

## D3.1. Selected powders supplied for WP3

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## REVISION HISTORY

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1.0	20-11-2023	Delphine QUARANTA / Christophe VOISIN / MTEC	First draft sent to the partners
1.1	11-12-2023	Marijke Jacobs / VITO	Approved by all WP3 partners

## EXECUTIVE SUMMARY

Deliverable 3.1, entitled “Selected powders supplied for WP3”, concerns the two powders: GDC10 and CSCO produced by MTEC following the request of the WP3 partners. The synthesis protocols and the characteristics of the powders delivered are detailed there.

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## List of Abbreviations

Abbreviation	Definition
CSCO	$\text{Ca}_{2.7}\text{Sr}_{0.3}\text{Co}_4\text{O}_9$
CNRS	Centre National de la Recherche Scientifique
GDC10	$\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$
MTEC	Marion Technologies
WP	Work Package

## 1 Introduction

The first tests carried out by the WP3 partners have demonstrated that two powder compositions were promising for the development of a composite with optimized thermoelectric properties:

- $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$  (GDC10) with a particle size between 0.4 and 0.9  $\mu\text{m}$
- $\text{Ca}_{2.7}\text{Sr}_{0.3}\text{Co}_4\text{O}_9$  (CSCO) with a particle size between 0.4 and 0.9  $\mu\text{m}$ .

These powders have been synthesized by MTEC in large quantities: 10 kg for GDC10 and 3 kg for CSCO to only use one batch of each composition during the entire time of the project. The synthesis protocols and characterizations of these powders, provided to the various partners (VITO, FIAXELL and CNRS), are described in this deliverable.

## 2 Synthesis protocols

### 2.1 GDC10

A single 10 kg batch of GDC10 has been produced by MTEC by a combustion method. The production method used is shown in Figure 1.

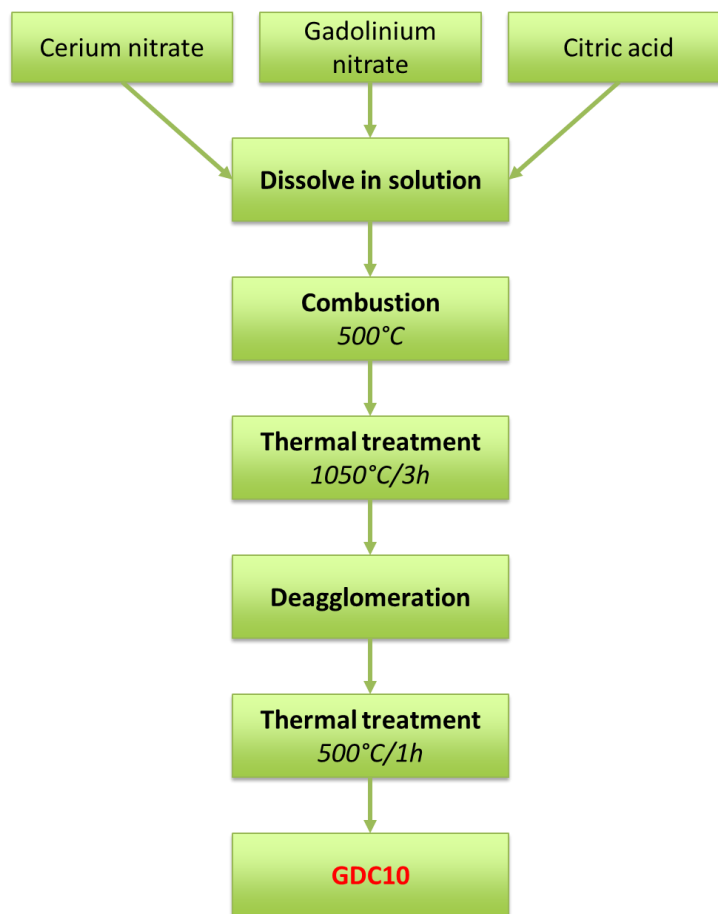


Figure 1 : Synthesis protocol of GDC10

At first, cerium and gadolinium nitrates has been dissolved in water in the right proportions. After a control of the stoichiometry, citric acid has been added to the solution. The solution is then heat treated at 500 °C in a continuous-type furnace in air. The obtained powder has been calcined at 1050 °C for 3 hours in a muffle furnace. After that, the product has been deagglomerated in ethanol with zirconia beads and dried at 80 °C in air overnight. The powder has been finally calcined at 500 °C for one hour to obtain  $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ .

