

Testing Method of LPG Residual Matter  
(Mass Analysis Method)

1. Scope

This Standard provides for the testing method of LPG residual matter.

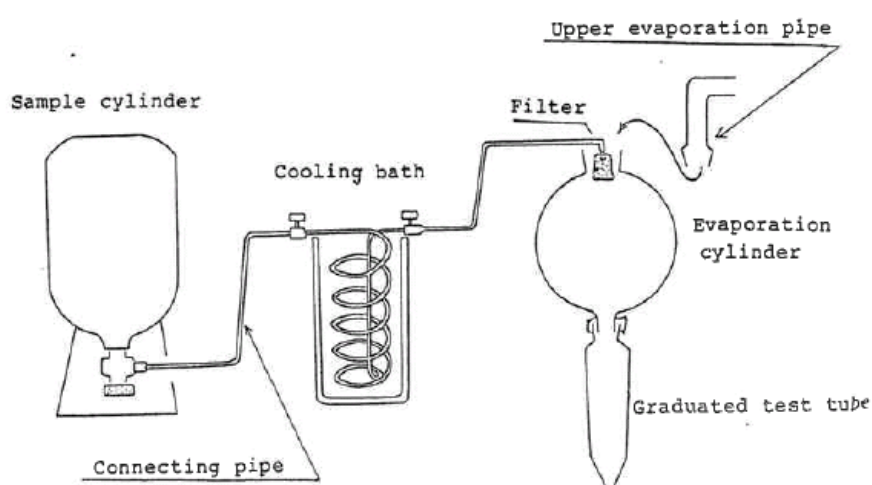
2. Summary of Method

After approximately 1 kg of liquid sample is evaporated in an atmosphere by using the prescribed testing apparatus, it is evaporated for 20 minutes in constant- temperature water bath at 75 °C and then the residual matter is weighed. It is evaporated for 30 minutes in oven at 105 °C, and then the residual matter is weighed, so as to obtain the residual matter at respective temperature.

3. Residual Matter Testing Equipment

The equipment consists of apparatus (1) through (6) described below, and its assembly drawing is shown in Fig. 1.

Fig. 1 Assembly Drawing of Residual Matter Testing Equipment



### (1) Sample Cylinder

This is of a construction shown in Fig.2. It is a cylinder made of stainless steel or aluminum alloy, withstanding a pressure of more than 31 kgf/cm<sup>2</sup> {3.04 MPa}, equipped with two or one nozzle opening(s) and capable of connecting a sample introduction pipe thereto. The cylinder valve should be equipped with a gas filling nozzle of a construction set forth in JTS B8245 (Valve for LPG Cylinder).

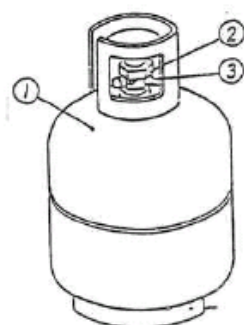


Fig. 2 Sample Cylinder (One Example)

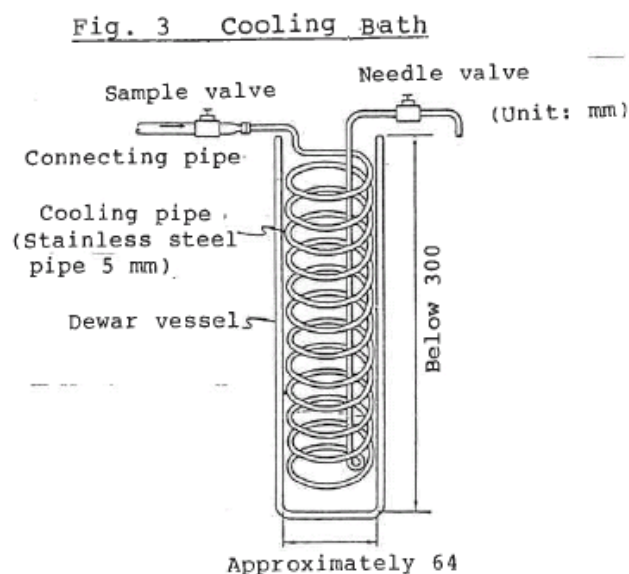
- ① Sample cylinder body
- ② Sample cylinder valve
- ③ Safety valve

