Original Research Article

DEVELOPMENT OF PARAMAGNETISM ANALYZER

Abstract:-

One of the quantities of fundamental importance in describing magnetic phenomena and materials is the magnetic moment. The research work focused on the development of a paramagnetic materials analyzer using locally sourced materials to determine magnetic moment of magnetic materials. The developed analyser involved the design of a sensing unit. The sensing unit is comprised of a colpit oscillator, preamplifier and shaping circuit, K – type thermocouple sensor, thermocouple amplifier, microcontroller, matrix keypad and a LCD. The designed colpit oscillator was used to determine the frequency change for each material considered. The output analogue signal from the oscillator was fed into the amplifier and shaping circuit. The shaped output signal from the shaping circuit was fed into the digital pins of the microcontroller which helps to measure the period of complete oscillations and hence the frequency. The K – type thermocouple sensor with a standard sensitivity of 41 µV/0C was used to determine the temperature of each paramagnetic material. The measured temperature was amplified and digitized using Max 6678 thermocouple amplifier with a resolution of 0.25 /0C and temperature range of – 200 0C to 700 0C. The matrix keypad was used to manually enter the mass and atomic mass number of magnetic samples. The developed instrument was calibrated using a known standard magnetic moment value of iron. The instrument was tested and it was able to determine the magnetic moment of available magnetic materials with a standard deviation of 0.0163±0.005. The value of magnetic moment obtained for the available known materials fell within the range of values obtained from literature. The maximum power consumption of the designed instrument is 3.85 watts.

Magnetic moment, Paramagnetic MAterial, Oscillator, Microcontroller, Temperature

1. INTRODUCTION

According to gauss's law of magnetism, there are no monopole sources of magnetic field. Since there is no magnetic charge, magnetic moment turns out to be a quantity of fundamental importance in describing magnetic phenomena and materials (Chiou and Williams, 2017). All substances possess magnetic properties and the ultimate source of their magnetism is the magnetic moment associated with their atom due to orbital motion and intrinsic spin. The magnitude of this magnetic moment is dependent on the species of atom. There are many ingenious and varied ways of realizing the measurement of magnetic moments (Foner, 1967, Bates, 1970). Most common methods for magnetic moment measurement are force method, induction method and indirect method. Foner (1956) was the first to describe an instrument for the measurement of magnetic moments; his design has become generic to all subsequent designs Hoon (1985). Hoon (1985) designed an instrument that can measure magnetic moment of magnetic materials. It had a robust nature and stability and one which offers great experimental flexibility. The instrument was calibrated against a known magnetic moment of an annealed high purity nickel. Pattnaik, (2014) also designed vibrating sample magnetometer, that can measure magnetic moment at room temperature. The design employed the principle of harmonic vibration of a magnetic sample in a magnetic field. The harmonicity was achieved by employing a colpitt oscillator containing sensing coils. The instrument was able to measure magnetic moments of both ferromagnetic and paramagnetic materials precisely and accurately; it was calibrated against Nickel. This paper is concerned with developing a paramgnetism analyzer using locally sourced material to determine magnetic moments of magnetic materials.

2. MATERIALS AND METHODS

Fig. 1 shows the block diagram of the developed instrument for measurement of magnetic moment. It comprises of the following (a) a Colpitt's oscillator that generates sinusoidal signal whose frequency increases or decreases when a magnetic sample poured inside a test tube is gradually inserted or withdrawn from the oscillator's coil; (b) a buffer amplifier, preamplifier and shaping circuit which amplifies the signal and shapes it into a square wave using CMOS Schmitt trigger NAND gate for accurate measurement of wave generated: (c) a thermocouple sensor which measures the temperature of the material sample and then links it to a thermocouple amplifier for amplification; (d) a microcontroller which forms the central processing unit for the whole system. It measures the period of complete oscillations; hence the frequency of magnetic samples measuring frequency, the temperature and also helps to convert all analogue signals to digital signal with the aid of analogue – to digital converter and sends output result to the display; and (e) a liquid crystal display which enables the user monitor the activity within the microcontroller and as well display the computed value of the magnetic moment. An automatic battery charger was incorporated to charge the 12 V battery and also act as a power source supplying 9 V to the entire system. The keypad was used to key in the mass and atomic mass number of each material sample considered during measurement. The activities within the microcontroller were controlled using an embedded C – program on arduino platform.

