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Filter Blocking Tendency

ASTM D2068, IP 387, CEN N 403, BS EN 590, IP PM EA

Cold Soak Test

ASTM D7501, IP PM EA, CEN N 403, CGSB 3.0 No. 142

Matthijs van der Spek Rev. 1.01

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Content of Presentation

- ✓ What is a Filter Blocking Tendency (FBT) Test?
- ✓ Why is the FBT important?
- ✓ FBT Background
- ✓ Standards
- ✓ The FBT Method
- ✓ Tamson Filter Blocking Tendency-Tester
- ✓ Recommended TFBT Set-Up
- ✓ Competitive Advantages of TFBT
- ✓ Cold Soak Methods
- ✓ Set-Up TLB50 for Cold Soak

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Filter Blocking Tendency (FBT)?

What is a FBT Test (1/2)?

- FBT is a calculated dimensionless value that defines the tendency of particulates in a fuel to plug or block a filter.
- For this test*, 300 mL of sample is pumped at a constant flow rate (20 mL/min) through a specified filter medium.
- Both pressure difference across the filter and the volume of fuel passing through the filter are monitored until the pressure reaches 105 kPa or the volume of fuel passing the filter medium reaches 300 mL.

*ASTM D2068 and IP 387

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Filter Blocking Tendency (FBT)?

What is a FBT Test (2/2)?

- When 300 mL of fluid is pumped, the end pressure is used to calculate the FBT number (please see table 1).
- Or, when the pressure reaches 105 kPa *before* the 300 mL of fluid is passed, the volume of fluid pumped at this point is used to calculate the FBT number (please see table 2).

Table 1: 300 mL Passed

kPa	FBT
20	1.017979
40	1.070105
60	1.151751
80	1.257179
100	1.380952
105	1.414214

Tabel 2: Overpressure

ml	FBT
10	30.01666
20	15.0333
30	10.04988
40	7.566373
50	6.082763
100	3.162278
150	2.236068
200	1.802776
250	1.56205
300	1.414214

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Filter Blocking Tendency Today

Importance Today?

- Fuels which have a high FBT could potentially block filters in the distribution network or during use in a vehicle, power plant, or aircraft.
- Cold flow issues with diesel containing FAME (biodiesel) and FAME material have resulted in the development of the new Energy Institute test method standard to check quality of FAME and (blended) diesel fuels to avoid major fuel operability problems.
- Fuel cleanliness is also an important issue as modern fuel injectors and injection pumps are being manufactured to more precise tolerances. Particle contamination, degradation, or corrosion of pipelines and storage tanks can quickly clog filtration systems.

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Filter Blocking Tendency Today

Importance Today, Examples?

Sweden: filter blocking problems reported initially winter 2011/12, continued 2012/13, 2013/14 and more serious during winter 2014/15:

- High FBTs measured 3.16 – 7.55.
- High levels of Saturated Monoglicerides (SMGs) and Sterol Glucosides (SGs) found in some samples.
- Some evidence of oxidation stability issues.
- Zn contamination found in some samples – vehicle fuel system components?
- Effect of detergents/dispersants?

Picture: contamination found in oil filter!

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Filter Blocking Tendency Today

Importance Today, Examples?

Italy: filter blocking problems result in vehicle power loss.

- Fame contaminants identified as polyethylene, nylon, and SGs in some cases.
- Centrifuge test (ASTM D2709) employed to isolate and determine level of contaminants.
- Correlation with FBT and EN 12662 total contamination test.

Picture: oil filter clogged after 9k miles using > B5 diesel.

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Filter Blocking Tendency Today

Importance Today, Examples?

Spain: filter blocking problems result in vehicle power loss.

- Problems appeared in Spanish market that caused filter blocking in diesel vehicles.
- Filter analysis showed that blocking were caused by some insoluble components coming from FAME (Saturated monoglycerides) that precipitated at low temperatures.

Picture 1: clogged filter!

Picture 2: filter analsys show insoluble components coming from FAME.

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Filter Blocking Tendency Today

Importance Today, Examples?

UK: Filter blocking problems reported initially winter 2012/13, continued 2013/14, slightly (19%) reduced in 2014/15 after introduction of voluntary FBT test limit of 2.52.

