## A BRIEF HISTORY OF THE MOLECULAR AND MATERIALS SCIENCE PROGRAM AT TRIUMF

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#### THE EARLY DAYS AND SERENDIPITY

RIUMF and the other so-called 'meson factories' of the era were planned primarily for conducting research in particle and nuclear physics. These plans were dramatically affected by the pioneering work of Ken Crowe and collaborators at Lawrence Berkeley Laboratory (LBL) demonstrating the promising capabilities of muons to probe the fundamental chemistry and physics of materials. And so TRIUMF's fortunes took an unexpected turn in 1972 when Donald Fleming, then a new Assistant Professor in the Department of Chemistry at the University of British Columbia (UBC), received a phone call from Joe Cerny, his former PhD supervisor at LBL, who had just come from a colloquium given by Crowe on the subject of 'Muonium Chemistry'. Cerny thought that might be something Fleming could get started at TRIUMF, since TRIUMF promised the world's most intense beam of continuous-wave pion beams in the world, and this type of research (later dubbed µSR for 'Muon Spin Rotation, Relaxation or Resonance') would ideally capitalize on the prolific availability of the required polarized muons arising from pion decay.1 So, Fleming went back to LBL to meet Crowe and his group, including his graduate student, Jess Brewer. Brewer was tremendously enthusiastic, and Fleming soon became enmeshed in the  $\mu$ SR program underway at the (now defunct) 184" synchrocyclotron.

Serendipity struck again. On a neighboring beam line, a University of Tokyo group — Toshimitsu ('Toshi') Yamazaki, Shoji Nagamiya (later a Director of the JPARC Laboratory in Japan), Kanetada ('Ken') Nagamine, and their graduate student, Ryugo ('Ryu') Hayano — were conducting research using negative muon ( $\mu^-$ ) capture to probe nuclear radii. Yamazaki was a postdoctoral fellow (PDF) at LBL when Fleming was a graduate student, and they reconnected at that time.

#### SUMMARY

A brief history of the 45 year development of the materials science program at TRIUMF, from its serendipitous beginning in 1975 through to the Centre for Molecular and Material Science today.

Soon after in 1974-76, Yamazaki, Nagamime, and Hayano came to TRIUMF to participate in the commissioning of the original 'M20'  $\mu$ SR beam line. Hayano would later play a pivotal role developing the data acquisition system for the embryonic  $\mu$ SR program, and in fact submitted TRIUMF's first Ph.D. thesis on March 29, 1979. Brewer came to TRIUMF around the same time, driven by his desire to develop a worldclass  $\mu$ SR program, and to partake in the world-class steelhead fly-fishing scene on the Cheakamus river near Squamish. He soon became faculty at the UBC Physics Department after a stint as a Killam Postdoctoral Fellow. This early complement of people — joined soon after by Fleming's first graduate student, Dave Garner, who played a very significant role in the beginning and would go on to become TRIUMF's second Ph.D. — formed the nucleus for the original  $\mu$ SR group at TRIUMF.

#### THE M20 µSR BEAM LINE AT TRIUMF

The cyclotron designer, Reg Richardson, was TRIUMF's Director in 1974 during the machine's construction phase. The "T2" pion-production target station was being installed, with one port for pions for cancer therapy ('M8'), and one for  $\mu^-$  beams ('M9') for muonic X-ray (and related) studies. Brewer urged the Director to install a port for a dedicated  $\mu$ SR beamline before the shielding blocks were put in. Richardson agreed. A 'President's Emergency Grant' from UBC, strongly supported by Chemistry head Charles McDowell, provided \$25,000 in funding — meagre, but enough to get a program started.

The first M20 beam line was not a budget item at TRIUMF, so it was 'home-made' using surplus magnets and power supplies from other laboratories. See Fig. 1 for a photo of its installation in 1974. Key players in constructing M20 were Ken Crowe and David Measday at UBC Physics, both helping to procure magnets and power supplies from their network of international contacts. Thanks to Measday, the first bending magnet ('Patty Jane') came from Harvard University. Crowe helped procure the subsequent string of five quadrupole magnets from the



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<sup>&</sup>lt;sup>1</sup>Please refer to Ref. [1] for an introduction to  $\mu$ SR.



Fig. 1 Beamline 1A at the T2 production target shortly before first beam in 1974, showing its three original secondary channels (M9, M20 and M8) and photos of their respective designers: Mike Pearce, Jess Brewer, and Ken Kendall. The proton beam (BL1A) is coming from the bottom left in this photo.

