Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.

- Supplementary Information -

Quantitative monitoring of the removal of non-encapsulated material external to filled carbon nanotube samples

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Table S1 – TGA residue with the corresponding filling yield (FY) of samples of $SmCl_3$ filled carbon nanotubes free of external $SmCl_3$. The TGA analysis was performed under flowing air. The same sample is marked (•). The iron content in the samples was calculated from the initial value of iron in empty single-walled carbon nanotubes (1.5 wt% of iron). The samples detailed below correspond to those analyzed in Figure 4; see Fig.4 caption for the detailed list of parameters employed.

	Washing	Residue/wt.%	FY/wt.%	Fe/wt.%
Time of stirring (*t*)	10 min•	15.8	20.9	1.2
	1 h	15.4	20.3	1.3
	1 day	15.4	20.2	1.3
Temperature (*T*)	50 ºC	16.7	22.2	1.2
	80 ºC•	15.8	20.9	1.2
	110 ºC	16.2	21.5	1.2
Volume (*V*)	50 mL	16.2	21.4	1.2
	100 mL•	15.8	20.9	1.2
	200 mL	16.1	21.2	1.2
Sonication time (t_s)	0 min•	15.8	20.9	1.2
	5 min	15.8	20.8	1.2
	10 min	15.4	20.2	1.3

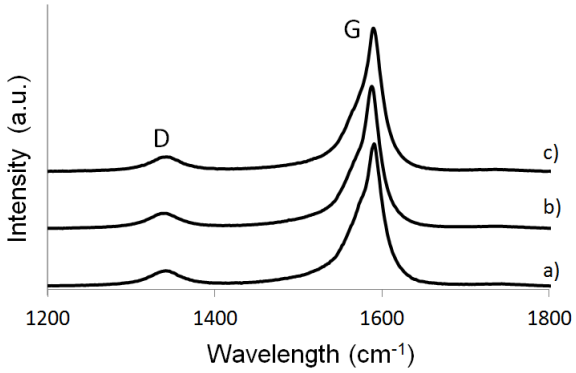

Fig. S1 – Raman spectra of $SmCl_3$ filled carbon nanotube samples cleaned a) without sonication, b) with 5 min sonication and c) with 10 min of sonication. The D to G band intensity ratio (I_D/I_G) of the samples is: non-sonicated: 0.123 ± 0.011; after 5 min sonication: 0.129 ± 0.025; after 10 min sonication: 0.125 ± 0.025.

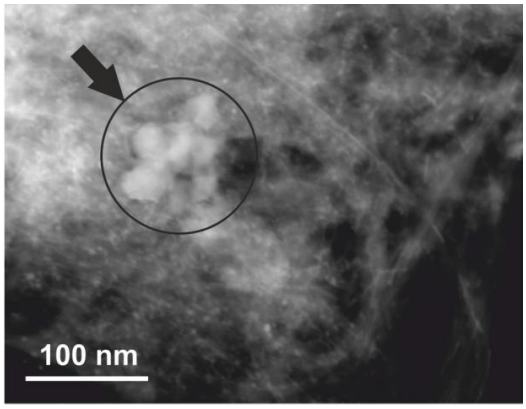

Fig. S2 – HAADF STEM image of $SmCl_3$ filled single-walled carbon nanotubes after partial washing, showing both the presence of external material (in the black circle where the arrow is pointing) and filling of carbon nanotubes (white strings).

Determination of the filling yield:

The filling yield (FY) can be calculated knowing the amount of iron in empty nanotubes[1]:

$$\mathrm{FY(wt\%)} = \frac{100 \times (R_2 - R_1)}{R_\mathrm{A} - R_1}$$

where R_1 and R_2 represent TGA residues of empty and filled carbon nanotubes respectively, while R_A can be calculated knowing the stoichiometry of the oxidative reaction which is taking place during the TGA analysis:

$$R_\mathrm{A} = \frac{100 \times y \times M_\mathrm{B}}{x \times M_\mathrm{A}}$$

Where M_A and M_B are molecular weights of A and B, while x and y are the reaction stoichiometric constants.

After burning the sample for TGA analysis, if the sample is not properly washed, the samarium(III) oxide content can be higher than expected due to the presence of external material that gives rise to false positive values.

[1] Ballesteros B, Tobias G, Ward MAH, Green MLH. Quantitative Assessment of the Amount of Material Encapsulated in Filled Carbon Nanotubes. Journal of Physical Chemistry C 2009;113:2653-6.