

Division des Sols



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C H L O R E

Analyse des Sols, Des Eaux et des Plantes

Damas

LE CENTRE ARABE
POUR LES ETUDES DES ZONES ARIDES ET DES TERRES SECHES
(ACSAD)

Le Centre Arabe
Pour les Etudes des Zones Arides et des Terres Sèches

A C S A D

N° . 500

C H L O R E

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Damas - Decembre 1981

Auteur : xxx / ARIANA
Titre : Dosage des chlorures par potentiometrie de zero.
Public : Methodes de dosages utilisees au Laboratoire de l'ARIANA.
Ministere de l'Agriculture, D.R.E.S, Tunis.
Pages/ref: 4 pages / 2 ref.

Resume:

Une electrode argent/chlorure d'argent est une demi-pile developpant un potentiel de 521 mV pour une concentration de 0.01 meq/l de chlore. Si on oppose une demi-pile de +521 mV de potentiel, ce systeme sera au zero electrique pour une concentration de 0.01 meq/l CL.

L'article decrit la preparation d'une electrode Ag/AgCl a partir d'une electrode Ag metallique, et d'une electrode a quinhydrone, ainsi que le montage de potentiometrie de zero.

Le dosage se fait par le nitrate d'argent N/35,5 .

Figures :- Preparation de l'electrode argent/chlorure d'argent .
- Preparation de l'electrode a quinhydrone .
- Montage galvanometrique .

Precision: 1 goutte de reactif correspond a 0.082 mgr de NaCl .

Materiel : Si on ne prepare pas les electrodes soi-meme:

- Electrode Ag/AgCl .
- Electrode a quinhydrone .
- Galvanometrie .

Produits : Pour preparer les electrodes soi-meme:

Alun de fer et d'amonium . Hydroquinone . Gelose (Agar-Agar) . Nitrate de potassium . Phtalate acide de potassium . HCL . Acide nitrique . Rouge de methyle.

Mots Cles: CHLOR / ELEC - TRAIN / EAUX /

ARIA

Auteur : XXX/ARIANA

Titre : Dosage des chlorures par colorimétrie

Public : Methodes de dosage utilisées au Laboratoire de l'ARIANA,
Ministère de l'Agriculture, D.R.E.S Tunis .

Pages/ref: 4 pages/ 2 ref .

Résumé:

Le chlore est précipité sous forme de chlorure mercurique par le thiocyanate mercurique . Les ions sulfocyanure en excès donnent une couleur rouge en présence d'ions ferriques. La coloration (480nm) est proportionnelle à la teneur en chlorure .

Méthode:

La prise d'essai est déterminée par la teneur en sodium déterminée préalablement, et varie de 10 ml à 0.1 ml si Na varie de 2.4 à 240 meq/l dans la même solution .

A l'échantillon on ajoute très exactement 2 ml de solution saturée (soit 1 gr/l à peu près), de thiocyanate mercurique $(\text{SCN})_2\text{Hg}$, puis 2 ml d'alun de fer et d'ammonium (6% dans HNO_3 6N) . On laisse la couleur se développer à l'obscurité .

Figure: Courbe d'étalonnage pour cuve de 2 cm, filtre 492nm de 0 à 16 meq/l de Cl .

Matériel: Colorimètre .

Produits: Solution saturée de thiocyanate mercurique . Alun de fer et d'ammonium . Chlorure de potassium .

Authors : Ayers (CORA W.)
 Title : Automated analytical technique for tobacco analysis.
 Public. : Technicon Int. symposium. Reprint by Techn. Int. CO. Ltd, London 1964
 Page/ref.: 11 / no ref.

Summary : The flow diagrams in use to determine elements of tobacco by continuous flow colorimetry are described for four elements (Iron, Manganese, Phosphorus, Chloride). Methods for extraction of Sodium and Potassium from tobacco leaves were also investigated during a collaborative test involving 16 laboratories. The methods tested were; 1) a simple aqueous extraction; 2) a dilute HCl extraction; 3) a column extraction and ; 4) (HNO₃, HClO₄) wet digestion. Flow diagrams are schematically as follows (flow rates are given in ml/min) :

Iron:

- Sample (1.2)/water (1.2)/13% ammonium thiocyanate (2x2,0)/
 Single Coil/Debubbler (2,0) to waste/480 mm-15 mm cell.

