DESIGN BRIEF FOR CDT GAS SENSOR SYSTEM

Gas Sensors Team
CDT

CDT

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Contents

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README: Highlighting used to indicate areas that need review. To interpret

YELLOW: These are specific aspects for your attention
e.g. changes in the design for 2021 compared to CDT's prototypes made in 2020

BLUE: The file repository is being prepared, and will be made available to the selected partner at a later date.

1. Introduction

CDT have, in partnership with Pace Intl, developed a prototype Gas Sensor for the detection and quantification of two key molecules in an apple store environment – 1-methylcyclopropene and ethylene. This prototype has undergone proof-of-concept (PoC) trials in apple stores during 2020. This document details operating conditions (Section 1), specifications (Section 2) and design (Section 3) of the current generation of prototypes, as well as proposed design alterations (Section 4) for the 2021 systems we are seeking quotes for. The intent of this document is to provide you with enough background information to design and build the systems for 2021 without having to start from scratch.

The two key target molecules are detected by two different sensors working in conjunction – a metal oxide sensor (Bosch SensorTec BME680/BME688) and an electrochemical sensor (Membrapor C2H4/S200). Several accommodations must be made for these sensors to operate with the required accuracy. This document details how this was achieved in the prototypes utilised in the PoC trials in 2020, as well as some suggested modifications for 2021. Where specific issues have been detected during the PoC trials, these have been added to the end of the relevant section. Please note this information is highly confidential and is subject to various patents and patent applications. It is therefore shared under the terms of the NDA between CDT and yourselves.

1.1 Using this document

A summary specification is included in Section 2. The tables included detail the specification of the system developed for the 2020 PoC trials, as well as a specification for the system to be developed in 2021.

The gas sensor system developed by CDT is outlined in Section 3. This includes specific references to the mechanical, electronic, operational and analytic systems. These details, along with an existing prototype system, will be passed to the outsource partner to allow for understanding of the current system.

In Section 4, the design brief for the 2021-onwards system is introduced, including detailed proposed changes that have arisen as a result from the PoC trials undertaken in 2020.

1.2 Introduction to partners

Introduction to CDT

Cambridge Display Technology Ltd. (CDT) is the European research and development arm of global chemicals giant Sumitomo Chemical. We are a wholly owned subsidiary of Sumitomo Chemical Co., Japan and benefit from the financial strength and market channels of our parent. Our current work spans projects from wave guide materials to water scarcity; from smart sensors to squeezed cities.

Our unique position within Sumitomo Chemical gives us the scope to combine genuine scientific curiosity with the satisfaction of creating solutions to real world problems.

- CDT encourages and promotes spontaneous and targeted innovation
- Our multifunctional teams are rewarded for individual contributions made
- We collaborate with partners in the Cambridge ecosphere and internationally, to share complementary skills and facilities

Introduction to Pace Intl

Pace International, also a wholly-owned subsidiary of Sumitomo Chemical Co., Japan, is a global leader in sustainable postharvest solutions for the fresh produce industry. In collaboration with growers, packers, and agricultural organizations, Pace develops innovative technologies that enhance, protect, and preserve fruit and vegetable quality, while reducing food waste and protecting the environment.

Through its extensive network of offices and distribution partners around the world, Pace develops and provides a comprehensive offering of organic and conventional products for use in storage and the packing line, that are designed to maintain freshness and flavour and extend shelf life of pome fruits, citrus, cherries, stone fruits, pomegranates, kiwifruits, tropical fruits, and vegetables. Pace is committed to delivering superior service and results through innovative products, leading application technologies, and unparalleled customer support, 24/7.

Pace's innovative and collaborative culture results in a team that is driven to find and develop the most sustainable food protection technologies that ensure fruit quality, extend shelf life, improve food safety, and reduce food waste and environmental impact. With a long history of partnering with technology companies, research and academic institutions, and customers, Pace is always finding new and better ways to protect the food supply, improve storage and packing efficiencies, and help meet the increasing demand for fresh produce. Through its own technology centres in North and South America, as well as access to Valent and Sumitomo Chemical's extensive global network of R&D centres and partners, Pace is at the forefront of researching and discovering the next generation of biorational decay control products, edible coatings, postharvest precision technologies, and ripening management solutions.

1.3 Apple Store Environment

After harvest, apples are typically stored in large rooms, such as the room shown in Figure 1. In order to extend the marketable life of the apples, several technologies are employed. These include cold storage, controlled atmosphere (and dynamic controlled atmosphere) and post-harvest treatment with 1-methylcyclopropene (1-MCP). A brief description of each is included, as they lay out the operating conditions for the Gas Sensors. In addition, two potential sources of interferents are also discussed.

Figure 1: A typical apple storage room. Each box contains ca. 500 kg of apples.

1.3.1 Cold Storage

In order to slow the respiration of the fruit being stored, the room is typically maintained at low temperatures (-0.5 – 1 °C). At the same time, to prevent the fruit from drying out the relative humidity of the room is typically in the region of 70-90%. These conditions are then maintained for the duration of the storage, typically <9 months after harvest, although in some cases the duration can be up to one year. The cooling is performed by the large units visible at the back of the room in Figure 1. Periodically, it is necessary to defrost the cooling units, which results in small increases in the temperature of the air.

Cold storage is typically enacted as fruit are being loaded into the room. The Gas Sensor device must therefore be able to operate in a range of temperatures from ambient temperature (which can be as high as 30 °C) to the minimum seen during cold storage. The rate of cooling of the air in a typical store is illustrated in Figure 2.

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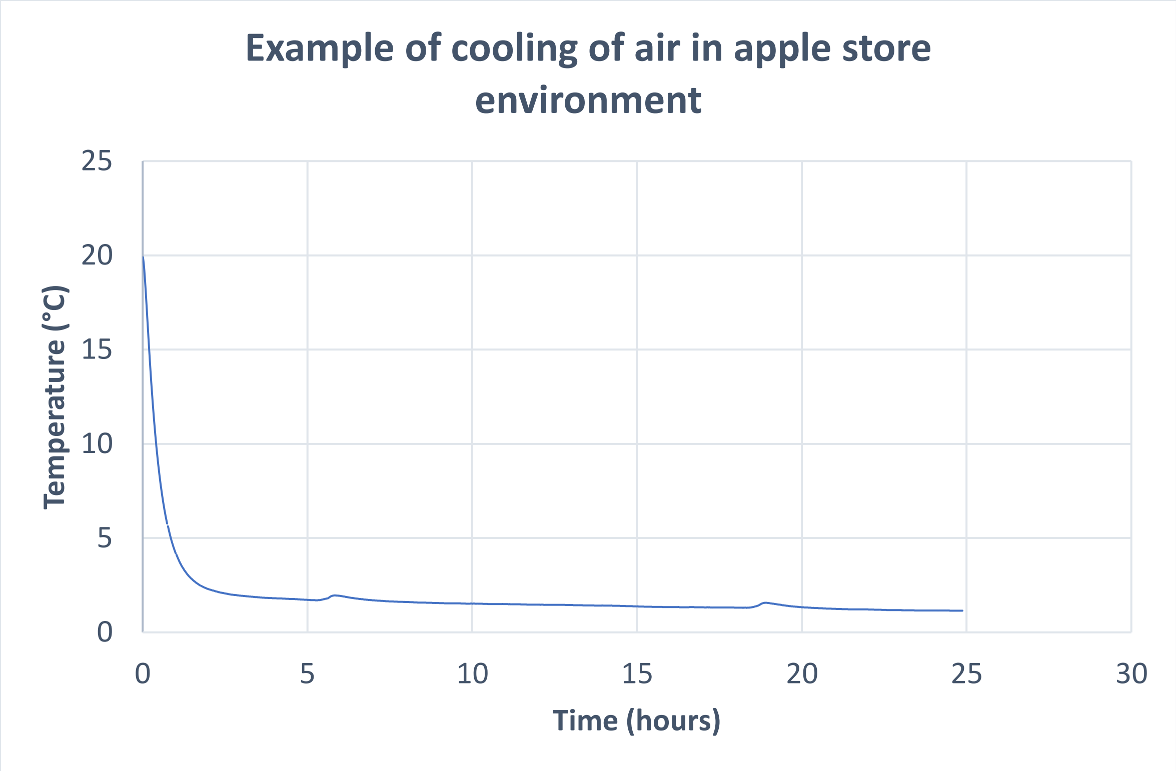


Figure 2: Illustration of air temperature across first 24h after cooling is started

1.3.2 (Dynamic) Controlled Atmosphere

Another approach to extend the life of fruit in cold storage is to utilise controlled atmosphere (CA) during storage. In CA storage, the amount of O_2 and CO_2 in the air is carefully adjusted to limit the respiration of the fruit. Values used range from 0.5-3% (v/v) O_2 and 3-5% CO_2. When the concentration of O_2 and CO_2 are automatically adjusted depending on the levels in the store (using scrubbers or adding gases in the appropriate amount) this is termed dynamic controlled atmosphere (DCA).

