Welcome!
Willkommen!
Bienvenue!
Benvenuto!
Recepción!
Καλώς ήρθατε!
Добро пожаловать!
欢迎！
歓迎！
स्वागत!
환영!

Condensation Particle Counter Calibration

Tim Johnson
Product Specialist
Particle Instruments

Global Aerosol Education

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Outline

• What is required in a Calibration
• Condensation Particle Counter – Basics
  – Instrument Schematics
• Specific issues for CPC calibration
• Description of Calibration Procedures
  – Basic Function checks
  – Instrument Flows
  – Optical Pulse alignment
  – Verification of temperatures
  – Concentration Checks
  – Special Tests
• Summary
Calibration

- **Calibration** \[\text{DEF}:\] The set of operations that establish, under specific conditions, the *relationship between values for quantities* indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

- Good calibration procedures are needed so that results are:
  - Repeatable
  - Reproducable
  - Quantitative
  - Traceable

- Why the interest in CPC Calibration?
  - Main reason in regulations
  - Engine Emissions (Euro 5 and later, PMP, Reg. 83).
  - Environmental Monitoring (not yet regulated but becoming more standardized)

---

CPC Calibration Method

‘Primary Absolute Calibration’ Method
Developed by B. Liu and D. Pui in 1974

CPC Calibration Papers

Butanol CPC Key components

- **Three Basic Components**
  - **Saturator**
    - Heated to produce vapor from Butanol
  - **Condenser**
    - Cooled to cool particles
    - Vapor condenses onto particles and they grow
  - **Optics**
    - Count particles

![Diagram of CPC system with labels for Saturator, Condenser, and Optics. The diagram shows a flow from a pump at 40 °C through a saturator at 10-22 °C, a condenser at 39 °C, and an optics section at ~10 µm. The flow is indicated by arrows.](image)
Butanol CPC

Key components

Three Basic Components

- Optics
- Condenser
- Saturator
Butanol CPC Activation Theory

$$D_{\text{kelvin}} = \frac{4\delta_S M}{\rho_L RT \log S}$$

1) Saturate
2) Condense
3) Detect

$$S \equiv \frac{P_v}{P_{\text{saturation}}(T)}$$

D = Kelvin Diameter
$$\delta_S$$ = Surface Tension of Working Fluid
M = Molecular Weight of Working Fluid
$$\rho_L$$ = Density of Working Fluid
R = Gas Constant
T = Temperature
S = Supersaturation Ratio
$$P_v$$ = Vapor Pressure
$$P_{\text{saturation}}(T)$$ = Saturation Vapor Pressure
CPC Comparison - Alcohol and Water

Traditional (Alcohol) vs. WCPC

- Condenser, 10°C
- Saturator, 35°C
- Growth Tube, 60°C

Traditional

\[ S = \frac{P_v}{P_{\text{sat}}} \]

WCPC

\[ S = \frac{P_v}{P_{\text{sat}}} \]

Centerline Axial Profiles

- Saturation (%)
- Temperature -->
- Kelvin Diameter -->

Axial Distance/Tube Radius (z/R)

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CPC Characteristics

- **Single Particle Counting device**
  - Can measure down to very low concentrations
  - CPCs are linear devices in single particle counting region
    - At high concentrations multiple particles can be in the single particle counting region simultaneously, resulting in lower detected concentration

- **Detection Efficiency varies with size**
  - Minimum Detection level often specified as D50 size
    - D50 = Diameter at which 50% of particles are detected (50% detection efficiency)
    - Other Dxx values are used to specify other Efficiency levels
  - Shape of efficiency curve depends on design of CPC
    - Flow path and flow rate, temperatures, working fluid, etc.
  - Detection Efficiency can depend on chemistry of particles being detected
    - Small effect with Butanol
    - Influences choice of calibration particles

- **Fast Response Time**
  - Testing done during product development
    - Not checked during Calibration
CPC Efficiency Curves

Counting Efficiency, %

Particle Size, nm

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TSI’s current Condensation Particle Counters
Models 3772 & 3790

3772 Specs

D$_{50}$: 10 nm
Aerosol Flow: 1 Lpm
Max. Conc.: $10^4$ pt/cm$^3$
Response Time: 3 sec
Saturator Temp. 39°C
Condensor Temp. 22°C

