

# Elemental mercury vapour in air: the origins and validation of the 'Dumarey equation' describing the mass concentration at saturation

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Received: 14 December 2009 / Accepted: 1 February 2010  
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**Abstract** The Dumarey equation has been the dominant relationship used to calculate the mass concentration of saturated elemental mercury vapour in air for the calibration of mercury vapour measurement equipment for over 25 years. However, the origin of the equation, and the validation data supporting its accuracy have never been published. This paper addresses that deficiency, compares the Dumarey equation with other data sets to which it has been wrongly attributed in the past, and describes why it remains superior to the use of mercury vapour pressure data in combination with the ideal gas law, for the purposes of calibrating mercury vapour measurement equipment.

**Keywords** Dumarey equation · Mercury vapour · Vapour pressure · Atomic spectrometry · Saturated mass concentration

## Introduction

Mercury has been recognised as an important environmental pollutant for many years. Its toxicity, cumulative health effects and potential for accumulation in terrestrial and aquatic biosystems has resulted in extensive research

into its emissions, transport, reactions, fate, and concentrations in environmental matrices, and effects on human health and environmental sustainability [1].

In particular, the pollution of ambient air by mercury has recently been of increasing interest. The population is exposed to mercury in ambient air mainly via mercury vapour (sometimes referred to as gaseous mercury), and the vast majority of this mercury vapour is elemental mercury [except at some industrial and coastal locations where a significant proportion of the gaseous mercury may comprise oxidised mercury in compounds such as mercury (II) chloride, or organics such as methyl mercury]. We henceforth use 'mercury vapour' to refer to 'elemental mercury vapour'.

Usually the final analysis step for mercury vapour determination is performed using cold vapour atomic fluorescence spectrometry (CVAFS), cold vapour atomic absorption spectrometry (CVAAS), or Zeeman atomic absorption spectrometry (ZAAS). All these measurements require calibration, a property which is not just limited to measurements of mercury vapour in ambient air, but is generic to all measurements whose final step is a determination of the amount or mass concentration of mercury in the gaseous phase. This is true for determination of mercury in water using purge and trap methodologies such as EN 1483 [2] and ISO 17852 [3]. All these measurements are ultimately dependant on the mass concentration of saturated elemental mercury in air at a given temperature, as this relationship underpins the calibration procedure.

The mass concentration of saturated mercury vapour in air has often been derived indirectly via the ideal gas law using a variety of empirical equations describing the vapour pressure of mercury, dating back to 1801, determined using a variety of experimental methods [4]. Rarely, though, did these measure the vapour pressure of mercury

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in air directly. The recent work [5, 6] of Working Group 25 of the European Committee for Standardization's Technical Committee on Air Quality (CEN/TC 264/WG 25—of which authors RJCB, WTC and PBS are active members) to develop methods for the measurement of mercury vapour in air and mercury in deposition in order to satisfy the implementation of the European Commission's Fourth Air Quality Daughter Directive [7], and the US EPA's requirement to enforce the Clean Air Mercury Rule [8], has brought into sharp focus the requirement to define the most suitable relationship to use to describe the mass concentration of saturated mercury in air when calibrating measurements of mercury vapour.

The 'Dumarey equation' has been the dominant relationship used to calculate the mass concentration of saturated mercury vapour in air for the calibration of mercury vapour measurement equipment for over 25 years. However, the origin of the equation, and the validation data supporting its accuracy have never been published. We now address that deficiency and compare the Dumarey equation with other data sets to which it has been wrongly attributed in the past, and describe why it remains superior to the use of mercury vapour pressure data in combination with the ideal gas law.