- Incidents linked to temperature <3°C
- Trend for higher FBTs (>2.52) in problem areas.
- European survey shows FBT <1.7 and most below 1.1.
- Evidence of base fuel quality issues – oxidation stability, poorly blended cold flow additives.
- Saturated FAME levels typical 20 - 25% but can be as high as 40%.
- Filter analysis shows SMGs, EVA and some evidence of polyethylene and polyamide (nylon?).
- Freezer rig test using suction through vehicle system confirms filter blocking on fuel from affected area but filter unblocks on warming.
- Designed experiment investigating high saturate FAMEs with various contaminants (SMGs, SGs, water).

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Filter Blocking Tendency Today

Importance Today, Examples?

Two examples of B5 samples with a floating layer at the top.

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Filter Blocking Tendency Today

Background to Sterol Glucosides (SGs)?

- The presence of SGs in vegetable oils (soybean and palm) is a well-known fact.
- Vegetable oil refining technologies don´t remove all of these SGs molecules.
- SGs are insoluble in FAME as well as in fossil fuel at very low contents. Ambient temperature and storage time of FAME are critical to establish a solubility limit.
- Some market issues in USA and Europe related with filter blocking have been linked with the presence of very low levels of SGs in the fuel.

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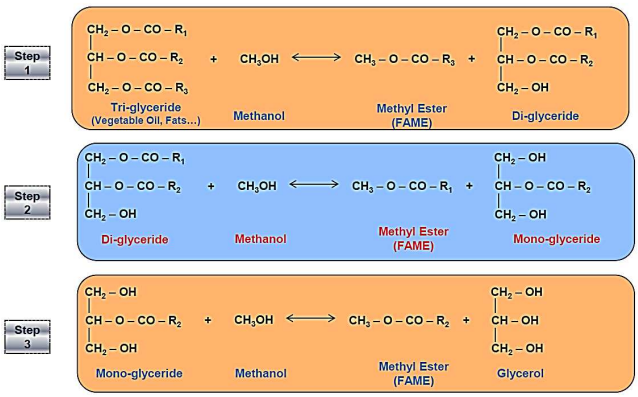
Filter Blocking Tendency Today

Background to Saturated Monoglycerides (SMGs)?



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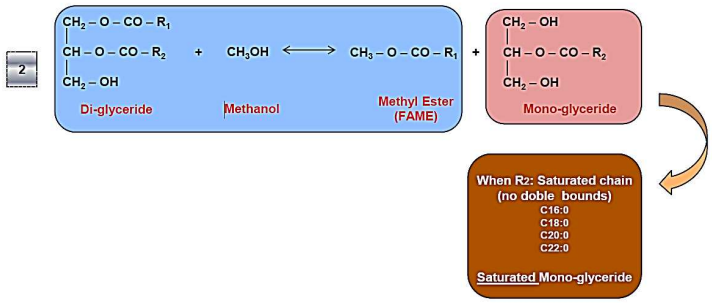
Filter Blocking Tendency Today

Background to Saturated Monoglycerides (SMGs)?

During the transesterification process it could happen that the reaction is not complete and finalizes in the di-glyceride step (1) or mono-glyceride step (2).



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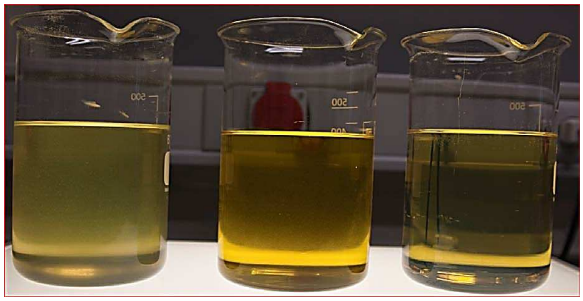
Filter Blocking Tendency Today

Background to Saturated Monoglycerides (SMGs)?

- Total Monoglyceride content in FAME depends on:
  - Optimization of the transesterification production process.
  - Feedstock used to produce FAME.
- In general, the ratio of Saturated Monoglyceride is equivalent to the ratio of Saturated Methyl Ester in the FAME.
- Saturated component ratio depends on feedstock used:
  - Saturated Content in Palm Methyl Ester: 45-50%
  - Saturated Content in Soy Methyl Ester: 12-17%
  - Saturated Content in Rape Methyl Ester: 5-8%
- Monoglycerides are compounds with a limited solubility in the FAME. SMGs solubility gets substantially worser.
- Higher solubility problems of SMGs in FAME than in diesel (fossil fuel).

