

## Tinkering with the wheel: can the goethite test run more smoothly?

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The goethite test is a beautifully engineered combination of thermal remanence application, AF demagnetization and heating-cooling cycles that allows, in principle, to separate the relative contributions of magnetite/maghemite, goethite and hematite in a specimen. It is particularly useful in environmental magnetism applications, specifically when trying to extrapolate variations in climatic signatures, such as temperature and precipitation from magnetic enhancement in samples that contain mixtures of these three phases. Carter-Stiglitz et al. (2006a) were the first to perform what became known as the goethite test, though it wasn't presented as a specific "test" until Guyodo et al. (2006) formalized it as such.

The test is mostly conducted within a Magnetic Properties Measurement System (MPMS) instrument and involves applying a pTRM to a specimen as it cools from 400 to 300 K through goethite's Néel temperature (~393 K), thus activating goethite's contribution. Typically, 2.5 T is used, so that any other magnetic mineral present that is "softer" than goethite (i.e. everything *s.l.*) will also be activated. The field is turned off at room T and the pTRM remanence is then measured upon cooling the specimen to low temperatures (~10 - 20 K, "LT") and warming back to 300 K, thus cycling through both the Morin ( $T_M$ , ~260 K) and Verwey ( $T_V$ , ~120 K) transitions (cycle 1). Of course,  $T_M$  will not be apparent if the magnetite/maghemite content makes up more than a few percent (1-2 %) of the total iron oxide content, nor if hematite is only present as nanocrystals or contains impurities (e.g., Al or Ti). Likewise, oxidation of magnetite will lower and broaden the  $T_V$  temperature and result in the characteristic "humpiness" of the cooling-heating curves (Özdemir and Dunlop, 2010). Increasing titanium substitutions will also suppress  $T_V$ .

At room temperature, the specimen is removed from the MPMS and AF demagnetized (typically using 200 mT fields) to remove the contribution of magnetite/maghemite ("Mt") will be used as a collective label for these phases hereafter). After re-inserting the specimen in the



Loess-paleosol sequence profile of Xihe basin at southern slope of West Qinling, [http://english.igg.cas.cn/Research\\_2015/Research\\_Divisions\\_2015/CGE/QE/Highlights7/201206/t20120611\\_86989.html](http://english.igg.cas.cn/Research_2015/Research_Divisions_2015/CGE/QE/Highlights7/201206/t20120611_86989.html)

MPMS the remanence is measured between 300-LT-300 K (cycle 2): this temperature cycling allows evaluating the shape of the curves *sans* "soft" phases, thus "looking through" the ferrimagnetic phases at goethite (Gt) and hematite (Ht) only. The specimen is subsequently warmed to 400 K to remove the contribution of Gt, and then the final cycle between 400-LT-300 K (cycle 3), reasonably reveals the contribution of Ht only. It is typically within these last two curves that if Ht grains larger than ~30 nm are present, the  $T_M$  will become visible. Subtracting the cooling remanence post-AF treatment from the initial cooling remanence (cycle 1- cycle 2) allows quantifying the contribution of Mt removed. This is typically done for the 300 K measurement only, but can be evaluated for any temperature between room and LT. The contribution of Gt can be determined by subtracting the remanence removed upon heating to 400 K, by computing the difference between the 300-LT-300 K remanences of cycles 3 and 2 as a function of temperature. As stated, any remaining remanence post-400 K heating should be representative of Ht only, but this is not always strictly the case, more on this below.

The classic goethite test suffers from the inconvenience of having to remove the specimen from the MPMS in order to perform the AF demagnetization (of course,

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pg. 9...*

## Visiting Fellow Reports

### Testing the universality and scale of the magnetic hydrocarbon migration hypothesis-Catcher development area, Western shelf, UK North Sea

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A variant magnetic fingerprint has been attributed to hydrocarbon migration pathways (e.g. Abubakar, 2016; Liu et al., 2004; Reynolds et al., 1990). Hydrocarbon migration generally occur through a complex network (Zhou et al., 2010) from the source rock (source kitchen) along available conduits. Depending on the amount of hydrocarbon generated and the subsurface structure and stratigraphy, these fluids may spill along the available conduit to potentially accumulate in a trapping system or seep out at the surface. The identification of these conduits via their magnetic signature has the potential to provide additional exploration data for a more efficient development of petroleum resources. A new magnetic hydrocarbon migration hypothesis has recently been developed (Badejo, 2019). This hypothesis suggests that hydrocarbon migration pathways have an increased presence of ferrimagnetic iron sulphides and siderite varying in a gradational manner along the pathway. It also establishes the presence of magnetic enhancement at the fluid-fluid contacts due to the precipitation of magnetite and greigite. As part of my PhD study, I intend to test the universality of these theories or delineate their scope of applications by describing the magnetic signature along the hydrocarbon migration routes. These routes will be established through well constrained basin models. This study is carried out in the Catcher development area of the Western Shelf, Central North Sea. This area is apt for this study due to the petroleum system in play. The absence of a matured source kitchen underneath the Catcher development area and the presence of the lateral continuous Palaeocene sandstone that serves as a conduit for migration of hydrocarbons reduces the uncertainty involved in modelling the basin history.

