
BENCH SCALE ANODE EVALUATION OF GREEN COKES USED FOR PREBAKED ANODES

PREPARATION AND TEST METHODS

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1 Summary

A bench scale procedure was developed to manufacture bench scale anodes (Ø 50mm, l=100mm) using an intensive impeller mixer and a bench press.

Batches of dry aggregate are mixed with five different pitch contents in the range of 13% to 17%. After baking in a high temperature furnace, the cylinders are tested according to the ISO standard methods.

Worldwide cokes were tested and a typical range of the anode properties as a function of the pitch content was defined. A precise determination of the pitch requirement and valuable information on the suitability of a given coke for prebaked anodes are provided. The evaluation of the results covers the fields of pitch requirement, of thermal shock anode resistance and of anode consumption related to the burning behaviour of the prebaked anodes.

2 Introduction

R&D Carbon has developed a standard procedure in order to evaluate the potential of coke for the manufacture of anodes used in the aluminium industry.

The first step of the procedure consists of the determination of the main properties of the green coke, its calcination, and the determination of the calcined coke properties.

Based on the properties of the calcined coke, bench scale anodes with five different binder contents, usually between 13% and 17%, will be produced, baked and tested.

The properties of the bench scale anodes will be compared to typical values obtained on bench scale anodes made with worldwide cokes used in the aluminium industry in order to determine the coke quality, especially in terms of pitch requirement, thermal shock resistance and burning behavior.

3 Experimental

3.1 Green Coke Testing

The entire sample is firstly divided in order to produce a 5 kg sample which will be tested in the R&D Carbon laboratory. The properties shown in the table below will be performed.

Pos	Properties	Unit	Method
1	Water Content	%	ISO 11412
2	Volatile Matter	%	ISO 9406
3	Hardgrove Grindability Index	-	ISO 5074
4	Sieving Analysis	%	ISO 12984
5	Elements XRF	%, ppm	ISO 12980
6	Ash Content	%	ISO 8005

Figure 1 : Properties of Green Coke

3.2 Pilot Calcining of Green Coke

The sample of needle coke will be calcined at R&D Carbon pilot plant in two steps. Firstly, the water and volatiles present in the green coke were removed in a pilot rotary kiln (D=10 inches and l of 6 m.) at a temperature of 750°C with load rate of 20 kg/h.



Figure 2: Pilot Rotary kiln

In a second step, the coke will be calcined in a static high temperature furnace in order to reach a real density of 2.07 kg/dm³. The following calcination program was applied:

- Room Temperature to 200°C : 200°C/h
- 200°C to 800°C : 100°C/h
- 800°C to 1125°C : 50°C/h, soaking time at 1125°C of 20 h

3.3 Calcined Coke Testing

The entire sample is firstly divided in order to produce a 5 kg sample which will be tested in the R&D Carbon laboratory. The properties shown in the table below will be performed.

Property	Method	Unit
Water Content	ISO 11412	%
Oil Content	ISO 8723	%
Sieving Analysis	ISO 12984	%
>32 mm		%
> 16 mm		%
16-8 mm		%
8-4 mm		%
4-2 mm		%
2-1 mm		%
1-0.5 mm		%
0.50-0.25 mm		%
<0.25 mm		%
Tapped Bulk Density	ISO 10236	kg/dm ³
8-4 mm		kg/dm ³
4-2 mm		kg/dm ³
2-1 mm		kg/dm ³
1-0.5 mm		kg/dm ³
0.5-0.25 mm		kg/dm ³
Hg Porosimetry	DIN 66133	kg/dm ³
Apparent Density (ø100µm)		kg/dm ³
Apparent Density (ø13µm)		%
Total Porosity		mm ³ /g
Pore Volume		
Grain Stability	ISO 10142	%
Pulverizing Factor	M168	-
Real Density in Xylene	ISO 8004	kg/dm ³
Crystallite Size Lc	ISO 20203	Å
Specific Electrical Resistance	ISO 10143	µΩm
Air Reactivity 525°C	ISO 12982-1	%/min
CO ₂ Reactivity	ISO 12981-1	%
XRF Analysis	ISO 12980	%
S		ppm
V		ppm
Ni		ppm
Si		ppm
Fe		ppm
Al		ppm
Na		ppm
Ca		ppm
P		ppm
Ash Content	ISO 8005	%

Table 1 : Properties of Calcined Coke

3.4 Dry Aggregate Preparation

The entire coke sample will be sieved in a pilot continuous sieving machine and the material larger than 8 mm will be crushed in a jaw crusher and sieved again.