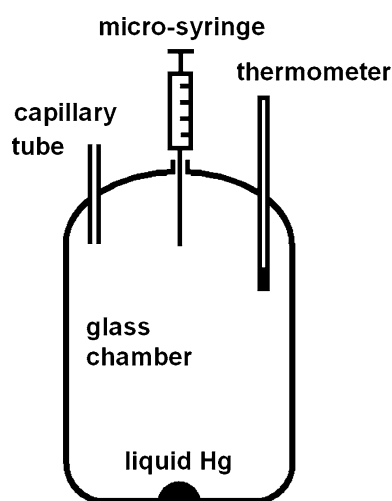
## Results and discussion

From 1978 to 1984, Ronny Dumarey was working towards a PhD at the University of Gent in Belgium developing a simple method to measure mercury in ambient air by CVAAS. The commonly used calibration method at that time was the reduction of mercury-containing solutions with  $\text{SnCl}_2$  followed by the purging of these solutions with inert gas to liberate the elemental mercury as mercury vapour [9]. Preparing and analysing mercury-containing solutions at the low concentrations required (in the ng/ml range) was time-consuming and also problematic owing to: instability over relatively short timescales [10, 11] meaning that fresh solutions needed to be prepared for every calibration, relatively high mercury concentrations in the blank reagents, and the requirement for numerous dilution steps thereby increasing the measurement uncertainty. The use of aqueous calibration solutions was therefore the limiting factor in terms of the cost and accuracy of the measurement at this time.

The solution to this problem was the calibration of the measurement using air saturated with elemental mercury vapour. This was achieved by employing a glass calibration vessel, open to the atmosphere via a capillary tube, with a small amount of elemental mercury at the bottom, such that a mercury-saturated atmosphere is allowed to develop within the vessel. A known volume of mercury-saturated

air may then be removed from the vessel through a septum, using a gas-tight syringe, and then used for calibration purposes. The mass of mercury within the volume removed is a function of the temperature of the air inside the calibration vessel, as recorded by a thermometer inserted into the vessel. A diagrammatic representation of such a calibration vessel, often known as a 'bell-jar', is shown in Fig. 1.

Having developed this calibration vessel, the remaining problem was to quantify the saturated mercury mass concentration in the air within the calibration vessel, and how this varied with temperature. Whilst many vapour pressure relationships are available for mercury [4], it was unclear which one should be used and, more fundamentally, whether the ideal gas law is appropriate to convert vapour pressures measured in vacuum into mass concentrations in air, because of the possible non-ideal behaviour of mercury vapour in air. The solution was to produce new data, which directly measured the mass concentration of saturated mercury vapour in air. Therefore, an extensive experimental program to determine these data at various temperatures was undertaken. This involved comparing the analytical responses measured for the known volumes of mercury-saturated air removed from the calibration vessel, to the analytical responses obtained from the measurement of known masses of mercury in standard solutions (prepared from  $\text{HgCl}_2$  and stabilised using cysteine) using the reduction and purge method, and capturing the mercury vapour evolved onto gold-coated silica absorption tubes. Following thermal desorption of the mercury vapour from the absorption tubes, measurements were made by CVAAS using a Coleman MAS-50 spectrometer [12]. The experiments were performed for calibration vessel temperatures



**Fig. 1** Diagrammatic representation of a calibration vessel for generating mercury-saturated air, at known temperatures, often known as the 'bell-jar'. The micro-syringe removes mercury-saturated air via a septum in the top of the vessel

of between 288 and 298 K and for mercury masses of between 0 and approximately 50 ng. In this way a determination of the mass concentration of saturated mercury vapour in air, at a given temperature,  $T$ , could be given by:

$$\gamma_{\text{Hg},T} = \left( \frac{m_{\text{std}}}{V_{\text{cal},T}} \right) \left( \frac{I_{\text{cal},T}}{I_{\text{std}}} \right) \quad (1)$$

where  $\gamma_{\text{Hg},T}$  is the mass concentration of saturated mercury vapour in air, at a given test temperature,  $T$ , in ng/ml,  $m_{\text{std}}$  is the mass of mercury in the standard solution, in ng,  $V_{\text{cal},T}$  is the volume of mercury-saturated air removed from the calibration vessel, at a given test temperature,  $T$ , in ml,  $I_{\text{cal},T}$  is the blank corrected analyser response from the measurement of the mercury-saturated air removed from the calibration vessel,  $I_{\text{std}}$  is the blank corrected analyser response from the measurement of the mercury in the standard solution.

