

Comparison of different kinds of frequency scanning methods used at low temperatures

Wenjing Liu^{1,2}, Haiyang Zhang^{1,2*}, Bo Gao^{1,2*}, Changzhao Pan^{3,1}, Dongxu Han^{4,1}, Jiangfeng Hu^{1,2,5}, Yaonao Song^{1,2}, Hui Chen^{6,1}, Xiangjie Kong¹, Ercang Luo^{2,1,5}, Laurent Pitre^{3,1}

¹ TIPC-LNE Joint Laboratory on Cryogenic Metrology Science and Technology, Chinese Academy of Science (CAS), China, Beijing, 100190

² Key Laboratory of Cryogenics, Technical Institute of Physics and Chemistry (TIPC), Chinese Academy of Sciences (CAS), China, Beijing, 100190

³ Laboratoire national de métrologie et d'essais-Conservatoire national des arts et métiers (LNE-Cnam), La Plaine-Saint Denis F93210, France, Paris

⁴ School of Mechanical Engineering, Beijing Institute of Petrochemical Technology, China, Beijing, 102617

⁵ University of Chinese Academy of Sciences, China, Beijing, 100490

⁶ Key Laboratory of Thermo-Fluid Science and Engineering of MOE, School of Energy and Power Engineering, Xi'an Jiaotong University, China, Xi'an, 710049

*Corresponding author, Tel: +86-10-82554512, E-mail: bgao@mail.ipc.ac.cn, zhy110@mail.ipc.ac.cn

Abstract – A novel single pressure refractive index gas thermometry (SPRIGT) has been collaboratively developing by the TIPC-CAS in China and the LNE-Cnam in France [1]. In the SPRIGT, microwave resonance frequencies are used to determine the refractive index of a working gas in a quasi-spherical resonator at a single pressure and temperature. Then an unknown temperature can be determined by comparing the refractive index with that at a reference temperature. Usually, the resonance frequencies were obtained by fitting the S21 scattering parameters with respect to the scanning frequencies, which are generated by a network analyzer using a frequency list generating routine. To realize high accuracy measurement in frequency at low temperatures, three kinds of frequency list generating method (scanning method), namely frequency uniform method, phase angle uniform method and their hybridization method (mixture method), have been investigated. The results show the phase angle uniform and mixture method is easier to realize high accuracy measurement than frequency uniform method at low temperature. Besides, long-time stability in frequency were also studied in this work, the results show that the phase angle uniform and mixture method have a same level stability, thus they can be used in the SPRIGT.

Keywords – SPRIGT, microwave resonator, frequency

scanning, quasi-spherical

I. INTRODUCTION

We can comprehend the nature temperature and measure the thermodynamic temperature through primary thermometry development. High accuracy measurements of thermodynamic temperature at low-temperature range, namely below the neon triple point at 24.5561 K, are of important strategic significance for both science and engineering. In the temperature range from 5K to 24.5561 K, there exist three modern types of primary gas thermometry: constant volume gas thermometry (CVGT), dielectric constant gas thermometry (DCGT) and acoustic gas thermometry (AGT) [2,4], which all have a harsh requirement of absolute pressure measurement. However, China has not yet set up any primary thermometry or devices below 24.5561 K on account of the limitation of absolute pressure measurement accuracy. To face this bottleneck, TIPC of CAS in China and LNE-Cnam in France have jointly developed a neoteric single-pressure refractive-index gas thermometry (SPRIGT) method of primary gas thermometry. This method successfully established the mathematical relationship of frequency, refractive index, gas density and thermodynamic temperature and made it becomes possible to determine the thermodynamic temperature by microwave resonance frequency measurements in a cavity where the pressure

and temperature of gas are accurately controlled. The media of frequency and thermodynamic temperature is index and can be expressed as a ratio method of frequency under vacuum and with pressure as equation:

$$\frac{n^2(T, P)}{n^2(T_{ref}, P)} = \frac{\left(\frac{(f_m + \Delta f_m)_{T,0} \left(1 + \frac{k_T(T)P}{3} \right)}{(f_m + \Delta f_m)_{T,p}} \right)^2}{\left(\frac{(f_m + \Delta f_m)_{T_{ref},0} \left(1 + \frac{k_T(T_{ref})P}{3} \right)}{(f_m + \Delta f_m)_{T_{ref},p}} \right)^2} \quad (2)$$

where $k_T(T)$ and $k_T(T_{ref})$ are the isothermal compressibility of the resonator at T and T_{ref} , the subscript pairs $(T,0)$ and $(T_{ref},0)$ denote the temperature T and T_{ref} at zero pressure.

