Towards a Model-Based Field-Frequency Lock for NMR

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Abstract: Nuclear Magnetic Resonance (NMR) relaxometry is a powerful technique that allows to investigate the properties of materials. More advanced NMR relaxometry techniques such as Fast Field-Cycling (FFC) require the magnetic field to reach any desired value in a very short time (few milliseconds) and field oscillations to stay within few ppms. Such specifications call for the introduction of a suitable Field Frequency Lock (FFL) system. FFL relies on an indirect measure of the magnetic field which can be obtained by performing a parallel NMR experiment with a known sample. In this paper we propose a PID controller to guarantee field fluctuations to stay below the desired level and short settling time. The tuning of the controller is based on a mathematical description of the entire process, which is validated by performing real experiments. Numerical simulations show promising results that we expect to be confirmed by real experiments.

Keywords: NMR, Field-Frequency Lock, PID, Bloch Equations, LS

1. INTRODUCTION

NMR allows to gather information about properties of an unknown sample by studying its resonance frequency ω_0 when placed in a known, constant magnetic field B_0. The resonance frequency ω_0 is in fact given by

$$\omega_0 = -\gamma B_0 \qquad (1)$$

where γ is the gyromagnetic ratio typical of the nuclear specie (may not be known a priori) and B_0 is the main magnetic field, typically generated by a resistive or superconducting magnet. If the value of B_0 is known and stable, it is then possible to identify the nuclear specie in the sample, as well as to study molecular structures and interactions. The FFL system is a well-known approach to avoid magnetic field oscillations, which may degrade the performance of the NMR experiment. The idea is to obtain an indirect but very fine grained measure of the magnetic field fluctuations from a parallel NMR experiment, that is carried out over a known sample (e.g. 2H) experiencing the same magnetic field we wish to control (Hoult et al. (1978); Kan et al. (1978); Maly et al. (2006); Samra (2008); Yanagisawa et al. (2008); Jiang et al. (2010); Li et al. (2011)).

If a field deviation $\Delta B(t)$ (which may arise from current oscillations or from external electromagnetic disturbances) is present, Equation (1) can be written as

$$\omega_0 + \Delta\omega(t) = -\gamma(B_0 + \Delta B(t))$$

therefore $\Delta B(t)$ results in a frequency deviation $\Delta\omega(t)$ such that

$$\Delta\omega(t) = -\gamma\Delta B(t) \qquad (2)$$

where the value of γ is known for the lock sample. The NMR lock signal is obtained after the quadrature detector and oscillates at $\Delta\omega(t)$; it is used to setup a closed loop which must reduce oscillations in the magnetic field.

In FFC NMR a standard control loop takes care of the tracking of the field profile, but cannot provide the desired precision during the measurement phase. The FFL loop must then be used when the field reaches a neighbourhood of the desired measurement value. This means that the FFL must deal with the following requirements:

- steady state perfect tracking of a step reference;
- maximum settling time T_{sett} smaller than a given value;
- disturbance rejection.

Since no FFL systems for FFC are currently available, it is necessary to update the conventional solutions to cope with the former specifications.

According to the literature, different approaches are possible to implement the FFL. The classical one is to realize the loop as a Phase Locked Loop (PLL), where the NMR lock signal is compared to a reference one and an error signal proportional to $\Delta\omega(t)$ is generated. This error signal can be used to feed a P or PI regulation block (see Kan et al. (1978); Hoult et al. (1978); Jiang et al. (2010)). Still, this approach suffers from low SNR and is ineffective in rejecting high frequency noise (Samra (2008)). To overcome these problems a different approach is required. The lock sample is stimulated with a series of low power, high repetition rate pulses, which bring the sample in the so called Steady State Free Precession (SSFP) regime (Carr (1958); Patz (1988); Gyngell (1989); Bagueira de Vasconcelos Azeredo et al. (2000)). However, this approach calls for a detailed model of the NMR lock experiment for a proper synthesis

Fig. 1. The magnetization vector M in the rotating reference frame xyz.

Fig. 2. The lock sequence, a series of identical RF pulses with inter-pulse period T and tip angle θ.