At the Institute of Rock Magnetism (IRM), thermomagnetic measurements and Mössbauer spectroscopy were carried out to enable the identification of the magnetic mineralogy of the study area. Indications of the presence of siderite were generally present in the oil stained core samples along the migration route for hydrocarbons of the Catcher development area (See figure 1a, b, c and d). Evidences of crystallographic changes associated with hexagonal pyrrhotite were also present in samples from the migration pathways (1b). Hexagonal pyrrhotite and greigite have been reported to form

in sediments under anoxic conditions (Hornig, 2018) and are known to occur in close association with one another (Larrasoana et al., 2007). Although the varying magnetic behaviour of greigite in different domain state (Chang et al., 2009) and a lack of low temperature transitions (Moskowitz et al., 1993; Chang et al., 2007) has made its identification challenging, microscopic techniques, together with high temperature techniques and evidences of other iron sulphides can be used for its identification. Unstained sedimentary samples from the same formation in the Western shelf which were isolated from the migrating hydrocarbons shown no evidence for the presence of siderite and iron sulphides (See figure 1e and f). The experiments generally revealed a Verwey transition indicative of magnetite or titanomagnetites with the unstained samples having a higher concentration of these minerals (see figure 1).

The information obtained from the experiments carried out at the IRM would be used to qualitatively and when possible quantitatively describe the magnetic signature along the migration pathways whilst focusing on the contrast in signatures between these pathways and the surrounding regions. More samples need to be analysed to achieve a holistic definition of these variations and to carry out a detailed reservoir scale magnetic study. The magnetic signature of petroleum reservoirs has been shown to relate to reservoir properties such as permeability (Ivakhnenko & Potter, 2004; Potter, 2007; Ali & Potter, 2011; Ali et al., 2013) and can be applied to prediction of the onset of production challenges such as scale formation (Imhmed, 2012).

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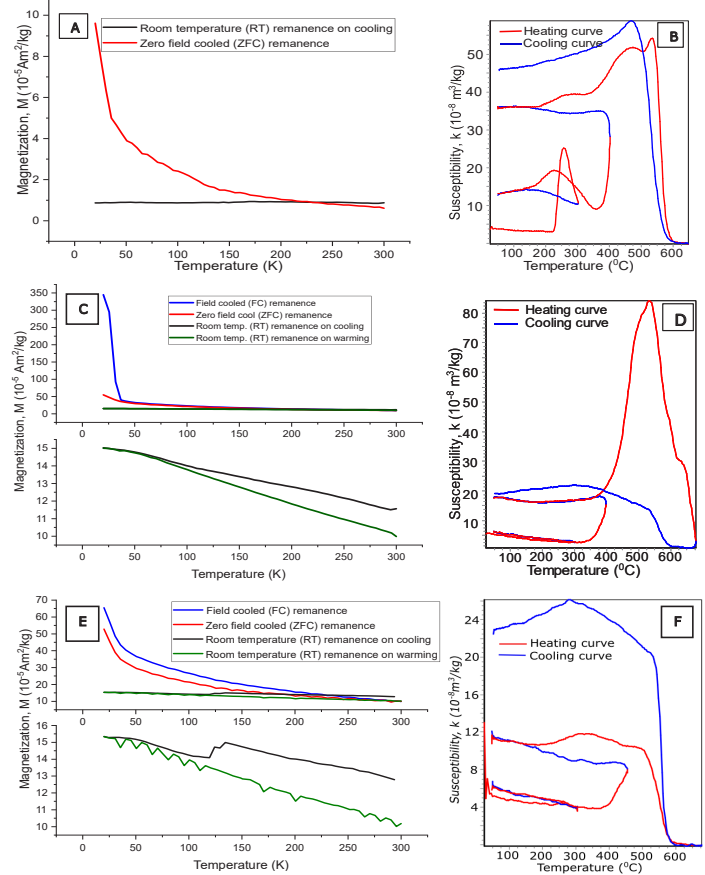


Figure 1: Representative results of the thermomagnetic experiments carried out on samples from the Catcher development area. The oil stained samples indicate the presence of siderite (a, b, c and d) and iron sulphide (d). The unstained samples showed no evidence for the presence of siderite or iron sulphides (e and f). Magnetite or/and titanomagnetite presence is evident for both the stained and unstained sample. These minerals however indicate a stronger presence in the unstained sample.





# Current Articles

A list of current research articles dealing with various topics in the physics and chemistry of magnetism is a regular feature of the IRM Quarterly. Articles published in familiar geology and geophysics journals are included; special emphasis is given to current articles from physics, chemistry, and materials-science journals. Most are taken from ISI Web of Knowledge, after which they are subjected to Procrustean culling for this newsletter. An extensive reference list of articles (primarily about rock magnetism, the physics and chemistry of magnetism, and some paleomagnetism) is continually updated at the IRM. This list, with more than 10,000 references, is available free of charge. Your contributions both to the list and to the Current Articles section of the IRM Quarterly are always welcome.