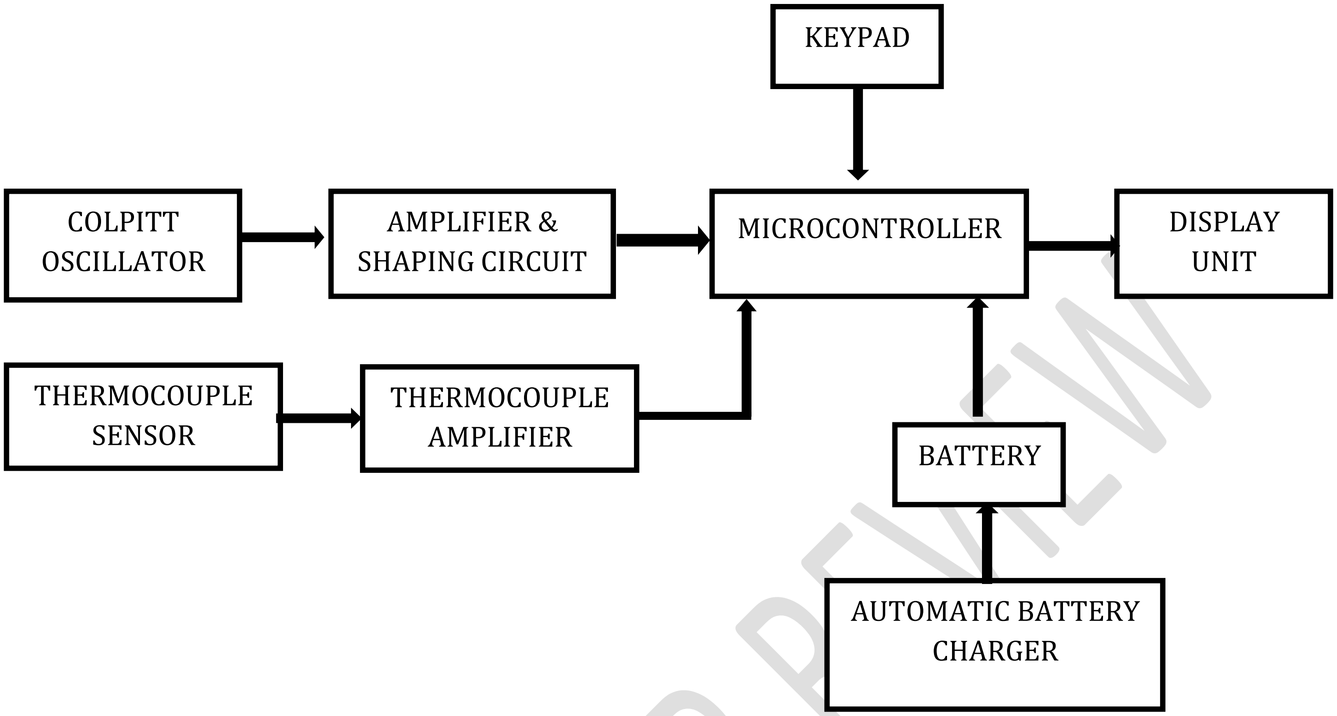


Figure 1 Complete Block Diagram of the Paramagnetism Analyser

a) The Oscillator Circuit

The emitter terminal of the transistor is effectively connected to the junction of the two capacitors, C1 and C2which are connected in series and act as a simple voltage divider. When the power supply is firstly applied,capacitors C1 and C2 charge up and then discharge through the coil L. The oscillations across the capacitors are applied to the base-emitter junction and appear in the amplified at the collector output.Resistors, R1 and R2 provide the usual stabilizing DC bias for the transistor in the normal manner while the additional capacitors act as a DC-blocking bypass capacitors. A radio-frequency choke (RFC) is used in thecollector circuit to provide a high reactance (ideally open circuit) at the frequency of oscillation, (fr) and a low resistance at DC to help start the oscillations. The required external phase shift is obtained from positive feedback obtained for sustained undamped oscillations. The amount of feedback is determined by the ratio of C1 and C2. These two capacitances are generally “ganged” together to provide aconstant amount of feedback so that as one is adjusted the other automatically follows.

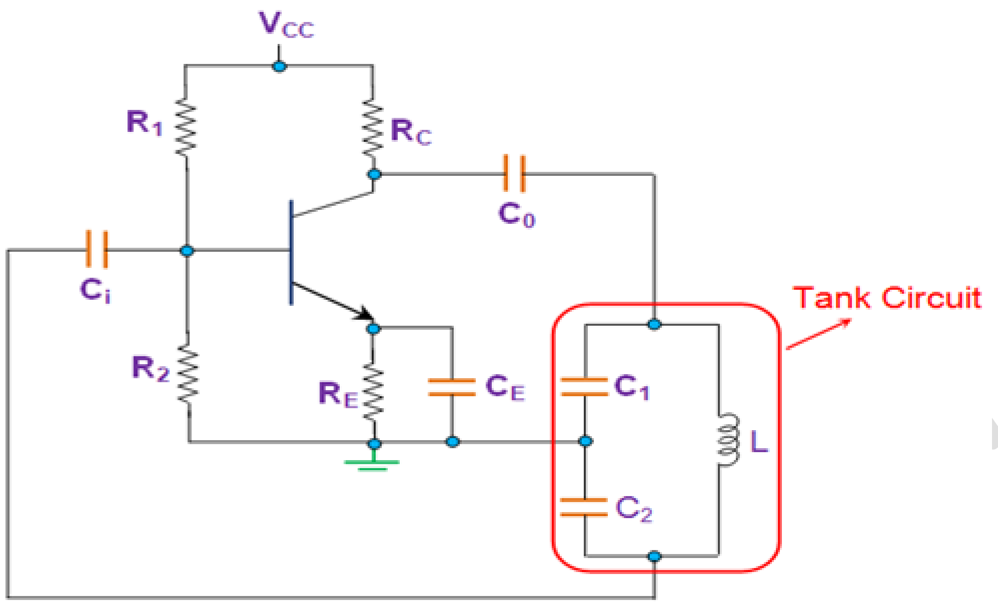


Figure 2 Colpitts Oscillator

In the design of the colpitts oscillator circuit, a single stage full biasing bipolar transistor amplifier (NPN) was used to produce a sinusoidal output. The capacitive voltage divider setup in the tank circuit works as the feedback source. The design Considerations for figure 2 are as follows:

V_B = 3.2 V, I_B = 0.87 mA,

Assuming $I_C \approx I_E$

$$V_E = 10\% V_{CC} \qquad (1)$$

The biasing resistors were obtained using the following expressions:

$$R_1 = \frac{V_{CC} - V_B}{I_B} \qquad (2)$$

$$R_2 = \frac{V_B R_1}{V_{CC} - V_B} \qquad (3)$$

$$R_E = \frac{V_B - V_{BE}}{I_E} \qquad (4)$$

$$R_B = R_1 // R_2 \qquad (5)$$

$$\text{Stability factor} = \frac{R_B + R_E}{R_E} = 10 \qquad (6)$$

The values of R_1, R_2, and R_E, are 10k, 3.7k, and 580 respectively.

