

# Characterization and Activation Study of Black Carbon Chars Derived from Cellulosic Biomass Pyrolyzed at Very High Temperature



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February 2017

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Materials Science and Technology Division

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CELLULOSIC BIOMASS PYROLYZED  
AT VERY HIGH TEMPERATURE**

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## CONTENTS

	<b>Page</b>
LIST OF FIGURES .....	v
LIST OF TABLES .....	vii
1. SUMMARY .....	1
2. OBJECTIVE .....	1
3. RESULTS .....	2
3.1 SAMPLES RECEIVED .....	2
3.2 METHODS .....	2
3.3 RESULTS .....	4
3.3.1 TGA ANALYSIS .....	4
3.3.2 GAS ADSORPTION RESULTS .....	5
4. CONCLUSIONS AND RECOMMENDATIONS .....	12
ANNEX: TGA ANALYSIS IN FLOWING NITROGEN .....	15
SIMULATION OF PHYSICAL ACTIVATION BY TGA IN FLOWING CARBON DIOXIDE .....	25
CHARACTERIZATION BY GAS ADSORPTION OF AS-RECEIVED AND ACTIVATED SAMPLES .....	64



## LIST OF FIGURES

	<b>Page</b>
Figure 1. Procedures used and analyses performed starting from fresh as-received chars .....	2
Figure 2. Equipment used in this project.....	3
Figure 3. Nitrogen adsorption isotherms for samples with common origin, at various levels of activation. Note that individual plots have different scales .....	8
Figure 4. Nitrogen adsorption isotherms for samples with common origin, at various levels of activation. Note that all plots have the same scale.....	10





## LIST OF TABLES

	<b>Page</b>
Table 1. Results of TGA analysis in flowing nitrogen .....	4
Table 2. Apparent kinetic parameters for activation in CO <sub>2</sub> estimated from TGA analysis .....	5
Table 3. Summary of surface and porosity properties measured by gas adsorption .....	7
Table 4. Best performing activated carbons derived from PPI chars (data selected from Table 3).....	13
Table 5. Open literature examples of activated carbons properties used for mercury removal .....	13
Table 6. Open literature examples of activated carbons properties used for gold recovery .....	13



## 1. SUMMARY

The State of Tennessee, in partnership with the University of Tennessee (UT) and the Oak Ridge National Laboratory (ORNL), has created the RevV! Manufacturing voucher program to help Tennessee manufacturers gain access to the world-class resources at ORNL. As a part of this program, ORNL was working with Proton Power, Inc. (PPI), a rapidly growing company located in Lenoir City, Tennessee. PPI has developed a patented renewable energy system that uses biomass and waste sources to produce inexpensive hydrogen gas or synthetic fuels which are economically competitive with fossil fuels. The pyrolysis process used by PPI in their manufacturing chain generates significant amounts of black carbon char as by-product.

The scope of ORNL collaboration with PPI was assessing the black carbon char as a potential feedstock for activated carbon production, as this could be a potentially new revenue stream. During 2015-2016 ORNL received eight char samples from PPI and characterized their initial properties, simulated their physical activation by carbon dioxide, prepared gram-size samples of physically activated carbons, and characterized their surface and porosity properties. This report presents a summary of the work methods employed and the results obtained in the collaborative project between ORNL and PPI.

## 2. OBJECTIVE

Pyrolysis of cellulosic biomass in the process patented by PPI generates a solid by-product in addition to gaseous streams (volatile organic compounds and hydrogen) that are further processed into synthetic fuel and clean hydrogen gas. The solid byproduct is pyrolyzed biomass, referred to as pyrogenic carbon or carbon black. This material will be produced at large scale (about 6,000 tons per year) at the Rockwood, Tennessee demonstration facility where PPI will produce about five million gallons of synthetic fuel. The pyrolyzed biomass is either a significant disposal problem or a real financial opportunity if its properties justify further conversion into a high value activated carbon. Based on PPI estimates, some grades of activated carbon have a market value of about ten times (or more) compared with regular coal used for its heating values. The question that ORNL needed to answer was whether the chars obtained in the pyrolysis process have the potential to be converted, by activation, into activated carbon with competitive properties. The prospect of converting the byproduct into a higher value carbon product is important not only for the economics of PPI technology in general, but also as a source for expanding the Tennessee economy and creating new jobs.

To answer this question, ORNL has analyzed the potential of converting PPI black carbons from different bio-renewable sources into activated carbons, and evaluated the range of useful properties of such carbons obtained by physical activation. Activated carbons are porous carbons with high surface area, high pore volume, and tailored pore sizes. They can be used for gas adsorption and storage (carbon dioxide, methane, propane, etc.), indoor air cleaning (carbon dioxide, odors), cleaning industrial gas emissions (mercury from flue gas, other toxic compounds), recovery of volatile organic compounds (dry cleaning units), etc. Other applications are in civil engineering (city water cleaning, drinking water purification), food industry (molasses discoloration), and pharmaceutical industry (advanced purification), etc. Every application requires carbons with some specific properties.

The problem is that not all chars can be successfully activated. The properties achieved depend on the raw material's structure and on the activation method. Physical activation is, in fact, a slow, controlled oxidation process caused by high temperature reactions with carbon dioxide or steam. It is the most common and less expensive process, in which part of the carbon mass is consumed to create the porous network. The properties vary, depending on the raw material (hard wood, soft wood, grass, etc.) and on the process conditions (temperature, duration, nature of oxidant). There is not known correlation between

raw material source, process conditions, and the useful properties achieved at the end of physical activation. ORNL's task was to explore the behavior of chars of various origins in process conditions and to compare the extent of usable properties obtained by activation. The activation agent was carbon dioxide.

### 3. RESULTS

#### 3.1 SAMPLES RECEIVED

ORNL received two groups of charred biomass from PPI (Table 1). The samples have been previously milled (maximum grain size  $< 178 \mu\text{m}$ , with the exception of sample 15-049) and acid-washed to remove metallic impurities.

#### 3.2 METHODS

Figure 1 shows a schematic of the procedures used in this project. As-received materials were first characterized in their original state by gas adsorption (nitrogen) at 77 K. This technique provided the initial Brunauer-Emmett-Teller (BET) method surface area, total pore volume, and the pore size distribution. An automated Autosorb 1C (Quantachrome Instruments) was used for volumetric gas adsorption measurements (nitrogen at 77 K).

Then the samples were analyzed by the thermogravimetric method (TGA) in nitrogen and (separately) in carbon dioxide. A TA Q5000 (Thermal Analytics) thermobalance was used, which was operated with either  $\text{N}_2$  or  $\text{CO}_2$  flowing gases. The runs in nitrogen up to  $1000^\circ\text{C}$  were used to determine the volatile content in each carbon. The runs in carbon dioxide were performed to simulate the activation step and to characterize the kinetics of oxidation by  $\text{CO}_2$  at two temperatures ( $700^\circ\text{C}$  and  $800^\circ\text{C}$ ). This exercise showed that each carbon had specific oxidation kinetics (activation energy, pre-exponential term). This information was used to estimate the duration of thermal treatment at each temperature that would lead to weight losses between roughly 20 and 30 %.

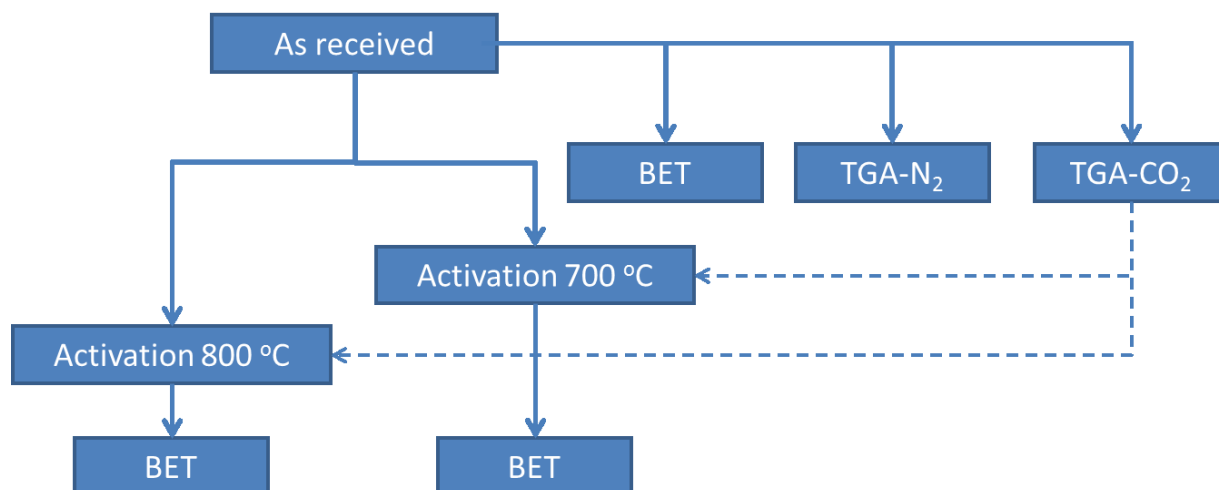


Figure 1. Procedures used and analyses performed starting from fresh as-received chars.

Activation of as-received materials was performed in flowing  $\text{CO}_2$  at 700 and 800  $^{\circ}\text{C}$  using samples every time. A horizontal furnace and a quartz tube (1 inch diameter) were used. The chars were held in porcelain boats and placed in the center of the uniform temperature zone. After heating in flowing  $\text{N}_2$  (in order to protect the carbon from oxidation) the gas was switched to  $\text{CO}_2$  and the sample was kept at the programmed temperature for a length of time estimated from prior TGA simulations. Cooling was done in flowing  $\text{N}_2$ . The weight loss recorded for each sample and activation temperature was used as the index for the degree of activation. Activated samples were kept in tightly closed vials. Each activated sample was then characterized by new measurements of BET surface area and porosity. Figure 2 shows the equipment used in the project.



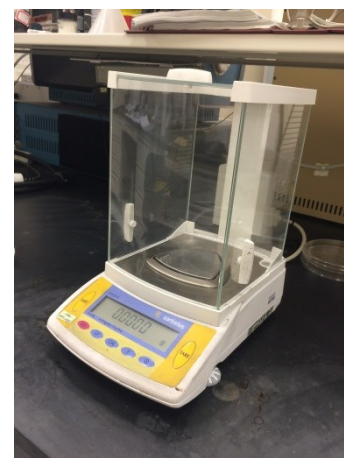
Thermogravimetric analyzer (TGA) used for volatile analysis and simulation of activation



Automated gas adsorption apparatus for BET and porosity analysis (Autosorb 1C)



Horizontal furnace and process tube used for activation in  $\text{CO}_2$



Analytical balance

**Figure 2. Equipment used in this project.**

### 3.3 RESULTS

#### 3.3.1 TGA Analysis

Table 1 shows the results of initial TGA analysis in nitrogen. The weight loss percentages are provided for four temperature ranges, corresponding to dehydration (30 to 120 °C), release of light volatiles (120 – 500 °C), pyrolysis and release of heavy volatiles (500 – 1000 °C) and isothermal treatment at 1000 °C. The residues vary between 80 and 90 %. The respective TGA plots are provided in the Annex.

**Table 1. Results of TGA analysis in flowing nitrogen**

Sample ID	Weight loss %					Origin
	30-120 °C	120-500 °C	500-1000 °C	1000 °C iso	Residue	
15-043	4.97	2.02	6.58	5.45	80.73	PPI Hardwood char
15-044	5.18	1.56	4.72	3.64	83.75	PPI Redwood char
15-045	5.99	2.76	5.44	3.60	82.21	PPI Switchgrass char
15-046	7.86	2.55	5.57	3.23	80.56	PPI Corn stoves char
15-049	5.96	1.92	4.20	1.89	85.96	PPI Hardwood char
16-004	3.04	2.64	4.91	2.97	87.10	PPI Chestnut oak
16-005	2.05	1.74	3.68	2.40	90.73	PPI White pine
16-006	1.62	1.63	5.48	3.23	88.70	PPI Yellow poplar

Table 2 shows the results of simulated activation runs using CO<sub>2</sub> as the physical activation agent. In these runs the rates of oxidation at constant temperatures (600, 700 and 800 °C) were measured and then used to estimate the apparent kinetic parameters  $A$  (frequency factor) and  $E_{act}$  (activation energy) characteristic to each material. The following equation was used;

$$Rate(\%/min)=100 \frac{\Delta m}{m_o} \frac{1}{\Delta t} = \frac{\Delta wt\%}{\Delta t} = A \exp\left(-\frac{E_{act}}{RT}\right)$$

In this Arrhenius-type equation  $\Delta m / \Delta t$  is the weight loss rate (mg/min) at constant temperature  $T$  (K),  $m_o$  is the initial sample weight (mg), and  $R$  is the gas constant (8.314 J mol<sup>-1</sup> K<sup>-1</sup>). The values of apparent kinetic parameters obtained from this analysis are shown in Table 2. The TGA plots for each char at two or three temperatures are provided in the Annex.

Data from Table 2 were used to estimate the duration of activation time corresponding to a target weight loss at the preselected temperatures of activation for each carbon material. The following equation was used:

$$\Delta t(min) = \frac{\Delta wt\%}{A(min^{-1})} \exp\left(\frac{E_{act}(\frac{J}{mol})}{8.314T(K)}\right)$$

A fair agreement was obtained between the actual weight loss obtained in practical conditions and the targeted weight loss. This procedure was efficient for producing activated samples with close degrees of activation, starting from materials with very different behavior. Indeed, one can see in Table 2 that and the frequency factor drops across seven orders of magnitude from sample 15-043 to sample 16-005. This trend correlates well with the variation of apparent activation energy which falls about seven times between the same two samples.

**Table 2. Apparent kinetic parameters for activation in CO<sub>2</sub> estimated from TGA analysis**

Sample ID	E <sub>act</sub>	A	ln(A)
	kJ/mol	min <sup>-1</sup>	
15-043	153	1.9E+07	16.74
15-044	81	2.2E+03	7.71
15-045	122	4.7E+05	13.07
15-046	139	2.9E+06	14.89
15-049	131	1.2E+06	14.02
16-004	35	1.0E+01	2.32
16-005	20	1.9E+00	0.63
16-006	38	1.8E+01	2.91

### 3.3.2 Gas Adsorption Results

Results obtained from N<sub>2</sub> adsorption (at 77 K) are provided in Table 3. The as-received and activated samples from the same char are grouped together to illustrate the progress of porosity with the activation level. The latter is estimated by the weight loss (BO, or “burn-off”) in column 2.

Other columns in Table 3 show the BET surface area ( $S_{\text{BET}}$ , m<sup>2</sup>/g) calculated according to the standard method developed by Brunauer, Emmett and Teller. The surface area values calculated from the density functional theory (DFT) method is provided in the next column ( $S_{\text{DFT}}$ , m<sup>2</sup>/g). It is normal that the two values differ a little. While DFT calculations are based on the most advanced molecular simulation method available now, and is more accurate than classical, thermodynamic methods, the BET method continues to be a standard used in most laboratories. Both results are presented here for the sake of comparison. Next, several values for the pore volume are provided. The total pore volume ( $V_t$ , cm<sup>3</sup>/g) is the volume of all pores with sizes below about 25-30 nm. It is calculated from the maximum amount of nitrogen condensed as liquid in the accessible pores within that size range (larger pores would not fill completely). The volume of micropores ( $V_{\text{micro}}$ , cm<sup>3</sup>/g) is the volume of pores with sizes < 2 nm. A subset of micropores called ultramicropores and having sizes below 0.7 nm is also shown ( $V_{\text{u-micro}}$ , cm<sup>3</sup>/g). The ultramicropores are the strongest adsorption sites of small molecule gases (H<sub>2</sub>, CO<sub>2</sub>, CH<sub>4</sub>, etc.). Larger molecules, such as volatile organic compounds, are adsorbed in the larger pores still belonging to the micropores space. The volume of even larger pores, mesopores with sizes > 2 nm, is shown next ( $V_{\text{meso}}$ , cm<sup>3</sup>/g). Mesopores are the avenues needed for quick access of gas molecules to their strong adsorption sites in the micropores. Mesopores are also the locus for adsorption of large organic molecules or

hydrated ions from aqueous media in applications such as water cleaning or electrical charge storage in supercapacitors. The presence of mesopores is recognized by the existence of hysteresis between the adsorption and desorption branches of the isotherms. The micropore volumes were calculated with the quenched surface DFT (QSDFT) approximation for slit-shaped pores of carbon. The mesopores volumes were evaluated from the desorption branch of isotherms using the method developed by Barrett, Joyner and Halenda (BJH).

More information on the efficiency of activation for each type of char, and a rough measure of porosity distribution between micropores (< 2 nm) and mesopores (> 2 nm), is available from the next two columns of Table 3, which contain the ratios between micropore (mesopores) volumes and the total pore volume. For most chars the ratio  $V_{\text{micro}}/V_t$  increases and the ratio  $V_{\text{meso}}/V_t$  decreases with the increase of the activation level (measured by BO). This shows that physical activation performed here did not only increase the total pore volume, but also did increase the proportion of micropores at the expense of mesopores.

The average pore size is evaluated in two different ways. The values calculated by the DFT method ( $d_{\text{DFT-mode}}$ , Å) represent the statistical mode of the pore size distribution calculated in the QSDFT approximation. These values are close to the size of ultramicropores (about 6-7 Å) because this pore size was present most frequently in the calculated distributions. The average pore size values ( $d_{\text{average}}$ , Å) is a rough estimate of the pore diameter in the hypothetical situation that all pores have cylindrical shape, and is obtained by the simple calculation  $d_{\text{average}} = 4 \times 10^4 \times (V_t / S_{\text{BET}})$ . These values have larger variation, as they depend on both  $V_t$  and  $S_{\text{BET}}$ .

The gas adsorption isotherms measured for each sample are provided in the Annex. Also provided are the pore size distributions evaluated using the QSDFT approximation for equilibrium adsorption of  $\text{N}_2$  in carbon slit-shaped pores.

The next two figures compare nitrogen adsorption isotherms on as-received and activated samples. In Figure 3 the  $\text{N}_2$  isotherms for samples with common origin are compared at various levels of modification: from the as-received state to the highest activated state (the state with the highest burn-off degree). This figure illustrates the increase of the amount adsorbed during activation. Note that the vertical axis has different ranges on each plot.

To ease the comparison between samples from different origins, the data are replotted in Figure 4 on a common scale. The plots now show clearly that two materials, derived from chestnut oak and white pine show the largest amounts adsorbed among all samples, with maximum BET surface area values around 950 – 1050  $\text{m}^2/\text{g}$  after activation at 800 °C.



Table 3. Summary of surface and porosity properties measured by gas adsorption

Sample	BO %	S <sub>BET</sub> m <sup>2</sup> /g	S <sub>DFT</sub> m <sup>2</sup> /g	V <sub>t</sub> cm <sup>3</sup> /g	V <sub>u-micro &lt;7Å</sub> cm <sup>3</sup> /g	V <sub>micro &lt;20Å</sub> cm <sup>3</sup> /g	V <sub>meso</sub> cm <sup>3</sup> /g	V <sub>micro</sub> /V <sub>t</sub>	V <sub>meso</sub> /V <sub>t</sub>	d <sub>DFT-mode</sub> Å	d <sub>average</sub> Å	Origin	File
15-043	0	152	139	0.15	0.01	0.05	0.09	0.33	0.60	7.85	40.1	PPI Hardwood char	AS621801
15-043-700	28	448	566	0.26	0.12	0.17	0.07	0.65	0.27	6.14	22.8		AS620401
15-043-800	38	730	894	0.40	0.17	0.27	0.10	0.68	0.25	6.14	21.8		AS612801
15-044	0	159	133	0.17	0.01	0.04	0.11	0.24	0.65	7.85	42.7	PPI Redwood char	AS622201
15-044-700	20	354	437	0.21	0.09	0.14	0.06	0.67	0.29	6.14	23.3		AS620901
15-044-800	44	625	751	0.33	0.15	0.24	0.07	0.73	0.21	6.14	21.3		AS620101
15-045	0	207	208	0.19	0.02	0.08	0.11	0.42	0.58	6.14	37.5	PPI Switchgrass char	AS622301
15-045-700	19	246	278	0.31	0.04	0.08	0.22	0.26	0.71	6.14	50.4		AS6B1101
15-045-800	34	612	692	0.41	0.12	0.22	0.18	0.54	0.44	6.14	26.8		AS640101
15-046	0	333	374	0.27	0.06	0.12	0.13	0.44	0.48	6.14	32.1	PPI Corn stoves char	AS622501
15-046-700	22	516	632	0.32	0.13	0.19	0.12	0.59	0.38	6.14	24.6		AS681601
15-046-800	29	611	731	0.38	0.14	0.22	0.15	0.58	0.39	6.14	25.2		AS681801
15-049	0	38	24	0.04	0	0.01	0.03	0.25	0.75	6.14	64.4	PPI Hardwood char	AS582401
16-004	0	241	238	0.39	0.02	0.07	0.32	0.18	0.82	6.14	64.1	PPI Chestnut oak	AS642901
16-004-800	27	915	999	1.15	0.14	0.28	0.85	0.24	0.74	6.14	50.2		AS6B1701
16-005	0	351	417	0.26	0.07	0.13	0.13	0.50	0.50	6.14	29.4	PPI White pine	AS650201
16-005-800	39	1050	1080	1.44	0.15	0.33	1.06	0.23	0.74	6.14	55.1		AS6B1601
16-006	0	529	644	0.4	0.12	0.19	0.21	0.48	0.53	6.14	30.7	PPI Yellow poplar	AS650301
16-006-800	47	686	763	0.51	0.13	0.25	0.25	0.49	0.49	6.14	29.6		AS292601

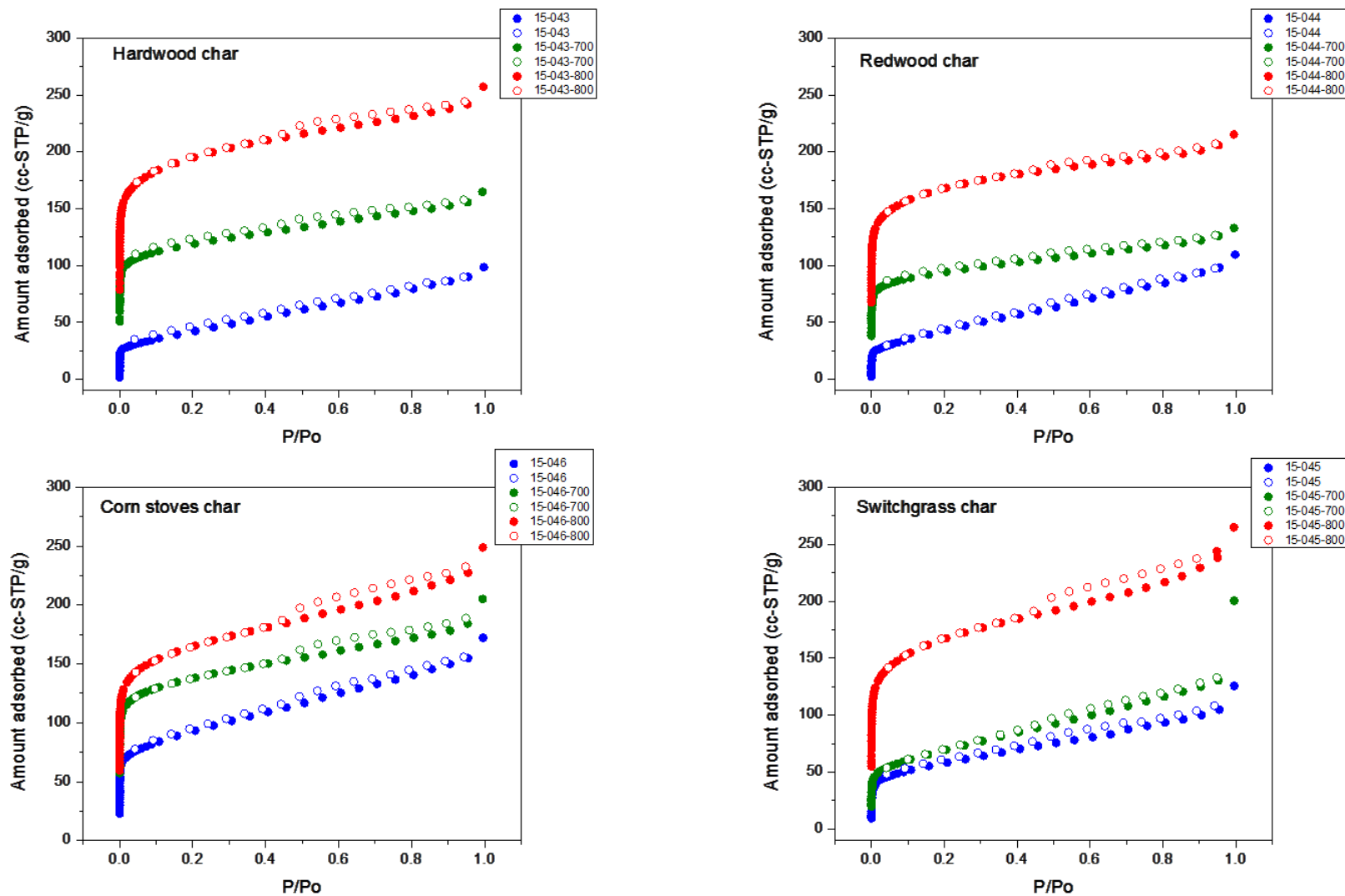


Figure 3. Nitrogen adsorption isotherms for samples with common origin, at various levels of activation. Note that individual plots have different scales.

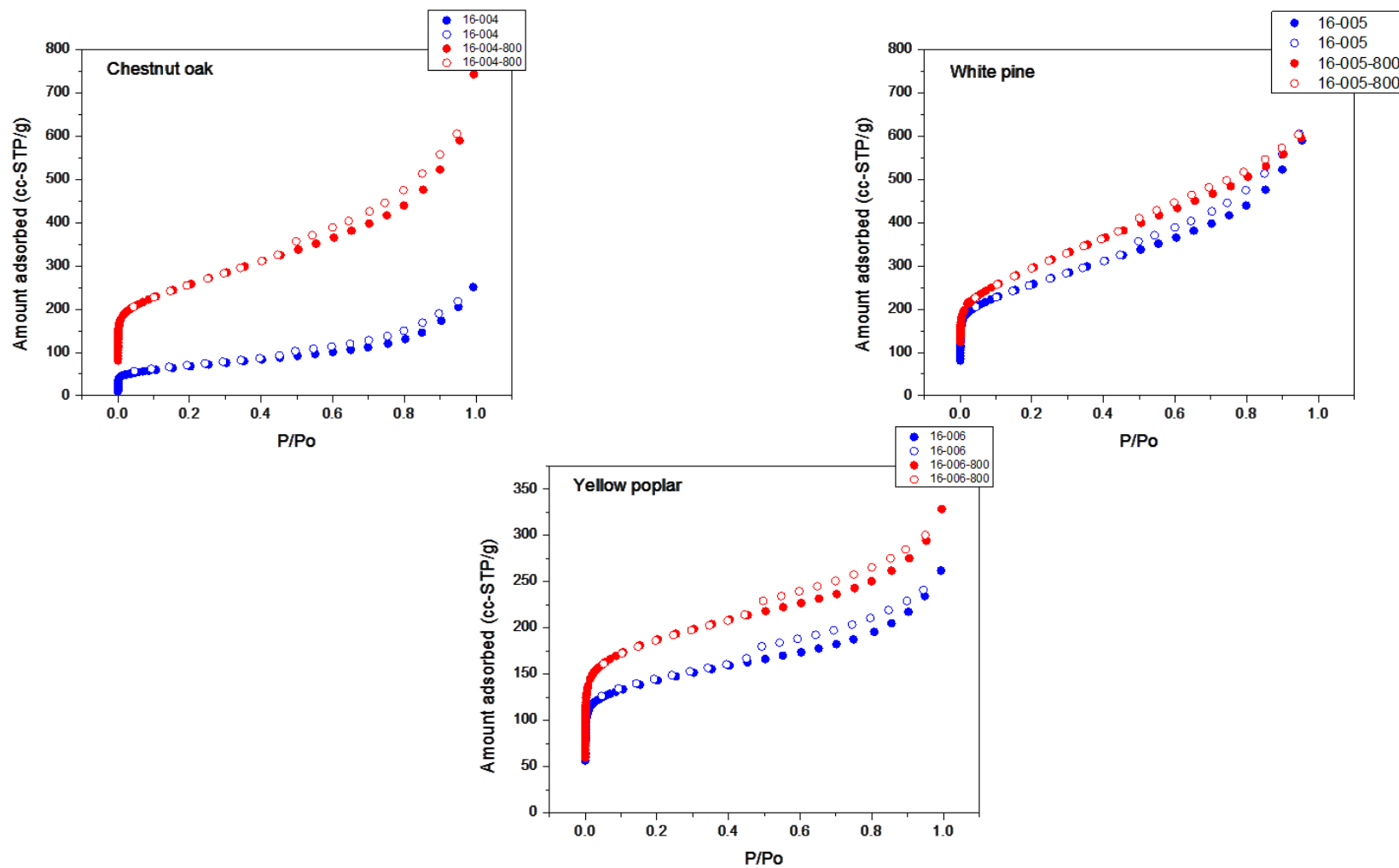


Figure 3 (cont.). Nitrogen adsorption isotherms for samples with common origin, at various levels of activation. Note that individual plots have different scales.

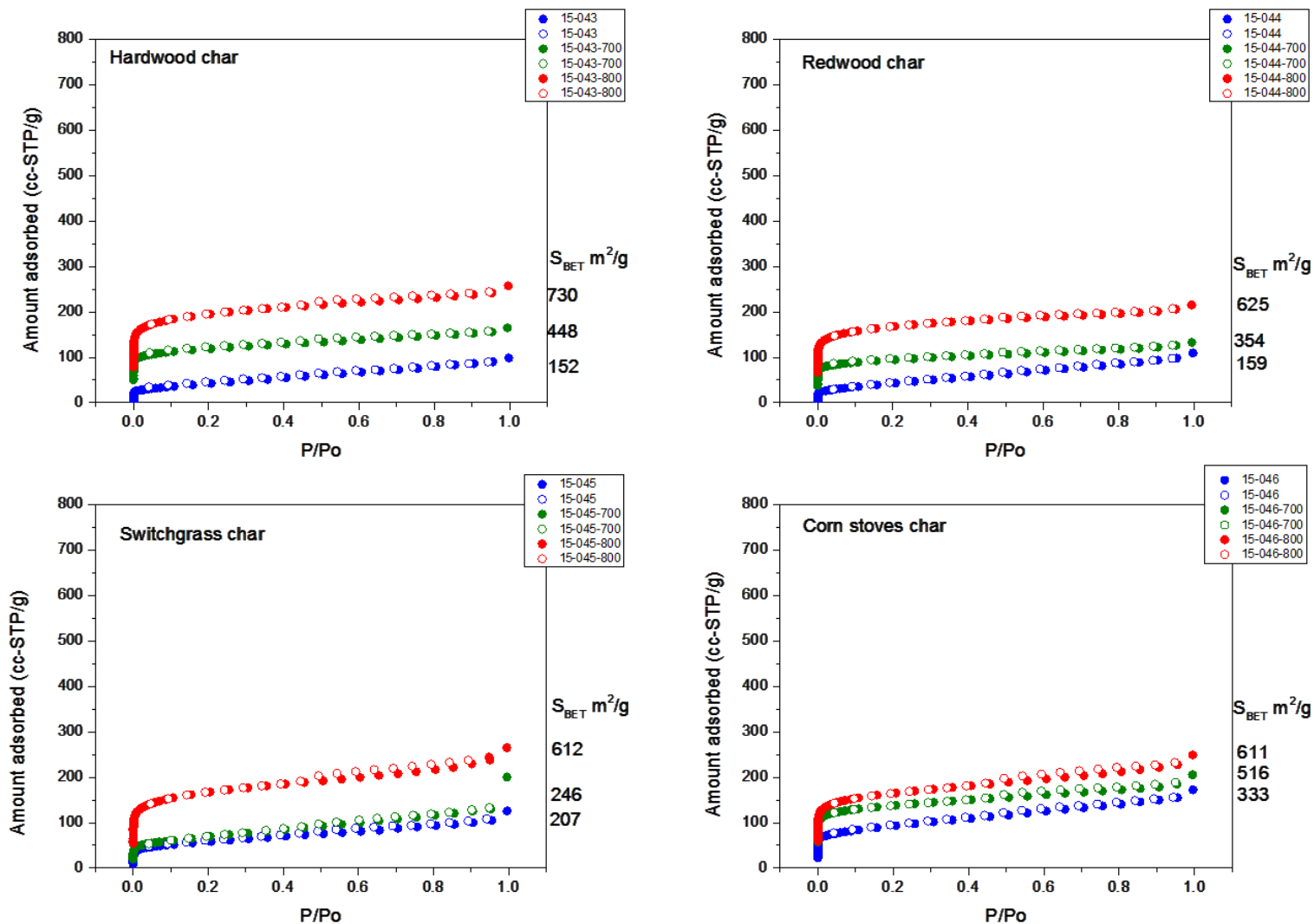


Figure 4. Nitrogen adsorption isotherms for samples with common origin, at various levels of activation. Note that all plots have the same scale.

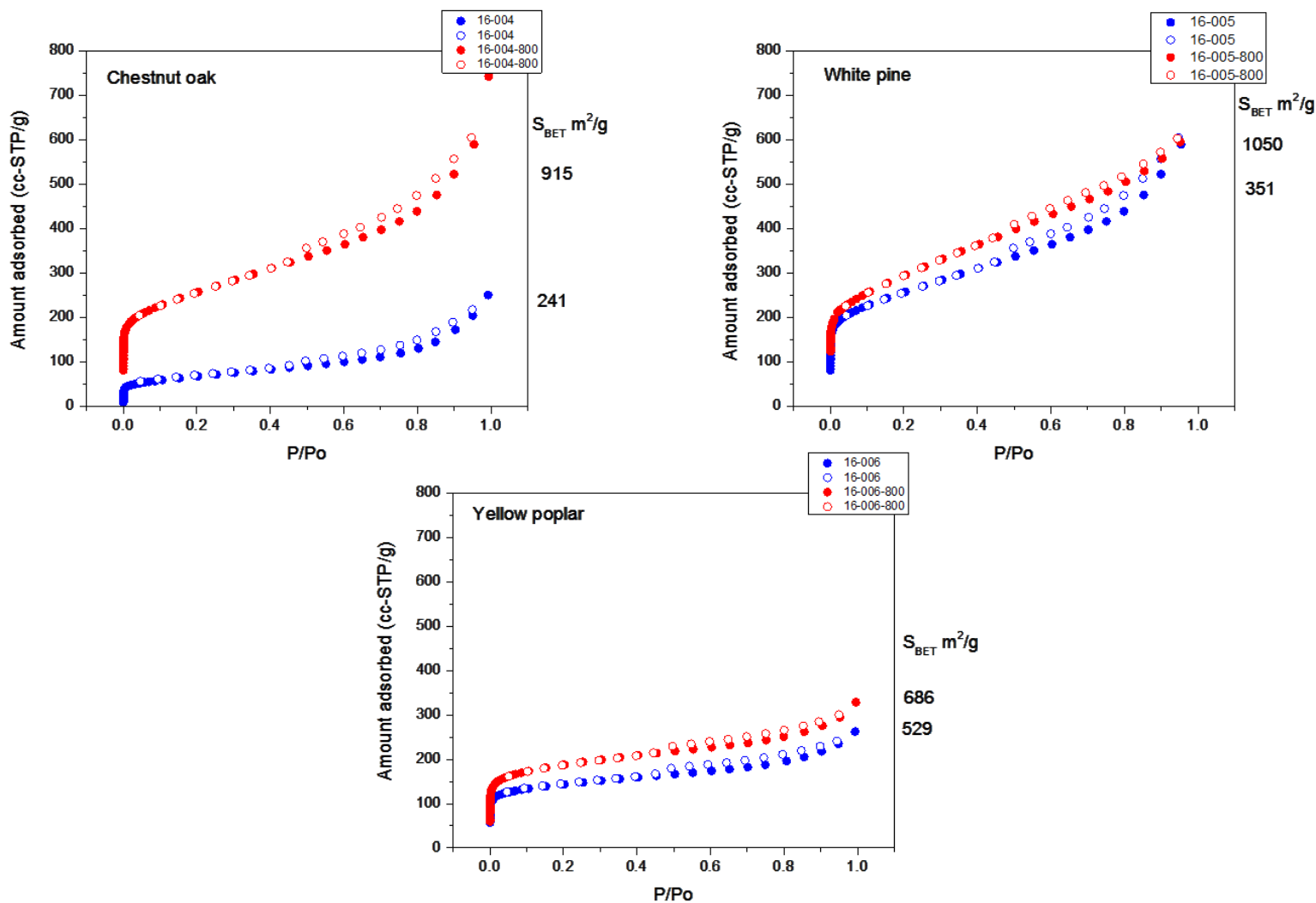


Figure 4 (cont.). Nitrogen adsorption isotherms for samples with common origin, at various levels of activation. Note that all plots have the same scale.

#### 4. CONCLUSIONS AND RECOMMENDATIONS

The results presented above suggest that the chars derived from Chestnut Oak and from White Pine have the potential of being converted into porous carbons with surface area in the range of about 1000 m<sup>2</sup>/g. Other wood essences (hardwood char, redwood char, yellow poplar) can also be activated to around 700 m<sup>2</sup>/g BET surface area. Based on data from open literature, the characteristics of these activated carbons obtained from PPI proprietary chars are comparable to those of commercial carbons reported in the open literature for applications in adsorption from solution.

In general, adsorption from solutions (aqueous or organic) follows a few rules of thumb<sup>1</sup> that indicate what type of applications can be successfully pursued with various types of carbons:

- Large molecules adsorb better than smaller molecules
- Non-polar molecules adsorb better than polar molecules
- Non-soluble or slightly soluble molecules adsorb better than soluble molecules
- pH may have an effect on the extent of adsorption (depends on polarity and solubility)
- Temperature favors diffusion kinetics but lowers adsorbability
  - Alcohols and aldehydes are poorly adsorbed because they are soluble and highly polar
  - Glycols are soluble and not very adsorbable
  - Amines adsorption is limited by polarity and solubility
  - Chlorinated organic compounds (aromatics, aliphatics) are quite adsorbable
  - High molecular weight organic compounds are more adsorbable

Granular activated carbon (GAC) adsorbents for liquid applications can be classified in three categories:

- Bituminous granular GAC – used for removing low concentrations of low molecular organic contaminants from waters, can have BET surface area up to 900 m<sup>2</sup>/g;
- Lignite GAC – used mostly for larger molecules and decolorizing applications, has BET surface area up to 650 m<sup>2</sup>/g
- Coconut shell-based GAC with BET surface area above 1000 m<sup>2</sup>/g – has narrower porosity which causes slower diffusion from liquid phase, and is better used in gas-vapor applications.

Table 4 compares the relevant properties of best performing activated carbons obtained from PPI chars. These data were selected from Table 3. For comparison, Table 5 shows the properties of several carbons tested from mercury adsorption from flue gas<sup>2</sup> and from aqueous solutions.<sup>3</sup> Similarly, Table 6 shows a few results from literature<sup>4</sup> on the activated carbons efficiency on metal recovery (Au) from aqueous solutions.

<sup>1</sup> \*\*\* Engineering and Design Adsorption Design Guide, Department of the Army, U.S. Army Corps of Engineers, Design Guide DG 1110-1-2 (1 Mar 2001).

<sup>2</sup> M M Maroto-Valer, Y Zhang, E J Granite, Z Tang, H W Pennline, Effect of pore structure and surface functionality on the mercury capacity of a fly ash carbon and its activated sample. Fuel 84 (2005) 105-108.

<sup>3</sup> M. Zabihi, A Haghighi, A Ahmadpour, Studies on adsorption of mercury from aqueous solution on activated carbons prepared from walnut shell, J. Hazard Mater. 174 (2010) 251-256.

<sup>4</sup> ON Kononova, AG Kholmogorov, NV Danilenko, SV Kachin, GL Paskov, YS Knonov, ZhV Dmitrieva. Sorption of gold and silver on carbon adsorbents from thiocyanate solutions, Carbon 43 (2005) 17-22.

