Validation and Demonstration of Relative Humidity Controlled Accelerated Corrosion Test: New Specification Development

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WP18 - 5087: Validation and Demonstration of Relative Humidity Controlled Accelerated Corrosion Test: New Specification Development

Performers: NAVAIR, SwRI, Boeing, ARL, AMCOM, AFRL, AFLCMC, Q-Lab

Technology Focus

- This effort is designed to develop accelerated corrosion tests which produce corrosion severity and damage mode which are similar to that produced by outdoor exposure.

Demonstration Site

- *Lead:* Patuxent River, MD
- *Round Robin at various sites*

Demonstration Objectives

- Develop accelerated corrosion test methods which:
  - Have positive correlation of corrosion damage modes to outdoor exposure samples.
  - Meet 1 year's corrosion damage in 1,000 hours or less.
  - Produce tunable corrosion severity and damage mode for both severe and moderate test conditions.
  - Display low lab to lab variability per ASTM E691.

Project Progress and Results

- Preliminary chamber performance characterization is underway.

Implementation Outlook

- Task 1 (Optimization of Cycle Period) is about to begin at NAVAIR and SwRI. NAVAIR currently working on chamber validation.

Project Team

- Victor Rodriguez-Santiago (PI), Alexander Lilly – NAVAIR
- James Dante, Erica Macha – SwRI
- Kristen Williams – Boeing
- Fred Lafferman – ARL
- Doug Hansen – UDRI
- Sean Fowler – Q-Lab
- Mike Spicer – AFRL
- Nancy Whitmire – AMCOM
- M. Casey Jones – Air Force CPCO/LCMC

Technical Objectives

- The proposed ESTCP effort is designed to develop existing corrosion tests sufficiently to allow its widespread use in testing labs across the DoD, original equipment manufacturers, DoD contractors, and other testing labs.
  - **Objective 1**: Mature the accelerated corrosion test protocol developed during the SERDP WP-1673, WP-1674, and SERDP WP-2521 efforts*
  - **Objective 2**: Characterize protocol variability by means of round robin testing with participation from DoD laboratories, contractors, and private industry
  - **Objective 3**: Establish a national standard to be used throughout the acquisition life cycle for testing and qualification of corrosion prevention schemes

*SERDP WP-1673: Accelerated Dynamic Corrosion Test Method Development
*SERDP WP-1674: Dynamic Multivariate Accelerated Corrosion Test Protocol
*SERDP WP-2521: Standardized Test Methodologies for Low Observable Coating Durability

Technical Approach

Task 1. *Optimization of Cycle Period*

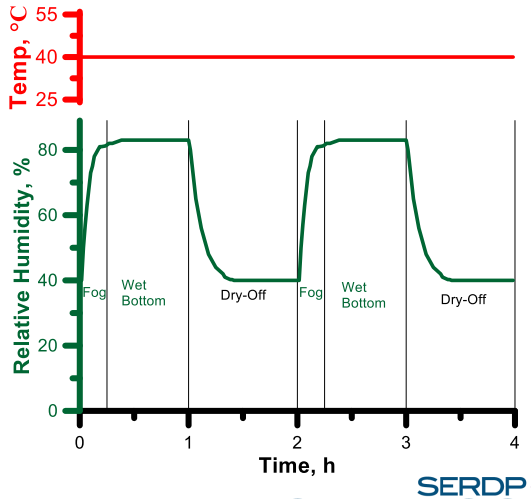
- Although the test protocol provides good agreement for several milder exposure locations, previous results indicate that small shifts in cycle duration and solution chemistry may have large effects on total damage. Therefore, a test protocol to simulate more aggressive environments will be developed.
- **Two chemistries** and **three RH cycles** will be tested. The cycles will be defined by NAVAIR, SwRI, Boeing, ARL, and AFRL. Testing during for this task will be performed by NAVAIR and SwRI.

Proposed Chemistries*

Reagent	g/L (Reagent) NE#1	g/L (Reagent) NE#2
NaCl	24.53	22.26
$MgCl_2.6H_2O$	11.10	11.10
Na_2SO_4	4.00	4.00
$NaNO_3$		3.27
HCl (1N)	(1 ml)	(1 ml)

*Normalized to ionic strength.

Base Cycle to be Modified



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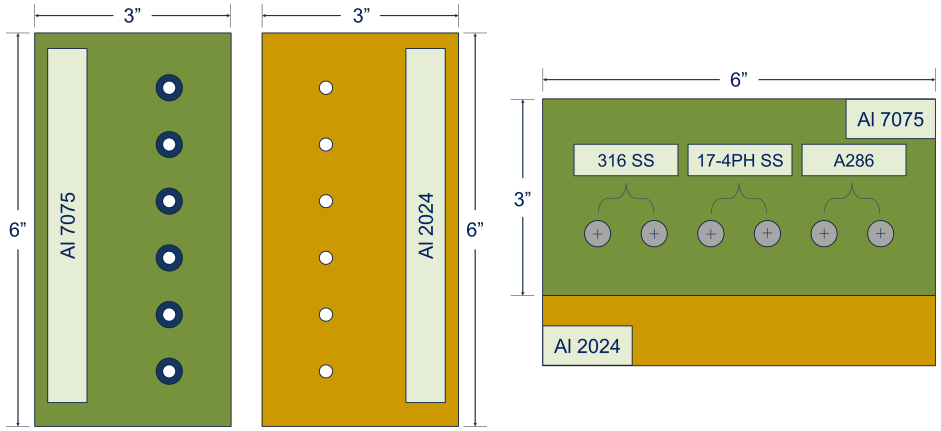
Technical Approach

- Task 1. *Optimization of Cycle Period*
  - Samples designed during SERDP WP-1673 will be used in this program
  - Samples include a lap-joint and fasteners to mimic non-ideal in-service equipment configurations



  - For aircraft applications, the plates will be made of 2024 and 7075
  - MIL-PRF-23377 class C will serve as a chromate control. A MIL-PRF-23377 class N and MIL-PRF-85582 class N primer will be tested to provide data on two non-chromated primers
  - Fasteners will be SS316, 17-PH, and A286

