INTEGRATING STANDARDS EDUCATION INTO THE NANOMATERIALS ENGINEERING CURRICULUM

NIST SSCD 70NANB22H206

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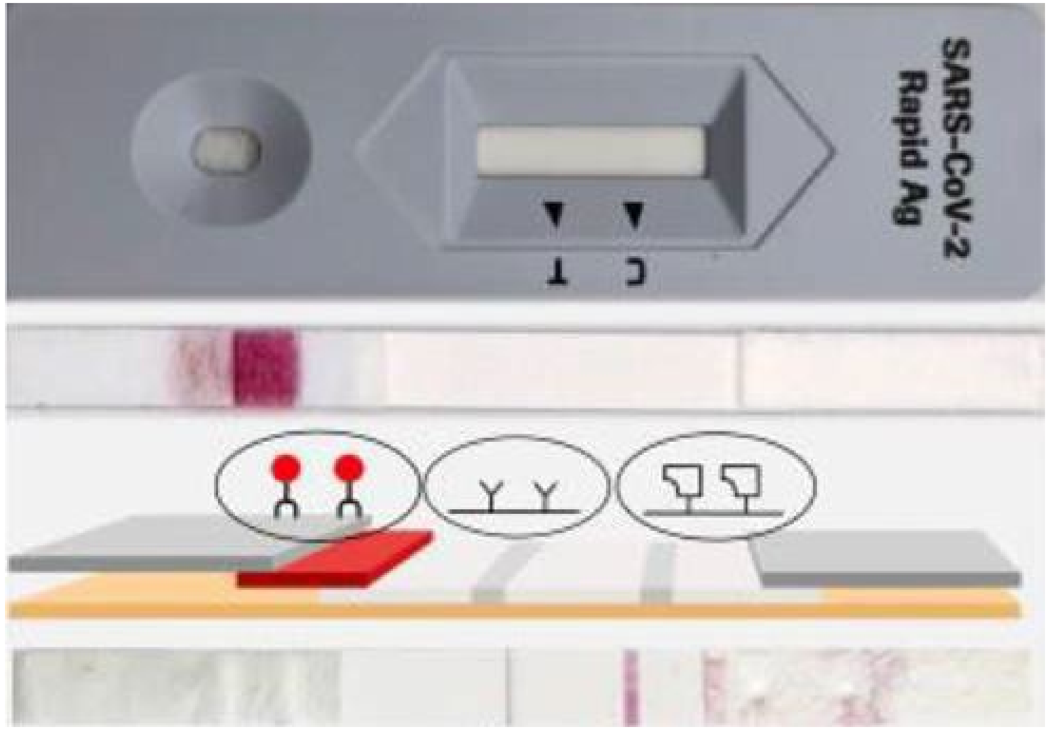
PROJECT MOTIVATION

- **Standards education not often a focus of undergraduate / graduate engineering curriculum**
  - Standards may be cited in course documents and laboratory protocols
  - However, students generally not assigned to look up standards themselves
    - Awareness of availability of documentary standards
    - Experience with reading and implementing standards
    - Opportunities to participate in the standards development process
- **Educational benefits**
  - Reinforcement of course theory
  - Improvements in lab technique and critical thinking skills
  - Practical training for future careers

WHY NANOTECHNOLOGY?