**Table 4. Best performing activated carbons derived from PPI chars (data selected from Table 3)**

Carbon	BET surface area (m <sup>2</sup> /g)	V <sub>t</sub> (cm <sup>3</sup> /g)	d average (Å)	Source
16-005-800	1050	1.44	55	PPI White pine - CO2 activation
16-004-800	915	1.15	50	PPI Chestnut oak - CO2 activation
15-043-800	730	0.4	22	PPI Hardwood char - CO2 activation
16-006-800	686	0.51	30	PPI Yellow poplar - CO2 activation
15-044-800	625	0.33	22	PPI Redwood char - CO2 activation

**Table 5. Open literature examples of activated carbons properties used for mercury removal**

Carbon	BET surface area (m <sup>2</sup> /g)	V <sub>t</sub> (cm <sup>3</sup> /g)	d average (Å)	Source	Mercury capacity (mg/g)	Reference
<b>From gas phase</b>						
DEM-OCCI	53	0.04	18	n/a	1.85	
AC-PCCI	863	0.49	23	n/a	0.23	2
Darco	700	n/a	n/a	commercial benchmark	2.77	
<b>From aqueous solutions</b>						
Carbon A	780	0.43	1640	Walnut shells, Zn impregnation	151	3
Carbon B	803	0.39	176	Walnut shells, Zn impregnation	101	

**Table 6. Open literature examples of activated carbons properties used for gold recovery**

Carbon	BET surface area (m <sup>2</sup> /g)	V <sub>t</sub> (cm <sup>3</sup> /g)	d average (Å)	Source	Gold capacity (mmol/g)	Reference
BAU	760	0.7	37	charcoal	2.3	4
ABG	420	0.56	53	brown coal	1.3	
UC	1450	0.52	14	cocoa stones	0.5	
LK4	1643	0.65	16	Siberian anthracite	3	

The tables shown above suggest that the activated PPI carbons have physical properties very much comparable with those reported in these applications. There is not much clarity in the literature<sup>5</sup> on what the main factors that control adsorption from solutions properties of activated carbons are. Along with physical properties, the surface chemistry (presence of functional groups), solution chemistry (pH, concentration, composition) and the nature of chemical species of interest play important roles. For this reason, testing of a given (recommended) carbon in the real conditions of its possible application remains a sure criterion for acceptance.

Adsorption from gas phase is better understood than adsorption from solutions. The gas adsorption properties of activated carbons depend primarily on their BET surface area and the pore size of prevalent

<sup>5</sup> H Marsh and F Rodriguez-Reinoso, "Activated Carbon". Elsevier, 2006.

pores. Carbons with a large volume of narrow pores (micropores, less than 20 Å) are most efficient for adsorption of small gas molecules. Possible applications include gas storage (e.g. methane), gas separation (CO<sub>2</sub> from methane), volatile organic compounds recovery (in chemical industry, refineries, dry cleaning units), etc. The benchmark of highly activated, ultramicroporous carbons with possible applications for demanding gas adsorption processes (such as H<sub>2</sub> storage at cryogenic temperatures, low pressure methane storage, etc.) have a large surface area (2500 – 2800 m<sup>2</sup>/g), significant volumes of ultramicropores (< 7 Å) and supermicropores (< 20 Å) and narrow pore size distribution.

Many volatile organic compounds can be removed from vapor phase using activated carbons. In general, non-polar organic compounds adsorb better than polar organic compounds, and higher molecular compounds adsorb better than lighter ones.<sup>1</sup> Adsorption on activated carbons is frequently used for removal of chlorinated solvents and aromatic hydrocarbon fuel components from air. It is more difficult to adsorb aldehydes, ketones and alcohols, although they adsorb better from vapor phase than from aqueous solutions where solubility is an additional factor.

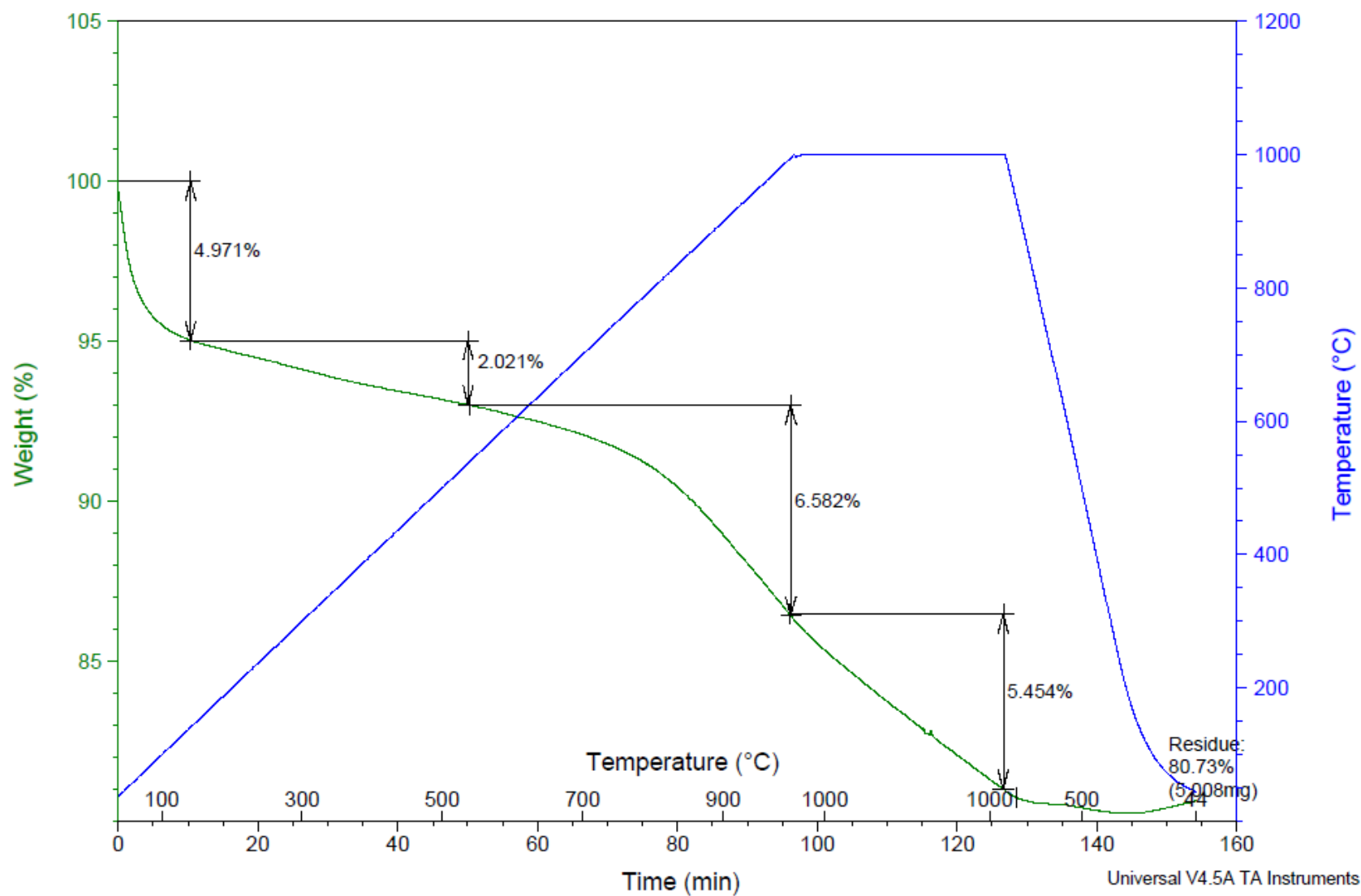
The importance of measuring adsorption isotherms specific to each application cannot be stressed enough when a decision is to be made about commercializing activated carbons. The reason is that adsorption processes are usually complicated by factors that cannot be foreseen a priori. One of them – and very important – is the competition exercised by other components, even minor, that might be present in a real situation. It is known that water vapors hinder adsorption of small molecules or volatile organic compounds from gas phase, and heavier hydrocarbons compete with lighter ones. Temperature is another very important factor for gas adsorption. Lowering temperature will increase the adsorption capacity. Since relative humidity in gases (air) depends on temperature, it is often necessary to determine which combination of gas temperature and relative humidity works efficiently for a given application.



## **TGA analysis in flowing nitrogen**

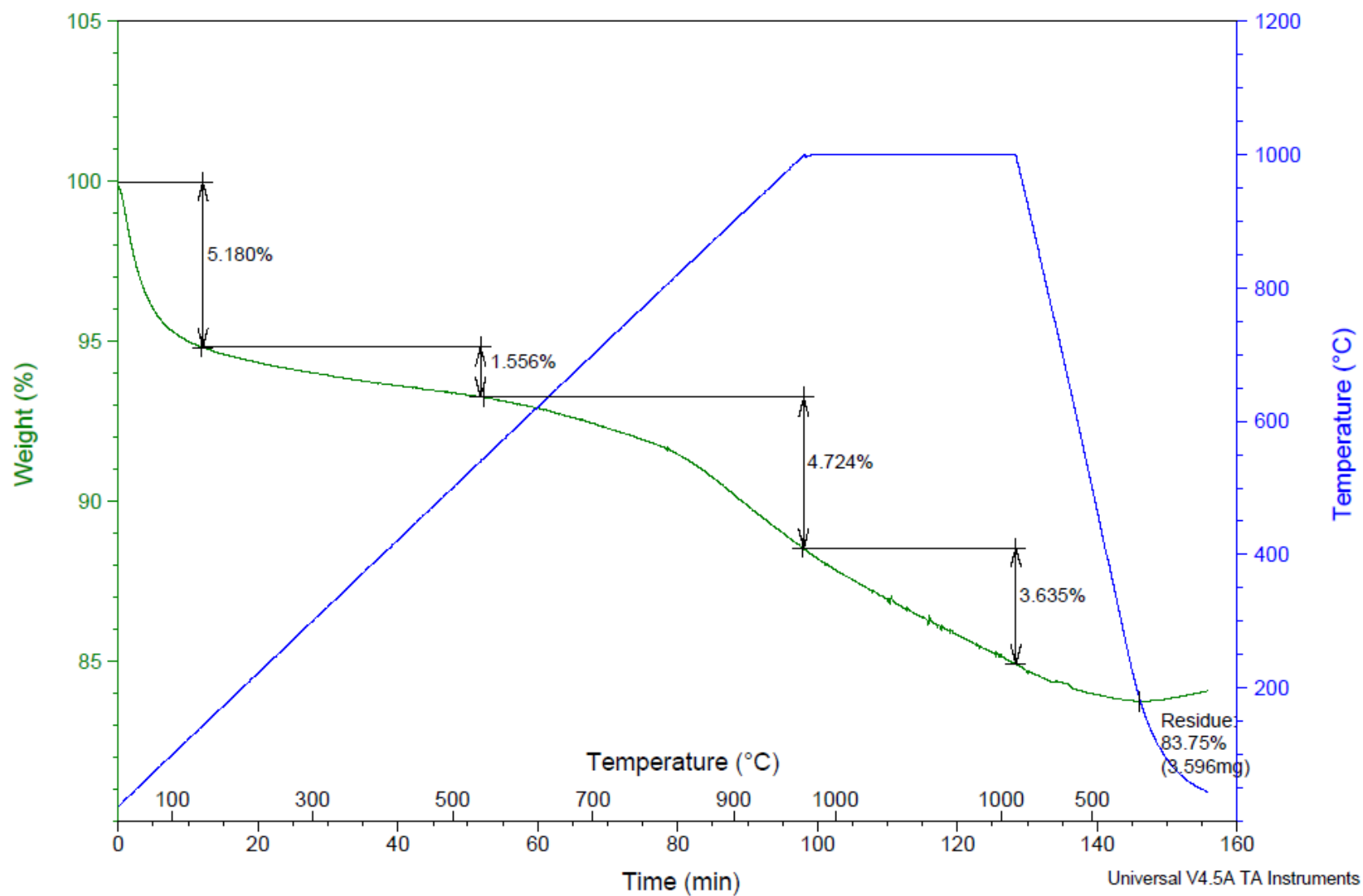
Sample: Proton Power 15-043  
Size: 6.2030 mg  
Method: Basic Carbon Ash

## TGA



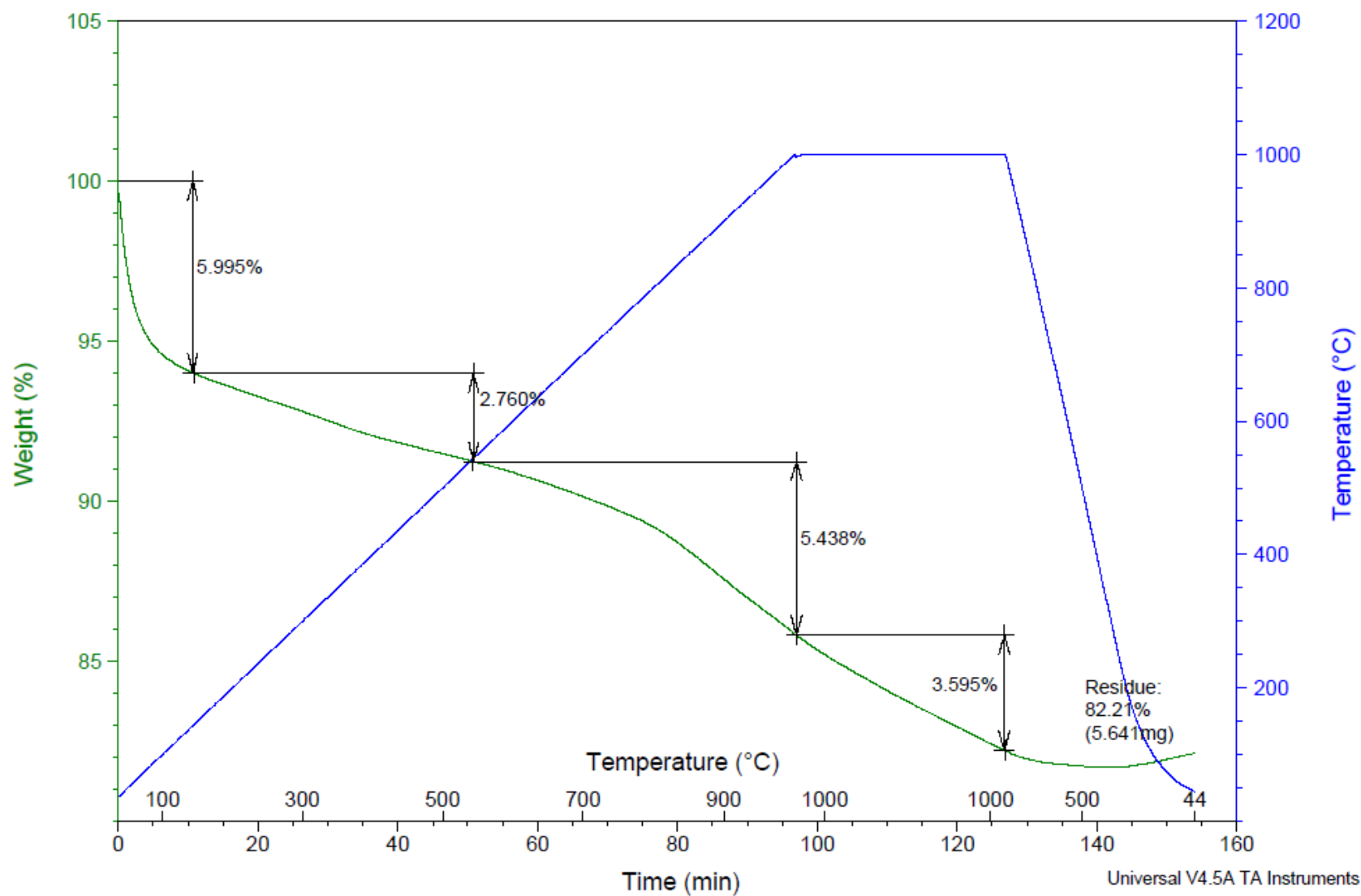
Sample: Proton Power 15-044 (repeat)  
Size: 4.2940 mg  
Method: Basic Carbon Ash

## TGA



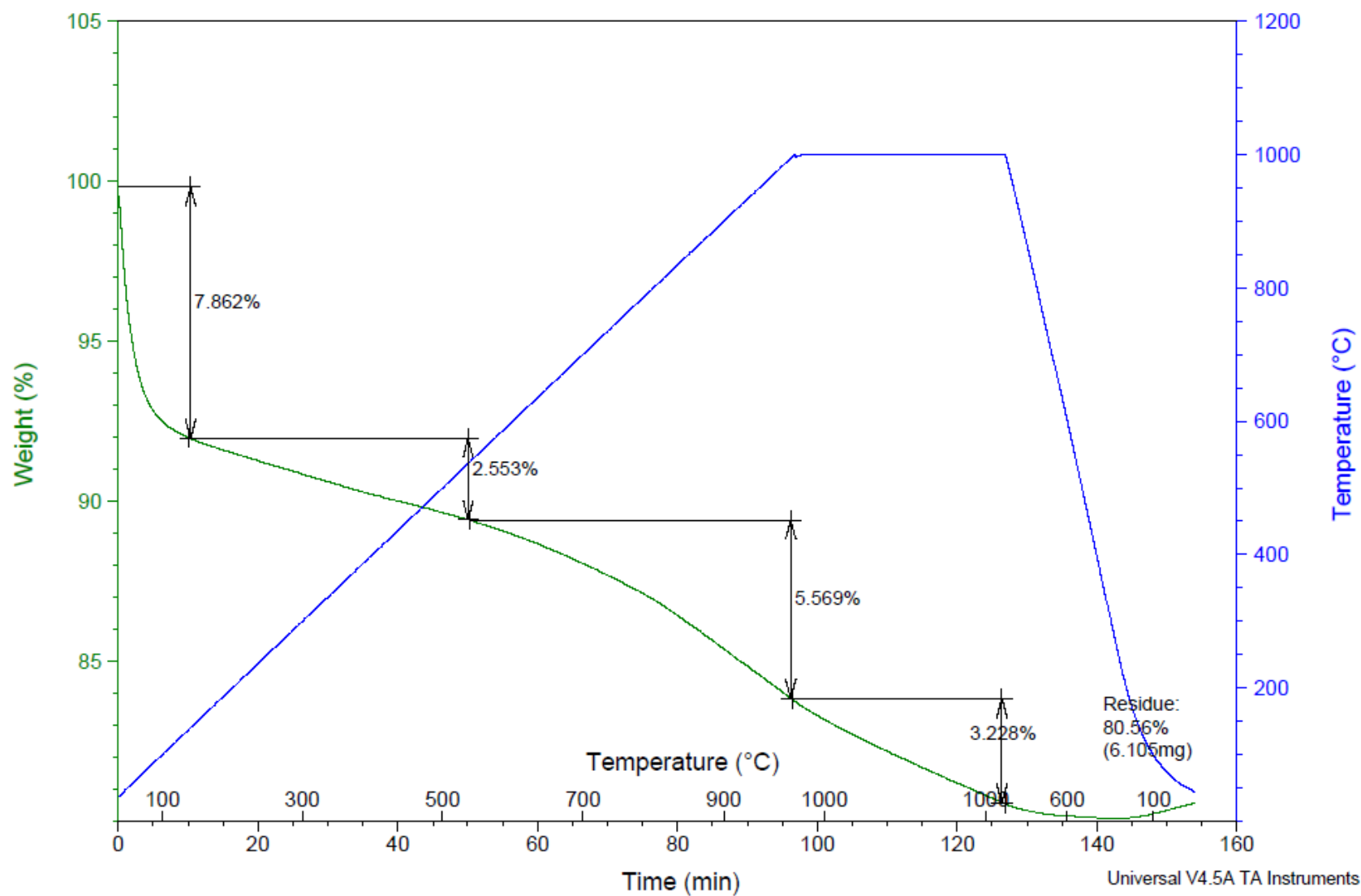
Sample: Proton Power 15-045  
Size: 6.8620 mg  
Method: Basic Carbon Ash

## TGA



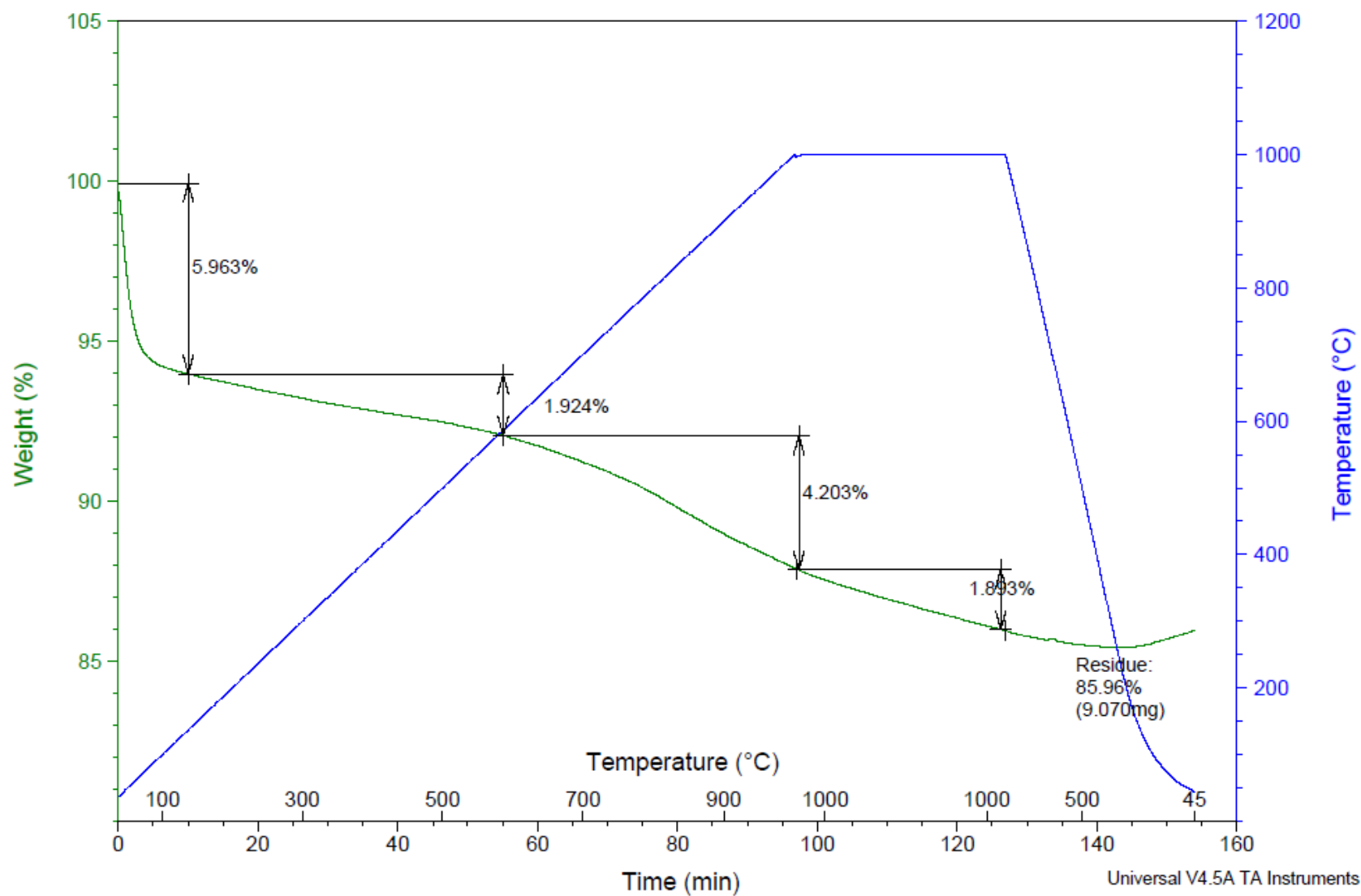
Sample: Proton Power 15-046  
Size: 7.5790 mg  
Method: Basic Carbon Ash

## TGA



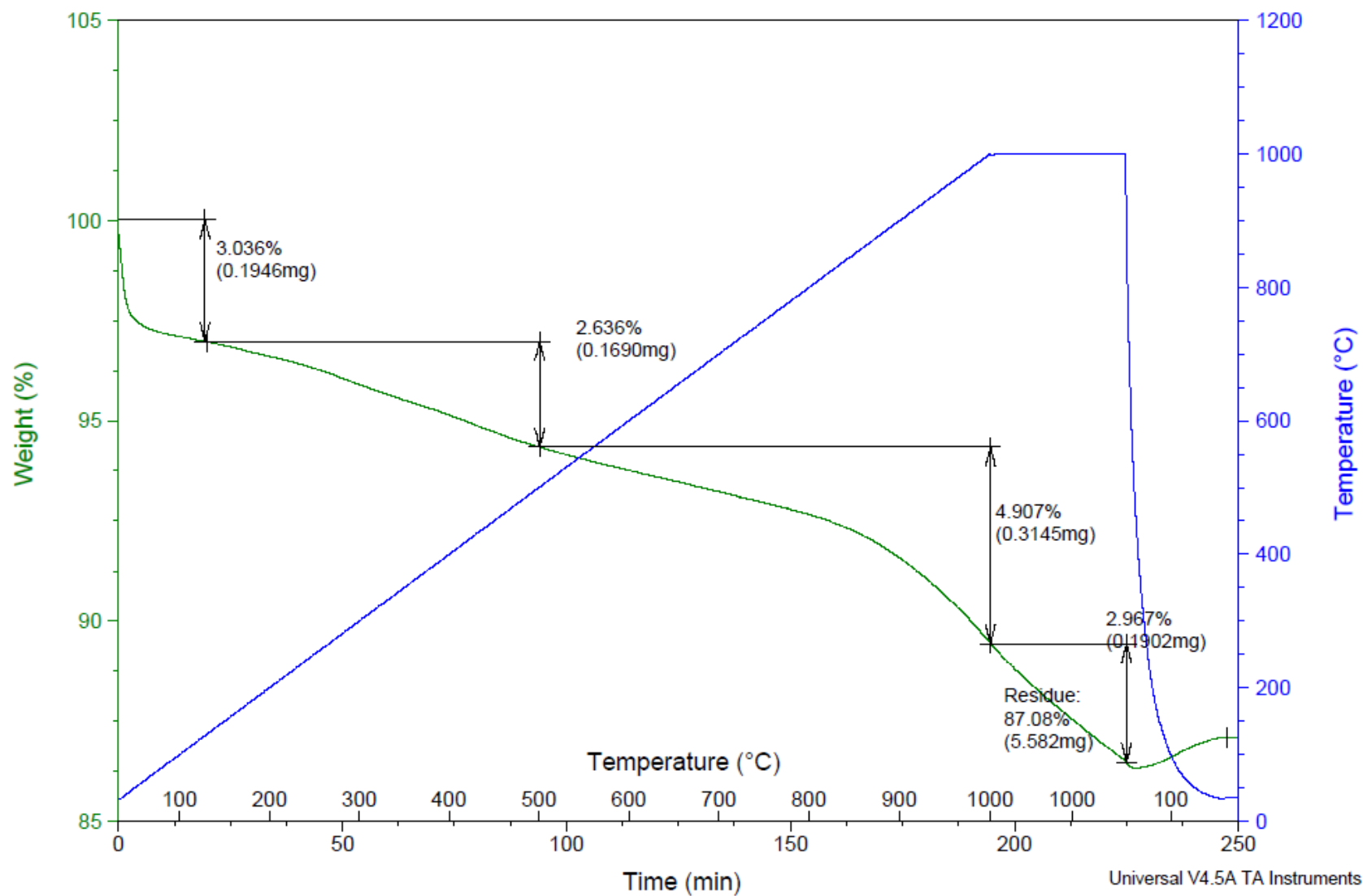
Sample: Proton Power 15-049  
Size: 10.5520 mg  
Method: Basic Carbon Ash

## TGA



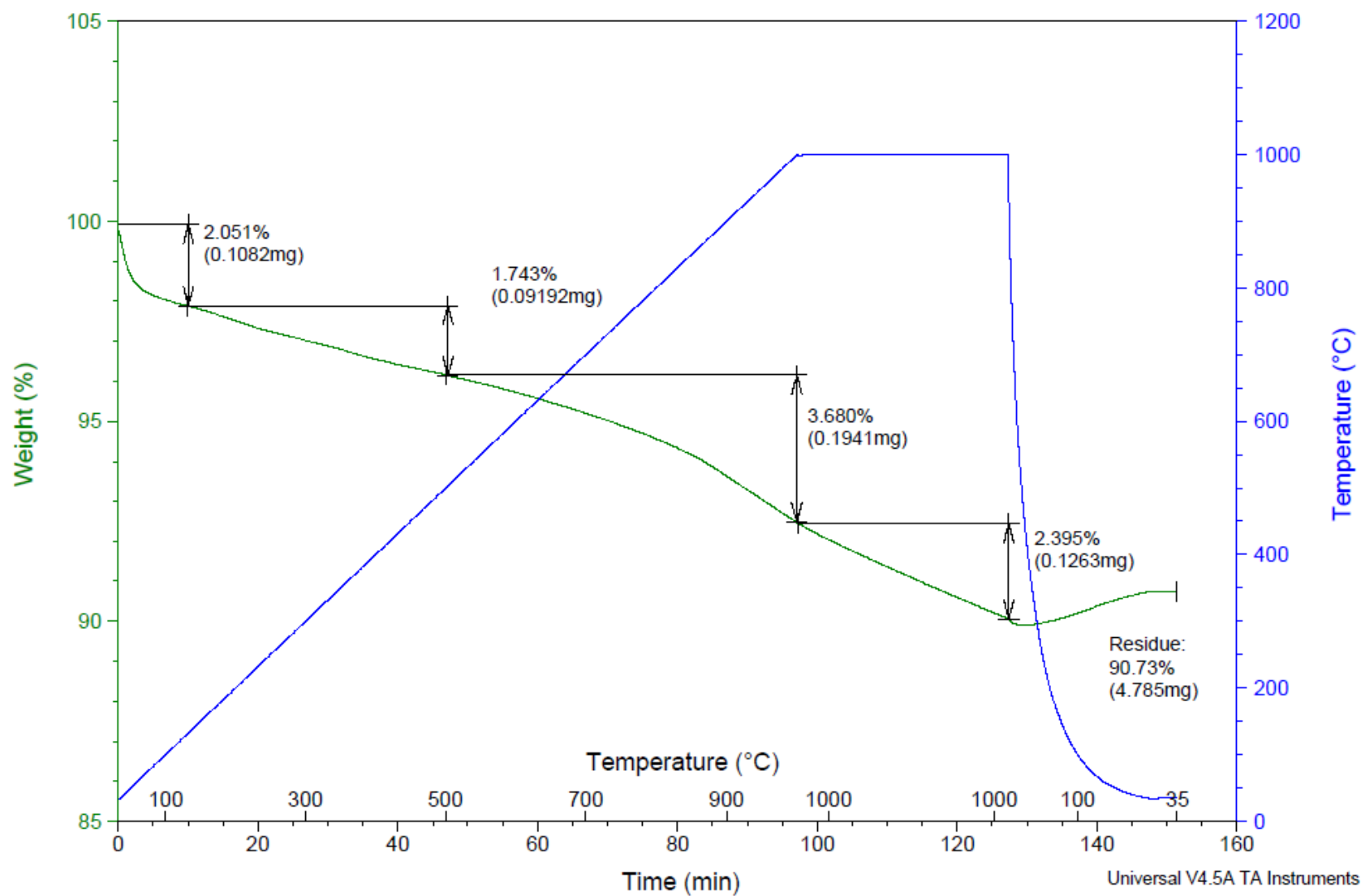
Sample: Sample-16-004 (Chestnut Oak)  
Size: 6.4100 mg  
Method: Heat Treatment

## TGA



Sample: Sample-16-005 (White Pine)  
Size: 5.2740 mg  
Method: Heat Treatment

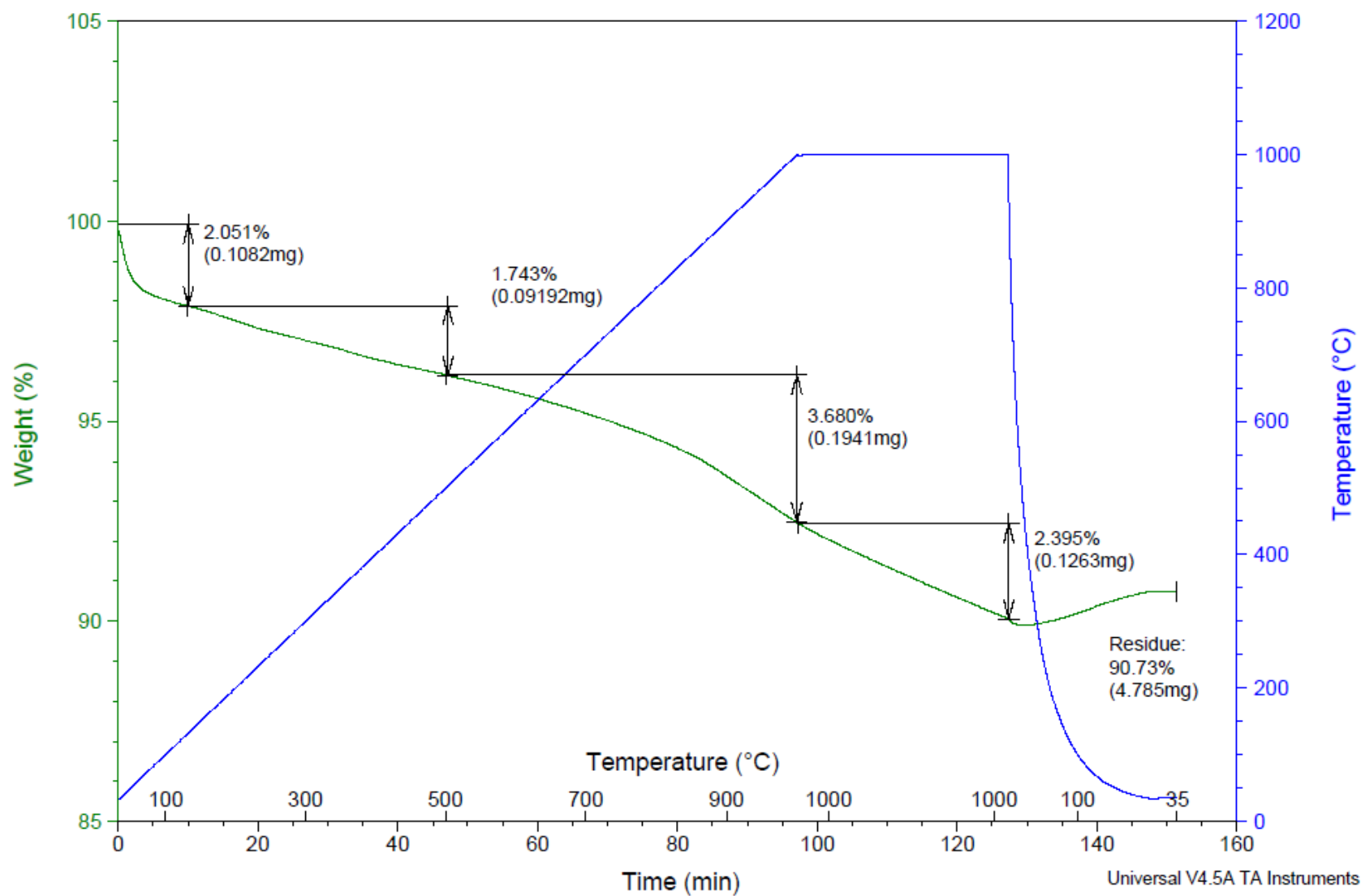
## TGA





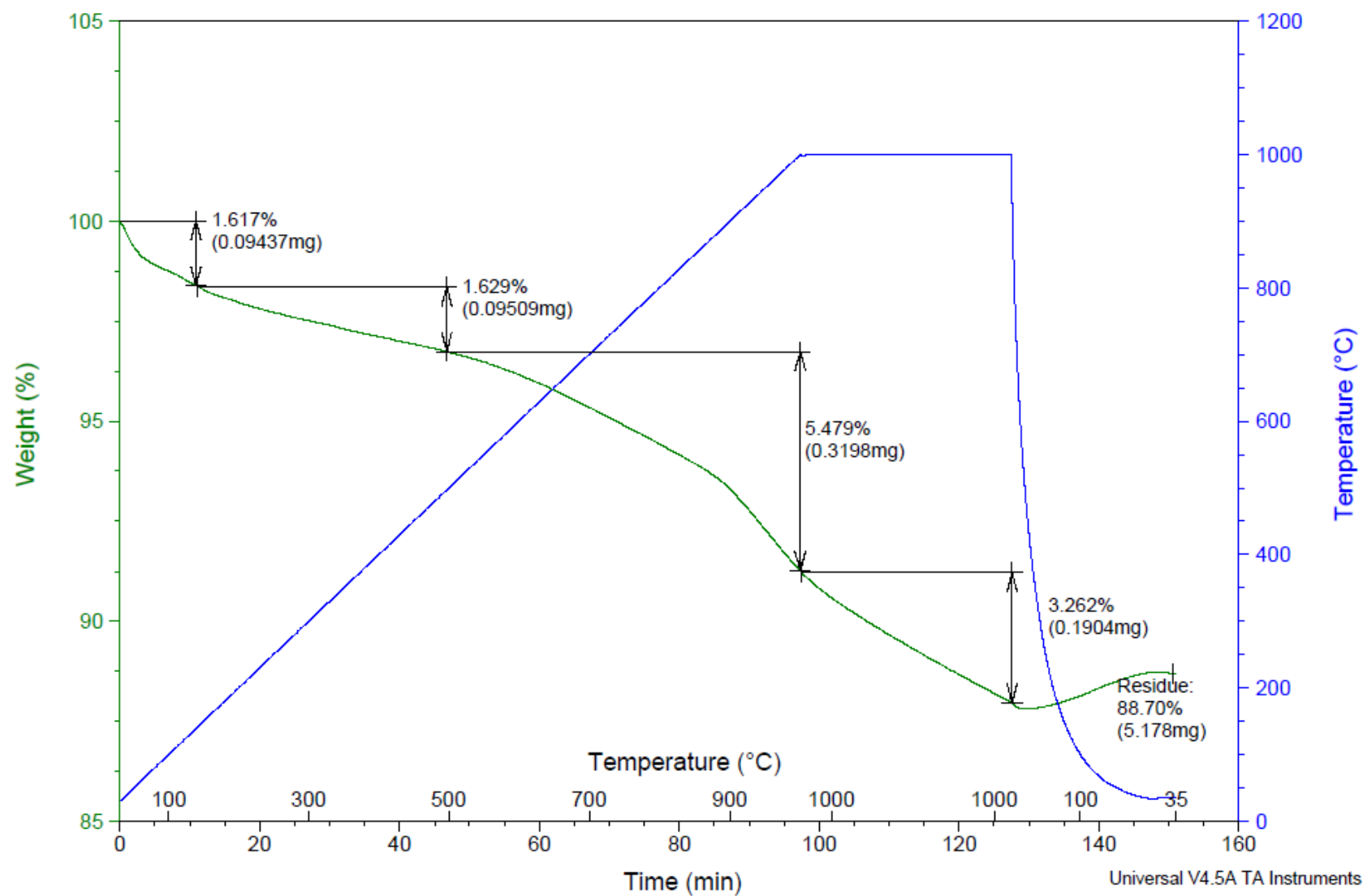
Sample: Sample-16-005 (White Pine)  
Size: 5.2740 mg  
Method: Heat Treatment

