SOURCE MONITORING
Estimating Measurement Uncertainty for Particulate Emissions from Stationary Sources

Presented by
Gerald Woollatt
LEVEGO
Overview

- Introduction
- Main objectives of the study
- Literature review / overview
- Key findings of Dutch study (1999)
- Data and Methodology
- Sampling Suitability Matrix
- Discussion
- Summary and conclusions
Introduction

- Particulate matter is a requirement for almost every listed process or category under the section 21 regulations.
- A relatively recent requirement is not only to report a measured result for compliance purposes but a reported uncertainty of the result needs to be reported with the result.
Introduction contd.

- The total uncertainty of the final reported emission result is difficult to quantify due to the physical properties of particulate matter.
- Tends to be inhomogeneous in the off-gas stream.
- Isokinetic sampling therefore required utilizing an internationally recognized validated method such as ISO 9096:2003 or US EPA Method 5 etc.
- Good quality data are essential in the decision making process for both the plant operator and regulator.
Decisions made on questionable data may lead to costly mistakes leading to wasteful expenditure and non action from the regulatory authorities.
Main Objectives of the Study

- Air quality still in its relative infancy in South Africa
- This project aims to coincide with the demands of the new legislation to ensure better data quality and reliability of reported emission results
- Ascertain the validity of source emission data through applying standardized statistical methods as described in the GUM 1995.
- To allow for a standardized method of assessment of data quality to be carried out by the regulator and plant operator.
**Limited studies and literature in the field of stack emission sampling have been conducted.**

**Assumption that the greatest components contributing to the uncertainty of the final measurement result are those factors / variables outside the control of the measurement technician namely:**

- Environmental conditions
- Plant operating conditions
- Sampling location
From the assumptions above several questions can be asked;

- Do the above variables have the greatest influence on the accuracy of the results obtained?
- Can the overall uncertainty of the measurement result be determined statistically?
- Can a suitable method of evaluating the acceptability of final emission data be developed?
In an attempt to answer these questions two sampling campaigns were conducted;

First sampling campaign was conducted on a plant were all the minimum requirements of the sampling method were complied with.

Second sampling campaign was conducted were all the minimum requirements were met except for the sampling location and process operating conditions.
The two sampling campaigns were compared to determine what influence the non-compliance of the sampling location and process operations would have on the final report emission results.

Worldwide the trend in industry is to build plants as compactly and cost effectively as possible.

Minimal consideration taken into account for sampling location compliance and potential influence on accuracy of emission results obtained.
Key findings of Dutch Study (1999)

- According to the study the results for the reproducibility were disappointing.
- Study found that off gas characteristics such as high water vapour content has a significant influence on the accuracy of the results obtained, led to classification or distinction to be made between “simple” and “difficult” sources.
- The reproducibility was estimated at approximately 44% RSD and a similar order of magnitude for all of the various concentration ranges measured.
Key findings of Dutch Study Contd.

- Errors and uncertainties with regards to temporal and spatial variations are too great to allow much value to be derived from an in-depth statistical analysis of the results obtained.

- Large uncertainties are present when trying to reproduce results utilizing current Isokinetic sampling methods.
Data and Methodology.

Two isokinetic sampling campaigns were conducted for particulate matter in accordance with ISO9096

- Campaign 1 (Source A): 12 one hour tests were conducted over two days on a fully compliant plant typical of a large industrial coal fired boiler power plant

- Campaign 2 (Source B): 3 one hour tests were conducted over an 8 hour shift on a typical cement kiln where the sampling location was not in compliance with the method requirements.
Data and Methodology contd.

- A critical element of a quality system and ensuring quality data is obtained is to ensure that the systems of calibration and measurement are traceable to national standards of measurement and that confidence can be placed in the quality of measurements carried out at all steps in the traceability chain (Clarke et al., 1998)

- ISO9096 is a validated, internationally recognized isokinetic sampling method
Most methods have been developed for steady state conditions and as such it is not unusual to have a 100 fold difference in emissions over time periods of 10 days to 10 months (Environment Agency Technical guidance note M2, 1993).

Therefore it is important to gain as much information about the process as possible.
A representative sample is obtained by withdrawing a sample isokinetically at several predetermined equal area points from the waste gas stream.

Sampling periods for each point should be of equal duration resulting in a composite sample.
Data and Methodology contd.

- Schematic of a typical Isokinetic Sampling train, courtesy EPA Method 17
Statistical approach -

The overall statistical approach utilised to estimate the method uncertainty - based on the Guide to the Expression of Uncertainty in Measurement” (generally known as GUM, 1995)

Important concepts of repeatability and reproducibility need to be understood

This project aimed to determine the reproducibility of the method under various operating times and conditions in the field.
Data and Methodology contd.

