Refractive Index of Reference Material under Different Measurement Conditions

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Abstract— **Refractive indices of different concentrations (5 ppm up to 200 ppm) of reference chemical solution (Fe(NO3)3) Nonahydrate were determined with an accuracy of ±10-5, the Brix of these chemicals were measured by using Digital multi-wavelengths (refractometer DSR-λ). Practically, the refractive indices of these solutions have been measured as a function of temperatures in the spectral visible range 0.4-0.7 µm; with increasing wavelengths and with increasing temperature the refractive index decreased monotonically. The refractive indices are increasing with increasing concentrations. Also, the Brix of this solution have been measured as a function of temperature (20 oC up to 30 oC) with wavelengths in the same visible spectral range. The Brix values are used as a tool of concentration of these chemicals samples. The empirical formula between the concentration and the Brix of these chemical are applied.**

Index Terms—**DSR, Brix, Dgital multi wavelength**

I. INTRODUCTION

There are an important relation between the accurate measurement of refractive index and the accurate specifications of chemical structure of such material. Refractive index is one of the physical parameters of chemical solutions could be precisely measured using an optical wavelength techniques (refractometer) [1]. There are many different standard reference materials commonly used in different applications. One of the most important transparent liquid Standard Reference Material is Ferric Nitrate of chemical compound with formula $Fe(NO_3)_3$.9H2O, it is also called Iron(III) Nitrate. This property is used by Chemists in a

Spectrometer to detect and measure the elemental composition of complex molecule and mixtures. Ferric Nitrate is a water soluble oxidizer [2]. It readily absorbs water from the atmosphere and must be kept tightly capped. Refractometers are used generally in oil, food and chemical industry to control the concentration during the production by monitoring the refractive index. It has different LED's at discrete wavelengths covering the visible range. The measurement principle refractometer relies on a complete refractive index measurement at visible wavelengths, which is also sensitive to absorption, leading to a full optical characterization of the measured substance. This Standard Reference Material (SRM) is intended for use as a primary calibration standard for the quantitative determination of iron. Since it is deliquescent, it is commonly found in its nona-hydrate form $Fe(NO_3)_3.9H_2O$ in which it forms colors to pale violet crystals. In chemistry laboratory, ferric nitrate is a common catalyst, which was chosen for synthesis of sodium amide as shown in the following equation [3].

$$2\ NH_3 + 2\ Na \rightarrow 2\ NaNH_2 + H_2.$$

Certain clays impregnated with ferric nitrate have been shown to be useful oxidants in organic synthesis. For example, ferric nitrate on montmorillonitea reagent called "Clayfen" has been employed for the oxidation of alcohols to aldehydes and thiols to disulfides [4].

A conversion of Brix (determined by refraction) to density is possible using labels. In case of high acid content, a Brix measurement using refractometer will give A somewhat lower values than Brix using density measurement device. The correct Brix value is obtained using specific table for the relation between the concentration and the density.

II. EXPERIMENTAL WORK

Liquid samples were prepared in the Reference Materials Lab at NIS with the concentrations as indicated in Table 1 [4, 8].
Table 1. (Different chemical concentrations of number of samples)

No. of Sample	Chemical Concentration
1- Blank	nitric acid 1%
2- Ferric nitrate	5 ppm
3- Ferric nitrate	10 ppm
4- Ferric nitrate	50 ppm
5- Ferric nitrate	100 ppm
6- Ferric nitrate	200 ppm

Properties:
Compound Formula: $H_{18}FeN_3O_{18}$,
Molecular Weight= (404 g/mole),
Density= 1.6429 g/cm^3,
Melting point= 47.2^{o}C (117.0 ^{o}F; 320.3 k),
Boiling point= 125^{o}C (257 ^{o}F; 398 k),
Solubility in water (150 g/100 ml).

