

Validated Protocols

for medical device characterisation along the AM process chain, based on advanced and routine characterisations

Deliverable D4

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Project 15HLT09 "MetAMMI Metrology for additively manufactured medical implants".

Due date: 31st May 2019

Submission date: 25th June 2019



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Introduction

With the ability to produce complex and customized patient specific medical devices on demand, the Additive Manufacturing (AM) technology is of growing interest for the medical sector. As the technology developed at a much faster pace than regulations or quality control, there is currently a lack of validated techniques to verify the finished parts. The intention of the MetAMMI project is to diminish these gaps and to increase confidence of the medical device industry in the AM technology.

In the framework of the MetAMMI project, various dense and lattice parts have been manufactured using different AM technologies and different materials. The parts have been characterized and typical geometrical deviations have been identified as well as their origin in the process chain. Based on these information, validated techniques for the identification of typical deviations have been investigated. This document gathers validated protocols for medical device characterisation along the AM process chain, for both advanced and routine characterisation techniques. For clarity purposes, the characterisation techniques are not described, as a more complete overview of each technique presented here can be found in *D2 Good practice guide on the correct choice of characterisation technique depending of the level of accuracy needed and the type of measurement required*.

X-ray computed tomography

X-ray computed tomography (CT or XCT) is a powerful non-destructive technique that allows obtaining a 3D digital image of a specimen under investigation. As such, both dimensional measurements and defect detection can be performed simultaneously. As a result, are presented in this section 3 validated protocols for CT, dealing with a specific task: dimensional measurements, dimensional errors, and defect detection.

Dimensions of AM parts

The protocol for dimension assessment of AM parts is presented in the workflow shown **Figure 1**, and is described in detail below.

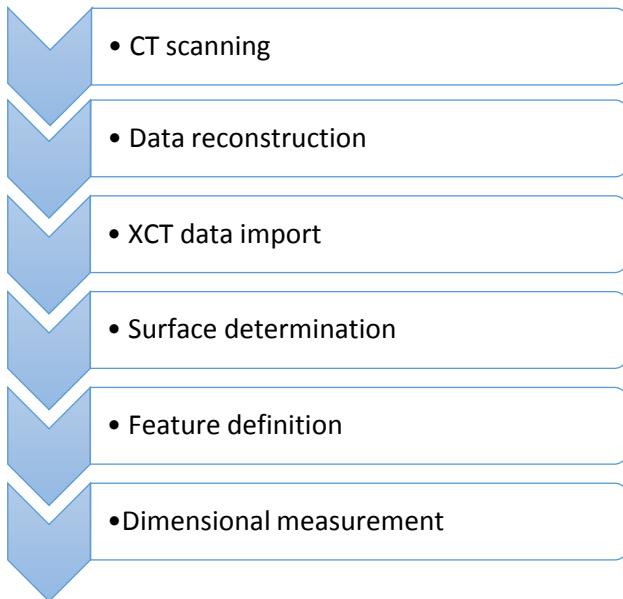
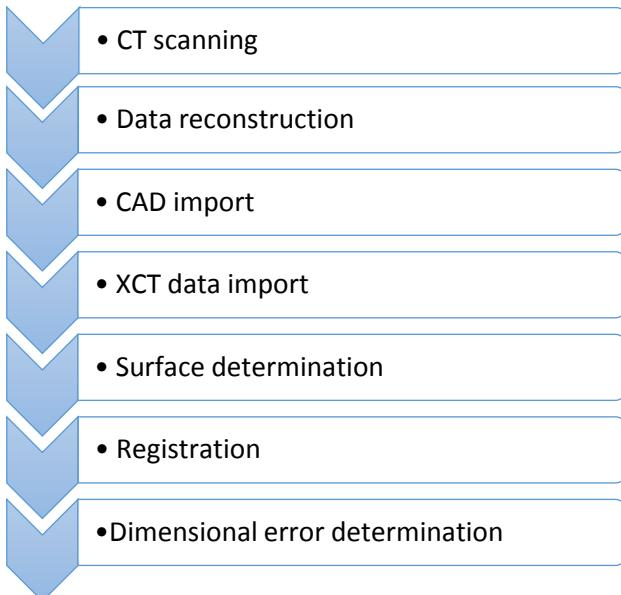


Figure 1: workflow for dimension assessment of AM parts by XCT.

