

CH 2252 Instrumental Methods of Analysis

Unit – I

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UV-vis Spectroscopy

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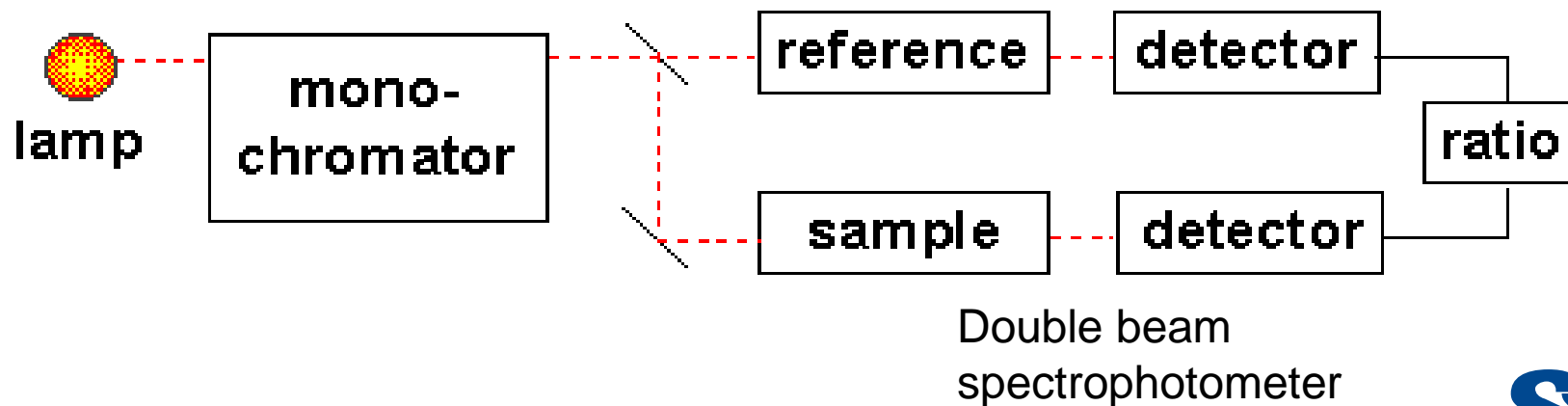
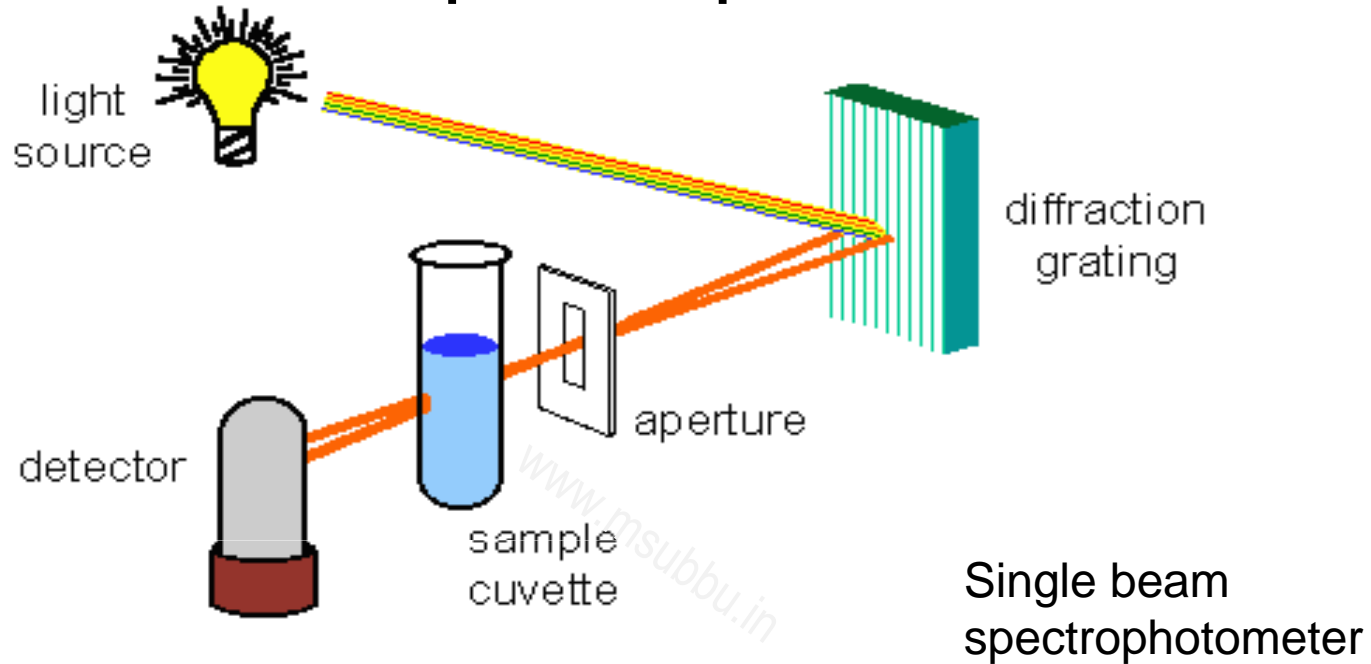