Fig. 3. The static input/output relation of the NMR as a sensor. $FWLR$ is the Full Width of the Linear Region, i.e. the width of the interval $[\Delta B^{min}; \Delta B^{max}]$. Here the curve can be approximated as a straight line.

of the regulator. Just few works in literature exploit an NMR model for the design of the controller (e.g. Hoult et al. (1978); Samra (2008); Schett and Schwilch (1994)), and none of them performs an exhaustive comparison of existing models to understand their advantages and limitations for control purposes. The main aim of this work is then the analysis of existing models for NMR in SSFP regime (Bloch (1946); Hahn (1950); Schiano et al. (1991); Samra (2008); Moraesa and Colnago (2014)), their validation by means of real NMR experiments and the derivation of a linear model that can be used to drive the synthesis of a PID regulator. A methodology for the synthesis is described and the overall closed-loop design is tested by performing numerical simulations which confirm the correctness of the approach.

2. LOCK SEQUENCE AND NMR AS SENSOR

As stated in the previous section, the lock sample must be stimulated using a sequence of low power, high repetition rate pulses to obtain an NMR signal which can be exploited as a measure of the magnetic field deviation ΔB. For sake of clarity let us introduce a rotating reference frame, xyz, with the z axis aligned with the magnetic field B_0 and rotating at the nominal resonance frequency $\Omega = -\gamma B_0$. The situation is depicted in Figure 1. Let θ be the angle the magnetization vector M moves away from the z axis because of each Radio Frequency (RF) pulse. Let T be the inter-pulse period. The lock sequence, shown in Figure 2, is then composed of all identical RF pulses with θ small (i.e. few degrees) and $T << T1, T2$ (possibly $T < T2^*$), where $T1$ and $T2$ are respectively the spin-lattice relaxation time constant and the spin-spin relaxation time constant, while $T2^*$ is the spin-spin time constant in a non-homogeneous magnetic field (Keeler (2011)). When the NMR lock sample is stimulated this way, the magnetization vector oscillates around a steady state position (Carr (1958)). If, in particular, RF pulses are applied along the y axis, then the magnetization reaches a steady state condition (SSFP) in the xz plane. If no field deviation is present (i.e. $\Delta B = 0$), the y component, M_y, is zero. If, instead, a field deviation is present, M_y provides a measure of the former quantity. Notice that the ΔB to M_y curve (see Figure 3) is bijective only if restricted around $\Delta B = 0$, meaning that we can properly sense field deviations in the interval $[\Delta B_{min}; \Delta B_{max}]$ (Samra (2008)).
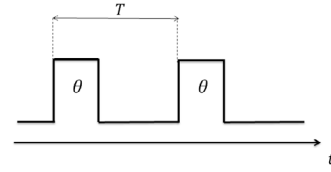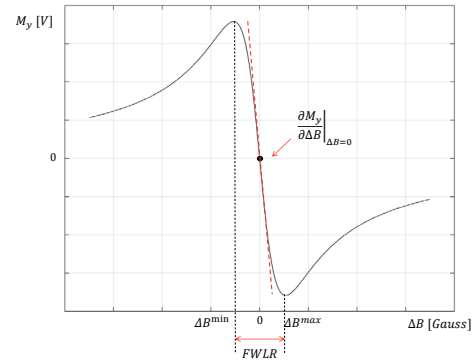
3. CLASSICAL MODELS FOR NMR: ANALYSIS AND VALIDATION

The usual way of describing the motion of M during an NMR experiment is through Bloch Equations (BE) (Bloch (1946)). Let M_0 be the magnitude of M at equilibrium (measured in Volt) and let B_1 be the amplitude of the RF magnetic field. We then write BE in the rotating reference frame xyz, assuming RF pulses to be applied along $-y$ (i.e. $B_1 < 0$), as

$$\begin{cases} \dfrac{dM_x(t)}{dt} = \dfrac{-1}{T2} M_x(t) + \gamma \Delta B(t) M_y(t) - \gamma B_1(t) M_z(t) \\ \dfrac{dM_y(t)}{dt} = -\gamma \Delta B(t) M_x(t) - \dfrac{1}{T2} M_y(t) \\ \dfrac{dM_z(t)}{dt} = \gamma B_1(t) M_x(t) - \dfrac{1}{T1} M_z(t) + \dfrac{M_0}{T1} \end{cases} \quad (3)$$

