



# Study of Calibration System for Liquefied Natural Gas (LNG) Dispenser Verification Device Based on Quality Method

Huancheng Yang, Jiamin Zhao, Hongyan Geng, Xiaocheng Xu, Min Zhang

*Inner Mongolia Institute of Metrology and Testing, Hohhot, Inner Mongolia, China*

*Inner Mongolia Institute of Metrology and Testing, Hohhot, Inner Mongolia, China*

*Baotou Testing Centre, Baotou, Inner Mongolia, China*

*Inner Mongolia Institute of Metrology and Testing, Hohhot, Inner Mongolia, China*

*Inner Mongolia Institute of Metrology and Testing, Hohhot, Inner Mongolia, China*

*nmg\_cfxz@163.com*

Abstracts: To fulfil calibrating of LNG dispenser verification device, the traceability system of the device was researched in this paper. Using a combination of dynamic and static quality measurement system, with high accuracy electronic balance as main standard, ultra-low temperature commutator with uncertainty of better than  $1.5 \times 10^{-4}$  was developed. The device could realize automation during the whole procedure of data collection under the specified flow, the calibration of LNG dispenser verification device, processing the data and generating the records and certificates.

Key words: verification device, calibration, the main standard device, commutator

## 1. Introduction

The Liquefied Natural Gas (LNG) dispenser verification device is metrological standard equipment for quantity traceability of Liquefied Natural Gas (LNG) dispenser. Liquid temperature of LNG is about  $-163^{\circ}$  in normal atmosphere, however, its critical temperature between gas and liquid in normal atmosphere is  $-162^{\circ}$ , consequently, its easily gasify when filling liquid, begins to have an impact on measurement accuracy of flowmeter. As metrological standard equipment for quantity traceability of Liquefied Natural Gas (LNG) dispenser, the accuracy and consistency of verification device is sincerely important. The program is mainly study the calibration system of verification device, its calibration and build (improve) detection ability for quantity traceability system of verification device. It can

not only calibrate with actual flow or liquid nitrogen, but also getting reference value of superior traceability from comparison between laboratories. This quantity traceability system is of a great deal of importance in LNG metrological verification, insuring fair trade and driving LNG industry improving.

## 2. Review

The Verification Device is mainly consist of liquid-phase flowmeter, gas-phase flowmeter and piping. When certifying, dispenser filling LNG to cylinder. Liquid LNG measured by liquid-phase flowmeter, gaseous LNG in cylinder measured by gas-phase flowmeter, then returned. After software processing, subtract the accumulative amount of the liquid-phase flowmeter and gas-phase flowmeter, meet as the final amount.

The verification device is calibrated with actual flow or liquid nitrogen. Combine with dynamic and static methods, it regards industrial electronic balance with high accuracy as the main instrument, pressure transmitter and temperature transmitter as the supporting equipment. The device is consist of weighing system, liquid-supplying system, reversing system, controlling system, data acquisition system and medium recycling system (actual flow calibration) During calibrating, calibrated verification device is series with gas-filling dispenser or liquid nitrogen-outgoing device. Controlling system will regulate flow of supplying system, with reversing system putting medium in liquid-phase flowmeter, gaseous LNG in gas-phase flowmeter, then return to cylinders. After filling up, weighing system will weigh the LNG in reservior bottle and collect data. With medium recycled by recycling

system, calibration of the verification device completed.

### 3. Working principle and process

#### 3.1 Precool adiabatic cylinder with low temperature

Connecting the liquid-phase input, filling into reservior bottle. It will be looping-in with connecting the bottle’s interface and output terminal of medium. Turn the valve on, cycling medium in the loop, and then finish precooling of reservior bottle within allotted time.

#### 3.2 Circulating process

(1) Install the calibrated-device on the platform. Checking if it be sealed firmly, and then turning on the power to preheating.

