

Supplementary Information

High-pressure Synthesis, Spin-Glass Behaviour, and Magnetocaloric Effect in $\text{Fe}_x\text{Ti}_2\text{S}_4$ Heideite Sulphides

R. S. Silva Jr.^{1,2}, J. Gainza¹, J. E. Rodrigues^{1,3}, L. Martínez¹, E. Céspedes¹, N. M. Nemes^{1,4}, J. L. Martínez¹, J. A. Alonso^{1*}

¹*Instituto de Ciencia de Materiales de Madrid (ICMM), CSIC, E-28049 Madrid, Spain.*

²*Department of Physics, Federal University of Sergipe, 49100-000, São Cristóvão-SE, Brazil.*

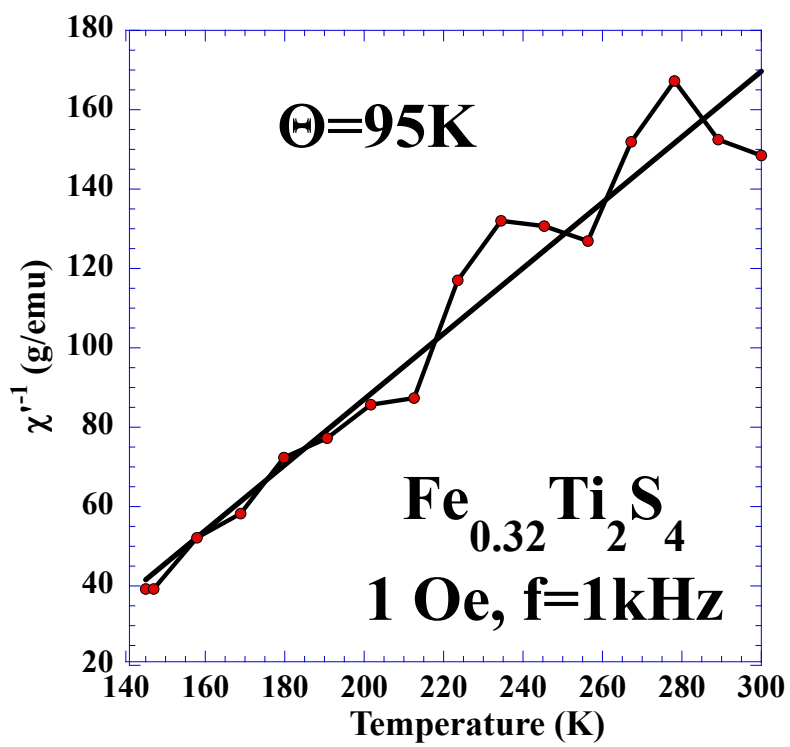
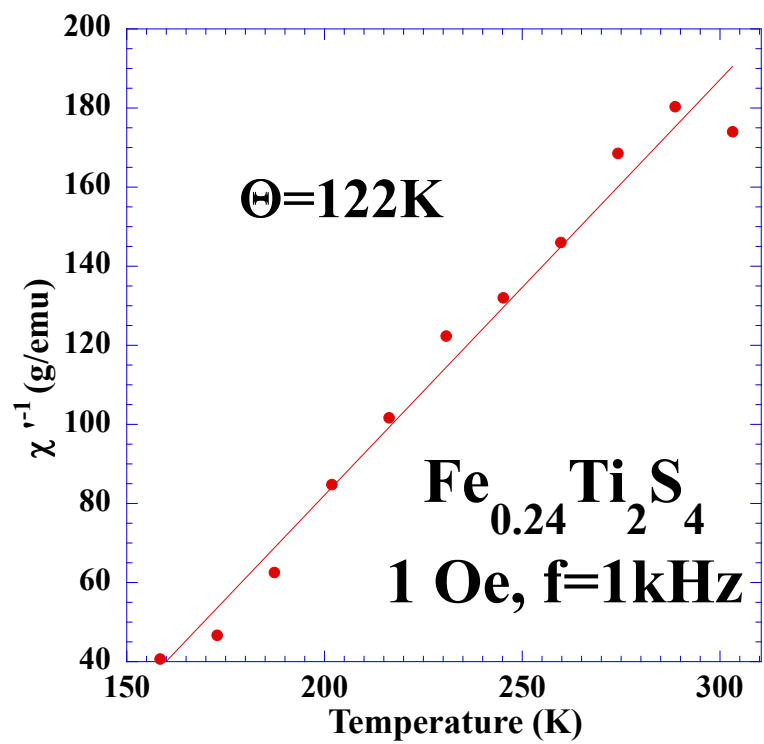
³*European Synchrotron Radiation Facility (ESRF), 71 Avenue des Martyrs, 38000 Grenoble, France.*

