

TECHBRIEF



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Considerations for Scalability of Alkali Concentrated Conditioning Solution

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SUMMARY

The following points provide a summary of this TechBrief.

- Artificial pore solution (APS) is a conditioning solution for use in determining formation factor in accordance with the American Association of State Highway and Transportation Officials (AASHTO) T 402.⁽¹⁾
- The use of a highly alkaline solution, such as APS, has precedence in the construction industry (e.g., for assessment of aggregate susceptibility to alkali-silica reaction). However, those applications use the solution as part of a prequalification process, not for construction quality assurance or quality control (QA/QC) programs. This TechBrief highlights the significant challenges associated with using APS in a QA/QC application.
- The goal of this document is to inform selection of repeatable, responsible, and relevant QA/QC tests for concrete materials. Formation factor can be used to predict service life but may not be practical to use for construction QA/QC. Meanwhile, resistivity may be more appropriate for construction QA/QC.
- This document discusses the considerations necessary for scaling APS from small numbers of bucket-sized volume solutions, which have been demonstrated in the research environment, to tank-sized volumes or large numbers of bucket-sized solutions necessary for larger scale QA/QC operations during construction operations.
- The scalability of APS introduces several challenges to the industry and requires technical, practical, and chemical considerations. Technical considerations include the APS ion equilibration with multiple concrete pore solutions and solution-to-sample volume ratios. Construction-style proportioning, transportation, space, and maintenance considerations can affect the feasibility of large volumes of APS. Chemical considerations present significant challenges in terms of personnel health and safety.
- To safely develop large volumes of APS, personnel should consider:
 - Using personal protective equipment (PPE), including well-fitting chemical respirator face masks and filters, safety glasses, gloves, appropriate clothing, and safety boots.
 - Mixing solutions outdoors or in well-ventilated areas.
 - Using nonmetallic tools.
 - Using ice to replace a portion of water by mass.
 - Incrementing chemical introductions into the water.

- Monitoring the solution temperature while chemicals are added.
- Safely storing, handling, and disposing to prevent negative effects to worker safety and the environment.
- Determining disposal cost and resources.
- Scaling APS for testing facility use is, overall, possible but involves new, thorough considerations for facility operations in addition to those aspects already considered for limewater (LW) usage.

INTRODUCTION

In the United States, owner agencies, consultants, and researchers are beginning to use electrical resistivity measurements, described in AASHTO T 402 and AASHTO T 358, for mixture design QA as a metric of material consistency and durability (See references 1–4). As a result, contractors, concrete suppliers, and independent laboratories are using resistivity measurements for a QC tool. For decades, concrete cylinder specimens used for resistivity and compressive strength testing have been conditioned in 100-percent relative humidity rooms or in tanks with a saturated calcium hydroxide ($\text{Ca}(\text{OH})_2$) solution, referred to as LW, to reduce leaching.^(5,6) These LW tanks, also known as “horse troughs,” use large quantities of solution to condition many specimens at one time. The tanks typically hold volumes greater than 390 L (103 gal).

In recent years, a modification to resistivity testing, known as the “bucket test,” was developed that requires conditioning concrete specimens in bucket containers with alkali-concentrated conditioning solution, also sometimes referred to as alkali or artificial pore solution (APS), or simulated pore solution.^(7–9) The APS solution outlined in AASHTO T 402 includes concentrations of 7.6 g/L (1.01 oz/gallon) of sodium hydroxide (NaOH), 10.64 g/L (1.42 oz/gallon) of potassium hydroxide (KOH), and 2 g/L (0.27 oz/gallon) of $\text{Ca}(\text{OH})_2$, which are typically used for 13.3 L (3.51 gallons) of solution per bucket.⁽¹⁾ Three 101-millimeters (mm) by 203-mm (4 inches by 8 inches) concrete cylinder specimens of the same mixture design are conditioned in one bucket, creating a solution-to-sample volume ratio of 2.7. APS reduces leaching of $\text{Ca}(\text{OH})_2$, similarly to LW, and alkali ions from concrete samples. The bucket test intends to mimic actual pore solution chemistry, which is needed to determine concrete’s formation factor.^(10,11)

