MINIFLASH TOUCH

ADVANCED

Rev. 3

Contents

1. Objective of this workshop

By finishing this advanced training program you will be able to perform complex flash point tests including sample handling for special applications in an integrated lab environment. Thus this workshop will first show how to set up the MINIFLASH Touch in a given IT infrastructure.

We will introduce to you some of the unique analysis features of the MINIFLASH TOUCH and will create a dilution curve for gasoline containing diesel fuel.

This workshop consists of:

1. Introduction and theoretical background to special FP applications (by trainer)
2. How to set up the MINIFLASH Touch with a PC and a printer
    a. Check or set network settings
    b. Remote operation
    c. Install printer
3. Simple remote controlled flash point tests (supervised)
    a. Check Anisol/Dodecane (CRM)
    b. Compare operation from the notebook vs. operation at the unit
    c. Verify CRM results
4. Exercises
    a. Study the effect of a single contaminant
    b. Dilution curve
5. Questions

2. Workshop preparation

Please make sure you have the following items available

1. MINIFLASH FLP (or FLPH) Touch incl. accessories
2. Power cable
3. Protective clothing (Gloves, Safety glasses, Lab coat)
4. Compatible printer (USB and/or network, compact printer)
5. Keyboard
6. Notebook with Windows Explorer
7. 2 patch (Ethernet) cables for network connection
8. Router with DHCP server
9. Samples: Dodecane, Anisole, Gasoline, diluted Gasoline, Perfume
10. 2 or 3 100ml-cups for sample mixing
11. Sample package (sample cup, pipette,…)

3. Introduction and Theory

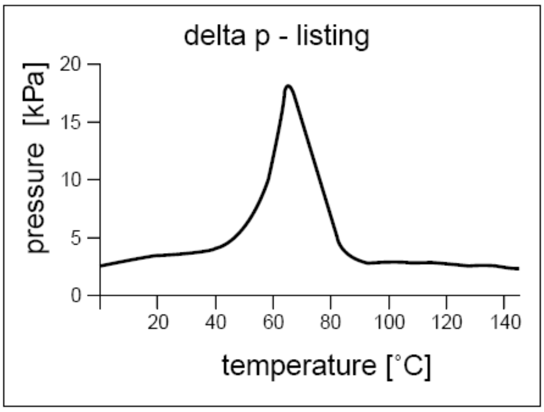
3.1. Ramp and Equilibrium measurements

For special applications it is important to know, if a ramp measurement, an equilibrium measurement or a flash/no flash measurement is performed.

3.1.1. Ramp measurements

Ramp measurements are used for a number of classical tests, like the Pensky Martens method ASTM D93, the Abel IP 170 and Tag D56 methods. For a ramp measurement the sample is heated to a starting point and afterwards the temperature is increased at a constant rate. At regular intervals a specified test flame or a glowing wire is lowered into the vapor space of the briefly opened test cup. The temperature at which a flash is observed is recorded as the flash point.

Sometimes small amounts of contaminations do not give a flashpoint. Instead flames are building a “halo” only, which is not considered sufficient for flashpoint detection in traditional methods. The MINIFLASH pressure measurement makes these “almost” flashpoints visible, by showing the pressure curve for all ignitions during a test.



During ramp measurements, with every ignition a small amount of vapor is being burned. Typically this effect is negligible or can be controlled. For example if the ignition is not being set every 1°C, but for example every 3°C. This allows more vapors to be formed, before the next ignition is being set.

In order to achieve comparable results it is also of critical importance to set the correct initial flash temperature. Typical ramp measurements have to start at least 18°C below the expected flash point. For samples containing small amounts of volatiles, this procedure can lead to fumes being slowly burned away.

Example: Diesel containing gasoline may give a flashpoint at 35°C, when tested with a ramp measurement beginning at 15°C. The same sample may yield a flashpoint at 25°C, when tested with a Flash/No-Flash test.

3.1.2. Flash/No-Flash measurements

For quick analysis, whether a flashpoint is within or outside specifications, a Flash/No-Flash Test can be performed. In this test a single ignition is set at a specified temperature.