The fines will be prepared out of the intermediate coke fractions 2 to 0 mm by using an air jet collision mill where the dust fineness can be adjusted to a target of 3'500 Blaine corresponding to a $d_{50\%}$ of 50 μm .

Six different fractions will be weighed to form a 4 kg dry aggregate according to the following straight line formulation for the grains:

- | | |
|--------------------------|-----|
| ➤ 8-4 mm: | 14% |
| ➤ 4-2 mm: | 14% |
| ➤ 2-1 mm: | 14% |
| ➤ 1-0.5 mm: | 14% |
| ➤ 0.5-25 μm : | 14% |
| ➤ Fines 3'500 Blaine: | 30% |



Figure 3: Dry Aggregate Preparation: Crusher, Sieving Machine, Air Jet Collision Mill

3.5 Paste Preparation

3.5.1 Binder Characteristics

A modern coal tar pitch showing a relatively low QI content with a typical 113°C Mettler Softening Point (SP) will be used as a binder. The binder was crushed to < 4 mm and will be used in the solid state in the mixing step.

Properties	Method	Unit	Value
Softening Point Mettler	ISO 5940-2	°C	113.0
Viscosity at 160°C	ASTM D5018	cP	1'550
Quinoline Insoluble	ISO 6791	%	8.4
Toluene Insoluble	ISO 6376	%	28.0
Real Density in Helium	ISO 21687	kg/dm ³	1.31
Ash Content	ISO 8006	%	0.25
Elements XRF	S	%	0.47
	Na	ppm	202
	Ca	ppm	59
	Si	ppm	106
	Fe	ppm	377
	Zn	ppm	311
	Pb	ppm	232

Table 2 : Binder Properties

3.5.2 Mixing

For each of the five pitch content levels, 2 x 4 kg portions will be prepared. The dry aggregates portions will be placed into steel containers for the overnight heating at 200°C in a drying cabinet.

An intensive Eirich impeller mixer of 10 liters capacity will be used. The hot dry aggregate will be transferred to the mixer and the corresponding cold crushed binder will be added. The mixing time will be 10 minutes and the paste temperature will be 173°C ±1°C (60°C above pitch SP).



Figure 4: Equipment for the Production of Bench Scale Anodes (RDC 161)

3.5.3 Forming

For each of the five pitch content levels, twenty bench scale anodes will be prepared from the two portions of paste. Portions of about 320 grams of paste will be successively pressed in a pre-heated (100°C) floating mould press for 30 seconds at a specific pressure of 400 bars in order to obtain bench scale anodes with 50 mm diameter and an approximate length of 100 mm. The complete unit including the mixer and the press is shown in the figure 2.

3.5.4 Baking

The set of 10 green cylinders will be baked in an electrically heated furnace BF12 (5 kW). It will be placed in a heat resistant steel recipient to form one layer of cylinders surrounded and covered by packing material pre sized to 2-1 mm. For each pitch percentage tested, the two batches of 10 cylinders will provide 20 cylinders for testing.

The following baking program will be applied:

- RT to 150°C: 100°C/h
- 150°C to 300°C: 10°C/h
- 300°C to 1'100°C: 50°C/h
- Soaking Time at 1'100°C: 20 hours

The baking furnace and its control unit are shown below.



Figure 5: RDC 166 Bench Scale Baking Furnace BF12

3.6 Bench Scale Anodes Testing

The green density of the cylinders will be measured after forming and cooling. After the baking step, the cylinders with the packing material glued at the surface will be weighed first, then cleaned with a metallic spatula and weighed again to determine the sticking propensity. The baked density of the cleaned cylinders will be eventually calculated along with the baking loss and the shrinkage related to the baking process.

The testing schedule of the baked anodes for a given pitch percentage (20 cylinders) is shown in the figure 4 below.

