



## Azo-Substitute of Ethoxy Acridine – A New Reagent for Extraction-Photometric Determination of Tellurium (IV)

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### Abstract

We studied associates of halogen telluride (Cl, Br) with 9-amino-4-ethoxyacridine-6-azo-N,N'-diethylaniline (AEADEA) by spectrophotometric method. It was established that associates are extracted well with chloroform-acetone (3:2). We determined the compositions, physical and chemical and analytical properties of complexes ( $\epsilon_k$ ,  $\beta_{KD}$ , D, R%). Maximum light absorption for chloride complexes is found to be  $\lambda_{max}$  520 nm, but for bromide complexes -  $\lambda_{max}$  530 nm. For establishing the composition of azo-substitute of ethoxyacridine the synthesized reagents were exposed to elemental analysis, as well titanometric titration by azo group. We developed new extraction-photometric determination methods of tellurium. Lambert-Beer law is observed in the range 0.5-15 mkg Te at 5 ml medium.

### Indexing terms/Keywords

9-amino-4-ethoxyacridine-6-azo-diethylaniline; extraction-photometric determination of tellurium; halogen tellurate.

### Academic Discipline And Sub-Disciplines

Chemistry

### SUBJECT CLASSIFICATION

Analytical chemistry; Inorganic chemistry

### TYPE (METHOD/APPROACH)

Spectrophotometry, extraction-photometric determination

### INTRODUCTION

Only sulfur containing chelating reagents are used for photometric determination of tellurium which is characterized with a high sulfur affinity. As a rule, the elements which have a high sulfur affinity and are often accompanied by tellurium impede the determination: Ag, As, Au, Cu, Hg, Se, Tl and platinum metals. That's why the preliminary separation of tellurium is often necessary, for which the use of diethyldithiocarbamate is relevant [1-2].

Methods of extraction-photometric determination of tellurium with basic dyestuffs are well known [3-4], among which butylrhodamine C is often used. Butylrhodamine C is low-selective, Fe (III), Cu (I), Pb (II), Sn (II, IV), Tl (III), Sb (V), In (III), Au (III), Ag (I) and  $Ga^{3+}$  interfere with the determination of tellurium. Results of determination are unrepeatable which force to apply severe conditions of analysis.

When determining the metals with no chromophoric properties as ion-associates, their metal halogenide complexes with cations of basic dyes, sensitivity of determination depends on intensity of reagent staining. Ethoxyacridine does not have a large connectivity system and, consequently blue color. The detection limit of elements by this dyestuff is high [5-6]. This defect can be eliminated by increasing conjugated chain due to formation of azo-substitute of ethoxyacridine.

In the present work we present the experimental results of associates of chloro-telluride and bromium telluride ( $HTeCl_6$ ,  $HTeBr_6$ ), with azoethoxyacridine-9-amino-4-ethoxyacridine-6-azo-N,N'-diethylaniline (AEADEA), and possibility of using them in photometric analysis was found out.

### EXPERIMENTAL

#### Reagents and instruments

$9.71 \cdot 10^{-3}$  M solution of tellurium (IV) was prepared from metal tellurium with high-purity (99.999%) by the method [7],  $9.71 \cdot 10^{-3}$  M (22 mkg) solution of tellurium (IV) was obtained by diluting the initial solution. Solution AEADEA (0.05%) was prepared by dissolving double-crystallized preparation in ethanol. Acidity of medium was created with solution of sulphuric acid (9.00 M), 1.00 M solution LiCl and 1.00 M KBr were used.

Spectrophotometric researches were conducted on spectrophotometer UB-1800 visible. While developing analytical formulas light absorption of painted extracts was measured on photoelectrocalorimeter KFK-2. pH value of solutions was determined by ionomer EB-74.

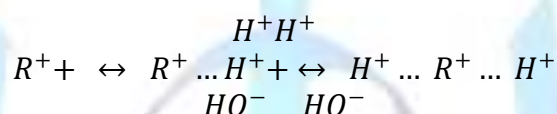
## Technique

Various quantities of tellurium (IV) were placed into test-tubes with friction lid, necessary acidity of sulphuric acid was created, 0.5 ml solution LiCl (KBr), 0.5 ml solution AEADEA were added, and the volume of mixture was brought to 5 ml with water. The mixture was slightly shaken to mix the components; 5 ml extractant (chloroform-acetone 3:2) was added into it. Then, we separated organic phase from water phase, measured light absorption of extracts relative to reference solution.

