Resonance frequency measurement with accuracy and stability at the $10^{-12}$ level in a copper microwave cavity below 26 K by experimental optimization

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Received 19 October 2019, revised 15 February 2020
Accepted for publication 24 February 2020
Published 4 May 2020

Abstract

Single-pressure refractive index gas thermometry (SPRIGT) is a novel primary thermometry technique, developed jointly by the Technical Institute of Physics and Chemistry (TIPC) of the Chinese Academy of Sciences (CAS) in China and LNE-Cnam in France. To help obtain a competitive uncertainty of 0.25 mK in thermodynamic temperature measurements, high-stability and low-uncertainty of microwave resonance frequency measurements better than 2 ppb have been demonstrated. This article describes how high-stability and low-uncertainty resonance frequency measurements were achieved using a copper microwave cavity. Microwave measurements were carried out under vacuum and isobarically at helium-4 pressures of 30, 60, 90, and 120 kPa over the temperature range of 5–26 K, with good consistency among microwave modes for the thermodynamic temperatures determined. Performance was optimized using an Allan variance analysis. In this way, with an integration time of 3 h, a stability and accuracy at the $10^{-12}$ level were obtained, an almost 20-fold improvement upon our previous result (Zhang et al 2019 Sci. Bull. 64 286–8).

Keywords: stability, microwave resonance frequency, resonator, Allan deviation, primary thermometry, thermodynamic temperature

(Some figures may appear in colour only in the online journal)

1. Introduction

Single-pressure refractive index gas thermometry (SPRIGT), developed jointly by the Technical Institute of Physics and Chemistry of the Chinese Academy of Sciences (TIPC-CAS) in China and Laboratoire national de métrologie et d’essais-Conservatoire national des arts et métiers (LNE-Cnam) in France [1], is a type of thermometry method based on gas polarizability. As a relative primary thermometry technique [2], SPRIGT measurements are conducted on single isobars rather than isotherms, which alleviates the need for accurate absolute-pressure measurement and increases the
measurement speed ten-fold. With state-of-the-art ab initio calculations of helium-4 properties [3–8], a competitive uncertainty of 0.25 mK is expected for the measurement of thermodynamic temperature below the neon triple point at 24.5561 K [1], which is of important strategic significance for the development of both cutting-edge scientific research and large scientific facilities, such as the Spallation Neutron Source in China and Large Hadron Collider in Europe [9].

Microwave measurements are widely used in the field of gas metrology [10–16]. SPRIGT allows the thermodynamic temperature to be determined from the ratio of gas refractive indices at a single pressure, one measured at the unknown temperature and the other at the reference temperature [1, 17], while the refractive index can be measured from the microwave resonance frequencies in a quasi-spherical high-conductivity resonator. The reference temperature can be the fixed point of neon or a known thermodynamic temperature measured using another absolute primary thermometry method, such as acoustic gas thermometry (AGT). In refractive index gas thermometry (RIGT) [2, 18], microwave measurements are used to determine the gas refractive index \( n(p,T) \). From the knowledge of \( n(p,T) \) and the pressure \( p \), the thermodynamic temperature \( T \) is deduced. RIGT has been successfully implemented at National Research Council (NRC) in Canada by Rourke to measure the temperatures 24.5561 K (neon triple point), 54.3584 K (oxygen triple point), and 83.8058 K (argon triple point), which are three ITS-90 defining fixed points [19]. Recent progress has also been achieved in the same laboratory on the measurement of the thermodynamic temperature of the triple point of xenon \( T = 161.40596 \) K by extrapolating the experimentally determined compressibility at the triple point of water [20]. In a complementary application of RIGT, where the pressure is deduced from the refractive index and temperature measurements, a quantum standard for absolute pressure measurements is currently being developed jointly by LNE in France and INRIM in Italy in the range from 0.2 kPa to 20 kPa by a superconducting microwave cavity [21]. In AGT [10, 13–16], microwave measurements are used to determine the dimensions or volume of the resonator so that the thermodynamic temperature can be deduced from acoustic measurements. Moreover, microwave measurements have also been used in other precise measurements, such as density and critical phenomena of helium [22], frequency [23], and quantum-gas and gravitational physics [24]. These were successfully implemented by using high-quality-factor niobium microwave cavities. The aforementioned gas metrology methods (SPRIGT, RIGT, AGT, and absolute pressure in a quantum standard) and precise measurements would benefit from any improvement in microwave measurements.