3.1.3. Equilibrium measurements

For some viscous samples, like paints, gums and viscous products containing solvents, international transport regulations (ADR) may require the sample to be tested with an equilibrium method, like ISO 1523, ISO 3679 and ISO 3680. The basic equilibrium method is based on the Abel method.

For an equilibrium measurement always a fresh sample is used. The cup is kept heated at a specified temperature, until equilibrium is being reached. This allows outgassing of volatiles, which are being kept “trapped” inside the viscous sample. After equilibrium time, only one ignition is being performed and then the sample is disposed of.

A single flash / no flash test at a specified temperature is required for determining, whether a samples´ flashpoint is above or below a certain limit. An exact flashpoint determination requires multiple tests, multiple samples and usually is a very time consuming procedure.

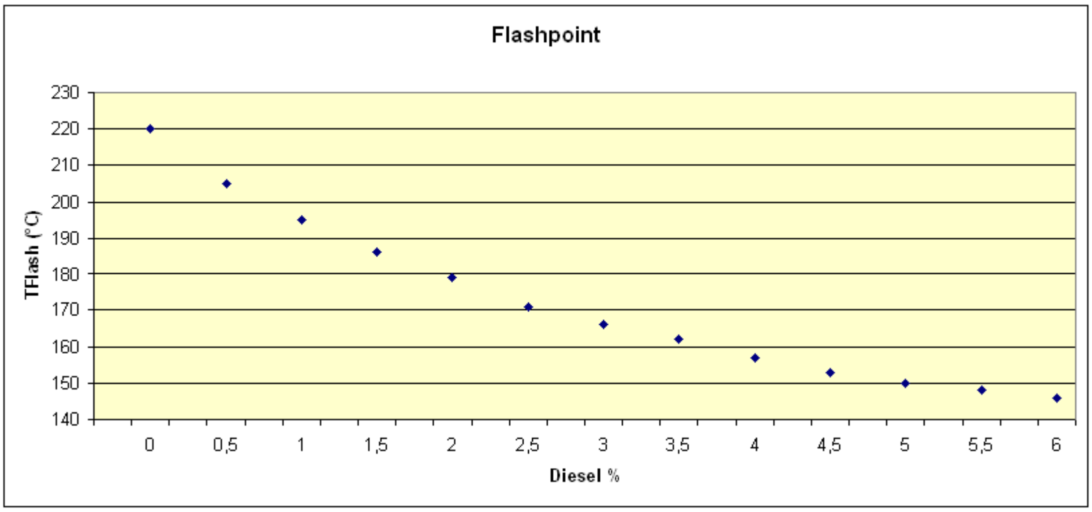
Since 2011 the ADR also to test viscous samples not only with an equilibrium method, but also with a slow ramp measurement, like procedure ASTM D93B. D93B has been developed, because the original D93 method did not give consistent results for viscous samples like lubricating oils. For D93B the heat rate is reduced to 1-1,6°C per minute, which produces outgassing conditions similar to an equilibrium method.

The MINIFLASH Touch includes ramp measurements, Flash/No-Flash Tests as well as equilibrium methods. By adjusting parameters like air feed, heat rate und step width, the MINIFLASH TOUCH can excellently simulate different closed cup methods. The MINIFLASH is accepted by the US D.O.T. for the determination of the flash point of hazardous goods.

3.2. Dilution measurements

3.2.1. Diesel in Lubricating Oil

Dilution measurements, as implemented in the MINIFLASH TOUCH, have first been required by the US Navy to detect contamination of the engine´s lubrication oil with Diesel. As an engine wears with normal use, it is not uncommon for small amounts of fuel to leak into and contaminate the lubrication oil in the engine. Diesel contamination reduces the ability of the lubrication oil to protect the bearings and other moving parts within the engine causing accelerated wear. The presence of diesel in lube oil dramatically lowers the oil's flashpoint temperature. Creating a dilution curve allows to measure the amount of Diesel, that has spilled into the engine. It allows machinery personnel to decide, whether it is required to change the oil in ships and machinery.



