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An evaluation of a Paar DMA 5000 density meter

D J Bayliss and S P Downes

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National Physical Laboratory
Teddington, Middlesex, United Kingdom, TW11 0LW

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ABSTRACT

NPL have recently acquired a Paar DMA 5000 vibrating cell density meter to replace the Paar DMA 55 which has been in use since 1993. The manufacturer's specification claims a higher accuracy and better repeatability for the new meter and, in addition, it has the capability to automatically correct for viscosity related errors. NPL has assessed this instrument over a density range of 1 kg m^{-3} to 1650 kg m^{-3}, a dynamic viscosity range of 1 mPa s to 600 mPa s and a temperature range of 10 °C to 50 °C. Within these parameters it was established that the instrument could, by using equations derived from multiple regression analysis of the test data, measure the density of an unknown liquid with an uncertainty of better than ± 0.05 kg m^{-3}. This compares with a measurement uncertainty of ± 0.1 kg m^{-3} when using the Paar DMA 55 over a similar measurement range.

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National Physical Laboratory
Teddington, Middlesex, United Kingdom, TW11 0LW

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Approved on behalf of the Managing Director, NPL,
by Dr Roy Preston, authorised by Head of
NPL Materials Centre

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1 INTRODUCTION

The Mass and Density section of the National Physical Laboratory have used a vibrating cell density meter (a Paar DMA 55) since 1993 for the density measurement of small volumes of liquids. A drawback in common with most first-generation instruments of this type is that the viscosity of the liquid under test causes an error in the measured density, although a correction can be applied if the viscosity of the liquid is known [1]. Several years ago Anton Paar, the manufacturer of the DMA 55, introduced a completely re-designed meter, the DMA 5000 with several new features [2]. The most potentially useful feature to NPL was the introduction of a means of correcting viscosity related errors in the density measurement of a fluid without knowledge of its viscosity being required.

The meter can be used as a direct reading instrument, with density measurements corrected automatically for viscosity errors by the instrument's internal software. However, work by Fitzgerald [3] shows that more accurate and reliable density measurements can be achieved by calibrating the instrument with a range of reference liquids of differing densities and viscosities.

2 PRINCIPLE OF OPERATION

Vibrating cell density meters work on the principle that a glass U-tube, rigidly mounted at the open end and filled with a sample of a fluid being measured, obeys the law of simple harmonic motion. The U-tube will oscillate when excited at a fundamental frequency f, which is a function of the combined mass of the U-tube m_1 and the fluid sample m_2. If c is the spring constant, the natural frequency of the system will be given by

$$f = \frac{1}{2\pi}\sqrt{\frac{c}{m_1 + m_2}} \qquad \textbf{(1)}$$

Since the mass of the fluid is the product of the U-tube volume v and the fluid density ρ, the oscillation period τ can be written

$$\tau = 2\pi\sqrt{\frac{m_1 + \rho v}{c}} \qquad \textbf{(2)}$$

Two constants, *A* and *B*, can be defined which contain terms for the U-tube's volume *v* and mass m_1, and the spring constant of the system *c*

$$A = \frac{4\pi^2 v}{c} \qquad B = \frac{4\pi^2 m_1}{c}$$

If these constants are substituted into Equation 2, the fluid density can be expressed as

$$\rho = \frac{(\tau^2 - B)}{A} \tag{3}$$

A and *B* are regarded as constants of the measurement cell and can be determined experimentally by calibrating the instrument using two reference fluids of known density, normally air and pure water. These constants are temperature dependent and are only valid at the calibration temperature. Once *A* and *B* have been established, the density of an unknown fluid (at the calibration temperature) can be determined from the measurement of the oscillation period τ of the U-tube which is filled with the fluid.

In practice, however, fluids have a damping effect on the oscillation of the U-tube that is dependent on the fluid viscosity; work by Fitzgerald [3] and others, including NPL, has established that for viscosities below 150 mPa s the error in the calculated density is related to the square root of the viscosity.

