Application Bulletin

Of interest to:	General analytical chemistry, automotive, lacquer and paints	

Iodine adsorption number of carbon black according to ASTM D 1510

General information

ProcessLab is a robust industrial analyzer that incorporates all needed equipment in a sealed case. It comes ready to use – including an industrial PC and operating unit as well as all needed analytical instruments. After the user has placed the sample(s) and pressed the start button, the system autonomously carries out the calibration and sample determination(s). Detailed results are available for export via corporate ethernet or analog output lines.

The ProcessLab setup described here carries out a fully automated determination of the iodine adsorption number (IAN) of a carbon black using a sample processor. That allows many samples to be analyzed in one single run. The setup includes the needed components for all measurements and, thanks to its flexibility, is able to meet specific demands on site.

Analyzer features

- Sealed and robust analyzer – ideal for harsh process environments
- Instruments not affected by carbon black due to sealed concept
- Automated and autonomous operation
- Easy and quick to control
- Reliable and established Metrohm technology
- Comprehensive communication possibilities (ethernet, RS232, digital/analog in- and outputs)
- Method according to ASTM D 1510
- Analytical determination according to ISO 1304

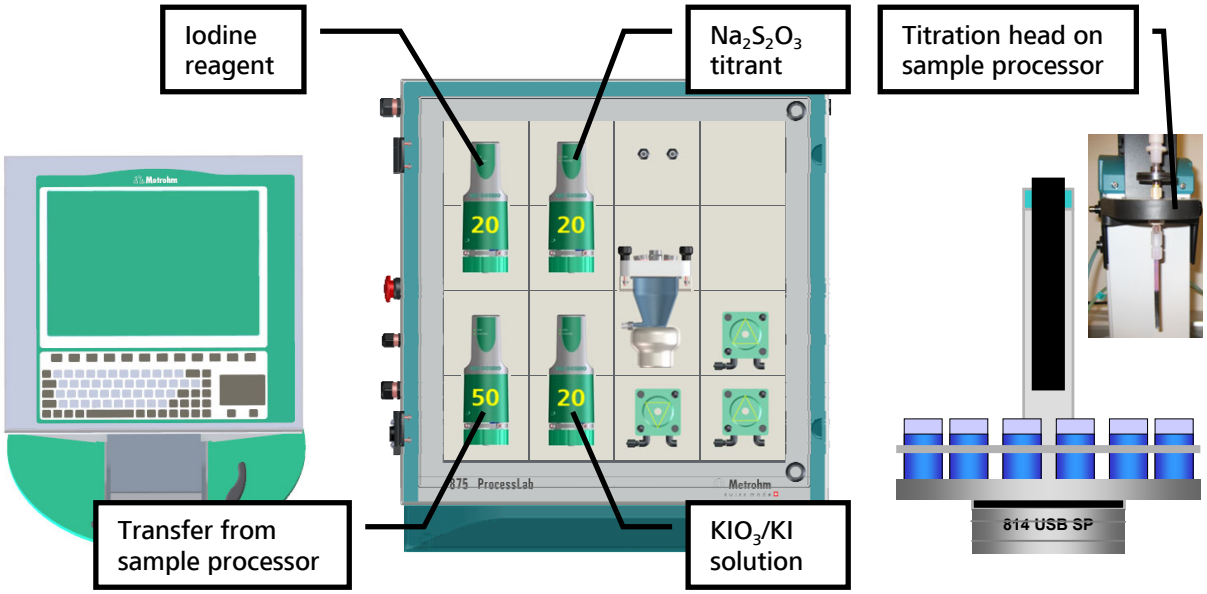
This system is very flexible and can be adapted very easily to any specific needs. If necessary an additional reagent cabinet is available and ProcessLab can be placed on top of it – this means sufficient space for all reagents, which makes ProcessLab even more practical.

Sigificance and use

The iodine adsorption number is related to the surface area of a carbon black and therefore a measure for the quality of this base material. In the presence of high contents of volatile or solvent-extractable substances it substantially decreases, thus allowing to draw conclusions concerning the suitability for a certain production process. The quality mainly depends upon the production conditions, ageing also has an effect on the iodine adsorption number (IAN).