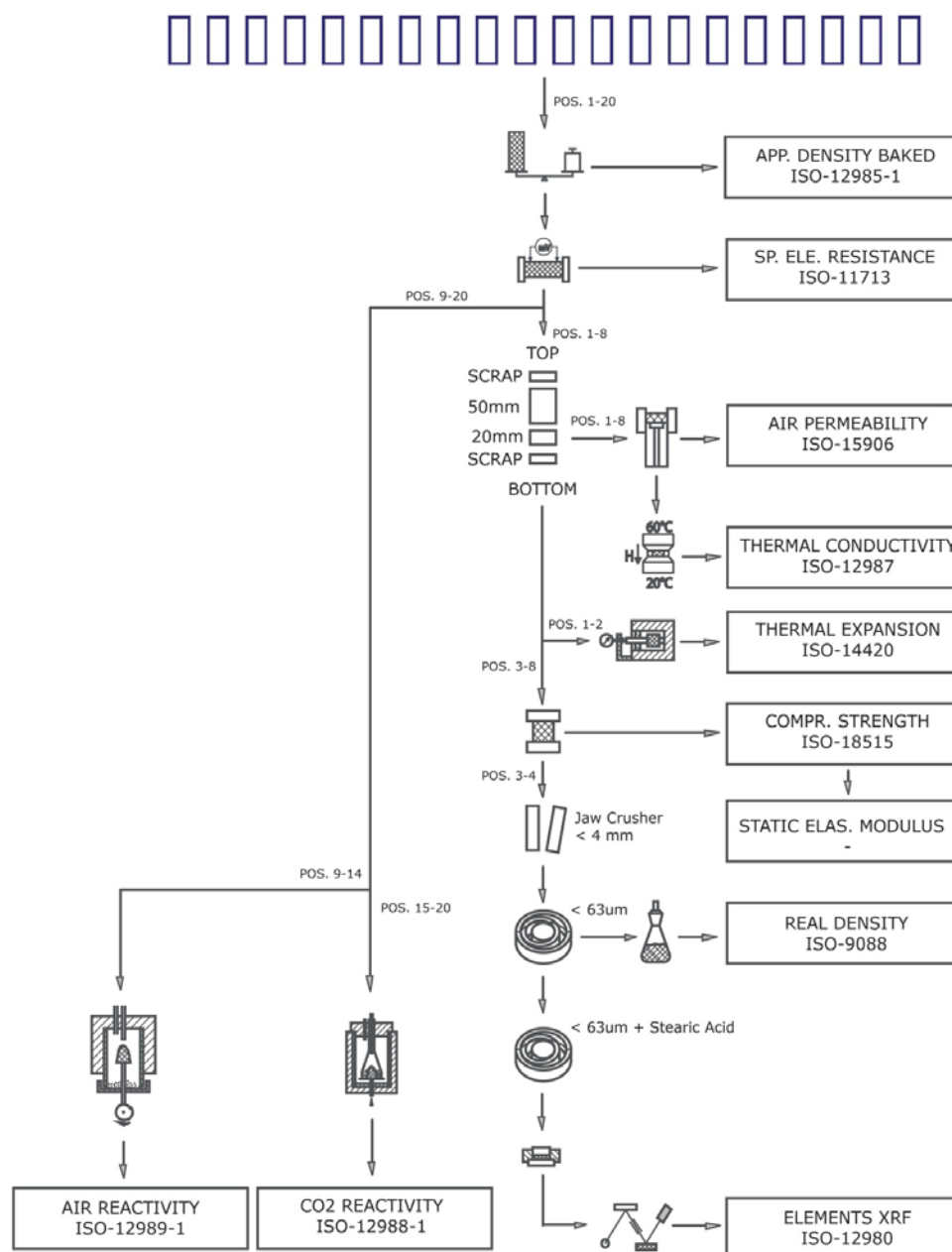


Figure 6: Bench Scale Anode Testing Schedule

4 Evaluation of the Results

The results are presented in the forms shown on the next pages. The ranges of results obtained on a worldwide basis for typical prebaked anode coke is shown as grey zones.

A coke giving anodes with properties lying in the grey zone shows a typical performance. When the curves or points are outside the grey zone the coke shows an inferior or a better performance potential than typical cokes.

