# Kandula Srinivasa Reddy Memorial College of Engineering (Autonomous)

Kadapa-516003. AP

(Approved by AICTE, Affiliated to JNTUA, Ananthapuramu, Accredited by NAAC)

(An ISO 9001-2008 Certified Institution)

# Department of Civil Engineering



# Certification Course

on

# Instruments used in environmental pollutant quantification

# **Course Instructor:**

Sri. V.V. Prasad, Assistant Professor, Civil Engg. Dept., KSRMCE

# **Course Coordinators:**

Sri. B. S. Praveen, Assistant Professor, Civil Engg. Dept., KSRMCE

Sri. K. Pramod, Assistant Professor, Civil Engg. Dept., KSRMCE

Date: 17/05/2021 to 02/06/2021



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Lr./KSRMCE/CE/2020-21/

Date: 10-05-2021

#### From

Sri. B. S. Praveen and Sri. K. Pramod, Asst. Professor, Dept. of Civil Engineering, KSRMCE, Kadapa.

To The Principal, KSRMCE, Kadapa.

Sub: Permission to Conduct Certificate Course – Reg.

Respected Sir,

The Department of Civil Engineering is planning to offer a certification course on "Instruments used in environmental pollutant quantification" for B. Tech. students of Civil Engineering. The course will start on 17th May. 2021 and the course will run for a total number of 30 hours. In this regard, I am requesting you to accept the proposal to conduct certification course.

Thanking you

Yours faithfully

(B. S. Praveen and K Pramod)

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Cr./KSRMCE/CE/2020-21/

Date: 12/05/2021

#### Circular

The Department of Civil Engineering is offering a certification course on Instruments used in Environmental Pollutant Quantification. The course will start on 17-05-2021 and the course will run for a total number of 30 hours. In this regard, interested students of Civil Engineering are required to register for the Certification Course. The registration link is given below.

 $\underline{https://docs.google.com/forms/f/g/kse5zsud9xclkdSKseLOasd\ pg3sng7lkMWsszPPsdl3wvkzBps2/viewform}$ 

The Course Coordinators Sri. B. S. Praveen and Sri. K. Pramod, Assistant Professor, Department of Civil Engg.- KSRMCE.

V. s.s. Muly

Principal

Cc to:

The Director, KSRMCE

The HoD-Civil, KSRMCE

**IQAC-KSRMCE** 

Fin Mal K.S.R.M. COLLEGE OF ENGINEERING KADAPA - 516 003. (A.P.)



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# Department of Civil Engineering

Registration list of Certification course

Instruments used in environmental pollutant quantification

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HoD-Civil Engg.

Head
Department of Civil Engineering
K.S.R.M. College of Engineering
(Autonomous)
(Autonomous)
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Syllabus of Certification Course

Course Name: Instruments used in environmental pollutant quantification

**Duration: 30 Hours** 

Module 1:

Introduction, Sampling, Storage, Importance of instruments in Environmental Planning and

management.

Module 2:

Separation Methods: Chromatography, Principle and Chromatogram; Types of Chromatography: Gas

Chromatography, High-performance liquid chromatography, Ion chromatography

Module 3:

Spectrophotometric Methods: Ultraviolet and visible absorption spectroscopy; Double-beam scanning spectrometer; Infrared spectroscopy; Fourier transform infrared spectrometer (FTIR); Attenuated total reflection (ATR); Atomic absorption and flame emission spectroscopy;

Measurements by AAS or by FES Measurements by FES; Hollow cathode lamps; Flame photometers.

Textbooks:

1. Francis Rouessac, Annick Rouessac - Chemical Analysis. Modern Instrumentation Methods and

Techniques-Wiley (2007)

2. Alfred R. Conklin, Introduction to soil chemistry: analysis and instrumentation – Wiley (2005)

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# Department of Civil Engineering

Certification course on "Instruments used in environmental pollutant quantification"

Date	Timing	Course Instructor	Topic to be covered
17/05/21	3 PM to 6 PM	Sri. V.V. Prasad	Introduction, Sampling, Storage
18/05/21	3 PM to 6 PM	Sri. V.V. Prasad	Importance of instruments in Environmental Planning and management.
19/05/21	4 PM to 6 PM	Sri. V.V. Prasad	Separation Methods
20/05/21	4 PM to 6 PM	Sri. V.V. Prasad	Chromatography, Principle and Chromatogram
21/05/21	4 PM to 6 PM	Sri. V.V. Prasad	Types of Chromatography
22/05/21	2 PM to 6 PM	Sri. V.V. Prasad	Gas Chromatography
24/05/21	4 PM to 6 PM	Sri. V.V. Prasad	High-performance liquid chromatography, Ion chromatography
25/05/21	4 PM to 6 PM	Sri. V.V. Prasad	Ultraviolet and visible absorption spectroscopy
27/05/21	4 PM to 6 PM	Sri. V.V. Prasad	Double-beam scanning spectrometer
28/05/21	4 PM to 6 PM	Sri. V.V. Prasad	Infrared spectroscopy
29/05/21	4 PM to 6 PM	Sri. V.V. Prasad	Fourier transform infrared spectrometer (FTIR); Attenuated total reflection (ATR)
1/06/21	4 PM to 6 PM	Sri. V.V. Prasad	Atomic absorption and flame emission spectroscopy; Measurements by AAS or by FES Measurements by FES;
2/06/21	4 PM to 6 PM	Sri. V.V. Prasad	Hollow cathode lamps; Flame photometers

Instructor: V. Ven
Coordinators: Ugrapud;
Bap

V. S. S. MWK Principal

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## Report of

# Certification Course on Instruments used in environmental pollutant quantification

# From 17/05/2021 to 02/06/2021

Target Group

Students

:

**Details of Participants** 

53 Students

Co-coordinator(s)

Sri. B. S. Praveen and Sri. K. Pramod

Organizing Department

Civil Engineering

Venue

Online (google meet)

Link: https://meet.google.com/lookup/ckseksjd38

#### Description:

The Department of Civil Engineering offered the Certification Course in "Instruments used in environmental pollutant quantification" from 17/05/2021 to 02/06/2021 and the course was organized for a total number of 30 hours. The course was instructed and coordinated by V.V. Prasad (Assistant Professor, Dept. Civil Engg.).

Environmental testing can identify and quantify compounds and pollutants in air, water, or soil. Alternatively, environmental testing can verify that a product or piece of equipment will perform as expected once it is out in the world. This can take the form of climate testing (e.g. temperature or humidity) or mechanical testing (e.g. measuring shock or vibration). Environmental test equipment is used in a variety of environments including: Environmental Chemical Testing, Agriculture, Public health and safety, Field testing, Petroleum, among others. The course is designed for the students to learn about the advanced machinery used in Environmental Engineering.