## TGA



Sample: Sample-16-006 (Yellow Poplar)  
Size: 5.8370 mg  
Method: Heat Treatment

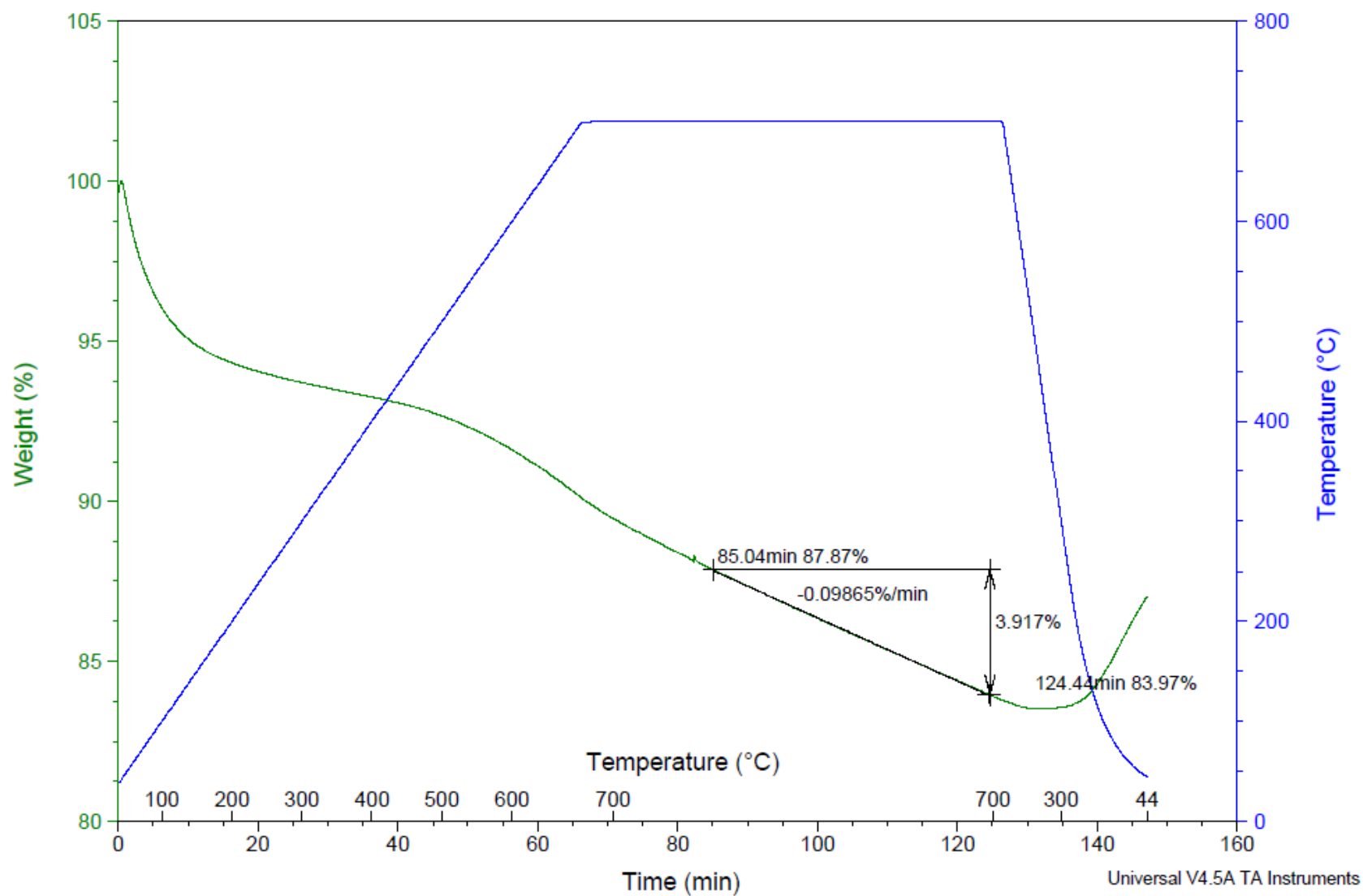
## TGA



**Simulation of physical activation by TGA in flowing carbon dioxide**

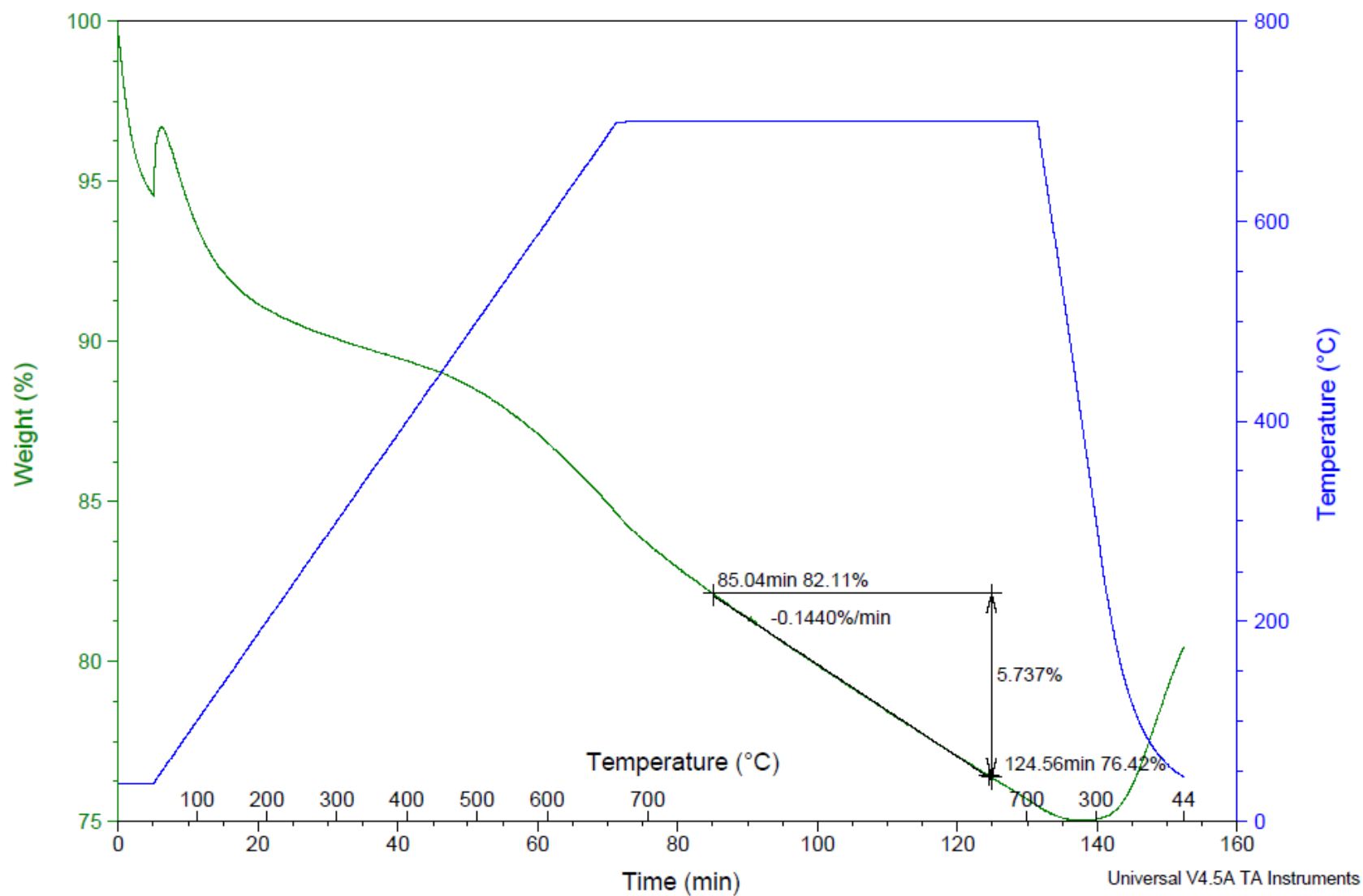
Sample: Proton Power 15-043  
Size: 10.5480 mg  
Method: Activation test 700 C - 60 min

## TGA



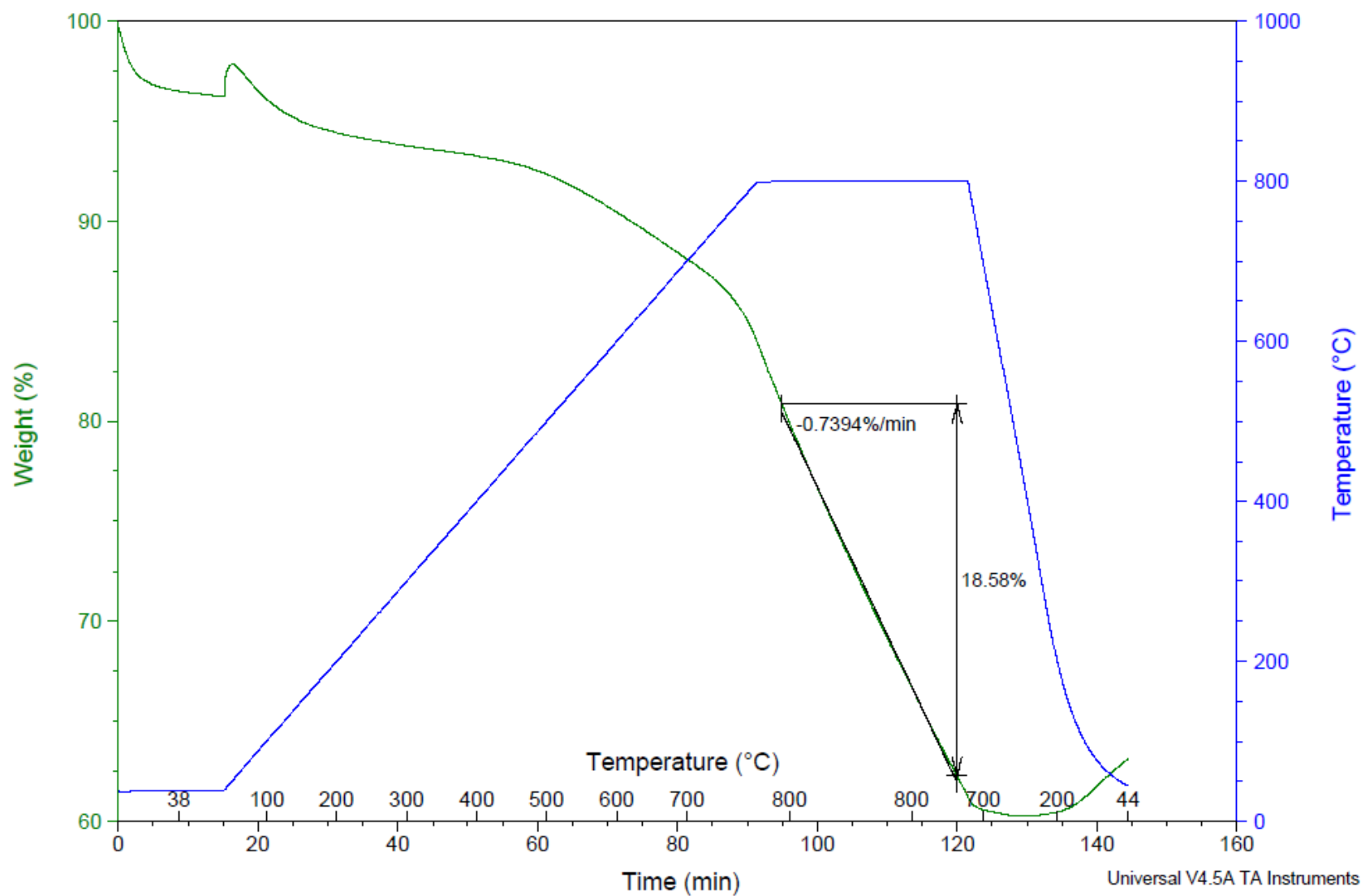
Sample: Proton Power 15-043  
Size: 7.2150 mg  
Method: Activation test 700 C - 60 min

## TGA



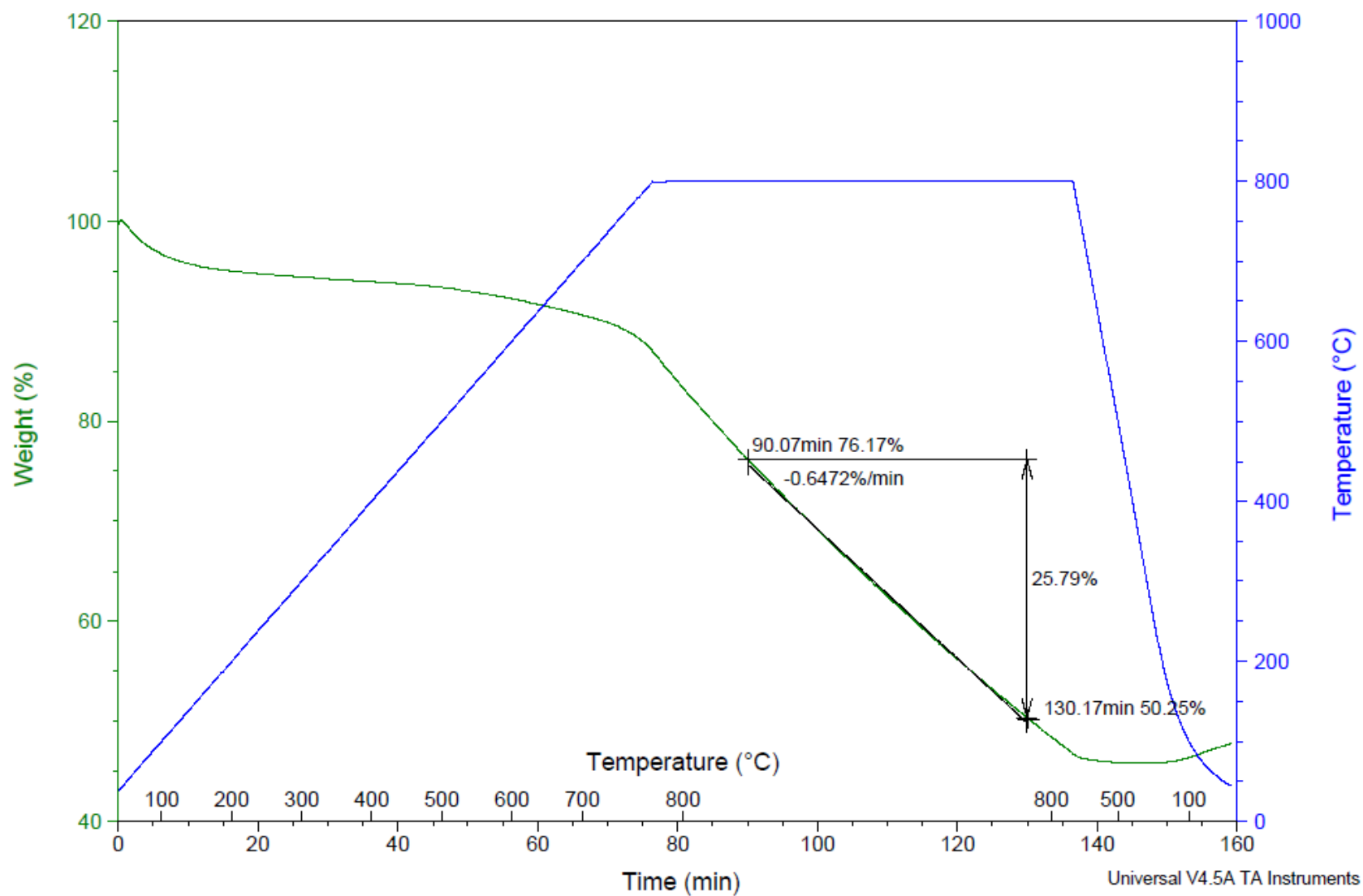
Sample: Proton Power 15-043  
Size: 9.9730 mg  
Method: Activation test to 800 C

## TGA

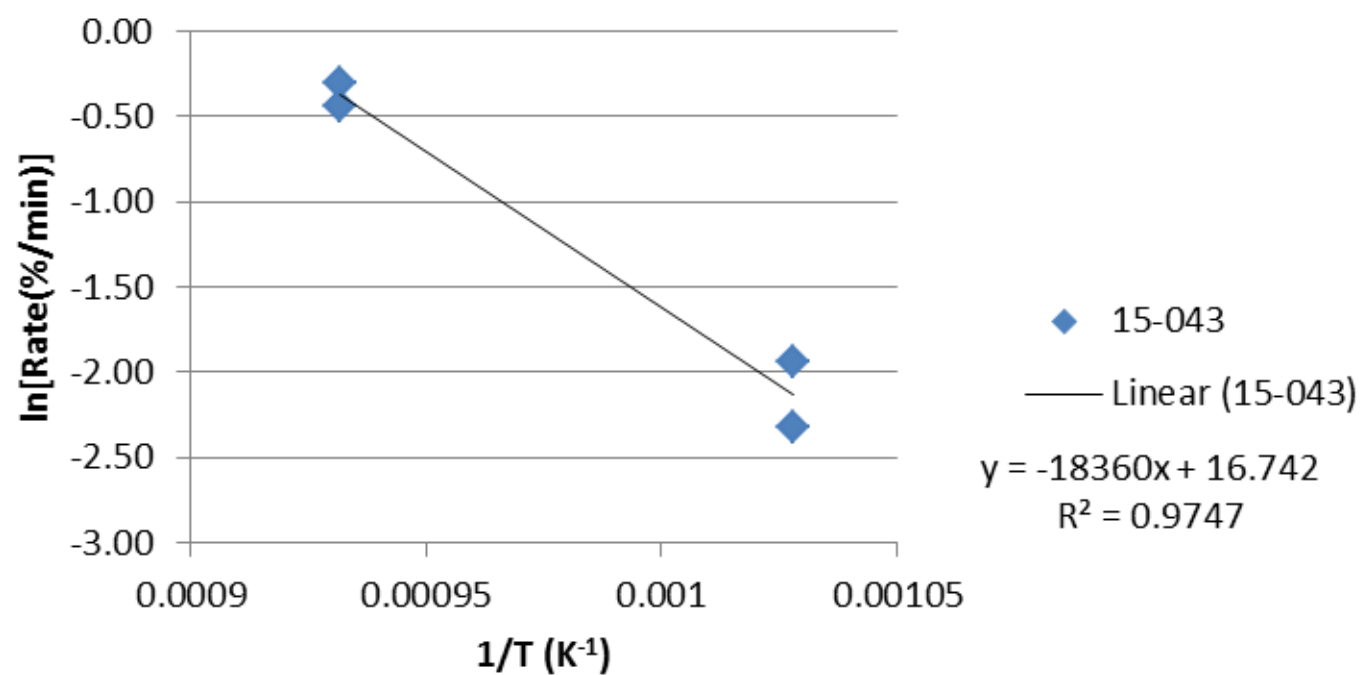


Sample: Proton Power 15-043  
Size: 8.7130 mg  
Method: Activation test2 800 C - 60 min

## TGA



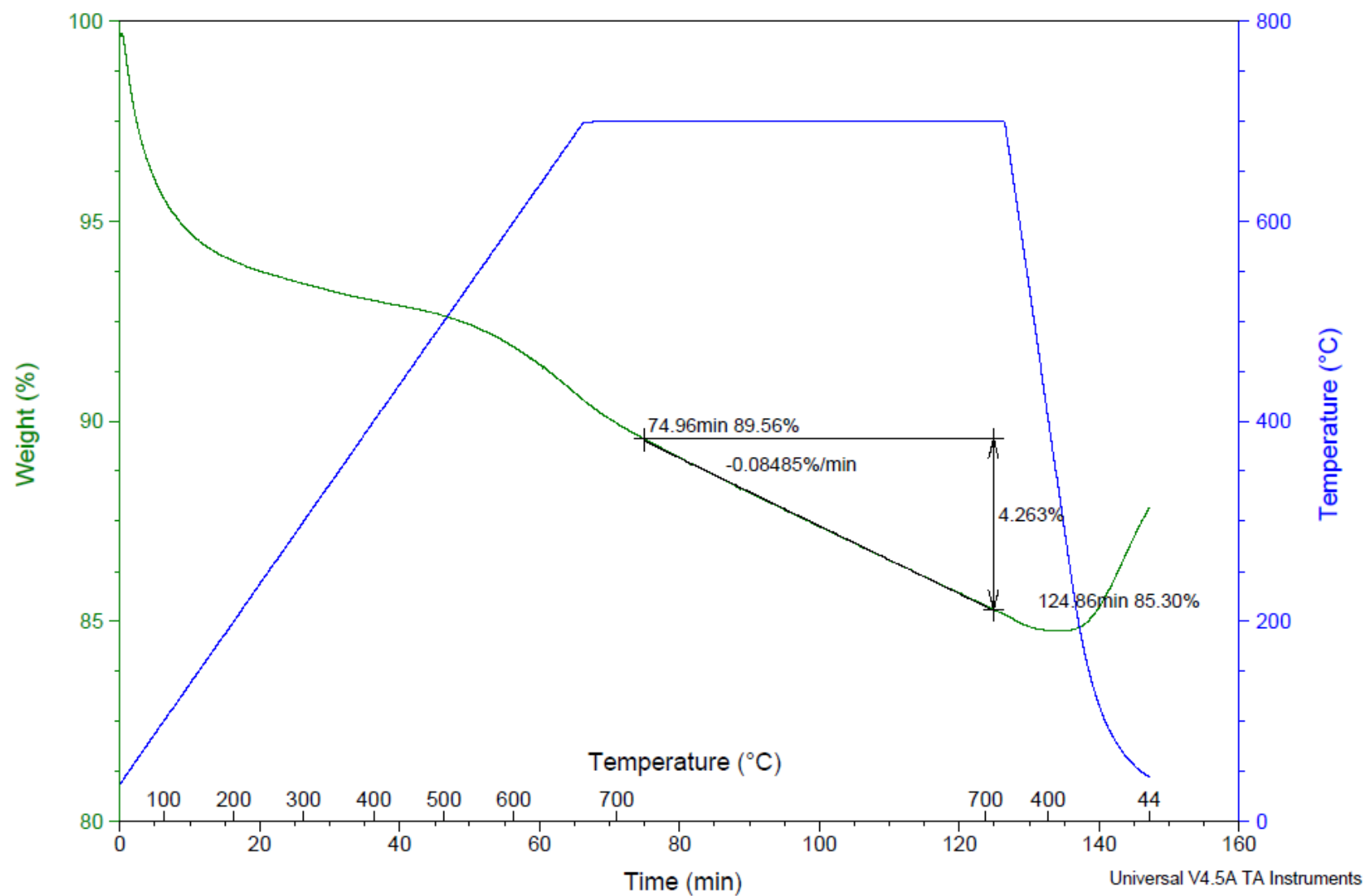
**15-043**





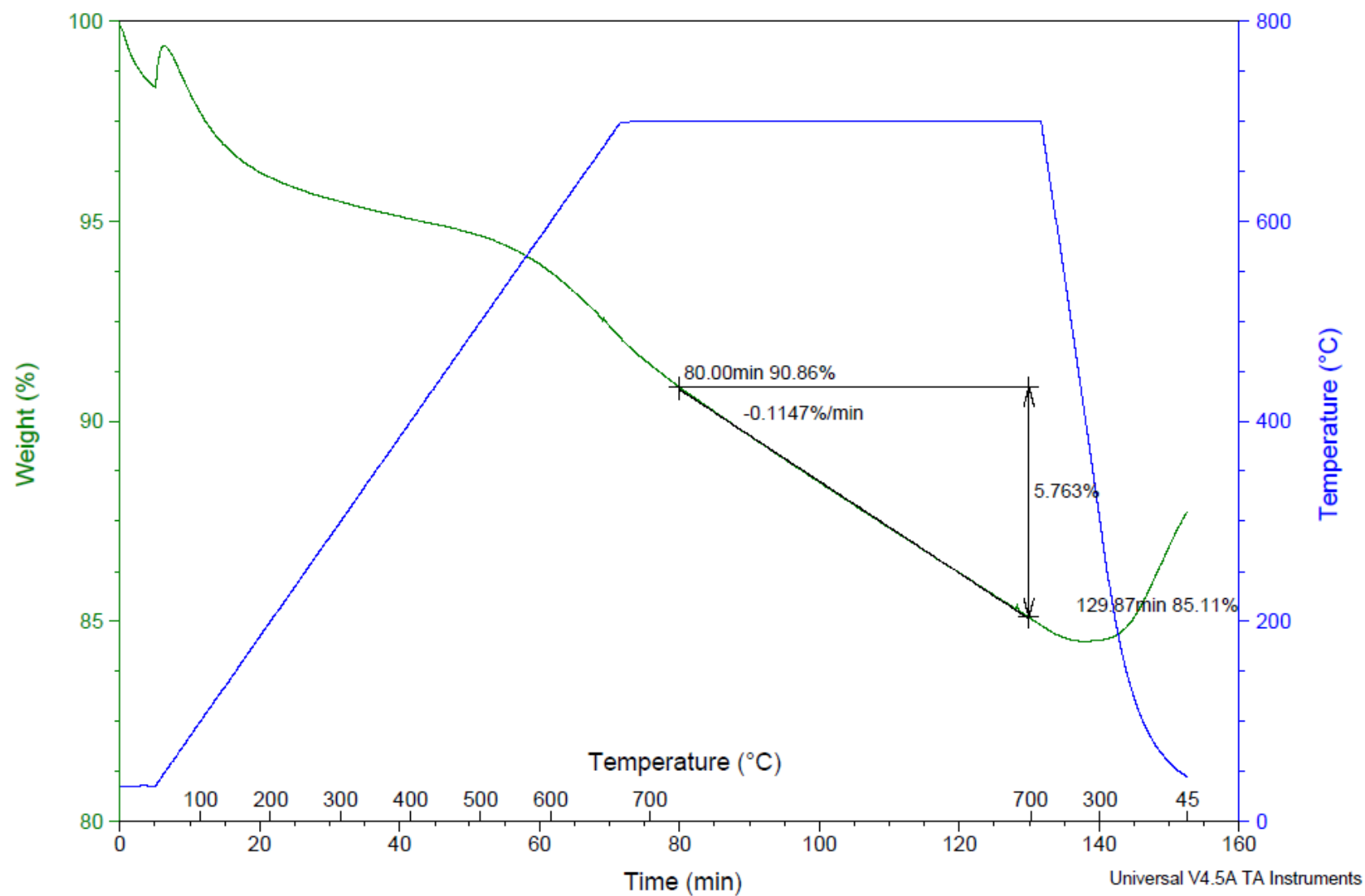
Sample: Proton Power 15-044  
Size: 9.6100 mg  
Method: Activation test 700 C - 60 min

## TGA



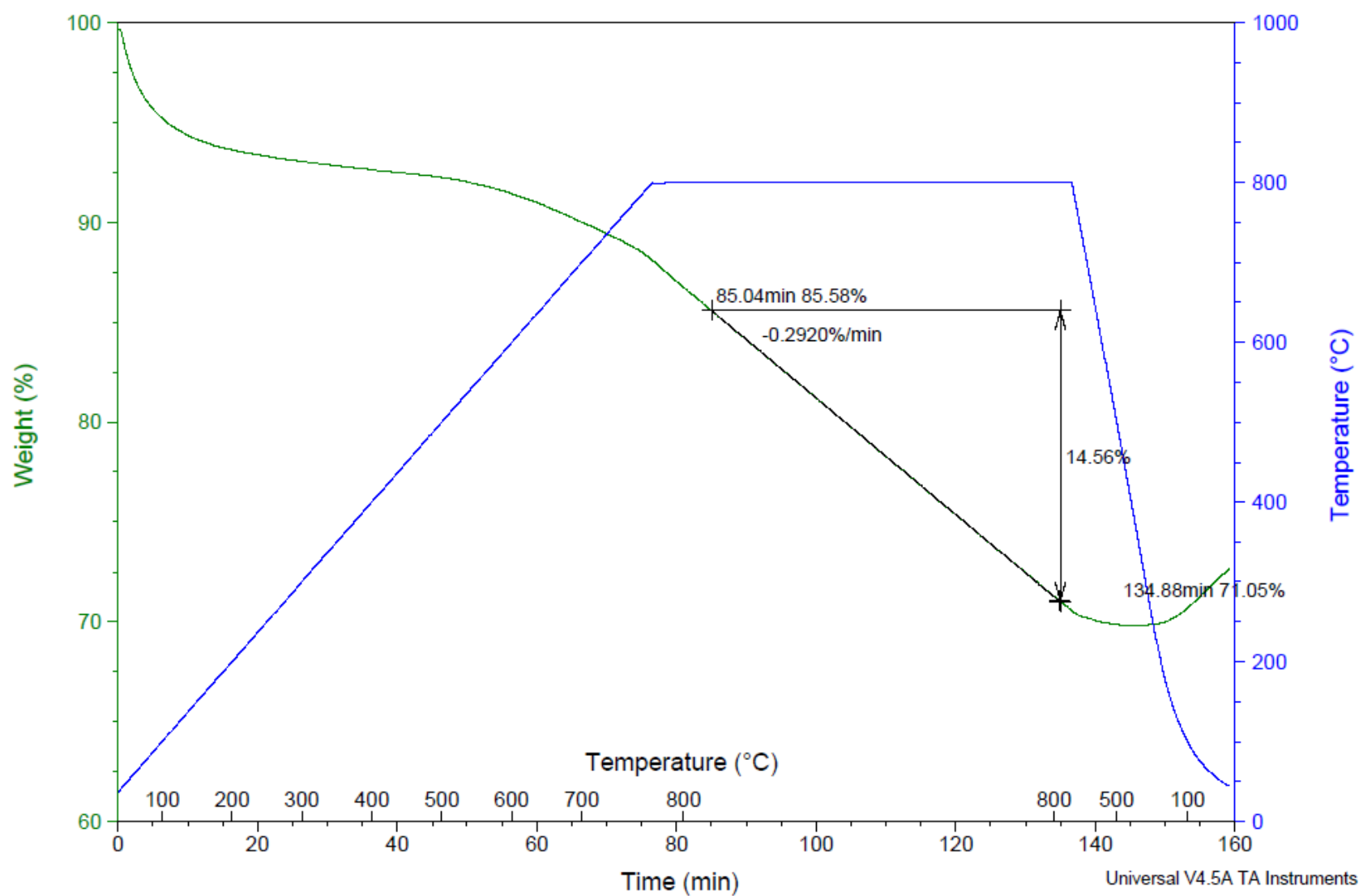
Sample: Proton Power 15-044  
Size: 6.8800 mg  
Method: Activation test 700 C - 60 min

## TGA



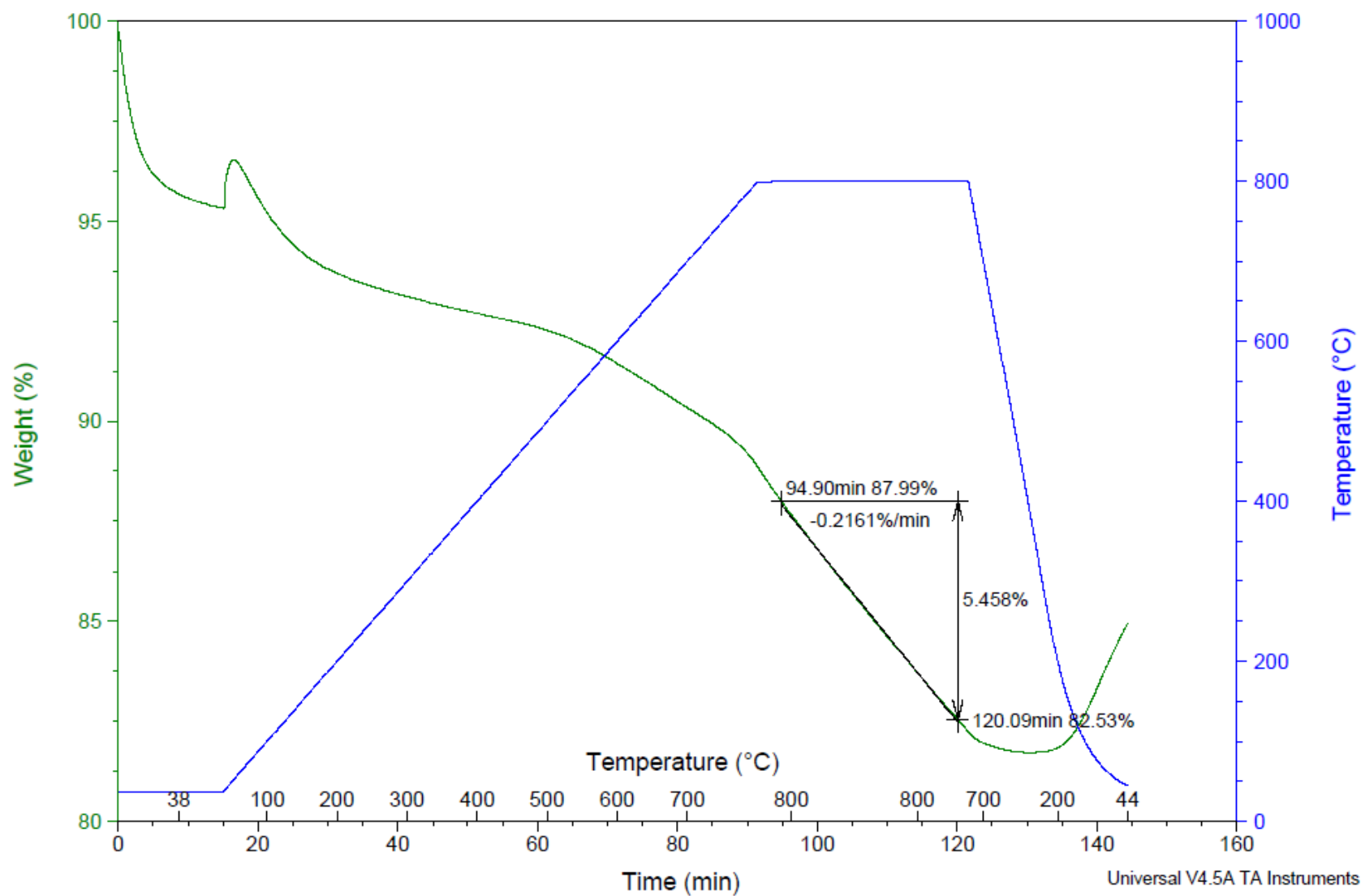
Sample: Proton Power 15-044  
Size: 9.1810 mg  
Method: Activation test2 800 C - 60 min

## TGA

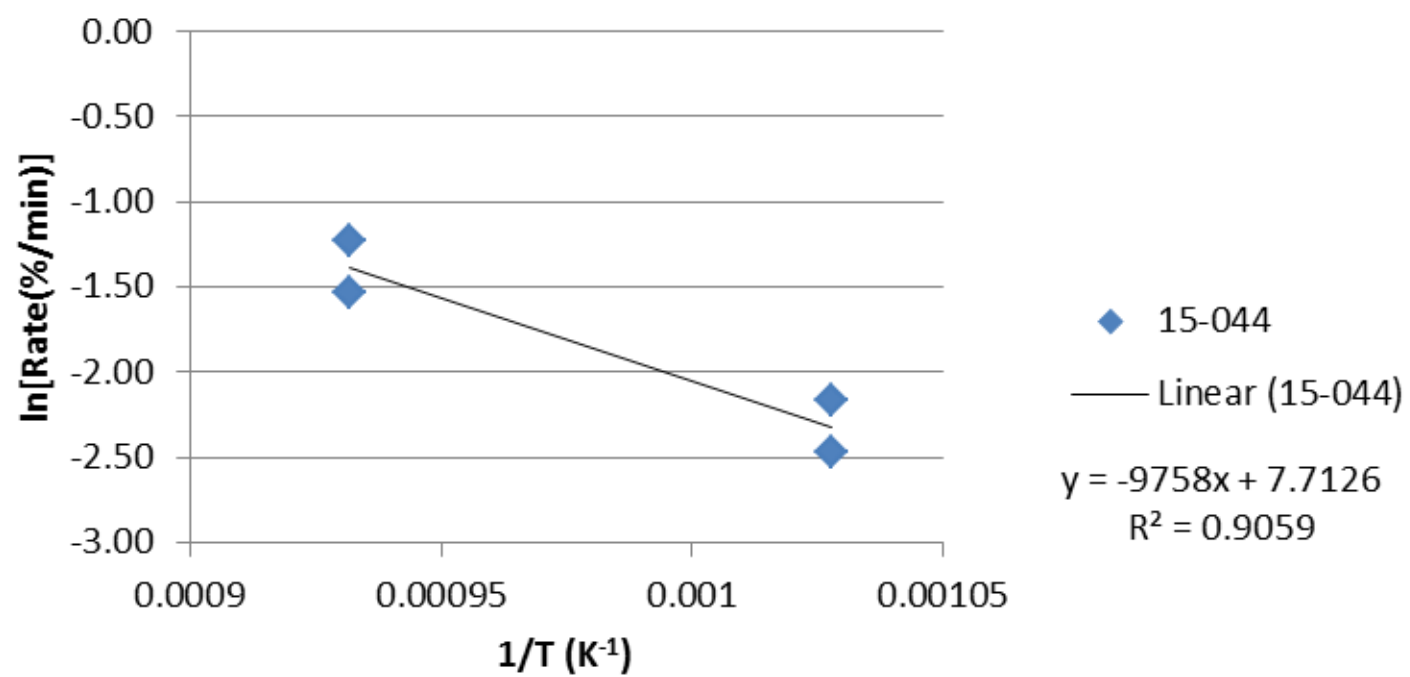


Sample: Proton Power 15-044  
Size: 12.3100 mg  
Method: Activation test to 800 C

## TGA

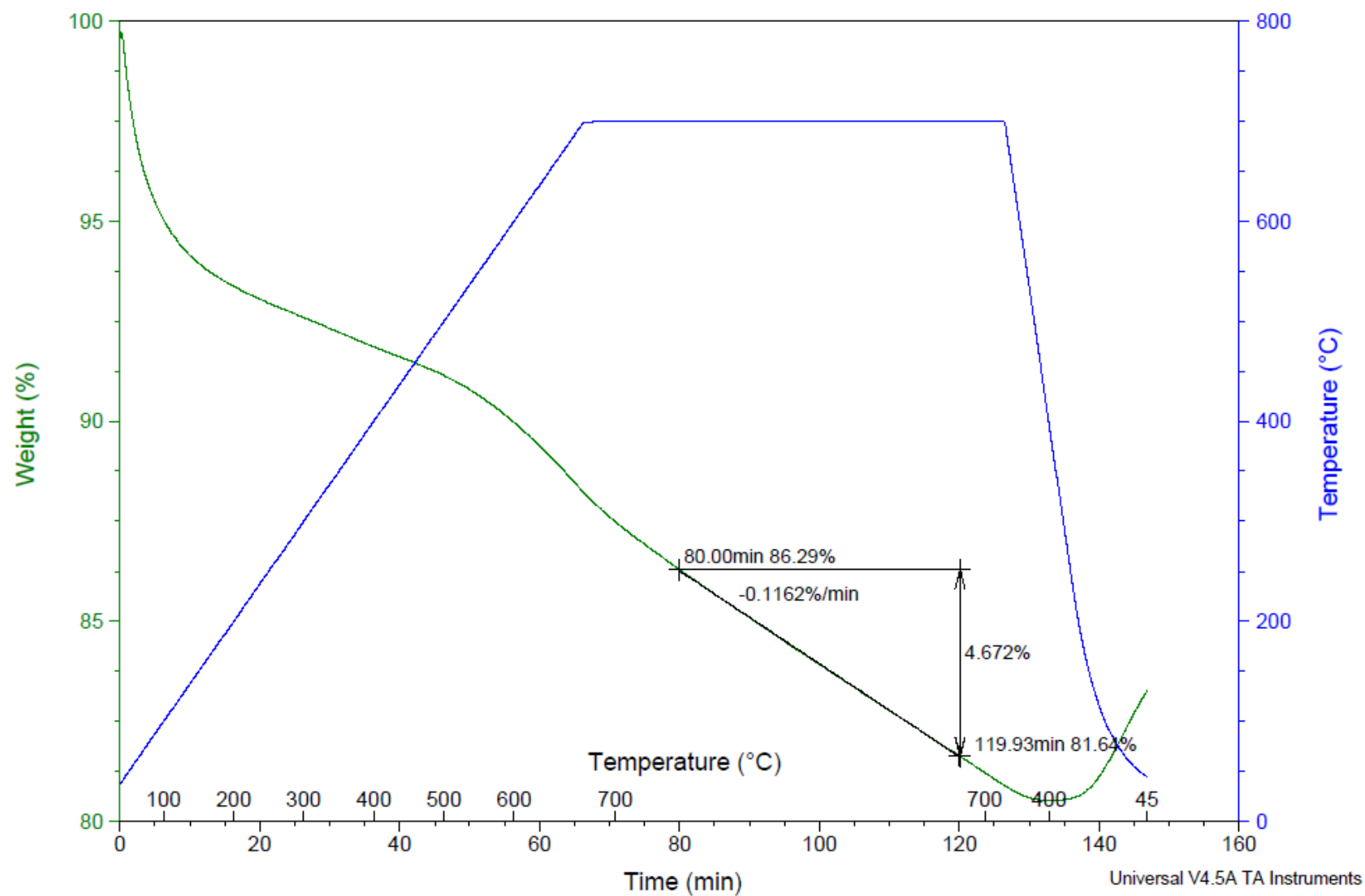


**15-044**



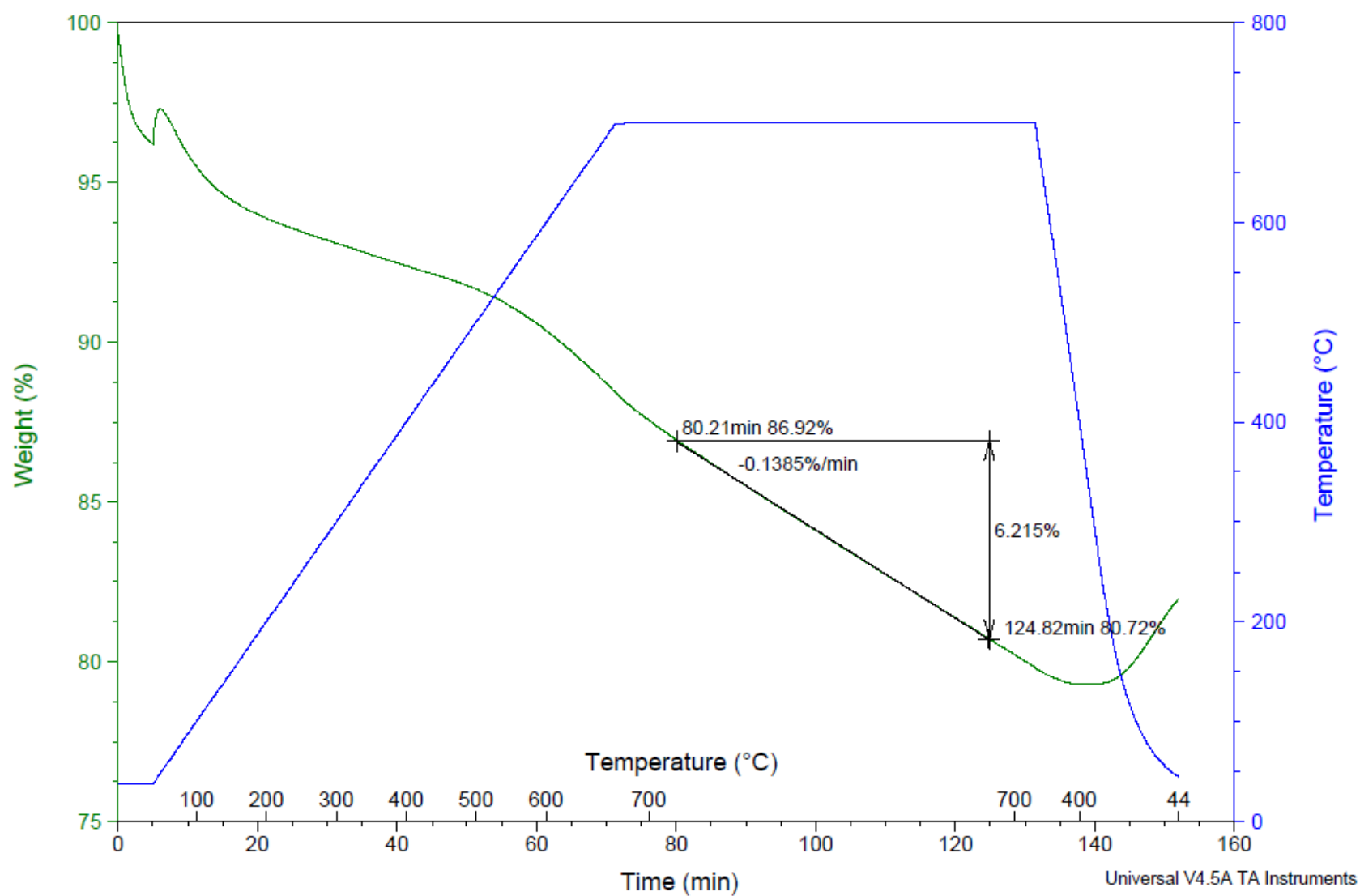
Sample: Proton Power 15-045  
Size: 8.6210 mg  
Method: Activation test 700 C - 60 min