## Archaeomagnetism

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## 17th Castle Meeting

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if it is known *a priori* that the specimens do not contain any Mt, then cycle 1 can be skipped altogether, with cycle 2 following the application of the pTRM). Variations of the classic test which avoid AF demagnetization altogether have been proposed (Maher et al, 2004; Lascu and Feinberg, 2011). Alternatively, Lagroix and Guyodo (2017) utilized the oscillation mode for field sweeping of the MPMS, which effectively produces a coarse AF demagnetization without removing the sample, by applying a series of DC fields of switching polarity and progressively decreasing amplitude (approximately 30% per half cycle) which converge to zero. A subsequent ultra-low field correction is applied to cancel any residual fields. The advantage of this protocol is obvious: limited handling of the specimen by the user to perform the AF demagnetization, thus allowing to perform the different temperature cyclings continuously and “hands free”. Additional benefit is to eliminate the need to re-center the specimen, which could result in inaccuracies if the post-AF remanence is weak and a poorly resolved centering peak is obtained. It also allows the “AF” demagnetization step to be done at temperatures other than 300 K opening up the possibility of doing a “LT-Goethite Test” and capturing the remanence contribution of SP sized goethite and hematite with blocking temperatures <300K.

Appealing as it is, the process is not fool-proof, however. The major limitation is the coarseness of the switching fields applied: upon demagnetization of a magnetite specimen Lagroix and Guyodo (2017) report that 98.6% of the remanence of the specimen is removed by a 300 mT peak field. Inspecting Lagroix and Guyodo’s test for the demagnetization routine (cf. their Fig 3a) one notices that certain steps past 300 mT result in negative magnetizations, whether “ultra low-field” corrected or not. In the case of Lagroix and Guyodo’s (2017) example, the magnetite remanence after 98.6% demagnetization is  $1.28 \times 10^{-1} \text{Am}^2/\text{kg}$ , but the negative remanences resulting from the subsequent “demagnetization” steps are larger still. The large field decrement of the oscillation mode thus actually quantizes the coercivity spectra into relatively wide, positively and negatively magnetized coercivity windows, and the net moment can have quite significant magnitude. Certain specimens are more prone to reveal such behavior than others, and application of the protocol on a number of specimens in our lab has resulted in net negative magnetizations after the demagnetization routine, constituting an obvious drawback.

Such behavior was the inspiration for a new alternative protocol that is also entirely executed within the MPMS and is essentially a combination of the “hard” IRM experiment (HIRM, Thompson and Oldfield, 1986) and the goethite test. The HIRM test involves applying a strong positive saturating IRM (e.g. 2.5 T) that will saturate all the phases in the specimen. Applying this in the form of a pTRM as is done in the goethite test ensures that Gt is fully activated in the positive direction, resulting in a positive magnetization of the specimen. The remanence subsequently measured (remanence

“A”) contains the sum-contribution of +Gt, +Ht, and +Mt (when these three phases are present). With the MPMS, this remanence can be measured as a function of temperature while cycling in zero field from 300 K to LT and back (the equivalent of cycle 1), and any Mt grains that lose remanence through  $T_v$  can be observed. A smaller field IRM (e.g. 200 or 300 mT) is then applied in the opposite direction, thus effectively reversing the contribution of the “soft” phases. The resulting remanence (remanence “B”) will thus be the summation of +Gt, +Ht, and -Mt (assuming that Ht and Gt remanences are unaffected by this intermediate field). Just as for the HIRM experiment,  $(A+B)/2$  yields the joint contribution of the “hard” phases (Gt and Ht), whereas  $(A-B)/2$  provides the contribution of the “soft” phases (Mt). The respective contributions can be readily calculated for any temperature between LT and 300 K by temperature-cycling remanence **B** (cycle 2). Extending the warming curve of cycle 2 to 400 K allows removing the contribution of Gt. Subsequent cycling (cycle 3) from 400 K to LT and back to 300 K, effectively eliminates the goethite contribution to the remanence, just as in the goethite test. A simple subtraction of the remanence removed by the 400 K warming (heating curve of cycle 2- heating curve of cycle 3) from the “hard” contribution between LT and 300 K results in the isolation of Ht’s remanence in that temperature range. Boom.

There are certain caveats, however, which may affect both the HIRM and the goethite test (old and new, but from now on I will only refer to the original goethite test) protocols, and if applicable require some additional data processing. These are recognizable from the data themselves and take different forms depending on the protocol performed. The caveats may inhibit a complete separation of the relative contributions of the individual mineral phases to the bulk remanence: for example there are cases in which nano hematite particles are “softer” than larger single domain hematite grains (e.g. Vallina et al., 2004) and can be demagnetized (or have their moments reversed) by ~200 mT fields. When present, these grains will contribute to the “soft” mineral fraction isolated in these tests and will erroneously be attributed to Mt. Some maghemite grains can “survive” 200 mT AF demagnetization but lose part of their remanence by 400 K, thus affecting determination of the relative contributions of all phases. These examples will be discussed in more detail below. In the following, I will first describe a case study for which the **caveats are not applicable**: goethite tests applied to the Mauch Chunk formation red beds, containing dominant hematite, some magnetite and minimal goethite.