Photo:



V.√e√ (Course Instructor)

(HoD, Civil Engg.)

Head Department of Civil Engineering K.S.R.M. College of Engineering (Autonomous) KADAPA 516 003. (A.P.)

V. S. S. Muly **Principal** 

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# DEPARTMENT OF CIVIL ENGINEERING

# Certificate Course

on

"Instruments used in Environmental Pollutant Quantification"

# Resource Person:

Sri V V Prasad

**Assistant Professor** 

**Department of Civil Engineering** 

**Coordinators:** 

Sri B. S. Praveen and Sri K. Pramod



17-05-2021 02-06-2021

# Department of Civil Engineering

Attendance sheet of Certification course on Instruments used in Environmental Pollutant Quantification

SI. N	Student Roll No.	Student Name	17/ 05	18/ 05	19/ 05	20/ 05	21/ 05	22/ 05	24/ 05	25/ 05	27/ 05	28/ 05	29/ 05	1/ 06	2/ 06
1	179Y1A0106	Nikhil Kumar Reddy Bhavanasi	~	V	~	V	a	V	V	V	V	V	a	V	V
2	179Y1A0107	Geetha Bhuma	V	V	V	V	V	V	V	V	V	1	V	V	V
3	179Y1A0113	Venkatesh Naik Bukke	V	V	V	v	V	L	V	L	V	V	L	L	V
4	179Y1A0115	Pallavi Chatta	V	V	V	a	V	V	V	~	V	V	V	a	V
5	179Y1A0119	Zaheer Dade	V	V	V.	V	V	V	V	V	V	V	V	L	v
6	179Y1A0122	Jayachandra Derangula	V	V	V	OI	V	V	V	V	V	V	V	V	V
7	179Y1A0123	Siddaiah Dollu	1	V	V	V	V	V	V	V	V	V	V	v	V
8	179Y1A0124	Suresh Gowd Ediga	a	~	V	V	V	V	V	V	V	V	V	V	V
9	179Y1A0126	Ashok Kumar Galeti	V	V	V	V	V	V	V	Q-	V	V	V	V	V
10	179Y1A0129	Pullaiah Gokula	V	a	V	V	V	V	V	V	V	V	V	V	V
11	179Y1A0130	Ramamanohar Reddy Gollapalle	V	V	ν		V	V	V	V	V	V	V	V	V
12	179Y1A0131	Sreekanth Gurakanivari	V	V	V	a	V	V	V	V	V	ν	V	V	a

			Dharani													
	13	179Y1A0133	Jonnavaram	V	~	V	V	V	V	L	V	V	V	V	a	L
	14	179Y1A0134	Manasa Juturu	V	V	V	V	V	V	V	V	V	V	V	V	a
	15	179Y1A0136	Himaja Kancharla	V	V	V	V	V	V	V	V	V	V	V	V	v
	16	179Y1A0139	Vivekananda Reddy Kota	V	V	V	V	V	V	V	V	V	V	v	v	v
A 4	17	179Y1A0147	Thimmappa Madiga	OI	V	V	v	V	v	a	V	V	· V	1	v	U
	18	179Y1A0155	Manjunatha Muttalahgari	V	v	V	V	~	V	v	v	v	V	V	V	1
	19	179Y1A0157	Prathima Nagooru	Ca	V	V	V	O1	V	V	V	V	L	V	a	1
	20	179Y1A0158	Vijaya Kumari Nalla	V	V	V	V	V	V	V	L	V	~	V	v	L
	21	179Y1A0174	Bhanu Prakash Peddaalankolla	V	V	/	V	a	V	V	L	V	~	L	V	-
	22	179Y1A0182	Susma Saraballa	V	V	V	V	V	V	V	V	V	V	V	L	1
	23	179Y1A0184	Abdul Rehaman Shaik	V	V	G	V	V	V	V	L	/	V	V	V	-
	24	179Y1A0197	Surya Thammisetty	V	v	V	~	V	V	V	V	V	0	V	V	V
N v	25	179Y1A0198	Rajesh Thotakanama	v	V	V	V	V	V	V	V	~	V	ν	V	V
	26	179Y1A01A1	Maheswari Undela	V	V	V	a	V	V	V	~	V	V	V	V	V
	27	179Y1A01A2	Siva Kumar Upparapalli	V	V	V	V	V	V	V	v	V	~	~	V	V

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28	179Y1A01A5	Vodiveeti	a	V	L	V	~	~	L	V	1	V	a	V	1
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30	179Y1A01A7	Lokesh Yarragolla	V	V	V	~	V	V	V	V	V	V	V	V	L
31	179Y1A01A8	Venkata Lakshmi Yarraguntla	V	V	V	V	V	V	V	V	ı	V	V	V	L
32	189Y5A0102	Siva Gangadhar Alavalapadu	V	V	V	V	G	V	V	c	~	v	V	1	
33	189Y5A0104	Dharani Kamalakara Rao Appalarajugari	v	V	W	V	V	V	a	V	v	L	C	L	-
34	189Y5A0107	Suresh Banka	V	V	V	V	V	V	V	V	レ	V	C	V	ι
35	189Y5A0109	Srinatha Reddy Bhumireddy	V	V	V	a	~	V	V	V	V	V	V	V	ı
36	189Y5A0110	Purushothamreddy Bijivemula	V	V	~	V	V	V	V	V	a	V	V	a	1
37	189Y5A0113	Kiran Kumar Bolleddu	V	v	V	V	V	v.	V	V	V	V	V	V	
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39	189Y5A0126	Shireesha Guramkonda	~	V	V	V	V	V	v	W	V	1	L	L	,
40	189Y5A0127	Nikitha Jaladi	V	v	V		V	/	ci	V	V	L	V	1	L
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42	189Y5A0132	Yaswanth Reddy Kambham	V	V	V	V	V	V	V	V	V	G-	~	V	7 ~
43	189Y5A0134	Sree Hari Reddy Katthi	~	V	V	V	V	V	v	v	0	a	V	v	C
44	189Y5A0135	Nagesh Kolliboina	V	V	V	e	V	V	v	V	V	V	V	V	V
45	189Y5A0138	Prasanth Kumar Kotturu	V	V	V	V	V	V	V	L	V	v	V	V	r
46	189Y5A0140	Suresh Kuruva	V	a	V	V	V	V	V	V	V	v		V	v
47	189Y5A0146	Venkata Ramana Reddy Nimmakayala	V	a	V	V	V	v	V	V	V	V	V	v	a
48	189Y5A0159	Abilash Reddy Sajjala	V	V.	a	V	V	V	V	V	V	ı	V	v	V
49	189Y5A0162	Naveen Kumar Sepuri	v	~	V	V	V	V	V	1	α	V	a	V	V
50	189Y5A0170	Chandra Kanth Thatamsetty	V	V	V	V	V	V	a	v	v	V	~	V	V
51	189Y5A0171	Divya Thonduru	V	V	V	V	V	V	V	V	~	V	V	V	
52	189Y5A0172	Parameswara Reddy Thummala	V	V	a	V	V	V	v	V	V	V	V	a	V
53	189Y5A0179	Narendra Kumar Reddy Yanamala	V	/	V	V	V	V	r	V	V		V	v	V

HoD-Civil Engg

Department of Civil Engineering
K.S.R.M. College of Engineering
(Autonomous)

KADAPA 516 003. (A.P.)

