The Sensitivity and Repeatability of Nanoparticle Measurements at High Dilution Ratios
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Abstract:

This contribution addresses the sensitivity and repeatability of nanoparticle measurements from diesel exhaust. The sampling method is critical in reproducing, as far as is possible, real tailpipe conditions. Currently presented results show marked variation due primarily to sampling method rather than instrumentation. In this case samples are taken from raw exhaust via a system capable of high dilution ratios and rates of dilution similar to that seen from the tailpipe.

The purpose of investigating sensitivity and repeatability is to provide a sound basis for evaluation of changes in particle emissions from base engines, the effect of fuels and additives, and the effect of all forms of aftertreatment.

By fixing certain parameters in the sampling system workable levels of both sensitivity and repeatability were observed over a wide range of engine operating conditions. The results have been translated into a daily system check for the Perkins test bed facility.

Introduction:

The importance of the effect of nanoparticle emissions on air quality and human health is now recognised world wide. Nanoparticle measurement presents a very real technical challenge. Large ambient number concentrations, particularly below 30 nm, have been observed by many researchers. The significant contribution from diesel exhaust is also well documented (1,2). The formation of nanoparticles (as observed close to the tailpipe) is a function of exhaust gas composition (solid, liquid and gas phase), temperature (exhaust and ambient), humidity and dilution rate. The influence of all these parameters has yet to be fully investigated, with the ultimate goal of establishing a reliable and robust engine/vehicle sampling and measurement method that simulates the real atmospheric process.

Perkins has been working in conjunction with The University of Minnesota to help establish a sampling and test protocol. It is recognised that the standard constant volume sampling method for regulated emissions does not necessarily replicate the atmospheric processes and much work still needs to be done (3). However, the process of reducing diesel particle emissions, particularly in the nano range, still needs to be investigated. Perkins has, through the work of The University of Minnesota, installed a sampling and dilution system capable of dilution ratio and temperature control (Figure 6). This system, over a considerable period of operation, has demonstrated a high degree of reproducibility, coupled with good resolution (the system is also described in Ref.3.). These features have now been developed into a test methodology which ensures reliable interpretation of major trends and changes in engine and aftertreatment performance.

The results as presented in Zurich are shown in figures 1-19. It must be stressed that the use of the absolute values is not recommended due to the unique combination of engine and sampling system.

Discussion:

The attached results are encouraging, but still require a high degree of skill and awareness in setting the various parameters. Of particular note are the following:

1. Primary dilution ratio and rate (plus residence time at the primary dilution ratio) have a major influence on the perceived distribution. The engine is known to be both relatively clean and also to behave as a very good polydisperse combustion aerosol generator. The secondary dilution ratio (to give overall dilutions of over 500:1) has a less marked effect.

2. Both the sample temperature and associated residence time in the sample line have a major effect on the particle distribution, but only below 30 nm, the largely carbon based larger particles being sensibly stable. The condition of the dilution air (temperature and humidity) must also be considered in conjunction with the sample condition. The length of sample line seems to be less influential when compared to sample line temperature gradient. The quantity of sample required is small and therefore rapid cooling in small bore pipework is to be expected.

3. The point of sampling for the raw exhaust is less critical (i.e. from just after T/C to some 3m downstream). The temperature drop and residence time in the main exhaust are both relatively minimal.

Conclusions:

Because of the lack of a recognised test method international comparison of results is not reliable, and consequently real performance improvements can only be approximated, particularly in the case of post tailpipe nanoparticle production. Continuing evaluation and investigation of the
THE SENSITIVITY and REPEatability of NANOparticle MEASUREMENTS at HIGH DILUTION RATIOS

By: CHRIS BARNES

Performance, Emissions and Combustion

PERKINS PRODUCT ENGINEERING

3rd NANOparticle WORKSHOP, ETH, ZURICH, 9/10th Aug 99
TEST FORMAT

• PERKINS STRATEGY:
  – FIX THE FOLLOWING:
    • HUMIDITY (via dilution air---approx. 15%).
    • DILUTION SYSTEM (Based on Minnesota's development).
    • ENGINE--T4.40 PHASER (a consistent “slave”).
    • SAMPLE POINT--ALLOWS FOR AFTERTREATMENT.
    • SAMPLE LINE TEMP.--300 deg C. (Average Exh. Gas temp.)
    • DILUTION RATIO---1000:1 (Based on Minnesota Work to represent atmospheric dilution from a tailpipe).
    • THE COMBINATION OF FIXED SAMPLE LINE TEMP. AND FIXED DILUTION EFFECTIVELY FIXES RESIDENCE TIMES.

ENGINE TEST BED VARIABLES

• ENGINE:
  – SPEED, LOAD, FUEL (SULPHUR), LUBE OIL, GAS TEMP.

• SAMPLE:
  – SAMPLE POINT (JUST AFTER T/C THROUGH TO AFTER AFTERTREATMENT).
  – LENGTH OF SAMPLE LINE (RESIDENCE TIME).
  – SAMPLE CONTROL TEMP.
  – DILUTION RATIO (Primary and Secondary).
  – RESIDENCE TIME DURING DILUTION.
  – HUMIDITY OF DILUTION AIR.
PRESENTATION of RESULTS

- PERKINS SAMPLING SYSTEM.