As with cold storage, (D)CA is only established once loading of the store has been completed. The drawdown in O_2 level is substantially slower than the drawdown in temperature, with a decrease of oxygen from 21% → 15% expected in the first 24 hours. A rough illustration of the changing levels is included in Figure 3.

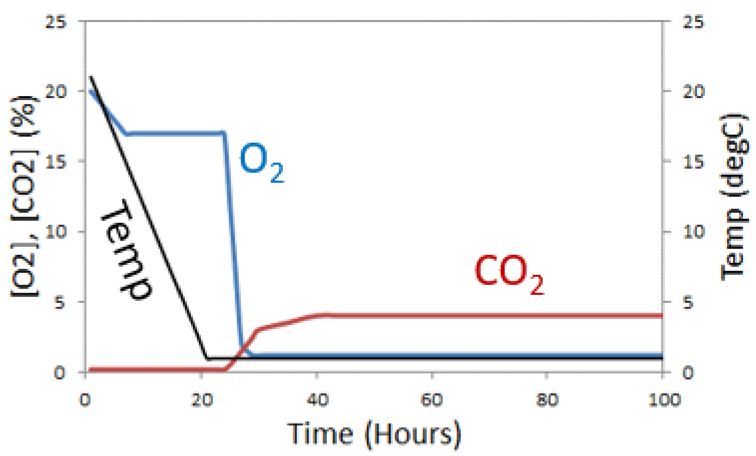


Figure 3: Changes in core temperature of the fruit and ambient O_2 and CO_2 levels in the store.

1.3.3 1-methylcyclopropene

1-methylcyclopropene (1-MCP) is a plant growth regulator that blocks ethylene receptors in fruits, resulting in the ethylene-induced ripening process being halted, as illustrated in Figure 4.

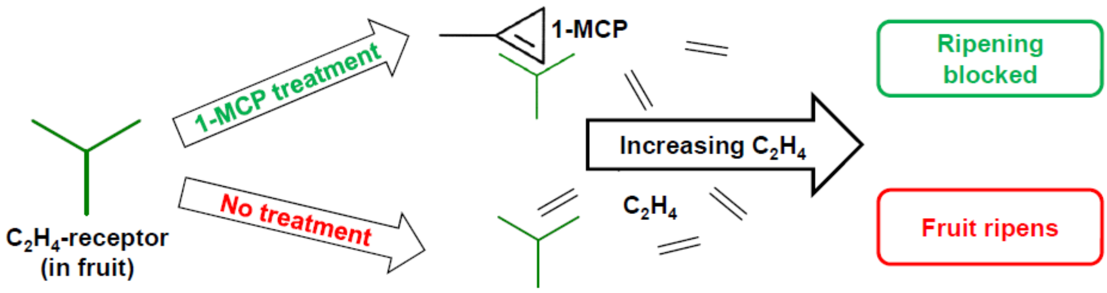


Figure 4: Illustration of the impact of 1-MCP treatment on fruit ripening

In apple stores, a treatment typically consists of releasing 1-MCP in the room to increase the concentration to 1 ppmv. This is done by calculating the volume of the apple store to ensure the correct amount is applied. If the treatment is incorrect due to improper release of 1-MCP, the expected storage life of the apples is decreased significantly. It is therefore highly beneficial to Pace International to be able to provide their customers (i.e. apple store owners) with confidence that the correct level of treatment was applied.

After some time in storage, new ethylene receptors are created in the fruit, so the 1-MCP treatment does not delay ripening indefinitely. As apples are climacteric fruit, the ethylene ripening process is autocatalytic. The onset of ripening is therefore marked by a significant increase in the concentration of ethylene present in the store. Being able to detect this rise is beneficial in providing apple store owners with additional information so that appropriate action (such as opening the stores and scheduling the sale of fruit) can be taken in good time. Being able to monitor ethylene therefore significantly improves the service that Pace International is offering.

1-MCP has shown to be effective for other climacteric fruits, including pears and kiwi fruits. In these instances, there is a risk that over-application of 1-MCP prevents ripening from occurring at all, so treatments are not routinely carried out. The targeted concentration of 1-MCP for pears and kiwi fruits is also typically lower than the concentration used for treating apples (500-650 ppbv), so greater accuracy of monitoring to prevent over-application would be necessary.

1.3.4 Fruit volatiles

As part of the maturing process, fruit release various volatile organic chemicals (VOCs). The most notable of these are summarised in Table 1.

Volatile	Low limit (ppmv)	High limit (ppmv)
Ethylene	4	60
Alcohols	<1	10
Esters	8	122
Total VOCs	20	288

Table 1: Summary of volatiles found in healthy fruit headspace. All values are in ppmv. Data generated by Waqar Ahmed/Roy Goodacre as part of UKRI project 132847 - "Low Cost Sensors to Reduce Storage Losses". Previously presented at InnoLAE 2019.

These volatiles have some impact on the sensors used, although the total impact is relatively low due to the low concentrations present. The levels and types of VOC present vary dynamically throughout the year, with the highest levels present at the start and end of the storage season.

One potential adverse event in the storage of fruit is the fruit rotting. This has an impact on the levels of volatiles present, particularly on the levels of alcohols present. The impact of a significant level of rot (equivalent to 4%) is shown in Table 2.

Volatile	Low limit (ppmv)	High limit (ppmv)
Ethylene	4	60
Alcohols	4	55
Esters	21	306
Total VOCs	49	714

Table 2: Summary of VOCs identified above when a significant level of rot is present in the store. All values in ppmv. Data generated by Waqar Ahmed/Roy Goodacre as part of UKRI project 132847 - "Low Cost Sensors to Reduce Storage Losses". Previously presented at InnoLAE 2019.

In addition to the above, another potential source of VOCs is if the O_2 level becomes too low, and the fruit begin respiring anaerobically. This can result in an unknown level of ethanol and acetaldehyde being release into the room. The impact of these volatiles is unknown, and the likelihood of anaerobic respiration occurring is considered low.

1.3.5 ecoFOG treatment and other volatiles

In addition to 1-MCP treatments, another popular treatment offered by Pace International is an antioxidant and fungicide treatment, with the brand name ecoFOG. This treatment involves creating a fog of solvent particles with contain the active ingredients. These solvents are known to have a deleterious impact on the sensors used, and the intention for 2021 is that sensor will not be exposed to ecoFOG treatment.

A summary of the ecoFOG solvents and other non-fruit sources of VOCs is included in Table 3.

VOC	Origin	Likelihood	Level
VOCs including glycols	ecoFOG	Highly Likely	Extremely high
C4-C10 esters	Component of ecoFOG	Highly Likely	Extremely high
Ozone	Stores are considering a constant trickle	Medium	20-30ppb
Ammonia	Leak from refrigerants	Low	Medium

Table 3: A summary of VOCs not associated with fruit that might be present in the store environment.

2. Specification of Gas Sensor

The purpose of the Gas Sensor created by CDT is to:

1. Report concentration of 1-MCP in apple storage room in the 24 hours after application
2. Periodically (at least 1/day) report concentration of ethylene in apple storage room for up to one year

The below tables lay out the specifications for the system developed in 2020, as well as the proposed specification for 2021. Changes from the previous specification are highlighted.

2.1 Operational

Property (Units)	2020	2021 target – target for new systms
Size (D x H x L, mm)	165 x 300 x 400	150 x 200 x 200
Supply voltage for charging (V)	110-240	110-240
Supply current for charging (A)	1	1
Power consumption (W)	1	1
Weight (kg)	6	4
Local data storage (MB)	8000	512
Battery capacity (days of continuous operation)	14	14
Wireless communication specification	Bluetooth 4.2 + LTE Cat-M1	Bluetooth 4.2 + LTE Cat-M1
Expected "active" use time (hours/year)	400	400
Operational lifetime (excluding filters* and sensors, years)	5 (estimated)	5
Pump flow rate (sccm)	45-55	45-55
Acceptable storage temperature (°C)	0 – 50	0 – 50
Bill of materials	£2,500	£1,300

* Filters to be replaced annually

2.2 1-MCP sensing performance

Property (Units)	2020	2021 target
1-MCP sensors present (CDT GS-FET)	8	0
1-MCP sensors present (Bosch BME680/BME688)	1 BME680	2 BME680/688
Operational range (ppmv of 1-MCP)	0.1 - 10	0.1 - 10
Accuracy (at 1000 ppbv, ppbv)	+/- 200	+/- 200
Resolution (ppbv)	10	10
Concentration reporting interval (hours)	0.5-1	0.25-0.5
Operational lifetime (years)	1-2 (to be confirmed)	1-2 (to be confirmed)

2.3 Ethylene sensing performance

Property (Units)	2020	2021
Ethylene sensors present (Membrapor S200/C2H4)	1	1
Operational range (ppmv of ethylene)	0.1 – 100	0.1-100
Accuracy (<10 ppmv, ppmv)	+/- 1	+/- 1
Accuracy (>10 ppmv, %)	+/- 10	+/- 10
Resolution (ppbv)	100	100
Concentration reporting interval (days)	2	1
Operational lifetime (years)	1-2 (estimated)	1-2 (to be confirmed)

3. Current prototype design

The key technology enabling CDT's Gas Sensor is utilising frequent periods of exposure to and isolation from the target gas to allow for frequent baseline recalibration, effectively countering the phenomenon of baseline drift. This principle, termed "Pulsed filtering", is illustrated in Figure 5.