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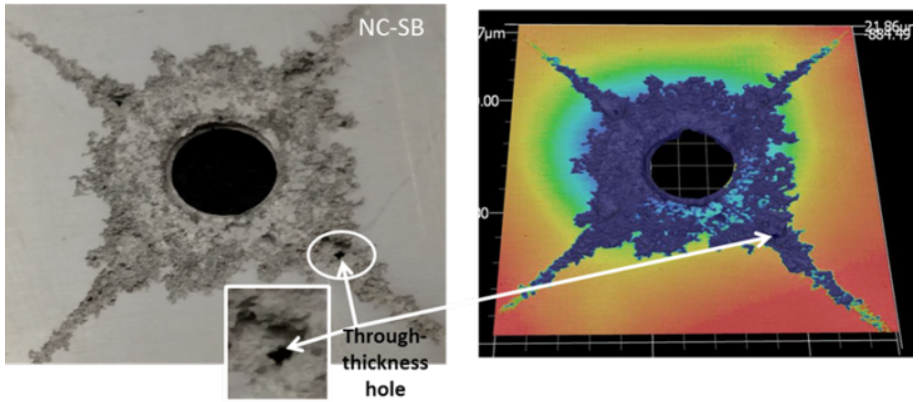
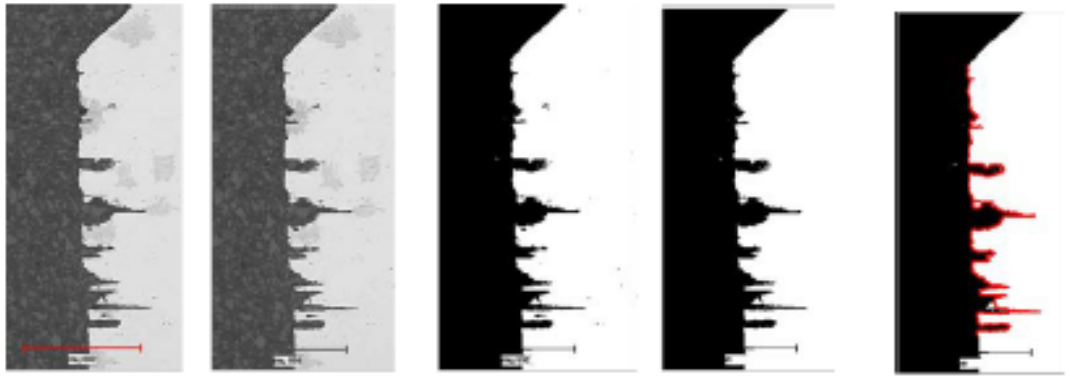
Technical Approach

- Task 1. *Optimization of Cycle Period*
  - The primary technique to quantify damage will be image analysis performed on 3-D optical images captured using an optical profilometer. This analysis will be utilized to generate ordinal rankings of coating performance and environmental severity. For exfoliation damage, image analysis and quantification will be utilized.



  - Go/No-Go Criteria:

Performance Objective	Data Requirements	Success Criteria
Observe similar corrosion damage modes	• Image analysis of test coupons and fielded samples • Mass-loss test coupons	*Positive correlation of damage mode between accelerated and outdoor exposure samples*
Decrease complexity and duration of testing	• Image analysis and mass loss measurements of test coupons and fielded samples • Time, equipment, and chemicals required to prepare test solution • User feedback	*Meet or exceed one year's corrosion damage in 1,000 test hours or less.* *No special mixing techniques required.*
Simulate both moderate and severe exposure conditions	• Image analysis of test coupons and fielded samples • Mass-loss test coupons	*Observe correlation between corrosion severity and damage mode for severe and moderate test conditions.*



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Technical Approach

- Task 2. *Round Robin Testing*
  - This task constitutes the centerpiece of the program. The **objective** of this task is to determine the amount of **variation** that should be expected from this test method
  - **Eight testing laboratories** will participate (NAVAIR, SwRI, Boeing, ARL, AMCOM, AFRL, AFLCMC, Q-Lab)
  - Eight testing labs is the recommended minimum number of labs for **ASTM E691** inter-laboratory studies to improve the accuracy of statements of precision (six is absolute minimum)
  - At the completion of testing, **repeatability and reproducibility** estimates will be obtained following ASTM E691 to determine test precision
  - Specimen material and coating systems will include common coating systems used on military **aircraft** and tested in Task 1(MIL-PRF-85582 Class N, MIL-PRF-23377 Class C and Class N) and ground assets
  - Specimens for **ground-based equipment** will include a steel and aluminum panels and will use MIL-DTL-53022 Type IV, and MIL-PRF-32550 Form A coatings
  - This round robin task is crucial for the development of the specification since it will provide the **inter- and intra-laboratory statistics** that will support the standard

Technical Approach

- Task 2. *Round Robin Testing*
  - Laboratories performing this testing will be provided with a testing chamber capable of providing the necessary relative humidity control. C-RH chambers will be obtained from Q-Lab
  - Participating laboratories will run a 500 to 1000 hour test on the provided specimens, and return all specimens and test data for analysis at the completion of testing
  - The moderate and severe test cycles are defined in Task 1

Performance Objective	Data Requirements	Success Criteria
Determine Lab to Lab Variability	• Image analysis and mass loss measurements of test coupons to determine severity of induced corrosion • RH/T logs from chamber and from sensors recorded during corrosion testing • Statistical analysis of induced corrosion and RH/T variability	*No participating laboratories are considered outliers using the outlier test in ASTM E691*

Technical Approach

- Task 3. *Standard Development and Acceptance*
  - The **objective** of this task is to **formalize** the test procedure into a NACE International laboratory accelerated corrosion test standard
  - A society standard offers several advantages over a MIL-STD:
    - OEMs can participate in development and drafting of the standard, thus reducing resistance to acceptance
    - Society standards require revisions every three to five years. This means the standard can be updated as new technical developments are made.
    - If DoD components wanted a MIL-STD version, it is easier to convert a pre-vetted standard
  - The approach is to create a NACE Task Group (TG) within STG 41 (Electric Utility Generation, Transmission, and Distribution) to drive the development of the specification
  - Victor Rodriguez-Santiago (PI, NAVAIR), Sean Fowler (Q-Lab), James Dante (SwRI, PI from WP-1673) and Kris Williams (Boeing, Key personnel on WP-2521) will serve on the TG as representatives from this ESTCP effort
  - The front end of the specification will define the overall testing approach
  - Specific environmental test cycles will be included within the annex section of the specification
  - Because of the flexible nature of the specification, additional annexes can be created to include other types of environments

Technical Progress

- Installation of Q-Fog Chamber
  - Complete 4/26/2019
- Samples painted, assembled, scribed, massed, and shipped to SwRI
  - Complete 10/7/2019
- Chamber Performance Characterization
  - In Progress
- Task 1 Test Method Design
  - In Progress
  - Performers responsible for test method design meeting 11/14/2019 to decide Task 1 testing protocol.

