

# theta

MAGAZINE

p. 8

## REACTIONS IN THE SOLID STATE

a safer more  
environmental  
approach to  
chemistry

## ACROSS THE GLOBE

an inside look at travelling  
with the PROTO team

p. 10

SPRING 2018 / ISSUE 1

# FROM THE TEAM

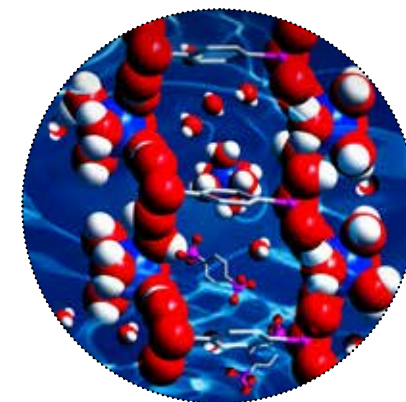
a magazine produced by PROTO

First off, thank you for taking the time to pick up a copy of 2Theta, a magazine produced by PROTO. We are proud to have published our first issue just in time for the 101st Canadian Chemistry Conference. We hope you enjoy reading through some of the diverse and interesting content we've put together for you. Thank you to our contributors and our extraordinary team that have made the launch of this magazine possible. We welcome any submission and suggestions for upcoming issues and encourage you to reach out to us at any time at [2theta@protoxrd.com](mailto:2theta@protoxrd.com).



2theta  
MAGAZINE

A magazine highlighting x-ray diffraction and its application in science and engineering.



4

## THE SCOOP

What's happening around PROTO

5

## NEW & NOTEABLE

Recent innovations to the product line

6

## HOLDING OPEN MICROPORES WITH WATER

Characterizing porous solids with XRD



8

## REACTIONS IN THE SOLID STATE

A safer more environmental approach to chemistry

10

## ACROSS THE GLOBE

An inside look at travelling with the PROTO team

12

## CONJUGATED MATERIALS

Towards wearable electronic devices

14

## UP & COMING

Interviews with emerging talent using XRD



17

## RIGHT BRAIN UNLEASHED

The artistic side of PROTO

18

## #FUNSCIENCE

When you need a break from the lab

# THE SCOOP

what's happening around PROTO



*As PROTO continues to grow, they are breaking new ground, not only with state-of-the-art technology, but literally with a brand new facility.*

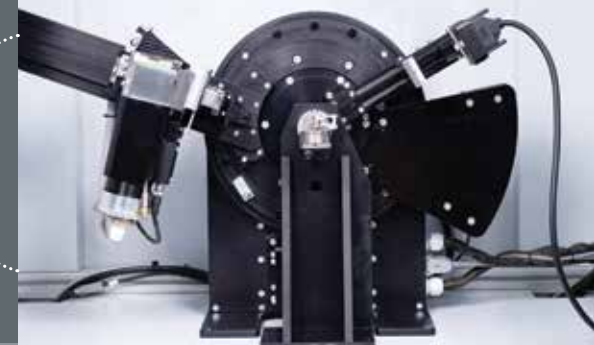
In the past three years PROTO has experienced significant growth across the entire company. With the introduction of new systems, development of additional product lines and constantly pushing the boundaries with technology and innovation, it was time to embrace the growth with the same enthusiasm that the team has continued to have over these past few years. With a twenty per cent increase in their workforce, and the addition of six new XRD systems to their product lines, it was time to find

*"We've quickly out-grown our current facility, and this move will be a great thing for our whole team and our customers."*

the space needed to embrace this exciting time. In 2017, PROTO acquired a new property in Windsor, Ontario. With plans to design and build a custom ground-breaking facility, the team is just buzzing with excitement. "We've quickly out-grown our current facility, and this move will be a great thing for our whole team and our customers," says Robert Drake, Sales Manager for PROTO. With plans to break ground in 2018, the 30,000 square foot facility will soon become the new home for the Canadian office.