As a dynamic system, Equations (3) require two inputs: the first one is the field deviation $\Delta B(t)$, the second one is the square wave representing the lock sequence of RF pulses. Notice that we are interested in $M_y(t)$ as the output of the system. When $\Delta B(t)$ varies as a step, the output $M_y(t)$ converges to a periodic behaviour rather than a single equilibrium value since $B_1(t)$ behaves as a square wave. This makes it difficult to perform linearization. To overcome this problem we move to a discretized version of BE, which is described in many works (Samra (2008); Moraesa and Colnago (2014); Carr (1958)).

The evolution of M is then given by

$$M(k+1) = A(T1,T2)W(\phi(k))V(\theta)M(k) + B(T1)$$

$$\phi(k) = -\gamma T \Delta B(k)$$

$$A(T1,T2) = \begin{bmatrix} e^{\frac{-T}{T2}} & 0 & 0 \\ 0 & e^{\frac{-T}{T2}} & 0 \\ 0 & 0 & e^{\frac{-T}{T1}} \end{bmatrix}$$

$$B(T1) = \begin{bmatrix} 0 \\ 0 \\ M_0(1 - e^{\frac{-T}{T1}}) \end{bmatrix} \quad (4)$$

$$V(\theta) = \begin{bmatrix} \cos(\theta) & 0 & \sin(\theta) \\ 0 & 1 & 0 \\ -\sin(\theta) & 0 & \cos(\theta) \end{bmatrix}$$

$$W(\phi) = \begin{bmatrix} \cos(\phi) & -\sin(\phi) & 0 \\ \sin(\phi) & \cos(\phi) & 0 \\ 0 & 0 & 1 \end{bmatrix}$$

Matrix A and vector B describe the process of relaxation in the dynamics of the system. Rotation matrix V describes the effect of each RF pulse as a rotation of M about the y axis of an angle θ. Rotation matrix W instead introduces the drift ϕ of the magnetization in the xy plane caused by a field deviation ΔB which is assumed as constant during T (See Figure 1). This model represents a discretization of BE with sampling time equal to T, the inter-pulse period, therefore all the oscillations of M occurring during each T are neglected. With this new Discrete time, Nonlinear Model (DNLM) the output $M_y(k)$ converges to a proper steady state value, making linearization an easier task. In addition, a single input (ΔB) is sufficient since the effect of the lock sequence is embedded in the model. Linearization of DNLM around $\overline{\Delta B} = 0$ provides a discrete time transfer function from $\Delta B(k)$ to $M_y(k)$ given by

$$G(z) = \frac{M_y(z)}{\Delta B(z)} = \frac{b_2}{z - e^{\frac{-T}{T2}}}$$

$$b_2 = \gamma T \frac{-e^{\frac{-T}{T2}}\sin(\theta)M_0(1 - e^{\frac{-T}{T1}})}{1 - \cos(\theta)(e^{\frac{-T}{T1}} + e^{\frac{-T}{T2}}) + e^{\frac{-T}{T1}}e^{\frac{-T}{T2}}} \quad (5)$$

where all the quantities in b_2 are known. In the following we will refer to this model as LM.

3.1 Validation

For the purpose of validation, a set of NMR experiments is carried out relying on a permanent magnet providing a stable field of 500 $Gauss$. The lock sequence is applied to different samples with different values of T and θ (for a detailed description of the trials refer to Tables 3 and 4). For gain evaluation, the response to a step field deviation $\Delta B = -0.235\,Gauss$ is concerned and the predicted steady state values of M_y, ($\hat{SS}_{DNLM}$ and $\hat{SS}_{LM}$), are compared to the real ones (SS). Results are shown in Table 1. A comparison of real settling times (ST) to DNLM and LM estimated ones ($\hat{ST}_{DNLM}$ and $\hat{ST}_{LM}$) is shown in Table 1 as well.