Technical Progress – Test Performance Monitoring Techniques

- Temperature and Relative Humidity
  1. Independent sensors co-located with test assemblies mounted at test assembly angle
     - Estimate of the local temperature and relative humidity collocated with test assemblies
     - Estimate of spatial variance in temperature and relative humidity
  2. Q-Fog wet and dry bulb measurement located in blower module
     - Estimate of bulk average temperature and relative humidity

- Salt/Solution Deposition
  1. Funnel and graduated cylinder with filter paper
     - Filter paper provides poor estimate for cumulative salt deposition weight at long exposure times
     - Graduated cylinder provides solution collection rate estimates and minimizes evaporative effects
     - Normalized to collection area of 80 cm^2
  2. Plexiglas panels mounted at the same angle as test assemblies
     - Provides estimated salt deposition on an angled surface which has the opportunity for solution run-off
  3. Spatial uniformity
     - A combination of the two collection methods provides an estimate for spatial variance in salt deposition within the chamber

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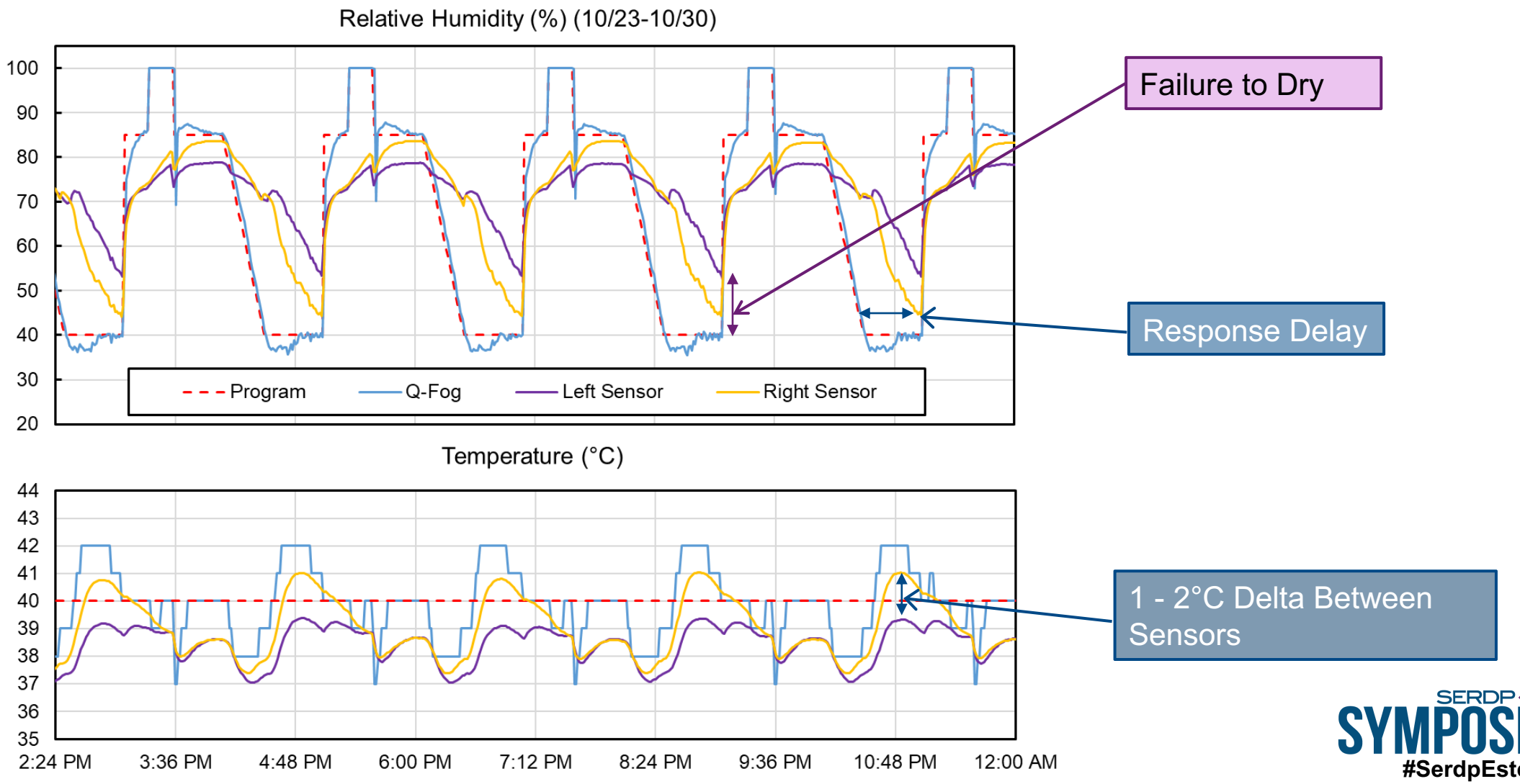
Test Performance Monitoring – Temperature and Relative Humidity