## TGA



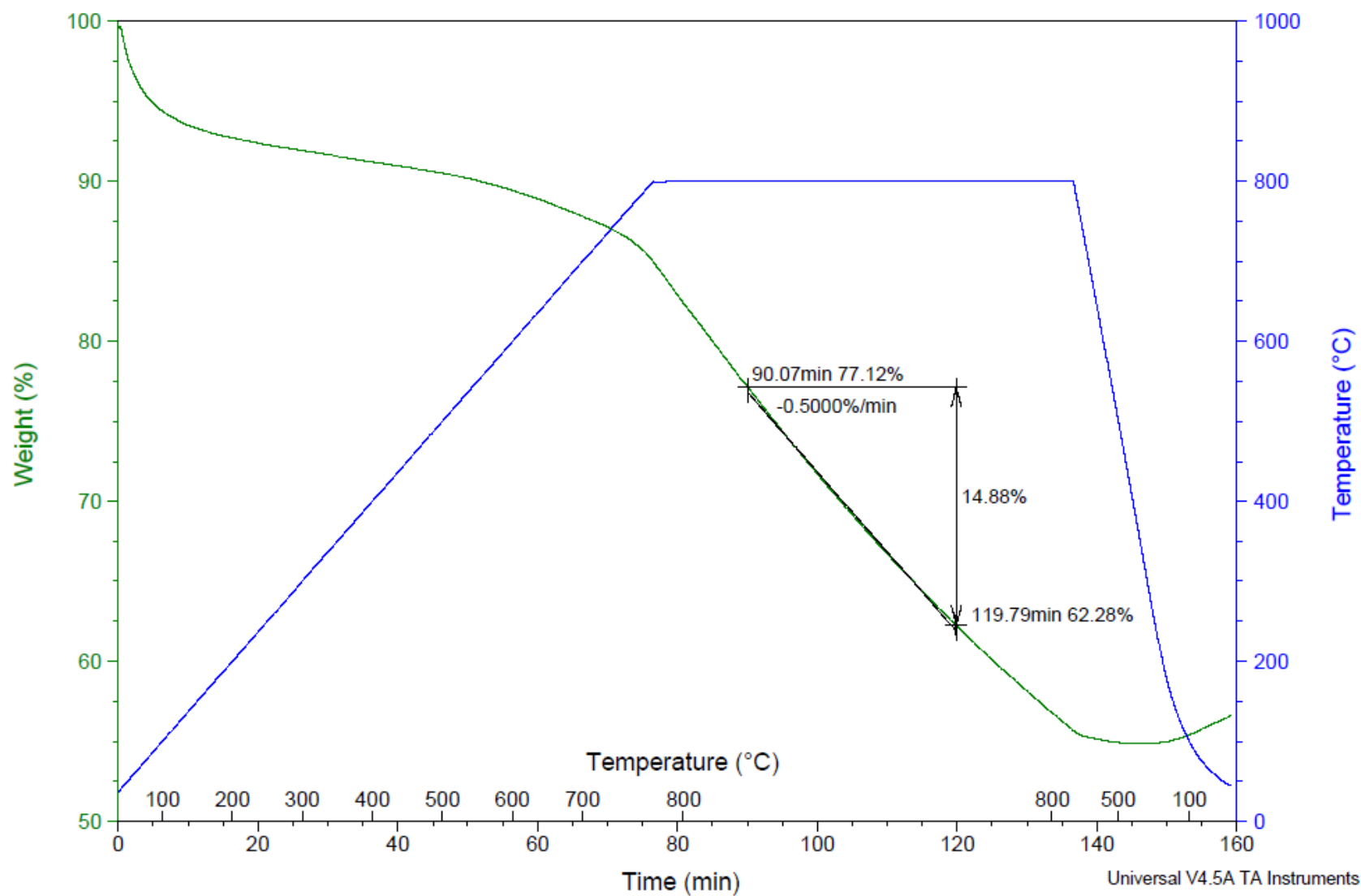
Sample: Proton Power 15-045  
Size: 6.5030 mg  
Method: Activation test 700 C - 60 min

## TGA



Sample: Proton Power 15-045  
Size: 10.3480 mg  
Method: Activation test2 800 C - 60 min

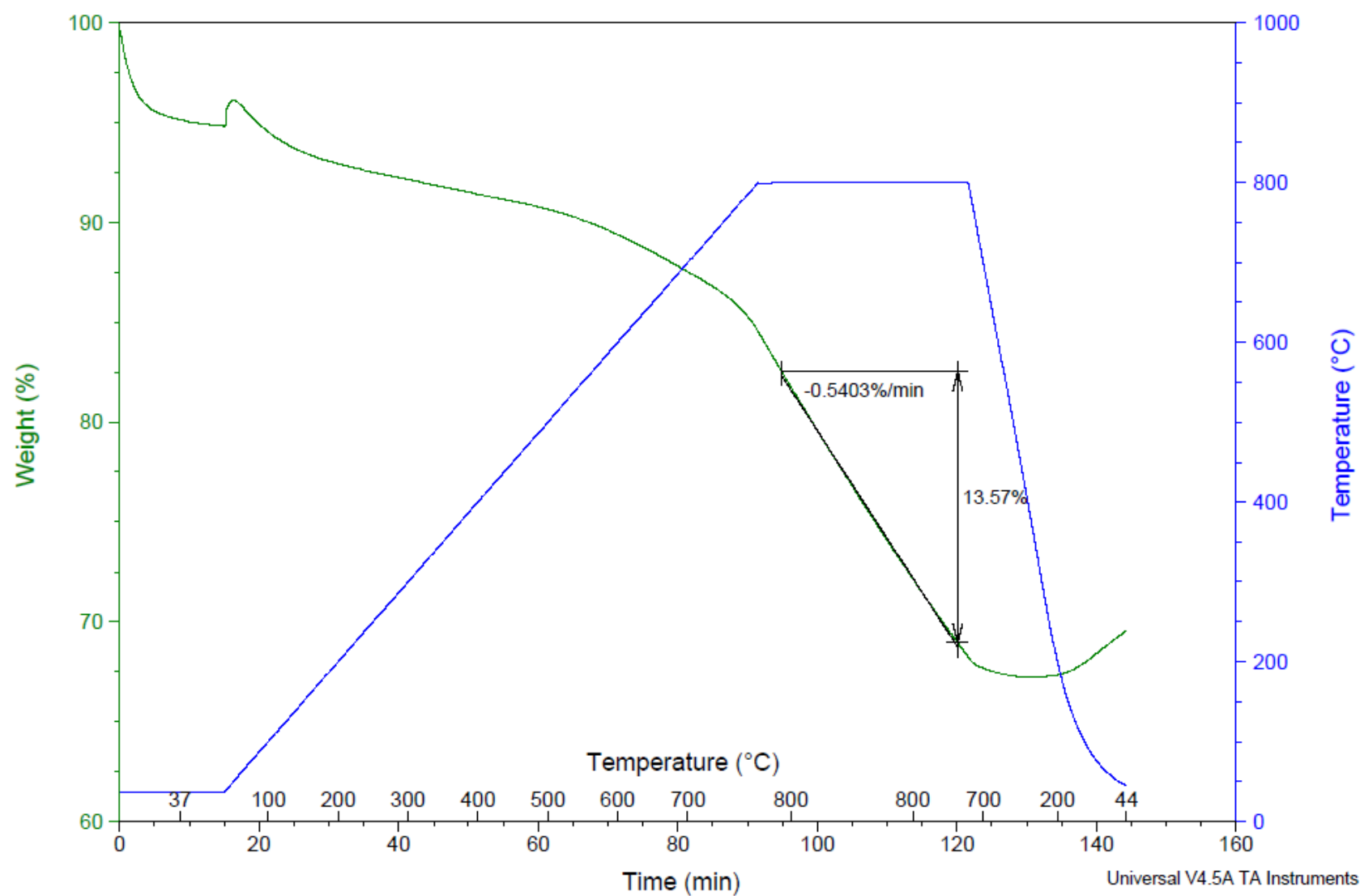
## TGA



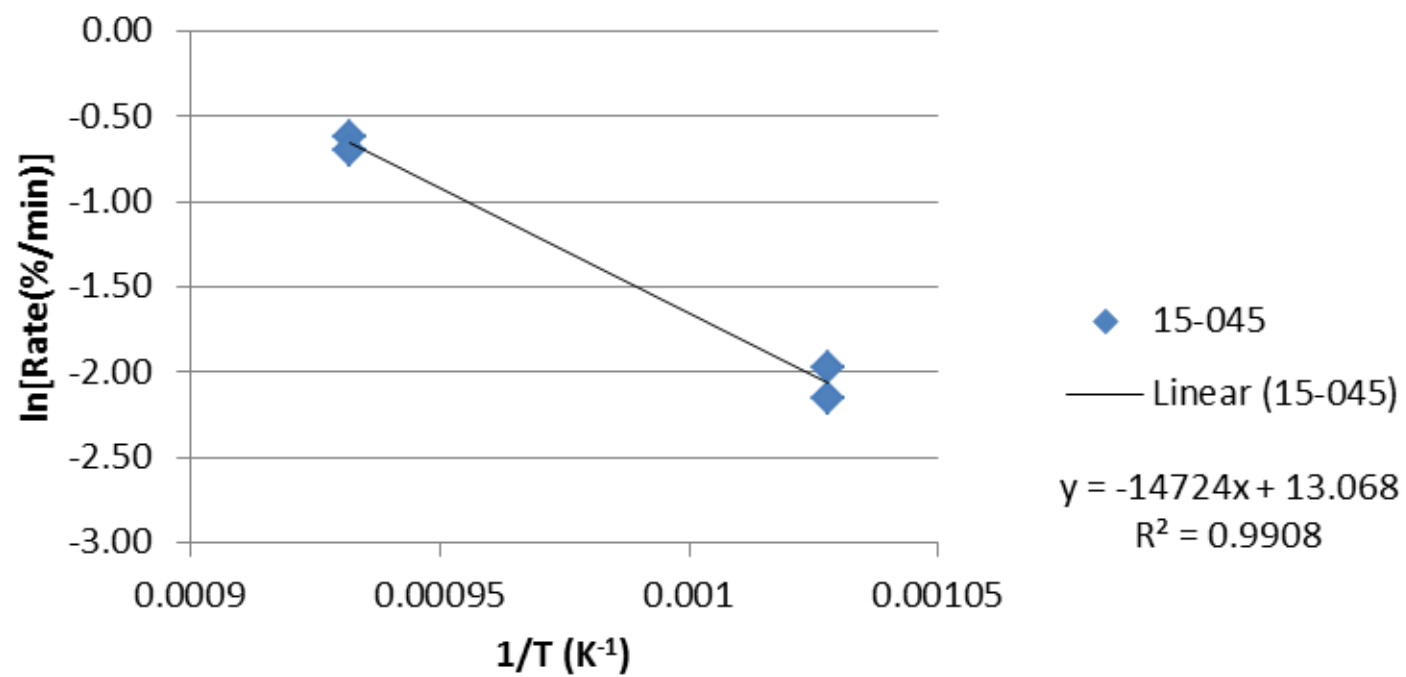


Sample: Proton Power 15-045  
Size: 8.6980 mg  
Method: Activation test to 800 C

## TGA

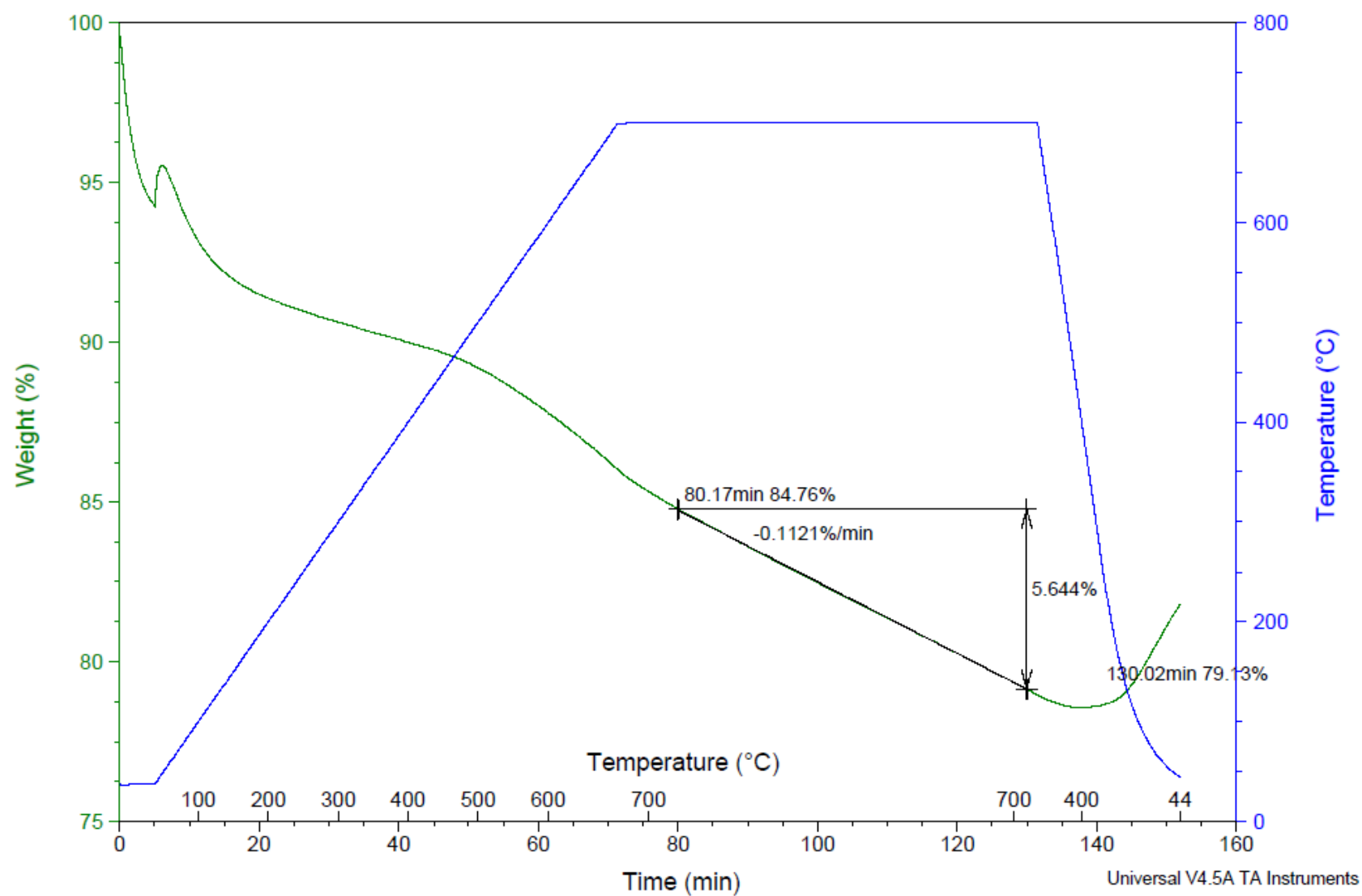


**15-045**



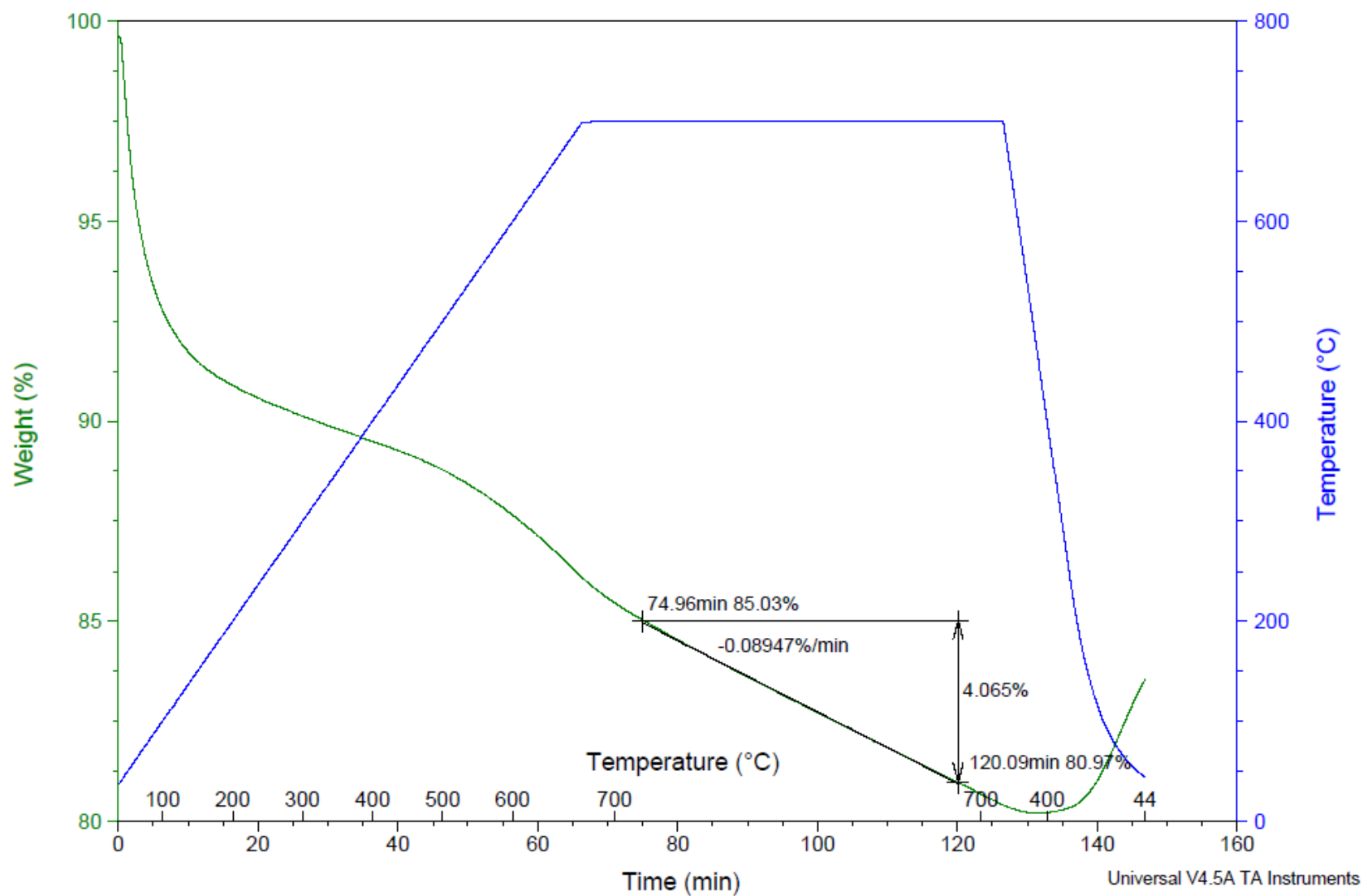
Sample: Proton Power 15-046  
Size: 8.2210 mg  
Method: Activation test 700 C - 60 min

## TGA



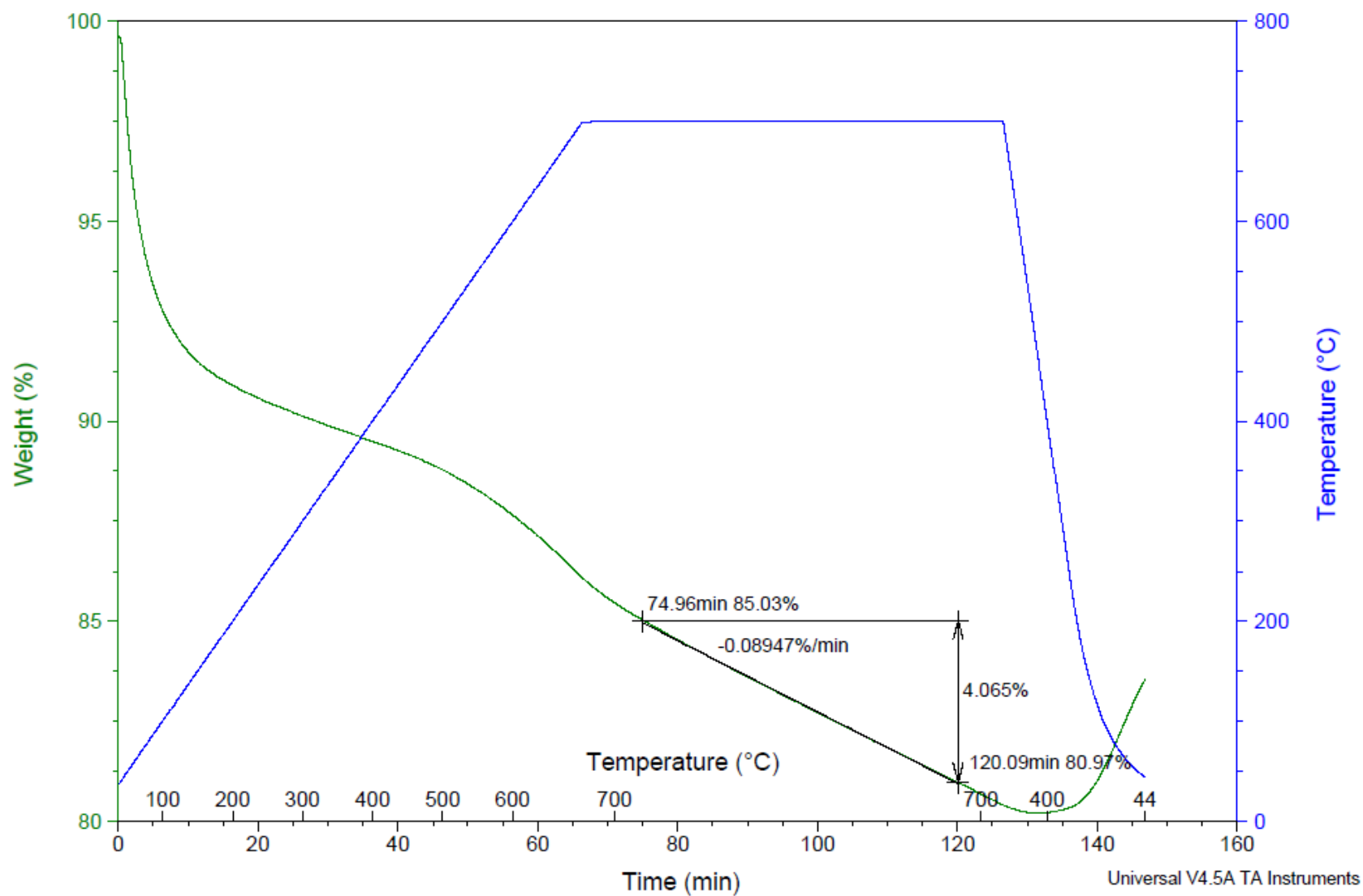
Sample: Proton Power 15-046  
Size: 11.7700 mg  
Method: Activation test 700 C - 60 min

## TGA



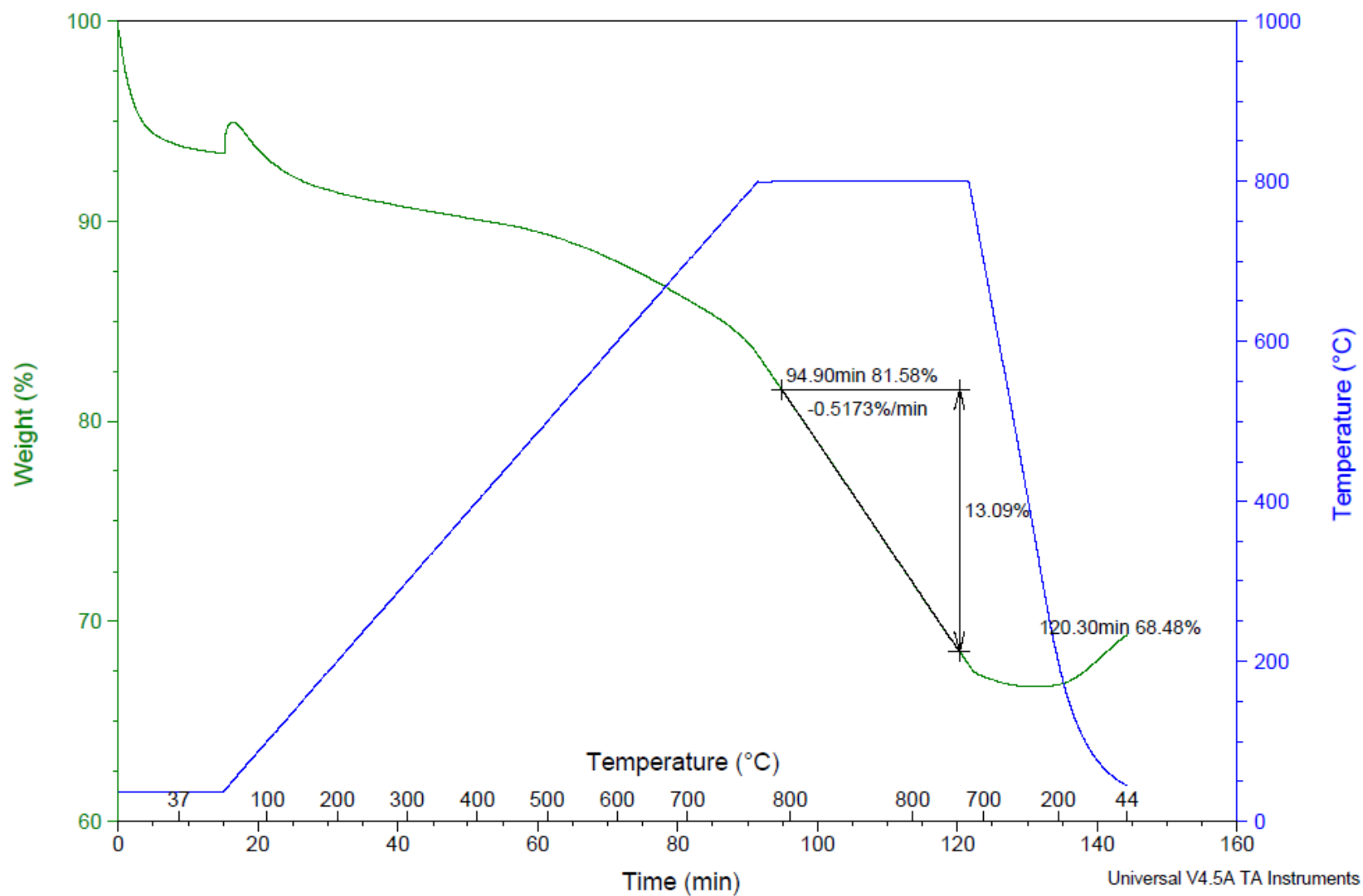
Sample: Proton Power 15-046  
Size: 11.7700 mg  
Method: Activation test 700 C - 60 min

## TGA



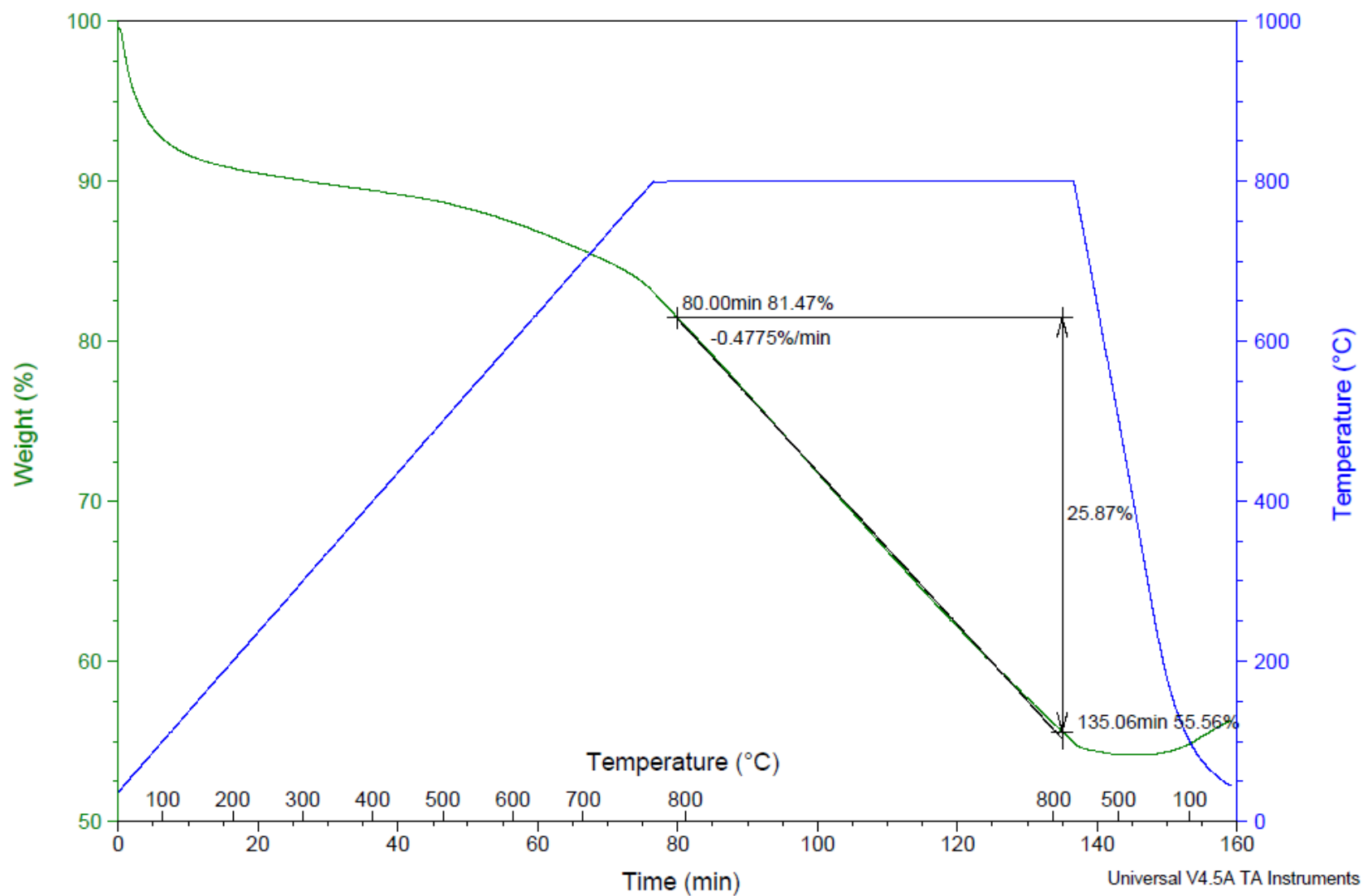
Sample: Proton Power 15-046  
Size: 8.2780 mg  
Method: Activation test to 800 C

## TGA

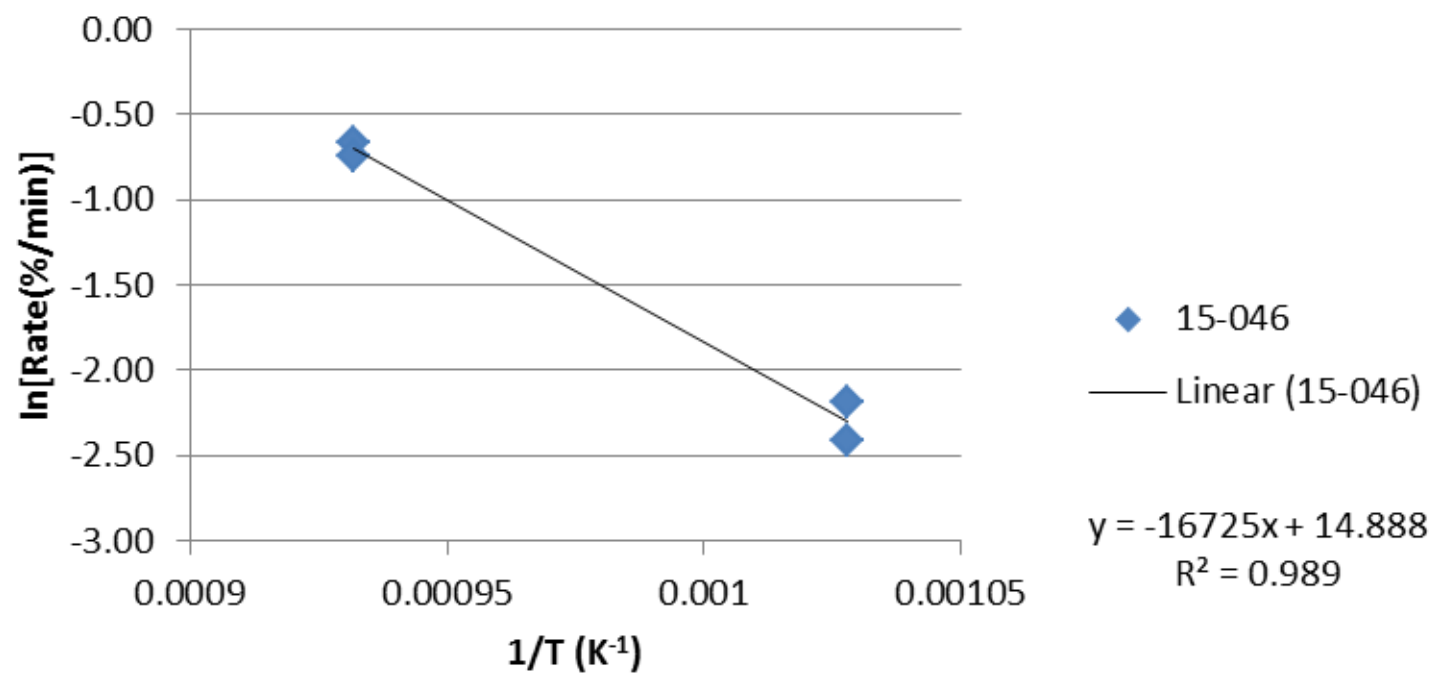


Sample: Proton Power 15-046  
Size: 9.7800 mg  
Method: Activation test2 800 C - 60 min

## TGA



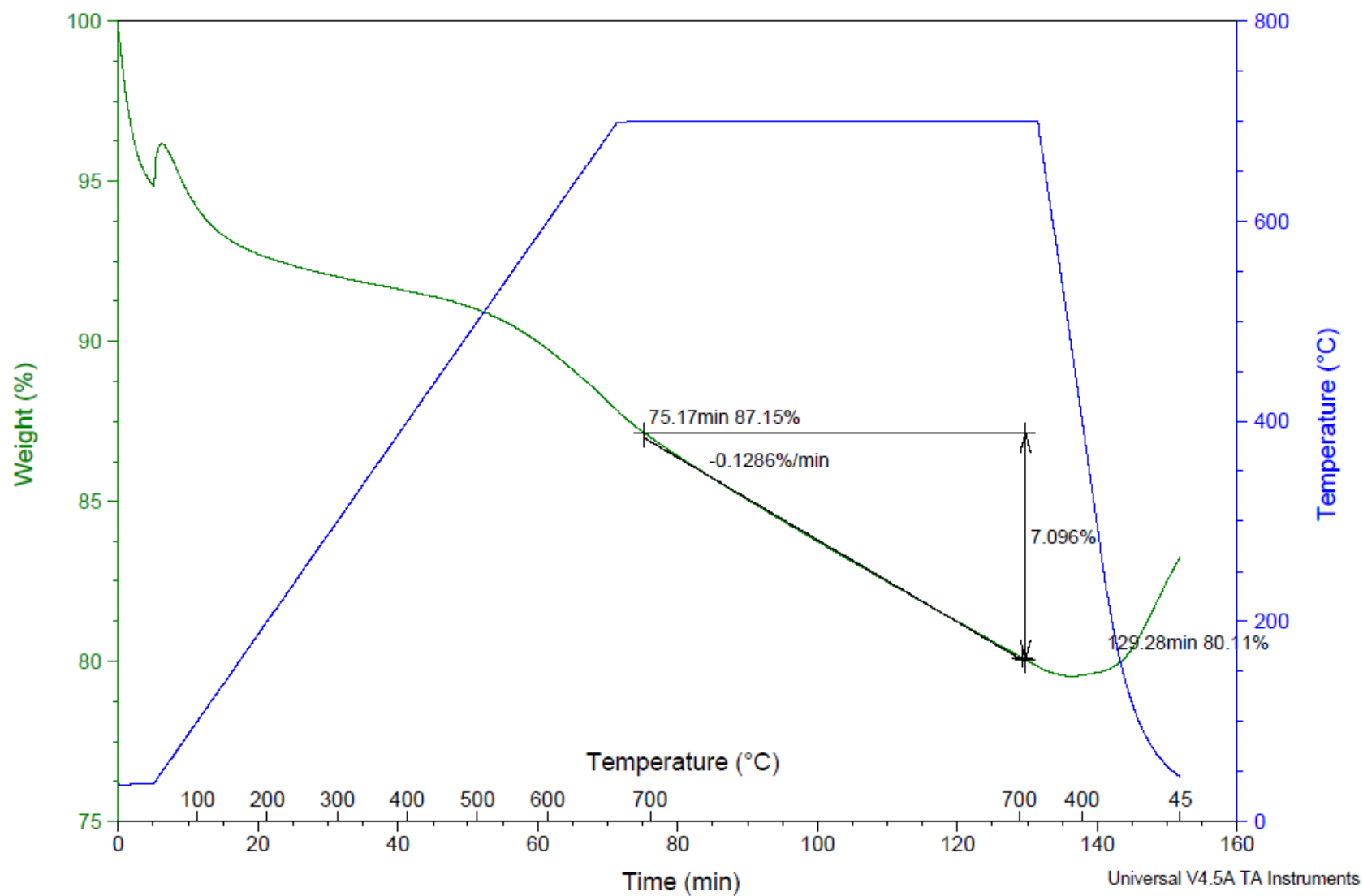
**15-046**





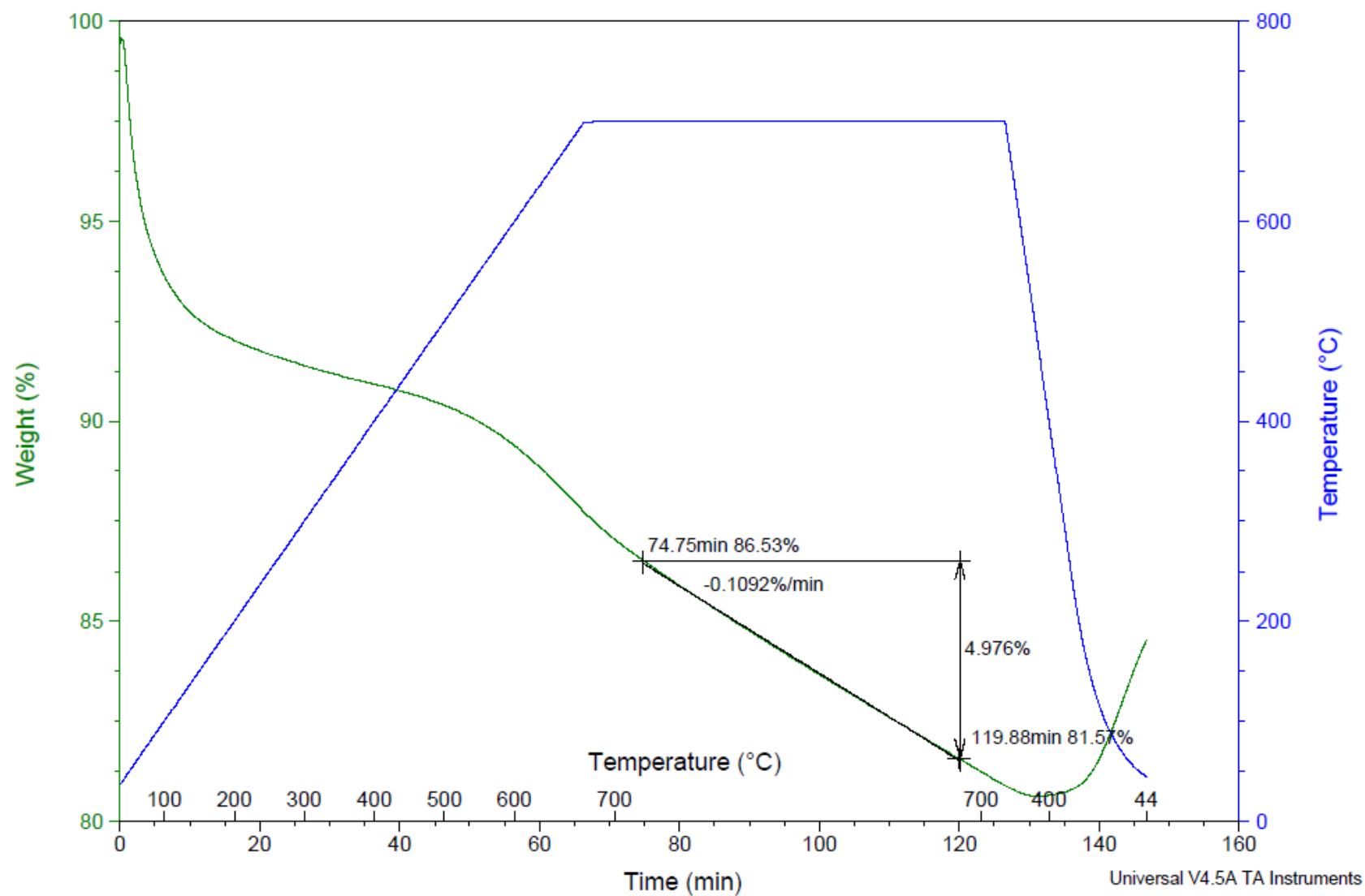
Sample: Proton Power 15-049  
Size: 8.6920 mg  
Method: Activation test 700 C - 60 min

