# ANALYSING THE SWABBING PROCESS TO IMPROVE THE ACCURACY OF CHARACTERISATION IN THE NUCLEAR INDUSTRY

A THESIS SUBMITTED TO THE UNIVERSITY OF MANCHESTER FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN THE FACULTY OF SCIENCE AND ENGINEERING

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## Abstract

## ANALYSING THE SWABBING PROCESS TO IMPROVE THE ACCURACY OF CHARACTERISATION IN THE NUCLEAR INDUSTRY Thomas Johnson A thesis submitted to the University of Manchester for the degree of Doctor of Philosophy, 2022

The nuclear industry requires routine monitoring and the characterisation of environments before they can be decommissioned. Part of the characterisation of potentially contaminated assets is done through analysing swabs that are taken by operators and analysed in laboratories to identify chemical, biological and radiological hazards. The use of human operators in potentially hazardous environments has safety, cost and time implications; and this is accompanied by an understanding that human operators have limited reliability in swabbing scenarios. For these reasons, there is a desire to develop robots that can replace human operators in accessing and characterising radioactive facilities.

Swabbing is a sample retrieval process which involves the removal of contamination from a surface using a porous substrate (swab). There is a lack of understanding of the cause of uncertainties in the swabbing process and this hinders the applicability and usefulness of swabbing. This project provides an analysis of swabbing which can inform best practice, and aid the development of a new generation of swabbing robots.

This thesis makes major contributions to our understanding of swabbing, presenting a systematic evaluation of swabbing inputs, as well as providing the first study which measures human performance in swabbing tasks. In addition to this, a feasibility study which focuses on the use of Chrysoidine G as a chemosensor used for the detection of common radionuclides is presented in this thesis. In addition to the contributions from these experiments, the development of equipment and a number of novel methodologies are detailed.

The effect of swabbing force, force application area, contaminant mass and the number of swab passes on the pick-up factor of loose contamination are all studied, with these experiments providing a foundation to better understand the role of different swabbing inputs on the swabbing process. It was found that altering swabbing force did affect swab efficacy, with swabbing force causing a total variation of approximately 16% in mean pick-up factor performance; with a 6% minimum and 22% maximum mean performance. Force application area was found to influence pick-up factor to a greater extent, with an approximate 20% range in mean pick-up factors observed. These results also highlight the importance of leading-edge accumulation, with this mechanism greatly impacting overall swabbing efficacy.

Human operators are widely thought to be a large source of swabbing uncertainty, and studies have been undertaken to quantify this uncertainty. It was found that swabbing area recreation had a coefficient of variation (C.O.V.) of approximately 18% and swabbing force recreation was subject to far greater variation with a C.O.V. of 61%. These results provide clear confirmation that human performance in swabbing tasks has a low repeatability, and that human operators do contribute significantly to overall swabbing uncertainty. The repeatability achieved with automated robotic systems in these swabbing tasks is far greater than that achieved by the participants in this study, with these results confirming that robotic platforms could be used to reduce uncertainty in swabbing tasks.

Further to increasing sample retrieval capability, the development of *in situ* sample analysis techniques is a crucial step in removing humans from harm in the characterisation of radioactive facilities. A feasibility study for one such *in situ* characterisation technique was performed. Chrysoidine G (CG) is seen as a promising candidate for colorimetric detection of the abundant fission products, Sr-90 and Cs-137, which are known to be prevalent across many nuclear sites. This sensor's performance in likely conditions for its use is assessed. Ultimately the response of this sensor was found to be incompatible for use on an *in situ* characterisation robot. The colorimetric response of CG was found to be greatly affected by pH, necessitating careful pH adjustment before sensing could be performed.

This thesis contributes towards efforts in the nuclear industry to improve *in situ* characterisation capability, through providing a greater understanding of sample retrieval and through advancing our knowledge of chemical sensing techniques. It is thought that through performing more characterisation in environments, the safety, costs and time for decommissioning can be improved.

Through analysing the performance of human operators in swabbing tasks, it is clear that humans are likely a large source for error in swabbing, and thus the use of automated systems can improve the accuracy of swabbing. This thesis has demonstrated the key variables which are important in determining swabbing repeatability, and this will assist in the development of new swabbing robots.

# Declaration

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# Chapter 1

# Introduction

The 20<sup>th</sup> century saw the inception and development of the nuclear industry which has grown around using the nuclear fission process to create energy [1]. Fission leads to the creation of radioactive particles which have contaminated many sites across the world [1].

The first commercialised nuclear power plant in the United Kingdom, Windscale, and the facilities at Sellafield present a variety of different decommissioning challenges [2]; from environments where there is little to no knowledge of the contamination to rooms containing large quantities of radioactive dust from the milling of fuel rods [3]. In the United Kingdom, the Nuclear Decommissioning Authority (NDA) manages 17 nuclear sites which account for over 1000 hectares of land and house over 800 buildings which require decommissioning and demolishing [4]. These decommissioning efforts require over £3 billion per year [4]. Scientific and technological advancement is intended to contribute a large part to efforts in reducing the associated cost with decommissioning [4].

The current forecast from the NDA for the cost of decommissioning in the UK over the next hundred years stands at £234 billion [5]. This cost is borne out of the complexity of handling nuclear waste. Although some of the materials in nuclear sites can currently be recycled or reprocessed [6], most waste has to be sorted into different categories dependent on their level of contamination and then stored or disposed of accordingly [7].

The IAEA (International Atomic Energy Association) has highlighted the importance of accurate radiological characterisation as it can inform the whole decommissioning process [8]. Assessing how much contamination is in an environment and the chemical identity of that contamination is essential for safer and better tailored practices around nuclear decommissioning [5].

Advances in the characterisation process currently focus on three general areas:

- Developing characterisation techniques Developing techniques that can provide more accurate, more precise or new characterisation information can increase the accuracy of waste consignments and also help create better tailored decommissioning plans which are more cost-effective.
- 2. **Removing humans from the process** Removing the need for humans to characterise environments is a key way to increase the safety of characterisation tasks.
- 3. **Increase the accuracy of sampling** Sampling is the process of taking a portion of the material in an environment in order to make predictions about that environment. Increasing the accuracy of sampling contributes to more accurate characterisation on the whole, with accurate sample collection reducing the need for more costly *in situ* sensing.

The IAEA states that the primary objective of characterisation is to "obtain representative calculations, *in situ* measurements and samples/analyses which provide an understanding of the radiological conditions that will be encountered during decommissioning" [8]. As radioactive environments pose dangers to humans [1] it would be preferable to be able to characterise without requiring human operators to enter these environments. There are many characterisation tasks that could be completed by robotic platforms, and there is a growing desire to deploy robotic solutions [9].

Characterisation involves the identification and quantification of physical, chemical and radiological hazards, and this is central in ensuring safe operation in the nuclear industry. Further to this, there is a regulatory requirement to assess dose rates in different locations and to track the location of fissile material [6]. These regulatory requirements are important in the efforts to control the access of states and individuals to fissile material which may be used as part of a weapon. Though characterisation encompasses a wide range of analytical techniques, the focus of this thesis is on the chemical and radiological determinations which are made to support work in the nuclear industry.

Though the nuclear industry is the primary focus of the work in this thesis, regulatory requirements for characterisation are found in many areas outside of the nuclear industry. Characterisation is required in order to maintain food standards [10], in the testing of weapons facilities [11], assessing hazards such as asbestos in the decommissioning of buildings [12] and in the control of drug development facilities [13]. It is hoped that the impact of this work is not limited to the nuclear industry, and can inform practice across the diverse range of fields where characterisation in hazardous environments is required.

In the nuclear industry, characterisation is important through the entirety of the life-cycle a radioactive facility. Through construction and operation of a facility, routine inspection is a matter of course. After a radioactive facility has ceased operation, it must be characterised in order to inform decommissioning plans. Once decommissioning has begun, routine characterisation (for instance after post-operational clean-out) is used to assess the remaining radiological, physical and chemical hazards. As characterisation is crucial through the life cycle of a facility, efforts to improve characterisation can increase the safety and reduce the cost of construction, operation, decommissioning and dismantling [6].

Radioactive facilities pose challenges in access to both humans and equipment. Where the dose rate is known to be high, the time that human operators can be in that environment is severely limited or access is prohibited [8]. In addition to this, high dose rates can cause damage to electrical components, which can limit the utility of equipment in these facilities [14]. The precautions taken to operate in high dose-rate areas must also be followed in legacy facilities where the dose rate is unknown; this significantly increases the cost associated with decommissioning facilities which are unlikely to pose a great radiological risk.

In order to assess the radiological risk of facilities, characterisation can either be performed *in situ* or samples can be taken from an environment to be analysed *ex situ*. While *in situ* sampling does not require the transfer of potentially hazardous material out of an environment, there are situations where *in situ* analysis is either not possible, or not desirable. There are many areas that sensors are prohibited from accessing either due to physical or geometric constraints. Where the dose rate is high, some sensing equipment may not function or have a limited life time. This has led to some *in situ* sensing equipment being rendered disposable. This is resource intensive and further complicates decommissioning of these facilities [15].

Sample collection is preferred in areas where *in situ* sensing is impossible or impractical. Characterisation through sample retrieval and *ex situ* analysis is common in areas where dose rates are high. Swabbing (a common sample retrieval process, defined in Section 4.1) is used routinely in the characterisation of glove-boxes and the

routine inspection of equipment [6]. Samples are also taken to inform radiation surveys done before decommissioning of facilities [16] as they are analysed with equipment that cannot currently deployed *in situ* to give more rich characterisation information.

The primary challenge in sample collection is ensuring that the sample gives an accurate representation of the environment from which it is taken, while also reducing the amount of potentially hazardous material that needs to be collected. There is a trade-off between increasing the volume of a sample, which reduces the fundamental sampling error (described in Section 3.3.3) while also preserving the environment from which the sample is taken, with the retrieval of larger sample volumes being more destructive to the environment. Further to this, sample size reduction is of particular interest in the nuclear industry where every sample extracted from a potentially radioactive environment poses a radiological risk, and so it is good practice to minimise the size of samples in order to reduce the hazards associated with sample handling and transportation.

Existing research in 'nuclear robotics' has focused on automating the use of nondestructive evaluation (NDE) techniques in radioactive environments [15, 17, 18]. These robotic platforms are capable of collecting radiometric and some chemical information from environments; however there are many more characterisation tasks that are not currently performed by robotic platforms [16]. Human operators are still often required to retrieve samples from radioactive environments, with these samples being removed from environments for *ex situ* analysis.

### **1.1** Aims and Objectives

This project aims to increase the accuracy of characterisation in the nuclear industry, and to enable the development of robotic characterisation capability. The following list of objectives has been created to achieve these aims.

#### 1.1.1 Aims

• Analyse Swabbing Inputs to determine their effect on Pick-Up Factor - Determining Pick-up factor (the proportion of matter removed from a surface) is an essential part of sample-based analysis. This work aims to quantify the extent to which different factors affect the pick-up factor. This work focuses on factors (swabbing inputs) which are controlled by the operator (and potentially in the future controlled by autonomous swabbing systems) in order to determine the most impactful swabbing inputs.

- Measure human performance in swabbing tasks There is an existing assumption that lack of repeatability from human operators is a major contributor to swabbing uncertainty. It is important to test this assumption so that we can determine which factors are most important to control when attempting to increase the accuracy of swabbing tasks.
- Develop analytical techniques which can be deployed *in situ* on a robotic **platform** It would be greatly advantageous to be able to provide full chemical and radiological characterisation of facilities *in situ*. To do this, there is a need for facile sensing techniques which could be readily deployed on a robotic platform.

### **1.2 Thesis Structure**

This thesis is presented in a sequence to aid readability and provide a consistent 'research arc'. Three technical chapters are presented in this thesis, with the remaining four chapters containing an Introduction, two extended literature reviews and conclusions. A brief description of each chapter is given below:

- 1. **Introduction:** This chapter provides an overview of the decommissioning challenges that the work from this thesis helps to address. There is also a statement of the key research outcomes from this thesis as well as a description of the thesis structure.
- 2. **Inspection Robotics:** This literature review gives a historical perspective on the development of robotics for the inspection of radioactive environments as well as highlighting the state of the art and the challenges that inspection robotics must address in the coming decades.
- 3. **Sampling and Swabbing:** This literature review details available literature on the theoretical underpinning for the adhesion and removal of contamination and subsequently analyses the sampling process, focusing on errors in sampling. This is followed by a review of available studies on swabbing, finishing by highlighting limitations of the current body of research.

#### 1.3. RESEARCH OUTCOMES

- 4. Determining the Effect of Swabbing Inputs on Pick-Up Factor: This technical chapter explores the relationship of various swabbing inputs with pick-up factor. The chapter begins with details of the methodologies developed and used in this chapter and is proceeded by results from the swabbing experiments. This chapter is concluded by a summary of the main findings from these experiments.
- 5. Human Performance in Swabbing Tasks: This technical chapter presents the findings from experiments which determined human performance in swabbing tasks. This is preceded by details of the experimental design.
- 6. Chrysoidine G for the Detection of Common Fission Products: This technical chapter presents the findings of an experiment looking at the viability of Chrysoidine G as a chemosensor for use in the nuclear industry.
- 7. **Conclusions:** This chapter provides an overview of the findings and outcomes of this PhD project. This chapter also analyses the research area and potential for future work.

While there are two separate literature review chapters, further context is provided in the technical chapters to aid readability; elucidating the motivations for the experiments and methodologies used.

### **1.3 Research Outcomes**

This thesis details contributions made in this PhD project, which are covered in greater detail in the subsequent chapters of this thesis.

- The Development of Techniques to Evaluate Swabbing Performance: This project required the development of new equipment and techniques to give us a greater understanding of the swabbing process. A force-controlled swabbing system, and a force-sensitive swabbing puck were developed for this purpose. A novel imaging technique which enabled rapid visualisation of contamination distribution across a swab, and on the swabbing surface was also developed.
- A Systematic Evaluation of Different Factors which affect the Swabbing Process: This project provides the first systematic evaluation of the effect of force input on the swabbing process. The effects of contaminant mass and swabbing area are also explored. These experiments give indications as to which

swabbing inputs are important to control in the swabbing process, and this can greatly aid the development of future swabbing robots.

- An Evaluation of Human Performance in Swabbing Tasks: Human operators are required to perform swabbing tasks across a range of industries and applications. It is expected that the force input of these operators is highly variable and this has a negative impact on the repeatability of swabbing. This project provides the first evaluation of human performance across multiple swabs. This work tests the assumption that human operators are a major reason for poor performance in swabbing.
- An Evaluation of a chemosensor, Chrysoidine G, used for the detection of caesium and strontium: Recent reports have demonstrated that Chrysoidine G may be a viable chemosensor for use in the nuclear industry. This thesis presents a paper which determines whether this chemosensor may be a viable candidate to provide *in situ* sensing during the characterisation process, identifying the challenges of progressing sensor technologies developed in laboratory conditions to deployment on-site through a robotic platform.

## **Chapter 2**

# **Inspection Robotics**

This chapter provides a literature review of inspection robotics, with a particular focus on robotics developed for and relevant to the nuclear industry. This review gives a historical perspective on the development of robotics in the nuclear industry, and highlights developments which are of interest to the development of swabbing robots.

The inspection robots which are described in this section demonstrate the ability for robotic platforms to successfully utilise a wide variety of inspection equipment, navigate autonomously and take samples from their environment. The robots described in this review are summarised in Table 2.1.

Robot(s) (Year)	Autonomous Navigation	Chemical Characterisation	Physical Characterisation	Radiological Characterisation	Sample Retrieval for Ex Situ Analysis	Sample Retrieval fo in situ Analysis
Rover, Louie I, Louie II, Workhorse, MOOSE [19] (1987)	х	Х	Camera feed.	Radiometric survey capability.	$\checkmark$	х
Robin, Surveyor, IMR, Amooty, Surbot [20] (1989)	~	Х	Camera feed.	Х	Х	х
MACS, RCS, Nomad, TSEE [21] (1997)	~	Х	Camera feed. Magnetometer. Ground penetrating radar.	Radiometric survey capability.	~	Х
Beagle 2 [22] (2002)	~	XRF spectrometer. Mossbauer spectrometer.	Camera feed.	X-ray detection to indicate alpha radiation.	х	~
LineScout [23] (2008)	Х	X	Camera feed.	Х	Х	Х
TALON, BROKK, PackBOT, Quince, Warrior, JAEA-3 [15] (2012)	~	Х	Camera feed. 2D and 3D reconstruction. Thermal imaging.	Radiometric survey capability. Gamma ray camera.	~	х
Curiosity Rover [24] (2013)	~	X-ray spectrometer. X-ray powder diffraction and fluorescence. LIBS. Neutron detection system to indicate hydrogen/water content.	Camera feeds. Humidity sensor. Pressure sensor. Temperature sensor. Wind sensor. UV radiation sensor.	X-ray detection to indicate alpha radiation. Gamma dosimeters.	х	~
RICA [25] (2017)	Х	x	Thermal camera. Camera feed.	Radiometric survey capability. Gamma ray camera. Gamma spectrometer.	~	х
DiddyBorg [26] (2017)	~	Х	Lidar for reconstructions. Camera feeds.	Х	Х	х
CARMA [17] (2018)	~	Х	Camera feed. 2D reconstruction.	Gamma counter. alpha/beta dosimetry.	х	х
TORONE [27] (2020)	~	LIBS	Stereo camera feed. 3D reconstruction.	Gamma counter. Alpha/beta dosimetry. Neutron Detection.	Х	Х

## Table 2.1: A summary of the capabilities of inspection and characterisation robots

### 2.1 Robotics for Characterisation

This section will review literature that is relevant to the development of ground-based characterisation robots for radioactive environments. While the focus of this review is on inspection capabilities that are of use in radiological characterisation, this review is not limited to research for the nuclear industry. There is a wealth of research from other sectors that can be considered useful in the development of characterisation robots.

## 2.1.1 Ground-Based Characterisation Robots Used in the Nuclear Industry

The application of robotics in the nuclear industry has long been required due to the inherent danger associated with exposure to radioactivity. In fact, ever since the inception of commercial nuclear power, remotely operated machines have been utilised [28, 20]. There has always been a desire for robots to take over more of the processes associated with maintaining and decommissioning nuclear power plants, with the first mobile prototypes being developed in the 1960s [29, 30]. This section details the historical development of robots used for the inspection in the nuclear industry, leading to the robotic platforms that are currently being used and developed in the industry.

This section concludes by highlighting the areas where further development must be made to inspection robots so that they may be used to replace human operators in the characterisation process.

The advantages of robots being used in the characterisation of environments are numerous, enabling safer, quicker and cheaper operation. Utilising the repeatability of robotic systems also promises more accurate characterisation.

The 1960s saw the first examples of robots being used to help with operations in nuclear power plants. Mobot [29], Little Ranger [30], Koelsch Models 601RC [30], 602RC [30], M2 maintenance system [28] and SM-229 [28] were examples of remotely operated manipulators that could handle large solid samples using a jaw gripper and also provide a camera feed to operators. These fully tele-operated manipulators gave operators the chance to interact with the environment in closed-cells that contain radioactive contamination levels which prohibit human entrance.

With the Three Mile Island accident in 1979, the need for robotics in the nuclear industry became more pertinent. The mobile platforms ROVER, LOUIE I and LOUIE II were developed for the clean up at Three Mile Island [19]. These platforms were tele-operated and could perform radiation surveys. These are the earliest reports of

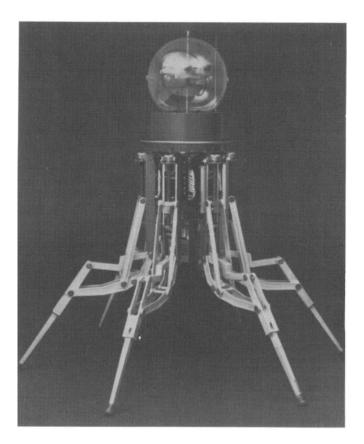


Figure 2.1: ODEX I: an early legged robot for cluttered environments [28].

robots being used for radiometric monitoring in the nuclear industry. ROVER was also fitted with an arm and an end-effector capable of sludge sampling. In addition to radiation survey robots, the Workhorse and MOOSE [19] were used in order to help with dismantling and clear-up of Reactor 2 at Three Mile Island.

The 1980s saw the development of the first non-manipulator style robots used in the nuclear industry. ODEX I [28], Robin [20], Surveryor [20], the Intelligent Maintenance Robot (IMR) [20], Amooty [20] and Surbot [20] were all developed to perform visual and radiological maintenance tasks in radioactive environments. Robin and ODEX I were legged robots which used feedback from touch sensors to navigate through cluttered environments. SURVERYOR II could climb stairs and also autonomously place inspection optics. Amooty and Surbot, developed in Japan, were capable of navigation through pre-mapped environments employing some level of autonomy. Amooty's autonomous capability used visual feedback to aid position control.

The events in 1986 at Chernobyl created environments where mobile robotic platforms were a necessity. Platforms such as, STR-1, Joker and Pioneer 1 [21] demonstrate the development of robotic systems in the aftermath of Chernobyl. Pioneer 1 was

#### 2.1. ROBOTICS FOR CHARACTERISATION

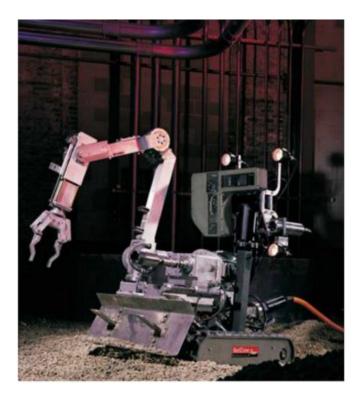


Figure 2.2: A picture of Pioneer 1 [21].

capable of 3D mapping and was designed to continue operating in highly radioactive environments, whereas STR-1 and Joker were designed to clear rubble.

MACS and RCS were used to provide radiation survey maps in various uncluttered environments across the Chernobyl site [21]. RCS and MACS both demonstrated an increasing level of autonomy for inspection robots. Both could avoid collision with walls using proximity sensors and would travel autonomously executing a random walk. The deployment of these robots marks the first reported deployment of robots that could perform monitoring tasks without supervision. The deployment of RCS also demonstrated an increased inspection capability through the presence of a ground-penetrating radar and a magnetometer.

Unfortunately, innovation in robotics for the nuclear industry slowed considerably in the years after Chernobyl [9], and consequently there are still many areas where nuclear robotics technology falls short of the demand in the industry. Currently, there is a need to develop greater sample retrieval capability as human operators are still used predominantly in sample retrieval tasks[8] [31]. This is not desirable due to the risk and associated cost of human exposure.

The Fukushima-Daiichi incident and clean-up operation demonstrated that there



Figure 2.3: The JAEA-3 platform [15].

was still a great deal of development required to more safely decontaminate and monitor nuclear environments [15]. This incident prompted more rapid development of robotics for the nuclear industry once again [9]. An example of the robots used in the response to the Fukushima incident is shown in Figure 2.3.

Robots were deployed in Fukushima to perform characterisation tasks as well as assisting with demolition and clean-up operations. Most of the robotic operations in the initial response to the Fukushima incident were completed by robots with little to no autonomy. The PackBOT [32] was a tele-operated rover that was equipped with the RC-1 that was developed for characterisation tasks in Fukushima. The RC-1 contained a gamma imager, web-camera, teletector, generator, 3D Lidar and a thermal camera [15]. The RC-1 equipment was also deployed on the TALON platform [33] as well as the JAEA-3 platform. The JAEA-3 [15] was used to provide radiometric maps of environments in the Fukushima-Daiichi plant.

Robotic development for the nuclear industry since the events at Fukushima has focused on increasing the level of autonomy of robotic platforms. Platforms such as the CARMA [17] and RICA [25] can now provide radiometric heat maps of environments with minimal human intervention. These platforms use commercial radiometric sensors. CARMA uses an  $\alpha$  sensor as well as  $\gamma$  dosimeters. These platforms provide useful information while reducing the exposure to ionising radiation for plant operators, however there remains a large amount of characterisation information from these environments that cannot be collected by current robotic platforms.

#### 2.1. ROBOTICS FOR CHARACTERISATION



Figure 2.4: The CARMA platform [17].

In order to gain more complete characterisation information from these environments, and ultimately eliminate the need for human operators to enter hazardous environments, the capability of inspection robots to perform different characterisation tasks must be increased. Platforms in the nuclear industry have limited capability to collect data about environment chemistry. Current research utilising laser diagnostics and other analytical techniques [18] exists, with TORONE representing the current state of the art for a non-contact characterisation robot [27, 34], in practice operators are still be required to enter these environments to use sensors and retrieve samples that are analysed in a separate lab, and so there is still a need to broaden the capability of robotic platforms deployed in to radioactive environments.

A summary of the robots discussed in this section can be found in the summary table in section 2.1.3. This table demonstrates the increasing levels of autonomy and inspection capability of characterisation robots in the nuclear industry, as well as the limitations pertaining to the characterisation information that these robots provide.

#### 2.1.2 Characterisation Robots in Space Exploration

Robots used for the characterisation of environments in space can be split in to two categories; the first of which being robots that perform *in situ* sensing and the second being robots designed to perform sample-return missions [35]. *In situ* sensing missions use rovers that can collect data in their environment without the need to transfer material back to Earth. Sample-return missions require samples to be retrieved for later analysis. There have been six successful robotic sample-return missions with the first being the Russian Luna 16 probe which launched in 1970 and brought approximately 100 g of lunar soil back to Earth [36]. There are fewer examples of platforms providing

*in situ* analysis, mainly the unsuccessful Beagle 2 lander [22] and the Curiosity rover currently in operation [24]. These two types of missions offer different advantages. The use of sample-return missions allow for samples to be analysed using more complex assay techniques and techniques that may not have been developed at the time of launch. However *in situ* analysis allows for results to be collected without posing the challenge of safely transferring material back to Earth.

Recent developments for *in situ* analysis are shown by the Curiosity rover [24]. The curiosity rover provides visual inspection capability [37] with non-contact environmental monitoring equipment [38] and assay techniques including Laser Induced Breakdown Spectroscopy (LIBS) [39] and alpha particle X-ray spectrometry [40]. Curiosity can measure pressure, wind, temperature, UV radiation, neutron dose and total radiation dose as well as recording visual information and processing dust and rocks to extract information about the chemistry and mineralogy of the Mars atmosphere. Curiosity provides a much more comprehensive characterisation suite than any competing platforms and demonstrates that integrated characterisation platforms can be designed and can be used to provide useful information.

The Perseverance rover demonstrates further developments for characterisation in space exploration platforms [41]. Perseverance provides stereo and panoramic imaging capability, a wide range of environmental sensors, ground-penetrating radar, X-ray fluorescence spectrometry, UV Raman spectrometry as well as having the ability to cache samples [42].

#### 2.1.3 Other Inspection Robots

Inspection is important in many sectors, and there are many examples of robots being used for inspection tasks. Although this project is targeted at the nuclear industry, it is valuable to determine whether developments in inspection robots for deployment in other sectors could be adapted to help with the inspection and characterisation of environments in the nuclear industry.

Many robots are developed only to perform visual inspection. Camera images are fed back to human operators in the inspection of power lines [23], crops [43, 44], pipes [45] and to monitor bridges [46] to name some applications. Current Lidar technologies allow robots to create full 3D reconstructions of environments [47]. These reconstructions are now commonplace and are used in the construction, defence and emergency response scenarios [26, 48, 49]. Robots are often designed to use visual inspection equipment (Lidars and cameras) for SLAM (simultaneous localisation and

mapping) and autonomous navigation as well as collecting data about environments [26, 43, 17].

Robots are now commonly employed in environmental science as data collection tools [50]. Temperature sensors, gas sensors, pH probes and humidity sensors are commonly utilised in the inspection of environments [50]. Robots used in this area often navigate autonomously for long periods of time while collecting environmental data [51]. Further to autonomous capabilities, it is common for environmental robots to be able to collect aquatic samples[52] and gaseous samples [53] using collection chambers.

CBRN robots [54], [55] are designed to assist in the assessment and clean-up of chemical, biological, radiological and nuclear material. ORPHEUS [54] is a robot designed to autonomously navigate through environments while providing a radiological heat map. The RESCUER [55] can perform radiological mapping tasks as well as use an arm to take samples of an environment.

