nanoScale
Biomagnetics
NANOSCALE BIOMAGNETICS SL CIF B99210171
Calle María de Luna 11 CEEI Aragón Nave 10B
+34 976 741714

19 -01- 2012

# Measuring technical report.

1 Commercial Data 2
2 Technical Information 3
1.1. Supplied Sample: MAGNO. 3
2.1 Tests requested 4
2.2 The DMC1+DM1 system. 5
3 Performed tests 5
4 Results 6
4.1 Single results for MAGNO Sample: 6
4.1.1 Results at day 1 (580 kHz-300G): 6
4.1.2 Results at day 7 (580kHz-300G): 7
5 SPA and ILP index 9
5.1 Sample Characteristics 9
5.2 Calculation of the $(\Delta T/\Delta t)_{max}$ 10
5.3 Calculation of the SPA index and ILP index 10
6 Graphs List 12
7 Tables list 12

NANOSCALE BIOMAGNETICS SL CIF B99210171
Calle María de Luna 11  CEEI Aragón Nave 10B
+34 976 741714

# 1 Commercial Data

| nB | |
|---|---|
| **Contact** | |
| **Expert Technician** | |
| **Report** | |
| **Test date** | |

nanoScale Biomagnetics
NANOSCALE BIOMAGNETICS SL CIF B99210171
Calle María de Luna 11  CEEI Aragón Nave 10B
+34 976 741714

# 2 Technical Information (As supplied by the customer)

### 1.1. MAGNO sample.

The supplied magnetic colloid is composed of a magnetite core coated with poly-ethylene imine (PEI) in aqueous solution. Physical properties are summarized in Table 1. Other physical parameters used to characterize the magnetic colloid are listed in Table 2.

| Volume | Concentration | Core Size | Hydrodynamic Diameter | Polidispersity | Description |
|---|---|---|---|---|---|
| **5 ml** | 2,5 mg/ml | 25±3 nm | 364 nm | 0,125 | $Fe_3O_4$-PEI NPs in water |

**Table 1.** MAGNO Sample.

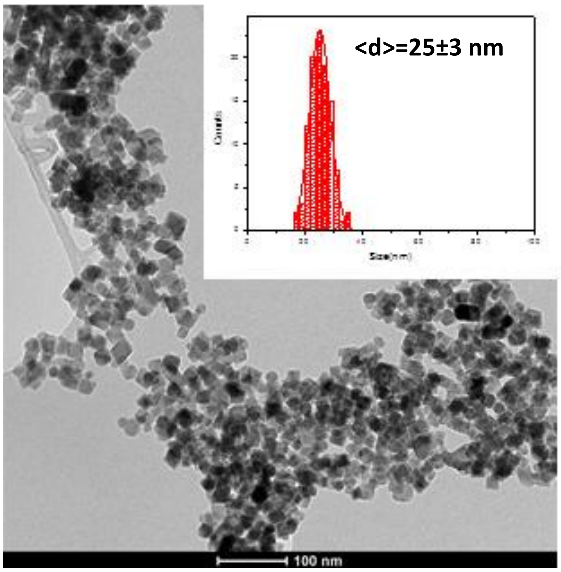


**Figure 1.** Transmission electron microscopy (TEM) images and histograms of magnetic colloid.

| Base Liquid | $C_L$ (Kcal/KgºC) | $\delta_L$ ($Kg/m^3$) | $\phi_L$ ($Kg/m^3$) |
|---|---|---|---|
| **Water [$H_2O$]** | 1.0 | 1000.00 | 2,50 |

**Table 2.** Physical parameters used to characterize the magnetic colloid.

### 2.1 Available tests

Basic test protocol: two different experiments are performed to characterize the magnetic colloid. First, studies at different field amplitudes and fixed frequency *(Table 2).* These experiments are made by quadruplicate, unless otherwise indicated by the customer. In this way a precise determination of SPA values and associated error is provided to the customer. In the second experiment of the basic protocol, the SPA value is studied at different frequencies *f*, at constant field amplitude $H_0$. The heating curves obtained, as well as the resulting SPA values from the fitting model are organized and tabulated as shown in *Table 3.*

| Test | Probe sample | Sample Volume | Test Time | Field Frequency | Field |
|---|---|---|---|---|---|
| **E1** | Insulation, 1ml | 1 ml | 180 s | 580 kHz ± 1 kHz | 300G ± 15G |
| **E2** | Insulation, 1ml | 1 ml | 180 s | 580 kHz ± 1 kHz | 270G ± 15G |
| **E3** | Insulation, 1ml | 1 ml | 180 s | 580 kHz ± 1 kHz | 240G ± 15G |
| **E4** | Insulation, 1ml | 1 ml | 180 s | 580 kHz ± 1 kHz | 210G ± 15G |
| **E5** | Insulation, 1ml | 1 ml | 180 s | 580 kHz ± 1 kHz | 180G ± 15G |
| **E6** | Insulation, 1ml | 1 ml | 180 s | 580 kHz ± 1 kHz | 150G ± 15G |