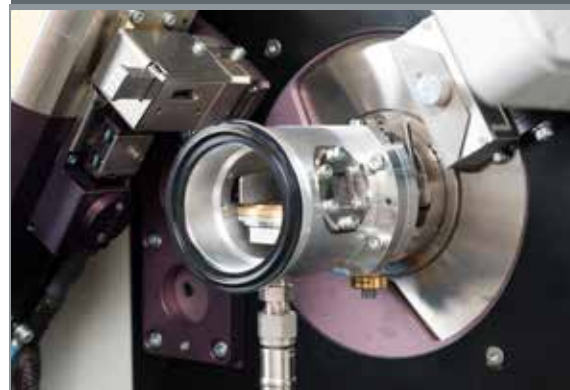
## AXRD LPD POWDER DIFFRACTOMETER

The LPD provides flexibility and customization in a lab unit configuration. It can be outfitted with cutting-edge technology including monochromators, X,Y sample stages, temperature and pressure cells, rotating phi stage and multi-sample exchangers.



## VARIABLE PRESSURE STAGE

PROTO has developed the only pressure cell available for benchtop diffraction systems. Investigate material-gas interactions directly at pressures ranging from  $10^{-3}$  atm (vacuum) up to 30 atm (440 psi).



## AXRD THETA-THETA SYSTEM

Designed to handle larger samples, or when more room is needed for experimental stages, such as temperature stages or pressure cells. The Flex-stage option makes mounting large samples easy, while the fully integrated cooling system keeps facility requirements to a minimum.



