

Certificate Number: IWTN/COA/W663/001 (14 May 2014)

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Material:	TUBALL SINGLE WALL CARBON NANOTUBES
Customer reference N°:	Lot 4 – 18032014 (Homogenised)
Date of Sample Receipt:	26 March 2014
Intertek Wilton Sample Ref. N° :	IWTN/W000000663

General Comments

The analyses carried out show that this sample contains a high concentration of single wall carbon nanotubes. All measurements were made on the dry powder as supplied; the sample was not dispersed or purified in any way prior to analysis.

Results of Analysis

Specification	Value	Method
Total carbon	~85 % w/w	Oxidative Combustion ¹ , TGA ²
Nanotube purity (T1%)	~74% +/- 1.5%	TGA ³
Primary oxidation peak (T _{ox})	Mean = 615°C, σ = 1.73°C	TGA
Main non-carbon species detected ⁴	Fe, O, Ni, Si, Cr, Na, S	EDX, XRF, CHNO
Raman G/D ratio	30.5 +/- 2.3 86.5 +/- 7.1 (non - homogenised)	Raman, 633nm ⁵
Main tube diameters - metallic	1.25, 1.30, 1.47, 1.58 nm	Raman 633nm and 785nm ⁶
Main tube diameters - semiconducting	1.43, 1.63, 1.66, 1.78, 2.01nm	Raman 633nm and 785nm ⁶
Approximate average tube diameter	~1.5nm	TEM ⁷

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Notes on Results of Analysis

1 Oxidative combustion with thermal conductivity detection. Average of 2 samples, effective combustion temperature ~ 1800°C.

2 Calculated from TGA residual weight at 800°C, assuming residue is 100% Fe₂O₃. Average of 3 samples.

3 T1% SWCNT purity figure calculated using method of R Jansen and P Wallis, Materials Matters (2009), 4.1, 23. Average of 3 samples, +/- 95% confidence limits.

4 Predominant non-carbon element is Fe.

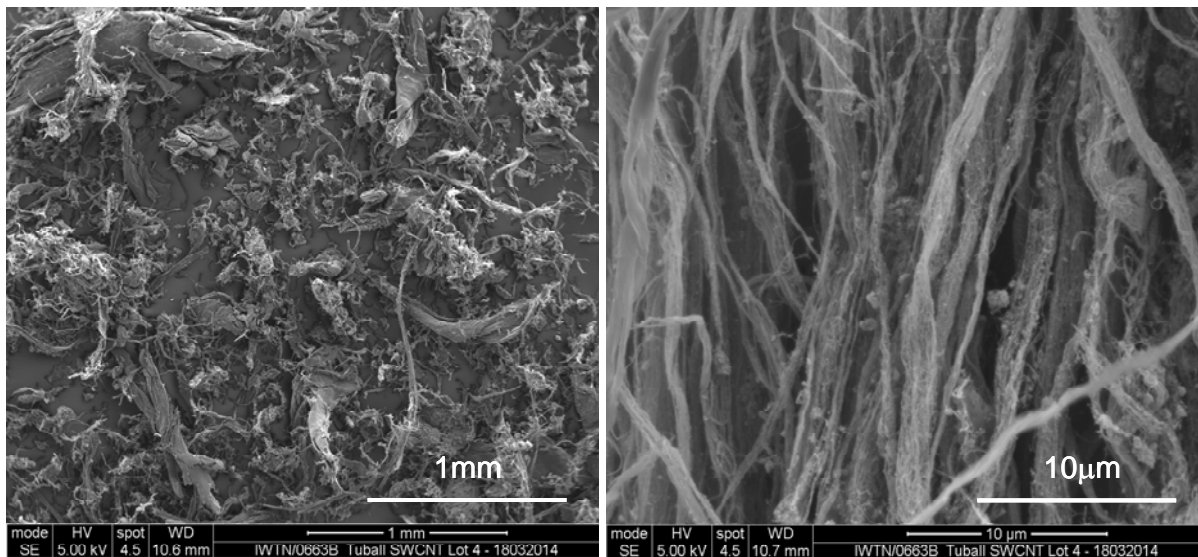
5 Raman G/D ratio computed using baseline-corrected peak heights for G⁺ and D band maxima. Average of 62 measurements, +/- 95% confidence limits. Result for non-homogenised sample also reported (61 samples).