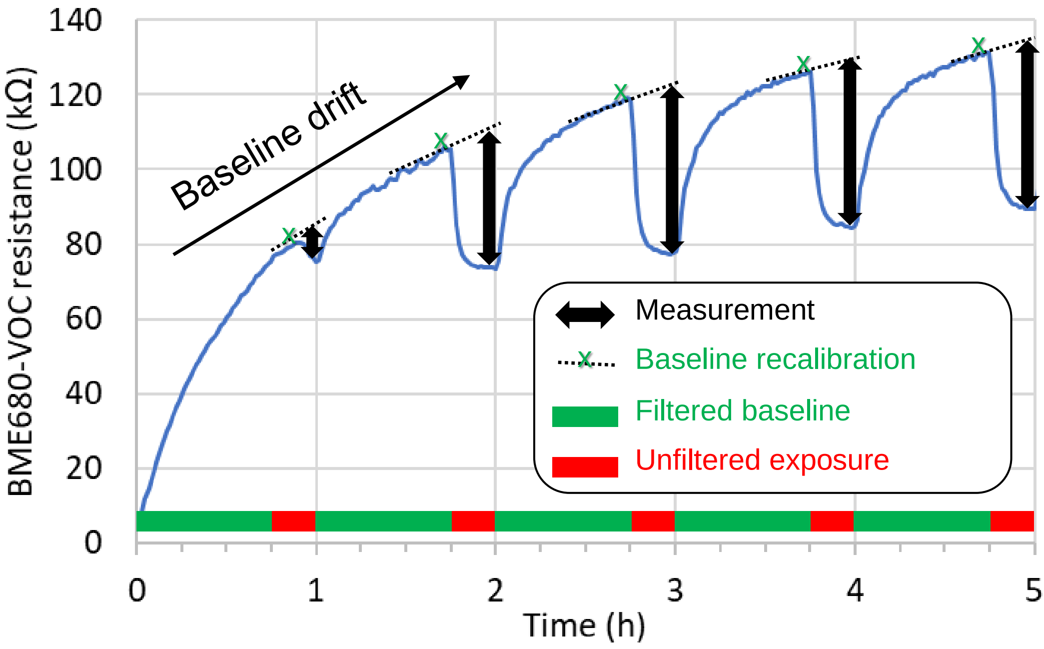


Figure 5: Illustration of the "Pulsed filtering" approach to maintain sensor accuracy

This approach requires extensive treatment of the flow. The specific approach taken in 2020 is outlined in Section 3.1. Details of the mechanical arrangement, including enclosure design, is included in Section 3.2. Section 3.3 outlines the electronic arrangement of the prototype, while Section 3.4 relates to the analytics. A section relating to the operating instructions provided to Pace International is included in Section 3.5.

3.1 Flow schematic and operation states

The flow setup below is designed to allow switching between various operating states needed over time. The switching between operating states is performed by switching three different 3/2 valves throughout the prototype. An overview of the system, with the valves indicated, is given Figure 6. The details of the operating states are given in Table 4.

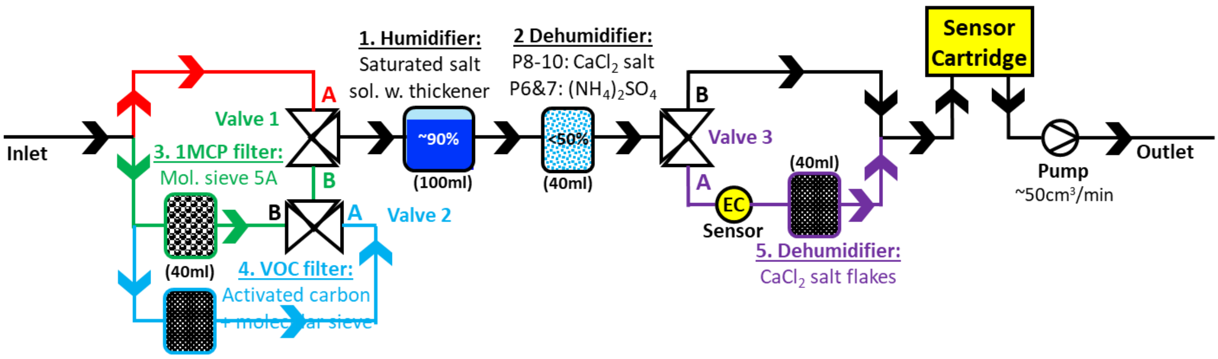


Figure 6: Flow schematic for the prototype in 2020. The specific filters and flow paths associated with are colour coded here and in Table 4

Operating state	Valve 1	Valve 2	Valve 3	Outcome
1MCP: Baseline	B	A	B	Humid without 1MCP/ethylene
1MCP: Exposure	A	A	B	Humid 1MCP/VOC/ethylene exposure

Ethylene: Baseline & drying OTFTs	B	B	A	EC: Humid w/o ethylene/VOCs/1MCP BME680: Dry w/o ethylene/VOCs/1MCP
Ethylene: Exposure	A	A	A	Humid ethylene/VOC/1MCP exposure

Table 4: Operating states for the prototype in 2020. The entries are colour coded according to the flow paths in Figure 6.

Details of the specific components used in constructing the flow paths is included in Section 3.2.

3.1.1 Default operation

The default operating states were subject to multiple iterations. A file detailing some of the versions is included in the file repository. The most recent version, labelled 3001, is reproduced in Table 5 and discussed below. This version is most like the proposed version for 2021.

			Valve 1	Valve 2	Valve 3	Pump	Meas. Interval
First 24h	**1MCP/Ethylene**						
	Power up		B	A	A	FWD	-
Repeat 47x (24h)	**12 min**	**Baseline/stabilisation**	B	A	A	FWD	10 sec
	3 min	**VOC exposure**	**B**	**B**	A	FWD	10 sec
	12 min	**Baseline/stabilisation**	B	A	A	FWD	10 sec
	3 min	**1MCP+... exposure**	A	A	A	FWD	10 sec
	5 min	Clearing lines	B	A	A	FWD	10sec
	Power Off		B	A	B	OFF	-
Total:	50 min						
Every 48h after	**Ethylene**						
	Power up		B	A	A	FWD	-
Repeat 2x (1h)	**12 min**	**Baseline/stabilisation**	B	A	A	FWD	10 sec
	3 min	**VOC exposure**	**B**	**B**	A	FWD	10 sec
	12 min	**Baseline/stabilisation**	B	A	A	FWD	10 sec
	3 min	**Ethylene+... exposure**	A	A	A	FWD	10 sec
	5 min	Clearing lines	B	A	A	FWD	10sec
	Power Off		B	A	B	OFF	-

Table 5: Default operation for a system operating with the 3001 firmware. The valve numbering reflects the numbering in Figure 6.

The default operation in the 2020 PoC trials consisted of an initial 24 hour period of 1-MCP monitoring, followed by periodic (every 48 hours) monitoring of ethylene.

1-MCP monitoring

To accurately monitor 1-MCP, it is necessary to alternate between two exposure types. The first exposure type is to measure the response of the sensor to all gases present in the atmosphere. The second exposure type involves exposing it to the atmosphere through a filter to remove the target gases (1-MCP/ethylene), which allows for background volatiles to be corrected for. The response of the sensors to the background volatiles was found to be minimal, so this additional setting is not required in 2021.

Ethylene monitoring

The same exposure system outlined above is used on a periodic basis to monitor the level of ethylene present in the store. In this case, the system operates only for one hour, before powering off for 47 hours.

In theory, this periodic waking could also be used for communication from the cloud to the system, such as updating firmware or scheduling future 1-MCP treatments that would require more intensive monitoring. This capability was not used in 2020.

3.2 Mechanical

A detailed CAD drawing of the 2020 prototype is included in the file repository. An image of an assembled unit is given in Figure 7.

