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Verification of the hydrostatic weighing system with existing gold purity instruments

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Abstract. Determination of gold bar purity remains one of the most challenging tasks in gold industry. Currently, the existing instruments have a limitation to determine the purity, which in turn have produced inaccurate gold purity measurement. For example, X-ray fluorescence (XRF) technique has a limitation to penetrate the gold bar and XRF was only used to determine the surface purity. Therefore, an improved non-destructive method and precise technique has been developed to determine the gold purity. Gold density measurement can verify the purity of the gold and in this study, the density of gold bar was calculated using a custom made hydrostatic weighing system (HWS). Several measurements of density were carried out for gold bar and tungsten bar. Using HWS, the density of gold bar and tungsten bar were 19.268 g/ml and 19.206 g/ml respectively. The expanded uncertainty was also evaluated and the value was 0.006 g/ml. The new developed HWS method was also verified with existing density measurement instruments where the same density results has been obtained but the existing instruments have produced bigger uncertainty of 0.2 g/ml. Thus, this study demonstrates that the developed HWS is appropriate to be used to measure the density of gold bar accurately. Furthermore, the new developed system of HWS have shown better measurement with smaller uncertainty and hence the improvement in gold bar purity measurement.

1. Introduction

Gold, silver, platinum and palladium are types of the precious metal and among these metal, gold remains the world's most precious commodities [1, 2]. For several centuries, it continues to be a secure investment for most of people. A gold with high purity is the most expensive metal therefore, its purity must be analysed through highly accurate and precise method [3]. The inspection and verification of the gold content in gold trading is also significant due to gold price is based on gold purity [4]. Pawn broking industry also need to measure the gold purity due to the requirement to pay the same value of gold to borrower by the pawn broker [5].

Presently, there are two main methods to determine the gold purity: Destructive method and Non-destructive method. Fire assay and Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) are the techniques in destructive method [6-11]. However, these techniques are inappropriate because the pawnbroker cannot cutting or drilling the gold items that want to be leased as stated in Pawnbroker Act 1972. For non-destructive methods, the equipment such as X-ray Fluorescence (XRF), needle, chemical solution, tri-electronic and touchstone can be used to determine the gold purity without destruct the sample [12-14].

Gold density measurement can verify the purity of the gold and because of this several density instruments such as densimeter and weighing balance with solid density accessories (WB) are capable to check the purity of gold. They are also non-destructive equipment, simple to use and cheaper to buy. Most pawn shops and jewellery shops use these two instruments, however, the densimeter and WB cannot detect the presence of tungsten in gold because the tungsten density is very close to that of gold. Distilled water is used as standard liquid for the densimeter and WB. The distilled water is placed in special container and the design of liquid container for densimeter and WB is unique. One of the special design of liquid container for densimeter is shown in Figure 1. To minimise the error of measurement, the temperature of distilled water must be similar to environment temperature. The gold mass in air is measured by placing the gold sample in the top of pan whilst the sample will be placed in the bottom of the pan to weigh the mass in the distilled water. Then, The density and purity of the gold sample is obtained from the display of densimeter and weighing balance.

These two density instruments are also economical, simple and non-destructive method but produced inaccurate density with uncertainty between 0.1 g/ml to 0.2 g/ml [15]. The density of pure gold and pure tungsten are 19.30 g/ml and 19.25 g/ml respectively [16, 17]. Therefore, it will be ineffective for any measurement setup that will not produce better uncertainty with two decimal places in order to differentiate pure gold and fake tungsten gold [17-20]. The failure to accurately measure the gold density has greatly affected the gold industry [21].