The frequency of oscillations was obtained using:

$$f_r = \frac{1}{2\pi\sqrt{LC_T}} \qquad (7)$$

The frequency of interest is 2 MHz; the capacitors C_1 and C_2 were selected such that the gain is 10 knowing that:

$$Gain = \frac{C_2}{C_1} \tag{8}$$

The inductance of the inductor L was obtained using equation 1.9.

$$L = \frac{1}{4\pi^2 f_r^2 C_T} \tag{9}$$

Where

$$C_T = \frac{C_1 C_2}{C_1 + C_2} \tag{10}$$

The value of the inductance of the inductor L obtained is 0.0696 nH and capacitors C_1 and C_2 are 27 nF and 270 nF respectively.

The number of turns for the air core coil with inductance value gotten above was calculated using the expression below:

$$L = \frac{0.394 r^2 N^2}{9r + 10l} \tag{11}$$

where r is the radius of the air core coil, *l* is the length of the air core coil and N is the number of turns. The selection of gauge of the copper wire depends on the current that passes the inductor. A single wire gauge of 40 was used with a maximum current capacity of 23.3 mA.

b) Amplifier & Shaping Circuit

This circuit consists of an emitter follower, amplification and shaping circuit is figure 3. To improve the weak output signal from the oscillator, the emitter follower was placed between the oscillator and the amplification circuit since it is usually characterized by high input impedance and low output impedance. The following parameters were considered for the emitter follower and amplifier with shaping circuit design.

$V_B = 2.1\,V$, $I_B = 10\,\mu A$, $hfe = 100$, $V_{CC} = 5\,V$, $V_{BE} = 0.7\,V$

$$R_5 = \frac{V_{CC} - V_B}{I_B} \tag{12}$$

$$R_6 = \frac{V_B - V_{BE}}{I_E} \tag{13}$$

Where $I_E = I_B\, hfe$

Required resistors values are $R_5 = 290\ k\Omega$ and $R_6 = 1.4\ k\Omega$

The amplification section is a simple common emitter amplifier because of its best combination of voltage gain and current gain.

$I_B = 4.3\ \mu A,\ hfe = 270,\ V_C = \frac{1}{2} V_{CC} = 4.5\ V$

$$R_6 = \frac{V_{CC} - 0.7\ V}{I_B} \tag{14}$$

$$R_7 = \frac{V_{CC} - V_C}{I_C} \tag{15}$$

Where $I_C = I_B\ hfe$

The required value of R_7 and R_8 are 1 MΩ and 2.2 kΩ respectively.

The shaping circuit unit was implemented using 74S132 CMOS Schmitt trigger NAND gate. When the voltage from the output of the emitter follower reaches 1.8 V, the NAND gate is high until it's below noise margin level thereby producing square wave. This square waveform is fed into microcontroller. To obtain the frequency of oscillation, the microcontroller determines the period when the signal is HIGH and LOW.