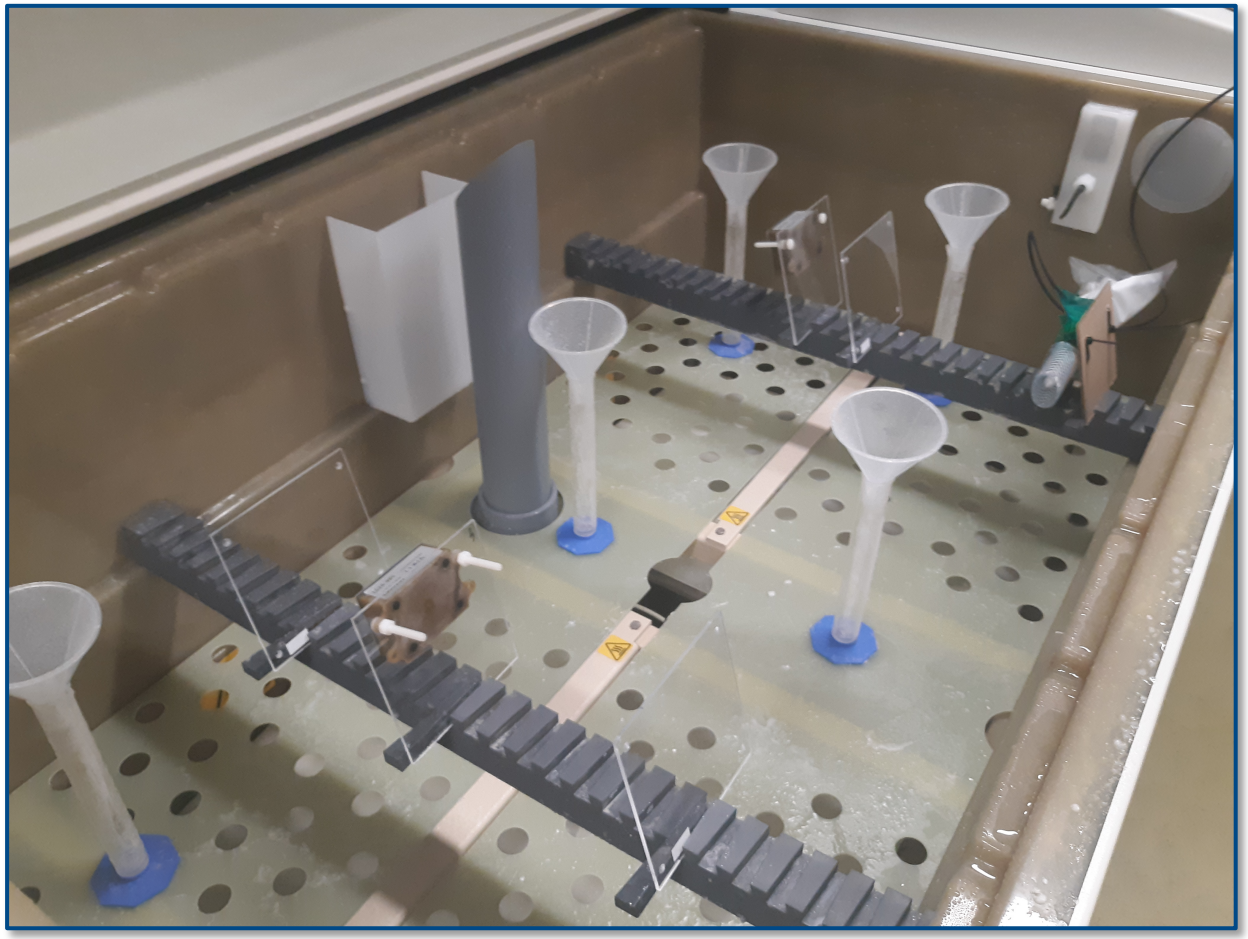


Technical Progress – Solution Collection

Max RH %	Fog or Spray	Time	Volume (mL)
80%	Fog	24 hr	0
80%	Fog	48 hr	0
90%	Fog	24 hr	3
80%	Spray	48 hr	6

10 second spray collects the same amount of solution as fog at 90% RH

Collection is consistent with other cyclic tests



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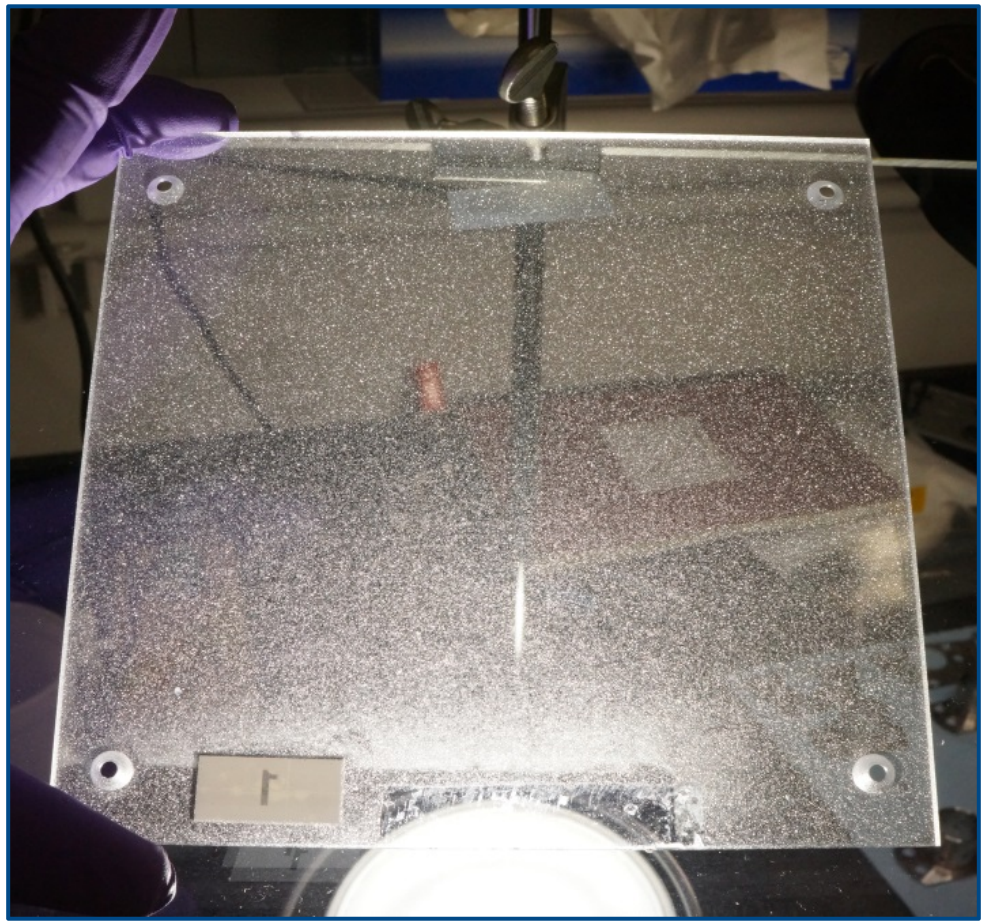
Technical Progress – Salt Deposition

Expt	Max RH %	Fog or Spray	Time	NaCl (g)
1	80%	Fog	24 hr	0.2217
2	80%	Fog	48 hr	0.6567
3	90%	Fog	24 hr	0.2833
4	80%	Spray	48 hr	0.5950

Salt deposition increase is non-linear (1&2)
Increase in humidity increased salt deposition (1&3)
Spray did not substantially decrease salt deposition (2&4)
Need to control salt deposition another way – Fog intermittently?



