HIGH ACCURACY MEASUREMENT OF GOLD PURITY USING HYDROSTATIC WEIGHING SYSTEM

MOHD FAZRULHISYAM BIN MOHD NOR

UNIVERSITI TEKNOLOGI MALAYSIA

HIGH ACCURACY MEASUREMENT OF GOLD PURITY USING HYDROSTATIC WEIGHING SYSTEM

MOHD FAZRULHISYAM BIN MOHD NOR

A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy

Faculty of Science
Universiti Teknologi Malaysia

JUNE 2020

DEDICATION

To my beloved parents

Mohd Nor bin Jaafar
Fatimah bte Ithnin
Sulaiman bin Suhil
Siti Nor Sham bte Mohd Hayram

To my beloved wife and children
who always there for me

Noor Zarina bte Sulaiman
Ahmad Farhan bin Mohd FazrulHisyam
Ahmad Farihin bin Mohd FazrulHisyam
Khairun Naajihah bte Mohd FazrulHisyam

You are always in my mind...

ACKNOWLEDGEMENT

I wish to express my sincere gratitude to my supervisor, Dr Abd Rahman Bin Tamuri and my co-supervisor, Dr. Abd Khamim bin Ismail for his encouragement, guidance, criticism and friendship. They did not only provide me with academic guidance but have also been of great support and encouragement to me in some of my blink moments during the period of this study.

My greatest appreciation goes to Mrs Hafidzah Othman, Dr Faridah Hussain and Dr Osman Hj Zakaria for their support in this project. My fellow colleagues such as Mr Syazwan Muhamad Ayub and Dr Siti Zaleha Mohd Nor should also be recognized for their support and assistance in various occasions. Their views and opinions are indeed useful.

I am also indebted to the National Metrology Institute of Malaysia (NMIM) and SIRIM for their financial support.

And last but not least, special thanks go to all my family members who have given me strength and moral support in completing this thesis.

ABSTRACT

Currently, existing instruments have limitation to determine the purity of gold bar and the method used is not reliable to detect the existence of tungsten in bar. Therefore, a new technique with high accuracy and non-destructive method has been developed to determine the purity of gold bar. In this study, three approaches were considered in the construction of hydrostatic weighing system (HWS); establishment of location and environmental condition, establishment of the density measurement of distilled water and establishment the mass measurement of samples. The X-ray fluorescence (XRF) analysis was used to determine the surface purity, while the HWS was used to determine the density of gold bar. The standard sample of gold bar, tungsten bar and fake gold bar were used in this study. Experimental results show that the density of gold bar is proportional to the purity of gold bar. The purity of 99.99% and 99.9% gold has density of 19.293 g/ml and 19.268 g/ml respectively. However, for tungsten, the density of 99.9% tungsten was only 19.206 g/ml with the expanded uncertainty of the density of 0.006 g/ml. Thus, this study demonstrates that the developed HWS is capable to be used to measure the density of gold bar accurately and hence it could detect the presence of tungsten in gold bar.

ABSTRAK

Pada masa ini, instrumen yang sedia ada mempunyai batasan untuk menentukan ketulenan jongkong emas dan kaedah yang digunakan tidak mampu untuk mengesan kewujudan tungsten dalam jongkong. Oleh itu, suatu teknik baharu dengan kaedah ketepatan tinggi dan tanpa-musnah telah dibangunkan untuk menentukan ketulenan jongkong emas. Dalam kajian ini, tiga pendekatan telah dipertimbangkan dalam pembangunan sistem penimbangan hidrostatik (HWS); penetapan lokasi dan keadaan persekitaran, penetapan pengukuran ketumpatan air suling dan penetapan pengukuran jisim sampel. Analisis pendarfluor sinar-X (XRF) digunakan untuk menentukan ketulenan permukaan, sementara HWS digunakan untuk menentukan ketumpatan jongkong emas. Jongkong emas piawai, jongkong tungsten dan jongkong emas palsu telah digunakan dalam kajian ini. Hasil eksperimen menunjukkan bahawa ketumpatan emas adalah berkadar terus dengan ketulenan emas. Ketulenan emas 99.99% dan 99.9% masing-masing mempunyai ketumpatan 19.293 g/ml dan 19.268 g/ml. Walau bagaimanapun, untuk tungsten, ketumpatan tungsten 99.9% hanya 19.206 g/ml dengan ketidakpastian ketumpatan adalah 0.006 g/ml. Oleh itu, kajian ini menunjukkan bahawa HWS yang dibangunkan mempunyai keupayaan untuk digunakan bagi mengukur ketumpatan jongkong emas dengan tepat dan dengan itu dapat mengesan kehadiran tungsten dalam jongkong emas.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

BAM	-	Bundesanstalt für Materialforschung und prüfung
BBC	-	British Broadcasting Corporation
EDXRF	-	Energy dispersive systems
HWS	-	Hydrostatic Weighing System
ICP	-	Inductively Couple Plasma
ICP-OES	-	Inductively Couple Plasma Optical Emission Spectrometry
KRISS	-	Korea Research Institute of Standards and Science
MINT	-	Malaysian Nuclear Agency
MyRM	-	Malaysian Reference Material
NMI	-	National Metrology Institute
NMIJ	-	National Metrology Institute of Japan
NIST	-	National Institute Standard and Technology
NMIM	-	National Metrology Institute of Malaysia
INPUT	-	Institute of Research and Consultancy YaPEIM
ISO	-	International Organization for Standardization
SI	-	Standard International
SKM	-	Suruhanjaya Koperasi Malaysia
SRM	-	Standard Reference Material
UK	-	United Kingdom
WDXRF	-	Wavelength dispersive system
WB	-	Weighing balance with solid density accessories
XRF	-	X-ray Fluorescence
YaPEIM	-	Koperasi Yayasan Pembangunan Ekonomi Islam Malaysia

