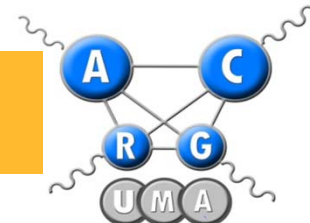




UNIVERSIDAD DE MÁLAGA



# ***The role of raw powder diffraction data in peer review past, present and future***

**Miguel A. G. Aranda**

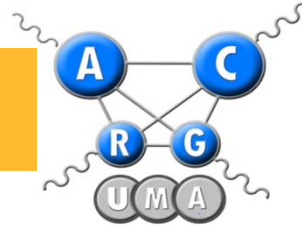
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**Inorganic Chemistry Department, University of Malaga, Spain**



# Outline



## 1. Motivation

## 2. Introduction

*2.1. Type of scientific data in powder diffraction*

*2.2. Type of information to be extracted*

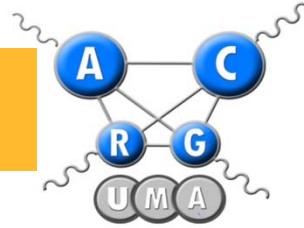
## 3. FAIR & FACT

## 4. Reviewing process

## 5. Conclusions



# 1. Motivation



- We are in this WS, so little motivation discussion is needed for world-wide Scientific (raw) Data Sharing

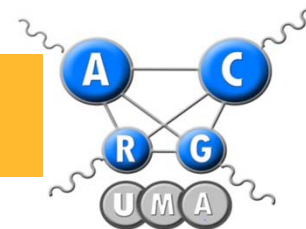
1. Helping experiment replication.
2. Likely better data analysis in the future (improvements)
3. New findings (and science) using ML and AI over many scientific datasets – our Big Data

## **Provocative:**

**Caveat related to #3**, and coming from the Large Facility environment, if Europe strongly promotes Scientific (raw) Data Sharing and other big countries do not:

**European congress - *Can finally Europe ends in a weaker position?***

For instance, we have been researching in Rietveld Quantitative Analysis of cements for more than a decade with different softwares (GSAS, Topas, HighScore+,....)  
We are sharing raw data since 2017, ML could/will take over and this subfield, as known today, could be 'dead' in a few years! *The know-how will be transferred through these training data sets!*



# 2. Introduction - Type of scientific raw data in PD Type of information to be extracted

**Primary raw data**  
*To be archived by the Facilities*

*(Ready-to-be-analyzed)*

**Processed raw data**

*To be shared by authors, along paper submission!*

**(Reduced data)**

**Derived data**

n point detectors

1D detector

2D detector

- Applying detector calibration
- Masking pixels (defective, etc.)
- Corrections (geometry, etc.)
- Radial integration
- Merging of data sets

hkl	$l/l_0$	$d_{obs}$ (Å)	$2\theta_{obs}$ (°)
012	26	6.282	17.480
104	18	4.4681	24.670
110	5	4.4152	24.970
113	100	3.7841	29.220
006	10	3.6712	30.140
202	1	3.6127	30.640
024	24	3.1412	35.380

◆ Atomic parameters

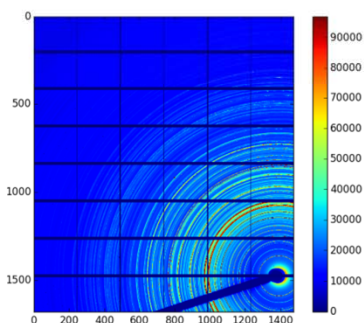
◆ Microstrain values

◆ Coherent diff. domain size

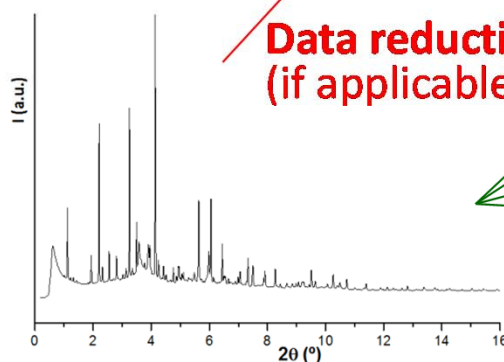
◆ QPA

◆ Total amorphous content

◆ Many other data (bulk modulus for high pressure, etc.)



**Data processing**  
(detector & geometry dependent)



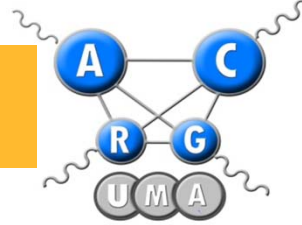
**Data reduction**  
(if applicable)

**Data analysis**  
(several types of software & programs)

Sharing powder diffraction raw data: challenges and benefits *J. Appl. Cryst.* (2018). **51**, 1739–1744



### 3. FAIR & FACT



**FAIR** : research data being findable, accessible, interoperable and reusable

Repositories and Large Facilities are addressing this.