## RESULTS AND DISCUSSION

Application of sulphuric acid allows us to vary acidity of medium in wider interval. It was established that in the interval 1-10 N acidity of medium basic azo-dye exists in orange color in the form of unprotonated single charged cation  $R^+$  with  $\lambda_{max}$  460 nm. In the interval 10-16 N reagent is in monoprotinated form: a new absorption band with two maximums appear from 16 to 33 N on curves of light absorption at  $\lambda_{max}$  380 nm and  $\lambda_{max}$  580-590 nm, which is related to the addition to dye molecule of the second proton  $H^+ \dots R^+ \dots H^+$ . This form was painted violet.

Thus, protonization of azo substitutes of ethoxyacridine can be represented with a scheme:



Condition of AEADEA in the interval 1-33 N by sulphuric acid was studied by spectrophotometric method (fig. 1).

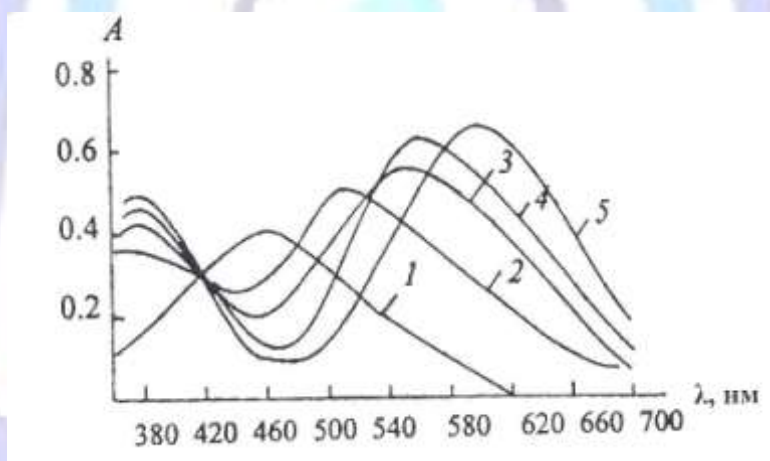


Fig. 1: Absorption band of AEADEA at different acidities of solution: 1-1N, 2-9 N, 3-15 N, 4-29 N, 5-33 N by sulphuric acid (SF-46,  $l=1$  cm,  $[C_{AEADEA}] = 4.18 \cdot 10^{-4}$  M).

Ethoxyacridine dyes were synthesized in the laboratory of analytical chemistry of the Institute of Catalysis and Inorganic Chemistry named after academician M.Nagiyev of NAS of Azerbaijan. Individuality of reagents was established by elemental analysis, titanometric titration by azo-group.

## Elemental analysis of ethoxyacridine dye.

To establish the composition of azo-substitutes of ethoxyacridine synthesized reagents were exposed to elemental analysis. First, the preparation was dried 2-3 hrs at  $50^{\circ}C$  in vacuum oven to eliminate humidity. Results of analysis are given in the table 1.

Table 1. Elemental analysis of azo-derivative of ethoxyacridine

Compound	Total content of components, %							
	Theoretically calculated				Practically found			
	C	N	H	Cl	C	N	H	Cl
AEADE	66.89	15.60	6.02	7.92	66.10	14.47	6.47	8.04

The formation and extraction of associates of chloride and bromide acido-complexes Te(IV) with AEADE were studied. Peaks of light absorption of chloride and bromide associates of tellurium (IV) with AEADE are observed in the interval 520-530 nm (fig. 2.)

Light absorption of extracts of associates is the same as the peak of ethoxyacridine which indicates electrostatic character of interaction and formation of complexes. During formation and extraction of complexes optimum acidity is in the interval 2.0-4.0 N by H<sub>2</sub>SO<sub>4</sub>. Under these conditions reagents are insignificantly extracted.

Organic solvents of various types: hydrocarbons, alcohols, simple and complex ethers, ketons, halogen derivatives of hydrocarbons were tested as extractants. It turned out that hydrocarbons (hexane, xylol, benzene) do not extract associates; simple salts of dyes, alcohols and halogen derivatives of hydrocarbons insignificantly extract associates, but ketons extract both associates and dyes. That's why we studied organic solvents as extractants which were taken in different combinations; the best extractant is found to be the mixture of chloroform-acetone (3:2).