### (2) Connecting Pipe

This is a pipe of approximately 5 mm in diameter, made of stainless steel or aluminum alloy, withstanding a pressure of more than 80 kgf/cm<sup>2</sup>. It is provided with a connector to the sample cylinder on one end and with a connector to the cooler on the other end.

### (3) Cooling bath

This is of a type set forth in Fig.3.



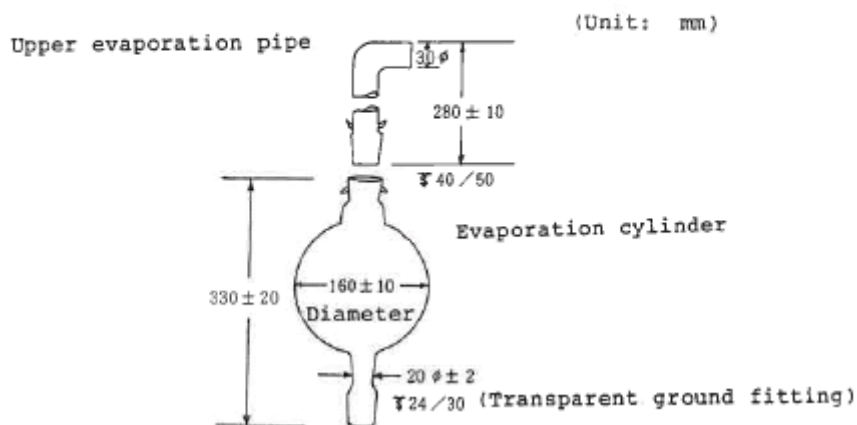
(4) Filter

Wire netting of stainless steel; 400 mesh.

(5) Evaporation Cylinder and Upper Evaporation Pipe

These are of glass with Class 1 hardness, and are of dimensions and a form shown in Fig.4.

Fig. 4 Evaporation Cylinder and Upper Evaporation Pipe

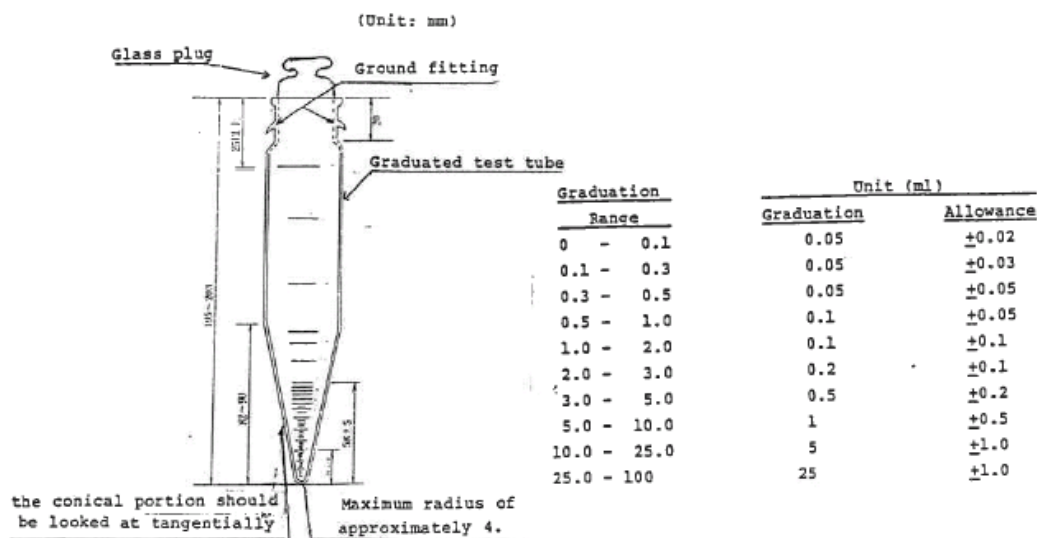


Remark: It is convenient to have a mark set at 2 liters in capacity.