The best current primary standards can produce the SI second with a relative standard uncertainty approaching \( 10^{-16} \), while the relative uncertainty of secondary frequency standards based on optical clocks is at the level of \( 10^{-18} \) [25]. A convenient way to perform experiments is to use a servo loop to lock the oscillator frequency to the maximum of a resonance. The frequency stability then depends on the shape and sharpness of the resonance, the signal-to-noise ratio, and the type of noise that predominates at the time scale under consideration [26–28]. One of the most commonly used techniques for servo-locking is the sideband method due to Pound and its optical equivalent [28]. In contrast, in SPRIGT, it is essential to perform scans over the resonance lines to check for any change in the width and shape (see section 3.1). These parameters are used both to measure electrical conductivity and monitor systematic effects. A scan typically takes two minutes. To implement SPRIGT, a system has been built at TIPC-CAS in China, which includes three subsystems, namely a temperature control system, a pressure control system, and a microwave system. The temperature and pressure control systems are designed to provide a very stable working environment for the microwave resonator and connecting cables. High-stability temperature control (0.2 mK), pressure control (4 ppm, 1 ppm \( \equiv 10^{-6} \)), and microwave resonance frequency (2 ppb, 1 ppb \( \equiv 10^{-9} \)) are required for thermodynamic temperature measurements with an uncertainty of 0.25 mK. A simple description of the three subsystems is presented in section 2.

In this paper, the structure of the quasi-spherical resonator (QSR) is described, and the experimental optimization of microwave measurement is presented. The rest of the paper is structured as follows. In section 2, we describe the experimental apparatus involving the main instrumentation for microwave, temperature, and pressure measurements. Detailed descriptions of the temperature [29, 30] and pressure measurement [31] can be found elsewhere and lie beyond the scope of the present paper. In section 3, we present how to realize high-stability and low-uncertainty of microwave resonance frequency measurement by optimizing the microwave background, the microwave emission power, mechanical stability, temperature control method, time reference, and microwave signal.

2. Experimental set-up

The apparatus of SPRIGT is shown in figure 1(a), including microwave, temperature control, and pressure control subsystems. For the microwave system, the core element is a copper (electrolytic tough pitch (ETP) copper, ISO norm) microwave cavity, i.e. a QSR, shown in figure 1(b). The high-quality-factor QSR was built from two hemispheres.

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1 On May 20th, 2019, the Bureau International des Poids et Mesures announced a major revision to the International System of Units (SI), in which the base unit, the kelvin, symbol K, was redefined by fixing the value of the Boltzmann constant. The practical realizations of the kelvin by primary thermometry are indicated in the ‘Mise en pratique for the definition of the kelvin in the SI’, in which low-uncertainty primary thermometry is required to promote the realizations of the new kelvin and the spread of high-accuracy, low-temperature metrology. (see https://www.bipm.org/utils/en/pdf/si-mep/SI-App2-kelvin.pdf).

2 Quality factors \( Q = f/2g \) for the four microwave modes at 5 K–26 K are: \( Q({\text{TM11}}) \approx 140 000 \), \( Q({\text{TE11}}) \approx 210 000 \), \( Q({\text{TM12}}) \approx 170 000 \), \( Q({\text{TE13}}) \approx 290 000 \).
whose inner surfaces were machined by precision diamond turning. The dimensions are the same as those of the resonator used at LNE-Cnam for a determination of the Boltzmann constant [11]. The inner shape is designed to be a tri-axial ellipsoid defined by

$$\frac{x^2}{a^2} + \frac{y^2}{a^2(1 + \varepsilon_2)^2} + \frac{z^2}{a^2(1 + \varepsilon_1)^2} = 1$$  \hspace{1cm} (1)