## TGA



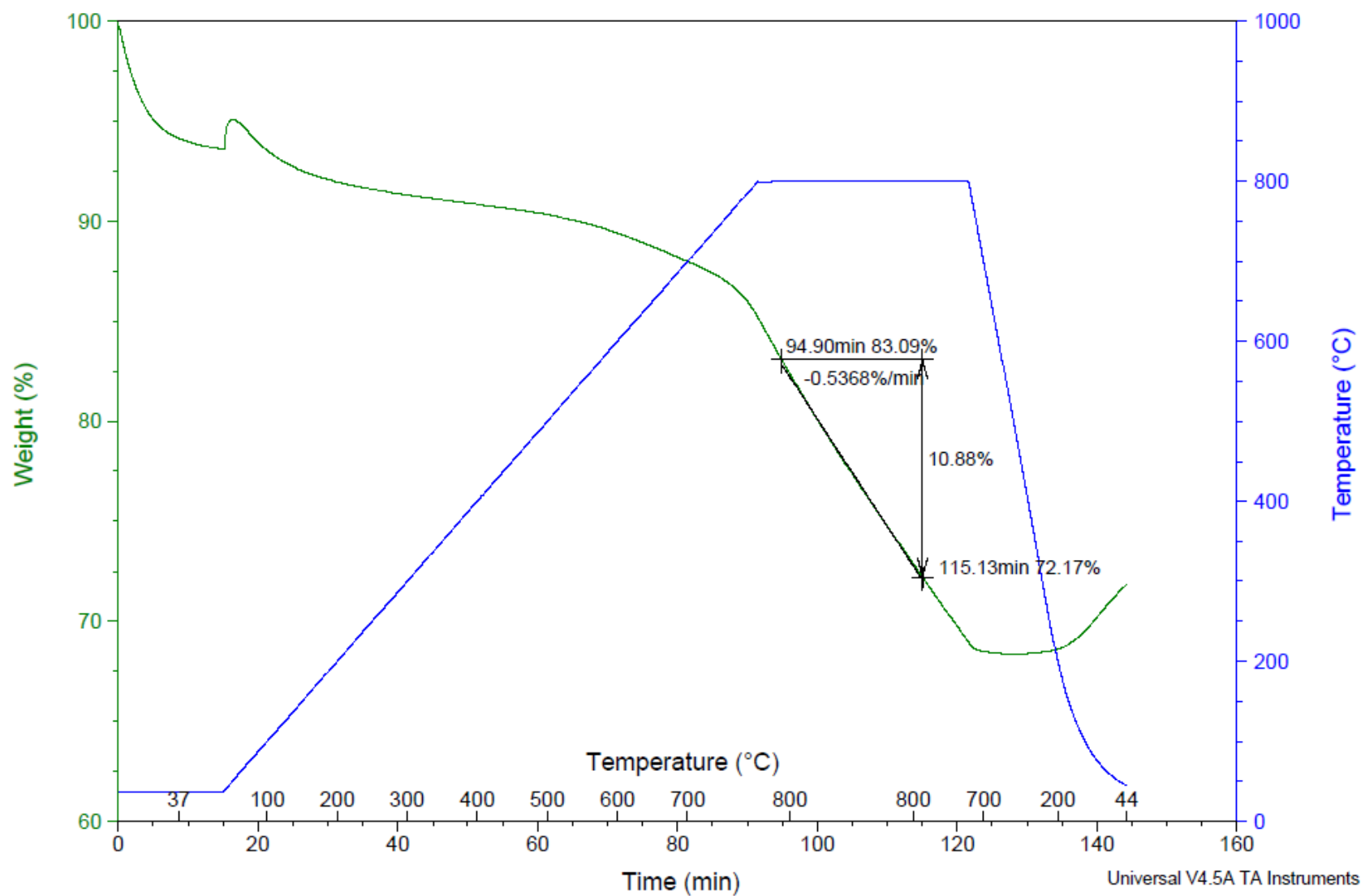
Sample: Proton Power 15-049  
Size: 9.6540 mg  
Method: Activation test 700 C - 60 min

## TGA



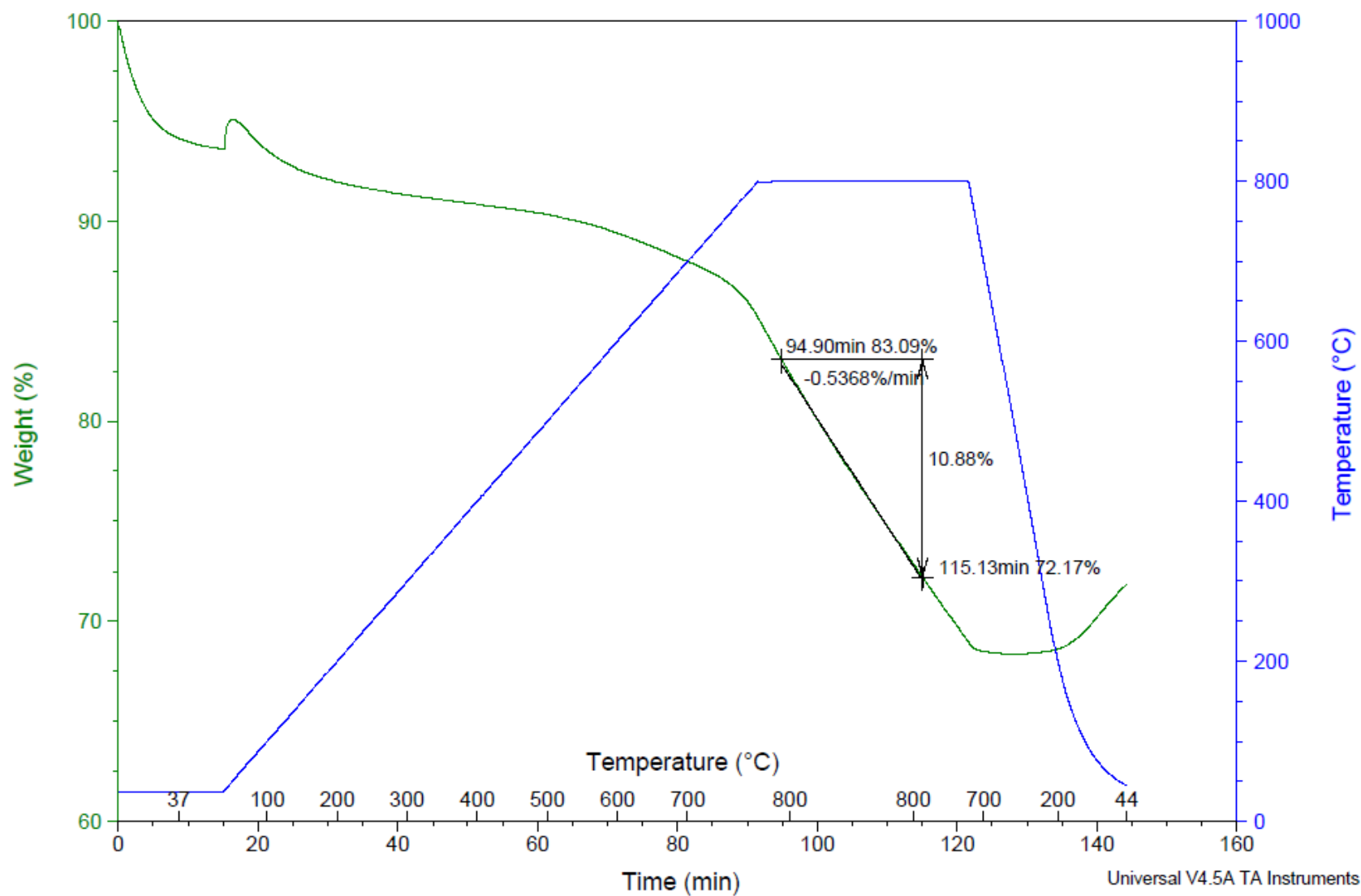
Sample: Proton Power 15-049  
Size: 14.5060 mg  
Method: Activation test to 800 C

## TGA

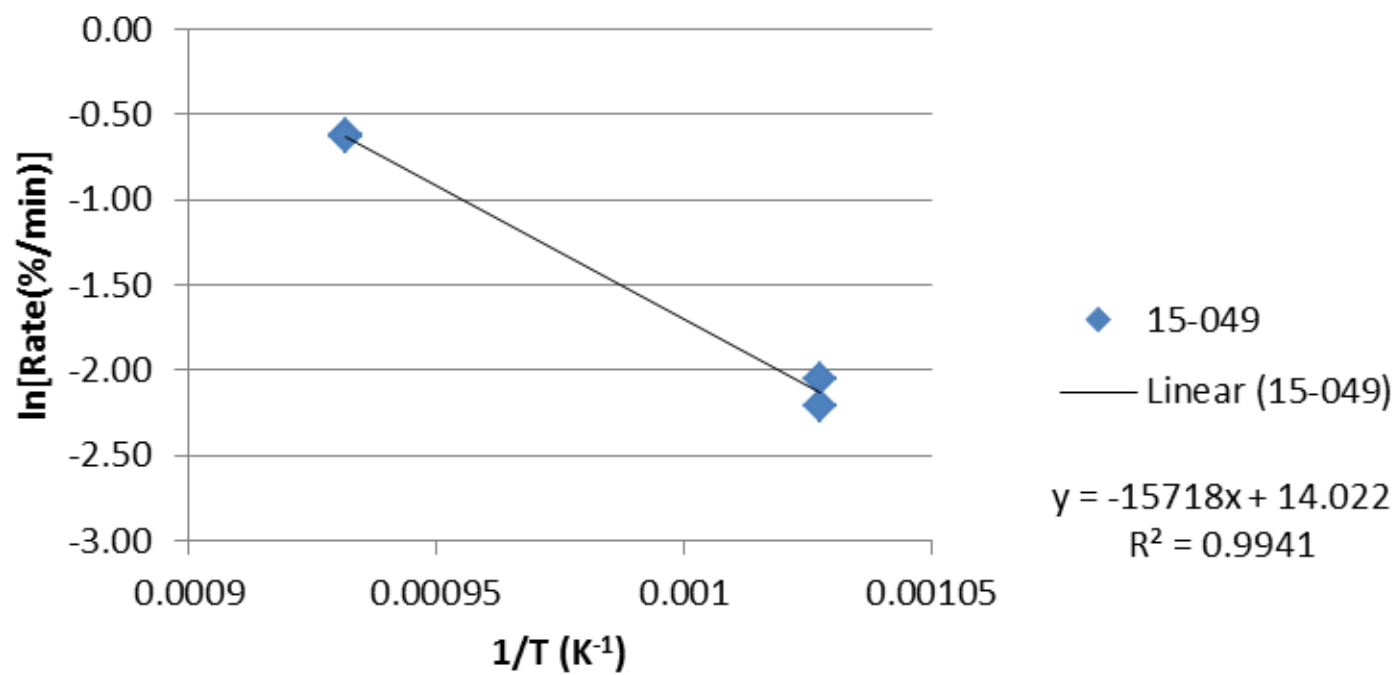


Sample: Proton Power 15-049  
Size: 14.5060 mg  
Method: Activation test to 800 C

## TGA

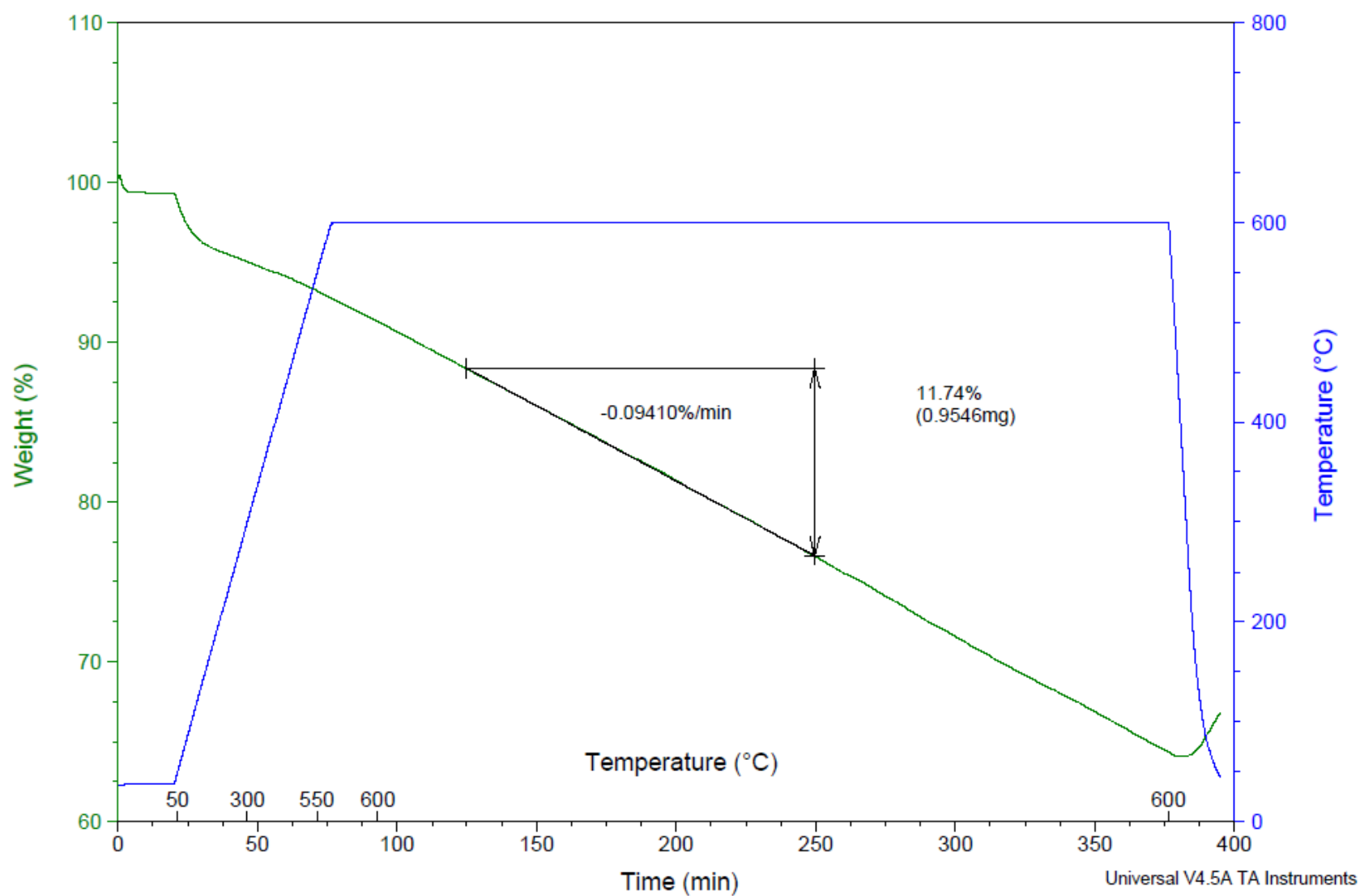


**15-049**



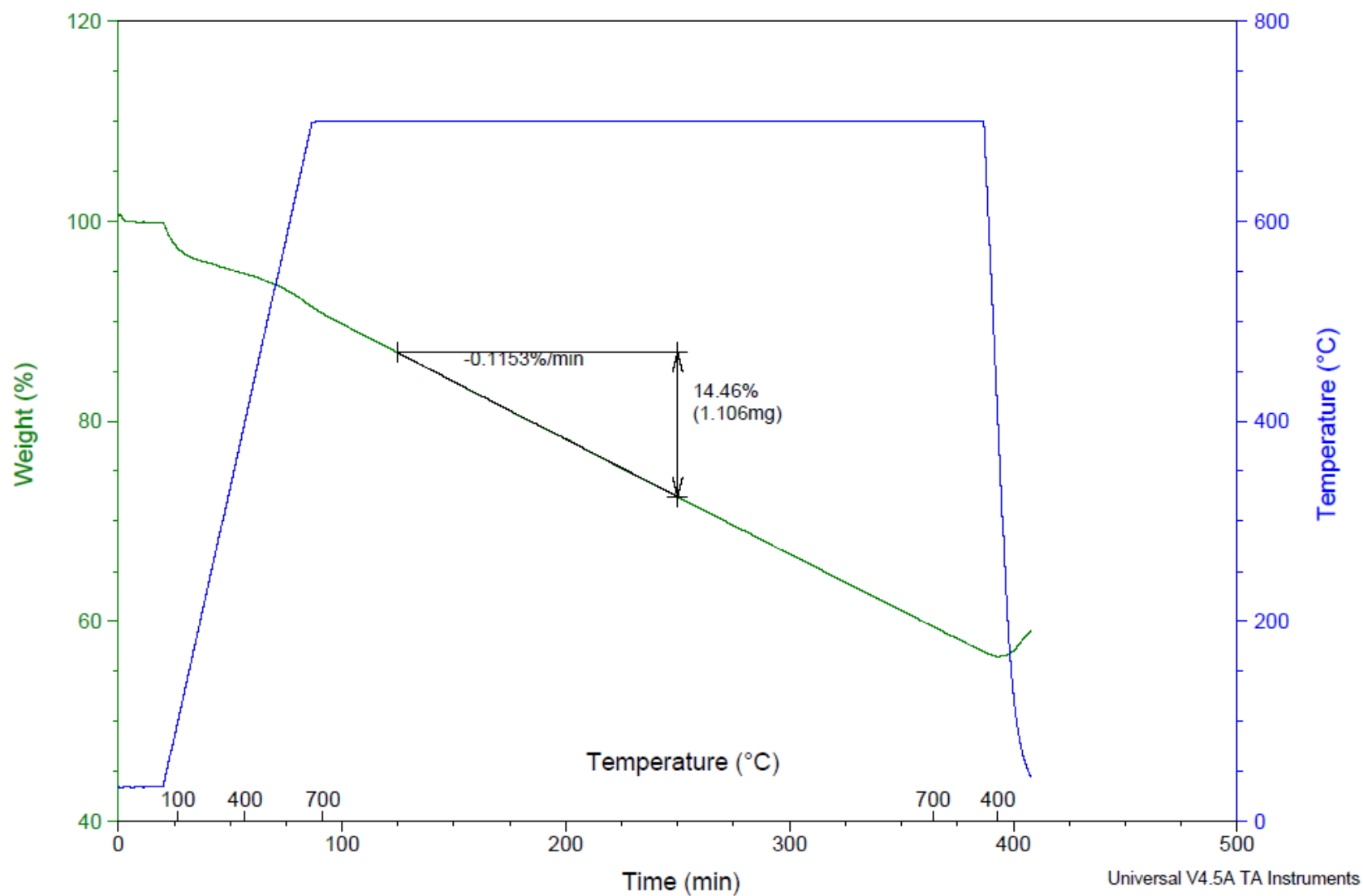
Sample: Sample-16-004 (Chestnut Oak)  
Size: 8.1300 mg  
Method: Activation test 600 C - 300 min

## TGA



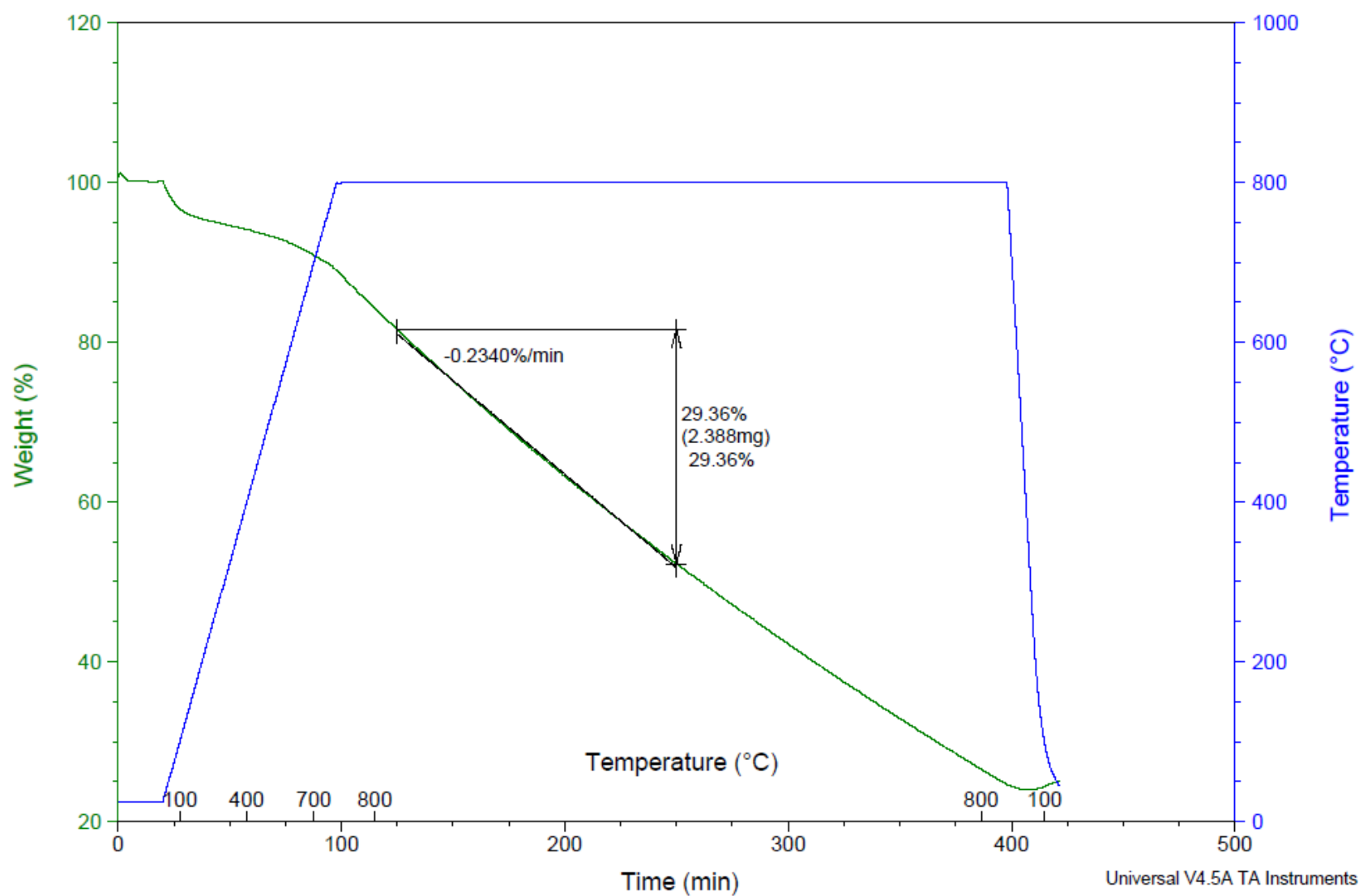
Sample: Sample-16-004 (Chestnut Oak)  
Size: 7.6500 mg  
Method: Activation test 700C - 300 min

## TGA



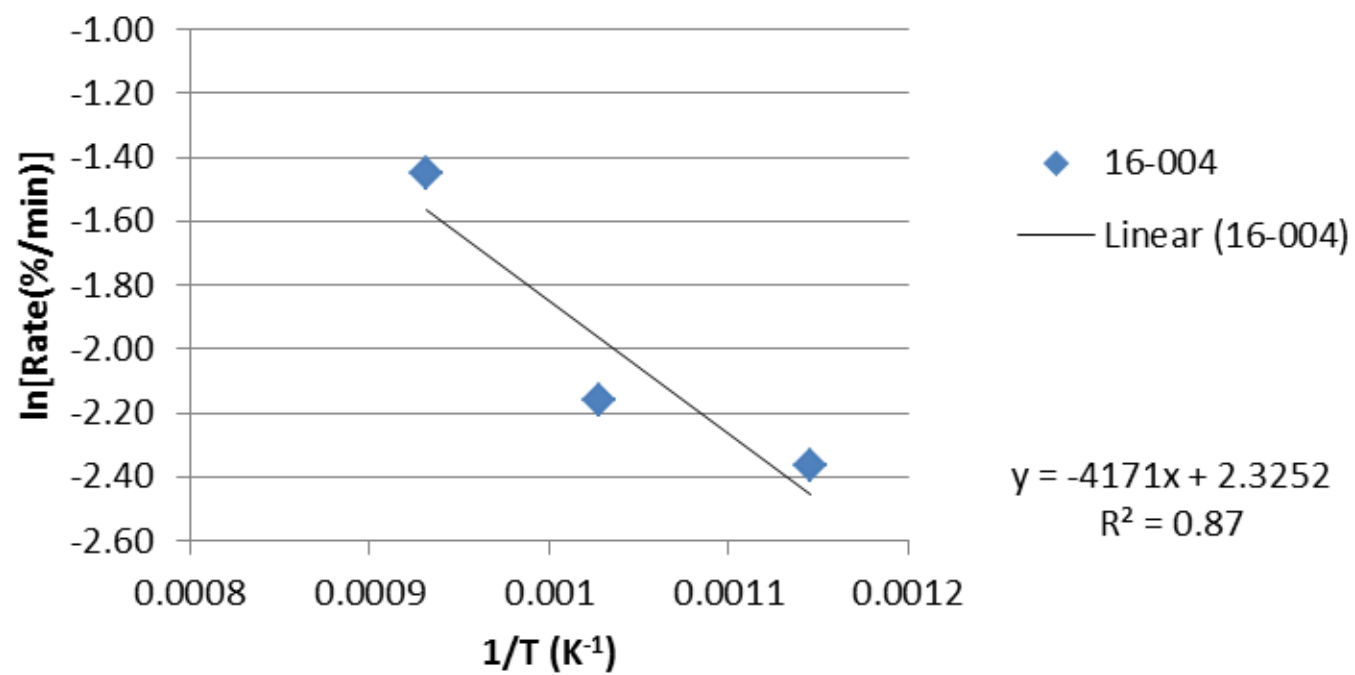
Sample: Sample-16-004 (Chesnut Oak)  
Size: 8.1330 mg  
Method: Activation test 800 C - 300 min

## TGA



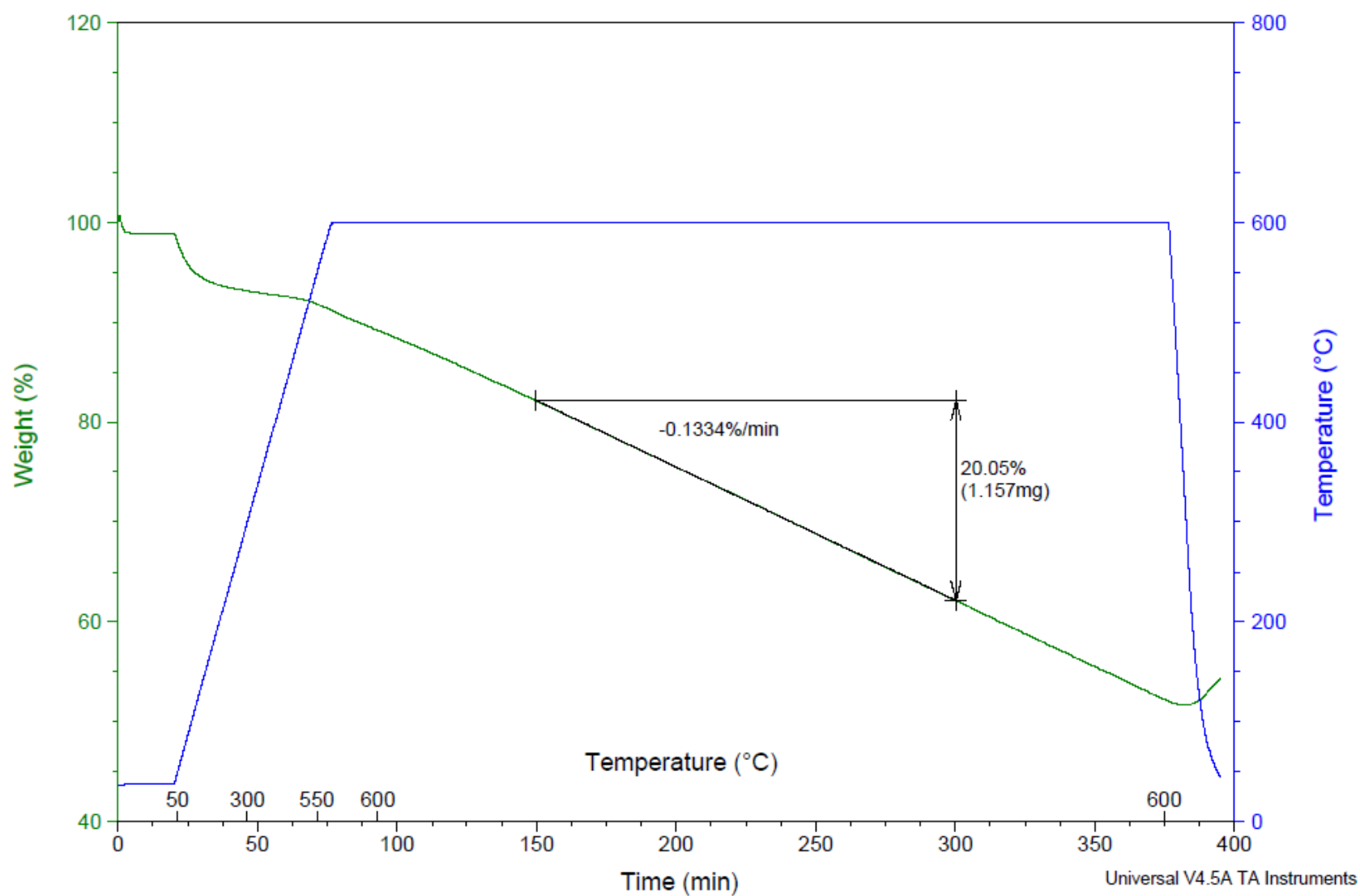


## 16-004



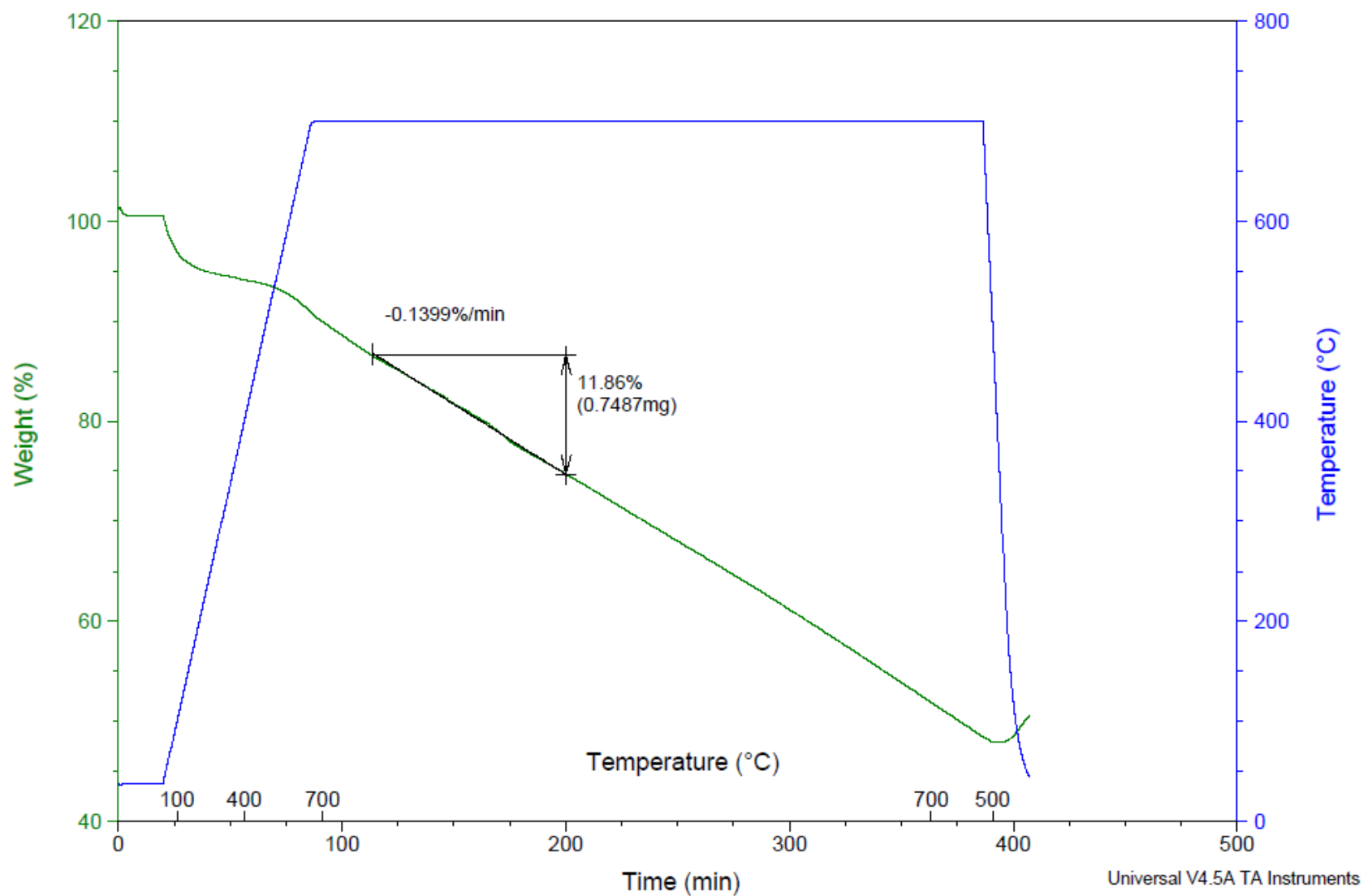
Sample: Sample-16-005 (White Pine)  
Size: 5.7720 mg  
Method: Activation test 600 C - 300 min

## TGA



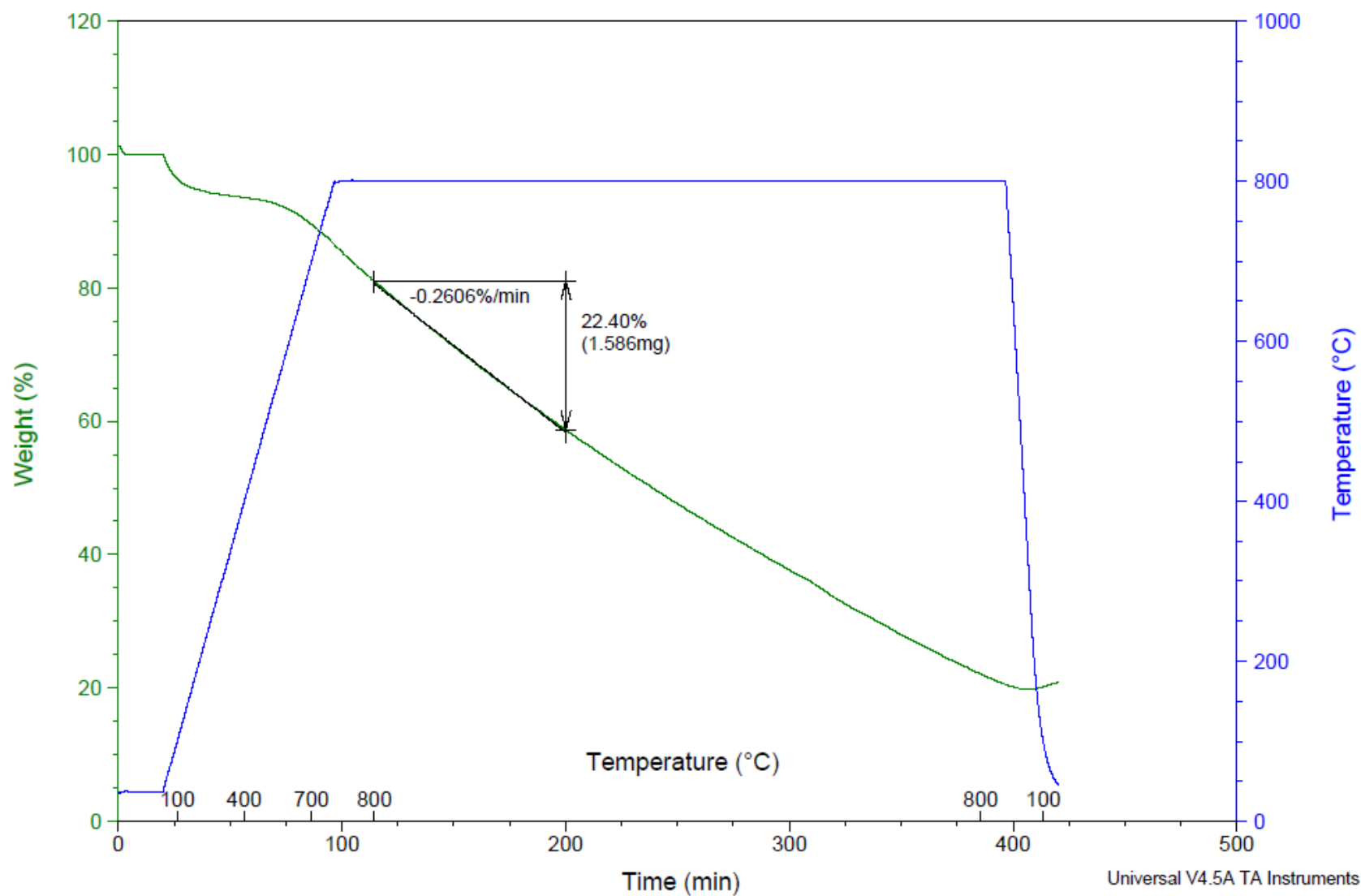
Sample: Sample-16-005 (White Pine)  
Size: 6.3120 mg  
Method: Activation test 700 C - 300 min

