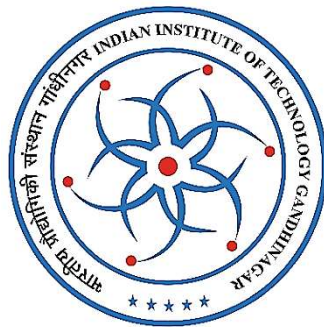


A training session report on  
**Single Crystal X-Ray Crystallography**

Under  
STUTI program funded by DST.



Co-ordinated by  
**Prof. Vijay Thiruvankatam**  
Department of Biological Engineering  
Indian Institute of Technology  
Gandhinagar, Gujarat  
**18<sup>th</sup> to 24<sup>th</sup> April 2022**

## **Acknowledgement**

We convey gratitude for the encouragement and support received from multiple sources during the execution of this training since its beginning. First and foremost, I want to express my sincere appreciation to the Department of Science and Technology (DST) for entrusting me with this project.

The workshop was coordinated by Prof. Vijay Thiruvankatam. The workshop was conducted on the Single Crystal X-Ray Crystallography funded by the FIST program (Project No.: RES/DST/CH/P0130/1818/0022 Date: 01-07-2017). Organizing team acknowledge the contributions of the training committee, in the implementation and the execution of the program to achieve the objectives of the project, particularly, Prof. Neha Jain (Department of Bioscience & Bioengineering, IITJodhpur), Prof. V. Madhu (Department of Chemistry, Karunya University), Prof. Sriram Kanvah (Department of Chemistry), Prof. Sivapriya Kirubakaran (Department of Chemistry), Dr. Gurudutt Dubey (PDF, Department of Chemistry), Ms. Delna (Ph.D. scholar) and Ms. Haritha (Ph.D. scholar).

I also acknowledge all the project staff and IITGN staff contributions without which these sessions could not have been possible.

### **Principal Investigators**

Prof. Vijay Thiruvankatam

Prof. Emila Panda

## ***Summary***

The goal of this training session is to popularize single crystal X-ray crystallography (SCXRD) facility among students, faculty, scientists and industry professionals through a week-long training workshop. The workshop was conducted at IIT Gandhinagar from 18<sup>th</sup> to 24<sup>st</sup> April, 2022 and comprised of lectures and hands on training sessions. This initiative is funded by Department of Science & Technology under the program ***STUTI*** (*Synergistic Training Program Utilizing the Scientific and Technological Infrastructure*). This workshop is aimed to provide an insight into the basic principles and various techniques of crystallization of small molecules and protein, functioning of Single Crystal X-Ray Diffraction (SCXRD) instrument, single crystal data analysis and interpretation, applications of SCXRD in material, pharmaceutical and biological sciences. The participants were introduced to the basic concepts of will be introduced to the basic concepts of image generation, instrumentation, troubleshooting and the advanced modes of operation. The focus of this workshop was to have “*a balance between theory and practical training on the equipment. Emphasis is on hands-on use of equipment for demonstration/characterization by each participant and analysis of participant’s samples*”.

## Introduction

Indian Institute of Technology Gandhinagar (IITGN) conducted 7-day long workshop on DST-FIST funded instrument 'Single Crystal X-ray Crystallography (SCXRD)' workshop in its campus for participants from various backgrounds such as Post Graduate, B.Tech., Professors, Scientists, Post-Doc Fellows, Ph.D. Fellows and Industry persons were invited (**Annex-1**). The following workshop's activities took place from 18<sup>th</sup> to 24<sup>st</sup> April 2022 (**Annex-2 & 3**). This report provides a quick overview of both the lecture and technical sessions.

