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## Data Article

# Experimental data for the synthesis of a new dimeric prodelphinidin gallate



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## ABSTRACT

This data article contains raw and processed data related to research published in Teixeira et al. (2016) [1]. Here we introduce data acquired from the synthesis of a prodelphinidin dimer gallate. All synthesis steps are described and a dataset for the removal of the protecting on prodelphinidin synthesis is presented. With hydrogenolysis *in situ* with triethylsilane the hydrogen required is produced and used at the same time, making the reaction possible without resorting to bottled hydrogen. Full NMR and HPLC-ESI-MS analysis data is also provided.

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## Specifications Table

Subject area	Chemistry
More specific subject area	Food Chemistry
Type of data	Table, figures
How data was acquired	NMR (Bruker Avance 400 spectrometer), mass spectroscopy (Finnigan Surveyor Plus HPLC system fitted with a PDA Plus detector, an autosampler Plus and a LC quaternary pump plus coupled to a Finnigan LCQ Deca XP Plus mass

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	detector equipped with a ESI source and an ion trap quadrupole equipped with an atmospheric pressure ionization (API) source).
Data format	Analyzed
Experimental factors	Synthesis procedure of a prodelphinidin dimer gallate starting from the two constitutive monomers.
Experimental features	Starting constitutive monomer protection with benzyl groupsDDQ oxidation at C4 of the upper monomer with benzyl alcohol as the nucleophile and DMAP as nucleophilic catalyst.Condensation of the two constitutive monomersHydrogenolysis <i>in situ</i> with triethylsilaneNMR and HPLC-ESI-MS data analysis
Data source location	Oporto, Portugal
Data accessibility	Data are available with the article

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## Value of the data

- The data for a simple method for prodelphinidin gallate synthesis with hydrogenolysis *in situ* is presented;
  - Full NMR analysis data is included for easy identification of the intermediate and final compounds;
  - This synthesis strategy may be applied to other proanthocyanidins.
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## 1. Data

The data here presented describes the synthesis procedure and analysis of epigallocatechin-(4 $\beta$ →8)-epigallocatechin gallate (EGC-EGCG), a prodelphinidin dimer (Fig. 1) [1]. The synthesis procedure starts from the two constitutive monomers: (-)-Epigallocatechin (EGC) and (-)-epigallocatechin gallate (EGCG), and includes a new approach to the removal of the protecting groups.

## 2. Experimental design, materials and methods

### 2.1. Materials

(-)-Epigallocatechin (EGC) and (-)-epigallocatechin gallate (EGCG) were purchased from Biopurify Phytochemicals Ltd. (Sichuan, China).

### 2.2. Methods

#### 2.2.1. Benzylation of monomeric flavan-3-ols

To a stirred solution of (-)-EGC **1** and (-)-EGCG **2** in dry DMF, under argon, was added potassium carbonate (K<sub>2</sub>CO<sub>3</sub>) (10 eq for **1**; 17.6 eq for **2**) and BnBr (7.7 eq for **1**; 13.6 eq for **2**). The solution was stirred at 0 °C for 2 h and left at room temperature for 72 h for **1** and 24 h for **2**. The mixture was extracted with ethyl acetate and water, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated. The crude product was purified with silica gel column chromatography (dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>) for **3** and hexane/ EtOAc 2:1 for **4**).

#### 2.2.2. Benzylation at C4

To a solution of EGC5Bn **3** and benzyl alcohol (BnOH) (10.3 eq) in CH<sub>2</sub>Cl<sub>2</sub> was slowly added, at 0 °C, 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) (2.3 eq). After reacting overnight at room

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