Formation factor is a metric of concrete transport properties that provides an indication of the concrete’s microstructure.^(7,8,12) Formation factor is calculated by dividing the concrete’s measured resistivity by the pore

solution resistivity. Since actual concrete pore solution extraction is labor and equipment intensive, the APS used in the bucket test provides a conditioning environment that is theoretically equivalent to the pore solution within a concrete specimen. The bucket test assumes that the internal concrete pore solution and APS solution in the bucket equilibrate. Therefore, the resistivity of the solution within the bucket is assumed equivalent to the concrete’s pore solution resistivity. Formation factor can be used in service life modeling.^(13,14)

AASHTO T 402 requires conditioning in a $\text{Ca}(\text{OH})_2$ -saturated solution that is representative of a typical concrete pore solution and specifies proportions of alkaline chemicals that should be added.⁽¹⁾ However, the bucket test, which this 2023 version requirement is based on, employed only the bucket-sized quantities of solution, and the standard does not consider the scalability of using APS. Since APS would be used instead of, or in addition to, the LW conditioning solution already used in many testing facilities for performing compressive strength and resistivity testing, concrete QA testers may be interested in using APS in large volumes similar to LW tanks.

This document discusses the considerations necessary for scaling APS from small numbers of bucket-sized volume solutions to tank-sized volumes or large numbers of bucket-sized solutions. Scaling APS requires care for technical, practical, and chemical considerations that can affect:

- Assumptions supporting formation factor.
- Large-volume solution feasibility.
- Health and safety of personnel.

TECHNICAL CONSIDERATIONS

Equilibration

The bucket test assumes that the concrete’s internal pore solution equilibrates with the solution in the bucket. However, when concretes of multiple different types and compositions are equilibrating within the same solution, ion exchange may occur between concrete specimens in addition to within the APS. This ion exchange between concretes of different types may affect the resulting resistivity values and the calculated formation factor. Therefore, a new solution should be used for each set of specimens, especially those having a different mixture design or being of a different batch. For tank-sized volumes of solution, the industry may intend to adapt the requirements for LW curing tanks written in ASTM C511 to APS solution.⁽⁵⁾ However, research has not yet been published investigating the effect on formation factor of having concrete specimens from different batches or of different types within one solution.

For equilibration to occur, the pore network of the concrete must allow the movement of ions into and out of its bulk material. For dense concretes with high particle packing, small pores, and a disconnected pore network, this assumption of equilibration may not be reality. Ultra-high performance and high-performance concretes are among those less likely to equilibrate with their surrounding solution because of their enhanced resistance to ion penetration and migration.^(15,16)

Pore Solution Composition

The chemical composition used for the bucket test and, therefore, within the APS solution specified in AASHTO T 402 was developed based on a 2016 multilaboratory study conducted with State department of transportation labs from around the country on concretes used in transportation projects.^(1,10) The concrete mixture designs consisted of a majority of ordinary portland cement and traditional supplementary cementitious materials (SCMs) such as fly ash and slag cement.^(7,10,11) As the industry moves toward using portland limestone cement instead of ordinary portland cement and the use of nontraditional SCMs expands, concrete mixtures will likely have different chemical compositions that may require reevaluation of the representative pore solution. The chemical composition currently specified for developing APS may no longer adequately simulate a typical concrete pore solution. Also, with the advancement and increasing replacement percentages of novel materials, the industry may no longer be able to represent most concrete mixtures using a single APS composition.

Solution-to-Sample Volume Ratio

Studies have illustrated the role of the volume of solution surrounding the test specimen and its impact on resistivity measurements when cured in LW.⁽¹⁷⁾ However, to date literature does not indicate whether