The binder requirements to get:

- a maximum anode density and minimum air permeability
- a given level of mechanical strength, sp. electrical resistance or thermal conductivity (under pitched side)

can be easily assessed and compared to typical values for worldwide cokes or to another tested coke.

The maximum percentage of binder guaranteeing no sticking of packing material as well as no baking swelling expansion of the bench scale anodes can be determined too.

The levels of the CO₂ reactivity residue and dust give valuable information on the burning behavior and on the dusting propensity of the prebaked anodes.

The levels of the compressive strength, of the elasticity modulus along with the coefficient of thermal expansion allow predicting the thermal shock behavior of the full size prebaked anode in the pots.

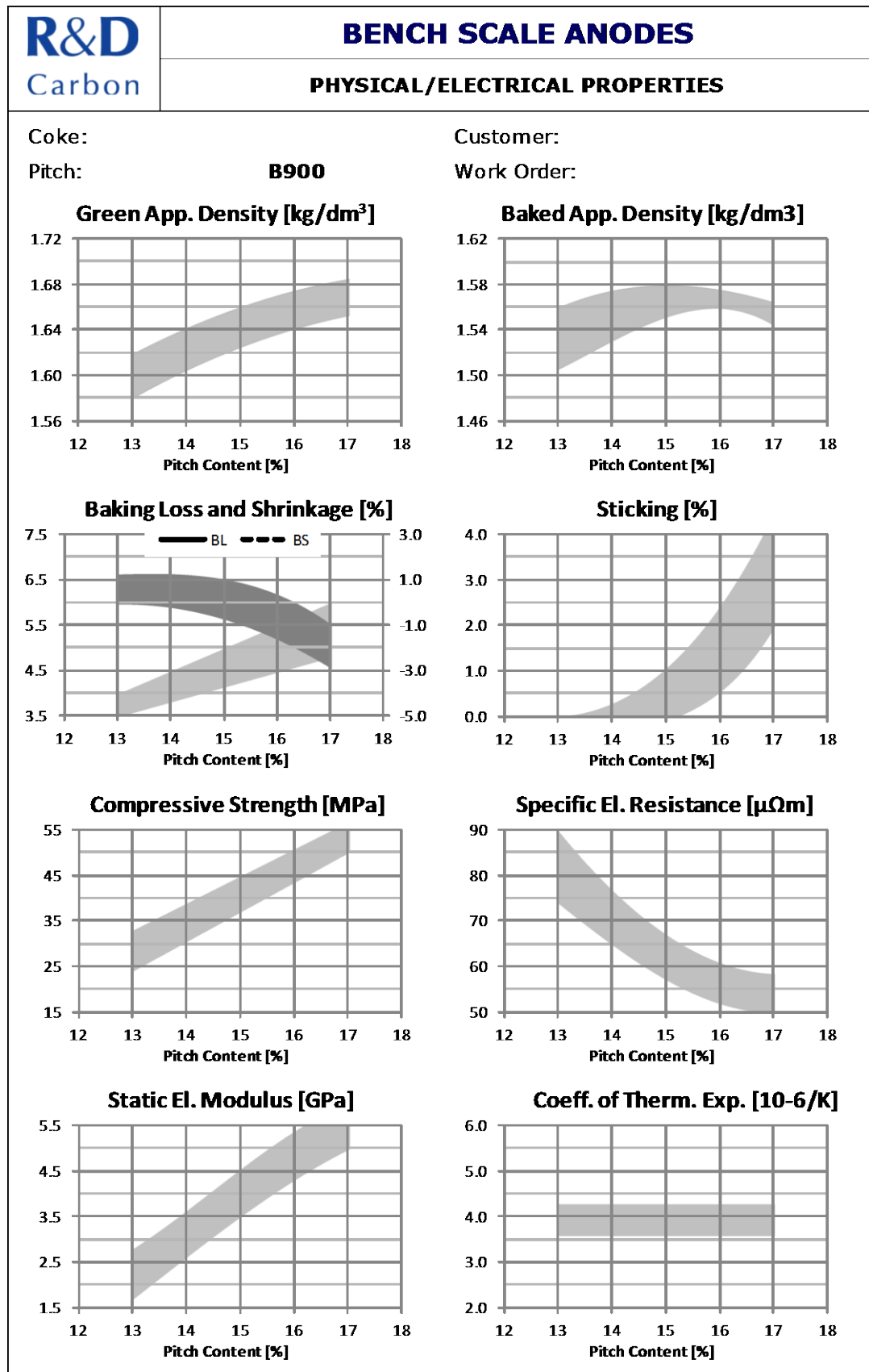


Figure 7 : Presentation of Bench Scale Results - Anode Physical and Electrical Properties