(2) Set the calibration system to precooling mode, observing it’s state. According to the default parameter values, the system will judge if it be completed by itself. Flow direction of the medium in precooling mode as chart 1:

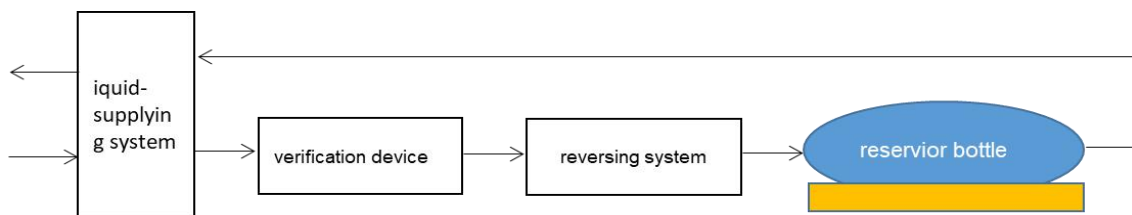


Chart1 Flow direction of the medium in precooling mode

As the device is mainly consisted by flowmeters, it must precool the flowmeters before calibration to meet the requirement of flowmeter calibration at low temperatures. During precooling, system will judge if the process is completed according to the gain amount of flowmeters, and then begins calibration.

### 3.3 Calibration process

After circulating process, weighing test the

liquid-phase flowmeter in verification device with this system. Set the system to calibration mode, at this time, flow direction of the medium as chart 2. Medium enter into reservior bottle through verification device and reversing system. After liquid-filling to specified weight, disconnect the device, separated the cylinder and piping. After weighing, empty the medium and recycle reservior bottle. The second calibration can’t be carried out until the pressure reduced to 0.1MPa.

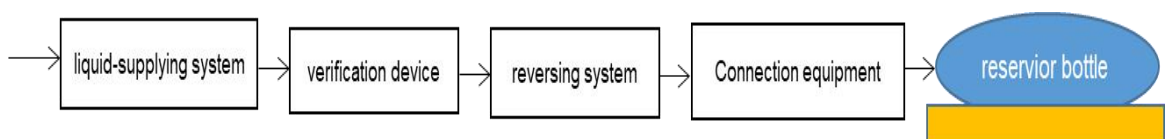


Chart2 Flow direction of the medium in calibration mode

After testing the liquid-phase flowmeter, calibrate the whole verification device. Connecting as chart 3.

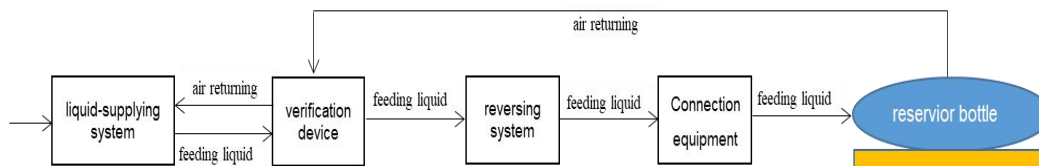


Chart 3 Connection in calibrating the whole verification device

Connects gas-phase air-returning-gun holder of the device and air-returning port of liquid-supplying system. While feeding liquid, recycling residual air to liquid-storage system. Comparing the cumulant of verification device with the value of electronic balance in weighing system, calibrate the whole verification device.

Before aerating, it should be purge the surface of liquid-feeding-gun, liquid-feeding-port, air-returning-gun and air-returning-port, on which easily frosting with compressed air. When calibration finished, take off the liquid-feeding-gun and air-returning-gun immediately. Disconnect the reservoir bottle and weighting. Calibration process as chart 4.