- Lecture sessions

**Prof. Vijay** (Department of Biological Engineering, IITGN) explained all of the essential principles and physics behind the operation during single crystal X-ray diffraction in significant detail, which made it easier for participants to understand the procedure while doing hands-on activities. **Prof. Sriram Kanvah** (Department of Chemistry, IITGN) discussed fluorescence spectroscopy as well as the importance of fluorescence and phosphorescence in organic and material science. **Prof. V. Madhu** (Department of Chemistry, Karunya University) gave a detailed lecture on organometallic compounds and crystallisation processes, as well as the benefits of single crystal X-ray crystallography. **Prof. Sivapriya Kirubakaran** (Department of Chemistry, IITGN) discussed key elements of polymorphism in organic chemistry, medicinal chemistry, and drug development employed in cancer treatment. **Dr. Gurudutt Dubey** (Department of Chemistry, IITGN) gave a comprehensive presentation on single crystal X-ray crystallography's applicability in the pharmaceutical industry and structure-based drug development. **Dr. Neha Jain** (Department of Bioscience & Bioengineering, IITJodhpur) discussed the structure guided discovery of amyloids modulators in the treatment of neurodegenerative diseases and explained biophysical characterization of newly developed anti-neurodegenerative drugs.

- Technical Sessions

On the **first day**, Dr. Vijay began the training session by giving a general overview of single-crystal X-ray crystallography, as well as its applications in the chemistry and pharmaceutical industries. Dr. Gurudutt (PDF scholar, IIT Gn) and Ms. Delna (Ph.D Scholar, IITGn) provided a thorough overview of the fundamental stages for sample preparation and other SCXRD setup activities. At the end of the day, all participants were taken for the campus visit. Then, all the participants were introduced to data analysis of the results obtained by the XRD setup on the **second day**. In addition, all participants were provided with the academic version of the installation setup as well as hands-on instruction on how to install the software. On the **third day**, all participants were granted access to the XRD setup in order to provided hands-on training by Dr. Achintya, Dr. Gurudutt, and Ms. Delna. In the **fourth day's** session, participants were briefed about how to get the parameters for diffraction data collection. also, they were taught about acquiring data from an unknown material. The **fifth day** session included a similar activity of reviewing the specifics of data gathering methods from software, as monitored by Prof. Vijay, Dr. Gurudutt, and Ms. Delna. The **sixth day** session included a discussion with participants on several fundamental principles and case studies, including crystal structure refinements, in-situ cryo-crystallization, and face indexation. The **seventh day** was a concluding session in which Prof. Vijay and Dr. Gurudutt handled many participant questions on technical and software difficulties.