6 Tube diameters computed using equation $d(\text{nm})=223/(\omega-10)$, where ω = Raman shift of radial breathing mode. Metallic/semiconducting assignment made on basis of Kataura plot (A. R. T. Nugraha, R. Saito, K. Sato, P. T. Araujo, A. Jorio, and M. S. Dresselhaus, Appl. Phys. Lett. 97, 091905 (2010)), and the position/shape of the G⁻ band.

7 Tubes observed by TEM were closely packed in ribbons so unable to accurately measure individual tube diameters.

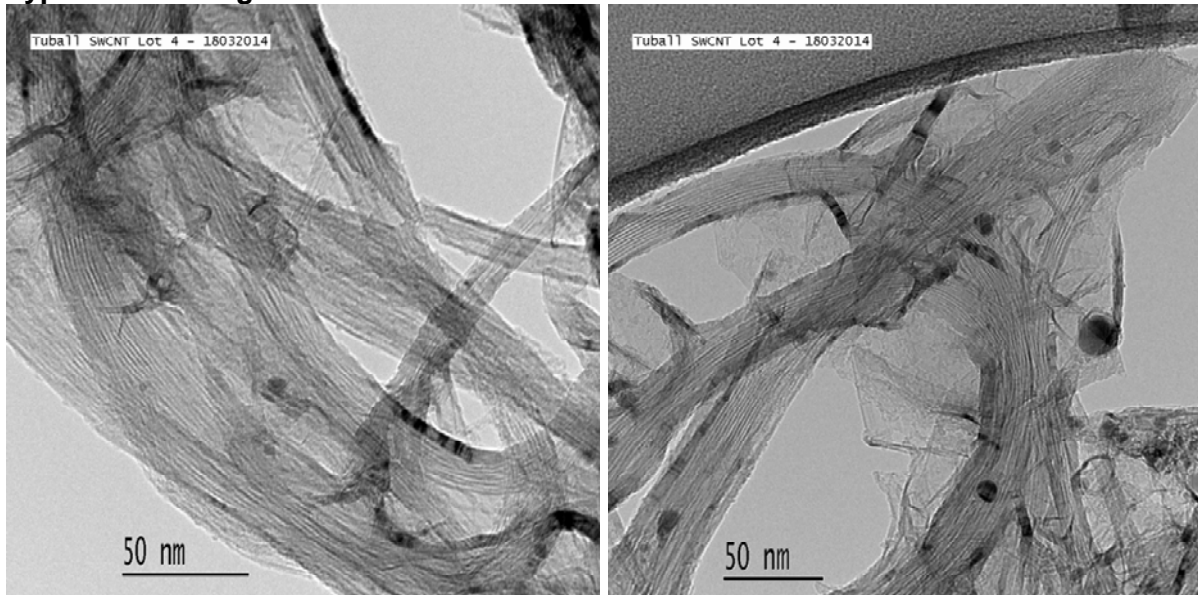
Supporting Information

Typical SEM Images

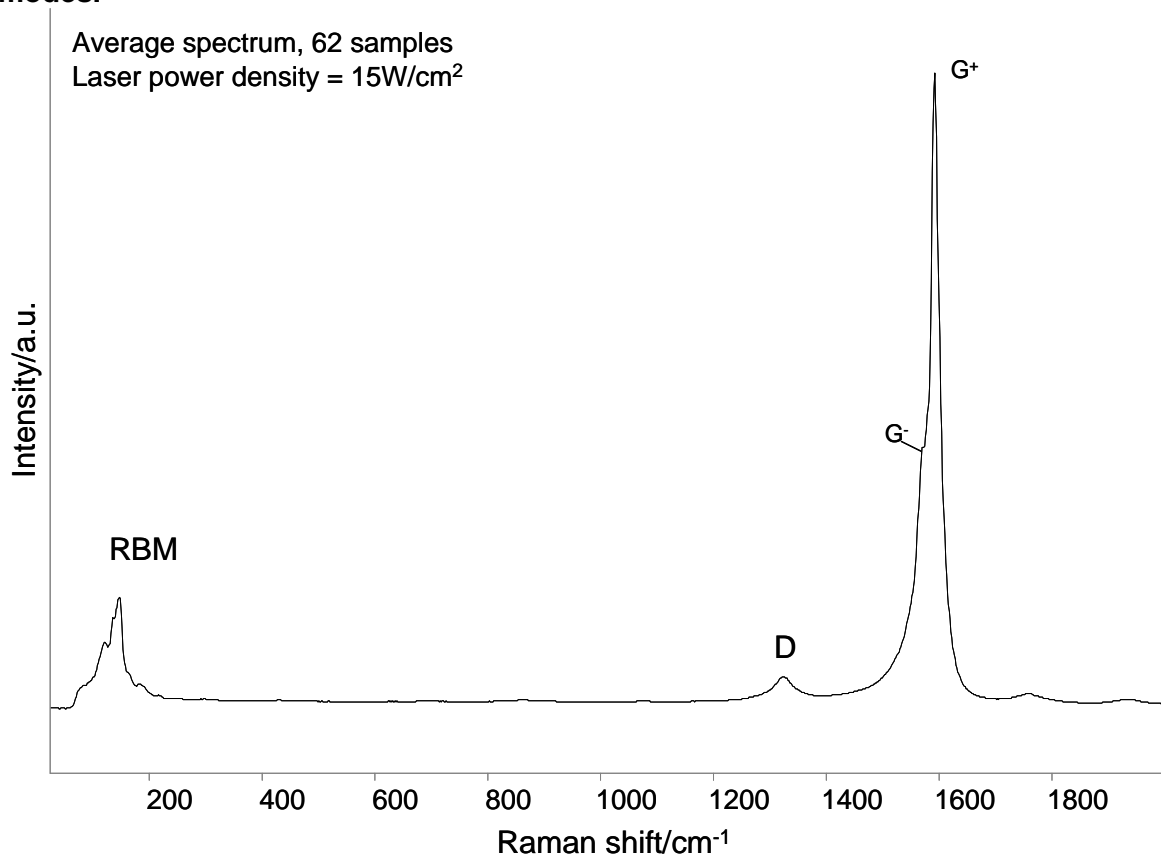


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Typical TEM Images

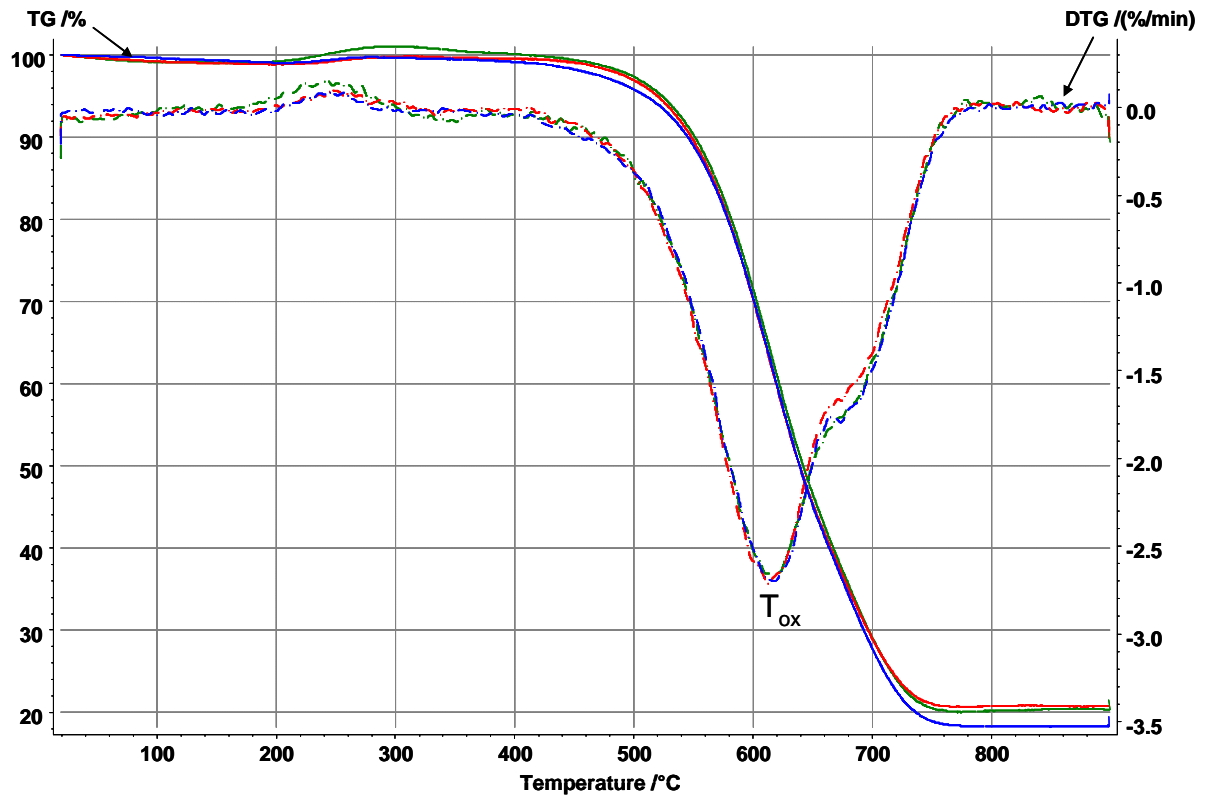


The SEM and TEM data show a high loading of nanotubes with varying degrees of aggregation and bundling. Dark regions in TEM are predominantly iron.