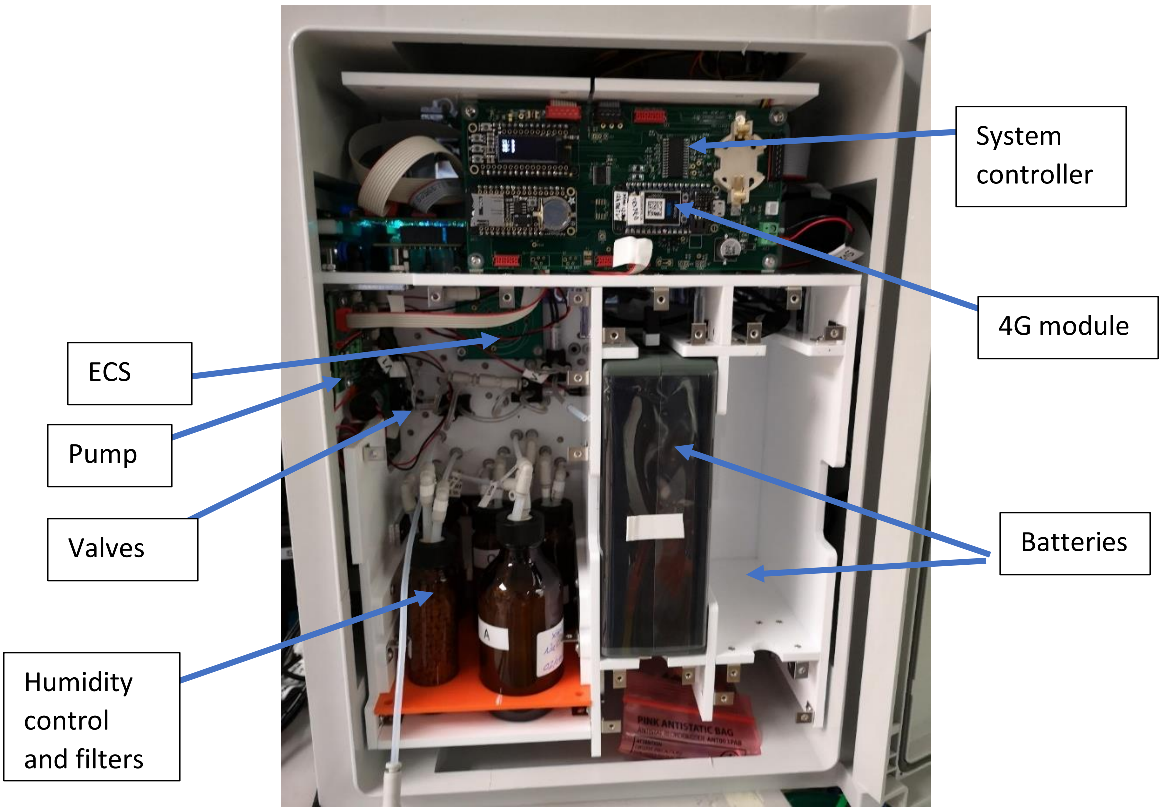


Figure 7: An image of an assembled prototype. The empty space on the right of the image is a holder for a second LiPo battery

3.2.1 Enclosure

A commercially available enclosure was taken and modified for this project. The modifications are listed below:

- Ports
    - Inlet port
    - Outlet port
    - Venting port
    - Cartridge port
- Cut-outs
    - Cut-out for status LEDs
    - Cut-out for power switch

        - Cut-out for mode button
- External additions
    - 4x wall mounting points

The internal supports were laser-cut from acrylic

Front-mounted plates to cover the areas where end user access is not required were designed, but ultimately not used

CAD files indicating the changes are available in the file repository

3.2.2 Flow control

Gas flow paths are constructed from a combination of tubing, glass vials for filters and humidity control, push-fit connectors, and 3/2 latching valves with a pump and needle valve for controlling flow rate. Latching valves are specified to reduce the power consumption of the prototype.

Much of the flow path is constructed from 1/8” OD transfer tubing, in combination with 1/8” fittings. Glass vials with septa are used to allow for easy, gas-tight connections by punching holes in the septum to accommodate the gas tubing. Interfacing to the 3/2 latching valves is performed using soft silicone tubing. Due to a perceived risk of the silicone tube slipping, a fitting was designed and printed to clamp the tubing to the valve.

In Figure 7, some of the flow tubing is located behind the back plate of the prototype. This approach is not recommended for future iterations, due to cost and decreased complexity of the flow path.

List of major components

- 3x 3/2 latching valve
- 4x 40 mL amber vial with septum
- 1x 100 mL amber vial with septum
- 1/16” transfer tubing
- Pump
- Needle valve

Several issues with the flow control system were identified

Issue:

A torque was not specified for tightening the screws holding the fitting to the body of the enclosure, resulting in some fittings being over-tightened. This resulted in some pressure being placed on the valve body, resulting in the valve not correctly switching due to suspected deformation. Loosening the valve resulted in correct switching behaviour resuming for all but one valve. We are in contact with the manufacturer to understand the issue with the remaining valve.

Proposed solution:

Avoid use of fitting or specify torque for tightening. Potential alternative valve also identified.

Issue:

Lower temperature could result in valve not switching

Proposed solution:

Pulse duration increased from minimum (~10 ms) to 60 ms.

3.2.3 Sensors

As noted previously, as two gases are to be monitored, more than one sensor is required in the prototype. The sensors for detecting 1-MCP were in an external cartridge, while the ethylene sensor was located inside the main body of the prototype.

1-MCP sensor cartridge

Due to the expected limited lifetime of the CDT GS-FET component, in the 2020 design the sensors were in an externally mounted replaceable cartridge. This cartridge contained 8x GS-FET devices, in addition to a single Bosch SensorTec BME680 environmental sensor. The BME680 sensor component monitors temperature, relative humidity, pressure and VOCs in the atmosphere. A commercial system was used for the outer cartridge, with a machined and printed plastic internal assembly to minimise headspace. An image of this is included in Figure 8.

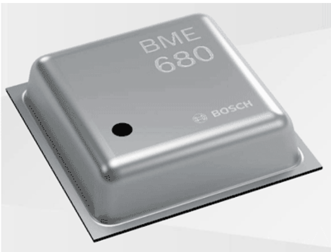


Figure 8: (Left) An image of the external cartridge used in the 2020 prototype. (Right) an image of the BME680 environmental sensor

As a result of the PoC trials in 2020, the decision was made not to proceed with the external cartridge model. Separately, the decision was made to proceed without the CDT GS-FET component. Therefore, the 2021 system should focus on having the BME680/688 component internally and there is no requirement for this "external" cartridge.

During a risk assessment exercise on the components used in the 2020 prototypes (excluding the GS-FET devices) only a single supply chain issue has been identified:

Issue:

Global supply of the BME680 component is very low. This is possibly due to imminent replacement by the BME688 component. Supply risks for BME688 component unknown.

Proposed solution:

An order for 50x BME688 sensor components has been placed by CDT with Mouser (US). Anticipated delivery is in May 2021. As required, these can be made available for the 2021 systems.

Ethylene electrochemical sensor (ECS)

The chosen ethylene sensor (Membrapor C2H4/S200) was in the main body of the prototype. A gas-tight cap with inlet and outlet was purchased from Membrapor. The interface to the cap was through a combination of two different types of silicone tubing. An image of the sensor in question is included in Figure 9. The specification for this part is laid out in Section 2.3. One additional requirement is that the ECS requires some O_2 to be present in the gas sample. As the level of O_2 in the CA store is always higher than the minimum level requires, no additional steps are required to address this specification.

Figure 9: An image of the standard housing for Membrapor sensors

One issue was identified with the sensor:

Issue:

The sensor is damaged to prolonged exposure to VOCs, including ethylene and apple fruit volatiles.

Proposed solution:

Sensor is only exposed periodically to volatiles, using the pulsed filtering approach

3.2.4 Filtration and humidity control

Various materials were deployed for the control of humidity and filtration. In addition to the requirements regarding capacity in lifetime, it is also necessary that all materials are tip-stable, to prevent normal handling of prototypes during installation resulting in contamination or blockage of the gas flow path.

Filtration

The pulsed-filtering approach outlined earlier requires the use of filter materials to remove the target gas at periodic intervals. As the system monitors multiple gases, different filters were utilised for different target gases, as outlined in Table 6.

Target	Filter material
1-MCP	Molecular sieves 5A
Ethylene	Activated carbon

Table 6: Filters deployed in the 2020 prototype

Due to incompatibility of the ECS to continuous exposure to VOCs, the filter for ethylene must also remove apple volatile VOCs as highlighted above.

One issue was identified with the filters used.

Issue:
Repeated exposure to ethylene resulted in the activated carbon releasing previously captured ethylene, giving a high baseline value
Solution:
Switch filter material to oxidising filter like Purafil SP. This can potentially be combined with another filter materials such as molecular sieves 13X to increase operational lifetime or tolerance to other volatiles that are poorly absorbed by Purafil SP (such as alcohols)

Humidity control

Work conducted at CDT's laboratories identified optimal humidity ranges for the various sensor components used in the 2020 PoC trials. These humidity ranges are summarised in Table 7

Component	Lower boundary	Upper boundary
CDT GS-FET	70	90
Bosch SensorTec BME680	0	50
Membrapor C2H4/S200	15	90

Table 7: Optimal humidity ranges for the sensor components used in 2020

As can be observed from the values in Table 7, there is no humidity value that allows for ideal operation for both the GS-FET and BME680 sensor. Therefore, the decision was made to proceed with two types of prototype, running at different humidity levels associated with the different sensors. The humidity control system results from the combination of three separate stages. A humidifier and two separate dehumidifiers, as illustrated in Figure 6. The materials used for the two systems identified above are summarised in Table 8, with the specific materials explained in Table 9.