**Table 3.** Description of the relevant experimental parameters for the heating data as a function of applied field amplitude $H_0$.

| Test | Probe sample | Sample Volume | Test Time | Field Frequency | Field |
|---|---|---|---|---|---|
| **E1** | Insulation, 1ml | 1 ml | 180 s | 828 kHz ± 1 kHz | 232G ± 15G |
| **E2** | Insulation, 1ml | 1 ml | 180 s | 580 kHz ± 1 kHz | 232G ± 15G |
| **E3** | Insulation, 1ml | 1 ml | 180 s | 444 kHz ± 1 kHz | 232G ± 15G |
| **E4** | Insulation, 1ml | 1 ml | 180 s | 355 kHz ± 1 kHz | 232G ± 15G |
| **E5** | Insulation, 1ml | 1 ml | 180 s | 315 kHz ± 1 kHz | 232G ± 15G |
| **E6** | Insulation, 1ml | 1 ml | 180 s | 265 kHz ± 1 kHz | 232G ± 15G |
| **E7** | Insulation, 1ml | 1 ml | 180 s | 248 kHz ± 1 kHz | 232G ± 15G |
| **E6** | Insulation, 1ml | 1 ml | 180 s | 229 kHz ± 1 kHz | 232G ± 15G |

**Table 4.** Description of the relevant experimental parameters for the heating data as a function of applied field frequency f.

**2.2 The DMC1+DM1 system.**

The tests are performed with a basic DM100 setup, integrated by the DMC1 system controller, and the DM1 applicator. The DM1 has been designed to perform calorimetric experiments in magnetic colloids. The DM1 includes a cylindrical probe-sample holder that is introduced in a dewar flask connected to a vacuum pump, in order to guarantee the best possible insulation. An inductor generates the magnetic field that is applied on the system dewar/probe-sample holder. The whole system is showed in the following figure.

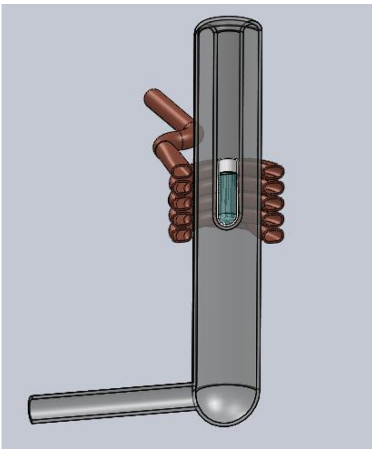

**Figure 2.** DM1 Applicator Scheme.

The field direction along the z-direction (vertical) has a high uniformity, better than 90 %, all over the sample volume. For a given working frequency, the ac field has a single component (i.e., is 'monochromatic') without any higher harmonics that could contribute to the heating process. The DM100's temperature measuring system uses a fibre optic sensor. The sensor is placed inside the sample. Data is registered at a rate of 200 ms with a 0.2 $^{o}$C resolution.

# 3 Test structure

- Both experiments of the basic protocol (Tables 2 and 3) can be performed on a n-fold basis as required by the customer.
- Additional requirements before or after measurements (i.e., sonication, cooling, quenching, etc) should be specified by the customer.
- By default the basic protocol is designed as follow:
  - Every test is made four times, evaluating the repeatability of the specific sample.
  - The sample is briefly sonicated before each test.
  - The sample is cooled in a non-forced way between tests.
- Before each test, the system dewar/probe/sample holder is left until thermal equilibrium is reached (thermalization of the system).

# 4 Results

The results obtained for the specified protocol are illustrated and the graphs provided. For the basic protocol, each test will be illustrated by two different graphical outputs:

- Graph 1: show the temperature curves of the four measurements (T1, T2, T3, T4).
- Graph 2: show the temperature increment curve (ΔT) of the four measurements (T1, T2, T3, T4).

*Note: The Graph 2 is provided as an easier way to compare between the different tests.