LIST OF SYMBOLS

f	-	Frequency of the ultrasonic wave
v	-	Velocity of the material
λ	-	Wavelength of the ultrasonic wave
ρ_{gold}	-	Density of gold
ρ_l	-	Density of liquid
m_a	-	Mass in air
m_l	-	Mass in liquid
$\rho_{24Carat}$	-	Density of pure gold
F_b	-	Buoyancy force
F_g	-	Gravitational force
g	-	Acceleration of gravity
m	-	Mass of the sample.
ρ_a	-	Air density
ρ_m	-	Density of the sample
V	-	Volume of the sample
F	-	Vertical force
F_A, F_L	-	Force on balance
R_1 ,R_2	-	Balance reading
ρ_w	-	Density of distilled water
ρ	-	Density of material/sample
t_w	-	Distilled water temperature
V_g	-	Volume of the sample
a	-	Length
b	-	Height
c	-	Width
V_1 , V_2 , V'	-	Volume of the sample
Δt	-	Temperature difference
γ	-	Volume thermal expansion
α	-	Liner thermal expansion
x	-	Mean measured by NMIM

X	-	Mean measured by reference laboratory
U_{lab}	-	Expanded uncertainties of NMIM
U_{ref}	-	Expanded uncertainties of reference laboratory
E_n	-	Reliability of the measurements
s	-	Standard deviation of a measurement
$\bar{x}$	-	Mean value
n	-	Total number of measurement reading
u_A	-	Standard uncertainty for Type A
v_{eff}	-	Effective degree of freedom
U	-	Expanded uncertainty
k	-	Coverage factor
U_c	-	Combined standard uncertainty
BS	-	Bath stability

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.0 Background

Precious metal is a rare metal and has a high value. Gold, silver, platinum and palladium are types of precious metals (Cayumil, Khanna et al. 2016; Ding, Zhang et al. 2019). Gold is a one of the world's most precious commodities and it remains to be a secure investment. A gold with high purity is the most expensive metal. Therefore, inspection and verification of the gold content in gold trading is significant due to gold price is based on gold purity (Bardi, Niccolai et al. 2008). Therefore, pawn broking industry have to measure the gold purity due to requirement to pay the same value of gold to borrower by the pawn broker (Ali, Johari et al. 2017).

The gold purity must be analyzed through highly accurate and precise method (Jotanovic, Memic et al. 2012). Carat is a unit for gold purity and it use in document standard such as Malaysian Standard and Indian Standard (IS2790 1999; MS1247 2005). The gold purity of 99.9%, 91.6% and 75.0% are of 24 Carat, 22 Carat and 18 Carat respectively. Other alloys or elements such as silver, zinc, copper, palladium and nickel must also be known. The different elements in gold will effect to gold colour (Cretu 1999).

The value of gold is depending on its purity; therefore, it is important to determine its purity. The density of 24 carat gold is 19.3 g/ml (Mercer 1992). Gold also can be easily bought and sold around the world (Kinneberg, Williams et al. 1998). Hence, some people can produce fake gold due to gold is universally accepted and valuable.

Fake gold was happened in Hieron II, king of Syracuse era (Heath 2002). Archimedes was assigned to check purity without cutting or drilling the King Hieron II new crown (Reese 2000). King Hieron II wants to assure that his new crown made from pure gold. During his era, there were many alloy of gold and silver in the market (Hughes 2005). It is show that the fake gold existed a long time ago. In modern era, tungsten was found plated with thin layers of gold (Arbutine 2008; Abdullah, Samsudin et al. 2015; Ismail, Sani et al. 2018). Tungsten is the only metal that has a density similar to the density of gold. Furthermore, gold is more expensive compare with tungsten. Therefore, tungsten is always used to produce the fake gold because of its density and a lot cheaper than gold.

Pawnbroker Act 1972 was introduced and has been gazette in Malaysia stated that, the gold that want to be leased cannot be destructed (Act81 1972; Dakian 2005). Pawnbroker Activity Guideline for Islamic pawn also stated that the pawnbroker must protect the gold from any damage (SKMGuideline 2013; Sulaiman, Kassim et al. 2014). Therefore, the pawnbroker in Malaysia is prohibited from cutting or drilling any jewellery made from gold or the gold bar which has been leased by customer.

There are four categories of Islamic pawn-broking: financial institutions, corporation sector, private company and stated link company (Shah 2018). The regulatory body that coordinate and standardize the activities of financial institutions, corporation sector, private company and stated link company activities are Bank Negara Malaysia, Suruhanjaya Koperasi Malaysia (SKM), Ministry of Housing and Local Government and SKM respectively. The main purpose of these regulatory bodies is to coordinate and standardize the Islamic pawn-broking activities to ensure the fair trade for public. However, the regulatory bodies do not have enough manpower and lack in technical knowledge to monitor the pawn activities especially in gold purity checking during the transactions.

There are destructive methods that can be used to detect tungsten in gold such as fire assay, Inductively Couple Plasma (ICP) or cutting cross the section of gold items (Hanrahan 1962; Brill and Wiedemann 1992; Brill 1997; Kinneberg, Williams et al. 1998; Karadjova, Arpadjan et al. 2000; Singh; Battaini, Bemporad et al. 2014). The method used to determine the gold purity are by using fire assay and Inductively

Coupled Plasma Optical Emission Spectrometry (ICP-OES) are also described in several International Standards and Standard Test Methods (ISO11426 1997; IS1418 2009; ISO15093 2015; ISO10378 2016). However, these methods will destruct the sample which against the Pawnbroker Act 1972 which clearly stated that the pawnbroker cannot cutting or drilling the gold items that wants to be leased.

All non-destructive methods can be determined the gold purity at the surface level, inside the sample or whole sample. It's difficult for instruments in non-destructive method to detect the presence of other metal in gold. X-ray Fluorescence (XRF), needle, chemical solution, tri-electronic and touchstone are the non-destructive methods which can only check at the surface level (Walchli 1981; Stankiewicz, Bolibrzuch et al. 1998; Khir, Badri et al. 2013). However, densimeter, weighing balance with solid density accessories (WB) and magnet can check for the whole gold sample.

Although the most commonly used instruments previously discussed have several advantages, there are still some major limitations and weaknesses such as inaccurate measurement, poor repeatability and not traceable to Standard International (SI) unit. Table 1.1 shows the limitation of non-destructive method instruments.