2.1 Measurements of Refractive Index and the Brix of Solutions.

Using the DSR-λ refractometer which is an automatic microprocessor controlled critical angle refractometer been designed to measure the refractive index of liquid media independent of opacity, viscosity and color with high

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resolution 0.00001 RI/0.01 Brix [5,6]. The instrument consists of an electronic unit separated from the stainless-steel measuring unit. 9 wavelengths (404.7, 435.8, 486.1, 546.1, 587.6, 589.3, 632.8, 656.3, and 706.5 nm) were employed during the dispersion measurement performance over the full visible range. A sample volume of 0.3 ml is sufficient for measuring process [7, 9]. The refractometer is equipped with a Peltier thermostat allowing measurement of refractive index at any selected temperature up to 80 ^{o}C, but the variation of the Brix with temperature up to 30^{o}C.

III. Results and Discussions

The refractive indices were measured between 20 ^{o}C and 50 ^{o}C in ascending and descending orders and the mean values were considers as shown in Figs (1- 5). Fig 6 shows the variation of refractive indices for the samples under investigation. The Brix variations with the temperature from 20 ^{0}C up to 30 ^{0}C of different concentration solution of Fe(III)Nitrate at different wavelengths are shown in Fig (7-9).

Since temperature has a very important influence on the refractive index measurement, the temperatures of the sample have to be controlled with high precision. The modern refractometers such as DSR-λ are several subtly different designs for controlling the temperature but there are some key common factors to all such as high precision temperature sensors and Peltier devices to control the temperature of the sample and the prism. The temperature control accuracy of these devices should be precisely calibrated, so that the variation in sample temperature is small enough that it will not cause a detectable refractive index change.

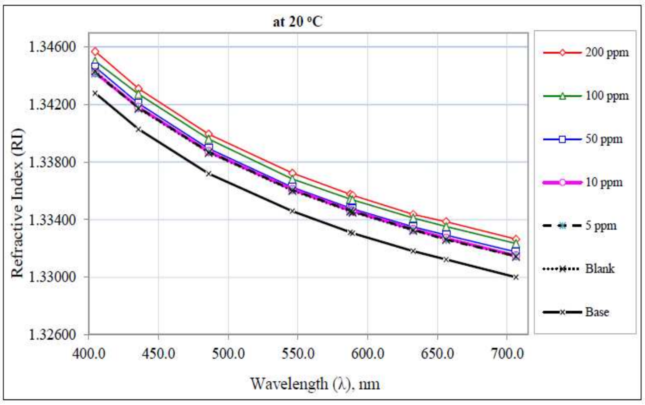

Fig (1) the variation of refractive indices of these samples at 20 ^{0}C in the visible wavelengths range

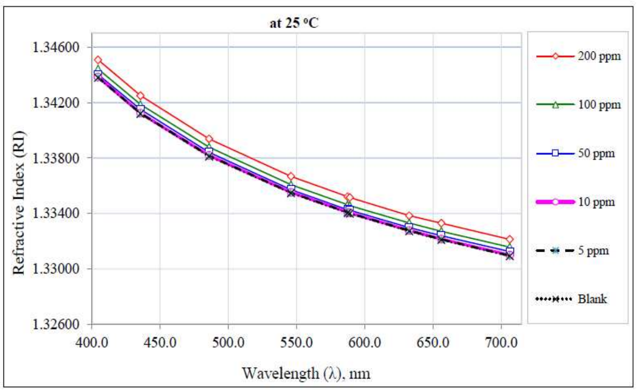

Fig (2) the variation of refractive indices of these samples at 25 ^{0}C in the visible wavelengths range

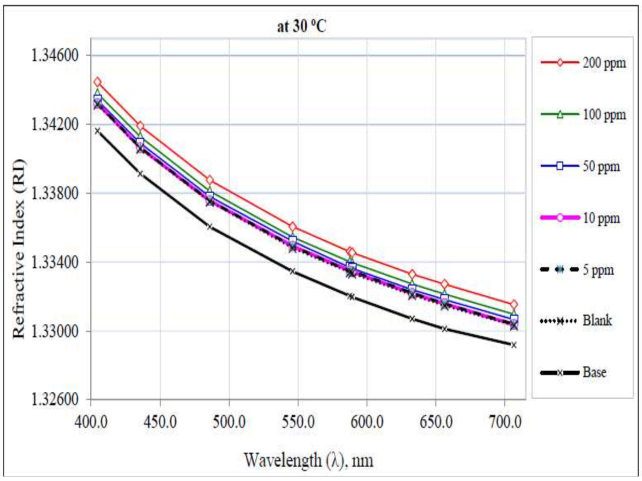

Fig (3) the variation of refractive indices of these samples at 30 ^{0}C in the visible wavelengths range

