20BDS0405 CHY 1701 2021/02/18

Quantitative colorimetric determination of Fe^{2+} metal ions using conventional and smart phone digital-imaging methods

1. Source of Iron in drinking water: Iron is a common metallic element found in Earth crust. Water percolating through soil and rock can dissolve minerals containing iron and hold them in solution. Occasionally iron pies may also be a source of iron in water. In deep well, where oxygen content is low, the iron bearing water is clear and colourless. But that water pumped from motor is exposed to surface oxygen and change into colored solid forms, initially into white, then yellow and finally to red-brown solid precipitate that settle out of the water.

2. Iron in drinking water: Iron is one of the many minerals that are essential for human health. Without iron, people may experience anaemia, fatigue, or an increase in infections. But how much iron is too much? Drinking water that contains iron can be beneficial to your health. However, excessive iron in drinking water may have negative effects like poor skin and metallic taste in water. Because water and iron don't physically mix well, people may notice leftover soap residue after showering or bathing. This soap build-up can also cause skin problems. Iron overload can also lead to hemochromatosis - which can cause damage to the liver, heart, and pancreas. Excessive iron can leave behind a residue in plumbing lines and this is yet another reason to consider removing iron from water. There are plenty of health concerns associated with too much iron intake, which is one of the main reasons people using well water should schedule annual water testing.

3. Importance of the experiment:

Metal ions such as Fe and Ni (Lewis acid) form complex with Lewis base and show strong colours even at lower concentrations. Ideal complexing agent should be stable, selective and be free from variations in color due to minor changes in pH or temperature. Colorimetric analysis is based on the change in the intensity of the colour of a solution with variations in concentration. Colorimetric method represents the simplest form of absorption analysis. The human eye is used to compare the colour of the sample solution with a set of standards until a match is found.

http://www5.csudh.edu/oliver/che230/labmanual/iron.htm

Expt. No.: 4 **Date:** 2021/02/18

Experiment	**Quantitative colorimetric determination of Fe^{2+} metal ions using conventional and smart phone digital-imaging methods**
Problem definition	Excess Iron in drinking water cause negative effects like poor skin and hemochromatosis (damage to liver, heart and pancreas). There are plenty of other health concerns associated with too much iron intake, and hence it is important to estimate the amount of iron in waste samples.
Methodology	Fe and 1,10-phenanthroline forms a stable deep-red colored complex. With increasing concentration of Fe in solution, its color intensity also increases, which in turn is a function of color coordinates (Red, Blue and Green, RGB) in the image taken using mobile phone camera.
Solution	Estimation of Fe concentration in different water samples can be found out using colorimetry technique from the calibration graph plotted based on different known Fe concentrations.
Student learning outcomes	Students will learn to perform colorimetric method, perform RGB response analysis and analyze Fe composition in different grades of steel

(i). Principle:

(a). Colorimetric method:

Photo-sensitive measurements are expressed in terms of absorbance, (A) as given in Eq. (1). Further, the linear relationship between absorbance (A) and concentration of the analyte

$$\varepsilon cl = A = \log(I_0/I) \quad \dots (1)$$

Where, I_0 is the incident light power, I the transmitted light power, ε = molar absorptivity, c = concentration of analyte and l = thickness of the solution.

(b). Digital-imaging method:

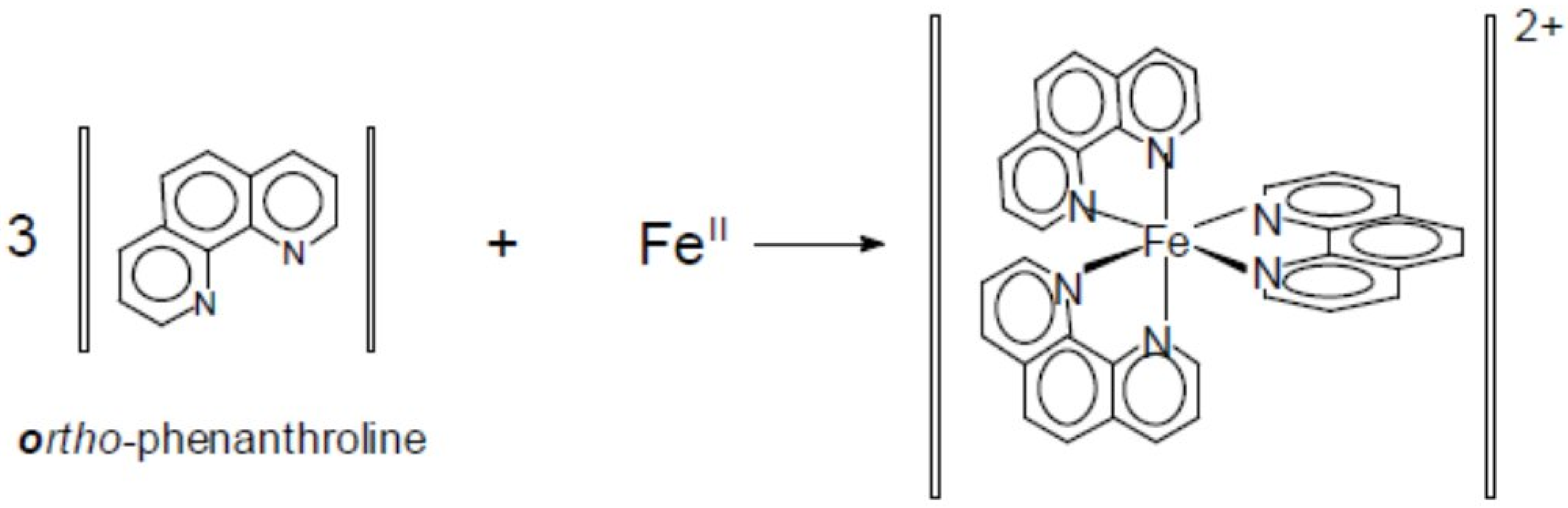
The color and intensity of digital image are usually 24 bit data (8 bit R + 8 bit G + 8 bit B) forming an additive color space, in which R, G and B lights are added together in various combinations to reproduce a broad range of colors. By using combination of R, G and B intensities, many colors can be displayed. The intensity of each color has 256 levels (from 0 to 255). The value of R = 0, G = 0, B = 0 refers to pure black while R = 255, G = 255, B = 255 is pure white. With this system, unique combinations of R, G and B values are allowed, providing for millions of different hue, saturation and lightness shades. These extensive dynamic colors of images provide the database for quantitative analysis. The goal of this study is to employ digital images-based colorimetry for the determination of Ni^{2+} concentration in aqueous samples.

The concentration of analyte is a function of color coordinates: $c = RGB$ … (2)

(ii) Scheme of the reaction and requirements

Iron (II) complex is formed with 1,10-phenanthroline $[Fe(C_{12}H_8N_2)_3{}^{2+}]$ and the absorbance of this colored solution is measured at **510 nm** with a colorimeter. Hydroxylamine (as the hydrochloride salt to increase solubility) is added to reduce any Fe^{3+} to Fe^{2+} and to maintain it in that state. The spectrum is plotted to determine the absorption maximum.

$4\ Fe^{3+}$	**$+ 2\ NH_2OH{\cdot}HCl$**	**$\rightarrow 4\ Fe^{2+}$**	**$+ N_2O$**	**$+ 4\ H^+$**	**$+ H_2O$**
Ferric Iron	Hydroxylamine Hydrochloride	Ferrous Iron	Nitrous Oxide	Proton	Water



ortho-phenanthroline

Ferrous tris-*o*-phenanthroline

Reagents, solutions and Instrumentation: Ferrous Ammonium Sulphate (10 ppm), 1,10-phenanthroline, Hydroxylamine hydrochloride and Sodium acetate solutions, Colorimetry and smartphone.