Table 1.1 Limitations for non-destructive method instruments

Instrument	**Method**	**Unit**	**Limitation**	**Reference**
XRF	Surface	%	Penetrate up 10 to 50 microns only	(Corti 2001)
Weighing balance	Whole	g/ml	Shows gold density similar with tungsten density	(Corti 2001)
Densimeter	Whole	g/ml	Shows gold density similar with tungsten density	(Abdullah, Samsudin et al. 2015)

There are difficulties to detect the presence of other metal in gold whenever instruments in non-destructive method are used. However, XRF and ultrasonic have limitations where XRF can only penetrate the gold sample within 10 to 50 microns (Corti 2001).

Research study on gold density measurement was conducted in 1992 (E.Mercer 1992). Hydrostatic weighing theoretical straightforward but their some practical difficulties such as analytical balance pan must be weighed first, air bubbles can decreased sample weight and water droplets on the wire holding the weighing tray can increase the sample weight. The evaluation of this study is hydrostatic weighing method unable to detect gold-plated with a metal that has similar density with gold.

Hydrostatic weighing method have a limitation and some problems on gold density measurement (Corti 2001). First problem is difficulty to measure the density of metal more than 3 types of materials. Secondly, this method unable to measure accurately the hollow sample and air bubbles trapped on sample. Density of sample will be decreased when weighing sample in liquid. Thus, hydrostatic weighing method is a simple method and quick non-destructive method but inaccurate assay.

The gold density measurement for binary alloys such as gold-silver or gold-copper is easy to measure (Kraut and Stern 2000). The main reason is the existing density instrument able to differentiate the density of gold, silver and copper. Density of gold, silver and copper are 19.3 g/ml, 10.5 g/ml and 8.96 g/ml respectively (Chang and You 1997; Kim, Kang et al. 2007; Martina Ortigosa, Valenstein et al. 2012; Shah, Gentile et al. 2016). It means that the binary alloys for gold-tungsten is easy to measure if the density instrument capable to differentiate their densities.

There are several studies on fake gold related to tungsten. All of the studies are from South East of Asia such as Malaysia and Indonesia. The study on fake gold is difficult to find in other region due to implementation of the gold hallmarking system. First literature related to fake gold was published on 10th February 2015 (Abdullah, Samsudin et al. 2015). Malaysian Nuclear Agency (MINT) and Institute of Research and Consultancy YaPEIM (INPUT) are the organizations involved in this research where MINT is the government agencies involved in nuclear field while INPUT is a

company under Yayasan Pembangunan Ekonomi Islam Malaysia (YaPEIM) involved in research of precious metal assay especially in Ar-Rahnu YaPEIM's pawn shop. They used XRF technique focusing on gold earrings through their research.

Fake gold bar was commonly produced by mixing the gold bar with other material such as tungsten due to similar density value. (Prasetiyo, Sihar et al. 2015). The purpose of their research is to develop testing method on gold bar purity through natural frequencies observation and Frequency Response Function of vibration response. However, this study is a propose concept and has not been validated through experimental result.

Fake gold was produced by gold plating with tungsten in different dimensions and weight (Ismail, Sani et al. 2018). Through their observation, the material covered by gold cannot be detected using XRF technique. They used ultrasonic method to detect fake gold. Several samples were used in this research such as bracelet, necklace and ear ring. It was complicated to detect fake gold due to shape and thin material of the sample.

1.1 Statement of the problem

Nowadays there is no single non-destructive method that can check the gold purity precisely and accurately. Effect of this problem is the presence of many cases of fake golds in the market (Ahmad 2002; Shamsuddin 2011; Alias 2012; Shamsuddin 2016; Rahman 2018). Currently, there are several density instruments capable to check the purity of gold. WB and densimeter using hydrostatic weighing method are the density instruments. This method can determine the density of gold and it is not destructive, simple and economic.

The density of gold can be measured using densimeter or WB through conventional hydrostatic weighing method where it can also determine the gold purity. It is economical, simple and non-destructive method but less accurate of about 0.1 g/ml to 0.2 g/ml (E.Mercer 1992). The density of pure gold and pure tungsten are 19.30 g/ml and 19.25 g/ml respectively (Abdullah, Samsudin et al. 2015; Guo, Gu et al. 2019). Therefore, it will be ineffective for any measurement setup that will not

produce better uncertainty in order to differentiate pure gold and fake gold (Oddy 1983; Raw 1997; Corti 2001; Abdullah, Samsudin et al. 2015). This problem will give a bad impact to gold industry.

Secondly, standard weight 200 g as a reference standard was used to calibrate densimeter and WB (Hildebrand 1993). It is not suitable for calibration of densimeter and WB due to standard weight is a reference standard for mass. So, it can affect the accuracy of the densimeter and WB. It is supposed reference material with known density value as a reference standard to calibrate densimeter and WB.

Thirdly, type of liquid used also important where some of pawnshops did not replaced the distilled water to determine the gold purity. Density of distilled water is important to ensure that the gold purity measurement is accurate (Kochsiek and Glaser 2010; Jenkins 2012; Morris and Langari 2012).

Lastly, the problem arises due to surface porosity of gold sample (Choi, Woo et al. 2008). For both instruments, the sample needs to be taken out of the liquid and immerse again for the next measurement. The trapping of air bubbles on the surface of porous sample has caused inconsistency in the measurement by influencing the liquid temperature and thus, the uncertainty of the measurement.

This study is conducted to explore and improve the hydrostatic weighing method by focusing on the method of density determination. The accuracy of hydrostatic weighing system will be improved to 0.01 g/ml because the density difference between gold and tungsten is only 0.05 g/ml. The establishment in measurement procedure of the hydrostatic weighing system is also important to obtain accurate result.