As carbon black is used as a filler material in the production of rubber and plastics and also as a pigment in colours it involves a certain IAN in order to reach and maintain the physical properties of the end product. For example the lifetime of a car tire dramatically increases as carbon black substantially improves its abrasion resistance and stability.

Wet part setup & system overview



Method details

The addition of iodine to a sample of carbon black results in an adsorption of the iodine on the (available) surface of the sample. After a certain reaction time the remaining (unadsorbed) iodine is separated using a 5 µm filter. The subsequent titration of the excess allows to calculate the amount of adsorbed iodine. The more iodine is adsorbed on the surface of the carbon black the less iodine can be found in the remaining liquid phase. The principle of a back titration is used for this measurement.

Please note that the adsorption reaction follows certain equilibration rules. It is necessary to maintain a particular ratio of volume of iodine to mass of sample size as specified in ASTM D 1510.

Reagents

- Iodine reagent, $c(I_2)$ = 0.0473 N (containing 5 mL pentanol/liter)
- Sodium thiosulphate titrant, $c(Na_2S_2O_3)$ = 0.05 N
- Potassium iodate/iodide solution, $c(KIO_3)$ = 0.05 N
- Sulphuric acid, $w(H_2SO_4)$ = 20%
- Pentanol
- Demineralized water

Analytical procedures

Titer of sodium thiosulphate titrant
15 mL of KIO_3/KI reagent are dosed into the determination vessel. After the addition of 50 mL of deionized water, add 3 mL 10% H_2SO_4 to acidify the solution. Then the titration is carried out using sodium thiosulphate as titrant. The titer is calculated at the end of the determination.

Concentration of the iodine reagent
15 mL of iodine reagent are added to the vessel and titrated using the sodium thiosulphate solution. The concentration of iodine may then be calculated from the known concentration of the sodium thiosulphate solution.

Blank value (total consumption of thiosulphate for total iodine)
50 mL iodine reagent are added to the titration vessel on the sample processor and stirred for 180 seconds. After the stirrer is stopped, let the solution rest for 30 seconds before 10 mL are transferred through the 5 µm filter to the determination vessel and titrated using the thiosulphate titrant.

Sample analysis (consumption of thiosulphate for iodine excess)
The corresponding amount* of sample is weighed into a beaker from the sample processor. Then follow the same steps as stated in the determination for the blank value.

* Table for determining the correct sample size

Expected/nominal iodine adsorption number **g/kg**	Sample size **mg**	Volume iodine/sample size ratio **mL/g**
0 to 130.9	500	50:1
131.0 to 280.9	250	100.1
281.0 to 520.9	125	200:1
521.0 and above	62.5	400:1

Reactions

$IO_3^- + 5\ I^- + 6\ H^+ \rightarrow 3\ I_2 + 3\ H_2O$ Titer determination using KIO_3/KI solution

$I_2 + 2\ S_2O_3^{2-} \rightarrow 2\ I^- + S_4O_6^{2-}$ Excess iodine titration for titer, blank and sample

Accessories used

- 1 x 2.875.0510; 875 ProcessLab Base Unit L Touch
- 1 x 6.7201.200; ProcessLab MDM Controller with 1 measuring amplifier
- 4 x 2.800.0010; 800 Dosino
- 3 x 6.3032.220; Buret 20 mL
- 1 x 6.3032.250; Buret 50 mL
- 1 x 6.7205.010; Peristaltic pump, 40 mL/min
- 2 x 6.7205.030; Peristaltic pump, 320 mL/min
- 1 x 2.814.0030; USB Sample processor with 1 tower
- Sartorius filter, disposable; #17594 K 5 µm with luer lock
- 1 x Sample rack as needed
- Reagent containers if needed (5, 10 and 20 L), incl. level switch