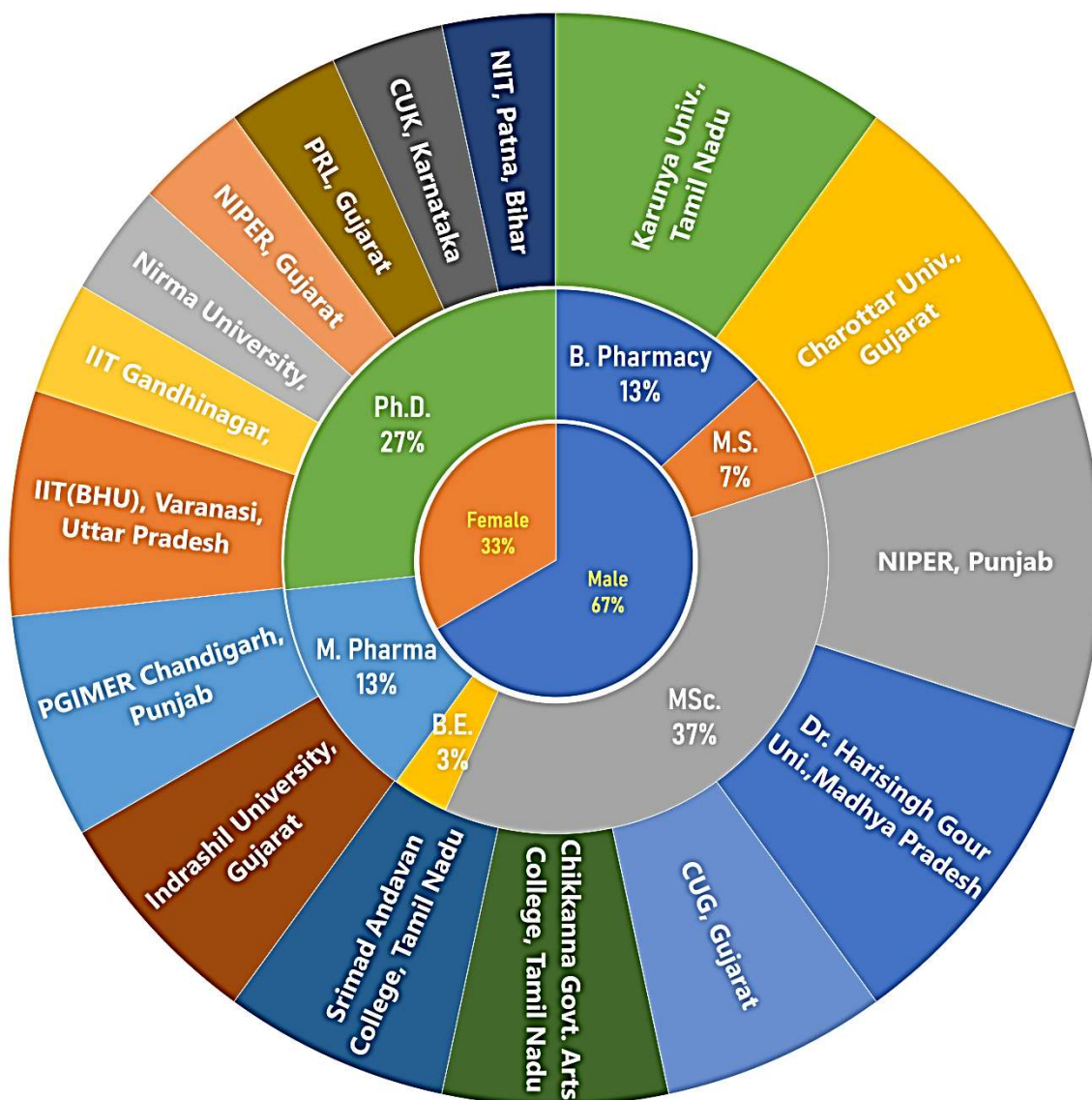
- Types of samples tested

During the technical session, all of the participants expressed an interest in learning from the workshop and characterized more than 25 no. of samples. In addition, it was observed that, three categories of samples were characterized utilizing FIST-SCXRD setup during the technical sessions: standard crystal of ylid, weak quality crystal and large size crystal. The samples tested were belonging to the following categories: a) Organometallic compound, b) Organic during molecule and c) In-organic compound. Additionally, crystal disorder, face indexation and few case studies of drug co-crystals were also discussed in detail during this training session.

## *Outcome of the workshop*

The STUTI workshop attracted participants from 15 different institutes (**Figure 1**). About 30 participants enrolled and attended the SCXRD training sessions. The goal of this training event was to bring together participants from many disciplines and raise awareness of the institute's advanced facilities. Throughout the sessions, participants asked major questions regarding theoretical and practical aspects of SCXRD instrumentations, techniques of developing good quality crystals, disorder treatments. This SCXRD workshop brought more than 5 collaborations from many small institutions and national level institutes. Finally, the feedback from the participants was considered in the evaluation of the workshop (**Annex4**). The majority of the participants were pleased with the training session and suggested that more workshops be held in the future. Few participants suggested organizing such a workshop/training sessions on more troubleshooting techniques of data collection.





**Figure 1:** Participants registered for the ‘Single Crystal X-Ray Crystallography’ workshop from 15 different institutes.

## Annex 1: Brochure for the program.

### Speakers



**Prof. Sivapriya Kirubakaran** is an Associate Professor in Chemistry at IIT Gandhinagar. Her work focusses on drug discovery and cancer chemical biology, synthesis of small molecules for cancer treatment.



**Prof. Sriram Kanvah Gundimeda** is an Associate Professor in Chemistry at IIT Gandhinagar. His work focusses on the synthesis of photosensitive conjugated materials for a variety of optical and biological applications.



**Prof. Vijay Thiruvankatam** is an Associate Research Professor in Biological Engineering at IIT Gandhinagar. He works on the structural biology of macromolecules, chemical crystallography and In situ cryo-crystallization of liquids.



**Dr. V. Madhu** is an Assistant Professor in Department of Chemistry at Karunya University. His research areas are optoelectronic materials, catalysis, bio-inorganic chemistry and coordination chemistry.