1.2 Objectives of the study

The main objectives of this study is to develop a hydrostatic weighing system (HWS) to determine the density of gold and tungsten. This main objective was pursued through the following sub-objectives:

a) To develop and optimize the HWS to measure density and volume of the gold and tungsten in order to improve the capability of density measurement to accuracy of 0.01 g/ml.

b) To determine and verify the density of pure gold, pure tungsten and fake gold accurately and precisely to differentiate these materials and verify with existing instruments such as WB and densimeter.

c) To identify and extent the factors effecting density measurement of gold and tungsten.

1.3 Scope of the study

The scope of this study includes determining the volume of gold and tungsten in order to obtain the density and volume of thermal expansion. The uncertainty measurement of the density and volume thermal expansion of the sample will be calculated. This study covers for solid gold and tungsten but not hollow sample. There are many shapes of solid gold and tungsten in the market, however bar, coin and rod shape will be used in this study. Gold and tungsten with purity 99.9% will be used in this study. The minimum and maximum weight of sample are 5 g and 500 g respectively. This study focused on an improved hydrostatic weighing method complete with analytical balance and distilled water as a standard density which traceable to a standard solid density and it is calibrated by National Metrology Institute (NMI). Distilled water then will be used as a standard liquid density. It is much cheaper and stable compared to other standard liquid density such as tridecane (KH Chang 2004). This study also focuses on non-destructive method and it will be verified with existing non-destructive instruments and destructive method such as cupellation method or fire assay. Distilled water will be used as a standard liquid and its temperature is set from 20°C to 30°C.

1.4 Significance of the study

Hydrostatic weighing method which uses either densimeter or WB is a reliable method and has been widely used to determine the purity of gold. It is a non-destructive method and offer more simple step. However, the measurement is highly depends on the instruments and procedures involved in order to get an accurate of gold purity.

The accuracy of both densimeter and weighing balance is 0.1 g/ml. The newly proposed system will be designed to get accurate purity results up to 0.01 g/ml and thus it can be used to differentiate between gold and tungsten. The function of this system is similar with other non-destructive method which will comply the Pawnbroker Act 1972 and Islamic Pawnbroker Activity Guideline that stated the pawnbroker cannot damage the gold. Furthermore, the system is able to determine the volume thermal expansion of gold. The used of dilatometer to determine the volume thermal expansion also can be avoided (Yamada, Abe et al. 2001). Therefore, this study has a great impact to public, gold industries and country as it will be able to resolve a fraud issue involving fake gold in Malaysia.

REFERENCES

Abas, A. A. (2013). "Determining the authenticity of gold jewelries by ultrasonic testing." Journal of Malaysian Society for Non Destructive testing(7): 246-250.

Abdullah, J., A. Samsudin, et al. (2015). "Non-Destructive Assaying Gold Jewellery Using Dual-Energy Micro-Computed Tomography." Jurnal Teknologi 73(3).

Act81 (1972). "AKTA PEMEGANG PAJAK GADAI " Pesuruhjaya Penyemak Undang-Undang, Malaysia.

Ahmad, Z. A. (2002). "Law to require gold, silver contents verified." The Star.

Ali, N. A. M., Y. C. Johari, et al. (2017). Ar-Rahnu: A Review of Literature and Future Research. SHS Web of Conferences, EDP Sciences.

Alias, W. N. H. W. (2012). "Bank tempatan rugi RM75 juta ditipu." Utusan Malaysia.

Arbutine, A. (2008). "Pawnbroker's Guide to Metals Testing." Today's Pawnbroker.

Ariffin, M. K. (2005). Termodinamik asas, Penerbit UTM Press.

ASTM (2017). "Standard Test Methods for Determination of Gold in Bullion by Fire Assay Cupellation Analysis (E1335-08)."

Avdiaj, S., J. Setina, et al. (2015). "Volume Determination of Vacuum Vessels by Gas Expansion Method." Mapan 30(3): 175-178.

Ayob, M. R. and M. Z. M. Yusof (1996). Termodinamik Gunaan untuk ahli Teknologi Kejuruteraan, Penerbit UTM.

Azli, R. M., M. R. Palil, et al. (2016). "Benefits-Sacrifices Exchange (BSX) in Islamic pawn broking transaction: An exploratory factor analysis." International Journal of Business, Economics and Law 10(1).

Azli, R. M., M. R. Palil, et al. (2017). "The Role of Ar-Rahnu's Benefits-Sacrifices Exchange towards Relationships between Customers and Islamic Financial Institutions." Pertanika Journal of Social Sciences & Humanities 25.

B.Boss, C. and K. J.Fredeen (2004). Concepts, Instrumentation and Techniques in Inductively Coupled Plasma Optical Emission Spectrometry, Perkin Elmer.

Bardi, U., F. Niccolai, et al. (2008). "Gold assay with Knudsen effusion mass spectrometry." International Journal of Mass Spectrometry 273(3): 138-144.

Battaini, P., E. Bemporad, et al. (2014). "The fire assay reloaded." Gold Bulletin 47(1-2): 9-20.

Bettin, H., M. Heinonen, et al. (2016). "Final report on EUROMET key comparison EUROMET. MD-K2 (EUROMET 627)" Comparison of density determinations of liquid samples"." Metrologia 53(1A): 07019.

Bigg, P. H. (1967). "Density of water in SI units over the range 0-40 C." British Journal of Applied Physics 18(4): 521.

Bignell, N. and B. Ward (2007). Density measurement at NMIA. Asia Pacific Symposium on Mass, Force and Torque (APMF 2007).

BIPM, I., IFCC, ISO, IUPAC, IUPAP, OIML (1995). "Guide to the Expression of Uncertainty in Measurement."

Blunt, E. (2008). "Fake fears over Ethiopia's gold." BBC News.

Bodryakov, V. Y. (2015). "Correlation of temperature dependences of thermal expansion and heat capacity of refractory metal up to the melting point: Tungsten." High Temperature 53(5): 643-648.

Brill, M. (1997). "Analysis of carat gold." Gold technology 22: 10.

Brill, M. and K. H. Wiedemann (1992). "Determination of gold in gold jewellery alloys by ICP spectrometry." Gold Bulletin 25(1): 13-26.

Brillo, J., I. Egry, et al. (2004). "Density and thermal expansion of liquid Au-Cu alloys." International Journal of Thermophysics 25(6): 1881-1888.

