

# PMA 500: Determination of the Degree of Contamination of Polluted Samples

Relevant for: Petro industry

Fast and precise determination of the contamination degree by serial dilution of a sample



## 1 Introduction

As a longstanding producer of flash point testing equipment, Anton Paar added a new innovative instrument to its portfolio – the PMA 500.

PMA 500 offers premium technologies for high sample throughput, an automatic motor-driven multi-function head and a long lasting encapsulated hot wire type electric igniter.

It guarantees straightforward and automatic handling, maximum safety and unrivaled ease of use in combination with a self-explanatory user interface.

### 1.1 Polluted samples

During the production, transport or the life cycle of your respective product it may get contaminated or diluted by chemical reactions or other substances (traces of volatile contaminants, water, engine oil, etc.) which can affect its flash point significantly. Depending on the concentration of the contamination or impurity, the flash point deviation might be greater or smaller.

Examples for such contaminations are:

- Pollution of motor oil by diesel fuel by oil dilution
- Pollution of diesel fuel by gasoline because of subsequent transportation of these fuels in pipelines or tank vehicles
- Change of quality of turbine oils while in use due to oxidation or thermal decomposition

PMA 500 is able to determine the grade of pollution fast and precisely.

With the aid of a specifically created “Product” definition the pollution grade of a series of samples can be monitored. This “Product” definition contains all required parameters for measuring the flash point of a given sample.

Within the “Product” definition, an impurity matrix lists a given impurity concentration of the sample with the respective flash point.

For this impurity matrix you need to establish a dilution series with the flash points of the differently contaminated sample. Simply mix the pure sample with the impurity at different percentages and perform a flash point test for each percentage. The dilutions should cover the range of the expected pollutions of the unknown samples. The more data (dilution and matching flash point) you enter into the impurity matrix, the more exact the calculated assumed impurity of a sample will be.

When you then perform a flash point test for a polluted sample with a product, the PMA 500 will calculate the assumed impurity concentration on the base of the found flash point.

This application report describes all necessary steps for creating a product definition with an impurity matrix to determine the contamination of a sample.

- Step 1 Create a dilution series for your sample.
- Step 2 Search for the flash point for each dilution percentage.
- Step 3 Repeat each flash point test to confirm the result.
- Step 4: Create a product definition and enter the dilutions with the respective flash points into the impurity matrix.

As example we used motor oil that has been contaminated with different amounts of diesel fuel.

## 2 Instrument



Figure 1: PMA 500

### 2.1 Accessories

The following accessories are required for testing:

- Standard test cup
- Lid for standard cup
- Multi-detector
- Electric igniter or
- Gas igniter

## 3 Step 1: Creating a Dilution Series

We used a 15W-40 motor oil and a B7 diesel as parent materials. From these materials we established a dilution series containing seven different degrees of contamination of diesel fuel in motor oil.

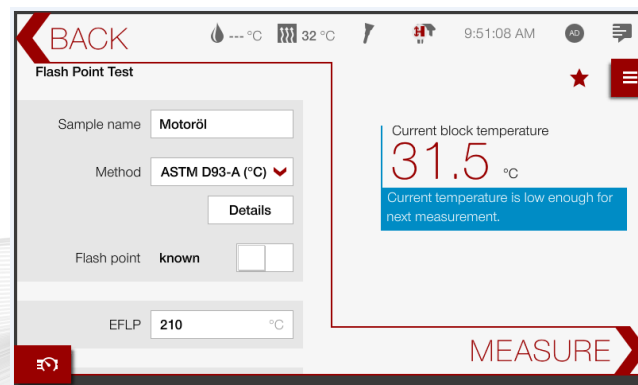
Sample Dilutions
100 % motor oil
1 % diesel in motor oil
5 % diesel in motor oil
10 % diesel in motor oil
20 % diesel in motor oil
40 % diesel in motor oil
60 % diesel in motor oil
80 % diesel in motor oil
100 % diesel

Table 1: Dilution series

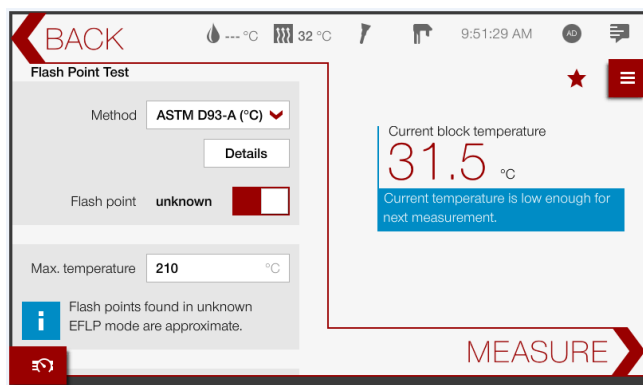
## 4 Step 2 and 3: Determination of the Flash Points for the Dilution Series

The flash points of the differently contaminated samples have been determined using method ASTM D93-A.