#### 2.1.4 Discussion

This review provides an overview of the history of development of inspection and characterisation capability for robots through to current platforms. The robots discussed in this section are briefly summarised in Table 2.1. This section has demonstrated the ability for robots to assist in the characterisation of environments as well as highlighting limitations of these platforms.

The nuclear industry uses robots to assist in the characterisation of radioactive environments. Robots provide a potential solution to the issue of human operators entering these hazardous environments to collect information. Current platforms such as CARMA [17] can be used to collect radiometric data and visual information with minimal human intervention. Although radiometric data can provide useful information about environments, it cannot provide a full understanding of the environment and this means that robots cannot currently be used to replace humans fully in the characterisation process. In order to do this, platforms capable of providing more complete chemical and physical characterisation data must be developed.

One key task in the characterisation of environments is sample retrieval. Sample retrieval is important because full information about an environment cannot currently be collected without being able to interact (physically and chemically) with material. Commonly samples are taken from environments to be analysed *ex situ* in a lab. In the nuclear industry this is far from ideal as this process takes a long time and introduces

risk due to the transfer of potentially hazardous materials. RICA [25] is a robotic platform currently capable of the retrieval of large solid items for *ex situ* analysis using a jaw gripper, however this sample type may pose radiological risks and is of limited analytical use in a laboratory. It is clear that platforms for the nuclear industry need to develop the capability to be able to retrieve different sample types. Without the ability to collect samples which are of use analytically (see Section 3.3.1 for a discussion of the factors which affect the analytical utility of samples), the goal of *in situ* sample analysis for the nuclear industry remains far off.

Characterisation robots used in space exploration have similar objectives and similar challenges to robots used in the characterisation of nuclear power plants. The Perseverance [41] and Curiosity rovers [24] act as the current state of the art for *in situ* analysis of samples and chemical characterisation capability. Curiosity has a powerful suite of analytical techniques including LIBS and X-ray fluorescence which can provide a wealth of characterisation information when used on appropriate samples. Developing a robot with similar capability to Curiosity for the nuclear industry could have a profound impact on the nuclear industry.

A limitation of current work is having some assurance about the sampling process. Without appropriate feedback, there is no assurance that a retrieved sample is representative of the area sampled. This can lead to biases and errors that can void the usefulness of analysis. Gaining assurance in the sampling process would provide a step-change in how samples can be used analytically. Specifically, samples may be able to be used for quantitative analysis and they could be treated as entirely representative.

### 2.2 Sample Retrieval Robotics

Retrieving samples for analysis is a crucial step in the characterisation process [8]. This review will detail the sample retrieval mechanisms that are used in the characterisation process. Further to this there will be a focus on how robots have been used in the past to retrieve samples, both in the nuclear industry and in other areas. This section will determine whether current robotic technology can be used to effectively assist with intrusive characterisation and also highlight areas where more development is required.

Table 2.2 summarises the robotic sample retrieval mechanisms that are discussed in this section.

Robot Name (Year)	Sampling Mechanism	Sample Retrieval Capability	
NEATER [56] (1993)	Dry swabbing.	Particulate contamination.	
Rocky 7 [57, 58] (1996, 2002)	Jaw gripper.	Large solid items and fine particulates.	
Beagle 2 [22] (2002)	Soil sampler and rock corer.	Rock samples and fine particulates.	
Curiosity [24] (2013)	Soil scooper and rock corer.	Rock samples and fine particulates.	
Rescuer [55] (2015)	Jaw gripper.	Large solid items.	
RICA [25] (2017)	Jaw gripper.	Large solid items.	
Perseverance [42] (2021)	Coring device	Regolith samples.	

Table 2.2: A summary of robotic sampling mechanisms

#### 2.2.1 Methods for Sample Retrieval of Solids

Sample retrieval methods used in the nuclear industry have remained largely unchanged since the 1960s [59, 60]. There has been little desire to develop new techniques while established techniques are capable of retrieving a range of samples which can be used for analytical purposes. Methods for sample retrieval are selected based on the specific constraints of the environment being sampled as well as the requirements for the desired analysis techniques.

The most basic and ubiquitous form of sample retrieval is the taking of dry swabs [59]. This method involves using a piece of paper (generally filter paper is used as its properties are well-known) or cotton swab, and applying pressure while swiping across a surface. This method can retrieve loose contamination from a surface and is employed widely in the nuclear industry [61].

Where dry swabs cannot extract sufficient amounts of contaminant from a surface, a wetting agent to increase the pick-up of stubborn contaminants [59, 62]. Wet swabs exhibit increased pick-up factors but have the significant disadvantages of increasing the attenuation of alpha radiation (making subsequent radiometric analysis more challenging) and also potentially creating additional liquid waste. These disadvantages make wet swabbing far less common in the nuclear industry.

Adhesive tapes can also be used to extract samples in a similar way to dry swabbing [59]. Adhesive tapes have the advantage of firmly holding contamination and offering spatial resolution to measurements, however they are generally much more difficult to process for chemical analysis.

Various reports have investigated the efficacy of using suction methods to retrieve

loose, surface contamination [63, 64, 65]. Suction methods could enable robotic platforms to retrieve particulate sample types, however the limited efficacy of these methods [65] and the potential for the spread of contamination limit the applicability of these methods in the nuclear industry.

Smair sampling [66] has been used to assess loose contamination. Smair sampling uses suction to draw particles towards an air sampling filter. Smair sampling has been shown to provide a relatively constant removal factor, unlike other methods. The main disadvantage to using smair sampling is the material requirements. Each sample requires its own sampling filter and avoiding cross contamination across samples can only be achieved by careful decontamination of the whole suction system. This disadvantage would mean that sampling would become incredibly expensive and time-consuming.

#### 2.2.2 Solid Sample Retrieval Mechanisms for Robots

There have been numerous examples of robotic systems that were capable of sampling of various physical forms of materials, and design features from these platforms can inform the design of future nuclear inspection robots. In the nuclear industry, robotic manipulators have been used to take swabs as part of regular contamination monitoring routines [56]. These robots operate by executing pre-defined paths to swab objects with relatively uniform dimensions. Despite their relatively common use in the nuclear industry, no reported work has determined the efficacy of these robots performing their sampling tasks.

Outside of the nuclear industry, One example of a robotic system capable of sampling was the Rocky 7 Mars rover prototype [57] [58] which was capable of extracting regolith samples with an end-effector which consisted of two driveable scoops. These scoops formed a "jaw gripper"[58] which meant that the rover was also capable of holding rock samples and cylindrical equipment. A representation of the Rocky 7 can be found in Figure 2.5.

The Rocky 7 design demonstrates the utility of a rover-type design that used an 'arm' to interact with its environment and extract samples. These features would be useful for the platform used in this project.

Other Mars exploration probes have also been able to take samples, such as the Beagle 2 [22], which could combine sample retrieval with preparation and *in situ* analysis of samples. The complete characterisation process offered by Beagle 2 would be desirable in the nuclear industry. The sampling capability of Beagle 2 and Rocky 7

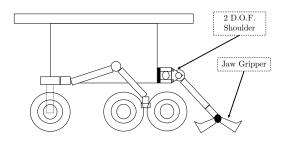


Figure 2.5: A diagram of the side view of Rocky 7.

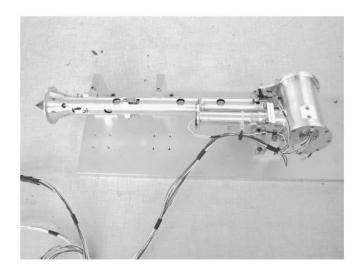


Figure 2.6: The PLUTO soil sampler developed for Beagle 2 [22].

was limited to regolith sampling and the sampling of rocks.

Currently the Curiosity rover is in operation on the surface of Mars [24]. This rover provides a much more sampling capability than any of its predecessors and marks a major leap in sample retrieval capability for robotic platforms. Curiosity uses a 5-degree of freedom arm which is capable of using three tools and two instruments [24]. A drill can be used to gather material from inside rocks, a sample scoop is combined with a processing system to allow for samples to be provided in measured sizes to the analytical equipment on the rover [24]. Further to this, a rotary brush can be used by Curiosity to allow for the removal of dust from rock surfaces [24]. The advanced retrieval capability of Curiosity enables a much more advanced suite of analytical techniques to be used. Curiosity can analyse dust, rock and surface samples with X-ray fluorescence spectrometry, LIBS, microscopy, radiography and neutron detection (used for the detection of near-surface hydrogen) [24].

The Perseverance rover represents the current state of the art in the characterisation and collection of samples in space exploration. Perseverance uses its Sample Caching System (SCS) to store drilled regolith samples which will be available for *ex situ* analysis [42]. Efforts were taken in the design of this system to avoid cross-contamination by using a titanium nitride coating on drill bits to limit the adsorption of organic contaminants [42].

CBRN robots [54], [55] are designed to assist in the assessment and clean-up of hazardous material in a disaster scenario. The RESCUER [55], again a modular design, was designed with a robotic arm with a gripper that was capable of holding sampling tools, and retrieving soil samples. Further to this, RESCUER could hold wire cutters or a drill. Its arm was teleoperated and a lack of feedback from this arm would mean that autonomous operation of this robot would not be possible.

RICA [25] was a robotic platform developed for use in the nuclear industry in France. The modular design of this platform enabled it to perform various characterisation tasks. The RICA could be configured to carry a payload of radiological sensors, including a gamma camera, gamma spectrometer, RGB camera and laser telemetry. This enabled the team operating RICA to collect extensive radiological data and create 3D radiometric heat maps.

Alternatively the RICA platform could be fitted with the "Romain 50 arm [25]". This arm enabled RICA to perform sampling or clean-up tasks with a jaw gripper. This platform is able to retrieve large solid samples for destructive assay later in a laboratory.

#### 2.2.3 Discussion

Dry swabbing is the predominant sample retrieval method for characterisation in the nuclear industry. The ubiquity of this method is due to its cost-effectiveness, its ease of application, and its compatibility with the various constraints imposed when operating in radioactive environments. Depending on surface conditions and the expected analysis techniques, other sampling methods may be used, but their application is limited.

Sampling methods and protocols are designed to be minimally destructive, in order to preserve the sampling environment and consequently increase the accuracy of characterisation information. Further to this, the nuclear industry requires volume and

### 2.2. SAMPLE RETRIEVAL ROBOTICS



Figure 2.7: A picture of RESCUER [55].

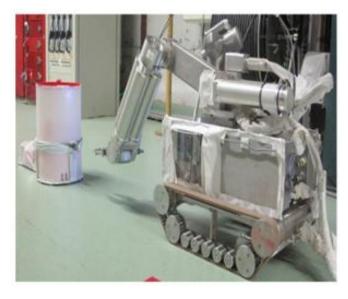


Figure 2.8: The RICA platform with the Romain 50 arm [25].

activity minimisation of samples to reduce the associated radiometric dose. These principles however, are at odds with the developed robotic platforms that are limited to retrieving large solid items. It is clear that current robotic platforms in the nuclear industry do not provide suitable capabilities for effective sample retrieval. An overview of robotic sample retrieval mechanisms is given in Table 2.2.

Sample retrieval mechanisms developed for robots in the nuclear industry thus far are not appropriate to allow *in situ* analysis of samples. The Curiosity Rover [24] and Beagle 2 [22] are platforms which provide *in situ* analysis, and the sample retrieval mechanisms designed for these platforms have been tailored to the techniques used on-board. If robots for the nuclear industry are to integrate sample collection and sample analysis, then the retrieval mechanisms used require development.

If sample retrieval is to be used in the characterisation of environments, it is essential to understand the errors that arise from sampling. The accuracy of sampling is of crucial importance in determining the ultimate accuracy of analyses. Robotic platforms provide far greater repeatability than has been possible when human operators were responsible for sampling, and this provides a unique opportunity to increase the accuracy of sample-based analysis. The design of current robotic sampling platforms rarely considers sampling accuracy, with the Perseverance rover [42] providing the only recorded example of this. Future development should begin to quantify sampling errors.

# **Chapter 3**

# **Sampling and Swabbing**

This chapter details available literature on sampling, with a particular focus on the technique of swabbing, and the implementation of solid sampling techniques on robotic platforms.

This chapter is included to provide context for the experimental work described in this thesis. The first section of this chapter reviews the theoretical understanding of the adhesion and removal of particles from surfaces. This is followed by a review of sampling, sampling methods and error considerations in sampling. This is proceeded by a review which collates studies that have looked at the method of swabbing. This chapter highlights the gaps in our current understanding, with special attention paid to this in Section 3.4.4

## **3.1 Definitions**

This section provides definitions for a number of technical terms which are included in this chapter.

- 1. Sample A collection of matter taken from an environment.
- 2. **Sampling** "A sequence of selective and non-selective operations ending with the selection of one or several assay portions submitted to the analytical process in their entirety."[67]
- 3. Lot "A well defined quantity of material whose heterogeneity is being studied; whose composition is to be estimated after sampling or whose homogenizing is carried out. As regards sampling, the lot L is the quantity that will be represented either by a single sample or by a set of twin-samples." [67]

- 4. **Particle** "The crucial property of a particle is that it is assumed to remain whole and unaltered in the mechanical, physical and chemical conditions that prevail, during sampling. With particulate solids, the particles are fragments. With liquids the particles are ions and molecules." [67]
- 5. **Swabbing** A sample retrieval process which involves the transfer of material from a surface on to a porous substrate (commonly filter paper).
- 6. Pick-Up Factor The percentage of activity [68] removed from a surface on to a swab. Alternatively called "collection efficiency" [69] or "removal factor" [61]. Pick-Up factor is preferred in this thesis to avoid confusion with measurements taken in the fields of decontamination (where removal factor is used widely) and other fields where collection efficiency is used, though the three terms can be used interchangeably.
- 7. **Swab Efficiency** Efficiency is the ratio of the rate of contaminant pick-up to the rate of contaminant redeposition from the swab.
- 8. **Swabbing Input** A collective term to describe the swabbing pressure profile, swabbing method and swab characteristics.
- 9. **Swabbing Efficacy** The ability for a swab to collect a contaminant. An efficacious swab is one in which a large volume of a particular analyte is collected.
- 10. Analyte A particle type of analytical interest [70].
- 11. **Porous** A porous material is one containing pores (voids) [71]. While every material could be said to be porous by this definition, porosity can be defined by considering the pore size in relation to the particle size which is interacting with the material.
- 12. **Collection Substrate** A general term for the porous substrate used for collection during swabbing tasks.

## **3.2** Theories for the Adhesion and Removal of Particles

Understanding and quantifying the mechanisms that determine adherence and the removal of particles from a surface has been important in a number of areas over decades [72]. Theories for adhesion have been of importance in the painting of cars, in the packaging of food and most widely in the fabrication of semiconductors for the electronics industry [72]. Mechanistic understanding of adherence and removal is of crucial importance in the analysis of the swabbing process.

#### 3.2.1 Adhesion

Much of our understanding of adhesion comes from the electronics industry, with most available models focused on replicating and predicting the behaviour of contaminants on silicon wafers [72]. Models for adhesion are generally considered valid only for particles with diameters  $< 40 \ \mu m$  as this size corresponds to the point at which gravitational forces begin to dominate over the electrostatic forces which are far more influential at smaller particle sizes [72]. Existing models have been developed subject to a number of generalisations made based on specific applications [73]. Surface and particle geometries are commonly assumed to be flat planes and spherical respectively, though a number of models are able to account for greater contact areas and different geometries [72].

The primary mechanism for adhesion arises from Van der Waals forces, with these forces being well quantified for macroscopic bodies [74]. Particles in aqueous media may interact with surfaces via "double layer forces" [75], with these forces and Van der Waals interactions being combined in to DLVO theory, which gives a complete picture of particle behaviour [75]. Figure 3.1 depicts double-layer interactions, explaining how metal particles can adhere to a metallic surface.

In aqueous media, adhesion is often affected by Lewis-acid/base interactions [72]. These polar interactions largely vary in nature from Van der Waals interactions, and often be comparatively much larger.

Double layer interactions are also observed with particles in air or vacuum [72], with charge transfer occurring wherever two interacting materials have different work functions.

Humidity can lead to capillary condensation, a mechanism that can eliminate electrostatic interactions in air. Examples show that the overall contribution of this effect to adhesion is small [72].

Where adhering particles or surfaces are compressible, elastic and plastic deformation can substantially alter van der waals interactions [72]. Deformation can increase (or decrease) the contacting area between surface and particle, which increases (or decreases) the strength of adhesion.

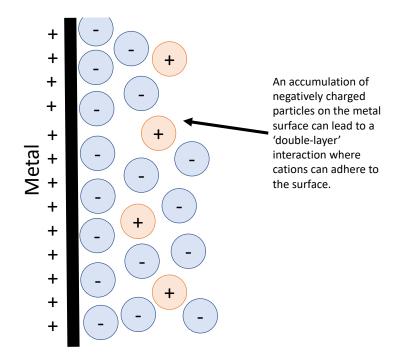


Figure 3.1: A diagram showing double-layer interactions.

For adhesion, theoretical models allow for a quantitative understanding of the interactions of particles and surfaces with a wide range of geometries and conditions [72]. Though these theories are not yet able to be applied to arbitrary geometries and dynamic physicochemical properties, they have been found useful over decades and in a broad range of areas [75].

#### 3.2.2 Removal

The removal of particles is a far more complex issue when compared to adhesion [69]. Removal can only be understood as a dynamic process, and a number of factors make quantification intensely challenging.

Where a particle is adhered to a surface, removal can only be achieved in two ways:

- 1. Reducing the size of the adhering force.
- 2. Applying an external force which is greater than the force of adhesion.

A common approach for removal is via immersion in a liquid [72]. This approach aims to reduce the size of interactions so that adhered particles can be freed. Water and other polar<sup>1</sup> liquids are generally poor choices, as their ability to reduce the size of Van der Waals interactions is countered by increasing the size of lewis acid-base interactions [72].

Common cleaning agents (i.e. laundry detergent) function primarily by reducing the strength of interactions. The high pH of these agents can increase the chemical potential or pH of adherents, consequently reducing the strength of adhesion. These agents are commonly much more effective for removal than immersion in a polar liquid [72].

For low levels of adhesion, removal can be achieved by Brownian motion [72]. Particles in a fluid are free to move and can collide with a surface. If the thermal energy of these particles is greater than the forces of adhesion, adherents can become dislodged from a surface.

Removal via induced fluid flow has also been studied [72] though system dynamics make this process difficult to model. If laminar flow is induced, there will be no removal of adherents as boundary conditions dictate  $v_x = 0$  at the surface, therefore agitation creating turbulent flow must be induced in order to remove particles. Turbulence is still unresolved analytically and so modelling in this case is impossible.

Removal via agitation from a brush or a swab has also been studied [69] and some attempts have been made to model this process [76]. These models have been found to disagree substantially with experimental results [69] due to oversimplifications in these models. Charge transfer from the surface to the agitator produce secondary electrostatic effects that are as of yet, unaccounted for in models.

Removal is a dynamic process, and current theories for removal do not capture the full complexity of the mechanisms which drive this process [69, 72]. Models are not yet able to be used to make useful quantitative predictions about the removal of particles from surfaces.

Thus far, no research has considered mechanisms for the removal of particles during the swabbing process. Understanding the interactions between particle, surface, and swab in this process could inform swabbing protocols and allow for more accurate characterisation information from swabs.

<sup>&</sup>lt;sup>1</sup>For clarity: a polar liquid is one which has a non-zero dipole moment, meaning that there is an imbalance of charges in the liquid. The charge imbalance in these liquids can promote electrostatic interactions with particles that are adhered to a surface.

## 3.3 Sampling

Sample retrieval is an important part of the monitoring and characterisation of radioactive environments [8]. Samples are taken from places that cannot be analysed *in situ* or to enable a wider range of analytical techniques to be used *ex situ*. Sampling is used to provide information that is representative [8] of the environment from which a sample is taken. Though this work focuses on nuclear decommissioning, a much wider variety of fields rely on sampling. An inexhaustive list of non-nuclear applications includes:

- Contaminant testing in water [77]
- Regulatory testing for chemical and biological agents in weapons facilities [78]
- Environmental sampling [50]
- Quality control for industrial processes [79]
- Health and safety Testing [80]

In order to meet the demands of sampling in these areas, many different sampling methods have been developed and are commonly deployed. This introduction will highlight the considerations that inform the choice of sampling methods, as well as describing the sampling methods that are commonly utilised.

#### 3.3.1 Principles of Sampling

The aim of sampling is to determine the properties (physical, chemical, radiological etc.) of an environment with as much accuracy as possible [59]. There are many factors requiring consideration in achieving this aim:

- Does the sampling method extract all potential analytes of interest? Certain analytes may be incompatible with some sampling methods. Alternatively in mixed systems (where multiple analytes are considered), the chosen sampling methods may be biased towards preferential selection of a certain analyte. This can provide a picture of the environment being sampled which is unrepresentative.
- How destructive is the sampling method? The act of sampling may significantly alter the environment being sampled, and this may undermine the purpose of sampling. Further to this, it is generally important to cause minimal damage to the sampling environment to reduce operational costs and ensure safety.

• What is the repeatability, accuracy and efficacy of the chosen sampling method? Without knowing the accuracy of the sampling method, no conclusions about the sampling area can be made. If a chosen method has a low repeatability, many samples will need to be taken to overcome this. Knowing the pick-up factor (removal) of the sampling method is crucial if quantitative analysis is required. Gaining an understanding of pick-up factor can be difficult for many sampling methods.

Choosing the most appropriate sampling method is crucial in obtaining useful information from sampling, The appropriate sampling method is chosen based on:

- Analyte Requirements: What analytes do you expect to find in the environment? Which sampling method is most suitable for the extraction of these analytes?
- Surface requirements: Does your sampling method work in the given sampling area? Do you know how well it works?
- Cost: Is there a cheaper alternative? Could a more expensive alternative provide information that could enable future cost savings?
- Safety Considerations: Does the sampling method place anyone in danger? If sampling for *ex situ* analysis, are you minimising the volume of potentially harmful material you are collecting?
- Analysis requirements: Can the sampling method provide samples that can be analysed? Further to this, are there considerations that can be made during retrieval which would simplify subsequent sample processing requirements.
- Geometrical requirements: Does the geometry of the environment make any method infeasible or particularly useful?
- Waste production: Does the sampling method generate secondary waste? Is the generation of secondary waste allowed in the operational environment?

The remainder of this section will describe common solid sampling methods, developed to address the requirements above for different applications.

## 3.3.2 Solid Sampling Methods

Solid sampling is widely required and focusing on this area is undoubtedly of use to the scientific community. Though solid, liquid and gaseous sampling are similarly important, the choice was made to narrow the focus of this project and this section to solid sampling.

Summary Table 3.3.2 offers an overview of solid sampling methods which are used commonly. Although used more widely, all of the presented solid sampling methods are used in different areas of the nuclear industry.

#### 3.3. SAMPLING

Dry and wet swabbing are commonly used in areas with high activity and areas with limited human access [60]. Swabbing offers a simplistic sampling method, with low material and energy costs. It has long been either the only or the most suitable sampling method in areas of high activity [66]. Though swabbing is widely used, swab samples often lack accuracy [83].

Grippers have been used on robots and manipulators to extract large solids from environments [15, 25]. This method is less widely applicable as collecting large samples generates larger volumes of waste and poses a greater radiological risk.

Vacuum cleaners and other suction devices have been used in the clearing of debris [15], but are generally avoided in characterisation tasks. Suction methods have been shown to have poor collection efficiencies [65] and pose cross-contamination issues in the environments where they operate and the extraction equipment. A number of sampling devices used on robotic platforms can be seen in the figures located in Section 2.2.

Soil samples are often obtained in the monitoring of land around nuclear sites; forming part of environmental impact assessments [84]. While soil samplers are generally found to provide samples which are accurate (given sample planning accounts for heterogeneity), their applicability to different sample types is limited.

Metal samples have been cut from fuel ponds [85], reactors [86] and from other structural components [86] in order to gain a detailed picture of contamination of these components. This method requires significant amounts of energy, can pose a high radiological risk and cannot be used on components that are in operation.

Ablation methods have gained popularity recently, and offer minimally destructive analysis of metal work and concrete [87]. Many view ablation methods as a viable method for the analysis of many sample types in the future [87]. However, ablation methods require the deployment of complex and relatively energy-intensive equipment which increases the cost of sampling. Further to this, matrix effects limit the accuracy of these methods currently [87].

The ubiquity of swabbing methods, combined with the clear gap in our understanding of the associated sampling errors demonstrate a need to further knowledge in this area.

Sampling Method	Description	Sample Types
Dry Swabbing [81]	A porous swab is rubbed along a surface and material is incorporated in to the swab.	Loosely bound solids including corrosion products, fine powders, organic matter, gels, greases, oils and paints.
Wet Swabbing [62]	A porous swab with a wetting agent (solvent or decontamination agent) is rubbed along a surface and contamination is incorporated in to the swab.	Fixed contamination including corrosion products, dried crusts and paints. Mobile contamination unsuitable for dry swabbing.
Adhesive Tape [8]	A substrate with an adhesive agent is placed on a sampling surface which removes material from the surface.	Fixed and loose surface contamination.
Grippers [25]	A gripper is used to extract large solid items.	Large solid items.
Suction [65]	A flow of air is used to remove material from a surface.	Loose surface contamination.
Soil Samplers [52]	Samples are collected in a pot which can be sealed.	Soil and rocks.
Cutting [25]	A sample area is cut from an environment.	Metal coupons, environmental samples, wood.
Laser Ablation [82]	A pulse of light is fired at a sampling area to vaporise some of the target material.	Concrete, metal work.
Other Ablation Methods [82]	An energy source (thermal, electrical, magnetic) is used to vaporise a target material which is then collected.	Concrete, metal work.

Table 3.1: An overview of solid sampling methods

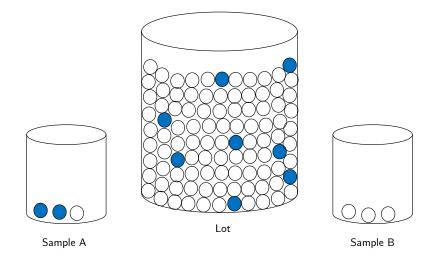


Figure 3.2: A depiction of fundamental error in sampling due to heterogeneity.

#### 3.3.3 Error Considerations during Sampling

Although sampling offers advantages it also introduces sources for additional uncertainty in measurements. Some of the contributing factors to this uncertainty can be reduced by implementing appropriate procedures and others are fundamental in the sampling process [88, 67]. The sources of sampling errors are explored in this review.

#### **Fundamental Error**

In the vast majority of instances, heterogeneity is present in environments on every scale. There is always an error when taking a sample of an environment due to this heterogeneity. This error is referred to as the **fundamental error**. The fundamental error is the minimum error that depends on various factors such as particle size distribution, shape and composition of a material or environment. This error cannot be affected by any sample preparation and cannot be avoided during sample collection. A diagram to explain how heterogeneity can lead to uncertainty in sample retrieval is given in Figure 3.2.

If there is suitable knowledge of the lot that is being sampled, the variance of the fundamental error can be calculated through using Gy's fundamental error equation [89], where  $\sigma_{FSE}^2$  is the variance of the fundamental sampling error,  $M_L$  and  $M_S$  are the

masses of the lot and sample respectively, f is the shape factor, g is the granulometric factor, c is the mineralogical composition factor, l is the liberation factor and  $d_N^3$  is the nominal volume of particles in the sample:

$$\sigma_{FSE}^2 = \left| \frac{1}{M_L} - \frac{1}{M_S} \right| fgcld_N^3 \tag{3.1}$$

Assuming that the mass of the lot from which the sample is taken is much larger than the mass of the sample itself, Gy's equation can be further simplified:

$$\sigma_{FSE}^2 = \left(\frac{1}{M_S}\right) fgcld_N^3, \, M_S \ll M_L \tag{3.2}$$

Gy's equation demonstrates that the variance of the FSE is directly proportional to the volume of particles in the sample and inversely proportional to the mass of the lot being sampled. The other factors effecting the fundamental error in Gy's equation have been found empirically to be independent of  $d_N^3$  and  $M_S$  and constant for a given sample type [90].