**Classic goethite test.** In the classic goethite test, the specimen behaves “as expected”: some remanence is lost during temperature cycling 1, reflecting non-recovery as the magnetic minerals present cycle through  $T_M$  and  $T_v$  and further remanence is lost through AF demagnetization in 200 mT fields (Fig. 1). Note that  $T_M$  is not particularly sharp, indicating a range of Ht grain sizes (specular and pigmentary), whereas  $T_v$  is most evident from FC

remanence curves (not shown here, but the reader may refer to Bilardello and Kodama, 2010).

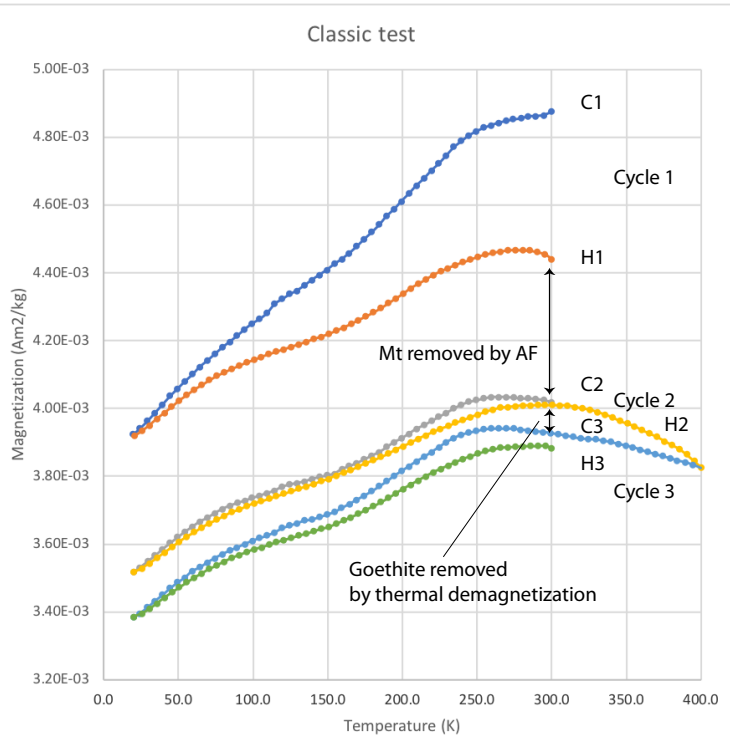


Fig. 1. Results of the classic goethite test for the Mauch Chunk formation red beds containing hematite, magnetite, and minimal goethite. The initial step of cooling in a 2.5 T field from 400 to 300 K is not shown. C and H stand for cooling and heating and each cooling-heating couple make up a temperature cycle. AF is the remanence removed upon 200 mT alternating field demagnetization.

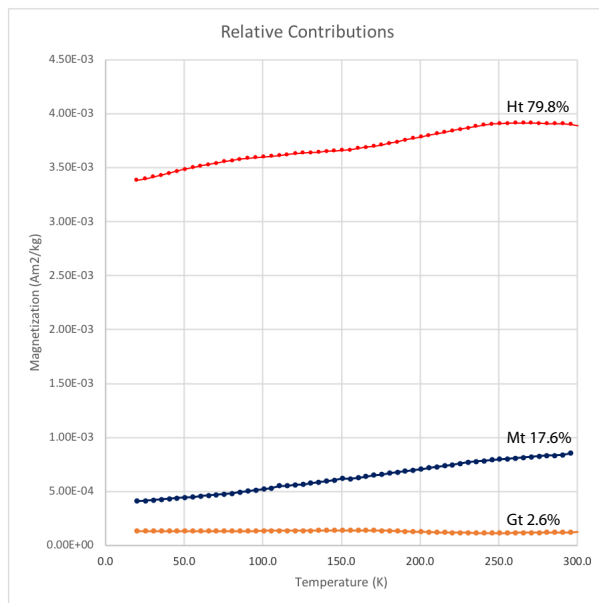


Fig. 2. Relative contributions of the magnetic phases present as determined from AF demagnetization (Mt, blue), thermal demagnetization to 400 K (Gt, orange), and remaining remanence (Ht, red).

The relative contribution of Mt is quantified as the dark blue curve in Fig. 2. Upon thermal demagnetization to 400 K a small amount of remanence that is attributed to Gt is lost (yellow curve in Fig. 1), and its relative contribution, shown as the orange curve in Fig. 2 can be

quantified by subtracting the subsequent warming curve H3 from H2. The remaining remanence, cycle 3 in Fig. 1 reflects the contribution of hematite. Some thermal hysteresis exists between cooling and heating curves C2 and H2, but the remanence is entirely recovered by 300 K. In percent, the relative remanence contributions at 300 K are 17.6% (Mt), 79.8% (Ht) and 2.6% (Gt). Note, that MPMS data are often prone to first-point artifacts, so the 300 K results may not always be the most representative: in this case, however, the relative contributions to the total remanence at 295 K are virtually identical.