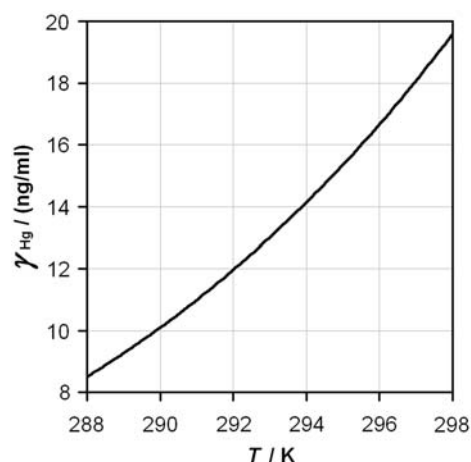
An empirical relationship between the mass concentration of saturated mercury vapour in air and temperature was calculated as the least squares best fit to a simplified Antoine-type equation [13] (often called an August equation) using all the data obtained (with the limited computing power in the 1980s) [14]. This relationship, now known commonly as the Dumarey equation, is given by:

$$\gamma_{\text{Hg}} = \frac{D}{T} 10^{-(A+[B/T])} \quad (2)$$

where  $\gamma_{\text{Hg}}$  is the mass concentration of saturated mercury in the air inside the calibration vessel, in ng/ml,  $T$  is the temperature of the air inside the calibration vessel, in K,  $A$  is a constant equal to  $-8.134459741$ ,  $B$  is a constant equal to  $3240.871534$  K,  $D$  is a constant equal to  $3216522.61$  K ng/ml.

The Dumarey equation is plotted between 288 and 298 K, the temperatures between which the relationship was tested, in Fig. 2. Those carrying out measurements of mercury vapour in ambient air over very short timescales (usually employing automatic instrumentation) often need very small masses of mercury for calibration, and choose to cool the calibration vessel below 288 K rather than use very low volume syringes. Whilst the equation is not validated below 288 K it continues to perform well against other relationships when extrapolated to lower temperatures, as shall be shown later. There are also other important practical issues to be considered when performing calibrations, at any temperature, the details of which are covered elsewhere [15].

The accuracy of the Dumarey equation was subsequently validated by the measurement of a number of certified reference materials in different matrices. Measurements were made by pyrolysis followed by the capture of the mercury liberated on absorption tubes containing gold-coated silica, and then subsequent measurement by thermal desorption and CVAAS [16, 17]. Much of this



**Fig. 2** The mass concentration of saturated mercury in air predicted by the Dumarey equation between 288 and 298 K

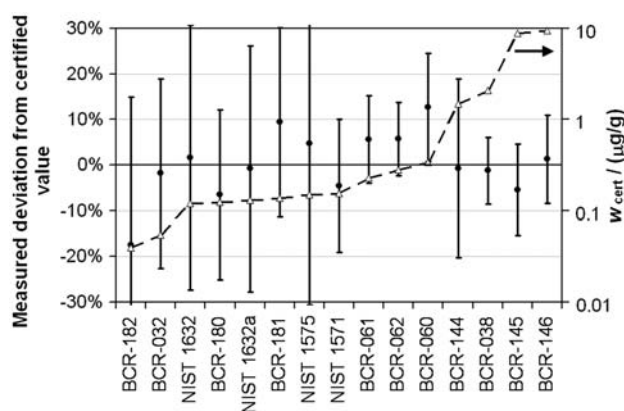
analysis was done as part of intercomparison exercises organised by the European Commission for the certification of BCR (Community Bureau of Reference—the former reference materials programme of the European Commission) reference materials. The results of this validation are shown in Table 1, and are displayed graphically in Fig. 3. The mean relative difference between certified and measured values across all the reference materials measured was 0.17% and in all but one case there was agreement between certified and measured values at the 95% confidence level. This is a good indication that the systematic bias between the Dumarey equation and reality is small. Figure 3 also shows that the performance of the equation is not dependent on the total mass being measured as there is also no evidence of differential bias as the masses measured change, only larger random uncertainties as the certified mass fractions decrease.