In the Paar DMA5000, the harmonic frequencies of the U-tube (filled with a sample fluid) are measured in addition to the fundamental frequency. The meter calculates the period of oscillation of the U-tube divided by the period of oscillation of a built in reference oscillator. This quotient is termed *Q*. As the damping of the harmonic frequencies is dependent on the viscosity of the sample, the density of the sample can be calculated as a function of *Q* and the *damping*.

The internal software of the meter compensates for errors caused by the viscosity of a sample by calculating the density ρ using the polynomial equation

$$\rho = C\tau^2(1 + D.\text{damping} + E.\text{damping}^2) - F + G\tau^4$$

where C, D, E, F and *G* are constants of the instrument.

A drawback to this equation is that it attempts to compensate for viscosity over the whole of the temperature range of the instrument. As such, the equation is relatively complex and includes an interaction term between Q and *damping*. An independent assessment of several DMA 5000s [3] has shown that better measurement accuracy can be obtained by deriving polynomial equations (usually with a quadratic form) at discrete temperatures. These equations are specific to an individual instrument. In use, the density of an unknown fluid at an intermediate temperature can be calculated by interpolation of the measurement data (see 6.2). Using the data obtained during the course of this investigation, a series of polynomial equations were obtained at discrete temperatures for this instrument (see 5.4), which relate fluid density to the measured parameters of Q and *damping*.

3 ASSESSMENT PROCEDURE

This report describes the following tests used to assess NPL's Paar DMA 5000 meter

- Comparison between the direct measurement of the fluid temperature within the U-tube, with the temperature displayed on the meter’s LCD screen which is provided via the built in platinum resistance thermometers;
- Measurement of the values of Q and *damping* for a variety of reference fluids (including air and water) at temperatures of 10 °C, 20 °C, 30 °C, 40 °C and 50 °C;
- Statistical analysis of the measurement data to establish the best-fit density equations at these temperatures.

4 MEASUREMENT OF FLUID TEMPERATURE

The measurement cell of the Paar DMA 5000 comprises a U-tube sealed inside a glass envelope. The glass envelope is fitted within a metal block, which supplies a rigid support for the vibrating U-tube. An inert gas possessing good thermal conductivity fills the space between the envelope and U-tube. The temperature of the metal block is maintained by a Peltier device and is monitored using two built-in PRTs adjacent to the measurement cell. Although Paar claim in their specification a [sample] temperature accuracy of 0.01 ºC, no provision is made to enable the PRTs to be removed for routine calibration. In use, the meter can be set in 10 mK increments to maintain the measurement cell at any temperature within the range 0 °C to 80 °C. An optional temperature scan function when enabled automatically changes the set temperature in variable increments. The meter records a density measurement after stabilisation at each increment.

NPL have carried out their own temperature assessment using two PRTs supplied by Sensing Devices Ltd, which have a nominal sensor diameter of 1 mm that is small enough to be inserted directly into the U-tube. The PRTs were calibrated by UKAS accredited laboratories over a temperature range of 0 ºC to 50 ºC, with and given an estimated uncertainty throughout the range of ± 0.005 ºC (at k=2)

4.1 Basis of temperature tests

The supplier of the PRTs had advised NPL that because the sheath of the sensing element was not sealed, contact with a fluid might result in erroneous readings. Since it was considered important to replicate working practice, one of the sensors was used to measure the temperature of the U-tube when it was filled with liquid, but the other was kept dry, thus providing a control to check the stability of the immersed sensor. It was consequently established that immersion in the test fluid had no detrimental effect of the performance of the PRT.

The U-tube, which is mounted in a horizontal plane within the glass envelope, can be viewed through a window in the front of the meter, although the field of view does not extend to the closed end of the U-tube. The built-in PRT sensors appear to be located adjacent to the rear of the measurement cell and in line with its mid-point.

Test 1 - Temperature gradients along the U-tube

Initial tests were carried out at a set temperature of 20 °C to check for any variation in temperature along the length of the U-tube, by filling it with silicone oil and noting any change in measured temperature when the sensor was moved along it. Similar tests were performed 18 months later, but at set temperatures of 10 °C, 20 °C, 30 °C, 40 °C and 50 °C. It was not possible to move the sensor more than a few millimetres further than the mid-point of the U-tube due to the limiting length of the PRT connecting wires.