Based on the advancement on the ab initio calculation of helium-4 properties and microwave resonance technology development, a competitive uncertainty of 0.25 mK theoretically for thermodynamic temperature is expected with ten-fold measurement speed compared with the other primary thermometry methods in SPRIGT[1].

It is a great challenge to realize this innovative approach practically and the SPRIGT system has been dedicated designed and constructed at TIPC-CAS. The resonant frequency is pivotal for getting thermodynamic temperature by SPRIGT. For the purpose of realizing high-precision microwave resonant frequency measurement, the quasi-spherical resonator has been perfectly closed and a stable enough environment of temperature and pressure has been realized [6-8]. Besides, microwave resonant frequency measurement adopted the frequency scanning method, mainly through transmitting microwave signal continuously by a signal source in a customized instrument. Then the microwave signal scans over a determined frequency bandwidth following by specific rules compoing the frequency scanning procedure. There are two kinds of scanning methods mainly used in microwave resonance measurement which will be narrated in detail later. To realize high-accuracy measurement in microwave resonant frequency at low temperatures, thes scanning methods will be investigated in this paper in order to achieve a low uncertainty [9].

II. DESCRIPTION OF THE METHODS

In SPRIGT, the thermodynamic temperature T of the working gas in the resonator cavity is determined by the microwave scanning procedure. And we decided to use the quasi-spherical cavity whose dimensions are precisely designed so as to successfully separate the signal into three peaks. The approach adopted to measure the

microwave resonances of the quasi-spherical resonator is the same as that previously determined to be optimal at NRC [10, 11]. The microwave signal transmitted by the signal source scans over a specific range of frequency as different rules in network analyzer. Afterwards, a scanning parameters called S_{21} can present the scanning procedure. The scattering parameter S_{21} was described three Lorentzian terms and a polynomial background term and fitted by Levenberg–Marquardt method, as shown in paper [12].

Practically, a two-port Agilent N5241A PNA-L network analyzer which has the best signal-to-noise until now is used to connect the resonator antennas with coaxial cables. Generally speaking, a time base is indispensable for the network analyzer, here the 10 MHz reference frequency signal is provided by FS725. Then the microwave resonances for a given triplet is measured by recording the complex S_{21} parameter. It can be expressed by the following equation [12].

$$S_{21}(f) = \sum_{n=1}^3 \frac{A_n f}{f^2 - (f_n + i g_n)} + B + C(f - f_*) + D(f - f_*)^2 \quad (2)$$

where A_n , B , C and D are complex constants which characterize the fitting parameters, f is the source frequency and f_* is an arbitrary constant.

The network analyzer data collection procedure has been optimized for better results through a variety of microwave modes including TM11, TE11, TM12 and TE13 modes. In general, TM modes are found to have better signal-to-noise ratios than TE modes and lower frequency modes have better signal-to-noise ratios than higher frequency modes.

Generally speaking, the procedure of microwave resonance frequency scanning is complicated mainly includes predicting the position of the triplet peaks, performing a coarse scanning encompassing all three predicted triplet peaks as the setting of specific parameters, determining the approximate positions of the actual peaks, performing fine scanning which centered on each of the three peaks in the triplet, determining initial parameters for fitting through coarse and fine scanning, extracting the triplet peak frequencies.

Microwave frequency scanning is the basement of high-precision measurements by the way of microwave resonances. There exist two main kinds of microwave generating and scanning list: frequency uniform scanning method and angle uniform scanning method. The difference of them is the different scanning rules. Frequency uniform method scans between two frequencies with a fixed frequency interval, angle uniform method generates scanning points of frequency with uniform phase angle for each peak.

Owing to the different scanning rule, distribution of the generated scanning points for these two methods has a great different as shown in figure 1. It is clear that for frequency uniform method, the points near the three

peaks are sparse but intensive on the bottom. While the distribution of angle uniform is quite opposite to the frequency uniform. The uneven distribution of scanning points may present the frequency unevenness. For the purpose of covering the scanning range much more uniform, a mixed method which integrate the advantages of these two methods is put forward. Here it is denoted by mixed scanning method as the blue lines in figure 1, it can be seen that the distribution of scanning points is much more uniform.