(6) Graduated Test Tube (with Plug)

This is of glass with Class 1 hardness, and is of a construction shown Fig.5.

Fig. 5 Graduated Test Tube and Plug



#### 4. Constant-Temperature Water Bath and Others

##### (1) Constant-Temperature Water Bath

This shall be capable of immersing the graduated cylinder down to the graduation line of 100 ml, and also capable of holding the bath temperature at  $37.8 \pm 0.1$  °C and also capable of holding it at  $75 \pm 1$  °C.

##### (2) Platform Scale

It shall have graduation of 0.01 kg or less.

##### (3) Oven

It shall be capable of holding the temperature at  $105 \pm 2$  °C, and shall not be provided with an ignition source.

#### 5. Preparations for Testing

(1) Glass apparatus, etc. to be used in the testing shall be washed with cleaning solvents, such as acetone, etc, and water and foreign matters are removed therefrom.

(2) A piece of zeolite (one piece of ceramics, etc. of approximately 2 mm in diameter) is put into the graduated test tube and the tube is plugged. Then the tube is hold in the oven at 105 °C for 30 min. , and cooled in the decicator for another 30 min. , the mass is measured to the digit of 0.1 mg. often opening the plug once and closing it again.

(3) The evaporation cylinder and the graduated test tube are assembled in a manner as shown in Fig.1, and all of the ground glass joints are clamped with springs.

#### 6. Collection of Sample

(1) Dry ice and isopropanol are put into the cooling bath, so as to spiral tube.

(2) Sample cylinder, connecting pipe, cooler and filters are connected as shown in Fig.1. <sup>(1)</sup>

Then, the valves are opened in the order of the sample cylinder valve, sample valve and needle valve, and the connecting pipe, spiral tube and filter are co-washed with the liquid sample.

Note <sup>(1)</sup>: In making connection, all places which will contact with the sample shall be of stainless steel or aluminum alloy. When connections are made with rubber pipe, the connecting portions shall not be anointed with grease or lubricating oil.

- (3) Sample cylinder valve is closed, and then the connecting pipe is removed from the sample cylinder. The mass of sample cylinder is weighed the digit of 0.01 kg.
- (4) After the connecting pipe is attached to the sample cylinder, the sample cylinder valve is opened, and the sample is led into the evaporation cylinder in liquid state. At this time, the sample is supplied little by little until the evaporation cylinder cools down. Needle valve should be used in adjusting the flow of the sample.
- (5) When approximately 2 liters of liquid sample is led into the evaporation cylinder valve is closed, and the sample cylinder is left as it is for approximately one minute.
- (6) Then, the connecting pipe is disconnected from the sample cylinder, and mass of sample cylinder weigh to the digit of 0.01 kg. Then, the collected quantity of sample is calculated from the difference between this weight and the weight measured in item (3) above.

## 7. Preliminary Evaporation of Sample

- (1) Upper evaporation pipe is attached to the evaporation cylinder and is fastened with springs.
- (2) While cautioning that sudden boiling will not occur <sup>(2)</sup>, the evaporation cylinder is exposed to atmosphere thus the sample will be evaporated until it is reduced to approximately 5 ml.

Note <sup>(2)</sup>: Evaporation shall be carried out so that bubbles will come out uniformly from bottom of the graduated test tube. If bubbles are not generated uniformly, it is recommended that the upper evaporation pipe is removed and the tip of copper wire (approximately 2 mm in diameter and 500 – 550 mm in length) is polished with rough-faced file and inserted from the top of the evaporation cylinder. When evaporation speed is low, the graduated test tube shall be immersed in water bath of below 20 °C.

