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CONTRIBUTION TO THE STUDY OF AN INORGANIC SEMI-QUANTITATIVE METHOD OF ANALYSIS: ANALYSIS OF CHROMIUM AND NICKEL

by

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I. GENERAL CONSIDERATIONS

In a previous communication* we have given a preliminary description of a semi-quantitative method of inorganic analysis, and have applied this procedure to the determination of copper. We will here briefly recapitulate the principle of the method. The systematic and thorough study of new analytical reagents, such as have been published in the Second Report of the International Committee on New Analytical Reactions and Reagents of the International Union of Chemistry, has lead us to the view that it should be possible to establish, in a certain number of cases at least, rapid semi-quantitative methods of determination, by making use, for one and the same element, of several of the recommended reagents. In order to have at our disposal an even wider field, we have also employed the reagents listed in the First Report of the above-mentioned Commitee.

By the dilution of a known solution of the desired element, one determines the limit of sensitivity of the reagents, the choice of which has been guided by the fact that they must present a sufficiently selective character. One can thus, by calculation from the values obtained, established tables which give directly the percentage of the limit being determined.

Since this work, we have pursued our researches in the semi-quantitative field and have applied this procedure to the determination of chromium, nickel, bismuth, molybdenum and manganese. The results obtained are, in the majority of cases, very satisfactory, provided always that notice is taken of a certain number of remarks made in the course of the present report.

The determination of the limit of sensitivity of the reagents must be made with the greatest care, since it serves for the establishment of calibration tables and hence determines the accuracy of all subsequent analyses. The values of the limiting sensitivities given in the literature, although in general reliable, cannot be considered as absolute. They vary from one publication to another, which is easily explained since a number of factors are able to modify the value of this limit.

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* P. WENGER, D. MONNIER and Y. RUSCONI, *Helv.*, 219 (1946) 1698.

Thus, for example, the volume of a drop is often taken as being 0.03 ml; it is clearly an approximate value which varies appreciably from one pipette to another and, for a given pipette, with the inclination which is given to it. On the other hand when working on drop plates the dimension and the colour of these play a not negligible rôle in the evaluation of the colour. If the determination is carried out on filter paper values are obtained which vary with the nature of the filter and the dimension of its pores. It is also necessary to take into account the degree of clarity, the angle at which the colour and the precipitate are observed, the weather and the personal coefficient of the observer. All these causes of variation can easily be eliminated by the same person carrying out the determination of the limit of sensitivity and the analyses of the unknown, with the same apparatus and under the same conditions. In doing this, the values given in the literature can be used as a first approximation. In the same way, it is preferable for each analyst to establish for himself the calibration tables, which we give below for chromium and nickel as a working guide.

The choice of these reagents (3 to 4 if possible) requires minute study and must fulfil several requirements. This choice permits, above all, avoiding the action of interfering ions. For this reason it is desirable that at least two of the reactions do not suffer interference from the same foreign ion.

It is also necessary that the limit of sensitivity be easy to determine, which is not always the case, particularly when the reagent itself possesses a colour. Finally, it is necessary to choose reactions which are readily employed, require the minimum of manipulation and are reasonably stable in solution.

This method of semi-quantitative analysis presents a number of advantages:

1. It is rapid; when the reagents have been prepared and the calibration tables prepared, the analysis can be carried out in a quarter of an hour (excluding the preliminary solution of the sample).

2. The employment of several reagents permit a check of the results obtained, and a determination of the order of the magnitude of the error.

3. A preliminary qualitative analysis is not necessary, since if there are any interfering ions the employment of such reagents will give different results. The analysis may be considered acceptable if two of the reagents give concordant results.

4. The apparatus is extremely simple and inexpensive; it comprises some pipettes, drop plates, filter paper and graduated flasks.

As with all methods, this type of determination presents certain difficulties. Thus, it is very difficult to find reagents sufficiently selective for the analysis to be possible in the presence of any foreign ion whatever. Under these circumstances a preliminary separation is necessary. Moreover, when the percentage of the element being determined is high, the results are very approximate, the error being 10 to 15 % and even more. By contrast, for quantities less than 1 %, the determination is very often quantitative.

II. SEMI-QUANTITATIVE ANALYSIS OF CHROMIUM

The chromium must be in solution in the form of chromate anions, CrO_4^{-2}.