## 2.2 $\text{Ca}_{2.7}\text{Sr}_{0.3}\text{Co}_4\text{O}_9$

A single 3 kg batch of CSCO has been produced by MTEC by a combustion method. The synthesis protocol is shown in Figure 2.

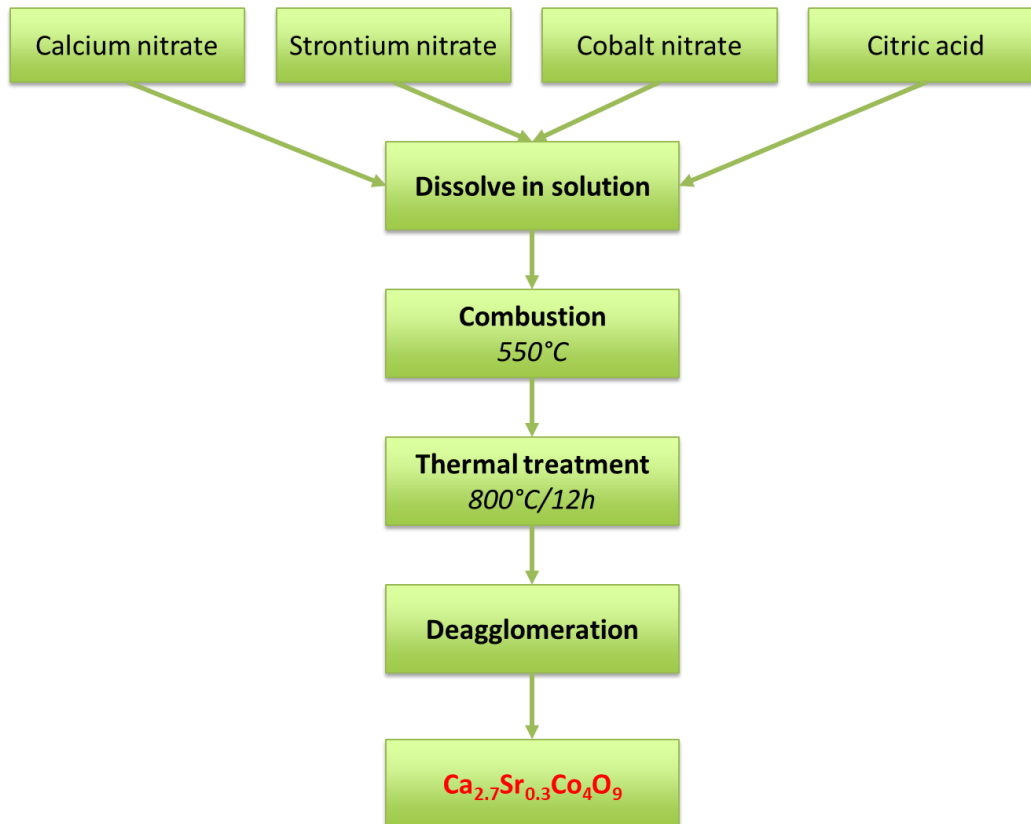


Figure 2 : Synthesis protocol of CSCO

At first, the metal nitrates have been dissolved in water. After controlling the stoichiometry, citric acid has been added to the solution. The solution is then calcined at 550 °C in a continuous-type furnace. The resulting powder has been treated at 800 °C during 12 hours in air. The powder has been finally deagglomerated in isopropanol with small zirconia beads and dried at 90°C in air overnight to obtain  $\text{Ca}_{2.7}\text{Sr}_{0.3}\text{Co}_4\text{O}_9$ .

### 3 Characterizations

The synthesized powders have been characterized by spectroscopy analysis: ICP-AES, laser particle size analysis, X-ray diffraction and BET adsorption method.

#### 3.1 GDC10

The GDC10 powder XRD pattern is shown in Figure 3.