#### **Grouping and Segregation Error**

Chemical and physical properties such as gravitational separation, chemical partitioning and electrostatic charge can cause some particles in a lot to cluster. When the sampling process does not correctly account for this possibility, this leads to sampling bias [88]. Grouping and segregation are depicted in Figure 3.3.

These effects can be overcome either by homogenizing the lot before sampling, or taking the more common approach of incremental sampling, where samples are taken from different locations within the lot and then combined before analysis. Both of these approaches are effective in reducing the potential for bias from this short-range heterogeneity.

#### **Increment Delimitation Error**

Increment delimitation error (IDE) occurs wherever a sampling device excludes or discriminates certain portions of a lot [91]. For example, using a sludge sampler that is too large to take samples from the floor of a fuel pond near to any large objects. IDE should be an important consideration when designing or specifying sampling equipment.

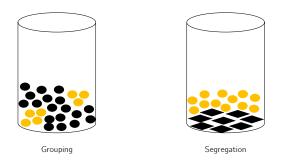


Figure 3.3: A depiction of grouping and segregation.

#### **Increment Extraction Error**

Increment extraction error (IEE) occurs when portions of a sample are lost or when contaminants are added to a sample during retrieval [92]. For example using a coring device that is too small to extract the largest fragments in a lot. IEE can be avoided by the selection of an appropriate sampling device.

#### **Preparation Error**

This error can occur when a sample is mishandled after retrieval and before final analysis. For example, a dry swab, commonly used to retrieve particulate samples in the nuclear industry, can both lose sampling matter from its surface and absorb airborne contaminants during transit if it is not properly processed. Preparation error can be reduced with an appropriate sample processing step and also by choosing more robust sampling equipment.

#### **Sampling Plans**

In characterising an environment, the implementation of an appropriate sampling plan is known to be vital to ensure representativeness [93]. Though determining the location and frequency of sampling is undoubtedly important in the development of *in situ* characterisation capability, it falls beyond the scope of this thesis.

Recent work has demonstrated (using the example of sampling strategies used for

spent fuel storage ponds) that inappropriate sample planning can have a significant impact on the statistical power characterisation [93]. An equally valid issue, however, is ascertaining the accuracy of individual samples taken during characterisation. Indeed, it is important to consider both sample planning and the accuracy of the chosen sampling technique to get a complete picture of the uncertainty of characterisation.

#### Summary

This section has described previous work done to understand error and uncertainty in the sampling process [67]. This work can be used in this project to help quantify uncertainty. The fundamental sampling error equation can be used to quantify part of the uncertainty in pick-up factor and can be compared to the uncertainties arising from other factors.

The goal of sampling method design is to eliminate (or reduce as far as possible) all sources for sampling error other than the fundamental sampling error. Though the work of Gy [67] has limited analytical use for this project, it provides a good understanding of the sources of error contribution and helps inform the sampling methods developed for this project.

## 3.4 Swabbing

When geometry, surface conditions or interference from surrounding areas makes the direct monitoring of surfaces impossible, it is common for samples to be retrieved using a variety of methods for *ex situ* analysis [59]. Those methods include dry swabbing, wet swabbing, large-area swabs, smair sampling, and sniffing techniques [59]. Swabbing (both wet and dry) are common in the characterisation of radioactive environments due to the simplicity of the method and equipment and also the advantage of generating minimal volumes of waste [88].

Swabs are used widely in the nuclear industry due to a lack of *in situ* characterisation capability and the increased sensitivity that traditionally lab-based techniques offer. For example, in determining strontium-90 contamination at the LLW threshold, liquid scintillation counting (LSC) is the preferred method [94]. While this technique provides high sensitivity, total counting times for each sample can be very long (1000 minutes in [94] though the total exposure and processing time can extend much longer depending on the required sensitivity and the chemical nature of the sampling matrix).

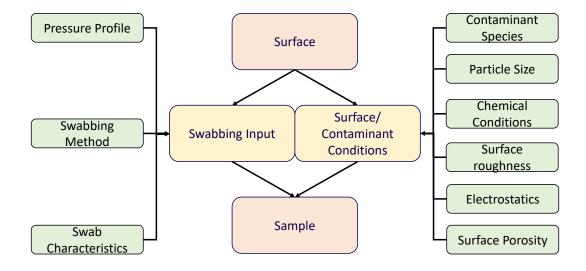


Figure 3.4: A flow diagram to show the factors which affect swabbing efficacy.

Due to these long exposure times, and the expensive sensing equipment used, it is unfeasible to use this method *in situ*.

Dry swabbing requires an operator to rub a surface with dry filter paper, allowing surface contamination to be transferred to the filter paper [59]. The material deposited on the filter paper is then assumed to be representative of the area sampled and can be analysed ex situ to give the chemical fingerprint of the area sampled [61].

Figure 3.4 presents a flow diagram which depicts the qualitative contributors to error in swab sampling. Swab samples are used to make predictions about a given surface, though there are a number of factors which will affect the relationship between swab and sample. Surface / contaminant conditions and the swabbing input will alter the reproduction of the surface which is present on the swab sample. In order to make an accurate understanding of the surface from this sample, the swabbing input and the surface/contaminant conditions must be well known, and their effect must be understood.

Swabbing input is a collective term for the swabbing pressure profile (forces applied to the surface and the area that they are applied over), swabbing method used (i.e. single or double pass, continuous, pulsed or static) and the swab characteristics (substrate material and form, wetting agent properties). The effect of each of these individual factors in the swabbing process is not widely understood and is likely dependent on other factors relating to environmental conditions.

Environmental conditions which may be important in the swabbing process are particle type and size, adhesion mechanisms, specific chemical conditions, surface roughness, porosity, surface/ particle charge and interference from co-contaminants. These many factors illustrate the profound challenge in enhancing knowledge of the swabbing process.

There are uncertainties involved in the dry swabbing process. Key questions about these uncertainties are outlined below [61]:

- 1. What is the true area swabbed? The method assumes that the operator collected material from a certain area and as this area is commonly estimated visually by the operator. This introduces a level of uncertainty in the area being swabbed. It is similarly important to consider the area of a swab which is available to collect matter. Reports have observed that matter transferred to the swab tends to be concentrated in a  $5 cm^2$  area around the fingertips of the operator[59]. This suggests that force application area can vary in practice, and this must be considered when determining the true area swabbed.
- 2. What is the pick-up factor of contamination present on the surface? Pick-up factor will vary between swabs due to both heterogeneity in surface properties and also variability in swabbing inputs. Though understanding pick-up factor variability is crucial in swab-based analysis, our current understanding of this area is severely limited.
- 3. What is the efficiency of the swab? The ratio of particles picked-up to particles deposited by the swab is often unknown and likely to be highly variable depending on surface conditions. Swab efficiency is also likely to be largely affected by the swabbing inputs, and understanding the impact of these inputs is crucial to determine swabbing errors.

Though the focus in this work in on sample retrieval, it is also important to consider that swabs will be analysed using techniques that add further uncertainties to the estimates they provide. It is important to understand that the radiation spectrum presented to a detector may be different to the 'true' spectrum expected from the analytes present on a swab. This is particularly pertinent when considering  $\alpha$ -emitting analytes collected on a swab. The collection matrix (and any wetting agents used) act to attenuate count-rate, and if this is not accounted for then under-estimates of activity can be made.

All of these questions are challenging to answer, but they are fundamental to accurate characterisation. Swab-based characterisation continues to be used to make decommissioning and waste consignment decisions in the nuclear industry [61], though anecdotally the use makes gross over-simplifications which are problematic in a number of ways. If operators treat swab-based estimates with the conservatism that our current understanding necessitates, activities will be over-estimated leading to reduced access to environments and over-consignment of waste (treating a larger volume of waste in higher hazard levels). If operators do not act with appropriate conservatism, then there is a potential that operators and the general public will be exposed to radioactivity levels which are in excess of environmental regulations.

#### 3.4.1 Pick-Up Factor for Dry Swabbing of Surface Contamination

This section presents literature that has focused on calculating the removal factor of contamination present on a surface from swabbing, to determine the current understanding of swabbing errors. There are few studies available in this area, and as will be seen, current literature falls far short of adequately addressing swabbing errors. This section will, however, provide information that can inform the reader as to the use of swabbing, and the expected variability in pick-up factor expected for a variety of surface/ contaminant conditions.

Pick-up factor is used as the predominant performance metric used in previous swabbing studies [66, 88]. Equation 3.3 gives a formal definition, where "P" is the pick-up factor and "A" is the activity, a term used to describe the response of the chosen analysis technique to the presence of the analyte. Most commonly, the activity will correspond to the radioactive count-rate.

$$P = \frac{A_{swab}}{A_{surface}} \tag{3.3}$$

When monitoring an unknown environment, it is unlikely that the pick-up factor of contaminants will be known. For this reason, a common approach is to assume a pick-up factor. Anecdotally, most sites will assign a site-fingerprint (an assumed pickup factor) which will be used when an expected pick-up factor cannot be estimated or

First Author (Year)	Contaminant	Surface Material	Pick-Up Factor (%)
Prince (1968) [95]	Tritiated nitrogen-acetate	Fibre-glass and shellstone	5-30 (dry swabbing)
Royster (1965) [66]	Thorium Oxide	Various materials	23.5-86
Mitchell (1964) [96]	Beryllium dust	Wood	3
Lichtenwalner (1992) [60]	Dust particulates	Unspecified	45
Chavalitnitikul (1984) [97]	Lead oxide dust	Formica and plywood	85 and 30
Saxby (1964) [98]	Loose actinyl dust	Unspecified	50
Verkouteren (2008) [69]	Polystyrene Latex beads	Various	1-98

Table 3.2: A summary of studies investigating pick-up factors for loose contamination

small-scale experiments can be conducted.

Through decades, common practice assumed a 10% pick-up factor regardless of the surface conditions, expected contaminants, and swab type used [61]. The 10% pick-up factor estimate reportedly dates back to the 1950s at Windscale [61] where the number was selected on the basis that 1% was too pessimistic and 100% was definitely too optimistic. Based on this reasoning, 10% was chosen "to be cautious" [61]. There have however been attempts to quantify pick-up factor in practice and these studies are summarised in Table 3.2. These studies highlighted that an assumed pick-up factor should, where possible, be supported by experiments using similar surface/contaminant conditions.

One report analysed the pick-up factor from fibre-glass and shellstone contaminated with tritiated nitrogen-acetate [95]. This work compared the performance of dry and wet swabbing. Pick-up factors ranged from 5 - 30% for dry swabbing and 5 - 32% for wet swabbing. This work showed a wide range for pick-up factor and no clear difference in performance between dry and wet swabbing. It is notable that a 10% pick-up factor assumption here could not be used as a cautious estimate as a number of swabs reported pick-up of less than this number.

Another report looked at thorium oxide particles deposited on to a wide range of different substrates [66]. The contamination in this work was deposited from a gentle air-stream. Results showed a range of pick-up factors from 23.5% for fibreboard to 86% for painted aluminium. Pick-up factors from environments in the nuclear industry are generally much lower than these reported values, demonstrating that contamination in practice is generally much more stubborn. These results also demonstrate that even loose contamination cannot be retrieved completely through dry swabbing. This is

#### 3.4. SWABBING

likely due to an operator's inability to maintain pressure throughout the swabbing motion and the likelihood of re-deposition of material from the swab to the surface.

Further work examined the pick-up factors of beryllium dust contamination on wood [96]. This work found a pick-up factor of just 3% after swabbing and 21% removal after washing and wiping. This work demonstrated the difficulty in accurately dry-swabbing a rough surface such as wood.

One study looked at dust pick-up in 18 locations [60]. This work used consecutive wiping to estimate the total amount of removable contamination. The average pick-up factor of removable contamination from first wipe in this work was 45%, however the total pick-up factor was not calculated in this work. A further finding from this study was that adjacent swabs varied massively in measured activity. Just 50% of adjacent ratios of activity were inside the range of 0.6 - 1.5. This finding demonstrates the large uncertainty in deposition of contamination across environments but also suggests that the uncertainty of swabbing pressure and force from human operators is likely a large source of error in this process.

A further study measured removable contamination from the surfaces of fuel flasks using consecutive swabbing [61]. Consecutive wipes were taken from the same area of 4 different flasks and the results were highly variable. The first wipe pick-up factor ranged from 24 - 64% and in some cases, later wipes removed more activity than initial wipes. This work supports other findings that show metal surfaces generally exhibit pick-up factors greater than those of rougher surfaces, showing that the surface type appears to have a large impact on the swabbing process. This work also shows that consecutive samples do not always follow a geometric sequence, and using consecutive swabbing to calculate total removable activity is not necessarily a reliable process. The uncertainty in consecutive swabbing may be caused by the transfer of contamination across the surface between swabs, the high variability in operator force input and also the efficiency of the swab.

Recent work on swab sampling for weapons testing [69] demonstrates the extent of the effect that swab and surface materials can have on pick-up factor. In an attempt to determine an the effect of different surface and swab materials, various surfaces were swabbed and the amount of polystyrene Latex beads removed were counted. The swabbing surface was not kept constant through these experiments, and though one of the two tested removal matrices consistently performed better than the other, there was a great range in the observed pick-up rates for both materials (1 – 98 % for muslin and 1 - 35 % for PTFE).

The studies detailed here show that the determination of pick-up factors is far from well understood. The few studies presented here have relied on the collection of empirical data which relates to specific forms of contamination on specific surfaces. As of yet there have been no attempts to develop understanding of this process which attempts to develop an understanding of swabbing that is useful for more than a specific material or contaminant.

These studies have demonstrated that pick-up factors are highly variable dependent on the surface chemistry. It seems that metal and smooth surfaces generally lead to much greater pick-up factors than rough surfaces. The poor reported pick-up from rough surfaces (just 3% for wood [96]) can inform sampling plans. Rough surfaces require much more measurement to get an understanding of the levels of contamination present.

Various studies used consecutive swabbing to estimate the total levels of contamination present on a surface [60], [61]. Although this method seems to be wellestablished, the results from these studies would not support the use of consecutive swabbing to determine total removable contamination levels [61].

Although some studies have posited that the force input from human operators in the swabbing process may be one of the largest sources of uncertainty in measured pick-up factor [60, 61], no studies have currently attempted to determine and quantify the effect of human input. Indeed the understanding of how different swabbing inputs (for example force application) affect swab efficacy is entirely absent from existing literature.

#### **3.4.2** Paper as a Swabbing Substrate

Swabbing requires the use of a porous substrate to collect material from a swabbing area. While there are many materials which may be appropriate to collect material from a surface, paper (commonly filter paper) is used predominantly in swabbing studies [81]. This section explores the role of paper as a swabbing substrate.

Paper is composed of layers of randomly interwoven cellulose channels. Filter paper is commonly manufactured from the raw material cotton due to its high cellulose content (> 95%), allowing for a high purity. A description of paper's chemical and physical composition is given in a previous review [99] and this may inform our understanding of the mechanisms which control pick-up.

The chemical properties of paper match those of cellulose. Other chemical processes such as the inclusion of brighteners are not employed when manufacturing filter paper, and this simplicity of chemical structure and form is in part a reason for its wide adoption in swabbing studies. Additional additives to the paper structure are thought to be detrimental to swab efficacy and repeatability. Further to this, most manufacturers of filter paper will provide information about the paper's thickness, pore size, weight, wetting rate and wet strength, which are important when choosing a substrate for swabbing experiments. During swabbing there are three likely mechanisms for contaminants to be picked up by the paper:

- 1. Where a liquid is used, the contaminant can be taken in to the liquid phase and this solution can reside in the cellulose paper channels.
- 2. The friction applied during swabbing can remove a contaminant from a surface and it can settle within a cellulose channel.
- 3. The contaminant can embed on the surface of the paper, deforming the paper structure.

#### **3.4.3** Uses of Swabbing in the Nuclear Industry

In order to provide some context for the use of swabbing in the nuclear industry, the following case studies are presented. These examples will detail the requirements for swab-based characterisation as well as highlighting some hypothetical issues with current swabbing practices. Although swabbing is used more widely than the cases highlighted in this section, these examples are chosen to highlight specific issues in the area.

#### Swabbing during the characterisation of Legacy facilities

In the United Kingdom, there are many 'legacy' facilities in which there are ares which require better characterisation before they can be safely decommissioned. Examples of these facilities can be found at Sellafield and at Dounreay [100]. Swabs are commonly used to retrieve samples for analysis in a lab as *in situ* characterisation techniques are currently limited. As there is little prior knowledge of the conditions in these environments prior to swabbing, it is intensely challenging to determine an estimated pick-up factor for this swab, and thus the utility of swabbing is limited. Due to the uncertainty associated with swabs taken from these facilities, proceeding operations are often made to be extremely cautious. Work which can be done to provide some

assurances of swabbing performance would facilitate the removal of layers of caution where appropriate, and lead to quicker and more cost-effective decommissioning.

#### Swabbing in Glove-boxes

Glove-boxes are commonplace in research and power-generating nuclear facilities [101], with their primary use being to add an additional layer of separation between a radioactive material and a laboratory worker. As glove-boxes commonly house potentially hazardous material, there is a requirement to ensure that contamination is found and managed appropriately within the glove-box. To do this, swabs are used as a control measure. Workers are required to swab their operational area and to routinely swab the remainder of the glove box to detect any contamination.

A problem with swabbing in glove boxes is that their conditions are not conducive to efficacious swabbing. Glove boxes may be air-fed with an inert gas which can introduce static build-ups and would be combined with a dry atmosphere. A dry atmosphere will limit the ability for contamination to be collected on a swab, and static may also have a similar effect. These factors raise serious questions about the efficacy of swabbing in glove boxes.

#### **Swabbing for Beryllium in Fusion Reactors**

In nuclear fusion reactors, beryllium is used widely due to its thermal properties. During fusion operators, beryllium-containing dust is liberated from reactor walls, with this dust being highly toxic [102]. To ensure the safety of operators in the fusion industry, it is important to identify any sources of beryllium contamination. To do this, swabs are routinely taken and analysed *ex situ*.

An issue with swabbing for beryllium contamination is that the surfaces from which these swabs are taken also contain beryllium. It is evident that the act of swabbing may liberate otherwise bound beryllium and create contamination where non existed.

#### The Costs of Swabbing

Swabbing is a technique which is inherently a cheap, resource-efficient method of sampling. Due to its low-cost and the minimal generation of secondary waste, it is a preferred method for sample retrieval in the nuclear industry [59]. The collection of swabs becomes considerably more expensive when these swabs are taken to a lab

for *ex situ* analysis. Swab samples which may be radioactively contaminated require special transport arrangements and control measures to be instituted in the analysis lab, with all of these control measures increasing cost. If more analysis of swabs was to be performed *in situ*, the costs of characterisation would be reduced significantly.

#### 3.4.4 Discussion

Sample retrieval forms an integral part of current characterisation practice. The ability to interact with a sample and take intrusive measurements enables a more comprehensive suite of analytical techniques to be used in characterisation. In addition to this, sample retrieval allows areas with difficult geometry and high levels of interference to be monitored where conventional *in situ* counting methods would not allow this.

In order for analysis by sampling to be accurate, any sources of error have to be well known, quantified where possible and controlled effectively. The literature reviewed in this section, summarised in Table 3.2, has shown that our understanding of the swabbing process is limited, and this limits the accuracy of the entire characterisation process. There is a clear need for work which addresses our limited understanding of errors in the swabbing process. In current practice it is common to make estimates based on assumed pick-up factors which are, in some cases [61], baseless. The scientific community needs to do more to address this area, especially considering the prominence of swabbing in the characterisation of radioactive facilities.

Replacing human operators with robot agents in the sample retrieval process offers the opportunity to reduce potential sources for error, as they are capable of achieving far greater repeatability of swabbing inputs. In order to maximise the potential of robotic platforms in the swabbing process, work needs to begin to quantify the effect of different error contributors, in order to determine which factors are most important to control. With a better understanding of the sources for error in swabbing, it may be possible to predict swabbing efficacy with greater accuracy, and to develop swabbing protocols which reduce the large variability between different swabs.

# Chapter 4

# Swabbing Inputs and their effect on Pick-Up Factor

This chapter presents the results of experiments, using an Automated Swabbing System (A.S.S., introduced in Section 4.2.1), aiming to determine the effects of different swabbing inputs (this term is defined in Section 4.1) on the swabbing process. The pick-up factor for these swabbing experiments is determined, and this is complemented with the imaging techniques introduced in Section 4.2.2 to provide an insight in to mechanisms that influence the swabbing process.

The need for accurate characterisation is crucial for safe and cost-effective operations in the maintenance and decommissioning of radioactive facilities [8]. Swabbing is widely used in the nuclear industry to assess contamination in highly active areas where access is limited and *in situ* sensing is challenging [59].

Section 3.4 made apparent the need for a greater understanding of swabbing, particularly understanding factors which influence pick-up factor. The large variability between different swabs taken from the same surface has widely been attributed to variable surface conditions and variable swabbing inputs [60, 61], as is detailed in section 3.4. This belief has yet to be tested systematically, so there is a clear need to quantify the error contributions from different sources in the swabbing process.

The experiments presented in this chapter alters swabbing inputs, and determines their effect on swabbing efficacy and repeatability. It is hoped that these experiments will inform future efforts which wish to improve the accuracy of sampling through swabbing.

This chapter presents the methodologies used in this chapter, this is proceeded by a section which details a series of experiments performed in order to ascertain the effect

of swabbing inputs on swab performance. This chapter is concluded with a summary of key findings from this work.

## 4.1 Definitions

This section provides definitions for a number of technical terms which are included in this chapter. While some of these definitions may appear in other chapters, they are included additionally here for the ease of the reader.

- 1. **Swabbing** A sample retrieval process which involves the transfer of material from a surface on to a porous substrate (commonly filter paper).
- Pick-Up Factor The percentage of activity [68] removed from a surface on to a swab. Alternatively called "collection efficiency" [69] or "removal factor" [61]. Pick-Up factor is preferred in this thesis to avoid confusion with measurements taken in the fields of decontamination (where removal factor is used widely) and other fields where collection efficiency is used.
- Swab Efficiency A measure of the ability for a swab to collect a larger sample. Efficiency is the ratio of the rate of contaminant pick-up to the rate of contaminant redeposition from the swab.
- 4. **Swabbing Input** A collective term to describe the swabbing pressure profile, swabbing method and swab characteristics.
- 5. **Swabbing Efficacy** The ability to swab in a desirable way. Swabbing efficacy mostly refers to the ability for a swab to 'pick-up' contamination. The definition of this term can be extended to the swab being representative of the surface from which it is taken.
- 6. Swab Passes The number of times the surface is covered during swabbing.
- 7. Force Application Area The area over which force is applied to a swab during swabbing.

## 4.2 Methodologies

This section details the equipment used and methods developed for use in these experiments. The Automated Swabbing System (A.S.S.) enabled swabbing inputs to be

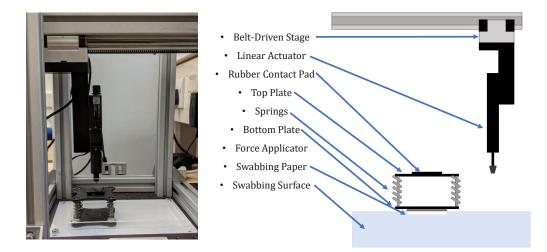


Figure 4.1: A labelled picture of the Automated Swabbing System with accompanying Schematic.

controlled with a high degree of repeatability, and the development and calibration of this system is presented. In addition to this, the imaging techniques which can be used to provide quantitative contaminant dispersal maps on swabs and swabbing surfaces are detailed here.

#### 4.2.1 Building a Force-Controlled Swabbing System

The force-controlled swabbing system developed in this project requires the ability to move in two-dimensions with a high degree of repeatability, while marrying this with a force applicator which can exert a pre-designated force to a swab through swab motion.

The developed system utilises a linear actuator (Zaber X-NA08A50-E09) mounted on a belt-driven linear stage (Zaber LC40B0150-KM). This system is installed in an aluminium scaffold, and is shown in Figure 4.1. The linear actuator makes contact with a spring-loaded swabbing pad, which consists of four springs (RS Components,  $K_s =$  $1.85 Nmm^{-1}$ ) suspended between two parallel plates. The spring-loaded swabbing pad sits on top of a rubber applicator (introduced to eliminate swab slipping) and swabbing paper.

Equation 4.1 can be used to determine the swabbing force, F from the linear actuator's extension, x. As the linear actuator is reported to have a total accuracy of  $\pm$  55  $\mu m$ ,

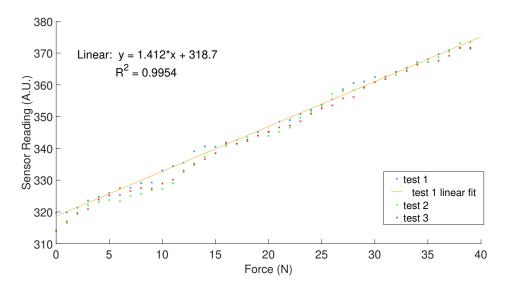


Figure 4.2: Calibration results for the Automated Swabbing System in three linear force increase experiments, with the intended swabbing force on the x-axis and the response of a capacitive force sensor given on the y-axis.

the total accuracy in swabbing force, calculated from Equation 4.1 is  $\pm 0.407 N$ .

$$F = 4K_s x \tag{4.1}$$

The repeatability of the linear stage is reported as  $< 20 \,\mu m$  which corresponds to 0.024 % of the swabbing area. It is notable that the error contribution from this stage is low compared to other sources of error.

To ascertain the performance of the automated swabbing system (A.S.S.), experiments were performed and the swabbing forces were measured by a capacitive force sensor (SingleTact CS8-100N) and a mass balance (Kern EMS-6K1), and the values given compared to the force calculated in Equation 4.1.

Figures 4.2, 4.3 and 4.4 show the results from these experiments. These results show a strongly correlated linear fit, giving confidence that the force production of the system is linear, as intended. This was confirmed in a increasing force step test, a random step test and a decreasing force step test. These results were consistent when analysed using both the capacitive force sensor and mass balance.

The intended swabbing force was often less than the measured swabbing force. This was thought to be due to deformation of the swabbing pad, as some of the applied energy did not go in to compressing the springs. As a result of these findings, subsequent experiments used the calibration shown in Figure 4.4 for the swabbing forces.

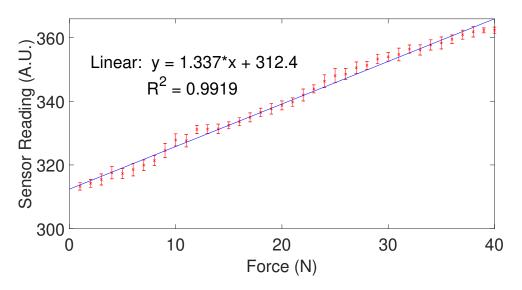


Figure 4.3: Calibration results for the Automated Swabbing System in a linear force decrease experiment.

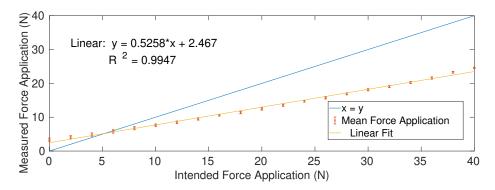


Figure 4.4: Calibration results for the Automated Swabbing System against a mass balance.

#### 4.2.2 Imaging for Swabbing Experiments

Imaging can provide useful insights in to the swabbing process and the mechanisms which influence the removal of contamination from surfaces. Previous studies in to swabbing have rarely used imaging techniques, with only one example [69] which was significantly limited in scope and analytical utility.

This previous work [69] used fluorescent polystyrene latex (PSL) beads and collected images using a fluorescent microscopy system, requiring manual counting of individual particles and a time-consuming process to create a tiled-image of the total  $2.5 \ cm \times 4.5 \ cm$  swabbing area. With modern imaging techniques and camera technology, there is no need to repeat this arduous imaging process.