**Dr. Neha Jain** is an Assistant Professor in Department of Bioscience & Bioengineering at IIT Jodhpur. Her research focusses on amyloid-dependent biofilms and their biophysical characterization.



**Dr. Gurudutt Dubey**, Discipline of Chemistry, IIT Gandhinagar. His research areas are co-crystallization of API and small drug like molecules, SC-XRD, *ab initio* modeling of small and macro molecules.

### Registration & Contact Details

Interested participants must register and only selected candidates would be invited for the workshop.

For selected candidates Registration fees, local travel, Boarding and lodging will be covered by IIT Gandhinagar.

Interested participants should register using the following link: <https://forms.gle/MytuEk4aKicanCmz8>

**Registration Deadline: 08<sup>th</sup> April 2022**  
Shortlisted candidates will be intimated by email, latest by 10<sup>th</sup> April 2022.

#### Eligibility criteria:

- Minimum qualification: Post Graduate (Science) or B.Tech. (Technology)
- Professors/Scientists/Post-Doc Fellows/Ph.D. Fellows/ Industry persons who are actively involved in R&D
- Not more than 2 participants from one institute

For more information:

Access: <https://events.iitgn.ac.in/stuti/>

Mail: [stuti@iitgn.ac.in](mailto:stuti@iitgn.ac.in)

Address: Block 4-316, IIT Gandhinagar, Palaj, Gandhinagar 382055

#### Acknowledgements



### Department of Science & Technology (DST) funded

Training workshop under **STUTI** (Synergistic Training Program Utilizing the Scientific and Technological Infrastructure)

*7 Days Workshop on*  
**Single Crystal X-Ray Crystallography**  
18<sup>th</sup> April to 24<sup>th</sup> April 2022  
IIT Gandhinagar, Gujarat

### Contents of the workshop

#### Day 1 - Session I & II

- Inauguration and Welcome note
- Introduction of the Participants and the Host
- Basic introduction to SC-XRD techniques
- Hands on: small molecule crystallization

#### Day 2 - Session I & II

- Crystallization of small molecules & proteins
- Hands on: Protein crystallization
- Lab visit

#### Day 3 - Session I & II

- Data collection & processing
- Hands on: Instruction to instrument
- Hands on: System check and basic precautions

#### Day 4 - Session I & II

- RLATT interpretation of diffraction data
- Industrial applications: API and drug like molecules
- Hand on: Crystal selection, mounting & centering
- Hands on: Fast scan vs. Matrix scan

#### Day 5 - Session I & II

- Structure refinement with SHELXL and CIF file generation
- Hands on: Strategy optimizer & set up a measurement
- Data reduction, scaling & structure solution

#### Day 6 - Session I & II

- In situ cryo-crystallization
- Crystal disorder & troubleshooting
- Case study with small molecules crystals
- Hands on: Structure solution
- Hands on: Face indexing and Scaling the data

#### Day 7 - Session

- Interactive and Problem solving session

### Overview of STUTI and Objectives of Workshop

DST welcomes all the participants for the workshop on Single Crystal X-Ray crystallography organised under STUTI. The STUTI program envisions hands-on-training and sensitization of the state-of-the-art equipment as well as towards sharing while ensuring transparent access to S&T facilities. Department of Science and Technology has identified IIT-Gandhinagar to function as a Project Management Unit (PMU) and as co-ordinator for this workshop.

This workshop is aimed to provide an insight into the basic principles and various techniques of crystallization of small molecules and protein, functioning of single crystal X-ray diffraction instrument, single crystal data analysis and interpretation, applications of SC-XRD in material, pharmaceutical and biological sciences. The participants will be introduced to the basic concepts of image generation, instrumentation, troubleshooting and the advanced modes of operation. The participants will be provided with hands on experience on the operation of the instrument and will have a chance to interact with subject experts and also analyse their own samples (with prior approval).