This method also has the problem arising from the surface porosity of gold sample as the gold sample in the liquid needs to be removed first and then will be immersed back for the next measurement [22]. This procedure will cause inconsistency due to air bubbles trapped in the sample and it will influence the liquid temperature and thus the uncertainty of measurement.
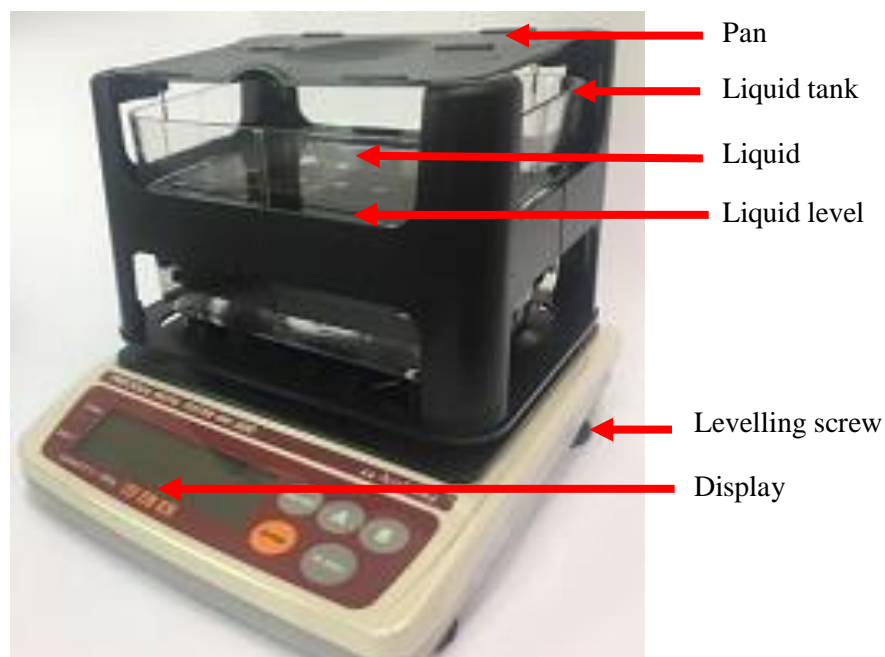

Figure 1. Composition of the densimeter

In order to get an accurate measurement, the liquid used in the densimeter and weighing balance is also must be traceable to the SI unit, where most pawnshops are not aware of this factor. The type of liquid used is also important where pure distilled water shall be used but some of the pawnshops did not replace the distilled water regularly. All these factors are important to make sure the gold purity

measurement is accurate [23-25]. Other parameters that can affects gold purity measurement are environment temperature, airflow, the liquid level in liquid container and levelling of densimeter.

Therefore, this study was conducted to explore and improve the hydrostatic weighing system by focusing on the procedure of density determination for gold purity check. The accuracy of the hydrostatic weighing system (HWS) needs to be improved at least to 0.01 g/ml due to reason that density difference between gold and tungsten is only 0.05 g/ml.

2. Method and Material
2.1. Densimeter
Densimeter is a combination of analytical balance and liquid container to measure the density of gold. Measureable range of the densimeter (AlfaMirage: Model GK-300) is 5 g to 300 g. The density readability of the densimeter is 0.1 g/ml. This method is based on Archimedes' principle where mass of the gold was determined by obtaining the different of the weight in air and the weight in distilled water. All measurements were repeated for ten times to ensure reading variability. Then using the weight difference, the density of the gold was calculated using the equation below:

$$\rho_{gold} = \frac{m_a \rho_l}{m_a - m_l} \tag{1}$$

Where:

ρ_{gold}	: Density of gold (g/ml)
ρ_l	: Density of liquid (g/ml)
m_a	: Mass in air (g)
m_l	: Mass in liquid (g)

2.2. Weighing balance with solid density (WB)
Figure 2 shows the weighing balance with density accessories consist of a support plate, glass beaker, thermometer and sample holder. The density accessories are easy to assembled and mounted to the weighing balance. The principle of this instrument is also based on Archimedes where the principle is similar to densimeter. Mass of gold sample in air and the distilled water were also measured to obtain the density of sample gold. The mass readability of the weighing balance is 0.0001 g. The density readability of the weighing balance is 0.00001 g/ml but the density accuracy is 0.1 g/ml [20].
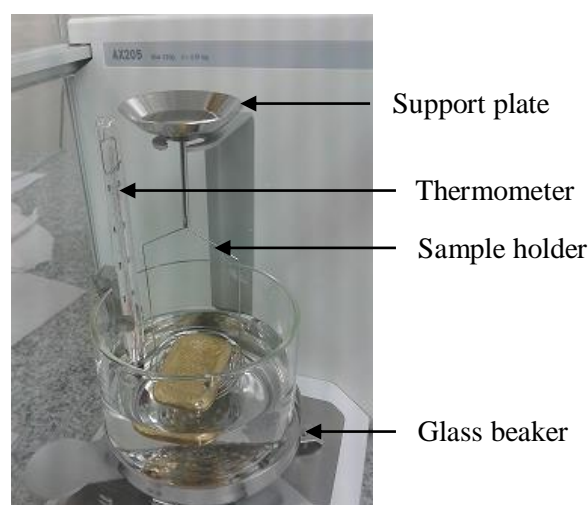

Figure 2. Weighing balance with density accessories.