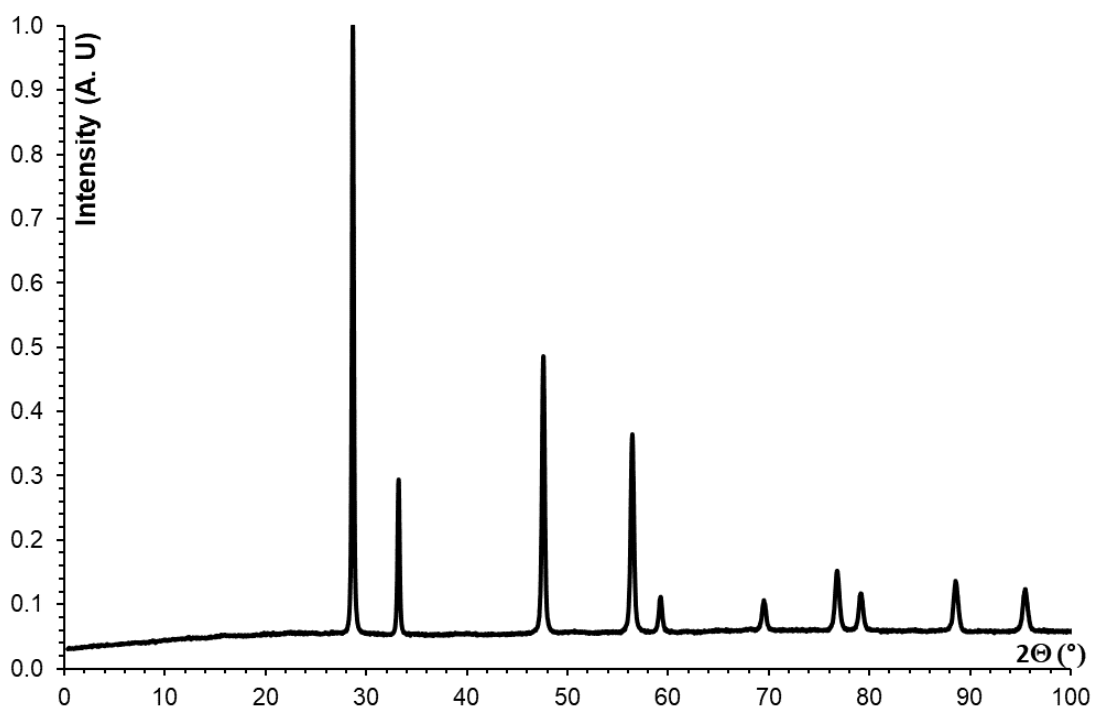


Figure 3: XRD pattern of GDC10

Only the  $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$  (JCPDS 750161) phase is identified.

The particle size analysis has been performed on a Malvern Mastersizer granulometer with the following conditions: 3000 rpm for the pump, US intensity at 100% and a solution of sodium metaphosphate at 2% for the dispersant. The particle size has been measured at 0.7  $\mu\text{m}$ .

The ICP-AES analysis has been realized on a Shimadzu device (ICPE 9820) and the results are shown on Table 1. The desired composition is obtained.



Table 1: ICP-AES analysis of GDC10

Element	Specification	ICP-AES analysis
Ce	0.90	0.90 ± 0.03
Gd	0.10	0.10 ± 0.01

A specific surface area of 9.2 m<sup>2</sup>/g has been measured by BET method on a Micromeritics Gemini device.

### 3.2 Ca<sub>2.7</sub>Sr<sub>0.3</sub>Co<sub>4</sub>O<sub>9</sub>

The XRD pattern of CSCO is shown in Figure 4.

