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# Calibration of ruby ( $\text{Cr}^{3+}:\text{Al}_2\text{O}_3$ ) and $\text{Sm}^{2+}:\text{SrFCl}$ luminescence lines from the melting of mercury: constraints on the initial slopes

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**Abstract:** We have measured the luminescence shifts of the ruby's R1- and R2-lines and the line of  $^5\text{D}_0 \rightarrow ^7\text{F}_0$  from  $\text{Sm}^{2+}:\text{SrFCl}$  corresponding to the melting pressure of mercury that is recommended by the AIRAPT task force as an International Practical Pressure Scale (IPPS). The linear coefficients of the pressure dependence of the R1-, R2-lines, and the luminescence line of  $\text{Sm}^{2+}:\text{SrFCl}$  are determined to be  $0.3722 \pm 0.002$  nm/GPa,  $0.3796 \pm 0.002$  nm/GPa, and  $1.123 \pm 0.002$  nm/GPa, respectively. The results not only put tight constraints on the initial slopes of ruby and  $\text{Sm}^{2+}:\text{SrFCl}$  gauges, but also link the luminescence-based pressure gauges to the more fundamental primary piston gauges.

## 1. Introduction

Since the invention of ruby ( $\text{Cr}^{3+}:\text{Al}_2\text{O}_3$ ) luminescence shift for pressure measurement [1] a half-century ago, ruby sensor as a pressure gauge has been widely used for pressure determination in experiments using diamond anvil cell (DAC) and other optically transparent pressure devices [2-4]. At pressures below  $\sim 20$  GPa, the shift of the R1 luminescence line may be approximated as linear [2]. At higher pressures, the R1-line shift displays significantly nonlinear behavior, which has been the main concern in developing an accurate extended ruby gauge. Among published ruby gauges [5-17] in the last 15 years, they are in reasonable mutual agreement within 2.5% in a pressure range up to 150 GPa. Recently, a ruby gauge (called Ruby2020) [17] has been proposed by an AIRAPT task group using a second-order polynomial form:

$$P \text{ (in GPa)} = \mathbf{A} \Delta\lambda / \lambda_0 (1 + \mathbf{B} \Delta\lambda / \lambda_0), \quad (1)$$

with the initial slope  $\mathbf{A}$  of  $1870 \pm 10$  GPa and the parameter  $\mathbf{B}$  of  $5.63 \pm 0.03$ , where  $\Delta\lambda$  is the ruby R1-line shift in nm, and  $\lambda_0$  is the wavelength of the R1-line near 694.25 nm at ambient condition.

Calibration at low pressures ( $< 10$  GPa) is dominantly affected by the initial slope in Eq (1),  $\mathbf{A} = \left( \frac{dP}{d \ln \lambda} \right)_{P \rightarrow 0}$ . Even though a precision in pressure using a ruby gauge can be 0.01 GPa [11, 18], its initial slope is not well constrained. The published  $\mathbf{A}$ -values were either fixed [3, 4, 10, 19] based on the linear coefficient averaged over a pressure range of 0-20 GPa [2], which tends to over-estimate the initial slope because of nonlinearity, or back-extrapolated using a large pressure range [5-7, 14, 15, 17, 20], which may involve unknown uncertainties.

In principle, reference points with their pressures calibrated by primary piston gauges can be used not only for tightly constraining the initial slope of ruby gauge but also for bridging the ruby gauge with the more fundamental primary gauges. Using six reference points, Forman et al [1] determined the ruby's R1-line shift with a linear coefficient of  $0.372 \pm 0.014$  nm/GPa with a relative uncertainty of  $\pm 3.8\%$ . Based on the melting of pure H<sub>2</sub>O, Grasset [18] calibrated the ruby's R1-line shift in a pressure range of 0-1 GPa, resulting in a linear coefficient of  $0.371 \pm 0.006$  nm/GPa with a relative uncertainty of  $\pm 1.6\%$ . Here, we report the calibration of the ruby luminescence line shift from the well-calibrated melting line of mercury which has a high precision of less than  $\pm 0.2$  MPa [21, 22]. The first AIRAPT task group on the International Practical Pressure Scale (IPPS) recommended the mercury melting line [21] be used as a pressure standard up to 1.2 GPa. Later the melting line was modified in 1991 [22] with reference to the then new International Temperature Scale of 1990 (ITS-90) [23].