The imaging techniques developed here are influenced by smart phone colorimetry techniques [103], analysing colour channels to contaminant intensities and spatial distribution. All of the images presented in this thesis were collected with a smartphone camera (Google Pixel 2) using the ProCam X application to control the collection settings. Two optical enclosures (opaque boxes) were built to enclose the swab and swabbing bed, removing ambient lighting effects from the images.

To be consistent with the other swabbing experiments performed in this thesis, a fluorescent particulate (Phosphorescent powder, Green XL) was chosen to provide a strong signal, without the need for illumination during image collection. This contaminant was first used for a student project [104], with the method used being adapted significantly to increase the analytical utility of collected images. Though the powder used for imaging and the sand used in the swabbing experiments are both particulate in nature, the fluorescent powder has a smaller particle size, and so is substantially influenced by adhesion to the bed [72].

These imaging experiments included a number of steps, firstly the swabbing bed was cleaned using 70 % isopropyl alcohol solution and a sheet of paper. This was followed by drying for approximately one minute and grounding the bed to reduce the effects of static. The fluorescent contaminant was then weighed and dispersed on the bed. After this, a swab wetted with deionised water was used to swab the surface. A light source was used to illuminate swab and surface for 20 seconds after swabbing. This ensured that the signal from the contaminant was not significantly diminished while the images were collected. This was followed by moving the swab to its optical enclosure and covering the swabbing bed with the other enclosure. The images were collected after this.

A 1 s exposure time was found to be appropriate for these images, with the focal

lengths fixed at 0.13 *m* and 0.23 *m* for the swab and swab bed images respectively. No filters and an ISO of 400 was used. These settings were found to provide sharp images with an appropriate amount of exposure. Three images were collected for each experiment to account for slight differences in camera position, and nudges to the camera during collection. Images were collected in a raw format (".dng") and exported to a photography workflow application (Darktable) where they were manually cropped and white-balancing was eliminated. Images were subsequently exported in an 8-bit compressed ".tif" format. The images were then processed in MATLAB, with details on the methods used detailed in Sections 4.2.2 and 4.2.2.

#### **Visual Contaminant Intensity Mapping**

Previous attempts to map contamination on surfaces and swabs have been limited in scale and analytical utility [69, 104]. New imaging techniques were developed for this project to allow greater insights in to performance in swabbing experiments.

Images were collected in a raw format (".dng"), with an application which allowed control of all of the image processing techniques. After this, images were cropped to size and exported to a format which was readable in MATLAB.

Images were read in MATLAB as an array with each pixel position being assigned three colour channel values (R,G,B). As the fluorescent pigment used was coloured green, and the background was black, the green channel was the only colour channel with any analytical utility. Images were then stored as an x \* y array where x and y denote the vertical and horizontal number of pixels in each image.

These images were visualised using MATLAB's scaled colour plot ("imagesc"), with a colour gradient representing the green value (0 - 255) for each pixel. Areas with greater contaminant intensity are shown with a strong yellow colour, with blue representing areas of lower contaminant intensity.

Intensity maps were created for all of the swabbing beds before and after swabbing, and the swabs after collection. A collection of swab contaminant intensity maps can be found in Figure C.1. Figure 4.5 gives a flow diagram describing the image processing method described in this section.

#### **Radial Contaminant Intensity Mapping**

Existing work [69, 104] does not apply quantitative analysis of contaminant dispersal on swabs and surfaces, and this is an important limitation. This section details a method

#### 4.2. METHODOLOGIES

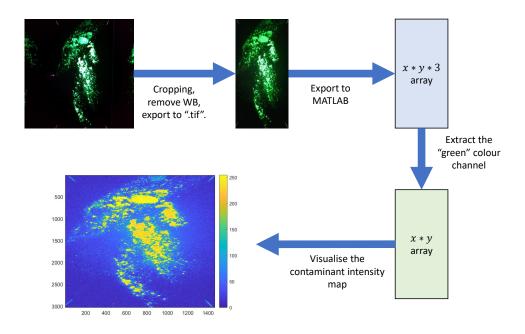


Figure 4.5: A flow diagram to show the process to produce contaminant intensity maps.

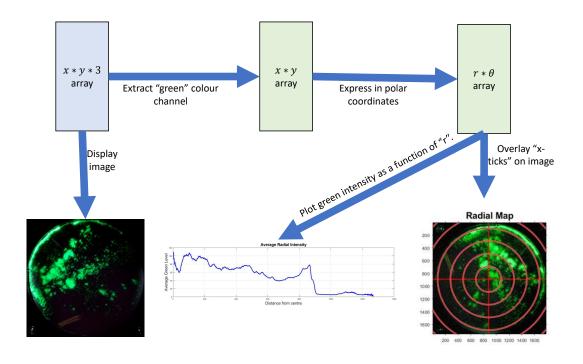


Figure 4.6: A flow diagram to show the process to produce radial intensity maps.

developed to analyse the radial distribution of contamination on a swab, which allows for quantitative analysis of swabbing images.

Figure 4.6 depicts a flow diagram which describes the radial contaminant intensity mapping method which was developed for this thesis.

Images were processed from a raw format (".dng") with the processing steps before analysis in MATLAB identical to the method utilised in Section 4.2.2. An x \* y \* 3 array, where x and y denote the vertical and horizontal number of pixels in the image and the three values corresponding to each pixel match the red, green and blue colour values of the image.

As was the case in Section 4.2.2, the green value for each pixel was extracted as the other two channels were extraneous in this analysis. The circular symmetry of the swabbing paper used, the *x* and *y* values were converted to polar coordinates using Equations 4.2 and 4.3, where *r* corresponds to the distance from the centre of the image and  $\theta$  corresponds to the angular displacement of the pixel from the y-axis. The centre of the image ((*x*<sub>0</sub>, *y*<sub>0</sub>)) was found and radii were calculated from this centre.

$$r = \sqrt{(x - x_0)^2 + (y - y_0)^2}$$
(4.2)

$$\theta = \arctan(\frac{y - y_0}{x - x_0}) \tag{4.3}$$

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After the image was expressed in polar coordinates, the mean green value as a function of radius was calculated and plotted. In order to aid visualisation, an image with the 'x-ticks' (the horizontal grid lines) from this plot were superimposed on the original image.

## 4.3 Swabbing Inputs and their effect on Pick-Up Factor

As has been detailed in Section 3.4, determining the extent to which swabbing inputs affect the swabbing process is of crucial importance. Ascertaining this will allow for more accurate estimates to be made from swabs and will inform the development of swabbing robots (see Section 2.2). Understanding the impact of swabbing force is important in developing force-controlled swabbing protocols for manipulator robots. In addition to this, swabbing force is posited to be a main contributor to swabbing error, and testing this is an important step in furthering our understanding of the swabbing process.

Force application area (FAA) was found through the course of the work in this thesis to play an important role in affecting swab efficacy. For this reason, FAA was explored in depth in these experiments. The effects of contaminant mass are also explored in this chapter to provide insights in to some of the mechanisms which influence the swabbing process. Finally, this chapter presents imaging experiments which were instructive as to the effects of wetting agents on the removal of contamination, and so these results are also presented.

## 4.3.1 Experimental

Swabbing experiments were performed, with a single contaminant dispersed across a smooth swabbing bed. A depiction of this is shown in Figure 4.7. The surface and contaminant conditions were chosen to model particulate contamination on a smooth surface, with consistency (as far as practicable) in these surface conditions across many

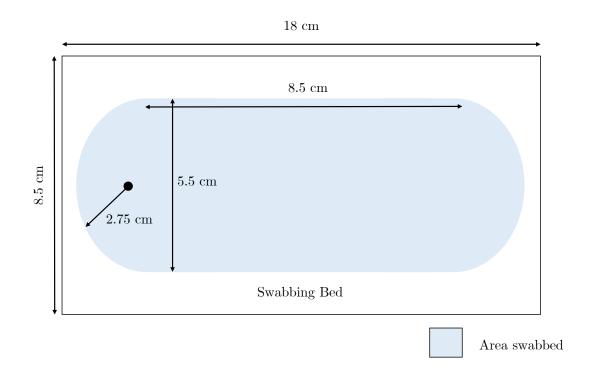


Figure 4.7: A depiction of the swabbing bed and swab path with measurements.

swabs. There was also a requirement for minimal preparation of surfaces and contaminants, with a need to perform many swabbing experiments.

The contaminant used in the swabbing experiments was sand (50-70 mesh particle size, Sigma-Aldrich 274739) and this was dispersed over a Perspex surface (Hobarts, UK). Whatman 41 filter papers (Ashless, 55mm diameter) were used in these experiments, with deionised water used as a wetting agent. Pick-up factors were determined using a mass-transfer method, measured with a mass balance (OHAUS Adventurer AX-224).

Surfaces were prepared prior to contaminant (sand) loading in order to mitigate the influences of a number of effects. Surfaces were cleaned with 98% Isopropyl alcohol to eliminate any oils, residues and dusts from the surface. After this, the surface, swabbing system and experimenter were grounded to remove any static which may affect contaminant pick-up. Changes in lab humidity level were not found to introduce any changes in contaminant grouping or adherence, and so this factor was not controlled actively.

Swabbing forces produced by the Automated Swabbing System (A.S.S.) were calibrated against capacitive force sensors (SingleTact CS8-100N) and were found to produce a well correlated linear relationship in the desired force range. The results from this calibration experiment can be found in Section 4.2.1.

Unless stated otherwise, swabbing experiments were performed using a two-pass method (i.e. the swab was moved down the length of the swabbing bed and then back to its starting position) with continuous force application. Where parameters other than force application were tested, a constant 10 N force was applied (obtained from the graph in Figure 4.4).

A manual operator was required to load the contaminant on the swabbing bed, place the swab on the swabbing bed, remove the swab, and transfer the swab on to the mass balance after experiments. This source of handling error was deemed to be unavoidable, and systematic for all results collected in these experiments.

Grouping errors (in the dispersal of the contaminant on the swabbing bed) could be caused by humidity levels and the build up of static. All equipment and materials were regularly grounded to reduce the build-up of static, and visual inspection found no sign of increased moisture levels in the contaminant or on the swabbing bed. Visual inspection indicated that there was no significant grouping during the swabbing experiments with sand, though there was more significant grouping during the imaging experiments. Figure 4.8 shows the swabbing bed after contaminant dispersal.

The grouping shown in Figure 4.8 is likely caused by larger forces of adherence to the surface and stronger inter-particle forces [72]. Despite this, the imaging techniques used were still able to replicate phenomena observed in the swabbing experiments and tests with different particle sizes further elucidate some mechanisms which are prevalent during swabbing.

Mass measurements were used analogously to activity measurements for swabbing experiments in this thesis. The definition for the pick-up factor equation used in this work is given in Equation 4.4, where P is the pick-up factor,  $m_f$  and  $m_i$  were the mass measurements of the swab after and before swabbing respectively, and  $m_s$  was the contaminant mass deposited on the surface. For the single contaminant systems tested this measurement functioned well, though if this work was to be extended to include mixed particle types, mass measurements could not be used.

$$P = \frac{m_f - m_i}{m_s} \tag{4.4}$$

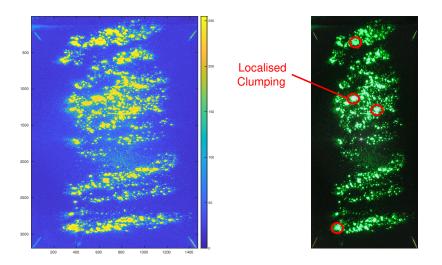


Figure 4.8: Left: A contaminant intensity map for the dispersal of contaminant on a swabbing bed, **right**: the image of the dispersal of fluorescent contaminant across the swabbing bed. The fluorescent contaminant formed local clumps on the swabbing bed, as opposed to the sand used in other swabbing experiments which did not mimic this behaviour.

## 4.3.2 Results

An initial experiment compared a human operator to the A.S.S., shown in Figure 4.9. Though these results could not be confirmed to be typical for all human operators in swabbing tasks, this initial result demonstrated the potential positive impacts for automated systems performing swabbing tasks. While the human operator achieved a standard deviation of  $\sigma = 13.1$  %, the force-controlled swabbing system achieved a much lower value of  $\sigma = 3.7$  %. Despite the sample size chosen comparing favourably to previous swabbing studies [81], there was still a desire to determine whether the sample size is a limiting factor in the statistical power of these experiments.

Swabbing is expected to act as an equilibrium process. Where material is transferred to and redeposited from a swab as the swab is moved. In order to test this, an experiment was performed to test pick-up against changing swabbing distance (presented as number of passes), given in Figure 4.10. For an equilibrium process, we would expect to see stabilisation of pick-up factor as the number of passes increases, however this is not observed in Figure 4.10. It was concluded that larger sample sizes were needed before ascertaining whether swabbing was acting as an equilibrium process.

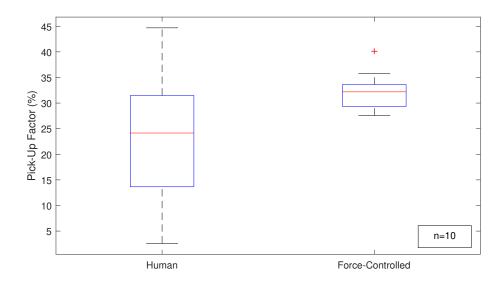


Figure 4.9: A comparison of a human operator and the A.S.S. first presented in [105].

Figure 4.10 was also hoped to help choose the most appropriate swabbing protocol used in the remaining experiments. Due to the inconclusive nature of these results, a two pass method was adopted for the swabbing experiments as it was consistent with previous literature described in Section 3.4.

Figure 4.11 shows the sensitivity of a swabbing experiment to changing sample size, while Table 4.1 presents the evolution of mean pick-up factor and standard deviation through this experiment. While the most significant change in variability and mean pick-up factor was seen between n=10 and n=20, there is still broad agreement between these two sample sizes. Indeed all of the subsequent mean values lie within one standard deviation of the initial mean (n=10).

Sample Size	Mean Pick-Up Factor (%)	Standard Deviation (%)
n=10	21.50	4.56
n=20	24.23	8.76
n=30	24.19	8.26
n=40	24.39	8.77
n=50	23.65	8.58

Table 4.1: Statistical metrics from Figure 4.11

As a result of the findings from Figure 4.11, n=20 was chosen as a sample size to test the effects of swabbing force on pick-up factor, as there was little statistical benefit to collect a sample size much larger than this.

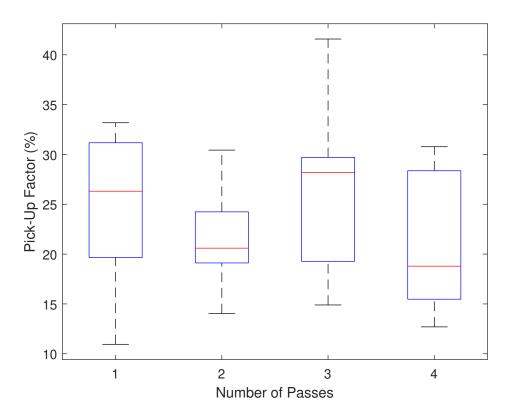


Figure 4.10: A effects of changing the number of passes on swab efficacy (n=10).

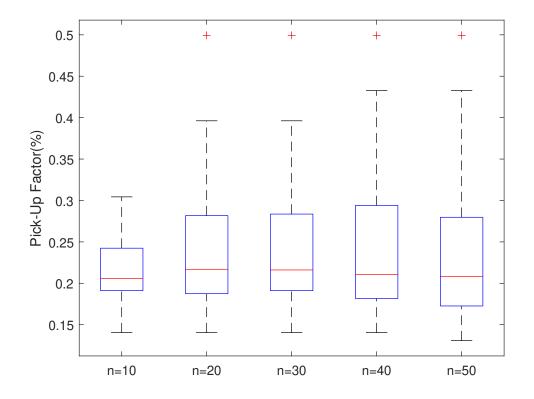


Figure 4.11: A box plot to show swabbing performance in experiments with increasing sample sizes.

## **Swabbing Force Effects**

Swabbing force is thought to be a key factor influencing the removal of particles during swabbing. It is widely posited that variability in swabbing forces adversely impacts swab repeatability [61], and so experiments were conducted to ascertain the effect of swabbing force on pick-up factor. Figure 4.12 demonstrates the effects of changing swabbing force on swabbing performance.

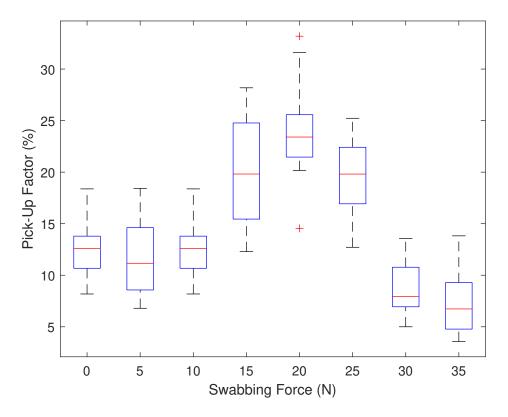


Figure 4.12: A graph showing the effect of swabbing force on pick-up factor (n=20).

Figure 4.12 demonstrates that swabbing force can have an effect on swabbing efficacy and repeatability. While these results may not be consistent for different contaminants, surfaces and swabbing systems, there is a clear region of forces which provide greater swabbing efficacy and repeatability. For this experiment, this region appears to be centred around 20 N, with swab efficacy falling sharply below 15 N and above 25 N.

It is notable in Figure 4.12 that swabbing forces greater than 25 N have significantly less swabbing efficacy than lower forces. It was observed that accumulation of particles at the swabs leading edge was significantly increased at these higher forces.

It is posited that leading edge accumulation is the primary mechanism responsible for reduced efficacy in this case. Figure 4.13 is presented to quantify the change in leading edge accumulation.

Figure 4.13 demonstrates that swabbing force can increase swab efficacy, with greater contaminant intensity seen throughout the radial profile with a 30 N swabbing force. Further to this, the leading edge of contamination accumulation peak is wider for the 30 N swabbing force, demonstrating the increase in leading edge accumulation with larger forces.

The results from Figure 4.12 have implications for the development of new swabbing protocols and swabbing systems. Swabbing force can impact swab efficacy and repeatability, consequently controlling swabbing force should be an important consideration. As these results only apply to the given surface and contaminant, further work must be done to test different surfaces and contaminants, determining which of these effects are specific to this system and whether are observed generally.

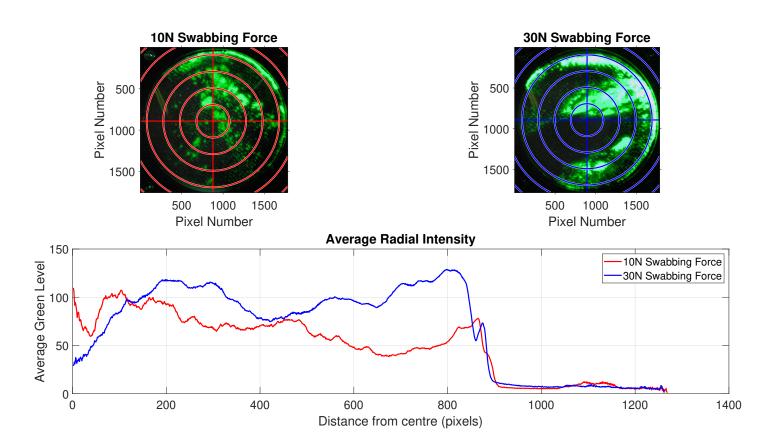


Figure 4.13: Radial contamination maps to compare swabs taken with two different swabbing forces.

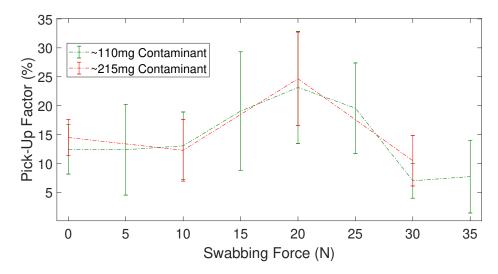


Figure 4.14: A comparison presenting the effects of swabbing force with different contaminant masses Taken from [83].

## **Contaminant Mass Effects**

Provisional results collected from the A.S.S. are presented in Figure 4.14. This graph suggests that there is agreement for pick-up factors independent of contaminant mass. A more thorough examination of the effects of changing contaminant mass is presented in Figure 4.15. The 215 *mg* contaminant mass tests were run for fewer swabbing forces, as they were only sought to determine whether contaminant mass impacts pick-up factor.

The uncertainties involved in the measurement of pick-up factor make any relationship between contaminant mass and pick-up factor challenging to discern. Figure 4.15 does not show any clear correlation between contaminant mass and pick-up factor, though it is notable that there is not a clear reduction or increase in pick-up factor for different contaminant mass. The results from Figures 4.14 and 4.15 may indicate that swab efficiency (see the definition for efficiency in Section 3.1) is constant across the mass range tested.

It is surprising that the swabs showed no sign of saturation across the contaminant range tested. Saturation (the point at which the swab is no longer effective in removing contamination) is an important consideration when making estimates about environments, and further work may wish to test over a wider range of contaminant masses to analyse saturation behaviour more closely.

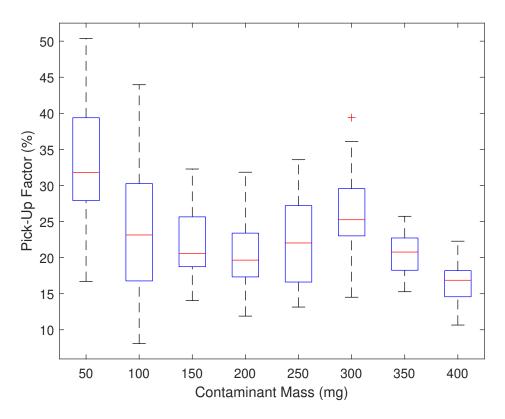


Figure 4.15: A comparison of swabbing performance for a range of contaminant masses (n=20).

#### Force Application Area and Swab Wetting Effects

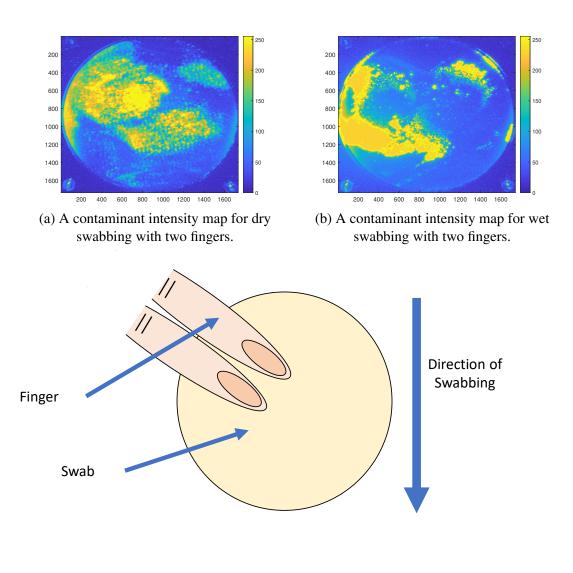
Force application area (FAA) is widely understood to be a factor influencing the removal of contamination during swabbing [59]. As well as influencing the swabbing pressure profile, FAA can change the geometry of a swab and thus impact the efficacy of swabbing. In order to systematically assess the impact of FAA on the swabbing process, a series of experiments were conducted.

It has been reported that swabbing from human operators led to the accumulation of contaminants around the area where the operator's fingers contacted the swab, with little contamination presenting in other areas [59]. Figure 4.16 presents the accumulation of contamination on a swab after a human operator applied force using two fingers. Figure 4.16 confirms the phenomenon seen in previous work [59], with contamination presenting predominantly where the fingers had touched the swab.

During wet swabbing, contamination clustered with a greater intensity around the edges of the fingers, while the spread of contamination was more diffuse after dry swabbing. It is apparent that the wetting agent (in this case deionised water) promoted contaminant particles to cluster.

In an effort to quantify the effects of FAA, a number of rubber circular force applicators with different diameters were employed. Figure 4.17 gives a comparison of these different force applicators' swabbing performance. It is evident that a larger swabbing area leads to greater efficacy, with an exception where the FAA. is equal to the area of the swab. Radial contamination maps, shown in Figure 4.18, were used to further elucidate the effects of FAA.

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(c) A depiction of the swabbing set-up.

Figure 4.16: A comparison of contaminant intensity maps for both wet and dry swabbing performed by a human operator.

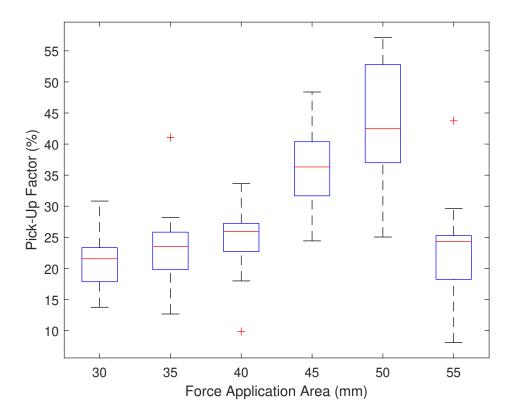


Figure 4.17: A comparison of swabbing performance for different force application areas, with a 55 *mm* diameter swab.

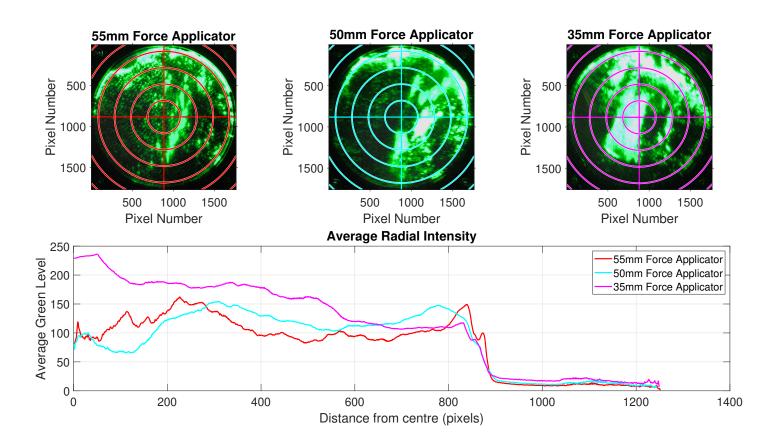


Figure 4.18: Radial contamination intensity maps for swabs (diameter 55 *mm*) taken with, **top left**: a 55 *mm* FAA, **top middle**: 50 *mm* FAA and **top right**: 35*mm* FAA. **Bottom**: a graph comparing the average radial intensity for these swabs.

#### 4.4. SUMMARY AND CONCLUSIONS

It is evident from Figure 4.18 that contamination is mainly collected around the leading edge of the swab, with some contamination spread more diffusely across the remainder of the swab. When comparing contamination maps for the 50 *mm* and 55 *mm* force applicators, it is evident that reducing the FAA to a size less than the swab area allows for contamination to be collected by a larger area of the swab. Comparing the peaks of the radial intensity graphs (around 850 from the centre) shows that the extent of leading edge contamination collected on the swab is greater for the 50 *mm* FAA. When comparing these results with the 35 *mm* applicator, it is clear that contamination is spread more diffusely with a smaller FAA and this may account for the reduced swab efficacy shown in Figure 4.17.

Figure 4.19 shows the contamination left on the swabbing bed after swabbing with different diameter force applicators. This figure provides further evidence for the importance of leading edge accumulation in swabbing. It is clear that a large volume of contamination not collected on the swabs is left around the leading edge of the swab, with this effect most pronounced for the 55 *mm* applicator. While these contamination maps are useful in elucidating contamination mechanisms, it is challenging to use these maps quantitatively as a relatively small amount of contamination led to optical saturation.

Swab wetting impacts the removal of contamination greatly. Figure 4.20 demonstrates the improved swab efficacy that a wetting agent provides, with dry swabbing leading to the smearing of contaminants across the contaminant bed.

It is clear that wetting agents allow for particulate contamination to 'clump' more easily and this positively impacts swab efficacy. Figure C.1 shows the effect of wetting on swabs taken with a range of force application areas. Dry swabbing leads to a more diffuse spread of contamination across the swab, whereas wet swabbing leads to more localised pick-up.

