

#### SCOPE

The measurement of sulfur and chlorine in activated carbon is demonstrated.

#### BACKGROUND

Activated carbon, also called activated charcoal, has many uses as a filter medium and absorbent, including water and gas purification, decaffeination, gas mask air filters, gold processing, metal extraction and sewage treatment. Sulfur and chlorine may occur naturally in charcoal and in the production and regeneration of activated carbon various solvents may be used, including sulfur-based and chlorine-based solvents. As the material is processed the sulfur and chlorine levels are monitored until they reach an acceptable content. EDXRF offers a fast and simple low cost method of measuring the sulfur and chlorine content with little or no sample preparation and relatively short analysis times. The NEX QC<sup>+</sup> is a self-contained touch screen analyzer ideal for use by non-technical operators throughout the quality control of activated carbon processing and recycling.



#### INSTRUMENTATION

<b>Model:</b>	Rigaku NEX QC <sup>+</sup>
<b>X-ray tube:</b>	4W Ag-anode
<b>Detector:</b>	Semiconductor
<b>Sample Type:</b>	Powder
<b>Film:</b>	Mylar 6um
<b>Analysis Time:</b>	300 sec
<b>Environment:</b>	Air
<b>Sample Prep:</b>	Grinder or mill Manual Compaction Press
<b>Optional:</b>	Autosampler



#### SAMPLE PREPARATION

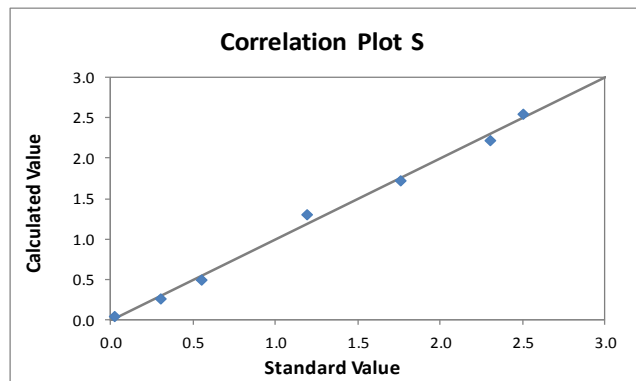
Samples are ground to a homogeneous medium powder of approximately 200 mesh (~75um particle size). Approximately 5g of material is transferred to a standard 32mm XRF sample cup and compacted to 250 inch-pounds torque using the Rigaku manual Compaction Press.

## CALIBRATION – Broad Range

Seven calibration standards assayed for S and Cl were used to generate empirical calibrations. Fe was also measured to enable correction factors which automatically compensate for its absorption/enhancement effects on the S and Cl X-rays.

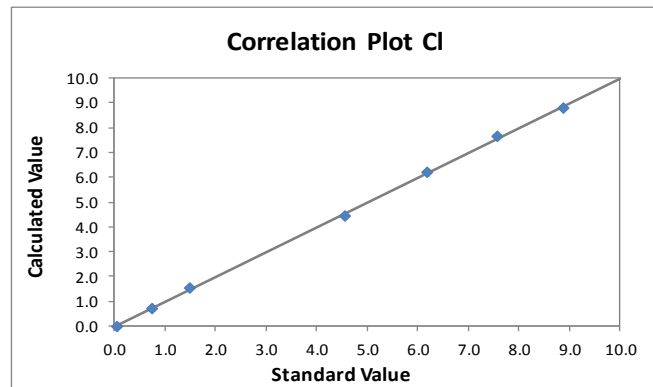
### S Calibration

Element: S		SEE: 0.068 %
Units: %		Correlation: 0.99525
Sample I.D.	Standard Value	Calculated Value
0	0.02	0.049
1	2.50	2.546
2	0.30	0.260
3	1.19	1.305
4	2.30	2.220
5	0.55	0.500
6	1.76	1.729



### Cl Calibration

Element: Cl		SEE: 0.070 %
Units: %		Correlation: 0.99960
Sample I.D.	Standard Value	Calculated Value
0	0.03	0.011
1	8.87	8.822
2	0.75	0.745
3	4.55	4.435
4	7.57	7.654
5	1.49	1.564
6	6.19	6.219



Aside from S and Cl a significant amount of Fe is also present in the samples. The Fe is measured in order to enable an “alpha” correction to compensate for its effects on the S and Cl X-rays. The calibration model can be optimized by adding a few more standards to the set in order to ensure enough degrees of freedom to enable alpha corrections for other elements present, such as P, K, Ca, Ti and Ni.