## 4.1 Examples: Single results (from MAGNO sample):

### 4.1.1 Results 1 (580 kHz-300G):

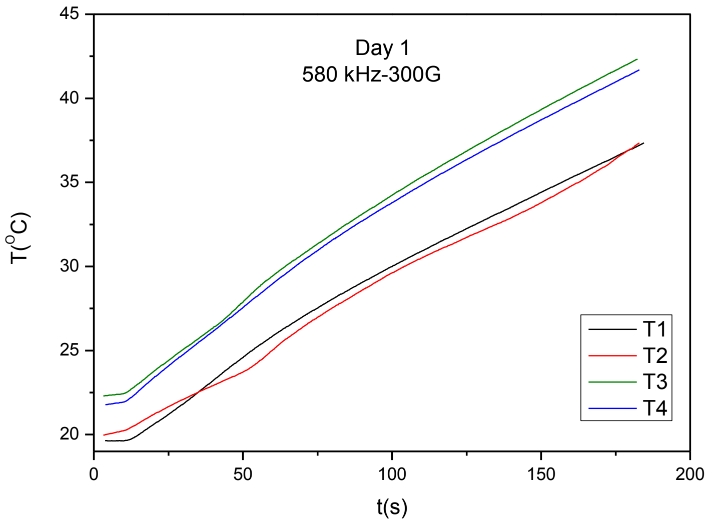


**Figure 3.** Graph 1 example for MAGNO (Tests E1).

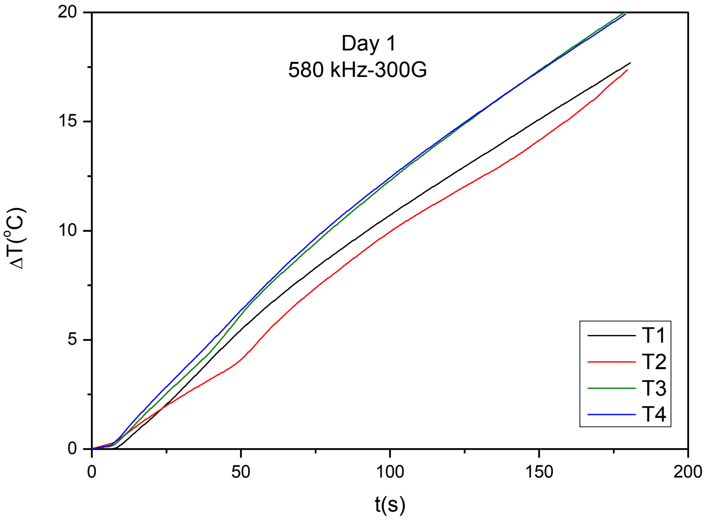


**Figure 4.** Graph 2 example for MAGNO (Tests E1).

### 4.1.2 Results 2 (580kHz-300G):

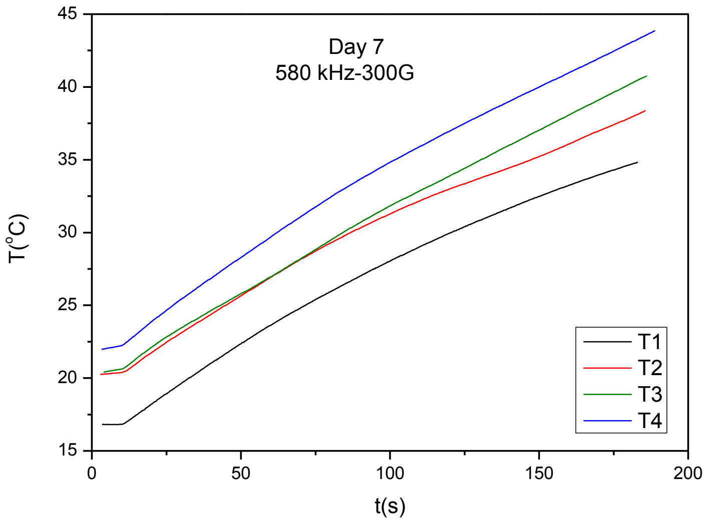


**Figure 5.** Graph 1 example 2 for MAGNO (Tests E2).

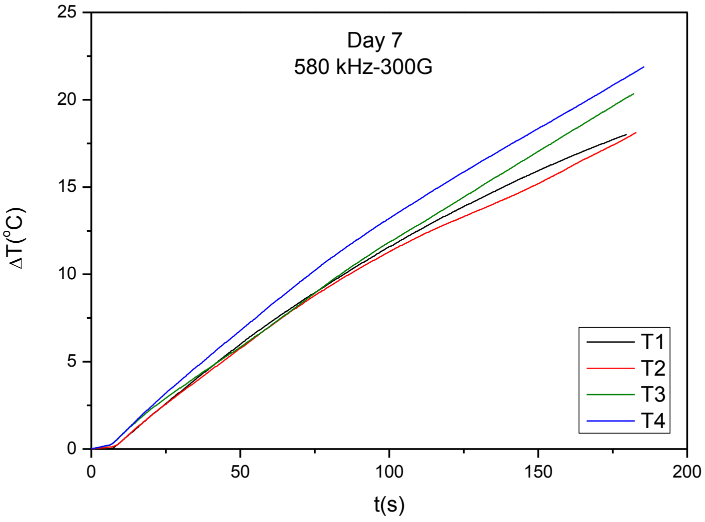


**Figure 6.** Graph 2 example 2 for MAGNO (Tests E2).

# 5 SPA and ILP index

The SPA index is the "Specific Power Absorption" of a sample subjected to heating. The SPA is calculated using the following formula:

$$SPA = \frac{C_L \delta_L}{\phi_L} \left(\frac{\Delta T}{\Delta t}\right)_{max} \ (Formula\ 1)$$

Where:

- $C_L$ : Calorific Capacity of the base liquid (J/Kg·K)
- $\delta_L$ : Density of the base liquid ($Kg/m^3$)
- $\phi_L$ :Concentration of the nanoparticles ($Kg/m^3$)
- $(\Delta T/\Delta t)_{max}$ : Differential of Temperature maximum respect to time of the temperature curve of the sample (K/s)
- SPA: Specific Absorption Ratio (W/g)

The SAR index depends linearly on the frequency and quadratically on the intensity of the applied field. For this reason, a new index is suggested: the ILP index (Intrinsic Loss Power). The ILP is used to the characterization of the intrinsic efficiency of the magnetic nanoparticles to absorb power. This new parameter is defined as:

$$ILP = \frac{SPA}{f H_0^2} \ (Formula\ 2)$$

Where:

- SPA: SPA index [W/g]
- f: frequency of the magnetic field applied [Hz]
- $H_o$: intensity of the magnetic field applied [A/m]

The units of the ILP index are $nH \cdot m^2/kg$

The ILP value of a magnetic colloid is independent of the used device.

## 5.1 Sample Characteristics

The base liquid of the simple supplied MAGNO is water [$H_2O$]. The nanoparticles concentrations are 2,5mg/ml.