with $a = 49.50$ mm, $\varepsilon_1 = 0.001$, and $\varepsilon_2 = 0.0005$, corresponding to the nominal semi-major axes of the tri-axial ellipsoid of 49.50 mm, 49.75 mm, and 50.00 mm on the $x$, $y$, and $z$-axes, respectively. The nominal shell thickness of the resonator is 10.0 mm. Two loop antennas are connected to a two-port vector network analyzer (Keysight Technologies N5241A PNA-X), one in each hemisphere used for emission and reception. The frequency reference of the vector network analyzer is a 10 MHz signal provided by a rubidium frequency standard (Stanford Research Systems FS725) or, for better stability, a GPS time and frequency system (Stanford Research Systems FS740, locked to GPS with oven-controlled crystal oscillator (OCXO) or rubidium frequency standard). For FS740, two kinds of antenna were used, one indoor, the other outdoor.

The QSR has been closed successfully at room temperature using a microwave method by monitoring the change in the relative excess half-width [32]. The resonator was designed to be used under vacuum and then with helium gas. For this reason, to avoid contamination, argon (purity of 99.999%, Beijing AP BAIF Gases Industry Co. Ltd) was flowed through it at a continuous rate of 80 SCCM until the resonator was coupled in the pressure vessel of the cryostat. After putting the resonator into the pressure vessel of the cryostat, a preliminary test on the performance of the QSR under vacuum was implemented without using any optimization methods involved in the present work. Frequency stability and uncertainty of 0.02 ppb and 0.03 ppb were realized by using 0 dBm microwave emission power with an integration time of 3 h [17], equivalently 0.063 ppb and 0.095 ppb for $-10$ dBm microwave emission power [1].

For the temperature control system, a cryogen-free cryostat was developed for SPRIGT in our previous work [29], using

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Figure 1. The structure schematic diagram of SPRIGT. (a) A simplified system diagram. (b) A 3D drawing of the QSR.
a two-stage Gifford-McMahon (GM) type pulse tube cryocooler as the cooling resource. Based on multi-layer radiation shields combined with the thermal-resistance method, gas type heat switch and proportional-integral-derivative (PID) control method, the pressure vessel temperature stabilities of 0.021 mK – 0.050 mK were realized with an integration time of 0.8 s in the temperature range from 5 K to 25 K. Based on further investigation on thermal characterization of the cryostat, the pressure vessel temperature stabilities were improved to 0.019 mK in the same temperature range with the same integration time [30]. In this work, the temperature of the resonator was measured by a rhodium-iron thermometer (Tinsley, SN226242) coupled to an AC resistance bridge (ASL F900). The 10 Ω standard reference resistance (Tinsley 5685A, SN1580409) was placed in an oil bath (Aikom Instruments MR 5100-L) with temperature stability better than 1 mK.

For the pressure control system, the detailed content is not emphasized in this work since it has been presented in our previous work [31]. The uncertainty contribution of pressure to measurements of thermodynamic temperature has also been reported in our previous work [1] and figure 2 in the review paper of RIGT [2]. To put helium-4 gas (purity of 99.9999%, Air Liquide) into the resonator and keep a single pressure, a gas compensation loop was built at room temperature to compensate for the leak of the piston gauge. The gas pressure was measured by an absolute-pressure piston gauge (Fluke PG 7601) and conducted by maintaining the piston at a constant height using a laser interferometer (KEYENCE LK-G80). Pressure stabilities of 0.0032 Pa at 30 kPa and 0.002 Pa at 90 kPa were realized with an integration time of 1.4 s at room temperature, corresponding to relative stabilities of 0.1 ppm and 0.02 ppm, respectively [31].

3. Experimental optimization and discussions

To realize high-stability and low-uncertainty microwave resonance frequency measurements at low temperatures, several experimental optimizations were implemented by using Allan analysis of variance in the present work. The detailed results are listed below.