3.2.2. Contamination with incompatible fuels

The dilution method can also be used to detect the contamination of an engine's fuel with an incompatible fuel. For example, the contamination of diesel fuel with more volatile turbine or combustion-ignition (e.g. gasoline) fuels results in a lower flashpoint temperature for the fuel. This type of contamination causes destructive pre-ignition, or detonation in the cylinders of the engine. If the contaminated fuel is a turbine fuel, the contamination can easily cause a significant change in the flame profile within the ignition zone of the turbine. This may result in damage to the turbine blades and premature engine failure or engine stalling.

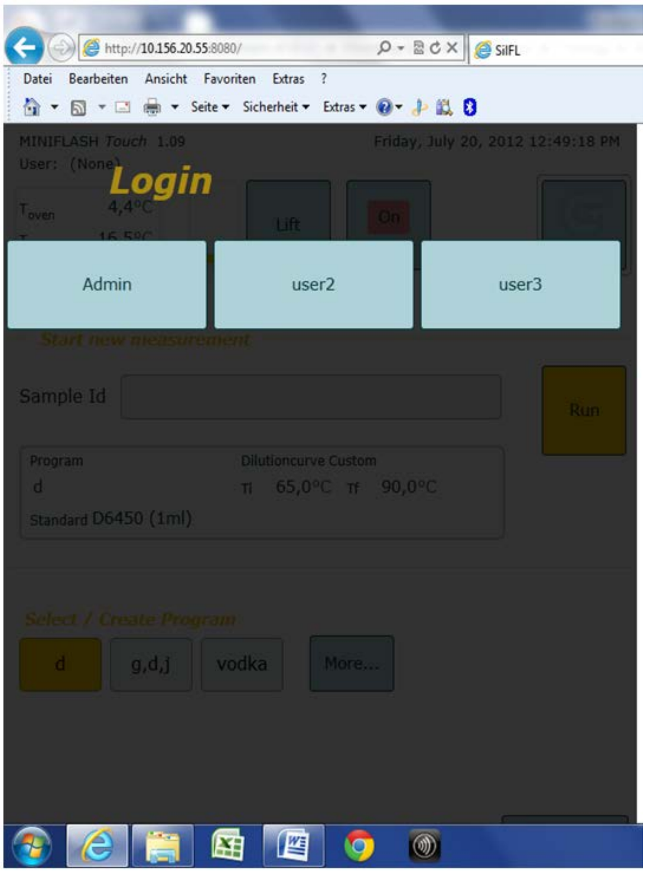
3.2.3. Preconditions for creating a dilution curve

If a dilution curve for lubricating oil is created, it is essential that the lubricating oil is known and can be used for mixing the dilution curve. Different lubricating oils have different flash points and consequently the dilution curve of one lubricating oil is different to another. It is not that important to know exactly about the diesel, as diesel is similar with a flashpoint slightly lower than 60°C.

4. How to set up the MINIFLASH Touch with a PC and a printer

4.1. Check or set network settings

1. Use the patch cables to connect the MINIFLASH Touch and your lab top to the net work (switch)
2. In the MINIFLASH Touch, click on MENU and then on NETWORK
3. Note down the instruments IP-Address and activate the DHCP-Server
4. Start Internet Explorer
5. Type the IP-address and the port no. (:8080) in your browser.
   E.g. if the internet address of your server is 169.254.213.29,
   then please type in http://169.254.213.29:8080/
6. Now you should see the following window:



If no network/switch is available you can connect the MINIFLASH Touch directly to your notebook with the patch cable. You have to use a fixed IP address on both, the MINIFLASH Touch and your PC. An IT person can support you with changing network settings on the PC.

For a measurement please use either the MINIFLASH Touch or your Laptop. Do not use them together because the MINIFLASH Touch supports multiple user instances which may conflict.

