence point of phenazone ($pK_B = 12.49$) and the third one of the second nitrogen atom of pyrazoline ring of aminophenazone ($pK_B = 15.00$). It is worth emphasizing that despite their very similar structure, as both compounds are pyrazoline derivatives, it is possible to determine them simultaneously in the mixture.

In the case of aminophenazone–caffeine mixture no satisfactory results were obtained neither in 100 % acetic acid, nor in 100 % acetic acid with acetic anhydride. Benzene with dioxan (1+1) proved to be the only suitable medium for differentiating titration of all the solvent systems examined. In the spectrophotometric titration curve three distinct breaks could be observed. The first and the last one corresponded to the neutralization of aminophenazone, whereas the second one – to the neutralization of caffeine ($pK_B = 13.39$).

The differentiating spectrophotometric titration of aminophenazone–phenacetin mixture was possible only in the benzene medium. The first two breaks indicated the equivalence points of aminophenazone, the third break – neutralization of the weaker base – phenacetin ($pK_B = 16.08$).

From the results presented it can be concluded that the method does not require preliminary separation of individual components of the mixtures by the time-consuming solvent extraction technique or by chromatography prior to determination.

The suggested method is simple and accurate and permits direct simultaneous quantitative determination of both basic components of the mixtures in spite of the fact that they absorb in the same region of the spectrum.

REFERENCES

1. Sell E., *Acta Pol.Pharm.*, **41**, 479 (1984).
2. Sell E., *ibid.*, **42**, 135 (1985).
3. Sell E., *Memoires Pharmaceutiques (France)*, **171**, 61 (1990).

Received June 1992
Accepted January 1993