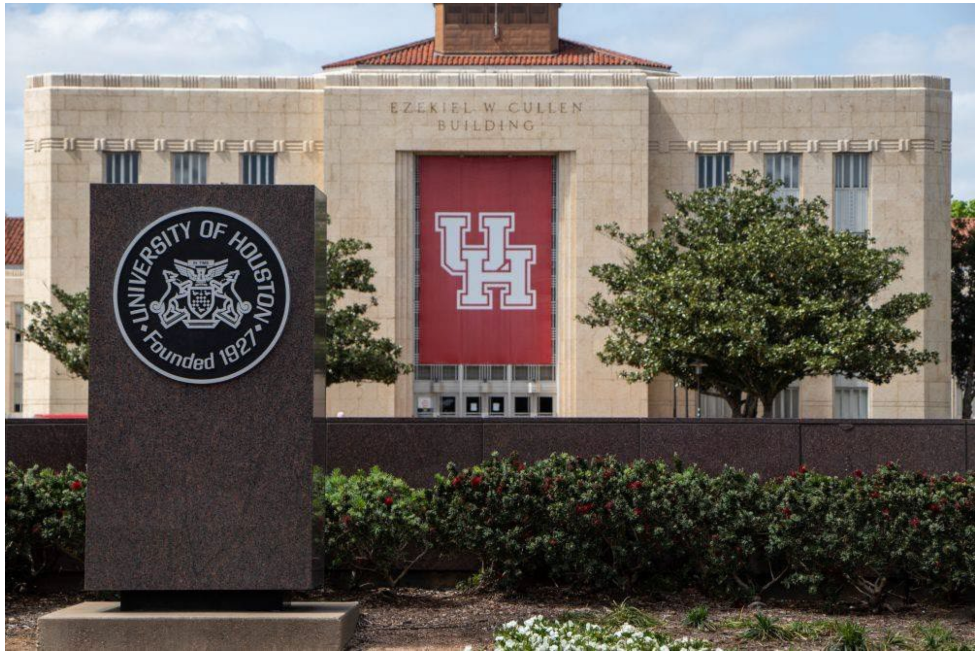
- **A fully established research field**
- **Commercial applications are rapidly expanding**
  - Electronics/semiconductors
  - Health and medicine (COVID tests & vaccine)
  - Environment, health & safety (EHS) applications and implications
- **Active development of standards**
  - ISO/TC 229 – Nanotechnologies
  - ASTM Committee E56 – Nanotechnology
  - IEC TC 113 – Nanotechnology for electrotechnical products/systems



Pölloth et al., 2022, *J. Chem. Educ.*, 99: 2579

PROJECT OBJECTIVES

- **Incorporate standards education related to nanotechnology into two engineering courses at the University of Houston**
  - CIVE 7397 – Experimental Methods in Nanomaterials Engineering (graduate level course)
  - ECE 5320/6307 – Nanomaterials and Solar Energy (undergraduate/graduate level course)
- **Two rounds of course deliveries in Spring 2023 and Spring 2025**
  - Curricular materials developed and delivered to 40-50 undergraduate and graduate students across both courses
  - Evaluation: Assignments and lab reports; Pre-/post-surveys



TEACHING GOALS

- **Develop an understanding of standards** pertaining to vocabulary, classifications, and characterization related to nanomaterials
- **Develop student skills and confidence** in navigating and implementing standard test methods
- **Promote an appreciation for the value of standards** and the standards development process

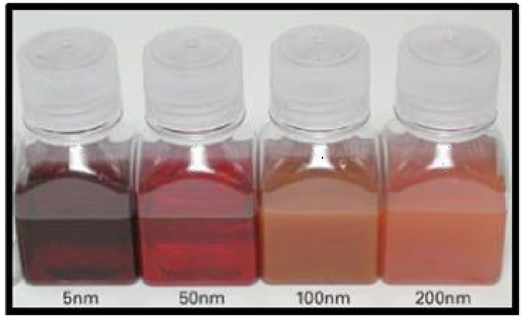
OVERVIEW OF COURSE STRUCTURE

General Curriculum

- Weeks 1-4 (Theory/Discussion)
  - Definition of nanomaterials
  - Important nanoparticle properties (size, surface charge, surface chemistry)
- Weeks 5-14 (Analytical Methods)
  - Measurement principles
  - Laboratory session

Standards-Focused Curriculum

- Weeks 1-4 (Introduce Standards)
  - Terminology standard
  - Reporting standards
  - Product characterization standard
  - Standard test methods