Remarks: When the evaporation work is conducted out-door, a place with good ventilation but without direct sunlight should be selected. Adequate consideration shall also be made for fire, explosion and offensive odor. When the work is conducted indoors, measures shall be taken to dispose Safety the evaporated gas from the upper evaporation pipe.

- (3) After the test tube is immersed in the constant-temperature water bath at 37.8 °C. Up to its shoulder for 20 minutes, the graduated test tube is taken out of the constant-temperature water bath.

#### 8. Measurement of Residual Matter at 75 °C

- (1) The graduated test tube is removed from the evaporation cylinder <sup>(3)</sup>, and the graduated test tube is immersed into the constant-temperature water bath at 75 °C down to the 100 ml graduation line. After being held there for accurately 20 minutes, air is blown into the same test tube for 10 seconds at 31. /minute <sup>(4)</sup>, and gas in the test tube is replaced by air.

Note <sup>(3)</sup>: Before removing the test tube, the water drops between the graduated test tube and the connecting portion of the evaporation cylinder shall be wiped off.

Note <sup>(4)</sup>: Methods of blowing the air into test tube shall be in accordance with Fig.6.

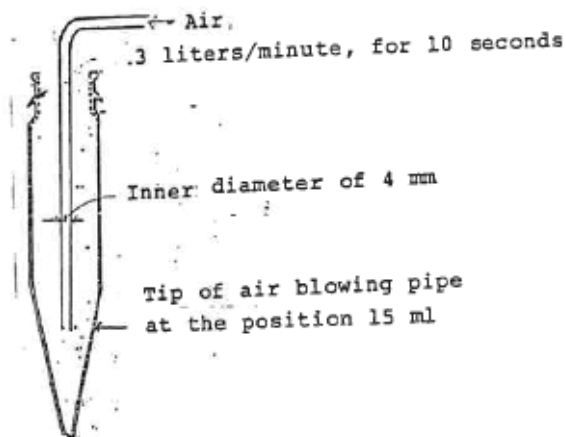


Fig. 6 Method of Blowing  
Air into Test Tube

- (2) The graduated test tube is taken out of constant-temperature water bath, and it is immediately plugged. Water adhered to the graduated test tube should be wiped off.

After being left cooling in the desiccator for 30 minutes, opening plug once and closing it again in order to prevent vacume situation, the graduated test tube is weighed to digit of 0.1 mg.

#### 9. Measurement of Residual Matter at 105 °C

- (1) Upon completing the operation in Section 8, the plug on the graduated test tube is removed, and the graduated test tube is horizontally placed in the oven at 105 °C for 30 minutes, and then air is blown into the graduated test tube for 10 seconds at a rate of 3 l./minutes (3), and the gas in the test tube is replaced by air.

- (2) The graduated test tube is taken out of the oven. After being left cooling in the desiccator for 30 minutes, the mass of the graduated test tube, together with the plug, is weighed to the digit of 0.1 mg.

#### 10. Calculation and Results

The residual matter is calculated in accordance with the following equation, and the result is rounded to an integer. Each of results will carry an additional remark of evaporation temperature.

$$S = \frac{(A-B) \times 10^3}{M}$$

Where S: Residual matter (mass in ppm)

A: Mass (g) of the graduated test tube (with plug)  
after evaporation at 75 °C or 105 °C.

B: Mass (g) of the graduated test tube (with plug).

M: Collected amounts of the sample (kg).

Remarks: When foreign matters, such as rust, etc. are found at the inspection of the filter, such effects should be recorded.



## Attachment 3

### Precision for Residual Matter

#### 1. Repeatability

Duplicate results by the same operator should be considered suspects if they differ by more than amounts shown in Fig.1. and 2 for repeatability.

#### 2. Reproducibility

The results of two laboratories should be considered suspect if the two results differ by more than the amounts shown in Fig.1. and 2 for reproducibility.