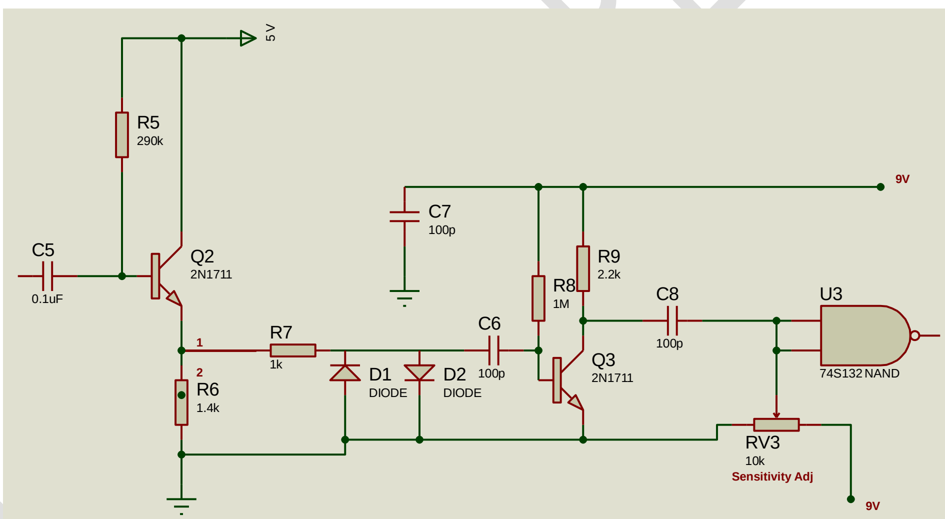


Fig. 3 Amplifier & Shaping Circuit

c) Temperature Sensing

Several types of temperature sensing techniques exist. This research work utilizes the thermo-junction Type K (chromel–alumel) temperature sensor. This sensor offers a wide temperature range, has low standard error, and has good corrosion resistance. The circuit in Figure 4 is a single-supply, type k thermocouple signal conditioning circuit with cold-junction compensation. It conditions the output of a Type K thermocouple, while providing cold-junction compensation for temperatures between 0°C and 250°C. The circuit operates from a single 3.3 V to 5.5 V supply and is designed to produce an output voltage transfer characteristic of 10 mV/°C. A Type K thermocouple exhibits a Seebeck coefficient of approximately 41 µV/°C; therefore, at the cold junction, the TMP35 (low voltage, precision centigrade temperature sensor), with a temperature coefficient of 10 mV/°C, is used with R_1 and R_2 to introduce an opposing cold-junction temperature coefficient of −41 μV/°C. This prevents the isothermal, cold-junction connection between the PCB tracks of the circuit and the wires of the thermocouple from introducing an error in the measured temperature. This compensation works extremely well for circuit ambient temperatures in the range of 20°C to 50°C. Over a 250°C measurement temperature range, the thermocouple produces an output voltage change of 10.151 mV. Because the required output full-scale voltage of the circuit is 2.5 V, the gain of the circuit is set to 246.3. Choosing R4 equal to 4.99 kΩ sets R5 equal to 1.22 MΩ. Because the closest 1% value for R5 is 1.21 MΩ, a 50 kΩ potentiometer is used with R5 for fine trim of the full-scale output voltage. Although the OP193 is a superior single-supply, micropower operational amplifier, its output stage is not rail-to-rail; therefore, the 0°C output voltage level is 0.1 V. The circuit is digitized by a single-supply ADC, by adjusting the ADC common to 0.1 V.

d) Display unit

A 16 x 2 LCD unit compatible with the Hitachi HD44780 driver was adapted for use as the display unit for the developed instrument. The LCD output was controlled by an arduino microcontroller unit which has an inbuilt ADC unit for converting analogue signals to digital signals. The Arduino microcontroller used a liquid crystal library to control the LCD display. The microcontroller manipulates several interface pins at once to control the display.

e) Microcontroller

For this research work, a 2560 arduino mega microcontroller was used owing to its flexibility, availability and huge libraries database. It functions as a frequency counter and thermometer by measuring the frequency of oscillation whenever magnetic materials are introduced into the oscillator’s coil and temperature required to compute magnetic moment of magnetic samples. A suitable micro-C code was written and embedded in the microcontroller so as coordinate the activity of the entire system, perform the necessary calculations and send output result to the display unit. The microcontroller was interfaced with a 2 by 16 Hitachi Liquid Crystal Display (LCD) so as to display the measured values of magnetic moment.