# Instruments Used in

Environmental Pollutant

Quantification.

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# Introduction

Environment is fragile. One cannot expect environment to be same over time. Anthropological activities have always changed the environment so much that there is phenomenal difference in its initial and current status. To quantify how much the nature has changed, it is important to know the difference between the initial condition of a parameter and the final quantity of the same parameter.

This can be done only with the help of instruments. Instrumentation plays a pivotal role in quantification of pollutants. The pollutants may be any where as simple as macroscopic particle to molecules of radioactive elements. It is unwise to try and learn about all the instruments in detail. This course however aims to tach you the basics of instruments that are used in environmental research.

Pollutants can be present anywhere and in any phase i.e., gas, liquid and solid. It is also possible for the pollutants to exist in other combinations of material phases such as aerosols, emulsions and suspensions. The first step in the quantification is sampling itself.

# Sampling

There are two types of water sampling strategies regarding the time frame when the samples are collected:

- (1) discrete samples and
- (2) composite samples

Discrete sample, also known as grab sample, is a single sample collected in an individual container. The sample is representative of the chemistry only at the time and place at which the

sample was taken. The time period is generally defined to be less than 15 min. Thus, discrete samples are appropriate when the sample composition is not time dependent.

Composite sample consists of a series of smaller samples collected at a predetermined time or after predetermined flow and mixed in the same container.

It is the choice of the researcher to determine what sample they have to use.

# Storage

After sampling, storing the sample properly is equally important. The samples depending on their compositions and phases, may be immediately analysed or stored under 4°C till the analysis is complete. Each and every sample has to be stored separately in separate container. No sample should intermix with other sample. It should not posess the potential to leak and contaminate other samples.

# Separation Methods

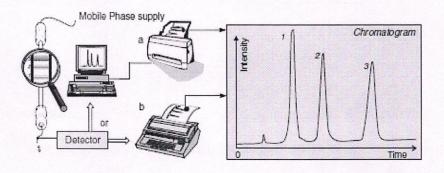
# Chromatography

Chromatography is a physico-chemical method of separation of components within mixtures, liquid or gaseous, in the same vein as distillation, crystallization, or the fractionated extraction. Chromatography, the process by which the components of a mixture can be separated, has become one of the primary analytical methods for the identification and quantification of compounds in the gaseous or liquid state.

# Principle:

The basic principleis based on the concentration equilibrium of the components of interest, between two immiscible phases. One is called the stationary phase, because it is is is is is immobilized within a column or fixed upon a support, while the second, called the mobile

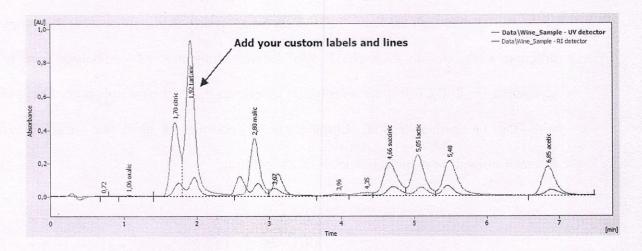
phase, is forced through the first. The phases are chosen such that components of the sample have differing solubilities in each phase. The differential migration of compounds lead to their separation. Of all the instrumental analytical techniques this hydrodynamic procedure is the one with the broadest application. Chromatography occupies a dominant position that all laboratories involved in molecular analysis can confirm.



Basic principle of Chromatography

# Chromatogram

The chromatogram is the representation of the variation, with time (rarely volume) of the amount of the analyte in the mobile phase exiting the chromatographic column. It is a curve that has a baseline which corresponds to the trace obtained in the absence of a compound being eluted. The separation is complete when the chromatogram shows as many chromatographic peaks as there are components in the mixture to be analysed.



Sample chromatogram

There are different types of chromatographic techniques that are actually for different samples.

Various forms of chromatography will be discussed in the following sections.

# Gas Chromatography

Gas chromatography (GC) is a widely used technique whose first applications date back more than 60 years. Since then, development has continued making the best use of the extreme sensitivity, versatility, the possibilities of automation and the ease with which new analyses can be developed. Because separation of compound mixtures on the column occurs while they are in the gaseous state, solid and liquid samples must first be vaporized. This represents, without hesitation, the greatest constraint of gas phase chromatography and weighs against it, since its use is limited to the study of thermostable and sufficiently volatile compounds. However, the applications are numerous in all domains and the development of high speed or multidimensional gas chromatography make this technique even more attractive. Its very great sensitivity permits detection of quantities of the order of picograms for certain compounds.

## Components:

A gas chromatograph is composed of several components within a special frame. These components include the injector, the column and the detector, associated with a thermostatically controlled oven that enables the column to attain high temperatures.



#### Typical Gas Chromatograph

#### Mobile Phase:

The mobile phase is a gas (helium, hydrogen or nitrogen), either drawn from acommercially available gas cylinder or obtained, in the case of hydrogen ornitrogen, from an on-site generator.

## Injector:

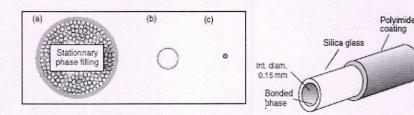
The injector is the sample's entrance to the chromatograph.

# Thermostatically controlled oven:

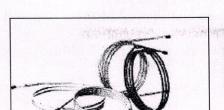
The gas chromatograph comprises an oven with sufficient volume to hold one ortwo columns easily and which can heat up to more than 400°C.

#### Columns:

There are two column types, which differ in their performance: packed columnsand capillary columns. For packed columns the stationary phase isdeposited or bonded by chemical reaction onto a porous support. For capillary columns a thin layer of stationary phase is deposited onto, or bound to the innersurface of the column.



Columns a) Stationary Column, b) 530 Column, c) 0.2 mm Column.





Columns Packed and Thin layer

### Detectors:

Some detectors are universal; that is they are sensitive to practically everycompound that elutes from the column. On the other hand, there are discriminating(selective) detectors that are sensitive only to specific compounds, yielding very uncomplicated chromatogram.