3.1. Microwave background polynomial

In this work, the microwave resonance frequencies $f_n$ and half-widths $g_n$ were determined from non-linear least-squares fitting (LM method [33]) of the measured complex scattering parameters $S_{21}$ as a function of frequency [34]. Here, we rewrite equation (15) in the literature to the following form:

$$S_{21} = \sum_{n} \frac{A_{nf}}{f^2 - (f_n + ig_n)^2} + \sum_{j=0} f_j (f-f_j)^j$$

Figure 2. Fitting results of the TM11 microwave mode for different background polynomial orders under vacuum: (a) $<f + g>$ against microwave background polynomial order $J$ at 5 K; (b) real and imaginary components of the parameter $S_{21}$ at 5 K, measured and fitted using the second-order background polynomial; (c) $<f + g>$ against the microwave background polynomial order $J$ at 24.5 K; (d) real and imaginary components of the parameter $S_{21}$ at 24.5 K, measured and fitted values using the second-order background polynomial.
where the fitting parameters were the complex constants $A_n$, $B_j$, and the three complex resonance frequencies $(f_3 + ig_n)$, one for each component of the triplet. In equation (2), $J$ is the background polynomial order, $f$ is the source frequency, and $f_s$ is an arbitrary constant; here, we made the same choice as the literature [34], i.e. $f_s = f_3$, to avoid numerical problems in the fitting program.

Figure 2 shows the fitting results of the TM11 microwave mode for different background polynomial orders at 5 K and 24.5 K under vacuum. With the polynomial order increasing, the average fitted triplet frequency $<f + g>$ and the uncertainty $u(<f + g>)$ have a little change, as shown in figures 2(a) and (c); the maximum relative changes in $<f + g>$ are about $2.0 \times 10^{-10}$ and $1.9 \times 10^{-10}$ for 5 K and 24.5 K, respectively. These changes are less than half of the relative standard uncertainty of $<f + g>$, indicating that increasing the microwave background polynomial order cannot obviously reduce their influence on the average fitted triplet frequency and the uncertainty in our system. Considering other microwave modes and those measured at room temperatures, a second-order-background polynomial is competent in this work. Table 1 lists the fitted frequencies and half-widths of the triplet for the second-order background polynomial. Figures 2(b) and (d) compare the results of the measured and calculated components of the scattering parameters $S_{21}$, where good agreement was found.

Moreover, the background noise from cables and feed-throughs influences the microwave measurement. To reduce the noise as much as possible, one can use some low-loss cables and feed-throughs, covering the working frequency range. For further improvement, the optimal combination of cables and feed-throughs, even the working microwave modes can be selected by measuring and analyzing the $S_{21}$ parameters.

3.2. Microwave emission power

In this work, the microwave signal was generated by a two-port N5241A vector network analyzer. The signal was transmitted from port 1 of the vector network analyzer, passing through the cables, feed-throughs, and resonator, later received by port 2. Since the relative loss of the cables and feed-throughs are nearly constant at stable operating conditions, the microwave emission power has a direct influence on the measurement inside the resonator. If the power is too small, the microwave signal may be drowned in the background noise, and thus leads to a relatively larger uncertainty of the fitted frequency or even no triplet. On the contrary, the microwave signal will heat the resonator too much; this will increase the temperature control instability and in turn reduce the frequency stability. Thus, it is necessary to experimentally optimize the microwave emission power.

Figure 3 shows the optimized results of the power at 5.0 K under vacuum based on the TM11, TE11, TM12, and TE13 microwave modes, which were employed in our preliminary measurements [17]. A bigger power makes it easier to increase the temperature instability, as shown in figures 3(a) and (b), where the temperature stabilities are about 0.042 mK and 0.024 mK for the microwave emission powers $P_{MW} = -7$ dBm and $P_{MW} = -10$ dBm, respectively. Figure 3(c) plots the temperature Allan standard deviation against the integration time for these two powers. It can be seen that there is an oscillation of the temperature Allan standard deviation for $P_{MW} = -7$ dBm, which is coupling with the measurements of the four microwave modes, measured in the TM11-TE11-TM12-TE13 loop. While for $P_{MW} = -10$ dBm, no oscillation was observed on the temperature Allan standard deviation, which means the microwave power has a negligible disturbance effect on temperature stability. Thus, the optimal microwave emission power was found to be $P_{opt} = -10$ dBm.

In the present work, we use one single microwave emission power $P_{opt}$ during all the low-temperature microwave frequency measurements, no matter whether the resonator is under vacuum or filled with high-purity helium-4 gas.

3.3. Mechanical stability, temperature control method, time reference, and signal intensity

In this section, mechanical stability, the temperature control method, the time reference, and signal intensity were also studied in this work under vacuum unless otherwise specified.