Filter Blocking Tendency Today

Background



Sample 1 Sample 2 Sample 3

Sample 1:
B5 made with FAME 0.69 TMG and 0.33 SMG (165 mg/kg estimated SMG in the B5). **Market Issues**

Sample 2:
B5 made with FAME 0.44 TMG and 0.20 SMG (100 mg/kg estimated SMG in the B5). **Market Issues**

Sample 3:
B5 made with FAME 0.52 TMG and 0.15 SMG (75 mg/kg estimated SMG in the B5) **No market issues**

Standards

ASTM D2068 / IP 387 Determination Filter Blocking Tendency:

A test portion of the fuel to be analyzed is passed at a constant rate of flow (20 mL/min) through a specified filter medium. The pressure difference across the filter and the volume of fuel passing the filter are monitored until the pressure reaches 105 kPa or the volume of fuel passing the filter medium reaches 300 mL. The pressure and flow are then used to calculate the filter blocking tendency, where a low number indicates a good fuel.

ASTM D7501 FBT of Biodiesel (B100) by Cold Soak Filtration Test:

In this test method, 300 mL of biodiesel (B100) is stored at 4.5 ± 0.5°C for 16 hours, allowed to warm to 25 ± 1°C, and vacuum filtered through a single 0.7 µm glass fiber filter at controlled vacuum levels of ~70–85 kPa.

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Standards

IP PM EA Cold Soak and Filtration method of FAME or Diesel containing FAME:

A test portion of FAME is conditioned to remove its thermal history by heating at 60°C for two hours and then allowed to cool to 20°C. The test portion is then dissolved in low aromatic kerosine to prepare a 10% blend. The blend is placed in a water bath maintained at 5°C for 16 hours. It is then allowed to warm to 20°C and tested in accordance with IP 387 Procedure B to determine its FBT.

IP PM ES Cold Filter Blocking Tendency:

Published in 2016. More research to follow. Basically two FBT tests are carried out at cold temperatures.

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Standards

Latest Development

- Recently, BS EN 590 has been updated to include Filter Blocking Tendency (IP 387 Procedure B) as a mandatory requirement.
- BS EN 590 describes the physical properties that all automotive diesel fuel must meet if it is to be sold in the UK. Since November 1st, 2015:
  - All UK diesel imports will be subject to new FBT limits.
  - IP 387 Procedure B must be performed on every batch of fuel covered by BS EN 590.
  - This specification change applies to all diesel sold.

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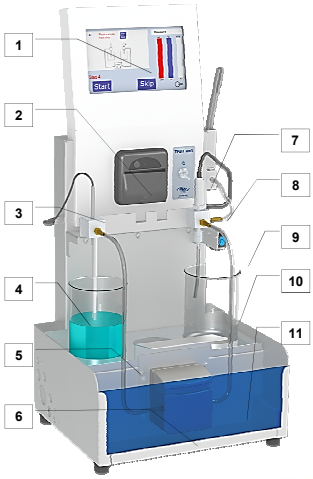
TFBT for ASTM D2068 /IP 387

TFBT



The automated TFBT (P/N 00T0942) provides a graphical guided user interface using a resistive touch screen. This screen guides the user through the test procedure. The guidance results in reliable performance of this test and the user can see what the apparatus is doing when it strictly follows the prescribed steps in the test method.

Operating the TFBT		
Item	Description	Remark
1	Touch Display	Graphical color
2	Printer	
3	Temperature sensor	PT100
4	Beaker 400ml	Sample
5	Hose	Tygon
6	Pump	
7	On/off	Press
8	Pressure sensor and filter	
9	Level sensor	Ultrasonic
10	Beaker	Receiver
11	Safety Window	Removable

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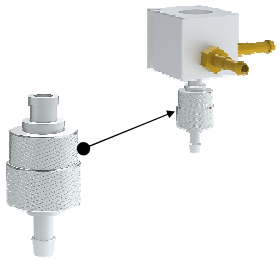
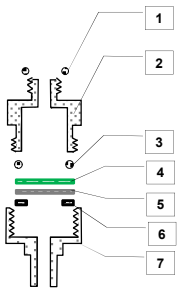
TFBT ASTM D2068 / IP387

Procedure A





Filter "A"	
1	Sealing ring
2	House top
3	Sealing ring PTFE
4	Filter
5	Filter support disc
6	Sealing ring flat PTFE
7	Housing bottom

- Adapter (P/N 15T0002) with filter housing (Millipore, stainless steel, nominal 13 mm diameter with a Luer fitting at the top where it connects with the TFBT).
- Filter A (P/N 24T0064), glass fiber, 1.6 µm nominal pore diameter, nominal 13 mm diameter and with an effective filtration area of 63.6 to 78.6 mm². Box with 98 pieces.

