

Technical Notes C001

"Trouble shooting when anodic solution becomes brown before end-point or dark brown at end-point in coulometric titration"

December 2004

Purpose of Operation:

To shorten an analysis time and/or gain more accurate results by cleaning an electrode tip when dehydration is not completed although Iodine exists sufficiently in excess or dehydration requires a longer time.

Apparatus:

Coulometric Moisture Meter CA-100 (Mitsubishi Chemical Corporation)
Aquamicron AKX, Aquamicron CXU (API Corporation)
Aquamicron Check Solution P (API Corporation)
Micro Syringe (100ul)
Paper Wiper
Balance
Self Protective Equipment

Measurement Condition:

Parameters of Moisture Meter: Normal (As default condition)

Procedures:

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Operation			Remarks
	1)	Electrolyte solutions (Aquamicron AKX and CXU) are charged into the anode and cathode chambers of an electrolysis cell. Then press the titration key to start electrolysis.	
	2)	Even when over Titration (Brown color) is observed, dehydration has not been completed.	
	3)	Press the titration key to stop dehydration. Then pull the detection electrode off the cell.	
	4)	Clean the two platinum electrodes carefully with paper wiper.	Do not polish electrodes with any cleanser.

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- 5) Insert the detection electrode again. When a displayed voltage shows a negative value, inject 100ul of Aquamicron Check Solution P in several times to gain some positive voltage.
- 6) Press the titration key, and the titration for dehydration will come to end in a smooth manner.

2-3ul of distilled water will give the same result.

Note:

Aquamicron AKX (anode solution), used for the analysis of ketones, becomes brown before end-point although Iodine exists sufficiently in excess, probably due to detection electrode not monitoring proper detection voltage because of undetected stain on the electrode tip.

In this case, there is no need to rinse out by inorganic acid or to polish the electrode with cleanser.