Manganese : (sampling by a double crook)

- Sample (1.2), water(1.2) sodium-periodate-phosphoric acid
 (2 x 2,0)/heating bath 95°C/Debubbler 2.0/520 mm-55mm cell/.

Phosphorus:

- Sample(0.23)/Ammonium molyb.(2.0)/Air(1.2) Ammonium vanadate (2.0)/ 4 time delay coil/ debubbler(2.0)/ 420 mm - 15 mm cell/.

Chloride:

- Sample (0.32), diluent (2.0), Air(1.2) /water (2.5)/water (2.5), Air (1.2) dialyzer 37°C/mercuric thiocyanate 50% ETOH (2 x 2,5) Mixing coil/debubbler (2.0)/480 mm-15mm cell/.

Tables : - Comparison of methods of extraction for potassium and sodium.
 - Comparison of results of three methods on K₂O analysis of different tobacco parts.
 - Variation of result with sampling rate.

Precision: An overall coefficient of variation between 1.5 and 5% was found for K at 3% level, Na at 250 ppm level, P at 0.25% level, Cl at 1% level, Fe at 220 ppm level, Mn at 80 ppm level.

Material: Single channel continuous flow colorimeter - recorder - flame photometer (sodium and potassium) - automatic sampler.

Authors : BROWN (E), SKOUGSTAD (M.W) FISHMAN (M.J)
 Title : Methods for collection and analysis of water samples for dissolved mineral and gases : Chloride and residual Chlorine .
 Public : US Geol . Survey . Book 5, Chapter A1 - 1970 .
 Pages/ref:(73 - 74) / 1 ref .

Summary:

Chloride is measured either by the Mohr's method or by titration with mercuric nitrate . These two methods are well-known. They are nevertheless described into detail.

Residual Chlorine may be found in waste water as a result of Chlorination or industrial discharge . In water , Chlorine hydrolyzes and usually ."free available chlorine" is accepted as including HOCl and OCl .

Oxydation of orthotolidine by chlorine gives a yellowish-known complex measured colorimetrically . The reaction is rapid with free forms of chlorine and slower with combined forms. The method, The reagents and the calculations are explained in detail .

Precision:- Mohr's method: r.s.d 4,5% at 27 ppm level , (n= 19); range of application 20 to 2000 mgr/l .
 - Mercuric : r.s.d 4% at 26 ppm level (n= 8); range of application 0.1 to 100 mgr/l .
 - For free chlorine, a precision of + 0.1 mgr/l can be expected at 1 to several ppm .

Iron, Manganese, Nitrite produce a positive interference . A measurement in situ is necessary, since free chlorine is released rapidly from samples .

Chemical/Material:

Color comparator for free chlorine

- Mohr's : NaCl - $\text{K}_2\text{Cr}_2\text{O}_4$ - AgNO_3
- Mercuric: NaCl - H_2O_2 - hydroquinone - Mercuric Nitrate- bromophenol, s. diphenyl carbazine - EtOH - HNO_3 - NaOH .

Key Words: - CHLOR -

Auteur : CANELLI (E)
Title : Simultaneous automated determination of chloride, nitrite,
nitrate and amonia in water and waste water.
Public. : Water, Air and Soil Pollution 5-1976.
Pages/Ref.: 339-348/11 ref.

Summary : A method for the determination of chloride and the three forms of nitrogen in water is described. The method uses a six channel continuous flow colorimeter requiring 2 ml sample only. The manifold is fully detailed, including tube length which was adjusted to achieve the best peak resolution.

Methods : The indophenol blue method is used for amonia, nitrite is determined as azo-compound, nitrate is reduced with an on-line column (Cd-Cu) and determined at 540 nm as nitrite. A general method to correct for the positive interference of nitrite on nitrate, is proposed. It is based on the assumption that the reduction efficiency of the column is reasonably constant within the range of concentrations investigated. The preparation and the conservation of reagents is described into detail. A recovery study was made on sample spiked with NaCl, KCl, NaNO₂. The range of recoveries over 10 samples were between 99 and 105%. No effect of pH was observed between pH 3,7 and pH 11,2. An overall time of analysis of 0.2 min-hour per sample and per batch of 100 samples is claimed. The technical points which may rise problems during the course of analysis, as the automatic filtration, the length of connecting tubes, the reagent concentration are explained. The proposed procedure has been used also to determine amonia-N after Kjeldhal digestion and on sample irradiated with UV light. The method seems to be applicable to saline water.