In the first step, the CT scanning of the AM part is performed. It is important for the scanning parameters to be recorded, and one output of the MetAMMI project was the creation of a measurement protocol (see Appendix 1). In such protocol, the general data, system settings, and acquisition parameters are recorded. The second step is the data reconstruction, which allows the acquired 2D set of projections to be reconstructed into a 3D volume. As several reconstruction parameters can significantly impact the data quality, hence the associated dimensional measurements, we recommend the recording of the main reconstruction parameters (such as beam hardening correction values...). In the third step, the XCT data is imported into a data processing software. For accurate dimensional measurements, a verification of the voxel size is recommended (global scaling error correction). Standard calibrated pieces are available and can be scanned before and/or after the sample, or placed within the measuring volume during scanning. The dimensional measurement of the calibrated artefact of a known dimension is used as a reference measurement and a global scaling error correction can thus be performed. In step 4, the surface of the AM part is defined. This can be done by several different ways, including global thresholding, local thresholding, adaptive thresholding, etc depending on the type of data processing software employed. The type of thresholding method employed will have an impact on the dimensional measurements. Once the surface of the AM part has been defined, the features of interest have to be defined. Geometrical primitives such as planes, cylinders or spheres can be fitted to act as reference features from which the dimensional measurements are performed. Dimensional measurements can also be performed from surface point to surface point.

Dimensional errors of AM parts

The protocol for dimensional error assessment of AM parts is presented in the workflow shown **Figure 2**, and is described in detail below.



Dimensional errors imply that the dimensional measurements are compared to a reference value. For AM parts, the reference value is usually the CAD file used as an input for the 3D printer. In the first step, the CT scanning of the AM part is performed. It is important for the scanning parameters to be recorded, and one output of the MetAMMI project was the creation of a measurement protocol (see Appendix 1). In such protocol, the general data, system settings, and acquisition parameters are recorded. The second step is the data reconstruction, which allows the acquired 2D set of projections to be reconstructed into a 3D volume. As several reconstruction parameters can significantly impact the data quality, hence the associated dimensional measurements, we recommend the recording of the main reconstruction parameters (such as beam hardening correction values...). In the third step, the CAD file is imported in the data processing software. Using the coordinate system from the CAD file allows for having the same reference system for each XCT sample, regardless of the XCT scanning orientation of the AM part. Then, the XCT data is also imported into a data processing software. For accurate dimensional measurements, a verification of the voxel size is recommended (global scaling error correction). Standard calibrated pieces are available and can be scanned before and/or after the sample, or placed within the measuring volume during scanning. The dimensional measurement of the calibrated artefact of a known dimension is used as a reference measurement and a global scaling error correction can thus be performed. In step 4, the surface of the AM part is defined. This can be done by several different ways, including global thresholding, local thresholding, adaptive thresholding, etc depending on the type of data processing software employed. The type of thresholding method employed will have an impact on the dimensional measurements. Once the surface of the AM part has been defined, that surface needs to be registered against the CAD file, which is acting as a reference. When possible, a feature-based registration should be performed, as it is the most robust type of registration. Alternatively, a manual registration followed by a best fit registration is advised. Finally, the dimensional error determination, or nominal-actual comparison, is performed, where the distance between the surface of the CT data and the corresponding surface of the CAD file is measured. The results can then be plotted as a histogram (probability in % versus deviation in μm) or visualized in a 3D viewer with a colour coding.