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## Thermal conductivity detector (TCD)

This general purpose and non-destructive detector, in use since the early days of GC, has for a long time remained a mainstay of the technique. Miniaturization has led to it being used as much for packed columns as for capillary columns. Of moderate sensitivity (400 pg/mL carrier gas) when compared with other detectors, it possesses nevertheless a very large linear range (six orders of magnitude). Its operating principle relies on the thermal conductivity of gas mixtures as a function of their composition.

### Flame ionization detector (FID):

Considered as almost universal for organic compounds this is effectively the detector par excellence, of GC. The gas flow issuing from the column passes throughthe flame of a small burner fed by a mixture of hydrogen and air. The detector destroys the organic compound present

whose combustion results in the releaseof ions and charged particles responsible for the passage of a very weak current10–12 A between two electrodes (pd of 100 to 300 V). One end of the burner, heldat ground potential, acts as a polarization electrode while the second electrode, called the collector, surrounds the flame rather like a collar. An electrometer amplifies the signal to a measurable voltage.

#### Photo-ionization detector (PID)

This detector is fairly selective but it has only a narrow range of application, convenient for hydrocarbons as well as for S or P derivatives. The operating principle consists to provoke ionization of the analytes by irradiation with a UV lamp emitting photons of high energy

#### Nitrogen phosphorus detector (NPD)

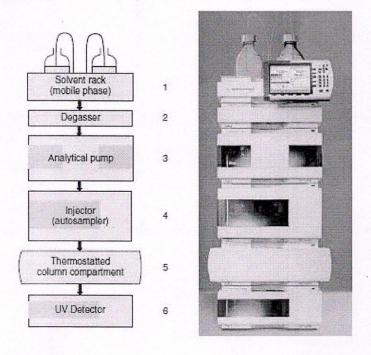
Compared with the FID, this thermoionic detector has a smaller flame in whichthe catalytic decomposition of compounds containing nitrogen (N), or phosphorus(P) yields, fairly specifically, negative ions which are received by a collectorelectrode. It comprises asmall ceramic cylinder doped with an alkaline salt (e.g. rubidium sulfate). Avoltage is applied to maintain a small plasma 800°C through the combustionof an air/hydrogen mixture. In these conditions the nitrogen present in airdoes not yield ions. Detector sensitivity is typically between 0.1 and 0.4 pg/s fornitrogen- or phosphorus-containing analytes, with a linear range of five orders ofmagnitude.

There are many other detectors in use.

# High-performance liquid chromatography

Of all the chromatographic techniques whose mobile phase is a liquid, high performance liquid chromatography (HPLC) is perhaps the best known. Its field of applications overlaps a large section of gas phase chromatography, to which can be added the analyse of many compounds that are thermolabile, or very polar or of high molecular weight. Its success is also due to the possibility, for the chromatographer, to act in a very precise manner upon the selectivity between compounds through an appropriate choice of columns and eluent composition by exploiting the solute/mobile phase/stationary phase interactions. Although the efficiency of the HPLC columns is less than those used for GC, new stationary phases that can operate in several modes such as ion pairing or increasing hydrophobic interactions, reveal further possibilities of HPLC. Finally, miniaturization of the technique (nanochromatography) has facilitated its working association with mass spectrometry.

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Typical HPLC arrangement

# The beginnings of HPLC

High-performance liquid chromatography, often called simply by its abbreviation, HPLC constitutes a general purpose analytical technique derived from the most ancient form of preparative liquid chromatography. The modern day technique is greatly enhanced in terms of selectivity, resolution, through miniaturization and the use of very elaborate stationary phases.

### Pumps

All HPLC systems include at least one pump to force the mobile phase through the column whose packing is fairly compact. The result of this is a pressure increaseat the injector which can attain 20 000 kPa (200 bars) depending upon the flowrate imposed upon the mobile phase, its viscosity, and the size of the particles of the stationary phase. Pumps are designed in order to maintain a stable flow rate, avoiding pulsations even when the composition of the mobile phase varies. These flow rate metered pumps contain, in general, two pistons in series, working in opposition, to avoid interruptions to the flow rate

# Injectors

In HPLC, the injection of a precise volume of sample onto the head of the columnmust be made as fast as possible in order to cause the minimum disturbance

#### Columns

The column is a straight stainless steel calibrated tube which measures between 3 and 15 cm in length and whose the inside wall is sometimes coated with aninert material such as glass.

#### Detectors:

## Spectrophotometric detectors

Detection is based upon the Lambert-Beer law, The absorbance of the mobile phase is measured at the outlet of the column, at one or several wavelengths in the UV or visible spectrum.

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#### Fluorescence detector

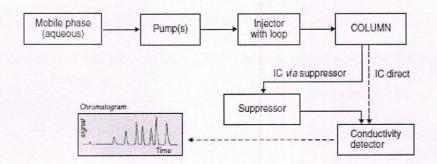
About 10 per cent of organic compounds are fluorescent, in that they have the ability to re-emit part of the light absorbed from the excitation source. The intensity of this fluorescence is proportional to the concentration of the analyte, as long as this concentration is kept low. Application to LC chromatography gave rise to fluorescence detectors, very sensitive and for this reason often used for trace analysis.

#### Refractive index detector (RI detector)

This type of detector relies on the Fresnel principle of light transmission through atransparent medium of refractive index n. It is designed to measure continuouslythe difference in the refractive index between the mobile phase ahead of andfollowing the column. So a differential refractometer is used. Schematically, abeam of light travels through a cell that has two compartments: one is filled withthe pure mobile phase while the other is filled with the mobile phase eluting the column. In practice, the optical dispersions of the media are likelyto differ, and consequently the refractive index will only match at one particular wavelength. this is used to quantify the pollutant or eluent.

# Ion chromatography

Ion chromatography (IC) is a separation technique which shares numerouscommon features with HPLC, yet possesses sufficient novel aspects such as its principle of separation or modes of detection, to make it the object of a separatestudy. IC is adapted to the separation of ions and polar compounds. The mobilephase is composed of an aqueous ionic medium and the stationary phase is anion-exchange resin.



Algorithm of IC

Besides the detection methods based on absorbance or fluorescence,ion chromatography also uses electrochemical methods based on the ionicnature of the species to be separated. Its greatest utility is for analysis of anionsfor which there are no other rapid analytical methods. Yet current applications of IC are far broader than the analysis of simple ions by which the technique first gained renown. The operating domain, comparable with that of capillary electrophoresis, concerns the separation of many kinds of inorganic or organics pecies such as amino acids, carbohydrates, nucleotides, proteins and peptides incomplex matrices.

### Basics of ion chromatography

This chromatographic technique is concerned with the separation of ions and polar compounds. Stationary phases contain ionic sites that create dipolar interactions with the analytes present in the sample. If a compound has a high charge density, it will be retained a longer time by the stationary phase. This exchange process is much slower when compared with those found in other types of chromatography. This mechanism may be associated, for molecular compounds, with those already dealt with by HPLC when equipped with RP-columns.