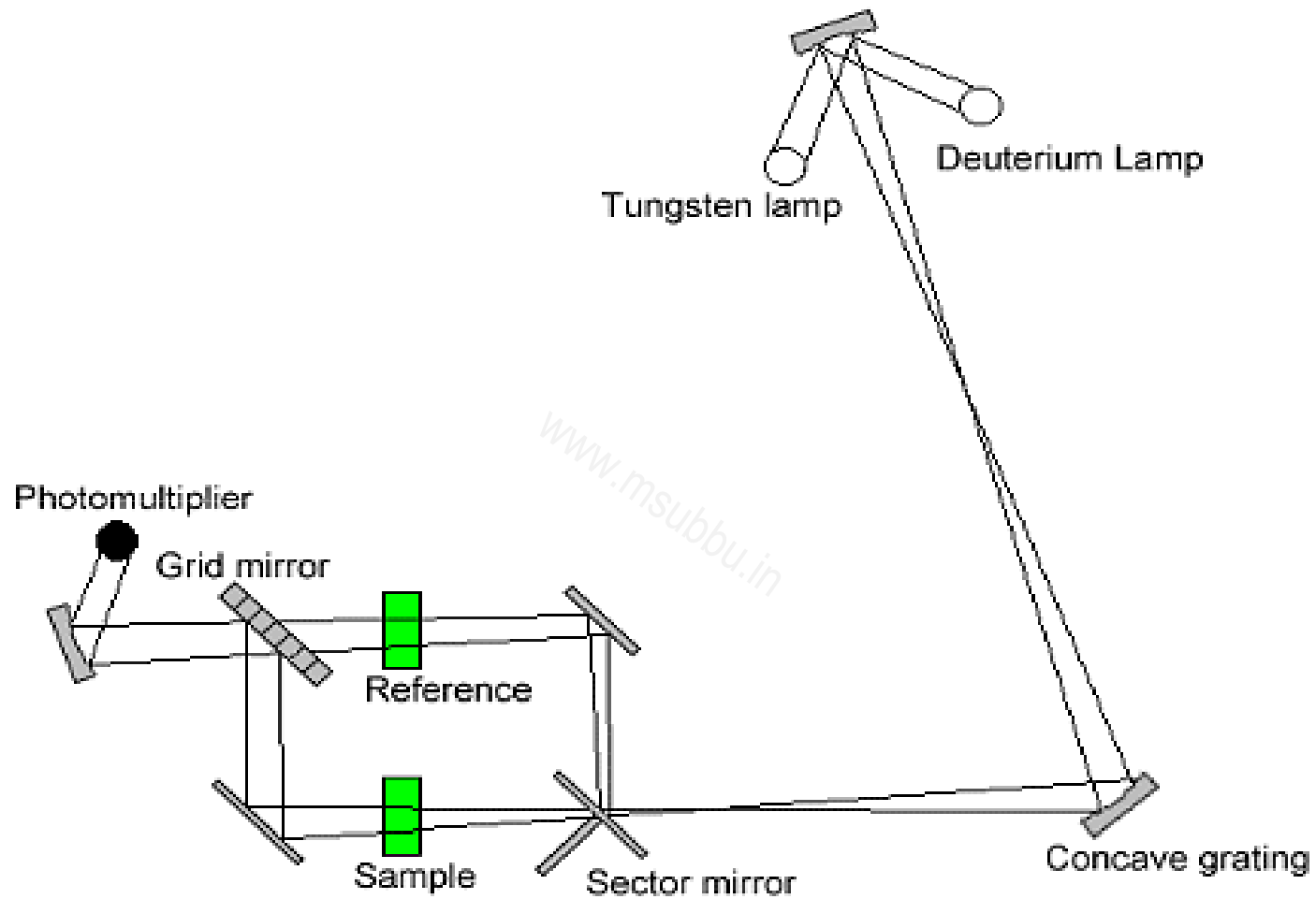
Contents

- Instrumentation (Source, Optical parts and Detectors)
- Estimation of inorganic ions such as Fe, Ni and Nitrite using Beer-Lambert's Law

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UV-vis spectrophotometer





Components of UV-vis

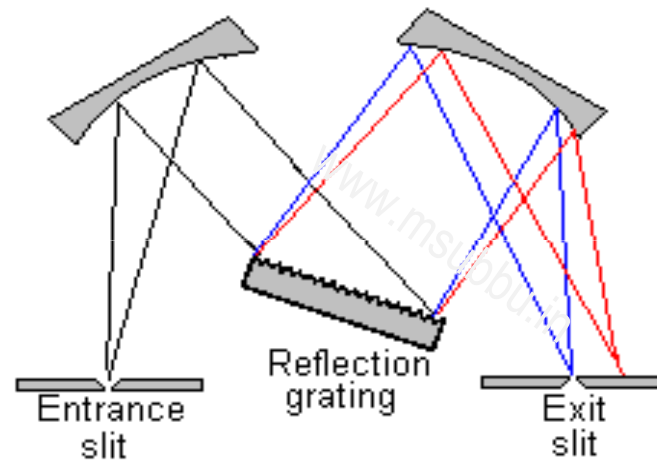
- Sources (UV and visible)
- Wavelength selector (monochromator)
- Sample containers
- Detector
- Signal processor and readout

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Sources of light

- It is important that the power of the radiation source does not change abruptly over its wavelength range.
- UV radiation:
 - Deuterium lamp
 - The electrical excitation of deuterium or hydrogen at low pressure produces a continuous UV spectrum. The mechanism for this involves formation of an excited molecular species, which breaks up to give two atomic species and an ultraviolet photon.
 - Both deuterium and hydrogen lamps emit radiation in the range 160 - 375 nm. Quartz windows must be used in these lamps, and quartz cuvettes must be used, because glass absorbs radiation of wavelengths less than 350 nm.
- Visible radiation
 - Tungsten halogen lamp
 - This type of lamp is used in the wavelength range of 350 - 2500 nm

Monochromator



Sample Containers (Cuvettes)

- The containers for the sample and reference solution must be transparent to the radiation which will pass through them.
- Quartz or fused silica cuvettes are required for spectroscopy in the UV region. These cells are also transparent in the visible region.
- Silicate glasses can be used for the manufacture of cuvettes for use between 350 and 2000 nm.