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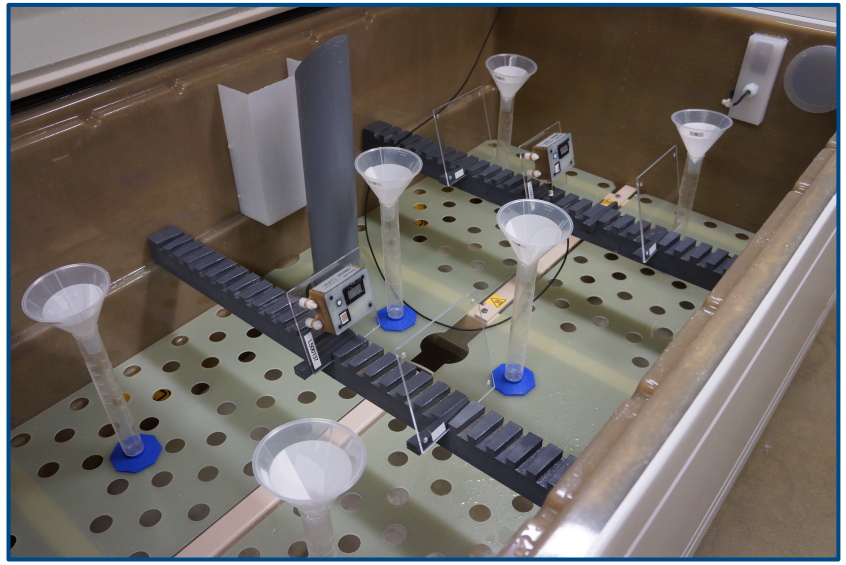
Test Performance Monitoring – Salt/Solution Deposition

Exp #	Fog/Spray Duration	# Cycles	Mass Collected by Plexiglas (g/m^2)
1	F: 15 min	12	28 ± 4.1
2	F: 30 min	12	42 ± 4.3
3	F: 15 min	71	29 ± 1.2
4	S: 10 sec	12	29 ± 2.4
5	S: 10 sec	57	28.5 ± 0.7



1 – 2: Doubled fog duration = increased total mass collected by Plexiglas 1.5 times.

Variability of each measurement method is the same between each experiment.

1 – 3: Increase in # cycles had no effect on the mean mass collected by Plexiglas.

Variability of each method is lower than those collected at shorter # of cycles.

1 – 4: 10 second spray = 15 minute fog. Variability half of that in fog.

4 – 5: Variability decreased as a function of exposure duration

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Future Work

- Additional test performance monitoring techniques will be investigated prior to initiation of Task 1 testing. The results from this will be used to inform the Joint Test Protocol and Demonstration Plan sections related to pre-demonstration testing and analyses.

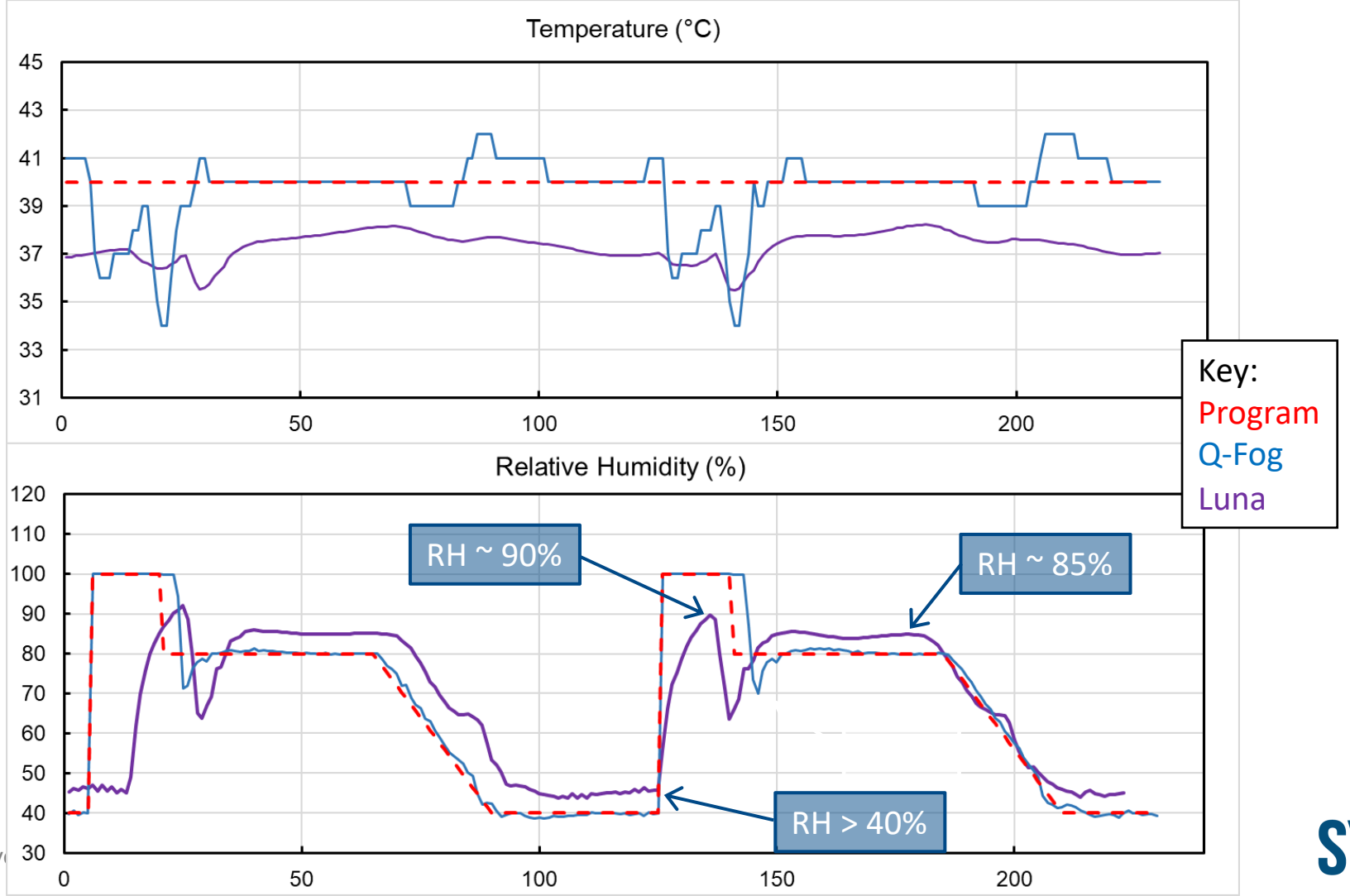
- Task 1 testing at SwRI and NAVAIR will begin shortly.

- Results will be analyzed by SwRI, and a Go/No-Go decision will be made based on project success criteria.

Thank you

Questions?