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## Stationary phases

Ion chromatography can be subdivided into cation exchange chromatography, in which positively charged ions bind to a negatively charged stationary phase and anion exchange chromatography, in which the negatively charged ions bind to a positively charged stationary phase. The column packings consist of a reactive layer bonded to inert polymeric particles. Stationary phases must satisfy implicitly a number of requirements as narrow granulometric distribution (mono-disperse), large specific surface area, mechanical resistance, stability under acid and basic pHs and rapid ion transfer.

#### Polymer-based materials

The best-known stationary phases are issued from copolymers of styrene and divinylbenzene, in order to obtain packings hard enough to resist pressure in the column. They are made of spherical particles with diameters of 5 to 15m that are modified on the surface in order to introduce functional groups with acidic or basic properties.

For cation separation the cation-exchange resin is usually a sulfonic or carboxylic acid. Thus, concentrated sulfuric acid is used to attack the accessible aromatic rings of the copolymer surface to link SO<sub>3</sub>H functional groups. A strongly acidic phase is obtained – for cation exchange.

### Silica-based materials

Porous silica particles can serve to support, through covalent bonding, alkylphenyl chains carrying sulfonated groups or quaternary ammonium groups. This fixation step is similar to that used to obtain bonded silica phases developed in HPLC.

## Resin films

A polymer called 'latex', prepared from a monomer that contains organic groups, is deposited as an array of tiny beads on a waterproof support to form a continuous film-like layer about  $1-2\mu m$  thickness.

## Mobile phases:

IC mobile phases are usually 100 per cent aqueous with organic or inorganic buffers to control selectivity and when necessary a small content of methanolor acetone used to dissolve certain samples. The pH is adjusted according to the separation to be achieved.

# Conductivity detectors:

Besides the spectrophotometric detectors based on absorbance or fluorescence of UV/visible radiation, and used when the mobile phase does not absorb appreciably, another mode of detection exists based upon electrolyte conductivity. Thus, at theoutlet of the column, the conductance (the inverse of the resistance) of the mobile phase is measured between two microelectrodes. The measuring cell should be of avery small volume.

## Ion suppressors

The mobile phase contains ions that create a background conductivity, making it difficult to measure the conductivity due only to the analyte ions as they exit the column. To improve the signal to noise ratio, when using a conductivity detector, a device called a suppressor, designed to

selectively remove the mobile phase ions isplaced after the analytical column and before the detector.

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# Spectrophotometric Methods

Visual colorimetry, one of the most ancient of analytical methods was already being used in the times of the Greeks and Romans although it began to take on a more modern scientific character when, in 1729, Pierre Bouguer postulated that 'if a given width of coloured glass absorbs half of the light issuing from a source, then a double width will reduce the light to a quarter of its initial value'.

Some 30 years later, Jean-Henri Lambert (1728–1777) proposed the first mathematical relationship 'the logarithm of the decrease in light intensity (today we would say the inverse of the transmittance) is equal to the product of the opacity of the medium times its thickness'. Finally in 1850, Auguste Beer established a relation between concentration and optical density now called absorbance), which led to the current form of the Beer–Lambert law (also called Lambert–Beer law or even Lambert–Beer–Bouguer law).

# Ultraviolet and visible absorption spectroscopy

The absorption by matter of electromagnetic radiation in the domain ranging from the near ultraviolet to the very near infrared, between 180 and 1100 nm, has been studied extensively. This portion of the electromagnetic spectrum, designated as the 'UV/Visible' since it includes radiation perceptible to the human eye, generally yields little structural information but is very useful for quantitative measurements. The concentration of an analyte in solution can be determined by measuring the absorbance at some wavelength and applying the Lambert– Beer Law. The method is known as colorimetry, considered as the workhorse of many laboratories. Colorimetry applies not only to compounds possessing an absorption spectrum in that spectral region but equally to all those compounds which, following modification by specific reagents, lead to derivatives which permit absorption measurements.

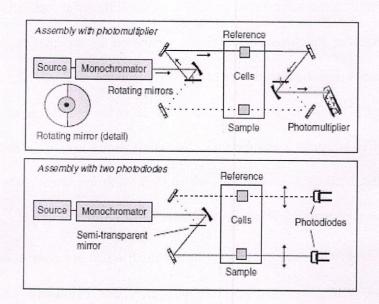
This can be achieved by a whole range of instruments, from colour comparators and other basic colorimetric devices to automated spectrophotometers that can carry out multi-component analyses. Liquid chromatography and capillary electrophoresis have favoured the development of improved UV/Vis detectors which reflects the current trend for acquiring chromatograms giving at the same time information concerning the nature and quantification of compounds.

## The UV/Vis spectral region

This region of the spectrum is conventionally divided into three sub-domains termed near UV (185–400 nm), visible (400–700 nm) and very near infrared (700–1100 nm). Most commercial spectrophotometers cover the spectral range of 185 to 900 nm. The lower limit of the instrument depends upon the nature of the optical components used and of the presence of air, oxygen and water vapour along the optical path which absorb intensity below 190 nm.

#### Instrumentation in the UV/Visible

A spectrophotometer is designed around three fundamental modules: the source, the dispersive system (combined in a monochomator), which constitute the optical section and the detection system.



UV VIS Spectrophotometer

These components are typicallyintegrated in a unique framework to make spectrometers for chemical analysis. Asample compartment is inserted into the optical path either before or after the dispersive system depending upon the design of the instrument.

#### Light sources

All spectrometers require a light source. More than one type of source can be used in the same instrument which automatically swap lamps when scanning between the UV and visible regions: For the visible region of the spectrum, an incandescent lamp fitted with atungsten filament housed in a silica glass; for the UV region a deuterium arc lamp working under a slight

pressure; alternatively, for the entire region 200 to 1100 nm, a xenon arc lamp can be used for routine apparatuses.

Land Mark Strategy

Dispersive systems and monochromators

#### Sequential instruments

The light emitted by the source is dispersed through either a planar or concave grating which forms part of a monochromator assembly. This devicepermits the extraction of a narrow interval of the emission spectrum. Thewavelength or more precisely the width of the spectral band, which is afunction of the slit width, can be varied gradually by pivoting the grating. Optical paths with long focal lengths (0.2 to 0.5 m) yield the bestresolution.

#### Simultaneous instruments

This category of instrument functions according to the spectrograph principle. The light beam is diffracted after travelling through the measuring cell.

#### Detectors

The detector converts the intensity of the light reaching it to an electrical signal. It is by nature a single channel device. Two types of detector are used, either a photomultiplier tube or a semiconductor (charge transfer devices or silicon photodiodes). For both of which the sensitivity depends upon the wavelength.