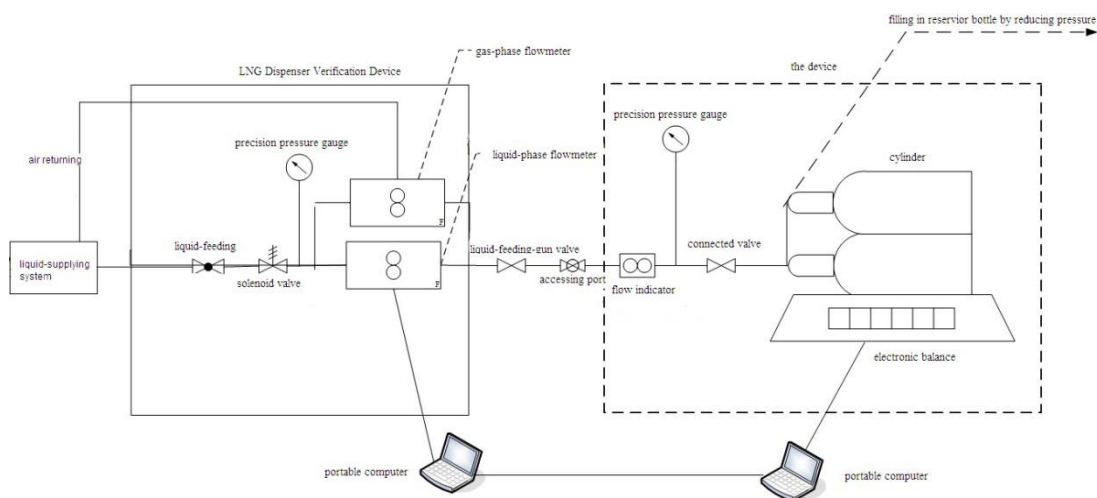


Chart 4 Calibration process

4. Technical indicators

- (1) Extended uncertainty: 0.1%(k=2);
- (2) Measuring range: (0 ~ 80)kg/min;
- (3) Medium: LNG、 liquid nitrogen;
- (4) Rated pressure: 1.2MPa
- (5) Maximal pressure: 1.6MPa

- (6) Temperature: -25°C ~ +55°C
- (7) Voltage:220V
- (8) Calibration port:20kg/min ≤ q<sub>1</sub> ≤ 32kg/min; 32kg/min ≤ q<sub>2</sub> ≤ 80kg/min
- (9) Times: Not less than 6 times in per point
- (10) Maximal measuring range of

electronic balance: 600kg; precision:50g.

### 5. Controlling system

The system sets in above and below. A portable computer is above, with which independent calibration-software of LNG dispenser verification device, mainly responsible for data collecting and processing.

Siemens PLC is below, in which controlled the process of calibration system. TCP/IP between these two equipments is of communication, to ensure data transmission timely and reliable.

### 5.1 Principle and structure of controlling system

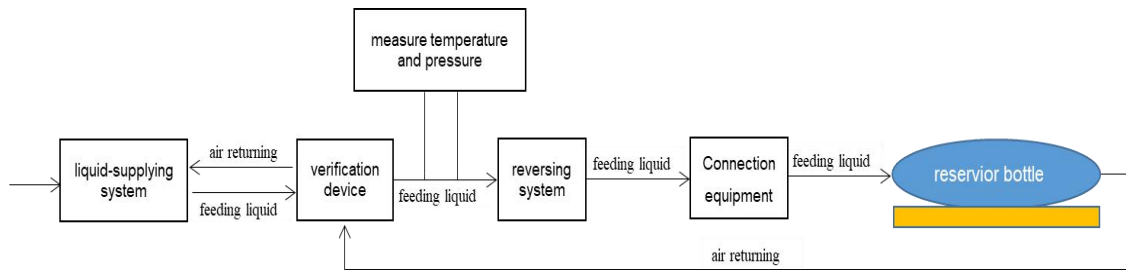


Chart 5 Principle of calibration process

With collecting data of equipment in real time, controlling system correct these data of flowmeters. And then, the commutator trigger cumulants of flowmeters during feeding the reservoir bottles. Thus the comparison can be achieved between data of LNG verification device and standard.

Software-processing of controlling system as chart 6.