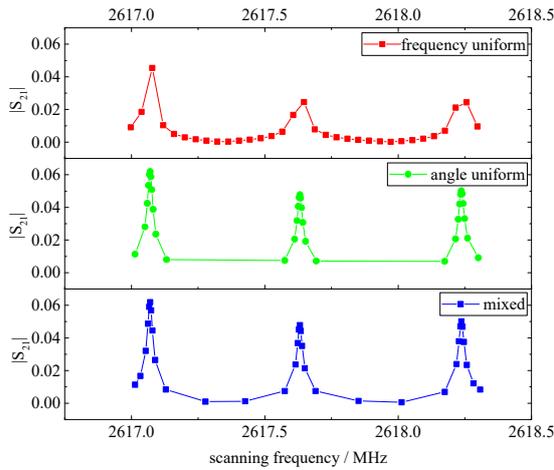


Figure 1 different distribution of scanning points of scanning approaches

Then another problem comes into being that what proportion of these two methods should be chosen. A series of research has been finished in order to find a suitable proportion for uniform distribution then a much more precise uncertainty will be obtained. Figure 2 shows the results of the relative change of fitting parameters in equation 1 through the measurement at different proportions. Here the trends of the $\sigma A/A_{avg}$ (figure2-a), $\sigma B/B_{avg}$ (figure2-b), $\sigma C/C_{avg}$ (figure2-c) and $\sigma D/D_{avg}$ (figure2-d) which present the stability of microwave signal are listed. We can see clearly that when we set 25% (frequency uniform occupies 25% and angle uniform holds 75%), A, B and D are the most stable during experiment. Therefore we can determine 25% as the best proportion for the microwave resonance frequency measurement in SPRIGT. Moreover, the proportion may be different for different experiment system.

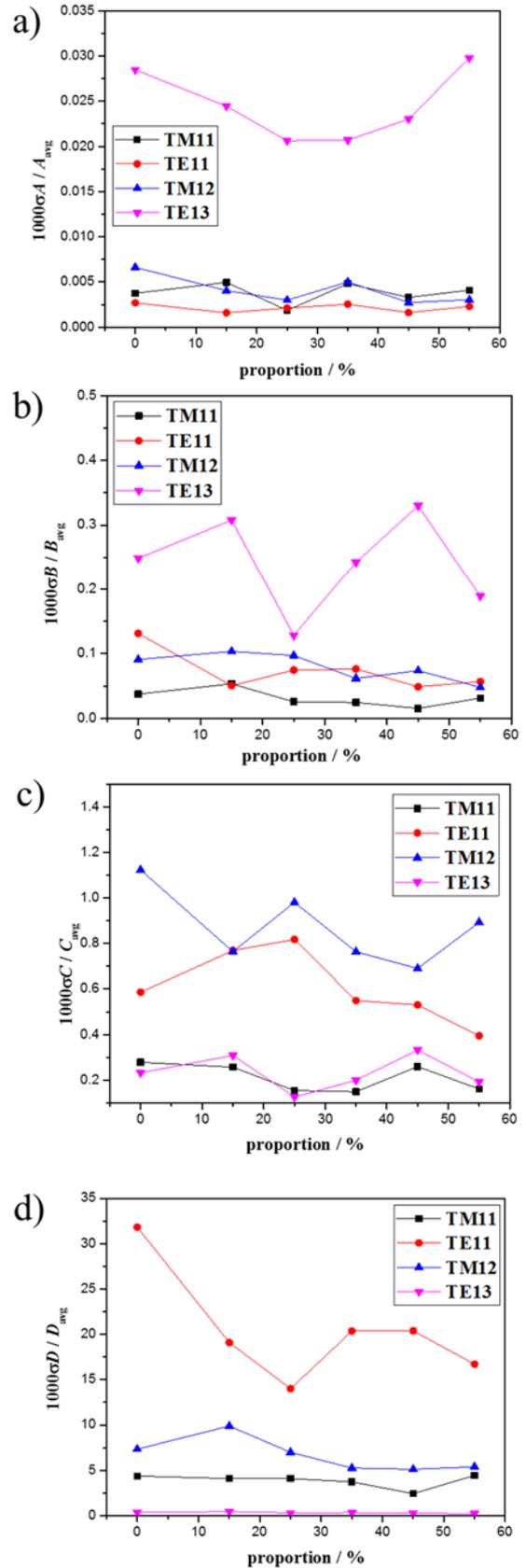


Figure 2 Different distribution of scanning points of scanning approaches

The proposition of this new method and the proportion determination are of great significance to the high-precision microwave resonance frequency measurements at low temperatures. It can not only improve the measurement uncertainty of microwave resonance but also guarantee the accuracy of the thermodynamic temperature obtained from the SPRIGT system.