(iii). Procedure:

(a). Colorimetry method: Take 5 standard 50 mL volumetric flasks (to prepare 4 known and 1 unknown solution). Fill the burette with Fe stock solution (10 ppm). Add 5, 10, 15 and 20 mL of the Fe solution in burette to the std. flasks to get 1, 2, 3 and 4 ppm of Fe(II) solutions. The unknown sample will be furnished in another 50 ml volumetric flask. Further, add 0.5 mL of hydroxyl ammonium chloride solution followed by 2.5 ml of 1,10-phenanthroline using a burette. The Fe(II)-phenanthroline complex forms at pH 2 to 9. Add 2.5 mL of sodium acetate buffer to neutralize the acid present and adjust the pH to a value at which complex forms. After that, make up the 50 mL mark in std. flask with distilled water. Allow at least 15 minutes before making absorbance measurements so that the color of the complex can fully develop. Once developed, the color is stable for hours. Obtain the absorption spectrum of the Fe solutions by measuring the absorbance at **510 nm**.

Record these absorbance readings in **Table 1**. Draw a calibration graph taking concentration of Fe^{2+} (in ppm) as X-axis and absorbance readings as Y-axis. A straight line that passes through the origin is an indication that the measured data obeys Beer's Law. From the calibration plot, measure the concentration of Iron in the given unknown sample.

Digital imaging method: The prepared standard solutions are lined up along with unknown concentration sample and blank. Using a white paper as background, take a photograph of the samples by holding the camera around 50 cm away. The calibration curve will be constructed through the RGB values of analytical response with different concentration of Fe^{2+} ions using smartphone app (RGB Tool). In the plotted graph, RGB response varies linearly *vs* the analyte concentration. In order to get precise analysis, follow the steps given below:

Transfer prepared standard solution and unknown solution
into different colorimetric test tubes

↓

Take image of all test tube solution
using smart phone camera

↓

Open the image processing app

↓

Go to gallery, open the image stored in app and
extract RGB values for each image/conc.