2.3. Hydrostatic Weighing System (HWS)

In this study, HWS which has been set up and operates based on Archimedes' principle. In density measurement, the sample was weighed using an analytical balance in air and noted as R_1. Then, the sample was immersed in standard distilled water and weighed again as R_2. Volume of the sample, V' will change due to temperature, Δt changes and the volume thermal expansion, γ of the sample. So, volume of sample, V' is calculated as:

$$V' = \left(\frac{R_1 - R_2}{\rho_w - \rho_a} \right)(1 + \gamma \Delta t) \qquad (2)$$

Finally, the density of sample, ρ_c is calculated after include the volume thermal expansion as:

$$\rho_c = \frac{R_1}{V'} \qquad (3)$$

To ensure accurate calculation, all factors affecting the gold purity such as density of distilled water, ρ_w air density, ρ_a mass in liquid and liquid temperature were considered and they have been included in the equation (2). These parameters were also identified to evaluate the expanded uncertainty of measurement. Figure 3 shows the schematic diagram for HWS.

The sample was attached to the balance (Mettler Toledo; Model XP504 with capacity of 500g) using a sample holder and hooks. A digital thermometer (Model F200 with accuracy of 0.01°C) was used to measure the temperature of the distilled water in the sample bath [26]. The sample bath is a glass cylinder containing the liquid for thermal control and stability. The use of glass has enabled to view the immersion level of the sample as it is transparent. A chiller was used to stabilize the temperature of the distilled water. The air density was obtained from the equation that included air temperature, humidity and ambient pressure.
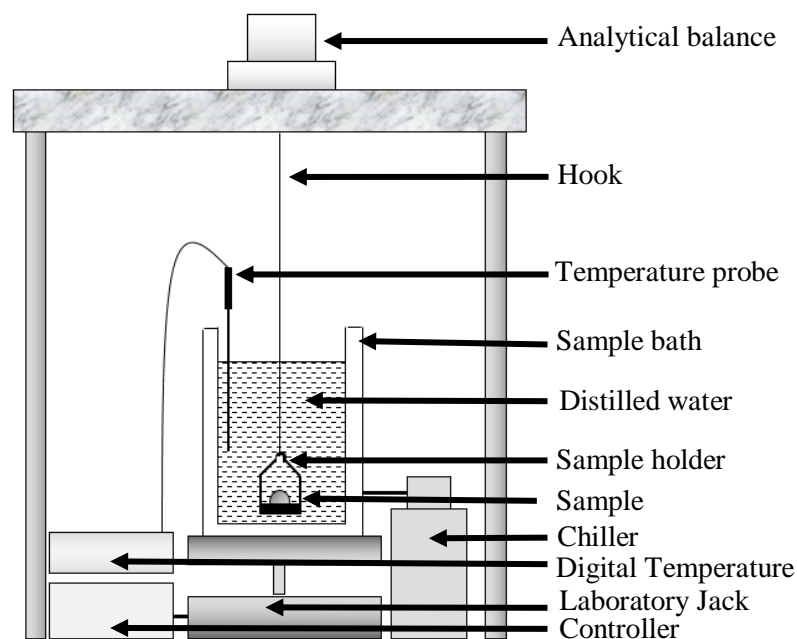

Figure 3. Schematic diagram of hydrostatic weighing system.

2.4. Material
Several samples of gold and tungsten bar with purity 99.9% were evaluated in this work due to the density of these materials is similar. So the purity and mass of gold and tungsten samples were measured using XRF (Thermo Scientific; Model ARL QUANT'X) and analytical balance (Mettler Toledo; Model XP504) respectively. Two groups of sample were measured as shown in Table 1. Sample S5 and S6 are gold samples with purity of 99.9% while S7and S8 are tungsten samples with purity of 99.9%.