- Statistical approach –
- If testing was conducted at an unsuitable location, or was carried out under fluctuating plant operating conditions, the validity of the sample may be questioned and the measurement results uncertain (ISO9096:2003)
Data and Methodology contd.

- Statistical approach –

- An assessment of the stability and uniformity of the flow in the flue will determine the suitability/compliance of the sampling location.

- For this reason the velocity flow profiles for Source A and B have been included to assess the quality and validity of emission results obtained.
Data and Methodology contd.

- Statistical approach –

- Series 1, 2 and 3 in the figures are representative of the flow profiles for each sampling port utilized for each sample conducted.
Flow Profiles compliant Location.
Flow Profiles non-compliant Location.
## ISOKINETIC TEST RESULTS

**PLANT:** COMPLIANT SAMPLING POSITION  
**DATE:** 01-Aug-05

<table>
<thead>
<tr>
<th>DATA NO.</th>
<th>Dust (conc) mg Nm³</th>
<th>CO₂</th>
<th>O₂</th>
<th>Static Pressure</th>
<th>Moisture</th>
<th>Gas Temp</th>
<th>Gas Velocity</th>
<th>Gas Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>287.65</td>
<td>11.10</td>
<td>8.20</td>
<td>-1.10</td>
<td>2.32</td>
<td>125.45</td>
<td>12.99</td>
<td>0.75</td>
</tr>
<tr>
<td>2</td>
<td>292.89</td>
<td>11.10</td>
<td>8.20</td>
<td>-1.11</td>
<td>2.39</td>
<td>125.65</td>
<td>13.04</td>
<td>0.75</td>
</tr>
<tr>
<td>3</td>
<td>315.09</td>
<td>11.10</td>
<td>8.20</td>
<td>-1.05</td>
<td>2.77</td>
<td>127.66</td>
<td>13.13</td>
<td>0.74</td>
</tr>
<tr>
<td>4</td>
<td>318.30</td>
<td>11.50</td>
<td>8.10</td>
<td>-1.05</td>
<td>2.80</td>
<td>129.06</td>
<td>13.08</td>
<td>0.75</td>
</tr>
<tr>
<td>5</td>
<td>362.23</td>
<td>11.50</td>
<td>8.10</td>
<td>-0.98</td>
<td>2.42</td>
<td>124.69</td>
<td>13.28</td>
<td>0.75</td>
</tr>
<tr>
<td>6</td>
<td>428.34</td>
<td>11.50</td>
<td>8.10</td>
<td>-0.98</td>
<td>2.19</td>
<td>126.93</td>
<td>13.22</td>
<td>0.75</td>
</tr>
</tbody>
</table>

### PARAMETER

- **Sum:** 2030.71  
- **Average:** 335.95  
- **Median:** 317.10  
- **Variance:** 252.22  
- **Standard Deviation:** 55.15

**Confidence (95%)**  
- 15.09  
- % Uncert: 33.20

**% Uncert (95% CI)**  
- 3.44  
- **Combined Standard Uncertainty:** 55.15
- **Combined Expanded Uncertainty:** 130.21
- **Combined Standard Uncertainty % Relative:** 29.75
- **Combined Expanded Uncertainty % Relative:** 72.99

*where CI = 95%, K = 2.45, degrees of freedom = 6*

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**PLANT:** COMPLIANT SAMPLING POSITION  
**DATE:** 04-Aug-05

<table>
<thead>
<tr>
<th>DATA NO.</th>
<th>Dust (conc) mg Nm³</th>
<th>CO₂</th>
<th>O₂</th>
<th>Static Pressure</th>
<th>Moisture</th>
<th>Gas Temp</th>
<th>Gas Velocity</th>
<th>Gas Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>156.66</td>
<td>11.50</td>
<td>8.10</td>
<td>-1.20</td>
<td>4.06</td>
<td>123.51</td>
<td>13.19</td>
<td>0.75</td>
</tr>
<tr>
<td>2</td>
<td>168.66</td>
<td>11.50</td>
<td>8.10</td>
<td>-1.20</td>
<td>2.73</td>
<td>125.63</td>
<td>13.14</td>
<td>0.75</td>
</tr>
<tr>
<td>3</td>
<td>182.53</td>
<td>11.50</td>
<td>8.10</td>
<td>-1.20</td>
<td>4.11</td>
<td>125.45</td>
<td>13.11</td>
<td>0.75</td>
</tr>
<tr>
<td>4</td>
<td>218.84</td>
<td>11.40</td>
<td>8.20</td>
<td>-1.07</td>
<td>5.26</td>
<td>122.91</td>
<td>13.12</td>
<td>0.75</td>
</tr>
<tr>
<td>5</td>
<td>219.83</td>
<td>11.40</td>
<td>8.20</td>
<td>-1.07</td>
<td>3.98</td>
<td>123.09</td>
<td>13.17</td>
<td>0.75</td>
</tr>
<tr>
<td>6</td>
<td>254.29</td>
<td>11.40</td>
<td>8.20</td>
<td>-1.05</td>
<td>4.63</td>
<td>124.00</td>
<td>13.12</td>
<td>0.74</td>
</tr>
</tbody>
</table>

