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## Sulfamic Acid as a Standard Substance in Non-Aqueous Titrimetry

Kwas sulfamowy jako substancja wzorcowa w środowisku niewodnym

Сульфамовая кислота как вещество-эталон для установки титра  
в неводной среде

Sulfamic acid,  $\text{NH}_2\text{SO}_3\text{H}$ , has been already proposed for the standardization of aqueous solutions of alkali metals hydroxides (1, 2, 4, 5, 7). It has an advantage of being stable in the solid state, non-hygroscopic and may be obtained in a pure form. Since the substance is soluble in some organic solvents (e.g. dimethylformamide), it was also employed to determine the titer of lithium methylate in a non-aqueous medium (3). Now it is conceivable to be tested as an acidic primary standard for a more widely used non-aqueous titrants — sodium methylate and sodium aminoethylate. The potentiometric investigation showed that the potential breaks are sharp and occur at stoichiometric ratios; an indicator method can also be used for the end point detection.

### EXPERIMENTAL

#### Reagents and apparatus

Sulfamic acid (POCH Gliwice) was twice recrystallized from water at  $70^\circ$  (discarding the first crystals separated at  $25^\circ\text{C}$ , and collecting the next, precipitated at refrigeration) and dried in a vacuum for at least 12 hrs; this simple procedure was almost as effective as Bishop's laborious method (1). Acidimetric assay gave 99,8% purity.

Sodium methylate (in benzene-methanol, 85:15), sodium aminoethylate (in ethylenediamine) — 0,1 N solutions. Dimethylformamide (BDH, reagent grade). Bromocresol green (BDH, reagent grade) — 0,05% solution in anhydrous methanol. Other solvents and reagents (POCH Gliwice, p.a.).

Potentiometer Titrator TTT 1 with scale expander PHA 630 T (Radiometer, Denmark); glass electrode G 202 C and silver chloride electrode (electrolytically coated silver electrode P 4011). Derivatograph type OD 102 (MOM, Hungary).

#### Derivatographic examination of sulfamic acid

The sample at a  $2^\circ\text{C}/\text{min}$  heating rate did not considerably change its mass from  $20^\circ\text{C}$  up to  $230^\circ\text{C}$ . DTA-curve showed in this range only one distinct peak between  $205\text{--}207^\circ\text{C}$ , owing to the melting of the substance. However, the specified examination is not consistent with the slow (and prolonged) heating of sulfamic acid, when it decomposes at a lower temperature (about  $140^\circ\text{C}$ ) (7).

### Potentiometric titrations.

A series of experiments were carried out, in which weighed amounts (20,0—60,0 mg) of sulfamic acid were dissolved in 30 ml of dimethylformamide (DMF) and titrated potentiometrically at 25°C with sodium methylate or sodium methylate or sodium aminoethylate solutions, using a glass indicator electrode. Exemplifying titration curves are shown on Fig. 1

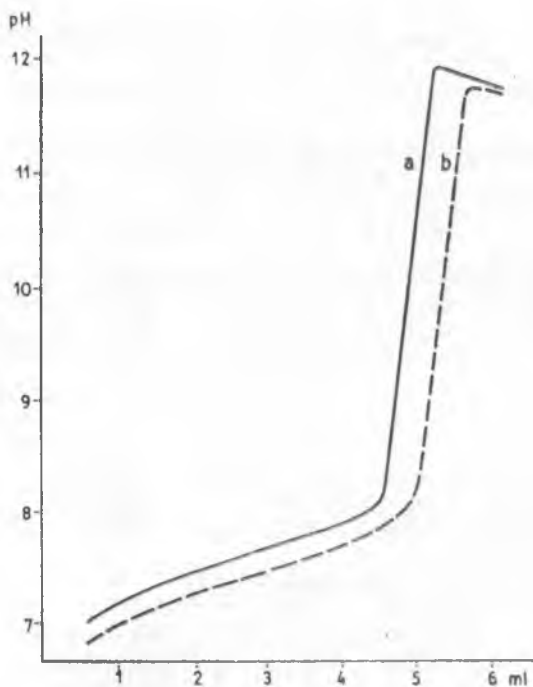


Fig. 1. Potentiometric titration of sulfamic acid with sodium methylate (curve "a"; w.: 0,483 g) and with sodium aminoethylate (curve "b"; w.: 0,0527 g)

In spite of the low acidic strength of sulfamic acid in DMF (6), the procedure ensures sharp potential breaks and stable potentials.

#### Titration with visual end-point detection

The samples (100—200 mg) of pure sulfamic acid, dissolved in DMF, were titrated with the titrants to be standardised. Several indicators (e.g. thymol blue, azo violet, thymolphthalein) were used in conjunction with pH titrations and bromocresol green was found to be most suitable, since the color change (yellow to blue) is sharp and it occurs almost exactly at the experimentally determined equivalence point. The evaluation of the experiments is given in Table 1.

Table 1. A statistical evaluation of the standardization of sodium methylate (I) and sodium aminoethylate (II) solutions with sulfamic acid  
 Statystyczna ocena oznaczania miana roztworów metanolanu sodowego (I) i aminoetylanu sodowego (II) za pomocą kwasu sulfanowego

Procedure	Number of variates (n)	Mean, % ( $\bar{x}$ )	Standard deviation, (%) (s)
I. Potentiometric			
end-point Visual	11	99,96	$\pm 0,052$
end-point	7	99,91	$\pm 0,086$
II. Potentiometric			
end-point Visual	7	99,91	$\pm 0,099$
end-point	7	99,84	$\pm 0,105$

### DISCUSSION

The presented experimental data show that sulfamic acid can serve as a satisfactory primary standard in acid-base non-aqueous titrimetry. The suitable medium is dimethylformamide. A given procedure enables a sufficiently precise determination of the titer of sodium methylate or sodium aminoethylate solutions with potentiometric or visual (recommended indicator is — contrary to Caso and Cefola (3) — bromocresol green) end-point titration.

### REFERENCES

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- Otrzymano 20 VI 1974.

### STRESZCZENIE

Kwas sulfamowy (kwas amidosulfonowy) może służyć jako substancja wzorcowa do oznaczania miana roztworów metanolanu sodowego lub aminoetanolanu sodowego. Odważkę kwasu sulfamowego rozpuszcza się w dwumetyloformamidzie i mierzkuje oznaczanym płynem potencjometrycznie (elektrody szklana i chlorosrebrowa) lub ze wskaźnikiem wizualnym (zieleń bromokrezolowa).

### РЕЗЮМЕ

Сульфамовую кислоту (амидосульфоновую кислоту) можна применять как вещество-эталон для установки титра метанолат натрия или аминокетанолат натрия. Навеску сульфамовой кислоты растворяется в диметилформамиде и потенциметрически титруется определяемым раствором (стеклянный электрод и хлоросеребряный электрод) или с визуальным индикатором (бромкрезоловый зеленый).