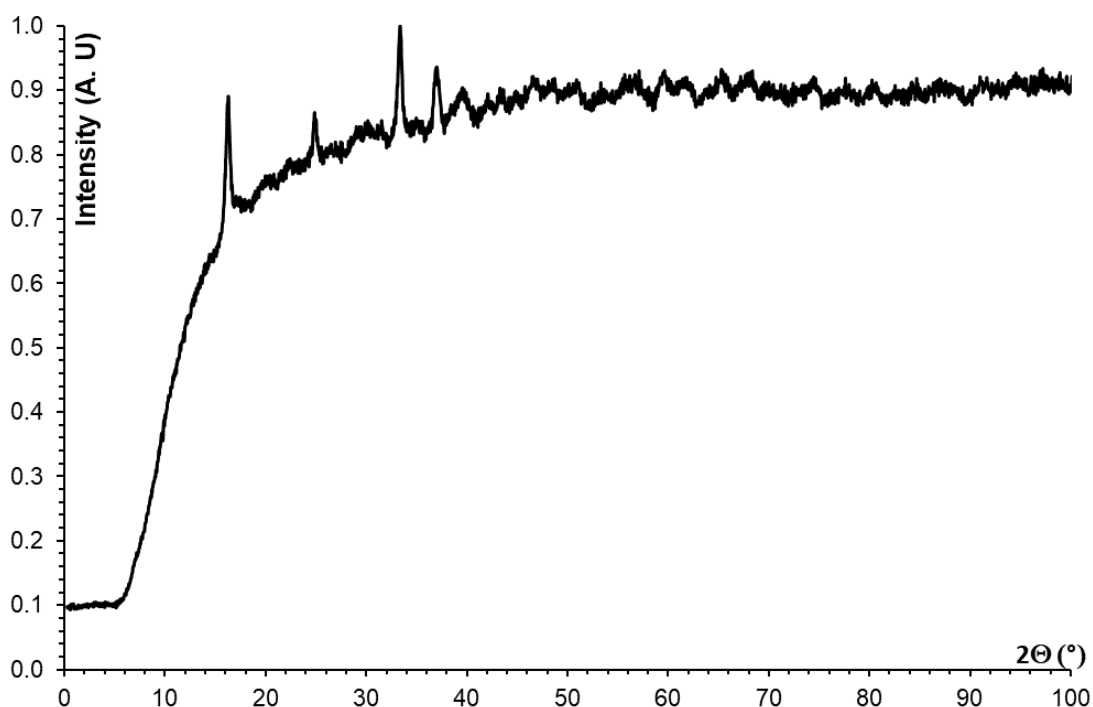


Figure 4: XRD pattern of CSCO

Only the Ca<sub>9</sub>Co<sub>12</sub>O<sub>28</sub> (JCPDS 210139) is identified.

The particle size analysis has been performed on a Malvern Mastersizer granulometer with the following conditions: 3000 rpm for the pump, US intensity at 100% and a solution of sodium metaphosphate at 2% for the dispersant. A particle size of 0.8 μm has been measured.

The ICP-AES analysis has been realized on a Shimadzu device (ICPE 9820) (Table 2).

Table 2: ICP-AES analysis of CSCO

Element	Specification	ICP-AES analysis
Ca	2.70	2.69 ± 0.03
Sr	0.30	0.32 ± 0.01
Co	4.00	3.99 ± 0.03

A specific surface area of about 38 m<sup>2</sup>/g has been measured by BET method on a Micromeritics Gemini device.

## 4 Powders delivered

The powders delivered by MTEC to the different partners within this WP are listed in the Table 3 below:

Table 3: Powders delivered by MTEC

Powder	Reference	Amount	Delivered on ... to ...
Ce <sub>0.9</sub> Gd <sub>0.1</sub> O <sub>1.95</sub>	158.064.019 Batch 004	1 kg	09-05-2023 to VITO
Ce <sub>0.9</sub> Gd <sub>0.1</sub> O <sub>1.95</sub>	158.064.019 Batch 004	1 kg	09-05-2023 to FIAXELL
Ce <sub>0.9</sub> Gd <sub>0.1</sub> O <sub>1.95</sub>	158.064.019 Batch 004	1 kg	09-05-2023 to CNRS
Ca <sub>2.7</sub> Sr <sub>0.3</sub> Co <sub>4</sub> O <sub>9</sub>	127.120.138 Batch 001	1 kg	21-08-2023 to VITO
Ca <sub>2.7</sub> Sr <sub>0.3</sub> Co <sub>4</sub> O <sub>9</sub>	127.120.138 Batch 001	1 kg	21-08-2023 to FIAXELL
Ca <sub>2.7</sub> Sr <sub>0.3</sub> Co <sub>4</sub> O <sub>9</sub>	127.120.138 Batch 001	1 kg	18-07-2023 to CNRS

## 5 Conclusion

Marion Technologies has produced two powders in large quantities: GDC10 and CSCO, respecting the specifications given by the WP3 partners. 1kg of each powder has been delivered to VITO, FIAXELL and CNRS.

## 6 Index for all figures

NUMBER	DESCRIPTION
1	Synthesis protocol of GDC10
2	Synthesis protocol of CSCO
3	XRD pattern of GDC10
4	XRD pattern of CSCO

## 7 Index for all tables

NUMBER	DESCRIPTION
1	ICP-AES analysis of GDC10
2	ICP-AES analysis of CSCO
3	Powders delivered by MTEC