Computer engineers are taking care.

To my understanding, not big challenges, just enough funding to accomplish the objectives.

**BUT**

*Flooding the repositories with poor (raw) data could harm/delay research advancement*

**FACT** : the shared data must have sufficient quality. They must be true facts.

*This spills over the narrative of the scientific publications!*

**HOWEVER**

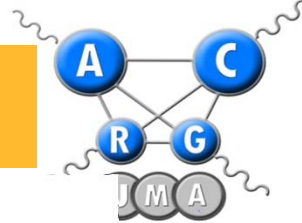
*How to address/ensure this, in the publishing step(s) ?*





## 4. Reviewing process – case example

Cement and Concrete Research 115 (2019) 361–366



Contents lists available at [ScienceDirect](#)

Cement and Concrete Research

journal homepage: [www.elsevier.com/locate/cemconres](http://www.elsevier.com/locate/cemconres)



Rietveld quantitative phase analyses of SRM 2686a: A standard Portland clinker

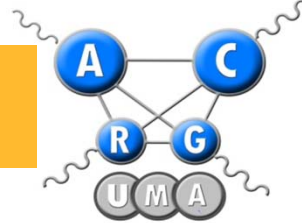


M. García-Maté<sup>a</sup>, G. Álvarez-Pinazo<sup>a</sup>, L. León-Reina<sup>b</sup>, A.G. De la Torre<sup>c</sup>, M.A.G. Aranda<sup>c,d,\*</sup>

SRM 2686a is a NIST reference Portland clinker with reported mineralogical analysis from powder diffraction and electron microscopy. This sample is used in ASTM C1365 test method for Rietveld quantitative phase analysis validation procedure. Here, we have analysed SRM 2686a by using three state-of-the-art powder diffraction configurations: i) strictly monochromatic  $\text{CuK}\alpha_1$  radiation in flat reflection geometry; ii) strictly monochromatic  $\text{MoK}\alpha_1$  radiation in flat transmission geometry; and iii) synchrotron radiation in rotating capillary transmission geometry. The silicate and aluminate enriched residues have also been studied by  $\text{CuK}\alpha_1$  powder diffraction. All the powder patterns were analysed by Rietveld method with the best available protocols. The results indicate that belite in SRM 2686a is composed of two polymorphs ( $\beta$ - and  $\alpha'_H$ -) that must be included in the analyses. The use of a unique phase for describing belite ( $\beta$ -polymorph) and improper peak shape modelling could explain the problems found for implementing ASTM C1365 in some cement manufacturing plants. Furthermore, all the patterns are deposited as open data access at Zenodo, so interested laboratories can analyse these data to verify their protocols.



## 4. Reviewing process – case example



**Table 1**

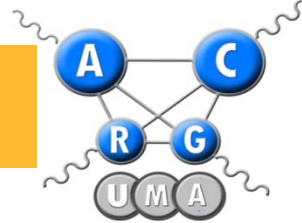
NIST mineralogical analysis reported for SRM 2686a from powder diffraction and electron microscopy [21]. The maximum variation allowed by ASTM C1365 test method [22] is also included.

	NIST SRM 2686a (wt%)	ASTM C1365 maximum variation allowed (%)	Range allowed by ASTM C 1365 test method
Alite	63.35 ± 1.29	5.9	57.45–69.25
Belite	18.68 ± 1.42	3.7	14.98–22.38
Aluminate	2.46 ± 0.67	2.1	0.36–4.56
Ferrite	10.76 ± 1.44	2.5	8.26–13.26
Periclase	3.4 ± 0.40	0.8	2.60–4.20
Alkali sulfates <sup>a</sup>	0.87 ± 0.27	0.9	0.00–1.80

The Compositional Analysis subcommittee of American Society for Testing and Materials (ASTM) C01.23 developed a test method, ASTM C1365, entitled ‘**Determination of the Proportion of Phases in Portland Cement and Portland-Cement Clinker Using X-Ray Powder Diffraction Analysis**’. This method considers the use of XRPD data analyzed by the Rietveld method and it is being used for cement industries to self-verify their RQPA procedures. **However, we were aware that some cement plants/labs. were having problems to validate their RQPA methodologies by using the ASTM C1365 test method.**



## 4. Reviewing process – case example



In our submission, “... All the patterns analyzed here can be accessed on Zenodo at <https://doi.org/10.5281/zenodo.1318500>, and used under the Creative Commons Attribution license... ” (doing this since 2017)

July 21, 2018 Journal article Open Access

### Rietveld Quantitative Phase Analyses of SRM 2686a: a Standard Portland Clinker

García-Maté, M; Álvarez-Pinazo, G; León-Reina, L; De la Torre, AG; Aranda, MAG

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Files (687.2 kB)

