



## prepASH<sup>®</sup> Series 340 Volatiles

Moisture, volatile matter, ash and fixed carbon can be analysed with prepASH219/229 following the ASTM D5142 in one working step.

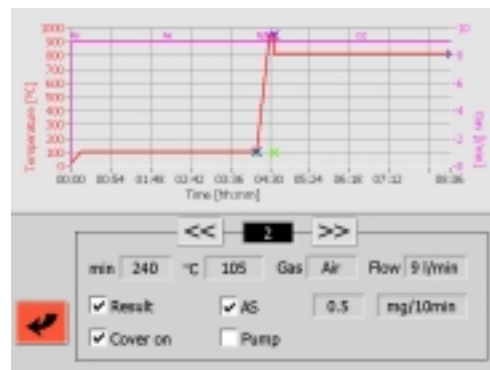
### prepASH Series 340 as a solution

The Series 340 is about fully automatic stand-alone devices of the second generation of thermogravimetric analysers to determine solid contents and ash of substances, highly sophisticated, and at the same time, very simple to operate, an intelligent combination of drying chamber, muffle furnace, precision balance and evaluation centre in one unit. The prepASH 229 e.g. is able to analyse up to 29 samples at once, allowing the user to program and run most complex, virtually unbounded procedures, in a temperature range beginning at +50°C up to +1000°C, optionally with various process gases.

### Eliminate potential for error – increase precision and reproducibility

Manual steps are the most common cause for inaccuracies. Those are reduced to a minimum by the new prepASH generation 340, because sample transfers to the drying chamber, the muffle furnace or the exsiccator are no longer needed. The samples are permanently weighed during the entire process and a compensation of the buoyancy ensures the measuring accuracy.

The basic system of the device that satisfies the most reference prescriptions, allows method-conform working under optimised analytic preconditions – with the effect of considerably enhanced reproducibility and comparability. With the optional control of process gases (oxygen, nitrogen, air), it is possible to treat even critical samples under definable conditions.



### Full control and documentation

Devices of the prepASH Series 340 do not need a PC to operate. During all program steps they permanently record the weight loss of every single sample to show it graphically on a colour touchscreen display. Furthermore, it is possible to embed all models via ethernet interface into a network and monitor them by the prepDATA software on a PC. All measuring data are saved in the device and the software can copy them on a memory stick or any PC which is part of the network.



### Saving time

Once the samples are weighed in, the device is able to perform its task autonomously. No more additional weighing, pre-incinerating, feeding and extraction of samples by hand delays the evaluation. The final result is at disposal in **up to 80% less time!**

### Content of this Applicaton:

1. prepASH as a solution for the ash, moisture and volatile content determination
2. General information on chemical properties of coal
3. ASTM D 5142 Standard Test Methods for Proximate Analysis
4. ASTM D5142 proximate analysis with prepASH (Practice)



## General information on chemical properties of coal

[http://en.wikipedia.org/wiki/Coal\\_assay](http://en.wikipedia.org/wiki/Coal_assay)

Coal comes in four main types or *ranks*: lignite or brown coal, bituminous coal or black coal, anthracite and graphite. Each type of coal has a certain set of physical parameters, which are mostly controlled by moisture, volatile content (in terms of aliphatic or aromatic hydrocarbons) and carbon content.

### 1. Moisture

Moisture is an important property of coal, as all coals are mined wet. Groundwater and other extraneous moisture is known as adventitious moisture and is readily evaporated. Moisture held within the coal itself is known as inherent moisture and is analysed.

### 2. Volatile matter

Volatile matter in coal refers to the components of coal, except for moisture, which are liberated at high temperature in the absence of air. This is usually a mixture of short and long chain hydrocarbons, aromatic hydrocarbons and some sulfur. The volatile matter of coal is determined under rigidly controlled standards. In Australian and British laboratories this involves heating the coal sample to  $900 \pm 5$  °C ( $1650 \pm 10$  °F) for 7 minutes in a cylindrical silica crucible in a muffle furnace. American Standard procedures involve heating to  $950 \pm 25$  °C ( $1740 \pm 45$  °F) in a vertical platinum crucible. These two methods give different results and thus the method used must be stated.

### 3. Ash

Ash content of coal is the non-combustible residue left after coal is burnt. It represents the bulk mineral matter after carbon, oxygen, sulfur and water (including from clays) has been driven off during combustion. Analysis is fairly straightforward, with the coal thoroughly burnt and the ash material expressed as a percentage of the original weight.

### 4. Fixed carbon

The fixed carbon content of the coal is the carbon found in the material which is left after volatile materials are driven off. This differs from the ultimate carbon content of the coal because some carbon is lost in hydrocarbons with the volatiles. Fixed carbon is used as an estimate of the amount of coke that will be yielded from a sample of coal. Fixed carbon is determined by removing the mass of volatiles determined by the volatility test, above, from the original mass of the coal sample.