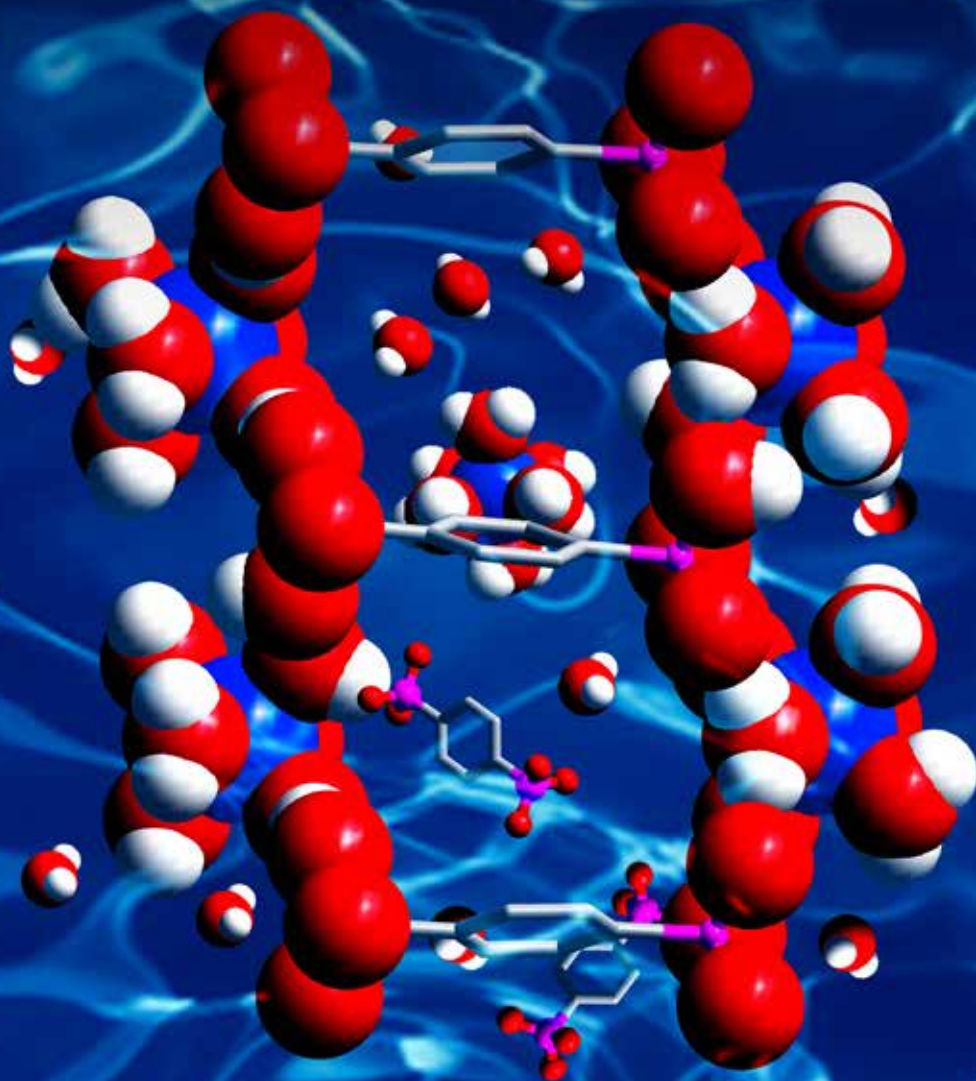
# NEW & NOTABLE

recent innovations to the product line



# HOLDING OPEN MICROPORES WITH WATER

Hydrogen-Bonded Networks Supported by Hexaaquachromium(III) Cations



Jared Taylor & George Shimizu use X-ray Diffraction to characterize the behaviour of porous solids.

Porous solids are materials that contain cavities, channels, or interstices. Depending on the size and shape of the pores and the chemical surfaces lining them, they can be used for applications ranging from separations and catalysis to sensing and delivery vessels.

Many of the newer classes of porous solids such as metal-organic frameworks (MOFs), covalent organic frameworks (COFs), polymers of intrinsic microporosity (PIMs), porous aromatic frameworks (PAFs), and porous molecular cages can be modified so as to tune the chemistry and engineering behavior of these materials.

At the University of Calgary, George Shimizu's group makes new nanoporous solids, primarily metal-organic frameworks (MOFs), directed by applications in gas separations and ion conduction.

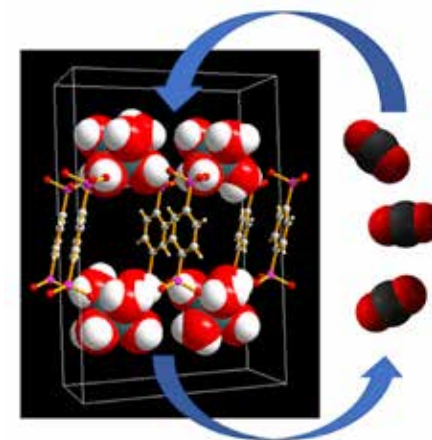
There are challenges to engineering these porous materials. "From surface enthalpic considerations, a void in a solid is always unfavorable," says Shimizu. There is an energetic penalty in forming a void and thus strong bonding and interactions play an important role in enabling pores to exist in these materials.

A second feature for modifying the performance of porous materials is order. Regular pore sizes and geometry give more predictable performance for adsorption and helps develop structure-property relationships. Weaker bonds such as hydrogen-bonds can form porous solids, provided sufficient complementarity and cooperativity exist between assembling partners.

A new family of hydrogen-bonded porous solids

was discovered and developed by the Shimizu group (CHEM, 2018, 4, 868-878)". These solids demonstrate reversible gas uptake in their pores and, owing to the hydrogen-bonded structure, can also show dynamic structures where pores open and close, like a gate, in response to the concentration/pressure of what is being adsorbed.

Along with the team at PROTO, research associate Dr. Jared Taylor, demonstrated this unusual feature occurred in a hexaaquachromium phosphonate structure where pore "gating" was observed at elevated pressures of CO<sub>2</sub>. In-situ



monitoring of the unit cell expansion at elevated CO<sub>2</sub> pressure was done using a PROTO AXRD Benchtop diffractometer outfitted with a pressure cell. This type of behaviour is unique to more weakly bonded solids and offers new avenues for smart sorbents and sensors.