University of Chicago, as well as the 2nd bending magnet, eponymously named 'Cal-Tech', followed by more 'Chicago' quads to focus the  $\mu^+$  beam.

The cyclotron proton beam was first extracted on December 15, 1974 (see article by Michael Craddock, this issue). The  $\mu$ SR era at TRIUMF was born just 7 months later on July 11, 1975, when the first  $\mu^+$  beam was delivered from the M20 channel (see Fig. 2). In those early days the M20 channel was tuned for 'forward muons', where the  $\mu^+$  produced from pion decay is emitted in the same direction as the pion momentum. The muon flux was minimal at first, gradually increasing with development of the cyclotron proton beam current, but nonetheless sufficient to initiate a robust molecular and materials science program (see e.g., Ref [2]).

The first published papers from M20 were in the field of magnetism, exemplified by Nishida *et al.* in Ref. [3]. Soon the first muonium chemistry papers began to appear, such as Ref. [4]. Muonium chemistry in low pressure gases was greatly facilitated by low energy "surface muons" (from pion decay at rest in the skin of the production target) — while these were observed initially on the M8 medical pion beamline, their widespread use at TRIUMF in  $\mu$ \*SR had to wait for the M20 channel. It was during this early period that arguably the most influential paper in  $\mu$ SR (to this day) was produced by the Japanese collaboration of Hayano *et al.* [5] using M20, which established the zero-field  $\mu$ \*SR technique which is to date a bulwark of  $\mu$ SR, e.g., in

probing the dynamics of quantum phase transitions, see e.g., Ref. [6].

The original M9 channel was upgraded in 1979-80 with a DC separator to remove unwanted  $e^+$  contamination. A test with surface muons on M9 demonstrated that the particle separator could also rotate the muon spins transverse to their momenta, allowing injection into strong magnetic fields. Such a "spin rotator" was incorporated into plans for an M20 upgrade in 1983, which enabled muon spin precession measurements in high magnetic fields parallel to the muons' momenta but transverse to their spins, a big advantage essential for (e.g.) Knight shift measurements. Spin rotation with surface muons was a global first and had a game changing impact on  $\mu^+$ SR worldwide.

M20 underwent a second refurbishment at the end of 2010, with major funding from the Canadian Foundation for Innovation (CFI).

The new design features a fast electrostatic kicker which can deliver a muon to one of two final legs independently (so-called "muons on demand" MOD), or deliver continuous beam to each leg simultaneously. Work was largely completed in August 2012, though kicker procurement issues have delayed implementation of the MOD capability. When commissioned, MOD will allow low-background  $\mu$ \*SR measurements on M20 out to many muon lifetimes — conferring to the heretofore continuous wave-only beamline a capability now enjoyed mainly by pulsed muon facilities.

Over the years, M20 has witnessed a multitude of groundbreaking experiments. Of particular relevance (beyond zero-field  $\mu$ SR) is an entirely new  $\mu$ SR experimental technique, called "Avoided Level Crossing Resonance" [7], whose muonium analog has become the mainstay of  $\mu$ SR's applicability in the chemistry of radicals.

# THE BEAM LINE THAT CLIMBS THE WALL: M15

The 'T1' target station on BL1A upstream from T2 initially had two beam ports: 'M11' for studies in pion physics; and 'M13' for studies using both pion and muon beams. M13 was suitable for some  $\mu^+$ SR experiments, but had an appreciable  $e^+$  contamination, making it less popular than M20. For some years M13 was used for numerous short trial  $\mu^+$ SR experiments, many of which led to new programs of research. Meanwhile, more and



has since been used mainly as a  $\mu^+$ SR facility.

## THE M9 DECAY-MUON CHANNEL

Though surface  $\mu^+$  beams are most in demand from the  $\mu$ SR community at TRIUMF, an initial kinetic energy of only 4.1 MeV limits their usefulness in probing dense materials. Forward muon beams are too energetic and have high  $e^+$  contamination; consequently, 'backward muons' (where the muon is emitted opposite to the pion momentum) with momenta typically 70-90 MeV/c remain essential for many experiments. Backward muons were also provided by the M20 channel, but more capacity was desired, particularly  $\mu^-$  beams for muon-catalyzed fusion studies.