Figure 2: workflow for dimensional error assessment of AM parts by XCT.

reference system for each XCT sample, regardless of the XCT scanning orientation of the AM part. Then, the XCT data is also imported into a data processing software. For accurate dimensional measurements, a verification of the voxel size is recommended (global scaling error correction). Standard calibrated pieces are available and can be scanned before and/or after the sample, or placed within the measuring volume during scanning. The dimensional measurement of the calibrated artefact of a known dimension is used as a reference measurement and a global scaling error correction can thus be performed. In step 4, the surface of the AM part is defined. This can be done by several different ways, including global thresholding, local thresholding, adaptive thresholding, etc depending on the type of data processing software employed. The type of thresholding method employed will have an impact on the dimensional measurements. Once the surface of the AM part has been defined, that surface needs to be registered against the CAD file, which is acting as a reference. When possible, a feature-based registration should be performed, as it is the most robust type of registration. Alternatively, a manual registration followed by a best fit registration is advised. Finally, the dimensional error determination, or nominal-actual comparison, is performed, where the distance between the surface of the CT data and the corresponding surface of the CAD file is measured. The results can then be plotted as a histogram (probability in % versus deviation in μm) or visualized in a 3D viewer with a colour coding.

Defect detection of AM parts

The protocol for defect detection in AM parts is presented in the workflow shown **Figure 3**, and is described in detail below.

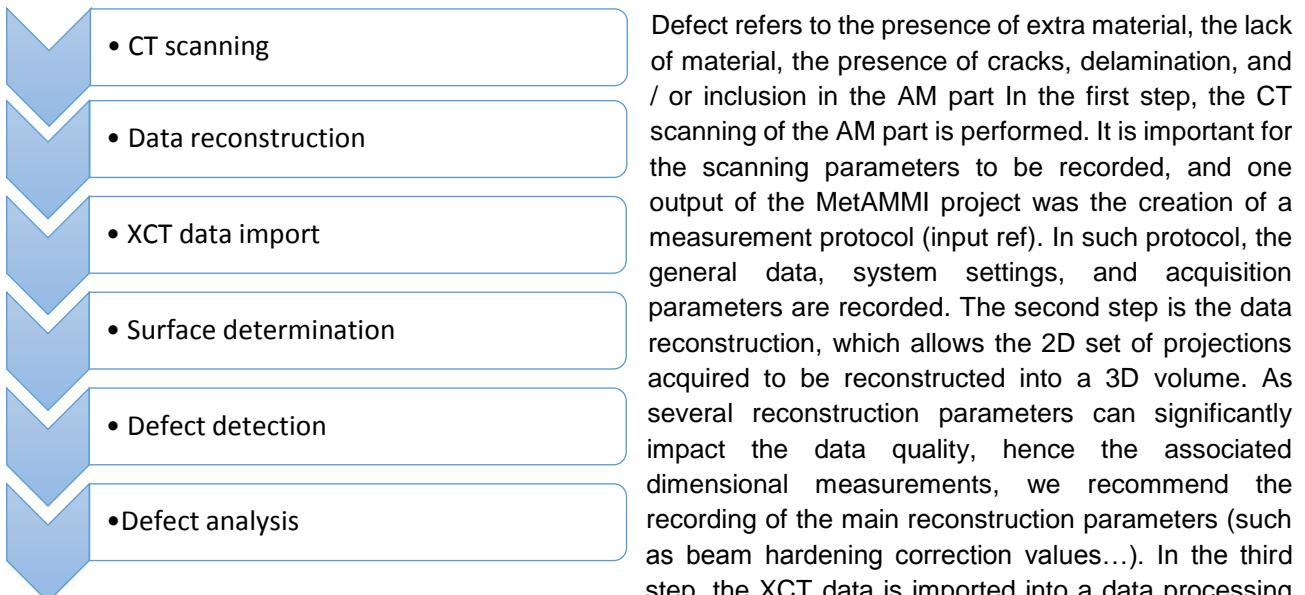


Figure 3: workflow for defect detection in AM parts by XCT.