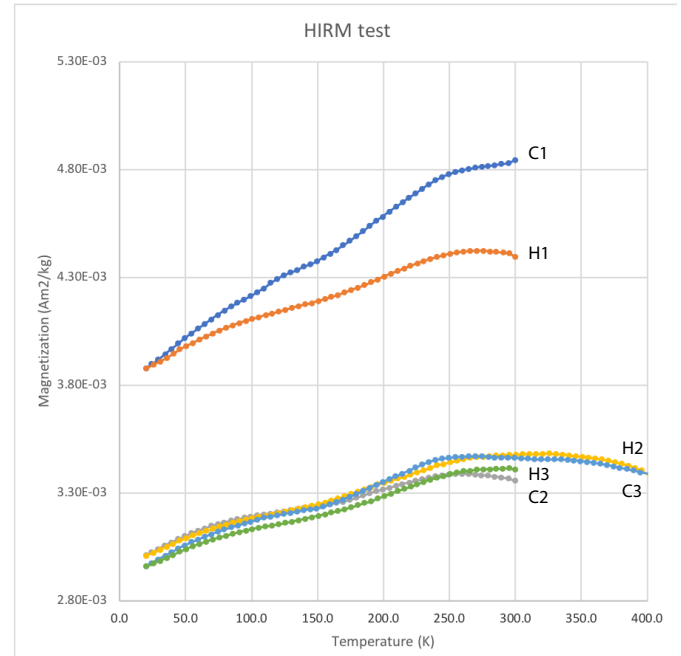


Fig. 3. HIRM-goethite test for the Mauch Chunk formation specimen. Note that the cooling curve 2 (C2, in gray) now has the lowest remanence which increases upon temperature cycling (heating curve H2, in yellow) as a consequence of non recovery of Mt remanence which points downwards.

**HIRM-Goethite test.** Subjecting the same specimen to the hybrid HIRM/goethite test similar results are obtained (Fig. 3). The first observed difference with the classic test is that the C2 curve now has the lowest remanence, owing to the magnetization of Mt pointing down. After cycling through  $T_v$  the remanence increases somewhat on warming (yellow curve H2), as a consequence of non recovery of some Mt remanence. Heating to 400 K removes the contribution of Gt, and further low temperature cycling removes additional remanence (green curve H3).

Calculating the “soft” (Mt) and “hard” (Gt + Ht) contributions at 300 K as shown above, one obtains 15.4% and 84.6% respectively. At 300 K, 1.4% of the initial remanence is removed by heating to 400 K, and can be attributed to Gt. Subtracting this from the total “hard” contribution one obtains 83.2% for hematite’s total contribution, with virtually identical results at 295 K.

The two techniques are thus in the ballpark of each other, with the greatest divergence around the relative contributions of Mt and Ht by a few %. The contribution of Gt is minimal and differs by ~1% of the total rema-

nence among both tests. In the HIRM-Gt test, 200 mT DC fields are considered the equivalent of 200 mT AF demagnetization, but strictly speaking they are not, and in fact IRMs are more effective at capturing the whole spectrum of grain coercivity, depending on the AF frequency they are compared to. The DC fields, because stronger, may also be more effective at reversing the moment of some nano-Ht grains, if present, thus affecting the “hard”/“soft” mineral ratio.

Case in which the **caveats are applicable**. An atmospheric dust containing maghemite, hematite and goethite (Reynolds et al., 2014) was subjected to both tests.

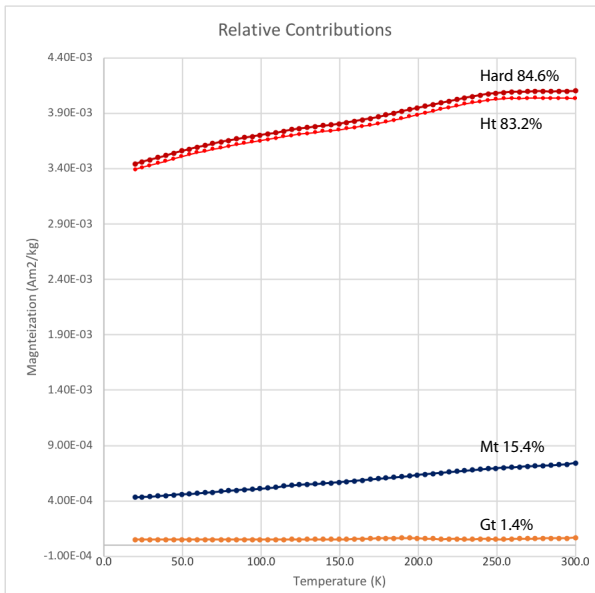


Fig. 4. Relative contributions of the magnetic phases present. These are comparable to those obtained through the classic goethite test. Colors as in figure 2, the dark red curve is for the “hard” components combined.