### Chart 6 Software-processing

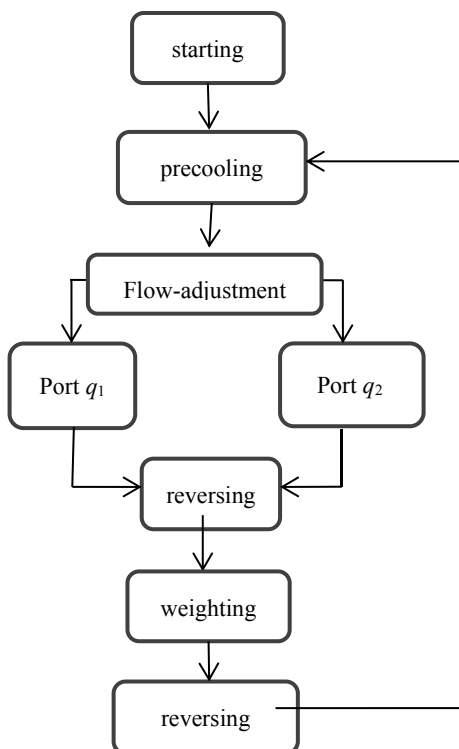
#### 5.2 Performance of controlling system

##### 5.2.1 Performance of data processing

Analog , the system is equipped with AD modules in 16 bits, with which used for collecting temperature and pressure, to ensure the accuracy of collecting analog signal. Thus reduct the raw temperature and pressure during testing.

Digital , the system is equipped with independent counting module in high speed. Frequency signals up to 10 Hz can be collected. At the same time, the fake interpolation method is used to process the low frequency signals. Restore the pulse signal during the measurement process accurately.

Communication , the system is equipped with RS485/232 interface. Also, it has unique communication software, with which collecting weighting of electronic balance and values in real time, to ensure the data of weighting stable and accurate.





### 5.2.2 Scaling performance

Extension interface is reserved .It can expand AD 、 AI 、 DO 、 DI and external communication equipment according to it's demands. At the same time, software designed in modular, if necessary, expanding the corresponding module, such as data processing, algorithm.

### 5.2.3 Security

System preset security plan. By monitoring weight, flow and pressure, anticipate possible risks. Choose necessary treatment according to the actual situation, such as tuning, alarming, crash-stop and so on, ensuring the whole system working in safe. Recording status and alarming as well, as a traceability basis for abnormal conditions.

### 5.3 Process-controlling system

The controlling method is in 3 steps, namely local control, remote control and automatic control.

Local control is manual, that is operating the equipment in panel. Usually, this method is mainly used in observing on site or the automatic system is out of order.

Remote control means the operator use the field equipment with the keyboard, the mouse or in the way of touching. For this way, operator can get the situation and data in real time, make adjustment based on changes. It's usually used when there is an experimental need.

Automatic control means a preset detection scheme. And then, the system will carry out the automatic work, including flow adjustment, process-controlling, data-recording. It is usually used in routine work where testing efficiency is required.

Appropriate mode of operation can be chosen. Local controlling method is with the highest priority. This is mainly in order to deal with the unexpected situations during testing, ensuring the safety of equipment and personnel.

### 5.4 Data-processing system

Data-processing system is used correcting the collected data and testing calculation.

Because of stress, air buoyancy and so on, data from testing can't be used as the final one. The system sends to computing module after correcting in it's unique method, ensuring data accuracy.

System processes data according to technical documents. It can getting errors, repeatability and so on and showing reports. Also, the data when you take a test can be store into database, for convenience of reviewing and tracing.

Testing-data can be classified and supervised if necessary. Users can query data in every procession until authorized. Then generate various reports using in analyzing and archiving.

## 6. Data-processing

### 6.1 Calibration error of verification device

(a)After gas-feeding , use formula(1) to calculate single measurement value relative error  $E_{ij}$  of calibrated dispenser verification device:

$$E_{ij} = \frac{(m_J)_{ij} - (m_B)_{ij}}{(m_B)_{ij}} \times 100\% \quad (1)$$

In the formula:

$(m_J)_{ij}$  —cumulative value of calibrated dispenser verification device in area  $i$  when take the  $J$  test, kg;

$(m_B)_{ij}$  —value of electronic balance in testing system in area  $i$  when take the  $j$  test, kg;

$E_{ij}$  —single relative error of value in area  $i$  when take the  $j$  test, %.

(b) After the whole 6 times testing completed in area  $i$ , use formula(2) to calculate the average of relative error combine with 6 times testing as the indication error  $E_i$  in this area.

$$E_i = \frac{\sum_{j=1}^n E_{ij}}{n} \quad (2)$$

In the formula:

$E_i$  —indication error in area  $i$ , %;

$n$  —times,  $n = 6$ .