3.3.1. Mechanical stability. When cooling the resonator from room temperature down to low temperatures (5 K–25 K) for each independent run in this work, it is important to keep the resonator in good mechanical stability before doing the microwave measurements. A simple way to release the mechanical stress is to carry out temperature cycling in the objective temperature range, for example, cooling and warming between 25 K and 5 K in this work. Although the procedure will take some times, it is good for temperature and frequency measurements. At the same set point for temperature control, after the temperature cycling, it is easier to realize highly stable temperature measurements, which have been observed in our system. The temperature stability, in turn, will improve the frequency measurement, as shown in section 3.3.2 below. When the temperature of the resonator reached the objective temperatures (5 K and 24.5 K), temperature regulations together with microwave measurements were implemented for several hours. If the frequencies of each microwave mode showed no obvious change anymore (changing within the microwave measurement uncertainty) at the objective temperatures, then it can be considered that the resonator has released its mechanical stress and is stable enough for the implementation of microwave measurements.

A cooling–warming temperature cycling for 55-hour was carried out in this work. The result is shown in figure 4(a), where the cooling and warming processes are respectively denoted as C and W in the figure legend, and the behind number is the sequence of the temperature cycle. The average amplitude of $|A_n|$ in equation (2) of the cooling and warming processes, denoted as $A$, was used to indicate the frequency change with the temperature change. They do not completely overlap, mainly because of a big difference in the temperature change speed between them. This will
Table 1. Fitted values of the triplet with the second-order background polynomial.

| T/K  | $f_1$/MHz       | $g_1$/MHz       | $f_2$/MHz       | $g_2$/MHz       | $f_3$/MHz       | $g_3$/MHz       |
|------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 5    | 2617.0688336(11)| 0.0093815(11)   | 2617.6300911(15)| 0.0093725(15)   | 2618.2397203(16)| 0.0098880(16)   |
| 24.5 | 2617.0614026(12)| 0.0094027(12)   | 2617.6226012(16)| 0.0093916(16)   | 2618.2321434(17)| 0.0099124(17)   |

Figure 3. Microwave emission power optimization at 5.0 K under vacuum. (a) Temperature evolution with $P_{MW} = -7$ dBm. (b) Temperature evolution with $P_{MW} = -10$ dBm. (c) The temperature Allan standard deviation $\sigma_T$ as a function of the integration time for $P_{MW} = -7$ dBm and $P_{MW} = -10$ dBm.

be improved later in our work future by controlling the cooling and warming speed automatically. Generally, after several cooling and warming cycles, microwave measurements can be implemented in the resonator when the temperature and the filled gas pressure are stable simultaneously. This is the case for the present work, where obvious resonance frequency changes were not observed for each microwave mode at the two objective temperatures, respectively.

3.3.2. Temperature control method. Due to the thermal expansion effect of the resonator, its dimensions will change if the temperature is not stable. The microwave resonance frequency is proportional to the reciprocal of the radius of the resonator, thus the temperature stability has a direct influence on microwave measurements. Figure 4(b) shows the influence on the microwave resonance frequency stability of the TM11 mode for two temperature control methods at $T = 15.0$ K and $p = 30$ kPa. Method 1 uses an 8½ digit multimeter (Keithley 2002) with a Cernox sensor as in our previous work [30], while method 2 uses an AC resistance bridge (ASL F18) with a calibrated rhodium-iron sensor as in CCT-K1 [35]. The temperature stabilities of the two methods are 38 $\mu$K and 8 $\mu$K with an integration time of 33.6 s, respectively. The frequency stability of method 2 is better than that of method 1 by about an order of magnitude with an integration time $\tau = 3$ h. Good temperature stability can improve the microwave resonance frequency stability. Thus, in our later measurements, method 2 was adopted all the time to maintain a good frequency stability.