The Dumarey equation, and its supporting validation data, was never formally published in the peer-reviewed literature, although the process of establishing the relationship was alluded to in the publication which described the method of using mercury-saturated air in a glass vessel for calibration [18]. The equation was spread mainly by personal communication and was recommended by the instrument manufacturers making the first commercially available CVAAS and CVAFS systems. Indeed, a description of such a system, in 1989, is thought to represent the first explicit appearance of the Dumarey equation (although not attributed at this stage) in the literature [19]. The relationship has been increasingly used over the last 25 years as measurements of mercury vapour have become more widespread and numerous, becoming colloquially known as the Dumarey equation, but only very rarely referred to as such in the literature [20]. It is now the dominant relationship used to calculate mass

**Table 1** The certified Hg mass fractions,  $w_{\text{cert}}$ , and measured Hg mass fractions,  $w_{\text{meas}}$ , in the certified reference materials measured during the validation of the Dumarey equation

Certified Reference Material	$w_{\text{cert}}/(\mu\text{g/g})$	$w_{\text{meas}}/(\mu\text{g/g})$
NIST SRM 1571 Orchard Leaves	$0.155 \pm 0.015$	$0.148 \pm 0.016$
NIST SRM 1575 Pine Needles	$0.15 \pm 0.05$	$0.157 \pm 0.018$
NIST SRM 1632 Trace Elements in Coal (Bituminous)	$0.12 \pm 0.02$	$0.122 \pm 0.029$
NIST SRM 1632a Elements in Coal (Bituminous)	$0.13 \pm 0.03$	$0.129 \pm 0.018$
BCR-032 Moroccan phosphate rock	$0.055 \pm 0.011$	$0.054 \pm 0.003$
BCR-038 Fly ash from pulverised coal	$2.10 \pm 0.15$	$2.074 \pm 0.035$
BCR-060 Lagarosiphon major	$0.34 \pm 0.04$	$0.383 \pm 0.007$
BCR-061 Platihypnidium riparoides	$0.23 \pm 0.02$	$0.243 \pm 0.010$
BCR-062 Olea europaea	$0.28 \pm 0.02$	$0.296 \pm 0.011$
BCR-144 Sewage sludge (domestic)	$1.49 \pm 0.22$	$1.48 \pm 0.19$
BCR-145 Sewage sludge (mixed origin)	$8.82 \pm 0.88$	$8.34 \pm 0.04$
BCR-146 Sewage sludge (industrial)	$9.49 \pm 0.76$	$9.61 \pm 0.53$
BCR-180 Gas coal	$0.123 \pm 0.008$	$0.115 \pm 0.020$
BCR-181 Coking coal	$0.138 \pm 0.011$	$0.151 \pm 0.029$
BCR-182 Steam coal	$0.040 \pm 0.007$	$0.033 \pm 0.009$

The value for BCR-032 is only indicative. Expanded uncertainties are quoted at the 95% confidence interval

