

Experimental report

15/09/2026

Proposal: 5-31-2929

Council: 10/2022

Title: Understanding Complex Synthesis-Structure-Property Relationships in the Frustrated S = 1 Spinel Antiferromagnet, ZnV₂O₄

Research area: Materials

This proposal is a new proposal

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Samples: ZnV₂O₄

Instrument	Requested days	Allocated days	From	To
D20	2	3	03/03/2023	05/03/2023
			23/05/2023	24/05/2023

Abstract:

Quantum magnets with frustrated magnetic interactions are predicted to give rise to a rich variety of exotic quantum materials properties. However, the complexity of such quantum materials often makes characterising the exact nature of their exotic properties extremely challenging. For instance, in frustrated quantum magnets, the degeneracy of possible magnetic ground states---that stems from the competition of magnetic interactions---means that the experimentally observed properties are extremely sensitive to any defects or disorder in the underlying materials structure, which can vary from sample to sample depending on the synthesis method used. This poses challenges for the development and application of condensed matter theory to understand the behaviour of real quantum materials, and ultimately limits our current ability to design and synthesise new quantum materials with properties tailored towards specific applications. Here, we aim to explore the complex synthesis-structure-property relationships in the frustrated S = 1 spinel antiferromagnet, ZnV₂O₄.

ZnV₂O₄ - D20 Experiment

September 15, 2026

Experiment Details

A summary of the run numbers and experimental details can be found in Tables 1 and 2. As the reactor was only running at 28 MW we were only able to collect data for the sintered sample.

	Sintered	Microwave
Can + screws (g)	30.49	44.97
Sample mass - start (g)	7.74	1.7
Sample mass - end (g)	7.67	-
Height (mm)	30	8
Diameter (mm)	12	10
Volume (g/cm ³)	3.4	0.5

Table 1: Sample mass and can.

Set	Run	Temperature (K)	λ (Å)
1	223766 - 223768	300	2.41
	223774 - 223776	300	1.3
	223777 - 223779	150	1.3
	223780 - 223785	1.5	1.3
First Dry	223786 - 223791	520	1.3
	223792 - 223795	300	1.3
	223796 - 223804	1.5	1.3
	223805 - 223813	25	1.3
	223814 - 223822	80	1.3
	223823 - 223831	150	1.3
	223832 - 223837	300	1.3
	223839 - 223853	545	1.3
Second Dry	223854 - 223865	520	1.3
	223866 - 223872	450	1.3

Table 2: Run numbers, temperatures, wavelength. Data are divided into 3 sets due to sample drying.

- We started the experiment measuring at 300 K with the wavelength, $\lambda = 2.41$ Å. We then realised that this was likely not going to give us a large enough integration window and so reduced the wavelength to $\lambda = 2.41$ Å.

- We then measured three temperatures, 300 K, 150 K and 1.5 K. During the measurements we noticed there was a problem with monitor 1 drifting, which was changing the background considerably as we were normalising the data. Switching to monitor 2 fixed this issue.
- Originally, we had hoped there would be no difference between 150 K and 300 K, meaning that we would've removed all the correlations from the system and be in the paramagnetic regime. But there was a large difference in the two temperatures.
- We then warmed to 520 K and measured for 2 hours. We saw a large background shift, which remained after cooling back to 300 K. The difference appeared to have a form factor dependence and we later decided this was water boiling off from the sample. After the experiment, we found our sample had a 0.07 g mass loss, which is probably due to this water evaporation.
- We flushed out the cryostat and with our new dry sample we decided to re-measure the temperatures we already had and add a few more between 1.5 K and 300 K. This data set is called the first dry in Table 2.
- The second 300 K run (#223832 - #223837) had half of the detectors at high angles drop out during the measurement. The data are still included in our analysis as we are only looking at the low Q region, which was unaffected. This problem was resolved by turning the detectors off and on again.
- We then warmed to 545 K, which we expected to show very little difference from the 520 K measurement. Unfortunately, this was not the case. The difference was fairly constant at low Q , but again, we decided that this must be more water within the sample that was being lost.
- We refer to the final set of measurements as the second dry in table 2 and includes the temperatures, 545 K, 520 K and 450 K. We used this as a way to check that we are in the paramagnetic regime, as we did not expect much difference between these temperatures. This is what we found, after a subtraction of 450 K – 520 K (second dry) and so we can assume that by 520 K we have reached the paramagnetic regime (or as close as we are able to get).

The analysis of the data is therefore grouped into the three sets in Table 2, with the temperature subtractions only being valid with temperatures measured in the same set.