The **classic goethite test** immediately reveals a dominant Mt contribution (cycle 1, pre 200 mT AF demagnetization) compared to the “hard” phases present (post-AF) (Fig. 5). After the removal of Mt though AF, an increase of remanence between 300 K and 20 K is apparent, which is typical of goethite. Some temperature hysteresis is present between ~150 and 300 K, thus around  $T_M$ . Further decrease of magnetization is experienced when warming to 400 K, as expected. Temperature cycle 3 still shows the increase in remanence upon cooling, though subdued, and the hysteresis is more pronounced (Fig. 5)

Determining the relative contributions to the remanence as a function of temperature, Mt makes up 93.4% at 300 K (and 295 K), leaving 6.6% for the “hard” phases. The goethite contribution at 300 K removed by heating is small (0.6%), however, the increase in remanence upon cooling observed in cycle 3 suggests that not all of the Gt was removed by thermal demagnetization at 400 K. There is also the likely possibility that this “unremoved Gt” is possibly “hard” maghemite that resists AF demagnetization. I will discuss this further below, for the time being I will keep referring to it as Gt. To obviate this effect, and assuming that all goethite remanence

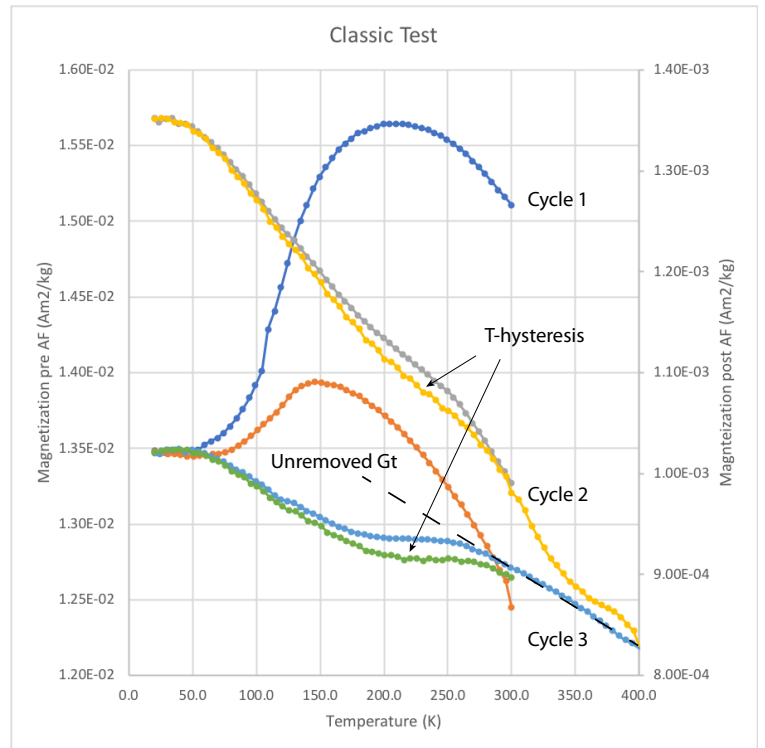


Fig. 5. Classic goethite test performed on a dust sample containing maghemite, hematite and goethite. Note the different scales for the pre- and post-AF measurements. Thermal hysteresis about the Morin transition is highlighted, as well as a negative slope corresponding to the contribution of goethite that was not removed by 400 K thermal demagnetization. See text for details.

should be removed by 400 K, the remanences in cycle 3 have been slope-corrected, by fitting a line through data above  $T_M$  and restoring this to horizontal by pivoting around the 400 K value of magnetization, thus allowing to determine the “true” contribution of Ht (Fig. 6). The difference (0.5%) can then be attributed to Gt not removed by 400 K and used to compile the total Gt contribution (1.1%). The corrected contributions of Gt and Ht are reported in Fig. 7. As expected, the Gt relative contribution removed is larger at low temperature.

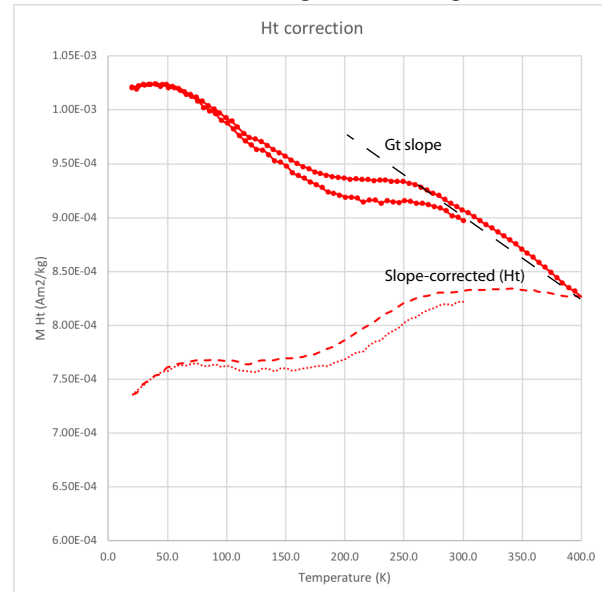


Fig. 6. Correction for the goethite slope of the hematite data from cycle 3 (dashed and dotted lines are the cooling and warming curves, respectively).