Brouwer, P. (2006). "Theory of XRF." Almelo, Netherlands: PANalytical BV.

BS718 (1991). "Specification for Density hydrometers." British Standard BS718:1991.

Cayumil, R., R. Khanna, et al. (2016). "Concentration of precious metals during their recovery from electronic waste." Waste management 57: 121-130.

Chang, J. Y. and S. M. You (1997). "Enhanced boiling heat transfer from microporous surfaces: effects of a coating composition and method." International Journal of Heat and Mass Transfer 40(18): 4449-4460.

Choi, I.-M., S.-Y. Woo, et al. (2008). "New density determination method using a reference gas." Metrologia 45(5): 524.

Clausen, J. L. and N. Korte (2009). "Environmental fate of tungsten from military use." Science of the total environment 407(8): 2887-2893.

Coggiola, G., S. Lorefice, et al. (1990). Characterization of a Tamson visibility bath for kinematic viscosity measurements. Proceedings of the 4th Symp. on Temp. and Thermal.

Corti, C. W. (1999). "Metallurgy of microalloyed 24 carat golds." Gold Bulletin 32(2): 39-47.

Corti, C. W. (2001). "Assaying of gold jewellery-Choice of technique." Gold technology 32: 20-30.

Council, B. H. (2010). "HALLMARKING: Guidance Notes - Practical guidance in relation to the Hallmarking Act 1973."

Cretu, C. and E. Van Der Lingen (1999). "Coloured gold alloys." Gold Bulletin 32(4): 115-126.

Dakian, M. (2005). Sistem kewangan Islam: instrumen, mekanisme, dan pelaksanaannya di Malaysia, Utusan Publications.

Davidson, S., M. Perkin, et al. (2004). "Measurement good practice guide, No. 71." The measurement of mass and weight. National Physical Laboratory, Teddington, Middlesex, YK 1.

Ding, Y., S. Zhang, et al. (2019). "Recovery of precious metals from electronic waste and spent catalysts: A review." Resources, Conservation and Recycling 141: 284-298.

Drnovsek, J., J. Bojkovski, et al. (1997). A general procedure for evaluation of calibration baths in precision temperature measurements. IEEE Instrumentation and Measurement Technology Conference Sensing, Processing, Networking. IMTC Proceedings, IEEE.

E.Mercer, M. (1992). "METHODS FOR DETERMINING THE GOLD CONTENT OF JEWELRY METALS." Gems and Gemology 28(4): 222-233.

Eames, D. A., G. A. Eames, et al. (2015). Device to test and authenticate precious metal objects, Google Patents. US20150308983A1.

Ellison, S. L. R., M. Rosslein, et al. (2000). Quantifying uncertainty in analytical measurement. Quantifying uncertainty in analytical measurement, Eurachem.

Espejo, F. and S. Armada (2012). "Determination of Volumetric Coefficients of Thermal Expansion in Alcoholic Beverages and Aqueous Ethanol-Sucrose Mixtures by Differential Volume Measurements." Food and Bioprocess Technology 5(7): 2805-2818.

Fassel, V. A. and R. N. Kniseley (1974). "Inductively coupled plasma. Optical emission spectroscopy." Analytical Chemistry 46(13): 1110A-1120a.

Fischer, J., M. Battuello, et al. (2003). "Uncertainty budgets for realisation of scales by radiation thermometry." CCT-WG5 on radiation thermometry: 631-638.

Fornasini, P. (2008). The uncertainty in physical measurements: an introduction to data analysis in the physics laboratory, Springer Science & Business Media.

Fujii, K. (2004). "Present state of the solid and liquid density standards." Metrologia(41): 1-15.

Fujii, K., A. Waseda, et al. (2001). "Development of a silicon density standard and precision density measurements of solid materials by hydrostatic weighing." Measurement Science and Technology 12(12): 2031.

Fukuda, M., A. Hasegawa, et al. (2018). "Thermal properties of pure tungsten and its alloys for fusion applications." Fusion Engineering and Design 132: 1-6.

Gasik, M. (2013). Handbook of ferroalloys: theory and technology, Butterworth-Heinemann.

Ghosh, S., V. L. Prasanna, et al. (2013). "Inductively Coupled Plasma-Optical Emission Spectroscopy: A Review." Asian J. Pharm. Ana 3(1): 24-33.

Giancoli, D. C. (2000). Physics for scientists and engineers, Prentice hall Upper Saddle River, NJ.

Guha, B. and G. Bandyopadhyay (2016). "Gold price forecasting using ARIMA model." Journal of Advanced Management Science Vol 4(2).

Guo, M., D. Gu, et al. (2019). "Formation of scanning tracks during Selective Laser Melting (SLM) of pure tungsten powder: Morphology, geometric features and forming mechanisms." International Journal of Refractory Metals and Hard Materials 79: 37-46.

Gupta, S. V. (2002). Practical density measurement and hydrometry, Taylor & Francis.

Gupta, S. V. (2006). Comprehensive Volume and Capacity Measurements, New Age International.

Hafizi, A. M., N. Halid, et al. (2016). Gold investment in Malaysia: its operation, contemporary applications and shariah issues. Derived from conference. qfis. edu. qa/app/media/7026.

Hanrahan, J. F. (1962). "The fire assay method as applied to high purity gold at the Rand Refinery, Limited." Journal of the Southern African Institute of Mining and Metallurgy 62(12): 712-727.

Heath, T. L. (2002). The works of Archimedes, Courier Corporation.

Heinonen, M. (2006). Uncertainty in humidity measurements. EUROMET Workshop P.

Henderson, J. B., W. D. Emmerich, et al. (1987). "A method for the temperature calibration of pushrod dilatometers." Journal of thermal analysis 32(6): 1905-1913.

Hildebrand (1993). "Operating Instructions Densimeter Model H-300S." Release 1.0.

Hughes, S. W. (2005). "Archimedes revisited: a faster, better, cheaper method of accurately measuring the volume of small objects." Physics Education 40(5): 468.