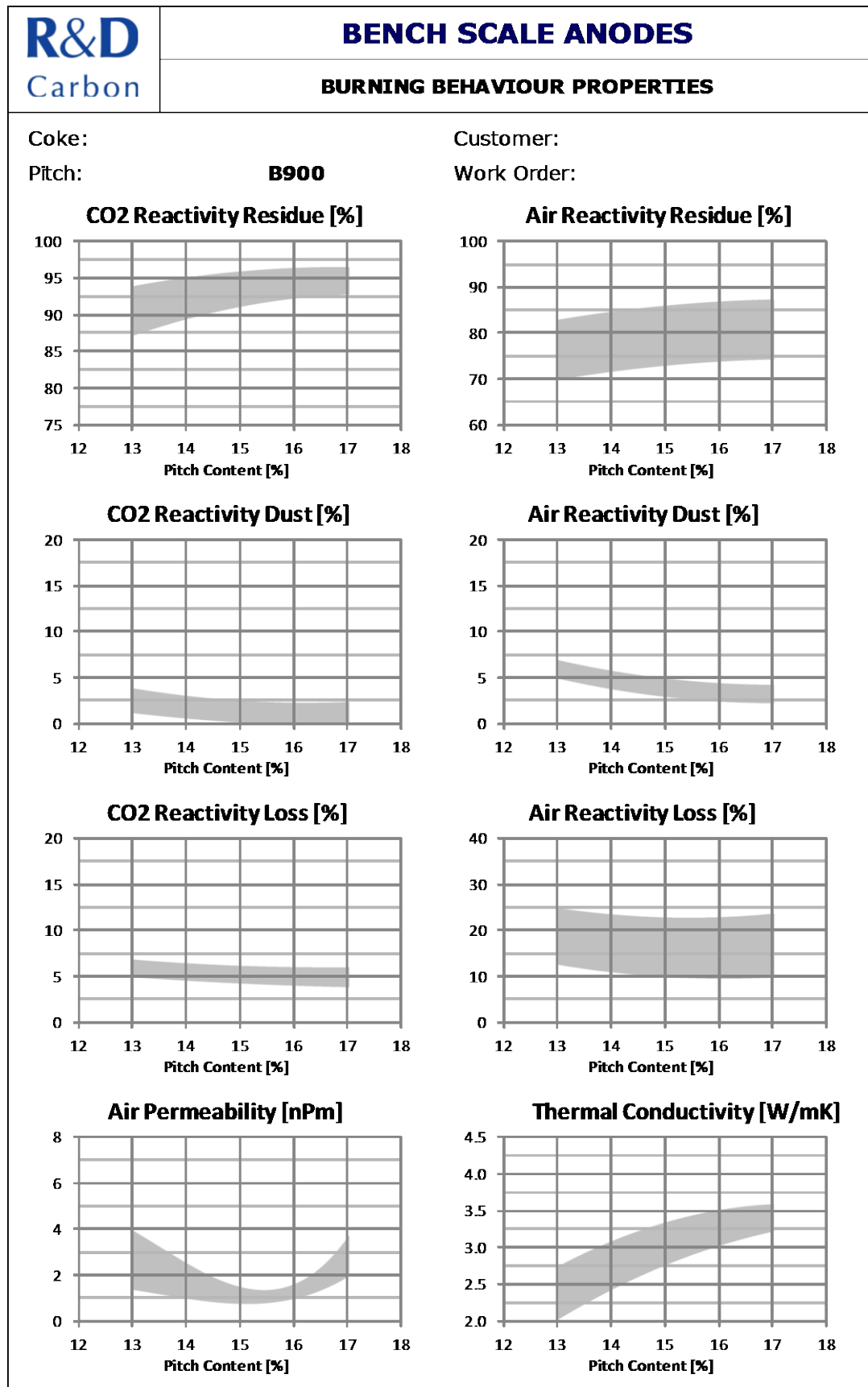


Figure 8 : Presentation of Bench Scale Results - Anode Burning Relevant Properties