Fullprof

A refinement of the cubic $Fd\bar{3}m$ model to the 520 K data (first dry) is shown in Fig. 1 and has good agreement with the data. Refinement as a function of temperature produced similar quality of fits, which matched the results from the high-resolution ID22/HRPD study. From this refinement, the fullprof scale was given to be 0.01503, which was used to convert the data to absolute units.

Conversion of data to absolute units

Data were converted to absolute units (b/ster./spin) using

$$s = \text{FPScale} \times \frac{2\pi^2 NV}{45\lambda^3} \quad (1)$$

where N is the number of magnetic atoms, V is the volume ($\text{\AA}^{-3}$) and λ is the wavelength ($\text{\AA}$). The values used for the conversion are summarised in Table 3.

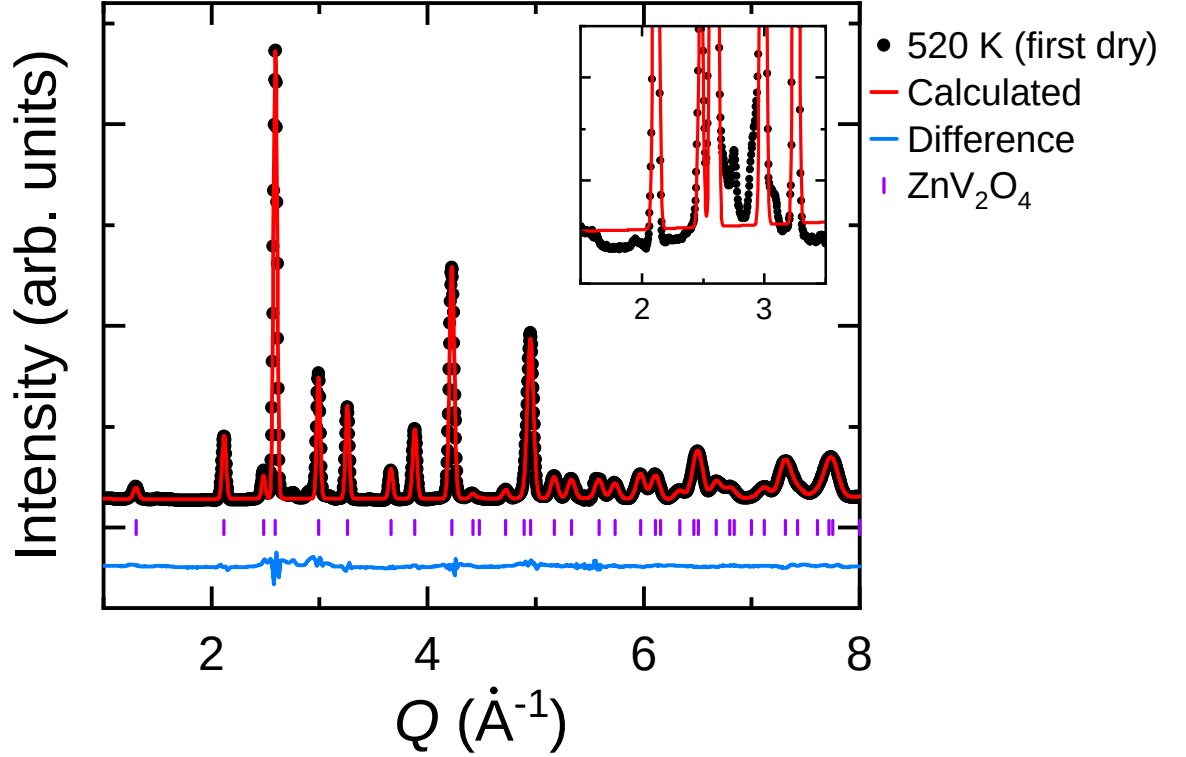


Figure 1: Rietveld fit of 520 K data (first dry) on Fullprof, $R_{wp} = 8.92\%$

Sample	FPScale	N	V ($\text{\AA}^{-3}$)	λ ($\text{\AA}$)	$\frac{2\pi^2 NV}{45\lambda^3}$	s
Sintered	0.01503	16	592	1.3	1891	28.42

Table 3: Values used for the conversion of data to absolute units

The temperature subtracted data in absolute units show a gradual evolution of the diffuse scattering. There is a sharp peak at $Q = 1.1 \text{ \AA}^{-1}$ which we have seen in the other experiments. The 450 K – 520 K (red) scattering is very nearly flat and so we conclude that 520 K is a good approximation to use for the paramagnetic state. We will fit the data with spinvert to determine the spin-spin correlation function.