3.3.3. Time reference. Usually, the vector network analyzer is connected to an external time reference, such as a rubidium atomic clock or GPS clock, to realize high-accuracy and high-stability frequency measurements. In this work, a rubidium
Figure 4. Optimized results of the mechanical stability, temperature control, time reference, and signal on the TM11 microwave mode under vacuum unless otherwise specified. (a) Evolution of the resonator temperature and average amplitude of $|A_n|$ in equation (2) during the temperature cycle. (b) The frequency Allan standard deviation with two temperature control methods at $T = 15.0$ K and $p = 30$ kPa. (c) Two-day frequency stability at 24.5 K with different time references. (d) Seven-day frequency stability at 5.0 K for the FS740 time reference locked to GPS with a rubidium atomic clock configured indoor antenna. (e) Real and imaginary components of $|S_{21}|$ of the triplet at 5.0 K with and without the two microwave amplifiers. (f) The frequency Allan standard deviation at 5.0 K using FS740 locked to GPS with a rubidium atomic clock configured outdoor antenna and amplifiers.

atomic clock (FS725) and GPS clock (FS740 locked to GPS) were used. While the long-term stability of GPS is excellent, its short-term stability is rather poor in comparison to modern oscillators [36]. Therefore, in this work, two different oscillators, an OCXO and a rubidium atomic frequency standard, were configured for FS740.

Figure 4(c) shows the typically measured stability of the microwave resonance frequency of the TM11 mode at 24.5 K in two days with the three types of time references, namely, FS725 (rubidium atomic clock), FS740 locked to GPS with a rubidium atomic clock and with an OCXO clock, with GPS configured indoor antenna. The frequency stability of FS740 locked to GPS with the rubidium atomic clock configured indoor antenna and that of the FS725 are comparatively equivalent: both stabilities are better than that of the OCXO clock. A resonance frequency stability of 0.02 ppb was realized using FS740 locked to GPS with rubidium atomic clock configured indoor antenna and FS725 with an integration time $\tau = 3$ h in the two-day microwave measurements. The SPRIGT measurement at different temperatures and pressures
may take one or two months; the time reference stability of the reference should be tested at this level to check if any other effect may perturb the measurement.

Furthermore, long-term microwave measurements at 5.0 K in seven days were carried out by FS740 locked to GPS with rubidium atomic clock configured outdoor antenna. The stability result is plotted in figure 4(d). A good resonance frequency stability of 0.04 ppb was achieved with $\tau = 3$ h.

3.3.4. Signal intensity. An amplifier is usually used to enhance the microwave signal intensity, as shown in the literature [37], where the amplitude of $s_{21}$ was amplified by nearly ten times with the help of a set of two 10 dBm amplifiers (Model ZX60-14 012 l, Mini-Circuits, bandwidth from 0.3 MHz to 14 GHz) connected in series at room temperature. The uncertainty was reduced by a factor of 5 to 12, even with a low-cost vector network analyzer.

In this work, with the same method and two amplifiers, as in the literature [37], measurements were carried out at 5.0 K under vacuum. Figure 4(e) shows the comparison results of the real and imaginary components of $s_{21}$ with and without the two amplifiers. With amplifiers, the amplitudes were amplified by about ten times and, in turn, the fitted relative standard uncertainty of the resonance frequency was reduced by a factor of 10 (from 0.3 ppb to 0.031 ppb). This is because the amplifiers increase the resolution of the peaks of the triple. A similar situation appears in the TE11, TM12, and TE13 microwave modes, with the fitted relative standard uncertainty of the resonance frequency reduced by a factor of 5, 5, and 10, respectively. It is a simple and effective way to solve signal problems, such as high-loss of microwave cable and feed-through, and even low-cost vector network analyzer. Moreover, low-temperature microwave amplifiers are expected to have a better performance than room-temperature amplifiers, and this will be one solution of ultra-high accuracy microwave measurements in the future work.

3.3.5. Overall performance. In section 3.3.3, the microwave measurements were carried out by using the indoor antenna, which was placed outside a window of the laboratory building, facing north against another building. Actually, the FS740 was supplied with two types of antenna: an indoor antenna, and an outdoor antenna. According to the user’s manual of the FS740 time reference [36], the best results can be realized if the antenna has a clear unobstructed view of the sky. It is highly recommended to put the outdoor antenna on the roof of the building, within which the FS740 is located. This is because it improves the quality and reliability of the GPS signals as more satellites will typically be visible and with a higher signal-to-noise ratio. This can improve the long-term stability of the FS740 time reference by a factor of three. This configuration was used for the following test described in this section.