When comparing the results obtained from DNLM and LM to the data collected from real NMR experiments these considerations arise:

Table 1. Comparison of model-predicted steady state values of M_y to real ones after the application of a step field disturbance.

Trial	$\hat{SS}_{LM}$ [mV]	$\hat{SS}_{DNLM}$ [mV]	SS [mV]	$\hat{ST}_{LM}$ [s]	$\hat{ST}_{DNLM}$ [s]	ST [s]
1.1	700	32	1.2	0.15	0.22	0.15
1.2	400	52	4	0.15	0.22	0.20
1.3	700	25	2.2	0.17	0.25	0.25
2.1	450	30	1.6	0.01	0.03	0.03
2.2	300	51	2.5	0.01	0.03	0.03
2.3	530	28	2.5	0.012	0.03	0.03

- the time constant of DNLM represents a good approximation of that of the real system;
- the time constant of LM represents an acceptable approximation of that of the real system, however it is slightly faster than both DNLM and the real data;
- the gain of both DNLM and LM heavily overestimates the gain of the real system.

The explanation for this error can be found in the field inhomogeneity, which is not taken into account by any of the previous models. They are in fact macroscopic models, intended to describe the evolution of the bulk magnetization vector, without considering the evolution of the small components which sum up into the M vector itself. In presence of a non-homogeneous field, the curve in Figure 3 shows a reduction of the slope in the linear region and a widening of the latter. The settling time instead is not affected by the non-homogeneity of the magnetic field. This phenomenon is consistent with the collected data. Since our goal is to obtain a linear model to drive the synthesis of the regulator, and since LM provides a good estimation of the process time constant, a possibility is to correct its gain. LM can be written as

$$G(z) = \frac{M_y(z)}{\Delta B(z)} = \frac{g(1 - e^{\frac{-T}{T2}})}{z - e^{\frac{-T}{T2}}}$$

with its static gain g estimated as

$$g = \frac{\overline{M_y^{ss}}}{\overline{\Delta B}} \quad (6)$$

where $\overline{\Delta B}$ is a known disturbance lying in the linear region of the curve in Figure 3 and $\overline{M_y^{ss}}$ is the steady state value of the transverse magnetization as a consequence of the application of $\overline{\Delta B}$.

The gain g can be obtained in two ways:

- modifying the existing nonlinear model to keep into consideration the missing phenomena and running a simulation to provide a gain estimation;
- developing a brand new model which allows to estimate the gain from a set of parameters via Least Square (LS) identification.

Both approaches will be investigated in the next sections. A similar problem appeared in Samra (2008), where the gain was modified by performing a normalization with respect to the maximum measured value of M_y. Still, in that case, the experiment was setup in a friendly environment, where the field disturbance was generated by a dedicated coil. In our case that approach may not be applied as all preliminary measures would be directly performed with the noisy magnet. Therefore,

we would like to reduce the number of measures which must be performed in open-loop.

4. GAIN CORRECTION STRATEGIES

4.1 Bloch-based Isochromat Model

When the main magnetic field B_0 is non-homogeneous the shape of the absorption spectrum is wider and shorter than the ideal case. This means that more frequencies around the Larmor one are involved in the resonance process. Each of these frequencies can be associated to a group of spins, called isochromat, which sees the same field value along the z axis. To properly study this phenomena, Hahn suggests in his paper (Hahn (1950)) to keep into account the separate behaviour of isochromats and then sum their contributions properly weighted. The same approach is adopted in Carr (1958); Patz (1988); Gyngell (1989); Bagueira de Vasconcelos Azeredo et al. (2000). In the following we will refer to this model as Bloch-based Isochromat Model (BBIM). The idea is to run BE for each isochromat i, precessing at speed ω_i, to compute its contribution $m_i(t)$. Then $M(t)$ is obtained summing all individual contributions $m_i(t)$ with proper weights a_i. So one can write

$$M(t) = \sum_i a_i m_i(t) \qquad (7)$$

The Lorentzian shape, centred at frequency ω_0, can be written as

$$S(\omega) = \frac{T2^*}{1 + (T2^*(\omega - \omega_0))^2} \qquad (8)$$

If an interval $[-\overline{\omega}; \overline{\omega}]$ is considered, $S(\omega)$ can be normalized as follows