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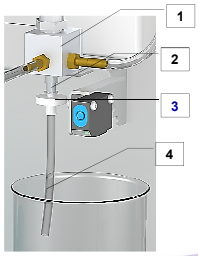
TFBT ASTM D2068 / IP 387

Procedure B



Filter "B"	
1	Adapter block
2	Filter adapter
3	Housing in "tapered"
3	Filter
4	Hose prevent splashing* (4 mm outher diameter) 24T0049

- Adapter block with filter adapter (P/N 15T0003) to allow the test portion to input through the taper fitting and exit from the Luer fitting (upside down).
- Filter B (P/N 24T0067), glass fiber grade GF/A, 1.6 µm nominal pore diameter and effective filtration area of 95.0 to 113.1 mm². Box of 98 pieces.

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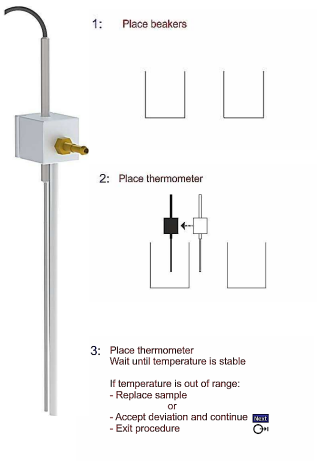
Sample Preparation



- Prepare sample per method instruction (check temperature, shaking, standing of sample for five minutes, etc.)
- Place two beakers (step 1 shown on the TFBT screen).
- Place at least 350 mL of the sample into the fuel reservoir beaker and check that the temperature is still within the range of 15 to 25ºC. Record the actual temperature (all done automatically with the integrated PT-100, step two on the TFBT screen).
- Press 'next'.

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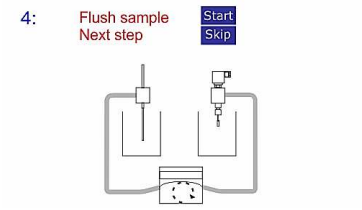
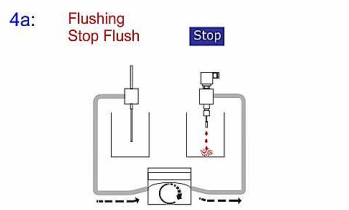
TFBT for ASTM D2068 / IP 387

Start the Test





- Flush the system through with the sample by allowing approximately 20 mL of the sample to flow into the receiver beaker (step 4 on the TFBT screen).
- Stop the pump (step 4a on the TFBT screen).
- Discard any fuel from the fuel receiver beaker.

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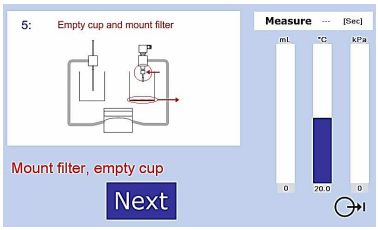
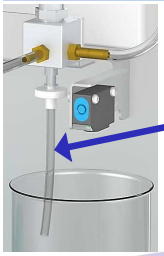
TFBT for ASTM D2068 / IP 387

Mount Filter and Anti-splash Tubing





- Mount filter to the adapter (picture shows filter for method B).
- Mount anti-splash tubing.
- Press ‘next’.

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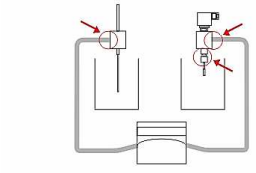
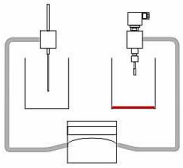
TFBT for ASTM D2068 / IP 387

Check Pressure

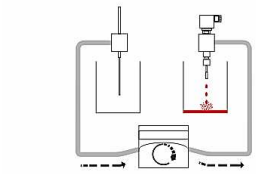
6: Check connections



7b: Start pressure in range?