Results

Sample No.	Titer determination sodium thiosulphate	Content determination of the iodine reagent	Blank of iodine reagent	Carbon black sample
1	1.0142	0.04846 mol/L	11.954 mL	124.04 g/kg
2	1.0135	0.04845 mol/L	11.950 mL	123.72 g/kg
3	1.0116	0.04837 mol/L	11.949 mL	123.29 g/kg
Mean	**1.0131**	**0.04843 mol/L**	**11.951 mL**	**123.68 g/kg**
s (abs.)	*0.0013*	*0.00005 mol/L*	*0.003 mL*	*0.38 g/kg*
s (rel.)	*0.13 %*	*0.10 %*	*0.02 %*	*0.30 %*

Result discussions

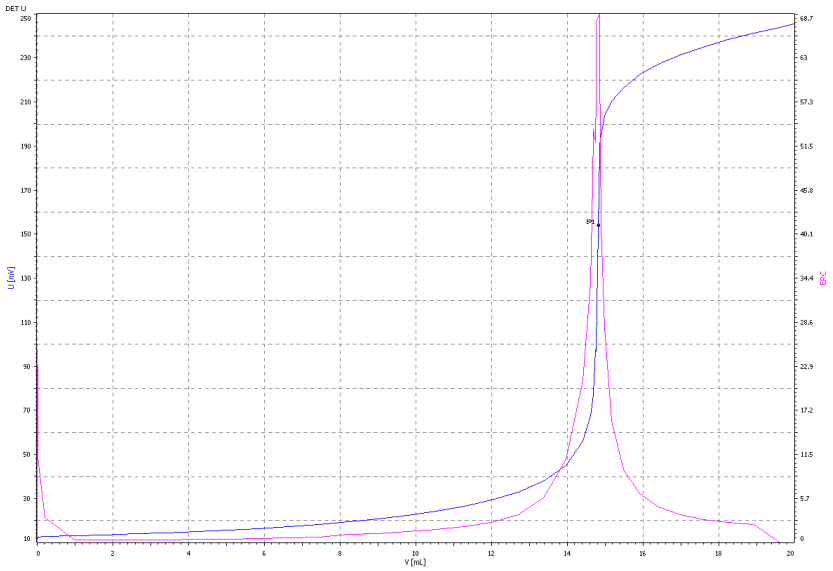
- The results show a very good repeatability and high accuracy
- The values were obtained under lab conditions

Details to be considered

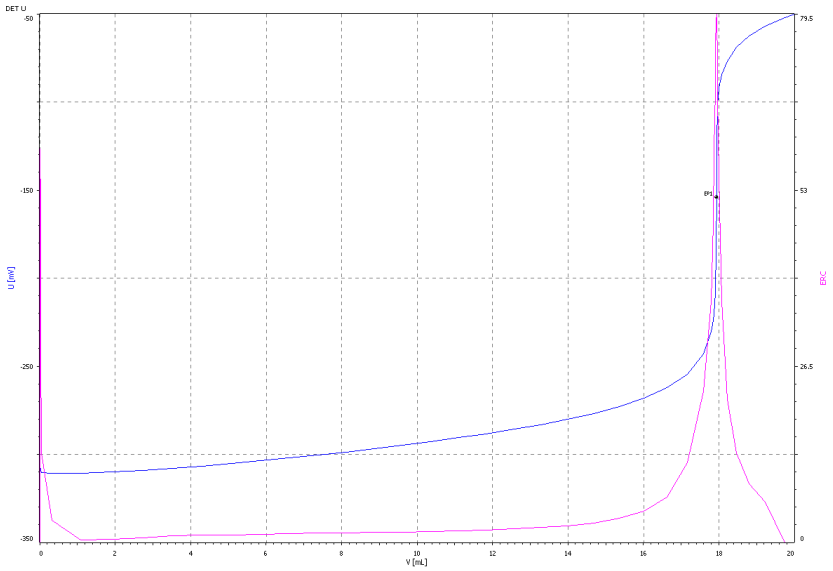
- The iodine adsorption reaction is an equilibrium reaction (also on the filter) that partially depends on particle size and reaction time in the vessel. These conditions need to be adjusted properly in order to obtain constant results. Every new filter needs to be saturated with iodine reagent before use – this is achieved by carrying out three blank determinations for system conditioning.
- A settling time of 30 seconds for the carbon black increases the lifetime of the filter (clogging). Please note that the filter should be changed regularly. It is recommended to do this on a daily base (50 samples/day), depending on the throughput the filter has to be changed more often.
- Keep tubing connections as short as possible and reduce dosing speeds to avoid bubble formation