No Precondition, 15 min Fog, 80% Max RH



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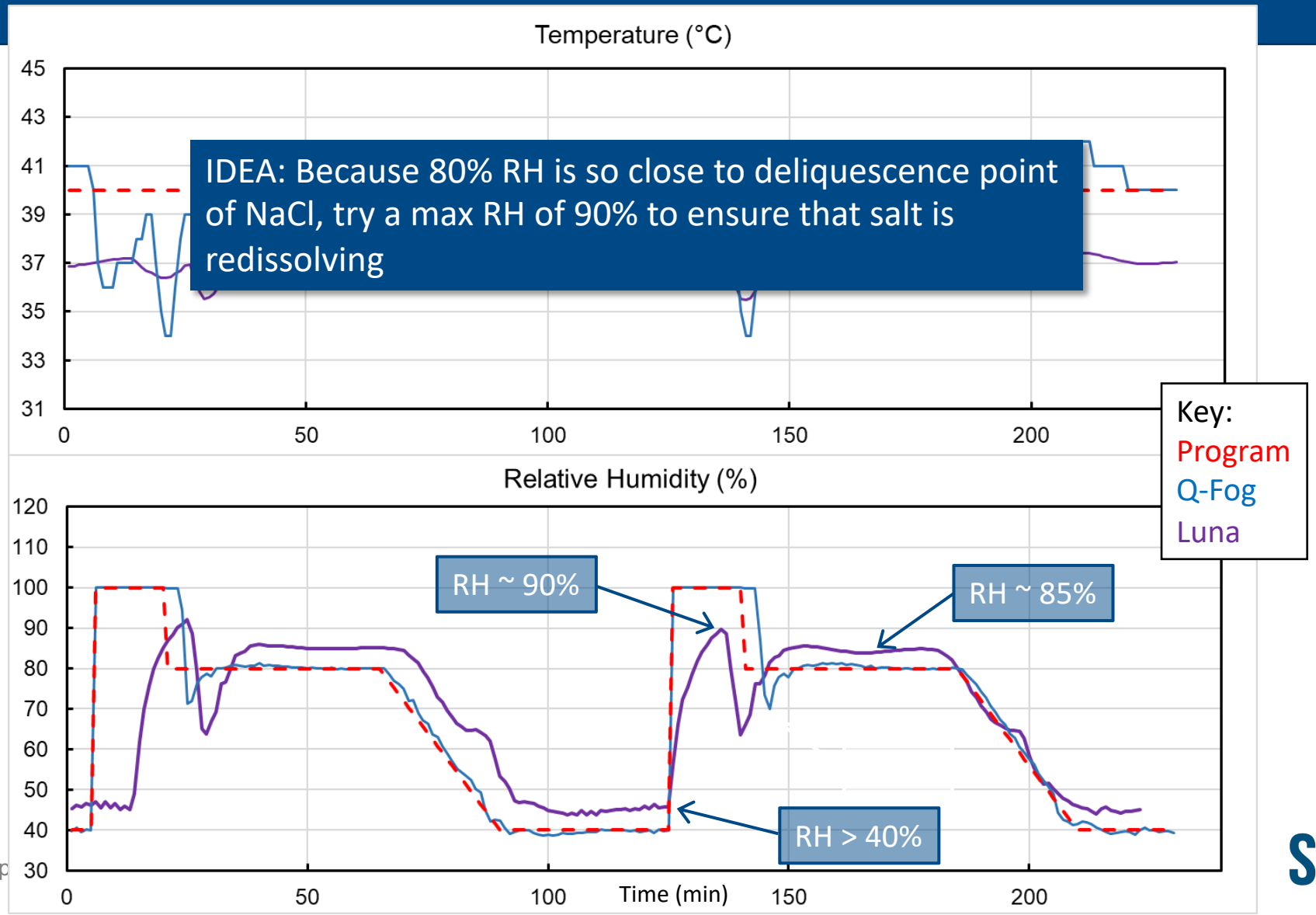
No Precondition, 15 min Fog, 80% Max RH



IDEA: Because 80% RH is so close to deliquescence point of NaCl, try a max RH of 90% to ensure that salt is redissolving