7a: Initial pressure build-up



- Check connections and press start (step 6).
- TFBT will pump for 20 seconds (step 7a).
- Pressure should be recorded, which should be within the range from 7 to 40 kPa. If this is not the case, check the apparatus for faults.
- If pressure is okay, please press 'next'.

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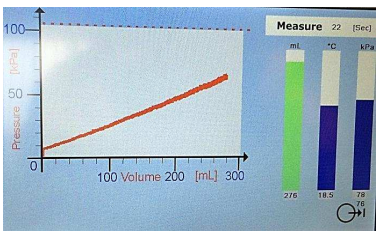
FBT Test



- The remaining sample is drawn from the integral fuel reservoir by the pump.
- A pulse damper provides smooth and continuous flow. The pressure and temperature of the fuel are continuously monitored, while it is pumped through the specified filter into the waste container.
- TFBT shows real-time graph of pressure built up on the touch screen.

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TFBT for ASTM D2068 / IP 387

FBT Calculation

300 mL Passed	
kPa	FBT
20	1.017979
40	1.070105
60	1.151751
80	1.257179
100	1.380952
105	1.414214

When 300 mL of sample is pumped at a pressure below 105 kPa, the following equation is used:

$$FBT = \sqrt{(1 + (\frac{P}{105})^2)}$$

P = maximum pressure reading obtained for 300 mL of fuel to pass through the filter, in kPa.

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TFBT for ASTM D2068 / IP 387

FBT Calculation

Overpressure	
ml	FBT
10	30.01666
20	15.0333
30	10.04988
40	7.566373
50	6.082763
100	3.162278
150	2.236068
200	1.802776
250	1.56205
300	1.414214

Or if maximum pressure is exceeded the following equation applies when the test has been discontinued as the pressure exceeded 105 kPa:

$$FBT = \sqrt{(1 + (\frac{300}{v})^2)}$$

v = volume of fuel in millilitres, passed prior to the pressure rising to 105 kPa.

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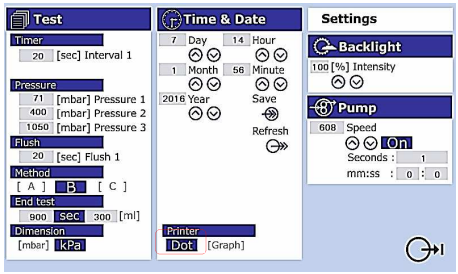
TFBT for ASTM D2068 / IP 387

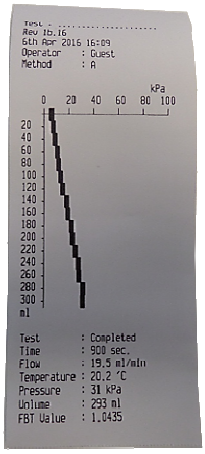
Result



- TFBT automatically calculates the FBT depending on test result.
- Result is shown on display (optional is result in mBar as shown in the picture, standard is in kPa).
- Standard integrated printer can print the result.

Set-up for ASTM D2068 / IP 387

Print-out



- Test result is displayed on screen and can be printed out, multiple copies if required.
- In the menu, different parameters can be set for the test and calibration of the temperature, pressure, pump speed, and level sensor.
- The display provides the operator with test procedure information. Following test results are printed:
  - Date and time, username, method (A or B),
  - Test result, time, flow (calculated), sample temperature, pressure, volume, and FBT value.

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Set-up for ASTM D2068 / IP 387

Recommended TFBT Set-up

- Tamson recommends procedure ‘B’:
  - Less risk for human error.
  - Better precision in ILS.
- Necessary part numbers:
  - TFBT (P/N 00T0942).
  - Adapter block with adapter for method B (P/N 15T0003).
  - Box with 98 method ‘B’ filters (P/N 24T0067).

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Set-up for ASTM D2068 / IP 387

Alternative TFBT Method ‘A’

- Necessary part numbers for procedure ‘A’:
  - TFBT (P/N 00T0942).
  - Adapter block with Millipore stainless steel housing (P/N 15T0002).
  - Box with 98 method ‘A’ filters P/N (24T0064).