Table/Fig.: - Recommended analytical concentration ranges for fresh water and waste water.
- General manifold
- A separate figure is given for each channel.

Precision: Range (in ppb of N) Nitrite (1-50) and (5-200) Nitrate (5-500) and (100-2500), amonia (5-500) and (100-1500), Chloride (10000-50000). The values of optimum concentration \pm relative standard deviation was (in ppt): 50 \pm 1 for N, 1000 \pm 1.1 for NO₃, 300 \pm 1.8 for NH₄ (number of successive det.: n = 11).

Authors : HANSON (N.W)

Title : Official standardized and recommended methods of analysis: Alkalinity.

Public : Soc . for Analyt . Chemistry - London - 1960 .

Page/ref: 334 - 337 / 1 ref .

Summary: Two methods are described for the determination of water alkalinity :

Firstly: Determination using indicators : 3 drops of phenolphthalein are added to a clear (centrifuged) sample, then the solution is titrated until the pink colour is discharged. The total alkalinity is determined using methyl-orange indicator solution , the titration is continued until the yellow colour changes into red . If the PH is below 8,3 there is not carbonate and phenolphthalein remains colourless .

- Free chlorine interference is removed by addition of minimal amount of sodium thiosulphate .

Secondly: Determination using electrometric titration: A PH meter is fitted with a combined glass electrode . After having standardized the PH meter , sulfuric acid is added to the sample and the values read from the PH meter are plotted, or more simply followed until PH 8,3 and then, until PH 4,5 .

- The end point of titration is made clearer by the use of a screened indicator: (xylene cyanol FF is screened indicator for methyl-orange end point).

Precision: Free chlorine interference can be removed by 1 drop of 1,0 N sodium thiosulphate .

- The PH of the bicarbonate neutralization depends on the relative quantities of CO_2 and total alkalinity . The PH of 4,3 should be replaced by:

PH 5,1 at alkalinity levels (as mgr/l of CaCO_3): 30
PH 4,8 at " " " " (as mgr/l of CaCO_3): 150
PH 4,5 at " " " " (as mgr/l of CaCO_3): 500

Material : PH meter - glass electrode .

Chemicals: Sodium thiosulfate- Na_2CO_3 - H_2SO_4 - methyl orange- phenolphthalein - Ethanol .

Key Words: CARB /

Auteurs : KREMPF (G) & MILLION (D)
Titre : Dosage de l'ion chlorure.
Public : Universite Louis Pasteur, Strasbourg 1975.

Resume:

Methode colorimetrique pour la determination de l'ion chlorure, l'echantillon est traite avec du thiocyanate mercurique qui forme un complexe mercurichlorure. L'ion thiocyanate en exces devient rouge en presence de fer ferrique. On colorimetre a 480nm une methode de dosage automatique est proposee:(en ml/min)

Echantillon: (2.5) / Air , (1.2)/reactif , (1.60) /
double bobine/ repompage (2)/.

Figures : Ecart type relatif:- 3% entre 30 et 400 micromoles/l en CL.
- 10 a 25% entre 30 et 10 micromoles/l.

Matériel : Colorimetre automatique equipe du montage chlorure.

Produits : Thiocyanate mercurieux . Methanol . Nitrate ferrique .
Acide nitrique .

Mots Cles: CHLOR / COLOR / EAU /

KREM

Auteurs : GREYER (C) , BRUTTEL (P) & ZIEGLER (R)
 Titre : Determination du chlorure, de l'hypochlorite et du chlorate dans une solution par titrage potentiometrique.
 Public : METROHM HERISAU No. A8f. 1971.
 Pages/ref: 2 pages / 2 ref.

Resume:

a. Teneur en hypochlorite:

On alcalinise 10 ml d'echantillon avec 2 ml NaOH, on titre avec anhydride arsenieux (gamme 1000 mV).

b. Chlorure:

Apres titrage de l'hypochlorite, on ajoute 5 ml acide sulfurique 2N, 100 ml d'eau distillee, on titre au nitrate d'argent 0.5N avec une electrode d'argent, chlorure d'argent et electrode de reference au sulfate mercurieux, on dose chlore + hypochlorite reduit. On obtient le chlore par difference (a) - (b).

c. Chlorate:

A 10 ml d'echantillon, ajouter 25 ml acide sulfurique 2N et 5 gr de sulfate de fer (II), pour reduire les chlorates en chlorure, chauffer, laisser bouillir 1 min. refroidir, diluer avec 150 ml eau distillee, titrer avec AgNO_3 0.5N comme precedemment (memes electrodes, meme gamme 1000mV). La teneur en chlorate est proportionnelle a (c) - (b).