Luminescence of Sm<sup>2+</sup> in host crystals (SrFCl, BaFCl) is widely used for pressure determination at low pressures (<10 GPa), largely because the pressure coefficient is almost three times larger than that of the ruby doublet [24, 25]. Sm<sup>2+</sup>:SrFCl or Sm<sup>2+</sup>:BaFCl is also used as a replacement for ruby sensors in sapphire anvil cells, since sapphire often contains trace amount of Cr<sup>3+</sup>, interfering with ruby luminescence signals. In addition, the luminescence spectrum of Sm<sup>2+</sup>:SrFCl or Sm<sup>2+</sup>:BaFCl shows a singlet line, well separated from the other lines, with a narrow linewidth. Due to the large pressure coefficient and the narrow linewidth, the precision of pressure measurement significantly improves, by more than three times with respect to the ruby gauge. In this paper, we also report the calibration of Sm<sup>2+</sup>:SrFCl luminescence line shift from the melting line of mercury.

## 2. Experimental procedures

We used a diamond anvil cell (DAC) with anvils of a culet size of 450  $\mu\text{m}$  in diameter. In all runs, we used rhenium gaskets and pre-indented each with the DAC to a thickness of 60-80  $\mu\text{m}$ . A  $\phi 275$   $\mu\text{m}$  hole was drilled at the center of the indentation, and subsequently loaded with a couple of ruby spheres or Sm<sup>2+</sup>:SrFCl chips (about  $\sim 3$ -10  $\mu\text{m}$  in size) and the mercury sample, well separated from the rhenium gasket, in the hole. The mercury sample is from Sigma-Aldrich (electronic grade, 99.9999% trace metals basis, product number 294594). The ruby spheres (3600 ppm-Cr<sup>3+</sup>:Al<sub>2</sub>O<sub>3</sub>, from BETSA) are annealed to relax internal strain [26]. The Sm<sup>2+</sup>:SrFCl sample contains nominally 0.1 at% Sm<sup>2+</sup> and comes from the same batch as the material studied in refs. [24] and [25]. A description of the synthesis conditions can be found in ref. [27]. We mechanically crushed a Sm<sup>2+</sup>:SrFCl crystal into small chips. These chips were used without annealing. The sample chamber was then loaded with neon gas as pressure transmitting medium (Fig. 1). In the pressure range of this study (<2 GPa), neon remains liquid, providing a hydrostatic environment. For precise controls of compression and decompression, we used a double-sided membrane technique [28]. The membrane pressure, which drives the DAC, was digitally changed by a controller (GE/Druck Model: Pace 5000). The compression or decompression of the DAC was applied by controlling membrane pressure over time with a step of 70-345 Pa/s (0.01-0.05 psi/s).

We also used an empty cell loaded with a ruby sphere from the same batch and a Sm<sup>2+</sup>:SrFCl chip, i.e., loaded in a drilled gasket hole without any pressure on the DAC. The luminescence signals from the empty cell ruby and Sm<sup>2+</sup>:SrFCl crystals were used as references at ambient pressure. Immediately

before and after each experimental run, the ambient pressure points were measured multiple times with their average number taken as the initial wavelength  $\lambda_0$ .

The luminescence measurements were performed using an Acton SpectroPro-300i spectrograph with a grating of 1800 grooves/mm, coupled with a PIXIS:400BR Digital CCD Camera System. A laser (Spectra Physics model Excelsior-532-300) of wavelength 532 nm was used for exciting luminescence sensors with a fixed laser power (<3 mW) at the sample position. The small laser power minimized the effect of local heating [26]. Typical luminescence spectra from the ruby spheres and the  $\text{Sm}^{2+}:\text{SrFCl}$  chips in the DAC are shown in Fig. S1 and Fig. S2, respectively. With a collection time of 0.25 s, the luminescence signals from both ruby and  $\text{Sm}^{2+}:\text{SrFCl}$  crystal are strong and can be well fitted to determine the peak positions with small uncertainties (< $10^{-5}$  nm) from the fitting statistics. In the fitting procedures, we used the Moffat function, defined as:  $I = a_0/(u+1)^{a_3}$ , where  $I$  is the intensity,  $u = [(x - a_1)/a_2]^2$ ,  $x$  is the wavelength, and  $a_1$ ,  $a_2$ , and  $a_3$  are fitting parameters. Uncertainties in peak position from peak-fitting are negligible (< $1.0 \times 10^{-5}$  nm) compared to those from other sources, such as repeatability and data scatters.