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Set-up for ASTM D2068 / IP 387

Recommended Consumables

- 400 mL beaker (P/N 31T2002). Six pieces.
- Tygon Hose 15m (P/N 24T0052).
- Printer paper, five rolls (P/N 28T7035).
- Verification fluid procedure B (P/N 25T2230)
- Anti-splash tubing 1m (P/N 24T0049).

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Set-up for ASTM D2068 / IP 387

Option

- Level and pressure calibration kit:
  - Volume scale 10 mL
  - Pressure resolution 1 mBar
  - Works certificate (pressure readout)

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TFBT

Unique Selling Points against Competition

- ✓ Single voltage from 85-230V, 50-60Hz.
- ✓ Excellent pump regulation guarantees a perfect constant flow. The flow is independent of the pump's counter pressure.
- ✓ Small dimensions, portable (suitcase model on request).
- ✓ Equipped with a graphical touch screen.

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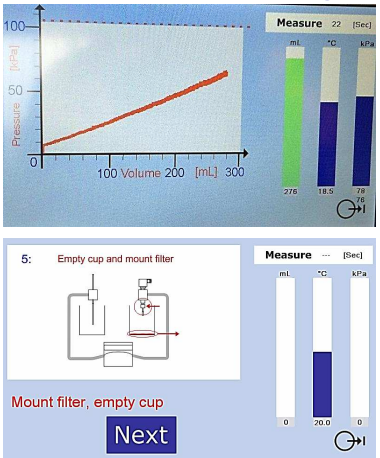
TFBT

Unique Selling Points against Competition



- ✓ Real-time curve is shown (PC not required to view).
- ✓ Visually guided test using step-by-step instruction graphs.
- ✓ Equipped with integrated printer.

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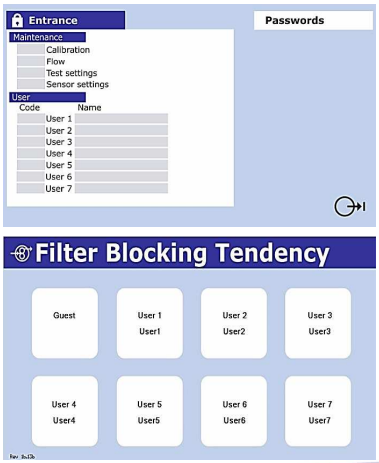
TFBT

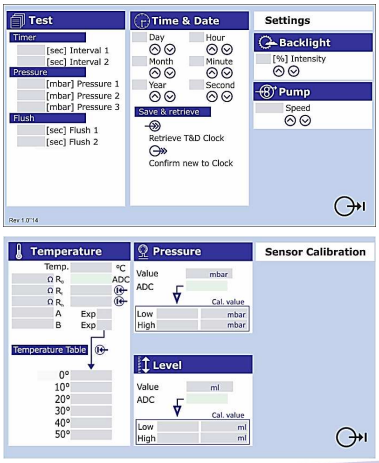
Unique Selling Points against Competition



- ✓ Password protection:
  - ✓ Service screen for calibration,
  - ✓ Service screen for test parameters, seven users and one guest,
  - ✓ If passwords protection is not required it can be switched off.
- ✓ Presettable user names.

TFBT

Unique Selling Points against Competition



- ✓ Touch screen is used to select how to perform the test, set the test parameters and calibrate the sensors.
- ✓ Service screen checks all sensors.
- ✓ Fully electronic calibration.
- ✓ Temperature calibration traceable to IEC 751.
- ✓ PT100 can be replaced and calibrated using standard 1/10 DIN and an IEC 751 certificate.

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TFBT

Unique Selling Points towards Competition

```
Tamson FBT Service valuesRev 1b.
13b - software
***    Temperature      ***
R0          : 100.00 Ohm
R1          : 47.49 Ohm
Rh          : 190.94 Ohm
A           : 3.9080 E-3
B           : -5.8020 E-7
0 degree  C : 7139713
10 degree C : 7418319
20 degree C : 7696096
30 degree C : 7973045
40 degree C : 8249165
50 degree C : 8524457

***    Pressure       ***
Cal Low     : 0 mBar
ADC Low     : 7
Cal High    : 1200 mBar
ADC High    : 1519

***    Level          ***
Cal low     : 0 mBar
ADC low     : 783
Cal high    : 300 mBar
ADC high    : 49

***    Pump           ***
Speed pump  : 624

***    General        ***
Graph       : Line
Interval    : 20 sec
Test method : A
P-Over value : 1050 mBar
Testresult  : kPa

***    End of test    ***
Ending time  : 900sec
Ending level : 300ml
Ending method: Time
```

- ✓ Resolution of temperature (± 0.05°C), pressure (non- linearity = 0.5%), flow (± 0.5 ml) and timer (±0.001 sec).
- ✓ Print-out of calibration data.
- ✓ Service screen to monitor sensor and pump speed.
- ✓ Integrated stopwatch / timer.

