ANALELE UNIVERSITATII BUCUREŞTI

Department of Physical Chemistry
4-12 Regina Elisabeta Blvd, District 3, Bucharest
phone: +40-21-3143508; fax: +40-21-3159249
ISSN: 1844-0401

ARS DOCENDI PUBLISHING HOUSE
Sos. Panduri 90, District 5 Bucharest
phone/fax: +40-21-4102575
e-mail: arsdocendi@yahoo.com

A NEW DESIGN FOR THE CRUCIAL EXPERIMENT INSTALLATION RELATED TO MAGNETIC SUSCEPTIBILITY DETERMINATION

A. Soare and Cristina Mandravel *

abstract: This paper presents a new design for a crucial experiment installation related to magnetic susceptibility determination. It was realized in 2003 after finishing the consolidation of the Faculty of Chemistry building, 4-12 Regina Elisabeta Bd., Bucharest.

key words: magnetic field, magnetic susceptibility determination, Faraday method

received: November 09, 2008 *accepted*: November 27, 2008

Introduction

Introduced in a magnetic field H, substances are diamagnetic or paramagnetic. Its behavior is described by the general equation:

$$J=\chi H \tag{1}$$

where: J – formed magnetic moment; χ – magnetic susceptibility; H – magnetic field.

For diamagnetic substances χ is comprised between -10^{-5} and -10^{-6}. In this category can be included all organic substances and non–metals with the exception of oxygen and sulphur [1].

For paramagnetic substances χ values are comprised between 10^{-5} and 10^{-6} and are dependent of temperature as P. Langevin established in 1905. In this category are included metals, organic radicals, oxygen and sulphur.

For ferromagnetic substances χ values are about 10^{-3}. In this category are included the metals from Fe group.

Hence it results that the magnetic susceptibility determination represents a crucial experiment among existent various methods established with this purpose in view. In laboratory of Physical Chemistry Department of Bucharest University in the seventies was realized a such installation based on very known Faraday method [2]. The principle scheme

* Department of Physical-Chemistry, Faculty oh Chemistry, University of Bucharest, 4-12 Bd. Regina Elisabeta, 030018, Bucharest, Romania, tel.: +40213143508*285, *corresponding authors e-mail*: aurel_soare@yahoo.co.uk, chrism@gw-chimie.math.unibuc.ro

of this Faraday method is shown in Fig. 1. It contains two magnets (1), which create a homogeneous magnetic field to z direction wherefore dH/dz = constant, and a torsional balance (3) with quartz rod (2). Onto the balance pan attached to the quartz rod the sample of substance is add and onto the other pan the weights are add. About 10 mg of analysis substance is weight in default of magnetic field. If a magnetic field is applied, we will observe a lost of the balance equilibrium: a paramagnetic or a ferromagnetic substance will apparetly be weightier and a diamagnetic substance will apparently be lighter. Thus we may deduce the type of the susceptibility [2].

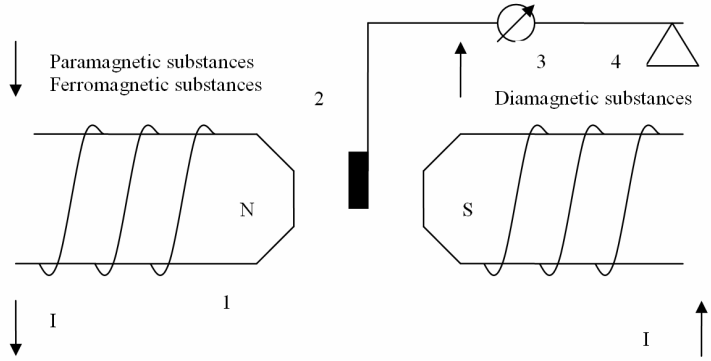

Fig. 1 *Principle scheme of Faraday method for magnetic susceptibility determination.*

This paper relates the changes of a hand-made old ins tallation after the consolidation of the Faculty of Chemistry building, executted between 1998 and 2002, including also the ″Methods for structural and molecular interactions study″ laboratory, from componence of Physical Chemistry Department, located on second floor (room E-215).

We must mention that the first hand-made installation for magnetic susceptibility determination, developed by lecturer dr. V. Mincu [1], was very voluminous. Half of this installation was placed in a created space into the laboratory wall (niche) and the other half was placed on an adiacent table with 1,5 x 1,5 m dimension.

Experimental part

Premiss for the new design of the installation:

- it was designed a protection for magnet which has not been moved during of the consolidation.
- it was plotted a poster containing the electrical schema and the operation mode of the installation before of demounting.
- because the consolidation of the central building of university, a hystorical monument, have been developed as a ″building inside the external walls″ we asked to constructor to keep and extend the intraspace into the wall (the niche). The final dimensions of the niche are: 2,1 m height, 1,2 m width and 1,1 m depth. So, after the consolidation of the

building all the installation for susceptibility determination have been placed into this niche.