	GS-FET optimised system			BME680 optimised system		
Stage	Material	Quantity (g)	Target RH	Material	Quantity (g)	Target RH
Humidifier	XG	80	90	XG	80	90
1st dehumidifier	$(NH_4)_2SO_4$	30	80	$CaCl_2$	30	50
2nd dehumidifier	$CaCl_2$	30	50	$CaCl_2$	30	50

Table 8: Summary of the systems controlled to different relative humidity levels

Material	Details	Form
XG	Saturated solution of calcium formate thickened with xanthan gum (1%)	Gel
$(NH_4)_2SO_4$	Ammonium sulfate	Granules
$CaCl_2$	(anhydrous) Calcium chloride	Flakes

Table 9: A summary of the humidity control materials used

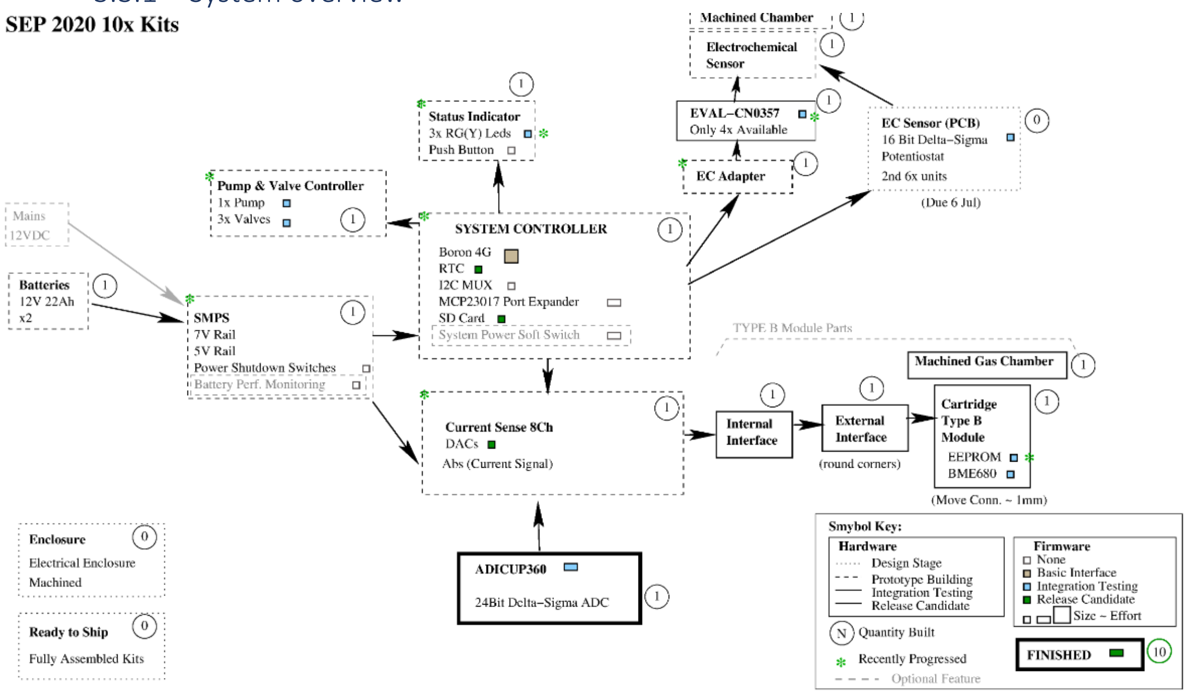
While these materials have been shown to be robust for handling, alternative materials could be more suitable. Humidity control measures based on micro- and mesoporous materials should be avoided, as these materials tend to also filter 1-MCP and/or ethylene. Simple salts are effective at controlling humidity without filtering 1-MCP.

3.3 Electronic

The control electronics for the 2020 prototype was a combination of custom PCBs with some commercial modules. An overview of the system is presented in Section 3.3.1, with details of the purpose of each component following.

In 2020, a modular design was undertaken to allow for potential iteration of individual components without requiring a new system controller board.

3.3.1 System overview



3.3.2 System controller

The main system controller board incorporates a real-time clock (RTC) and interfaces directly to the wireless communications module (Particle.io Boron 4G module) and the local memory (SD card). The board is also linked to the external “mode” button to allow a different set of operating states to be accessed.

3.3.2.1 Boron 4G module

The Boron 4G module uses an internal SIM card to connect to the particle cloud. Details of the cloud connections can be found in Section 3.4. The publish frequency was set at every 30 seconds.

The Boron 4G module also includes the ability to communicate over Bluetooth. This functionality was not used in 2020.

3.3.2.2 SD card module

A module for writing data values to SD card was included to allow for local storage in data in case of failure of the LTE-Cat M1 connection.

Two issues were detected with the SD card module:

Issue:
In one module, the SD card was corrupted, to the point that writing data, deleting data and formatting the SD card was not possible. The origin of this error was not established. This occurred while the SD cards were in use in the systems.
Proposed solution:
Review functions for writing to SD card to determine origin.

Issue:
Several prototypes ended up with an error where invalid characters were written to the SD card. This interrupted the data recording and resulted in data loss. The origin of this error was not established
Proposed solution:
Review functions for writing to SD card to determine origin.

3.3.3.3 Internal display

An OLED display is included to allow for monitoring of operation, including date and time, SD file size and the most recent reading from a sensor. This component isn't likely to be required in final, commercial versions of the Gas Sensor. The component also included 3 small buttons that were used as part of the flow tests.

3.3.3.4 Mode button

An external button, the "mode" button allowed different operation modes (aside from the default operating mode outlined in Section 3.1.1) to be accessed. These modes were accessed by holding the "mode" button before power-on, and then releasing when the status LEDs reached a specified colour.

The most important alternative operating mode was the manual flow control mode. In this mode, the buttons located alongside the OLED display were used to manually switch the 3/2 latching valves between states, allowing for different valve configurations to be tested to ensure that no leaks were present in the system

3.3.3 Switched-mode power supply + low power management

Take 12V power from batteries and adapt to 5V and 7V. Incorporates external power switch for powering on prototype.

One issue was observed with the power supply.

Issue:
Quiescent power consumption associated with Boron 4G module was too high
Solution:
A low power management board was created and implemented in prototypes 6-10. This allowed for the power to be removed from the Boron 4G component, decreasing power consumption significantly. A detailed schematic is included in the file repository

3.3.4 Current Sense 8ch

Board to measure currents of the CDT GS-FET components.

This part is not required in the 2021 system

3.3.5 Cartridge type B + internal and external interfaces

This component contains two types of sensors – CDT's Gas-sensing field effect transistors (GS-FET) and the BME680 environmental sensor.

This part is not required in the 2021 system

3.3.6 Pump and valve controller

This part is a secondary board used to control the pump and valve components. As noted above, this was included as a module but would be suitable to move to the main board in the 2021 system.

3.3.7 Battery

The systems are powered using 2x lithium polymer batteries. The part chosen was a 22Ah 12V battery produced the Tracerpower.

One supply chain issue has been identified with this component.

Issue:
Tracerpower batteries have been previously purchased from Deben Group Industries. Batteries are currently listed as out of stock, but more stock is expected from May onwards.
Proposed solution:
Place order with new the supplier.

3.3.8 Status Indicators

3x RG(Y) LEDs are used to indicate status of kit. The interpretation according to the current firmware is laid out in Table 10.

Light	Green Flash	Red Flash
Status	GS-FET working	GS-FET not working
SD Card	Succeeded writing to SD	Failed writing to SD
Cellular	Successful connection to 4G	Failed connection to 4G

Table 10: Status light codes in normal operation

3.3.9 EC sensor + adaptor

Integration for the Membrapor C2H4/S200 component, including measurement circuitry. Although a commercial board was identified that could directly monitor the sensor, supply issues resulted in an equivalent system being developed in-house. This component would also be suitable for inclusion on the system controller board in 2021.

3.4 Cloud platform and analytics

Pace Intl.'s intention is to create a customer accessible cloud platform, allowing the end user to access information regarding their store at will. For the PoC trials in 2020, two cloud services were utilised to create a web-accessible view of analysed data: the particle.io cloud to transfer data from device to the web, and thingspeak.com for storing and analysing data. A schematic overview of this is included in Figure 10. This "Cloud Analytics" platform is described in more detail in Section 3.4.2.