Reagents:

1. Gum guaiacum* (1 % in ethyl alcohol) gives a blue precipitate.
2. Strychnine (1 % in 35N sulphuric acid)gives a rose colour.
3. Diphenylcarbazide (0.25 % in a mixture 1/1 acetone-water) gives a violet colour.

All the reactions are carried out on drop plates. The reagents are stable for several days.

Establishment of a reference system

Starting from a standard solution of potassium dichromate containing 0.1 g/l of chromium, Cr, we prepare two solutions; one containing 0.000125 g/l of chromium (solution E), the other 0.00125 g/l of chromium (solution D).

For the determination of the limit of detection of diphenylcarbazide, we use solution E in the following manner: in cup No. 1 of the drop plate are placed one drop of solution E and 9 drops of water; this new solution is described as E_1. In cup No. 2 of the drop plate are placed two drops of solution E and 8 drops of water; this new solution is described as E_2. In cup No. 3 of the drop plate are placed three drops of solution E and 7 drops of water; this new solution is described as E , etc. In cup No. 10 of the drop plate are placed 10 drops of solution E and no water; this new solution is described as E_{10}.

To each cup are added one drop of dilute sulphuric acid and one drop of diphenylcarbazide. The violet colour is not visible in the 4 first cups but appears in cup No. 5. The limit of detection of this reagent is, therefore, given by solution E_5.

The limit of detection of the gum guaiacum is determined in an identical manner, by using solution D. (A more concentrated solution is employed because the sensitivity of this reagent is less than that of the preceding one.) The dilution is effected in the cups (D_1, D_2 . . . D_{10}) and to each is added one drop of concentrated sulphuric acid and one drop of the solution of the resin. A blue precipitate is observed in cup No. 5 (solution D_5).

With regard to strychnine, account must be taken of the fact the reaction only takes place in concentrated sulphuric acid medium. In order to avoid dilution one drop is taken of solution D_1, D_2 . . . D_{10}, and the test is carried out with one drop of the solution of strychnine on a second drop plate. The limit is obtained with solution D_5.

Note. For the determination of these limits, it is necessary that the last ones be obtained with the solutions 5, 6, 7, 8, 9 or 10 since, as can be seen by consulting the tables and Figure 1, the values obtained for the dilutions 1, 2, 3 are too approximate.

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* In general, we refer the reader to the two reports of the Commission on New Analytical Reagents . . . *loc. cit.*, for the formula and constitution of the reagents and for their method of preparation.

The above results permit the establishment, by a simple calculation, of a system of references. This is done according to the following procedure: the starting solution contains 0.1 g/l of chromium. By dilution there are obtained on the one hand the solutions E, then $E_1, E_2 \ldots E_{10}$, on the other hand the solution D, then $D_1, D_2 \ldots D_{10}$.

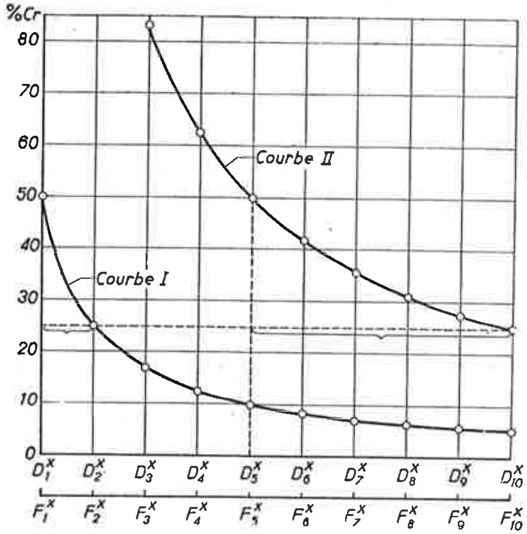

Fig. 1. Curves enabling the percentage of chromium to be determined from the limit of detection. Curve I corresponds to Table D^x, Curve II corresponds to Table F^x.

Let us now suppose that we start with a solution containing 0.1 g/l of a sample whose chromium content is unknown. We can prepare solutions corresponding to those used in the case of pure chromium. These are called $E_1^x, E_2^x, \ldots E_{10}^x, D_1^x \ldots$ etc. If, in determining the limit of detection with diphenylcarbazide we find this, not for E_5, but, for example, for E_{10}^x, we deduce from this that this sample contains 50 % of chromium, since the solution limit is only half as dilute as that containing 100 % chromium. It is possible to calculate by analogous reasoning to what percentage of chromium dilutions $E_1^x \ldots E_{10}^x$ and $D_1^x \ldots D_{10}^x$ etc. correspond.