## 4.4 Summary and Conclusions

This chapter has elucidated key factors which influence the swabbing process. Swabbing experiments were analysed by measuring mass pick-up and various imaging techniques. Section 4.3.2 provides an insight in to the effects of changing swabbing force. It was observed that swabbing force can impact the efficacy of pick-up and the repeatability of swabbing, with an optimum force range found for this swabbing system. This section also demonstrated the effects of leading edge accumulation, a mechanism

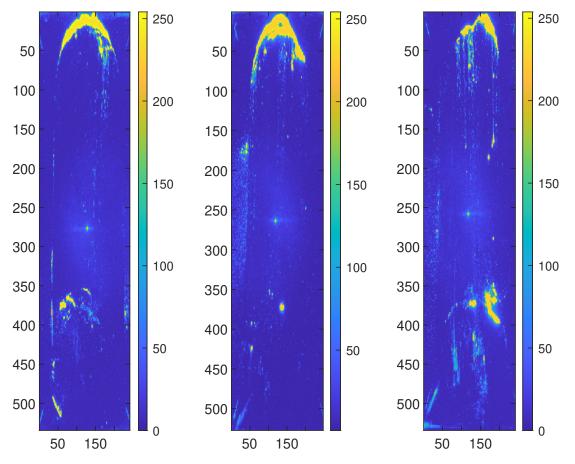


Figure 4.19: A comparison of the contamination left on the swabbing bed after wet swabbing with, **left**: a 55 *mm* force applicator, **centre**: a 50 *mm* force applicator and **right**: a 35 *mm* force applicator.

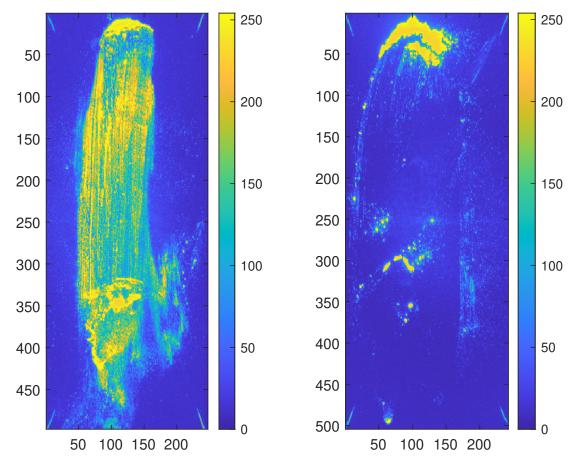


Figure 4.20: A comparison of the swabbing bed after, **left**: dry and **right**: wet swabbing.

which appears to be highly influential in the swabbing of particulate sample types.

Section 4.3.2 aimed to determine whether contaminant mass played a significant factor in the efficiency of a swab. While it is notable that there was no clear change in swab efficiency over the range of contaminant masses tested, the uncertainties involved in measuring pick-up factor dictate that no clear relationship can be discerned. Future work may wish to test over a wider range of contaminant masses, or to test different particle sizes and types.

This chapter also analysed the impact of force application area (FAA) on the swabbing process. Results demonstrated that a larger force application area provided more efficacious pick-up, though this may adversely affect swab repeatability. FAA appears to be a major influencing factor in the swabbing process, with FAA determining the extent of leading edge accumulation.

Results presented in this chapter begin to quantify the role of wetting agents, showing the increased grouping of contaminants when a wetting agent was used. Without a wetting agent, swabbing efficacy is limited, and swabbing results in the smearing of contamination across a surface. While the surfaces and contaminants which were the focus of this work are trivial to remove, and interact very weakly with the surface on which they were dispersed, further work exploring different chemical wetting agents and determining their impact on the swabbing process will be invaluable; especially when examining surfaces which are more representative of conditions that may be found in operational facilities.

The simple swabbing motions used in the experiments presented in this chapter are widely employed due to a desire for repeatability across multiple tests. With the advent of robotic swabbing systems, the requirement for the 'continuous' swab is no longer necessary, and more complex motions are achievable with a greater repeatability. Future work in this area should implement complex motions, for example using motions designed to limit leading-edge accumulation, and determine the impact of these motions on swab efficacy.

# **Chapter 5**

# Human Performance in Swabbing Tasks

The variability of human swabbing inputs is postulated as a primary influencing factor in the removal of contamination in swabbing tasks [60, 61]. Specifically, an inability for humans to provide a repeatable pressure profile over multiple swabs, coupled with an assumed lack of repeatability in area coverage is cited as the main reason for large swabbing uncertainties. Though this is widely assumed, there have been no previously reported attempts to measure human performance in these tasks and to determine to what extent human operators are to blame for swabbing uncertainty. This chapter details the results of a human participant study which provides the first record of human performance in swabbing tasks.

As human operators are still used predominantly in the collection of swabs across the nuclear industry [106], bench-marking their performance is an important step towards increasing the accuracy of predictions that are informed by information taken from swabs. The results from this study provide a valuable point of comparison for the next generation of swabbing robots, while also providing an assessment of swabbing accuracy for sites where robots may not be deployable.

This chapter is comprised of a section of definitions for technical terms used in this chapter, a detailed methodology which covers considerations in the design of this study as well as considerations in the collection and processing of data in this study. The results in this chapter are presented alongside a discussion of their implications. This chapter is concluded with a short summary which details the key findings and contributions of this chapter.

## 5.1 Definitions

- 1. **Swabbing** A sample retrieval process which involves the transfer of material from a surface on to a porous substrate (commonly filter paper).
- 2. **Swabbing Input** A collective term to describe the swabbing pressure profile, swabbing method and swab characteristics.
- 3. Swab Pass The number of times the swab area is covered.
- 4. **Convex Hull** The convex hull of a set of points is the intersection of all convex sets containing that set of points [107].
- 5. Pick-Up Factor The percentage of activity [68] removed from a surface on to a swab. Alternatively called "collection efficiency" [69] or "removal factor" [61]. Pick-Up factor is preferred in this thesis to avoid confusion with measurements taken in the fields of decontamination (where removal factor is used widely) and other fields where collection efficiency is used.

## 5.2 Methodologies

This section describes the study design, experimental procedures, chosen apparatus and the data processing techniques employed in the study which is detailed in this chapter. This study involved the collection of spatial, temporal and force data from 21 human participants who performed 30 swabbing tasks each. The requirements for this study can be seen in Table 5.1.

Data Type	Desired Collection Range	Required Precision
Spatial	100 <i>cm</i> (in 3D)	1 <i>cm</i>
Temporal	30 s	2 Hz
Force	50 N	0.1 <i>N</i>

Table 5.1: A table detailing the data collection requirements for this participant study.

The required spatial range was chosen to provide tracking over a volume that is expected to be covered by a human's hand during swabbing tasks, with the precision chosen to produce a tracking error of 1 %, with a higher error being undesirable. The

Data Type	Chosen Equipment	Collection Range	Precision	Sampling Frequency (
Spatial	Vicon Tracking System [109]	5 m	10 µm	250
Force	SingleTact Force Sensors [110]	100 N	0.1 <i>N</i>	3.3

Table 5.2: A table detailing the equipment used in this study.

temporal range is chosen to reflect an expected maximum for swabbing time, with the precision chosen to match the frequency at which humans can consciously control their behaviour [108]. This will allow for any intentional actions to be measured, without the need to measure high frequency tremors. The force collection range was chosen after an initial experiment, which showed that for an individual participant, applied swabbing forces were unlikely to exceed 50 N, with the precision again chosen to provide an error of 1 %.

Table 5.2 is included to provide an overview of the experimental equipment used in this study, and to provide a comparison against the requirements detailed in Table 5.1.

## 5.2.1 Study Design

The aims of this study were to measure human performance during swabbing tasks (using metrics seen to be important in determining swab efficacy, detailed in Chapter 4) with a sample that is representative of human operators who take swabs, and to collect data from a sample which has statistical significance. The key considerations in the design of this study were to develop a task that allows for the measurement of all relevant parameters while retaining the 'feeling' of real swabbing scenarios for the participants. This subsection details the design of this study and discusses the decisions taken in order to best meet the aims of this study.

As this study necessitated the recruitment of human participants, all of the relevant promotional material and experimental procedures were subject to a proportionate review by the University's Research ethics Committee. The promotional material can be found in Appendix E.

A key consideration in the design of equipment for this study was creating force sensing apparatus that could be integrated easily for swabbing tasks. The chosen force sensor had to be lightweight and small enough to not alter the properties of a paper swab significantly when they were fixed under one. For this reason, small capacitive

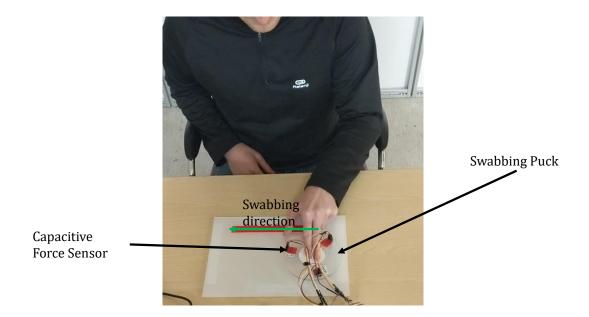


Figure 5.1: A picture of a participant completing the designed swabbing task.

force sensors were chosen, and this is further discussed in Section 5.2.2.

Figure 5.1 shows a model participant completing the designed swabbing task. Participants were asked to complete a number of double-pass swabbing tasks. The swab was placed on top of the swabbing puck, designed for this task. This puck housed three capacitive force sensors inside two sheets of acrylic. This apparatus was tested by the experimenter to ensure that using the swabbing puck did not drastically alter the feeling of the swabbing task.

Spatial tracking was achieved using a Vicon capture system, detailed in Section 5.2.2. Participants were made to place a tracking object on the top of their hand which was fastened with Velcro. This tracking object and the tracking system was not found to impede participants significantly.

#### **Sample Recruitment**

This study aimed to provide a sample size which was significantly larger than any analogous studies [99], where small sample sizes are commonly the main limitation of these studies. For this reason, recruitment aimed to enlist as many participants as possible to participate in the two-week collection time for this study.

The recruitment of participants was limited to engineers or those working in the fields of engineering (to replicate the operators found in the nuclear industry as closely as possible) who did not state an impairment which may affect their ability to swab.

Building access provided further limitations to recruitment, as only university staff and students could visit the lab where collection was taking place, due to control measures implemented to control the spread of Covid-19.

A total of 22 participants were recruited for this study, with data from 21 of these participants being suitable for analysis. The data from the remaining participant was not usable as the tracking data was not recorded correctly for this participant.

Each participant was tasked with completing 30 swabbing tasks, and although some of the participants completed more (one participant got up to 33), analysis was limited to 30 to simplify the data processing requirements.

The resulting analysis was based on these 630 swabbing trials (N=630), with this sample representing an order of magnitude increase on the samples utilised in previous analogous swabbing studies [99].

## 5.2.2 Experimental Design

This study required the collection of spatial, temporal and force data in order to benchmark typical human force application, area coverage and swab times. Spatial data was collected using a Vicon tracking system [109], and force data was collected independently using SingleTact force sensors [110]. The specifics of the equipment and methods used, as well as the data processing techniques used are detailed in this subsection.

## **Spatial Tracking**

Participant hand positions were tracked by 12 Vicon (Vero v1.3 X) cameras in an enclosure depicted in Figure 5.2. Participants were fitted with a tracking object, which housed 5 IR-reflective markers. Using this tracking object. the Vicon cameras were able to provide position and rotation information for the participant's hand at a frequency of 250 Hz. The accuracy of similar tracking systems is known to be between  $63 - 290 \mu m$ , which would provide spatial tracking with an accuracy and frequency far greater than required (see Table 5.1).

During the study, the participant was first fitted with the tracking object, with the distance from the centre of this object to the centre of the participant's index finger being recorded. After the participant's test runs, the Vicon system began recording. After the trials were completed by the participant, the recording was saved. This gave

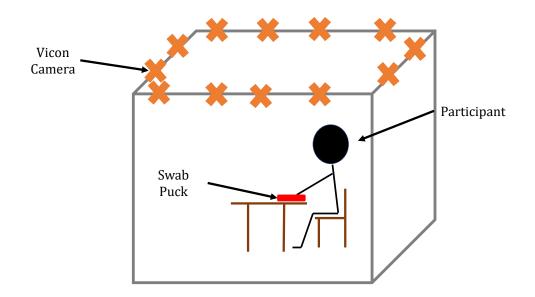


Figure 5.2: A depiction of the tracking enclosure used in this study.

a record of spatial coverage for a continuous period which contained all of the participant's trials. In order to provide spatial information for only the time periods of interest (during each swabbing trial) the tracking data was replayed and the frames of interest were recorded manually. This process introduced a potential source for error, with the exact start of each trial difficult to distinguish.

After the spatial data was refined so that it included only frames of interest, the spatial data (from the centre of the participant's hand) was converted to the estimated position of the centre of the swab, using the transformation shown in Equations 5.1 to 5.5. The processing of spatial data is shown in Figure 5.3.

$$\vec{x_s} = \underbrace{R_{xyz}}_{c} \vec{L} + \vec{x} \tag{5.1}$$

$$\vec{x}_s = \begin{bmatrix} x_s \\ y_s \\ z_s \end{bmatrix}$$
(5.2)

$$\vec{x} = \begin{bmatrix} x \\ y \\ z \end{bmatrix}$$
(5.3)

$$\vec{L} = \begin{bmatrix} 0\\L\\0 \end{bmatrix}$$
(5.4)

$$\underline{\underline{R}_{xyz}} = \begin{bmatrix} \cos\beta\cos\gamma & \sin\alpha\sin\beta\cos\gamma - \cos\alpha\sin\gamma & \cos\alpha\sin\beta\cos\gamma + \sin\alpha\sin\gamma\\ \cos\beta\sin\gamma & \sin\alpha\sin\beta\sin\gamma + \cos\alpha\cos\gamma & \cos\alpha\sin\beta\sin\gamma - \sin\alpha\cos\gamma\\ -\sin\beta & \sin\alpha\cos\beta & \cos\alpha\cos\beta \end{bmatrix}$$
(5.5)

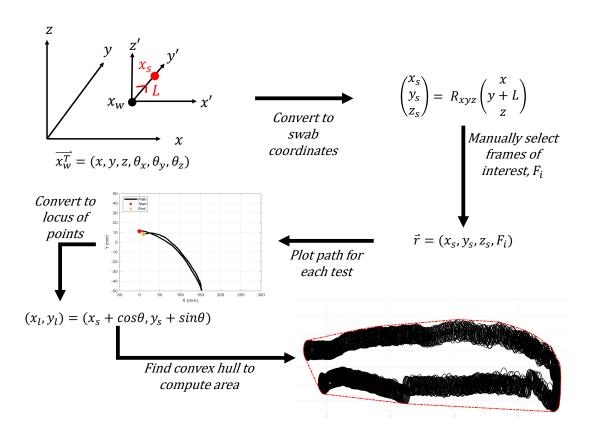


Figure 5.3: A visualisation of processing techniques used to calculate area coverage for each trial.

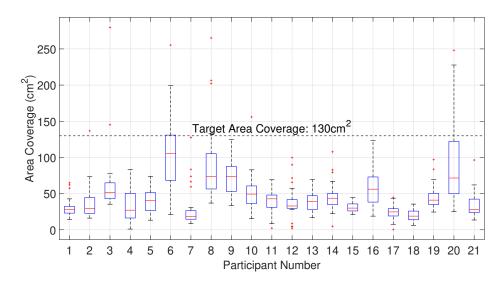


Figure 5.4: A box plot showing the estimated area coverage for each participant across their trials without using the locus of points method. The dashed line shows the area that should be covered in the goal swab path.

After spatial data were converted to reflect the position of the swab centre, a locus of 100 points was created around each (x, y) swab position to reflect the full area of the swab. This step was performed to reflect the physical situation. Areas calculated using only the centre points were a major under-estimation of area coverage, and in the limit where the executed path was perfectly straight (as participants were instructed) the area coverage would tend towards zero. A summary of the area estimations without using the locus of points method are shown in Figure 5.4.

The spatial data were collected and processed for each trial and every participant, with the area covered being recorded for each of these trials. Area coverage was estimated by calculating the convex hull for the set of points representing the swab's position.

#### **Force Sensing**

Figure 5.5 shows a force diagram for a swabbing puck during swabbing.  $F_v$  and  $F_h$  are the vertical and horizontal forces applied during (commonly by an operator). R is the reaction force from the surface,  $\mu_s$  and  $\mu_c$  are the coefficients of friction caused by the swab-surface and swab-contaminant interfaces respectively and  $W_p$  is the weight of the swabbing puck and swab combined.

During swabbing tasks it is common for operators to apply a larger vertical force

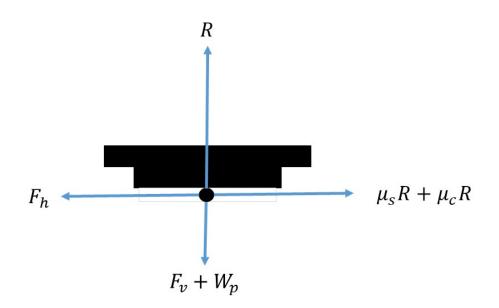


Figure 5.5: A Force diagram showing the forces on a swabbing puck during swabbing, adapted from [83].

to displace contaminants which are more strongly adhered to the surface. This vertical force will also be known as the swabbing force and it functions to remove contamination by increasing the frictional force between interfaces.

In order to measure swabbing forces from participants in a way that is representative of common swabbing scenarios, the chosen force sensor must be lightweight and flexible. It was desirable to choose force sensing equipment that would not interfere with the swabbing process. Lightweight capacitive force sensors were found to operate with adequate precision, over the specified force range (see Table 5.1), while being readily integrable for this study.

Three force sensors were placed in a triangular formation on the surface of a swabbing puck, shown in Figure 5.6. Three sensors were used to provide some insight in to the pressure profile across the swab through the swabbing motion.

Force data was communicated via serial, with the data being recorded on a control PC during each trial. Each sensor shared the same serial port, hence the frequency for each sensor reading was 3.3 Hz (one third of the value of a single sensor). This

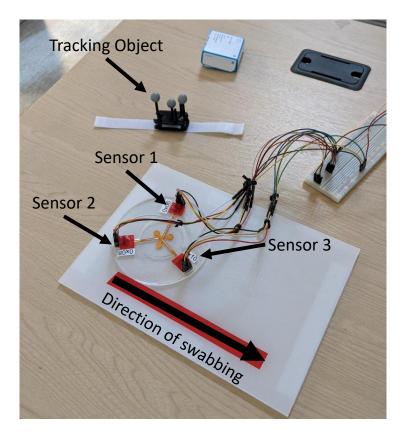


Figure 5.6: An annotated picture showing the force sensing equipment used in this study.

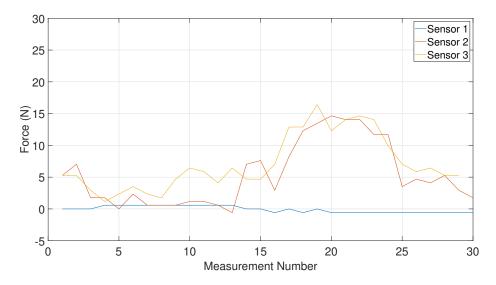


Figure 5.7: An example of the force readings taken from a trial (P1R2).

collection frequency still exceeded the threshold between intentional and unintentional movements for humans of 2 Hz [108]. The force sensors used 10-bit encoding for messages (1024 distinct values) across a range of 100 N, giving a total precision of < 1 N.

The digitised voltage response from these sensors was converted to a force estimation using the manufacturers calibration curve (which has been tested separately in Figure 4.2). These force values were collected for every trial for each participant, with the mean force inputs and variance being recorded. Force balance (see Equation 5.6) was also recorded for these runs, in order to give an understanding of participants' pressure profiles. In Equation 5.6 *B* is the force balance and  $\bar{F}_2$  is the mean of readings on force sensor 2.

$$B = \frac{\bar{F}_2}{\bar{F}_3} \tag{5.6}$$

Figure 5.7 shows the force readings from one trial (P1R2). It can be seen that the reading from Sensor 1 was close to zero throughout the trial. This could be due to a lack of force being applied to this sensor, or by sensor malfunction. The sensor was tested (and found responsive) prior to the participant's arrival, so sensor malfunction is considered unlikely. This trend of low readings from sensor 1 was consistent through the study. It is thought that this is a consequence of the sensor being placed below the usual point of application, with little motion expected from the participants fingers along the vertical axis.

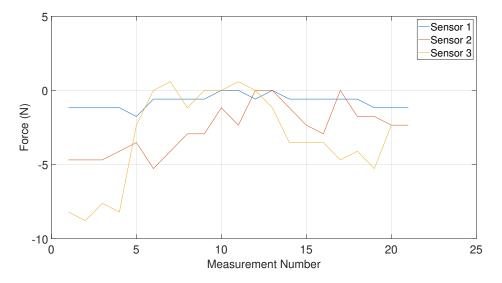


Figure 5.8: An example of the force readings taken from a trial (P21R5) showing consistent under-pressure readings.

A significant number of trials (N = 51) showed under-pressure readings for the entirety of swab motion. This occurred when no sensor was being contacted by the participant's fingers and may have been caused by the paper swab lifting the sensor up, the participant deforming the sensor significantly or an issue with a malfunctioning sensor. As the situation of under-pressure is not physically possible, any under-pressure values were taken to be zero when the force readings were analysed.

## 5.3 Results

Through this study the spatial coverage and force application from 21 human participants was recorded over 30 swabbing trials. The collected data were analysed to produce a representative understanding for human performance in area coverage and force application in swabbing tasks. The uncontrolled variable of swabbing time was also analysed. The data were considered separately as well as combined, in order to provide insights in to potential correlations between different swabbing inputs.

## 5.3.1 Changes of Performance

As the participants in this sample were not selected on the basis of having prior experience of swabbing tasks, it was important to ascertain whether there were any significant changes in performance throughout the trials.

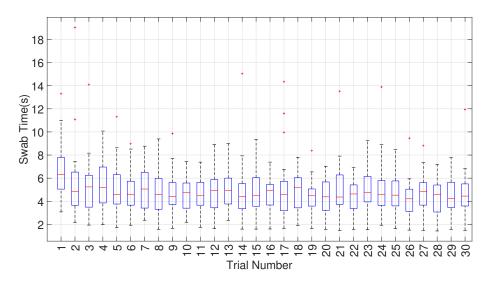


Figure 5.9: A box plot presenting the evolution of swab time for all participants as their trial number increased.

Table 5.3: A table to show the evolution of swab time standard deviation with increasing trial number.

Run Numbers	$\sigma_t$ (s)	Normalised $\sigma_t$ (%)
1-5	2.7995	82.1343
6 - 10	1.7728	52.0107
11 - 15	1.8987	55.7059
16 - 20	1.8295	53.6743
21 - 25	1.9665	57.6952
26-30	1.6892	49.5578

Figure 5.9 presents the changes in swab time for participants as they progressed through their trials. After an initial period of adjustment through the first 5 trials, there is a clear settling (showed in a decreased spread of swab times) from this point on. This finding is reinforced in Table 5.3. As participants were not explicitly told to control their swab time, these results are unsurprising. After an initial adjustment period, participants settled in to a routine with relatively less variance in swab time. These results show no clear evidence of changes in performance with a large number of trials, and hence this reinforces the validity of the participant selection protocols.

Results for area coverage and force application, two variables intended to be controlled by participants, were analysed similarly to Figure 5.9. The evolution of swab

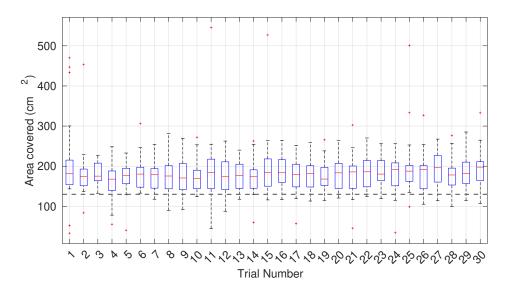


Figure 5.10: A box plot presenting the evolution of area coverage with increasing trial number. The dashed line shows the target area coverage for these tasks.

area coverage is shown in Figure 5.10 and the change in mean force application is shown in 5.11.

The effect of trial number on swabbing area, shown in Figure 5.10 and Table 5.4, is far less pronounced than any changes in swabbing time. There is just a 5 % reduction in standard deviation for area coverage, when compared to approximately 30 % for swab time. This is an indication that there was no change in performance throughout the trials.

Figure 5.11 and Table 5.5 detail the changes in mean force application through the trials. There is a 7 % reduction in mean force application on average through the last

Table 5.4: A table to show the evolution of area coverage standard deviation with increasing trial number.

Run Numbers	$\sigma_A (cm^2)$	Normalised $\sigma_A$ (%)
1-5	69.9679	82.1052
6 - 10	74.8872	87.8778
11 - 15	61.876	72.6067
16 - 20	58.4884	68.6343
21 - 25	58.9041	69.1222
26 - 30	66.7784	78.3624

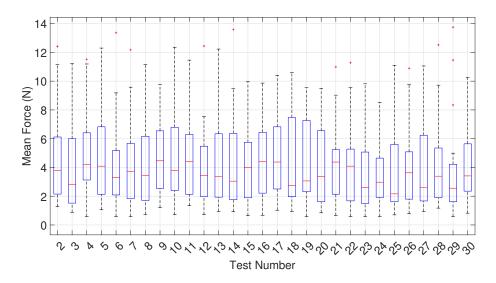


Figure 5.11: A box plot presenting the evolution of mean swabbing force with increasing trial number. **Note:** trial 1 is not included in this analysis due to a number of corrupted tracking files.

five tests, when compared to the first five. This trend is not significant to suggest any significant changes in performance by the sample throughout the tests.

## 5.3.2 Spatial Tracking and Area Coverage Performance

Participants were given a task to execute a straight, double-pass swab across a 20 *cm* length, 30 times while their hand positions were tracked using a Vicon system. The collected tracking data were used to provide the coordinates of the swab centre, which was used to show the area covered in each swabbing trial, with the methods used

Table 5.5: A table to show the evolution of mean force standard deviation with increasing trial number.

Run Numbers	$\sigma_F(N)$	Normalised $\sigma_F$ (%)
1-5	3.3504	82.4774
6 - 10	2.9998	73.8464
11 - 15	2.9530	72.6929
16 - 20	2.9736	73.2002
21 - 25	2.6623	65.5379
26 - 30	3.0620	75.3773

being detailed in Section 5.2.2. Figure 5.12 shows the area covered (in black) and the area estimated by the convex hull of tracking points (in red, dashed) for one trial. It is evident that the convex hull will always provide an over-estimate for the area coverage, though quantifying this over-estimate is beyond the scope of this work.

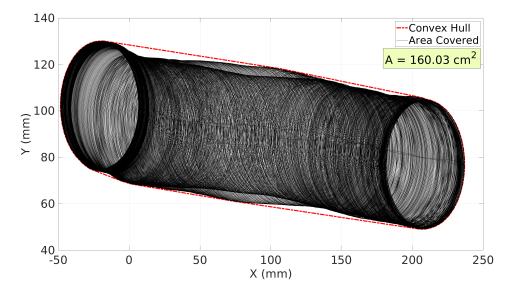


Figure 5.12: A plot showing the swab coverage and area estimation for one trial (P5R5).

The area coverage for each participant was collected and is presented in Figure 5.13. It is evident that most participants' area coverage was on average over the goal area coverage. For a number of participants, their means were significantly higher than the target (P6 had a mean area coverage that was more than double the goal). This was primarily a consequence of vertical movement (perpendicular to the proposed swab path), as opposed to an inability to recreate the path length well.

Figure 5.14 is an extreme case of swabbing area over-estimation in this study, where there appears tracking points which do not match physical intuition. Despite the potential for over-estimations caused by this type of error, the area coverage of many participants was seen to be reasonably repeatable, as shown in Figure 5.13. The coefficient of variation (Equation 5.7) is presented alongside mean area coverage in Table 5.6.  $C_v$  is the coefficient of variation,  $\sigma$  is the standard deviation and  $\mu$  is the mean.

$$C_{\nu} = \frac{\sigma}{\mu} \tag{5.7}$$

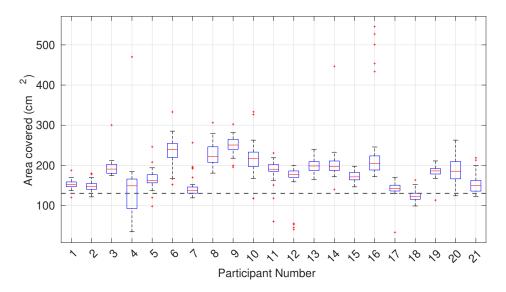


Figure 5.13: A box plot to show the area coverage performance for each participant. The dashed line shows the target area coverage.