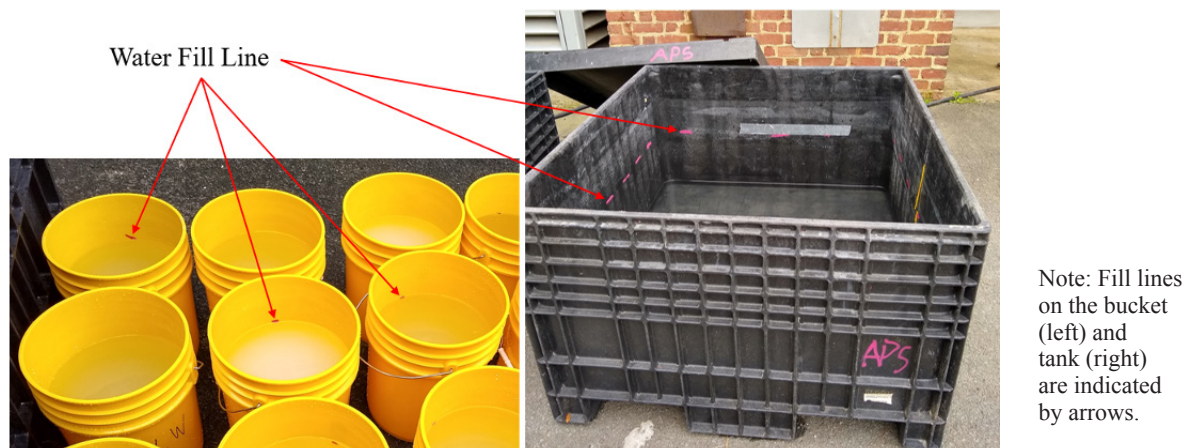
the storage solution volume when using APS can affect the formation factor results, through either the measured concrete specimen resistivities or the solution resistivity. The solution-to-sample volume ratio may influence the resistivity values of the concrete and solution. Previous versions of AASHTO T 402 (AASHTO TP 119) specified a solution-to-sample volume ratio between 2.0 and 3.0.⁽¹⁸⁾ While a 2.7 ratio holds for three concrete cylinders within one bucket, maintaining this ratio within a tank is more difficult, especially if concrete specimens are continuously rotating in and out of the tank as they age. The change in solution-to-sample volume ratio may affect the calculated formation factor.

PRACTICAL CONSIDERATIONS

Construction-Style Proportioning

Minute differences in chemical proportions and dilution can change the solution conductivity. Exacting chemical and water proportions are needed to achieve a target solution conductivity consistent with that of the bucket test. Including more or less water than specified is a likely outcome for construction industry-type applications when developing large volumes of solution, such as a tank of 390 L or numerous buckets. The process of weighing out that specific amount of water would be time intensive. Testing laboratories would either need a scale that can weigh the entire tank as water is added, or smaller quantities of water would need to be measured and added to the tank. More likely, to reduce time and labor involved, testing laboratories would determine, based on tank or bucket dimensions, the height level to which water should fill the container, as depicted in figure 1. However, even with careful calculations and measurements, consistency is difficult to obtain, resulting in concentration variability. During a recent Turner Fairbank Highway Research Center study, with control and precision during development of

Figure 1. Photograph. Construction-style proportioning of water with water fill lines on buckets and on a tank.



Source: FHWA.

the APS solutions, a collection of 24 buckets containing 8 different concrete mixture designs obtained a solution conductivity variability of 21 percent. While LW is currently used in large volumes, a strictly $\text{Ca}(\text{OH})_2$ saturated solution does not require the same level of precision needed for calculating formation factor from APS-conditioned concretes, nor is the same amount of variability possible given the nature of adding only one ingredient until the water has reached saturation.

Transportation

Consider, however, the feasibility of moving a large volume of solution from one location to another. If the solution is mixed outdoors, it will likely need to be moved inside a laboratory for use. Any transportation of the solution should involve a well-fitting lid over the container, a plan for containing and cleaning spills or splashes, and consideration of the effects of a sloshing liquid on the load distribution.

Space

Using large volumes of APS for specimen conditioning and storage increases space requirements. Testing facilities have limited space available for specimen storage and conditioning. The expenses associated with expanding the available space to store dozens and potentially hundreds of 18.9-L (5 gal) buckets would be extensive. One 390-L (103 gal) tank can potentially hold up to 210 concrete cylinders 101 mm in diameter by 203 mm high (4 inches by 8 inches), while only 16 buckets or 48 of the same-sized concrete cylinders are able to fill the same amount of space. These facts equate to a bucket storage efficiency of 22.9 percent of the tank storage capability. Figure 2-A and figure 2-B display the disparity in space requirements between the numerous buckets or a single tank needed to condition concrete specimens.