Matériel : a. Electrode de platine.
 b. Electrode d'argent, chlorure d'argent, electrode de reference au sulfate mercurieux. Enregistreur potentiometrique. Poste de titrage.

Produits : Sulfate de fer . Acide sulfurique . Nitrate d'argent . Anhydride arsenieux . Hydroquinone (fac) . Soude .

Mots Cles: CHLORE / ELEC / EAUX /

METR

Authors : MAC LEOD (S) & ZARCINAS (B)
 Title : The determination of amonium and chloride by an autoanalyser for the measurement of cation exchange capacity of soils.
 Public : Communication in Soil Science and Plant Analysis 7-8-1976.
 Pages/ref: 743 - 750 / 8 ref.

Summary:

A continuous flow colorimetric method is developed for the simultaneous determination of amonium and chloride from amonium chloride used to saturate the exchangeable complex of soils.

Method: - Salts are removed by a glycol-ethanol wash.
 - Soil is saturated with 1N amonium chloride PH 8,5 in 60% ethanol. Amonium adsorbed is replaced by a mixture of calcium nitrate / potassium nitrate (0.25 M / 1.5 M) .

Colorimetry: Amonium is determined by Berthelot's sodium phenate - hypochlorite reaction and chloride by mercury (II) thiocyanate, ferric ions being provided by a ferric nitrate solution.

Diagrams (shown here separately) are:

1. Sample (1.6) / air (3.4) / water (1.6) / single mixing coil / iron (III) nitrate 20 gr/l in nitric acid (1.6) / single mixing coil / (II) thiocyanate 0.8 gr/l (1.6) / single mixing coil / colorimeter 480 nm / repump to waste 2.9 / .
2. Sample (1.2) / air (3.9) / EDTA (disodium) M/60 (3.9) / sodium phenate (2.0) / single mixing coil / sodium hypochlorite (3% free chlorine) (2.0) / single mixing coil / bath 95°C (20 feet) / cooling coil / colorimeter 620 nm / repump to waste (2.9) / .

Correction for free amonia used to adjust the PH of saturating solution to 8.5 is made by applying a constant correction factor to amonium results when high retention of saturating solution is suspected (swelling clays). Analysis of variance was made on 23 soil samples. The results for manual and automatic determination of chloride was the same. Standard deviation of amonium determination was higher than in manual titrimetric method.

Precision: R.s.d is 2% at 48 me/100 g amonium level and 0.7% at 15 meq/100 gr level of chloride.

Chemicals: Glycol . Ethanol . Sodium hypochlorite . Phenol sodium hydroxyde . EDTA (disodium) Mercury (II) . Thiocyanate . Iron (III) nitrate . Amonium chloride . Calcium nitrate . Potassium nitrate .

Key Words: CHLORIDE / AMONIUM / AUTOANALYSE / CEC /

MACL

Author : xxx / ORION
 Title : Chloride ion activity electrode model 92 - 17.
 Public : ORION Research Incorporated 1968.
 Pages/ref: 4 pages / --

Summary:

The electrode responds to the activity of free unbond chloride ion in solution; some anions interfere with the response to chloride ion (halide, nitrate, acetate, hydrogen carbonate, sulfate).

The chloride ion activity electrode can be used at different PH (2 to 10) especially at low concentration of Cl^- . It is free from sulfide, cyanide and ammonia interference.

The chloride electrode is made of a thin layer of water immiscible liquid ion exchanger which is held in place by a porous membrane disc, inside the electrode body, there is an aqueous filling solution, and the outside is a surface of Ag/AgCl internal reference element. As the filling solution contains a fixed level of chloride there is an equilibrium state, and any change in chloride ion activity in the sample solution produces a change in the electrode potential.

Tables :- Typical electrode response to chloride activity and concentration.
 - Interfering ions and their selectivity constants.
 - Electrode potential behavior versus solution PH (for NaCl solution).