#### UV/Vis spectrophotometers

UV/Vis spectrometers are classified into three optical designs: a fixed spectrometer with a single light beam and sample holder; a scanning spectrometer with double light beams and two sample holders for automatic measurement of absorbance; and a non-scanning spectrometer with an array detector for simultaneous measurement of multiple wavelengths.

#### Single-beam mono-channel optical spectrometers

Many routine measurements are conducted at fixed wavelengths by basic photometers fitted with interchangeable interference filters or simple grating monochromators. In single-beam instruments, obtaining a spectrum requires measuring the transmittance of the sample and of the solvent at each wavelength. A control corresponding to the solvent alone or a solution containing the reagents of the measurement (but without the compound to be measured, an analytical blank), is first placed in the optical path, then is replaced by the solution prepared from the sample of unknown concentration. These instruments sometimes have a built-in electronic compensation arrangement for variations in light source intensity known as a split-beam. A part of the light beam is diverted before it reaches the sample permitting the stabilization of the source intensity (this is not a true reference beam). These instruments yield absorbance and led to the analyte concentration.

#### Array-detector spectrophotometers

This type of instrument resembles a spectrograph closely since it allows the simultaneous recording of all wavelengths of the spectrum.

#### Double-beam scanning spectrometer

The double-beam design greatly simplifies the process of the single-beaminstrument by measuring the transmittance of the sample and solvent almost simultaneously. One beam passes through the sample while the other passes through the reference solution. Most spectrometers use one (or two) mirroredrotating chopper wheel to alternately direct the light beam through the sampleand reference cells. This permits the detector to compare the two intensities transmitted by reference or sample solutions for the same wavelength

#### Infrared spectroscopy

Analytical infrared studies are based on the absorption (or reflection) of theelectromagnetic radiation that lies between 1 and 1000μm. This is one of themost common spectroscopic techniques used for compound identification andmeasuring of concentrations in many samples. This spectral range is sub-divided into three smaller areas, the near infrared (near-IR, 1-2.5 µm), the mid infrared(mid-IR, 2.5-50µm) and the far infrared (beyond 25µm). Although the near-IRis poor in specific absorptions, it is considered as an important method by qualitycontrol laboratories for quantitative applications. By contrast the mid-IR regionprovides more information upon the structures of compounds and consequently it is much used as a procedure for identifying organic compounds for which itremains a form of functional group fingerprinting. The far IR requires the useof specialized optical materials and sources. To conduct these analyses there is afull range of instruments from Fourier transform spectrometers to a multiplicity of analysers of dispersive or non-dispersive types, specialized in the measurement of pre-defined compounds (e.g. analysis of gases and vapours) or which allowcontinuous analyses on production lines. The Fourier transform infrared spectrometry It offers numerous possibilities for the treatment of spectra and has applications for the analysis of structured microsamples (infrared microanalysis).

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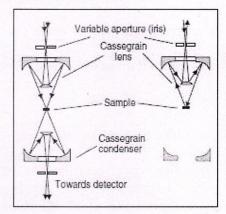
#### Absorptions in the infrared

Infrared absorption information, which varies with the radiation wavelengthsselected, is generally presented in the form of a spectrum, which is the basicdocument issued from the spectrometer. The ordinate of the graph records theratio of the transmitted intensities, that is with and without sample, calculated foreach wavelength marked on the abscissa.

#### Fourier transform infrared spectrometer (FTIR)

An FTIR spectrometer contains a single beam optical assembly with, as an essential component, an interferometer – often of the Michelson type – located between the source and the sample (Figure 10.7c). It consists of three active components:

- · a moving mirror,
- · a fixed mirror and
- a beam-splitter.





**FTIR** 

The radiation issuing from the polychromatic source impacts on the beam-splitteracting as a separator, which is made of a semi-transparent film of germanium deposited on a KBr support. This device divides the original beam into two halves, one of which is directed towards a fixed mirror and the other towards a moving mirror whose distance from the beam-splitter varies. Recombined, these two beams follow the same optical path passing through the sample before reaching the detector that measures the global light intensity received. This is amultiplexing procedure applied here to the domain of optical signals. The heart of the Michelson interferometer is a moving mirror, the sole mobile component that oscillates between two

extreme positions. When its position is suchthat the pathways travelled by the two beams have the same length until the detector (zero optical path difference), then the light composition of the beamleaving the interferometer is identical to that which enters. In contrast, when themobile mirror changes of this particular position, the light leaving has a spectral composition which depends upon the phase difference of the two beams: the signal transmitted over time by the detector is recorded in the form of an interferogram.

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This FTIR technique of obtaining spectra, also adapted to the near-IR, hassignificantly modified traditional procedures of obtaining IR spectra. Adopted by almost all manufacturers of spectrometers

This method hasseveral advantages:

The entrance slit is replaced by an iris furnishing a better signal to the detectorwhich receives more energy (multiplexing advantage);

The signal-to-noise ratio is much higher to that of the sequential method since itcan be improved by the accumulation of successive scans (Fellgett advantage);

#### Light sources

In the mid-IR several type of sources are used. They are either a lamp filamentor a hollow rod, 1–3mm in diameter and 2 to 4 cm long, made of fused mixtures of zirconium oxide or rare earth oxides (Nernst source) heatedby Joule effect by the means of an internal resistor.

#### Detectors

The detection of photons in the infrared region was difficult to achieve for along time and a major cause of this was the mediocre sensitivity of the firstspectrophotometers. The principle

relies upon the thermal effect of IR radiation. Sensors that measureradiation by means of the change of temperature of an absorbing material are classified as thermal detectors.

Numerous devices have been used, thermistors, thermocouples, thermopiles and other sensors rather imaginative. For FTIR instruments, one of the most common thermal sensors is the pyroelectric detector.

#### Transmission procedures

Gases, for which the absorbances are weak, require cells for which the optical pathway can be very long, at least of several centimetres but sometimes reaching several hundreds of metres through a complex arrangement of mirrors giving multiple reflections of the infrared beam inside the cell volume then becomes important (0.2 L).

In contrast, for the hyphenated techniques such as GC/IR threadlike gas cells(light pipes) whose volume does not exceed a few tens of microlitres (l=10cmand diameter <1mm) are used. These cells use gilded side walls to provokemultiple reflections.

Liquids are usually analysed with cells, which have two dismountable IRwindows. For qualitative analysis, a droplet of the sample is compressed between two NaCl or KBr discs without spacers to create a film. For quantitative analysis, depending upon the wavelength of the measurement, either quartz cells (with optical pathway of 1 to 5 cm), or sealed cells that have a variable or fixed path length can be used. The optical path length must be calibrated and periodically controlled.