## TGA

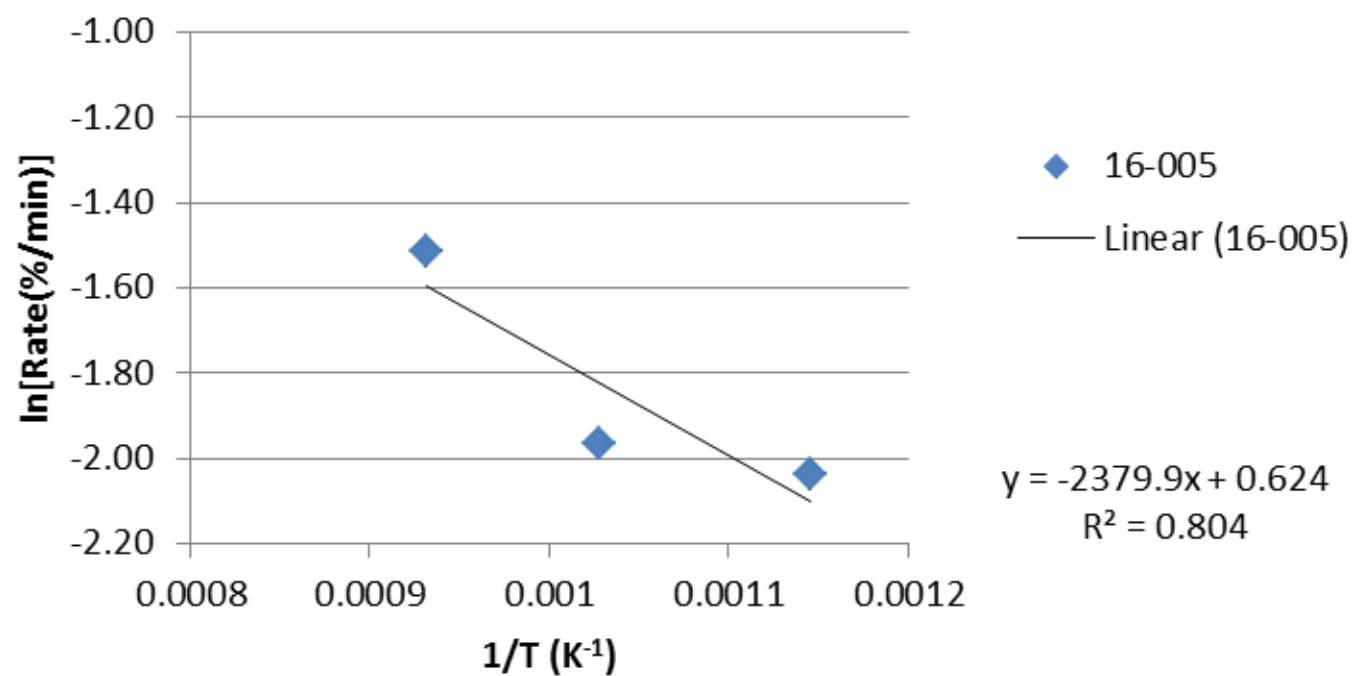


Sample: Sample-16-005 (White Pine)  
Size: 7.0790 mg  
Method: Activation test 800 C - 300 min

## TGA

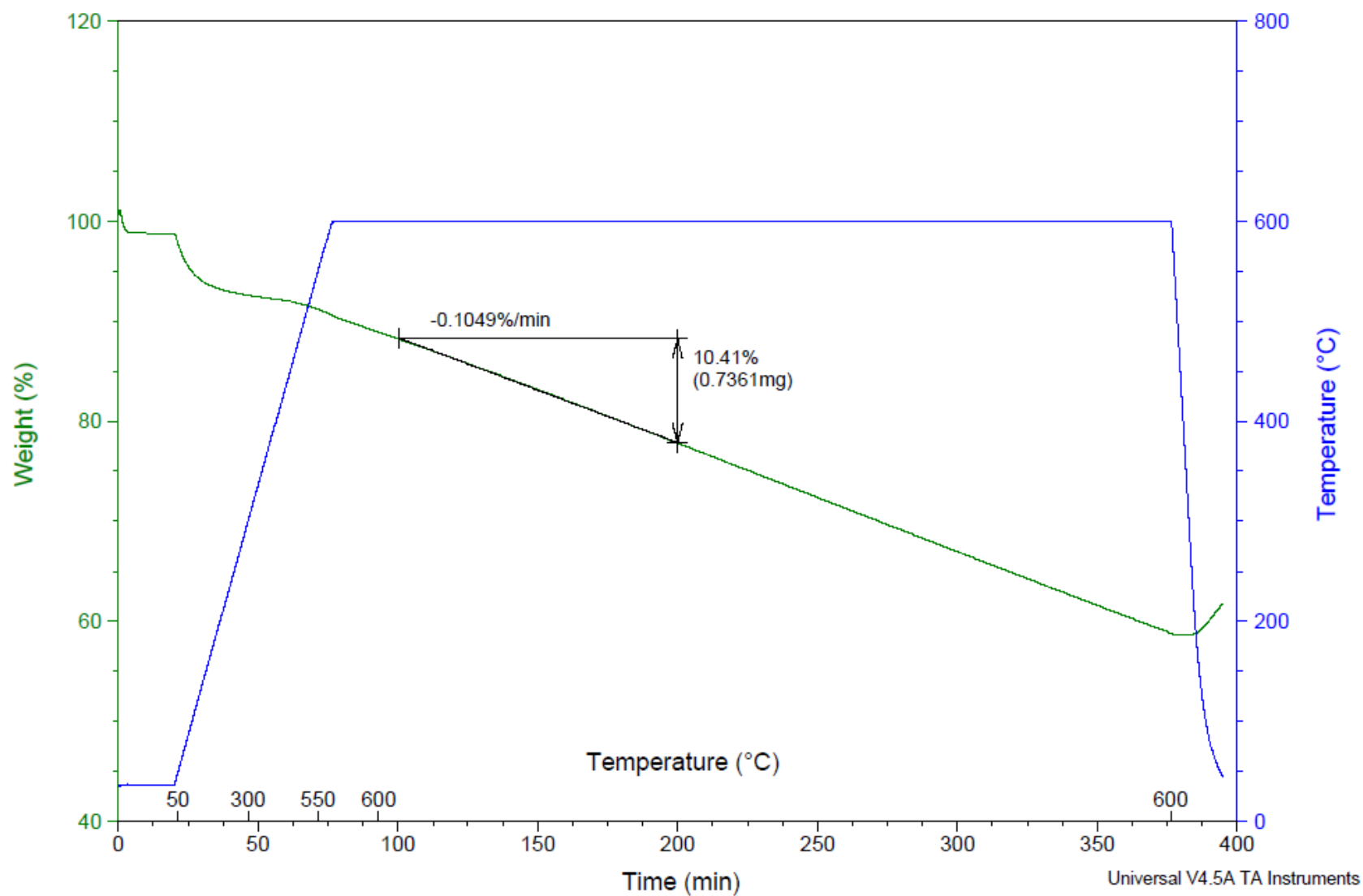


## 16-005



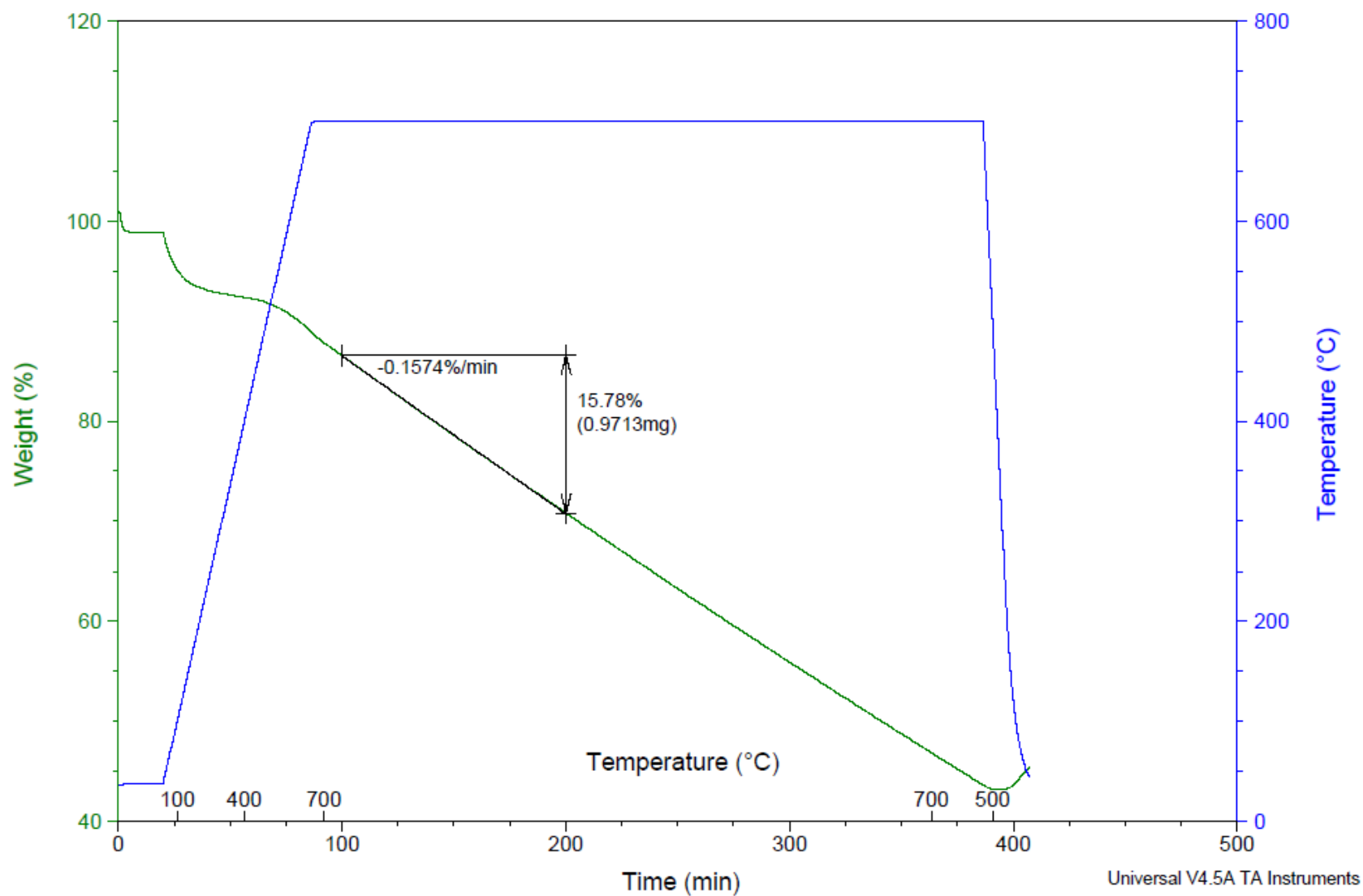
Sample: Sample-16-006 (Yellow Poplar)  
Size: 7.0690 mg  
Method: Activation test 600 C - 300 min

## TGA



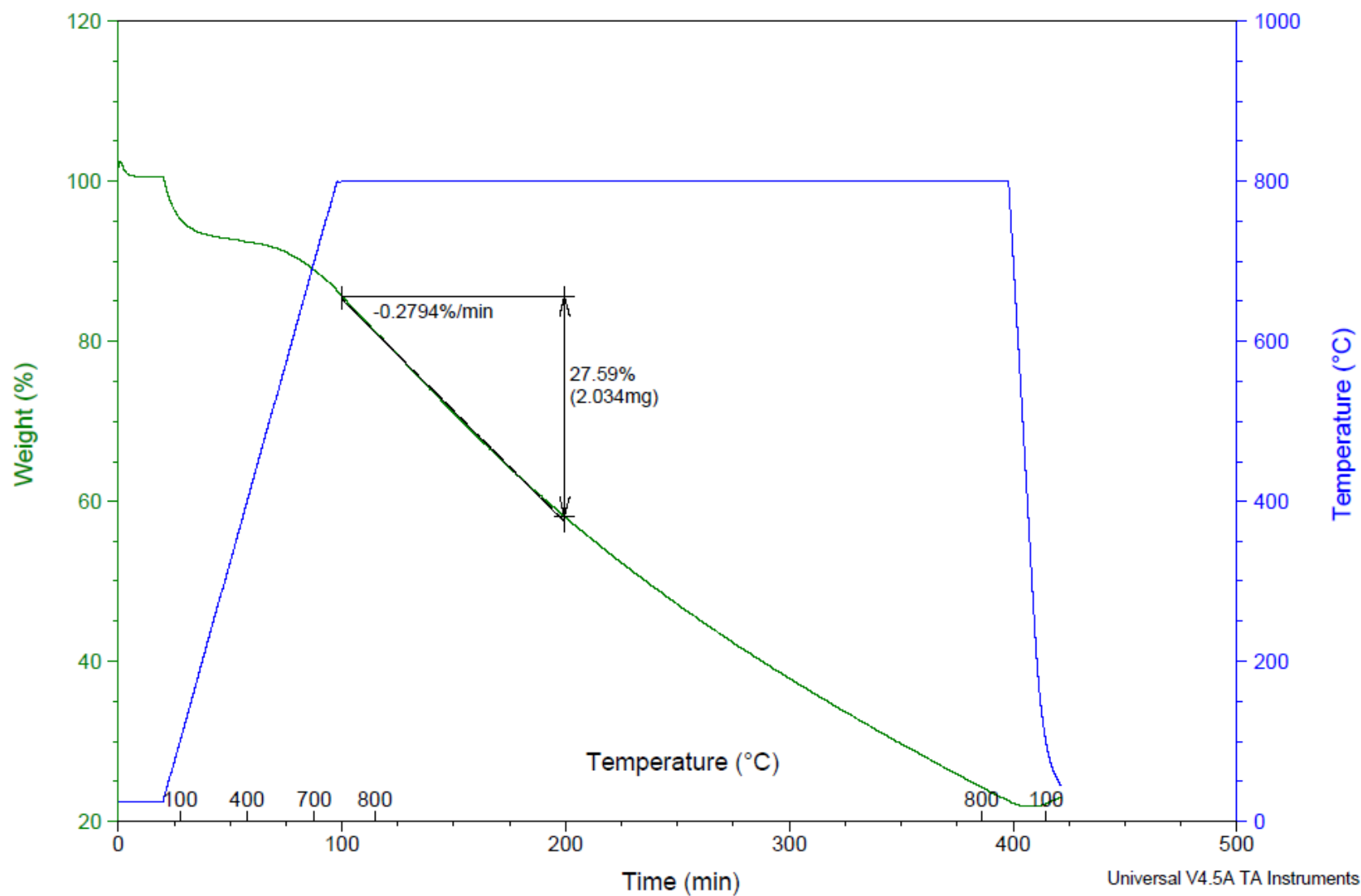
Sample: Sample-16-006 (Yellow Poplar)  
Size: 6.1570 mg  
Method: Activation test 700 C - 300 min

## TGA



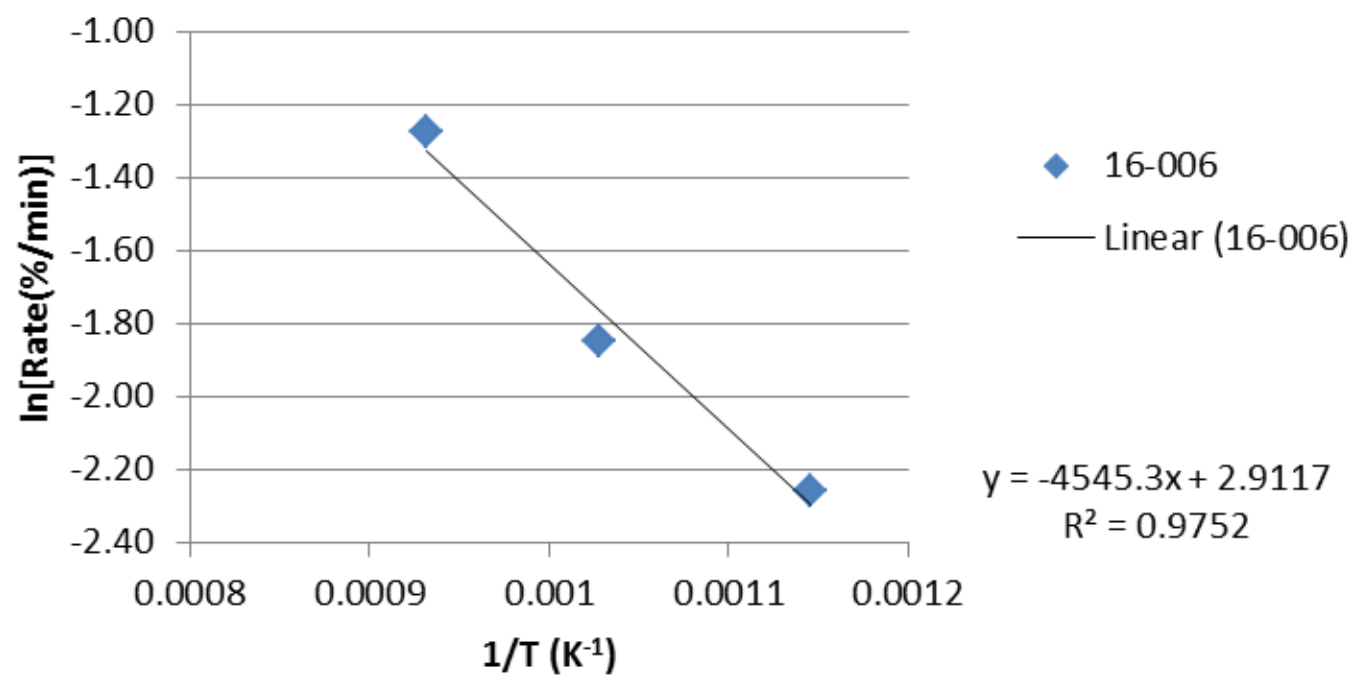
Sample: Sample-16-006 (Yellow Poplar)  
Size: 7.3740 mg  
Method: Activation test 800 C - 300 min

## TGA



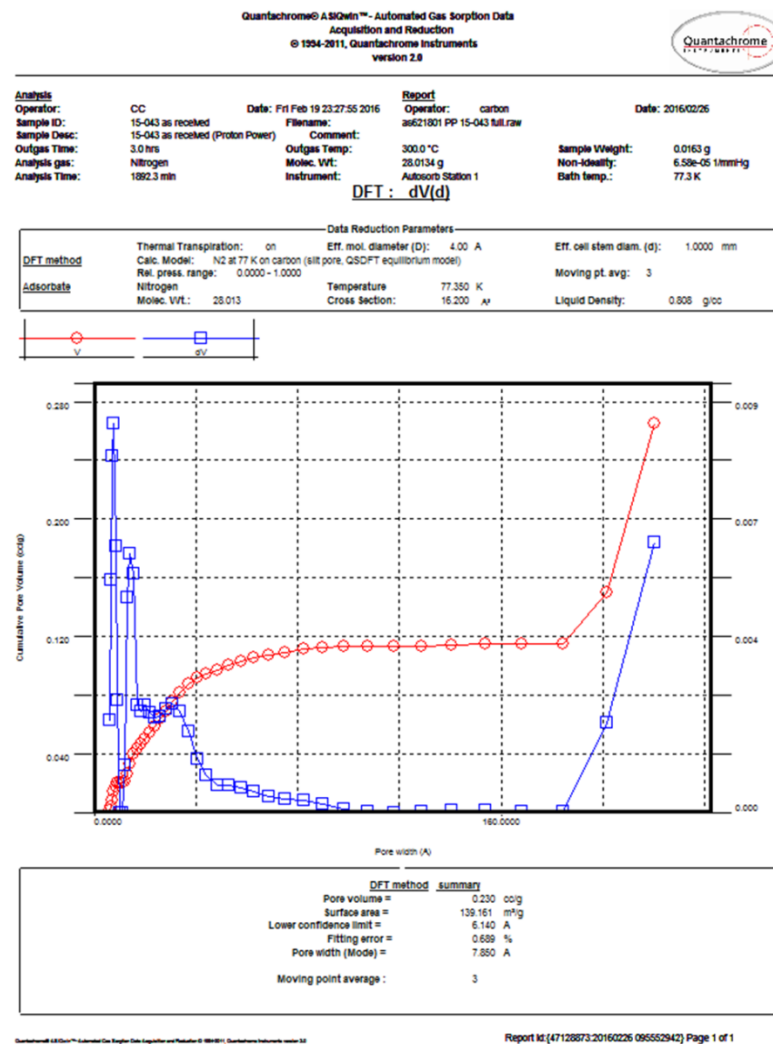
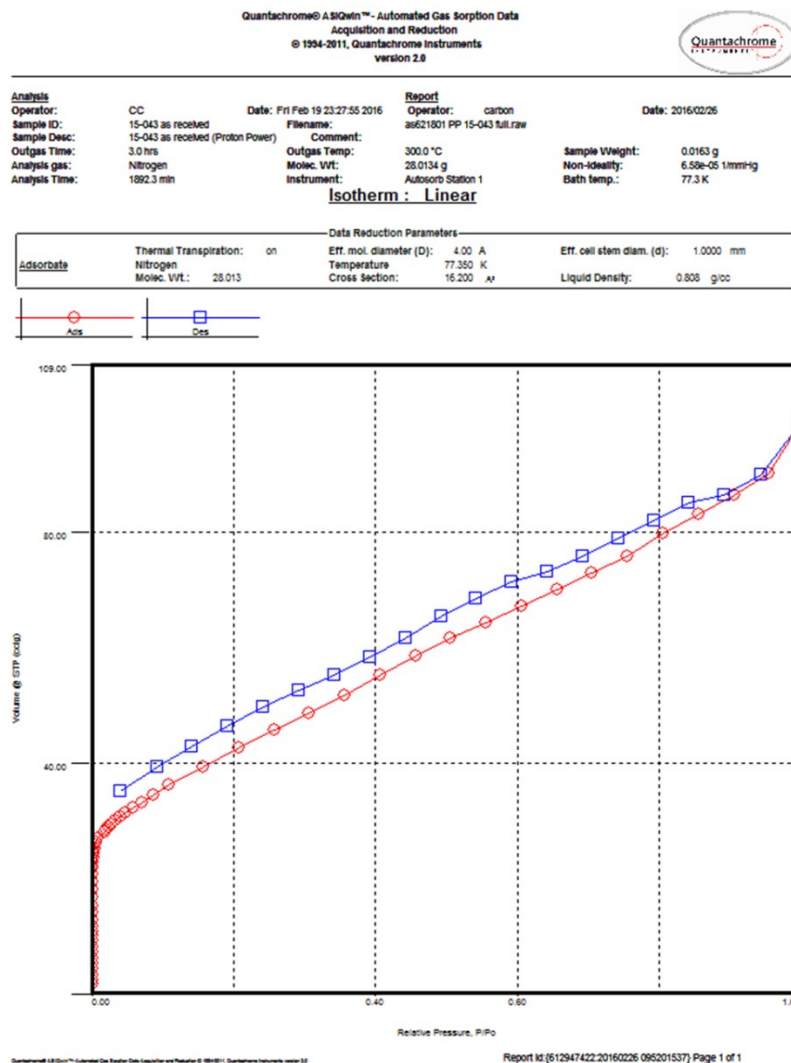


## 16-006

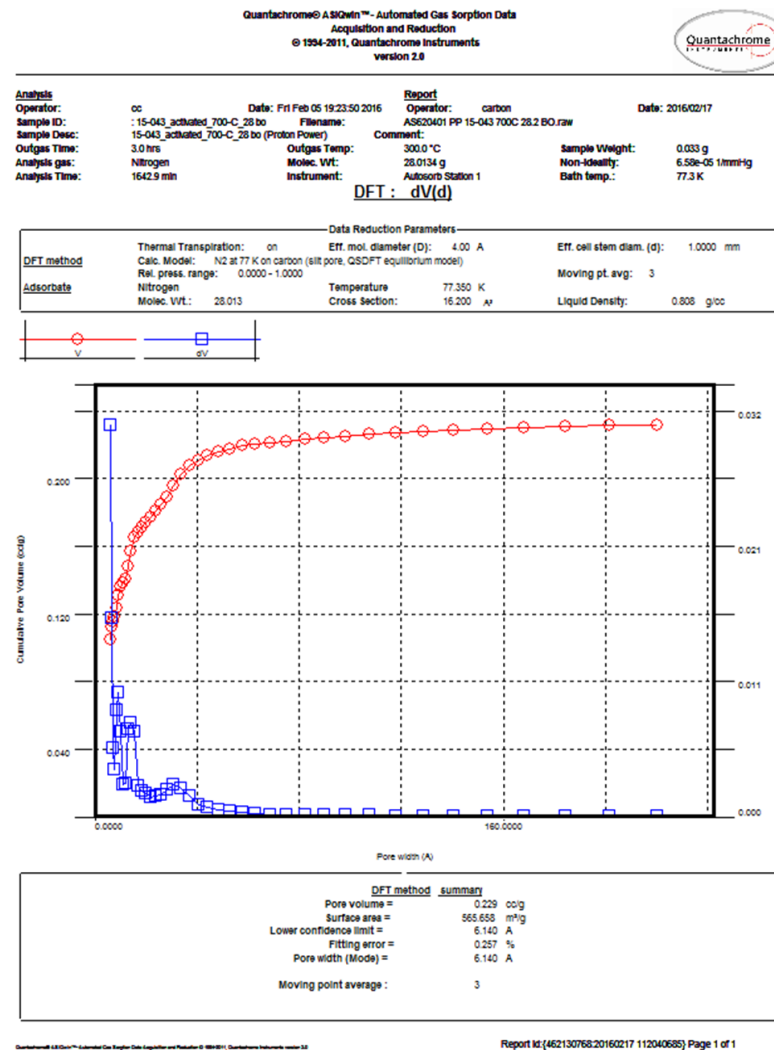
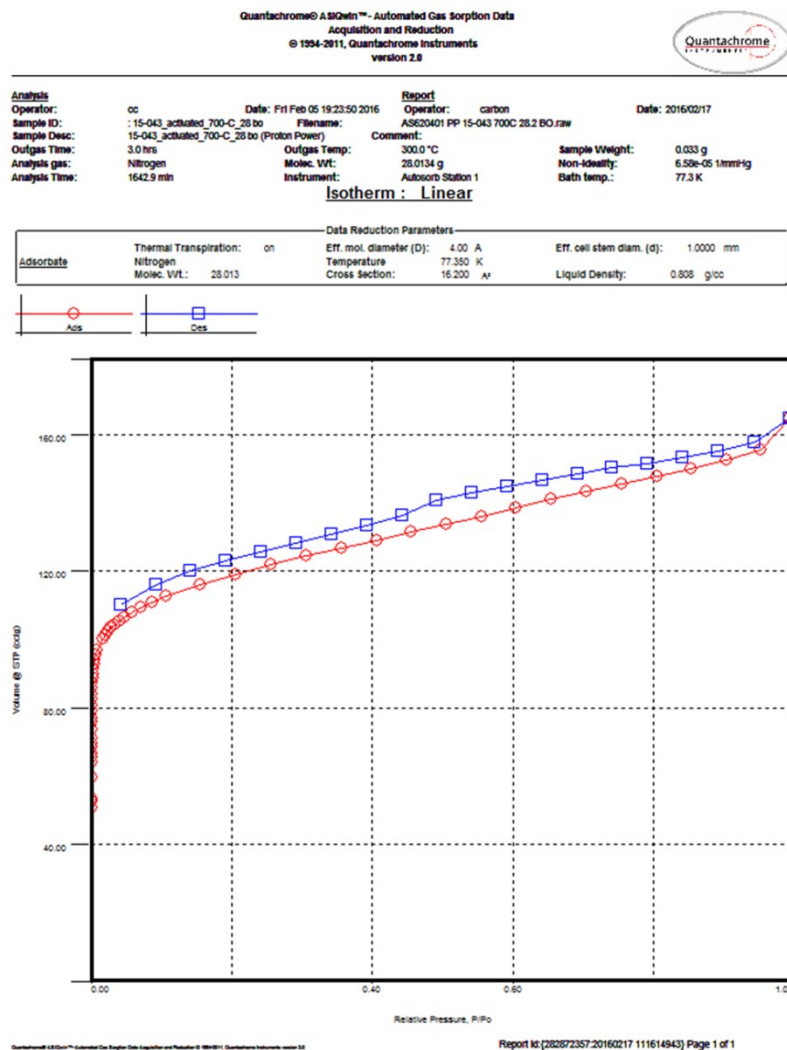


## **Characterization by gas adsorption of as-received and activated samples**

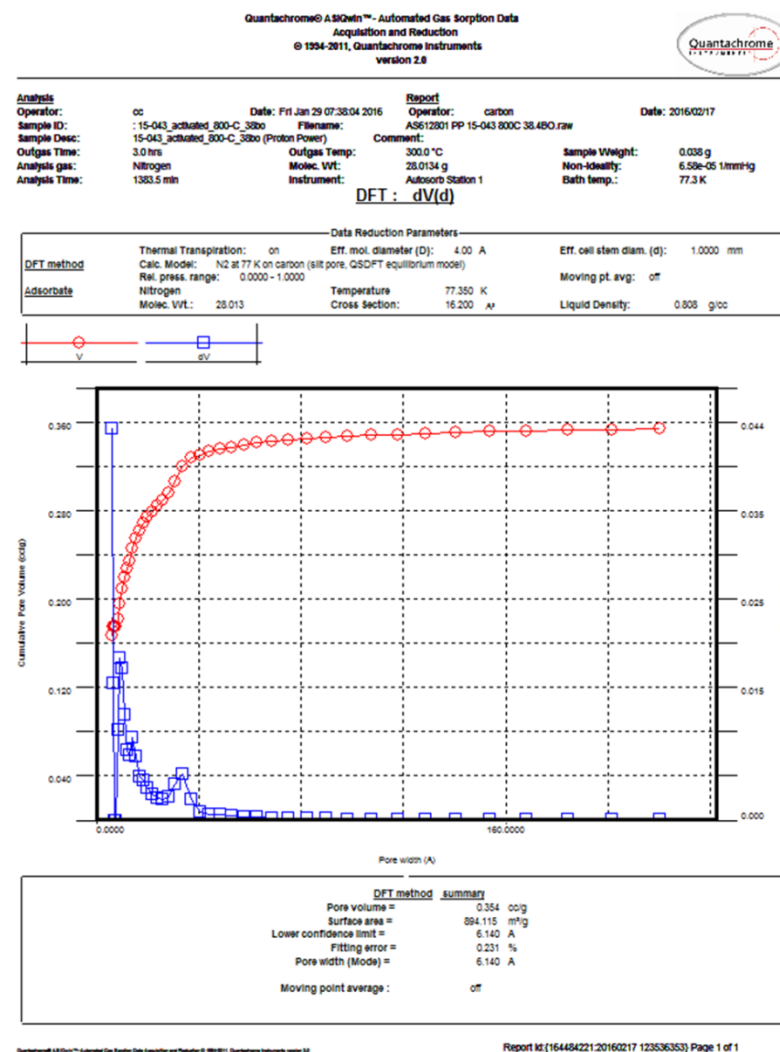
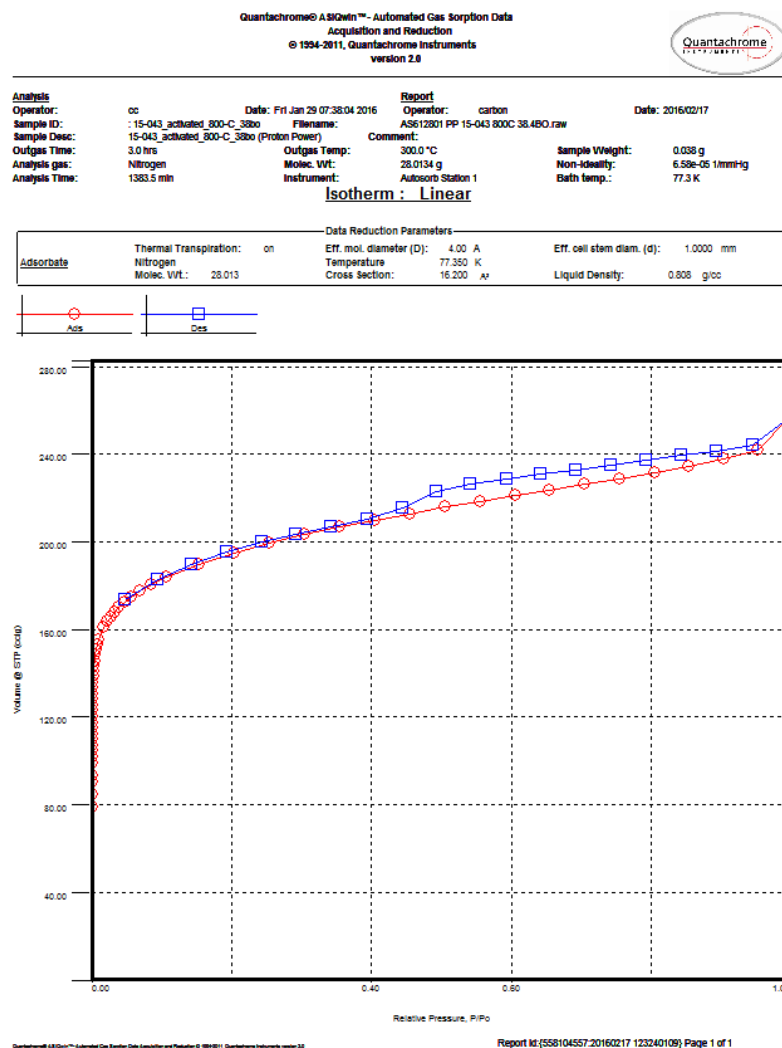
# 15-043 as received



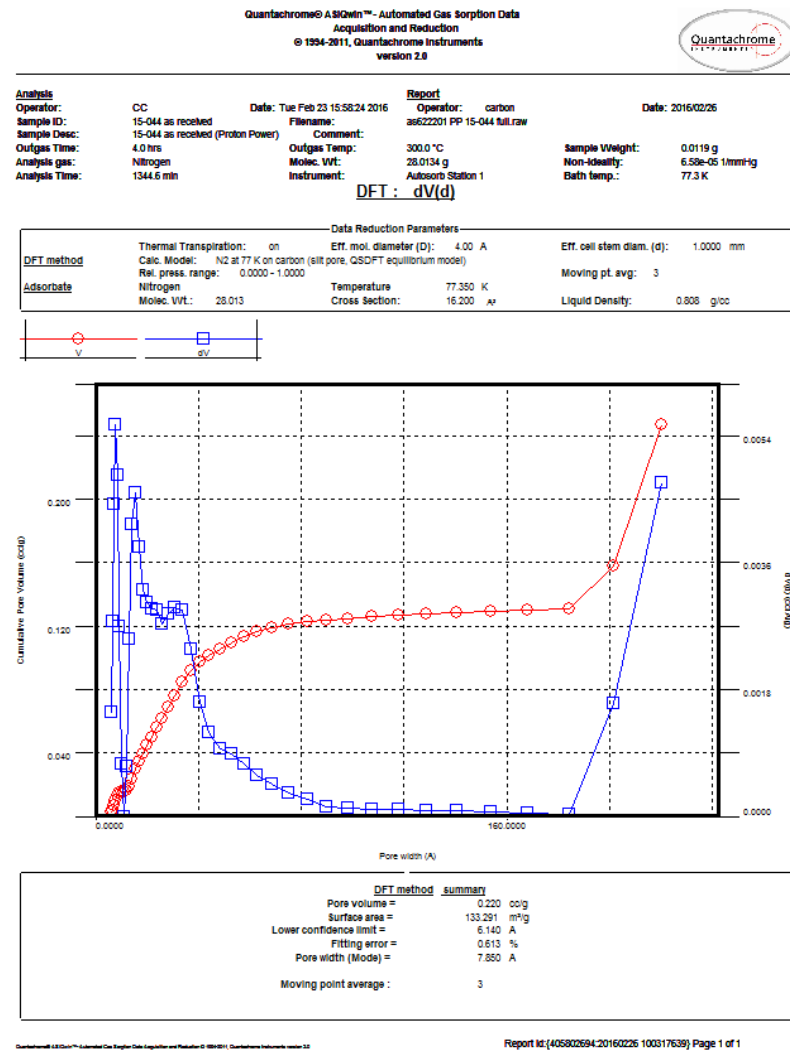
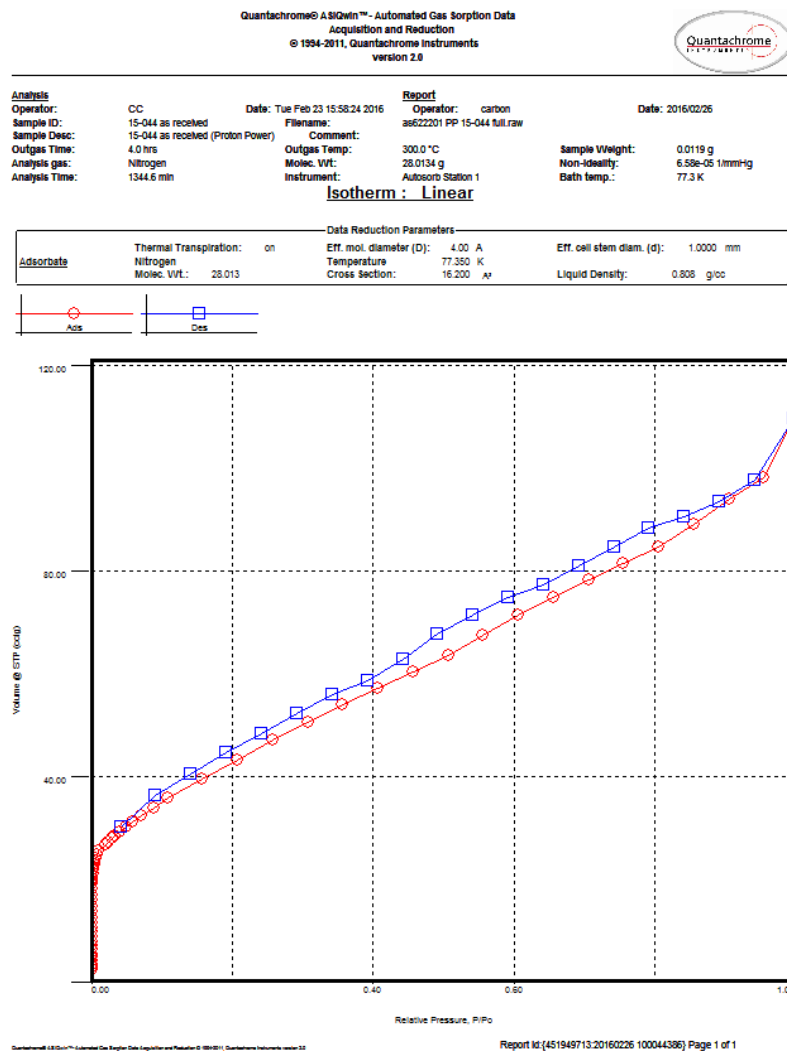
# 15-043 activated at 700 °C (28 % BO)




# 15-043 activated at 800 °C (38 % BO)



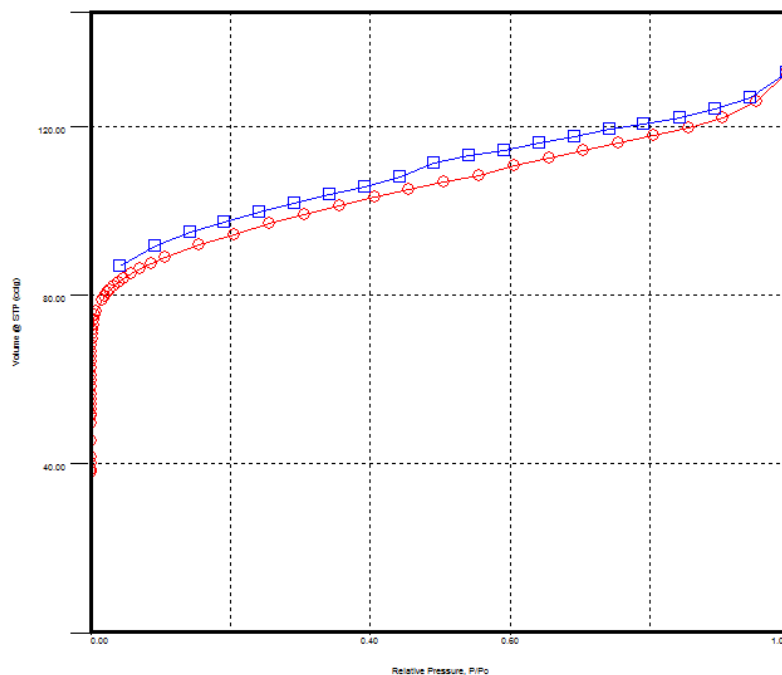
# 15-044 as received



# 15-044 activated at 700 °C (20 % BO)


Quantachrome® ASiQwin™ - Automated Gas Sorption Data Acquisition and Reduction					
© 1994-2011, Quantachrome Instruments					
version 2.0					
<hr/>					
<b>Analysis</b>		<b>Report</b>			
Operator:	cc	Date:	Wed Feb 10 12:06:01 2016	Operator:	carbon
Sample ID:	15-044_activated_700-C_20 bo	Filename:	AS620901 PP 15-044 700C 20 BO.raw	Date:	2016/02/17
Sample Desc:	15-044_activated_700-C_20 bo (Proton Power)	Comment:			
Outgas Time:	3.0 hrs	Outgas Temp:	300.0 °C	Sample Weight:	0.034 g
Analysis gas:	Nitrogen	Molec. Wt:	28.0134 g	Non-ideality:	6.59e-05 1/mm <sup>3</sup> -g
Analysis Time:	1580.5 min	Instrument:	Autosorb Station 1	Bath temp.:	77.3 K
<hr/>					
Isotherm : Linear					

