京都電子工業株式会社 KYOTO ELECTRONICS MANUFACTURING CO, LTD.

Application Note Factor determination of potassium permanganate solution

Industry Instrument Measurement method Standards Inorganic chemical Automatic potentiometric titrator Potentiometric titration / Redox Titration JIS K8001-2017

1. Scope

The method for factor determination of a 0.02 mol/L potassium permanganate solution is stipulated in the JIS K 8001 General rules for test methods of reagents. This standard describes an example of the measurements for finding the factor determination of a 0.02 mol/L potassium permanganate solution. In this method, a 0.02 mol/L potassium permanganate solution is titrated using sodium oxalate, a volumetric analysis standard. The titration reaction is as follows.

 $2KMnO_4 + 5H_2C_2O_4 + 3H_2SO_4 \rightarrow 2MnSO_4 + K_2SO_4 + 10CO_2 + 8H_2O_2 + 2MnSO_4 + K_2SO_4 + 10CO_2 + 8H_2O_2 + 2MnSO_4 + K_2SO_4 + 10CO_2 + 2MnSO_4 + K_2SO_4 + 10CO_2 + 2MnSO_4 + K_2SO_4 + 10CO_2 + 2MnSO_4 + 2MnSO$

2. Precautions

Store the potassium permanganate solution in a dark place in a lightproof, airtight container.

3. Post-measurement procedure

After cleaning the electrode in pure water, store it immersed in pure water so that its liquid junction does not dry out.

| 4 A | | 1 |
|-----------------|------|------|
| $\Delta \Delta$ | ppar | atus |
| T . / \ | ppur | atuo |

| Main unit | Automatic potentiometric titrator (preamplifier : STD) |
|-----------|---|
| Electrode | Combined platinum electrode |
| | (Inner filling (outer) 3.3 mol/L potassium chloride solution) |

5. Reagents

| Titrant | 0.02 mol/L (0.1 N) potassium permanganate solution |
|--------------------|--|
| Standard Substance | Sodium oxalate* |
| Reagent Added | Sulfuric acid + ion exchanged water $(1 + 1)$ |

* Heat to 200 °C for 60 minutes to dry.

6. Procedure

- 1) Add 0.20 to 0.24 g of pretreated sodium oxalate to a tall, 300 ml beaker.
- 2) Add 200 mL of pure water, then add 20 mL of sulfuric acid (1 + 1) and heat to 70 °C.
- 3) While stirring the mixture, immediately inject a set volume (28 mL) of prepared 0.02 mol/L potassium permanganate.
- 4) Allow the reaction to continue while stirring for 300 seconds.
- 5) Next, titrate it with the potassium permanganate solution.
- 6) After measurement, immerse the electrode in water for 5 minutes. Wait for the potential to stabilize, and then proceed to the next measurement.

Separately, repeat steps 2) to 6) without sodium oxalate. Treating this titration volume as the blank test value, subtract it from the titration volume when the standard substance was measured.

In the test using potentiometric titration, heat the sample solution to 70 $^{\circ}$ C. Then inject 28 mL of the titrant at a fixed rate using constant volume dispensing mode.* Wait 300 seconds, and then resume titration.

In the procedure described here, steps 3) to 5) above are performed automatically in accordance with the parameter settings.

* Constant volume dispensing mode

In this mode, a specified volume of titrant is dispensed automatically at a fixed rate.

| 7. Calculation | | |
|--------------------------|---------------------------------------|--|
| Factor = SIZE \times R | \times K1 / (EP1 – BL1) \times C1 | |
| SIZE | Amount of sodium oxalate used (g) | |

| SIZ | ZE | Amount of sodium oxalate used (g) |
|-----|----|---|
| R | | Purity of the sodium oxalate $= 0.995$ |
| EP | 1 | Titration volume (mL) |
| BL | .1 | Titration volume for the blank test (mL) |
| C1 | | Concentration conversion coefficient = 6.70 mg/mL |
| K1 | | Unit conversion coefficient $= 1000$ |

8. Example

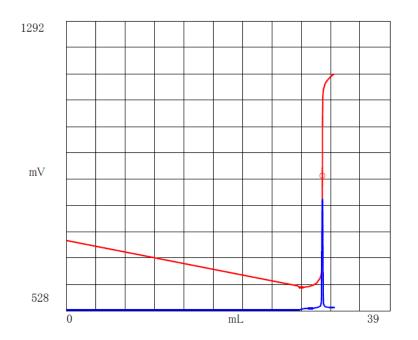
- Parameter -

| <titr. mode=""></titr.> | Auto Int. | <u><ctrl. para.=""></ctrl.></u> | |
|---------------------------------|-------------|---------------------------------|----------|
| <titr. form=""></titr.> | EP Stop | Number of EP | 1 |
| | | End Sense | Auto |
| <u><titr. para.=""></titr.></u> | | Gain | 1 |
| Burette No. | 1 | Data Sampling | Auto |
| Channel/Unit(Ctrl.) | Ch1, mV | Ctrl. Speed | Standard |
| pH Polarity | Standard | Other Control | Standard |
| Direction | Auto | Stirrer Speed | 4 |
| Wait Time | 300 (s) | Auto Int. Mode | Standard |
| Doze Mode | Volume Stop | | |
| Stop Volume | 28 (mL) | | |

(The above condition is an example. The setting condition depends on the model.)



- Example of Titration curve -



- Measurement results -

| Table 1 Measurement result | | | | |
|----------------------------|------------|----------------|--------------|--------|
| | Sample (g) | Titration (mL) | Time (h:m:s) | Factor |
| 1 | 0.2017 | 30.299 | 0:12:40 | 0.9886 |
| 2 | 0.2020 | 30.391 | 0:13:15 | 0.9871 |
| 3 | 0.2034 | 30.617 | 0:13:07 | 0.9866 |
| Mean | | | | 0.9874 |
| SD | | | | 0.0011 |
| RSD (%) | | | | 0.11 |

9. Summary

Only manual titration is stipulated in JIS K 8001. However, the use of potentiometric titration results in stable measurements free from variance due to differences between analysts.

10. References

JIS K 8001-2017 General rules for test methods of reagents