Due to a strong commitment from the U. of Tokyo, who provided a superconducting solenoid with additional beam line components, a second "B" leg to the original M9 beam line was installed and became operational in 1988. It was commissioned by two founding fathers of  $\mu$ SR at TRIUMF, Ken Nagamine and Toshi Yamazaki, for their program of muon-catalyzed fusion studies (see e.g., Ref. [9]). The M9B leg would deliver backward muons from 40 MeV/c to 100 MeV/c, facilitating measurements on high-pressure solids, liquids and even gases in thick-

more users from Canada and abroad began asking for spinrotated surface muon beams on M20, which made it apparent that another such beam line was needed at TRIUMF.

Meanwhile, UBC physics Professor John Warren's group was anxious to search for spontaneous conversion of muonium  $(\mu^+e^-)$  to antimuonium  $(\mu^-e^+)$ , and so (then Director) Erich Vogt ordered construction of the world's best dedicated surface  $\mu^+$  beamline. There was insufficient real estate to accommodate a new beamline and experimental area on the floor of the Meson Hall, hence the new beamline (dubbed 'M15') had to go vertical (as shown in Fig. 3) and then south to a new purpose-built experimental area, two stories above. Warren's experiment [8] was the first to use M15, but the channel walled vessels. An official dedication for the M9B beamline was held in 1989 (see Fig. 4).

The backward  $\mu^{\pm}$  beams of M9B were first used for liquid-phase muonium chemistry experiments [10] and solid state samples in high-pressure cells [11], where surface muons could not penetrate the target containers.

After a serendipitous magnet coil failure, it was discovered that M9B could be tuned to produce transversely-polarized  $\mu^{\pm}$  beams by extracting them off-centre as they exited the solenoid, thus approximating the important advantages of ( $\mu^{+}$  only) spin-rotated surface muon beams. In a landmark experiment this capability was used to make high precision measurements of



Fig. 3 The vertical section of the M15 surface muon channel, installed and commissioned in 1984. At the top, M15 turns 90 degrees to pass horizontally through the wall and into the M15 Hall, which houses its dual DC Separators and a quadrupole triplet field lens for the world's only achromatic spin rotator.

relativistic shifts in muonic atoms, which illustrated the fact that the  $\mu^-$  wave function is essentially completely inside the nucleus for nuclei heavier than tin [12].

Most recently the negative muons from M9B were used to make the first measurement [13] of the chemical reaction rate of the heaviest isotope of the H atom, muonic helium (He++ $\mu^-e^-$ ), with a mass 4.1 times that of hydrogen, in a gas target at 500 bar pressure.

Eventually, age caught up with M9B's original infrastructure and it became impractical to operate and maintain. However, given M9B's worldly unique capability to produce a spin-rotated decay beam, the beamline's loss could not be long tolerated. In 2012. Simon Fraser University (SFU) Professor Paul Percival penned a letter (signed by 54 other molecular and materials scientists from around the world) making the case for the important and varied scientific program at M9B. This led to a CFI Innovation Fund proposal submitted by SFU Professor Jeff Sonier in 2016, which was eventually funded and dubbed 'M9H'. As of 2020 this project is actively underway and will eventually provide transversely spin polarized  $\mu^{\pm}$  beams into a variety of extreme physics and chemistry environments, supporting a broad program in quantum materials, green chemistry, energy storage devices, and even potentially, archaeological materials characterization.

## HIGH TC SUPERCONDUCTIVITY AND OTHER RESEARCH HIGHLIGHTS

Without question the emergence of the field of high temperature superconductivity (HTSC) brought the TRIUMF  $\mu^+$ SR program to the next level of prominence. In 1986, Bednorz & Muller discovered that Sr-doped lanthanum cuprate could be superconducting at the highest temperatures then recorded. By January 1987 a sample was brought to TRIUMF by Gabe Aeppli of Bell Labs to be measured with  $\mu^+$ SR (see Ref. [14]). Within a few months the even higher-*Tc* "YBCO" supercon-

ductor (YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub>) was discovered and samples from various groups around the world were brought to TRIUMF for testing with  $\mu^+$ SR, which had unique capabilities ideally suited for these materials.