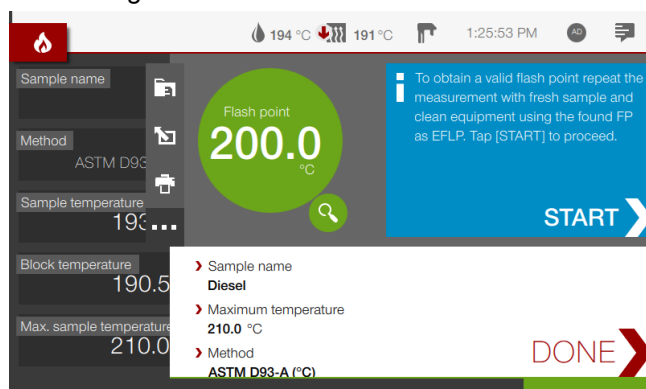
1. At the PMA 500 open the "Flash Point Test" screen.
2. Enter a sample name.
3. Select ASTM D93-A as method.



4. As the flash point of the diluted samples is not known, set the "Flash point" switch to "unknown".
5. Set the maximum test temperature. When this temperature is reached without finding a flash point, the test is stopped. In this example we set the "Max. temperature" to "210 °C".



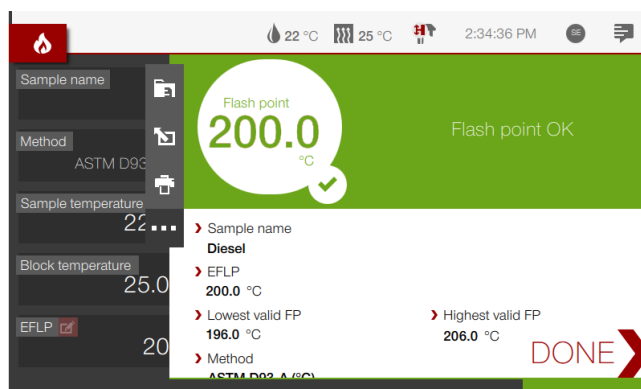
6. Fill the sample into the test cup, place the lid with the multi-detector on to the cup and insert the test cup into the PMA 500.
7. Tap <MEASURE> to start the test.
8. When the test is finished the found flash point temperature is displayed and the device starts cooling.



**Please note:** Flash points found in “unknown flash point” mode have to be considered as approximate. Therefore, the flash point test must be repeated with clean equipment and a fresh portion of the sample. For this repeated flash point test, the found flash point from the search will be used as expected flash point.

9. The PMA 500 will guide you through the necessary steps. Simply tap <START> in the blue segment of the result screen.
10. When the instrument has reached the safe-handling temperature, tap <UP> to move the multi-function head up.
11. Remove the test cup from the instrument and discard the sample.
12. Clean and dry all equipment thoroughly.
13. Fill a fresh portion of the sample into the test cup.
14. Insert the assembled test cup into the instrument.
15. Tap <DONE>.
16. Tap <START>. The measurement is started using the flash point found in the previous measurement as EFLP.

17. When the test is finished the found flash point temperature is displayed and the device starts cooling.



18. Repeat the steps above for each sample in your dilution series.

After performing two flash point tests (as detailed above) for each sample dilution, the following flash points have been determined:

Sample Dilutions	Flash Point [°C]
100 % motor oil	208.0
1 % diesel in motor oil	183.0
5 % diesel in motor oil	135.0
10 % diesel in motor oil	113.0
20 % diesel in motor oil	97.5
40 % diesel in motor oil	81.5
60 % diesel in motor oil	74.5
80 % diesel in motor oil	69.5
100 % diesel	65.0