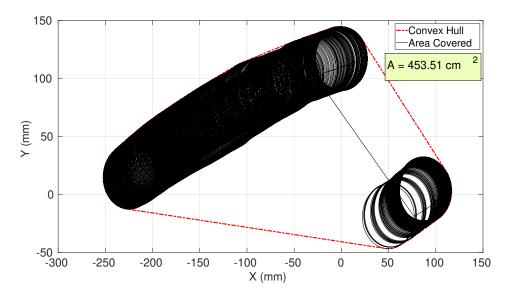


Figure 5.14: A plot showing the swab coverage and area estimation for one trial (P16R2).

Participant	$Mean(cm^2)$	$C_{v}$ (%)	Participant	Mean $(cm^2)$	$C_{v}(\%)$
P1	153.00	7.63	P12	162.46	28.75
P2	147.85	10.50	P13	198.50	9.27
P3	194.42	11.81	P14	204.73	24.12
P4	143.73	51.81	P15	172.68	6.80
P5	165.22	15.84	P16	248.30	45.72
P6	233.44	16.35	P17	140.62	16.04
P7	146.91	19.55	P18	124.05	11.78
P8	227.93	12.23	P19	183.95	9.03
P9	250.03	9.21	P20	189.10	15.18
P10	218.10	20.63	P21	153.99	15.02
P11	185.87	16.94			

Table 5.6: A table showing the mean and coefficient of variation of area coverage for each participant.

Table 5.7: A table showing the mean area coverage and coefficient of variation for the whole sample.

Participant	Mean $(cm^2)$	$C_{v}$ (%)
Full Sample	191.84	17.82

Table 5.6 shows a  $C_v$  range of between 6.8 – 51.8 %. These values can be combined to give a representative area coverage across the sample. Our representative area coverage is shown in Table 5.7. These values represent the first attempt to bench-mark the area coverage of human participants in swabbing tasks.

A histogram for the area coverage across all test was collected by separating area coverage linearly across sixty bins. This is visualised in Figure 5.15. It is evident that the spread of data is visually similar to a normal distribution. Further work may test this assertion, to determine whether area coverage can be well-modelled by a normal distribution. This has implications for the characterisation community, as it suggests that this aspect of human input may be predictable, therefore resulting errors may be accounted for.

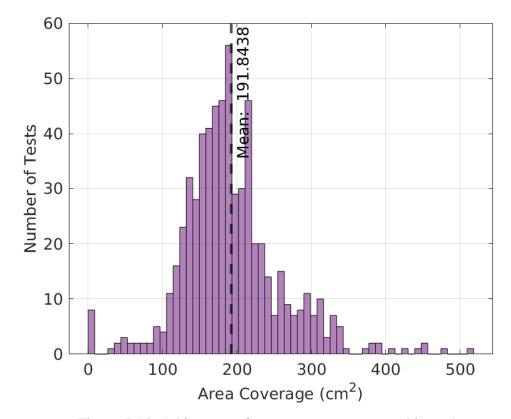


Figure 5.15: A histogram for area coverage across this study.

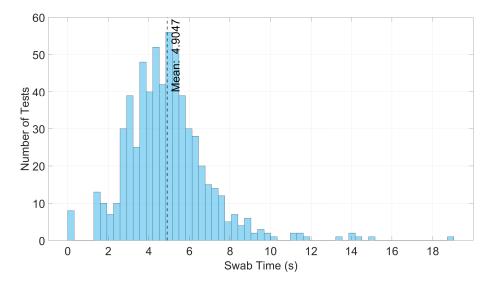


Figure 5.16: A histogram for swab time across this human operator swabbing study.

#### **5.3.3 Temporal Performance**

Swab time was not explicitly told to be controlled to participants, though these data were collected with the view to determine whether there is any subconscious control of this variable and to see whether there are any patterns between controlling swab time and area coverage performance or force application performance.

The distribution of swab times for the whole study is shown in Figure 5.16, with a clear skew to swab times which were smaller than the mean. This implies that there were a considerable number of trials which were far longer than the mean.

The swab times for each participant were collated and are shown in the box plot in Figure 5.17. This figure shows similarities to Figure 5.13, with a few participants demonstrating a far larger spread of swab times than the rest of the sample. To determine any correlation between area coverage variance and swab time variance, the coefficients of variation for both were compared for each participant in Figure 5.18. The correlation between the two was fairly strong with a coefficient of determination of 0.47. This suggests that controlling swab time can have a positive impact on reducing the variance in area coverage.

#### 5.3.4 Force Application Performance

Force data were collected from three sensors placed on the underside of a swab (see Figure 5.6) for each trial in this study. An example of the force data collected for

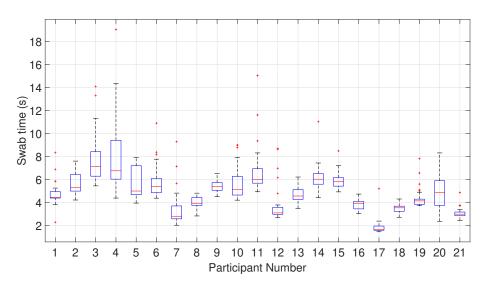


Figure 5.17: A box plot showing swab time performance for each participant.

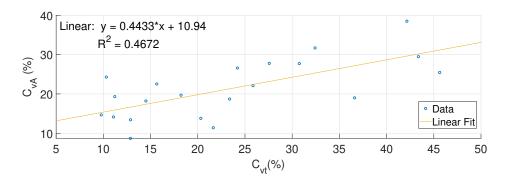


Figure 5.18: A plot comparing the coefficients of variation for swab area and swab time.

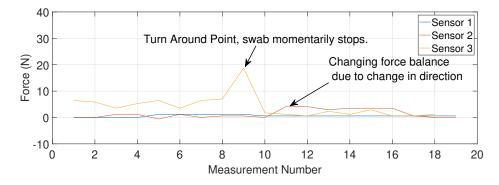


Figure 5.19: A graph showing the calibrated force response measured in one trial (P6R28)

each of these trials is presented in Figure 5.19. It is evident that the force applied to each sensor was not constant, and the relative pressure applied to each sensor was not consistent through each trial. To quantify these features, a number of metrics were employed. Mean force application was calculated using Equation 5.8.

$$\bar{F} = \frac{\sum_{n=1}^{N} (F_1(n) + F_2(n) + F_3(n))}{N}, \forall F > 0$$
(5.8)

In addition to mean force, the coefficient of variation (see Equation 5.7) was also collected for each trial to act as a metric for the 'consistency' of force application through each trial. The maximum force for each of these trials was also recorded. Histograms for each of these metrics were collated (each with 60 bins) and can be seen in Figures 5.20, 5.21 and 5.22. These three histograms are all skewed to values lower than the mean, and further work may investigate whether these findings can be used to predict swabbing errors.

Box plots were also collected for each of the three force application metrics. With the results presented in Figures 5.23, 5.24 and 5.25. Table 5.8 provides a summary of the mean force application and consistency for each participant. It is clear from these results that force application through swabbing tasks is highly inconsistent. Only one participant (P12) was able to achieve a  $C_{\nu}$  of less than 50 % of the mean force application. This finding supports the common assertion in previous reports that force application is inconsistent through swabbing tasks [99].

Figures 5.23 and 5.24 give clear indications of representative swabbing mean and max forces. All participants applied mean forces of < 10 N in their trials, with only one participant (P2) applying a peak swabbing force of over 20 N with regularity. While the values may change in different scenarios (where the coefficient of friction

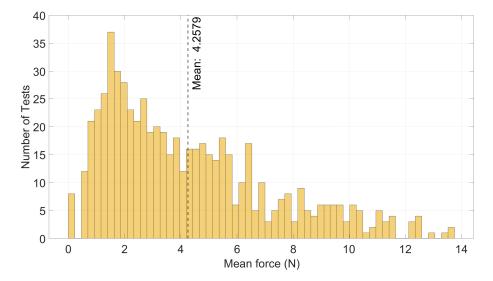


Figure 5.20: A histogram showing the mean force application in this human operator swabbing study.

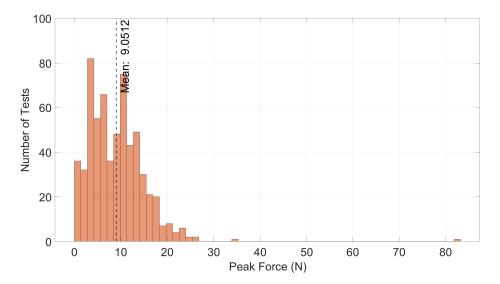


Figure 5.21: A histogram showing the maximum force application in this human operator swabbing study.

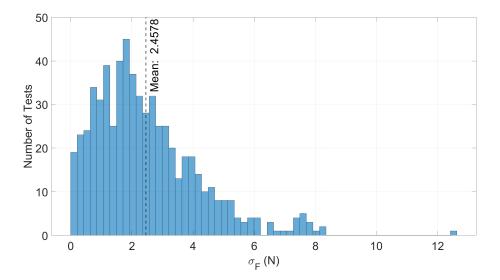


Figure 5.22: A histogram showing the standard deviation of force application for each participant in this swabbing study.

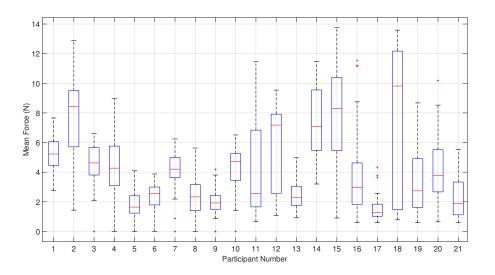


Figure 5.23: A box plot showing the mean force application for each participant in this study.

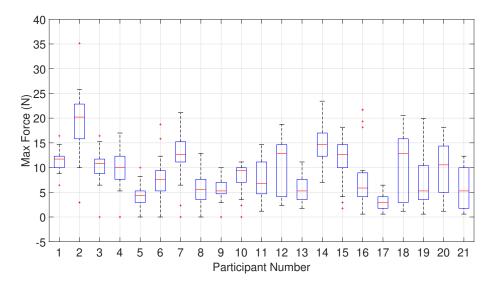


Figure 5.24: A box plot showing the maximum force application for each participant in this human operator swabbing study.

is different, or when the geometry of the swabbing surface changes the ability for the operator to apply force) these are still useful values to inform the characterisation process. Further to this, these values act as a point of comparison in the development of swabbing protocols for robotic platforms.

The values for  $C_v$  shown in Figure 5.25 further demonstrate inconsistency in force application during swabbing tasks. These values would correspond to force range through each swabbing trial of between 1 - 10 N, a variation that could have a large impact on swabbing efficacy (when consulting the results in Chapter 4).

Table 5.8 gives an overview of the force application performance for each participant involved in this study. To ascertain any correlations between mean force application and consistency of force application, Figure 5.26 was produced. The correlation between these two factors is not strong enough to suggest any interdependence between these two factors.

In order to provide a bench-mark value for force application through this study, the mean force application and mean coefficient of variation are presented in Table 5.9. These values may be used in future work to predict swabbing error contributions from force application.

Finally, the relationships between swab time and area coverage and consistency of force application were processed and are presented in Figures 5.27 and 5.28. These figures show no clear correlation when compared to the correlation between swab time

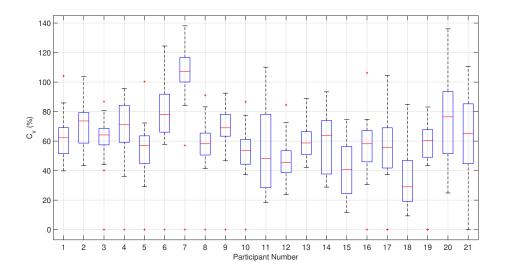


Figure 5.25: A box plot showing the coefficient of variation of force application for each participant in this human operator swabbing study.

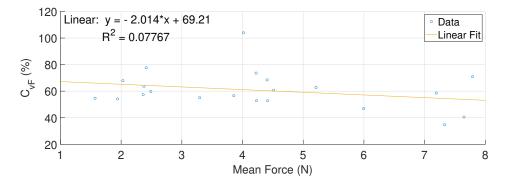


Figure 5.26: A graph showing the relationship between mean force application and coefficient of variation for force, a metric used to measure the consistency of force application.

Participant	Mean (N)	$C_{v}(\%)$	Participant	Mean (N)	$C_{v}$ (%)
P1	5.21	62.78	P12	6.00	46.88
P2	7.79	70.93	P13	2.49	59.79
P3	4.51	60.73	P14	7.19	58.57
P4	4.41	68.43	P15	7.65	40.51
P5	1.94	54.16	P16	3.86	56.62
P6	2.41	77.61	P17	1.57	54.67
P7	4.02	103.94	P18	7.33	34.68
P8	2.36	57.41	P19	3.29	55.07
P9	2.033	67.92	P20	4.22	73.62
P10	4.23	52.85	P21	2.38	63.66
P11	4.41	52.78			

Table 5.8: A table showing the mean force application and average coefficient of variation for each participant

Table 5.9: A table showing the mean force application and coefficient of variation across all participants.

Participant	Mean (N)	$C_{v}(\%)$
All Participants	4.09	60.65

and area coverage (see Figure 5.18). The data presented suggest that force application acts independently to swab time and area coverage. Thus, there is no evidence that controlling swabbing force will impact area coverage or swab time.

#### 5.3.5 Study-Wide Analyses

While previous results in this section have determined correlations between average performances of participants using a number of different metrics, these correlations have not been investigated for each trial separately. The analysis of individual trials gives greater understanding as to whether any of the measured swabbing inputs depend on one another.

The majority of the analyses performed showed very weak correlations, and can be seen in Appendix D. Figure 5.29 is included in this chapter as a relatively strong correlation was found between C.O.V. of area coverage and C.O.V. of swabbing time,

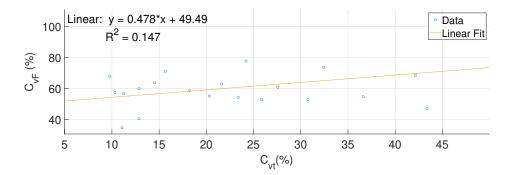


Figure 5.27: A graph showing the relationship between coefficient of variation for force and swab time.

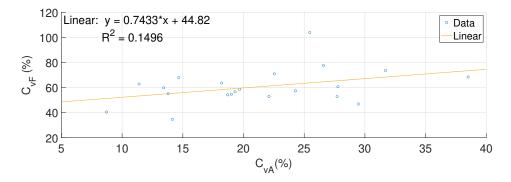


Figure 5.28: A graph showing the relationship between coefficient of variation for force and area coverage.

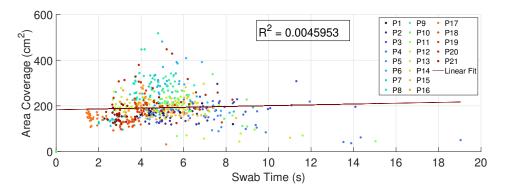


Figure 5.29: A figure showing the relationship between area coverage and swabbing time in this human operator swabbing study, with each swabbing test recorded. Colour-coded groups are given for each individual participant.

this is shown in Figure 5.18.

The relationship between coefficients of variation in Figure 5.18 does not extend to the relationship between area coverage and swab time, as the correlation displayed in Figure 5.29 is very weak.

## 5.4 Summary and Conclusions

This chapter presents the results of a human participant study aimed at providing an understanding of typical performance in swabbing tasks. This study uses spatial, temporal and force data to determine typical area coverage, force application and swab time for the participants in this study. This chapter also explores correlation between these metrics, in order to provide a greater understanding of errors which may contribute towards swabbing performance.

This study is the first of its kind to measure human performance in swabbing tasks, with a total sample of N = 630 swabbing tasks, where its scale far exceeds other analogous swabbing studies (i.e. an order of magnitude higher than studies in [99]).

A novel method combining a locus of points method with convex hull calculation was developed to estimate area coverage in this chapter. Across this study the average area coverage was found to be 191.8  $cm^2$  (compared to the goal area coverage of 130  $cm^2$ ) and the average coefficient of variation for area coverage was found to be 21.3 % of the mean coverage. This value can be used as a representative figure for human area coverage in swabbing tasks, which further work may attempt to link this to uncertainties in pick-up factor.

The mean force application for all participants in this study was found to be 4.09 N, with the average peak force being 9.05 N. The consistency of force application was also measured and found to be 60.7 % of the mean force application. These values may be used in future work to determine the error contributions from inconsistent force application on pick-up factor.

The results from this study were also analysed to determine any correlations between swabbing inputs. The most compelling correlation was between consistency of swabbing time and consistency of area coverage, with a coefficient of determination of 0.4672, suggesting that the control of swab time can positively impact area coverage consistency, and vice versa. This has implications for the development of swabbing protocols for human operators. Table 5.6 shows a  $C_v$  range of between 6.8 – 51.8 %. These values can be combined to give a representative area coverage across the sample. Our representative area coverage is shown in Table 5.7. These values represent the first attempt to bench-mark the area coverage of human participants in swabbing tasks.

These results demonstrate a lack of repeatability of human in swabbing tasks. This raises serious issues around current swabbing practices, and the use of human-taken swabs. Future work should determine human pick-up factor repeatability using the large sample sizes used in this study, as this would provide a strong justification for the changing of swabbing practices.

## **Chapter 6**

# The Feasibility of Chrysoidine G for *in situ* Characterisation

This chapter focuses on a feasibility study performed to ascertain whether the chemosensor, Chrysodine G is a suitable candidate for use to detect the fission products, strontium and ceasium, in radioactive environments. The contents of this chapter are an adaptation of material presented in paper format, adapted to fit the style of this thesis.

## 6.1 Definitions

This section provides definitions for a number of technical terms which are included in this chapter. While some of these definitions may appear in other chapters, they are included additionally here for the ease of the reader.

- 1. **Chemosensor** A molecular structure which provides a detectable signal in response to the presence of some analyte [111].
- 2. Analyte A particle or species of analytical interest.
- Identification In the nuclear industry, identification tasks are taken in order to ascertain the specific hazards associated with an environment. Identification tasks can pertain to the identification of physical, chemical and specific radiological hazards.
- 4. **Characterisation** Characterisation tasks are undertaken in the nuclear industry to assess the chemical 'make-up' and the quantity of chemical and radiological hazards.

- 5. **Monitoring** Monitoring is a routine task in the nuclear which necessitates the collection of dose rates from different locations across an environment. The requirement for monitoring satisfies regulatory requirements and also contributes to operational and decommissioning decision-making.
- 6. *in situ* For the purposes of this chapter, *in situ* is defined as in the place which is being characterised (or inspected, or monitored).

## 6.2 Introduction

Assessing the location and volume of radioactive contaminants is essential throughout the life-cycle of facilities in the nuclear industry [6, 112]. Accurate characterisation is important in maintaining the safety of workers and also developing suitable decommissioning plans for radioactive facilities [8]. While this characterisation has previously been performed using expensive radiometric counters and by extracting samples for *ex situ* analysis, the development of low-cost and facile chemical sensing technologies [111] could potentially be greatly advantageous in reducing the time and cost, as well as increasing the safety of characterisation tasks.

Identification, characterisation and monitoring tasks aim to locate, speciate and quantify hazardous contaminants. These tasks are performed in environments with widely varying physicochemistry, and often without prior knowledge of the environment [6, 106]. These analytical challenges are further complicated by the risks associated with exposure to radiation in these environments, imposing limitations on the complexity of technologies that can be deployed in these environments as well as imposing strict requirements for the robustness of sensing technologies that are suitable for deployment [14].

Development of robots for the inspection and monitoring of nuclear facilities has grown rapidly since the 2012 incident at the Fukushima Daiichi Nuclear Power Plant. Much development has focused on increasing the level of autonomy and increasing characterisation capability of robotic platforms. Platforms such as the CARMA [17] and RICA [25] can now provide radiometric heat maps of environments with minimal human intervention. These platforms use commercial radiometric sensors. CARMA uses an  $\alpha$  sensor as well as  $\gamma$  dosimeters. These platforms provide useful information while reducing the exposure to ionising radiation for plant operators, however there remains a large amount of characterisation information from these environments that cannot be collected by current robotic platforms, and therefore such characterisation is reliant on person ingress.

In order to better characterise information from these environments, and ultimately eliminate the need for human operators to enter hazardous environments, the capability of inspection robots to perform different analytical techniques must be improved. Current research utilising laser diagnostics and other analytical techniques [18] exists, with TORONE representing the current state of the art for a non-contact characterisation robot [27, 34]. In practice operators are still be required to enter these environments to use sensors and retrieve samples that are analysed in a separate lab, and so there is still a need to broaden the capability of robotic platforms deployed in to radioactive environments.

#### 6.2.1 Strontium and Caesium Detection

Strontium-90 is a focus in the characterisation process as it is among the most hazardous radionuclides to plant and animal life and it has a large abundance in SNF immediately after reactor operations [113]. Its half-life (27.7 years) means that strontium can be problematic for many decades after decommissioning [114, 115].

Strontium-90 detection is predominantly radiometrically measured in the laboratory through the use of liquid scintillation counting (LSC) [116], though non-radiometric methods such as is Inductively Coupled Plasa- Mass Spectrometry (ICP-MS) [117] are also utilised. Despite low limits of detection  $(10^{-9} gkg^{-1}$  for ICP-MS, 20.1  $Bqkg^{-1}$  for LSC) [117, 116], both require complex sample preparation and also expensive sensing equipment. A sensing technique which does not have these disadvantages may be much more readily adapted for *in situ* inspection. Recent chemical sensing developments have made progress towards more facile strontium detection [118].

Caesium-137 has a similar half life (30.2 years) to strontium-90 [6] and is a gammaemitter. It is known to persist in the muscles of organisms with fish being adversely affected upon exposure [6]. Caesium-137 is commonly detected radiometrically [119] using techniques such as gamma spectroscopy( $\gamma$  - spec). Radiometric detection of Cs-137 can be challenging in highly-active areas, or areas where sensor geometry is incompatible.

Caesium and strontium contamination are found in both aqueous and solid forms. In radioactive effluent treatment plants, it is essential to monitor levels of strontium and caesium, down to the parts per trillion level, before waste can be discharged [6]. Both of these fission products are also known to have been found in solid environmental

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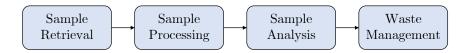


Figure 6.1: An overview of the tasks involved to perform *in situ* robotic characterisation and important considerations for each task.

samples [120], be a focus in the characterisation of environments post-accident [121] and also found to contaminate material components in various nuclear process plants [85].

In the nuclear industry, detection techniques for radioactive strontium and caesium should have a low limit of detection. Ideally techniques should be able to detect strontium and caesium down to the parts per billion level (ppb) [6] so that waste can be cleared outside the scope for radioactive waste handling. There is also a requirement for detection techniques to cope with the varying chemical, physical and radiological conditions under which it will be required to operate. Resilience to pH changes are particularly important due to the amount of characterisation work done after POCO [106] where many components and environments are exposed to strong nitric acid.

#### 6.2.2 *In-Situ* Chemical Analysis

Chemical analysis is a multi-stage process as shown in Figure 6.1. The development of each stage of this process, from sample retrieval to sensing, may be constrained and informed by the specifics of each other stage. In a lab-based environment, these stages can (i) take a long time, (ii) require carefully controlled environments and/or (iii) make use of large or expensive analytical hardware.

To implement 'lab-style' chemical sensing *in-situ* using a robotic platform is very challenging due to the requirement to complete the full process in a very confined space. Figure 6.2 shows a picture of the Clearpath Husky, which is one of the larger commercially available mobile platforms that could be used for *in-situ* chemical analysis. These size constraints would limit the range of analytical techniques that could be readily utilised. The sizes and payload capacity of a number of other commercially available platforms are shown in Table 6.1.

#### 6.2. INTRODUCTION



Figure 6.2: An image of the Clearpath Husky robot fitted with an arm. Dimensions for the operational area where equipment may be mounted are highlighted in red.

Table 6.1: A table detailing the payload capacities for a selection of commercial robotic platforms commonly used in inspection robotics.

Platform	Dimensions (cm)	Payload Capacity (kg)
Clearpath Husky	99 x 67 x 39	75
Clearpath Jackal	51 x 43 x 25	20
AgileX Scout 2.0	93 x 70 x 35	50
AgileX Scout Mini	61 x 58 x 25	50
Boston Dynamics Spot	110 x 60 x 51	14

An arm to manipulate samples and a flat surface area to allow for sample processing are likely to be required to perform most sample-based chemical sensing techniques. The limited space on commercially available platforms means there is a need to develop characterisation techniques which require only a small footprint that do not necessitate bulky sensors or complex multi-stage processing.

#### **Sample Retrieval**

Sample retrieval forms an integral part of current characterisation practices. The ability to interact with a sample and take intrusive measurements enables a more comprehensive suite of analytical techniques to be used for characterisation. In addition to this, sample retrieval allows the monitoring of areas with difficult geometries and high levels of interference where the application of conventional non-contact *in-situ* counting methods would not allow this.

In order for analysis by sampling to be accurate, any sources of error and bias have to be well known, quantified where possible and controlled effectively. Dry swabbing is used widely in the characterisation of radioactive facilities, and past research has shown the relatively poor repeatability that has been achieved using this approach [81]. As the limited repeatability of this method places limitations on the estimates that can be made from characterisation using dry swabbing, efforts to increase the repeatability of swabbing remain important. Current research has demonstrated that repeatability of force-application and other swabbing inputs can positively impact the repeatability of swab sampling [83].

Replacing human operators with robot agents in the sample retrieval process offers the opportunity to reduce potential sources for error. Humans are assumed to apply a constant force and pressure over a certain area during the swabbing process. One investigation postulated that the force input from human operators varies greatly from swab to swab, the area covered is not constant and the pressure applied is not even [60]. Work that could quantify the input from human operators would provide a greater understanding of the sources of error in the swabbing process.

Current sample retrieval robots primarily use manipulators with jaw grippers to collect larger items which are not suitable for many chemical analysis approaches. These systems would have to be modified so that they could perform swabbing tasks to allow for the retrieval of particulate samples.

#### Sample Processing

Sample processing is often required to ensure that the retrieved sample is suitable for the chosen analytical technique(s). Minimising the processing tasks required is essential in ensuring that sensing is feasible, due to the size constraints on most robotic platforms. Further to this, waste is commonly generated through preparation techniques such as dissolution and pH adjustment. As waste generated in the nuclear industry can

#### 6.2. INTRODUCTION

have large cost implications [112], minimising preparation requirements has additional significance.

#### Sample Analysis

Characterisation of radioactive environments is performed in order to assess risk, to optimise waste consignment, and for regulatory compliance. The primary concern of a characterisation technique is to provide an accurate representation of the environment from which it is taken. It is equally important, however, to consider the resources requirements of each analytical technique. For instance, although proven to be highly accurate, characterisation through liquid scintillation counting (LSC) [116] is highly intensive in terms of operator time and cost, which limits its utility in decommissioning scenarios [6].

#### Waste Management

Waste management is a central consideration in the characterisation process. Accurate characterisation is ultimately unviable if it generates waste which is more costly to process than the savings through characterisation.For this reason, it is crucial to assess the waste generation of a technique when considering its feasibility.

#### 6.2.3 Chrysoidine G

Colorimetric chemical sensors are a particularly promising candidate for *in situ* inspection as they potentially only require vision detection systems, which are already commonplace on robotic platforms [9] and have a very small footprint.

As an optical chemosensor, Chrysoidine G (4-phenylazo-m-phynylenediamine, alternatively CG), has been reported to complex with cobalt [103], strontium [122] and caesium [123], undergoing a colour transition that is clear to the 'naked-eye'. These metals are collectively responsible for a large quantity of radiotoxicity in spent nuclear fuel (SNF) [124]. The complexation behaviour of CG with these metals [103] makes it a promising candidate for identification and characterisation tasks in the nuclear industry.