In a typical run, we first compressed the DAC via the membrane controller at a given step until the sample pressure reached  $\sim 1.5$  GPa. We then decompressed the DAC at a similar rate to a pressure of  $\sim 0.9$  GPa. In both compression and decompression paths, we continuously measured luminescence signals every 0.25 seconds, while the mercury sample crossed its melting point [22]. In a pressure interval of 0.6 GPa, our compression (or decompression) rates allowed us to record 600-6000 luminescence spectra, providing fine recordings of the pressure conditions in the sample chamber.

The DAC temperature was monitored by a K-type thermocouple attached to the cell. We applied a correction of  $-0.1$  °C to the thermocouple readings, based on the temperature measurement of the thermocouple in the icy water, which displayed  $0.1$  °C. The average temperatures during the ruby runs and  $\text{Sm}^{2+}:\text{SrFCl}$  runs were measured to be  $23.75 \pm 0.05$  °C and  $23.0 \pm 0.05$  °C, respectively. According to the melting line of mercury [22],

$$P \text{ (MPa)} = 19.32845d + 0.0018333d^2 + 0.000059791d^3 \quad (2)$$

where  $d = 273.15 + t - 234.3156$ , and  $t$  is temperature in °C, the melting pressures at those temperatures are  $1231.5 \pm 1.0$  MPa for the ruby runs and  $1216.3 \pm 1.0$  MPa for the  $\text{Sm}^{2+}:\text{SrFCl}$  runs in this study. The uncertainty of  $\pm 1.0$  MPa is largely from the uncertainties in temperature ( $\pm 0.05$  °C). Note that the melting pressures here are both slightly above the upper pressure limit of 1200 MPa in the reported melting line of mercury [22]. As discussed by Molinar et al [21, 22], the third-order polynomial form (Eq. 2) precisely represents their experimental data covering a pressure range of 965 MPa (227.4 - 1192.5 MPa), with a very small residual standard deviation ( $\sigma$ ) of 0.06 MPa. If we assume a  $3\sigma$  value (0.18 MPa) as the uncertainty in the extrapolation of the melting curve (Eq. 2) by 32 MPa to pressures up to 1232 MPa, the total uncertainties are still close to  $\pm 1.0$  MPa, which is predominantly from the uncertainties in temperature ( $\pm 0.05$  °C).

### 3. Results and discussion

Fig. 2a shows the measured shifts of the ruby's R1-line ( $\Delta\lambda = \lambda_p - \lambda_0$ , here  $\lambda_p$  is the measured wavelength at high pressure and  $\lambda_0$  is the reference at ambient pressure), as we slowly compressed and decompressed the DAC. The rate of compression or decompression in each run was fixed by setting a

step of either 70 Pa/s or 345 Pa/s using a membrane controller. Under compression paths, the mercury sample undergoes a liquid to solid transition (freezing). We observe an overall increase in  $\Delta\lambda$  with time under compression. Notably, when the shift reaches a certain level ( $\sim 0.475$  nm), there is a sudden drop in  $\Delta\lambda$  by  $\sim 0.012$  nm. Because the sample chamber's volume is nearly constant in the pressure range of this study, this large drop is likely associated with a sudden shrinkage of the mercury sample upon freezing, and a consequent expansion of the remaining materials in the sample chamber (Ne and ruby). Although the drop in  $\Delta\lambda$  provides a clear indication of the freezing of mercury, the corresponding pressure may be slightly above the melting pressure of mercury, because liquid mercury can often be super-pressurized [21] under compression, a phenomenon similar to supercooled liquid below melting temperature.

Under decompression, the solid mercury at high pressure undergoes melting via a path of decompression. As shown in Fig. 2a, we observe an obvious plateau in each decompression run of all our experiments. The plateaus are likely caused by the mercury's volume expansion upon melting. Similar to latent heat under heating a solid to melting, the observed plateaus here reflect a latent component on melting, which may be referred as "latent compression". Apparently, the melting process is slower than the freezing. At the time scale in our experiments (seconds), superheating (or over-decompression) of liquid mercury is unlikely. Therefore, we assign the plateaus to the onset of the mercury melting. Because of the well calibrated melting curve of mercury [21, 22], the plateaus thus provide a precise calibration for the  $\Delta\lambda$  at the mercury melting pressure (Table 1). In Fig. 2b, similar plateaus are observed for the ruby's R2-line, with the numerical data also listed in Table 1.