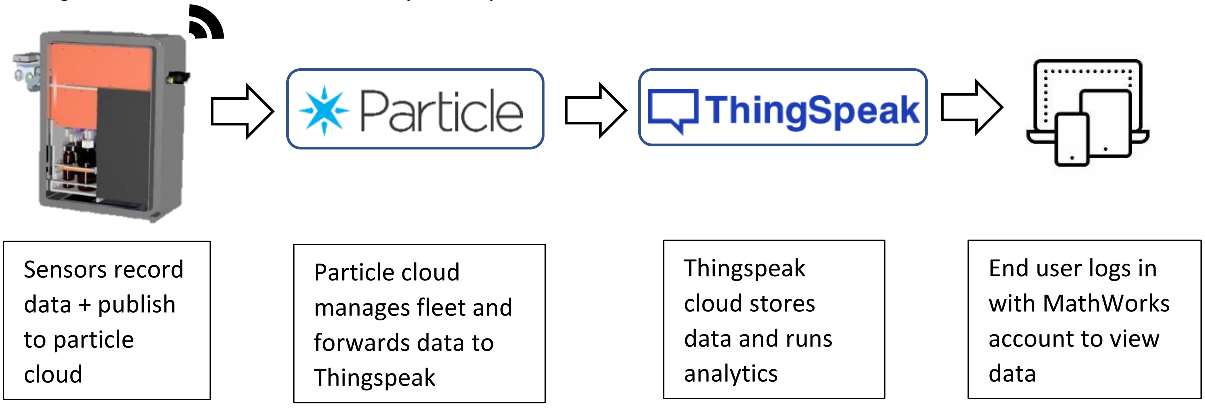


Figure 10: Schematic of data transport in 2020 PoC trials

A secondary analytics path was also used, which involved offline data analytics performed by the Gas Sensors team at CDT. More details are found in Section 3.4.3, below.

3.4.1 General comments on analysis

The intention of this section is to capture know-how on how to approach analysing data collected by the prototypes in 2020. This includes reference to specific values that arose from experiments conducted prior to the 2020 PoC trials at CDT's laboratories. This know-how can be broken down by the sensor it applies to. It is worth recalling that the range of relative humidity and temperatures experienced in the store environment is relatively limited, but even within these ranges some impacts can be seen.

3.4.1.1 BME680/688 sensor

Several environmental factors influence the BME680 sensor. In addition, there are some non-environmental factors that must be included.

Relative humidity

The response of the BME680 to a fixed concentration of 1-MCP is impacted by relative humidity (RH), as illustrated in Figure 11.

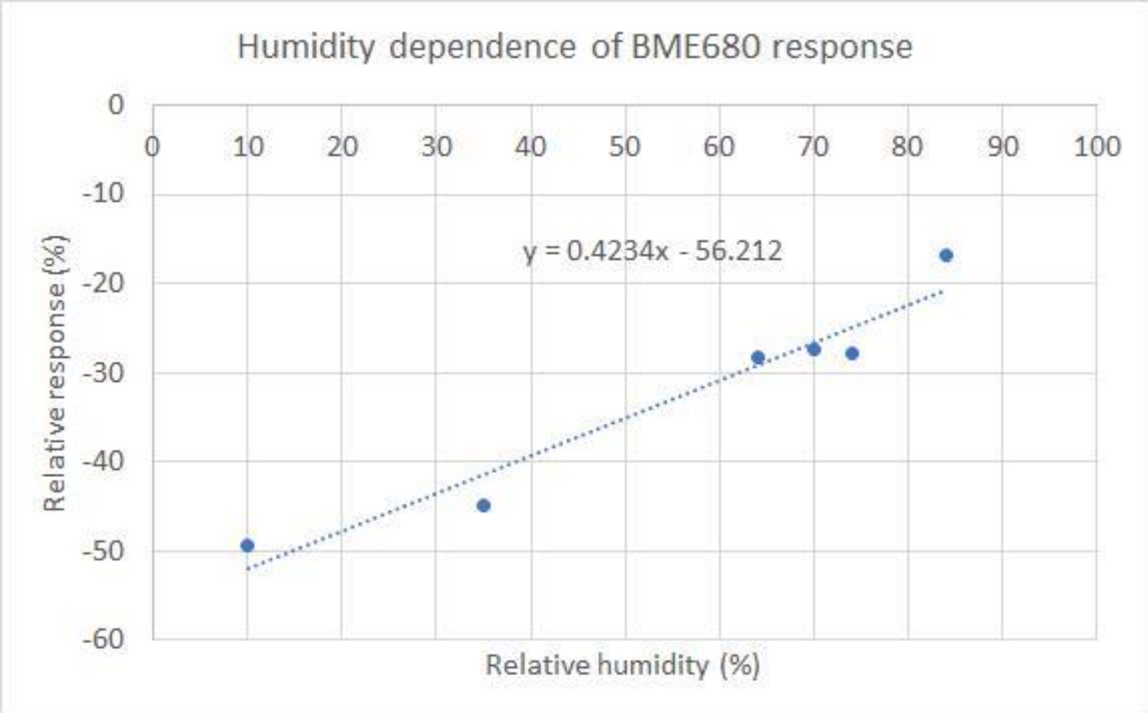


Figure 11: Relationship between relative humidity and response for sensors exposed to 1-MCP at a 1 ppmv concentration.

Any difference in the RH between the calibration and the measurement conditions will need to be corrected for. This can be minimised by conducting the calibration *in situ*, which will result in minimal differences between calibration and measurement. Any changes during operation can be corrected for in a similar fashion, using the RH values recorded by the BME680 sensor.

Temperature

The impact of temperature on the sensor response to a fixed concentration of 1-MCP is minimal. Provided that calibration is conducted at a similar temperature, the impact of temperature can be ignored.

Ethylene

The BME680 shows significant cross-sensitivity to ethylene, which must be corrected. This ethylene sensitivity significantly varies sensor-to-sensor. This will require utilising the ethylene measurements from the ethylene-sensing component (Membrapor C2H4/S200) as well as calibration values of the impact of ethylene on the individual BME680 sensor. This correction was not applied in the 2020 PoC trials.

Burn-in

BME680 sensors show significant burn in. We recommend that sensors undergo burn-in (i.e. are powered on and run) for a minimum of 100 hours before calibration, slightly beyond the 50 hours of burn-in recommended by the manufacturer.

Run-in

Significant run-in has been observed for these sensors. Run-in is defined as the period after powering on the sensor before the sensor is stable for use. In particular, the BME680 sensor shows significant increase in sensitivity to 1-MCP in the hours after powering on (see Figure 12).

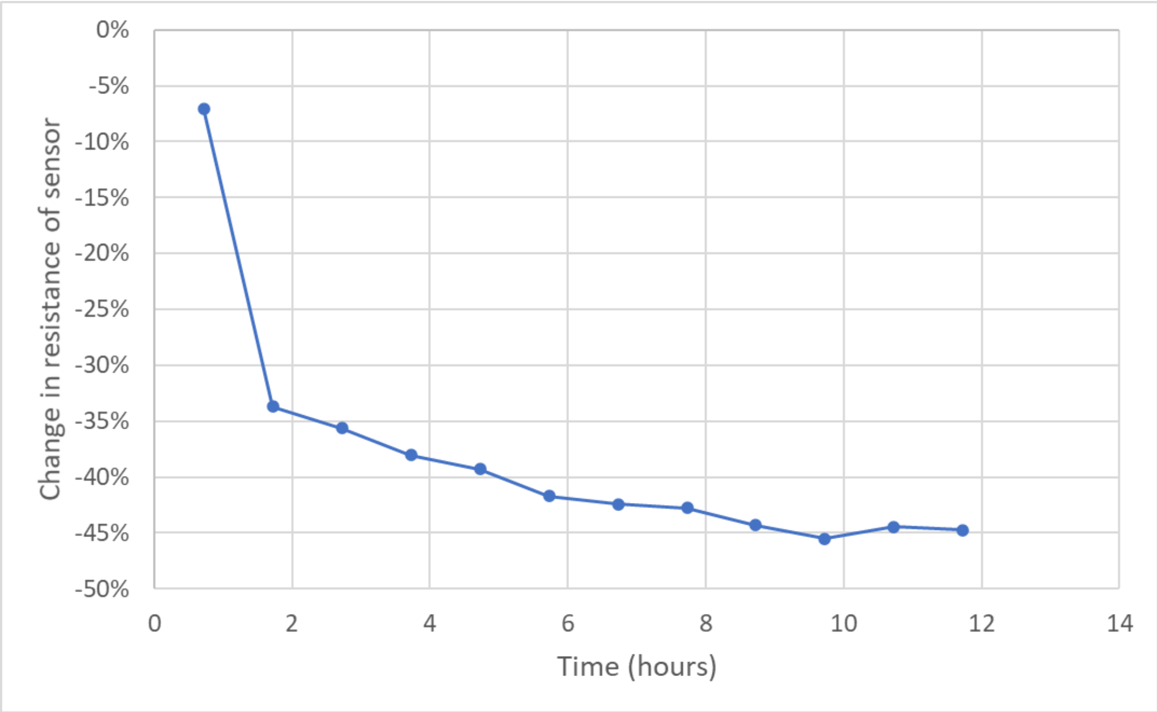


Figure 12: Response of the sensor to repeated 15-minute exposures of 1-MCP at a fixed concentration of 1 ppmv.

