

Australian Government Department of Industry, Innovation and Science National Measurement Institute

#### Establishment of an Ultra-High Accuracy 670 PVTt Gas Flow Primary Standard at NMIA

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## In this Presentation:

- The Need of a New Standard
- PVT Design Methodology
- Uncertainty Analysis & Working Example
- Conclusions

#### **NMI Gas Flow Facility**

### Currently used at NMIA,

 $\Box$ 2 x primary standards:

□ 300L Bell Prover (±0.1%)

□5 x Mercury Sealed Piston Provers (±0.084-0.100%)

□ 3 x Nozzle Arrays used as transfer standards for flowrates from 0.005 to 500 m<sup>3</sup> h<sup>-1</sup>:

□Rotor Sonic Nozzle Array, or RSNA (0.005 to 8 m<sup>3</sup> h<sup>-1</sup>)

□Satellite Sonic Nozzle Array, or SSNA (0.5 to 180 m<sup>3</sup> h<sup>-1</sup>)

□Rotor Sonic Nozzle Array, or RSNA (50 to 300 m<sup>3</sup> h<sup>-1</sup>)

#### **NMI Gas Flow Facility**



300L Bell Prover at NMIA



Schematic Diagram of the Critical Flow Venturi Sonic Nozzle Array at NMIA

#### **NMI Gas Flow Facility**

Completed recently at NMIA,

□670L <u>Pressure</u>, <u>Volume</u>, <u>Temperature and time</u> (PVTt) primary standard (<u>Discussed today</u>!)

□7000 m<sup>3</sup> h<sup>-1</sup> Critical Flow Venturi Nozzle Array (nicknamed the Blue Spaghetti Monster Array or BSMA)

#### **NMIA Gas Flow Facility**

Disadvantages of using bell and mercury-sealed piston provers:

- (1) safety concerns due to the use of mercury and oil as seals in these standards,
- (2) the limitation on using these standards for measurement with pressures higher than atmospheric due to the oil and mercury liquid seals,
- (3) difficulty in reducing the large spatial temperature non-uniformity in and around these standards while used in ambient air, which is currently assessed to be 150 mK, and
- (4) difficulty in determining the volumes of these provers to better than 400 ppm (0.04%).

#### **PVTt System**

What is a PVTt System?

- A well-determined volume with ultra-accurate pressure, temperature and chemical composition measurements used to calculate the density of gas inside this volume to determine the gas mass.
- Used mainly in calibrating nozzles by having a diverter valve with a start-stop timer.
- Usually housed inside a water tank for best temperature uniformity and stability (reducing a major source of uncertainty).

#### **PVTt System**

Reasons to have a PVTt System:

- (1) replace the existing bell and mercury-sealed piston provers,
- (2) increase the range of flowrate up to 120 kg h<sup>-1</sup> as well as increasing CVFN calibration pressures to 7 bars, and
- (3) improve the measurement uncertainties from  $\pm 0.1\%$  to better than  $\pm 0.02\%$ .

#### **PVTt System**

#### NMIA's 670L PVTt System









#### **PVTt System - Design Methodology**

Design was based on existing standards but with several improvements made on:

Design and Mode of Operation.

□ PVTt Tank Volume determination.

□ Water Tank Temperature Control/Uniformity.

#### **PVTt System – Design & Mode of Operation**

Design and mode of operation

- □ Based on annular cylinders to allow heat transfer from the outside and inside walls.
- □ Placed vertically for better convective heat transfer.
- □ Fast 3-way valve to minimise uncertainty associated with Start/Stop of flow (<2ms).
- □ Full automation of calibration to minimise user errors and streamline data collection.
- Reduction of inventory volume as compared to that of total volume (<25 mL), hence leading to negligible effect on measurements.</p>

#### **PVTt System - Design & Mode of Operation**





#### **PVTt System – Volume Determination**

Two measurement methods were employed:

□ Gravimetric method using water – conducted by NMIA MRQ team with an uncertainty of ±80 ppm.

□ Gravimetric method using nitrogen (aka expansion method) – conducted by Gas Flow and Chemical Metrology Groups with assistance from Dr Li Chunhui (NIM) with an uncertainty of ±221 ppm.

#### **PVTt System – Volume Determination**



#### **PVTt System – Tank Temperature**

Water Tank Temperature Control/Uniformity

- Water bubbling at centre of cylinders to mix water and provide cooling effect.
- PID controller connected to heater elements and an SPRT for accurate temperature control.
- Seven PRTs used at various locations inside the tank to assess uniformity at all times – better than 2 mK observed.

#### **PVTt System – Tank Temperature**



Graph of temperature measurement of NMIA water bath versus time. Standard deviation was calculated to be 0.9mK

#### **PVTt System - Uncertainty Analysis**

Mathematical model used (conservation of mass):

$$Q_{m} = \frac{(m_{PVTt}^{e} - m_{PVTt}^{s}) + (m_{Inv}^{e} - m_{Inv}^{s})}{t} , \text{ or}$$
$$Q_{m} = \frac{V_{Inv}(\rho_{Inv}^{e} - \rho_{Inv}^{s}) + V_{Inv}(\rho_{Inv}^{e} - \rho_{Inv}^{s})}{t}$$

For a mathematical model with correlated uncertainty components, the following equation can be used:

$$u_c^2(y) = \sum_{i=1}^N c_i^2 u^2(x_i) + 2 \sum_{i=1}^{N-1} \sum_{j=i+1}^N c_i c_j u(x_i) u(x_j) r(x_i, x_j)$$

For fully correlated components,  $r(x_i, x_j) = 1$ . For non-correlated components,  $r(x_i, x_j) = 0$ .