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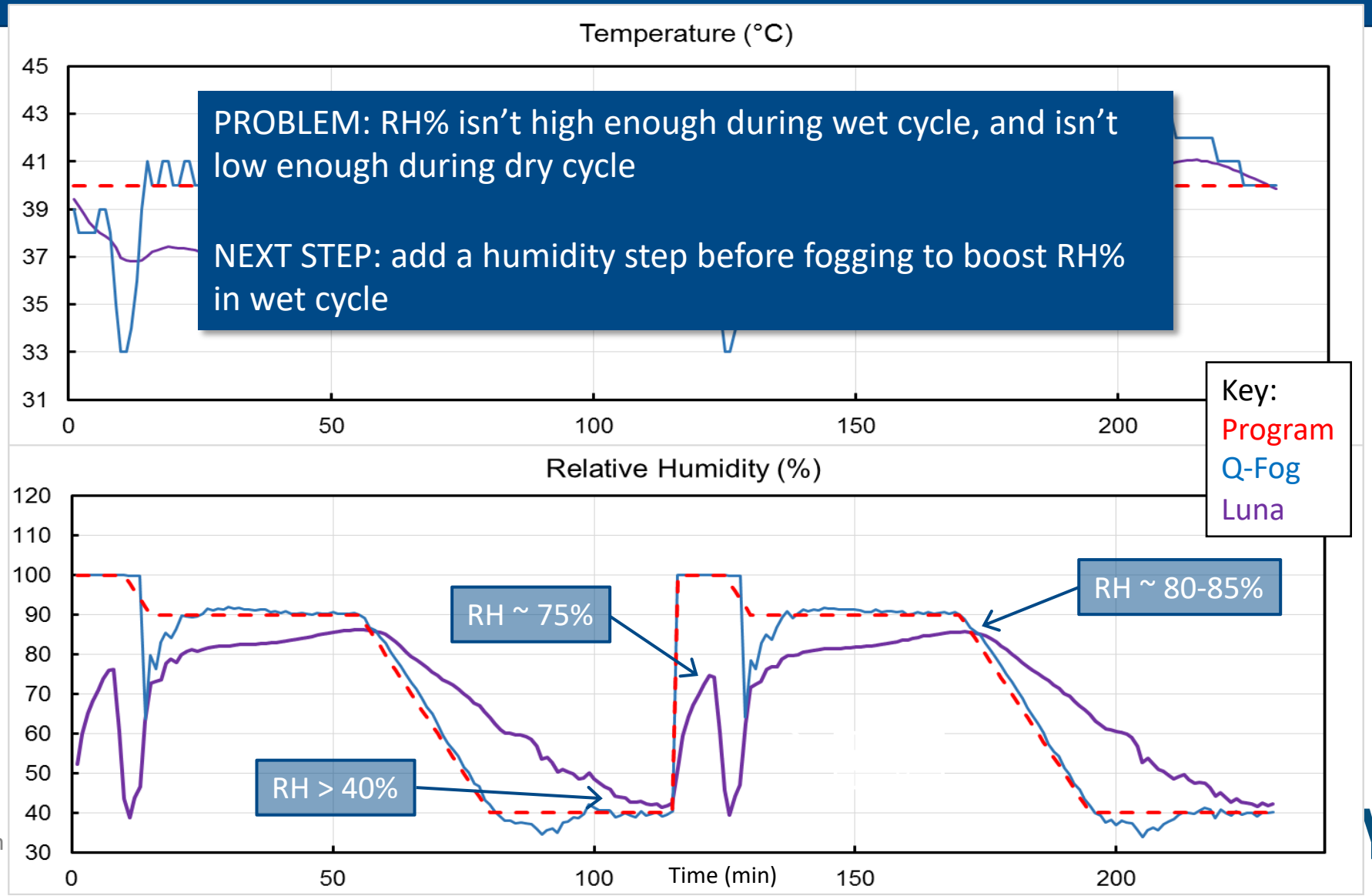
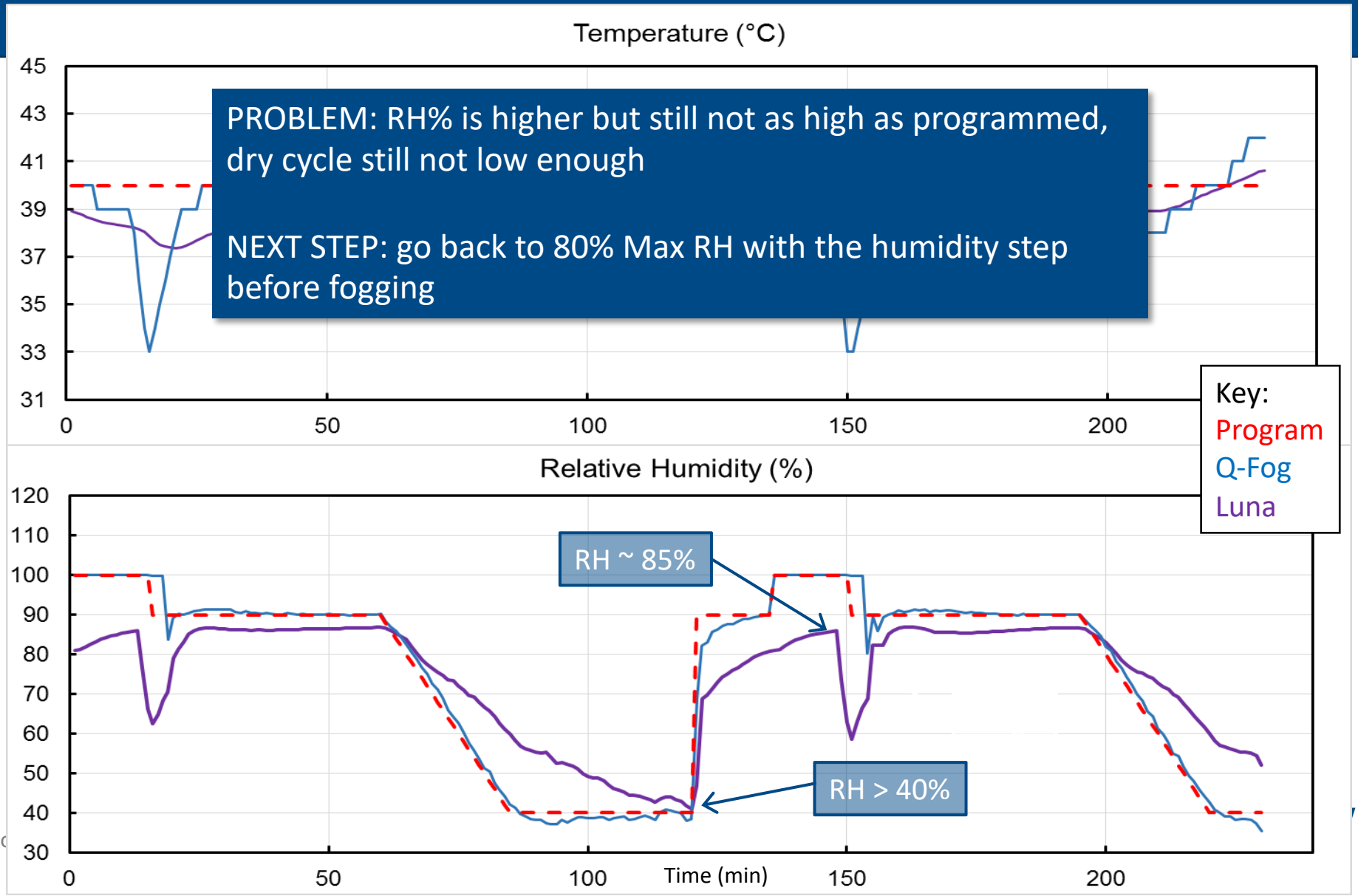
No Precondition, 15 min Fog, 90% Max RH



Distribution

90% RH Precondition, 15 min Fog, 90% Max RH



PROBLEM: RH% is higher but still not as high as programmed, dry cycle still not low enough