4.2. Remote operation

Connect the MINIFLASH Touch to a network and check some new features remotely:

- ☐ Use the patch cable and connect the instrument to the router
- ☐ Switch the instrument on and enter in Admin mode (no password required)
- ☐ Find the Network setup in the settings menu and note the IP address. Make sure the instrument is in DHCP mode (default).
- ☐ Open your browser and connect to the IP address of your instrument at port 8080 (example: http://192.168.2.12:8080)
- ☐ Please note that once connected remotely, you access the instrument in your own instance. This means that you are
  - i. Not replicating the screen from the instrument
  - ii. You can check results and create new programs but
  - iii. You should not start measurements
- ☐ Please enter the Settings menu and find the different languages installed.
- ☐ Open the address http://192.168.2.12:8080/reslist. You will see what data is on the unit. It is transferred as XML file to your computer as extensible markup language, a common data description language, which most of modern LIM-Systems can understand and parse. Come back later to check, once data gets generated for the dilution curve.

4.3. Install Printer

Connect the printer to the MINIFLASH Touch:

- ☐ Connect the USB printer to the MINIFLASH Touch
- ☐ In the set up menu, verify that the printer is installed and select it
- ☐ Print a test page
- ☐ Alternatively, connect a network printer to the router

Installation of the network printer via router

- ☐ Note down the routers IP-Address
- ☐ NOTE: You cannot install ".exe"- or ".zip"-files, MINIFLASH Touch requires a ".inf"-file for printer installation. Load the printer driver .INF-File for your printer on a USB-Device. Alternatively use the generic printer drivers delivered on the Grabner MINIFLASH Touch USB flash device.
- ☐ On the MINIFLASH TOUCH press on MENU and PRINTER MANAGEMENT
- ☐ Select the printer-driver from the USB-device and press INSTALL
- ☐ Follow the installation wizard requirements: Choose "Create a new port" and select "Standard TCP/IP port". Press "Next"
- ☐ Type in the routers IP address and press "Next"
- ☐ Finish by adding a printer name
- ☐ A black screen will pop up: Commit the changes by pressing "Continue"
- ☐ The printer is now ready to use

5. Simple remote controlled flash point tests

Measure the flash point of Anisol and Dodecane using D6450 standard. Connect the MINIFLASH Touch to a network and check some new features remotely:

- ☐ Use the notebook and verify the settings of the Anisol program (you have to be logged in at least as advanced user)
- ☐ Prepare the sample and start the Anisol test from your notebook
- ☐ Check what happens on the screen of you notebook and compare to the screen on the MINIFLASH Touch.
- ☐ Note the FP of Anisol and compare to the value in the instruments calibration sheet
- ☐ Next, switch to the Dodecane program and verify its settings.
- ☐ Prepare the sample and start the Dodecane run again from your notebook
- ☐ Note the differences between the Notebook screen and the MINIFLASH Touch screen.
- ☐ Finally, note the FP of Dodecane and compare also to the result in the instruments calibration data sheet.

	1st result	2nd result	Res. per calib	Offset
Anisol				
Dodecane				

6. Exercises

6.1. Study the effect of a single contaminant (time ~1h)

To study the effect of a contaminant we will check how a sample with lower flashpoint, that is spiked into a sample with a high flashpoint, influences the flashpoint. We will also slowly increase the concentration of low FP sample and monitor the results. In special cases we can also see, how vapors of the low FP sample are being burned away during the test: The ignition will not produce enough pressure for a flashpoint detection, but the pz-curve will show a small increase. When the flash point is reached we will check the final flash pressure: The flash pressure graphic indicates, how fast a sample is fully ignited and as such gives information about the quality of the ignition (e.g. for Diesel fuel).

- ☐ Set up a new program called "Spike Test" using the following settings:
    - i. Ti=50°C, Tf=90°C, select D7094SCR (screening) standard with a heate rate of 5.5°C per minute. Also use "Manual" mode.
    - ii. NOTE: with MANUAL sample handling, the analyzer waits until the oven reaches the initial temperature, then the message „Insert Sample" will appear. After you have done this you have to press the CONTINUE button a second time to start the measurement.
- ☐ Run pure n-Dodecane in the 2ml sample cup.
- ☐ While waiting for the first result put 10ml dodecane in a glass beaker and spike it with 3 drops of Anisol.
- ☐ Measure the spiked dodecane with same program
- ☐ The remaining solution spike again with 3 drops Anisol for another measurement.
- ☐ Repeat the last step until you are running out of sample.
- ☐ During the measurements monitor the flash pressure and the pz course (press on the flash pressure profile to toggle between both graphs)
- ☐ Finally compare the results in the results list, look at detail results to study the pz course and check especially the flash pressure profile during the flash point.