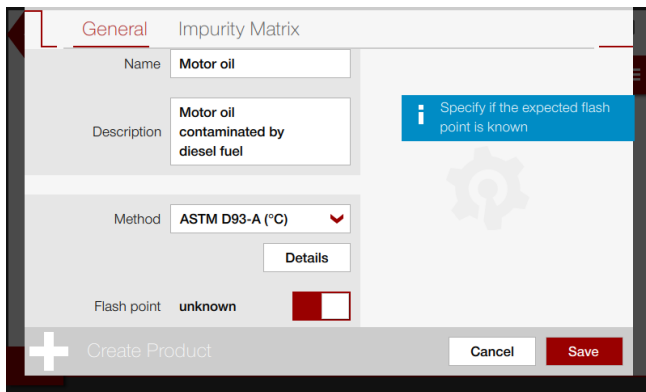
Table 2: Flash points for dilution series

## 5 Step 4: Creating the Product Definition

As the final step we created a product definition with an impurity matrix with the flash point data we gathered for the dilution series in the steps above.

To define a new product:

1. From the menu select **"Products"**.
2. Open the context menu and select **"New"**.
3. Enter a name for the product and an optional description.

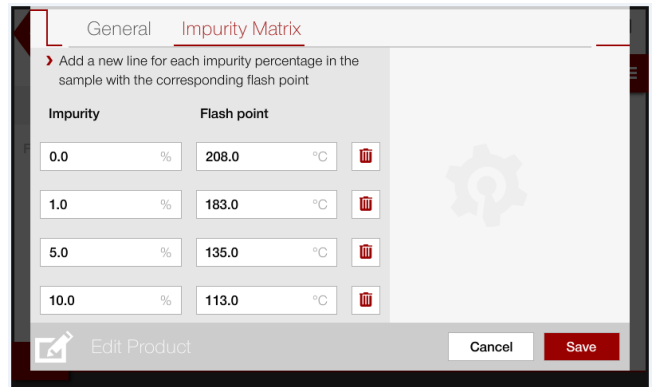


4. As the aim of this product definition is to determine the flash point of the samples and thereby calculate their impurity, set the **"Flash point"** switch to **"unknown"**.
5. Swipe the screen up to set the maximum test temperature. When this temperature is reached without finding a flash point, the test is stopped.

The maximum temperature should be preset at least to the highest expected flash point plus an additional safety margin to make sure that the flash point will be detected in any case and the test will not be terminated by reaching the overflow temperature.

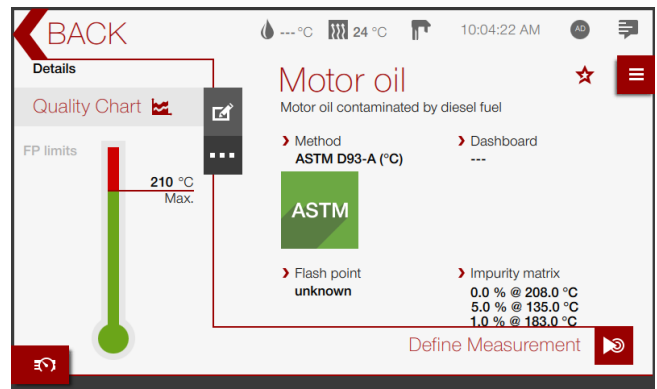
In this example we set the **"Max. temperature"** to **"210 °C"**.

6. Select the **"Impurity Matrix"** tab.
7. Select **"Add"** to create as many new lines as required.
8. Now enter the dilutions and detected flash points which were determined earlier (see table 2 above).



Impurity	Flash point
0.0 %	208.0 °C
1.0 %	183.0 °C
5.0 %	135.0 °C
10.0 %	113.0 °C

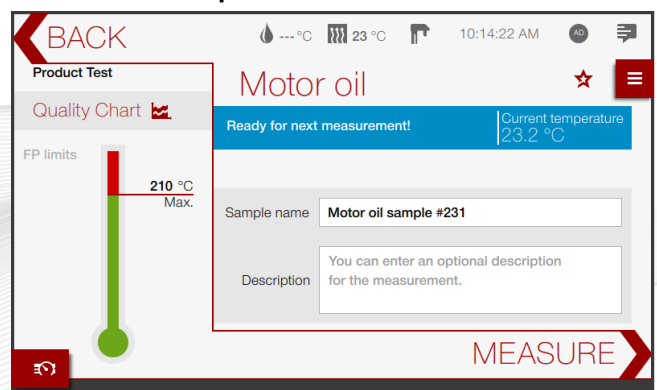
9. Select **<Save>** to save the product definition. The product details screen will be displayed. You can scroll the **"Impurity matrix"** section if you wish to view more data.