Solids are more difficult to study. If the solid can be dissolved in a suitable solvent, the same method as for aliquid is followed although no solvent exists which is transparent across the wholerange of the mid-IR. This procedure permits of quantitative measurements to be performed at all wavelength except those absorbed by the solvent.

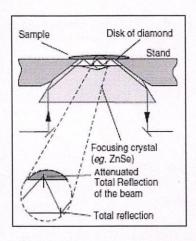
Pellets are used for solid samples that are difficult to melt or dissolve in anysuitable IR-transmitting solvents.

Attenuated total reflection (ATR)

ATR consists of imposing on an optical beam one or several reflections at theinterface between the sample and a material upon which the sample has beendeposited. This material must be transparent in the region of the wavelengthchosen.

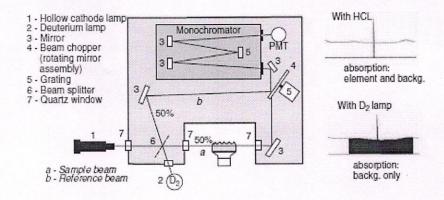
#### Atomic absorption and flame emission spectroscopy

Atomic absorption spectroscopy (AAS) and flame emission spectroscopy (FES), also called flame photometry, are two analytical measurement methods relying on the spectroscopic processes of excitation and emission.



#### **ATR**

Methods of quantitative analysis only, they are used to measure of around seventy elements (metal or nonmetal). Many models of these instruments allow measurements to be conducted by these two techniques although their functioning principles are different. There exists a broad range of applications, as concentrations to the  $\mu g/L$  (ppb) level can be accessed for certain elements.

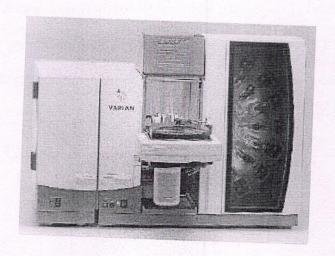


#### AAS Principle

## Effect of temperature upon an element

The principle of atomic absorption and flame emission is that incandescent gases absorb at the same wavelengths as they areable to emit. When the light of an electricarc (serving as a source of white light), is dispersedby a prism, a continuous spectrum is obtained. If this radiationsource is replaced by a Bunsen burner onto which a few crystals of sodium chlorideis sprinkled, then an emission spectrum of sodium is obtained which possesses thewell-known yellow doublet located at 589 nm, among other lines.

Finally, if the two previous sources, electric arc and theflame of the Bunsen burner are placed in series along the same optical path aspectrum will be obtained which, contains darklines in place of the emission lines of sodium (Figure 13.1, bottom). This is theresult of the presence in the flame of a large proportion of ground state sodiumatoms that can absorb the same frequencies as the excited sodium atoms emit. This is a manifestation of atomic absorption.



#### Applications to modern instruments

To measure an element by one or other of these two methods, it must be in theform of free atoms. To this end, the sample is heated to a temperature of at least2000 °C, in order to dissociate all chemical combinationsIn AAS, the concentration can be deduced from the measurement of lightabsorption by the atoms remaining in the ground state when they are irradiated an appropriate source of excitation.

In FES, conversely, the concentration can be deduced from the intensity of the radiation emitted by the fraction of atoms that have passed into excited states.

#### Measurements by AAS or by FES

The quantification of elements by these two methods implies that a relation exists between the concentration and the intensity of the corresponding light absorption emission. They make use of protocols which comprise a calibration curve from standard solutions of the analyte.

The absorbance of the element in the flame depends upon the number of ground state atoms in the optical path. The absorbance of theelement in the flame depends upon the number of ground state atoms. The method relies on the Lambert–Beer relationship.

#### Measurements by FES

For a population of excited atoms, the emitted light intensity depends upon the number of atoms that return to the ground stateBasic instrumentation for AAS

In its simplest form, an AAS resembles a single beam spectrophotometer. Theoptical scheme is illustrated in Figure 13.5 which shows a basic model. It contains four principal components: the light beam issuing from the source passes through the burner in which the element is brought to

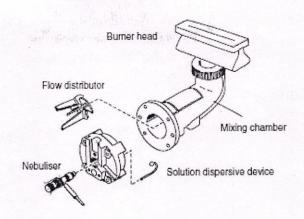
its atomic state beforebeing focused upon the entrance slit of the monochromator which selects avery narrow wavelength interval. The optical path ends at the entrance slit of thedetector.

Hollow cathode lamps (HCL) and electrodeless discharge lamps (EDL)

For the above reasons, atomic absorption instruments use two basic types of lamps. Hollow cathode lamps (HCL) are discharge lamps that contain a fill gas (argon orneon). Depending upon the element which constitutes the cathode, the emission spectrum of these sources are different. Hence, to measure an element such aslead, the cathode must contain lead. Each cathode contains the element of choicein a very pure form (99.99 per cent) where possible.

The spectrum emitted by the lamp corresponds to the superimposition of radiation emitted by the cathode and by the gaseous atmosphere within the lampThermal devices for obtaining atomic aerosolsAtomization using a flame – burner and nebulizerThe atomic aerosol for the instrument is provided by a combination of a nebulizerand a burner. The sample in aqueous solution is sucked up by the Venturi effect.

Pressurized air is passed through a tube causing the sample solution to be drawninto the burner as a fine mist where it is mixed with a combustible gaseousmixture to produce a flame, which finally contains the atomic aerosol. Naturally,the sample must be in solution form in order to employ this process. This robustmechanical assembly, called the burner, has a rectangular base of about 10 cm inlength by 1mm in width. The optical axis of the instrument is aligned with thelongest dimension of the flame.



#### Nebuliser and Atomiser

The flame is characterized principally by its chemical reactivity for a givenmaximum temperature and its spectrum. This is a complex mediumin dynamic equilibrium, containing free radicals, at the origin of a spectrum inthe near UV which results from the superimposition of emission and absorptionlines. This can interfere with the measurement of some elements. That is why it is not just any flame which may be used for any element. The chemical reactivity of the flame not being homogeneous, it is important to find a good position for the optical path of the instrument. The graphite tube is surrounded by a double sleeve. One contains an inert gas, such as argon, that circulates to protect the elements from oxidation while theother cools the entire device, using water.

In comparison with the burner, this flameless atomization produces a veryhigh atom density and a longer confinement period which multiplies the overallsensitivity by a factor of 1000.

#### Chemical vaporization

Some elements such arsenic (As), bismuth (Bi), tin (Sn) or selenium (Se) are difficult to reduce to atoms in a flame when in higher oxidation states. In order to measure these elements the sample is reacted with a reducing agent constituted by sodium borohydride or tin chloride in an acidic

medium, just prior to analysis. A volatile hydride of the element is formed which is swept up by make-up gas into a quartz cell placed in the flame of the burner.