	Flash Point	Final pz
Dodecane (C12)		
10ml C12 + 3 drops Anisol		
8ml spiked C12 + 3d Anisol		
6ml spiked C12 + 3d Anisol		
4ml spiked C12 + 3d Anisol		
2ml spiked C12 + 3d Anisol		

6.2. Dilution Curve to check contamination of Diesel (time ~1h)

With a series of known concentrations of a contaminant one can measure and store a dilution curve. We will collect such a curve of diesel samples contaminated with gasoline at specific concentrations. Further, we will check what happens to unknown diesel samples, if they are contaminated.

We will use ASTM D7094 and a stirrer.

- ☐ Create a measuring program called “Diesel” with Ti = 50°C, Tf = 75°C
- ☐ Create another program called “Diesel lo gas” with Ti = 40°C, Tf = 75°C
- ☐ Create a third program called “Diesel hi gas” with Ti = 20°C, Tf = 75°C and Ti-T=2°C.
- ☐ Create a concentration series and corresponding measuring programs:
  - i. Program “Diesel hi gas” for 3% to 2% gasoline contamination
  - ii. Program “Diesel lo gas” for 1.5% and less gasoline contamination
  - iii. Program “Diesel” for the pure Diesel sample
- ☐ If the 3% sample gives an immediate FP at 20°C and if time allows you may modify the “Diesel hi gas” and start at Ti=15°C with pre-cooled sample and cup.
- ☐ Of all measured samples in the group, calculate the mean value and modify the program “Diesel” as follows:
  - i. Set the Ti down to 40°C
  - ii. In the programs menu click on the button “Dilution Curve”. This button is shown in the “Name” field.
  - iii. Add the mean values for each corresponding concentration
  - iv. Select the colour you deem appropriate for each concentration
- ☐ Take a diesel sample with unknown gasoline contamination and measure the flashpoint using the “Diesel” program.
- ☐ Enter into the results list to see the concentration of the corresponding samples.

Dilution Curve

Samples	S/N	S/N	S/N	S/N	Mean
+ 3% Gas					
+2.5% Gas					
+ 2% Gas					
+ 1.5% Gas					
+1% Gas					
Pure Diesel					

7. Questions

1. How to find the IP Address of your MINIFLASH Touch in the network?
   - ☐ Menu → Settings
   - ☐ Menu → Network
   - ☐ Check on the switch/router
   - ☐ Ask your IT manager

2. At which port is the web server of the MINIFLASH Touch listening for connections?
   - ☐ Port Nr.: 80
   - ☐ Port Nr.: 21
   - ☐ Port Nr.: 8080
   - ☐ Port Nr.: 0815

3. What indicates the default printer used by the instrument?
   - ☐ A blue dot
   - ☐ A green star
   - ☐ A red flag
   - ☐ The highlighted name

4. A single, lower flash contaminant in a higher flash matrix
   - ☐ Decreases the FP
   - ☐ Increases the FP

5. The higher the concentration of the contaminant
   - ☐ the higher the final flash pressure
   - ☐ there is no effect
   - ☐ the lower the final flash pressure
   - ☐ effect depends on profile

6. Why can the 3% gasoline give an immediate FP at 20°C but can show a FP around 35°C when starting with Ti of 15°C?
   - ☐ Starting at 20°C there are already enough fumes producing sufficient energy to ignite the full sample.
   - ☐ When starting from 15°C the sample has time to be more homogenised when passing 20°C, so real FP comes around 35°
   - ☐ Starting from 15°C, fumes forming gradually burn away so at 20°C there is no FP.
   - ☐ At 15° the gasoline fumes condensate on the oven surface preventing a flash at 20°C.