For the rest of the laboratory, after consolidation, the work space is smaller than before, but the space for susceptibility determination increased!

The general view of the new installation is shown in Fig. 2. and general electric schema is shown in Fig. 3.

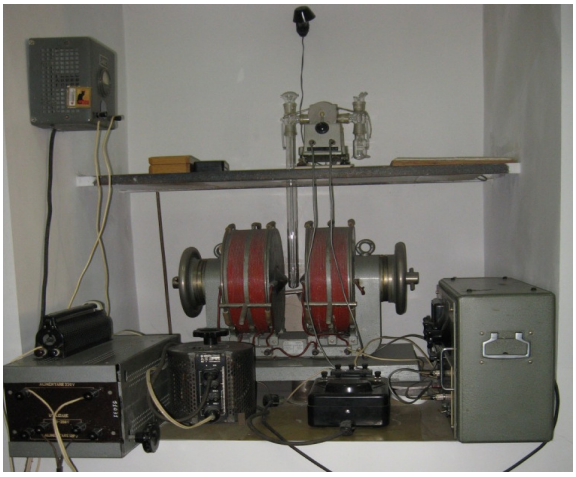

Fig. 2 *General view of new designed installation.*

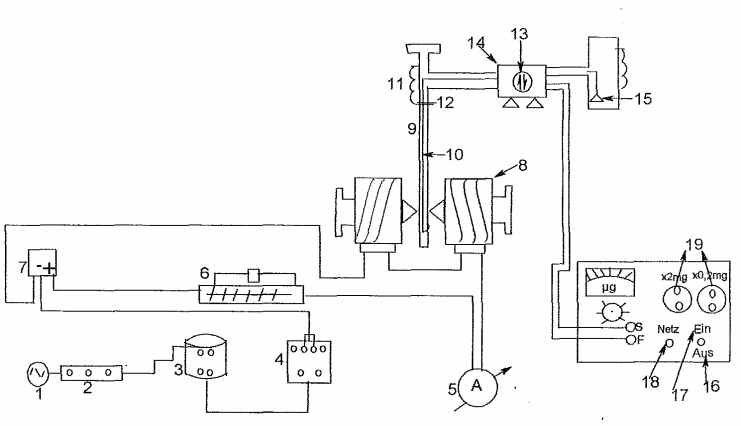

Fig. 3 *Electrical schema of new realized installation. Where:*
1- alternative currrent source; 2-electrical extension; 3-adjustable auto-transformer ATR1; 4-stabilizer; 5-ampermetre; 6-rheostat; 7-console; 8-electromagnet with two reels; 9-quartz tube; 10-quartz rod; 11-spring; 12-schliff of the quartz tube; 13-drummer for fine adjustment; 14-Gouy balance; 15-balance plate; 16-Sartorius balance; 17-starter; 18-control electric bulb; 19-compensator.

When the installation was reassembled all the electrical devices were verified and all the electrical contacts were cleaned.

The electromagnet is made of two conical polar pieces. The distance between the two conical pieces may be varied with an accuracy of ± 0,05 mm. The magnetic field is yield with the aid of four copper reels grouped in pairs. These reels which are serial interlinked have a resistance of 7 Ω and the terminals voltage is 125 V for a maximum current of 18 A. During the earthquake from 1977, a part of the spool (reel) wire of the magnet was compromised and maximum amperage suported now by the installation can be increased only to 8A.

For the calibration of the installation we used as a standard substance, $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ (Mohr salt) and for checking we used $K_3[Fe(CN)_6]$, Merk, p.a.

Results and discussion

The assamblage of the installation was the first part of the dissertation practical work [3], entitled ″Achievement of a resurgent experiment installation for magnetic susceptibility determination based on Faraday method″ for obtainning master degree in Applied Physical Chemistry and Radiochemistry.

The second part of the above named dissertation was planned to the elaboration of the work instructions.