(c) measurement repeatability  $(E_r)_i$  use formula(3) to calculate:

$$(E_r)_i = \sqrt{\frac{\sum_{j=1}^n (E_{ij} - E_i)^2}{n-1}} \times 100\% \quad (3)$$

In the formula:

$(E_r)_i$  —measurement repeatability in area  $i$ , %.

## 6.2 Experiment

6.2.1 The device weighing at port  $q_1$ , port  $q_2$ . Selecting 10 evenly distributed points within the usage( $j=1, 2, \dots, m, m \geq 10$ ). Adding  $F_2$ -level weight from  $j=1$  to  $j=m$  step by step, once calibration; then loading-off from  $j=m$  to

$j=1$  step by step, twice calibration. Repeat in  $n$  times( $n \geq 10$ ). Selected ports as follow: 20, 30, 40, 50, 60, 70, 80, 90, 100, 110kg, 5, 10, 15, 20, 25, 30, 35, 40, 45, 50kg, use formula(4) to calculate,  $(m_j+R_0)$  means D-value at  $j$  with time  $i$ .

$$\Delta m_i = R_{mi} - (m_j + R_0) \quad (4)$$

In the formula:

$m_j$  —weight of  $F_2$ -level at  $j$ , kg;

$R_{mi}$  —reading of weighting system when testing  $m_j$ - $F_2$ -level weight with time  $i$ , kg;

$R_0$  —average reading of weighting system for empty cylinder of 6 times testing, kg.

$$\text{average at } j \quad \Delta m = \frac{1}{n} \sum_{i=1}^n \Delta m_i \quad (5)$$

Relative standard uncertainty in A of a single testing at  $j$

$$s_j = \frac{1}{m_j + R_0} \left[ \frac{\sum_{i=1}^n (\Delta m_i - \Delta m)^2}{n-1} \right]^{1/2} \times 100\% \quad (6)$$

Relative standard uncertainty in B of a single testing at  $j$

$$u_j = \frac{\Delta m}{2(m_j + R_0)} \times 100\% \quad (7)$$

Take the maximum of relative standard uncertainty in A and B as calibration result.

$$s = (s_j)_{\max} \quad (8)$$



$R_0=$ 380.000 kg port $q_1$									
order	$m_j(\text{kg})$	$Rm_i(\text{kg})$	$\Delta m_i(\text{kg})$	$s_j(\%)$	$u_j(\%)$	$Rm_i(\text{kg})$	$\Delta m_i(\text{kg})$	$s_j(\%)$	$u_j(\%)$
1	20.000	400.010	0.010	0.001	0.001	400.000	0.000	0.001	0.001
2	30.000	410.000	0.000	0.001	0.001	410.010	0.010	0.001	0.001
3	40.000	420.010	0.010	0.001	0.001	420.000	0.000	0.001	0.001
4	50.000	430.000	0.000	0.001	0.001	430.000	0.000	0.001	0.001
5	60.000	440.010	0.010	0.001	0.001	440.000	0.000	0.001	0.001
6	70.000	450.000	0.000	0.001	0.001	450.010	0.010	0.001	0.001
7	80.000	460.000	0.000	0.001	0.001	460.000	0.000	0.001	0.001
8	90.000	470.010	0.010	0.001	0.001	470.010	0.010	0.001	0.001
9	100.000	480.000	0.000	0.001	0.001	480.010	0.010	0.001	0.001
10	110.000	490.010	0.010	0.001	0.001	490.010	0.010	0.001	0.001

$\Delta m=$	0.0050	kg				$\Delta m=$	0.0050	kg	
$s=$	$(s_j)_{\max}=$	0.00132	%			$s=$	$(s_j)_{\max}=$	0.00132	%
$u=$	$(u_j)_{\max}=$	0.00062	%			$u=$	$(u_j)_{\max}=$	0.00062	%