Optimum volume of water and organic phase equals to 5 ml. Molar ratio of components in extractable compounds was studied by methods of isomolar series, shift of equilibrium and straight line [8]. It was found that halogenide associates of tellurium (IV) are associated with AEADE in the ratio 1:1.

Based on the established ratio of components in extractable compounds we calculated the extraction constants by limiting logarithmic method of Asmus [8].

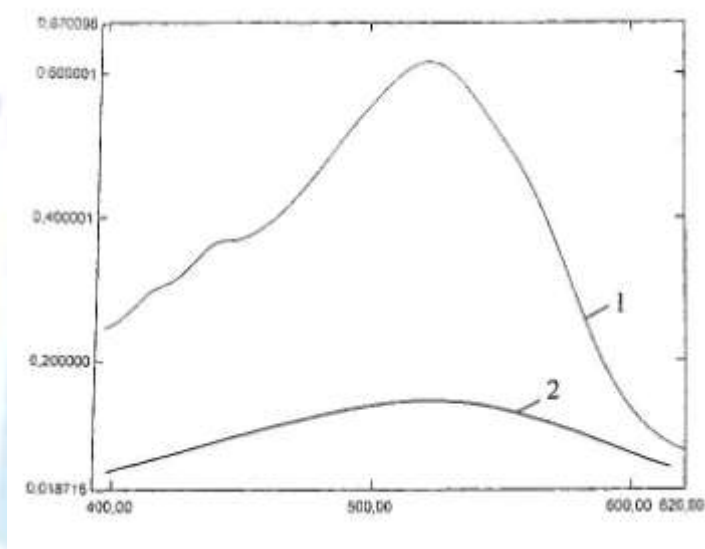


Fig. 2: Absorption spectrum of associate extract of chlorotelluride with AEADE – 1 [C<sub>Te</sub>]=4.85·10<sup>-3</sup> M and reagent – 2 (SF-UB-1800) [C<sub>AEADE</sub> = 4.18·10<sup>-4</sup> M]

True values of molar coefficients of absorption and biphasic stability constants of associates were found by Komar, Sommer and saturation methods (Table 2). We determined distribution coefficient and extraction degree of tellurium (Table 2).

Table 2. Physical and chemical characteristics of halogentellurites with AEADE

Associate	Optimum acidity of medium, N	$\lambda_{max}$ , NM	$\epsilon \cdot 10^{-4}$	$\beta_{KD} \cdot 10^5$	D	R, %	Ratio Te:hal.:R <sup>+</sup>
[HTeCl <sub>6</sub> ] · AEADE	3.0	520	6.20	4.70	105.80	98.64	1:6:1
[HTeBr <sub>6</sub> ] · AEADE	4.0	530	6.80	5.90	125.25	98.80	1:6:1

### The execution of definition

0.5-15.0 mkg of tellurium (IV) was placed into test-tubes with friction lid, and 0.4 ml 1.0 M KBr, 1.6 ml 9 M H<sub>2</sub>SO<sub>4</sub> were added. 0.5 ml of 0.1 alcohol solvent AEADEA was added into the mixture, the volume of mixture was brought to 5 ml with distilled water and light absorption of extracts were measured in the cell with 1=0.5 cm at 490 nm wavelength on KFK-2. Lambert-Beer law is observed in the range 0.5-15 mkg Te at 5 ml medium.

The followings do not interfere with the determination of tellurium: large amount of alkali and alkaline-earth metals Co(II), Co(III), Ni(II), Al<sup>3+</sup>, Cu(I), Cu(II), Tl(I), Pb(II), Sn(II), Fe(II), Ta(V), Nb(V), as well as anions PO<sub>4</sub><sup>3-</sup>, NO<sub>3</sub><sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, C<sub>2</sub>O<sub>4</sub><sup>2-</sup>, tartrate-



ion, citrate-ion, EDTA. Au(III), Fe(III), Se(IV), Tl(III), Hg(II), In(III), as well as thiocarbamide interfere with the determination of tellurium.

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## Author' biography with Photo



Dr. Namig Ismayil Ismayilov works in the laboratory "Analytical Chemistry" in M.Nagiyev Institute of Catalysis and Inorganic Chemistry of Azerbaijan National Academy of Sciences. His scientific direction: development of new extraction-photometric determination methods of Ga, Te, In, Au, Hg and others. He is the author of more than 45 scientific works, including one patent.