Data Reduction Parameters			
Adsorbate	Thermal Transpiration: on	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Nitrogen	Temperature: 77.350 K	
	Molec. Wt.: 28.013	Cross section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc

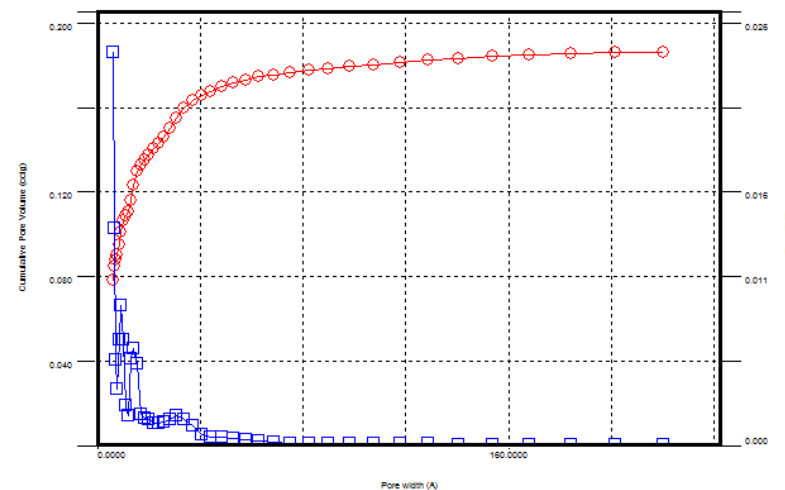


Quantachrome® ASiQwin™ - Automated Gas Sorption Data Acquisition and Reduction © 1994-2011, Quantachrome Instruments version 2.0

Report ID: (774249008:20160217 123924760) Page 1 of 1

Quantachrome® ASiQwin™ - Automated Gas Sorption Data Acquisition and Reduction © 1994-2011, Quantachrome Instruments version 2.0				
<b>Analysis Operator:</b> cc		<b>Report Operator:</b> carbon		<b>Date:</b> 2016/02/17
<b>Sample ID:</b> 15-044_activated_700-C_20 bo		<b>Filename:</b> AS620901 PP 15-044 700C 20 BO.raw		
<b>Sample Desc:</b> 15-044_activated_700-C_20 bo (Proton Power)		<b>Comment:</b>		
<b>Outgas Time:</b> 3.0 hrs		<b>Outgas Temp:</b> 300.0 °C		
<b>Analysis gas:</b> Nitrogen		<b>Molec. Wt.:</b> 28.0134 g		
<b>Analysis Time:</b> 1580.5 min		<b>Instrument:</b> Autosorb Station 1		
		<b>Sample Weight:</b> 0.034 g		
		<b>Non-ideality:</b> 6.59e-05 1/mm <sup>3</sup> -g		
		<b>Bath temp.:</b> 77.3 K		
<b>DFT : dV(d)</b>				

Data Reduction Parameters			
DFT method	Thermal Transpiration: on	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Calc. Model: N2 at 77 K on carbon (slit pore, QSDFT equilibrium model)		
	Rel. press. range: 0.0000 - 1.0000		Moving pt. avg: 3
Adsorbate	Nitrogen	Temperature: 77.350 K	
	Molec. Wt.: 28.013	Cross section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc

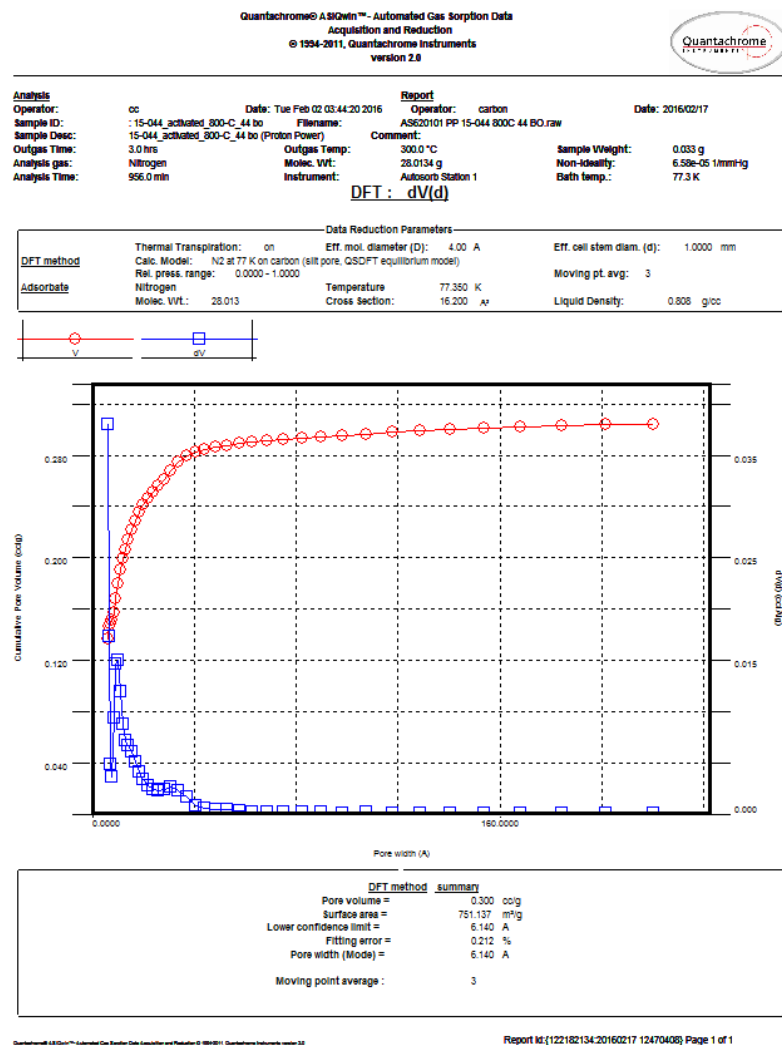
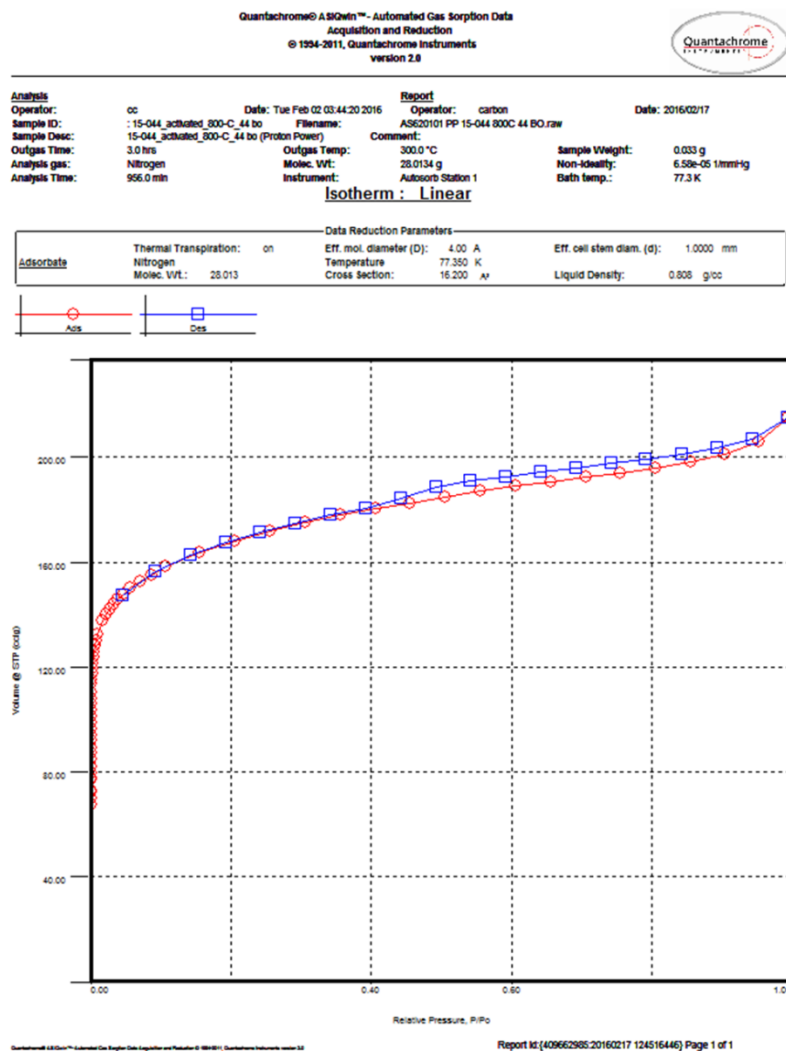


DFT method summary	
Pore volume =	0.184 cc/g
Surface area =	437.292 m <sup>2</sup> /g
Lower confidence limit =	6.140 Å
Fitting error =	0.292 %
Pore width (Mode) =	6.140 Å
Moving point average :	3

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Report ID: (759146779:20160217 124145374) Page 1 of 1

# 15-044 activated at 800 °C (44 % BO)





# 15-045 as received

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**Analysis**  
Operator: CC  
Sample ID: 15-045 as received  
Sample Desc: 15-045 as received (Proton Power)  
Outgas Time: 4.0 hrs  
Analysis gas: Nitrogen  
Analysis Time: 1049.9 min

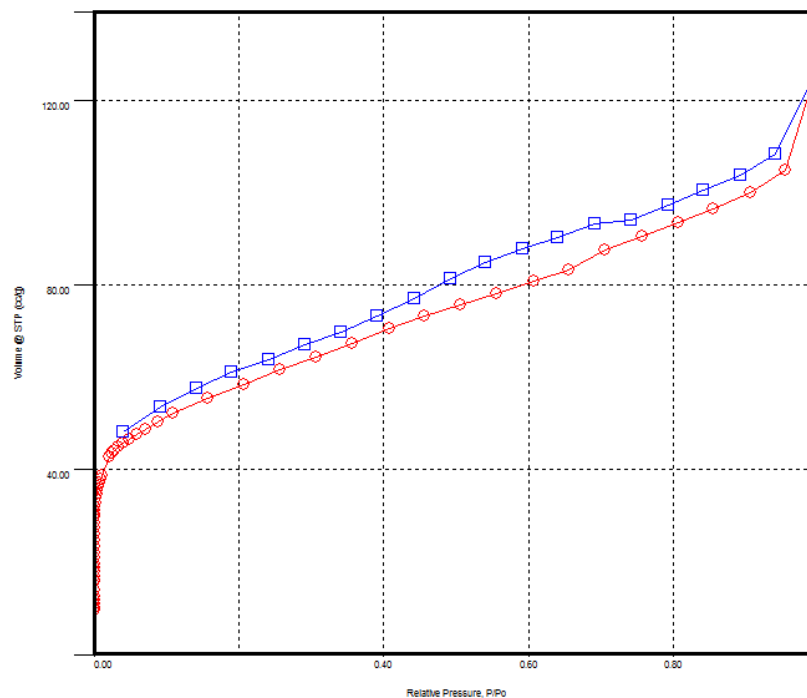
**Report**  
Date: Wed Feb 24 11:41:50 2016  
Filename: as622301 PP 25-045 full.qps  
Comment: 300.0 °C  
Outgas Temp: 28.0134 g  
Molec. Wt.: 28.0134 g  
Instrument: Autosorb Station 1

**Operator:** carbon  
**Date:** 2016/11/14

Sample Weight: 0.012 g  
Non-ideality: 6.58e-05 1/mmHg  
Bath temp.: 77.3 K

**Isotherm : Linear**

Data Reduction Parameters			
Adsorbate	Thermal Transpiration: on Nitrogen Molec. Wt.: 28.013	Eff. mol. diameter (D): 4.00 Å Temperature: 77.350 K Cross Section: 16.200 Å <sup>2</sup>	Eff. cell stem diam. (d): 1.0000 mm Liquid Density: 0.808 g/cc



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Report id:[68456554:20161114 115731425] Page 1 of 1

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**Analysis**  
Operator: CC  
Sample ID: 15-045 as received  
Sample Desc: 15-045 as received (Proton Power)  
Outgas Time: 4.0 hrs  
Analysis gas: Nitrogen  
Analysis Time: 1049.9 min

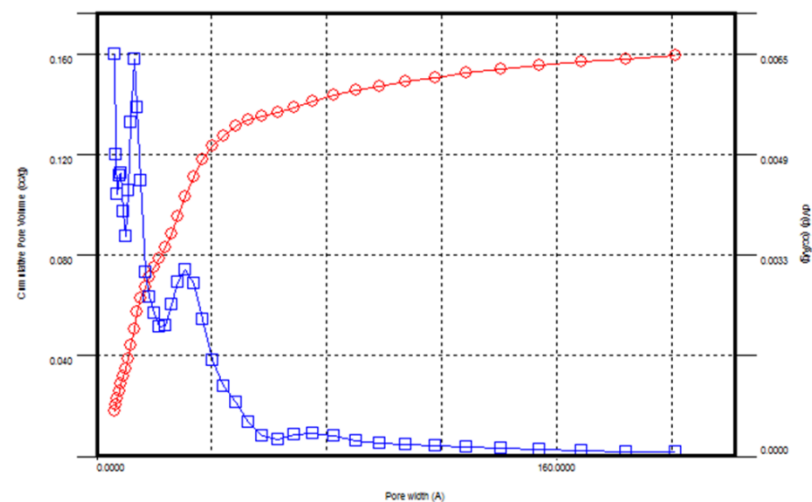
**Report**  
Date: Wed Feb 24 11:41:50 2016  
Filename: as622301 PP 25-045 full.qps  
Comment: 300.0 °C  
Outgas Temp: 28.0134 g  
Molec. Wt.: 28.0134 g  
Instrument: Autosorb Station 1

**Operator:** carbon  
**Date:** 2016/11/14

Sample Weight: 0.012 g  
Non-ideality: 6.58e-05 1/mmHg  
Bath temp.: 77.3 K

**DFT : dV(d)**

Data Reduction Parameters			
DFT method	Thermal Transpiration: on Calc. Model: N2 at 77 K on carbon (slit pore, QSDFT equilibrium model) Rel. press. range: 0.0000 - 1.0000	Eff. mol. diameter (D): 4.00 Å Temperature: 77.350 K Cross Section: 16.200 Å <sup>2</sup>	Eff. cell stem diam. (d): 1.0000 mm Moving pt. avg: 3 Liquid Density: 0.808 g/cc



**DFT method summary**  
Pore volume = 0.159 cc/g  
Surface area = 208.262 m<sup>2</sup>/g  
Lower confidence limit = 6.140 Å  
Fitting error = 6.543 %  
Pore width (mode) = 6.140 Å  
Moving point average : 3

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Report id:[718866139:20161114 115917592] Page 1 of 1

# 15-045 activated at 700 °C (19 % BO)

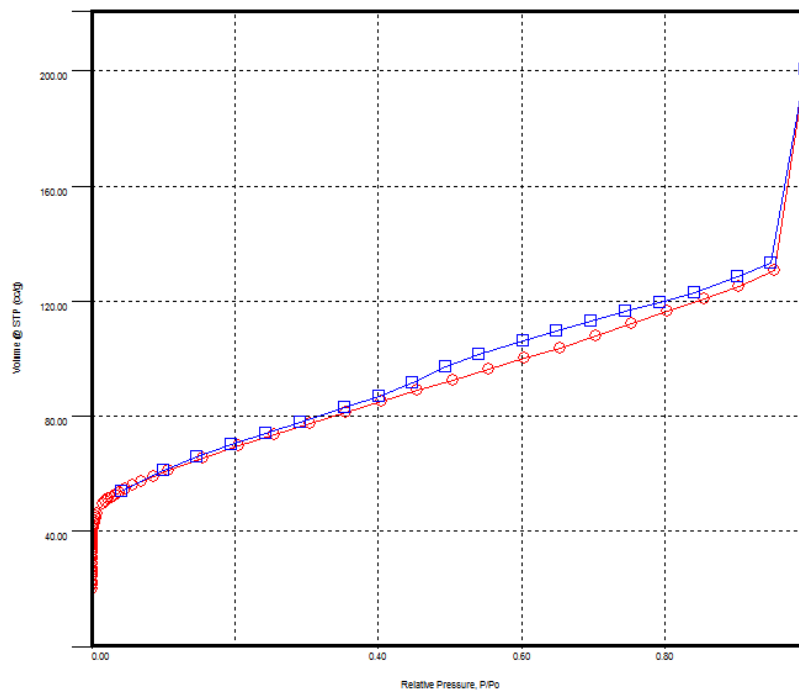
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**Analysis**  
Operator: cc Date: Sat Nov 12 07:47:45 2016 Operator: carbon Date: 2016/11/14  
Sample ID: Proton Power 15-045-700 (19% BO) Filename: AS8B1101 PP 15-045 700 C 19 BO (R).raw  
Sample Desc: repeat Comment: 390.0 °C  
Outgas Time: 3.0 hrs Outgas Temp: 390.0 °C  
Analysis gas: Nitrogen Molec. Wt.: 28.0134 g Non-ideality: 6.58e-05 1/mmHg  
Analysis Time: 824.7 min Instrument: Autosorb Station 1 Bath temp.: 77.3 K

**Isotherm : Linear**

Data Reduction Parameters			
Adsorbate	Thermal Transpiration: on	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Nitrogen	Temperature: 77.350K	
	Molec. Wt.: 28.013	Cross Section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc



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Report id:{167418241:20161114 113836833} Page 1 of 1

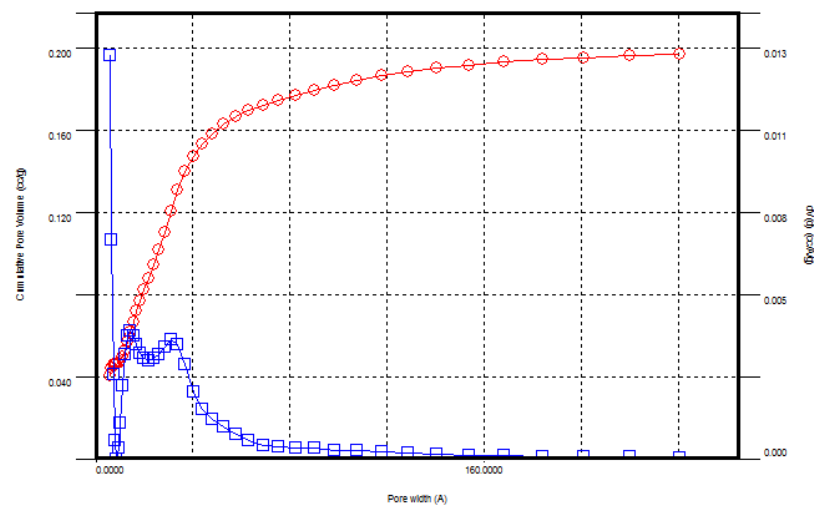
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**Analysis**  
Operator: cc Date: Sat Nov 12 07:47:45 2016 Operator: carbon Date: 2016/11/14  
Sample ID: Proton Power 15-045-700 (19% BO) Filename: AS8B1101 PP 15-045 700 C 19 BO (R).raw  
Sample Desc: repeat Comment: 390.0 °C  
Outgas Time: 3.0 hrs Outgas Temp: 390.0 °C  
Analysis gas: Nitrogen Molec. Wt.: 28.0134 g Non-ideality: 6.58e-05 1/mmHg  
Analysis Time: 824.7 min Instrument: Autosorb Station 1 Bath temp.: 77.3 K

**DFT : dV(d)**

Data Reduction Parameters			
DFT method	Thermal Transpiration: on	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Calc. Model: N2 at 77 K on carbon (slit pore, QSDFT equilibrium model)		
Adsorbate	Rel. press. range: 0.0000 - 1.0000	Temperature: 77.350K	Moving pt. avg: 3
	Nitrogen	Cross Section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc
	Molec. Wt.: 28.013		



DFT method summary	
Pore volume =	0.196 cc/g
Surface area =	277.700 m <sup>2</sup> /g
Lower confidence limit =	6.140 Å
Fitting error =	0.528 %
Pore width (Mode) =	6.140 Å
Moving point average :	3

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Report id:{1177961072:20161114 114313963} Page 1 of 1

# 15-045 activated at 800 °C (34 % BO)

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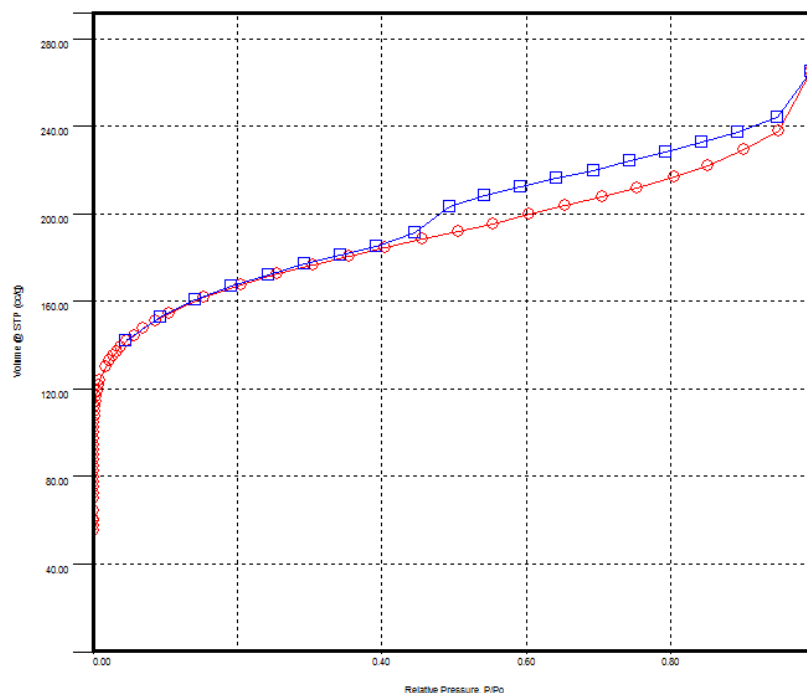


**Analysis**  
Operator: CC  
Sample ID: 15-045 - 34.4% b/o at 800 °C (Proton Power)  
Sample Desc: 15-045 - 34.4% b/o at 800 °C (Proton Power)  
Outgas Time: 6.0 hrs  
Analysis gas: Nitrogen  
Analysis Time: 965.1 min

**Report**  
Date: Sat Apr 02 04:41:48 2016  
Operator: carbon  
Filename: AS840101 PP 15-045 800C 34 BO.raw  
Date: 2016/11/10  
Comment:  
Outgas Temp: 300.0 °C  
Molec. Wt: 28.0134 g  
Instrument: Autosorb Station 1  
Sample Weight: 0.0295 g  
Non-ideality: 6.58e-05 1/mmHg  
Bath temp.: 77.3 K

Isotherm: Linear

Data Reduction Parameters			
Adsorbate	Thermal Transpiration: on	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Nitrogen	Temperature: 77.350 K	
	Molec. Wt.: 28.013	Cross Section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc



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Report id: {1119924908:20161110 160855784} Page 1 of 1

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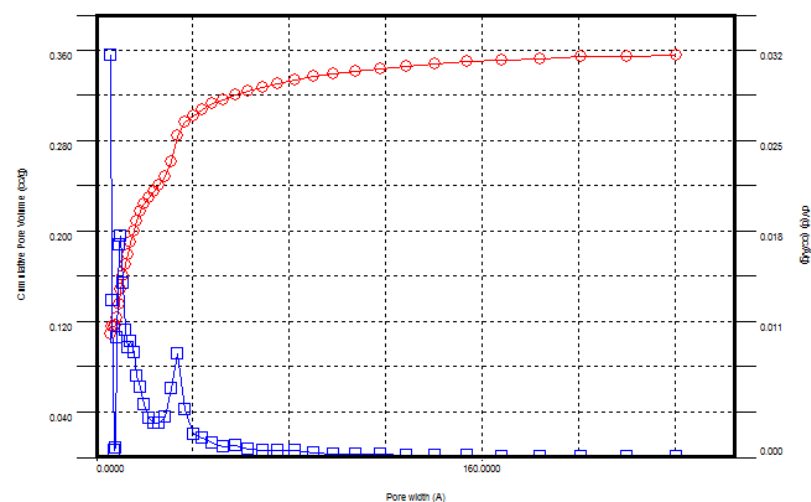


**Analysis**  
Operator: CC  
Sample ID: 15-045 - 34.4% b/o at 800 °C (Proton Power)  
Sample Desc: 15-045 - 34.4% b/o at 800 °C (Proton Power)  
Outgas Time: 6.0 hrs  
Analysis gas: Nitrogen  
Analysis Time: 965.1 min

**Report**  
Date: Sat Apr 02 04:41:48 2016  
Operator: carbon  
Filename: AS840101 PP 15-045 800C 34 BO.raw  
Date: 2016/11/10  
Comment:  
Outgas Temp: 300.0 °C  
Molec. Wt: 28.0134 g  
Instrument: Autosorb Station 1  
Sample Weight: 0.0295 g  
Non-ideality: 6.58e-05 1/mmHg  
Bath temp.: 77.3 K

DFT: dV(d)

Data Reduction Parameters			
DFT method	Thermal Transpiration: on	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Calc. Model: N2 at 77 K on carbon (slit pore, QSDFT equilibrium model)	Temperature: 77.350 K	
Adsorbate	Rel. press. range: 0.0000 - 1.0000	Cross Section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc
	Nitrogen		Moving pt. avg: off
	Molec. Wt.: 28.013		

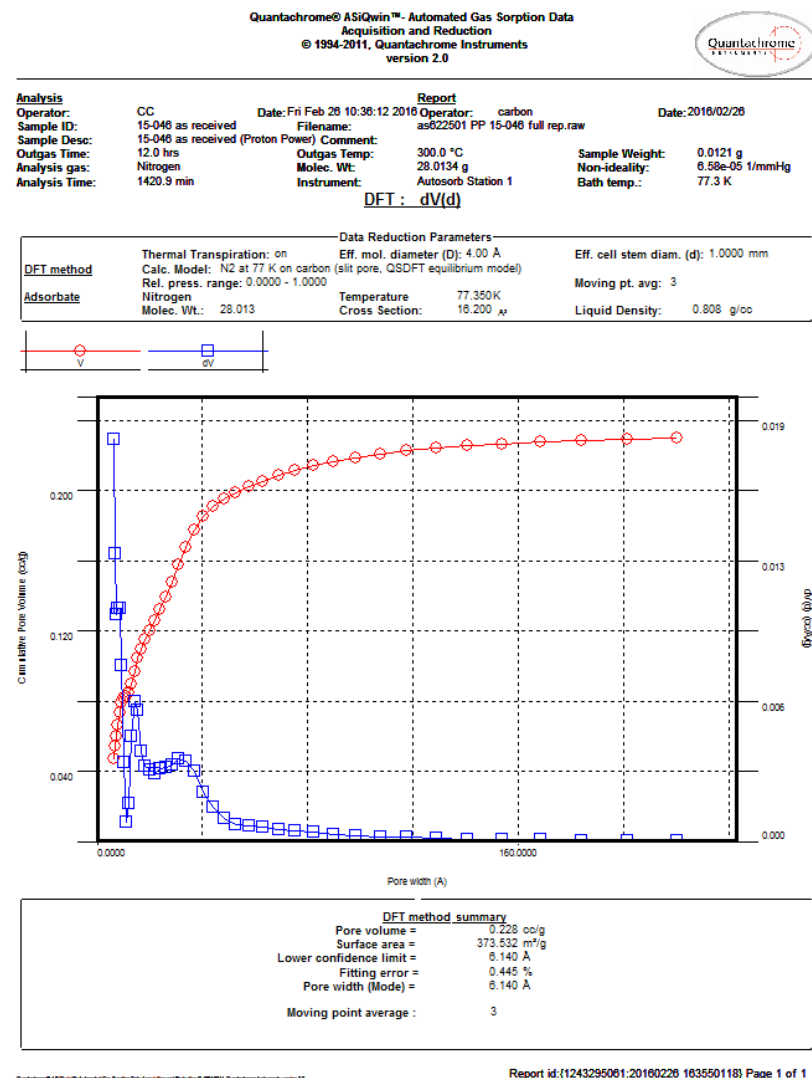
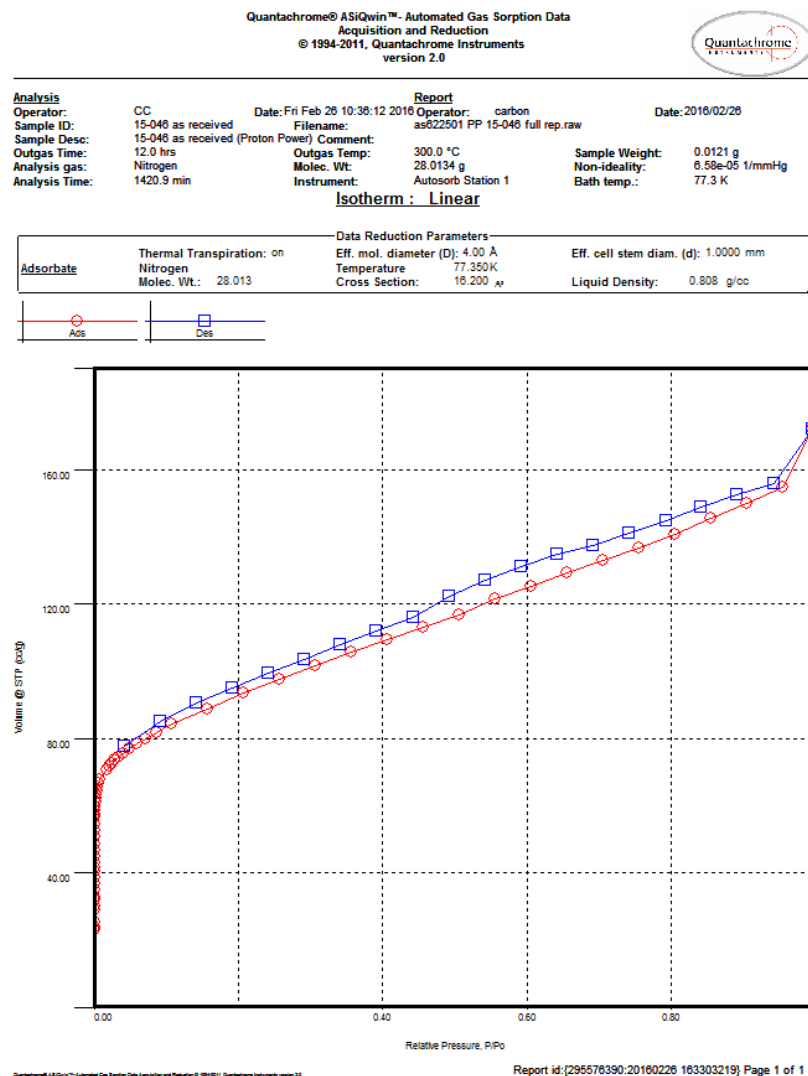


**DFT method summary**  
Pore volume = 0.355 cc/g  
Surface area = 891.591 m<sup>2</sup>/g  
Lower confidence limit = 6.140 Å  
Fitting error = 0.355 %  
Pore width (Mode) = 6.140 Å  
Moving point average: off

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Report id: {857239903:20161110 161112247} Page 1 of 1

# 15-046 as received



# 15-046 activated at 700 °C (22 % BO)

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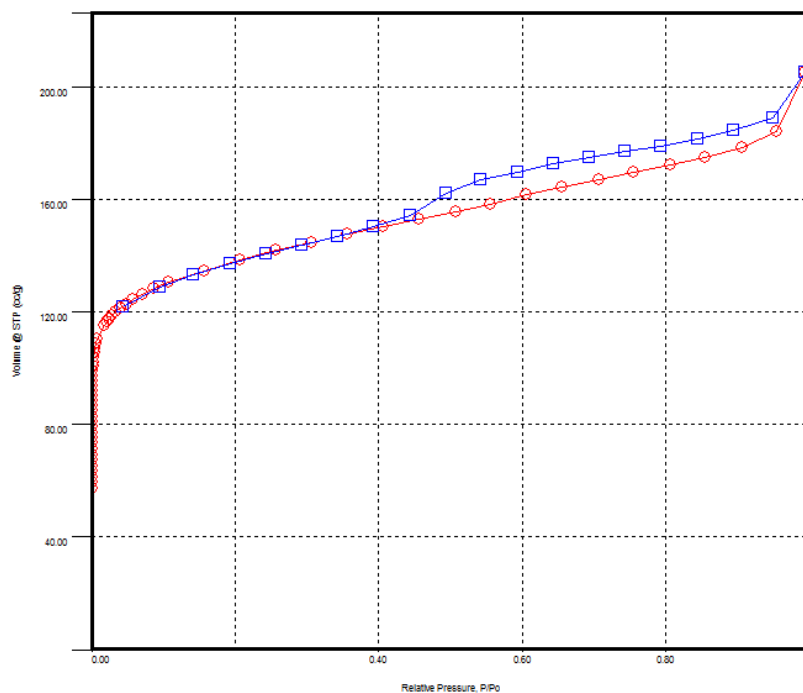
**Analysis**  
Operator: ng  
Sample ID: : Proton Power 15-046 activ. @ 700C - 21.8 % b/o  
Sample Desc: Proton Power 15-046 activ. @ 700C - 21.8 % b/o  
Outgas Time: 2.5 hrs  
Analysis gas: Nitrogen  
Analysis Time: 836.7 min

**Report**  
Date: Wed Aug 17 03:59:30 2016  
Operator: carbon  
Filename: AS881601 PP 15-046 700C 22 BO.raw  
Date: 2016/11/10

**Data**  
Outgas Temp: 300.0 °C  
Molec. Wt: 28.0134 g  
Instrument: Autosorb Station 1  
Sample Weight: 0.0153 g  
Non-ideality: 6.58e-05 1/mmHg  
Bath temp.: 77.3 K

Isotherm : Linear

Adsorbate	Data Reduction Parameters		
	Thermal Transpiration: on	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Nitrogen	Temperature: 77.350 K	
	Molec. Wt.: 28.013	Cross Section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc



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Report id:[466251869:20161110 161838655] Page 1 of 1

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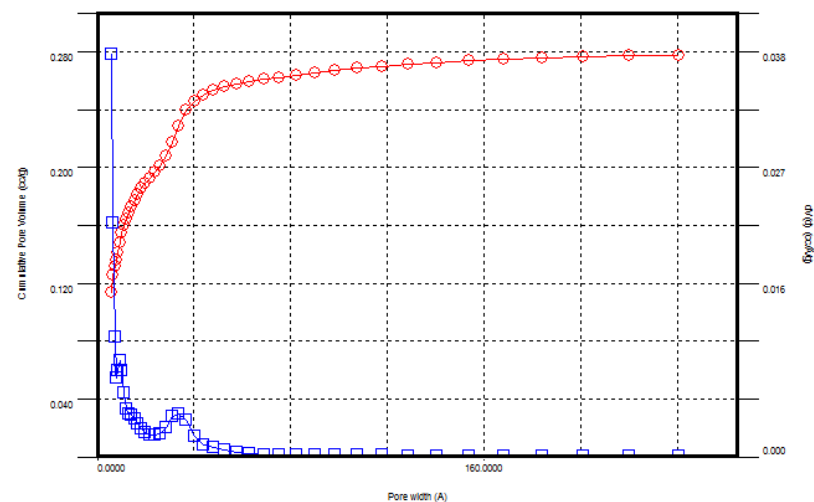
**Analysis**  
Operator: ng  
Sample ID: : Proton Power 15-046 activ. @ 700C - 21.8 % b/o  
Sample Desc: Proton Power 15-046 activ. @ 700C - 21.8 % b/o  
Outgas Time: 2.5 hrs  
Analysis gas: Nitrogen  
Analysis Time: 836.7 min

**Report**  
Date: Wed Aug 17 03:59:30 2016  
Operator: carbon  
Filename: AS881601 PP 15-046 700C 22 BO.raw  
Date: 2016/11/10

**Data**  
Outgas Temp: 300.0 °C  
Molec. Wt: 28.0134 g  
Instrument: Autosorb Station 1  
Sample Weight: 0.0153 g  
Non-ideality: 6.58e-05 1/mmHg  
Bath temp.: 77.3 K

DFT : dV(d)

DFT method	Data Reduction Parameters		
	Thermal Transpiration: off	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Calc. Model: N2 at 77 K on carbon (slit pore, QSDFT equilibrium model)	Temperature: 77.350 K	
	Rel. press. range: 0.0000 - 1.0000	Cross Section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc
Adsorbate	Nitrogen	Molec. Wt.: 28.013	Moving pt. avg: 3



**DFT method summary**  
Pore volume = 0.274 cc/g  
Surface area = 832.320 m<sup>2</sup>/g  
Lower confidence limit = 6.140 Å  
Fitting error = 0.288 %  
Pore width (Mode) = 6.140 Å  
Moving point average : 3

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Report id:[513626862:20161110 162135292] Page 1 of 1

# 15-046 activated at 800 °C (29 % BO)

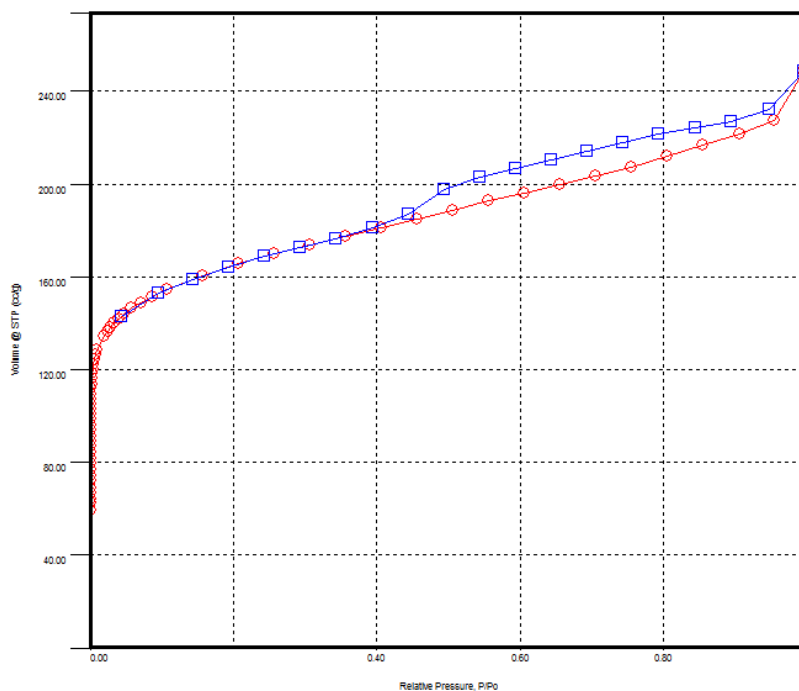
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**Analysis**  
Operator: cc Date: Fri Aug 19 06:08:14 2016 Operator: carbon Date: 2016/11/10  
Sample ID: : Proton Power 15-046 activ. @ 800C - 29.3 % b/o R Filename: AS881801 PP 15-046 800C 29 BO.raw  
Sample Desc: Proton Power 15-046 activ. @ 800C - 29.3 % b/o repe Comment:  
Outgas Time: 2.5 hrs Outgas Temp: 300.0 °C Sample Weight: 0.0138 g  
Analysis gas: Nitrogen Molec. Wt: 28.0134 g Non-ideality: 6.58e-05 1/mmHg  
Analysis Time: 758.7 min Instrument: Autosorb Station 1 Bath temp.: 77.3 K

**Isotherm : Linear**

Adsorbate	Thermal Transpiration: on	Data Reduction Parameters	Eff. cell stem diam. (d): 1.0000 mm
	Nitrogen	Eff. mol. diameter (D): 4.00 Å	
	Molec. Wt.: 28.013	Temperature: 77.350 K	
		Cross Section: 18.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc



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Report id: (896157127:20161110 162834470) Page 1 of 1

