

Application Note AN-T-100

# Chloride in acidic copper baths

## 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.

## EXPERIMENTAL

This analysis is carried out on an automated system consisting of an 814 Sample Processor and a 905 Titrand equipped with an iAg-Titrode with  $\text{Ag}_2\text{S}$  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.

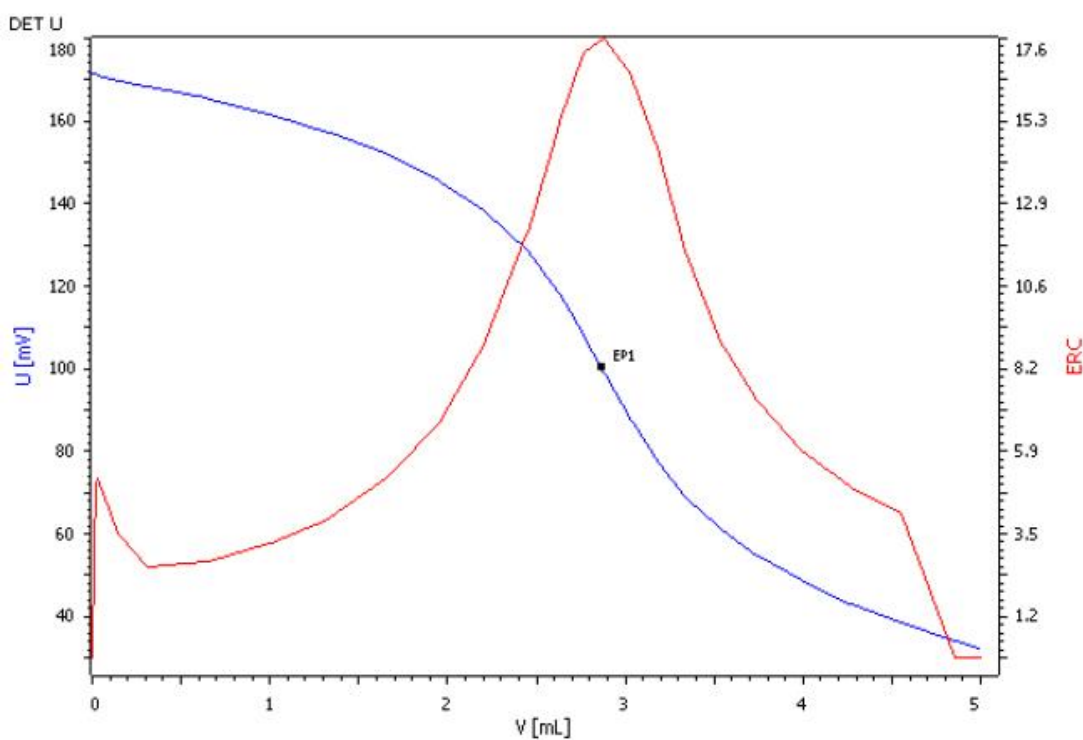


**Figure 1.** 814 Sample Processor and 905 Titrand equipped with an iAg-Titrode with  $\text{Ag}_2\text{S}$  coating controlled by tiamo software.

## 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.

## 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

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## CONFIGURATION



### 905 Titrando

用于使用 Dosino 加液系统一个量接口位分析滴定的高端滴定。

- 多四套 800 Dosino 加液系
- 等当点滴定(DET)、等量等当点滴定(MET)和点定滴定(SET)
- 使用子性量(MEAS CONC)
- 控的加液功能,LQH
- 用于外拌器或加液器系的四个 MSB 接口
- 智能“iTrode”
- USB 接口
- 使用 OMNIS-Software、*tiamo*-件或 Touch Control
- 如果需要,足 GMP/GLP 和 FDA 要求,比如 21 CFR 第 11 部分



### 814 USB Sample Processor (1T/0P)

USB Sample Processor 包括一个工作站,可用于自理少量至中等量的常品系列。可再接多台(隔膜或蠕)和三台加液器用来行 LQH 加液理。

由于其用范很广,因此必根据具体用来合的品、拌器、滴定和 Swing Head 以及品容器并独。

通 Touch Control 通 "stand alone" 控制。有以下 PC 控制用件品可供: 滴定件 tiamo™、色分析件 MagIC Net、伏安法件 viva 或 OMNIS。



### Sample Processor 843 Pump Station–rinse/aspirate

843 Pump Station(蠕式)个内置的蠕,可直接由 Sample Processor 自器通命令来控制。冲洗/抽吸 (rinse/aspirate)型中包含了用于自清空滴定杯和清洗滴定装的完整配件。此附件用于与 814 和 815 Sample Processor 一起使用。



### iAg Titrode Ag<sub>2</sub>S

pH 玻璃膜且集成了感器数据存芯片的智能合式形,用作参比。

具有硫化物 (Ag<sub>2</sub>S),其用于高的敏度和更好的指示限。

免用于 pH 恒定的滴定(硝酸滴定),例如:

- 化物、化物、化物
- 硫化物
- 硫化
- 硫醇
- 化物

存放在蒸水中。

iTodes 可以在 Titrande、Ti-Touch 或 913/914 Metern 上使用。