

*N. Βέη*: Βραχὺ Ἑπειρωτικὸν χρονικὸν ἐκ Βερατίου.

ΑΝΑΚΟΙΝΩΣΕΙΣ ΜΗ ΜΕΛΩΝ

ΑΝΑΛΥΤΙΚΗ ΧΗΜΕΙΑ. — **Ascorbic acid (Vitamin C) as an analytical reagent. II Detection of selenium\***, *par E. C. Stathis*. Ἀνεκοινώθη ὑπὸ τοῦ κ. Κ. Ζέγγελη.

It has been shown in a previous paper that the ascorbic acid, commonly called vitamin C, can be used as reagent for the detection of gold (I).

The adopted method is based on the reducing activity of ascorbic acid.

It is evident from the formula of the ascorbic acid that its peculiar reducing activity is due to the characteristic endiol group.

Since the appearance of the first paper, further studies have been carried out on the use of ascorbic acid as reagent.

The purpose of this paper is to describe a methode for the detection of selenium which is based on the reduction of the selenite to metallic selenium, by means of ascorbic acid.

EXPERIMENTAL

A selenium solution containing strong hydrochloric acid in the cold gives with an aqueous solution of ascorbic acid the amorphous red variety which, on warming, goes to the gray crystalline form. A selenate or selenic acid must first be reduced to a selenious acid.

For the detailed investigation of the reaction, solutions of selenious acid of various concentrations were prepared.

Metallic selenium, free from any foreign matter, is placed in a beaker, and nitric acid sp. gr. 1,2 - 1,3 is added. After the sample has dissolved in the beaker, it is evaporated to dryness on the water bath and taken up in 100 ml. concentrated hydrochloric acid.

The following solutions of selenious acid were prepared to determine the sensitivity of the reaction.

(A) solution. 0.00666 gr. of selenium in 100 ml. of HCl sp. gr. 1,19.

(B) solution. 10 ml. of (A) solution are diluted with HCl to 100 ml.

An aqueous solution 0,2 % of ascorbic acid freshly prepared makes a satisfactory reagent solution.

\* ΕΛΕΥΘ. ΣΤΑΘΗ: Τὸ ἀσκορβινικὸν ὀξύ (βιταμίνη C) ὡς ἀντιδραστήριον εἰς τὴν ἀναλυτικὴν Χημείαν. II Ἀνίχνευσις σεληνίου.

The tests were carried out in the usual test tubes.

To 3 ml. of (A) solution 1 ml. of water and 1 ml. of the reagent are added. A red precipitate of metallic selenium is formed.

If the amount of selenium is less than 0.0006 gr. in 5 ml. a turbid red solution is produced and with amounts of selenium less than 0.000001 gr. a yellow red solution is obtained.

The following results, obtained by the above method, indicate the sensibility of the reaction.

Milliliters of selenious acid sol.	Milliliters of HCl 1.19	Milliliters of Water	Milliliters of the reagent	Color of the reaction	Selenium in grams
3 (A)	—	1	1	Red precipitate	0.0002
1.5 (A)	1.5	1	1	» »	0.0001
0.75 (A)	2.25	1	1	Turbid red solution	0.00005
3 (B)	—	1	1	Red color	0.00002
1.5 (B)	1.5	1	1	Pink color	0.00001
0.75 (B)	2.25	1	1	Yellow-red color	0.000005

The procedure was further tested for the detection of selenium in the presence of other elements, usually occurring in seleniferous materials.

The reaction has been found to be unaffected by the presence of tellurium, copper, bismuth and arsenic in strong hydrochloric acid solutions.

Silver ions are precipitated in hydrochloric acid solution as chlorides and can be separated.

Mercury is reduced by the reagent and should be removed.

Gold is precipitated at the same time but can be separated by treating the well-washed precipitate with nitric acid, sp. gr. 1.25, which will dissolve the selenium but not the gold.

The reaction was also applied for the detection of selenium in ores.

The following procedure was adopted in the analysis of an ore of which the principal constituents were Berzelianit ( $\text{Cu}_3\text{Se}$ ) and Calcite ( $\text{CaCO}_3$ ).

A gram portion of the sample is placed in a beaker and 5 ml. of water are added, followed by 20 ml. concentrated nitric acid.

After the sample has dissolved in the beaker, it is evaporated to dryness on the water bath and the residue taken up in 10 ml. concentrated nitric acid and 30 ml. of water. The insoluble matter is filtered off and the

solution received in 250 ml. volumetric flask. 25 ml. of the filtrate are evaporated to dryness on the water bath and the residue is dissolved in concentrated hydrochloric acid. After the nitric acid is expelled, the solution is transferred in 100 ml. volumetric flask and diluted with concentrated hydrochloric acid to the mark.

For the detection of selenium to 1 ml. of the above solution 2 ml. of concentrated hydrochloric acid and 2 ml. of the reagent were added. After 15" a red turbid solution was produced.

#### CONCLUSION

A method has been described for the detection of selenium and its sensibility has been shown.

The method has been found uneffected by the presence of tellurium and other elements contrary to the old methods.

Its application to ores, that have been explored, indicates a satisfactory procedure.

#### Π Ε Ρ Ι Λ Η Ψ Ι Σ

Εἰς προηγουμένην ἡμῶν ἀνακοίνωσιν ἐν τῇ Ἀκαδημίᾳ Ἀθηνῶν ἐδείξαμεν ὅτι τὸ ἀσκορβινικὸν ὀξύδὺν δύναται νὰ χρησιμοποιηθῆ ὡς ἀντιδραστήριον διὰ τὴν ἀνίχνευσιν τοῦ χρυσοῦ.

Συνεχίζοντες τὰς ἐρεῦνας ἡμῶν ἐπὶ τῆς ἐφαρμογῆς τοῦ ἀσκορβινικοῦ ὀξέος ὡς ἀναλυτικοῦ ἀντιδραστηρίου περιγράφομεν ἐν τῇ παρούσῃ μελέτῃ νέαν μέθοδον ἀνιχνεύσεως τοῦ σεληνίου, ἣτις στηρίζεται ἐπὶ τῆς ἀναγωγῆς τοῦ σεληνιώδους ὀξέος εἰς ἐρυθρὸν μεταλλικὸν σελήνιον.

Ἐκ τῶν ἀναγραφόμενων ἀποτελεσμάτων εἰς τὸν παρατιθέμενον πίνακα προκύπτει ὅτι ἡ μέθοδος αὕτη ἀνιχνεύσεως σεληνίου εἶναι λίαν εὐαίσθητος.

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*N. M. Μαγκάκη*: Παρατηρήσεις τινὲς ἐπὶ τῆς παθολογικῆς ἀνατομικῆς τοῦ ἔξανθηματικοῦ τύφου.