Some of the physical properties of the sample are showed below.

| Sample | Base Liquid | $C_L$ (Kcal/KgºC) | $\delta_L$ ($Kg/m^3$) | $\phi_L$ ($Kg/m^3$) |
|---|---|---|---|---|
| **MAGNO** | Water [$H_2O$] | 1.0 | 1000.00 | 2,50 |

**Table 5.** Properties physical of the sample.

### 5.2 Calculation of the $(\Delta T/\Delta t)_{max}$

In order to obtain the temperature differential respect to time of the sample in every test, the following process has been done:

1. Exponential adjustment of the experimental temperature curve obtained in every test.
2. Calculation of the derivate of the exponential adjustment curve respect to time.
3. Calculation of the $(\Delta T/\Delta t)_{max}$ as the value of the derivate of the expoential adjustment curve.

### 5.3 Calculation of the SPA index and ILP index

The index SPA and ILP have been calculated using the formulas described at the beginning of this section (Formula 1 and Formula 2) with the data obtained during the tests and the characteristics of the sample.

The SAR has been calculated for every repetition (T1, T2, T3 and T4) of every type of test (E1, E2, E3, and E4). Then, the mean value of SPA and the standard deviation of SAR have been calculated for every type of test. The calculated values of SPA for every test are showed in the following tables.

- SPA for tests at 580 kHz:

| Test | Frequency [KHz] | Field [G] | SPA [W/g] | | | | SPA (*) [W/g] | SD (**) |
|---|---|---|---|---|---|---|---|---|
| | | | T1 | T2 | T3 | T4 | | |
| **E1** | 580 | 300 | 216 | 184 | 227 | 241 | **217** | 24 |
| **E2** | 580 | 270 | 142 | 121 | 118 | 131 | **128** | 11 |
| **E3** | 580 | 240 | 95 | 94 | 83 | 74 | **87** | 10 |
| **E4** | 580 | 210 | 59 | 38 | 54 | 73 | **56** | 14 |
| **E5** | 580 | 180 | 40 | 29 | 30 | 32 | **32** | 5 |
| **E6** | 580 | 150 | 9 | 9 | 13 | 16 | **12** | 3 |