Raman spectrum of powder showing high G/D ratio and well-resolved radial breathing modes.

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TGA data



This figure shows the TG and DTG traces for three samples, confirming good reproducibility of the number of carbon species, the primary oxidation temperature (T_{ox}), and the residual mass. Scan rate 5°C/min

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Procedures/Guidelines followed

Technique	Method reference	Primary Report Identifier(s)
TGA	BSI Standard DD ISO/TS 11308:2011(E)	IWTN/W000000663ARL001
SEM/TEM	BSI Standard DD ISO/TS 10798:2011(E) BSI Standard DD ISO/TS 10797:2012(E) MSG-LAB-SOP-33,58,59	IWTN/W000000663BRL001
Raman	MSG-LAB-SOP-IR-60 NIST special publication 960-19 (2008) section 4 pages 36-54	IWTN/W000000663RL001 IWTN/W000000828RL001
Oxidative Combustion (CHNO)	MSG-LAB-SOP-XRF-101	IWTN/W000000663FRL001 IWTN/W000000663GRL001
Semi-quant XRF	MSG-LAB-SOP-XRF-99	IWTN/W000000663GRL001

Deviations from Guidelines

Method reference	Section	Details
ISO/TS 11308:2011(E) (TGA)	6.1 Sample pan selection	Method specifies use of aluminium pans, but these would melt under the conditions specified for the scan. Alumina pans were substituted.
R. Jansen and P. Wallis, Materials Matters (2009), 4.1, 23. (T1% Purity Calculation)	TGA	Equation given in reference for T1% calculation is incorrect. Correct equation (shown below) was used: $T1\% = 100 \cdot \left(\frac{\text{Weight\% at } 200^{\circ}\text{C} - T1\% \text{ measured}}{\text{Weight\% at } 200^{\circ}\text{C} - \text{Weight\% at } 800^{\circ}\text{C}} \right)$

Certificate Number: IWTN/COA/W663/001 (14 May 2014)**Authorisations**

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