### PARAMETER

- **Sum:** 1250.74  
- **Average:** 208.46  
- **Median:** 205.58  
- **Variance:** 1007.68  
- **Standard Deviation:** 31.74

**Confidence (95%)**  
- 19.77  
- % Uncert: 37.31

**% Uncert (95% CI)**  
- 1.07  
- **Combined Standard Uncertainty:** 31.77
- **Combined Expanded Uncertainty:** 77.88
- **Combined Standard Uncertainty % Relative:** 25.52
- **Combined Expanded Uncertainty % Relative:** 63.22

*where CI = 95%, K = 2.45, degrees of freedom = 6*
# ISOKINETIC TEST RESULTS

**PLANT**  NON-COMPLIANT  SAMPLING POSITION  
**DATE**  10-Nov-05  

<table>
<thead>
<tr>
<th>DATA NO.</th>
<th>Dust [conc] mg/Nm³</th>
<th>CO₂</th>
<th>O₂</th>
<th>Static Pressure</th>
<th>Moisture</th>
<th>Gas Temp</th>
<th>Gas Velocity</th>
<th>Gas Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1694.52</td>
<td>23.00</td>
<td>11.00</td>
<td>-1.00</td>
<td>9.68</td>
<td>93.17</td>
<td>52.44</td>
<td>0.83</td>
</tr>
<tr>
<td>2</td>
<td>1813.39</td>
<td>22.00</td>
<td>11.00</td>
<td>-1.00</td>
<td>12.89</td>
<td>95.00</td>
<td>53.50</td>
<td>0.82</td>
</tr>
<tr>
<td>3</td>
<td>2051.77</td>
<td>22.00</td>
<td>12.00</td>
<td>-1.08</td>
<td>12.72</td>
<td>95.75</td>
<td>53.39</td>
<td>0.82</td>
</tr>
</tbody>
</table>

**PARAMETER**  

<table>
<thead>
<tr>
<th>PARAMETER</th>
<th>SUM</th>
<th>AVG</th>
<th>MEDIAN</th>
<th>VARIANCE</th>
<th>SD</th>
<th>SD % Relative</th>
<th>% Uncert (95% CI)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>sum</td>
<td>5559.68</td>
<td>1853.23</td>
<td>1813.39</td>
<td>22064.74</td>
<td>181.93</td>
<td>9.82</td>
<td>31.22</td>
</tr>
<tr>
<td>average</td>
<td>1853.23</td>
<td>22.33</td>
<td>22.00</td>
<td>0.22</td>
<td>0.58</td>
<td>2.59</td>
<td>5.09</td>
</tr>
<tr>
<td>median</td>
<td>1813.39</td>
<td>22.00</td>
<td>11.00</td>
<td>-1.00</td>
<td>0.58</td>
<td>0.05</td>
<td>0.01</td>
</tr>
<tr>
<td>variance</td>
<td>22064.74</td>
<td>0.22</td>
<td>0.2222</td>
<td>0.0014</td>
<td>1.81</td>
<td>5.74</td>
<td>15.35</td>
</tr>
<tr>
<td>SD</td>
<td>181.93</td>
<td>0.58</td>
<td>0.58</td>
<td>0.05</td>
<td>1.81</td>
<td>5.74</td>
<td>15.35</td>
</tr>
<tr>
<td>confidence (95%)*</td>
<td>578.53</td>
<td>1.84</td>
<td>1.84</td>
<td>0.15</td>
<td>4.22</td>
<td>14.31</td>
<td>48.83</td>
</tr>
<tr>
<td>SD % Relative</td>
<td>9.82</td>
<td>2.59</td>
<td>5.09</td>
<td>4.50</td>
<td>1.40</td>
<td>5.09</td>
<td>15.09</td>
</tr>
<tr>
<td>% Uncert (95% CI)*</td>
<td>31.22</td>
<td>8.22</td>
<td>16.20</td>
<td>14.31</td>
<td>4.46</td>
<td>16.20</td>
<td>48.83</td>
</tr>
</tbody>
</table>