TABLE E^x

0.000125 g/l of sample

	E_1^x	E_2^x	E_3^x	E_4^x	E_5^x	E_6^x	E_7^x	E_8^x	E_9^x	E_{10}^x
Diphenyl-carbazide	—	—	—	—	100	83.3	71.4	62.5	55.6	50
Gum guaiacum	—	—	—	—	—	—	—	—	—	—
Strychnine	—	—	—	—	—	—	—	—	—	—

TABLE D^x

0.00125 g/l of sample

	D_1^x	D_2^x	D_3^x	D_4^x	D_5^x	D_6^x	D_7^x	D_8^x	D_9^x	D_{10}^x
Diphenyl carbazide	50	25	16.7	12.5	10	8.3	7.1	6.3	5.6	5
Gum guaiacum	—	—	—	—	100	83.3	71.4	62.5	55.6	50
Strychnine	—	—	—	—	100	83.3	71.4	62.5	55.6	50

The chromium content in the sample is directly given by the solution which corresponds to the limit of detection. The above tables must be extended since they do not give all the percentages of chromium. It is easy to establish, by calculation, by starting with other dilutions, tables for any content of chromium (equal to or greater than the limit of detection). For solutions containing respectively 0.01, 0.1 and 1 gramme per litre of sample (solutions C^x, B^x and A^x) we have:

TABLE C^x

0.01 g/l of sample

	C_1^x	C_2^x	C_3^x	C_4^x	C_5^x	C_6^x	C_7^x	C_8^x	C_9^x	C_{10}^x
Diphenylcarbazide	6.3	3.1	2.1	1.6	1.3	1.0	0.89	0.78	0.69	0.63
Gum guaiacum	62.5	31.3	20.8	15.6	12.5	10.4	8.9	7.8	6.9	6.3
Strychnine	62.5	31.3	20.8	15.6	12.5	10.4	8.9	7.8	6.9	6.3

TABLE B^x

0.1 g/l of sample

	B_1^x	B_2^x	B_3^x	B_4^x	B_5^x	B_6^x	B_7^x	B_8^x	B_9^x	B_{10}^x
Diphenylcarbazide	0.63	0.31	0.21	0.16	0.13	0.10	0.089	0.078	0.069	0.063
Gum guaiacum	6.3	3.1	2.1	1.6	1.3	1.0	0.89	0.78	0.69	0.63
Strychnine	6.3	3.1	2.1	1.6	1.3	1.0	0.89	0.78	0.69	0.63

TABLE A^x

1 g/l of sample

	A_1^x	A_2^x	A_3^x	A_4^x	A_5^x	A_6^x	A_7^x	A_8^x	A_9^x	A_{10}^x
Diphenylcarbazide	0.063	0.031	0.021	0.016	0.013	0.010	0.009	0.008	0.007	0.006
Gum guaiacum	0.63	0.31	0.21	0.16	0.13	0.10	0.089	0.078	0.069	0.063
Strychnine	0.63	0.31	0.21	0.16	0.13	0.10	0.089	0.078	0.069	0.063

If we have to determine by means of diphenylcarbazide a substance of which the chromium content is between 25 and 50 % (Table D^x) the results obtained are only approximate; but even here, it is possible to improve matters by using dilutions such that the limit of detection is arrived at not only in the first cups, but in the cups 5, 6, 7, 8, 9 or 10 (see Figure 1).

In the present case, we choose a solution containing 0.000250 g/l of sample (solution F^x).

TABLE F^x

	F^x_1	F^x_2	F^x_3	F^x_4	F^x_5	F^x_6	F^x_7	F^x_8	F^x_9	F^x_{10}
Diphenyl-carbazide	—	—	83.3	62.5	50	41.7	35.7	31.3	27.8	25

The precision of the results can also be increased by working with a total of 20 drops instead of 10, provided, however, that the determination of the limit is still possible (Figure 1).