Figure 2. Photographs. Disparity in space requirements between buckets and a tank.



A. Photograph. 47 buckets needed to condition 141 concrete cylinder specimens in APS.

B. Photograph. A single tank that can condition up to 210 concrete cylinder specimens.

Source: FHWA.

Maintenance

If using a tank of solution, that solution should be replaced at regular intervals. ASTM C511 for LW tanks specifies cleaning and refilling each tank with potable water and 3 g/L of $\text{Ca}(\text{OH})_2$ at intervals no longer than 24 mo.⁽⁵⁾ The same standard requirements apply to all water storage tanks, which includes both LW and APS. The addition of APS tanks would require complete cleaning of equipment and remixing of solution at regular intervals.

In addition, for reasons to be explained in the Reactivity section, any equipment needed to maintain temperature and circulation of the solution should not have metal components. While plastic tools and equipment can perform the same duties, care must be taken to ensure that the correct materials are used.

Temperature

Large volumes of solutions often present challenges to maintain specified temperature. Covering the solution can help reduce evaporative cooling. However, bucket lids or covers for tanks can be one additional item that requires maintenance. Heating, ventilation, and air-conditioning systems are often powered down when the workforce is out of the testing lab, which can cause conditioning or testing outside of ranges allowed by the test method. In LW systems, this phenomenon is often mitigated using a plug-in heater to maintain temperature, but the caustic nature of APS has been demonstrated to quickly destroy traditional immersion heaters.

CHEMICAL CONSIDERATIONS

Due to the nature of the chemicals used to develop an alkali-concentrated conditioning solution, health, safety, and reactivity considerations should be reviewed before developing a large volume of solution.

Chemical Volume

For a typical bucket test, the AASHTO T 402 APS proportions equate to 13,250 g of water, 143.9 g of KOH, 102.6 g of NaOH, and 27 g of $\text{Ca}(\text{OH})_2$.⁽¹⁾ However, a tank of APS totaling 390 L requires 388,533.8 g of water, 4,219.6 g of KOH, 3,008.6 g of NaOH, and 791.7 g of $\text{Ca}(\text{OH})_2$. The chemical amounts were linearly scaled to maintain the same proportioning while achieving a large-volume APS. This large amount of chemicals is shown in figure 3. For comparison purposes, LW tanks use only a single bowl of the chemicals pictured in figure 3, to which the arrow points.

Figure 3. Photograph. Chemicals used to develop one tank of APS, along with the single bowl of Ca(OH)₂ needed to develop a tank of LW.



Note: Arrow shows the single bowl of Ca(OH)₂.

Source: FHWA.

Health and Safety

The powder chemicals used to develop APS can be health hazards if the proper precautions and PPE are not considered. Table 1 summarizes the health hazards presented in the safety data sheets for each chemical found in an AASHTO T 402 APS solution.⁽¹⁾

For the chemicals listed in table 1, the safety data sheets recommend not breathing the dust or vapors, washing exposed skin thoroughly after handling, and avoiding release into the environment.^(19–21) Recommended PPE includes wearing eye protection, face protection, protective clothing, and protective gloves.^(19–21) Once

the reactions occur, the resulting APS is highly alkaline, which can cause skin and eye irritation upon contact.

To address these health and safety concerns, personnel handling or within the general area of these chemicals should wear the following PPE: a well-fitting chemical respirator face mask, safety glasses, nitrile or latex gloves, long-sleeved clothing, long pants, and steel- or composite toed safety boots. In addition, the chemicals should be handled and mixed into the solution under a fume hood or outside. These precautions are even more necessary when mixing large volumes of solution because there is a greater possibility for powder and vapors to enter the air.

For comparison, Ca(OH)₂ is the only chemical added to water to develop LW conditioning solutions. Therefore, only those health and safety risks in table 1 associated with Ca(OH)₂ apply to LW. While many of the same safety data-sheet recommendations and PPE requirements apply to LW conditioning solutions, the risks associated with LW are not as severe.

