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National Standard
of the People's Republic of China

GB/T 7600-2014

Replacing GB/T 7600-1987

Determination of water content in transformer oils and turbine oils in service by coulometric method

运行中变压器油和汽轮机油水分含量测定法（库仑法）

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Issued on: September 3, 2014

Implemented on: April 1, 2015

**Issued by: General Administration of Quality Supervision, Inspection
and Quarantine of the People's Republic of China;**

**Committee of Standardization Administration of the
People's Republic of China.**

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Foreword

This Standard was drafted according to the regulations specified in the GB/T 1.1-2009.

This Standard replaces GB/T 7600-1987 "Determination of water content in transformer oils in service by coulometric method". Compared with GB/T 7600-1987, the main changes are as follows:

- EXTEND the scope of this Standard by adding the item "turbine oils";
- ADD the item "reference standard";
- DELETE the items "preparation of Karl Fischer reagent" and "preparation of electrolyte" in the original Standard;
- REARRANGE the contents of the original Standard properly;
- ADD the item "cleaning of electrolytic cells" in the Appendix.

This Standard was proposed by China Electricity Council.

This Standard shall be under the jurisdiction of the National Technical Committee for Standardization of Electrochemistry (SAC/TC 322).

Drafting organizations of this Standard: Xi'an Thermal Power Research Institute Co., Ltd, and Anhui Electric Power Research Institute.

Main drafters of this Standard: Xiao Xiuyuan, Feng Liping, and Qi Jiong.

This Standard was first-time released in 1987. This is the first revision.

Determination of water content in transformer oils and turbine oils in service by coulometric method

1 Scope

This Standard specifies the method for determining the water content in transformer oils and turbine oils in service by coulometric method.

This Standard applies to the determination of water content in transformer oils and turbine oils in service. This method can be used as reference for determining the water content in phosphate ester fire-resistant oil.

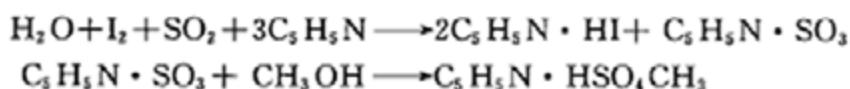
2 Normative references

The following documents are essential to the application of this document. For dated references, only the versions with the dates indicated are applicable to this document. For undated references, only the latest versions (including all the amendments) are applicable to this document.

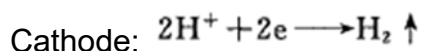
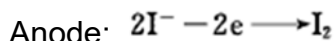
GB/T 7597 Method of sampling for transformer and turbine oils in electric power industry

3 Method and principle

The principle is as follows: The iodine is reduced by sulfur dioxide when there is water, and then generates hydroiodic pyridine and methyl hydrogen sulfate pyridine when there are pyridine and methanol. The equations are as follows:



In the process of electrolysis, the electrode reaction is as follows:



6 Determination steps

6.1 COLLECT oil samples according to the provisions specified in the GB/T 7597.

6.2 SEE the Appendix A for the cleaning of electrolytic cells.

6.3 DEBUG the instruments according to the instrument instructions.

6.4 TURN on the electromagnetic stirrer, so as to start electrolyzing the remaining water in the electrolytic cell. If there is excessive iodine in the electrolyte, USE a 0.5 μ L microsyringe to inject appropriate amount of pure water. The electrolyte color gradually fades at this moment. CONDUCT electrolysis when the electrolyte becomes yellow eventually.

6.5 When the electrolyte comes up to the titration end point, USE a syringe to extract a certain amount of standard sample with known water content, or USE a 0.5 μ L syringe to extract 0.5 μ L of pure water. PRESS down the start button. INJECT the extracted standard sample with known water content or pure water into the electrolytic cell from the upper injection port of the electrolytic cell for instrument calibration. The relative error between the display values of instruments and the standard values shall be $\pm 5\%$. The instruments can only be considered to be adjusted when three consecutive calibrations come up to the required values. If the calibration values go beyond the range, the electrolyte shall be replaced.

6.6 When it is difficult for the electrolyte to come up to the titration end point, TURN off the stirrer first, so as to stop the titration. PICK up the electrolytic cell afterwards. SHAKE for several times. START stirring and titrating again, so as to fully electrolyze the water on the surrounding wall. The above-mentioned steps can be repeated for several times, so as to come up to the end point as soon as possible. If it is still difficult to meet the requirements, the electrolyte shall be replaced prior to conducting the test according to the Article 6.5.

6.7 After levelling the instrument, USE the sample to be tested to flush the syringe for at least three times. EXTRACT 1mL of the sample to be tested afterwards (the injection volume can be adjusted according to the water content in the sample).

6.8 PRESS the start button, so as to inject the sample to be tested into the electrolytic cell from the upper injection port of the electrolytic cell. The instrument conducts automatic electrolysis until the sample to be tested comes up to the end point. NOTE down the display value. The same test shall be repeated at least twice. TAKE the mean value.

Appendix A

(Informative)

Cleaning of the electrolytic cell

A.1 Solvent cleaning

The electrolytic cell is made from glass, platinum, polytetrafluoroethylene, and other materials, which can resist strong acid and most of the solvent. When cleaning the electrolytic cell, it is best to use the solvent being compatible with the samples for analysis, such as acetone, methanol, etc.

FILL the solvent into the cathode chamber. USE a rubber stopper to seal the connector of the drying tube. SHAKE thoroughly to remove the precipitates inside (can be repeated). POUR all solvent into the anode chamber for flushing afterwards. PAY attention not to flush the electrode lead.

A.2 Nitric acid cleaning

If the solvent fails to clean the electrolytic cell, the nitric acid, water, and methanol shall be used successively for cleaning. In addition, USE a vacuum pump or other similar devices to extract the acid through the filter plate (can be repeated), until the holes on the filter plate become clean. The cleaning shall be conducted according to the following specific steps:

- a) TAKE out the cathode chamber first. USE water to flush it. ADD about 50mL of 75% nitric acid in the anode chamber. PUT the cathode chamber into the anode chamber slowly so that the filter plate is immersed in acid. CONNECT a vacuum pump onto the cathode chamber. EXTRACT sufficient amount of nitric acid through the filter plate so that the cathode is immersed in nitric acid. The precipitates start to be dissolved by nitric acid. The color of nitric acid becomes darker. After discarding the nitric acid, ADD about 50mL of 75% nitric acid in the anode chamber again. EXTRACT nitric acid through the filter plate (can be repeated), until the color of nitric acid no longer changes.
- b) If the electrolytic cell still cannot be cleaned according to the steps given in item a), it is necessary to heat the nitric acid before cleaning the electrolytic cell according to the steps given in item a).
- c) USE water to thoroughly clean the electrolytic cell. USE methanol to flush afterwards.

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