Precision:- Concentration range : 0.35 - 35000
 - PH range : 2 - 10
 - Temperature range : 0 - 50°C .

Key Words: CHLOR / ELEC - INST / EAUX /

ORIO

Auteur : xxx / ORSTOM
Titre : Determination des ions chlorure dans les extraits aqueux de sols.
Public : ORSTOM, PARIS. Roneo.
Pages : 4 pages.

Resume:

L'aliquote ayant servi au titrage des carbonates et bicarbonates par potentiometrie est utilisee pour doser le chlore suivant la methode de Volhard: Un exces d'ions Ag est titre par du sulfocyanure de potassium de titre connu. La fin de reaction est indiquee par des ions Fe^{+++} qui donnent une coloration rouge avec l'exces de sulfocyanure. Il faut isoler le precipite par du benzene pour eviter la redissolution du precipite d'AgCl.

La methode potentiometrique utilisant une electrode d'argent et une electrode de reference au KNO_3 / calomel est decrite en detail.

Materiel :- PH-metre utilise en potentiometre
ou- Potentiometre enregistreur (fac), Electrode d'argent,
Electrode au colomel.

Produits : Methode potentiometrique:

Acide sulfurique . Bicarbonate de sodium . Nitrate d'argent . Gelatine . Nitrate de potassium . Chlorure de potassium .

Methode manuelle:

Thiocyanate de potassium . Nitrobenzene ou ether .
Sel de fer ferrique .

Mots Cles: CHLOR / ELEC - POTENT / EAUX / METH /

ORST

Auteur : XXX / ORSTOM

Titre : Determination des ions carbonate($\text{CO}_3^{=}$) et bicarbonate (CO_3H^-) dans les extraits aqueux de sols .

Public : Roneo ORSTOM . non publié .

Page/ref: 6 pages / pas de references .

Résumé:

- On dose les ions carbonates et bicarbonates par H_2SO_4 . Les points equivalents des reactions de neutralisation peuvent être suivis par des indicateurs colorés . La difficulté pour les extraits de sols souvent troubles est de visualiser le virage . Plusieurs colorants sont possibles :

- 1)- Le bleu de thymol-sulfonephtaléine: PH 9,6 bleu, PH 8,0 jaune .
ou La phénolphtaléine:..... PH 10,0 rouge, PH 8,3 incolore
- 2)- L'hélianthine :..... PH 4,4 rouge, PH 3,1 jaune
ou- Le bleu de bromophénol :..... PH 4,6 bleu, PH 2,8 jaune

C'est le couple phénolphtaléine puis hélianthine qui donne les meilleurs resultats .

- On peut plus simplement suivre les variations de PH en cours de neutralisation , à l'aide d'un electrode de verre combinée :

- PH 8,3 : neutralisation de la moitié des carbonates .
- PH 4,1 à 4,3 : neutralisation des bicarbonates .

Les points equivalents peuvent être déplacés suivant la force ionique du milieu . On utilise 50ml d'échantillon dosé avec H_2SO_4 0,05 N .

On peut suivre la reaction avec un potentiomètre et une burette dont le debit est asservi à la pente de la courbe de neutralisation . En fin de reaction on peut doser les chlorures sur la même prise d'essai(s'il n'y a pas eu de colorants ajoutés) .

Materiel:- PH metre - electrode de verre - electrode de reference calomel .

Produits: Tampon PH 8,0 et 4,0 - H_2SO_4 - Na_2CO_3 - phénolphtaléine- bleu de thymol - Ethanol - bleu de bromophénol .

Mots Cles: CARB / ELECT / TRAIN

Author : xxx / PINTA
Title : Indirect determination of non-metallic elements.
Public.in: PINTA (M), Atomic Absorption Spectroscopy, HILGER 1974.
Pages/ref: page 228 / -

Summary:

Indirect method for the determination of chloride ions. The method is based on the addition of an excess silver nitrate of a known concentration which precipitate chlorine, as silver chloride: Excess silver is determined by Atomic Absorption Spectroscopy.

The sample is diluted before analysis so that the concentration lies in the range between 20-80 mg/l CL.

The detection limit of silver being 8 ppm the water should have at least twice as much chloride for the method to be applicable.

Figure : Standard curve for determination of chloride in fresh water.