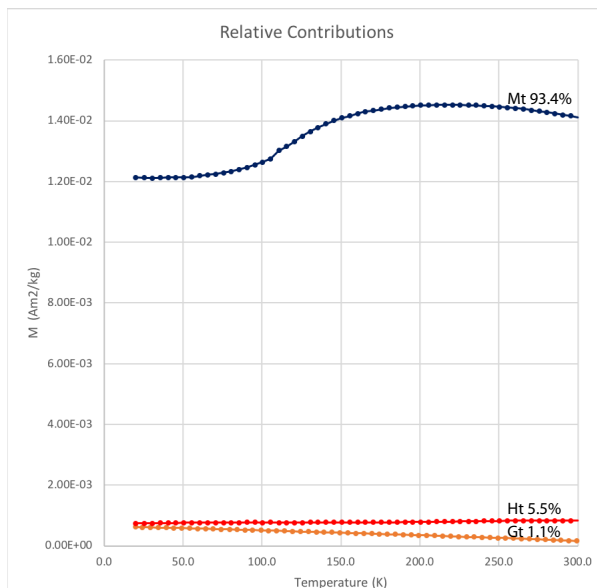


Fig. 7. Relative contributions of the different iron oxides. The contributions of hematite (red) and goethite (orange) are calculated after correcting for the slope of goethite in cycle 3 (see Fig. 6).

**HIRM-Goethite test.** Performing the alternate test on the same specimen, the remanences of cycles 2 and 3 are negative, owing to the dominance of Mt present (Fig. 8a). Evaluating the remanence in absolute values is sometimes helpful (Fig. 8b), however one must bear in mind that the Mt contribution is never removed, simply reversed, and contributes to all cycles shown.

Computing the “soft” and “hard” contributions, 93% and 7%, respectively are obtained (Fig. 9), providing an excellent comparison with the classic test. Separating the relative contributions of Gt and Ht, however, is trickier. The “Gt” remanence removed upon heating to 400 K, obtained by subtracting the heating curve 3 (cycle 3, -Mt, +Ht) from the heating curve 2 (cycle 2, -Mt, + Ht,

+ Gt), in fact appears to be larger than the total “hard” contribution (Fig. 9), amounting to 10.6% of the total remanence. In the previous example, Gt appeared to not be completely removed by the 400 K thermal demagnetization, however, if this were the case in the HIRM test, then the Gt contribution would be even larger than 10.6%. More likely, some nano-maghemite is thermally demagnetized by 400 K, enhancing the apparent Gt contribution. In fact, Carter-Stiglitz et al. (2006b), following Smirnov and Tarduno (2000), acknowledge that maghemite may have similar low temperature behavior to goethite. That the hematite slope in Fig. 6 was corrected for some maghemite unremoved by AF demagnetization thus remains a likely possibility.

Despite these issues, the excellent agreement of the relative “soft” and “hard” contributions for the two tests is encouraging, and attests to the validity of 200 mT AF in removing most of the Mt contribution, and 200 mT DC demagnetization in reversing the polarity of the same grains. Whether it is the goethite or the maghemite contribution that needs to be adjusted after the slope correction, however, and thus affecting the total “hard” and “soft” contributions determined through both tests, still remains a matter of debate. Unlike the classic test, however, in the HIRM test it is not possible to correct the Ht contribution by adjusting the slope of the cycle 3 curves, because the Mt contribution is still heavily present in those curves (rather, one can adjust the slope, but the magnitude will still be off). This contribution cannot be effectively subtracted without making heavy assumptions on how much of its remanence is removed/remains through non-recovery across the  $T_v$  during and after temperature cycles 2 and 3. For similar specimens, then, the classic goethite test thus remains the preferred option, though if a slope in cycle 3 remains, then the culprit mineralogy (Gt or Mt) should be investigated more closely. Performing the Lacroix and Guyodo (2017) test on Mt rich samples may also introduce uncertainties