$$Sn(\omega) = \frac{S(\omega)}{\sum_{-\overline{\omega}}^{\overline{\omega}} S(\omega)} \qquad (9)$$

therefore we can define

$$a_i = Sn(\omega_i) \qquad (10)$$

In order to validate BBIM, a set of trials with different samples and different experimental parameters is carried out. As for LM validation, trials are performed with the permanent magnet. For comparison, a field deviation of $\Delta B = -0.235\,Gauss$ is considered since it lies well within the linear region. Real data are filtered to remove high frequency noise. The model predicted values are obtained as the average of the steady state behaviour of M_y. The predicted steady state values of M_y ($\hat{SS}_{BBIM}$) and the measured ones (SS) are compared in Table 2. The static gain is now much closer to that of real experiments. The static gain for LM, g, can then be obtained running a simulation of BBIM, according to (6). An example of BBIM simulation is shown in Figure 4, the corresponding real data in Figure 5.

4.2 Constrained Least Squares Model

A second approach consists in estimating g using LS optimization starting from the set of collected data. Notice that one can write g as

$$g = \frac{\overline{M_y^{ss}}}{\Delta B} = -\gamma y \qquad (11)$$

with

$$y = \frac{\overline{M_y^{ss}}}{\Delta \omega} \qquad (12)$$

where

$$\overline{\Delta \omega} = -\gamma \overline{\Delta B} \qquad (13)$$

Let Y be the vector containing the measured y for each trial and let Φ be the matrix of regressors

$$Y = \begin{bmatrix} y_1 \\ y_2 \\ \dots \end{bmatrix} \quad \Phi = \begin{bmatrix} \varphi_1 \\ \varphi_2 \\ \dots \end{bmatrix}$$

with

$$\varphi_i = [T1_i \; T2_i \; T2_i^* \; T_i \; \theta_i \; M_{0_i}]$$

Then we can setup a Constrained LS (CLS) identification problem as

$$\hat{\beta}^{CLS} = min_{\beta,p}(Y - \Phi\beta)'(Y - \Phi\beta)$$
$$subject\,to : min_p\, p\beta > 0$$
$$\beta(3), \beta(5) > 0 \qquad (14)$$
$$\forall \underline{\Phi} < p < \overline{\Phi}$$

where $\underline{\Phi}$ and $\overline{\Phi}$ are the lower and upper bounds for the matrix of regressors (each regressor is supposed to lie within these bounds, which must be chosen according to the experimental setup).

Notice that we use constraints to force y to be positive and to increase whenever $T2^*$ or θ increase (a standard LS approach does not ensure that these points are satisfied a priori). The resulting optimization problem is a quadratic program which is solved with the Matlab optimization tool Yalmip (Löfberg (2012)). With real data shown in Table 2, related to the trials reported in Tables 3 and 4, the optimal value for β is given by
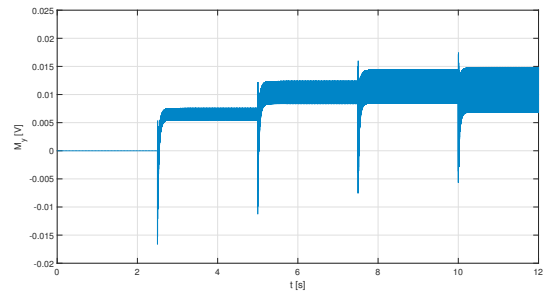

Fig. 4. BBIM simulation for Trial 1.2. M_y as a response to a series of steps of $\Delta B = -0.235\,Gauss$ each one. For gain evaluation only the first step is considered.
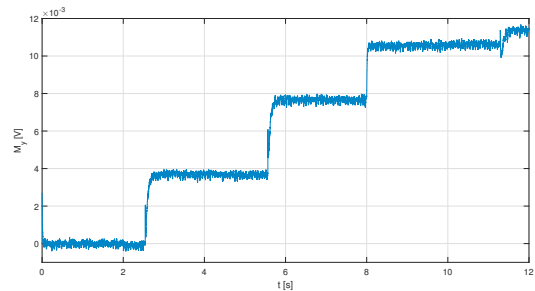

Fig. 5. Collected data for Trial 1.2. M_y as a response to a series of steps of $\Delta B = -0.235\,Gauss$ each one. For gain evaluation only the first step is considered.