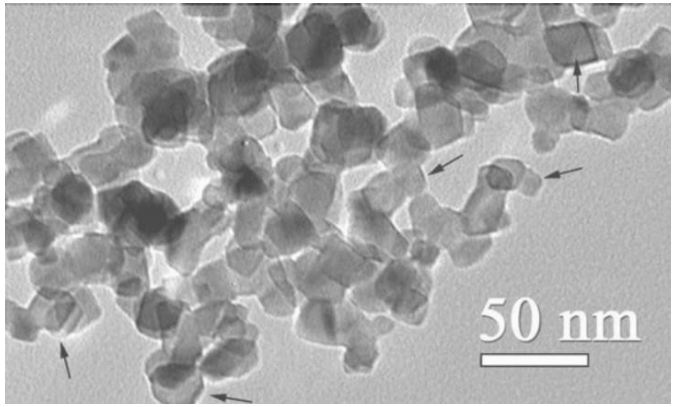
Gold nanoparticles:
An ideal nanomaterial
for discussion and laboratories

(Ted Pella, Inc., https://www.tedpella.com/gold_html/goldsols.aspx)

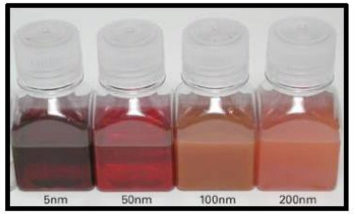
IN-CLASS ACTIVITIES / DISCUSSION

- **Nanotechnology documentary standards integrated with lectures and hands-on activities**

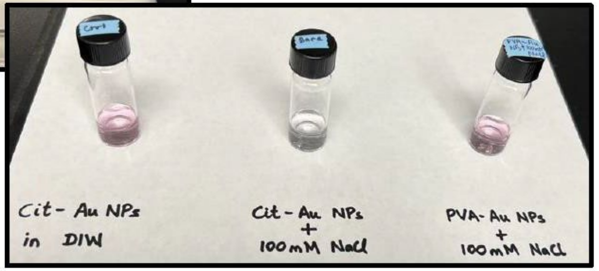
  - Terminology (ASTM E2456-06)
  - Key nanomaterial properties (ASTM E3144/E3206)
  - Detection in products (ASTM 3025)



(NIST SRM 1898)



(Ted Pella, Inc., https://www.tedpella.com/gold_html/goldsols.aspx)



- **Discussion motivates appreciation of challenges in evaluating nanomaterials in samples and products, and need for standards**
- **Assessment tool:** Assignment on classifying standards and identifying sections of standards that address topics discussed in class

GUEST LECTURES

- **Dr. Vincent A. Hackley (NIST)**
  - Defining standards
  - Benefits of using and developing standards
  - Process to develop international consensus standards
- **Dr. Nemal Gobalasingham (Waters-Wyatt Technology)**
  - Industry contributions to ISO and ASTM standards

➢ **Guest experts share direct experience with students and encourage participation in the standards development process**

➢ **Assessment tool:** Assignment related to lecture content

LABORATORIES

- **Standards reading during lecture sessions paired with hands-on laboratories**
  - **UV-Vis spectroscopy:** Optical detection and size distribution (ISO/TS 17466)
  - **Dynamic light scattering:** Size distribution (ASTM E3247)
  - **Electrophoretic light scattering:** Surface potential (NCL Method PCC-2)
  - **FTIR spectroscopy:** Surface chemistry (ISO/TS 14101)
  - **Asymmetric flow field – flow fractionation (AF4):** Size distribution (ISO/TS 21362; ASTM E3409)
  - **Single particle inductively-coupled plasma mass spectrometry:** Size distribution (ISO/TS 19590)

➢ **Students gain familiarity with reading / implementing standards, understanding procedures in depth, and critically evaluating data**

➢ **Assessment tool:** Lab report discussion questions related to standards

EXAMPLE LABORATORY HANDOUT AND DISCUSSION QUESTIONS

CIVE 7397 – Laboratory 2: Dynamic Light Scattering (DLS)