III. RESULTS AND DISCUSSIONS

So as to be convinced that this new microwave scanning approach is in effect and can be used to measure for long time, we carried out a set of experiments. The table below shows the single point uncertainty of different scanning approaches for 4 microwave modes in one microwave resonance frequency scanning cycle. It is obvious that the uncertainties for 4 microwave modes of frequency uniform scanning method are much bigger than those of the other approaches. To ensure the microwave resonance measurement uncertainty, we decided to abandon the frequency uniform scanning approach. Compared with angle uniform method, the uncertainties of mixed scanning method are slightly smaller for the 4 modes. Therefore, it can be the foundation for long-time measurement with angle uniform and mixed scanning methods.

Table 1. The single point uncertainty of different scanning approaches for 4 microwave modes

Method	TM11	TE11	TM12	TE13
Frequency uniform	1.56	14.39	3.6	3.23
Angle uniform	0.32	0.17	0.14	0.96
Mixed	0.31	0.16	0.13	0.94

The following states the analysis results of measuring for 12 hours at 5 K in these two approaches. Here only shows the analysis results of only TM11 mode, the other three modes are the similar to TM11. Figure 3 demonstrates the frequency result of angle uniform scanning method by Allen variance. And the result of analysing that of mixed method in the same way is shown in Figure 4. These two measurement are both taken under vacuum, unfortunately, the level of temperature control is totally different owing to the influence of external environment. The 12 hours measurement of angle uniform method are implemented when the standard deviation of temperature control is 0.029 mK. However, it is 0.0694 mK of mixed method, much bigger than angle uniform. Temperature control precision has a big impact

on the frequency stability, but we can see from the figures that the long-time stability of angle uniform is about 0.009 ppb and 0.008 ppb of mixed method (the integration time are both 3 hours). It is also clearly to see that collecting by mixed method is much more stable then we could use it for long-time measurement.

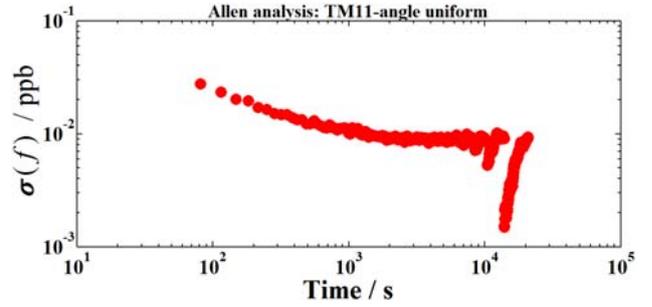


Figure 3 Allen variance result of angle uniform scanning method measurement for 12 hours

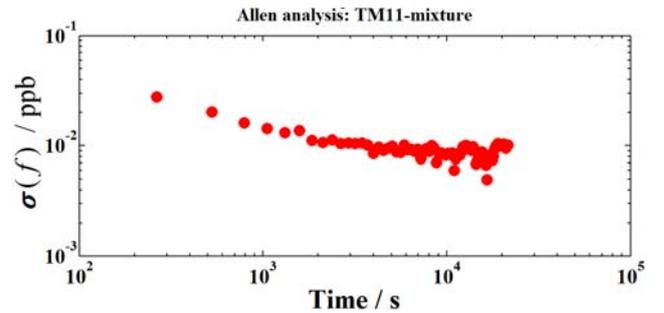


Figure 4 Allen variance result of mixed scanning method measurement for 12 hours

As can be seen above, so as to get the thermodynamic temperature T of SPRIGT system, the data collection from 5K to 25K is indispensable. Forasmuch, it is necessary to test the result of mixed scanning method. We test these three kinds of scanning approaches from 16 to 22K for 4 microwave modes. The results of the three scanning methods for 4 modes is shown in Figure 5, through comparing the single point uncertainty of three methods for 4 modes, we can see that the uncertainty at different temperature has no big difference for 4 modes. Then we can say that this new method can be used in SPRIGT system.