NEXT STEP: go back to 80% Max RH with the humidity step before fogging

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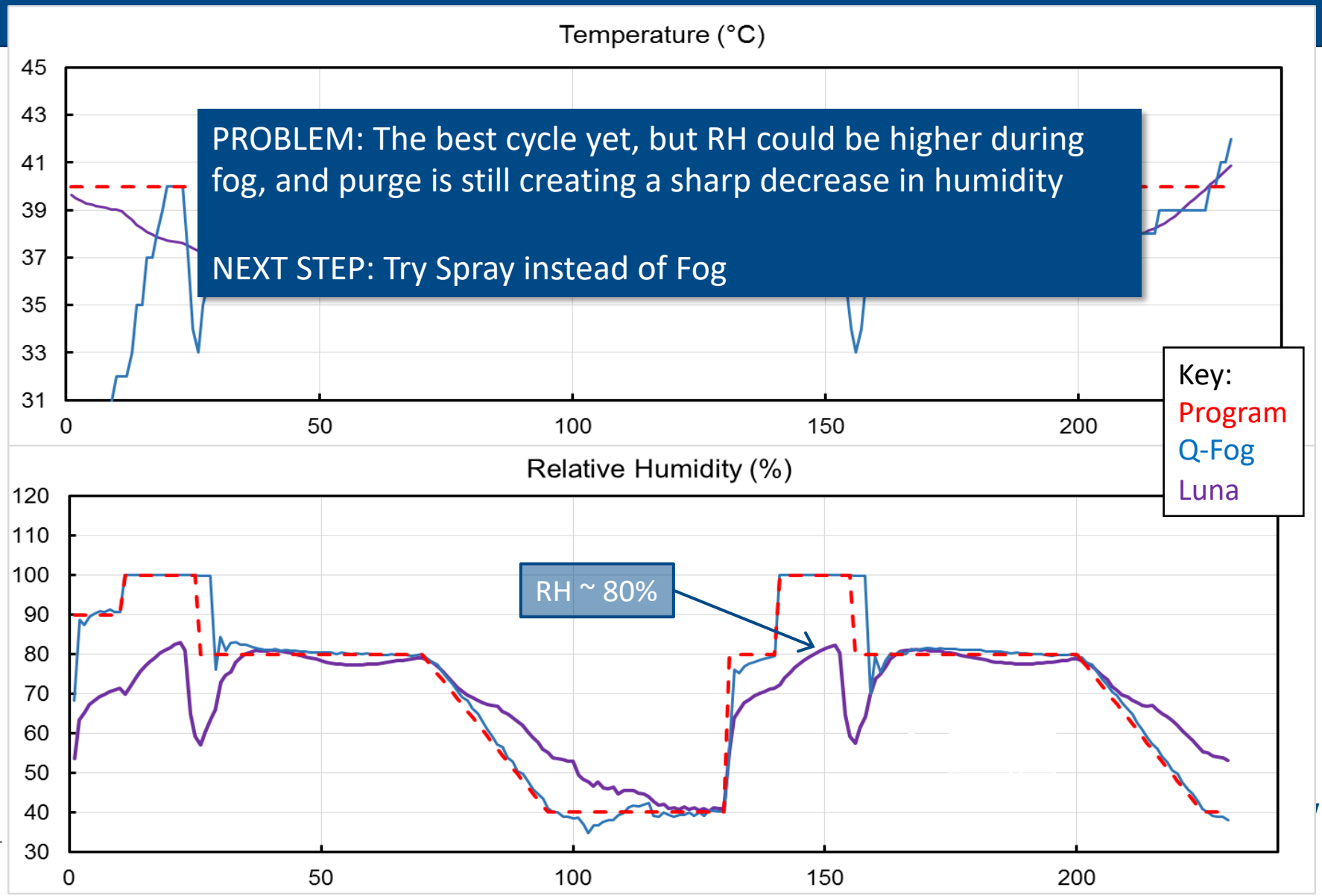
80% RH Precondition, 15 min Fog, 80% Max RH



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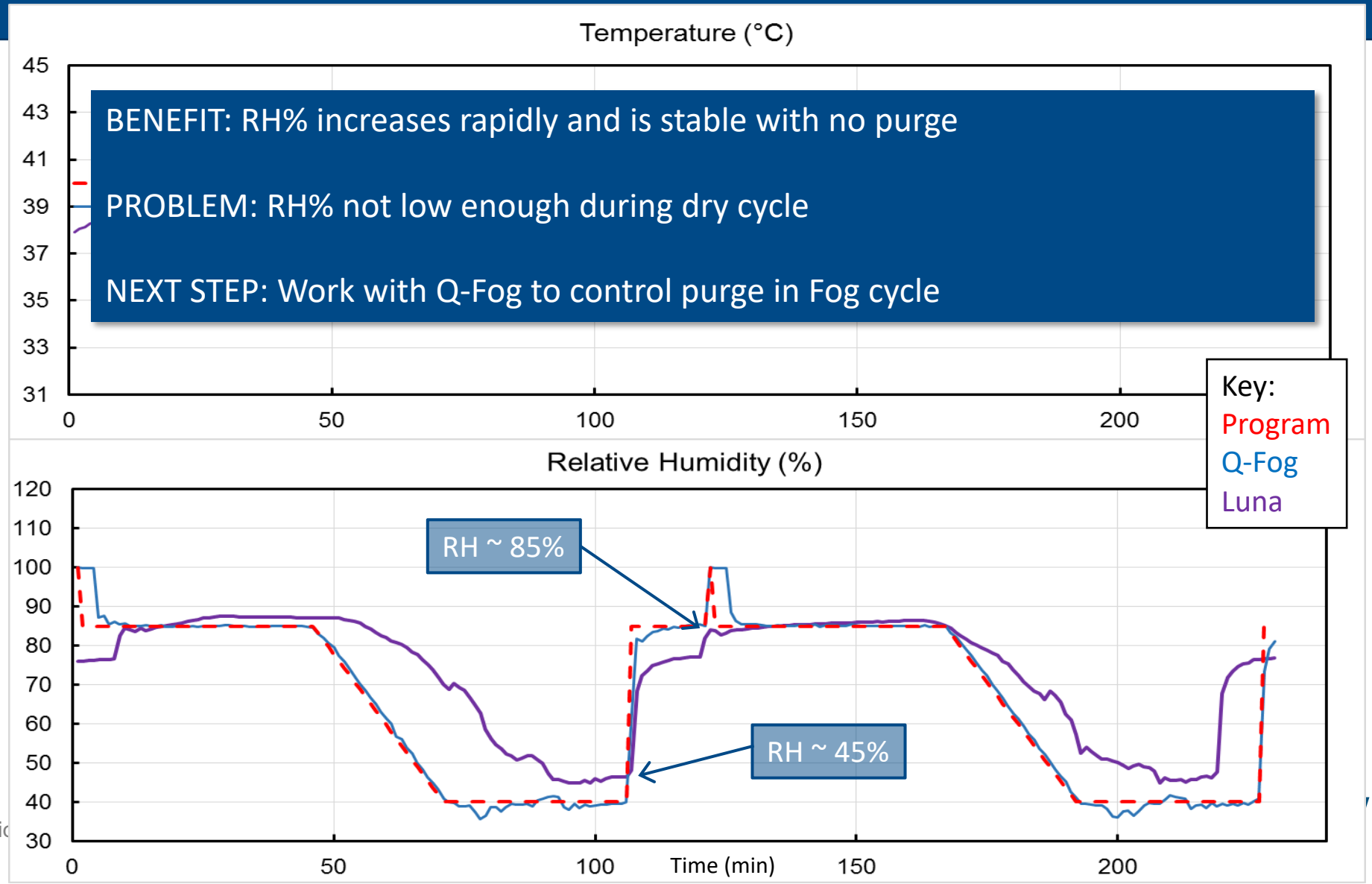
85% Precondition, 10 sec Spray, 85% Max RH



BENEFIT: RH% increases rapidly and is stable with no purge

PROBLEM: RH% not low enough during dry cycle

NEXT STEP: Work with Q-Fog to control purge in Fog cycle

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Acronyms and Symbols

Abbreviation	Meaning	Unit
T, Temp	Temperature	*C
RH	Relative Humidity	%

Publications

- New NACE standard with cyclic test method expected at conclusion of project