3790 Specs

D$_{50}$: 23 nm
Aerosol Flow: 1 Lpm
Max. Conc.: $10^4$ pt/cm$^3$
Response Time: 3 sec
Saturator Temp. 38°C
Condensor Temp. 32°C

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3775 ‘High Concentration’ CPC

3775 Specs

- $D_{50}: \quad 4 \text{ nm}$
- Max. Conc.: $10^7 \text{ pt/cm}^3$
  - Using Photometric mode
- 95% Response Time
  - High Flow: 4 sec
  - Low Flow: 5 sec
3776 Ultrafine Butanol CPC

3776 Specs

D$_{50}$: 2.5 nm
Max Conc: 3x10$^5$ pt/cm$^3$
Aerosol Flow: 0.05 Lpm

95% Response Time
High Flow: <0.8 sec
Low Flow: 5 sec
## CPC Specs Summary

<table>
<thead>
<tr>
<th>Specifications</th>
<th>3007</th>
<th>3781</th>
<th>3783</th>
<th>3772</th>
<th>3790</th>
<th>3787</th>
<th>3775</th>
<th>3776</th>
<th>3788</th>
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<tr>
<td>D_{50} Min. Size (nm)</td>
<td>10</td>
<td>6</td>
<td>7</td>
<td>10</td>
<td>23</td>
<td>5</td>
<td>4</td>
<td>2.5</td>
<td>2.5</td>
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<tr>
<td>Max. Concentration (particles/cm³)</td>
<td>100,000</td>
<td>400,000</td>
<td>1,000,000</td>
<td>10,000</td>
<td>10,000</td>
<td>250,000</td>
<td>50,000 &lt;10^7*</td>
<td>300,000</td>
<td>400,000</td>
</tr>
<tr>
<td>Concentration Accuracy (%)</td>
<td>±20</td>
<td>±10</td>
<td>±10</td>
<td>±10</td>
<td>±10</td>
<td>±10</td>
<td>±10</td>
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<tr>
<td>Response - T95 (s)</td>
<td>&lt;9</td>
<td>&lt;2</td>
<td>&lt;3</td>
<td>~3</td>
<td>~3</td>
<td>~0.7</td>
<td>~4</td>
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<td>~0.25</td>
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<tr>
<td>Sample Flow (LPM)</td>
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<td>0.12</td>
<td>1.0</td>
<td>1.0</td>
<td>0.6</td>
<td>0.3</td>
<td>0.3</td>
<td>0.3</td>
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<tr>
<td>Total Inlet Flow</td>
<td>0.7</td>
<td>0.6</td>
<td>0.6</td>
<td>1.0</td>
<td>1.0</td>
<td>0.6/1.5</td>
<td>0.3/1.5</td>
<td>0.3/1.5</td>
<td>0.6/1.5</td>
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<tr>
<td>Flow Source</td>
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<td>External</td>
<td>External</td>
<td>External</td>
<td>Internal</td>
<td>Internal</td>
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</tr>
<tr>
<td>Working Fluid</td>
<td>Isopropyl</td>
<td>Water</td>
<td>Water</td>
<td>Butanol</td>
<td>Butanol</td>
<td>Water</td>
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<td>Butanol</td>
<td>Water</td>
</tr>
<tr>
<td>Display</td>
<td>Digital LCD</td>
<td>Digital LCD</td>
<td>Touch w/graph</td>
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<td>Digital LCD</td>
<td>Touch w/graph</td>
<td>LCD w/graph</td>
<td>LCD w/graph</td>
<td>Touch w/graph</td>
</tr>
<tr>
<td>Data Logging/Storage</td>
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<td>On-board</td>
<td>Flash drive</td>
<td>N/A</td>
<td>N/A</td>
<td>Flash drive</td>
<td>Memory Card</td>
<td>Memory Card</td>
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<tr>
<td>SMPS Compatibility</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
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</table>

* Photometric Mode
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<td>&lt;10⁷⁺</td>
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</table>

* Photometric Mode
Issues for CPC calibration

- Calibration Standard
  - Primary Calibration – Aerosol Electrometer (AE)
  - Secondary Calibration – Reference CPC
- Need Special Aerosols for calibration
  - Monodisperse Aerosol needed for Counting Efficiency measurements
  - Single charged particles needed for AE
  - High concentrations needed for concentration linearity tests
- Some tests only done if required for regulations
  - Counting Efficiency Tests
    - Required for PMP (Engine Emission) standard
      - D50 Efficiency = 50% ± 12% at 23 nm and D90 Efficiency > 90% at 41 nm
    - Other CPCs - Efficiency curve determined during product development
      - Variation between units is very small.
Traceability: Aerosol Electrometer (AE)