Combining the above optimized results, namely the second-order background polynomial, optimized power $P_{opt} = -10$ dBm, good mechanical stability, temperature control using an AC resistance bridge and rhodium-iron resistance sensor, the time reference FS740 locked to GPS with a rubidium atomic clock but configured outdoor antenna and two room-temperature amplifiers connected in series, preliminary stability measurements on the TM11 mode were implemented at 5.0 K under vacuum. Figure 4(f) plots the Allan standard deviation of the resonance frequency. With the same time reference, FS740 locked to GPS with a rubidium atomic clock, the stability for the outdoor antenna with amplifiers is about an order of magnitude better than that for only indoor antenna, as shown in figures 4(d) and (f). High-stability of 3.7 parts per trillion (ppt, 1 ppt $= 10^{-12} = 10^{-3}$ ppb) and low-uncertainty of 5.2 ppb were realized with an integration time of 3 h. The frequency stability and uncertainty in the present work are nearly 20 times better than those in our previous work [17]: 0.064 ppb and 0.095 ppb with the same integration time and $P_{opt} = -10$ dBm [1].

In SPRIGT, the thermodynamic temperature uncertainty has many influence factors, and frequency is one of the major factors, as shown in our previous work [1] and the RIGT review paper [2]. The improvement in frequency measurement accuracy can reduce its influence on thermodynamic temperature measurement by a factor of 20, which is good for realizing the high-accuracy measurement of thermodynamic temperature by SPRIGT. The above optimization methods have the potential to be used in gas metrology and other research fields, where high-stability and low-uncertainty microwave measurements are necessary.

3.4. Results with pressures

After many experimental optimizations under vacuum, preliminary microwave measurements were performed from 5 K to 26 K with the resonator filled with high-purity helium-4 at the following conditions: (1) fitting $s_{21}$ with a second order background polynomial; (2) setting the microwave emission power $P = P_{opt} = -10$ dBm; (3) carrying out a temperature cycle to make the resonator have a good mechanical stability; (4) controlling the temperature with an AC resistance bridge and rhodium-iron sensor; (5) using the time reference FS725; and (6) without the two amplifiers. The measurements were implemented on isobars, and the results are plotted in figure 5.

Figure 5(a) shows the total relative standard uncertainty of $< f + g >$ of the TM11 mode at temperatures from 5 K to 26 K and pressures up to 120 kPa with $\tau = 80 \text{ s}^2$. Most uncertainties are located within 0.9 ppb, except the $p-T$ region with pressures near 120 kPa and temperatures near 5 K. The main reason is that at lower temperatures, the pressure stability inside the resonator becomes a little worse than that

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4 It is usually used as a constant integration time for all the microwave modes. Figure 5(a) only shows the uncertainty of the TM11 microwave mode for different pressures and temperatures. However, for the high-frequency TE13 mode, the maximal total relative standard uncertainty is about 1.8 ppb. Moreover, the integration time was also chosen to be in the same ‘time’ as the other instruments, such as the resistance bridge. The integration time of 20 000 s is used to check and characterize the stability of the measurements.
at higher temperatures, which disturbed the frequency stability by the change in helium-4 density inside the resonator. Figure 5(b) plots a one-day microwave measurement of the TM11 mode at 24.5 K with different pressures. The frequency stabilities are about 0.03, 0.02, 0.04, and 0.05 ppb for 30, 60, 90, and 120 kPa with $\tau = 3$ h, respectively, and the temperature stabilities are about 6.9, 8.3, 7.9, and 6.6 $\mu$K with $\tau = 33.6$ s. Due to the pressure stability, 60 kPa has the best frequency stability in the one-day stability measurement. These are still better than our previous work (0.063 ppb under vacuum provided with $P_{\text{opt}} = -10$ dBm); even the pressure instability increases the microwave frequency measurement instability. The stability and uncertainty still have room for improvement, provided later using the time reference, FS740 locked to GPS with rubidium atomic clock configured outdoor antenna, and microwave amplifiers.