### 5. Chemical analysis

Coal is also assayed for oxygen content, hydrogen content and sulfur. Sulphur is also analysed to determine whether it is a sulfide mineral or in a sulfate form. This is achieved by dissolution of the sulfates in hydrochloric acid and precipitation as barium sulfate. Sulfide content is determined by measurement of iron content, as this will determine the amount of sulfur present as iron pyrite.

Carbonate minerals are analysed similarly, by measurement of the amount of carbon dioxide emitted when the coal is treated with hydrochloric acid. Calcium is analysed. The carbonate content is necessary to determine the combustible carbon content and incombustible (carbonate carbon) content.

Chlorine, phosphorus and iron are also determined to characterise the coal's suitability for steel manufacture.

An analysis of coal ash may also be carried out to determine not only the composition of coal ash, but also to determine the levels at which trace elements occur in ash. These data are useful for environmental impact modelling, and may be obtained by spectroscopic methods such as ICP-OES or AAS

## 2. ASTM D 5142 Standard Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures

**ASTM D 5142 Document Information: Publication Date: Feb 1, 2009**

### Scope:

These instrumental test methods cover the determination of moisture, volatile matter, and ash, and the calculation of fixed carbon in the analysis of coal and coke samples prepared in accordance with Method D 2013 and Practice D 346. Results obtained through the use of the instrumental tests have been shown to differ from those obtained with Test Methods D 3173, D 3174, and D 3175 on some coals and cokes. Where a relative bias between the instrumental methods and Test Methods D 3173, D 3174, and D 3175 for proximate analysis of coal and coke are shown to exist, the instrumental results shall be corrected or the instrument calibrated using samples of known proximate analysis. Test Methods D 3173, D 3174, and D 3175 shall be considered the referee test methods. The instrumental test methods are not applicable to thermogravimetric analyzers using microgram size samples.

The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.



### 3. ASTM D5142 proximate analysis with prepASH

- Moisture, volatile matter, ash and fixed carbon can be analysed with prepASH219/229 following the ASTM D5142 in one working step.
- The needed heating steps in the correct atmosphere including the calculation of the four results are fixed programmed. Time and temperature of the steps can be adapted by the supervisor to the needs of samples and regulations.