Primary Concentration Reference

Resistor (R)
- 1% precision, measured using NIST traceable standard

Particle charge \((n_p)\)
- Verified to be unity (1.0) by SMPS

Flow rate \((q_e)\)
- NIST traceable flow meter

\[
N = \frac{V}{e \cdot R \cdot n_p \cdot q_e}
\]

- \(e\) = elementary unit charge 
  \(1.602 \times 10^{-19} \text{ C}\)
- \(n_p\) = average number of charges per particle
- \(q_e\) = volumetric aerosol flow rate
Reference Aerosol Electrometer versus Reference CPC

- **Primary Calibration** – Aerosol Electrometer (AE)
  - Calibration against a Reference Electrometer is used to establish traceability to SI units.
  - **Reading** must be corrected if particles carry multiple charges.
  - **Calibration result** for Efficiency curve must be corrected if particles have more than one mode.
  - Needs high enough concentration for good signal.

- **Secondary Calibration** - Condensation Particle Counter (CPC)
  - Reference CPC calibrated against a Reference Electrometer is a valid, traceable transfer standard.
  - **Calibration result** for Efficiency curve must be corrected if particles have more than one mode.
Electrostatic Classifier: Traceable Particle Size

In a cylindrical DMA, $Z_p$ of selected particles is

$$Z_p = \frac{v}{E} = \frac{n_p eC}{3\pi \mu D_p}$$

Inversely proportional to $D_p$

$$Z_p = \frac{[q_t - 1/2(q_p + q_m)] \ln( r_2 / r_1 )}{2\pi VL}$$

Flow rates ($q_t$, $q_p$, $q_m$)
- NIST traceable flow meters

Geometric parameters ($r_1$, $r_2$, $L$)
- NIST traceable bore gage, micrometer and caliper

Voltage on center electrode (V)
- Calibrated with NIST traceable kilovolt divider

Output is Single Electrical Mobility $\approx$ Single Particle Charge

New ISO standard (ISO 15900-2009) describes Classifiers
CPC used in PMP Standard

- Europe has a particle number emission standards for certain vehicle types
- PMP Standard has special requirements for CPC
- CPC requirements
  - Butanol working fluid
  - Single Flow (no flow splits)
  - Counting Accuracy of ±10% over full concentration range
  - No photometric mode but Dead-time correction allowed
  - Requirements for Efficiency curve check and Concentration Linearity
- Calibration must be done with Aerosol Electrometer
  - Counting Efficiency specified for D50 and D90
  - Linearity checked at 5 equally spaced concentrations between zero and 10,000 particles/cc
    - Determine Slope and $R^2$ of CPC under test versus a Reference Detector (Electrometer or CPC)
    - Ratio must be between 0.9 and 1.1 at all concentrations
Atomized Calibration Aerosol

NaCl Particles

Width ($\sigma_g = 1.84$) Crystalline shape

Calibration Particles Percentage
- 50 nm – single charge 74.39%
- 72 nm – double charge 19.05%
- 91 nm – triple charge 6.56%

Problems for Calibration
- Multiply charged particles
  - Worse for smaller sizes
CAST Soot Calibration Aerosol

Oxidation air
Compressed air
N₂
C₃H₈

CAST Generator

Concentration Control

Electrostatic Classifier & Nano-DMA

Raw Emery CAST Particles
Example: 61nm (sₙ = 1.59)

Calibration Particles | Percentage
--- | ---
22 nm – single charge | 90.868%
32 nm – double charge | 8.663%
40 nm – triple charge | 0.47%

Classified Soot is Multimodal
Non-spherical (agglomerates) Multiply charged

> Corrections must be applied
Emery Oil Calibration Aerosol

Filtered air Supply

Electrospray Aerosol Generator

Concentration Control

Electrostatic Classifier & Nano-DMA

Emery Oil Particles for D50 Calibration

<table>
<thead>
<tr>
<th>Calibration Particles</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>23 nm – single charge</td>
<td>99.919%</td>
</tr>
<tr>
<td>32 nm – double charge</td>
<td>0.081%</td>
</tr>
<tr>
<td>40 nm – triple charge</td>
<td>0.000001%</td>
</tr>
</tbody>
</table>

Monodisperse ($\sigma_g = 1.04$)