Table 1. The information of the samples

Sample	Material	XRF, Purity (%)	Mass (g)	Remarks
S5	Gold	99.9	19.9990	Bar
S6	Gold	99.9	20.0113	Bar
S7	Tungsten	99.9	19.3875	Bar
S8	Tungsten	99.9	19.1537	Bar

2.5. Calculation of Performance Statistics
According to MS ISO/IEC 17043:2010 (Conformity Assessment-General Requirements for Proficiency Testing), the quality of each measurement is based on a calculated error normalised (E_n value) with respect to the stated uncertainty [27]. Hence, to ensure the measurements that were carried are reliable, E_n was calculated by comparing the result of expanded uncertainty from the uncertainty obtained by existing instruments. The calculation used is shown in the equation (4):

$$E_n = \frac{HWS - REF}{\sqrt{U_{HWS}^2} + \sqrt{U_{REF}^2}} \tag{4}$$

where:
|	
HWS	: Mean measured by HWS
REF	: Mean measured by existing instruments (Densimeter and WB)
U_{HWS}	: Expanded uncertainties of HWS
U_{REF}	: Expanded uncertainties of existing instruments (Densimeter and WB)
$|E_n| \leq 1$	: Indicates the satisfactory performance of the HWS
$|E_n| > 1$	: Indicates the unsatisfactory performance of the HWS and requires root cause analysis followed by corrective actions.

3. Results and discussion
The newly developed HWS needs to be verified with other existing instruments. Verification of the HWS was done by comparing its performance with currently existing measurement instrument that are densimeter (Alfa Mirage, GK 300) and WB (Mettler Toledo, AX205). Several samples of 99.9% gold (S5, S6) and 99.9% tungsten (S7, S8) were used to verify the HWS and existing instruments. Figure 5 show the density of samples and uncertainty of measurement results for S5, S6, S7 and S8 using three different instruments. Figure 4 shows the density measurement of samples by densimeter, WB and HWS. Figure (a) shows the density of sample S5 by using HWS, densimeter and WB are 19.268 g/ml, 19.3 g/ml and 19.27 g/ml respectively. Figure (b) shows the density of sample S6 by using HWS, densimeter and WB are 19.267 g/ml, 19.2 g/ml and 19.25 g/ml respectively. Figure (c) shows the density of sample S7 by using HWS, densimeter and WB are 19.205 g/ml, 19.2 g/ml and 19.20 g/ml respectively. Lastly, Figure (d) shows the density of sample S8 by using HWS, densimeter and WB are 19.208 g/ml, 19.2 g/ml and 19.27 g/ml respectively.

The expanded uncertainty for HWS, densimeter and WB are 0.006 g/ml, 0.2 g/ml and 0.2 g/ml respectively. A list of significant measurement uncertainties for HWS obtained from Equation (2) and (3) are given in the Table 2. The expanded uncertainty determined at 95% confidence level was 0.01 g/ml.

Table 2. Analysis of expanded uncertainty of HWS at 95% confidence level

| Parameter	Uncertainty (g/ml)
Mass in air (g)	0.00192
Air Density (g/ml)	0.00039
Liquid density (g/ml)	0.00001
Mass in liquid (g)	0.00214
Thermal Expansion (/°C)	0.00008
Liquid Temperature (°C)	0.00007
Combined standard uncertainty, u_c	0.0029
Expanded uncertainty, U	0.0058