Detectors

- Photomultiplier tube
- Photodiode array

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Estimation of Iron

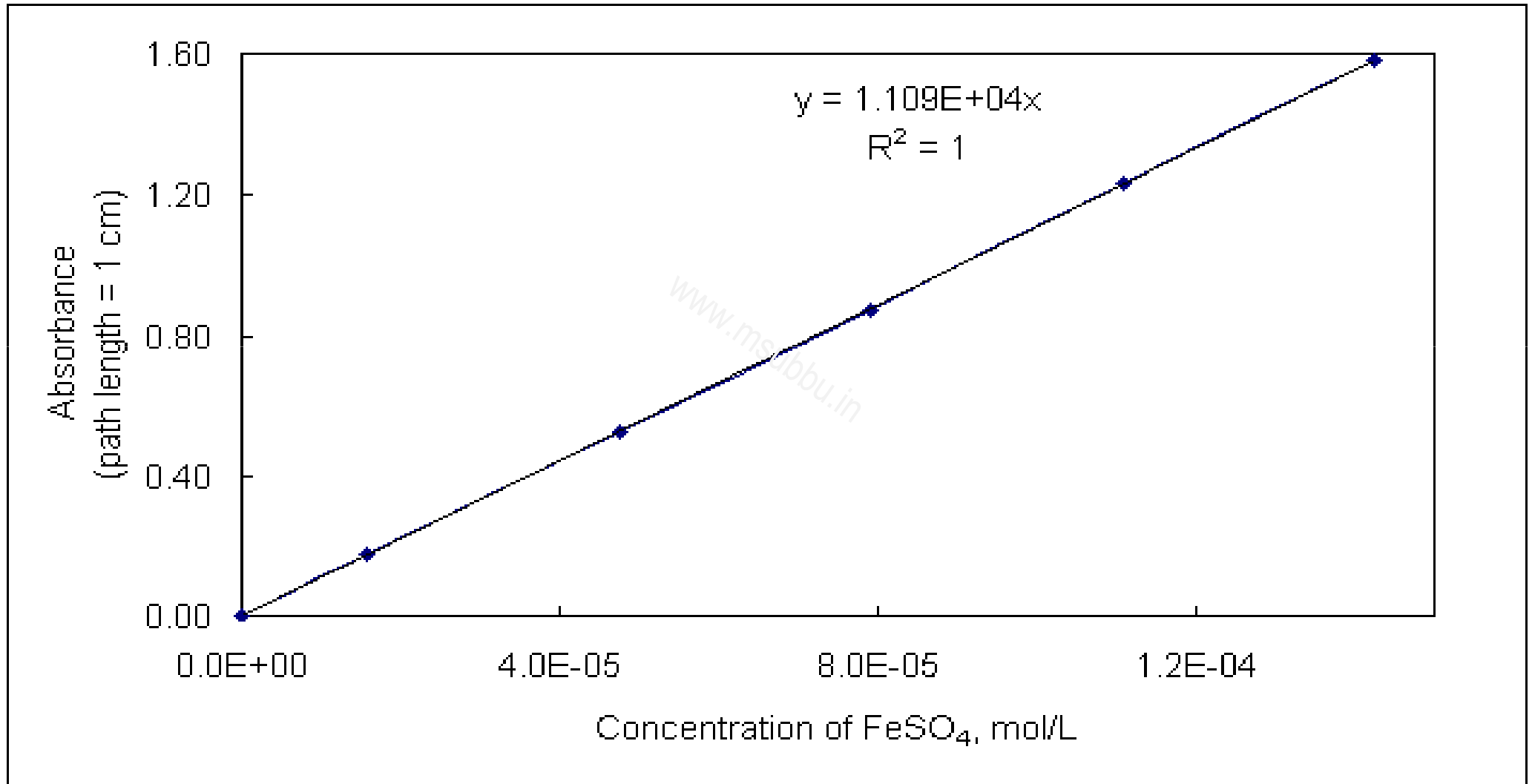
- Fe(II)
- Colouring complex: 1,10-phenanthroline
- Measurement at λ_{\max} of 510 nm