### Schedule

Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7
08:30 Registration	09:00 Session I	09:00 Session I	09:00 Session I	09:00 Session I	09:00 Session I	09:00 Interactive session
09:00 Inaugural Session	11:00 Break	11:00 Tea Break	11:00 Tea Break	11:00 Tea Break	11:00 Tea Break	11:15 Tea Break
10:45 Tea Break	11:30 Expert Talk	11:30 Expert Talk	11:30 Expert Talk	11:30 Expert Talk	11:30 Expert Talk	11:30 Closing remarks
11:00 Session I	12:30 Lunch	12:30 Lunch	12:30 Lunch	12:30 Lunch	12:30 Lunch	
12:30 Lunch	14:00 Session II (Hands on)	14:00 Session IIA	14:00 Session IIA	14:00 Session IIA	14:00 Session IIA	
14:00 Session II (Hands on)	15:30 Tea Break	15:30 Tea Break	15:30 Tea Break	15:30 Tea Break	15:30 Tea Break	
15:30 Tea Break	16:00 Lab visit	16:00 Session IIB	16:00 Session IIB	16:00 Session IIB	16:00 Session IIB	
16:00 Campus Visit		17:00 Discussion	17:00 Discussion	17:00 Discussion	17:00 Discussion	
						Networking Dinner on Day 5

Sessions IIA & IIB will be Hands on training



**Annex 2: List of participants registered and their attendance for the Single Crystal X-Ray  
Crystallography workshop.**

Sr. No.	Candidate Name	Gender	Educational Qualification	Email address	Host Institute
1	Priyanshu Nema	Male	B. Pharmacy	priyanshunema9908@gmail.com	Dr. Harisingh Gour University, Sagar
2	Twinkle Gupta	Female	B. Pharmacy	twinkle Gupta1970@gmail.com	
3	Priyadarshi Kendya	Male	B. Pharmacy	ankit.kendya@gmail.com	
4	Amritha G. Nambair	Female	M. Pharmaceutics	amrithagnambiar.rs.phe21@iitbhu.ac.in	IIT (BHU), Varanasi
5	Maan Singh	Male	M. Pharmaceutics	maansingh25may97@gmail.com	
6	Soumalya Chakraborty	Male	M. Pharmaceutics	sc280562@gmail.com	NIPER Mohali
7	Astha Gupta	Female	M.S. (Medicinal Chemistry)	asthaorai.289@gmail.com	
8	Rahul Yadaorao Kapse	Male	B. Pharmacy	kapserahul2015@gmail.com	
9	Dimpy P. Patel	Female	MSc. (Medical Laboratory Technology)	Dimpypatel@gmail.com	Charotrar Univ., Charusat
10	Abhishek Dadhania	Male	Ph.D. (Chemistry)	abhishekdadhania.bt@charusat.ac.in	
11	Unnati Dipakkumar Patel	Female	MSc. (Chemistry)	16drche001@charusat.edu.in	
12	Namrata Sangwan	Female	MSc. (Zoology)	NMSGWN@GMAIL.COM	PGIMER Chandigarh
13	Arushi Chauhan	Female	MSc. (Zoology)	arushichauhan.2294@gmail.com	
14	V. Madhu	Male	Ph.D. (Chemistry)	madhu@karunya.edu	Karunya Univ., Tamil Nadu
15	Jerome Issac	Male	MSc. (Chemistry)	jeromeissac321@gmail.com	
16	Deepanjaly K.S	Female	MSc. (Chemistry)	deepanjalydas@gmail.com	
17	Sunil Kumar	Male	MSc. (Chemistry)	sunilk.phd20.ch@nitp.ac.in	NIT, Patna
18	Tahir Mehmood	Male	MSc. (Chemistry)	tahirmahmoodisu@gmail.com	Indrashil University, Ahmedabad
19	Patel Bhumiben Chandubhai	Female	MSc. (Chemistry)	pbumi141@gmail.com	
20	Naveena S V	Male	MSc. (Chemistry)	naveena.sv.snp@gmail.com	Central Univ. Karnataka
21	Surendra Vikram Singh	Male	Ph.D. (Physics)	surendra@prl.res.in	Physical Research Laboratory, Ahmedabad
22	J. Sivaraman	Male	Ph.D. (Chemistry)	sr91084@gmail.com	Srimad Andavan Arts and Science College, Tiruchirappalli
23	P. Venkatesan	Male	Ph.D. (Chemistry)	venkat@andavancollege.ac.in	
24	P. Vijayan	Male	Ph.D. (Chemistry)	pvisayan@gmail.com	Chikkanna Govt. Arts College, Tiruppur
25	S. P. Rajasingh	Male	Ph.D. (Chemistry)	sprajasingh@gmail.com	
26	L Raju Chowhan	Male	Ph.D. (Chemistry)	rchowhan@cug.ac.in	Central University of Gujarat
27	Biplob Borah	Male	MSc. (Chemistry)	biplobborah18@gmail.com	
28	Dattatraya Jalindhar Yadav	Male	M. Pharmaceutics	18ftphdp50@nirmauni.ac.in	Nirma University, Ahmedabad
29	Saumya Kapoor	Female	M.S. (Medicinal Chemistry)	saumya.kapoor@niperahm.res.in	NIPER-Ahmedabad
30	Arvind Chadhar	Male	B.E. (Electronic and Communication)	aravind.chadhar@iitgn.ac.in	IIT Gandhinagar