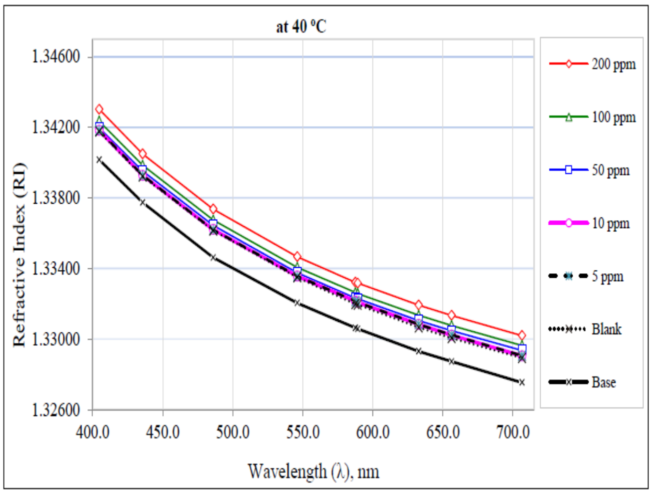

Fig (4) the variation of refractive indices of these samples at 40 ^{0}C in the visible wavelengths

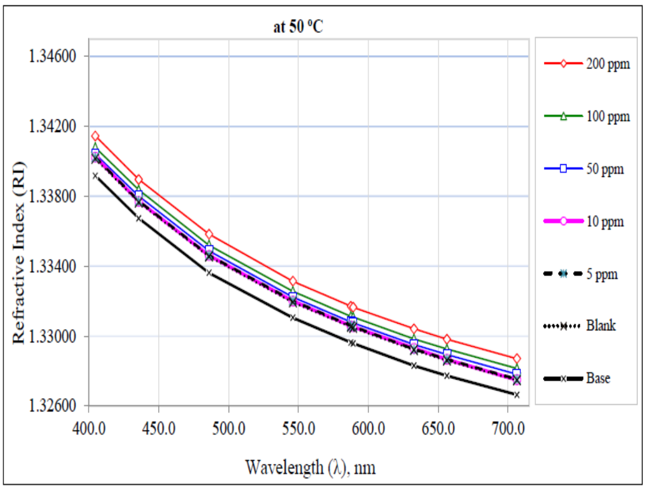

Fig (5) the variation of refractive indices of these sample at 50 ^{0}C in the visible wavelengths

In modern refractometers, the wavelength is tuned to bandwidth of +/- 0.2 nm to insure correct results for samples in different dispersion; the refractive indices values are decreased monotonically with increasing temperature and wavelengths. No absorption is observing but the value of refractive index is increased with increasing of concentrations [10].

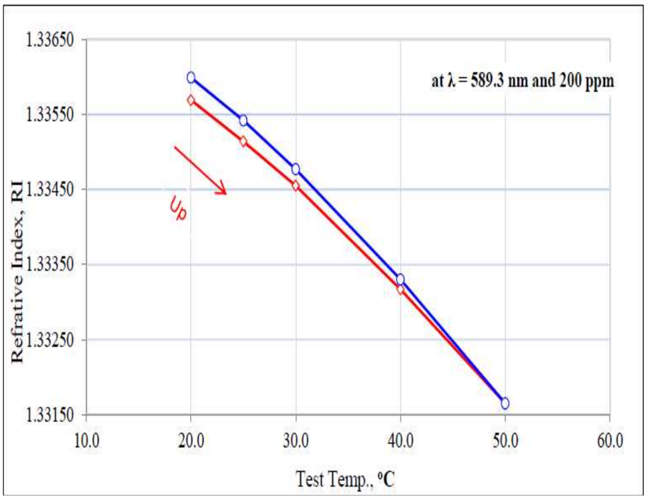

Fig (6) the hysteresis curve due to the variation of refractive index in ascending and descending orders.

