The Various Analytical Techniques for the Analysis of Sodium Chloride in Food Samples and Their Advantages and Disadvantages

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Approaches for determination of NaCl (salt)

- Direct determination of NaCl (solids only)
- Determination of Na\(^+\) and Cl\(^-\) (or Cl)
- Determination of Na\(^+\) only
- Determination of Cl\(^-\) only
- Determination of Total Cl only
Techniques

- **XRD** (X-Ray Diffraction) (NaCl directly in solids)
- **XRF** (X-Ray Fluorescence) (Na & Tot. Cl in solids)
- **Optical Atomic Emission**
  - Flame (Na only)
  - Plasma (Na, Cl? & other elements)
Techniques Cl⁻ only

- Titration with silver nitrate (AgNO₃)
- Ion Chromatography (IC)
- Ion Selective Electrode (ISE)
- Colourimetric methods
X-Ray Diffraction (XRD)

- Direct determination of NaCl
- NaCl is a crystalline salt
- Arranged in a crystal lattice with a unique structure
- Only solids
Techniques

• XRD (X-Ray Diffraction) (NaCl directly in solids)

• XRF (X-Ray Fluorescence) (Na & Tot. Cl in solids)

• Optical Atomic Emission
  – Flame (Na only)
  – Plasma (Na, Cl? & other elements)
Advantages: X-Ray techniques (XRD & XRF)

- **XRD**: Sees only actual NaCl as NaCl. Can check for other particular Na salts and/or chlorides.
- **Both**: Not necessary to dissolve or extract solid samples. Same sample preparation used for both.
- **XRF**: Na plus Cl, plus other elements of interest.
Disadvantages: X-Ray techniques (XRD & XRF)

**XRD & XRF**

- Instrumentation is expensive and requires a highly skilled operator.
- Limitations on sample throughput.
- Must be dry, preferably also ashed and compressed to withstand vacuum conditions.

**XRD**

Precision (repeatability) not very good.

**XRF**

Less sensitive to lighter elements (Cl and particularly Na).
Periodic Table

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<th>B</th>
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Lanthanide Series:

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<td>Tm</td>
<td>Yb</td>
<td>Lu</td>
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<th>Pa</th>
<th>U</th>
<th>Np</th>
<th>Pu</th>
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<th>Cm</th>
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<td>Fm</td>
<td>Md</td>
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<td>Lr</td>
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Techniques

- **XRD (X-Ray Diffraction)** (NaCl directly in solids)
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- **Optical Atomic Emission**
  - Flame (Na only)
  - Plasma (Na, Cl? & other elements)
Optical Atomic Emission

**Flame Instruments**
- Flame Photometer (LPG flame)
- Atomic Absorption (AA) in emission mode (air-acetylene flame)

**Plasma Instruments**
- ICP (Inductively Coupled Plasma) (ICP-OES): Most popular
- DCP (Direct Current Plasma) and MIP (Microwave Induced Plasma) (both possibly obsolete)
Advantages: Optical Atomic Emission techniques

- **Flame Instruments**: Low cost instrument and running costs (LPG or acetylene)
- **AA**: Can also determine wide variety of other metal elements (separately)
- **ICP**: Can determine Na plus most other elements together

Highly selective: very little spectral interference (none for Na)
Disadvantages: Optical Atomic Emission techniques

- **All:** Need to dissolve analytes: Extraction
  Possible problems with high levels of sugars, fats etc. Digestion or ashing to destroy

- **Flame Instruments:** Limited to metal elements

- **ICP:** Instruments expensive
  High running costs (high argon consumption)
Techniques Cl⁻ only

- Titration with silver nitrate (AgNO₃)
- Ion Chromatography (IC)
- Ion Selective Electrode (ISE)
- Colourimetric methods
Silver Nitrate Titration

Precipitation titration (AgCl precipitates)

- No indicator
- Potassium chromate indicator (Mohr’s method)
- Back titration with potassium thiocyanate, Fe$^{3+}$ indicator (Volhard's method)
- Dichlorofluorescein indicator (Fajans method)
- Potentiometric titration
Advantages of Silver Nitrate titrations

**Potentiometric:**
- Can be automated

**All:**
- Low Cost
- High repeatability if sharp end-point
Disadvantages of Silver Nitrate titrations:

- Can see other halides (bromide and iodide) as chloride
- Can’t see if Cl\textsuperscript{-} is from NaCl or other chlorides
Techniques Cl\textsuperscript{-} only

- Titration with silver nitrate (AgNO\textsubscript{3})
- **Ion Chromatography (IC)**
- Ion Selective Electrode (ISE)
- Colourimetric methods
Advantages of Ion Chromatography

- Very selective, negligible interference
- Very sensitive – can determine low concentrations
- Can also determine other anions at same time
Disadvantages of Ion Chromatography

- Instruments expensive
- Low sample throughput
- Can’t use acids to extract samples
- Problems with high fat content in the samples
- Can’t see if Cl\(^-\) is from NaCl or other chlorides
Techniques Cl⁻ only

- Titration with silver nitrate (AgNO₃)
- Ion Chromatography (IC)
- Ion Selective Electrode (ISE)
- Colourimetric methods
Advantages of ISE & Colourimetric methods

- Both: Relatively low cost
- Fairly high sample throughput
- Colourimetric: may be automated (flow systems)
- ISE: Relatively simple operation
Disadvantages of ISE & Colourimetric methods

- Both: Can’t see if Cl\(^{-}\) is from NaCl or other chlorides
- Susceptible to interferences
- Colourimetric: Inaccurate if solution is coloured
References


3. **CHLORIDE – DISSOLVED mg/L Cl - Silver Nitrate Potentiometric**

4. **New colorimetric determination of chloride using mercuric thiocyanate and ferric ion**
   I Iwasaki, S Utsumi... - Bulletin of the Chemical Society of ..., 1952 - Journal@rchiv
Periodic Table