This laboratory will apply DLS measurements to determine particle sizes for a series of purchased gold (Au) nanoparticles (nominally 10 nm, 30 nm, 60 nm, and 100 nm), a mixture of the four sizes of Au nanoparticles, two unknown Au nanoparticle samples synthesized in class with different visible colors (pink and purple), the 60 nm Au nanoparticles with an adsorbed polymeric surface coating (polyvinylpyrrolidone), and titanium dioxide (TiO_2) nanoparticles. The learning objectives of the laboratory include: (1) evaluating and explaining similarities or differences between the DLS sizes and the manufacturer's reported sizes; (2) identifying uncertainties or errors in the measurement based on the results acquired; (3) interpreting and critically evaluating the quality of the raw data collected, the autocorrelation function, and the model fitting results; and (4) implementing best practices for sample preparation and handling for DLS measurements.

The laboratory is paired with **ASTM E3247–20: Standard Test Method for Measuring the Size of Nanoparticles in Aqueous Media Using Dynamic Light Scattering**, to support the learning objectives.

DISCUSSION QUESTIONS (Due one week after the lab session)

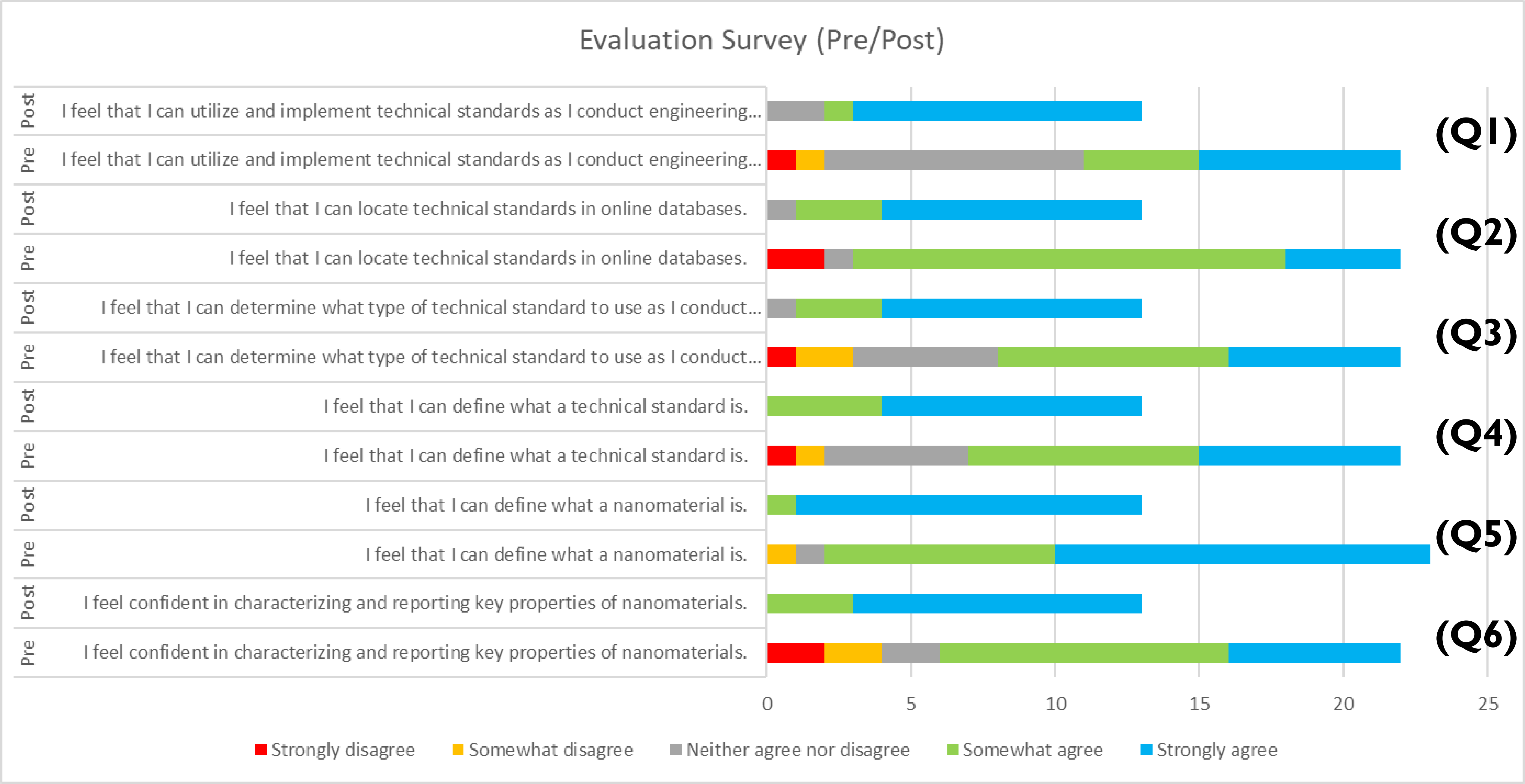
1. ***Measurement settings***

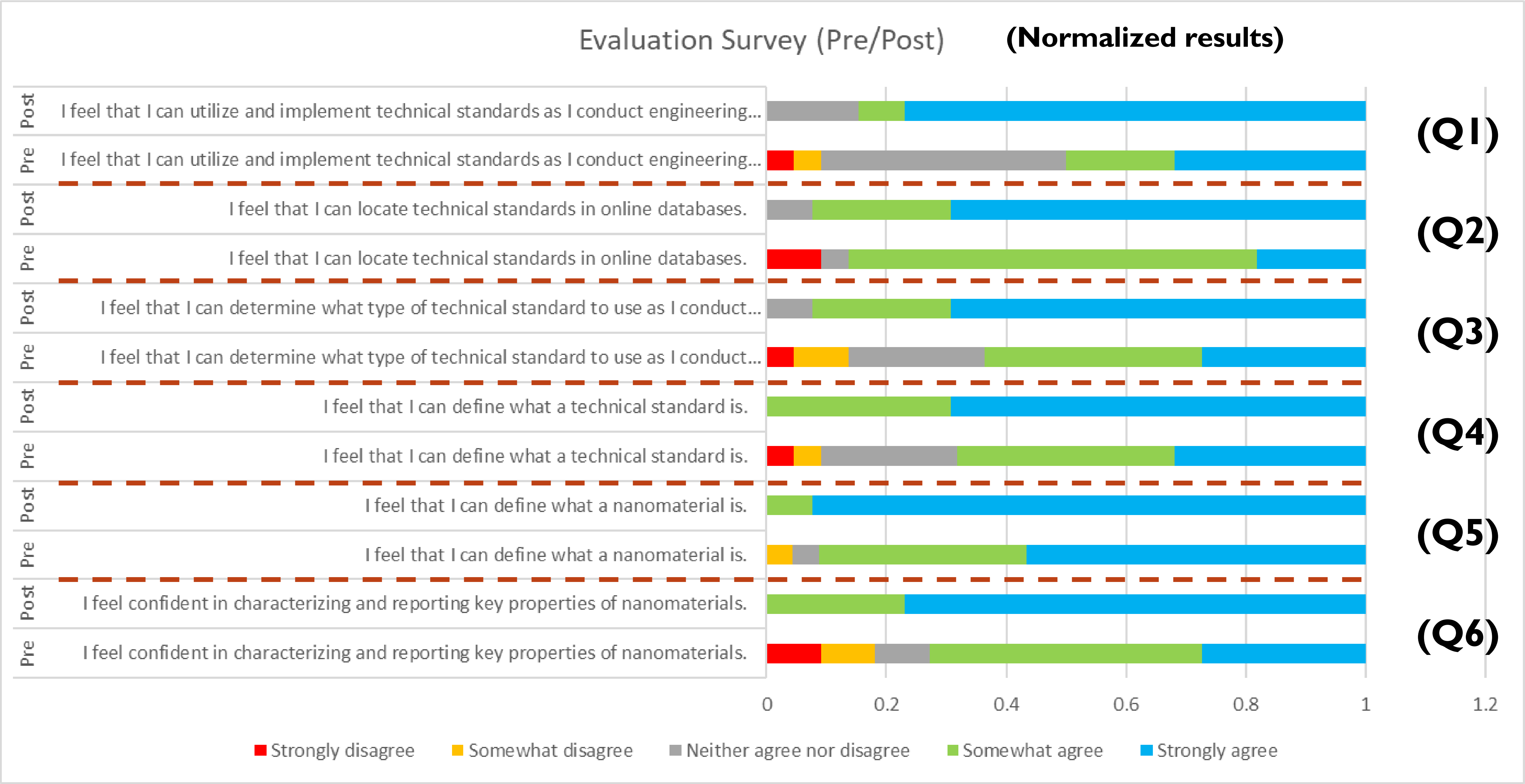
   Several pieces of information related to the sample and measurement properties were carefully inputted to set up the DLS measurement (as recorded in your Data and Observations). Discuss why the **temperature** and **solvent viscosity** are important to input accurately by identifying where (e.g. what model equation) errors in these parameter inputs would propagate to errors in the size determination by DLS. *For full credit, you must specify the relevant slide(s) from the course lecture notes and/or the relevant section from the ASTM standard that support your answer.*