available and can be scanned before and/or after the sample, or placed within the measuring volume during scanning. The measurement of the calibrated artefact of a known dimension is used as a reference measurement and a global scaling error correction can thus be performed. In step 4, the surface of the AM part is defined. This can be done by several different ways, including global thresholding, local thresholding, adaptive thresholding, etc depending on the type of data processing software employed. The type of thresholding method employed will have an impact on the dimensional measurements. Once the surface of the AM part has been defined, the defect detection can be performed. Depending on the type of software used, this can be done automatically or manually, but in all cases requires a difference in the grey value corresponding to the defect and that corresponding top the AM part material. When the defects are detected many parameters can be assessed, both on a global (overall volume), or local (individual defect) basis.

CMM

The protocol for dimensional measurement of AM parts using a tactile coordinate measuring machine (CMM) is presented in the workflow shown **Figure 4**, and is described in detail below.

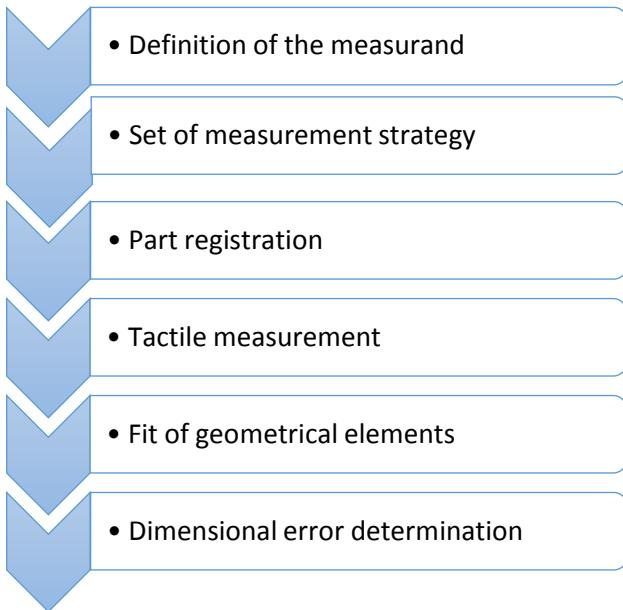


Figure 4: workflow for dimensional error assessment of AM parts by a tactile CMM.

As a first step, for tactile CMM, it is extremely important to define what exactly is to be measured and how (definition of the measurand). Based on the defined measurand, the measurement strategy must be set. This includes the selection of the probe (e. g. T- or star probe configuration, diameter and material of the probing sphere), the selection of the probing mode (single point probing or scanning), the probing force and the distribution of the points. This strategy depends on the measurement task, the needed measurement uncertainty and the characteristics of the measurement object. When AM parts are measured, in particular the roughness of the part must be taken into account. Also, the strategy to register the part must be defined to become able to set the coordinate system. For this, geometrical elements which allow to define a coordinate system must be identified on the part. One possibility to do this is the 3-2-1 registration measuring a plane, a line and a point.

These elements must be measured first and the coordinate system must be set. After that, the geometrical elements are probed following the measurement strategy.

In the next step geometrical elements are fitted in the measured probing points. Here the dimensions (e.g. cylinder diameter) but also the form deviations are of interest. The latter can become large, when rough AM parts are measured which causes a large measurement uncertainty.

The last step is the determination of the geometrical errors. The measured dimensions are compared with the corresponding dimensions of the drawing or CAD-representation or compared to the pre-defined tolerances of the workpiece.

Archimedes

The following protocol describes a process for the post production verification of features such as volume, density & porosity of an AM sample using the Archimedes' method. Presented in **Figure 5** is the workflow for the characterisation of AM samples using an Archimedes' method.