**Annex 3: Schedule date and activities during the workshop.**

<b>Day 1</b>			<b>Day 5</b>		
<b>18<sup>th</sup> April 2022</b>	08:30	Registration	<b>22<sup>th</sup> April 2022</b>	09:00	Session I
	09:00	Inaugural Session		11:00	Tea Break
	09:30	Session IA		11:30	Expert Talk
	10:30	Tea Break		12:30	Lunch
	11:00	Session IB		14:00	Session IIA
	12:30	Lunch		15:30	Tea Break
	14:00	Session II		16:00	Session IIB
	15:30	Tea Break		17:00	Discussion
	16:00	Campus Visit	19:30	Networking Dinner	
<b>Day 2</b>			<b>Day 6</b>		
<b>19<sup>th</sup> April 2022</b>	09:00	Session I	<b>23<sup>th</sup> April 2022</b>	09:00	Session I
	11:00	Tea Break		11:00	Tea Break
	11:30	Expert Talk		11:30	Expert Talk
	12:30	Lunch		12:30	Lunch
	14:00	Session II		14:00	Session IIA
	15:30	Tea Break		15:30	Tea Break
	16:00	Lab visit		16:00	Session IIB
			17:00	Discussion	
<b>Day 3</b>			<b>Day 7</b>		
<b>20<sup>th</sup> April 2022</b>	09:00	Session I	<b>24<sup>th</sup> April 2022</b>	09:00	Interactive session
	11:00	Tea Break		11:00	Tea Break
	11:30	Expert Talk		11:30	Closing remarks
	12:30	Lunch			
	14:00	Session IIA			
	15:30	Tea Break			
	16:00	Session IIB			
	17:00	Discussion			
<b>Day 4</b>					
<b>21<sup>th</sup> April 2022</b>	09:00	Session I			
	11:00	Tea Break			
	11:30	Expert Talk			
	12:30	Lunch			
	14:00	Session IIA			
	15:30	Tea Break			
	16:00	Session IIB			
	17:00	Discussion			

#### Annex 4: Feedback summary

<b>Sr. No.</b>	<b>Content</b>	<b>% Rating</b>
<b>1</b>	Overall grading of the Programme with reference to relevance of course, module/content etc.	97 % rated above 8 points
<b>2</b>	Overall grading of the facilities provided by the institute, i.e., Hostel, Mess, Class Rooms, Transport/infrastructure etc.	95% rated above 8 points
<b>3</b>	Overall grading of the faculty members conducting the training	95% rated above 8 points
<b>4</b>	How do you rate the overall training methodology	100% rated above 8 points
<b>5</b>	How far the field visit is relevant and related to your research study	80% rated above 8 points
<b>6</b>	Usefulness of this training in your current role	100% rated above 8 points
<b>7</b>	Usefulness of this training in future work/job you may handle	97% rated above 8 points
<b>8</b>	How far have you benefitted from interaction with the fellow participants of the training	100% rated above 8 points
<b>9</b>	How far the course material supplied relevant and related to the training curriculum	100% rated above 8 points
<b>10</b>	Overall grading of the process of training	97% rated above 8 points
<b>11</b>	Your recommendation to your peers/ colleagues for the training Programme	95% rated above 8 points