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Fully automated determination

Acid copper baths are mainly used for the copper deposition on semiconductor wafers. Small amounts of chloride increase the speed of deposition and reduce anode polarization. However, higher concentrations are undesired, as this will decrease the quality of the copper deposition. Therefore, it is quite important to monitor the amount of chloride to have an effective, yet high-quality copper deposition process.

In this Application Note, a fully automated solution based on titration is presented. In comparison to ion chromatography, titration offers the benefit that no dilution of the sample is necessary, and the hardware is comparatively low-priced. Furthermore, the fully automated solution allows users to minimize handling errors, to reduce workloads, and to guarantee outstanding reproducibility.

SAMPLE AND SAMPLE PREPARATION

The method is demonstrated for an acid copper bath. No specific sample preparation is required.

Ω Metrohm

EXPERIMENTAL

This analysis is carried out on an automated system consisting of an 814 Sample Processor and a 905 Titrando equipped with an iAg-Titrode with Ag_2S coating.

To a reasonable amount of sample, 5 mL of nitric acid is added to acidify the sample. Then, deionized water is added to cover the glass membrane and silver ring of the electrode, and the sample is titrated with standardized silver nitrate titrant until after the equivalence point.

Figure 1. 814 Sample Processor and 905 Titrando equipped with an iAg-Titrode with Ag2S coating controlled by tiamo software.

RESULTS

The analysis demonstrates an acceptable result and well-defined titration curves. The sample analyzed contained 49.17 mg/L chloride with a relative standard deviation of 0.31% (n = 10). An example titration curve is displayed in **Figure 2**.
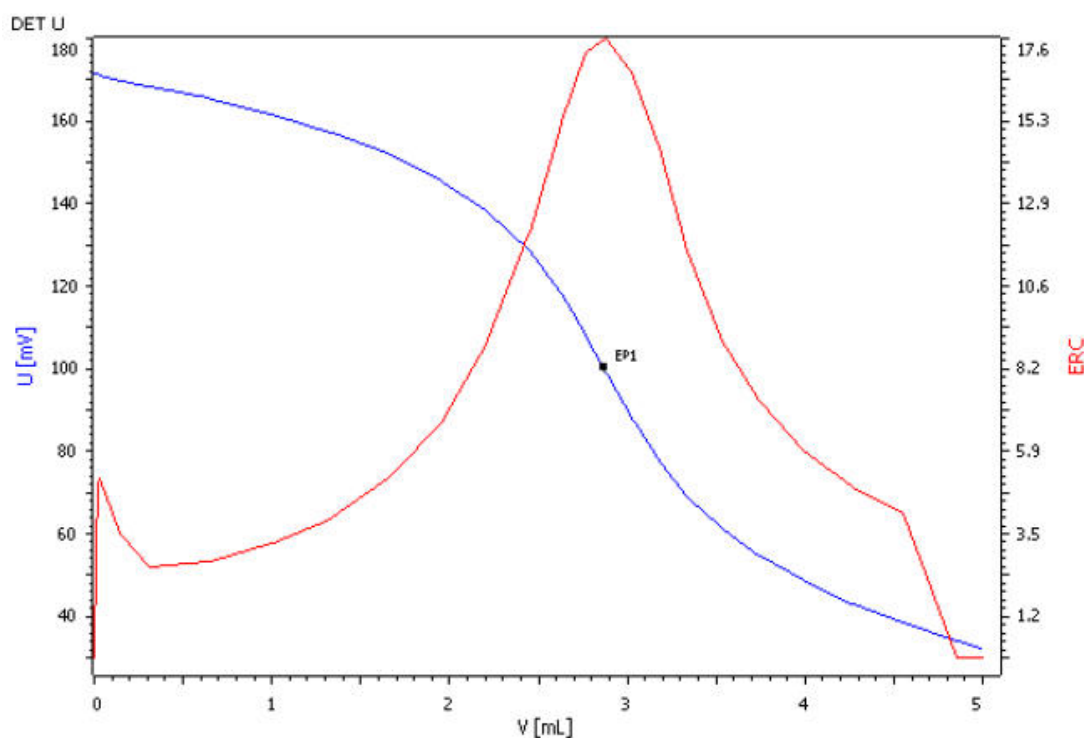

Figure 2. Example titration curve of the chloride determination in an acid copper bath.

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CONCLUSION

Titration is a precise and reliable method to determine the chloride content in acid copper baths. Using the 814 Sample Processor allows a fully automated determination, freeing up valuable time of the operator and thus increasing the productivity in the lab. Furthermore, by fully automating the analysis, the reproducibility can be increased and sample analysis failures due to improper handling can be reduced.

Internal reference: AW TI CH1-1130-022013

CONTACT

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CONFIGURATION

905 Titrando

Dosino
- 800 Dosino
- (DET)(MET)(SET)
- (MEAS CONC)
- ,LQH
- MSB
- "iTrode"
- USB
- OMNIS-Software*tiamo*- Touch Control
- , GMP/GLP FDA , 21 CFR 11

814 USB Sample Processor (1T/0P)

USB Sample Processor ,() LQH
, Swing Head
Touch Control "stand alone" PC : tiamoTM MagIC Net viva OMNIS

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Sample Processor 843 Pump Station– rinse/aspirate

843 Pump Station(), Sample Processor / (rinse/aspirate) 814 815 Sample Processor

iAg Titrode Ag2S

pH ,

(Ag$_2$S) ,

pH (),:

-
-
-
-
-

iTrodes TitrandoTi-Touch 913/914 Metern

Metrohm