Table 2. Comparison of $\hat{S}S_{BBIM}$ and $\hat{S}S_{CLS}$ to real SS after the application of a step field disturbance.

Trial	$\hat{S}S_{BBIM}$ [mV]	$\hat{S}S_{CLS}$ [mV]	SS [mV]
1.1	3.5	2.3	1.2
1.2	6	3.5	4
1.3	3	4.1	2.2
1.4	12	3.6	3
2.1	2.5	2.3	1.6
2.2	4	3.5	2.5
2.3	2	4	2.5
2.4	3	3.3	3
3.1	6	2.5	3
3.2	9	3.7	6
3.3	4.5	4.2	3
4.1	3	5.1	3
4.2	6	6.3	8
4.3	1	6.8	5
5.1	3.5	3.4	3
5.2	9	4.6	6
5.3	3	5.1	5

$$\hat{\beta}^{CLS} = \begin{bmatrix} 0.000000068046973176 \\ 0.000000083235109192 \\ 0.000550233592575485 \\ 0.000869743805386847 \\ 0.000002707415573927 \\ 0.000000216962856730 \end{bmatrix} \tag{15}$$

and the predicted steady state values of M_y, ($\hat{S}S_{CLS}$), are reported in Table 2.

5. SYNTHESIS OF THE REGULATOR

The methodology introduced in the following allows to synthesize a PID regulator in a parameterized way, thus allowing to obtain the desired loop functions independently on the magnet and on the NMR sample used for the lock experiment. Notice that in Samra (2008) a correction coil is used as actuator for the control action. The latter should be carefully engineered to generate a spatially uniform correction. Moreover, some space is required to properly place the correction coils. A PI controller was used to compensate for the NMR time constant only, since no settling time requirement was considered. In our case instead the control action will result in a current directly injected into the main magnet. This will allow to save space and to preserve field homogeneity. Our approach will require to properly model the behaviour of the magnet and to use a PID regulator to compensate for both the magnet and the NMR time constants, allowing us to achieve the desired settling time. The scheme in Figure 6 shows the overall closed-loop setup for the FFL in terms of transfer functions. In particular C is a known conductance, while

$$G_{mag}(s) = \frac{\mu_{mag}}{1 + s\tau_{mag}} \tag{16}$$

is the transfer function of the magnet generating B_0,

$$G(s) = \frac{\mu_{nmr}}{1 + s\tau_{nmr}} \tag{17}$$

is the continuous-time version of equation (5) with a correct gain.

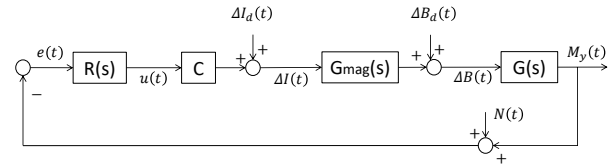$$R(s) = \frac{\mu_r(1 + s\tau_{z1})(1 + s\tau_{z2})}{s(1 + s\tau_p)} \tag{18}$$

Fig. 6. Block scheme with transfer function for the FFL. $u(t)$ is the control action, $\Delta I(t)$ is the overall current deviation from the nominal one; it is the sum of the current from the regulator and the current disturbance $\Delta I_d(t)$; $\Delta B(t)$ is the overall field deviation seen by the NMR sample; it is the sum of the field from the magnet and the field disturbance $\Delta B_d(t)$; $M_y(t)$ is the y component of the transverse magnetization in the rotating frame; $N(t)$ is measurement noise; $e(t)$ is the error signal feeding the regulator.

is a PID controller in the realizable form.