EVALUATION

- **Pre/Post Surveys**
  - I feel that I can **define what a technical standard is**
  - I feel that I can **locate technical standards in online databases**
  - I feel that I can **determine what type of technical standard to use** as I conduct engineering work
  - I feel that I can **utilize and implement technical standards** as I conduct engineering work
  - I feel that I can **define what a nanomaterial is**
  - I feel confident in **characterizing and reporting key properties of nanomaterials**

- **Students generally reported improvement in all areas**


Evaluation Survey (Pre/Post)
Post
I feel that I can utilize and implement technical standards as I conduct engineering...
Pre
I feel that I can utilize and implement technical standards as I conduct engineering...
Post
I feel that I can locate technical standards in online databases.
Pre
I feel that I can locate technical standards in online databases.
Post
I feel that I can determine what type of technical standard to use as I conduct...
Pre
I feel that I can determine what type of technical standard to use as I conduct...
Post
I feel that I can define what a technical standard is.
Pre
I feel that I can define what a technical standard is.
Post
I feel that I can define what a nanomaterial is.
Pre
I feel that I can define what a nanomaterial is.
Post
I feel confident in characterizing and reporting key properties of nanomaterials.
Pre
I feel confident in characterizing and reporting key properties of nanomaterials.
(Q1)
(Q2)
(Q3)
(Q4)
(Q5)
(Q6)
0
5
10
15
20
25
Strongly disagree
Somewhat disagree
Neither agree nor disagree
Somewhat agree
Strongly agree


Evaluation Survey (Pre/Post)
(Normalized results)
Post
I feel that I can utilize and implement technical standards as I conduct engineering...
Pre
I feel that I can utilize and implement technical standards as I conduct engineering...
Post
I feel that I can locate technical standards in online databases.
Pre
I feel that I can locate technical standards in online databases.
Post
I feel that I can determine what type of technical standard to use as I conduct...
Pre
I feel that I can determine what type of technical standard to use as I conduct...
Post
I feel that I can define what a technical standard is.
Pre
I feel that I can define what a technical standard is.
Post
I feel that I can define what a nanomaterial is.
Pre
I feel that I can define what a nanomaterial is.
Post
I feel confident in characterizing and reporting key properties of nanomaterials.
Pre
I feel confident in characterizing and reporting key properties of nanomaterials.
(Q1)
(Q2)
(Q3)
(Q4)
(Q5)
(Q6)
0
0.2
0.4
0.6
0.8
1
1.2
Strongly disagree
Somewhat disagree
Neither agree nor disagree
Somewhat agree
Strongly agree