In spite of the good selectivity of the reagents certain foreign ions render the determination impossible without a preliminary separation: such as, for example, copper, arsenic, antimony, tin, and vanadium. The analysis can be carried out in the presence of iron if it is there in not too great a quantity. In sulphuric acid solution it does not interfere with the diphenylcarbazide test and it can be masked by sodium fluoride in the gum guaiacum test. The strychnine test cannot be employed since the presence of iron diminishes the sensitivity of the reaction.

Analysis of chromium alloy: Composition of the alloy: Cr 0.2 %, C 0.3 %, Si 0.4 %, Mn 1.1 %, Fe: remainder.

Procedure, this comprises 4 operations:

1. Weighing;
2. Dissolving and oxidation to chromate;
3. Determination of the limit of detection by successive dilutions;
4. Determination of the chromium content of the sample, by means of the tables and consideration of the various results obtained.

0.1 g of the sample is weighed with a precision of about 1 %, dissolved in as little aqua regia as possible, evaporated to dryness and the residue dissolved in dilute nitric acid. The solution is neutralised with sodium hydroxide, solid sodium peroxide is added and the solution boiled until the excess of peroxide has been completely decomposed. The liquid is filtered and the filtrate collected in a calibrated flask and diluted to 1.000 ml by the addition of distilled water. This is the type solution, from which solutions A^x and B^x are prepared.

With gum guaiacum (solution A^x) the precipitate appears in cup 4 (A^x_4) and is not visible in cup 3 (A^x_3). From table A^x it is deduced that the chromium content of the alloy is between 0.16 and 0.21 %.

With diphenylcarbazide (solution B^x) no colour is produced by solution B^x_2, but appears in cup 3 (B^x_3) which corresponds (table B^x) to a chromium content between 0.21 % and 0.31 %.

The test is impossible with strychnine, since traces of Fe remain in solution.

The chromium content of the alloy is therefore between 0.16 and 0.31 %, the mean being 0.23 %.

III. SEMI-QUANTITATIVE ANALYSIS OF NICKEL

Reagents:

1. Cyclohexanedionedioxime (saturated solution in ethyl alcohol) gives a rose coloured precipitate, in the presence of zinc sulphate (50 % aqueous solution).
2. Rubeanic acid (saturated solution in ethyl alcohol) gives a blue coloration.
3. Diammonium tetrathiocyanatomercurate (solution of 2.7 g of mercuric chloride + 3 g of ammonium thiocyanate in 100 ml of water + zinc sulphate (10 % aqueous solution)) gives a greenish precipitate.

Establishment of a reference system

A standard solution of nickel sulphate ($NiSO_4 \cdot 7H_2O$) containing 0.1 % of nickel is diluted until the limit of detection is reached.

Reaction 1 (cyclohexanedionedioxime).

The standard solutions of decreasing concentration (total volume 10 drops) are placed in 10 cups of the drop plate, by the same technique as employed in the case of chromium.

Then to each cup are added one drop of zinc sulphate solution, one drop of reagent and one drop of concentrated ammonia. In the presence of nickel there is produced a rose coloured precipitate.

Limit obtained: F_4 (solution 0.0001 %).

The table below presents another type of lay-out, allowing a direct reading of the nickel content (one table per reagent).

TABLE I
cyclohexanedionedioxime

H_2O drops	Ni drops	A^x 0.1 % solution	B^x 0.01 % solution	C^x 0.001 % solution	D^x 0.0005 % solution	E^x 0.0002 % solution	F^x 0.0001 % solution
9	1	0.4	4.0	40	80		
8	2	0.2	2.0	20	40	100	
7	3	0.13	1.3	13.3	26.6	66.6	
6	4	0.1	1.0	10	20	50	100
5	5	0.08	0.8	8	16	40	80
4	6	0.07	0.7	6.6	13.3	33.3	66.6
3	7	0.06	0.6	5.7	11.4	28.6	57.1
2	8	0.05	0.5	5.0	10	25	50
1	9		0.44	4.4	8.8	22.2	44.4
0	10		0.4	4.0	8.0	20	40

Remarks: The addition of $Zn^{\cdot\cdot}$ eliminates interference from this element. We have, in fact, found an increase in sensitivity in its presence. On the other hand, it is advantageous to effect a preliminary separation of the nickel by formation of the soluble complex amine, particularly in the presence of large amounts of iron (see determination of Ni-Cr in a steel, later).

The iron can alternatively be masked by the fluoride anion.