# REACTIONS IN THE SOLID STATE: A SAFER, MORE ENVIRONMENTAL APPROACH TO CHEMISTRY

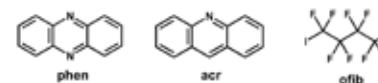
Making chemistry safer and more environmentally-friendly, while improving efficiency and speed, is a fascinating challenge being tackled by the Tomislav Friščić group at McGill University. A number of advantages are gained by performing chemical reactions in the solid state instead of using traditional solvents. Eliminating the use of solvents greatly reduces toxic waste streams while enabling access to novel reactions, materials, and molecules. So how does this “Chemistry 2.0” work? “Thinking about reactions between solids means you

must forget most things you know about chemistry and focus on surface, particle size, rheology, and nucleation effects,” says Friščić. Their latest paper published in *Crystal Growth & Design* (2018, 18 (4), 2387–2396) studies the bench stability of halogen-bonded cocrystals that are synthesized via mechanical milling, using a 10ml stainless steel jar that contains 7mm stainless steel balls. The milling is done for 15 minutes during which time the grinding action of the balls with a small amount of liquid (known as liquid-assisted-grinding



Igor Huskić, Graduate Student from the Tomislav Friščić group at McGill University, uses X-ray Diffraction and Rietveld analysis to understand solid-state reactivity mechanisms and kinetics.

or LAG) causes the reaction to occur. Halogen-bonded cocrystals involving the volatile halogen bond donor octafluoro-1,4-diiodobutane (ofib), with phenazine (phen) and acridine (acr) as acceptors are then synthesized. These types of materials are of interest, as halogen bonds have found use as building blocks for creating novel molecular solids and functional materials in crystal engineering.



These cocrystals are the first examples of acridine or phenazine combined with an aliphatic halogen bond donor and bench stability studies help provide further insight into halogen bond-driven cocrystallization as a route to stabilize volatile compounds in the solid state.

Crystal engineering by cocrystal formation is often focused on structural analysis and the development of more efficient designs and routes for synthesis. The overall stability of cocrystals is still not well understood. “This is surprising, as understanding and manipulating cocrystal stability are central to development of pharmaceutically relevant materials with improved solubility and in the

design of materials for the capture of volatile compounds,” says Friščić.

Cocrystal stability and characterization of the solid-state reactivity and phase transformations of these materials can be studied using Powder X-ray Diffraction.

As pioneers in this field, the group has to sometimes create novel equipment. Graduate student Igor Huskić developed a home-made environmental cell which can be placed inside their PROTO AXRD Benchtop X-ray diffractometer. The cell can be used to modify the atmosphere around the sample. Conducting real-time studies of solid-state reactivity and phase transformations within the environmental cell, is a central point of Huskić’s doctoral work.

Other not yet published work in his doctorate also shows how the environmental cell can be used for monitoring transformations of microporous metal-organic frameworks (MOFs) under different environmental conditions, as well as for development of solvent-free, clean transformations for extraction of rare elements from minerals under high humidity conditions.

Some of these novel materials can be evaluated for new ways to sequester CO<sub>2</sub>, store gases or extract critical elements.



# ACROSS THE GLOBE

an inside look at travelling with the PROTO team



*Our team shares with you some of their best experiences while travelling across the Globe for work.*

## BRIAN SIMPSON

*X-ray Detector Scientist*

As the lead X-ray Detector Scientist at Proto Manufacturing, I support our global customers to the best of my ability. In addition to my daily tasks, I'm often involved in installation and training of our Laue product line, which has allowed me to see more of the world than I'd ever imagined doing on my own. Despite my near total lack of sleep on trips, I can't help but feel energized when treated to such amazing locations and cultures.



▲ Anshun Bridge – Chengdu, China



▲ Isa Khan's Garden Tomb – Delhi, India

## I'VE BEEN TO...

*India, China, Poland, Turkey, and across Canada & USA*

## COOLEST THING I'VE SEEN

*Hiking up Yunding Mountain and visiting the temple complex near the top. (Near Chengdu China).*



## NATAN CARATANASOV

*Global Service Manager*

Being the Global Service Manager for Proto I am really forced to put the term Global in bold... No matter where I am in the world I am still connected with my peers and friends, at the same time making sure Proto has excellent reputation by providing top notch service to our customers, even despite my Jet Lag or time zone difference.