**Combined Standard Uncertainty**  181.94  
**Combined Expanded Uncertainty**  578.5778  
**Combined Standard Uncertainty % Relative**  19.71  
**Combined Expanded Uncertainty % Relative**  62.69  

*where CI = 95%, K = 3.18, degrees of freedom = 3
If one compares the calculated overall uncertainties for the Tables, one would notice that the non-compliant data set returns a similar overall uncertainty (62.69% RSD @ 95% LOC) when compared to the compliant stack (62.52-72.98% RSD @ 95% LOC).
Combined / expanded uncertainty

The data were deemed to be of a normal distribution. Due to a lack of sufficient data, this is assumed to be the case and could not be statistically verified.
Combined / expanded uncertainty

- The overall uncertainty is also much higher than anticipated; this once again is mainly attributed to the small data sets utilized and the number of external/random variables that cannot be accounted for (i.e. fluctuating process operating conditions, changes in environmental conditions etc.)
Combined / expanded uncertainty

- An estimate of the overall uncertainty was attempted but the results were not conclusive as not enough data were obtained to enable any valid statistical inferences to be made.
Sampling Suitability Matrix

- Alternative tool developed to assess data quality
- Developed due to lack of data and high uncertainties
- Based on adherence to the requirements of the method
- Qualitative in nature but allows regulator and process operator to assess how reliable reported data is.
### Summary of requirements - Apparatus and sampling conditions