International, A. (2015). "Standard Test Methods For Rubber Products - Chemical Analysis." ASTM D297-15.

IS1417 (1999). "Gold and Gold Alloys, Jewellery/Artefacts - Fineness and Marking Specification (Third Revision) " Indian Standard(IS 1417:1999).

IS1418 (2009). "Determination of Gold in Gold Bullion, Gold Alloys and Gold Jewellery/Artefacts - Cupellation (Fire Assay) Method." International Standard Third Revision(IS 1418:2009).

IS2790 (1999). "Guidelines for Manufacture of 23, 22, 21, 18, 14 and 9 Carat Gold Alloys." Indian Standard IS 2790:1999.

Ismail, M. P., S. Sani, et al. (2018). Ultrasonic inspection of fake gold jewelry. IOP Conference Series: Materials Science and Engineering, IOP Publishing.

ISO10378 (2016). "Copper, lead and zinc sulfide concentrates - Determination of gold and silver - Fire assay gravimetric and flame atomic absorption spectrometric method ISO 10378:2016 (E)." International Standard(3rd Edition).

ISO11426 (1997). "Determination of gold in gold jewellery alloys - Cupellation method (fire assay)." International Standard ISO 11426(2nd Edition:1997(E)).

ISO15093 (2015). "Jewellery - Determination of precious metals in 999% gold, platinum and palladium jewellery alloys - Difference method using ICP-OES." International Standard ISO 15093:2015 (E)(2nd Edition).

Ivekovic, A., N. Omidvari, et al. (2018). "Selective laser melting of tungsten and tungsten alloys." International Journal of Refractory Metals and Hard Materials 72: 27-32.

Jalas, P., J. P. Ruottinen, et al. (2002). "XRF analysis of jewelry using fully standardless fundamental parameter approach." Gold technology 35: 28.

Jenkins, S. (2012). "Measurement and Instrumentation: Theory and Application." Chemical Engineering 118(12): 9-10.

Jones, F. E. and R. M. Schoonover (2002). Handbook of mass measurement, CRC Press.

Jotanovic, A., M. Memic, et al. (2012). "Comparison of x-ray fluorescent analysis and cupellation method for determination of gold in gold jewellery alloy." Glasnik hemicara i tehnologa Bosne i Hercegovine 38: 13-18.

Junior, J. J. P. S., R. G. Pereira, et al. (2018). Inmetro hydrostatic weighing system - determination of solids volume. Journal of Physics: Conference Series, IOP Publishing.

Karadjova, I., S. Arpadjan, et al. (2000). "Determination of trace metals in high purity gold." Fresenius' journal of analytical chemistry 367(2): 146-150.

Karamanov, A., B. Dzhantov, et al. (2013). "Glass transition temperature and activation energy of sintering by optical dilatometry." Thermochimica acta 553: 1-7.

Karmakar, B., P. Kundu, et al. (2002). "Crystallization Kinetics and Mechanism of Low Expansion Lithium Aluminosilicate Glass Ceramics by Dilatometry." Journal of the American Ceramic Society 85(10): 2572-2574.

KH Chang, Y. L. (2004). "Hydrostatic Weighing at KRISS." Metrologia(41): 95-955.

Khir, M. F. A., M. B. Badri, et al. (2013). "Critical appraisal of rahn-based Islamic microcredit facility from Shariah perspective." Advances in Natural and Applied Sciences 7(3): 221-232.

Kim, S. T., D. Y. Kang, et al. (2007). "Separation and Quantitation of Silver Nanoparticles using Sedimentation Field-Flow Fractionation." Journal of Liquid Chromatography & Related Technologies 30(17): 2533-2544.

Kinneberg, D. J., S. R. Williams, et al. (1998). "Origin and effects of impurities in high purity gold." Gold Bulletin 31(2): 58-67.

Kochsiek, M. and M. Glaser (2010). Handbook of Metrology, John Wiley & Sons.

Kraut, J. C. and W. B. Stern (2000). "The density of gold-silver-copper alloys and its calculation from the chemical composition." Gold Bulletin 33(2): 52-55.

Kuchler, R., T. Bauer, et al. (2012). "A compact and miniaturized high resolution capacitance dilatometer for measuring thermal expansion and magnetostriction." Review of scientific instruments 83(9): 095102.

Kumar, A., H. Kumar, et al. (2013). ESTABLISHMENT OF TRACEABILITY OF REFERENCE GRADE HYDROMETERS AT NATIONAL PHYSICAL LABORATORY, INDIA (NPLI). International Journal of Modern Physics: Conference Series, World Scientific.

Kumar, T. K. S., N. N. Viswanathan, et al. (2015). "Estimation of sintering kinetics of oxidized magnetite pellet using optical dilatometer." Metallurgical and Materials Transactions B 46(2): 635-643.

Kurtz, R. A. and H. Utzinger (1976). Selectively adjustable darts, Google Patents.

Lai, J. H. and A. E. Johnson (1993). "Measuring polymerization shrinkage of photo-activated restorative materials by a water-filled dilatometer." Dental Materials 9(2): 139-143.

Lange, B., S. Recknagel, et al. (2008). "Analysis of pure copper- a comparison of analytical methods." Microchimica Acta 160(1-2): 97-107.

LaPorte, M. M., M. J. Sanger, et al. (2009). "Using soda cans to teach physical science students about density." Journal of chemical education 86(2): 209.

Lee, Y. J., K. H. Chang, et al. (2004). "Automatic alignment method for calibration of hydrometers." Metrologia 41(2): S100.

Lee, Y. J., W. G. Lee, et al. (2012). "HYDROSTATIC WEIGHING APPARATUS FOR THE DENSITY DETERMINATION OF LARGE WEIGHTS." XX IMEKO World Congress.

Lorefice, S. and A. Malengo (2006). "Calibration of hydrometers." Measurement Science and Technology 17(10): 2560.

Majcen, N., M. Majer, et al. (2002). "Traceability of results of measurements of gold in precious metal alloys by XRF." Acta chimica slovenica 49(1): 187-194.