The closed-loop lock system should provide:

- stability of the closed-loop system;
- perfect tracking of a step reference at steady state
- settling time $T_{sett} < 10\,ms$;
- rejection of current/field oscillations;

The overall process transfer function is then given by

$$P(s) = G(s)G_{mag}(s)C = \frac{\mu}{(1 + s\tau_{nmr})(1 + s\tau_{mag})} \tag{19}$$

Since both poles in $P(s)$ have negative real part, it is possible to use the two zeros of the PID (see Equation (18)) to cancel them. This means setting

$$\tau_{z1} = \tau_{mag}$$
$$\tau_{z2} = \tau_{nmr}$$

τ_p is placed out of the desired bandwidth and works as a filter for SSFP oscillations of M_y (see Carr (1958)), which act as measurement noise in this framework; at this point the closed loop bandwidth depends on the gain only. To keep the synthesis parametric one can set

$$\mu_r = \frac{1}{\mu} b_w$$

where b_w represents the desired closed-loop bandwidth expressed in rad/s. A careful choice of b_w and τ_p allows to provide the required disturbance rejection and settling time, while obtaining a sufficient phase margin to achieve robust stability.

To verify the correctness of the design of the PID regulator a set of simulations is performed over a case-study sample ($T1 = 0.007\,s$, $T2 = 0.002\,s$, $M_0 = 0.29\,V$, resulting in $\tau_{NMR} = 1.66\,ms$). Notice that we simulate a superconductive magnet, whose dynamic is very slow ($\tau_{mag} = 1.49\,s$) when compared to the rest of the system. As a consequence its behaviour can be considered in practice as the one of an integrator. The homogeneity of the field generated by the magnet results in $T2^* = 0.00042\,s$. Furthermore, a saturation is introduced at the output of the regulator to model the presence of the DAC working on an interval of $[-3V; 3V]$. The NMR process is modelled with BBIM to keep the simulation as close as possible to the real experiment. In particular, a limit-case situation is described. Figure 7 shows the response of the system to an initial condition of $\Delta B_d = -0.0235\,Gauss$. The closed loop simulation shows that the disturbance is correctly compensated and the $M_y(t)$ signal is brought to zero. The static precision requirement is fulfilled. The settling time requirement is achieved ($T_{sett} = 10\,ms$) even
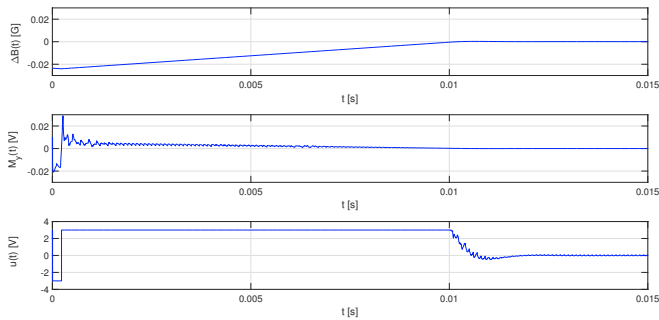
Fig. 7. Closed-loop simulation. $\Delta B(t)$ is the overall field deviation seen by the NMR sample; $M_y(t)$ is the y component of the transverse magnetization in the rotating frame; $u(t)$ is the control action.

if the saturation of the control action $u(t)$ is active. Still, if $|\Delta B_d|$ assumes a higher value, the regulator may not be able to ensure the maximum settling time because of saturation.

6. CONCLUSION

This paper aims to obtain a linearised model to properly describe the behaviour of an NMR lock sample in SSFP regime. Along with a model of the magnet which generates B_0, this is used to synthesize a parametrized PID regulator which should bring the magnetic field to its desired value in a given time interval. The parametrized approach allows to obtain these goals independently on the magnet and on the NMR sample chosen for the experiment. Closed loop simulations highlight the correctness of the approach but call for a detailed analysis of the disturbances action on the plant to properly scale the control action as a current. In addition, a suitable field sensor is required to open the loop when the NMR sensor is brought out of its linear region.

Table 3. Details of lock sequences used for NMR trials.