Name	Size
aluminate enriched residue clinkerNIST 170718 R1.xrdml	24.2 kB <a href="#">Download</a>

88 views 136 downloads [See more details...](#)

Indexed in **OpenAIRE**

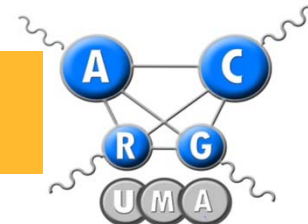
Publication date: July 21, 2018

DOI: [10.5281/zenodo.1318501](https://doi.org/10.5281/zenodo.1318501)

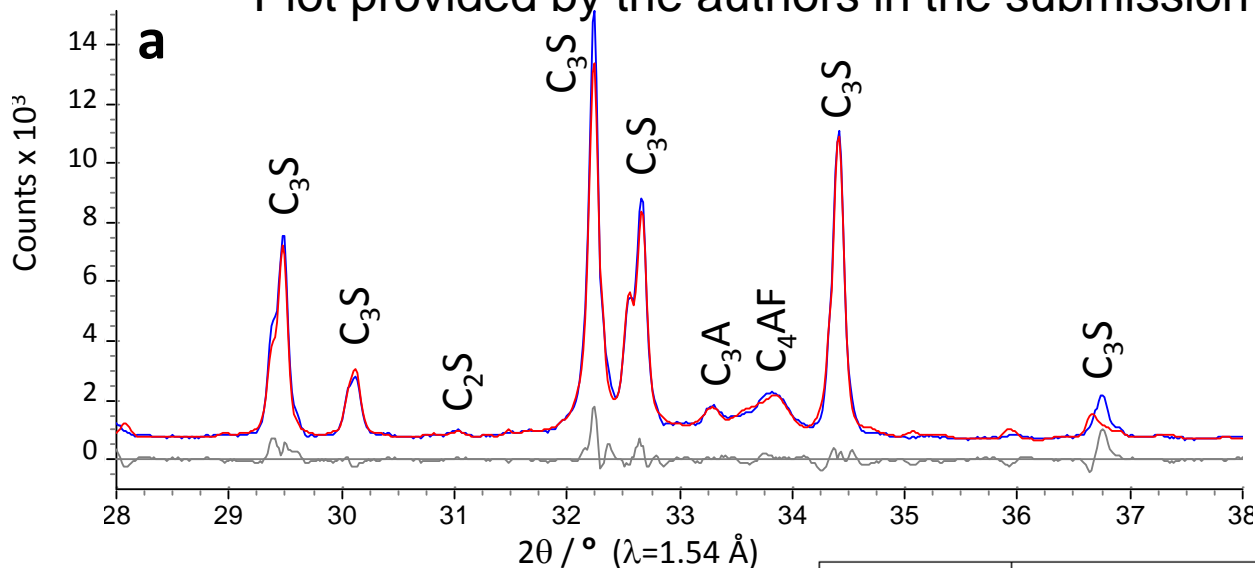
Keyword(s): Mineralogical content, synchrotron radiation, powder



## 4. Reviewing process – case example



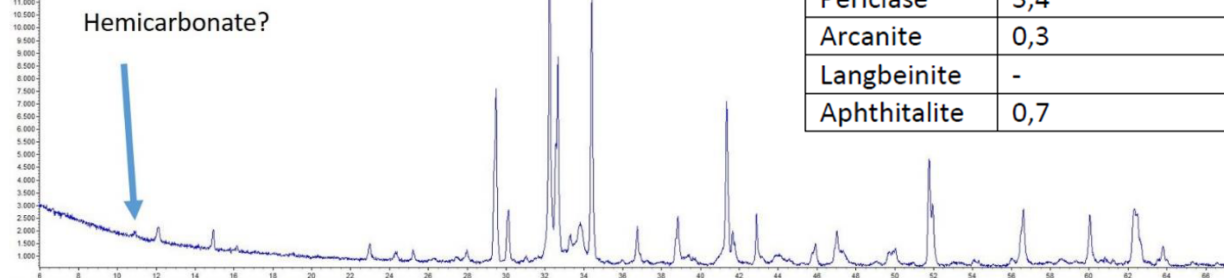
Plot provided by the authors in the submission



The reviewer re-analyzed the deposited data (Cu-Ka1, Mo-Ka1, synchrotron)

This is a plot of your data provided in the internet.