In the runs of  $\text{Sm}^{2+}:\text{SrFCl}$ , we do not observe clear pressure drops under compression. The plateaus under decompression are not as clear as those in the experiments on ruby (Fig. 3), which is likely related to the relatively small volumes of the mercury sample in the  $\text{Sm}^{2+}:\text{SrFCl}$  experiments. Nonetheless, the turning points from the mercury melting are still clearly visible, as illustrated in the derivative plots in Fig. 3. Using the data points in the vicinity of the turning points, we can define the shift of the  $\text{Sm}^{2+}:\text{SrFCl}$  luminescence line  ${}^5\text{D}_0 \rightarrow {}^7\text{F}_0$  corresponding to the mercury melting (Table 1).

**Table 1: Shifts of luminescence lines corresponding to the melting of mercury at room temperature**

Ruby data	Cell1 Run 1	Cell1 Run 2	Cell1 Run 3	Cell2 Run 4	Cell2 Run 5	Mean of all runs (nm)
Number of data points on the plateaus	60	71	75	16	16	
Rate of membrane pressure (in Pascal per second)	70	70	70	345	345	
<b>Ruby R1 shift:</b> Mean value (nm) Standard deviation (nm)	0.4559 $6.7 \times 10^{-4}$	0.4597 $6.4 \times 10^{-4}$	0.4580 $6.3 \times 10^{-4}$	0.4595 $5.8 \times 10^{-4}$	0.4586 $7.2 \times 10^{-4}$	0.4584 $\pm$ 0.0015
<b>Ruby R2 shift:</b> Mean value (nm) Standard deviation (nm)	0.4650 $7.8 \times 10^{-4}$	0.4675 $7.5 \times 10^{-4}$	0.4661 $6.0 \times 10^{-4}$	0.4690 $5.8 \times 10^{-4}$	0.4680 $9.0 \times 10^{-4}$	0.4671 $\pm$ 0.0016

<b>Sm<sup>2+</sup>:SrFCl data</b>	Cell1 Run1	Cell2 Run2				
Number of data points on the plateaus	3	20				
Rate of membrane pressure (in Pascal per second)	70	70				
<b><sup>5</sup>D<sub>0</sub>→<sup>7</sup>F<sub>0</sub> line shift</b>						
Mean value (nm)	1.3663	1.3665				1.3664±0.0005
Standard deviation (nm)	6×10 <sup>-4</sup>	3.6×10 <sup>-4</sup>				

Therefore, in a narrow pressure range up to 1.2 GPa, we can determine the linear coefficients,  $\Delta\lambda/\Delta P$ , from the measured shifts of these luminescence sensors (Table 2). The uncertainty in pressure ( $\Delta P$ ) is small, about  $\pm 1$  MPa from the uncertainties in temperature ( $\pm 0.05$  °C). The uncertainty in  $\Delta\lambda$  consists of two major components: those from  $\lambda_p$  and  $\lambda_0$ . From the five runs in the ruby experiments (Table 1), the uncertainty in  $\lambda_p$  is estimated to be  $\sim \pm 0.0016$  nm (Table 1). Note that the precision in  $\lambda_p$  in each run is  $< \pm 0.0001$  nm (Table 1). The larger uncertainty from multiple runs is mainly caused by the repeatability of the spectrometer. A similar level of uncertainty is also obtained from the multiple measurements of ruby spheres in the empty cell before and after the experimental runs, which is  $\pm 0.0018$  nm. Combining these two components, we obtain a total uncertainty in  $\Delta\lambda$  of  $\sim \pm 0.0024$  nm in the experiments on ruby. The linear coefficients for ruby's R1 and R2 lines are then determined to be  $0.3722 \pm 0.002$  nm/GPa and  $0.3796 \pm 0.002$  nm/GPa, respectively (Table 2).

Because of only two runs in the Sm<sup>2+</sup>:SrFCl experiments, it is difficult to directly estimate the uncertainty in  $\lambda_p$ . However, due to the narrow linewidth of the luminescence line in Sm<sup>2+</sup>:SrFCl, the uncertainty in  $\Delta\lambda$  is unlikely to exceed that in ruby's experiments. This is reflected from the multiple measurements of the Sm<sup>2+</sup>:SrFCl chip in the empty cell before and after experimental runs, which gives an uncertainty in  $\lambda_p$  of  $\pm 0.0016$  nm. Considering that the uncertainty in  $\Delta\lambda$  is largely limited by the repeatability of the spectrometer, here we use a similar level of uncertainty of  $\pm 0.0024$  nm as that in ruby's experiments. The linear coefficient for the Sm<sup>2+</sup>:SrFCl luminescence line (<sup>5</sup>D<sub>0</sub>→<sup>7</sup>F<sub>0</sub>) is determined to be  $1.123 \pm 0.002$  nm/GPa.