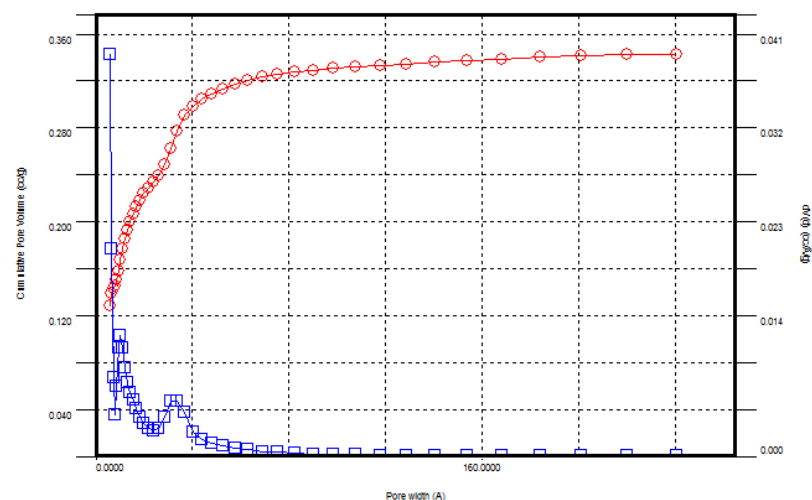
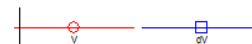
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**Analysis**  
Operator: cc Date: Fri Aug 19 06:08:14 2016 Operator: carbon Date: 2016/11/10  
Sample ID: : Proton Power 15-046 activ. @ 800C - 29.3 % b/o R Filename: AS881801 PP 15-046 800C 29 BO.raw  
Sample Desc: Proton Power 15-046 activ. @ 800C - 29.3 % b/o repe Comment:  
Outgas Time: 2.5 hrs Outgas Temp: 300.0 °C Sample Weight: 0.0138 g  
Analysis gas: Nitrogen Molec. Wt: 28.0134 g Non-ideality: 6.58e-05 1/mmHg  
Analysis Time: 758.7 min Instrument: Autosorb Station 1 Bath temp.: 77.3 K

**DFT : dV(d)**

Adsorbate	Thermal Transpiration: on	Data Reduction Parameters	Eff. cell stem diam. (d): 1.0000 mm
	Nitrogen	Eff. mol. diameter (D): 4.00 Å	
	Molec. Wt.: 28.013	Temperature: 77.350 K	
		Cross Section: 18.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc



**DFT method summary**  
Pore volume = 0.338 cc/g  
Surface area = 730.591 m<sup>2</sup>/g  
Lower confidence limit = 6.140 Å  
Fitting error = 0.324 %  
Pore width (Mode) = 6.140 Å  
Moving point average : 3

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Report id: (896053545:20161110 16305487) Page 1 of 1

# 15-049 as received

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**Analysis**  
Operator: CC  
Sample ID: 15-049 as received  
Sample Desc: 15-049 as received (Proton Power)  
Outgas Time: 6.0 hrs  
Analysis gas: Nitrogen  
Analysis Time: 712.0 min

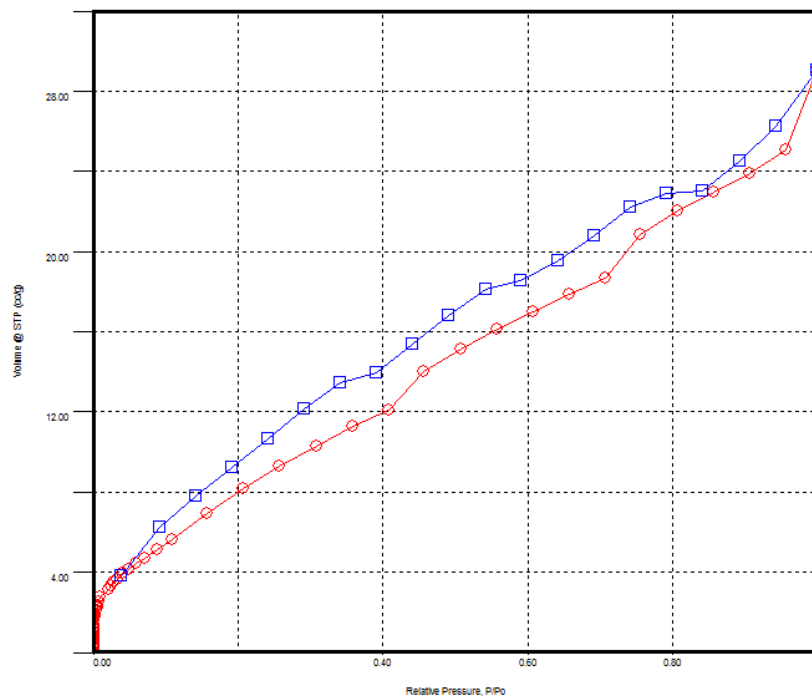
**Report**  
Date: Tue Mar 01 03:10:35 2016  
Filename: as622901 PP 15-049 full.raw  
Operator: carbon  
Comment: as622901 PP 15-049 full.raw  
Date: 2016/03/04

**Data Reduction Parameters**  
Eff. mol. diameter (D): 4.00 Å  
Temperature: 77.350 K  
Cross Section: 18.200 Å<sup>2</sup>  
Eff. cell stem diam. (d): 1.0000 mm  
Liquid Density: 0.808 g/cc

**Sample Weight:** 0.0185 g  
**Non-ideality:** 6.58e-05 1/mmHg  
**Bath temp.:** 77.3 K

**Isotherm : Linear**

<b>Adsorbate</b>	Thermal Transpiration: on	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
Nitrogen		Temperature: 77.350 K	
Molec. Wt.: 28.013		Cross Section: 18.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc



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Report id: [38471847:20160304 091422176] Page 1 of 1

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**Analysis**  
Operator: CC  
Sample ID: 15-049 as received  
Sample Desc: 15-049 as received (Proton Power)  
Outgas Time: 6.0 hrs  
Analysis gas: Nitrogen  
Analysis Time: 712.0 min

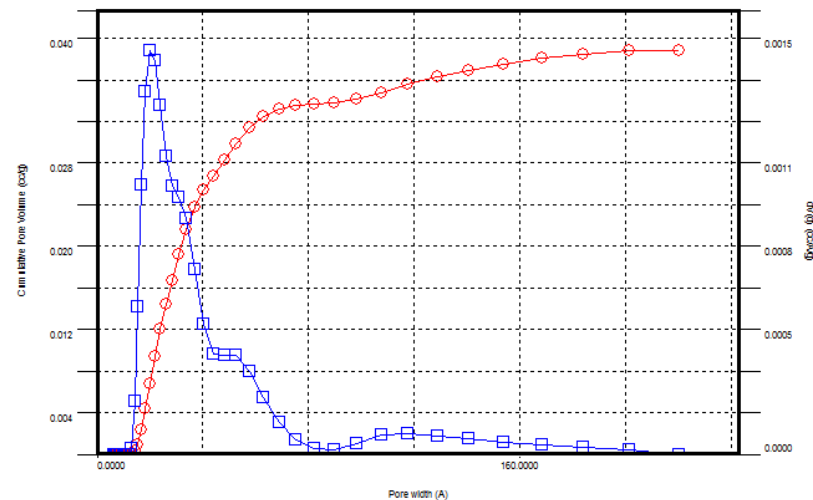
**Report**  
Date: Tue Mar 01 03:10:35 2016  
Filename: as622901 PP 15-049 full.raw  
Operator: carbon  
Comment: as622901 PP 15-049 full.raw  
Date: 2016/03/04

**Data Reduction Parameters**  
Eff. mol. diameter (D): 4.00 Å  
Temperature: 77.350 K  
Cross Section: 18.200 Å<sup>2</sup>  
Eff. cell stem diam. (d): 1.0000 mm  
Liquid Density: 0.808 g/cc

**Sample Weight:** 0.0185 g  
**Non-ideality:** 6.58e-05 1/mmHg  
**Bath temp.:** 77.3 K

**DFT : dV(d)**

<b>DFT method</b>	Thermal Transpiration: on	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Calc. Model: N2 at 77 K on carbon (slit pore, QSDFT equilibrium model)		
	Rel. press. range: 0.0000 - 1.0000		Moving pt. avg: 3
<b>Adsorbate</b>		Temperature: 77.350 K	
Nitrogen		Cross Section: 18.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc
Molec. Wt.: 28.013			



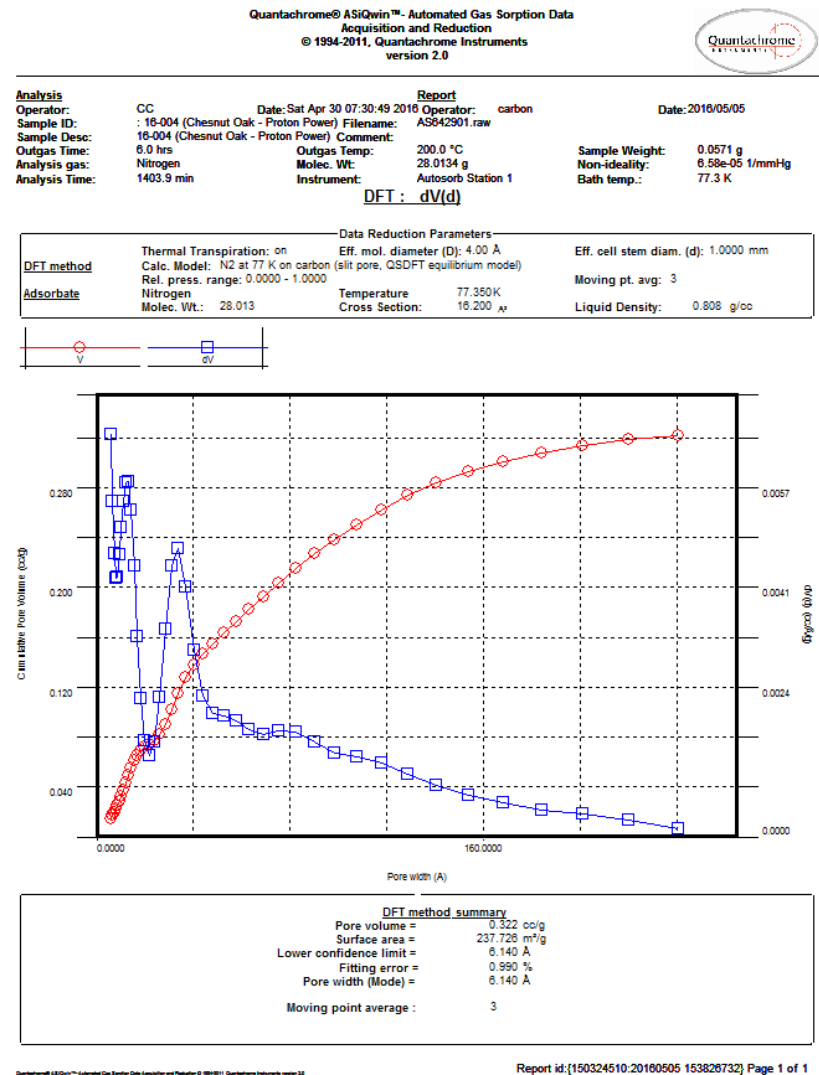
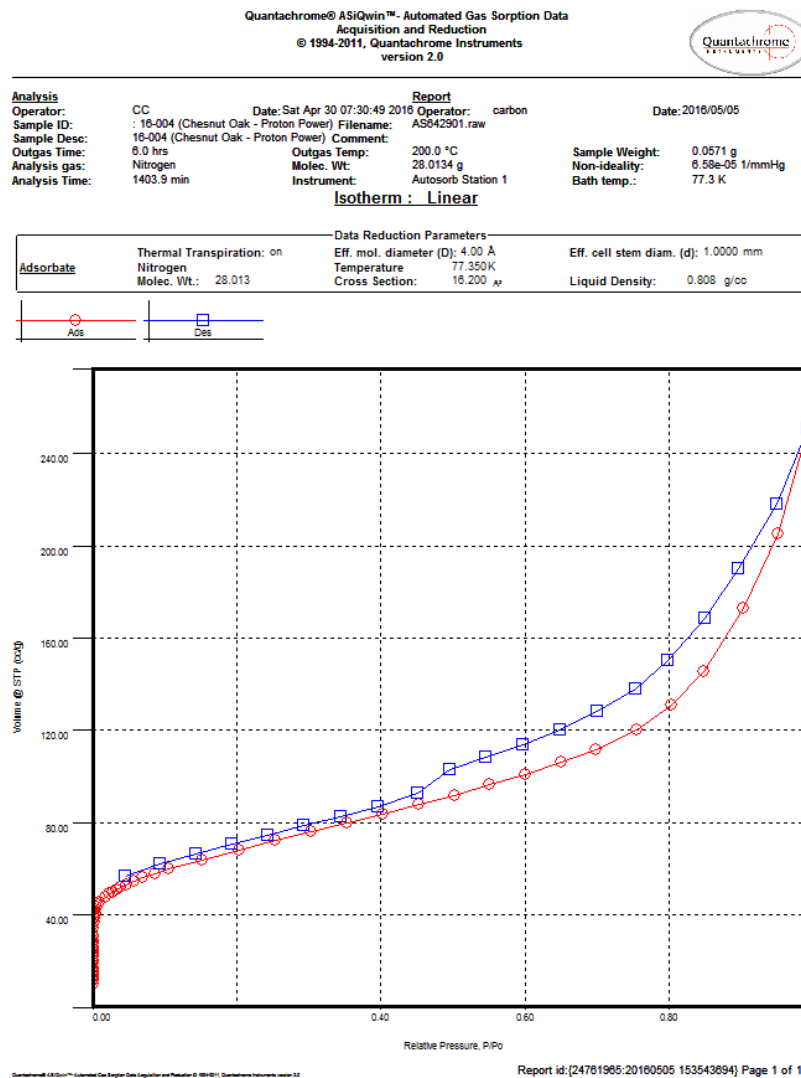
**DFT method summary**

Pore volume = 0.039 cc/g  
Surface area = 24.431 m<sup>2</sup>/g  
Lower confidence limit = 6.140 Å  
Fitting error = 2.851 %  
Pore width (Mode) = 20.000 Å  
Moving point average : 3

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Report id: [634696737:20160304 091902933] Page 1 of 1

# 16-004 as received





# 16-004 activated at 800 °C (27 % BO)

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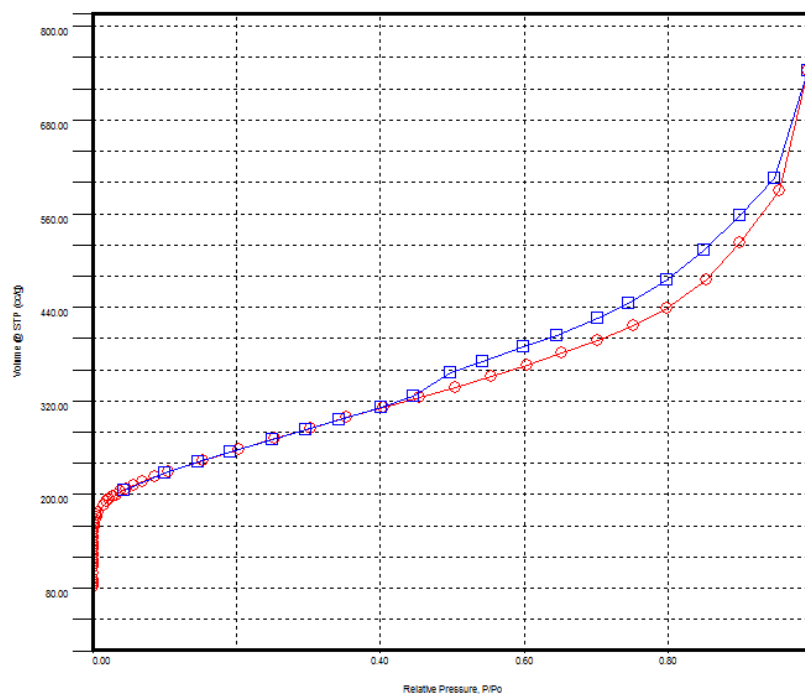
**Analysis**  
Operator: cc  
Sample ID: 16-004-800C 27% BO  
Sample Desc: 16-004-800C 27% BO  
Outgas Time: 21.0 hrs  
Analysis gas: Nitrogen  
Analysis Time: 799.7 min

**Report**  
Date: Thu Nov 17 22:59:00 2016  
Operator: carbon  
Filename: AS6B1701 PP 16-004 800C 27% BO.qps  
Comment: 350.0 °C  
Outgas Temp: 350.0 °C  
Molec. Wt: 28.0134 g  
Instrument: Autosorb Station 1

Date: 2016/11/18  
Sample Weight: 0.0101 g  
Non-ideality: 6.58e-05 1/mmHg  
Bath temp.: 77.3 K

Isotherm : Linear

Data Reduction Parameters			
Adsorbate	Thermal Transpiration: off	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Nitrogen	Temperature 77.350 K	
	Molec. Wt.: 28.013	Cross Section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc



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Report id: (809094074:20161118 120417245) Page 1 of 1

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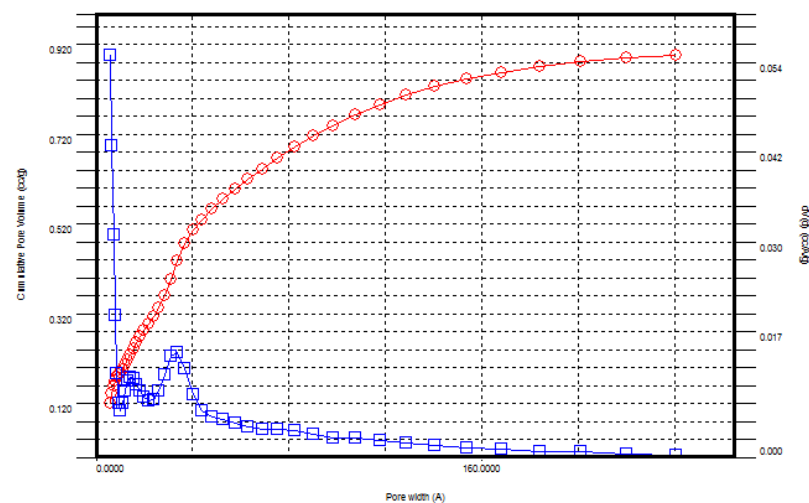
**Analysis**  
Operator: cc  
Sample ID: 16-004-800C 27% BO  
Sample Desc: 16-004-800C 27% BO  
Outgas Time: 21.0 hrs  
Analysis gas: Nitrogen  
Analysis Time: 799.7 min

**Report**  
Date: Thu Nov 17 22:59:00 2016  
Operator: carbon  
Filename: AS6B1701 PP 16-004 800C 27% BO.qps  
Comment: 350.0 °C  
Outgas Temp: 350.0 °C  
Molec. Wt: 28.0134 g  
Instrument: Autosorb Station 1

Date: 2016/11/18  
Sample Weight: 0.0101 g  
Non-ideality: 6.58e-05 1/mmHg  
Bath temp.: 77.3 K

DFT : dV(d)

Data Reduction Parameters			
DFT method	Thermal Transpiration: off	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Calc. Model: N2 at 77 K on carbon (slit pore, QSDFT equilibrium model)		
	Rel. press. range: 0.0000 - 1.0000		
Adsorbate	Nitrogen	Temperature 77.350 K	Moving pt. avg: 3
	Molec. Wt.: 28.013	Cross Section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc



**DFT method summary**  
Pore volume = 0.352 cc/g  
Surface area = 998.565 m<sup>2</sup>/g  
Lower confidence limit = 6.140 Å  
Fitting error = 0.896 %  
Pore width (Mode) = 6.140 Å  
Moving point average : 3

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Report id: (204197539:20161118 120844573) Page 1 of 1

# 16-005 as received

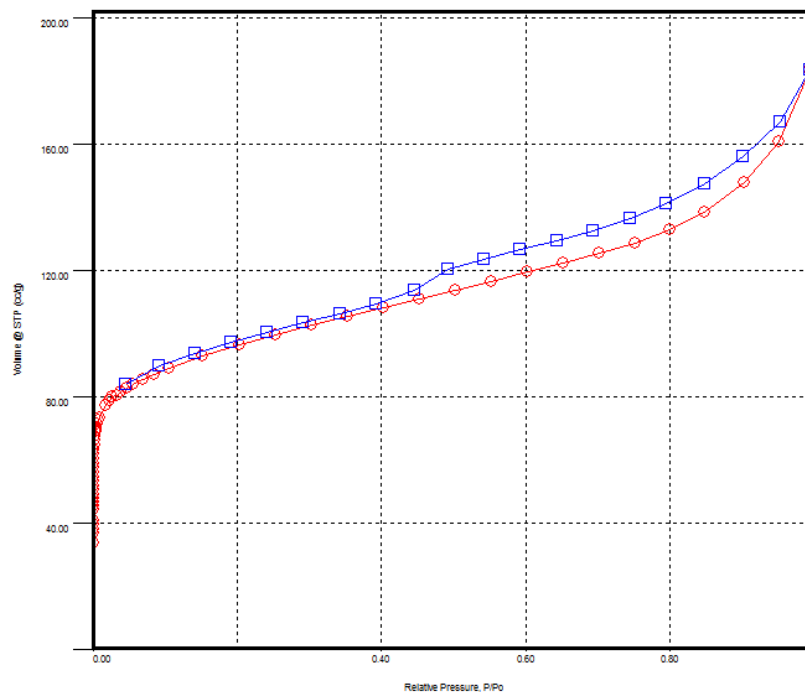
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**Analysis**  
Operator: CC Date: Tue May 03 11:27:05 2016 Report Operator: carbon Date: 2016/05/05  
Sample ID: : 16-005 (White Pine - Proton Power) Filename: AS650201.raw  
Sample Desc: 16-005 (White Pine - Proton Power) Comment:  
Outgas Time: 6.0 hrs Outgas Temp: 300.0 °C Sample Weight: 0.0571 g  
Analysis gas: Nitrogen Molec. Wt: 28.0134 g Non-ideality: 6.58e-05 1/mmHg  
Analysis Time: 1294.7 min Instrument: Autosorb Station 1 Bath temp.: 77.3 K

**Isotherm : Linear**

Adsorbate	Thermal Transpiration: on Nitrogen Molec. Wt.: 28.013	Data Reduction Parameters		Eff. cell stem diam. (d): 1.0000 mm
		Eff. mol. diameter (D): 4.00 Å Temperature 77.350 K Cross Section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc	



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Report id: {1384489782:20160505 154341358} Page 1 of 1

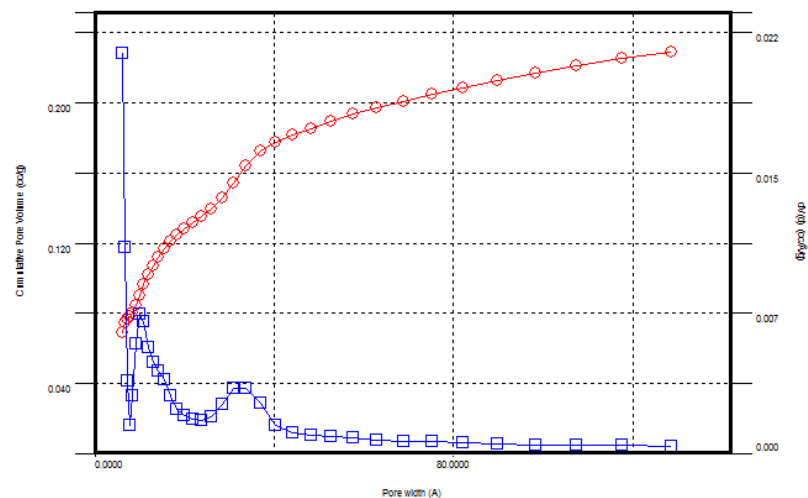
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**Analysis**  
Operator: CC Date: Tue May 03 11:27:05 2016 Report Operator: carbon Date: 2016/11/10  
Sample ID: : 16-005 (White Pine - Proton Power) Filename: AS650201.raw  
Sample Desc: 16-005 (White Pine - Proton Power) Comment:  
Outgas Time: 6.0 hrs Outgas Temp: 300.0 °C Sample Weight: 0.0571 g  
Analysis gas: Nitrogen Molec. Wt: 28.0134 g Non-ideality: 6.58e-05 1/mmHg  
Analysis Time: 1294.7 min Instrument: Autosorb Station 1 Bath temp.: 77.3 K

**DFT : dV(d)**

Adsorbate	Thermal Transpiration: on Calc. Model: N2 at 77 K on carbon Rel. press. range: 0.0000 - 1.0000 Nitrogen Molec. Wt.: 28.013	Data Reduction Parameters		Eff. cell stem diam. (d): 1.0000 mm
		Eff. mol. diameter (D): 4.00 Å (slit pore, QSDFT equilibrium model) Temperature 77.350 K Cross Section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc	

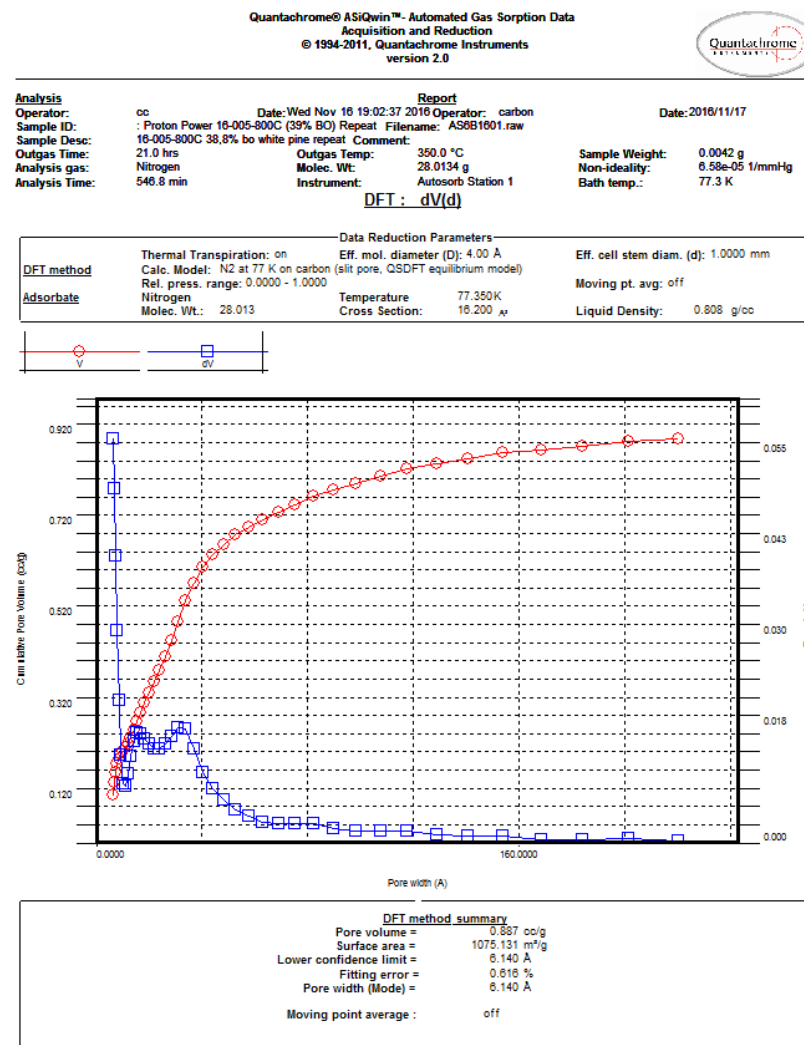
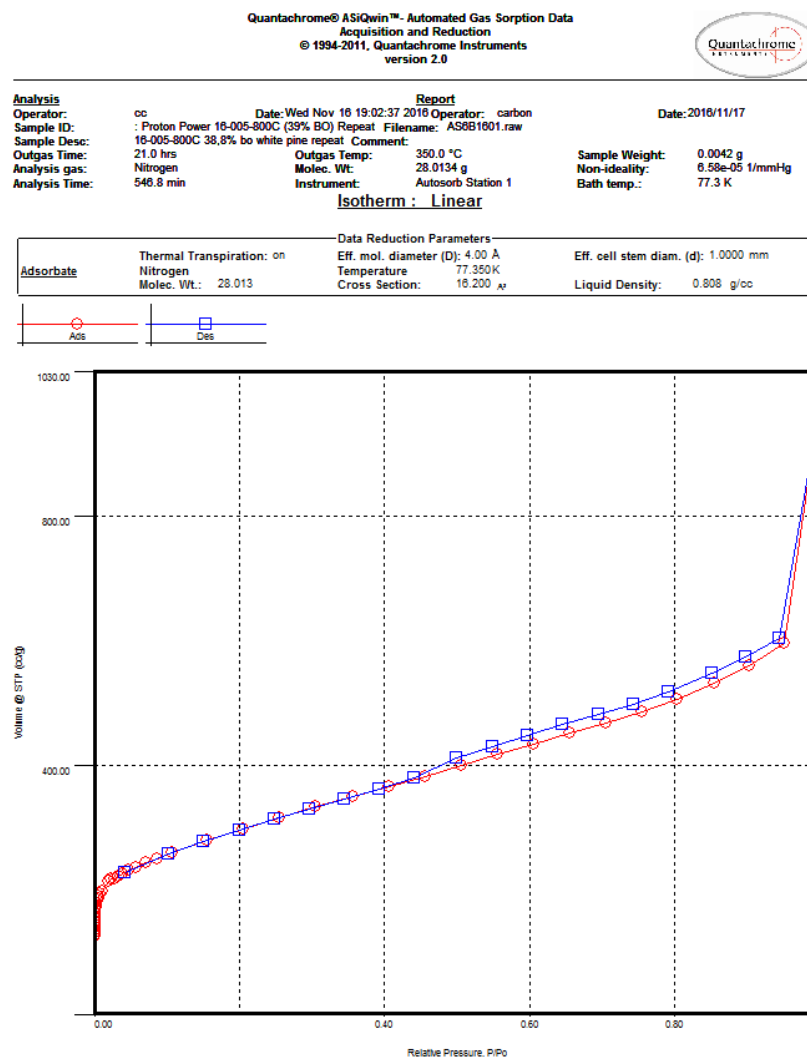


**DFT method summary**  
Pore volume = 0.227 cc/g  
Surface area = 416.833 m<sup>2</sup>/g  
Lower confidence limit = 6.140 Å  
Fitting error = 0.494 %  
Pore width (Mode) = 6.140 Å  
Moving point average : 3

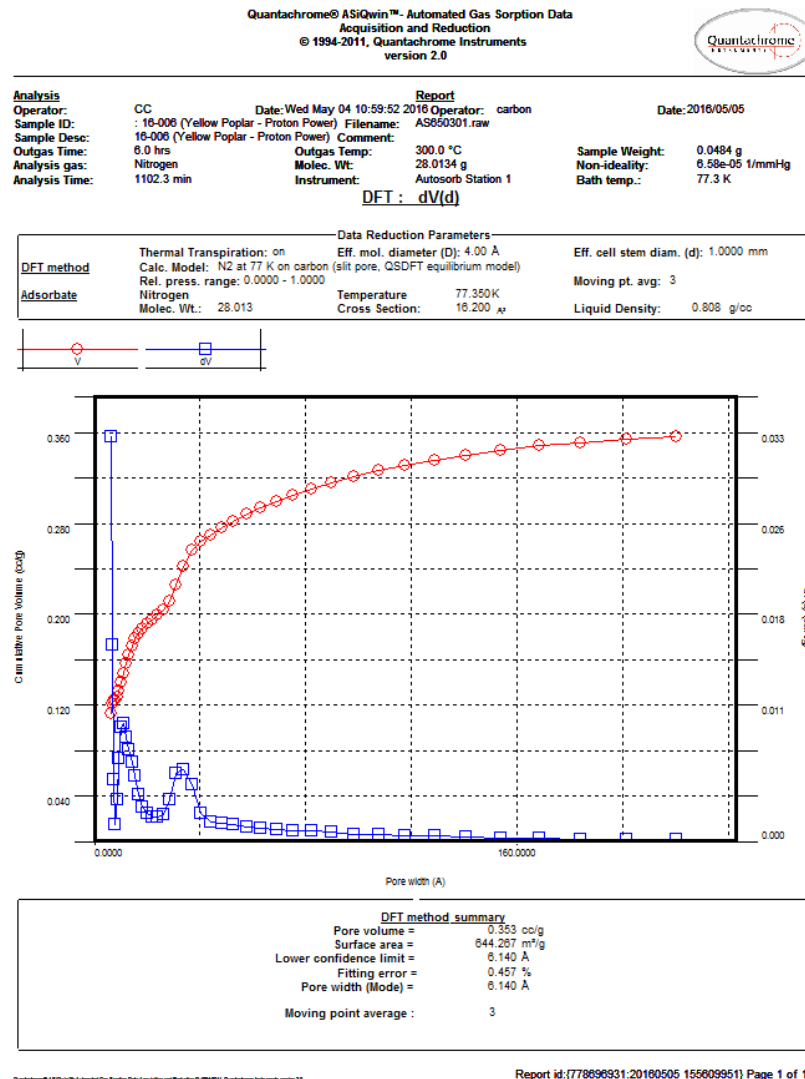
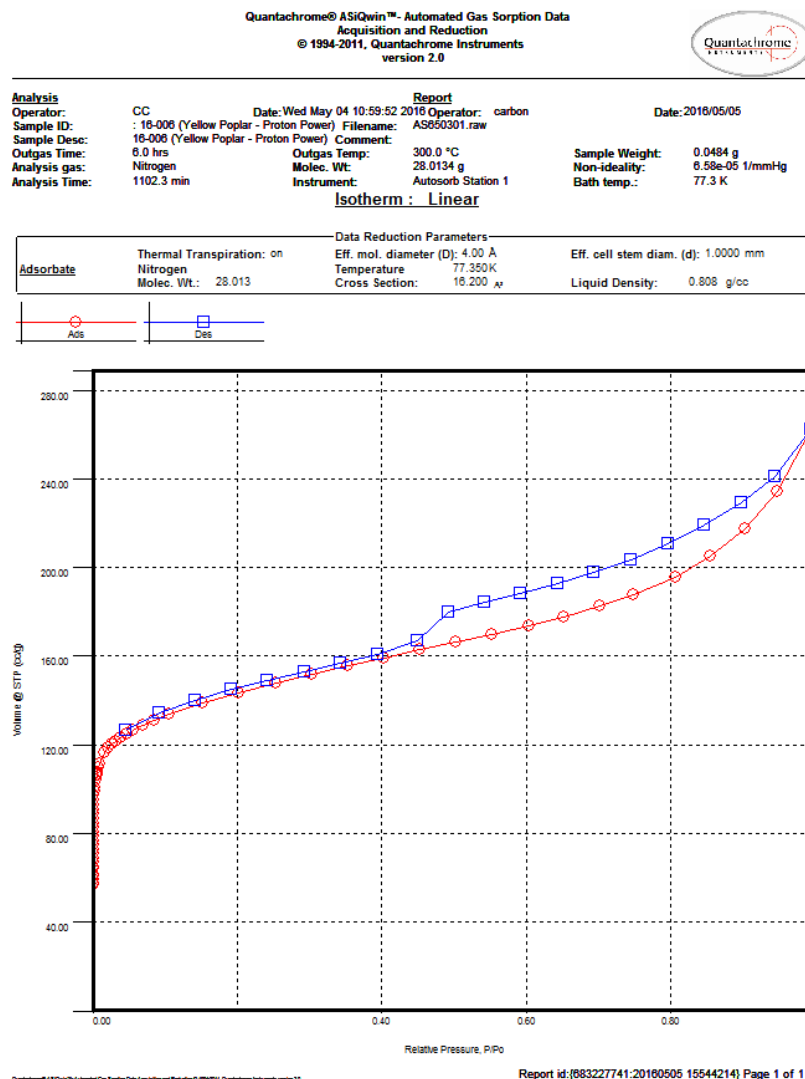
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Report id: {726196321:20161110 164753841} Page 1 of 1

# 16-005 activated at 800 °C (39 % BO)



# 16-006 as received



# 16-006 activated at 800 °C (47 % BO)

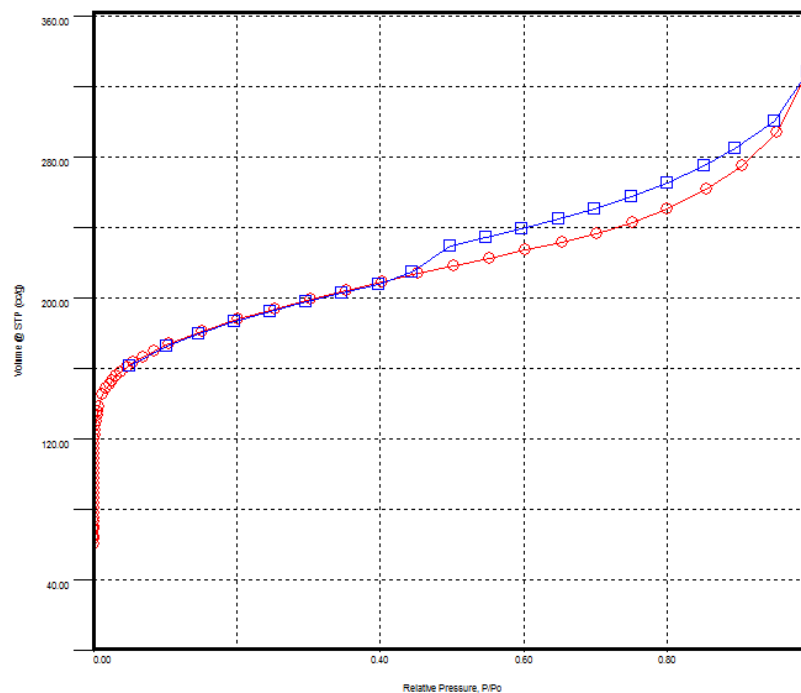
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**Analysis**  
Operator: cc Date: Tue Sep 27 05:22:51 2016 Operator: carbon Date: 2016/11/10  
Sample ID: : Proton Power 16-006 activ. @ 800C - 47.14 % b/o Filename: AS892601 PP 16-006 800C 47 BO.raw  
Sample Desc: : Proton Power 16-006 activ. @ 800C - 47.14 % b/o Comment:  
Outgas Time: 3.0 hrs Outgas Temp: 300.0 °C  
Analysis gas: Nitrogen Molec. Wt.: 28.0134 g Non-ideality: 6.58e-05 1/mmHg  
Analysis Time: 1069.2 min Instrument: Autosorb Station 1 Bath temp.: 77.3 K

Isotherm : Linear

Adsorbate	Data Reduction Parameters		
	Thermal Transpiration: on	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Nitrogen	Temperature: 77.350 K	
	Molec. Wt.: 28.013	Cross Section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc



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Report id:[629781909:20161110 17044752] Page 1 of 1

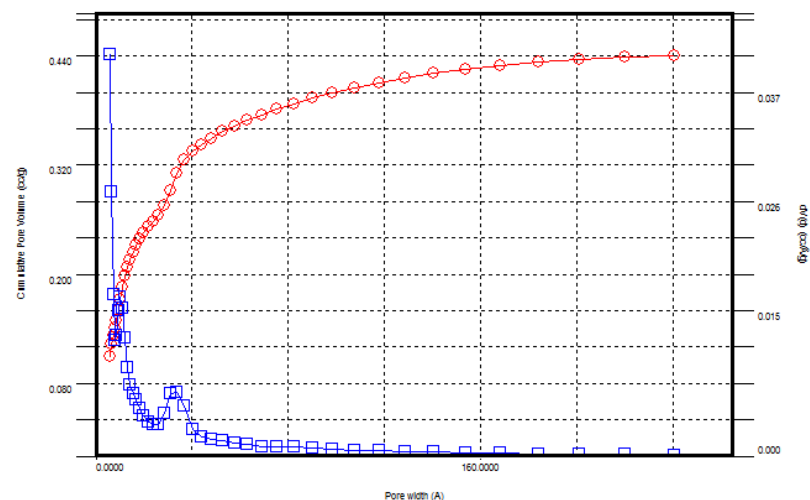
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**Analysis**  
Operator: cc Date: Tue Sep 27 05:22:51 2016 Operator: carbon Date: 2016/11/10  
Sample ID: : Proton Power 16-006 activ. @ 800C - 47.14 % b/o Filename: AS892601 PP 16-006 800C 47 BO.raw  
Sample Desc: : Proton Power 16-006 activ. @ 800C - 47.14 % b/o Comment:  
Outgas Time: 3.0 hrs Outgas Temp: 300.0 °C  
Analysis gas: Nitrogen Molec. Wt.: 28.0134 g Non-ideality: 6.58e-05 1/mmHg  
Analysis Time: 1069.2 min Instrument: Autosorb Station 1 Bath temp.: 77.3 K

DFT : dV(d)

DFT method	Data Reduction Parameters		
	Thermal Transpiration: off	Eff. mol. diameter (D): 4.00 Å	Eff. cell stem diam. (d): 1.0000 mm
	Calc. Model: N2 at 77 K on carbon (slit pore, QSDFT equilibrium model)		
	Rel. press. range: 0.0000 - 1.0000		Moving pt. avg: 3
Adsorbate	Nitrogen	Temperature: 77.350 K	
	Molec. Wt.: 28.013	Cross Section: 16.200 Å <sup>2</sup>	Liquid Density: 0.808 g/cc



**DFT method summary**  
Pore volume = 0.437 cc/g  
Surface area = 783.246 m<sup>2</sup>/g  
Lower confidence limit = 6.140 Å  
Fitting error = 0.404 %  
Pore width (Mode) = 6.140 Å  
Moving point average : 3

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Report id:[280870057:20161110 170705691] Page 1 of 1