Over the next several decades a huge variety of new HTSC materials passed through TRIUMF's  $\mu^+SR$  facilities, partly as a result of the establishment of a Superconductivity Program (later renamed "Quantum Materials") in the Canadian Institute for Advanced Research in 1987-8. From the outset the  $\mu^+SR$  technique was paired with microwave and other techniques at UBC, where the group of Hardy,



Fig. 4 (from right) Japanese Prime Minister Toshiki Kaifu, (then) TRIUMF Director Erich Vogt (red tie), BC Premier Bill van der Zalm, and BC Minister Stan Hagen, at the official M9B beamline dedication in 1989.

Bonn, and Liang eventually developed the world's most perfect single crystals of YBCO, after an initial sample prepared for a UBC Physics Open House display. This led to the demonstration [15] of the *d*-wave character of the superconductivity in YBCO, a breakthrough contribution to the field. To this day HTSC is one of the bread-and-butter staples of TRIUMF's  $\mu^+$ SR program.

### TRIUMF'S $\beta$ -NMR PROGRAM

In the early 1990s TRIUMF was in friendly competition with other  $\mu$ SR facilities around the globe, in particular the Paul Scherrer Institute (PSI) in Switzerland. PSI's very high intensity proton cyclotron enabled the practical realization of very low energy (LE) muons, the proof-of-principle having first been demonstrated at TRIUMF [16]. The LE muon flux at TRIUMF was far too low to compete, so a different approach to probing thin films and surfaces was needed. In 1995, (then) TRIUMF Director Alan Astbury approached UBC Physics Professor Robert Kiefl about the potential for a materials science program at the radioisotope production facility, ISAC, that was being proposed at the time (see ISAC History article, this issue). Discussions in the fall of 1995 led to the idea of using an ISAC-produced polarized 8Li isotope beam coupled with beta-detected nuclear magnetic resonance (βNMR) to realize a new probe of materials on the nano-scale.  $\beta$ NMR was well established - e.g., for characterizing states of impurity atoms implanted in bulk materials - but applications in materials research had languished due to the lack of dedicated infrastructure. Like the muon, 8Li nuclei decay asymmetrically, so the nuclear spin can be monitored via the anisotropy of the decay products. Unlike muons, which are created fully polarized as a consequence of pion decay, the 8Li must be polarized by inflight laser excitation, which was the most challenging part of the βNMR installation, ably realized under the leadership of TRIUMF's polarized beam expert Phil Levy. The combination of a large nuclear spin polarization, shallow depth, and signal detection via nuclear decay would enable NMR experiments complementary to LE µSR. In December 1995 the TRIUMF Experiments Evaluation Committee agreed with the argument for such a facility, so in 1996 Kiefl and others investigated <sup>8</sup>Li-βNMR's potential, finding applications in a variety of areas, including the nature of magnetism

in thin films, multilayer structures, and interfaces where conventional NMR would be unable to obtain a signal from such tiny amounts of material.

The "Workshop on Experiments and Equipment at Isotope Separators", held in Harrison Hot Springs, B.C. in April 1997 with presentations from several leading European  $\beta$ NMR experts marked the transition of this concept into reality. The promise built momentum in the community and spurred a dedicated effort to quickly realize the new facility. Several key aspects differed from all previous implementations of  $\beta$ NMR — in particular, drawing on the model of the  $\mu$ Sr facility at TRIUMF, it was envisioned as a permanent user facility expected to provide a new capability to a wide community of researchers on an ongoing basis.

In 1997, designs were initiated by Kiefl and a newly-hired post-doc, Gerald Morris, and the first  $\beta$ NMR experiments were approved. Construction began in early 1999 and by May 2000 the first experiment with polarized beam was performed — with a <sup>8</sup>Li beam rate of just 10<sup>7</sup>/sec, a huge resonance with a 50:1 signal-to-noise ratio was seen after a single 1-second pulse of beam! This made it clear that  $\beta$ NMR could accumulate high-quality data as quickly and efficiently as its more established  $\mu$ SR big brother and fill the LE  $\mu$ SR niche that TRIUMF was missing. The facility (Fig. 5) was commissioned in 2001 and during that year resonance and spin relaxation measurements on thin metallic films and



Fig. 5 UBC Professor Rob Kiefl in the cage of the  $^{8}$ Li- $\beta$ NMR facility he helped conceive.

insulators were performed, and the  $^8\text{Li-}\beta\text{NMR}$  program was off and running.

Subsequent facility improvements included a second spectrometer for beta-detected nuclear quadrupole resonance  $\beta$ NQR in zero magnetic field and low field  $\beta$ NMR was commissioned in 2003, with the high voltage systems for variable implantation energy added later in 2007.