## I'VE BEEN TO...

*Japan, South Korea, Brazil, Argentina, India, South Africa, China, Germany, Poland, Czechoslovakia, Ukraine, Indonesia, Thailand, England, Ireland, Scotland, Mexico, Chile, Australia, France, Romania, Norway, Austria, and Netherlands*

## COOLEST THINGS I'VE SEEN

*The sunrise over the Arctic on a flight back to Japan, and the northern lights seen from Norway. Seeing the night sky and the stars under the Arizona Desert sky, is probably one of the coolest things I've seen.*



▲ City center square – Rzeszow, Poland



▲ TOP LEFT: Navy base on Coronado Island – outside of San Diego  
TOP RIGHT: Saturn V Apollo Moon Rockets at the U.S. Space and Rocket Center Museum – Huntsville Alabama

## KRISS LEFTWICH

*Application Scientist, U.S. Sales Lead*

As the U.S. sales lead, I interact with a lot of our foreign representatives. Since starting this job I have been to China and India. Upcoming international travel will include Poland, Scotland, Indonesia, and again to India! Last year, I was lucky to be in Hyderabad and Mumbai during Ganesh festival



## COOLEST THING I'VE SEEN

*My best friend's wedding in Jaipur with elephants everywhere.*

RIGHT:  
Indian food -  
Vada (a.k.a. wada).



▼ BELOW: Celebration of Ganesh Festival at IUCr in Hyderabad



# CONJUGATED MATERIALS - TOWARDS WEARABLE ELECTRONIC DEVICES.



The internet has connected our world in ways no one could have imagined. No longer just connecting our personal computers, the internet now connects our cars, phones, personal electronics, and even our refrigerators. This is just the beginning of the new “internet-of-things” that attempts to connect us in even more unique ways. We can imagine wearable devices built into our clothing and portable devices utilizing flexible screens that roll-up like a piece of paper. For this to become a reality, new materials with enhanced

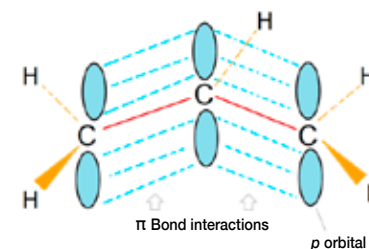
stretchability and robustness need to be developed.

Conjugated materials, especially polymers, are a particularly interesting class of compounds. Conjugated materials are materials where the p-orbitals of adjacent atoms are overlapped. This overlap causes a delocalization of the electrons in the molecule, resulting in favorable electronic properties that can be used in organic electronics. Additionally, these materials possess good solubility that allows device manufacturing and fabrication via ink-



The University of Windsor's Simon Rondeau-Gagné and his student Michael Ocheje use X-ray Diffraction to understand the next generation of novel materials for electronics.

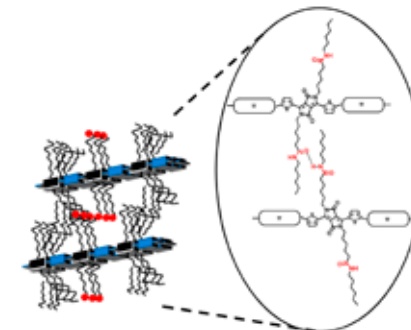
jet printing. “Despite these interesting properties, a lot of improvements must be achieved to expand their use in wearable and flexible electronic devices,” says Simon Rondeau-Gagné at the University of Windsor. His research group focuses on the development of novel strategies to design new nanostructured conjugated polymers with properties such as stretchability, self-healing, biodegradability, and near-infrared absorption.



One of Rondeau-Gagné's graduate students, Michael Ocheje, is currently investigating the incorporation of dynamic supramolecular interactions into  $\pi$ -conjugated materials in order to control their morphology or overall structure and allow for better mechanical compliance. In Ocheje's recent publication in *Macromolecules* (2018, 51, 1336–1344), amide containing alkyl side chains on

conjugated polymers are shown to form intermolecular hydrogen bonds between adjacent amide-containing side chains. This helps to control the morphology of the final polymer, which in turn can help improve the flexibility and stretchability of the material, without harming the electronic properties.

Ocheje uses a Proto AXRD benchtop powder diffractometer in his research to get a better understanding on the influence of dynamic interactions on the final thin-film morphology, which can be directly correlated to mechanical and electronic properties. Controlled self-assembly of conjugated polymers through hydrogen-bonding side chains is a promising strategy toward more efficient semiconducting polymers for thin film transistors and other organic electronics.