- MINNESOTA SAMPLING SYSTEM.

- ILLUSTRATION of Perkins v. Minnesota Agreement.

- SPREAD of RESULTS @ 1600 rpm/10% load and 2600 rpm/100% load.

- ILLUSTRATION of LOAD DISCRIMINATION @ 1600 rpm.

PERKINS TEST PROCEDURE

- START of EACH DAY:
  - RUN 1600 rpm/10% LOAD and 2600 rpm/100% LOAD and COMPARE AGAINST "STANDARD CURVES".
  - THIS BUILDS UP A BANK OF CONSISTENCY RESULTS AND CONFIRMS OVERALL SYSTEM FUNCTION.

- BENEFITS:
  - ALLOWS FUEL EFFECTS TO BE OBSERVED.
  - ALLOWS AFTERTREATMENT TO BE EVALUATED.
  - ALLOWS ENGINE CHANGES TO BE EVALUATED. (and engine types, once the standard curves have been generated).
SCHEMATIC OF DILUTION SYSTEM

Exhaust Gas Emission Probe
Pressure Gauge
Particle Sampling Probe

Temperature Controller
Thermo-Couple
Coarse Needle Valve
Vacuum

Insulated and Heated Sampling Line (100-300 deg C) as required.
Critical Flow Orifice just before Primary Ejector/Diluter
Primary Flow Mixer and Expander

Pressure Gauge
HEPA Filter
Pressure Regulator
Activated Carbon for ambient NOx reduction
Scanning Mobility Particle Sizer (SMPS)
Condensation Particle Counter (CPC)

Air Supply Line
Primary Excess Flow
NOx Measurement and Excess Flow

Engine

Product Engineering
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07/9205 G1/5201000015/EN/4.01 ENGINEERING

Perkins
REPEATABILITY EXERCISE -- 1600 RPM, 1000:1 DILUTION RATIO.
18 data sets over 2 months - 10% LOAD, 300 degC Sample line

C.Barnes 2 Aug 99
Bed 65 - SMPS Results - REPEATABILITY

Comparison between Perkins Two Stage Dilution at 1600 rpm, 50% Load at 1000:1 Dil., 300 degC sample temp., and a recent result from Minnesota on the same engine type, and with their sampling and two stage dilution system (Dilution ratio unknown).
ENGINE MAP- LOAD EFFECT @1600rpm, 100%-10% load at 1000:1 Dil., 300 degC sample temp

CONCENTRATIONS CONVERTED TO RAW EXHAUST

100%
75%
50%
25%
10%

2 Results for EACH LOAD POINT

Particle Size nm

C. Barnes 03 Aug 99
Bed 65 SMPS Results-MAP

REPEATABILITY EXERCISE-- 2600 RPM, 1000:1 DILUTION RATIO.
18 Data sets over 2 months--100% LOAD, 300 degC Sample line

CONCENTRATIONS CORRECTED TO RAW EXHAUST

Particle Size nm

C. Barnes 2 Aug 99
Bed 65 SMPS Results-REPEATABILITY
PRESENTATION of RESULTS

- MEAN and Std DEV. @ 1600/10% and 2600/100% points.

- ILLUSTRATION of HEATED SAMPLE LINE TEMP. EFFECTS @ 1600 rpm/10% load--
  Maximum Suppression of losses due to Warming Effect on sample from 94 deg C
  to 119 deg C. with line at 300 deg. C ?
REPEATABILITY-Mean of 18 sets at 2600 rpm, 100% Load, 1000:1 Dil., 300 degC over 3 months-Std 450 ppm sulphur fuel

ALL CONCENTRATIONS CORRECTED to RAW EXHAUST

VALUES at 10.37 and 11.14 nm are estimates—mean minus std dev. is negative due to spread of results in this region.

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Bed 65 SMPS Results-REPEATABILITY
P:/kgall/combust/wip/P3811/RPT26b.xls

REPEATABILITY EXERCISE—Mean of 24 data sets at 1600 rpm, 10% load, 1000:1 Dil., 300 degC over 3 Months-Std 450 ppm Sulphur Fuel

ALL CONCENTRATIONS CORRECTED TO RAW EXHAUST

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Bed 65 Results-SMPS-REPEATABILITY-
P:/kgall/combust/WIP/P3811/RPT16a.xls
PRESENTATION of RESULTS

- HEATED LINE EFFECTS @ 2600 rpm/100% Load---Slightly increased losses with Reducing Temp. from a sample at 390 deg. C?

- ILLUSTRATION of Heated line EXTENSION effects-nano-particle growth suppression at 1600/10%, and possible growth @ 2600/100%?
**Repeatability**

Mean of 18 sets at 2600/1600 rpm, 100%/10% Load respectively:
1000:1 Dil., 300 degC PLUS results WITH and WITHOUT NEW HEATED BOX EXTENSION (250599)

**Concentrations Corrected to Raw**

**Effect of Sample Line Temp. 2600 RPM. 500:1 Dilution Ratio. 100% Load**

**Concentrations Corrected to Raw Exhaust**

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Bed 65 SMPS Results

P/kgall/combust/wip/ P3811/heatex1a.xls

T4.40 AEU64420BY-4 Cyl on Highway 81 kW @ 2600 rpm

Perkins Product Engineering Job P3811