The temperature differences encountered along the U-tube were recorded as offsets from the measured temperature at the mid-point of the U-tube. Figure 1 shows the offsets for each set temperature.

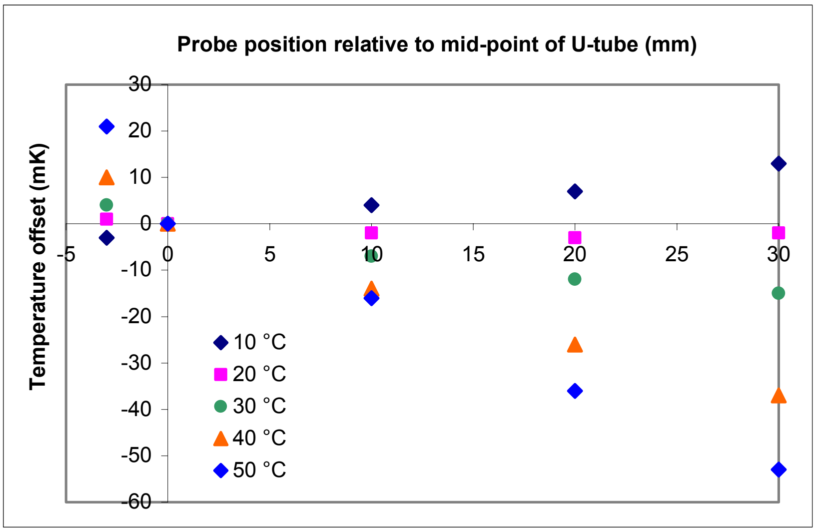

Figure 1. Temperature variation along the U-tube

Test 2 - Temperature comparison with air

The U-tube was unfilled - i.e. it contained only air at ambient pressure. A PRT sensor was inserted as far as the mid-point of each limb of the U-tube. The set temperature was adjusted from 50 ºC down to 10 ºC and up again to 50 ºC in 10 °C increments. Following stabilisation at each set temperature, both the 'displayed' temperature (that determined from the instrument's built-in PRTs) and the 'measured' temperature (that determined from the PRT's inserted into the U-tube) were recorded for both limbs. This procedure was then repeated after swapping over the test PRT sensors within the limbs of the U-tube.

Test 3 - Temperature comparison with water

The U-tube was filled with distilled water and a PRT sensor was inserted as far as the mid-point of its rear limb. The temperature was cycled over the range 50 ºC to 10 ºC as described in Test 2. Following stabilisation at each set temperature, the displayed and the measured temperatures were recorded. The same PRT sensor was then inserted in the front limb of the U-tube and the procedure repeated.

Test 4 - Temperature comparison with silicone oil

Test 3 was repeated except that silicone oil was used in place of distilled water.

Test 5 - Temperature comparison with air

Test 2 was repeated at temperatures of 20 ºC and 50 ºC to establish if immersion in water and silicone oil had affected the accuracy of the PRT sensor during Test's 3 and 4.

4.2 Results of temperature testing

After completion of the tests, the PRT sensor that had been immersed in liquids was checked against the control sensor. As no significant change in its performance was apparent, it was concluded that no noticeable adverse effects had resulted from the immersion of the sensor in water and oil.

Within the temperature range assessed, once the meter had stabilised the displayed temperature always agreed with the set temperature to within ±2 mK

Comparison of the temperature measurements made at the mid point of the U-tube taken 18 months apart, showed a maximum difference of 4 mK. This demonstrates a high degree of stability of the built-in temperature measurement and control system. Furthermore, the short-term repeatability of the instrument when controlling the temperature of the measurement cell was shown during Test 2 and Test 5 to be better than 4 mK.

Figure 2 shows the difference between the 'measured' T_M and 'displayed' T_D temperatures of the U-tube when filled with air, water and oil over the range of temperatures tested. Mean values of T_M and T_D have been calculated at each temperature.