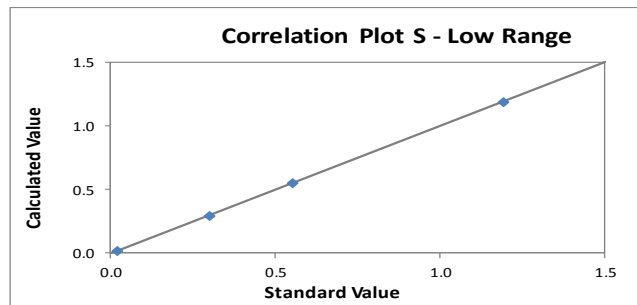
## SPLIT CALIBRATIONS

Calibration accuracy depends mainly on the goodness of the set of calibration standards. Accuracy can also be further optimized by splitting the range into a low range and a high range using two separate calibrations. In this way each range can be optimized as a separate calibration. For very low ranges of S and Cl it is recommend to make the powder sample as a hydraulically pressed pellet and use helium purge for the ultra-low levels.

As an example, shown here is low range S calibration when split from the broad range. As with the broad range, adding a few extra standards to the low and high range calibrations will best define the X-ray absorption effects creating an optimum model for the material.

### S Calibration for Low Range

Element: S SEE: 0.006 %		
Units: % Correlation: 0.99986		
Sample I.D.	Standard Value	Calculated Value
0	0.02	0.023
2	0.30	0.292
3	1.19	1.189
5	0.55	0.556



## REPEATABILITY (Precision)

To demonstrate recovery and repeatability, a low and a high calibration standard were selected and measured against the broad range calibration. Each was measured in 10 repeat analyses using an analysis time of 180 sec per measurement without moving the sample between measurements to determine the average and standard deviation (Std Dev) for precision.

Sample ID: Standard 1 Units mass%				
Element	Standard Value	NEX QC+ Average Value	Std Dev	% Relative
S	2.50	2.542	0.011	0.4
Cl	8.87	8.84	0.021	0.2

Sample ID: Standard 2 Units mass%				
Element	Standard Value	NEX QC+ Average Value	Std Dev	% Relative
S	0.30	0.262	0.002	0.7
Cl	0.75	0.737	0.002	0.3

Precision is mainly due to measurement time and the repeatability of the instrumentation in making the measurement. Both precision and detection limits can be optimized by using longer measurement times.

## DETECTION LIMITS (LLD – Lower Limit of Detection)

In the empirical method, ten repeat analyses of a blank powder sample (Chemplex SpectroBlend or similar) are taken with the sample in static position, and the standard deviation ( $\sigma$ ) is determined. The LLD (Lower Limit of Detection) is then defined as  $3\sigma$ . The total measurement time for the LLDs shown below is 180 sec per analysis (100 sec S & Cl, 80 sec other elements).

Element	LLD
S	0.0036% (36 ppm)
Cl	0.0018% (18 ppm)

Detection limits are dependent on many factors, including measurement time, sample preparation and calibration concentration range. For example, optimum S and Cl detection limits can be achieved using 300 sec measurement time or greater, calibrating only over a low concentration range and making samples as hydraulically pressed pellets.

## CONCLUSION

The NEX QC<sup>+</sup> offers the lab analyst or at-line QC technician a simple and fast tool for measuring sulfur and chlorine in activated carbon. The versatility of the NEX QC<sup>+</sup> EDXRF analyzer also allows for the measurement of other elements in the material, given assays for them in the set of calibration standards.