Precision: Comparison with Volhard's titrimetric on 8 samples ranging from 0.1 to 4 gr/l CL gave a \pm difference varying from 1 to 8% with an average of 3%.

Apparatus: AAS (328 nm): Silver Hollow Cathod Lamp . Normal air acetylene burner .

Chemicals: Silver nitrate solutions. Nitric acid. Hydrochloric acid solutions.

Key Words: CHLOR / AAS - METH / H₂O /

PINT

Authors : SAFFIGNA (P.G) , KEENEY (D.R) & HENDRICKSON (L.L)
 Title : Halide analysis in soils with a chloride titrator and a bromide electrode.
 Public : Communication in Soil Science and Plant Analysis 7-8-1976.
 Pages/ref: (691 - 699) / 13 ref.

Summary:

Amperometric measurements for accurate determination of chloride, bromide and iodide separately and in all possible equimolar concentration (0.125 to 45 mM potassium salt) using the chloridometer.

The experiment made was to evaluate on field and lysimeter the concentration of chloride and bromide after distributing in the soil KCl and KBr solution and making a comparison study.

Chloride and bromide ions in the sample were determined together with the chloridometer, after that the bromide was determined directly with Br electrode, and chloride by difference. An accurate estimate of the sum (Br + Cl) is possible because titration speed is the same for Cl and Br separately.

The Chloride did not interfere in bromide determination when using the bromide electrode at a 10:1 ratio Br/Cl, in the mixture Br - Cl containing 20 mM Cl the measured bromide concentration must be reduced to 0.9mM.

Tables :- Amperometric measurement of chloride, bromide and iodide with the chloridometer.
 - Recovery and ratio of chloride and bromide in the field profiles and the lysimeter leachate.

Precision:- The response of electrode to KBr (from 0.5 micromoles to 1 millimole), to KCl and $MgCl_2$ (from 0.3 to 282 millimoles) and to $Ca(NO_3)_2$, (1.6 to 161 millimoles) as well as mixture of these salts was investigated.
 - The Br electrode provided reproducible data with an average coefficient of variation of 4% from 5 micromoles to 1 millimole of KBr in solution. Sensitivity is 0.5 micromoles of Br.

Material : Chloridometer . Bromide specific ion electrode + junction reference electrode + ion PH-meter .

Chemicals: Potassium chloride . Potassium bromide solution .

Key Words: CHLOR / ELEC - INSTR /

SAFF

Authors : SMART (R. ST. C) , THOMAS (A.D) & DROVER (D.P)
 Title : Selective ion electrode measurements of chloride concentrations
 in the determination of cation exchange capacities of soils.
 Public : Communication in Soil Science and Plant Analysis 5-1-1974.
 Pages/ref: 1-11/ 5 ref.

Summary:

Comparison of two procedures for the determination of chloride ions. The first method was electrometric titration using a quinhydrone silver-silver chloride electrodes and silver nitrate as titrant. The second method used an Cl selective ion electrode connected to an PH-ion meter calibrated against standards of known chloride ion concentration (1, 10 and mM in equimolar sodium chloride, sodium sulfate solution 0.5M solution).

The possibility of applying the method to measurement of CEC in soils using amonium chloride was investigated.

Method: For CEC determination, soil was leached with 1N amonium chloride, fixed amonium was displaced by 0.5M sodium sulfate. CEC is proportional to amonium ion in sodium sulfate provided excess amonium is corrected for by measuring chloride as index ion the method is not suitable for saline soils. Soil samples investigated had PH5 to 6.6. Interference of amonia was shown. Calibration of electrode with amonium chloride instead of sodium chloride reduced amonia interference to 5% at amonium concentration less than 20 millimoles. Application of systematic activity coefficient (0.75) for index chloride ion in extracts gave results within $\pm 7\%$ in all cases.

Tables : Comparison of Ion Selective Electrode and titration method for 12 CEC leachates.

Material : 1. Titration method: Quinhydrone electrode . Silver-silver chloride electrode . Null point galvanometer (or potentiometric recorder).
 2. Selective electrode: Chloride selective electrode . PH-ionmeter.

Chemicals: Ethanol . Amonium chloride . Amonia . Sodium sulfate . Sodium chloride. Silver nitrate .

Key Words: CHLORIDE / AMONIUM / CEC / SOILS / ELEC /

SMAR

Auteur : XXX / TECHNICON Cy .