Sample	Trial	T [μs]	θ [deg]
1	1.1	100	4
1	1.2	100	8
1	1.3	200	8
1	1.4	200	8
2	2.1	100	4
2	2.2	100	8
2	2.3	200	8
2	2.4	200	8
3	3.1	100	4
3	3.2	100	8
3	3.3	200	8
4	4.1	100	5
4	4.2	100	9
4	4.3	200	9
5	5.1	100	4
5	5.2	100	8
5	5.3	200	8

Table 4. Details of samples used for NMR trials.

Sample	γ [$rad/(s*Gauss)$]	$T1$ [s]	$T2$ [s]	$T2^*$ [s]	M_0 [V]
1	26751.3	0.06	0.02	0.000055	0.28
2	26751.3	0.007	0.002	0.000042	0.29
3	26751.3	0.18	0.056	0.000075	0.28
4	26751.3	2.37	2.14	0.000030	0.59
5	25166.2	1	0.8	0.000050	0.45

REFERENCES

Bagueira de Vasconcelos Azeredo, R., Colnago, L.A., and Engelsberg, M. (2000). Quantitative analysis using steady-state free precession nuclear magnetic resonance. *Analytical chemistry*, 72(11), 2401–2405.

Bloch, F. (1946). Nuclear induction. *Physical review*, 70(7-8), 460.

Carr, H. (1958). Steady-state free precession in nuclear magnetic resonance. *Physical Review*, 112(5), 1693.

Gyngell, M.L. (1989). The steady-state signals in short-repetition-time sequences. *Journal of Magnetic Resonance*, 81(3), 474–483.

Hahn, E.L. (1950). Spin echoes. *Physical review*, 80(4), 580.

Hoult, D., Richards, R., and Styles, P. (1978). A novel field-frequency lock for a superconducting spectrometer. *Journal of Magnetic Resonance*, 30(2), 351–365.

Jiang, D., Chen, H., Chen, Z., and Zheng, Z. (2010). The digital field-frequency lock system of high-resolution nmr spectrometer. In *Electrical and Control Engineering (ICECE), 2010 International Conference on*, 2328–2331. IEEE.

Kan, S., Gonord, P., Fan, M., Sauzade, M., and Courtieu, J. (1978). Automatic nmr field-frequency lock - pulsed phase locked loop approach. *Review of Scientific Instruments*, 49(6), 785–789.

Keeler, J. (2011). *Understanding NMR spectroscopy*. John Wiley & Sons.

Li, M., Schiano, J.L., Samra, J.E., Shetty, K.K., and Brey, W.W. (2011). Reduction of magnetic field fluctuations in powered magnets for nmr using inductive measurements and sampled-data feedback control. *Journal of Magnetic Resonance*, 212(2), 254–264.

Löfberg, J. (2012). Automatic robust convex programming. *Optimization methods and software*, 27(1), 115–129.

Maly, T., Bryant, J., Ruben, D., and Griffin, R.G. (2006). A field-sweep/field-lock system for superconducting magnetsapplication to high-field epr. *Journal of Magnetic Resonance*, 183(2), 303–307.

Moraesa, T.B. and Colnago, L.A. (2014). Simulação de sinais de rmn através das equações de bloch. *Quim. Nova*, 37(8), 1410–1416.

Patz, S. (1988). Some factors that influence the steady state in steady-state free precession. *Magnetic resonance imaging*, 6(4), 405–413.

Samra, J. (2008). *A field-frequency lock implemented with a sampled-data feedback control algorithm derived from a small-signal NMR model*. Ph.D. thesis, The Pennsylvania State University.

Schett, O. and Schwilch, A. (1994). Method and device for compensating of field disturbances in magnetic fields. US Patent 5,302,899.

Schiano, J., Magin, R.L., and Wright, S. (1991). Feedback control of the nuclear magnetization state: Modeling and control design. *IEEE transactions on medical imaging*, 10(2), 138–147.

Yanagisawa, Y., Nakagome, H., Hosono, M., Hamada, M., Kiyoshi, T., Hobo, F., Takahashi, M., Yamazaki, T., and Maeda, H. (2008). Towards beyond-1ghz solution nmr: Internal 2 h lock operation in an external current mode. *Journal of Magnetic Resonance*, 192(2), 329–337.