# UP & COMING

interviews with emerging talent using XRD

## MICHAEL OCHEJE

Graduate Student in Chemistry at the University of Windsor



### WHERE YOU WENT TO SCHOOL:

University of Windsor (B.Sc.), Riverside Secondary School

### PERSON YOU'VE LEARNED THE MOST FROM:

The person I've learned the most from is my graduate advisor. I've been introduced to a whole new and exciting field of science that affords me the opportunity to make significant contributions. I've also learned a lot about how to make my writing technical without sacrificing comprehensiveness, and the importance of collaboration and teamwork.

### MOST INTERESTING PROJECT YOU HAVE WORKED ON:

The most interesting project I've worked on would have to be the first one I pursued as a graduate student. It entailed the supramolecular assembly of conjugated polymers through the use of dynamic interactions towards the development of stretchable and self-healable conductive materials. We showed that installing hydrogen-bonding motifs through side-chain engineering is an effective strategy for imparting both stretchability and self-healing into otherwise nonstretchable conjugated polymers.

### HOW YOU HAVE USED XRD IN YOUR RESEARCH:

Knowledge of the solid-state, thin-film morphology of materials is very important for understanding the influence that our approach has on the final electronic and mechanical properties. XRD assists us in this respect by providing us information about the ordering of our materials on scales that we can correlate to final device performance.

### BIGGEST ACCOMPLISHMENT OR WHAT YOU'RE MOST PROUD OF:

My biggest accomplishment to date would have to be completing my Bachelor's degree.

### WHY YOU LOVE WHAT YOU DO:

I love doing research because it allows you to fully exercise your imagination and creativity in order to tackle your academic interests.



## IGOR HUSKIĆ

PhD candidate at McGill University's Department of Chemistry

### WHERE YOU WENT TO SCHOOL:

I finished my Bachelors' and Masters' degrees at the University of Zagreb, in Zagreb, Croatia. While there I worked on stopped flow solution kinetics.

### PERSON YOU'VE LEARNED THE MOST FROM:

It takes a village to raise a child and scientists are kind of similar. I'm not even going to mention the small army of professors that taught me during my schooling. My supervisor, Tomislav, has definitely taught me the most during these 5 years of my PhD. Another person who I must mention and who taught me everything about crystallography would be Dr. Vladimir Stilić from Zagreb.

### MOST INTERESTING PROJECT YOU HAVE WORKED ON:

It's like Sophie's choice, trying to pick a favourite project. I loved my project on naturally occurring metal-organic frameworks. The crystals I was getting with these materials were breathtaking. I also got to do neutron diffraction and scattering on these at the Oak Ridge National Lab which was pretty cool.

### HOW YOU HAVE USED XRD IN YOUR RESEARCH:

XRD is my go to tool. In my research I run accelerated aging reactions with solid reactants under non-ambient conditions. Powder XRD is probably the best method to see how the structure of the reactants vs. products changes in situ in real time. I also do a lot of single crystal studies of my products, or just for fun.

### BIGGEST ACCOMPLISHMENT OR WHAT YOU'RE MOST PROUD OF:

Our naturally occurring MOFs publication got picked up by a newspaper in Croatia and I got a phone call from my grandma that all the neighbors were dropping by and congratulating her.

### WHY YOU LOVE WHAT YOU DO:

I've always been curious about things, to a fault. I just want to know how things happen and why they happen, I can't leave mysteries unsolved. Also, you get to work with some terrifyingly smart people and you get to see how other people think. And being paid while doing it is just a happy coincidence.





**JARED TAYLOR**

*Lead for laboratory research at Sanjel Energy Services in Calgary*

**WHERE YOU WENT TO SCHOOL:**

I attended the University of Calgary for my BSc and PhD, then went to Kyoto University for 1.5 years as a Post Doc and 1.5 years as an assistant professor.

**PERSON YOU'VE LEARNED THE MOST FROM:**

Probably my PhD supervisor George Shimizu. He not only trained me in many aspects of materials and inorganic chemistry, but also how to write impactful papers and grants, and the value of networking to advance my career in the sciences.