Titre : Dosage des chlorures - Methode industrielle N° 99 - 70W .

Public : Publication comp . Technicon - non datée .

Page/ref: 3 / 0 ref

Resume :

L'ion chlore se combine avec le mercure et en presence de thiocyanate mercurique libere l'ion thiocyanate . En presence d'ion ferriques l'ion thiocyanate forme un complexe rouge , dont l'absorption , mesurée à 480nm , est proportionnelle à la concentration initiale en chlorures . Le manifold pour des concentration de 0 à 100 ppm est le suivant :

Air (0.32)/eau (1.6 / echantillon (0.42) / bobine 10 tours/evier repompage (0.32) / eau (0.80) / Air (0.32) / bobine 10 tours / thiocyanate 0,417 % (0.80) / bobine 20 tours colorimetre 15 m/m , 480nm / debullage (0.80) / .

L'etage de dilution 1/5^e peut être supprime pour les concentrations plus faibles .

Une technique pour eliminer la pollution mercurique des effluents est expliquée . On precipite le mercure sous forme de sulfure à l'aide d'une solution de thioacetamide, on garde ensuite le precipité en vase clos .

Precision : Limite de detection 7,5ppm - coefficient de variation au niveau de 56 ppm : 1,5 % .

Produits : thiocyanate mercurique - Nitrate ferrique - Methanol. NaCl-HNO₃ .

Mots cles : CHLOR / COLOR /

TECH

Authors : XXX / TECHNICON

Title : Methyl orange alkalinity in water and waste water .

Public : Industrial Method N° 111-71 W/B Technicon Cy - 1976 .

Page/ref: 2 / no reference .

Summary:

A methyl orange indicator solution is buffered by potassium acid phthalate ($C_8H_5KO_4$) at PH 3.1 . At this PH the reagent changes only slightly of color with a change in alkalinity of the solution. The reaction may be used to determine the alkalinity of water samples at 550nm by using inverse colorimetry .

The following flow diagram may be used with a continuous flow colorimeter :

- Water (2.00) / Air (0.32)/ Methyl orange PH 3.1(0.80)/ sample(0.32)/ double coil (20 turns)/debubbler to waste(0.60)/ colorimeter 15mm - 550nm./
- Flow rates are given between brackets in ml/mm .

Tables : Assembly of flow tubes (manifold) .

Precision:- Free forms of chlorine interfere. Addition of sodium thiosulfate suppresses this interference . Since the reaction does not follow Beer-Lambert's law it is necessary to calibrate the standard solutions .

- Detection limit is 10 mg/l . At 250 ppm level (as $CaCO_3$), the relative standard deviation is 0.28% (the number of samples is not given) .

Reagents : $C_8H_5KO_4$ - HCL - Methyl orange - Na_2CO_3 . Reagents must be prepared with water free of CO_2 .

Key Words: CARB / COLOR /

Author : xxx / VARIAN
Title : Atomic Absorption: Chloride.
Public : VARIAN TECHTRON No. 85.10.009.00 1978.
Pages/ref: 2 pages / -

Summary:

Standard conditions for silver measurement by Atomic Absorption. Application to chloride:

a) To an aliquot containing 0.5 to 5.0 meq CL add 20 ml of 1000 meq/l silver (as silver nitrate). 1 ml nitric acid (extra pure, free of chloride) make up to 200 ml. Allow to stand overnight in a dark place. Centrifuge, take an aliquot measure silver by AAS.

Standard are prepared from sodium chloride in the same conditions.

b) Alternative procedure: wash the precipitate as obtained in a) with 10 ml of 1% nitric acid (twice). Dissolve in 10 ml 50% ammonia. Rinse and complete dissolution with 10 ml more. Transfer to volumetric flask, adjust. Measure silver by AAS. Both methods may be used together, providing a cross-check for the analysis.

Precision:- Iodide, bromide, cyanide and sulphide interfere by precipitating silver.
- Optimal working range 1 to 5 ppm silver i.e 0.5 to 2 milligrams chloride.

Material : AA Spectrophotometer . Silver Hollow Cathod Lamp.

Chemicals: Silver nitrate . Nitric acid . Sodium chloride.

Key words: CHLOR / AAS - METH / EAUX / TOTAL /

VARI