↓

Process the RGB values (R/G) or (R/B) or (G/B)
etc., till to get linear response

↓

Plot the calibration curve using RGB linear
response vs concentration

↓

Find the unknown conc using the
calibration curve

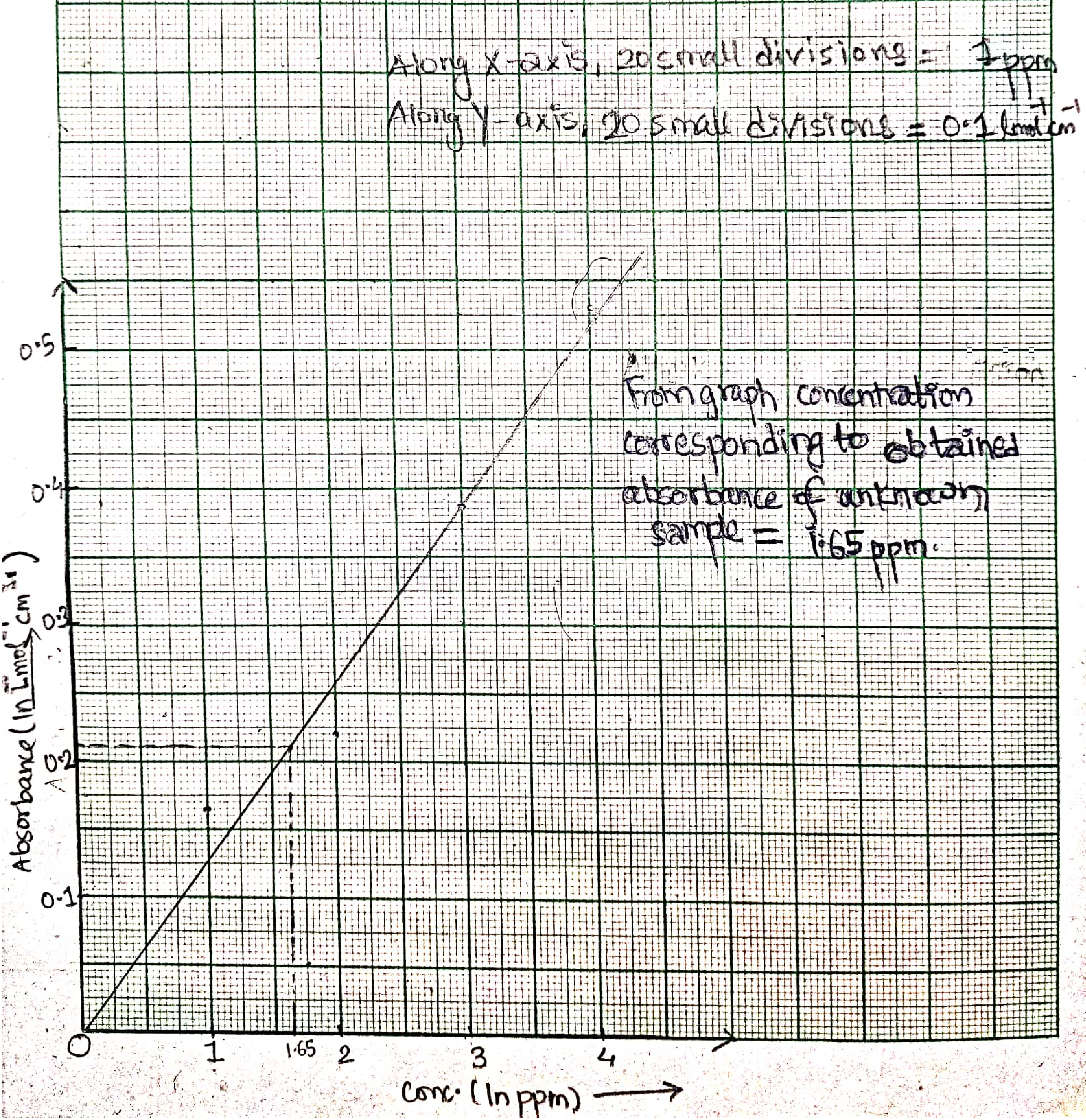
Table 1: Experimental Data

S. No.	Data collected from Colorimetric device		Data collected from smartphone device*					
	Conc. (ppm, X-axis)	Abs (Y- axis)	R	G	B	R/G	G/B	R/B
1.	1	0.165	132	50	14	2.64	3.57	9.43
2.	2	0.219	205	62	8	3.31	7.75	25.62
3.	3	0.385	237	71	9	3.34	7.89	26.33
4.	4	0.530	276	75	12	3.68	6.25	23
5.	Unknown	0.208	201	58	9	3.46	6.44	22.33

* Corresponding ratio that is linearly increasing with analyte concentration is used for plotting.

Result:

(i). Concentration of Fe in steel sample (using colorimetry) = 1.65 ppm (mg/L)
(ii). Concentration of Fe in steel sample (using digital imaging) = 22 ppm (mg/L)


Along X-axis, 20 small divisions = 1 ppm
Along Y-axis, 20 small divisions = 0·1 $L\,mol^{-1}\,cm^{-1}$
From graph concentration corresponding to obtained absorbance of unknown sample = 1·65 ppm.
0·5
0·4
0·3
0·2
0·1
O
1
1·65
2
3
4
Absorbance (In $L\,mol^{-1}\,cm^{-1}$)
Conc. (In ppm)