From the results shown, it is clearly show that, for the same sample, the measurement obtained from HWS are consistent with the existing instruments. Using HWS method, the density of gold bar and tungsten bar were 19.268 g/ml and 19.206 g/ml respectively. This shows that, there is an improvement in new developed system of HWS that can be used to determine the purity of gold. Furthermore, the uncertainty evaluation for HWS is much smaller compared to the densimeter and WB and hence more accurate.
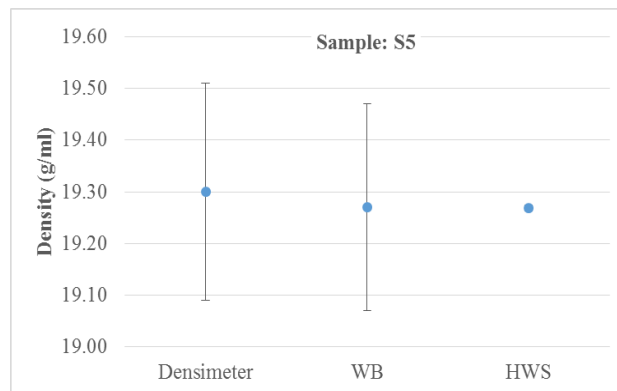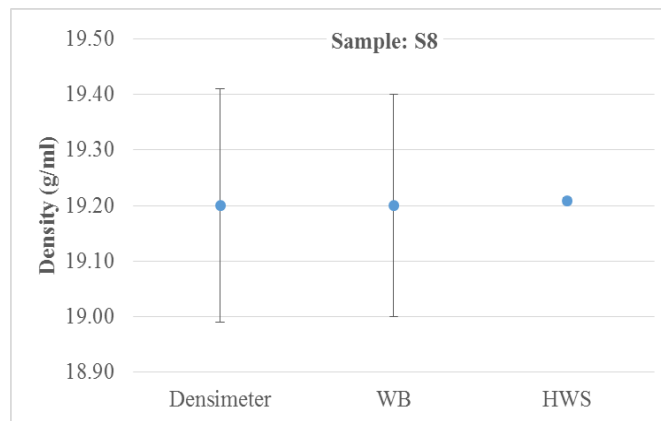

(a). Density measurement of S5 by density measurement instruments
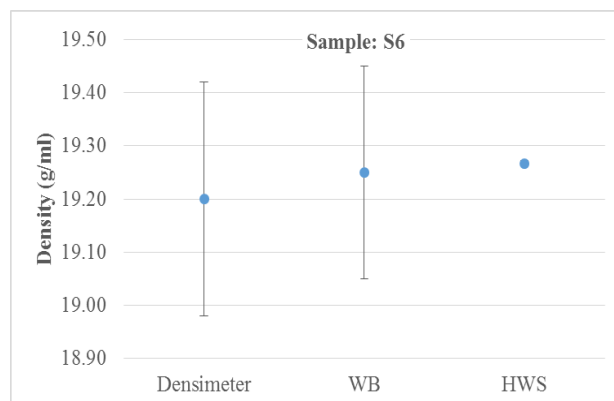
(b). Density measurement of S6 by density measurement instruments
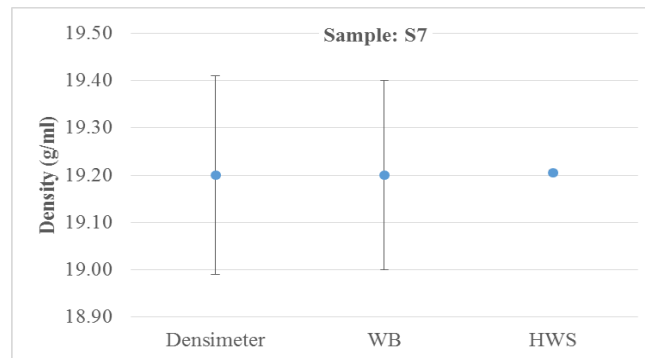
(c). Density measurement of S7 by density measurement instruments

(d). Density measurement of S8 by density measurement instruments

Figure 4. Density measurement of samples by densimeter, WB and HWS.

The quality of HWS performance is based on the calculated error normalized (E_n value) with respect to the result of uncertainty as depicted in Figure 4 [28]. E_n value was calculated by comparing the result between HWS and existing instruments. Table 3 shows the E_n value of the HWS by comparing with densimeter and WB. E_n value of the HWS is below than 1, so it indicates the satisfactory performance of the HWS. Therefore, the results obtained are acceptable and thus HWS is reliable for density gold measurement.

Table 3. E_n value of the HWS by comparing with densimeter and WB

Sample	Densimeter	WB
S5	0.15	0.01
S6	0.30	0.08
S7	0.02	0.02
S8	0.04	0.04

4. Conclusions

New findings in this study has enabled HWS to be proposed as an instrument to determine the density of gold and tungsten accurately. Therefore, HWS was successfully developed to differentiate gold and tungsten and it was also verified with existing instruments. Furthermore, the quality of HWS performance is also comparable with the existing density instrument depicted by the E_n value.

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