It should also be noted that copper interferes when present in large amounts.

Reaction 2 (rubeanic acid).

This reaction is carried out on filter paper (LOUIS SCHLEIFFER No. 576, 5.5 cm diam).

The procedure is by successive dilutions in cups in the drop plate, as in the previous case.

One drop from each of the solutions is placed on a filter paper impregnated with rubeanic acid and dried, then exposed to the vapours of ammonia and allowed to dry for 5 minutes. A blue stain indicates nickel. In the case of low concentrations of nickel a blue ring is formed.

Limit obtained: F_4 (0.0001 % solution).

TABLE II

rubeanic acid

Ni drops	A^x 0.1 % solution	B^x 0.01 %	C^x 0.001 %	D^x 0.0005 %	E^x 0.0002 %	F^x 0.0001 %
1	0.4	4.0	40.0	80		
2	0.2	2.0	20.0	40	100	
3	0.13	1.3	13.3	26.6	66.6	
4	0.1	1.0	10.0	20	50	100
5	0.08	0.8	8.0	16	40	80
6	0.07	0.7	6.6	13.3	33.3	66.6
7	0.06	0.6	5.7	11.4	28.6	57
8	0.05	0.5	5.0	10	25	50
9		0.44	4.4	8.8	22.2	44.4
10		0.4	4.0	8.0	20	40

Remarks: As in the case of the previous reaction, it is often preferable to separate the nickel by ammonium hydroxide (particularly in the presence of iron). Copper and cobalt also give the reaction.

Reaction 3 (diammonium tetrathiocyanatomercurate + $Zn^{\cdot\cdot}$).

10 successive dilutions are carried out on a drop plate (see above). To these solutions are added one drop of 10 % zinc sulphate solution and one drop of a solution of diammonium tetrathiocyanatomercurate. There is formed slowly, in the

presence of nickel, a greenish precipitate (up to about 15 minutes should be allowed).

Limit obtained: E_3 (0.01 % solution).

TABLE III

diammonium tetrathiocyanatomercurate + $Zn^{\cdot\cdot}$

Ni drops	A^x 0.1 % sol.	B^x 0.08 %	C^x 0.04 %	D^x 0.02 %	E^x 0.01 %
1	30	37.5	75		
2	15	18.7	37.5	75	
3	10	12.5	25	50	100
4	7.5	9.5	18.7	37.5	75
5	6	7.5	15	30	60
6	5	6.25	12.5	25	50
7	4.3	5.4	10.75	21.5	43
8	3.75	4.75	9.4	18.7	37.5
9	3.33	4.25	8.3	16.6	33.3
10	3	3.75	7.5	15	30

Remarks: Copper, cobalt and iron react and interfere. The iron may be eliminated by ammonium hydroxide.

Analysis of nickel alloy: composition of the alloy: Ni 30.5 %, Fe 69.5 %.

1. Assay by cyclohexanedionedioxime (see Table I). The assay is carried out with solution C (0.001 %).

Results obtained: C_2^x reaction positive (more than 20 % of nickel), C_1^x reaction negative (less than 40 % of nickel).

The nickel content of the alloy is, therefore, between 20 and 40 %. Closer figures may be obtained by using a solution of different dilution, for example, solution E (0.0002 %). The limit of detection is found to be between 7 and 6 drops, i.e., between 28.5 and 33.3 %.

2. Assay by rubeanic acid (see Table 2). Solution C (0.001 %) is first used and then solution E (0.0002 %), the results obtained being similar to that of the preceding assay. The nickel content is between 25.5 and 33.3 %.

3. Assay by diammonium tetrathiocyanatomercurate (see Table 3). Solution E (0.01 %) is used and the limit lies between E_{10}^x (more than 10 %) and E_9^x (less than 33.3 %). Then the solution is dilute and the assay is carried out with solution D (0.02 %): D_5^x reaction positive (more than 30 %), D_4^x reaction negative (less than 37.5 %).

IV. DETERMINATION OF CHROMIUM AND NICKEL IN A STEEL

To complete the preceding investigations, we have endeavoured to determine, in a similar alloy, the content of chromium and of nickel by the method described below.

Composition of the alloy:
8.6 % of nickel
18.5 % of chromium
0.7 % of silicon
iron: remainder.