Mansouri, S. S., A. Farsi, et al. (2011). "Density calculation of liquid organic compounds using a simple equation of state up to high pressures." Journal of Molecular Liquids 160(2): 94-102.

Mark Clarkson, F. M. N. (2004). "Bilateral Comparison of Standard Hydrometer between IRL, New Zealand and NML-SIRIM, Malaysia." 2nd International Symposium on Metrology & Physics Application and Energy Measurement (ISMPAEM-2004).

Markowicz, A. (2011). "An overview of quantification methods in energy-dispersive X-ray fluorescence analysis." Pramana 76(2): 321-329.

Martina Ortigosa, S., J. S. Valenstein, et al. (2012). "Parameters affecting the efficient delivery of mesoporous silica nanoparticle materials and gold nanorods into plant tissues by the biolistic method." small 8(3): 413-422.

Maryani, A. and H. M. Rusmahafi (2017). "ANALISIS PENGARUH KOMPETENSI SYARIAH, KOMPENSASI ISLAMI DAN KECERDASAN SPIRITUAL TERHADAP KINERJA KARYAWAN (Studi Kasus Bank Muamalat Cabang Ternate)." Jurnal Ekonomi Islam 8(1): 51-70.

Mattlage, M. and C. Coody (2005). Fishing hook, Google Patents.

Merlone, A., L. Iacomini, et al. (2007). "A liquid bath for accurate temperature measurements." Measurement 40(4): 422-427.

Michalski, L., K. Eckersdorf, et al. (1991). Temperature measurement, J. Wiley.

Mohd Nor, F., A. K. Ismail, et al. (2014). "An improved ring method for calibration of hydrometers." Measurement 2014 48: 1-5.

Morris, A. S. and R. Langari (2012). Measurement and instrumentation: theory and application, Academic Press.

MS1247 (2005). "Description and Marking of Articles of, or containing Precious Metal - Code of Practice." Malaysian Standard MS 1247:2005.

MS1365 (1994). "Methods of Test for Standard of Fineness of Precious Metals: Part 1: Gold and Articles of Gold Alloy by Cupellation Method." Malaysian Standard(MS 1365: Part 1: 1994).

Nayak, A. K., R. K. Singh, et al. (2010). "Measurement of volumetric thermal expansion coefficient of various nanofluids." Technical Physics Letters 36(8): 696-698.

Nicolaus, R. A. and G. Bonsch (1997). "A novel interferometer for dimensional measurement of a silicon sphere." IEEE transactions on instrumentation and measurement 46(2): 563-565.

Ning, Y. (2005). "Properties and applications of some gold alloys modified by rare earth additions." Gold Bulletin 38(1): 3-8.

Nor, F. M. (2012). "Ring Method for Hydrometer Calibration." International Measurement Confederation, IMEKO.

Oddy, A. (1983). "Assaying in antiquity." Gold Bulletin 16(2): 52-59.

Organ, B. R. M. (2010). "Palladium Hallmarking in the UK." Platinum Metals Review 54(1): 51-52.

Pagtalunan, V. C. and L. K. S. Tolentino (2019). "Characterization of Silicon Oil Calibration Bath Through Stability and Uniformity Tests Using Standard Platinum Resistance Thermometer and Precision Multimeter." International Journal of Engineering Research and Technology 12: 705-710.

Paradisa, P. F., T. Ishikawaa, et al. (2008). "Density of liquid gold measured by a non-contact technique." Gold Bulletin 41(3): 242-245.

Patterson, J. B. and E. C. Morris (1994). "Measurement of absolute water density, 1 C to 40 C." Metrologia 31(4): 277.

Pavlik Iii, A., S. V. Ushakov, et al. (2017). "Structure and thermal expansion of Lu2O3 and Yb2O3 up to the melting points." Journal of nuclear materials 495: 385-391.

Philipps, V. (2011). "Tungsten as material for plasma-facing components in fusion devices." Journal of nuclear materials 415(1): S2-S9.

Piorek, S., S. I. Shefsky, et al. (2013). System and method for identification of counterfeit gold jewelry using xrf, Google Patents.

Poterasu, M. (2008). "Time drift compensation of the electronic balance during the measurement of liquid crystal density." Measurement Science and Technology 19(2): 025102.

Prasetiyo, I., I. Sihar, et al. (2015). A Gold Bar Purity Testing Method Based on Vibration Characteristics. Applied Mechanics and Materials, Trans Tech Publ.

R.K.Rajput (2010). "Engineering Metrology And Instrumentation (Including Laboratory Practicals)." S.K.Kataria & Sons(4th Edition).

Rahman, D. A. A. (2018). "Rantai emas palsu pun nak digadai." Harian Metro.

Rastrelli, A., M. Miccio, et al. (2009). "Modern and ancient gold jewellery attributed to the Etruscans: a science-based study." ArcheoSciences. Revue d'archeometrie(33): 357-364.

Raw, P. (1997). "The assaying and refining of gold, a guide for the gold jewellery producer." World Gold Council.

Raza, N., S. J. H. Shahzad, et al. (2016). "Asymmetric impact of gold, oil prices and their volatilities on stock prices of emerging markets." Resources Policy 49: 290-301.

Reese, R. L. (2000). University physics, Brooks/Cole.

Riski, K. and H. Kajastie (2004). "Study on temperature and density gradients in water for hydrostatic density determination." Measurement Science and Technology(15): 1687-1693.

Rotter, M., E. Gratz, et al. (1998). "A miniature capacitance dilatometer for thermal expansion and magnetostriction." Review of scientific instruments 69(7): 2742-2746.

Saunders, J. B. (1972). "Ball and cylinder interferometer." Res. Natl Bur. Stand. C 76: 11-20.

Saunders, P., J. Fischer, et al. (2008). "Uncertainty budgets for calibration of radiation thermometers below the silver point." International Journal of Thermophysics 29(3): 1066-1083.

Saw, C. K. and W. J. Siekhaus (2005). "Thermal expansion of AuIn2." Scripta materialia 53(10): 1153-1157.

Schaffer, S. (2003). Golden means: Assay instruments and the geography of precision in the Guinea trade. Instruments, Travel and Science, Routledge: 32-62.