Based on the SPRIGT principle [1], thermodynamic temperatures were determined from different microwave modes for the isobar $p = 60$ kPa. Figures 5(c) and (d) present the differences between mode thermodynamic temperatures $T_{\text{mode}}$ and the average values $T_{\text{avg}}$, $(T_{\text{mode}} - T_{\text{avg}})$, at temperatures from 5 K to 26 K, where microwave measurements with $\tau = 80$ s were used. Good mode consistency was observed on $(T_{\text{mode}} - T_{\text{avg}})$ within $\pm 80 \mu$K, and a small relative standard uncertainty component for the thermodynamic temperatures, from mode consistency, was realized with $u(T_{\text{mode}})$ values $<60 \mu$K.$^5$

### 4. Conclusion

The object of this study was the optimization of microwave frequency measurements at low temperatures. The measurements in question concern the resonance frequencies of a high-quality-factor tri-axial ellipsoidal copper resonator, either under vacuum or else filled with high-purity helium-4 gas (at pressures from 30 kPa to 120 kPa). The ratio of frequencies with and without gas yields the refractive index from which the temperature can be determined with high accuracy once the effects of pressure are taken into account. This is called RIGT. The application here concerns a novel variant of the technique exploiting single isobars such that pressure-dependent effects almost cancel—single-pressure RIGT or SPRIGT. Microwave

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$^5$ The uncertainty budget for SPRIGT has been reported in our previous work [1] and the review paper of RIGT [2]. Since this work is focused on microwave measurements, it will be conducted in our future work.
resonances are detected using pairs of antennas linked to a vector network analyzer.

The study dealt with both experimental parameters and data analysis. Frequency was characterized using the two-sample (Allan) variance widely used in time and frequency metrology. To minimize self-heating, yet with an acceptable signal-to-noise ratio, a microwave power of $-10 \text{ dBm}$ was found to be optimal. A slight improvement of the quality of the fitted lineshape compared with our previous work [17] was obtained using a quadratic function for the background rather than a linear one. The use of temperature cycling between 5 K and 25 K turned out to improve the reproducibility of frequency measurements. Fine control of the resonator temperature is achieved using a calibrated rhodium-iron resistance thermometer read using an AC resistance bridge instead of an 8½ digit multimeter. The local frequency reference was a GPS referenced commercial rubidium clock. It was essential to use a roof-based outdoor antenna for the GPS system. Frequency resolution was also improved by the introduction of two microwave pre-amplifiers in series, albeit close to the network analyzer located at room temperature. Increased performance is expected with the installation of cryogenically cooled pre-amplifiers located closer to the microwave resonator.

In this way, for an integration time of three hours, microwave resonance frequency measurements with a stability at the $10^{-12}$ level were demonstrated, which represents an almost 20-fold enhancement compared with those of our previous work [17]. The present observations could help in other high-stability, low-uncertainty frequency measurements in copper microwave cavities, not only for improving thermodynamic temperature measurement in SPRIGT, but also for other precise measurements, where high-stability and low-uncertainty microwave measurements are necessary.

The final uncertainty achieved using SPRIGT in our laboratory is equal to or better than 0.17 mK in the range 5 K to 25 K. An extensive description of the uncertainty budget will be presented in a future publication. As far as frequency measurements are concerned (i.e. the subject of the present work), had they not been possible at the $10^{-12}$ level, the subtle influence of microwave cables and feed-throughs would perhaps not have been detected as early as it was. The effect of thermal cycling is not one of uncertainty but rather of potential mechanical instability.

Acknowledgments

This work is supported financially by the National Key R&D Program of China (Grant No. 2016YFE00204200), the National Natural Science Foundation of China (Grant No. 51627809), the International Partnership Program of the Chinese Academy of Sciences (Grant No. 1A1111KYSB20160017), and the EMRP project Real-K (Grant No. 18SIB02). The author Changzhao Pan was supported by funding provided by the Marie Skłodowska-Curie Individual Fellowships-2018 (Grant No. 834024). The authors gratefully acknowledge Richard Rusby from the National Physical Laboratory, UK for sharing his long experience and constant advice on temperature measurements. We are deeply grateful to Wei Wu from the City University of Hong Kong, China for his kind reading and helpful suggestions with the manuscript. We would also like to thank Kun Liang from the National Institute of Metrology, China, and Chongxia Zhong from the Beijing Institute of Metrology, China for their helpful discussion on time reference.

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