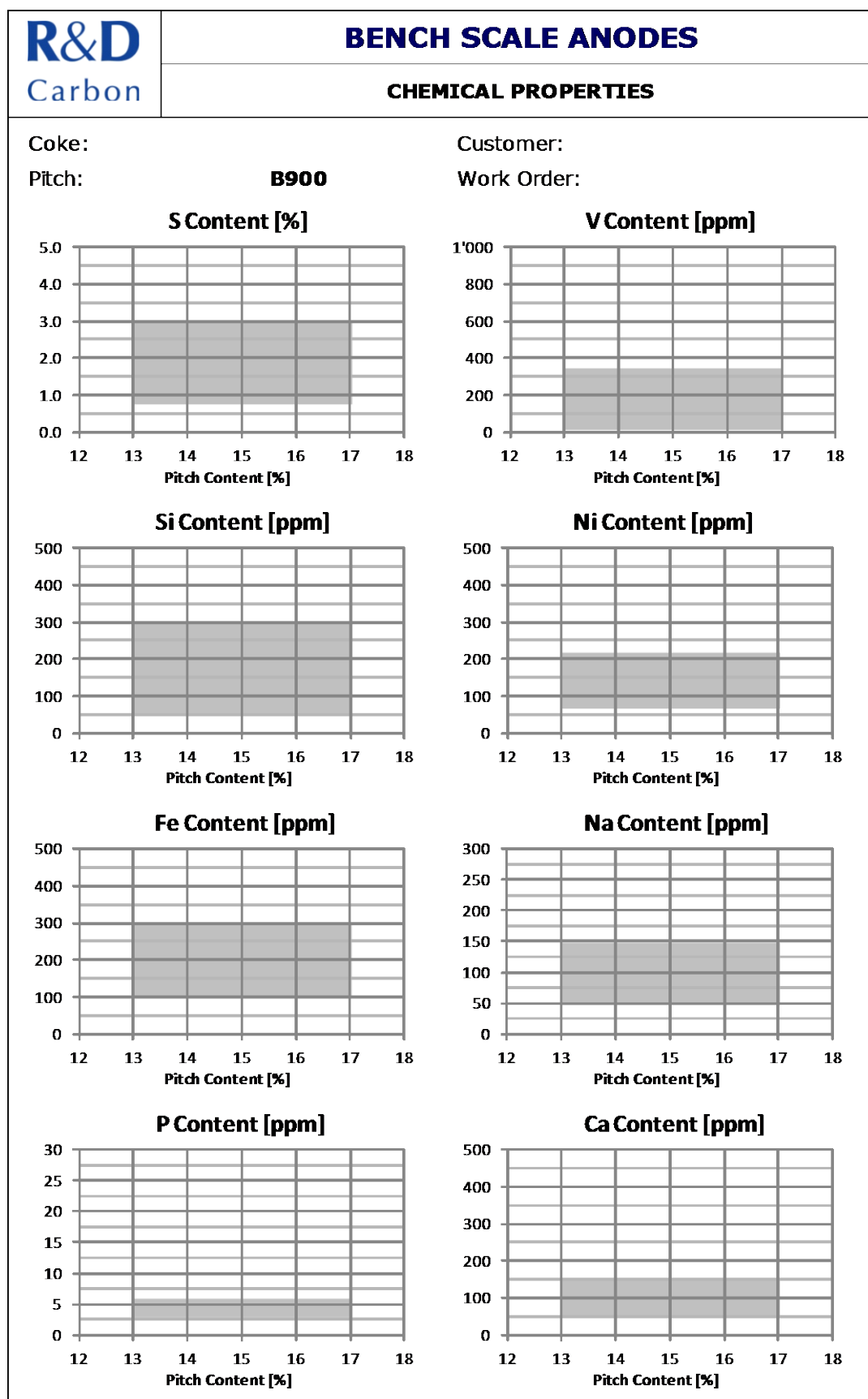


Figure 9 : Presentation of Bench Scale Results - Chemical Properties