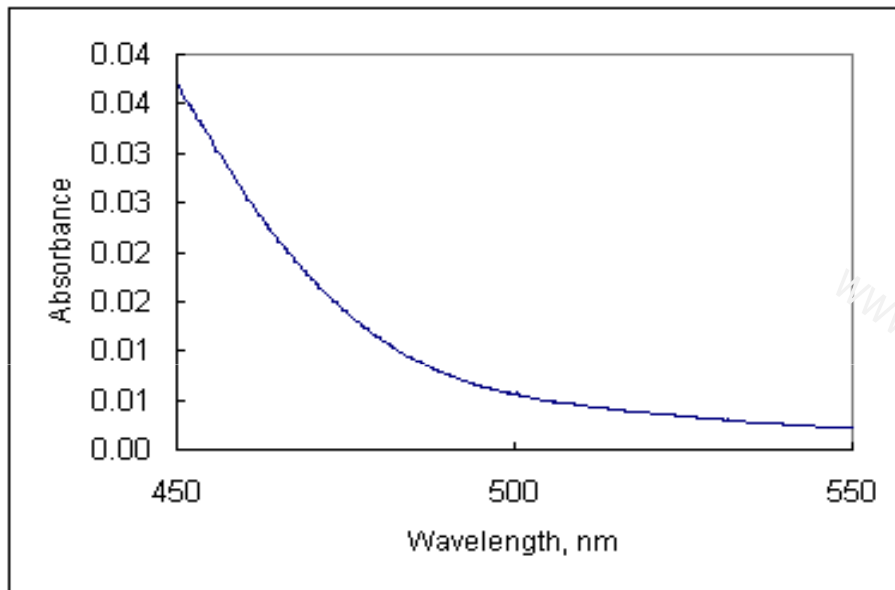
Ferrous sulfate

Conc. Ferrous $\mu\text{mol/L}$	Abs1	Abs2	AvgAbs
0	-		0
15.8	0.1750	0.1764	0.176
47.5	0.5238	0.5236	0.524
79.2	0.8748	0.8750	0.875
111.0	1.2311	1.2309	1.231
143.0	1.5829	1.5828	1.583

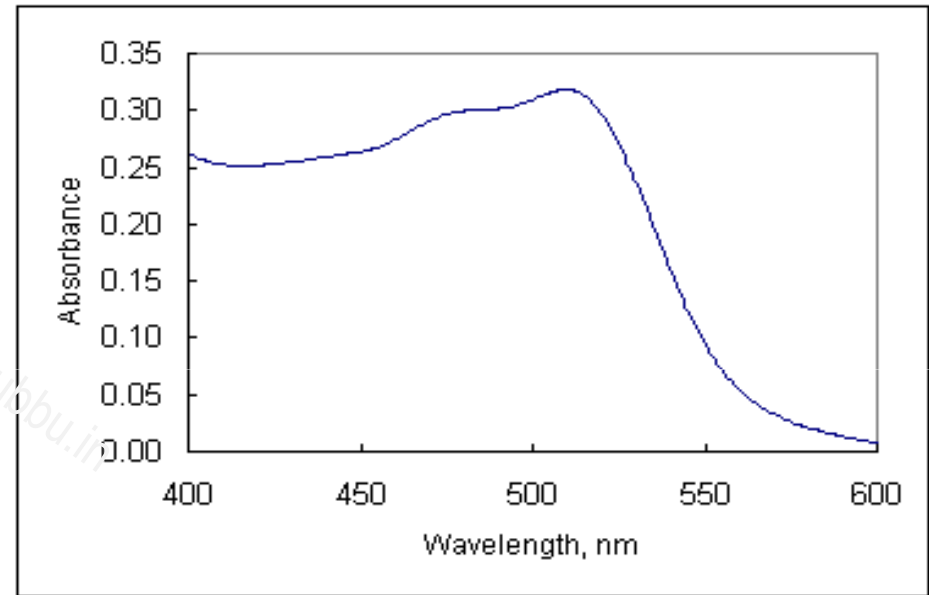
Extinction Coefficient of Fe²⁺-phenanthroline complex



