

Technical Notes K001

"Trouble shooting when solvent becomes brown before end-point or dark brown at end-point in volumetric titration"

December 2004





Purpose of Operation:

To shorten an analytical 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:

Volumetric Moisture Meter KF-100 (Mitsubishi Chemical Corporation) Aquamicron Titrant SS-Z 3mg, Aquamicron Solvent KTX (API Corporation) Micro Syringe (10ul)

Paper Wiper

Balance

Self-protective Equipment

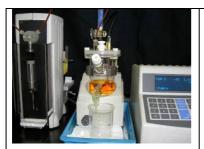
Measurement Condition:

Parameters of Moisture Meter: End Sense 140mV (Others are default setting)

Procedures:

| Operation | | Remarks |
|-----------|---|---------|
| | Aquamicron Solvent KTX is charged into a flask and press the titration key to start dehydration with Aquamicron Titrant SS-Z 3mg. | |
| | 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. | - |

AQUAMICRON Technical Notes K001



- 5) Insert the detection electrode again. When a displayed voltage shows a negative value, inject 2-3ul of distilled water 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.
- 7) If dehydration fails again, change the end-sense voltage to 450mV and repeat the process of 3) to 6).

Refer to instrument's manual to change the parameter.

Note:

Aquamicron Solvent KTX, 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 and or to polish the electrode with cleanser.