Fig . 1 Precision For Residual Matter 75 °C

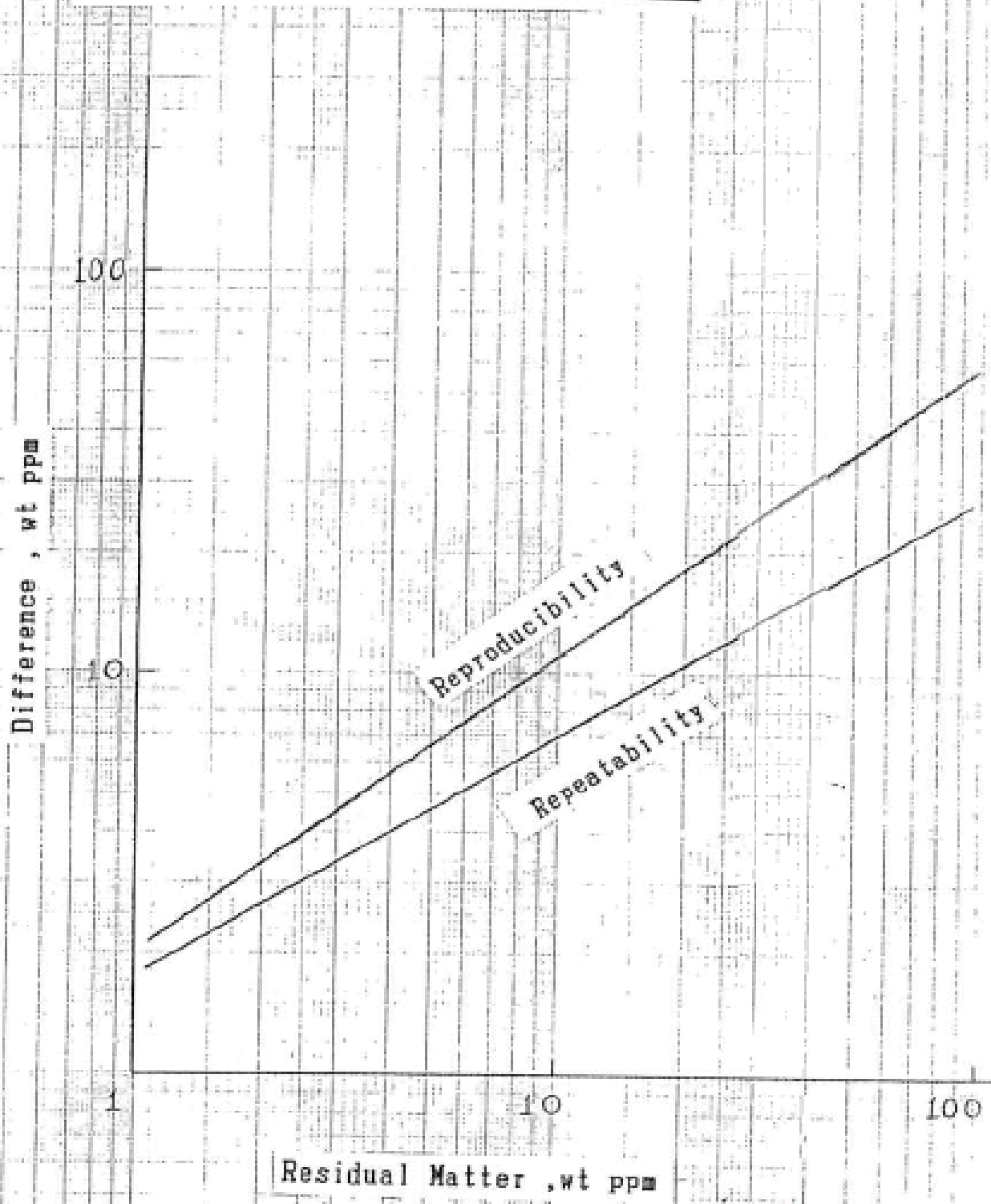


Fig . 2 Precision For Residual Matter 105 °C