$R_0=$ 120.000 kg port $q_2$									
order	$m_j(\text{kg})$	$Rm_i(\text{kg})$	$\Delta m_i(\text{kg})$	$s_j(\%)$	$u_j(\%)$	$Rm_i(\text{kg})$	$\Delta m_i(\text{kg})$	$s_j(\%)$	$u_j(\%)$
1	5.000	125.000	0.000	0.003	0.000	125.000	0.000	0.003	0.000
2	10.000	130.000	0.000	0.003	0.000	129.995	-0.005	0.003	0.000
3	15.000	135.000	0.000	0.002	0.000	135.005	0.005	0.003	0.000
4	20.000	140.000	0.000	0.002	0.000	140.005	0.005	0.003	0.000
5	25.000	145.000	0.000	0.002	0.000	145.000	0.000	0.003	0.000
6	30.000	150.000	0.000	0.002	0.000	150.000	0.000	0.002	0.000
7	35.000	155.005	0.005	0.002	0.000	155.005	0.005	0.002	0.000
8	40.000	160.005	0.005	0.002	0.000	159.995	-0.005	0.002	0.000
9	45.000	164.995	-0.005	0.002	0.000	165.000	0.000	0.002	0.000
10	50.000	169.995	-0.005	0.002	0.000	170.000	0.000	0.002	0.000

$\Delta m=$	0.0000	kg				$\Delta m=$	0.0005	kg	
$s=$	$(s_j)_{\max}=$	0.00267	%			$s=$	$(s_j)_{\max}=$	0.00295	%
$u=$	$(u_j)_{\max}=$	0.00000	%			$u=$	$(u_j)_{\max}=$	0.00020	%

$u=(u_j)_{\max}$  (9) Results as table 1, parametric coordinate diagram as chart 7.