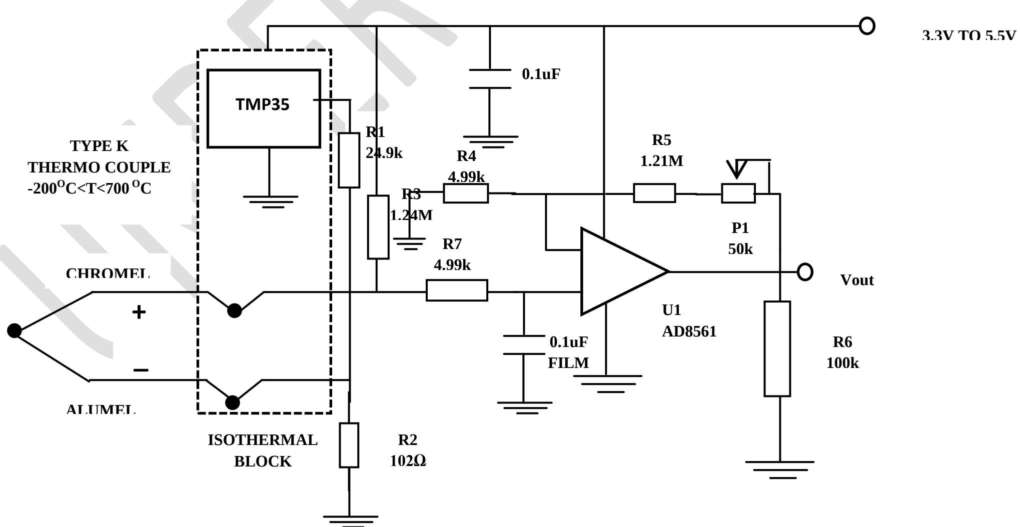


Figure 4: Thermocouple Amplifier

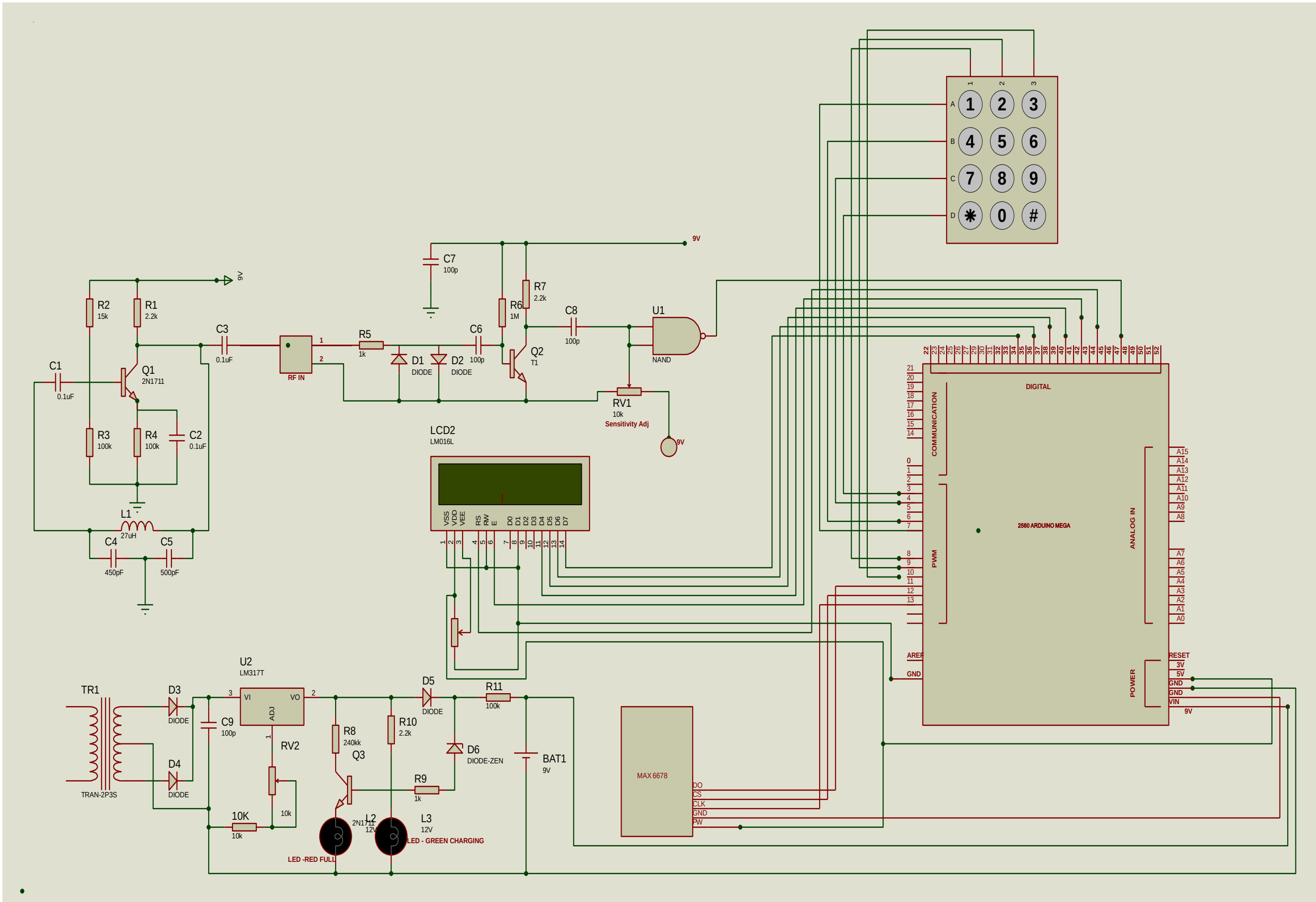


Figure 5: Complete Circuit Diagram of the Developed Paramagnetism Analyser.