Two of the earliest applications of BNMR studying metals and insulating perovskite oxides (related to the cuprate high Tc superconductors) remain some of the most important and clearly illustrate the unique power of the technique. In the first, the Teslarange magnetic field available for the first time in a BNMR experiment allowed the first reliable measurements of the Knight shift, a resonance shift in metals due to the weak polarization of the conduction electron spins. Remarkably, the "Korringa" relaxation of the initially highly-polarized spin towards thermal equilibrium could also be measured in metals, which, in combination with the Knight shift, offered all the power of conventional NMR in metals, but now in thin films and as a function of depth [17]. In contrast, the muon lifetime is so short that this type of relaxation is almost never observable. This clearly demonstrated that <sup>8</sup>Li, as a probe of solids, was not simply a heavier version of the muon, but a complementary modality sensitive to different phenomena by virtue of its million times longer lifetime.

The <sup>8</sup>Li nucleus is spin-2, enabling a coupling between the <sup>8</sup>Li nuclear spin and the gradient of the electric field at the <sup>8</sup>Li site.

The resulting splitting provides a useful fingerprint of the specifc crystallographic site of the 8Li when its symmetry is lower than cubic. In contrast, the muon is spin 1/2, a pure magnetic probe which doesn't feel this interaction at all. The quadrupole splitting in SrTiO, was by far the largest seen using 8Li, enabling a number of novel investigations, including a study of magnetism in mutlilayer structures of SrTiO, with LaAlO, [18]. Remarkably, the interfaces of these two nonmagnetic insulators exhibits both superconductivity and a form of weak magnetism!

## THE TRIUMF CENTRE FOR MOLECULAR AND MATERIAL SCIENCES (CMMS)

Prior to 1990, the  $\mu$ SR facilities at TRIUMF were not oriented towards being true 'user facilities', even though they were developing future

Canadian leaders in material science (e.g., Rob Kiefl at UBC and Graeme Luke at McMaster). But demand from visitors was increasing, prompting a new operational model. So in 1990, thanks to generous support from NSERC, TRIUMF, UBC, and SFU, the TRIUMF  $\mu$ SR User Facility was created to provide the most productive environment possible for visitors doing  $\mu$ SR at TRIUMF. The change had the desired effect of attracting many new users from across the globe, and gaining new prominence at TRIUMF, where it assumed a more significant role in the development of the lab's 2005 Five-Year Plan, specifically the intent to build a new (3<sup>rd</sup>) surface muon beamline in M9A.

The program's ambitions were growing with its prominence. After years of being dispersed around TRIUMF, the program finally gained a 'home' in 2002 when the former 'Batho' biomedical facility was turned into laboratory and office space for local facility scientists, grad students, and post-docs, as well as national and international visitors. The emergence of the BNMR program necessitated a name change, so in 2003 the "Centre for Molecular and Materials Science" (CMMS) was born. This was coincident with a major CFI application that led to a drastically enhanced infrastructure, including the two new surface muon beamlines, M20 and M9A, described above. The CMMS provided the umbrella for an improved organizational structure in concert with TRIUMF Management for both  $\mu$ SR and  $\beta$ NMR science that led to even further growth, culminating most recently in the M9H CFI project. A critical measure of TRIUMF's support was its response to the ongoing world-wide helium supply crisis, which threatened the life-liquid of the CMMS. In 2013 a dedicated liquefier was purchased and commissioned, thereby rescuing the CMMS and its Ultra Cold Neutron experimental neighbour from oblivion.

Today, the CMMS at TRIUMF is composed of a dedicated and experienced group of seven permanent scientists (all experts in specific aspects of the art of  $\mu$ SR/ $\beta$ NMR) and three technicians. The scientists are variously involved in managing the day-to-day operations of the Centre in parallel with a renewed emphasis in engaging their own and/or collaborative research initiatives.

As such, these efforts support a wide range of visiting scientists (including 5 CAP prize winners [19]) from practically everywhere in the world. About 100 peer-reviewed research papers are published each year in such diverse areas as atomic, molecular and chemical physics, reaction rate and free-radical chemistry, condensed matter physics, and nanoparticle science, with a small but important number of applied science efforts for industry thrown into the mix. With the deployment of the full capabilities of the M20, M9A, M9H and a significant increase of  $\beta$ NMR beam availability all on the horizon, the CMMS is looking forward to a long and ever more prosperous future.

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