⁴*Departamento de Física de Materiales, Universidad Complutense de Madrid, E-28040 Madrid, Spain.*

* Corresponding author: ja.alonso@icmm.csic.es

Table S1. Elemental composition (atomic %) of the samples. Percentage of the different oxidation states found in the analysis of the Ti 2*p* core level peak.

Samples	<i>Composition (% at)</i>						<i>Ti 2p</i>		
	Fe	Ti	S	O	C	N	%Ti²⁺	%Ti³⁺	%Ti⁴⁺
Fe_{0.24}Ti₂S₄	2.6	6.3	5.9	41.0	44.2	0.0	33.5	62.4	4.1
Fe_{0.32}Ti₂S₄	2.8	5.1	4.3	37.9	49.3	0.6	45.5	49.1	5.4
Fe_{0.42}Ti₂S₄	1.1	2.0	3.1	23.8	69.3	0.7	19.4	75.1	5.5



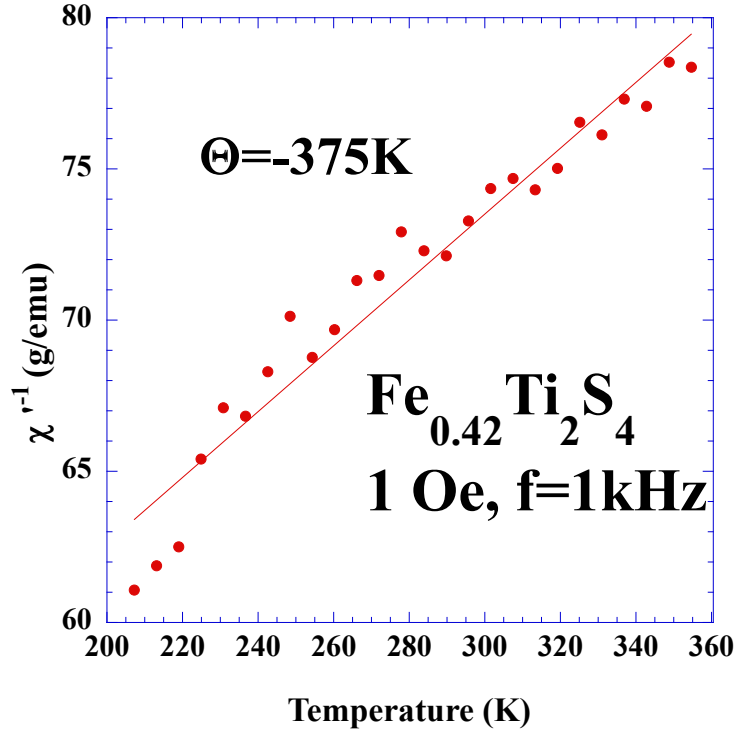


Figure S1: Curie-Weiss fitting of the inverse of the real part of the *ac* magnetic susceptibility measured at $f = 1$ kHz with an amplitude of 1 Oe in the temperature range from 150 K to 300 K, and 320 K to 355 K, for the $x = 0.24$ and 0.32 , and $x = 0.42$, respectively.