Metal complexation of CG (Chrysoidine G), an azo dye [125], changes the absorption spectrum of CG, with a characteristic peak growth at 460nm [103] resulting in a changing the colour from yellow to dark-orange.

#### Chrysoidine G for in-situ detection

Previous work has suggested that CG could be used as the basis of a new chemical sensor for the detection of strontium and caesium in the nuclear industry [103, 122] and could provide a low-cost alternative for the sensing requirements in the characterisation of facilities. This paper assesses the viability of CG for use in the *in situ* characterisation of radioactive facilities.

As outlined in Sections 6.2.2 - 6.2.2, the development of an *in situ* sensor requires the automation of a number of processes. When considering sample types of interest, there are three main areas where CG may prove useful. Detection of strontium and caesium from effluent waste streams is required before discharge [126, 127], distributed radioactive particulates are common in characterisation scenarios [128] and the identification and quantification of strontium in soil samples in crucial in the characterisation of land [129]. Each of these sample types require different sample retrieval methods, sample preparation steps and may interact differently with a potential CG-based chemosensor. Simulant sample types analogous to liquid nuclear effluents were used in this study, as they were deemed to pose fewer challenges in sample preparation when compared to simulant solid samples.

When developing a robotic inspection platform, each stage of sample preparation may introduce new hardware requirements and may generate new waste. These considerations are non-trivial on inspection platforms with limited space to integrate hardware, and in radioactive environments where the generation of waste can have large associated costs. For this reason, this paper aims to determine the feasiblity of CG with minimal sample preparation steps.

### 6.3 Materials and Methods

The performance of CG complexation behaviour with strontium and caesium was tested using different concentrations of aqueous solutions of strontium and caesium salts (specified in Section 6.3.1). The colour response of the CG was measured using UV-Vis spectrophotometry. This method was chosen to determine whether the colour change offered by CG can be used for analytical purposes. As this paper aimed only to determine feasibility, new colour-sensing analytical equipment and a full integrated sensor was not deemed to be required. However it is recognised that if CG is identified as a feasible *in situ* sensor, the development of such supporting infrastructure to relate the CG colour change to quantifiable compositional analysis will be needed.

Metal Salt	Analyte Concentration Range (ppm)	CG Concentration (ppm)
Strontium Nitrate	0.05 - 10.0	20
Strontium Chloride	0.1 - 0.5	20
Strontium Carbonate	0.1 - 0.5	20
Caesium Nitrate	0.1 - 0.8	20
Caesium Chloride	0.1 - 0.8	20
Caesium Sulfate	0.1 - 0.8	20

Table 6.2: A table detailing the tests performed.

#### 6.3.1 Preparation of Chrysoidine and Analyte Solutions

An aqueous solution of Chrysoidine G (Analytical Standard, Sigma Aldrich, UK, used as received) was prepared in accordance with previous work (1000 ppm) [123].

Strontium and caesium metal salts were purchased (99.9% purity, Sigma Aldrich, UK, used as received) and added to  $18.2 M\Omega$  deionised water to produce 1000ppm stock solutions. Samples were drawn from these stock solutions and further diluted to chosen concentrations. The analyte concentration ranges were chosen to align with the ranges tested in previous relevant reports [103, 123, 122] and are presented in Table 6.2. The CG solutions were added to the metal salt solutions before the solution containers were shook by hand in order to ensure proper mixing of solutions, with no additional adjustment of pH or changing of chemical conditions, in order to reflect the likely conditions for the application of this chemosensor. The concentrations CG shown in Table 6.2 reflect the concentrations in the combined solutions of metal salts and CG.

#### 6.3.2 Collection of UV-Vis Spectra and pH Measurements

After the solutions were prepared, absorption spectra were recorded by a UV-Vis spectrophotometer (Analytik Jena Specord 200 plus).

The measured absorbance can be related to the concentrations of an analyte by considering the Beer-Lambert law given in Equation 6.1, where A is the absorbance which is a function of the relative measured intensity,  $\varepsilon$  is the molar absorption coefficient, c is the concentration of an analyte and l is the mean path length of light.

$$A = \log_{10}\left(\frac{I_0}{I}\right) = \varepsilon(\lambda)cl \tag{6.1}$$

The Beer-Lambert equation can be extended depending on the number of analytes

to be considered. Equation 6.2 shows the form of this law in the case where two analytes are to be considered.

$$A = \varepsilon_1(\lambda)c_1l + \varepsilon_2(\lambda)c_2l \tag{6.2}$$

The metric used in [123] to demonstrate the changing colour of solutions is used in this paper, and is given explicitly in Equation 6.3, where  $A_{460}$  is the absorbance at 460 *nm* and  $A_{380}$  is the absorbance at 380 *nm*.

$$A^{\dagger} = \frac{A_{460}}{A_{380}} \tag{6.3}$$

Solution pH was measured for the CG and strontium carbonate solutions using an S220 pH/ion meter (Mettler Toledo) as the ligand is reported to be pH sensitive [130]. pH was recorded in triplicate for each solution with the probe being cleaned thoroughly between each measurement.

## 6.4 **Results and Discussion**

Validation of the performance of CG in the detection of strontium and caesium [103, 122] was performed for each metal salt (specified in Table 6.2), with concentration ranges specified in Table 6.2. Measurements of pH were performed for the strontium carbonate tests only, with the results of this experiment presented in Figure 6.7.

UV-Vis Spectra for solutions of these metals salts and the CG indicator are presented in Figures 6.3, 6.4, 6.5, 6.6 and 6.7.

Though the locations of absorbance maxima across Figures 6.4 - 6.7 agree with previous work [103], Figure 6.3 shows broad disagreements with previous results on the trends in absorbance at maxima with respect to concentration [103, 122], no clear linear increase in peak absorbance with increasing caesium concentration was observed irrespective of the anion from all three metal salts tested.

Figure 6.3 (bottom right) presents a selected absorbance ratio indicating the colour change observed from these solutions, Equation 6.3. These caesium solutions demonstrate the disagreement with previous literature, with no linear increase in this absorbance metric with respect to caesium concentration [122]. The lack of a clear pattern in absorbance behaviour indicates that CG may not be viable to quantify caesium in these solution conditions.

Figure 6.4 presents the UV-Vis response of CG in the presence of strontium nitrate.

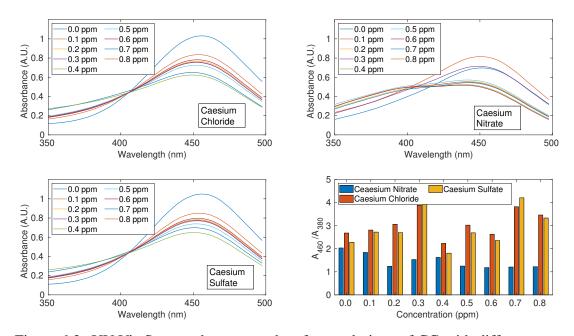


Figure 6.3: UV-Vis Spectrophotometry data from solutions of CG with different caesium salts. **Top left, Top right, Bottom left**: UV-Vis spectra each salt over the tested concentration ranges. **Bottom right**: Mean values for the ratio of absorbance of 460 *nm* to 380 *nm* light for each caesium salt. Error bars are omitted from **bottom right** as they are too small to be easily visible.

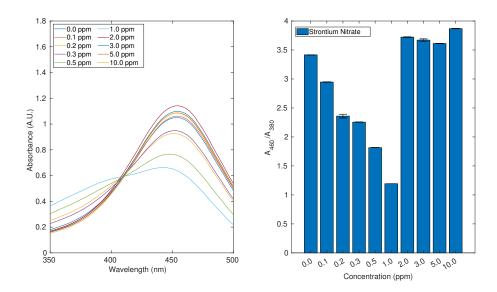


Figure 6.4: UV-Vis Spectrophotometry data from solutions of CG with different concentrations of aqueous strontium nitrate solutions. Left: UV-Vis spectra collected from these solutions. **Right**: A comparison of the ratios of absorbance at 460 *nm* to 380 *nm*. Error bars are included, though most are relatively small (< 1%) of the mean.

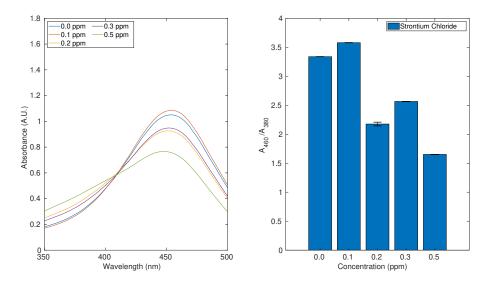


Figure 6.5: UV-Vis Spectrophotometry data from solutions of CG with different concentrations of aqueous strontium chloride solutions. Left: UV-Vis spectra collected from these solutions. **Right**: A comparison of the ratios of absorbance at 460 *nm* to 380 *nm*. Error bars are included.

These results showed a decrease in the ratio of  $A^{\dagger}$  between  $0.0 - 1.0 \, ppm$ , opposite to the trend reported previously [103]. There was an increase of this metric with respect to increasing caesium concentrations.

The reduction in  $A^{\dagger}$  shown in Figure 6.4 was repeated in Figure 6.5, though the decrease in  $A^{\dagger}$  in Figure 6.5 was not monotonic. These results do indicate complex formation, but it is unlikely that the resulting colour change can be used for strontium quantification *in situ*.

Figure 6.6 shows the collected spectra and the  $A^{\dagger}$  relationship for increasing concentrations of strontium carbonate. The observed relationship for different strontium carbonate solutions was consistent with the results for both strontium nitrate and strontium chloride. When comparing these results to that of previous work, the response of CG could be considered to be more sensitive to solution pH than to strontium concentration. To illustrate this point, Figure 6.7 is presented. Figure 6.7 (right) shows a strong correlation between pH and solution colour. The requirement for solution pH to be carefully adjusted before measurement is impractical *in situ*.

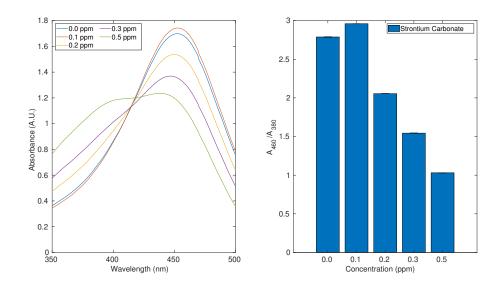


Figure 6.6: UV-Vis Spectrophotometry data from solutions of CG with different concentrations of aqueous strontium carbonate solutions. **Left**: UV-Vis spectra collected from these solutions. **Right**: A comparison of the ratios of absorbance at 460 *nm* to 380 *nm*. Error bars are included.

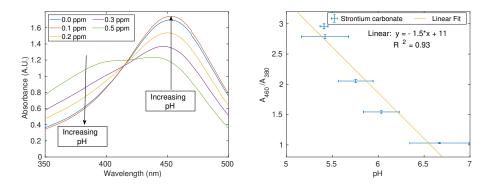


Figure 6.7: UV-Vis Spectrophotometry data from solutions of CG with different concentrations of aqueous strontium chloride solutions. Left: UV-Vis spectra collected from these solutions. **Right**: A comparison of the ratios of absorbance at 460 *nm* to 380 *nm* plotted against the pH of the different solutions. A linear fit is provided to demonstrate the correlation between the two variables.

## 6.5 Conclusions and Future Work

The results presented here suggest that Chrysoidine G (CG) is not suitable to be utilised *in situ* for the quantification of radioanalytes in the conditions tested, without prior pH balancing. The strong correlation between the colorimetric response and pH means that significant development must address the necessary sample preparation requirements before CG can be used as part of an *in situ* sensor. Ideally sensors should not necessitate extensive sample preparation, and minimise the amounts of waste generated. Appropriate molecular design, synthesis and testing of dye molecules needs to be undertaken to deliver the appropriate changes in absorbance upon complexation that can be used to quantify strontium and/or caesium content without being adversely affected by extrinsic factors, like pH, that may be encountered in sampling environments.

## **Chapter 7**

## Conclusions

This project made advancements which are aimed at increasing capability and understanding in the characterisation of radioactive environments. The work presented in this thesis provides insights in to the swabbing process, a common sample retrieval process used widely in the nuclear industry as well as many other areas (see Chapter 3). The primary contributions are made in the area of sample retrieval, while work was also done to investigate potential analysis techniques which may be implemented on robotic platforms in the coming years.

This chapter includes a chapter-by-chapter summary of the contributions and implications of the work presented in this thesis, followed by a broader overview of the progress made in this thesis and the feasible next steps required to advance this work. This chapter in concluded by a list of the publications resulting from the work presented in this thesis.

## 7.1 Findings and Conclusions: Chapter by Chapter

This section details the contributions made in each chapter of this thesis. The introduction and this chapter are excluded from this as no contributions are presented primarily in these chapters.

#### 7.1.1 Inspection Robotics

Chapter 2 presents a literature review which gives an historical overview of the development of robotics in the nuclear industry. While this topic has been covered by other authors [9], the review presented in this thesis adds significant discussion on current work, while other work [9] ceases covering advances after the initial response to the incident at Fukushima-Daiichi nuclear power plant.

Perspectives from the nuclear industry are complemented in Chapter 2 by a summary of inspection robotics development in a broad range of other areas. This work shows that autonomous navigation, visual feedback and other 'non-contact' analyses are performed fairly routinely. There is however a distinct lack of sample retrieval capability found in inspection robotics.

The second half of Chapter 2 focuses on the platforms which have been developed to retrieve samples in inspection and characterisation tasks. There is a highlighted lack of platforms which have been developed to perform the retrieval of loose, solid (often particulate) sample types. This problem is of great significance in the nuclear industry, where the location and quantity of solid radioactive contaminants must be recorded. The best example of solid sample retrieval capability which can be adapted for the nuclear industry comes from space exploration missions. Sample return missions are relatively commonplace, with some more recent platforms (Beagle 2 and Curiosity) retrieving samples for *in situ* analysis. *In situ* analysis would be greatly advantageous in the nuclear industry as it allows for characterisation of facilities without the need to transfer potentially harmful samples around site. The capability of the Curiosity rover can act as a standard which the nuclear industry should aim for.

The literature presented in Chapter 2 indicate a clear direction for future characterisation robot development, and it is hoped that this chapter can inform those in the nuclear robotics field.

The key findings from this chapter can be summarised as follows:

- The use of robotic platforms in the nuclear industry is long-established due to radiological hazards, making human operation in many environments infeasible.
- Mobile robots which are able to perform sample retrieval tasks and *in situ* analytical techniques could provide a step-change in the way we characterise radioactive facilities. Robotic platforms developed for space exploration, and inspection in other areas demonstrate that existing technology could be used to develop characterisation robots in the nuclear industry. There is however, a gap in the appreciation of sampling uncertainties in current research on sampling robots.

#### 7.1.2 Sampling and Swabbing

Chapter 3 presents a literature review which details the swabbing process as well as presenting various aspects of sampling. There are few studies which have focused on swabbing; this chapter analyses this small body of work, providing insights as to the current understanding of this technique. It is clear from the analysis in Chapter 3 that the errors associated with the swabbing process are large and there has been no effort to reduce or quantify these errors in any systematic way.

In order to provide a mechanistic understanding of the swabbing process, available literature on particle adhesion is presented in Chapter 3. It is clear that variations in surface and contaminant conditions contribute largely to swabbing uncertainty, with different conditions leading to variation in the level of adhesion. Swabbing is a dynamic process which involves both the removal and re-deposition of contaminants from/to a surface. It is clear that there is a lack of understanding of mechanisms which are important influencing factors in contaminant removal during swabbing, and more work is required to understand these mechanisms.

Chapter 3 presents a summary of errors which are pertinent in the swabbing process, taken from Gy's Theory of Sampling [67]. These error sources are important to consider when analysing the swabbing process, though the Theory of Sampling is unlikely to provide any quantitative utility [91].

Chapter 3 provides a theoretical underpinning for the work done in this thesis, drawing from various different areas. It is evident, however, that there is much more work required to establish an understanding which gives a mechanistic understanding of the swabbing process, a better quantitative understanding of the errors associated with this process and finally to provide a framework which can be used to inform the design of swabbing protocols and future swabbing systems.

The key findings from this chapter can be summarised as follows:

- Addressing uncertainties in sampling processes is of crucial importance, and there is a need to further efforts in this area, to develop a robust quantitative framework which can inform sampling plans.
- Swabbing is widely used in the nuclear industry, as well as many other areas, to aid characterisation. It is necessary to develop a greater understanding of the swabbing process so that characterisation can be made more accurate.

#### 7.1.3 Swabbing Inputs and their effect on Pick-Up Factor

A major contribution of this thesis is the work conducted in Chapter 4; a systematic analysis of the impact of different swabbing inputs on the swabbing process. This work determines the effect of changing swabbing force and force application area on the pick-up factor for loose particulate contamination. This work furthers our understanding of the error contributors in swabbing tasks, and is also valuable for the development of swabbing robots.

It was found that having the ability to control swabbing force impacts the efficacy and repeatability of pick-up in the experiments in Chapter 4. In these experiments there was an optimum force range where pick-up factor was increased, though the effect on repeatability was far-less pronounced. Though it is clear that swabbing force does contribute to swabbing performance, the results in Chapter 4 suggest that this factor is not the sole contributor to swabbing uncertainty. Even with highly repeatable swabbing inputs, there were still large ranges of pick-up factors observed in these experiments. There is still a large amount of work required to fully understand the impact of all of the major error contributors in the swabbing process.

Chapter 4 also demonstrated the effects of force application area on the swabbing process. The results presented show that the pressure profile (the way force is applied to a swab) can greatly influence the efficacy and repeatability of swabbing. This area should be more widely explored in future as this may enable greater control of swab efficacy.

Leading edge accumulation is evident in the swabbing experiments presented in Chapter 4, and this mechanism is important in the swabbing process. the tendency for a large proportion of contaminants to be collected around the leading edge of force application can reduce over all swab efficacy, with less of the swab area able to collect contamination. This mechanism can potentially be used, with an appropriate swabbing pressure profile, to control swabbing efficacy more accurately.

- Addressing uncertainties in sampling processes is of crucial importance, and there is a need to further efforts in this area, to develop a robust quantitative framework which can inform sampling plans.
- Swabbing is widely used in the nuclear industry, as well as many other areas, to aid characterisation. It is necessary to develop a greater understanding of the swabbing process so that characterisation can be made more accurate.

Measurement	Value	Associated Error
Target Area Coverage	$130 \ cm^2$	N/A
Mean Area Coverage	$191.84 \ cm^2$	17.82%
Mean Force Application	4.09 N	60.65%

Table 7.1: A table summarising some of the key performance metrics presented in Chapter 5.

#### 7.1.4 Analysing Human Performance in Swabbing Tasks

A lack of repeatability in human input during swabbing tasks is thought widely [99] to be a major contributor to swabbing uncertainty. Though this thought is often posited in previous literature [61], there have been no attempts to measure human performance in these tasks. Chapter 5 provides the first measurements of humans in swabbing tasks.

The study detailed in Chapter 5 collected force sensor data, combined with spatial tracking data, from 21 participants each performing 30 double-pass swabbing tests. The results of this study allow us to begin to quantify human performance and repeatability during swabbing. Table 7.1 presents a summary of the average performance of the whole sample.

The results for area coverage and force application indicate that human repeatability in this process is low, though it is not possible at this time to make any definitive estimations as to how the repeatability in force application and area coverage relates to swabbing performance. Continuity of force application is found to be extremely low, with an average coefficient of variation of approximately 60% of the mean force application. When comparing this value to the results in Chapter 4, it is evident that inconsistent force application from operators would have an appreciable impact on swabbing performance.

- Force application and area coverage for the human participants were both found to have low repeatability and this would likely be detrimental to swabbing repeatability.
- Force application was seen to be considerably more variable than application area in this study. The repeatability achieved for force application would be far less than would be expected for a robotic system. This implies that robotic systems may be used to reduce swabbing uncertainty.

## 7.1.5 A Feasibility Study on the use of Chrysoidine G for the *in situ* detection of Common Fission Products in the Nuclear Industry

Chapter 6 details work done to assess the viability of Chrysoidine G for use in *in situ* inspection tasks. It was thought that optical chemosensors such as Chrysoidine G are well-suited for integration with inspection robotics due to their ease of use and unso-phisticated sensing requirements; optical chemosensors can be analysed using cameras which are commonly utilised by inspection robots. The work in Chapter 6 ultimately demonstrates that the use of Chrysoidine G in this project was unfeasible, with the sensor's performance greatly affected by chemical changes which would be difficult to control *in situ*.

The results presented in Chapter 6 show the UV-Vis spectra of Chrysoidine G when in solution with a number of different strontium and caesium salts. This work attempted to replicate the likely use-case for this sensor, with no control of pH or other solution conditions permitted. CG's response to the caesium salts tested was found to result in no colour change, which was in disagreement with previous literature [122]. Though a colour change was observed when CG was in solution with different strontium salts, the observed colour change was not in agreement with previous literature [103], and this was not able to be used to quantify the presence of strontium in solution.

The results in Chapter 6 show that CG's colour change is pH-dependent, with a colour response  $(A_{460}/A_{380})$  that was found to be well-approximated using a linear fit. This finding highlights the need for pH adjustment to any solution before CG can be used for analytical purposes. The requirement for additional sample preparation was considered to be impractical using current inspection robotics, with the additional consideration that any additional chemical processing would generate additional waste. Further to this, pH adjustment would dilute the solution, adding an additional complication in analysing CG solutions.

- Chrysoidine G was not found to be suitable for the use-case of *in situ* robotic characterisation. further work is required to develop sensors using CG, or determining other potential candidate sensing technologies.
- CG's colour change was strongly affected by pH. Potential sensing technologies for *in situ* characterisation are required to be more robust to chemical changes, so that the requirements for sample preparation (and the requirements for extra hardware and subsequent disposal of all waste products) is limited.

### 7.2 Overview of Progress and Further Work

This thesis presents work that has made significant progress in furthering our understanding of the swabbing process, with a further contribution towards the development of chemical sensing technology which may be used on robotic platforms. This work will help in the assessment of contamination, and has particular pertinence in the nuclear industry. It is hoped that the work in this thesis can stimulate further research in to the swabbing process so that it can be understood more thoroughly.

The following items have been highlighted by the results and discussion in this thesis as salient areas where further investigation is required or would be advantageous:

- The Development of Swabbing Robots There is a lack of capability for robots to retrieve loose samples from solid surfaces, and this is important in the assessment of contamination, particularly in the nuclear industry. The implementation of swabbing on robotic platforms would prove an important step in the 'roboti-fication' of the characterisation of radioactive facilities.
- Furthering Understanding of Swabbing Mechanisms Although efforts to quantify error contributors in the swabbing process are important, the ultimate purpose of this work is to more accurately control and predict swab efficacy. A promising method to achieve this aim relies on understanding mechanisms (such as leading edge accumulation) and controlling the relevant swabbing inputs so that these mechanisms can be manipulated. First efforts to implement this work could control the swabbing pressure profile to maximise the efficacy of swabbing, and in doing so increase the accuracy and repeatability of this process.
- Understanding the Effects of Surface/Contaminant Conditions on the Swabbing Process - The swabbing experiments conducted in this thesis all used a large particulate contaminant on a smooth surface. While this was useful in providing repeatable surface conditions, this is unlikely to match application conditions in every instance. In order to provide an understanding of swabbing that is most useful in the 'real world' there needs to be an effort to test different surface and contaminant conditions.
- Understanding Swabbing as a Dynamic Process Swabbing is a process that involves both the retrieval and the redeposition of contaminants. A limitation of the work in this thesis is that the understanding of how swab efficiency changes

throughout the swabbing process is not explored. Future work should explore this area.

- Furthering Efforts to measure Human Performance in Swabbing Tasks -A primary limitation of the human participant study presented in this thesis is the quality of the force data collected. Future work should utilise force sensors which can provide greater spatial resolution so that typical human pressure profiles can be analysed.
- Developing Chemosensors which can be integrated on to Robotic Platforms - Chapter 6 highlights the difficulties in implementing chemical sensing technology on robotic platforms. Future work should aim to develop sensors which can be more easily integrated on to inspection and characterisation platforms. This is particularly important in the context of nuclear decommissioning, where the generation of additional waste, or the requirement for additional hardware, has long-term implications.
- Furthering work with Chrysoidine G Although Chrysoidine G was found to be unsuitable for this project, there is still evidence for complexation with certain metal cations. Future work should further our understanding of the behaviour of Chrysoidine and consider whether this indicator can be optimised by some structural alteration.

## 7.3 The Development of Swabbing and Swabbing Robots

This section is included to provide insight as to where the developments made in this thesis (particularly the understanding gained from the experiments in Chapters 4 and 5) will be applied, and to show the impact of this work. While parts of this section are speculative, it is intended to provide a greater appreciation for the impact of this work.

This project was conceived to address a disconnect between robotic development and analytical science (developments in the characterisation process). It was seen that robotic development in the nuclear industry had largely avoided sample retrieval and this severely limited the role that robotics could play in the characterisation of radioactive facilities.

When tasked with developing a 'swabbing robot', a roboticist would address this as a control problem, though in the case of swabbing it became evident that there were no clear parameters that were known to be important in this control problem. Chapter 4 attempts to address this issue. From this work it is clear that treating swabbing as a force-control problem has merit. In addition to this, it has become clear that existing swabbing protocols (continuous force application through the entire swabbing motion) limit swab efficacy and repeatability. This work has prompted the development of a robotic system which can change swabbing pressure-profile, apply force in a discontinuous manner while still retaining a high degree of repeatability. Future work will determine whether this system can provide a step-change in the way that swabbing is conducted in practice.

The work presented in Chapter 5 provides evidence to test the common assertion that human operators are a major contributor to swabbing uncertainty [60, 81]. The force application performance (mean application 4.09 N with a C.O.V. of 60.7 %) is a clear indication that human operators have low repeatability in these tasks. The mean force application is low when compared to the values tested with the A.S.S. in Chapter 4, with swabbing efficacy likely adversely impacted by this low force application. The large C.O.V. observed for force application of the participants is far larger than the variation in forces applied from the A.S.S. or could reasonably be expected from a robotic system. This result has promising implications for the potential performance of swabbing robots.

Participant area coverage was observed to have a C.O.V. of 21.3 % which again highlights the poor repeatability of human operators. This result reinforces the need the involvement of robotic systems in the characterisation process. In order to determine the implications of poor force application repeatability on swabbing performance, more work is required to determine swab efficiency behaviour during swabbing.

It is also noteworthy that the results from this chapter have wider implications for future practices in swabbing. It is highly likely that human operators remain responsible for the collection of swabs in instances were automated solutions are not appropriate. The results presented in this chapter provide a benchmark from which can inform estimates of swabbing uncertainty. Further to this, the methods proposed in this chapter may be adapted and tailored to specific swabbing scenarios so that more robust assessments for swabbing performance can be made. This would be useful in ensuring that characterisation is made more accurate and these assessments can be used to inform the development of swabbing procedures.

The work presented in Chapters 4 and 5 are foundational in addressing swabbing uncertainty in a systematic way, and this work has implications for best-practice in swabbing as well as informing the development of swabbing robots. The work from these chapters has led to the creation of a follow-on project as part of the Robotics for Nuclear Environments project grant. This work has also led to the creation of an EU MCSA Doctoral Network proposal, RAICAM, focused specifically on autonomous sample retrieval in hazardous environments.

### 7.4 The Development of Autonomous Characterisation Robots

While swabbing forms a major component of this thesis, there are many more areas where contributions can be made to improve the characterisation process in radioactive environments. As was highlighted in Chapter 2, there is an aim to perform more characterisation techniques *in situ*, as this would be advantageous in terms of safety, cost and time. The development of sample retrieval capability will help with this, as it will enable for more sample types to be handled, which imposes fewer sample preparation requirements, however there is still a great need to develop characterisation techniques which can be deployed on robotic systems. Particularly in radioactive environments, there is a need for analysis techniques that are cheap and facile. This need arises as any equipment deployed in to radioactive environments may become contaminated, and/or may have a short operational lifetime before it has to be disposed of. Aside from wanting to minimise the amount of waste produced, there are massive cost incentives to reduce the burden for waste disposal in the nuclear industry. For these reasons, this thesis aimed to determine the feasibility of a potential candidate chemical sensing technology for use *in situ*.