**Table 6.** SPA for tests at 580 kHz at day 1.

| Test | Frequency [KHz] | Field [G] | SPA [W/g] | | | | SPA (*) [W/g] | SD (**) |
|---|---|---|---|---|---|---|---|---|
| | | | T1 | T2 | T3 | T4 | | |
| **E1** | 580 | 300 | 223 | 215 | 189 | 221 | **212** | 15 |
| **E2** | 580 | 270 | 143 | 120 | 141 | 114 | **130** | 15 |
| **E3** | 580 | 240 | 103 | 94 | 109 | 76 | **95** | 14 |
| **E4** | 580 | 210 | 48 | 30 | 64 | 59 | **50** | 15 |
| **E5** | 580 | 180 | 34 | 15 | 33 | 22 | **26** | 9 |
| **E6** | 580 | 150 | 15 | 17 | 15 | 16 | **16** | 1 |

**Table 7.** SPA for tests at 580 kHz at day 7.

- SPA for tests at 232 G:

| Test | Frequency [KHz] | Field [G] | SPA [W/g] | | | | SPA (*) [W/g] | SD (**) |
|---|---|---|---|---|---|---|---|---|
| | | | T1 | T2 | T3 | T4 | | |
| **E1** | 828 | 232 | | | | | | |
| **E2** | 580 | 232 | | | | | | |
| **E3** | 444 | 232 | | | | | | |
| **E4** | 355 | 232 | | | | | | |
| **E5** | 315 | 232 | | | | | | |
| **E6** | 265 | 232 | | | | | | |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **E7** | 248 | 232 | | | | | | |
| **E8** | 229 | 232 | | | | | | |

**Table 8.** SPA for tests at 232 G.

(*) Mean value
(**) SD: standard desviation.

**Comparative graphs between the mean values of SPA:**

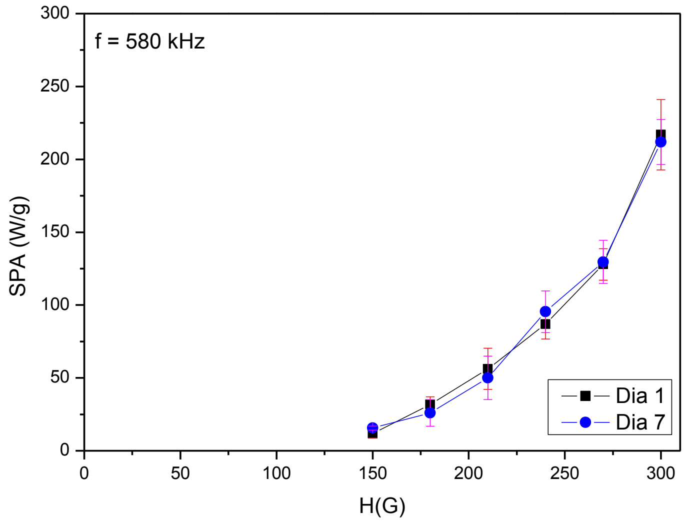


**Figure 8.** SPA comparison at 580 kHz .

# 6 Graphs List

**Figure 1.** Transmission electron microscopy (TEM) images and histograms of magnetic colloid. 3

**Figure 2.** DM1 Applicator Scheme. 5

**Figure 3.** Graph 1 which shows the temperature curves obtained at day 1 post-synthesis (Tests E1). 6

**Figure 4.** Graph 2 which show the temperature increment curves obtained at day 1 post-synthesis(Tests E1). 7

**Figure 5.** Graph 1 which shows the temperature curves obtained at day 1 post-synthesis(Tests E1). 7

**Figure 6.** Graph 2 which show the temperature increment curves obtained at day 1 post-synthesis(Tests E1). 8

**Figure 8.** SPA comparison at 580 kHz . 11

# 7 Tables list

**Table 1.** Supplied Samples. 3

**Table 2.** Physical parameters used to characterize the magnetic colloid. 3

**Table 3.** Description of the tests characteristics 4

**Table 4.** Description of the tests characteristics **¡Error! Marcador no definido.**

**Table 5.** Properties physical of the sample. 9

**Table 6.** SPA for tests at 580 kHz at day 1. 10

**Table 7.** SPA for tests at 580 kHz at day 7. 10

**Table 8.** SPA for tests at 232 G. 11