Plot produced by the referee based on the deposited raw data

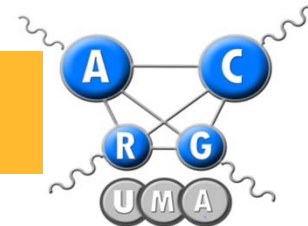


	1st person (reviewer)	2nd person (reviewer)	Your CuKα	Your Average
Alite	67,5	67,6	70,2	67,3
Belite	13,6	12,1	11,4	13,5
C3A	3,2	2,4	4,2	3,6
C4AF	11,8	12,9	9,4	10,7
Periclase	3,4	3,1	3,8	3,6
Arcanite	0,3	0,4	0,3	0,3
Langbeinite	-	0,5	0,3	0,4
Aphthitalite	0,7	0,5	0,5	0,5

Very insightful comments



## 4. Reviewing process – ideas



**We are moving from reviewing the scientific publications**  
–including reduced and derived data–

Towards reviewing raw data

For PD, I advocate for [ready-to-analyze data](#).

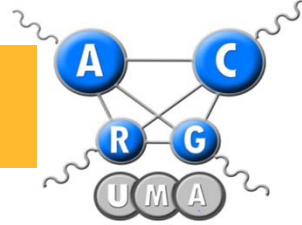
The files used by common software, which depends upon the field and the results to be obtained, Rietveld/PDF

### **Pros. and cons.**

Workload will be likely larger for the referees



## 4. Reviewing process – ideas



### 1. To encourage the motivation of reviewers. **Persons vs. Groups?**

*In addition to recognition with new metrics (publons, etc.).*

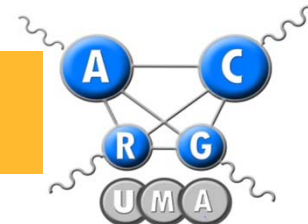
1.1. Reduction in processing fees for referees (re)analyzing data when needed?

1.2. Accompanying the papers with the reviewer assessment on the data with doi?

1.3. Promote the submission of reviews or lead articles by very active reviewers/groups providing thorough reports on deposited (raw) data?



## 4. Reviewing process – ideas



### 2. Helping with revision of the shared/deposited (raw) data

2.1. In addition to specific software developed by IUCr, checkcif etc., the IUCr referee database could contain the software expertise to facilitate the reviewing of the raw data [including (re)analysis?]

For instance, for RQPA: GSAS/Topas/etc...; for PDF: PDFgui...

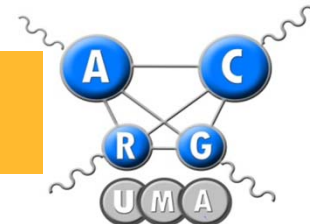
2.2. Should IUCrJ request the raw data *confidentially BUT compulsory* for the reviewing step?

2.3. *For PD reviewing, could be very beneficial to request also the control file.*

*Is it feasible? Larger transfer of know-how?*



## 4. Reviewing process – ideas



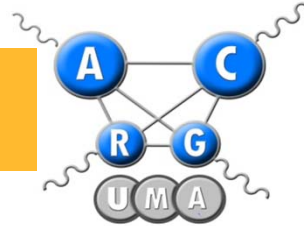
### 3. Pilot-plan for Powder Diffraction?

- 3.1. To choose one subfield as standard as possible to implement this strategy. In a first thought, to be more elaborated, it could be Quantitative Phase Analysis or Pair Distribution Function
- 3.2. To decide to compulsory request raw data associated to the paper for the reviewing process.  
To decide about requesting (confidentially) the control file(s)?
- 3.3. To open a call for reviewers willing to (also) review the raw data when adequate. (How do we deal with proprietary software?)
- 3.4. Results??





## 4. Reviewing process – ideas



### 4. Final thought not specific of PD

4.1. How can we measure the quality of the reviewing step ?

4.2. In a connected, collaborative scientific environment, could the reviewers be marked by authors and editors.

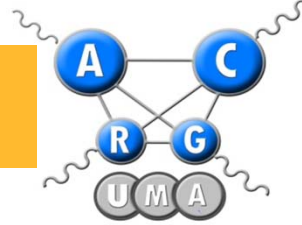
The individual marks can be anonymous, at the time of the evaluation, but the final output could be openly reported to encourage high quality reviews !

Could this be quantified, 1-star to 5-stars reviewers? Not based on the amount of reviews but on their quality.

If I publish about 5-8 papers a year, I should produce, at least, 15-20 reviews!



## 5. Conclusions

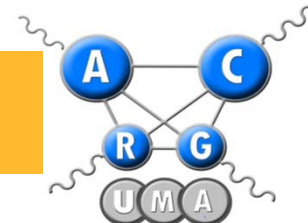


### My personal view

- I. Reviewing raw [deposited] powder diffraction data (when needed) is complex but not impossible
- II. Recognition/encouraging actions (for the reviewers) have to be adopted
- III. A subfield should be identified for a pilot-action.



## Acknowledgements



To all my collaborators

Thank you very much for your attention!



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