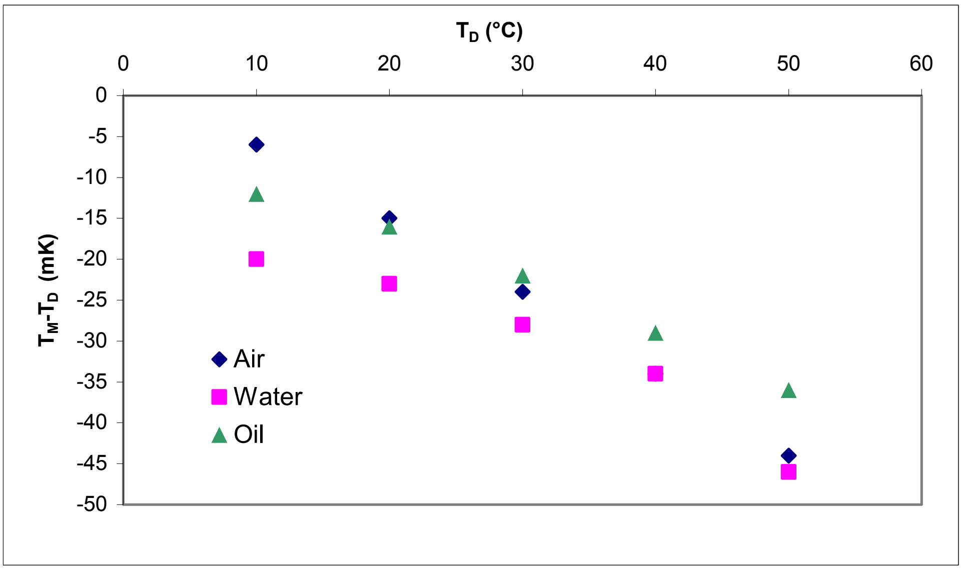

Figure 2. Difference between the measured T_M and displayed T_D U-tube temperature

4.3 Conclusions from temperature testing

It is apparent from Figure 2 that the magnitude of the temperature gradient along the U-tube is broadly proportional to the difference between the cell and the ambient temperatures. This suggests that heat is conducted through the open end of the cell by the copper connecting wires of the PRT sensor, and the heat flow from the relatively low energy Peltier device is insufficient to compensate, thereby resulting in a false temperature indication.

At 20 °C (the ambient temperature of the laboratory), the temperature of the measurement cell and laboratory would be in equilibrium and any net heat flow along the sensor wires should be insignificant. This would explain the approximately zero temperature gradient along the U-tube at a set temperature of 20 °C as shown in Figure 1.

However, as seen from Figure 2, at 20 °C the measured temperature within the U-tube is approximately 20 mK lower than the displayed temperature. This is probably due to a systematic difference between the temperature measured by the inserted PRTs and that calculated by the meter from the inbuilt ones. In any event, using the method of assessment described in this report (see 5.1), a repeatable systematic error of this magnitude in the U-tube temperature would not affect the accuracy of the measurement of an unknown liquid.

5 ASSESSMENT USING REFERENCE LIQUIDS

5.1 Basis of assessment

The aim of the assessment was to provide a series of equations which would enable the density of an unknown sample to be determined with the best achievable accuracy at any temperature within the assessment range, i.e. 10 °C to 50 °C. This was realised by testing several certified reference liquids of known density[1], together with air and pure de-saturated water[2] (see Table 1), encompassing a wide density and viscosity range.

Fluid	**Nominal density at 20 °C** ($kg\ m^{-3}$)	**Certified temperature range** (°C)	**Nominal dynamic viscosity at 20 °C** (mPa s)
Air	1.2	-	0.0
2,2,4 Trimethylpentane	692	10-50	0.5
Base lubricating oil	867	10-50	17
Lubricating oil A90	887	10-30	583
Water	998	-	1.0
Tetrachloroethylene	1 623	10-50	0.9

Table 1. Fluids used for the investigation of the Paar 5000 density meter

Measurements were taken of *Q* and *damping* of each fluid at temperatures of 10 °C, 20°C, 30 °C, 40 °C and 50 °C. Regression analysis on the measurement data enabled a best-fit polynomial equation to be derived for each discrete temperature relating fluid density to *Q* and *damping*.