Practical examples

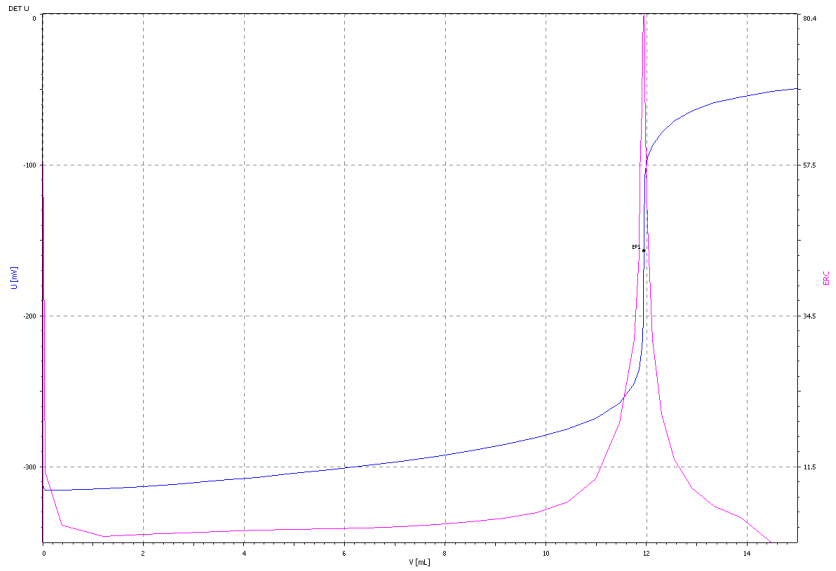
Titer determination of sodium thiosulphate titrant using KIO_3/KI solution



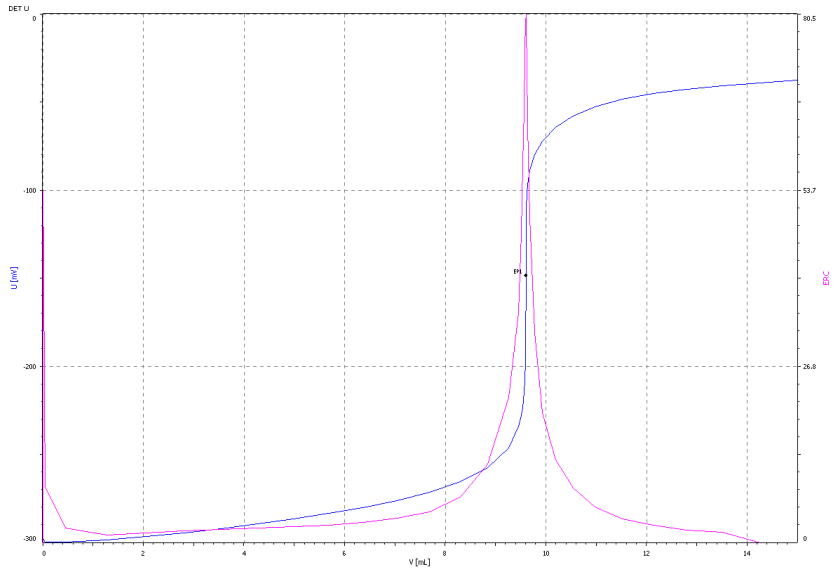
Content determination of the iodine reagent using sodium thiosulphate titrant



Blank value of the pure iodine reagent (without sample)



Analysis of carbon black sample (500mg)



References

ASTM D 1510 – 08c; Standard Test Method for Carbon Black – Iodine Adsorption Number

ISO 1304 (fourth edition, 2006-02-15); Rubber compounding ingredients – Carbon black – Determination of iodine adsorption number

ICBA; International Carbon Black Association – http://www.carbon-black.org

Donnet, Jean-Baptiste, Carbon black, Hardcover book second edition, Publisher: CRC, ISBN-13: 978-0824789756