The obtained linear coefficient of ruby's R1 line ( $0.3722 \pm 0.002$  nm/GPa) is in excellent agreement with that of Grasset [18] ( $0.371 \pm 0.006$  nm/GPa) and that of Forman et al [1] ( $0.372 \pm 0.014$  nm/GPa). Both of the previous studies [1, 18] used reference pressure points that are calibrated from the primary piston gauges (see Table 2). The relative uncertainty in this study is  $\pm 0.5\%$ , compared to  $\pm 1.6\%$  [18] and  $\pm 3.8\%$  [1] in previous studies. The linear coefficient of ruby's R2-line ( $0.3796 \pm 0.002$  nm/GPa) is in reasonable agreement with those in previous studies [1, 18] (Table 2). The relative uncertainty ( $\pm 0.5\%$ ) is significantly improved, compared to  $\pm 2.4\%$  [18] and  $\pm 3.7\%$  [1] in previous studies.

As far as we know, no calibration studies have been reported on the  $\text{Sm}^{2+}:\text{SrFCl}$  luminescence line shift based on pressure reference points from primary piston gauges. The line shifts of  $\text{Sm}^{2+}:\text{SrFCl}$  chips were measured together with ruby sensors [24, 25] up to a pressure over 10 GPa, from which the pressure dependence of the line positions was defined. The obtained linear coefficient in this study (Table 2) is calibrated against a reference point of the mercury melting at room temperature [21, 22] in a narrow pressure range of 0-1.2 GPa.

The shifts of luminescence lines against pressure in ruby and  $\text{Sm}^{2+}:\text{SrFCl}$  display clear nonlinear behavior above  $\sim 20$  GPa and  $\sim 5$  GPa, respectively. Among various forms to account for the nonlinear term [11], a polynomial form to the second order (Eq. 1) is widely used. According to Eq. 1, the nonlinear term at the mercury's melting pressure can be estimated to be 0.0046 GPa for the ruby's R1 line, based on the parameters in Ruby2020 [17]. The nonlinear term is small,  $\sim 0.4\%$  at 1.2 GPa. Then, after correcting the nonlinear term, we can determine the initial slope  $\mathbf{A}$  in Eq (1) to be  $1858 \pm 10$  GPa. The  $\mathbf{A}$  value here is smaller than, but still comparable to, that of  $1870 \pm 10$  in Ruby2020 [17] within uncertainties. Similarly, for the  $\text{Sm}^{2+}:\text{SrFCl}$  line, the nonlinear term at the mercury's melting pressure is estimated to be 0.0121 GPa (or  $\sim 1\%$  at 1.2 GPa), based on the data in Fig. S3. Thus, the initial slope  $\mathbf{A}$  in Eq (1) for the  $\text{Sm}^{2+}:\text{SrFCl}$  luminescence line is determined to be  $620.6 \pm 1.1$  GPa.

**Table 2: The determined linear coefficients and initial slopes in Eq (1)**

Emission line	Initial slope $\mathbf{A}$ (GPa) back-extrapolated according to Eq (1)	Linear coefficient $\Delta\lambda/\Delta P$ (nm/GPa)	Comment
Ruby-R1 $\text{Cr}^{3+}:\text{Al}_2\text{O}_3$	$1858 \pm 10^*$  *The parameter $\mathbf{B}$ of 5.63 in Eq (1) is from Ruby2020 [17]	$0.3722 \pm 0.002$	This study
		$0.371 \pm 0.006$	Grasset 2001 [29]: melting of $\text{H}_2\text{O}$ to 0.8GPa
		$0.372 \pm 0.014$	Forman et al 1972 [1]: up to 2.2 GPa using data of melting of $\text{CCl}_4$ , $\text{H}_2\text{O}$ , n- $\text{C}_7\text{H}_{16}$ , $\text{C}_2\text{H}_5\text{Br}$ , and of solid-solid transitions in $\text{CCl}_4$ (III-IV) and $\text{H}_2\text{O}$ (VI-VII)
Ruby-R2 $\text{Cr}^{3+}:\text{Al}_2\text{O}_3$		$0.3796 \pm 0.002$	This study
		$0.377 \pm 0.009$	Grasset 2001 [29]
		$0.406 \pm 0.015$	Forman et al 1972 [1]

Sm <sup>2+</sup> :SrFCl	620.6±1.1**	1.123±0.002	This study
	**The parameter <i>B</i> of -5.0 is used, based on data in Fig. S3	1.106±0.005	Lorenz et al 1994 [24]
		1.10	Shen et al 1991 [25]

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