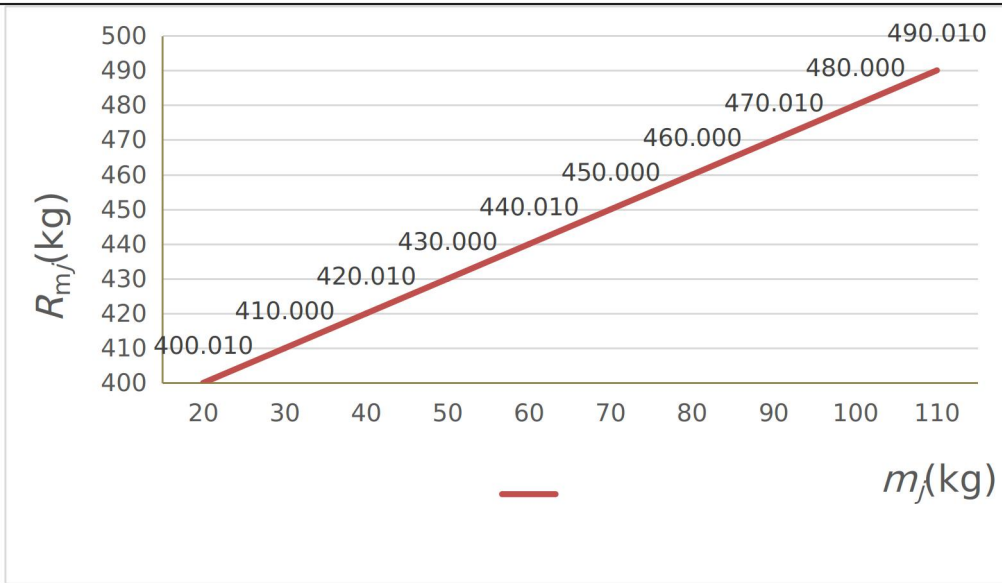


Table1 Calibration results of weighing system

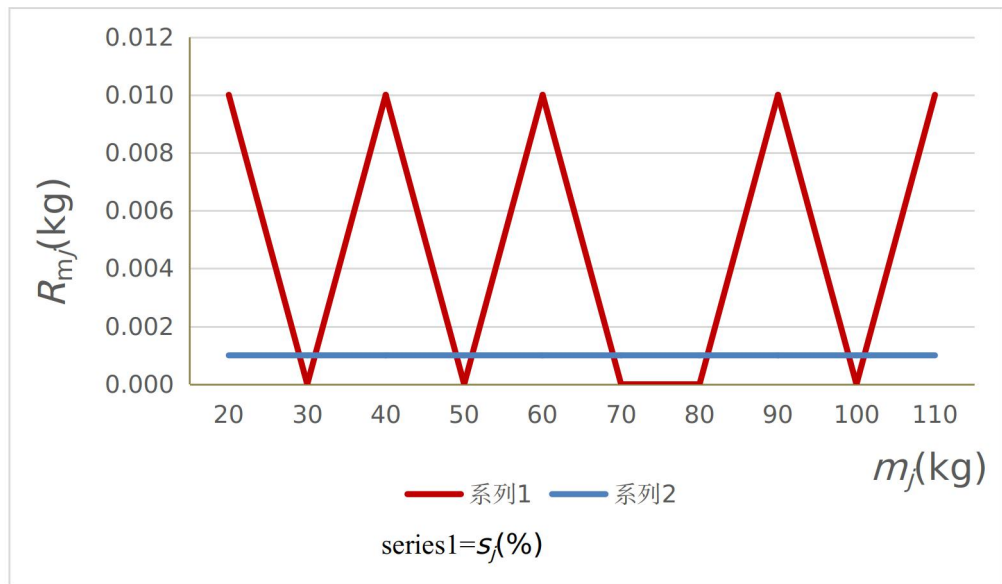


Chart7 Parametric coordinate diagram

Take the maximum as calibration result:  
 $s=2.67 \times 10^{-5}$ ,  $u=0.62 \times 10^{-5}$

uncertainty. Testing at port  $q_1$  and port  $q_2$  with route-subtracting, the minimum testing-time are  $t_{q1min}=200s$ ,  $t_{q2min}=120s$ .

6.2.2 Calibration of commutator

Results as table2 and table3.

time(ms) order $i$	1	2	3	4	5	6	7	8	9	10	average (ms)
$t_{q11i}$	20	20	21	22	23	24	21	20	20	21	21.2
$t_{q12i}$	22	23	23	23	22	20	20	26	28	25	23.2

Table2 Commutator measurement results of port  $q_1$





$t_{q11}$  standard deviation of a single experiment:

$$s_{tq11} = \sqrt{\frac{\sum_{i=1}^n (t_{q11} - \bar{t}_{q11})^2}{n-1}} = 1.40\text{ms} \quad (10)$$

relative standard uncertainty in A:

$$u_r(t_{q11}) = \frac{s_{tq11}}{1000t_{q1\min}} \times 100\% = 7.00 \times 10^{-6} \quad (11)$$

$t_{q12}$  standard deviation of a single experiment:

$$s_{tq12} = \sqrt{\frac{\sum_{i=1}^n (t_{q12} - \bar{t}_{q12})^2}{n-1}} = 2.53\text{ms} \quad (12)$$

relative standard uncertainty in A:

$$u_r(t_{q12}) = \frac{s_{tq12}}{1000t_{q1\min}} \times 100\% = 1.27 \times 10^{-5} \quad (13)$$

relative standard  $\left| \frac{t_{q11} - t_{q12}}{4t_{q1\min}} \right|$  uncertainty in B:

$$u_{rB}(t_{q1}) = 1/1000 \times \times 100\% = 2.50 \times 10^{-6} \quad (14)$$

composite relative standard uncertainty:

$$u_r(t_{q1}) = \sqrt{u_r^2(t_{q11}) + u_r^2(t_{q12}) + u_{rB}^2(t_{q1})} = 1.47 \times 10^{-5} \quad (15)$$

time(ms) order $i$	1	2	3	4	5	6	7	8	9	10	average (ms)
$t_{q11i}$	20	22	26	27	21	25	22	22	20	21	22.6
$t_{q12i}$	21	22	23	24	25	28	28	29	20	20	24.0