Spectrum of Fe(II)



Blank solution



Fe(II)

Using: Jasco V-530 UV-Vis Spectrophotometer



Estimation of Ni

- NiSO_4 solution (Nickel (II) Sulfate)



Estimation of Ni (contd)

S.No.	Wave length, nm	Absorbance at				
		Con 0.04 mol dm ⁻³	Con 0.05 mol dm ⁻³	Con 0.06 mol dm ⁻³	Con 0.07 mol dm ⁻³	Con 0.08 mol dm ⁻³
1	340	0.05	0.053	0.057	0.071	0.086
2	350	0.057	0.063	0.069	0.084	0.092
3	360	0.086	0.09	0.098	0.119	0.123
4	370	0.116	0.132	0.159	0.187	0.205
5	380	0.172	0.203	0.233	0.275	0.309
6	385	0.180	0.229	0.27	0.324	0.358
7	390	0.185	0.245	0.295	0.347	0.395
8	391	0.197	0.242	0.304	0.356	0.392
9	392	0.20	0.24	0.292	0.357	0.404
10	393	0.204	0.258	0.306	0.361	0.410
11	394	0.19	0.244	0.298	0.36	0.404
12	395	0.188	0.215	0.249	0.295	0.322
13	400	0.185	0.213	0.248	0.292	0.322
14	410	0.156	0.179	0.208	0.248	0.272
15	420	0.112	0.129	0.15	0.178	0.195
16	430	0.072	0.081	0.093	0.109	0.12
17	440	0.048	0.053	0.06	0.069	0.075

Mathpal, S., Kandpal, N.D., E-Journal of Chemistry, 6 (2009) 445-448

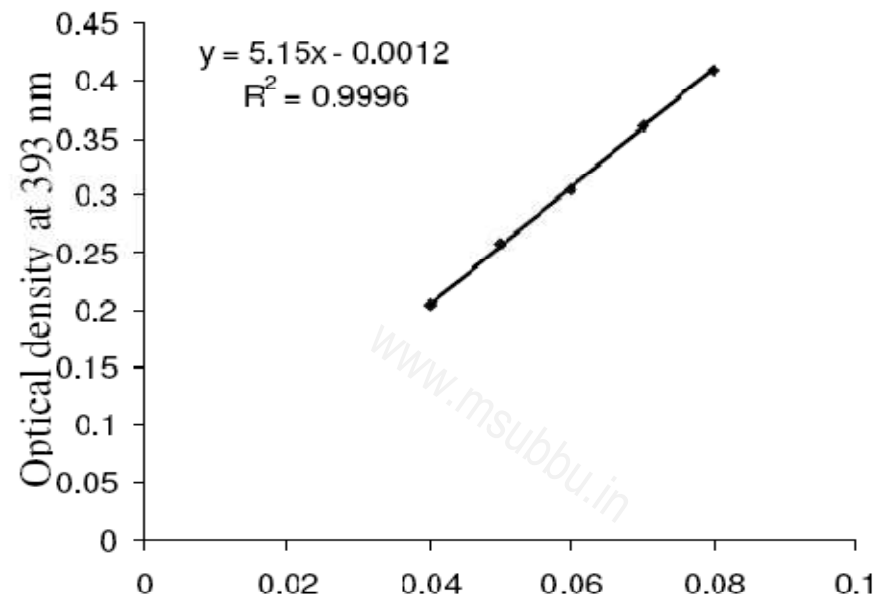


Figure 1. Concentration of nickel sulphate, mol dm⁻³.