0.1 g of the sample, weighed to about 1 %, is dissolved in aqua regia, the solution evaporated to dryness, and the residue taken up with dilute nitric acid. The solution is neutralised with sodium hydroxide and solid sodium peroxide added. The excess of peroxide is destroyed by boiling and the solution is then filtered.

For determination of the chromium, the filtrate is transferred to a graduated flask and diluted to 1.000 ml with distilled water. From this solution, solutions C^x and D^x are prepared.

Reaction with diphenylcarbazide: solution D^x_4 gives the limit of detection, hence the chromium content is between 12.5 and 16.7 %.

Reaction with gum guaiacum: solution C^x_4 gives the limit of detection. The chromium content is between 15.6 and 20.8 %.

Reaction with strychnine: solution C_4 gives the limit of detection, hence the chromium content is between 13.5 and 15.6 %.

The chromium content of the alloy, therefore, lies between 12.5 and 20.8 %, with a mean value of 17 %.

For the determination of nickel, the precipitate of the hydroxides is dissolved in dilute nitric acid. The solution is treated with ammonia; the iron is precipitated while nickel remains in solution in the form of the complex amine. The solution is filtered and diluted to a litre in a graduated flask (0.01 % solution). The following assays are then carried out:

1. Assay with cyclohexanedionedioxime (see Table I). The reaction is still distinct with B^x_1, from which the alloy contains more than 4 % of nickel.

Tests are then carried out with a solution diluted twentyfold, solution D (0.0005 %); the limit of detection is then found to be between D^x_{10} (more than 8 % and D^x_9 (less than 8.8 %).

2. Assay by rubeanic acid (see Table II). Identical results are obtained. The percentage of nickel in the alloy therefore lies between 8 and 8.8 %.

After giving the principle of this method and several examples of its application, we think the reader will appreciate the value of such assays for the rapid control of alloys and various industrial products whether solid or liquid.

In addition, this semi-quantitative method has a great advantage of being carried out easily, where there are limited facilities, with the most simple of apparatus. It presents great flexibility; thus in the above examples, we have established the analysis in the most general case, the reagents chosen being as selective as possible, if not the best. But if it had been necessary, for example, to establish a reference system for an alloy whose composition had been qualitatively determined and where there were, in this case, only several interfering ions, our choice would probably have been different. We should have been able to employ reagents which were less selective but more sensitive and with a greater factor of visibility, which would have increased the precision of the method.

We hope to publish, at a later date, results obtained with other ions but even now we think it advisable to warn the chemist that although this method is capable of being applied in a very wide field it will not always give accurate results. It should be reserved, in the first place, for the determination of the constituents of a substance which are presents only in small amounts. Under these circumstances the analysis may be considered as quantitative.

SUMMARY

The authors propose a new semi-quantitative method of analysis, simple and very rapid, based on the use of sensitive and specific organic reagents, by determination of the limit of detection of the reaction.

Two methods are described:

1. Determination of chromium by use of the following reagents: gum guaiacum, strychnine or diphenylcarbazide.
2. Determination of nickel by means of cyclohexanedionedioxime, rubeanic acid or diammonium tetrathiocyanatomercurate.

RÉSUMÉ

Les auteurs proposent une nouvelle méthode semi-quantitative, simple et très rapide, basée sur l'emploi de réactifs organiques, sensibles et spécifiques, par détermination de la limite de perceptibilité de la réaction.

Deux procédés sont décrits:

1. Dosage du chrome, à l'aide des réactifs suivants: résine de gaïac, strychnine ou diphénylcarbazide.
2. Dosage du nickel, à l'aide de: cyclohexanedionedioxime, acide rubéanique ou tétrathiocyanatomercurate diammonique.

ZUSAMMENFASSUNG

Die Verfasser schlagen eine neue semiquantitative, einfache und sehr schnelle Methode vor, um die Wahrnehmbarkeitsgrenze einer Reaktion zu bestimmen. Diese Methode beruht auf der Verwendung von empfindlichen und spezifischen organischen Reagenzien.

Zwei Verfahren werden beschrieben:

1. Chrombestimmung, mit Hilfe volgender Reagenzien: Guajakharz, Strychnin oder Diphenylcarbazid.
2. Nickelbestimmung, mittels: Zyklohexandiondioxym, Rubeansäure oder Diammoniumrhodanidomercurat.

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