In fig(6), the hysteresis curve due to the variation of refractive index in ascending and descending order from increasing temperature from 20 ^{0}C up to 50 ^{0}C for the samples under investigation are due to evaporation. These results are in agreement with its melting point at 47.2 ^{o} C.

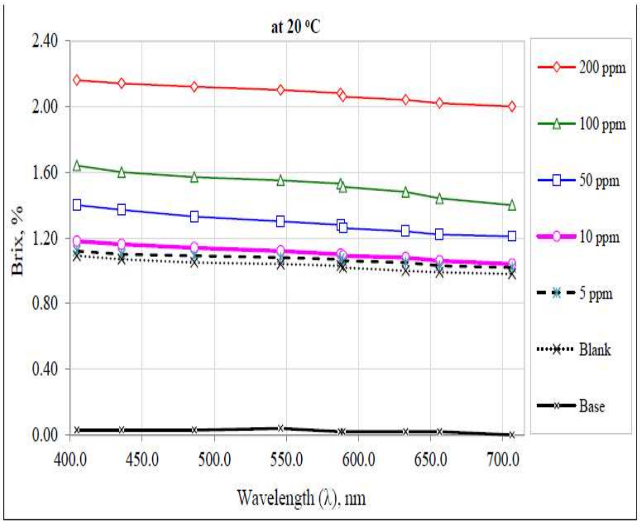

Fig (7) the variation of refractive indices of these samples at 20 ^{0}C in the visible wavelengths range with the zero reference.

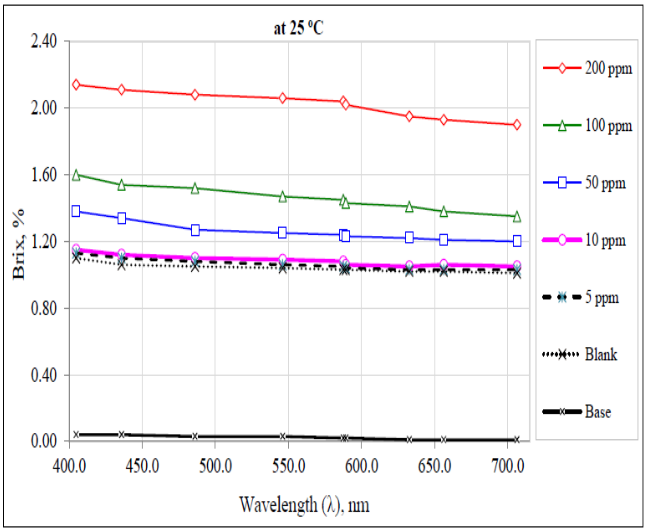

Fig (8) the variation of refractive indices of these samples at 25 ^{0}C in visible wavelengths range with the zero reference

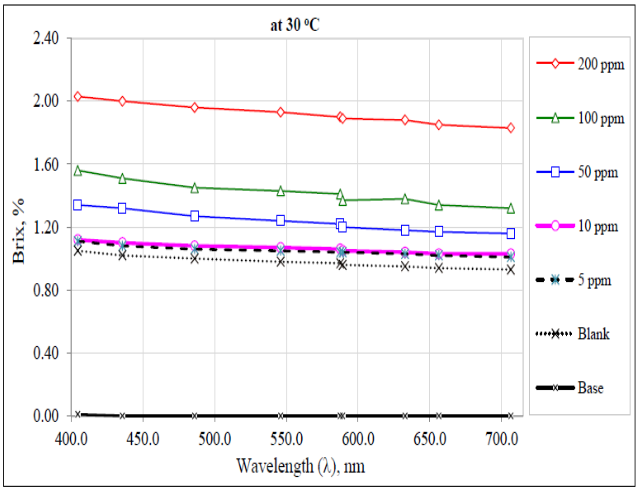

Fig (9) the variation of refractive indices of these samples at 30 ^{0}C in visible wavelengths range with the zero reference

The study of the Brix at different temperature (20 ^{0}C, 25 ^{0}C, 3o ^{0}C) and different wavelengths in the visible range are shown in figures. (7- 9). These studies are used as a tool of the concentration of these samples [11]. The Brix were decreased smoothly with increasing temperature and wavelength.