## Flame photometers

Measurements by flame photometry are carried out either using atomic absorptionspectrometers with a burner (but without the light source), or flame photometers. The latter are less sophisticate instruments.



(AUTONOMOUS) KADAPA, ANDHRA PRADESH, INDIA-516003

### DEPARTMENT OF CIVIL ENGINEERING

#### CERTIFICATE OF COURSE COMPLETION

This certificate is presented to

Vijaya Kumari N. (Reg. No. 179Y1A0158), Student of KSRM College of Engineering (Autonomous) for successful completion of certification course on "Instruments used in environmental pollutant quantification" offered by Department of Civil Engineering, KSRMCE -Kadapa.

Course Duration: 30 Hours;

From 17/05/2021 to 02/06/2021

Course Instructor:

Sri V.V. Prasad,

Assistant Professor, CE, KSRMCE-Kadapa

Coordinators

Head of the Department

Principal



(AUTONOMOUS) KADAPA, ANDHRA PRADESH, INDIA-516003

### DEPARTMENT OF CIVIL ENGINEERING

## CERTIFICATE OF COURSE COMPLETION

This certificate is presented to

Bhanu Prakash P. (Reg. No. 179Y1A0174), Student of KSRM College of Engineering (Autonomous) for successful completion of certification course on "Instruments used in environmental pollutant quantification" offered by Department of Civil Engineering, KSRMCE-Kadapa.

Course Duration: 30 Hours; From 17/05/2021 to 02/06/2021 Course Instructor:

Sri V.V. Prasad,

Assistant Professor, CE, KSRMCE-Kadapa

Coordinators

Head of the Department

Principal



(AUTONOMOUS) KADAPA, ANDHRA PRADESH. INDIA-516003

## DEPARTMENT OF CIVIL ENGINEERING

## CERTIFICATE OF COURSE COMPLETION

This certificate is presented to

Rajesh T. (Reg. No. 179Y1A0198), Student of KSRM College of Engineering (Autonomous) for successful completion of certification course on "Instruments used in environmental pollutant quantification" offered by Department of Civil Engineering, KSRMCE-Kadapa.

Course Duration: 30 Hours; From 17/05/2021 to 02/06/2021 Course Instructor:

Sri V.V. Prasad,

Assistant Professor, CE, KSRMCE-Kadapa

Coordinators

Head of the Department

Principal

V.S.S. Muly



(AUTONOMOUS) KADAPA, ANDHRA PRADESH, INDIA-516003

## DEPARTMENT OF CIVIL ENGINEERING

## CERTIFICATE OF COURSE COMPLETION

This certificate is presented to

Srinatha Reddy B. (Reg. No. 189Y5A0109), Student of KSRM College of Engineering (Autonomous) for successful completion of certification course on "Instruments used in environmental pollutant quantification" offered by Department of Civil Engineering, KSRMCE -Kadapa.

Course Duration: 30 Hours;

From 17/05/2021 to 02/06/2021

Course Instructor:

Sri V.V. Prasad,

Assistant Professor, CE, KSRMCE-Kadapa

Head of the Department

V s.s. mw/4 Principal

### Department of Civil Engineering

Feedback of students on Certification Course on "Instruments used in environmental pollutant quantification"

Sl. No.	Roll. No.	Name of The Student	Are the course lecture hours sufficient?	Do you got extra knowledge related to Environmental pollution?	Rate the course instructor	Rate the entire course?
1	179Y1A0106	Nikhil Kumar Reddy Bhavanasi	Yes	Yes	Excellent	5
2	179Y1A0107	Geetha Bhuma	Yes	Yes	Excellent	5
3	179Y1A0113	Venkatesh Naik Bukke	Yes	Yes	Excellent	5
4	179Y1A0115	Pallavi Chatta	Yes	Yes	Excellent	5
5	179Y1A0119	Zaheer Dade	Yes	Yes	Excellent	5
6	179Y1A0122	Jayachandra Derangula	Yes	Yes	Excellent	5
7	179Y1A0123	Siddaiah Dollu	Yes	Yes	Excellent	5
8	179Y1A0124	Suresh Gowd Ediga	Yes	Yes	Excellent	5
9	179Y1A0126	Ashok Kumar Galeti	Yes	Maybe	Excellent	4
10	179Y1A0129	Pullaiah Gokula	Yes	Yes	Excellent	5
11	179Y1A0130	Ramamanohar Reddy Gollapalle	Yes	Yes	Excellent	5
12	179Y1A0131	Sreekanth Gurakanivari	Yes	Yes	Good	5
13	179Y1A0133	Dharani Jonnavaram	Yes	Yes	Excellent	5
14	179Y1A0134	Manasa Juturu	Yes	Yes	Excellent	4
15	179Y1A0136	Himaja Kancharla	Yes	Yes	Good	5
16	179Y1A0139	Vivekananda Reddy Kota	Yes	Yes	Good	5
17	179Y1A0147	Thimmappa Madiga	Yes	Yes	Excellent	5

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38	189Y5A0120	Swetha Damsetty	Yes	Yes	Excellent	4
39	189Y5A0126	Shireesha Guramkonda	Yes	Yes	Excellent	5
40	189Y5A0127	Nikitha Jaladi	Yes	Yes	Excellent	5
41	189Y5A0128	Venkata Subbaiah Janapati	Yes	Yes	Good	4
42	189Y5A0132	Yaswanth Reddy Kambham	Yes	Yes	Excellent	4
43	189Y5A0134	Sree Hari Reddy Katthi	Yes	Yes	Excellent	5
44	189Y5A0135	Nagesh Kolliboina	Yes	Yes	Excellent	5
45	189Y5A0138	Prasanth Kumar Kotturu	Yes	Yes	Excellent	5
46	189Y5A0140	Suresh Kuruva	Yes	Yes	Excellent	5
47	189Y5A0146	Venkata Ramana Reddy Nimmakayala	Yes	Yes	Excellent	4
48	189Y5A0159	Abilash Reddy Sajjala	Yes	Yes	Excellent	4
49	189Y5A0162	Naveen Kumar Sepuri	Yes	Yes	Good	5
50	189Y5A0170	Chandra Kanth Thatamsetty	Yes	Yes	Good	4
51	189Y5A0171	Divya Thonduru	Yes	Yes	Excellent	5
52	189Y5A0172	Parameswara Reddy Thummala	Yes	Yes	Excellent	5
53	189Y5A0179	Narendra Kumar Reddy Yanamala	Yes	Yes	Excellent	5

Coordinators BAR

HoD-Civil Engg.

Head
Department of Civil Engineering
K.S.R.M. College of Engineering
(Autonomous)
KADAPA 516 003. (A.P.)