5.2 Preliminary operations

The DMA5000 was configured to display, and automatically send to a printer, the following information as soon as the U-tube temperature had stabilised at the required set temperature (normally within 20 minutes):

- Sample number, date and time
- U-tube temperature as measured by the internal PRTs

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[1] Supplied by H & D Fitzgerald Ltd.

[2] De-ionised and distilled water, boiled for10 minutes to ensure de-saturation, and allowed to cool.

- Q value
- Damping value
- Density values calculated by the internal software, with and without viscosity correction

Initially, a second measurement was taken after a further 5 minutes had elapsed to check that the U-tube temperature had fully stabilised, but was later discontinued after it was established that there was no significant difference between the measurements.

5.3 Assessment procedure

At the start of each day, the normal adjustment procedure as described in the meter's Instruction Handbook [2] was performed using air and water at a temperature of 20 °C. According to the Handbook, this procedure determines the instrument constants described in Section 2.

The measurement procedure for each reference fluid was as follows:

- Water at 20 ºC
- Clean and dry U-tube
- Air at 20 ºC
- Reference liquid at maximum certified temperature
- Reference liquid at intermediate certified temperatures
- Reference liquid at minimum certified temperature
- Reference liquid at intermediate certified temperatures
- Reference liquid at maximum certified temperature
- Clean and dry U-tube
- Air at 20 ºC
- Water at 20 ºC

The U-tube was cleaned after measuring a liquid by flushing with a suitable solvent.

The meter was programmed to cycle automatically in 10 °C increments through the temperature range being measured. The measurements of water at 20 °C served to monitor the stability of the meter and also ensure that the U-tube had been effectively cleaned after testing each sample.

Each reference liquid was supplied in a sealed glass ampoule. Two samples from each ampoule were measured, except for tetrachloroethylene. With this fluid it was found that the measured density of the second sample was significantly lower than the first. Although there was an interval of about five hours between the commencement of processing the first and second samples, tetrachloroethylene is not significantly hydroscopic. The inconsistency was attributed to contamination.

5.4 Analysis of results

The following information was collated at each nominal temperature:

- The displayed temperature
- The densities of the fluids calculated at the displayed temperature
- The measured value of *Q*
- The measured values of *damping*

The densities of the reference liquids were taken from their respective calibration certificates. For water, the densities were calculated using the polynomial equation[3] given by Patterson and Morris [4]; for air, the densities were calculated using the equation of Giacomo [5].

This data was then analysed with the fixed non-linear regression option of Statistica[4] using the reference densities as the dependent variables, and *Q* and *damping* as the independent variables. Analysis was carried out both with, and without, the air data included, although in practice measurement of the density of gases using a Paar meter

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[3] Strictly, this equation is only valid for temperatures between 1 °C and 40 °C, but examination of water density determinations by other authors which do extend to 50 °C show that no significant error would be introduced by extrapolating the Patterson and Morris equation to 50 °C.

[4] Statistica for Windows, v6.0, © Statsoft, Inc.

would not normally be undertaken by NPL. This is because gases are of the order of 1 000 times less dense than liquids which leads to correspondingly higher relative measurement uncertainties, making the data less reliable when fitting to an equation.

Comparison of the reference and predicted densities (calculated from the best-fit polynomial equation) revealed a few outliers[5], so the data was re-analysed with these cases excluded.

The resulting best-fit equations all have the form:

$$\rho_T = a_T Q^2 + c_T \sqrt{D} + k_T \qquad (4)$$

or

$$\rho_T = a_T Q^2 + b_T D + c_T \sqrt{D} + k_T \qquad (5)$$

where ρ_T is the density of the test liquid at temperature T °C

a_T, b_T and c_T are the regression coefficients at temperature T °C

k_T is the intercept value at temperature T °C

The values of the coefficients and intercepts are shown in Table 2, together with the associated standard errors of the equations. As predicted, the results obtained by omitting the air data gave rise to lower standard errors; for this reason the values are taken as those with the air data excluded.