#### STANDARD-METHODE

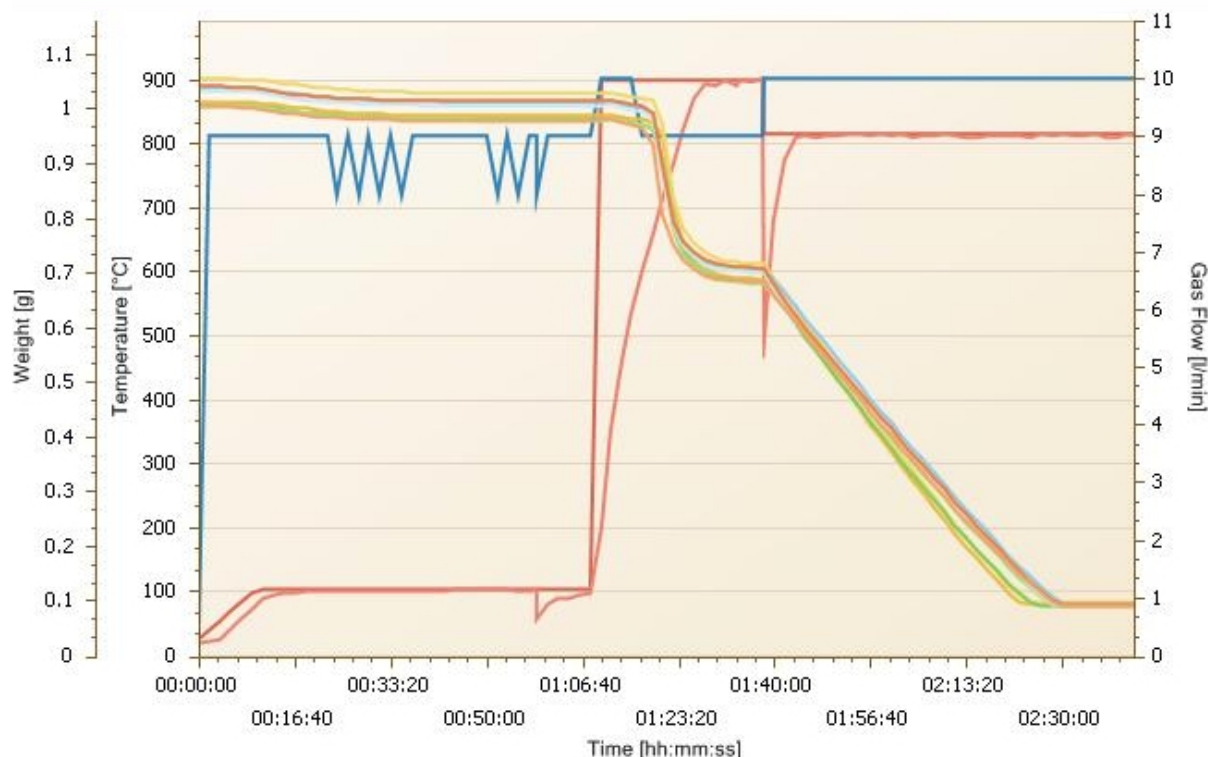
Method: ASTM#3.log

Step	Temp 1 [°C]	Temp 2 [°C]	Gas	Gas Flow [l/min]	Time [min]	Auto Stop [mg/min]	Manual Stop	Result
1	25	105	Air	9	10			
2	105	105	Air	9	180	0.5 mg/10	On	Moisture [%] (A)
3	105	900	N2	9	1			
4	900	900	N2	9	7		On	Volatile [%] (B)
5	900	815	O2	9	480	0.5 mg/10		Ash [%] (C)

- The user is guided through the needed manual steps as weighing in, cover and uncover the crucibles.
- prepASH can be calibrated with reference material of known volatile matter.
- Reference coal and Calibration

Reference material is used to calibrate the methods for the determination of volatile matter content of samples. prepASH® is delivered with a standard method and pre-calibrated with 3 standards. Customers calibration: the reference materials must cover the full range of expected volatile matter contents of the coal and coke samples that will routinely be analyzed.

#### Graphic presentation of the analysis





**Result:**

Group	Pos.	Sample	ID	A: Mois[%]		B: Vola[%]		C: Ash[%]		D: Carb[%]
				Tare [g]	Weight [g]	Result	Weight [g]	Calc.	Time	
1	1	1	-	33.2620	1.0126	A	0.9871	2.518	00:55	
						B	0.6559	32.711	01:37	
						C	0.0934	9.224	02:34	
						D		55.547	00:00	
1	2	1	-	30.0925	1.0550	A	1.0285	2.512	00:54	
						B	0.6838	32.677	01:37	
						C	0.0968	9.175	02:34	
						D		55.636	00:00	
1	3	1	-	27.2393	1.0058	A	0.9802	2.545	00:52	
						B	0.6520	32.630	01:37	
						C	0.0924	9.187	02:36	
						D		55.638	00:00	
1	4	1	-	27.0490	1.0048	A	0.9792	2.548	00:52	
						B	0.6494	32.824	01:37	
						C	0.0918	9.136	02:40	
						D		55.492	00:00	
2	5	2	-	23.3289	1.0401	A	1.0125	2.654	00:58	
						B	0.6733	32.608	01:37	
						C	0.0948	9.115	02:42	
						D		55.624	00:00	
2	6	2	-	29.9311	1.0324	A	1.0053	2.625	00:53	
						B	0.6690	32.576	01:37	
						C	0.0940	9.105	02:40	
						D		55.694	00:00	
2	7	2	-	29.4264	1.0415	A	1.0141	2.631	00:53	
						B	0.6759	32.471	01:37	
						C	0.0953	9.150	02:40	
						D		55.748	00:00	
2	8	2	-	27.0839	1.0043	A	0.9777	2.649	00:53	
						B	0.6522	32.415	01:37	
						C	0.0920	9.161	02:41	
						D		55.776	00:00	

**Statistic:**

Group: 1	A: Mois[%]		B: Vola[%]		C: Ash[%]		D: Carb[%]
	A-Result	A-Calc.	B-Result	B-Calc.	C-Result	C-Calc.	D-Calc.
mean	0.99375	2.53078	0.66028	32.71045	0.09360	9.18050	55.57825
std	0.02343	0.01838	0.01591	0.08248	0.00223	0.03610	0.07127
rstd	2.35788	0.72631	2.40948	0.25217	2.38577	0.39328	0.12823
n	4	4	4	4	4	4	4

Group: 2	A: Mois[%]		B: Vola[%]		C: Ash[%]		D: Carb[%]
	A-Result	A-Calc.	B-Result	B-Calc.	C-Result	C-Calc.	D-Calc.
mean	1.00240	2.63950	0.66760	32.51735	0.09403	9.13260	55.71055
std	0.01691	0.01375	0.01065	0.09013	0.00145	0.02700	0.06700
rstd	1.68651	0.52110	1.59581	0.27719	1.54459	0.29562	0.12026
n	4	4	4	4	4	4	4



**prepASH: Equipment:**

Moisture and volatile matters are determined under protection nitrogen atmosphere. The ashing step is done under oxidising atmosphere with oxygen. Therefore **prepASH 219/229** has to be equipped with the **process gas flow unit (340-8502)**.

For the volatile matter step the **crucibles (340-8032, set of 5 pieces(340-4024))** have to be covered with a lid (**340-8033, set of 5 pieces(340-4025)**).

**Set “Volatiles”**

Free of charge we supply prepASH® with Al<sub>2</sub>O<sub>3</sub> crucibles with covers, tweezers and steel turn table for the crucibles.