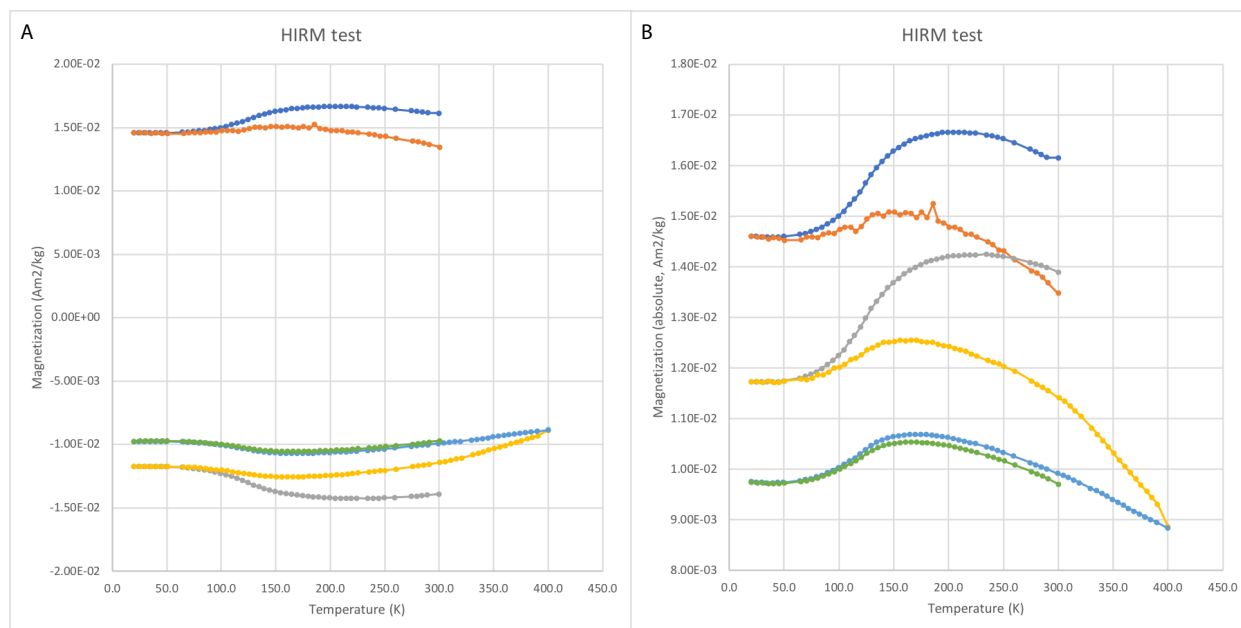


Fig. 8. Results of the HIRM test on the atmospheric dust specimen: A) Measured data; B) absolute values of the magnetizations. Note the further loss of maghemite remanence during temperature cycle 3.

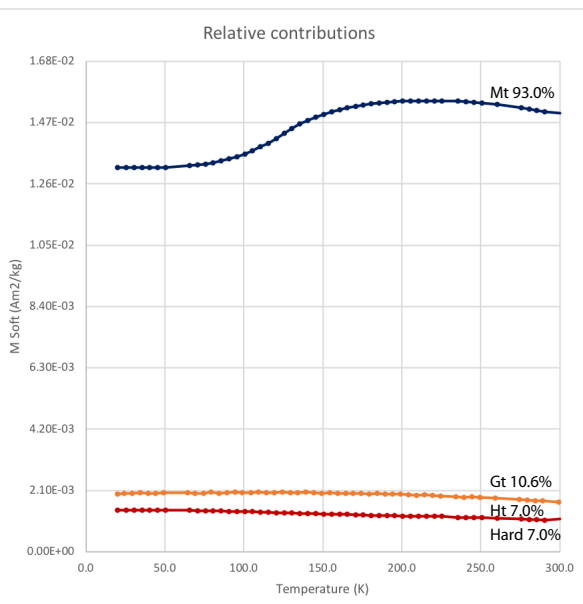


Fig. 9. Relative contributions of the “soft” (maghemite, blue) and “hard” (hematite plus goethite, dark red) remanences isolated. Using the HIRM protocol it is not possible to separate the contributions of goethite and hematite, as evidenced by the apparent goethite contribution (orange) that is larger than the bulk “hard” contribution. See text for details.

around how much Mt contribution is effectively demagnetized, which, if the bulk of the remanence is positive, may largely go undetected.

As briefly mentioned, there is also the possibility of introducing error due to partial demagnetization/switching of the “hard” phases, especially hematite. In soils hematite is generally very hard and the standard assumption that it’s unaffected by 200 mT probably holds, however, small authigenic particles (or aggregates) at the SP-SD boundary may have lower coercivity. Likewise larger hematite crystals in igneous/metamorphic rocks may also have very low coercivity. While these tests are generally geared towards sedimentary magnetism they are not completely general and the possible caveats must always be considered.

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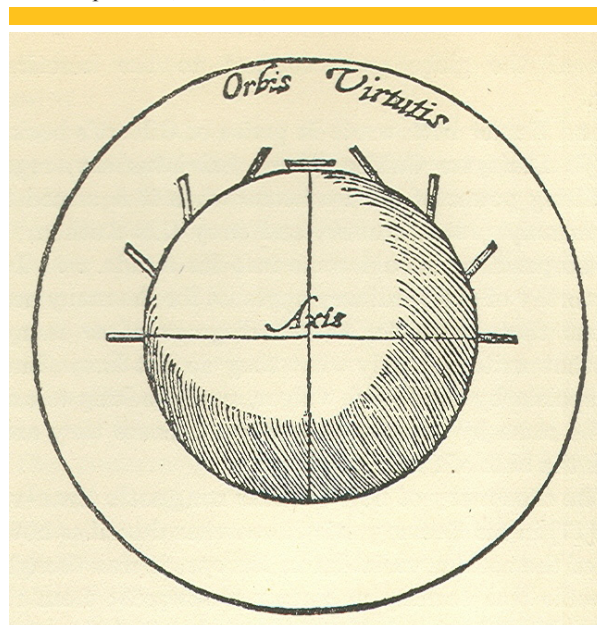
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