INSTRUCTOR ASSESSMENT

- **Benefits of standards education**
  - Valuable and unique approach to **enhance analytical methods courses**
  - Guest lecturers from industry and government **raise student interest**
  - Students have expressed interest in engaging with ASTM as student members or participating further through their formal careers
- **Challenges**
  - **Length of standards (20-50 pages):**
    - Selected readings assigned/discussed in class
    - Explicitly prompting students to reference standards in the lab discussion questions improved standards utilization <u>and</u> students' ability to answer the questions thoroughly and accurately
  - **Difficulty level:** Advanced theory (e.g., light scattering, electrophoretic mobility) more appropriate for graduate level

EXTERNAL DISTRIBUTION (slide 1 of 4)

- **Course materials and “lab kits” distributed to other IHEs**
  - Utah State University
  - Swarthmore College
- **Direct hosting and mentorship of undergraduate researchers**
  - Two undergraduate researchers advised by Dr. Illya Medina-Velo, Houston Christian University
  - Students directly mentored by PI and a PhD research assistant in reading and application of two standards (dynamic light scattering and electrophoretic light scattering) for measurements conducted for their research
  - Standards were successfully applied for data quality evaluation and designing approaches to improve data quality

EXTERNAL DISTRIBUTION (slide 2 of 4)

- **Course book in development as "open educational resources"**
  - Integrated theory, activities, and laboratories

Exercise 2.1: Virtual Tour of NIST SRMs

1. Watch the linked video from the YouTube channel, Veritasium, titled "The world depends on a collection of strange items. They're not cheap." (Veritasium, 2022, https://youtu.be/esQyYGezS7c)

(i) Would the materials covered in the video be classified as **physical standards** or **documentary standards**?

(ii) Identify examples discussed in the video of the benefits of SRMs toward the following sectors:

a. Environment

b. Human health or safety

c. Industry

d. Regulation

2. Visit the catalog of SRMs on the NIST shop, and navigate to the section specifically designated for nanomaterials. Select one of the nanomaterials from the list, and download the Certificate/Report for the SRM from its catalog page. Report the following:

(i) The name of the SRM

(ii) Nanomaterial properties reported in the certificate

(iii) Instrument used for each measurement

Exercise 2.2: Classifying Documentary Standards Related to Nanotechnology

Review the examples provided in The ABCs of Standards Activities of the different types of documentary standareds. Then, open the links below to review the lists of nanotechnology-related standards that have been published by ASTM and ISO:

- ASTM Committee E56 on Nanotechnology
- ISO Technical Committee (TC) 229 on Nanotechnologies:

Based on the title and abstract (freely available for standards published by either organization), provide one example from the available nanotechnology standards that can be categorized as the following types of standards:

(i) Terminology standard

(ii) Testing standard

(iii) Product standard

(iv) Process standard

(v) Standard on data to be provided

EXTERNAL DISTRIBUTION (slide 3 of 4)

- **Course book in development as “open educational resources”**
  - Integrated theory, activities, and laboratories

Exercise 3.1: Terminology standards for nanotechnology

Review the following two documentary standards related to definitions of nanomaterials:

- ASTM E2456-06(2020) — Standard Terminology Relating to Nanotechnology
- ISO 80004-1:2023(en) — Nanotechnologies – Vocabulary — Part 1: Core vocabulary

It is noted that ASTM standards may be freely accessible at institutions with an ASTM COMPASS subscription. At the time of this writing, the text of ISO 80004-1:2023(en) is accessible from the ISO catalog page linked above by selecting “Read Sample.”

