

Application Note AN-H-127

Thermometric analysis of aluminum by back-titration

Fast and robust technique for aluminum determination

A thermometric complexometric titration procedure has been adapted to the determination of aluminum in solutions, where the direct titration with fluoride is not practicable because of the interference of silica (e.g., from digestion of clays, zeolites, or other alumino-silicate-containing substances).

The new method involves the use of a thermometric indicator (hydrogen peroxide) to give a sharp temperature change at the endpoint. When all the excess EDTA has reacted with the copper(II) titrant, the first trace of free Cu^{2+} ions causes the H_2O_2 to decompose very rapidly, causing a sudden increase in the temperature of the solution. The heat of reaction ΔH_f for $H_2O_2 \rightarrow H_2O + [O]$ is approximately -98 kJ/mol, or twice the heat created during the reaction of a strong acid with a strong base. This makes the technique very robust.

Additionally, thermometric titrations have very short titration durations as the titrant is added continuously while monitoring the temperature. Results are usually obtained within 2–3 minutes.

SAMPLE AND SAMPLE PREPARATION

Aluminum sulfate and potassium alum salts are used as samples. To an Erlenmeyer flask containing the aluminum salts, EDTA solution in excess and ammonia solution is added. Then the obtained solution is stirred for five minutes while boiling to facilitate the complexation reaction between aluminum and EDTA.

EXPERIMENTAL

After cooling down to room temperature, an aliquot of the solution is used for titration. Ammonia buffer and hydrogen peroxide are added subsequently. The excess of EDTA is back titrated with Cu^{2+} solution.
The thermometric titration is carried out automatically with the OMNIS software in combination with an OMNIS Titrator and a dThermoprobe.



Figure 1. OMNIS Titrator equipped with a dThermoprobe. Example setup for the analysis of aluminum.

RESULTS

The analysis of aluminum is very reproducible. Relative standard deviations <0.3% are obtained with this method.

Table 1. Results of the aluminum determination in aluminum sulfate (Al2(SO4)3 · 16 H2O) and potassium alum (AlK(SO4)2 · 12 H2O).

	Assay Al in $Al_2(SO_4)_3 \cdot 16\ H_2O$ / %	Assay Al in $AlK(SO_4)_2 \cdot 12\ H_2O$ / %
n =	8	10
Mean	7.87	5.11
SD(abs)	0.02	0.01
SD(rel)	0.25	0.20

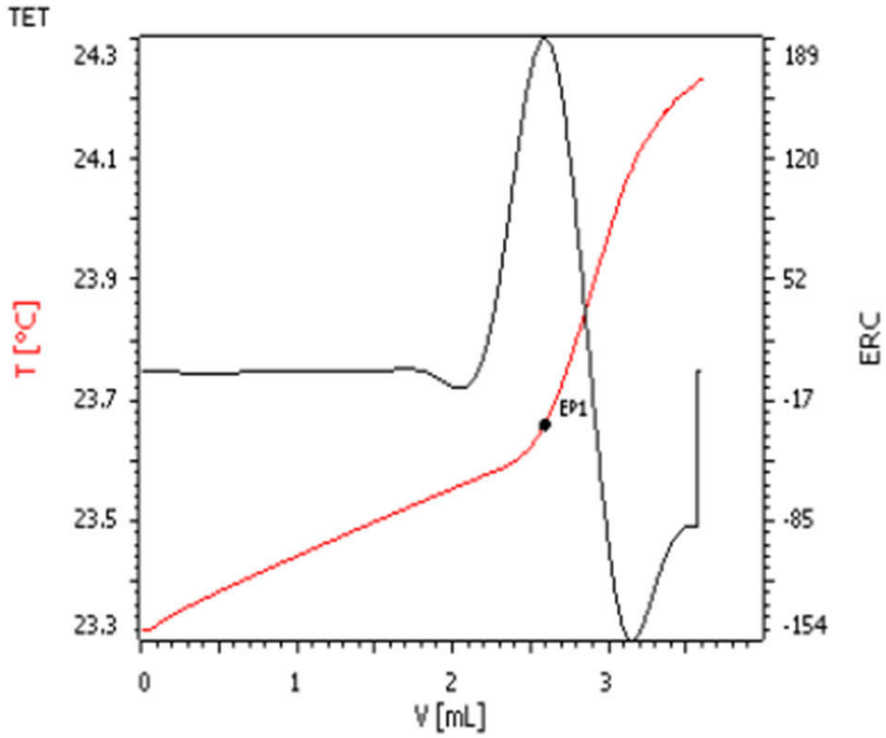


Figure 2. Example curve of thermometric determination of aluminum in aluminum sulfate.

CONCLUSION

This application shows a fast alternative method to the potentiometric titration of aluminum which also can be used in the presence of silicates.
Thermometric titration is a very fast and maintenance-free technique, which leads to reliable and precise results. The addition of peroxide enhances the reaction enthalpy and therefore additionally increases the reproducibility.

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CONFIGURATION



OMNIS Titrator with magnetic stirrer, without function license

Innovative, modular potentiometric OMNIS Titrator for stand-alone operation or as the core of an OMNIS titration system. Thanks to 3S Liquid Adapter technology, handling chemicals is more secure than ever before. The titrator can be freely configured with measuring modules and cylinder units and can have a stirrer added as needed. Thanks to various software function licenses, various measuring modes and functionalities are possible.

- Control via PC or local network
- Connection option for up to four additional titration or dosing modules for additional applications or auxiliary solutions
- Connection option for one rod stirrer
- Various cylinder sizes available: 5, 10, 20 or 50 mL
- Liquid Adapter with 3S technology: Secure handling of chemicals, automatic transfer of the original reagent data of the manufacturer

Measuring modes and software options:

- Endpoint titration: "Basic" function license
- Endpoint and equivalence point titration (monotonic/dynamic): "Advanced" function license
- Endpoint and equivalence point titration (monotonic/dynamic) with parallel titration: "Professional" function license



dThermoprobe

High-sensitivity digital temperature sensor for thermometric titration with OMNIS.

The Thermoprobe has a short response time and a high resolution, which enables precise recording of even the smallest temperature changes.

This sensor can be used in aqueous and nonaqueous solutions which do not contain any HF, for determinations such as:

- Acid number (TAN) in accordance with ASTM D8045
- Total base number (TBN)
- Free fatty acids
- Ca/Mg determination
- Phosphate



Cylinder unit OMNIS special, 10 mL

Intelligent 10 mL cylinder unit for one OMNIS Titrator, Titration Module or Dosing Module. This cylinder unit is especially recommended for the following solutions:

- Aqueous alkaline solutions
- Titrant 5
- Silver nitrate solutions
- Nonaqueous alkaline solutions
- Permanganate solutions
- EDTA solutions

Includes dosing tubing and antidiffusion tip.

OMNIS

A WHOLE NEW LEVEL OF PERFORMANCE

Function license Thermometric Titrator

Function license "Thermometric Titrator" for the OMNIS Titrator

Contains the function modes

- Thermometric Titration (TET)
- MEAS U/T/pH
- Liquid Handling
- Titration only with internal buret of an OMNIS Titrator