Spherical Singly Charged

Ideal for Calibration

TRUST. SCIENCE. INNOVATION.
PMP Counting Efficiency Requirements

Original PMP requirement

A lot of sizes needed to be generated
Long test - Expensive

<table>
<thead>
<tr>
<th>% Particle Efficiency</th>
<th>Lower Size (nm)</th>
<th>Upper Size (nm)</th>
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<tbody>
<tr>
<td>D10</td>
<td>15</td>
<td>17</td>
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<tr>
<td>D25</td>
<td>16</td>
<td>20</td>
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<tr>
<td>D50</td>
<td>20</td>
<td>26</td>
</tr>
<tr>
<td>D90</td>
<td>33</td>
<td>41</td>
</tr>
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</table>

Current PMP requirement

<table>
<thead>
<tr>
<th>% Particle Efficiency</th>
<th>Particle Size (nm)</th>
<th>Nominal Efficiency</th>
<th>Lower Efficiency</th>
<th>Upper Efficiency</th>
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</thead>
<tbody>
<tr>
<td>D50</td>
<td>23 ± 1</td>
<td>50% ± 12%</td>
<td>38%</td>
<td>62%</td>
</tr>
<tr>
<td>D90</td>
<td>41 ± 1</td>
<td>&gt;90 %</td>
<td>90%</td>
<td>100%</td>
</tr>
</tbody>
</table>
Electrospray Aerosols Classified

• Solution concentrations need to be adjusted for each particle size needed
  – Aerosols needed for Efficiency Curve calibration are 23 nm and 41 nm
    • Counting Efficiency at 23 nm is 50% ± 12%
    • Counting Efficiency at 41 nm is >90%
  – Aerosol needed for Concentration Linearity
    • Must have very high percentage of singularly charged particles
    • Need concentration ≥ 10,000 (to all instruments) to cover full concentration range
What is included in calibration?

• Description of Calibration Procedures
  – Basic Function checks
  – Instrument Flows
  – Optical Pulse alignment
  – Verification of temperatures
  – Concentration Checks
    • Zero count
    • Basic Linearity
  – Special Tests
    • Counting Efficiency Test
      – For PMP Standard Checks at D50 and D90 points
    • Concentration Linearity testing (for PMP – special requirements)
    • Photometric Calibration
What is included in calibration?
- Basic Function Checks
  - Circuit board Voltages
  - Analog inputs and outputs, pulsed output
  - Communications
    - Serial connection
    - USB connection
    - Ethernet
    - Flash Memory card
  - Liquid Level sensor setup
  - Water Removal System
What is included in calibration?

- Instrument Flows

• Concentration is linearly related to Flow
• Some CPCs have multiple flows
  – Selectable Inlet Flows
  – Capillary Flows (Ultrafine CPCs)
  • Requires Calibration
What is included in calibration?
- Optical Alignment

- Particles all grow to uniform size
- Pulses need to be uniform (within a range) in both height and width
What is included in calibration?

- Temperatures

- Saturator and Condenser Temperatures determine Counting Efficiency

- Optics need to be heated to prevent condensation on optics
Concentration Checks

• Reference Instrument
  – Reference CPC
  – Aerosol Electrometer - Primary Calibration Standard
    • Requires Known Charge Level on Particles
    • Not single Particle Counting – Requires hundreds or charges

• Linearity Checks over concentration range

• Zero Count Test – 12 hour test
  – Average value, Particle Burst Events

• Dead Time Correction verification
  – What is Dead-time?
Particle Concentration Calculation

Correcting for Coincidence - Dead Time

Concentration = \( \frac{\# \text{ Particles}}{(\text{Flow rate} \times \text{“Sample time”})} \)

“Sample Time” ≡ \( t_{\text{live}} = t_{\text{interval}} - (t_{\text{dead}} \times \tau) \)

\( \tau \) = Dead Time Correction Factor
Dead-Time Correction

\[ DTC = \tau = \frac{T_m}{T_d} \]

Coincident Gaussian Pulses

<table>
<thead>
<tr>
<th>Signal</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>( v_s )</td>
<td>( T_m )</td>
</tr>
<tr>
<td>( v_s )</td>
<td>( T_d )</td>
</tr>
</tbody>
</table>

\( T_d = \text{Discriminator Time} \)
\( T_m = \text{Minimum Detectable Time} \) (between particles)
\( \tau = \text{Dead-time Correction factor} \)
Dilution Bench