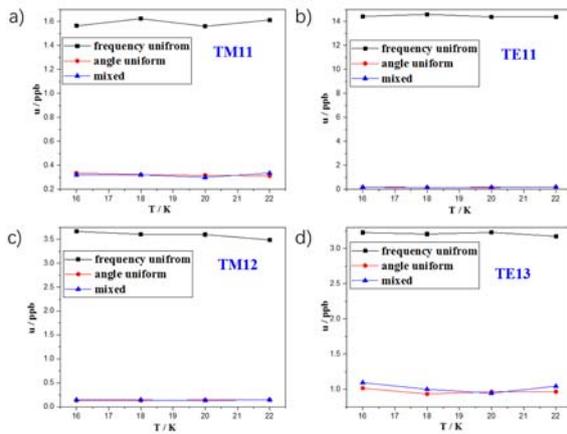


Figure 5 Comparison results of three scanning methods from 16K to 22K

IV. CONCLUSIONS

The SPRIGT experiment system has been established in TIPC so as to verify the feasibility of single pressure refractive index gas thermometry. The microwave resonance frequency scanning approaches have been investigated in order to accomplish high-precision microwave measurement. And one novel method called mixed method has been put forward. We have described the measurement effect of this method for not only one microwave scanning cycle at different temperature for different microwave modes but also long-time stability measurement. After comparing the results of this new one with the other two methods, we insure that this method can be used in the SPRIGT system covering the studied temperature range. When we set the integration time as 3 hours, the standard deviation of continuous measurement for 12 h was 0.008 ppb. At our knowledge, it is the first time that such scanning method successfully used in the experiment system. And this mixed method can be widely used in other fields to improve the measurement uncertainty.

ACKNOWLEDGMENT

This work is supported financially by the National Key R&D Program of China (Grant No. 2016YFE0204200), the National Natural Science

Foundation of China (Grant No. 51627809), the International Partnership Program of the Chinese Academy of Sciences (Grant No. 1A1111KYSB20160017), the EMRP project InK2 (No. 15SIB02) and the China Postdoctoral Science Foundation (Grant No. 2017LH031).

REFERENCES

- [1] B. Gao, L. Pitre. et al. "Feasibility of primary thermometry using refractive index measurements at a single pressure," *Measurement*, vol. 103, pp. 258-262, June 2017.
- [2] L. Pitre, F. Sparasci. et al. "New measurement of the Boltzmann constant k by acoustic thermometry of helium-4 gas," *Metrologia*, vol. 54, no. 6, pp. 856-873, October 2017.
- [3] W.J. Liu, L. Pitre. et al. "Microwave Method to Close Two Hemispheres," *CPEM 2018 Conf. Digest*, Paris, 2018.
- [4] M. R. Moldover, W. L. Tew and H. W. Yoon, 2016, Advances in thermometry, *NATURE PHYSICS*, 12, 7-11.
- [5] L. Pitre, L. Risegari. et al. "Determination of the Boltzmann constant k from the speed of sound in helium gas at the triple point of water," *Metrologia*, vol. 52, no. 5, pp. 263-273, August 2015.
- [6] Gao B, Pan C, Pitre L, et al. Chinese SPRIGT realizes high temperature stability in the range of 5–25 K[J]. *Sci. Bull.*, 2018, 63: 580-594.
- [7] Chen Y Y, Zhang H, Song Y, et al. Thermal response characteristics of a SPRIGT primary thermometry system[J]. *Cryogenics*, 2019, 97: 1-6.
- [8] Han D, Gao B, Chen H, et al. Ultra-stable pressure is realized for Chinese single pressure refractive index gas thermometry in the range 30-90 kPa[J]. *Science Bulletin*, 2018, 63(24): 1601-1603.
- [9] H.Y. Zhang, W.J. Liu, B. Gao. et al. A high-stability quasi-spherical resonator in SPRIGT for microwave frequency measurements at low temperatures[J]. *Science Bulletin*, 2019.
- [10] Rourke P M C. NRC microwave refractive index gas thermometry implementation between 24.5 K and 84 K[J]. *International Journal of Thermophysics*, 2017, 38: 1-27.
- [11] Hill K D. Progress toward development of low-temperature microwave refractive index gas thermometry at NRC[J]. *International Journal of Thermophysics*, 2015, 36(2-3): 205-228.
- [12] May E F, Pitre L, Mehl J B, et al. Quasi-spherical cavity resonators for metrology based on the relative dielectric permittivity of gases[J]. *Review of scientific instruments*, 2004, 75(10): 3307-3317.