Relation between Brix and Chemical Concentration for SRM

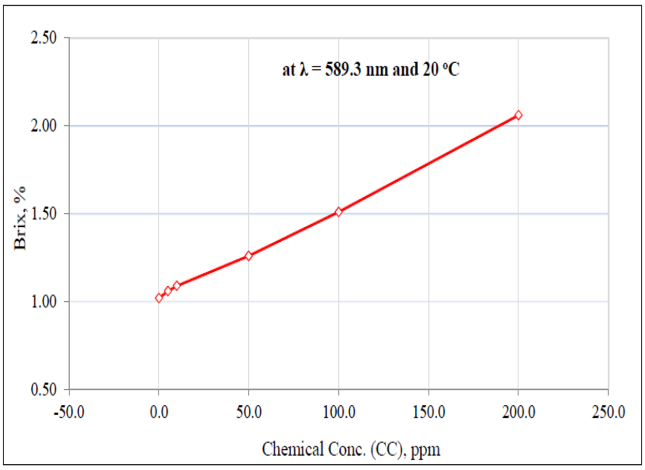

Fig (10) the relation between the chemical concentration of samples and Brix

Relation between the concentration of samples and Brix are shown in figure 10, it is formulated as in Equation below when λ = 589.3 nm at 20 ^{0}C. The empirical formula can be presented as:

$Brix_{\%} = 4\times10^{-6}\, CC^{2} + 0.0044\, CC + 1.0334$

IV. Conclusion

Five samples of Iron(III)Nitrate Nonahydrate are prepared in the Standard Reference Material Lab at NIS with concentrated variation from 5 ppm to 200 ppm as shown in table (1). We studied the variation of refractive indices of these samples as a function of wavelength and temperature. The refractive index decreases monotonically with increasing wavelength and temperature, but the refractive index increases with increasing of concentration.

Also from fig (6) which is called the hysteresis curve due to the variation of refractive index in ascending and descending order from increasing temperature due to evaporation. These results agree with its melting point at 47.2 ^{o}C. The variations of the Brix of these samples are shown in figures (7- 9). The Brix values of these samples are used to compare with the concentration of solutions. Fig (10), illustrates the relation between the Brix and chemical concentration CC at wavelength λ=589.3 nm and temperature 20 ^{o}C (directly proportional); the empirical formula is given by the last mentioned equation.

REFERENCES:

[1] Wei, D., K. Takamasu, and H. Matsumoto, *Is the Two-Color Method Superior to Empirical Equations in Refractive Index Compensation?* Optics and Photonics Journal, 2016. **6**(08): p. 8.

[2] Naveen, Togati, et al. "A Predictably Selective Nitration of Olefin with Fe (NO3) 3 and TEMPO." *The Journal of organic chemistry* 78.12 (2013): 5949-54.

[3] Khodier, S.A., *Refractive index of standard oils as a function of wavelength and temperature.* Optics & Laser Technology, 2002. **34**(2): p. 125-128.

[4] HSNO Chemical Classification Information Database, *New Zealand Environmental Risk Management Authority*, retrieved 2010-09-19.

[5] Musso, M., et al. "Interferometric determination of the refractive index of liquid sulphur dioxide." *Measurement Science and Technology* 11.12 (2000): 1714-20.

[6] Rheims J, Koser J, Wriedt T, *Refractive- index measurements in the near –IR using an Abbe refractometer*. Meas. Sci. Technol 1997, 8:601-5.

[7] Nemoto, Shojiro. "*Measurement of the refractive index of liquid using laser beam displacement.*" Applied optics 31.31 (1992): 6690-4.

[8] Standard Reference Material Lab at NIS –Egypt, 5, (2015).

[9] National Institute for Occupational Safety. *NIOSH pocket guide to chemical hazards*. DIANE Publishing, 2000.

[10] Rao, S. Madhusudana. "Spectrographic technique for determining refractive indices." *Optical Engineering* 36.1 (1997): 162-166.

[11] Bruno, Thomas J., and Paris DN Svoronos. *CRC handbook of basic tables for chemical analysis*. CRC press