1. How is a “nanomaterial” defined in each standard?

2. Read the definitions for “transitive” and “non-transitive” in the ASTM standard, as well as “nanoscale phenomenon” in the ISO standard. Is it required for a nanomaterial to exhibit a “transitive” property or “nanoscale phenomenon” to meet the definition of a nanomaterial?

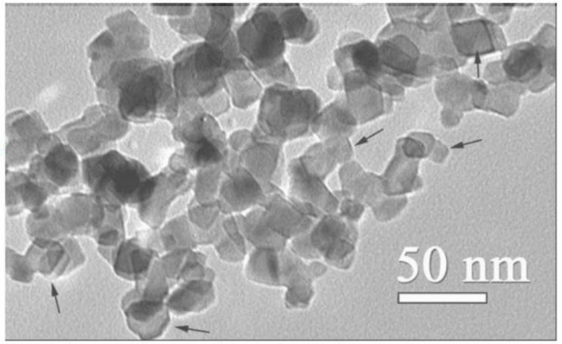
3. What is the definition of a “primary particle,” an “agglomerate,” and an “aggregate”? *Test your knowledge on this concept using Interactive Question 3.2 at the end of this chapter.*

Interactive Question 3.2

The Certificate of Analysis for **NIST Standard Reference Material® 1898 (Titanium Dioxide Nanomaterial)** presents the following set of TEM and SEM images demonstrating three different levels of structure: individual crystallites with sizes < 50 nm (arrows on top image), fused particle structures with sizes ≈ 100 nm (middle images), and large clusters of particles with microscale sizes (bottom images) that can be fragmented into the smaller structures with energy input, e.g., sonication.

1. Drag and drop the terms “primary particle,” “agglomerate,” and “aggregate” to label the appropriate structure in the image.

Note that the drop zone for the upper image refers to each individual crystallite indicated with an arrow (not the entire structure shown).



EXTERNAL DISTRIBUTION (slide 4 of 4)

- **Conference Presentations**
  - American Chemical Society National Meeting – Education Session, August 2023
  - Sustainable Nanotechnology Organization – Education Session, November 2023
  - ASTM International E56.02 (Nanotechnology – Physical and Chemical Characterization) – October 2024
- **Brochure of Course Book to be Distributed**
  - Sustainable Nanotechnology Organization – Fall 2025

OTHER OUTPUTS

- **Publications and Dissertations**
  - Meiraba Chirom, Chengzhen Qin, Feng Lin, Aniqa Lim, Steven Baldelli, Francisco Robles-Hernandez, Dong Liu, Viktor G. Hadjiev, Xiaonan Shan, Stacey Louie, and Jiming Bao. Non-contact thermometer for measuring surface temperature of photothermal catalysts using near-infrared black-body radiation spectrum. *Device*, 2024, 100467, DOI: 10.1016/j.device.2024.100467
  - Haoran Wu, Alaia Homawoo, Saba Shariati, Carlos E. Astete, Debora F. Rodrigues, Cristina M. Sabliov, Elham H. Fini, and Stacey M. Louie. Evaluation of drug release from polymeric nanoparticles in simulated saliva and gastric media by asymmetric flow field–flow fractionation (AF4). *RSC Pharmaceutics*, 2024, 1, 994-1007, DOI: 10.1039/D4PM00175C.
  - Riya A. Mathew, Effects of Heterogeneous Foulant Layers on the Photoreactivity and Aggregation of Titanium Dioxide Nanoparticles, Doctoral dissertation, 2023.
  - Haoran Wu, Evaluation of Drug Release from Polymeric Nanoparticles in Gastrointestinal Media, Doctoral dissertation, 2025.

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  - **Dr. Vince Hackley, NIST**
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  - Riya Mathew, UH
  - Haoran Wu, UH
  - Meiraba Chirom, UH
  - Abigail Fick, HCU*
  - Tchemongo Berte, UH
  - Shwetha Jayakumar, UH
  - Nusrat Antora, UH
  - Hong Zhong, UH
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