# T10 Integrated Infrared Oxidation Measurement Procedure
## Draft 4 9/06/2005

**Scope:**

This test method is designed to measure oxidation in used diesel engine oils.  The test was designed around the Mack T10 engine test, but may be applicable to used diesel engine oil samples from other sources.

This method does not purport to address all of the safety concerns associated with its use.  It is the responsibility of its user to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

**References:**

E168-XX

**Summary of test method:**

Fresh (new) and used diesel engine oil samples are scanned in a Fourier Transform Infrared spectrometer (FTIR) instrument using a 0.05mm path length $BaF_2$ transmission Cell.  The spectral data is integrated over the region of carbonyl absorbance to obtain a result for the increase in concentration in this region.  The results are calculated in absorbance * wavenumbers (cm-1) units relative to the path length of the cell standard of 0.05mm (Abs. * $cm^{-1}$ / 0.05mm).  The final result is corrected to 1 cm of path length (Abs./$cm^2$).

**Apparatus:**

A.  FTIR capable of the following:
    1.  Mid IR Capable.
    2.  DTGS Detector **or equivalent**.
    3.  Resolution minimum 4 wavenumbers and 16 scans per sample.
B.  Transmission Cell:
    1.  Path length nominally 0.05mm.
    2.  Material – Barium Fluoride ($BaF_2$)
C.  Instrument Control and/or Data Processing software:
    1.  Performs and Calculates results for specific integrals with baseline correction under the integral region and baseline correction outside the integral region.
D.  Analytical Balance capable of measuring to +/- 0.001grams.
E.  Glassware for dilutions:
    1.  10 ml graduated cylinder with stopper.
    2.  Pipette (glass or plastic).
    3.  25 ml volumetric pipette (May use other size as available, but weights listed in (Cal.A.1) must be adjusted proportionately.).

**Materials:**

A.  Carbon tetrachloride ($CCl_4$) – reagent grade or better.
B.  Base oil – **suggest a 150 Neutral base stock**.
C.  Other solvents or chemicals required by FTIR equipment manufacturer for path length calibration and cell cleaning.

**Calibration:**

A.  Linearity of Detector must be determined in the following manner:
    1.  Blend Base oil into $CCl_4$ in approximately the following wt./vol. Ratios using the analytical balance and 25 ml volumetric pipette:  (wt. Base oil/vol. $CCl_4$) 0.0g oil/25 ml CCl4 (0.0%), 0.25 g /25 ml (1%), 0.50 g/25 ml (2%), 0.75 g/25 ml (3%), 1.00 g/25 ml (4%), 1.50 g/25 ml (6%), 2.00 g/25 ml (8%), 2.5 g/25 ml (10%) and higher concentrations as necessary to establish linearity to the specification listed in (Cal.A.7).  Record actual weights obtained to the nearest 0.001 grams.

2. Obtain spectra using the 0.05 mm BaF$_2$ cell for all Base oil/ CCl$_4$ blends prepared in (Cal.A.1).
3. Calculate and record the net absorbance for each blend at ~2926 cm-1 using a baseline correction from 3100 – 2700 cm-1.
4. Plot absorbance vs. Concentration values.
5. Calculate the best-fit linear equation for all points (minimum 4 points) obtained at or below 1 absorbance.
6. Calculate the deviation from the above linear equation of the remaining absorbance values.
7. The point at which the absorbance vs. Concentration values above 1 absorbance unit deviate from the line by more than 6% (delta/absorbance predicted) is the "limit of linearity" (LOL) for the detector.

B. Cell path length measurement:
1. Cell path is to be calculated using the "fringe" technique. A spectral scan of and empty cell should be stored and analyzed by counting the number of complete sin wave forms in a know wavenumber region. The path length is calculated by the equation below. Spectral Scans should be performed in triplicate and measurements should not deviate by more than +/- 1%.

$$P (mm) = (n \div (2 \times W)) \times 10$$

Where:
P (mm) = path length in mm.
n = number of complete sin waves counted
W = total wavenumbers in the region counted for sin waves.

2. Obtain an actual value of path length to 0.001 mm of the cell to be used for analysis. This path length will be used to correct results to the nominal 0.05 mm path length.
3. Path length measurements are to be performed each time a demountable cell is taken apart for cleaning and verified daily prior to each set of samples to be analyzed.

**Procedure:**
A. Obtain 3 spectra of the Fresh oil and each available used oil according to E168.
B. Verify that the raw absorbance of the peak near 1725 cm-1 does not exceed the LOL obtained in (Cal.A.7) for each spectra obtained.
1. If the limit is NOT exceeded proceed to the Data Analysis sections.
2. If the limit is exceeded on a given sample follow the dilution technique described in (Pro.C).
C. Dilution technique for samples which exceed the LOL.
1. Dilute used oil with Fresh oil of the same origin in a 1:1 ratio by volume (**may dilute to just under LOL if desired to maximize signal).**
2. Using a 10 ml graduated cylinder (w/stopper) fill to the 5ml mark with Fresh oil.
3. Fill the same cylinder the rest of the way to the 10 ml mark with the used oil requiring dilution.
4. Place the stopper in the cylinder to seal it from leakage and shake vigorously (by hand or mechanical shaker) until a homogeneous mixture is obtained (at least 2 minutes). Warming the mixture may aid in obtaining homogeneity.
D. Obtain spectra for diluted samples as necessary.
E. Discard spectra that exceeded the LOL.

**Data Analysis**
A. Process the triplicate spectra of each sample to obtain the spectral average.
B. Correct the results of the spectral averages to the 0.05 mm path length equivalent based on the actual path length obtained in (Cal.B.2).
C. Subtract the Fresh oil spectral average from each used oil spectral average (1:1 ratio for undiluted uses a factor of 1.0, 1:2 ratio for diluted samples uses a factor or 0.5).

D.  Calculate a baseline from 1800 – 1665 cm-1 on the subtraction result spectra obtained in (Data.C) above.
E.  Integrate the corrected area between the baseline points calculated in (Data.D) above.
F.  Multiply the area obtained by 200.
G.  Report the results for all samples (Fresh oil should be zero) in units of Absorbance * $cm^{-1}$/1 cm (**Abs./cm$^2$**).

**Bias:** not available
**Precision:**
A.  Repeatability:  not available.
B.  Reproducibility : Initial estimate at the 95% (+/-1.96 sigma) confidence interval:
      1.   +/- 12 Abs. *$cm^{-1}$/1 cm (**Abs./cm$^2$**).