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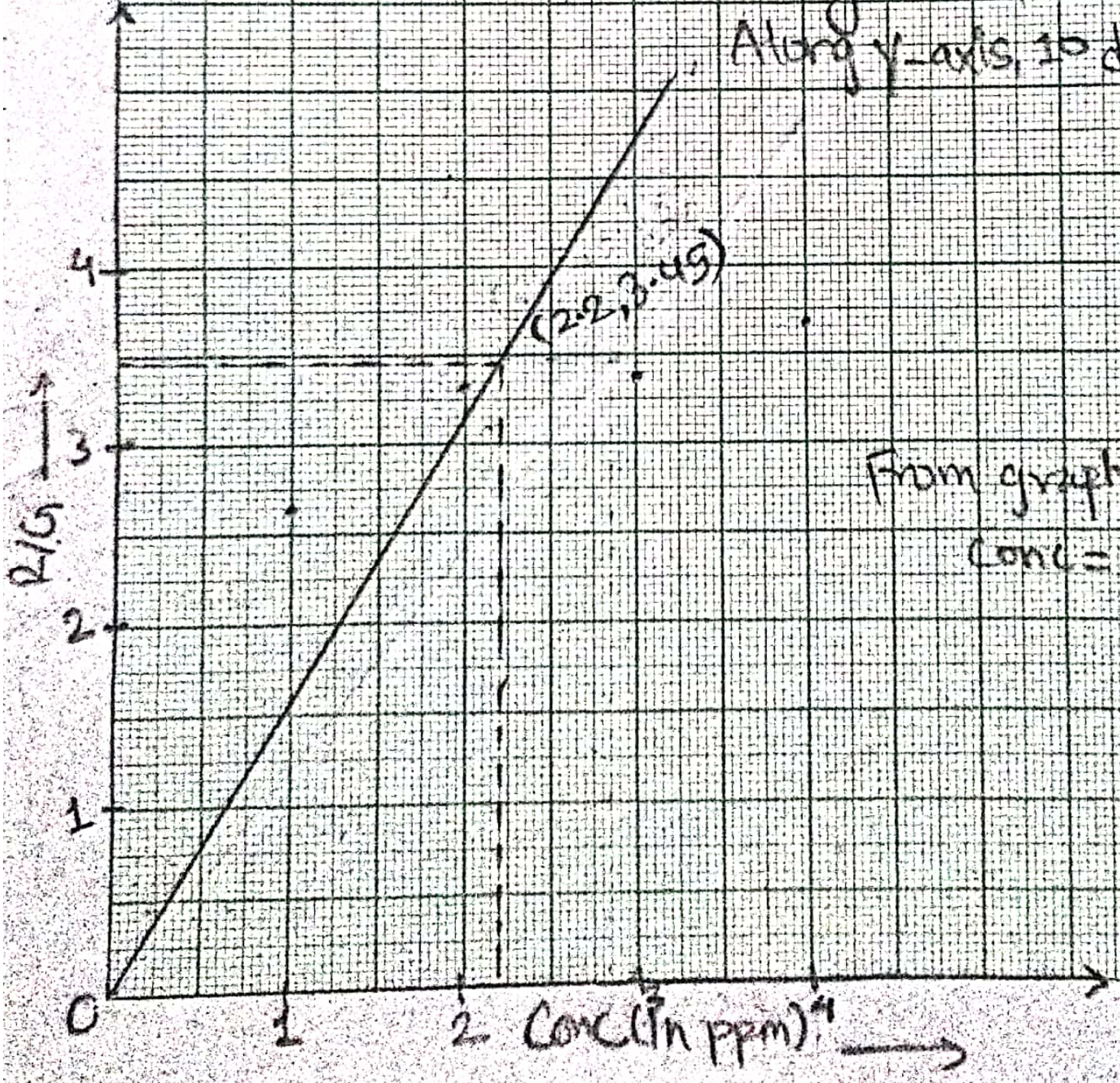
Along X-axis, 10 division = 0.5 ppm
Along Y-axis, 10 divisions = 0.5



From graph,
Conc = 2.2 ppm.