<table>
<thead>
<tr>
<th>SAMPLING LOCATION</th>
<th>Approx. Value</th>
<th>Measured Value</th>
<th>compliance y/n</th>
<th>Rating</th>
<th>Key</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow angle</td>
<td>&lt;15 °</td>
<td></td>
<td></td>
<td>10</td>
<td>P</td>
</tr>
<tr>
<td>Pressure difference (pitot tube)</td>
<td>&gt; 5 Pa</td>
<td></td>
<td></td>
<td>10</td>
<td>P</td>
</tr>
<tr>
<td>Ratio of max gas velocity to min gas velocity</td>
<td>3:1</td>
<td></td>
<td></td>
<td>10</td>
<td>P</td>
</tr>
<tr>
<td>Negative flow</td>
<td>None</td>
<td></td>
<td></td>
<td>10</td>
<td>P</td>
</tr>
<tr>
<td>Straight length before the sampling plane</td>
<td>&gt; 5 hydraulic diameters</td>
<td></td>
<td></td>
<td>9</td>
<td>P</td>
</tr>
<tr>
<td>Straight length after the sampling plane</td>
<td>&gt; 2 hydraulic diameters</td>
<td></td>
<td></td>
<td>9</td>
<td>P</td>
</tr>
<tr>
<td>Straight length before emission point</td>
<td>&gt; 5 hydraulic diameters</td>
<td></td>
<td></td>
<td>9</td>
<td>P</td>
</tr>
<tr>
<td>Number of sampling points</td>
<td>dependant on duct size</td>
<td></td>
<td></td>
<td>9</td>
<td>P</td>
</tr>
<tr>
<td><strong>EQUIPMENT FOR DUST COLLECTION</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Alignment of the nozzle</td>
<td>10%</td>
<td></td>
<td></td>
<td>8</td>
<td>FS</td>
</tr>
<tr>
<td>Isokinetic Criteria</td>
<td>+15% and - 5 %</td>
<td></td>
<td></td>
<td>8</td>
<td>FS</td>
</tr>
<tr>
<td>Leak test</td>
<td>&lt;2%</td>
<td></td>
<td></td>
<td>8</td>
<td>FS</td>
</tr>
<tr>
<td>Condenser, drying tower: residual gas moisture</td>
<td>&lt; 10 g/m³</td>
<td></td>
<td></td>
<td>7</td>
<td>FS</td>
</tr>
<tr>
<td>Gas meter volume measurement uncertainty</td>
<td>2%</td>
<td></td>
<td></td>
<td>7</td>
<td>FS</td>
</tr>
<tr>
<td>Absolute pressure measurement uncertainty</td>
<td>1%</td>
<td></td>
<td></td>
<td>7</td>
<td>FS</td>
</tr>
<tr>
<td>Absolute temperature measurement uncertainty</td>
<td>1%</td>
<td></td>
<td></td>
<td>7</td>
<td>FS</td>
</tr>
<tr>
<td>Filter efficiency (test aerosol 0.3μm)</td>
<td>&gt; 99.5 %</td>
<td></td>
<td></td>
<td>6</td>
<td>EQ</td>
</tr>
<tr>
<td>Filter material (adsorption of components)</td>
<td>No reaction or adsorption</td>
<td></td>
<td></td>
<td>6</td>
<td>EQ</td>
</tr>
<tr>
<td>Nozzle straight length before the first bend</td>
<td>&gt; 30 mm</td>
<td></td>
<td></td>
<td>5</td>
<td>EQ / P</td>
</tr>
<tr>
<td>Nozzle tip: distance to obstacles</td>
<td>&gt; 50 mm</td>
<td></td>
<td></td>
<td>5</td>
<td>EQ / P</td>
</tr>
<tr>
<td>Nozzle: Length with constant internal diameter</td>
<td>&gt; 10mm</td>
<td></td>
<td></td>
<td>4</td>
<td>EQ</td>
</tr>
<tr>
<td>Nozzle: variation in diameter angle</td>
<td>&lt; 30 °</td>
<td></td>
<td></td>
<td>4</td>
<td>EQ</td>
</tr>
<tr>
<td>Nozzle Internal diameter</td>
<td>&gt; 4mm</td>
<td></td>
<td></td>
<td>4</td>
<td>EQ</td>
</tr>
<tr>
<td>Nozzle area: measurement uncertainty</td>
<td>10%</td>
<td></td>
<td></td>
<td>4</td>
<td>EQ</td>
</tr>
<tr>
<td>Elbow: Radius of the bend</td>
<td>&gt; 1.5 d</td>
<td></td>
<td></td>
<td>4</td>
<td>EQ</td>
</tr>
<tr>
<td>Balance resolution (ng)</td>
<td>0.01mg to 0.1mg</td>
<td></td>
<td></td>
<td>3</td>
<td>L</td>
</tr>
<tr>
<td>weighing uncertainties</td>
<td>&lt; 5% of the LV for process</td>
<td></td>
<td></td>
<td>3</td>
<td>L</td>
</tr>
<tr>
<td>Thermal stability (filter)</td>
<td>&gt; 8h</td>
<td></td>
<td></td>
<td>3</td>
<td>L</td>
</tr>
<tr>
<td>Overall Blank Value</td>
<td>&lt; 10% LV or 2 mg/m³</td>
<td></td>
<td></td>
<td>3</td>
<td>L</td>
</tr>
<tr>
<td>Sampling time measurement uncertainty</td>
<td>5secs</td>
<td></td>
<td></td>
<td>2</td>
<td>FS</td>
</tr>
<tr>
<td>Linear measurement uncertainty</td>
<td>1% duct .2mm / 5% Nozzle</td>
<td></td>
<td></td>
<td>2</td>
<td>FS</td>
</tr>
<tr>
<td><strong>EQUIPMENT FOR FLUE GAS CHARACTERISTICS</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Absolute temperature</td>
<td>1%</td>
<td></td>
<td></td>
<td>1</td>
<td>C</td>
</tr>
<tr>
<td>Flue gas density</td>
<td>0.05 kg/m³</td>
<td></td>
<td></td>
<td>1</td>
<td>C</td>
</tr>
<tr>
<td><strong>Total possible Score</strong></td>
<td>188</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Validity of Results obtained**

- **Excellent (Fully compliant)**: 188 1.00
- **Fair (mostly compliant)**: > 150 <180 0.80
- **Poor**: <=60 0.32

**Key:**
- P: Plant Restrictions
- FS: Field Sampling Restrictions
- EQ: Equipment Restrictions
- L: Laboratory Restrictions
- LV: Limit Value
- C: Calculated / Measured in the Field
Discussion

- Tabulated data is divided broadly into three groups namely:
  - Plant restrictions
  - Field sampling restrictions
  - Equipment restrictions
  - Laboratory restrictions
Discussion

- Divided into groups as per the rating that corresponds with the control or lack thereof of the sampling technician to control these variables.
- The less direct control the higher the rating and therefore the more if not complied with the more potentially unreliable the reported results would be.
Summary and Conclusions

- Although subjective and semi-quantitative in nature the matrix will provide invaluable supplementary information in lieu of comprehensive uncertainty data.
- The matrix will allow the regulator and the plant operator to make more informed decisions about the reliability of the emission results reported.