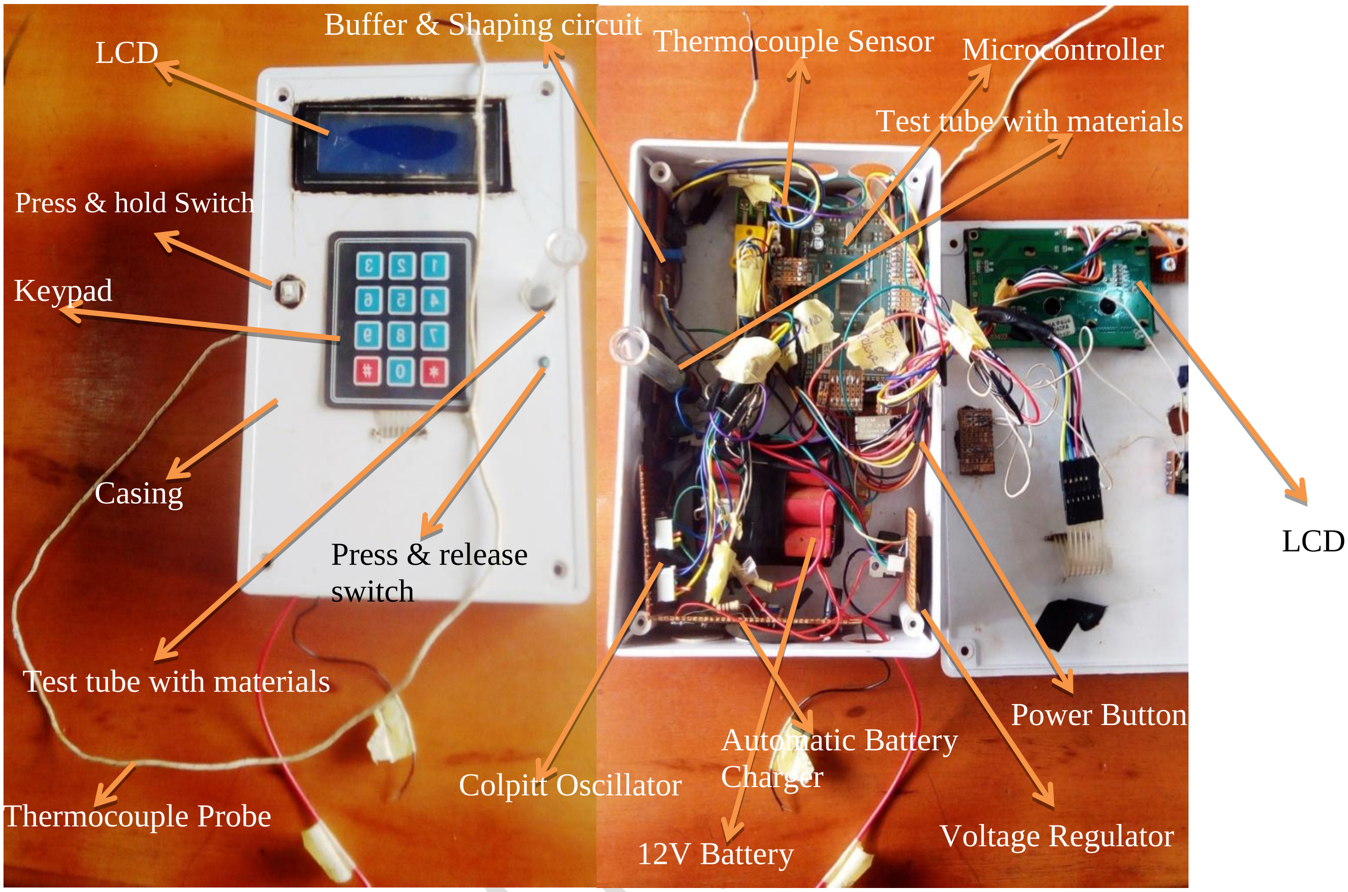


Figure 6: Image of the developed Paramagnetism Analyzer

3. TESTING, PERFORMANCE EVALUATION AND CALIBRATION

a) Testing and Examination of Counter Developed and Oscillator Circuit

The figure 5 shows the completed circuit of the developed paramagnetism analyser under test and evaluation. Table 1 shows the data obtained from the analysis carried out during the testing of the digital counter developed and available digital frequency meter. The testing was done by simultaneously passing a varying signal from a standard signal generator into the developed frequency counter and a standard frequency meter (MEGGER M7029) to verify if there is variation between the two measurements. Although the actual measurement recorded by the two instruments differs a little due to marginal discrepancy, however there is a consistency in the variation of two sets of measurement with respect to the input signal from the signal generator. The factor by which the measurement of the standard frequency meter increases or decreases is the same factor by which the measurement of the designed meter increases or decreases. The correlation factor (R^2) obtained from table 1 is 0.99950, show a reliably good agreement between the measured values with the standard data values. Statistical analysis revealed a mean percentage error value of 1.17% and accuracy of 98.83%. This shows that the designed frequency counter compared favorably well with the standard frequency meter (MEGGER M7029). The comparison plot of standard frequency meter against the designed frequency counter was plotted on an excel spreadsheet and the graph is shown in Figure. 7.

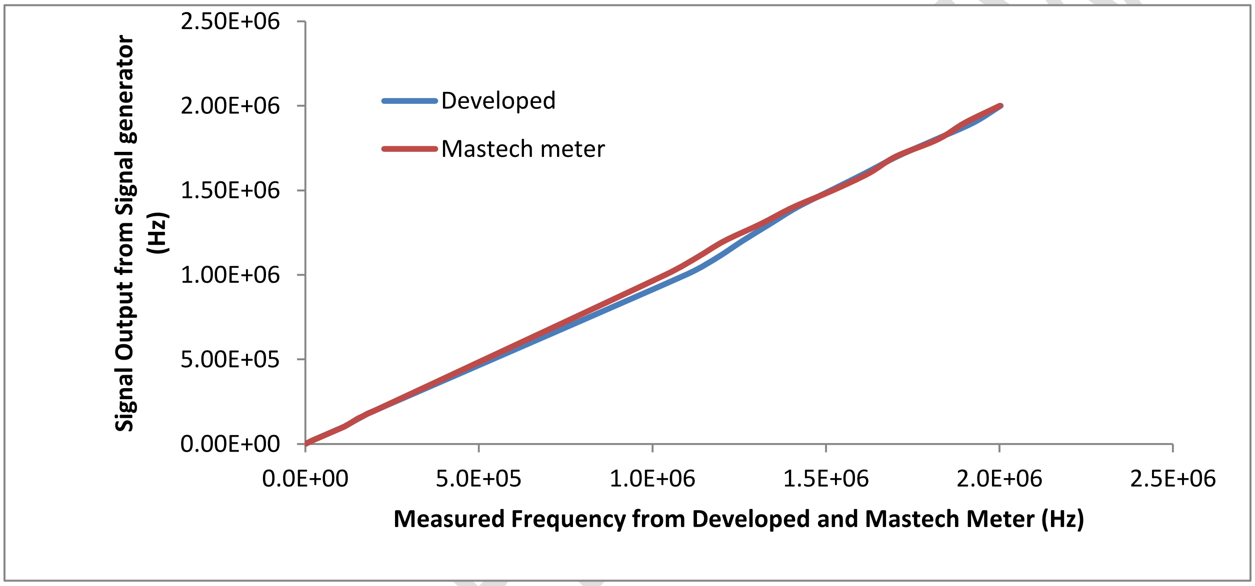
Table 1: The output frequency from the developed frequency counter and the standard meter with respect to the varying input signal from the signal generator and the errors