## 6 Measurement

Now we can start a measurement with a motor oil sample with unknown diesel contamination.

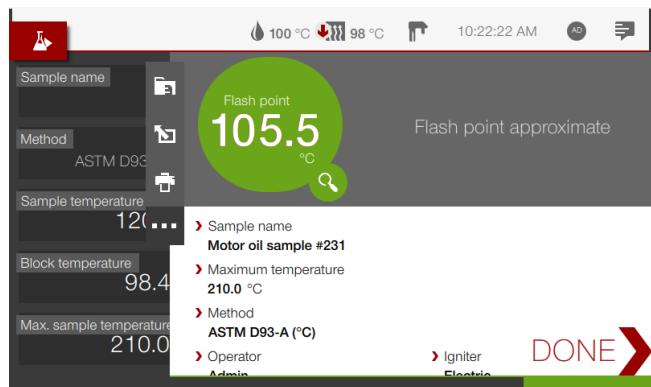
1. In the product details screen, select **"Define Measurement"**.
2. Enter a **"Sample name"**.



3. Fill in the sample and place the test cup with lid and multi-detector into the PMA 500.
4. Tap **<MEASURE>**.

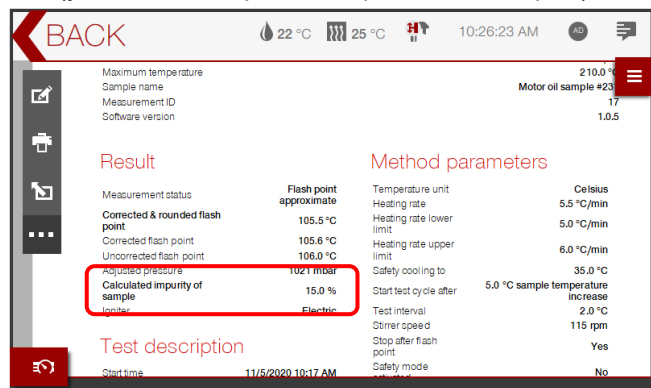
## 7 Results

When the test is finished, the found flash point is displayed.



To view the calculated impurity of the sample, open the test report:

1. From the menu select "Data Explorer".
2. Select "Test Results".
3. Select the respective test from the list. A preview of the test report is shown.
4. Slide the report to view the calculated impurity (you could also print or export the test report).

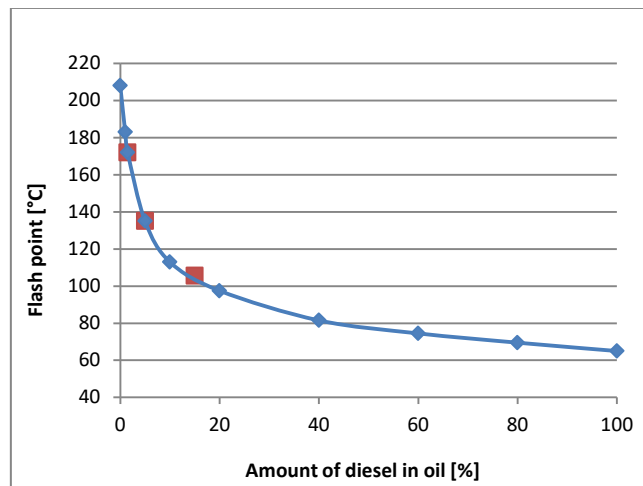


The following mixtures have been successfully tested by using the predefined product:

% Diesel in motor oil	FP [°C]	Calculated impurities [%]
1.5	172.0	1.5
5	135.0	5
15	105.5	15

Table 3: Flash points and calculated impurities

The diagram below shows the dilution series with the found flash points for the unknown samples.



## 8 Discussion

With the new PMA 500 it is possible to determine the degree of pollution in unknown samples fast and efficiently.

Mixtures with known contaminations have to be prepared and measured.

A product definition is created using the flash points of the contaminated mixtures as well as of the pure samples.

When an unknown sample is measured using the "Product" feature the flash point and degree of contamination are listed in the test report.

### Abbreviations used in this application report:

FP: Flash point

EFLP: Expected flash point

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