The classical principle of the Archimèdes' method involves measuring the apparent mass of the sample in air, then in twice-distilled water to deduce its density. However in the case of lattice structures, in water bubbles form at the interface air-lattice preventing the water to penetrate deep inside the lattice because the surface tension of water is too high ($72.8 \cdot 10^{-3} \text{ N.m}^{-1}$ at 20°C). In order to avoid this phenomenon, which distorts the measurements, absolute ethanol is used.

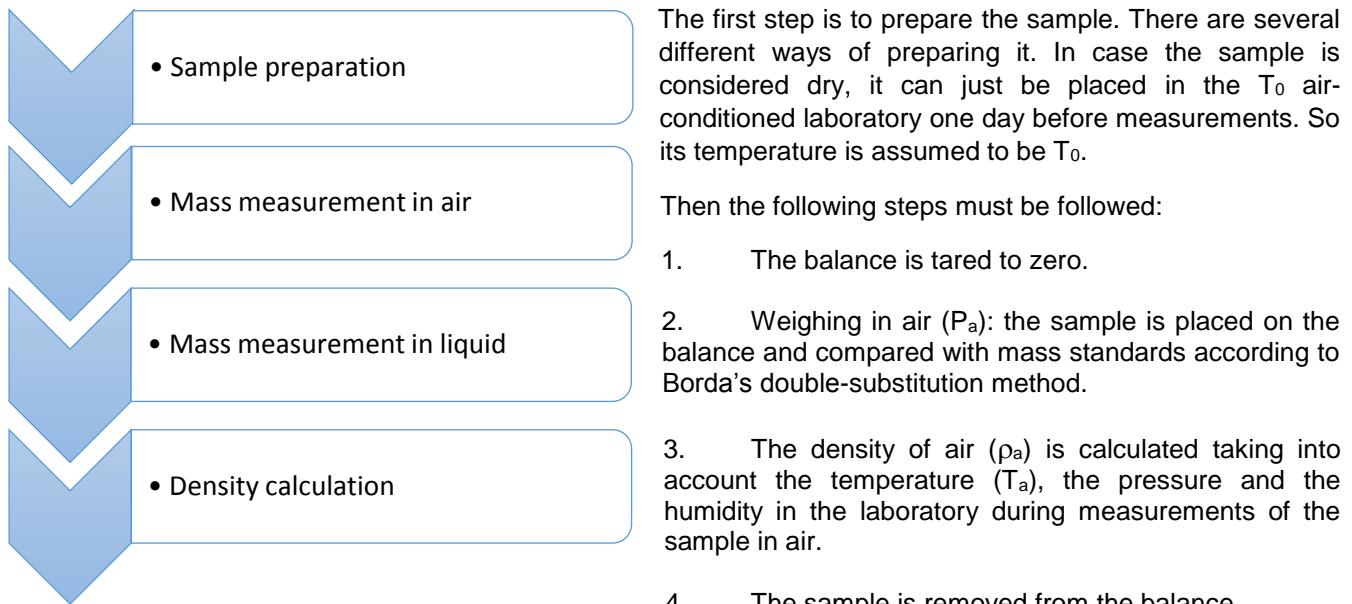


Figure 5: workflow for the characterization of AM samples using Archimedes' method.

6. The balance is tared to zero
7. Weighing in liquid (twice-distilled water or absolute ethanol) (P_l): the sample is placed in the liquid on a suspension device. Once the sample and the temperature are stabilised, the mass of the sample is compared with mass standards placed on the weighing pan according to Borda's double-substitution method. This comparison in liquid has to be carried out twice. In between each measurement an eventual derivation of the tare has to be checked and then the balance is tared to zero if no derivation is observed.
8. The temperature of the liquid inside the container is taken (T_l) in order to take into account the variation of temperatures on the liquid.
9. The density of air (ρ_a) is calculated taking into account the temperature (T_a), the pressure and the humidity in the laboratory during measurements of the sample in the liquid.
10. Weighing in air (P_a): the sample is placed for the second time on the balance and compared with mass standards according to Borda's double-substitution method.
11. The final result (ρ) is expressed as the mean of the two measurements carried out using the following relation:

$$\rho = \frac{A_a}{A_a - A_l} \left\{ \rho_{l,T_0} [1 + C_l(T_l - T_0)] [1 + C_s(T_l - T_0)] - \rho_a [1 + C_s(T_a - T_0)] \right\} + \rho_a [1 + C_s(T_a - T_0)] \quad (1)$$

where:

$$A_a = P_a \left(1 - \frac{\rho_a}{\rho_e} \right) \text{ and } A_l = P_l \left(1 - \frac{\rho_{a,l}}{\rho_e} \right) \quad (2)$$

with C_l and C_s the thermal expansion coefficient of the liquid and of the sample respectively and $\rho_e = 8000 \text{ kg.m}^{-3}$, the conventional value of the density of the standards.

This equation takes into account the air buoyancy correction.

Pycnometer

The following protocol describes a process for the post production verification of features such as volume, density & porosity of an AM sample using the gas pycnometry method. Presented in **Figure 6** is the workflow for the characterisation of AM samples using gas pycnometry.

Gas pycnometry is a measurement method based on gas displacement and the relationship between volume and pressure.

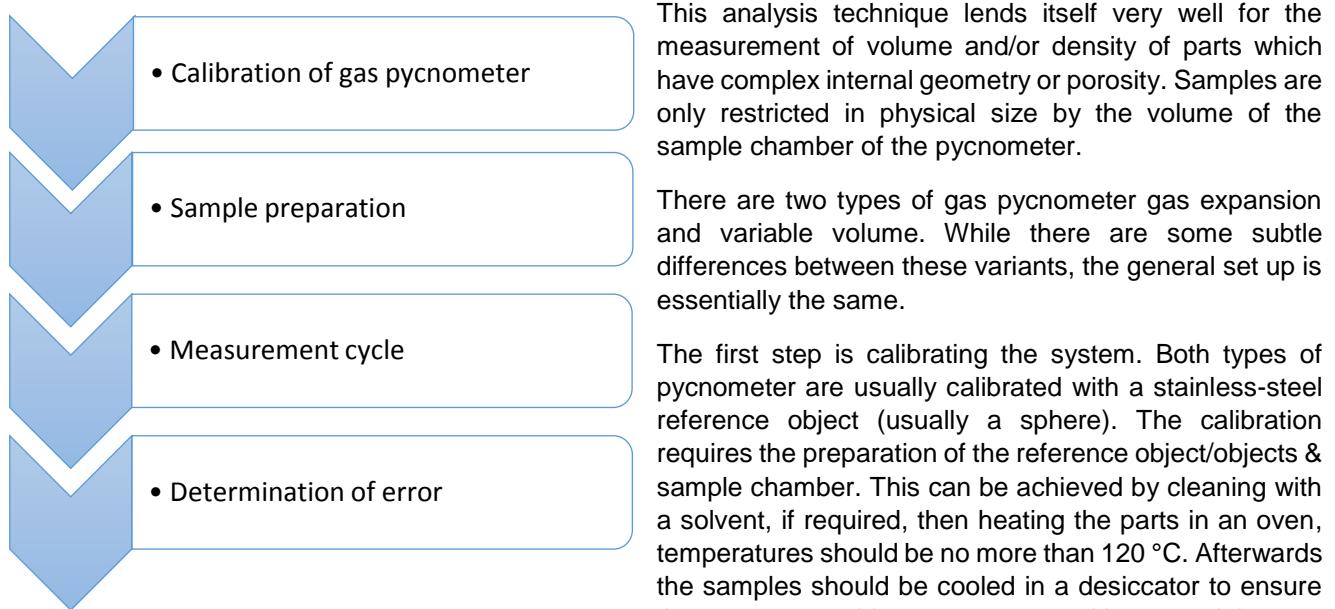


Figure 6: workflow for the characterization of AM samples using Gas Pycnometry.

using powder free polymer gloves and forceps. Carry out the calibration of the system in accordance with the manufacturers protocol. This will generally involve approx. 10 purge cycles with the reference object in the chamber.