CDT's solution in the 2020 PoC trials was to utilise the data collected over 12 hours during calibration to create a profile of how the sensitivity varied with time. By fitting this relationship to a curve, this relationship could be extended to predict the changes in sensitivity over 24 hours, allowing for correction of burn-in

Two alternative solutions have also been proposed to Pace Intl. for consideration:

1. Calibrate the sensors over a 24 hour period, to avoid need for fitting profile to model
2. Power-on BME680 sensor component 12 hours before treatment starts, to minimise the impact of burn-in on the sensitivity.

3.4.1.2 Membrapor C2H4/S200

The influence of the environment on the ethylene-sensing component are more limited

Relative humidity/temperature

No significant impact of RH/T has been observed with this sensor. Provided the calibration RH/T is not too dissimilar to the operating conditions, no corrections need be applied

Cross-sensitivity

Significant impacts of some VOCs at high concentrations (>100 ppmv) have been observed. These volatiles are not expected to be encountered in these quantities during storage, and so the impact can safely be ignored.

3.4.2 Thingspeak analytics

Due to limitations in the Thingspeak platform, the data from each prototype is broadcast to one of three "channels". The data from different channels must then be combined back into a coherent data format before analysis can be performed. Due to the nature of cloud communications, with options for data to be delayed or missed, this can result in significant missing data. For this reason, we recommend that Thingspeak is not used as a data platform for 2021.

A copy of the analysis file used in the cloud platform for 1-MCP analysis is included in the file repository. This file is written using MATLAB code. This version is outdated compared to the python version, and it is not recommended to be used as a starting point for creating analytic algorithms.

3.4.3 Python analytics

A copy of the analysis file used for offline analysis is included in the file repository. This is the most up-to date version possible. There are some references to external files: where these occur, a comment explains the type of file required for the script to work. A general process flow for the 1-MCP analysis is included in Figure 13.

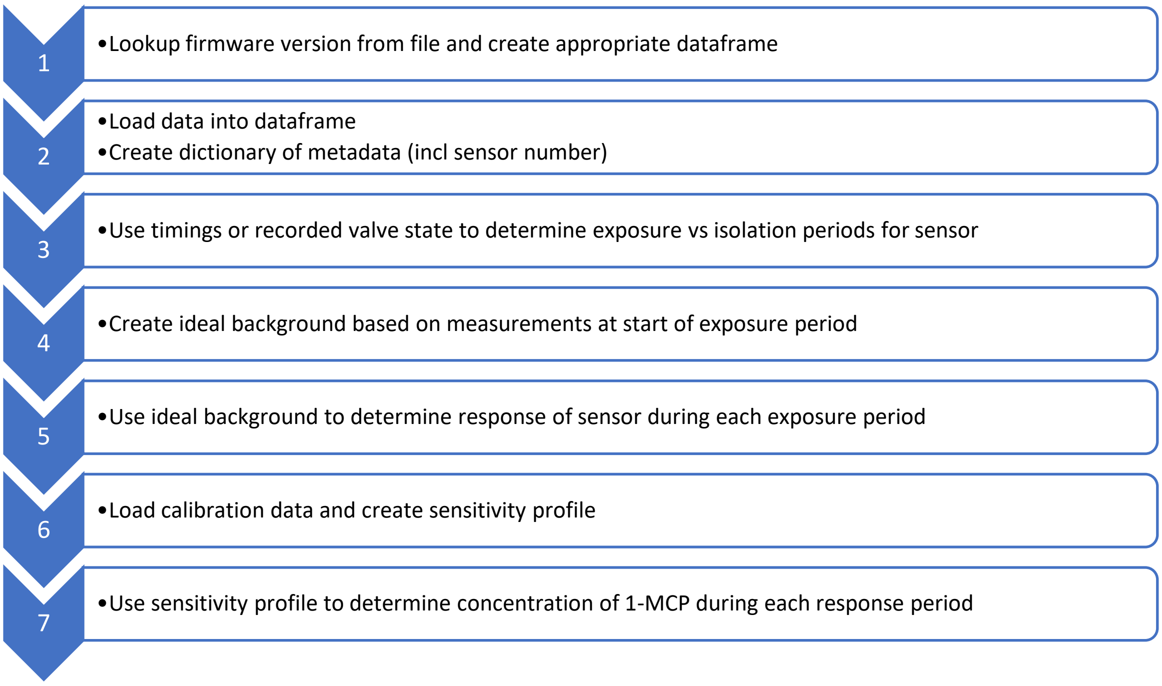


Figure 13: Process flow for 1-MCP analysis

A second script is included also [filename]. This is for analysis ethylene data collected in the Thingspeak cloud platform. The data must first be exported from the Thingspeak channels associated with the cloud platform into local .csv files. The process flow for this analysis is included in Figure 14.

1. Load each channel as a dataframe
2. Create index for joining data on
3. Join dataframes into single dataframe
4. Select periods for analysis
5. Identify peaks in signal from ethylene sensor using scipy.signal.find_peaks
6. Apply calibration from measurements taken at CDT

Figure 14: Process flow for ethylene analysis

3.5 Operating instructions

Included in the file repository is a copy of the setup and operating instructions that accompanied the 2020 prototypes. Two issues in the PoC trials were later attributed to these instructions, and will need to be remedied in 2021:

Issue:
During initial setup, flow tests were carried out as indicated in the setup instructions. These tests were conducted in a warm office environment, and the prototypes were immediately powered off. When deployed in the PoC trials, the sensors were placed in a cold store, which caused condensation and damaged the sensor cartridges
Proposed solution:
Proposed changes to operating humidity should resolve (see Section 4.2.1). Alternatively, flow tests should be conducted in a cold environment.

Issue:
Some leaks were identified, despite tests conducted. More detailed instructions were drafted (see file repository), but issues persisted
Proposed solution:
Lower complexity of system in 2021 should resolve. Alternatively, alternative connections to filters/humidity control could be considered.

4. 2021 prototype design

4.1 Design brief

The brief is to create 25-35x of a modified version of the system outlined in Section 3. A summary of the required modifications is included below. These systems must be delivered to Pace Intl. no later than September 2021. Included in the design is the requirements for a cloud platform that is accessible to staff representatives of Pace Intl. The systems must be suitable to be deployed by Pace Intl. staff, after calibration at Pace's facilities. Instructions for carrying out the setup and in-house tests should also be provided. Sensor accuracy will be validated using a gas chromatograph equipped with a flame-ionisation detector (GC-FID).

The system must be robust to handling for the transport from Pace's facility to the customer store. At the customer store, the system must be wall-mountable, as well as able self-stand on the floor of the customer unit, depending on if the unit is to undergo short- or long-term tests. The system must be simple to operate, with detailed operating instructions provided.

The intention is for analytics to be carried out remotely utilising a cloud platform. Therefore, the system must be able to communicate to an appropriate cloud platform, ideally using LTE Cat-M1 communications. The ability to report readings locally via Bluetooth 4.2 is also desirable. The cloud analytics will be like the version outlined in Section 3.4.3.

4.2 Flow schematic and operating states

A simplified version of the system operating in the PoC trials is proposed for 2021. A schematic of this system is included in Figure 15. The operating states are also simplified, with a summary included in Table 11.

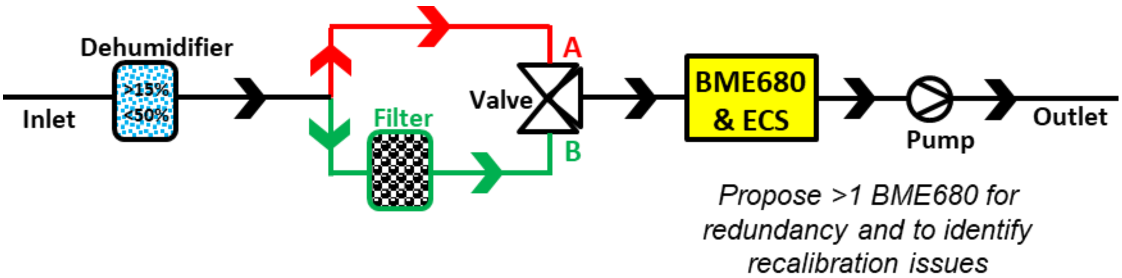


Figure 15: A schematic of the proposed system for 2021. Proposed dehumidifier is either LiCl or $CaCl_2$, proposed filter is a blend of molecular sieves 13X and Purafil SP layered in a vial, with exposure first to molecular sieves 13X.

Phase	Valve	Outcome
Baseline	B	w/o 1MCP/ethylene/VOCs
Exposure	A	1MCP/VOC/ethylene exposure

Table 11: Proposed operating states for 2021. The text colours indicate the associated flow path in Figure 15.