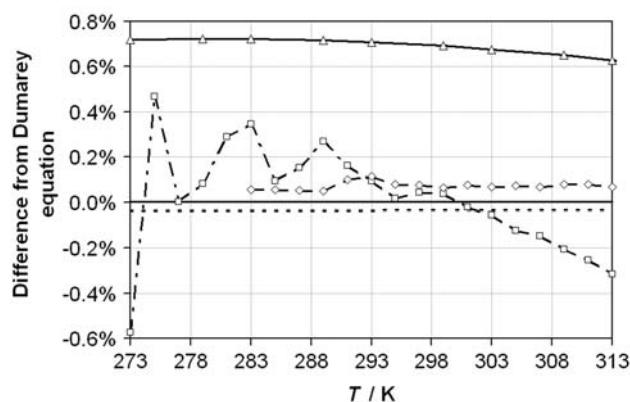


**Fig. 3** The Hg mass fractions measured in several certified reference materials during the validation of the Dumarey equation expressed as a percentage deviation from the certified value (*solid circles*), left hand axis. The uncertainties in these values have been calculated as the relative uncertainties in the certified and measured values added in quadrature, and expressed at the 95% confidence interval. The right hand axis displays the certified Hg mass fraction in the certified reference materials,  $w_{\text{cert}}$  (*open triangles*)

concentrations of saturated mercury vapour in air when calibrating measurements of mercury vapour.

One of the advantages of the Dumarey equation is that it can be used to gauge the mass concentration of saturated mercury in air at any temperature between 288 and 298 K and not just at the discrete temperatures for which data is often supplied in reference handbooks. The Dumarey equation shows fortuitous agreement with the commonly used data given in the 52nd and 60th Editions of CRC Handbook of Chemistry and Physics (which contain identical data) [21, 22]—to both of which the Dumarey equation is often wrongly attributed on occasions. (The CRC Handbook has been cited here as it is the source to

which most people refer, although it is recognised that in most cases this is not the original source of the data.) It also agrees fortuitously with the Knudsen relationship [23], which is also commonly used and to which the Dumarey equation is periodically similarly wrongly attributed. The reason this agreement is fortuitous is because these relationships are based on data sets expressed as vapour pressures converted into mass concentrations using the ideal gas law. This then suggests that either these determinations of mercury vapour pressure were made in air and this conversion is appropriate, or they were made in vacuum where this conversion is not appropriate but they exhibit a serendipitous negative bias. A comparison between these relationships is shown in Fig. 4. Figure 4 also displays selected data from ISO 6978 for the determination of mercury in natural gas [24], which is thought to be based on the Dumarey equation, with the discrepancy observed thought to occur from rounding errors (only four significant figures are quoted in the tabulated data) and the use of a numerical factor of 273.16, rather than 273.15, when converting between kelvin and degrees celsius (possibly as a result of confusion between the IPTS-68 and the ITS-90 temperature scales). Included in Fig. 4 are two other more recent applications of the Dumarey equation: the relationship used by CEN/TC 264/WG 25 in their draft standard for total gaseous mercury measurement [25], and the relationship used by Brown and co-workers during a recent series of publications describing research into ambient mercury vapour measurement [15, 20, 26, 27]. These publications also represent the only other instances of the Dumarey equation being explicitly stated in the published literature, to the best of the authors' knowledge. The only difference between these relationships and the Dumarey equation are the number of significant figures



**Fig. 4** The difference observed at the temperatures indicated between the Dumarey equation and the: Knudsen data [23] (triangles, solid line), CRC Handbook of Chemistry and Physics 52nd and 60th Edition data [21, 22] (squares, dot-dashed line), ISO 6978 data at selected temperatures [24] (diamonds, dashed line), and the 'Dumarey-based' relationships: CEN/TC 264/WG 25 (solid line), and Brown and co-workers [15, 20, 26, 27] (dotted line)

used to describe the constants  $A$ ,  $B$  and  $D$ . Dumarey stated  $A$ ,  $B$  and  $D$  to 10, 10 and 9 significant figures, respectively, in the original Dumarey equation. CEN/TC 264/WG 25 used 6, 6 and 7 significant figures, respectively (resulting in a relative average discrepancy of approximately +0.002%), whilst Brown and co-workers used 5, 5 and 7 significant figures, respectively (resulting in a relative average discrepancy of approximately −0.04%). These discrepancies are negligible when compared to the uncertainty of any relevant analytical measurement. Indeed it is noteworthy that even the use of only four significant figures for constants  $A$ ,  $B$  and  $D$  still results in a discrepancy of less than −0.2% from the original Dumarey equation.