The second step is to prepare the AM sample/samples for measurement. In general, metallic parts can be heated to approximately 120 °C, other materials should be heated to a temperature as hot as the sample material can handle without compromising the features/structure. Once heating is complete allow the AM samples to cool in a desiccator. Before transferring the AM sample to the pycnometer record the mass.

The third step is to transfer the AM sample to the chamber for measurement. Before beginning a measurement series always calibrate the pycnometer. When measuring a sample use between 10 and 20 purge cycles with the AM sample in the chamber. With more modern pycnometers this may be automatically determined by the system. The pycnometer will normally measure and display the volume & density of the sample. Porosity can be calculated by measuring the envelope volume of the sample by a suitable method e.g. Vernier caliper, CMM, Optical Comparator. The porosity is the percentage of free space in the envelope volume, this can be calculated by subtracting the sample volume from the envelope volume.

In the fourth and final step the result can be compared with a reference value for the sample, in this case the reference value is usually the relevant nominal measurement from the CAD file used as the input for the 3D printer.

Weighing

A simple measurement of the final parts mass by weighing is one approach to identify defects in manufactured parts. It would be a fast and inexpensive method, easy to integrate in the manufacturing process chain for the evaluation of the quality of a printed part.

The expected mass (m) of the part can be calculated by the product of final density of the solid material (ρ) and the nominal part volume (V) retrieved from the corresponding STL files, when taking into account typical shrinkage for the material used:

$$m = \rho * V$$

The results from weighing of the printed part can be compared to the calculated mass or to the mass of a reference part. Investigations within the project showed, that weighing allows to identify defects leading to a change in volume and mass of around 20 % compared to a reference part. Also, the detection of minor defects is possible if the process is very stable (see also *D6, Good practice guide for the medical device industry to be able to identify geometrical deviations to enable process chain corrections to be implemented*).

Appendix 1 Measurement protocol template

European Metrology Programme for Innovation and Research
Project: 15HLT09 MetAMMI - Metrology for additively manufactured medical implants

Measurement Protocol

T2.1 Volumetric Characterisation

1 GENERAL DATA

Measuring Organisation:	
Measurement System:	
Limits of measurement system:	
Name of Operator:	
Date of Measurement:	
Measurement No.:	
Measurement related to Activity Number of JRP Protocol:	
Measured Part:	Name: Part ID:
Manufactured by: in Activity:	
Additive Manufacturing Technology:	

2 SYSTEM SETTINGS

Voltage (kV):	
Current (μ A):	
Pre-filter (Material and thickness in mm):	
Integration time (ms):	
# of projections:	
Voxel size (μ m)	

3 NOMINAL/ACTUAL COMPARISON

ALIGNMENT PROCEDURE:	
SCALING FACTOR <i>When the overall dimension of AM part is different, a scaling of CT data will be necessary. Factor should be entered here. Can be different in x, y and z direction.</i>	in x direction:
	in y direction:
	in z direction:
DEFINITION OF X, Y AND Z DIRECTION <i>Describe or upload a picture.</i>	
DEVIATION DISTRIBUTION	90% < 50% <
DEVIATION HISTOGRAM	
POROSITY	Total material volume: Total defect volume: Ratio: Minimum pore diameter:

4 OBSERVATIONS & COMMENTS

Please describe in the following table your observations and detected deviations.

Further observations regarding:	Comments	Pictures
Missing material:		
Extra material:		
Distortions:		
Cracks:		

5 FURTHER COMMENTS

6 DATA SHARED