Shah, A. A. (2018). "Tadbir Urus dan Pengawalseliaan Ar-Rahnu di Malaysia: Bidang Kuasa Siapakah?" KANUN: Jurnal Undang-undang Malaysia 30(No2).

Shah, C. M., R. C. Gentile, et al. (2016). "PERFLUOROCARBON LIQUIDS'ABILITY TO PROTECT THE MACULA FROM INTRAOCULAR DROPPING OF METALLIC FOREIGN BODIES." Retina 36(7): 1285-1291.

Shamsuddin, N. (2011). "Penipuan Emas: Kaji Ketulenan." Harian Metro.

Shamsuddin, N. (2016). "PENIPUAN EMAS: LUAR BETUL DALAM TIPU." Harian Metro: 6.

Singh, N. (2012). "A rugged, precise and accurate new gravimetry method for the determination of gold: an alternative to fire assay method." SpringerPlus 1(1): 1-6.

SKMGuideline (2013). "GARIS PANDUAN AKTIVITI PAJAK GADAI ISLAM (AR-RAHNU)." Suruhanjaya Koperasi Malaysia.

Smith, G. T. (2002). Industrial metrology: surfaces and roundness, Springer.

SmrCka, L., K. MiSek, et al. (1965). "The density of quenched gold." Cechoslovackij fiziceskij zurnal B 15(6): 418-424.

Stankiewicz, W., B. Bolibrzuch, et al. (1998). "Gold and gold alloy reference materials for XRF analysis." Gold Bulletin 31(4): 119-125.

Sulaiman, N. S. H., S. F. Kassim, et al. (2014). "The comparison between ar-rahnu and conventional pawn broking." Global Business and Economics Research Journal 3(5): 57-65.

Sun, G.-D., K.-F. Wang, et al. (2019). "A low-cost, efficient, and industrially feasible pathway for large scale preparation of tungsten nanopowders." International Journal of Refractory Metals and Hard Materials 78: 100-106.

Tanaka, M., G. Girard, et al. (2001). "Recommended table for the density of water between 0 C and 40 C based on recent experimental reports." Metrologia 38(4): 301.

Taylor, B. N. and C. E. Kuyatt (1994). "Guidelines for evaluating and expressing the uncertainty of NIST measurement results."

Terentyev, D., J. Riesch, et al. (2018). "Plastic deformation of recrystallized tungsten-potassium wires: Constitutive deformation law in the temperature range 22-600°C." International Journal of Refractory Metals and Hard Materials 73: 38-45.

Thalmann, R. (1995). "Intercomparison of thermal expansion measurements." EUROMET Project 275.

Toledo, M. (2006). "Operating Instructions: Excellence Plus XP Analytical Balances."

Toledo, M. (2008). "Proper Weighing with Laboratory Balances."

Walchli, W. (1981). "Touching precious metals." Gold Bulletin 14(4): 154-158.

Warke, V. S., R. D. Sisson Jr, et al. (2011). "The effect of porosity on the austenite to ferrite transformation in powder metallurgy steels." Materials Science and Engineering: A 528(10-11): 3533-3538.

Watanabe, H., N. Yamada, et al. (2002). "Development of a laser interferometric dilatometer for measurements of thermal expansion of solids in the temperature range 300 to 1300 K." International Journal of Thermophysics 23(2): 543-554.

White, G. K. and J. G. Collins (1972). "Thermal expansion of copper, silver, and gold at low temperatures." Journal of Low Temperature Physics 7(1-2): 43-75.

Willink, R. (2013). Measurement uncertainty and probability, Cambridge University Press.

Wright, J. D., V. E. Bean, et al. (2010). "NIST calibration services for hydrometers." NIST Special Publication 250: 78.

Yaacob, S. E. (2018). "CABARAN DAN ALTERNATIF PENYELESAIAN BAGI PENYEDIA PERKHIDMATAN AR-RAHNU DI MALAYSIA (CHALLENGES AND SOLUTIONS FOR AR-RAHNU SERVICE PROVIDERS IN MALAYSIA)." UMRAN-International Journal of Islamic and Civilizational Studies 5(1).

Yadav, S., V. K. Gupta, et al. (2005). "Proficiency testing through interlaboratory comparison in the pressure range up to 70 MPa using pressure dial gauge as an artifact."

Yamada, N., R. Abe, et al. (2001). "A calibration method for measuring thermal expansions with a push-rod dilatometer." Measurement Science and Technology 12(12): 2121.

Yang, X. (2018). "Beneficiation studies of tungsten ores-A review." Minerals Engineering 125: 111-119.

Zhang, X., Q. Yan, et al. (2017). "Preparation of pure tungsten via various rolling methods and their influence on macro-texture and mechanical properties." Materials & Design 126: 1-11.

Zoroddu, M. A., S. Medici, et al. (2018). "Tungsten or Wolfram: Friend or Foe?" Current medicinal chemistry 25(1): 65-74.

LIST OF PUBLICATIONS

Indexed Journal

1. Mohd Nor, F., Tamuri, A.R., Ismail, A.K. (2019). "New Development Of The Metal Volume Thermal Expansion", Journal of Advanced Research in Fluid Mechanics and Thermal Sciences, page 106-110 **(Indexed by SCOPUS)**

Non-indexed Journal

1. Mohd Nor, F., Tamuri, A.R., Ismail, A.K. (2019). "Fake Gold: Gold Purity Measurement Using Non Destructive Method", International Journal of Engineering & Technology, page 165-172

Indexed Conference Proceeding

1. Mohd Nor, F., Tamuri, A.R., Ismail, A.K., Mazuki,A.A.M. (2019). "Verification of the hydrostatic weighing system with existing gold purity instruments", International Conference on Mechanical and Manufacturing Engineering **(Indexed by SCOPUS)**

Non-indexed Conference Proceeding

1. Rahmat,N.R., Mohd Nor, F.,Othman,H. Zakaria, O. (2016). "Density Measurement of Solid Density by using Hydrostatic Weighing System", National Physics Conference 21-22 December 2016