**MOST INTERESTING PROJECT YOU HAVE WORKED ON:**

My recent project involving the synthesis and characterization of porous hexaaqua chromium(III) salts. The project involved significant utilization of techniques I was not familiar with, such as ab initio structure solutions from powder x-ray diffraction data, and in situ gas sorption/PXRD. It is always a pleasure to be able to learn new methods while working on a project you are passionate about.

**HOW YOU HAVE USED XRD IN YOUR RESEARCH:**

XRD has been fundamental to every paper I have published to date. Structural characterization from single crystals and PXRD; phase characterization, transformation and purity from PXRD; particle size analysis for nanomaterials; defect characterization and more. Its value cannot be overstated, as the ability to make predictions about structure-property relationships from quality x-ray data has driven the majority of my research.

**BIGGEST ACCOMPLISHMENT OR WHAT YOU'RE MOST PROUD OF:**

I would have to say it was the acceptance of my first publication from my research at Kyoto University. It was the first publication that I had fully written and gone through the submission and peer review process from start to finish. After many rejections, additional experiments and a final submission while coming down with food poisoning, it was finally accepted.

**WHY YOU LOVE WHAT YOU DO:**

I am fascinated with how the universe works, and how we go about refining and furthering our understanding of it. Being able to take some of that knowledge and apply it to manipulating and tuning solids at a molecular level, then realizing how and why those changes affect the macroscopic properties is what makes me love materials science. Being able to share what I've learned and advance some of our collective knowledge of science is what drives me.

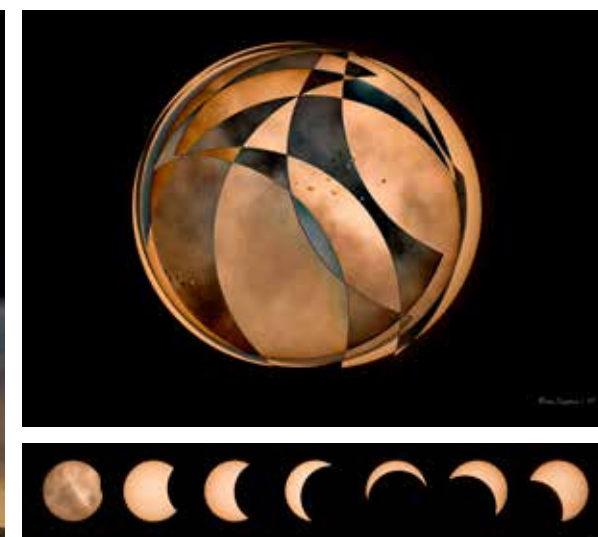
# RIGHT BRAIN UNLEASHED

The artistic side of PROTO.

*Application Scientist by day, Photographer by night. Although it is often believed that you are either 'left' or 'right' brain dominated, Brian Simpson proves that theory wrong with his magnificent portfolio of photography. This snapshot of his work shows his extraordinary talents as well as his unique ability to tie science together with art.*



**WHAT TO DO ON A FRIGID WINTER MORNING?** Photograph freezing soap bubbles! The cold air quickly freezes the inner layer of water, while a soapy film remains to support the bubble structure. At the nucleation sites, crystals form. Requires very cold, very calm air.



**"THE GREAT AMERICAN ECLIPSE."** I wanted an "artsy" way to display this amazing event. Before making the composite (bottom) I needed to align the individual frames in software. While playing with the layer settings in the software, I discovered that unique quality that I was looking for (top).

# #FUNSCIENCE

when you need a break from the lab

## FOLLOW THIS!

some fun & interesting social media accounts to follow in science



### BRIAN COX @ProfBrianCox

Cox is an author, physicist, and broadcaster... and has a pretty interesting twitter feed about all his travels.



### JOANNE MANASTER @sciencegoddess

Manaster is a faculty lecturer in biology, and her passion for science, support of women and sense of humour are apparent on her twitter feed.