### 7.5 Final Remarks

The purpose of this work was to address issues which are pertinent in the context of nuclear decommissioning. Nuclear power exists as a strong candidate to form a large portion of global energy generation through the coming decades [115], though the challenges facing wider acceptance of the nuclear industry are based around the long-term cost and safety implications for nuclear decommissioning, waste management and the responses to accidents [131]. Alongside these issues of perception, there is a real need to ensure that the costs of nuclear decommissioning and waste management can be reduced with new technological development [132]. It can be contended that

### 7.5. FINAL REMARKS

better characterisation is the key to ensuring safety, reducing costs and reducing the time taken across many areas in the nuclear industry.

This work addressed the need for greater characterisation capability for the robotic platforms which were being developed at the time of this project's commencement. The role of robotic systems for characterisation has taken on renewed importance in recent months, with the announcement of accelerated decommissioning schedules for the United Kingdom's fleet of Magnox reactors [132]. With the pursuit of accelerated decommissioning programmes, there is a greater need for robotic systems to enter highly radioactive areas to characterise and inform decommissioning plans, as these areas would have previously been sealed for long time periods before any decommissioning activities were undertaken.

Results presented in this thesis have shown that the use of automated swabbing systems can typically reduce the interquartile range for pick-up factor to around  $\pm 5\%$ , and though direct comparison to previous literature using human operators is challenging, this still appears to be a far greater repeatability than the best reported performance of  $\pm 10\%$ . These results highlight the importance of developing automated systems to swab as well as providing further evidence to the assumption that human variability is a major cause for the variability in swabbing performance in previous reported literature.

The deployment of robotic characterisation platforms is becoming more commonplace, and activities such as the deployment of the Lyra robot at Dounreay [100] are illustrating the cost and time savings that can be achieved through the deployment of robotic characterisation platforms. This project has targeted the development of sample retrieval capability, with swabbing being used widely in the characterisation of nuclear power plants. The superior repeatability achievable from robotic platforms provides a unique opportunity to both increase the accuracy of characterisation through swabbing as well as removing the radiological risks posed to operators who are deployed in characterisation environments.

The limited existing understanding of swabbing was highlighted and addressed in this work. The large uncertainties associated with swabbing and limited understanding of the contributors to these uncertainties has meant that estimates informed by swabs were forced to be largely conservative, with this conservatism driving up the costs and time taken to decommission facilities. It is hoped that the results presented here can be used to inform, change practices where appropriate and ultimately help to secure the role of nuclear power in the low-carbon energy sector of the coming decades. Through this project, a number of issues with practice in the nuclear industry have been raised. The first of these issues would be the use of estimated pick-up factors where these estimates are not based on a statistically robust sample of 'similar' surface profiles. Though established practices advocate for the use of a 'site fingerprint' where no better estimate can be found, the work presented in this thesis strongly disagrees with this approach. Further, the results presented here strongly suggest there is a need to augment human capabilities to conduct swabbing. The results presented in Chapter5 strongly suggest that human performance in force application and area coverage is likely to have significant impact on swabbing performance. To overcome this issue, the author would recommend the development of automated systems to swab, and/or the use of aids to help operators to provide consistent force application and area coverage. Through adopting these changes, there is an opportunity to increase the accuracy of characterisation, and thus drive greater safety and lower costs in nuclear decommissioning.

This project has also highlighted a number of methodological improvements which could be made to improve the accuracy and efficacy of current swabbing practices. The first is to utilise swabbing protocols which work to remove contamination more repeatedly, using more complex motions than a consistent single-pass swab, and developing methods which allow for better contaminant removal. Secondly, there is scope to increase the use of wetting agents which are not deionised water. Commonly, the use of deionised water to increase swab efficacy can have negative effects on swab efficacy due to the introduced chemical interactions of a polar liquid. Finally, there is scope to select force application areas which maximise pick-up. It is evident from the work presented in this thesis that contaminant pick-up is focused close to the leading-edge of force application, thus manipulating this leading-edge can positively impact swab efficacy and consequently repeatability can be improved.

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- [132] NDA, "Nuclear decommissioning authority strategy," tech. rep., The Nuclear Decommissioning Authority, 2021.

## **Appendix A**

# **Publications**

The work presented in this thesis has been used as the basis for a number of publications. The author's publications so far include:

- T. Johnson, C.A. Sharrad, S. Watson and T. Carey, "Investigating the Effect of Force Input on the Pick-Up Factor of Loose Contamination", *Waste Management Symposium*, 2021.
- T. Johnson, C.A. Sharrad, S. Watson and T. Carey, "Analysing Human Operators and and Automated Swabbing System in Swabbing Tasks", *ANS, Decommissioning Environmental Science and Remote Technology*, 2021.
- T. Johnson, S. Zhou, W. Cheah, W. Mansell, R. Young and S. Watson, "Implementation of a Perceptual Controller for an Inverted Pendulum Robot", *Journal of Intelligent and Robotic Systems*, 2020, pp. 683-692, 99(3-4).

There are also a number of publications which are still in the process of being published. There is an indication (in bold) indicating the stage of each potential publication. These include:

- T. Johnson, C.A. Sharrad, S. Watson and T. Carey, "Swabbing Inputs and their effect on Pick-Up Factor", *Journal of Radiological Protection*, Advanced Draft.
- T. Johnson, C.A. Sharrad, S. Watson and T. Carey, "Analysing Human Performance in Swabbing Tasks", *Journal of Radiological Protection*, Advanced Draft.

- T. Johnson, C.A. Sharrad, S. Watson and T. Carey, "A Feasibility Study on the use of Chrysoidine G for the *in situ* detection of Common Fission Products in the Nuclear Industry", *Waste Management Symposium 2023*, Under Review.
- D. Barton, T. Johnson, A. Callow, T. Carey, S. Bibby, S. Watson, C. Sharrad, "A Review of Contamination of Metallic Surfaces within Aqueous Nuclear Waste Streams", *Progress in Nuclear Energy*, Under Review.

# **Appendix B**

# **Continuing Research**

This project has directly led to a number of follow-on projects:

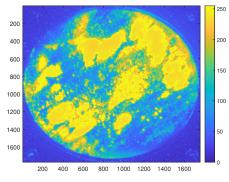
- A 15-month PDRA position on the EPSRC RNE Project.
- An EU MCSA Doctoral Network grant, RAICAM, focused on autonomous robotic sampling in hazardous environments.
- An application to the EPSRC Doctoral Prize Fellowship Scheme, titled "Analysing Swabbing to Improve the Accuracy of Beryllium Characterisation in the Nuclear Fusion Industry".

It is hoped that research continues in this area, and that autonomous robotic swabbing is realised in the 'real-world'. This research promises a step-change in how we are able to characterise radioactive environments. Accurate characterisation is essential in minimising the waste burden of the nuclear industry, which is an important component of ensuring the viability of nuclear energy as a feasible source of power in to the future.

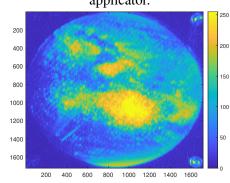
# **Appendix C**

# **Swab Contamination Maps**

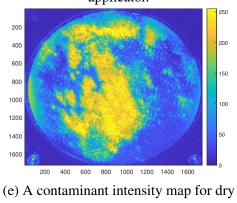
Chapter 4 presents a number of contamination maps for swabs and swabbing beds, providing a novel insight in to the swabbing process. Many of the images collected are not presented in the main body, and are presented here. Figure C.1 shows images from the swabs taken in 6 separate swabbing experiments. These maps demonstrate the differences in observed swab contamination during both wet and dry swabbing.



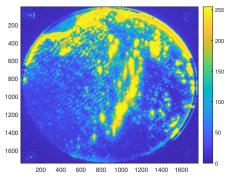
(a) A contaminant intensity map for dry swabbing with a 55 *mm* circular force applicator.



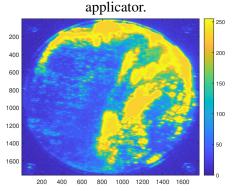
(c) A contaminant intensity map for dry swabbing with a 50 *mm* circular force applicator.



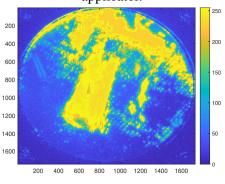
swabbing with a 35 *mm* circular force applicator.



(b) A contaminant intensity map for wet swabbing with a 55 *mm* circular force



(d) A contaminant intensity map for wet swabbing with a 50 mm circular force applicator.



(f) A contaminant intensity map for wet swabbing with a 35 *mm* circular force applicator.

Figure C.1: Contaminant intensity maps for dry and wet swabbing with different force applicators.

# **Appendix D**

# Swabbing Performance Study Analyses

Chapter 5 presented results from a human participant study, where force application, area coverage and swabbing time were measured. This appendix presents a number of correlation graphs, with none showing any compelling correlation.

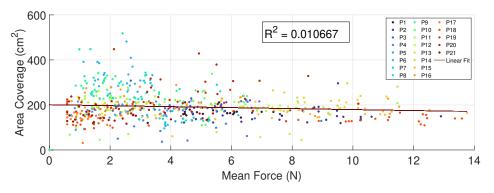


Figure D.1: A graph showing the relation between area coverage and mean force in this study.

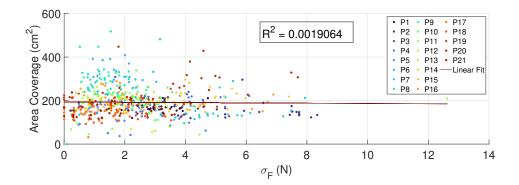


Figure D.2: A graph showing the relation between area coverage and force application consistency in this study.

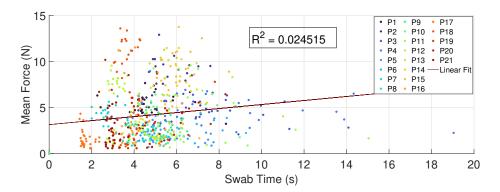


Figure D.3: A graph showing the relation between mean force application and swabbing time in this study.

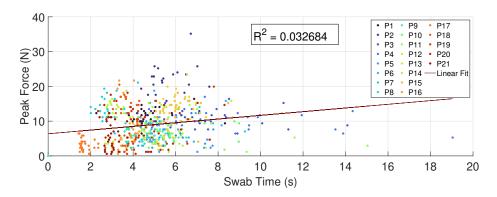


Figure D.4: A graph showing the relation between maximum force application and swabbing time in this study.

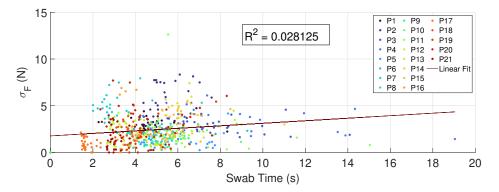


Figure D.5: A graph showing the relation between consistency of force application and time in this study.

# **Appendix E**

# **Ethics Review Material**

This chapter includes all of the relevant supplementary material from the ethical review process undertaken for the study detailed in chapter 5.

### Investigating the Performance of Human Operators in Swabbing Tasks

#### 0. Proposal name

#### 0. Enter the proposal name

Investigating the Performance of Human Operators in Swabbing Tasks

#### 1. Description of the data

#### 1.1 Type of study

This study will involve participants swabbing a surface repeatedly with a specially designed puck. Spatial information and the force applied to the puck will be recorded during these swabbing tasks. This study will be used to analyse the performance of human operators in swabbing tasks.

#### 1.2 Types of data

Quantitative force data and temporally resolved spatial tracking data.

#### 1.3 Format and scale of the data

CSV files will be collected for both the force data and the tracking data. The total scale of the data collected will be <<1TB.

### 2. Data collection / generation

#### 2.1 Methodologies for data collection / generation

Spatial tracking data will be collected using a Vicon Tracking. This system has been used in previously published work and is considered as an acceptable ground-truth for tracking data in robotics.

Force data will be collected using a capacitative force sensor and will be transferred to a computer using the I2C protocol. This should allow for force data to be collected at a high frequency with limited information loss during tracking.

#### 2.2 Data quality and standards

The Vicon tracking system has its own calibration method which can be operated using the Vicon software suite. This will be done before every participant begins the study. The Vicon system produces an indication of uncertainty while tracking and this can be checked to ensure the tracking system is accurate during operation.

The force sensors used have been calibrated before purchase, however only raw data will be collected during this study as absolute force values are not important. Each force sensor will undergo calibration against a mass-balance to ensure that the sensor is operating lineraly.

#### 3. Data management, documentation and curation

#### 3.1 Managing, storing and curating data

Data will be collected on a computer and will be saved directly to my university DropBox account. The data collected will be in CSV format so it can be managed and curated using Microsoft Excel or Matlab. This data will be kept on the university DropBox until the completion of my PhD. This dataset may be placed in the digital appendices for my thesis.

#### 3.2 Metadata standards and data documentation

Tracking data will be accompanied with Vicon's accuracy of tracking data.

#### 3.3 Data preservation strategy and standards

This data will be stored until the end of my PhD. Beyond this time this dataset will be placed in my digital appendices for my thesis. This dataset may also accompany any publications that result from this work.

### 4. Data security and confidentiality of potentially disclosive information

#### 4.1 Formal information/data security standards

None applicable.

#### 4.2 Main risks to data security

If the spatial tracking data is used, it may be possible to make some conclusions about the motion of people during swabbing tasks. The level of risk from people using this information is negligible, but it is still important that participants cannot be identified through this study. For this reason, the identity given to each participant to ensure that data from each participant does not get mixed, will not be shared with anyone other than the principal investigator.

#### 5. Data sharing and access

#### 5.1 Suitability for sharing

This data is suitable for sharing as it contains no personal information about participants. This data is wholly anonymised and so the force and spatial tracking data could potentially be shared.

#### 5.2 Discovery by potential users of the research/innovation data

This dataset could accompany any publications that result from this study. This dataset will be given in the digital appendices of my thesis.

#### 5.3 Governance of access

The principal investigator will decide whether to share this information with interested parties.

#### 5.4 The study team's exclusive use of the data

This data will be available for sharing at the culmination of my PhD.

#### 5.5 Restrictions or delays to sharing, with planned actions to limit such restrictions

None applicable.

#### 5.6 Regulation of responsibilities of users

None applicable

### 6. Responsibilities

#### 6. Responsibilities

University of Manchester Research IT.

### 7. Relevant policies

#### 7. Relevant institutional, departmental or study policies on data sharing and data security

Policy	URL or Reference
Data Management Policy & Procedures	http://documents.manchester.ac.uk/DocuInfo.aspx?DocID=33802
Data Security Policy	https://documents.manchester.ac.uk/DocuInfo.aspx?DocID=6525
Data Sharing Policy	https://www.library.manchester.ac.uk/using-the-library/staff/research/research-data-management/sharing/index.htm
Institutional Information Policy	
Other	
Other	

### 8. Author and contact details

8. Author of this Data Management Plan (Name) and, if different to that of the Principal Investigator, their telephone & email contact details

Thomas Johnson, thomas.johnson-2@manchester.ac.uk

Analysing the Performance of Human Operators in Swabbing Tasks - Participant Consent Form

### Overview

This study is being conducted to obtain an understanding of how humans perform in the swabbing of surfaces to analyse radiological contamination. This study is part of a PhD student project at the University of Manchester to better understand the swabbing process so that we can characterise radioactive environments more accurately.

You have been asked to take part in this study so that we can analyse the force profiles and swabbing area that you produce during swabbing tasks. This will enable us to see how repeatable this process is and to determine how this varies across different participants.

Should you agree to participate, it should take less than 30 minutes of your time. You will be asked to swab a surface 20 times and we will collect the force information and spatial information from these swabs. The data collected in this study will be limited to force data and tracking data from the puck. You will not be identifiable in any of this data.

The data collected will be used with similar data from other participants to determine trends in force profile and area recreation. This data will be used in this PhD project and may be published in journals or conferences in the future.

Throughout the study, you are free to ask for a break or to stop at any time.

This form will be scanned and kept in a password protected DropBox folder within 3 hours of signing. This paper copy will be shredded after it is scanned. The scanned copy of this virtual consent form will be deleted within one year of it being signed.

Consent

I am of a working age (16-75). 🛛

I do not know of any impediment that would affect my performance during swabbing.

Participant Signature:

Date: \_\_\_\_\_

### Participant Information Sheet - Investigating Human Performance in Swabbing Tasks

It is important for scientists to know the types and quantities of materials present in certain environments, and for this we often take samples by swabbing our environment. In order for us to make good judgements about the environments we sample, we need to understand how our swabs relate to the environment. This study aims to give us a better picture of how humans perform while swabbing. If we can know the forces used and areas covered by our human operators more accurately, we can make better estimates.

If you choose to be part of this study, you would be expected to spend 30 minutes performing swabbing tasks, while we measure the force of your swabbing and the positions your swab covers. This study is planned to take place in our lab in the Sackville Street Building, University of Manchester, so you would be expected to make your own way to us.

Below are a few key points that may answer some questions you have:

- Your participation is always voluntary and you may withdraw from the study at any time.
- In order to participate you need to have no pre-diagnosed problems that may affect your ability to swab. Conditions that would exclude you would include shoulder mobility issues, hand and finger issues and arthritis in the hands of arms.
- No health risks have been identified during the risk assessment review process.
- Your data will be anonymised so your personal information will not be shared with anyone. The only data we will store will be force sensor data and swab tracking data.

If you decide to participate in this study, it would be a great help to our research. You will be playing a part in helping us to survey environments better.

Please contact us if you have any questions or would like to volunteer.

Tom Johnson,

PhD Student, University of Manchester, Robotics for Extreme Environments Group.

Email: thomas.johnson-2@manchester.ac.uk

### Data Collection Protocol

This study will require the collection and storage of both spatial and force measurements. Force measurements will be taken from four "SingleTact" capacitive force sensors. Spatial tracking data will be taken from a Vicon tracking system which consists of 12 IR tracking cameras mounted around the perimeter of an aluminium extrusion scaffold. A 2D diagram of the experimental set-up is shown below.

SingleTact sensors communicate with the Arduino Microcontroller via the I2C communication protocol. A computer programme has been written to allow for the force data from these sensors to be stored in a CSV at a set frequency. CSVs will follow the naming convention:

### "FS1-A-001"

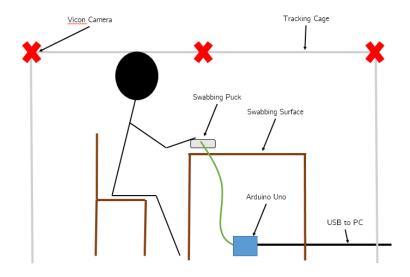
Where "FS1" designates the force sensor identifier (1-4), "A" is the participant identifier (A-Z and AA-DD for the 30 participants) and "001" is the swab number for the participant. This naming convention will remove the need to segment force data across different swabs and participants and will make it easier to synchronise data with the Vicon Tracking data.

Vicon tracking data will record directly to a PC outside the tracking cage and can be exported to CSV. Tracking data will follow a similar naming convention:

### "VT-A-001"

Where "VT" designates Vicon tracking data. The recording of tracking data will be stopped and exported after each swab to reduce the burden of segmenting tracking data.

After the collection is finished, both the tracking data and force data will be clipped to remove data entries while there is no swabbing occurring. These clipped files will be saved with the modifier "- clip" to distinguish from the raw data.



Hello,

I am a PhD student at The University of Manchester, in the Department of Electrical and Electronic Engineering. I am investigating how effective people are at swabbing surfaces when they collect samples for lab analysis, such as you would do at industrial facilities.

I would like to invite you to volunteer to take part in a study I am conducting. We are looking for participants who are able to visit our lab in the Sackville Street Building for up to 30 minutes and perform some swabbing activities whilst we record your performance.

Please find an information sheet and consent form attached, these will give you more information about the study if you are interested. If you know anyone else who works in science/engineering, feel free to pass this email on to them.

If you wish to participate, please send me an email and we can arrange a time for you to attend.

Kind regards,

Tom Johnson.

To whom it may concern,

Thank you for taking the time to review my application, and for the points you have raised. The application has been amended in light of your comments. Below are the responses to each comment:

C1 "C3.1 talks of a non CE medical device, you therefore need to tick the appropriate box here and answer the supplementary questions. Please note, the use of non CE medical devices requires consultation through the clinical trials team and therefore it may take slightly longer to review your revisions"

Response: The response to question C1 is given as "The physical testing of participants" and I believe this best describes the trial. I have to tick one of the boxes in C3.1, and am unable to say that I will not be using medical devices. In the trial I will be using external sensors, some of which are not CE marked. Therefore I chose the non CE marked option.

# C2.8 "Please describe the procedures to be undertaken Have any non-human and proof of concept trials been carried out and published?"

Response: The answer to C2.8 has been appended with "Non-human trials using the force sensors have been conducted in work that has been accepted for publication. 'Analysing Human Operators and an Automated System in Swabbing Tasks', ANS Winter Meeting 2021.

A small scale trial is planned to test the vicon tracking system with the force sensors before this study is carried out."

### C2.8 "So there is some risk just not significant risk? Please detail the risk and all the potential harm. Are any groups at higher risk?"

Response: The response to C2.8 has been changed to "These sensors are purchased commercially and connect to a computer using USB, 5V. As these sensors use such a low voltage, there is no electrical risk". The original phrasing of "negligible risk" was inaccurate. The full risk assessment for the use of the equipment in this trial is provided in L42.

C3.1 "Which of the following describes the medical device or potential medical device that you will be using? C1 says no medical device, which is it?"

Response: The "Non CE Marked" box has now been ticked. However, as mentioned previously, we do not believe any of the sensors used in this study to be medical devices.

### C3.4 "Is the intention to develop a new product and make this a commercial endeavour?"

Response: There is no intention to develop a new product or make this a commercial endeavour. The equipment has been developed solely for the purposes of this trial. This has now been explicitly stated in the response.

### D3.3 "Why are you intending to only save the data results until the completion of your PhD?"

Response: The results will now be made available permanently on Figshare. This will allow others in the University to access this data after the completion of my PhD. The DMP has now been amended to reflect this.

L15 "What is the background to your study? What has previously been published?"

Response: Swabbing is used as a standard sample retrieval mechanism in the characterisation of environments in industrial facilities. Understanding the relation between samples and the environments from which they are taken allows us to characterise more accurately. Previous studies focussed on swabbing have only used inputs from one operator. This work aims to collect data from multiple operators to give greater insight in to human performance in these tasks.

This work will ultimately contribute to the development of autonomous robotic systems used for sample retrieval in industrial facilities.

An external review of the proposed methodology was given by Dr. Paul Bremner (Bristol Robotics Laboratory), who has previously designed and run studies using human participants in the area of human-robot interaction.

Previous non-human trials have been conducted and published:

- Johnson, T., Sharrad, C., Watson, S., & Carey, T. (2021). Investigating the Effect of Force Input on the Pick-Up Factor of Loose Contamination 21093. *Waste Management Symposium*.
- Johnson, T., Sharrad, C., Watson, S., & Carey, T. (2021). Analysing Human Operators and an Automated Swabbing System in Swabbing Tasks. *ANS Winter Meeting 2022.* (Accepted for Publication)

L20.2 "You've detailed later on that you will be obtaining written consent, please clarify how this will work. will you be asking for participants to sign the form, scan it and email it to you? Will you accept a typed signature instead? Where will you store the consent forms? Will you print them off and put them in a locked cupboard or will you encrypt them and store them securely in a digital format?

Please clarify specifically how the recruitment process will work. You state later on that you will approach people directly. How will you do this? If you will be using email, how will you obtain their email addresses? Direct approach carries a risk of potential coercion so if you will be doing this, please add this as an ethical issue and provide an appropriate mitigation strategy"

Response: The response to L20.2 now reads "The project aims to measure the force input over an area during a swabbing task that is representative of a characterisation procedure in nuclear facilities.

The ultimate aim is to automate this process using robotics to increase safety and consistency.

This task is currently undertaken by graduate employees in the nuclear industry, so participants with a background in engineering or science will be recruited.

Participants will identified through sending an email to other researchers at the University through the Robotics for Extreme Environments Mailing List (80+ members). This email will encourage receivers to forward the email on to other researchers. A previous study last year found participants using the same strategy, so this email should be sufficient to attract enough participants. This email will have a participant information sheet and consent form attached. Participants can sign this form with either a scanned copy with their signature, or a typed signature, for convenience. These consent forms will be saved on an encrypted hard drive.

Participants will be asked to watch an instructional video, and then swab an area with a force sensing puck twenty times as was shown in the video.

Force data and position data will be recorded for analysis by the researcher running the study."

# L21 "Transmission of pathogens would be a risk, not an ethical issue. Please outline the ethical issues associated with this study and your mitigation for these issues. You may wish to consider confidentiality and informed consent."

Response: This answer now reads "The main ethical issues associated with this study are ensuring confidentiality of the participants, providing informed consent and avoiding the coercion of potential participants.

Efforts must be taken to pseudo anonymise all data collected during the study to ensure that no individuals can be identified from the data while the data from each participant is still distinguishable. Each participant will be given an identifier (Participant letter) which will be used to ensure that all data collected from each participant is distinguishable. The identifiers given to each participant will not be collected and so the only way this data could be de-anonymised would be if an observer of the study collects this data.

Informed consent for this study will be given primarily from the participant information sheet. The University templates have been used. Participants will also be encouraged to ask any questions that they may have on the recruitment email.

Recruitment will only be pursued by the email. No further efforts will be made to coerce people to participate in this study."

# L22 "Since signed Consent forms are to used please detail how they will be saved, and pseudo anonymised"

Response: Consent forms will be sent by participants over email. These forms will be saved on an encrypted hard drive and the email will be deleted within 7 days of being received. Data will be pseudo anonymised by giving each participant's data a unique identifier. No list will be created to link participant names to these identifiers.

### L23.3 & L23.4 Various issues with the consent forms and PIS.

Response: These forms have been redrafted using the university templates. These new forms address the points raised.

# L32 "It is a little unclear in regards to how you will contact any potential participants - do you intend to email everyone or just those of your choosing. Please ensure participation is voluntary"

Response: Recruitment will now take place through an advert sent to the research group's (Robotics for Extreme Environments Group) mailing list. This list has 80+ members and previous approved studies have been able to recruit effectively from this list. The email advert will now state that participants may forward this email on to others working in science/engineering in the University who may be interested in participation. This should avoid any potential coercion of participants.

### **Other Changes**

• The consent form now contains two extra conditions which were initially on the prescreening questionnaire. This was done to reduce the amount of documents containing personal information that need to be stored for this project. The pre-screening questionnaire can still be found in L42 and can be used if required.

Kind regards,

Tom Johnson.



# Participant Information Video

Swabbing Study – October 2021



# Why are we doing this study?

- Swabbing is an important task in assessing the condition of nuclear power plants.
- We want to understand the typical force application and area coverage for an operator.
- With data collected from multiple participants we can work out averages and get a representative view.

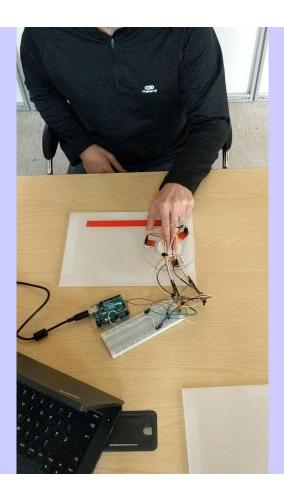


# The task

We need you to swab across a 20cm length multiple times (30) while we collect data on the force that you apply to the surface and the position of your hand:

- 1. Press around the centre of the swab with two fingers.
- 2. Apply a steady pressure as you swipe across a 20cm length.

Imagine that you are trying to collect up dust as you are swiping.





# The task

You will do the following:

- 1. 5 test runs (with the 20cm line)
- 2. 30 tests without the line.

We will monitor the position of your hand continuously. We will monitor the force you apply for each test. Please wait for the sensor to be reset between tests.