The major changes in this version are summarised below

4.2.1 Removal of humidifier & relocation of dehumidifier

The humidifier component was required in 2020 to ensure adequate humidity for the GS-FET sensing component. As this component is removed, the system in 2021 can operate at a lower overall humidity, provided that this humidity falls in the region of 15-50 %RH. During operation, this RH level should then stay within 10%.

4.2.2 Removal of additional filtration loops

Some of the exposure loops identified in Figure 6 and Table 4 are no longer required, as the impact of VOCs associated with the fruit on both sensors was less than expected. This means that only a single filter is required, as opposed to the two separate filters used in 2020. This filter being easily replaceable without requiring significant disassembly is desirable.

4.2.3 Removal of additional valves

Due to the removal of some filters and humidity control options, it is not necessary to include as many parallel flow paths as previously. Therefore, less valves are required in the system. Although a 3/2 valve is indicated in Figure 15, a combination of two 2/2 bi-stable valves could be used instead.

4.3 Mechanical changes from 2020

One major change is proposed to 2021. Significant changes are likely to also be required associated with changes in the electronic parts.

4.3.1 Change to internal sensor cartridge

It is proposed that the external cartridge in the 2020 prototypes is replaced with an internal cartridge, or a series of internal headers containing the different sensors. Although the BME680 and ECS components are indicated as being in the same unit in Figure 15, it is preferred that these units are replaceable independent of each other.

This internal cartridge will require designing a new gas-tight header, or incorporating a commercially available gas-tight header, such as the header available from Membrapor.

4.4 Electronic changes from 2020

Two major changes are proposed. Several other significant changes, both electronic and other, are likely to be required as a result of the major changes. This will likely involve a complete redesign of many of the PCBs used in this kit.

A modular approach was used in 2020 to allow for flexible iteration of specific components, but it may be suitable to use a single board for the purpose of saving cost.

4.4.1 Removal of GS-FET component

Due to potential supply difficulties, the GS-FET component will not be included in the 2021 system. Much of the associated measurement circuitry can therefore be removed, including the 8CH Current Sense board. Partial redesign of the PCBs will therefore be required.

4.4.2 Removal of interface boards

With the removal of the external cartridge (see Section 4.3.1), the interface boards are no longer required. As it is possible that an approach involving the sensing components being separated, it may be practical to reconsider the approach used in interfacing to the sensor components from 2020.

5. Calibration and validation testing

Due to the variable response from both sensors, calibration of the response of the sensors to the target gasses is necessary to obtain sufficient accuracy when operating in the store. Calibration testing involves recording the sensors responses and the concentration of gas (as determined by GC-FID) to create a concentration-corrected sensitivity value. Two potential approaches for calibration are indicated in Section 5.1.

In addition to calibration, it will be necessary for some validation testing to be performed. In this instance, validation tests refer to tests of the integrity of the system, without any testing of the response to the target gasses. While the expectation is that this testing will be performed by the manufacturing partner, if there are substantial barriers or a substantial cost to these tests there may be the possibility of the validation testing being performed by Pace International or CDT.

As it is expected that shipment of the units will require some disassembly, we expect that some secondary validation tests will need to be performed by Pace International after receiving shipment. These tests should be limited to only testing the aspects of the system impacted by the disassembly.

5.1 Calibration process

The proposed calibration process is outlined in Figure 16. This process involves the calibration being conducted by Pace International after receiving shipment of the prototype systems, alongside validation tests. In addition, there are consideration in how the generated calibration data will be included in the cloud analytic platform. A proposed process is included in Figure 17

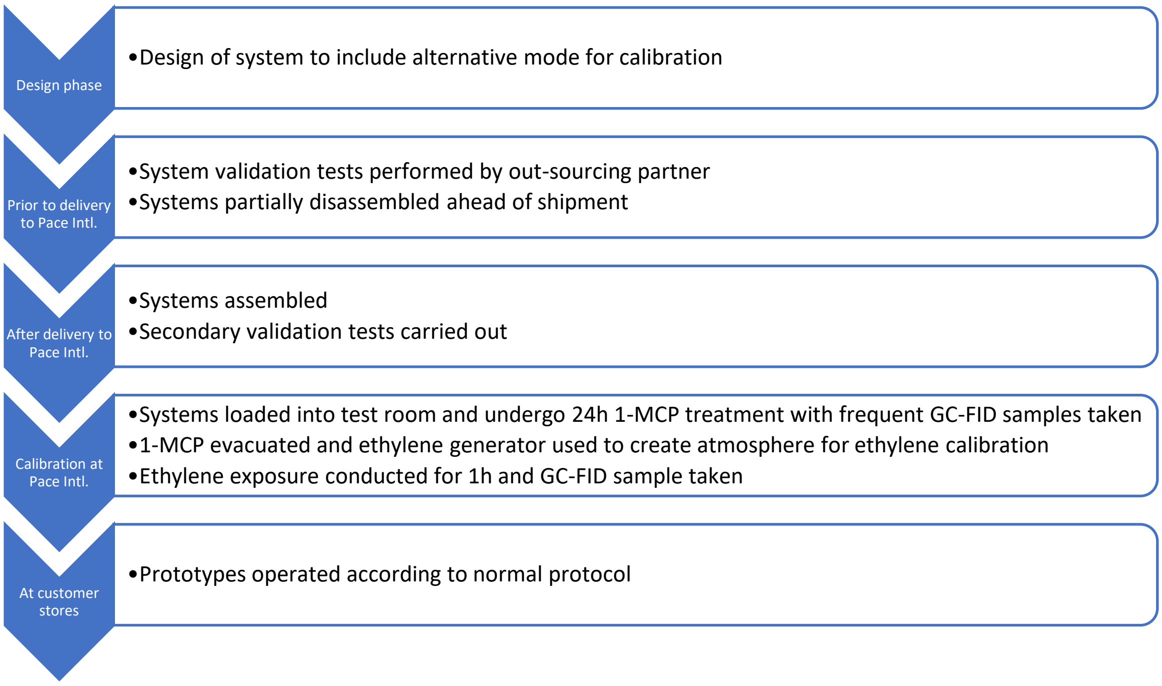


Figure 16: Proposed process for calibration of the prototype systems

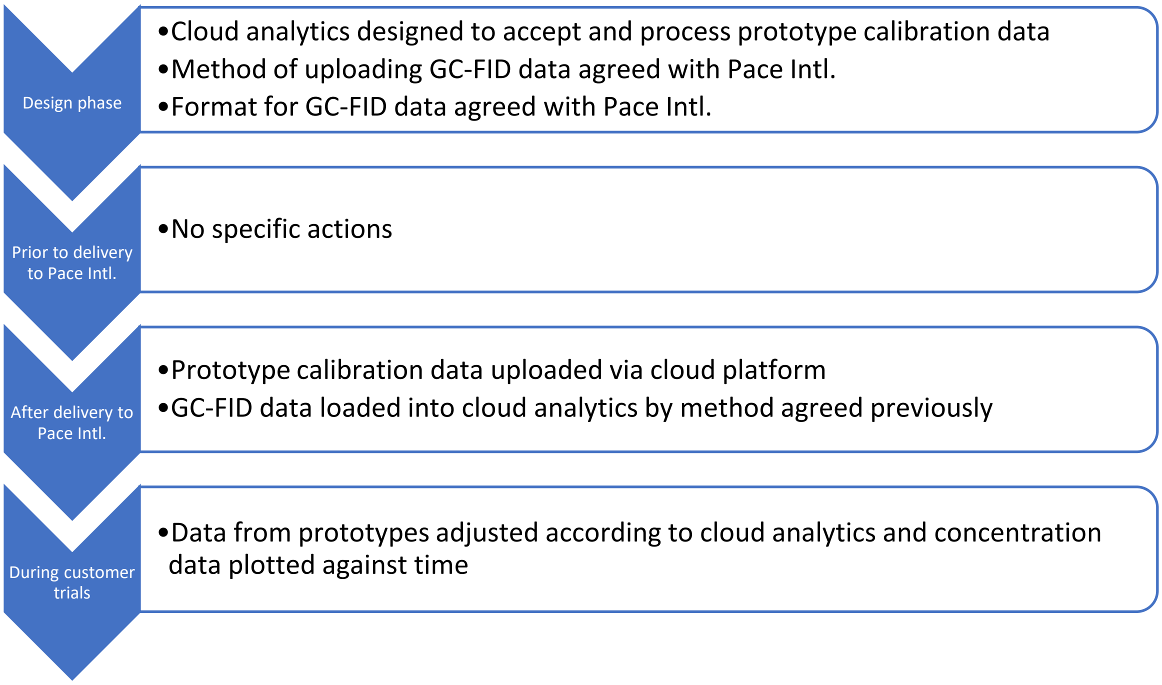


Figure 17: Proposed process for including calibration data into the cloud analytics platform