Table3 Commutator measurement results of port  $q_2$

$t_{q21}$  standard deviation of a single experiment:

$$s_{tq21} = \sqrt{\frac{\sum_{i=1}^n (t_{q21} - \bar{t}_{q21})^2}{n-1}} = 2.50\text{ms} \quad (16)$$

relative standard uncertainty in A:

$$u_r(t_{q21}) = \frac{s_{tq21}}{1000t_{q2\min}} \times 100\% = 2.08 \times 10^{-5} \quad (17)$$

$t_{q22}$  standard deviation of a single experiment:

$$s_{tq22} = \sqrt{\frac{\sum_{i=1}^n (t_{q22} - \bar{t}_{q22})^2}{n-1}} = 3.40\text{ms} \quad (18)$$

relative standard uncertainty in A:

$$u_r(t_{q22}) = \frac{s_{tq22}}{1000t_{q2\min}} \times 100\% = 2.83 \times 10^{-5} \quad (19)$$

relative standard uncertainty in B:

$$u_{rB}(t_{q2}) = 1/1000 \times \left| \frac{t_{q21} - t_{q22}}{4t_{q2\min}} \right| \times 100\% = 2.92 \times 10^{-6} \quad (20)$$

composite relative standard uncertainty:

$$u_r(t_{q2}) = \sqrt{u_r^2(t_{q21}) + u_r^2(t_{q22}) + u_{rB}^2(t_{q2})} = 3.25 \times 10^{-5} \quad (21)$$

6.2.3 Relative standard uncertainty of

$$U_r = 2 \times u_r = 8.5 \times 10^{-5} \quad (23)$$

$F_2$ -level weight, the maximum  $u_F = 2.5 \times 10^{-6}$ .

6.2.4 Making relative standard uncertainty

Take the maximum value of per uncertainty component, as follow:

$$u_r = (s^2 + u^2 + u_r^2(t_q) + u_F^2)^{1/2} = 4.26 \times 10^{-5} \quad (22)$$

6.2.5 Relative extended uncertainty

When  $k=2$ , relative extended uncertainty:

6.2.6 Verification

Take two LNG dispenser verification device with relative extended uncertainty in 0.30% to compare with above measurement system, and test 6 times. The results as table 4, comparison as chart 8.

devices times	1	2	3	4	5	6	remark
uncertainty $\times 10^{-4}$							
Device 1	45	47	46	48	48	47	
Device 2	49	48	49	48	48	46	
Device 3	0.85	0.86	0.83	0.85	0.84	0.85	

Tips: Device1-LNG dispenser verification device1; Device2-LNG dispenser verification device2; Device3-calibration system for LNG dispenser verification device.

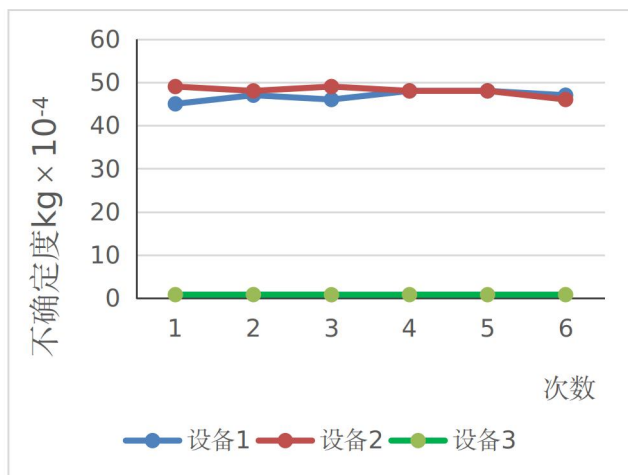


Chart 8 Comparison

7. Conclusion

The experiments show that the relative extended uncertainty of calibration system for LNG dispenser verification device is better than 0.1%. It can be used for calibration of LNG dispenser verification device with relative extended uncertainty in 0.30% ( $k=2$ ).

References:

1. JJG164-2000 《Liquid-flow Standard Device》;
2. JJG1114-2015 《LNG Dispenser》;
3. GB/T36126-2018 《Liquefied natural gas dispenser for vehicle》;
4. OIML R81-2006《Dynamic measuring devices and systems for cryogenic liquids》.