Work Instructions:

I. Preliminary operations:

1. the assurance of the Gouy balance (14) rightness with the aid of the four screwes such as the air bubble of the balance level can be centered.
2. washing and cleaning of the quartz vial with distilled water and filter paper.
3. the attachment of the vial to the quartz rod (10) of the Gouy balance between the two magnets (8);
4. insulating of the vial from ambience by attachment of the quartz tube (9), fitted with schliff (12) and grip, to the lever of Gouy balance with the aid of a spring (11).
5. coupling of the installation to the alternative current source (1);
6. Sartorius balance starting by switching the starter button (17) from ¨EIN″ to ″AUS″ position; if the bulb ″NETZ″ is turn on it comes to that Sartorius balance works.
7. Gouy balance equilibrating, first by placing of a counterweight on the balance plate (15) and than by fine adjustment of the drummer (13). When Gouy balance is equilibrated, the indicator needle of the Sartorius balance scale must indicate 0 value.
8. Sartorius balance equilibrating: first the balance must be started. After a few minutes of waiting the ″Faktor″ button is fixed on 200 division; the indicator needle of the balance scale must indicate 10 value, if not, we have to put on or to take up small weights (it is recommended to use tinfoil instead of paper because it does not absorb humidity) until the needle indicate 10 value. The same operations are done by switching the ″Faktor″ button to 100, 10 and 5 divisions. Than the ″Faktor″ button is switched to 2 division and the indicator needle is fixed to 0 value with the aid of the drummer for fine adjustment (13) of the Gouy balance.

II. Work operations:

1. the quartz vial is empty.
2. it is increase the voltage of the auto-transformer ATR1 (3) resulting an increment of the magnetic field intensity which is proportionate with the intensity of the electric current.
3. it is read the current intensity values on the apermetre (5).
4. simultaneous with the increment of the voltage, the indicator needle of the Sartorius balance vary on the scale. It is fixed to 0 value with the aid of the two compensators (19). Thus the mass variation of the empty vial in magnetic field (Δm_f), when the current intensity increases, is measured. If the indicator needle is out of scale it is returned with the aid of the compensator (19).
5. the steps 2-4 are repeated with quartz vial filled with the analysis substance. Thus the mass variation of the vial filled with analysis sample in magnetic field (Δm_{f+s}) when the current intensity increases, is measured.

For the experiments related with paramagnetic susceptibility dependence on temperature, the quartz tube (9) is substituted with a double walls tube for circulation of refrigerent liquid. The routine determinations described above have to begin only after the achievement of the equilibrium state.

The improvement of this experiment type in future may be realized by the encloser of the niche with a ″termopan″ glass.

The third part of the master dissertation was planned to **the calibration of the installation**.

Step 1: The mass variation measurement of the empty vial (Δm_f) in magnetic field.

Table 1 The mass variation of the empty vial in magnetic field with the current intensity.

I (A)	Δm_f (μg)
1	59
1.5	139
2	236.5
2.5	339
3	454
3.5	569
4	716.5
4.5	824
5	939
5.5	1065
6	1174
6.5	1298
7	1419
7.5	1483
8	1578

Step 2: The mass variation measurement of the vial filled with standard substance (Δm_{f+st})in magnetic field and the calculation of H (dH/dz) factor.

Table 2 The mass variation of the vial filled with standard substance (Δm_{f+st}) in magnetic field and variation of H (dH/dz) factor with the current intensity

I (A)	Δm_f (μg)	Δm_{f+st} (μg)	Δm_{st} (μg)	$H\frac{\partial H}{\partial z}$ x 10^2
1	59	81	22	7.77
1.5	139	187	48	16.95
2	236.5	303	66.5	23.49
2.5	339	461	122	43.09
3	454	636	182	64.28
3.5	569	815	246	86.88
4	716.5	978	261.5	92.36
4.5	824	1187	363	128.21
5	939	1374	435	153.64
5.5	1065	1587	522	184.37
6	1174	1782	608	214.75
6.5	1298	2002	693	244.77
7	1419	2112	704	248.66
7.5	1483	2235	752	265.61
8	1578	2342	764	269.85

Standard substance is $Fe(NH_4)_2(SO_4)_2{\cdot}6H_2O$ with magnetic susceptibility χ=32,3x10^{-6} [1,7].

H (dH/dz) factor is calculated with equation:

$$H\frac{\partial H}{\partial z}=\frac{\Delta m_{st}\cdot g}{m_{st}\cdot \chi} \tag{2}$$

where: m_{st} = 8,59 · 10^{-3} g - the wheight of the standard substance; g = 9,8 m/s^2 - gravitational acceleration; $\Delta m_{st} = \Delta m_{f+st} - \Delta m_f$ - is the mass variation of the standard substance.

Step 3: Indirect checking of the balance calibration.

Used substance is $K_3[Fe(CN)_6]$ with magnetic susceptibility χ = 8,1 x 10^{-6} [1,7].

The magnetic susceptibility is calculated with the equation:

$$\chi=\frac{\Delta m_{sx}\cdot g}{m_{sx}\cdot H\frac{\partial H}{\partial z}} \tag{3}$$

where: m_{sx} = 49,24 · 10^{-3} g is the wheight of $K_3[Fe(CN)_6]$; g = 9,8 m/s^2 is gravitational acceleration; $\Delta m_{sx} = \Delta m_{f+sx} - \Delta m_f$ is the mass variation of the checking substance.

