) (*see* 3.3.18), to aliquo
Let layers separate and
ous solution, let layers
g once. Then add 5 mL
rate, and discard CCl_4.
Discard CCl_4 layer and
nnel evaporate before

1.03B(i) (*see* 3.3.18),
ute dithizone reagent,
e reagents from pipet
eparate and draw off
cted to aspirator with
om CCl_4 layer, add
) s.

sh out with ca 2 mL
equate portion of
suitable container,
re convenient, but
ept in dark until *A*

pectrophotometer
for Zn in blank
lution from curve

, 8, 10, 12, and
n. To each flask
M NH_4OH, add
). Pipet 25 mL
5, 7.5, 10, 12.5,
separators, and
beginning with
nd plot values

Final Action 1965

A. Reagents

(a) *Dilute sulfuric acid.*—0.18M. Dilute 10 mL H_2SO_4 to 1 L.

(b) *Calcium hydroxide saturated solution.*—Filter before use.

(c) *Quinalizarin solution.*—Dissolve 45 mg quinalizarin in 1 L 95–96% H_2SO_4.

(d) *Boron standard solution.*—0.5 mg B/mL. Dissolve 2.860 g H_3BO_3 and dilute to 1 L with H_2O. Prepare working standards by further dilution with H_2O.

B. Determination

Place 1.00–2.00 g dry, ground plant material in Pt or SiO_2 dish. Add 5 mL saturated $Ca(OH)_2$ solution and dry at 105°C. Carefully volatilize over burner, ash in furnace 1 h at 600°C, and cool. Add exactly 10 or 15 mL 0.18M H_2SO_4, break up ash with glass rod, stir gently, and filter. Transfer 2 mL filtrate to colorimeter tube, add an exact amount (e.g., 15 mL) quinalizarin reagent, stopper, and mix by swirling gently. Let stand at room temperature 24 h (or until both unknowns and standards have cooled to same temperature). Shake tube again immediately before reading in photoelectric colorimeter with 620 nm filter.

Adjust colorimeter to 100% *T* with blank solution prepared as above but using 2 mL H_2O in place of test solution. Prepare standard curve with series of standards containing 0.5–10 μg B/mL.

Reference: *JAOAC* **41**, 304(1958).

CAS-7440-42-8 (boron)

3.4.03

AOAC Official Method 928.04
Chloride in Plants
Gravimetric Method
First Action 1928
Final Action

(If bromides or iodides are present in significant amounts, correct results accordingly.)

A. Preparation of Solution

Verify complete retention of chloride in each kind of material by trial, since losses can occur, especially in products high in carbohydrates, if insufficient sodium carbonate is present during ignition, or in any case if excessively high temperature is used.

Moisten 5 g test portion in platinum dish with 20 mL 5% sodium carbonate solution, evaporate to dryness, and ignite as thoroughly as possible at ≤500°C. Extract with hot water, filter, and wash. Return residue to platinum dish and ignite to ash. Dissolve in HNO_3 (1 + 4), filter, wash thoroughly, and add this solution to the original aqueous extract.

B. Determination

To the prepared solution, A, add 10% silver nitrate solution, avoiding more than a slight excess. Heat to boiling, protect from light, and let stand until precipitate coagulates. Filter on weighed Gooch crucible, previously heated to 140°–150°C, cool, and weigh. Report as % Cl.

References: *JAOAC* **11**, 209(1928); **12**, 195(1929); **21**, 107(1938).

CAS-7782-50-5 (choline)

3.4.04

AOAC Official Method 915.01
Chloride in Plants