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[5] These outliers were defined as having a residual value (i.e. the difference between the reference and predicted density) of > 0.05 kg m $^{-3}$.

Assessment temperature	**Equation coefficients and intercepts**				**Standard error**
	Q^2	**Damping**	**$\sqrt{\text{Damping}}$**	**Intercept**	
T (°C)	a_T	bT	c_T	k_T	(kg m^{-3})
Data with air included					
10	285.331 6	-0.296 0	0.706 0	-1 015.932 1	0.012 8
20	285.310 5	-	-0.506 8	-1 014.683 3	0.009 1
30	285.275 9	-	-0.517 6	-1 014.551 4	0.010 4
40	285.246 9	-	-0.458 1	-1 014.728 7	0.010 3
50	285.220 7	-	-0.505 2	-1 014.461 3	0.005 9
Data with air omitted					
10	285.345 8	-0.333 3	0.886 4	-1 016.240 1	0.009 4
20	285.314 9	-	-0.5029	-1 014.724 4	0.008 7
30	285.283 8	-	-0.512 3	-1 014.618 9	0.007 6
40	285.255 4	-	-0.526 6	-1 015.525 2	0.006 7
50	285.220 7	-	-0.505 2	-1 014.461 3	0.005 8

Table 2. Summary table giving the coefficients, intercept and standard error obtained from a regression analysis of the values of *Q* and *damping* at each assessment temperature

5.5 Effect of U-tube temperature uncertainty

Although, as shown in 4.3, it was not possible to determine precisely the true temperature of a fluid in the U-tube at the time of measurement, the short-term temperature stability and repeatability was consistent to within a few mK.

The coefficients and intercepts shown in Table 2 were determined using reference densities of the test fluids calculated for the displayed temperature at the time of measurement. The density of an unknown fluid at one of the assessment temperatures can be found by measuring *Q* and *damping*, and applying the coefficients and intercept given in the table for the appropriate temperature as described in 6.1. This will give a density value accurate for the displayed temperature, even though the true fluid temperature could be offset from it by up to 40 mK.

However, a long-term drift of the meter's built-in temperature measurement system could result in significant errors, as described at 7.5.

Because of the uncertainty about the accuracy of the built in PRTs, the raw data was subsequently re-analysed but using reference densities of the test liquids calculated by assuming a temperature offset of Δ_T from the displayed temperature, where

$$\Delta_T = T_D - (20\ \mathrm{mK})$$

or

$$\Delta_T = T_D - T_M$$

In both cases, the analysis gave slightly higher residuals; the best fit for the data occurred if it were assumed that the displayed temperature was correct. No definite conclusions were drawn.

6 PRACTICAL METHODS OF DENSITY MEASUREMENT

6.1 Density measurement at an assessment temperature

The density of an unknown fluid sample at a temperature used in the assessment of the instrument can be determined by measuring the *Q* and *damping* values of the sample. These values, together with the associated coefficients and intercept given in Table 2, are substituted in Equation (4) or Equation (5) as appropriate. The amount of test liquid permitting, at least five independent measurements of the density should be made, to obtain a representative standard deviation of the mean density value.

6.2 Density measurement at an intermediate temperature

To determine the density at an intermediate temperature, or a value for the expansivity of the liquid, the *Q* and *damping* values are measured at discrete temperatures of 10 °C, 20 °C, 30 °C, 40 °C and 50 °C (or as many as is practical subject to the limitations of the liquid’s physical properties) and the density at each temperature calculated using the appropriate equation as described in 6.1. A polynomial regression analysis is then performed with density as the dependent variable and temperature as the independent variable. The resultant best-fit equation can then used to interpolate the density at any required intermediate temperature.

7 EVALUATION OF UNCERTAINTIES

The following sources will contribute to the combined measurement uncertainty when determining the density of a test fluid. A worked example of an uncertainty budget is given in Table 3.

7.1 Fluid temperature

When assessed in the manner described, calibration errors of the built-in PRTs will not affect the accuracy of measurement (see 5.5). However, any drift with time (i.e. PRT stability) will cause an error in the calculated density due to the thermal expansivity of the liquid. Experience with the characteristics of small PRTs used at NPL shows that a temperature stability of better than ±0.01 °C per year can be expected.