• Reference: Single flow, single particle counting CPC
• Adjusted diluter - Exact diluter ratio determined
• Can measure up to 10 million particles/cc
1000:1 Diluter

- Use to compare concentrations
  - Reference CPC is 3772 single particle counting CPC
  - Test 3775 and 3776

- 3775 has photometric mode
  - $10^7$ maximum concentration
- 3776 has 300,000 maximum concentration
Checking Concentration Linearity

Adjust 1000:1 Diluter - Sample to determine “Mean Dilution Ratio” (~ 1000:1)

⇒

Adjust variable diluter to adjust concentrations

<table>
<thead>
<tr>
<th>Table Diluter Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Minimum</td>
</tr>
<tr>
<td>16000</td>
</tr>
<tr>
<td>40000</td>
</tr>
<tr>
<td>96000</td>
</tr>
<tr>
<td>160000</td>
</tr>
<tr>
<td>240000</td>
</tr>
</tbody>
</table>

![Graph showing concentration vs. reference counter](image)
### Linearity Test

**Example model 3776**

<table>
<thead>
<tr>
<th>I.U.T Conc</th>
<th>3772 Conc</th>
<th>Ref Conc</th>
<th>%diff</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.695e+004</td>
<td>1.707e+001</td>
<td>1.690e+004</td>
<td>0.306</td>
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<tr>
<td>3.032e+004</td>
<td>3.036e+001</td>
<td>3.005e+004</td>
<td>0.872</td>
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<tr>
<td>3.848e+004</td>
<td>3.789e+001</td>
<td>3.751e+004</td>
<td>2.572</td>
</tr>
<tr>
<td>5.128e+004</td>
<td>5.040e+001</td>
<td>4.990e+004</td>
<td>2.752</td>
</tr>
<tr>
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</table>

**Dilution Ratio:** 0.9999

**Slope:** 1.0029
CPC Zero Count Test

Attach a large HEPA filter to inlet
Set Unit to HIGH FLOW RATE.
Perform “Zero Count Test”

Test Criteria

• Overall Concentration
• Burst Count (# over 20 in 10 minutes)
• Maximum Burst Concentration
Photometric Calibration

 Photometric Mode Calibration Table - CpcCal2.cxx

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<tr>
<th>Conc</th>
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</table>

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Example for a Fully SI-Traceable CPC Calibration Setup

AIST Calibration System

Presented by Hiromu Sakurai @ ISO/TC 24/SC 4/WG 12 meeting in U.K. in September 2008
CPC Calibration - Summary

• Basic Calibration is important for all applications
  – Regulations add new requirements including traceability
• Different CPC designs require different tests
• Reference Standards
  – Primary or Secondary Calibration Standards can be used
  – They both have advantages and disadvantages
• Challenge Aerosols affect calibration results
  – Ideal particles are single-mode, monodisperse, and spherical
  – High concentrations and single charge per particle are sometimes needed
• All CPCs calibrations require checks or adjustment of: Flow, Optical alignment, Temperature, Concentration and Zero
• Addition Requirements can include: Efficiency curves (or portions of the curve), specific concentration linearity requirements
• Working on a CPC calibration standard is underway
  – ISO/TC 24/SC 4/WG 12
Webinars

February 10th  
Particle Surface Area Concentrations and Number Size Distributions in View of Exposure Assessment  
by Dr. Christof Asbach (IUTA)

February 24th  
On-line Nanoparticle Characterization Studies of Fe-Ni Catalysts for Carbon Nanotube Growth  
by Dr. Mohan Sankaran (Case Western Reserve)

March 10th  
Fluorescent Biological Aerosol Particle Concentrations and Size Measured with UV-APS  
by Dr. Alex Huffman (Max Planck Institute)

Optical Particle Sizer Model 3330

- Size resolution <5% at 0.5μm  
- User adjustable size channels  
- Size range: 0.3 – 10μm in up to 16 channels  
- Wide concentration range from 0 to 3000 particles/cm³  
- Fully compliant with ISO 21501-01/04

New WCPC's Models 3787 & 3788

- 2.5nm detection  
- Single particle counting to 4x10⁵ particles/cm³  
- <100 ms rise-time response w/ 42 ms time constant (fastest CPC available)  
- Convenient, eco-friendly water as working fluid
Thank You For Your Attention

Any Questions?

Tim Johnson (tim.johnson@tsi.com)