After the mass variation measurement of the checking substance $K_3[Fe(CN)_6]$, we must verify R ratio:

$$R=\frac{\chi_{sx}}{\chi_{st}}=\frac{8,1\cdot 10^{-6}}{32,3\cdot 10^{-6}}=0,250 \tag{4}$$

where: χ_{st} is the magnetic susceptibility of the standard substance $Fe(NH_4)_2(SO_4)_2{\cdot}6H_2O$; χ_{sx} is the magnetic susceptibility of the substance $K_3[Fe(CN)_6]$.

Table 3 The magnetic susceptibility variation and the mass variation of the vial filled with $K_3[Fe(CN)_6]$ (Δm_{f+sx}) in magnetic field with the current intensity

I (A)	Δm_{f+sx} (μg)	Δm_{sx} (μg)	$H\frac{\partial H}{\partial z}$ x 10^2	χ x 10^{-6}
1	111.5	52.5	7.77	13.44
1.5	239.5	100.5	16.95	11.80
2	341	104.5	23.49	8.85
2.5	526	187	43.09	8.64
3	725.5	271.5	64.28	8.41
3.5	986.5	417.5	86.88	9.55
4	1163	446	92.36	9.62
4.5	1394	570	128.21	8.85
5	1598	659	153.64	8.54
5.5	1831	766	184.37	8.27
6	2023	849	214.75	7.87
6.5	2295	997	244.77	7.98
7	2554	1135	248.66	9.23
7.5	2675	1140	265.61	8.93
8	2718	1192	269.85	8.41

From eqns. (3) and (4) results:

$$R = \frac{\Delta m_{sx}}{\Delta m_{st}} \frac{m_{st}}{m_{sx}} \tag{5}$$

where: $m_{sx} = 49{,}24 \cdot 10^{-3}$ g is the mass of the substance $K_3[Fe(CN)_6]$; $m_{st} = 8{,}59 \cdot 10^{-3}$ g is the mass of the standard substance $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$; Δm_{st} - is the mass variation of the standard substance $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$; Δm_{sx} - is the mass variation of the checking substance $K_3[Fe(CN)_6]$.

Table 4 *R* ratio values corresponding to different current intensities (*I*)

I (A)	Δm_{sx} (μg)	Δm_{st} (μg)	R
1	52.5	22	0.416
1.5	100.5	48	0.365
2	104.5	66.5	0.274
2.5	187	122	0.267
3	271.5	182	0.260
3.5	417.5	246	0.296
4	446	261.5	0.298
4.5	570	363	0.274
5	659	435	0.264
5.5	766	522	0.256
6	849	608	0.244
6.5	997	693	0.247
7	1135	704	0.286
7.5	1140	752	0.276
8	1192	764	0.260

Excepting the first two values of the current intensity, R values are quite close to 0,250 value, hence the experiment installation works and it is calibrated.

Determinations of type and values of magnetic susceptibility are very important characteristics of the new substances and composite materials. This type of determination can be also used for the study of the corrosion products nature [4,5] closer by modern methods as are X-ray diffraction (XRD), electron diffraction (ED), FTIR spectroscopy and Mossbauer spectroscopy [6] which are rapid but very expensive methods.

Next improvement of this installation is the interfacing with a computer for the automate obtaining of the results.

Conclusions

A new design for the crucial experiment installation concerning magnetic susceptibility determination was realized.

Work instructions were elaborated and the calibration of the new designed installation was realized.

Magnetic susceptibility determination can be used for the characterization of the corrosion products.

REFERENCES

1. Mincu, V. (1986) *Ph.D Thesis*, University of Bucharest, Bucharest.
2. Chiosa, V., Stănculescu, I. and Mandravel, C. (2006) **Structură moleculară**, Ed. Univ. Bucuresti, 134.
3. Soare, A. (2003) *Master Dissertation*, University of Bucharest, Bucharest.
4. Soare, A. and Mandravel, C. (2005) *Analele Universităţii Bucureşti. Chimie*, anul XIV vol. **2**, 352-60.
5. Soare, A., Chiosa, V. and Mandravel, C. (2008) *Rev. Chim* **59**(11), 1264-9.
6. Mandravel, C. and Chiosa, V. (2005) **Metode de studiu ale structurii moleculare**, Ed. Univ. Buc., 117-26.
7. Taraban, M.B., Leshina, T.V., Anderson, M.A. and Grissom, C.B. (1997) *J. Am. Chem. Soc*. **119**, 5768-9.