It is important to emphasise that the intention when the Dumarey equation was produced was not to prove the applicability of the ideal gas law for mercury vapour in air nor to judge the correctness of existing published data, but to provide a method to replace the time-consuming solution-based calibration method with a faster but equally accurate procedure. In the process, a relationship to describe the mass concentration of saturated mercury vapour in air was derived, which was independent of any previous data. These considerations provide important background against the recent increase in interest surrounding identification of the 'correct mercury vapour pressure equation', which has been provoked by the requirements of the US EPA to support enforcement of the Clean Air Mercury Rule, and of the European Commission to support the requirements of the Fourth Air Quality Daughter Directive.

In this context, the Dumarey equation provides a validated direct measure of the mass concentration of saturated

mercury vapour in air. Other data commonly used are generally determinations of the vapour pressure of mercury (usually in vacuum and rarely in air) from which a link is made to the mass concentration of saturated mercury vapour in air, via the ideal gas law. In order to make this link one must be certain that the mercury behaves as an ideal gas under the conditions in the calibration vessel, else quantify this non-ideality. Neither requirement is easy to fulfil. Indeed, as we have previously suggested [27], and have had recently confirmed [28], it seems that mercury does not behave ideally under the conditions encountered in the calibration vessel—possibly as a result of interaction between the mercury atoms and the component atoms and molecules of air. Hence, whilst new correlations for the vapour pressure of mercury [4] may therefore be correct [28], they are not necessarily appropriate for calculating the mass concentration of saturated mercury vapour in air for the calibration of mercury vapour measurement equipment. For this purpose we need a relationship that is a direct measure of the mass concentration of saturated mercury in air, not involving the use of the ideal gas law. The Dumarey equation answers this practical requirement, has been used in this capacity for over 25 years, and has recently been shown to be correct within the uncertainty of measurement of modern techniques [28].

## Conclusions

We have presented for the first time the origins of the Dumarey equation for calculation of the mass concentration of saturated mercury vapour in air, and the validation data supporting the accuracy of this equation. We have also compared the equation with other mercury vapour pressure relationships to which it is often wrongly attributed. We intend that this presentation of the data underpinning the relationship will elucidate its independence from other data sets, and improve understanding of the distinction between the Dumarey equation and other data, based on the vapour pressure of mercury, to which it is compared. The advantages of using a data set that does not rely on use of the ideal gas law for its implementation to calibrate mercury vapour measurements have been presented. The accuracy of the Dumarey equation, and the dangers of using alternative equations based on the vapour pressure of mercury, have recently been confirmed using state-of-the-art measurement techniques [28]. We hope this demonstrates why the Dumarey equation remains the most appropriate relationship to describe the mass concentration of saturated mercury vapour in air for the calibration of mercury vapour measurements over a quarter of a century after it was first produced.

Furthermore, it now seems appropriate that the Dumarey equation be used exclusively in future by all legislators,



instrument manufacturers, and scientists involved in the measurement of mercury vapour and be included in all relevant future European and international standards, or incorporated into existing standards when they come up for technical review. For this purpose the constants  $A$ ,  $B$  and  $D$  in the Dumarey equation should always be quoted to a minimum of five significant figures, but preferably more. Given the increasingly common requirement for very small calibration masses of mercury for high time resolution measurements, and the Dumarey equation's continued good agreement with other data sets above and below the temperature range over which it has been validated, we would propose that it may be used for the calculation of the mass concentration of saturated mercury vapour in air for all temperatures between 273 and 313 K. Furthermore, based on comparison with current state-of-the-art measurement techniques [28], we suggest that an indicative relative uncertainty for the Dumarey equation of approximately 2% would be appropriate.

**Acknowledgments** The UK Department for Business, Innovation and Skills' funding of the National Measurement System Chemical and Biological Metrology Programme is gratefully acknowledged. The very useful discussions with members of CEN/TC 264/WG 25 and with Dr Steve Long (NIST) are gratefully acknowledged.

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