Signal Generator (Hz)	Designed Counter (Hz)	Standard Meter(Hz)	Error
1.0K	1088	1072.7	0.01426307
1.1K	1185	1164.8	0.01734203
1.2K	1259	1241.3	0.01425924
1.3K	1341	1311.2	0.02272727
1.4K	1414	1407.8	0.00440403
1.5K	1520	1520.8	0.00052604
1.6K	1612	1605.4	0.00411112
1.7K	1723	1705.8	0.01008325
1.8K	1812	1805.9	0.00337782
1.9K	1922	1901.9	0.01056838
2.0K	2011	2002.6	0.00419455
10K	10805	10605.0	0.01885903
11K	11765	11524.0	0.02091288
12K	12393	12410.0	0.00136986
13K	13254	13216.0	0.0028753
14K	14059	14285.0	0.01582079
15K	15050	15272.0	0.01453641
16K	16143	16529.0	0.02335289
17K	17029	17349.0	0.01844487
18K	18062	18008.0	0.00299867
19K	19040	19086.0	0.00241014
20K	20230	20176.0	0.00267645
100K	110004	110330.0	0.00295477
110K	119482	118600.0	0.00743676
120K	127780	127910.0	0.00101634
130K	136467	135720.0	0.00550398
150K	154168	151420.0	0.01516921
160K	163226	161920.0	0.0181482
170K	171929	171500.0	0.00806571
180K	181696	180380.0	0.00250146
190K	192921	191770.0	0.00729571
200K	203359	203350.0	0.00600198
1.0M	1096355	1037500.0	4.4259E-05
1.1M	1185157	1126300.0	0.05672771
1.2M	1259316	1207300.0	0.05225695
1.3M	1338146	1311500.0	0.04308457
1.4M	1417516	1405900.0	0.02031719
1.5M	1514649	1520800.0	0.00826232
1.6M	1612017	1623500.0	0.00404458
1.7M	1705464	1701800.0	0.00707299

1.8M	1815580	1821900.0	0.00215301
1.9M	1924996	1901100.0	0.00346891
2.0M	2003709	2001200.0	0.01256956
		0.99950	0.01172

Figure. 7: Comparative plot of developed Frequency Counter against Standard Frequency meter with respect to a varying signal generator.

The oscillator's response was examined when a magnetic material is introduced into its coil by



gradually lowering granulated magnetic material contained in a test tube inside the oscillators coil. This causes a change in the inductance of the coil which in turn causes changes in the output frequency of the oscillator. The corresponding frequency values were measured using both the standard frequency meter (MEGGER M7029) and the developed oscillator with counter. The results obtained are shown in Table 2. Obviously from the table 2, the result showed that the developed oscillator with counter compared favorably well with one obtained with the standard frequency meter. That means that oscillator is fairly stable and reliable.

Table 2 Frequency Count from both Meters when Sample Materials were gradually lowered into the Coil

Frequency Counter	Frequency Meter	Deviations
1502813	1502712	6.72118E-05
1512812	1512701	7.33787E-05
1601812	1601702	6.86769E-05
1661281	1661171	6.62183E-05
1700302	1700221	4.76409E-05
1700412	1700302	6.46944E-05
1801328	1801218	6.10698E-05
1802787	1802621	9.20881E-05
		0.0000676

b) Calibration of Paramagnetic Analyser

The developed instrument was calibrated using iron as a standard magnetic sample with known magnetic moment value as provided in literature. It is necessary to compute the calibration constant K in order to ascertain the accuracy and efficiency of the developed system. The calibration of the paramagnetic analyzer was done using equation 16

$$\mu_{eff} = \frac{KTM_r\Delta f}{mf_0} \qquad 16$$

Whereμ_{eff} is the effective magnetic moment (Am^2), K is the calibration constant (Am^2/K), M_r is the atomic mass (kg), T is the Temperature (K), Δf is the frequency change (Hz), m is the mass of sample materials (kg). The microcontroller was used to measure the frequency and temperature; the mass of sample material was obtained using an electronic balance. These made the computation of the calibration constant easier. The following procedures were observed: firstly, the mass and the temperature of the material samples were determined using an electronic balance and a temperature sensor respectively. Thereafter, the frequency of the oscillator when an empty tube was inserted and when a known mass of known material with known magnetic moment was inserted were measured and this gave the frequency change. The table 3 gives the record of the measured and constant parameters. The calibration constant was computed using the expression below and was incorporated into the microcontroller for accurate computations of magnetic moment for the available materials considered.

Table 3: Parameters for Calibration Constant Computation

Parameter	Values
Initial frequency, f_0 (Hz)	1502813
Final frequency, f_f (Hz)	1802787
Frequency change, Δf (Hz)	299974
Measured mass, m (kg)	0.005
Obtained atomic mass number, M_r (kg)	0.056
Obtained magnetic moment of known sample μ_{eff}, (Am^2)	5.60
Temperature, T (K)	303

$$Calibration\ constant,\ K = \frac{\mu_{eff} m f_0}{M_r T \Delta f} = 0.00000185\ Am^2/K \qquad 17$$

This value was incorporated into the microcontroller for accurate computation of the magnetic moment of the available material considered.