## ONE HUNDRED YEARS AGO...

what was happening in the world

### ERNST OTTO FISCHER

Ernst was a German chemist who won the Nobel Prize for pioneering work in the area of organometallic chemistry was born in 1918.



### SPANISH FLU

In January 1918 the spanish flu was first observed in Kansas. By June of 1918 it became the 'spanish flu pandemic'

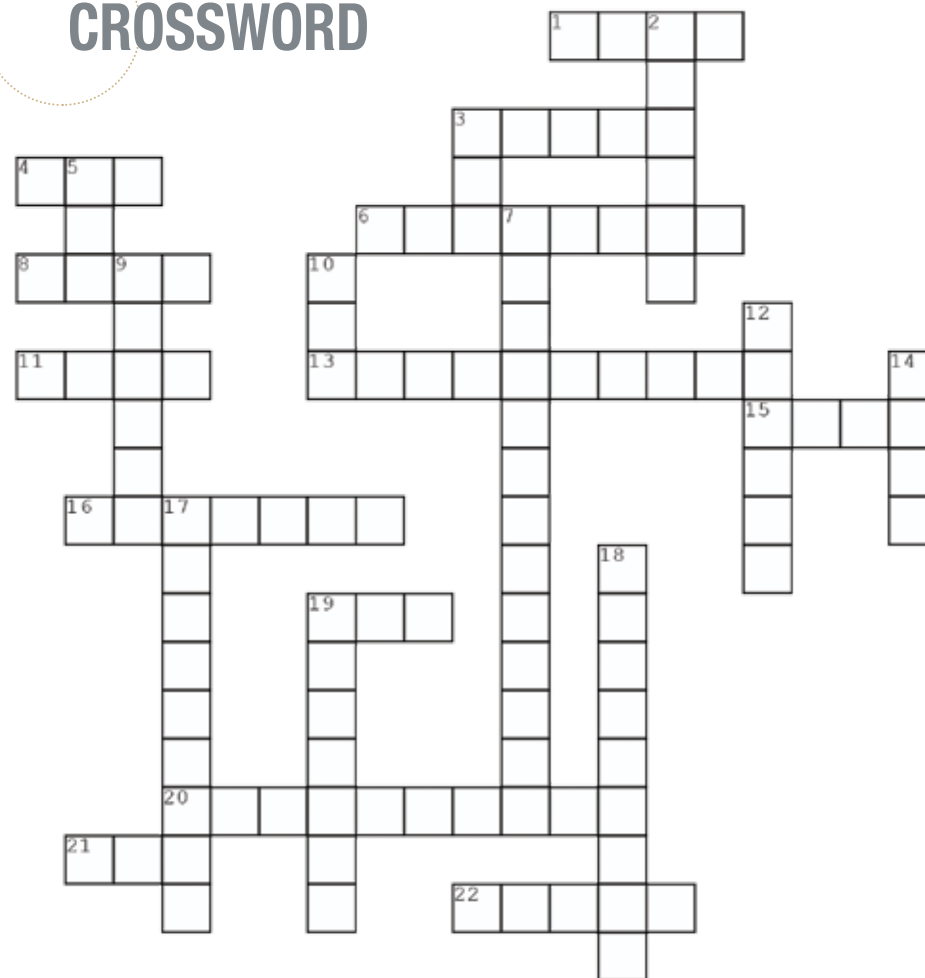


### POP-UP TOASTER

Although the first toaster was developed in Scotland in 1893, it wasn't until 1918 that the 'pop-up' feature was invented by Charles Strite.



## CROSSWORD



### Across

1. Ion source
3. Dynamite personality
4. Blind as a...
6. Tire gauge readout
8. Time of light
11. Bake in a kiln
13. one hundred and seventeen
15. Personal transport
16. White coat, typically
19. Valuable find in a mine
20. Bohr and Plank
21. A fixed charge.
22. Three is not a crowd for particles in protons

### Down

2. Greasers on ice
3. Calgary-to-Edmonton dir.
5. It may be on tap
7. Glasswear
9. Borealis
10. N. of Alta
12. Vessel
14. Music Genre
17. Great bear constellation
18. Charm
19. Quick learning method, much more gradual.



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