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Cold Soak Filtration Test of Biodiesel

To Simulate Winter Behaviour

- The method combines a cold soak step (cooling the sample), and a subsequent filterability step, to determine filter blocking tendency.
- The combination of two test methods provides manufacturers, fuel blenders and suppliers with a means of checking operability for both B100 FAME materials and B5 or any other BX blended diesel fuels.

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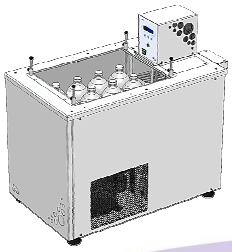
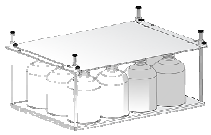
Cold Soak Filtration Test of Biodiesel

Cold Soak Test Requirements

Method	Cold Soak	Heating of Sample
IP PM EA, CEN N 403	+5°C for 16 hours	20°C for 2 hours
ASTM D7501	+4.5°C for 16 hours	25°C for 2 or 4 hours
CGSB	+1°C for 16 hours	25°C for 2 hours





- ✓ Tamson TLB50 to keep samples at +4.5°C for 16 hours and heat it up to +25°C for 2 or 4 hours
- ✓ Position for 12 x 500 mL jars
- ✓ Smart rack to position flasks
- ✓ Free Tamcom software plus labtop can regulate TLB50 to follow a presetted temperature curve (e.g. 16 hours @ 5°C and than 2 hours @ 20°C.

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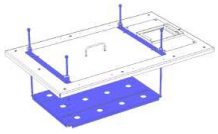
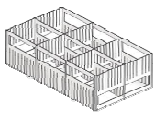
Cold Soak Filtration Test of Biodiesel

ASTM D7501 Set-up





- ✓ TLB50 bath:
  - ✓ 230V/50 Hz (P/N 00T0072),
  - ✓ 115V/60 Hz (P/N 00T0071),
  - ✓ 230V/60 Hz (P/N 00T0073).
- ✓ Adjustable leveling platform TLB50 (P/N 03T0071).
- ✓ 2 * Rack for 500 mL jars (P/N 03T1049).
- ✓ 4 * Bottle bracket to prevent floating (P/N 03T1041).
- ✓ 4 * Rail to hold bottle bracket (P/N 03T1040).

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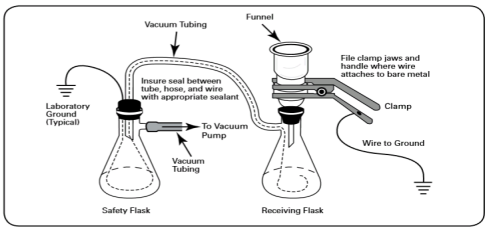
Cold Soak Filtration Test of Biodiesel

ASTM D7501 Set-up



- ✓ Complete manual filtration set (P/N 31T2000) as per ASTM method.
- ✓ Vacuum pump P/N 11T0031 (230V) or P/N 11T0032 (115V).
- ✓ Rod stand with clamps (P/N 13T8044). Two pieces recommended.
- ✓ Please see our special specification sheet for ASTM D7501 which can be downloaded from our website www.tamson.com.

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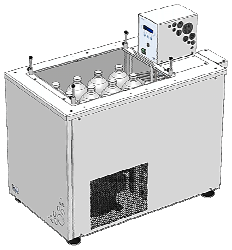
Cold Soak Filtration Test of Biodiesel

ASTM D7501 Alternative Recommendation



TLB50 for cold soak!!

TFBT for Filtration Test!!

- ✓ Please note that manual filtration set-up (P/N 31T2000) can be replaced by the TFBT procedure 'B' for the energy institute method: IP PM EA.
- ✓ Tamson also recommends to use our TFBT instead of the manual filtration set-up for reliable, easy and better filter blocking tendency tests.

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The end

Thank you for you time and consideration!