

Flash point determination of Asphalt, Tar and Bitumen by use of GRABNER MINIFLASH

1. Problem

Measuring flashpoint of asphalt, bitumen or tar is a most challenging test. Industrial coal tar pitch for example is solid at ambient temperature, softens around 100-130°C and becomes fully liquid at 180°C. When cooled down the tar sample becomes rock solid in seconds, making it difficult to clean flashpoint testing equipment and to continue with the measurements.

Tar is used for a variety of industries. E.g. for aluminium production, coal tar pitch is mixed with petroleum coke and baked at high temperatures to produce anodes and cathodes for smelting aluminium. Combustible coal tar pitch also poses a risk to the health of industry workers, if inhaled during the production process. When heated or incinerated, coal tar pitch produces hazardous odours. For safety reasons it is therefore of critical importance to observe the flash point. This is best done by testing flashpoint in a sealed cup without an open flame, using the least possible amount of sample to ensure that no hazardous odours are spreading.

2. MINIFLASH versus Open Cup

In general the open cup method is used for testing tar, bitumen and asphalt samples at high temperature. The drawbacks of this method are evident especially when used for coal tar pitch: A large amount of sample (70mL) is heated in an open sample cup, so a harmful concentration of hazardous airborne particles can form and spread quickly. Open cup flashpoint measurements are not highly reproducible and cleaning of the big cups is a time consuming and cumbersome thing to do.

The Grabner MINIFLASH on the other hand is using only 1-2 mL of sample and tests the flashpoint in a completely closed cup. The flashpoint is detected via a patented method, without an open flame. The small sample cup guarantees faster heating of tar samples, cleaning is also easier with the MINIFLASH than with classical open or closed cup testers. The fast sample throughput and an improved repeatability make the MINIFLASH an unmatched analyzer for this application.

3. Measurement procedure

MINIFLASH offers a very convenient way to test coal tar pitch:

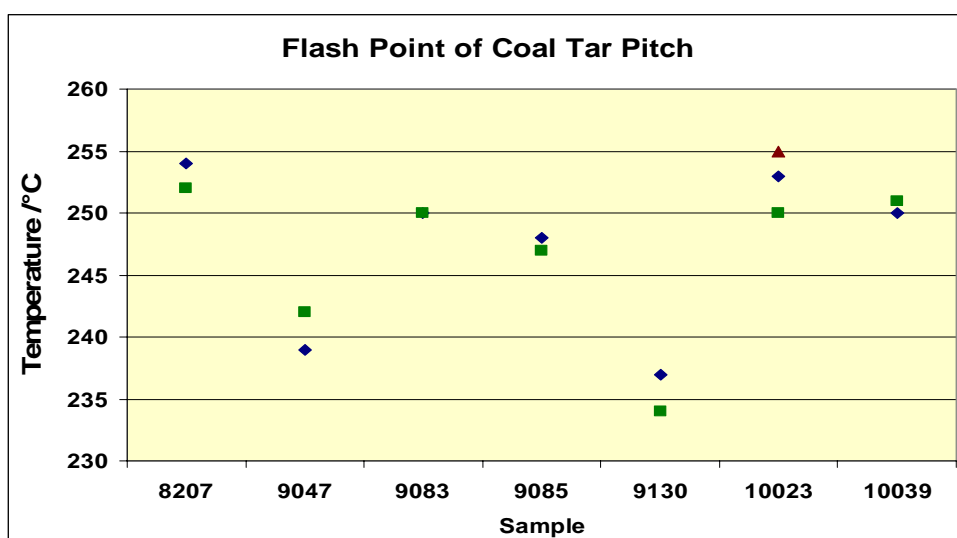
- 1. Create a test program:** Use the preprogrammed D7094 or D6450 method and set starting temperature (Ti) at least 18°C below the expected flash point, e.g. at 200°C. D7094 has a lower heating rate and is preferable for testing bituminous samples. The Sample has to be liquid at start temperature to guarantee that it is not hardening after the test, as the instrument is cooling down to start temperature, before the sample cup can be accessed.

2. **Create a cleaning program to clean arc electrodes:** Use the D7094 or D6450 program and set Tf as well as Ti to 200°C.
3. **Fill in the coal tar pitch** into the sample cup and start the measurement immediately. Use the stirrer, as for some kind of samples it might be necessary.
4. **Upon test completion:** Put 1-2 mL of GRABNER INSTRUMENTS piston oil into the still hot and liquid sample and stir the liquid mixture with a wooden stirrer, until a homogenous mixture evolves after 20s.

4. Results

Seven unknown different samples of coal tar pitch have been tested, showing very good repeatability. The screening method was used to identify the expected flashpoint and set the initial temperature Ti, prior to the actual tests. In the below results list Ti was set at least 30°C below the expected flashpoint.

Sample name	Ti	Tf	Method	Tflash/[°C]	Mean [°C]
8207	200	300	D6450	254	253,00
8207	200	300	D6450	252	
9047	200	300	D6450	239	240,50
9047	200	300	D6450	242	
9083	200	300	D6450	250	250,00
9083	200	300	D6450	250	
9085	200	300	D6450	248	247,50
9085	200	300	D6450	247	
9130	200	300	D6450	237	235,50
9130	200	300	D6450	234	
10023	200	300	D6450	253	252,67
10023	200	300	D6450	250	
10023	200	300	D6450	255	
10039	200	300	D6450	250	250,50
10039	200	300	D6450	251	



Table/Fig. 1: Flash point of unknown Coal Tar Pitch samples, tested with the GRABNER MINIFLASH.

5. Cleaning procedure

In order to get best results, cleaning of the flash point tester is most important, especial after testing sticky samples. The small MINIFLASH sample cup and the user friendly design guarantees easy cleaning of the instrument:

1. Remove the stirrer from the cup and do a rough pre-cleaning
2. Use a tissue to remove the oil-tar mixture from the sample cup and do a rough cleaning
3. Let the cup cool down
4. Remove the cup from the cup carrier, put the stirrer into the cup, fill the cup with Toluene and put it into an ultrasonic bath for approximately 15 min.

On the instrument

5. Clean the cup temperature sensor and the arc pin with the brass brush
6. Use Toluene to clean the oven surface of the MINIFLASH after every 20 measurements
7. Start the cleaning program twice before inserting the next sample to be tested. By using the cleaning program, an electric arc is set to the electrodes multiple times to ensure that no sample accidentally stays on the electrodes.

If another sample cup is available you may consider to skip cleaning points 1-4. You only need to clean the instrument before inserting the next sample. Thus a high sample throughput is guaranteed.

As the cleaning procedure is done a fairly high temperature, the use of protective gloves and pliers when handling the sample cup is recommended.

6. Summary and Conclusion

By using the Grabner Instruments flashpoint method and testing only 1-2 mL of sample in a completely sealed metal cup, the MINIFLASH flash point analyzer guarantees highest safety and eliminates the risk of generating open flames and hazardous fumes when testing flash point. The very good repeatability and an easy cleaning procedure make the GRABNER INSTRUMENTS analyzer a clearly superior analyzer when testing the flash point of **tar, asphalt or bitumen samples.**