4. RESULT

Table 4: Measured and standard magnetic moment of magnetic materials available

Materials	Measured μ_{eff} (Am^2)	[1]Standard μ_{eff} (Am^2)
Aluminium	3.65	3.63 – 4.00
Copper	1.99	1.90 – 2.10
Iron	5.70	5.00 – 5.60

[1] http//:web.uvic.ca/~djberg/Chem324/Chem324-12.pdf

5. MEAN STANDARD DEVIATION ESTIMATION

The mean standard deviation of the designed instrument was obtained by repeatedly measuring the magnetic moment of a magnetic sample (iron) for good ten times in order to test repeatability or deviation from true value. The repeated measured values obtained are shown in table 5

Table 5: Mean Standard deviation Table.

Measured values (x)	$x_i - \bar{x}$	$(x_i - \bar{x})^2$
5.70	0.015	2.25×10^{-4}
5.69	0.005	2.50×10^{-5}
5.70	0.015	2.25×10^{-4}
5.65	- 0.035	1.23×10^{-3}
5.68	- 0.005	2.50×10^{-5}
5.71	0.025	6.25×10^{-4}
5.67	- 0.015	2.25×10^{-4}
5.68	- 0.005	2.50×10^{-5}
5.69	0.005	2.50×10^{-5}
5.68	- 0.005	2.50×10^{-5}
		2.655×10^{-3}

$$\text{Standard deviation}, \sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{N}((x_i - \bar{x})^2)} = \sqrt{\frac{2.655 \text{ x } 10^{-3}}{10}} = 0.0163 \approx 0.02$$

$$\text{Standard deviation error}, \mu = \frac{\sigma}{\sqrt{n}} = \frac{0.0163}{\sqrt{10}} = 0.005$$

6. DISCUSSION

The colpilt oscillator was designed to give an output frequency of 2 MHz and above with a resolution of 1 Hz. Obviously from Table 1, the designed oscillator with counter compared favorably well with the standard frequency meter (MEGGER M7029). Although the actual readings recorded by the two instruments differ a little due to marginal discrepancy, however there is a consistency in the variation of the two sets of measurement with respect to the input signal from the signal generator. The factor by which the measurement of the standard frequency meter increases or decreases was the same factor by which the measurement of the designed meter increases or decreases as shown in Figure 1. Statistical analysis revealed a mean percentage error value of 1.17% and accuracy of 98.83%. The correlation factor (R^2) of 0.99950 obtained also show a reliably good agreement between the designed oscillator with counter measured values and the standard meter measured values. The oscillator's performance and response when a magnetic material is introduced into its coil were also verified. This was achieved by gradually lowering granulated magnetic material contained in a test tube inside the coil of the oscillators. This causes a change in the inductance of the coil which in turn causes changes in the output frequency of the oscillator. The corresponding frequency values were measured using both the standard frequency meter (MEGGER M7029) and the developed oscillator with counter. The results obtained are shown in Table 2. Obviously from the table, the result showed that the developed oscillator with counter compared favorably well with the standard frequency meter with an absolute mean deviation of 0.0000676. This means that when any material that has paramagnetic or ferromagnetic properties inserted into the coil of the oscillator there will be a corresponding frequency change. The temperature sensing device used measures temperature between – 200°C to 700°C with a sensitivity of 41 µV/°C and a resolution of 0.25/°C. To ascertain the accuracy and efficiency of the developed system, the instrument was calibrated using iron as a standard magnetic sample with known magnetic moment value. The calibration constant K was computed and the value obtained is 0.00000185A/m^2. After the design, examination and performance test was carried out on the developed paramagnetism analyzer. It was found that the instrument measures the magnetic moment of the materials available accurately with a resolution of 0.01 *A/m^2* and a mean standard deviation of 0.0163 ± 0.005. This means that error in the instrument is very insignificant and that it can accurately measure magnetic of magnetic materials once without repeated measurements. The standard deviation was obtained using Table 5 by repeatedly measuring the magnetic moment of the magnetic samples tested for ten numbers of times. The results are shown in Table 4. Obviously from the table, the measured values of magnetic moment for the available known materials fall within the range of values obtained from literature. In the case of iron, a difference of 0.1Am^2 was observed. This may be as a result of impurities arising from metal recycling processes. The maximum power consumption of the developed instrument is 3.85 watts.

CONCLUSION

The aim of this study was carried out to a conclusive end. This involves the design and construction of a paramagnetism analyzer. The developed instrument showed a good response and the performance was excellent when the measured magnetic moment values were compared with existing standard magnetic moment values obtained from literature with a standard deviation of 0.0163 ± 0.005 and a resolution of 0.01Am^2. Aluminum had a magnetic moment of 3.65 Am^2, copper had 1.99 Am^2 and Iron had 5.70 Am^2.. The correlation factor (R^2) of 0.99950 obtained for the two frequency counter also show a reliably good agreement between the designed frequency counter measured values and the standard meter measured values. Statistical analysis revealed a mean percentage error value of 1.17% and accuracy of 98.83%. The temperature sensing device measures temperature between – 200°C to 700°C with a sensitivity of 41 µV/°C and a resolution of 0.25/°C. Conclusively therefore, the instrument performed well and it is recommended for measurement of magnetic moment of magnetic sample (ferromagnetic and paramagnetic) in material science/condensed matter laboratories. The developed instrument is a portable handheld device which operates well on a rechargeable DC power source. It is cheap, easy to repair if malfunctioned and does not require any special skill to operate.

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