7.2 Equation for density calculation

The uncertainty contribution associated with using an equation for calculating the density of a liquid is taken as the standard error of the equation, as given in Table 2.

7.3 Density of reference liquids

All certified reference liquids used in this investigation had an uncertainty of ±0.005 kg m^{-3} (at k=2). The uncertainty of calculating the density of water using the Patterson and Morris equation [4] varies with temperature up to a maximum of ±0.002 8 kg m^{-3} (at k=2) at 40 °C. Therefore, the uncertainty contribution from the density values of the reference liquids is taken as ±0.005 kg m^{-3}.

7.4 Repeatability of the meter

If several measurements of a test fluid are made at a single temperature, and the density is calculated as described at 6.1, the uncertainty contribution due the meter's measurement repeatability will be given by the standard deviation of the density values obtained.

If the density is calculated at an intermediate temperature as described at 6.2, the uncertainty contribution will be the standard error of the polynomial equation used to fit the measurement data.

7.5 Stability of the meter

Because the density of de-ionised and distilled water can be calculated to better than ± 3 ppm, it provides a good medium with which to assess the long-term stability of the Paar meter. The meter was first checked by NPL in October 2001 with pure water, at a displayed temperature of 20 °C, and gave values for Q in the range 2.656 748 and 2.656 749 for 5 samples. During the testing for this report, the value for Q determined on 37 samples varied from 2.656 753 to 2.656 761, showing a distinct trend to increase in value over the three week period. Performing the manufacturer’s adjustment procedure as described in the Instruction Handbook [2] had no noticeable effect on this change.

Several factors could account for this slow but definite increase: very slight contamination of the U-tube, a drift in the oscillation measurement system of the meter or a drift in cell temperature measurement due to either the built-in PRT's or the meter's resistance bridge. More experimentation over a period of time will be needed to establish whether this drift continues, but it represents a change in the calculated value of the density of water during the course of this evaluation of about ± 0.009 kg m^{-3}.

7.6 Typical uncertainty budget

Table 3 gives an example of an uncertainty budget for tetrachloroethylene, a liquid with a high cubic expansivity (1.06 x 10^{-3}), measured at 20° C.

Uncertainty Contribution		**Divisor**	**Input standard uncertainty**	**Output standard uncertainty (± kg m^{-3})**
PRT stability	± 0.01 °C	√3	± 0.005 8 °C	± 0.010
Density equation	± 0.010 kg m^{-3}	1	± 0.010 kg m^{-3}	± 0.010
Reference liquid	± 0.005 kg m^{-3}	2	± 0.002 5 kg m^{-3}	± 0.003
Measurement repeatability	± 0.002 kg m^{-3}	1	± 0.002 kg m^{-3}	± 0.002
Meter stability	± 0.009 kg m^{-3}	√3	± 0.005 2 kg m^{-3}	± 0.006
			Combined uncertainty (k=1)	± 0.016
			Expanded uncertainty (k=2)	**± 0.04**

Table 3. Estimation of the uncertainty of the density of tetrachloroethylene

8 CONCLUSIONS

The Paar DMA5000 has proven a valuable addition to the measurement capability of the NPL Mass and Density section. It provides a much faster and more efficient method of measuring small liquid samples with greater accuracy than the Paar DMA55. A criticism is that the internal PRTs cannot be removed for periodic calibration. However, assessing the instrument in the manner described will negate errors arising from systematic temperature offsets, provided the instrument is re-assessed and the coefficients are re-calculated periodically.

The drift in the resonant frequency of the U-tube with time is something that requires monitoring, and it is suggested that initially the meter should be checked with reference liquids, and revised equations established at yearly intervals. At that time, a re-evaluation of the temperature measurement capability of the meter will help establish this contribution. Since slight contamination of the U-tube is a possible cause of the drift, particular care should be taken to clean the U-tube thoroughly after use. For the lubricating oils, it was found that a satisfactory method was to flush thoroughly with diethyl ether followed by acetone[6].

9 ACKNOWLEDGEMENTS

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