#### **PVTt System - Uncertainty Analysis**

The uncertainty components  $V_{PVTt}$ ,  $V_{Inv}$  and t are obtained by various measurement methods and therefore considered to have non-correlated uncertainties, hence their  $r(x_i, x_j) = 0$ .

On the other hand, the two sets of densities,  $(\rho_{PVTt}^e, \rho_{PVTt}^s)$  and  $(\rho_{Inv}^e, \rho_{Inv}^s)$ , are calculated from measurements of pressure and temperature in conjunction with a published equation of state and therefore considered to be correlated.

It follows from the above:

$$u_{Q_{m}} = \sqrt{\frac{\left(c_{V_{PVTt}}u_{V_{PVTt}}\right)^{2} + \left(c_{\rho_{PVTt}}u_{\rho_{PVTt}}\right)^{2} + \left(c_{\rho_{PVTt}}u_{\rho_{PVTt}}\right)^{2} + \left(c_{V_{Inv}}u_{V_{Inv}}\right)^{2} + 2c_{\rho_{PVTt}}c_{\rho_{PVTt}}c_{\rho_{PVTt}}u_{\rho_{PVTt}}u_{\rho_{PVTt}}r(\rho_{PVTt}^{e},\rho_{PVTt}^{s}) + 2c_{\rho_{Inv}}c_{\rho_{Inv}}u_{\rho_{Inv}}u_{\rho_{Inv}}r(\rho_{Inv}^{e},\rho_{Inv}^{s})}$$

#### **PVTt System – Working Example**

A CFVN with a nominal diameter of 2 mm was connected in series with the PVTt standard. The starting pressure in the PVTt tank was set to ~100 Pa. Dry nitrogen, with a purity better than 99.999% produced by the boil-off of liquid nitrogen at NMIA cryogenic facility, was allowed to flow into the PVTt tank through the CFVN for a period of 420 s giving an end pressure of ~39 kPa. The temperature of the tank's water was set to 20.450°C and this set point was maintained within  $\pm 0.9$  mK. Measurements of the PVTt tank pressures and water temperatures were recorded. These measurements were repeated seven times.

#### **PVTt System – Working Example – Uncertainty**

Gas used: dry nitrogen, N <sub>2</sub> Starting Pressure = 100 Pa, End Pressure = $39 \times 10^3$ Pa Temperature = $20.450^{\circ}$ C, $V_{PVTt}$ = 0.663 358 m <sup>3</sup> , $V_{Inv}$ = $18 \times 10^{-6}$ m <sup>3</sup> , $t$ = 420 s					
Components	<b>u</b> (@1SD or <i>k</i> =1)		Source		
Componente		ppm	000100		
V <sub>PVTt</sub>	27 mL	40	Cal report		
$\rho_{PVTt}^{e}$					
Pressure	5 Pa	132	[6]		
Temperature	6 mK	21	[6]		
Equation	2.2×10 <sup>-5</sup> kg m <sup>-3</sup>	50	[5]		
$\rho_{PVTt}^{s}$					
Pressure	5 Pa	131	[6]		
Temperature	6 mK	0.06	[6]		
Equation	6.2×10 <sup>-8</sup> kg m <sup>-3</sup>	0.2	[5]		
V <sub>Inv</sub>	0.18 mL	0.1	[6]		
$\rho_{Inv}^e$					
Pressure	5 Pa	0.004	[6]		
Temperature	6 mK	<0.001	[6]		
Equation	2.3×10 <sup>-5</sup> kg m <sup>-3</sup>	0.002	[5]		
$\rho_{Inv}^{s}$					
Pressure	5 Pa	0.004	[6]		
Temperature	6 mK	<0.001	[6]		
Equation	2.3×10 <sup>-5</sup> kg m <sup>-3</sup>	0.001	[5]		
t	5×10 <sup>-3</sup> s	12	[6]		
if all uncertainties are non-correlated $u_{Q_m}(k=1, r=0) = 182 \text{ ppm}$					
Let $r = 0.95$ (highly correlated uncertainties)					
$u_{Q_m}(k=1, r=0.95) = 57.8 \text{ ppm}$					
$U_{Q_m}$ (k = 2, r = 0.95) = 116 ppm					

Components		<b>u</b> (@1SD or <i>k</i> =1)		Source	
			ррт	oource	
$Q_{m,STD}$		3.9×10 <sup>-8</sup> kg s <sup>-1</sup>	58	Table 1	
$\rho_N$					
	Pressure	7.9 Pa	39	Combined	
	Temperature	4 mK	7	Combined	
	Equation	5.9×10⁻⁵ kg m⁻³	25	[5]	
$p_N$		7.9 Pa	39	Combined	
Repeatability		1.4×10 <sup>-10</sup> m <sup>2</sup>	15	Measured	
(7 trials)					
$u_N(k=1, r=0) = 85.5 \text{ ppm}$					
$U_N(k=2, r=0) = 171 \text{ ppm}$					

#### Conclusions

- NMIA's gas flow measurement uncertainty has been improved from ±0.1% to ± 0.012% credited primarily to better volume determination of ±0.008% and stringent water temperature control and uniformity of 2 mK.
- Further improvements on the uncertainty can be achieved (1) by using more accurate transducers, and (2) increasing the correlation among various components from highly correlated, r = 0.95, to fully correlated, r = 1.
- Improvements on the measurement uncertainty of calibrating a CFVN can be made by (1) using better pressure transducers, and (2) placing the CFVN in a more uniform and better controlled temperature environment (water tank!).
- Preliminary comparison with NMIA standards has shown good agreement however a formal comparison that involves other national institutes around the world is needed.

# Thank you Questions?