Reactivity

When these chemicals are mixed into the water to develop solution, chemical reactions occur that also can present health and safety hazards. The molecules dissociate into potassium, sodium, calcium, and hydroxide ions. However, if any metals are present in the water, KOH can react with the metals to form hydrogen gas, which is an extremely flammable gas that can burn with invisible flame and can displace oxygen. For these reasons, KOH should only be used outdoors or in a well-ventilated area.⁽¹⁹⁾ To reduce

Table 1. Health hazards summarized from safety data sheets for chemicals in APS.

Chemical	Hazard	Category
KOH	Corrosive to metals	1
	Acute oral toxicity	4
	Skin corrosion/irritation	1A
	Serious eye damage/eye irritation	1
	Specific target organ toxicity (single exposure)—respiratory system	3
NaOH	Skin corrosion/irritation	1A
	Serious eye damage/eye irritation	1
	Hazardous to the aquatic environment—acute	3
Ca(OH) ₂	Skin corrosion/irritation	2
	Serious eye damage/eye irritation	1
	Specific target organ toxicity (single exposure)—respiratory system	3

Note: Based on references 19–21.

the possibility of producing hydrogen gas, no extraneous metals should be introduced to the solution. The container, stirring implements, thermometer, and other tools should be composed of plastic, glass, or another nonmetal, as shown in figure 4.

Upon introducing APS chemicals to water, the molecules disassociate and generate heat. At smaller volumes typical of the bucket test, the increase in temperature that results from the chemical reaction is minimal. However, the solution's temperature change is significant when mixed in large volumes. Care should be taken to ensure that the temperature of solution remains low during reactivity and that the solution container will not be damaged as a result of the heat generated. Plastic, especially polyvinyl chloride (PVC) plastics used in many construction applications, has a relatively low melting point of only 75 °C (167 °F).

To reduce the solution temperature, a portion of the water mixed into an APS tank can be accounted for by a mass of ice, as depicted in figure 4. Another technique for reducing the solution temperature is to add the entire volume of water before adding any chemicals, and then slowly introducing partitioned increments of chemicals. The charges on the potassium, sodium, and hydroxide ions are more stable when surrounded by greater numbers of water molecules than when fewer water molecules surround the dissociated ions. Therefore, providing plenty of water when introducing the chemicals can dissipate the effects of exothermic reactions more quickly. Monitoring the solution temperature can help personnel determine when the next increment of chemicals should be added.

For comparison purposes, the chemical reactions that develop LW are more stable and less exothermic than those that occur to develop APS. Therefore, extreme reactivity precautions are not needed. Also, because there is no KOH in the LW system, there is a low likelihood that hydrogen gas will form.

Figure 4. Photograph. Reactivity precautions that must be taken to develop large volumes of APS, including ice to reduce the temperature of the solution and a plastic shovel to stir without introducing metals to the solution.



Source: FHWA.

Environmental

Table 1 mentions hazards to human health and safety and to aquatic life. The solution and its chemicals cannot be poured down a drain or released into the environment. Care must be taken to ensure the proper protocols are followed for handling, storing, and disposing of such solution. Failure to follow appropriate chemical procedures can result in contamination of water supply for humans and wildlife. Such a high-alkaline solution, especially in large volumes, can increase water pH. For humans, drinking or using high-alkalinity water can result in bitter taste, upset stomach, skin irritation, and eye irritation.⁽²¹⁾ High water pH can also lead to calcium and magnesium carbonate buildup in pipes and possibly result in heavy metal toxicity.

High-alkalinity water significantly affects aquatic life. Minor increases in pH can reduce dissolved oxygen levels in water, killing fish and plant life, as well as affecting the solubility and toxicity of chemicals and heavy metals in the water.^(22,23)

Disposal

Once finished with an investigation that used APS, the solution must be disposed of. With a large volume of APS, either tanks or numerous buckets, disposing of the highly alkaline solution poses a difficult task. The health and safety hazards to humans and aquatic life already mentioned in the Reactivity and Environmental sections suggest that specific chemical disposal procedures are needed.

Few options exist for disposing the solution. The tank can be opened in a well-ventilated area and placed in front of fans to evaporate off the liquid, leaving the chemical deposits and minimal liquid for chemical waste disposal, or the entire solution can be disposed of. In either option, a chemical waste company will need to be contracted to pick up and dispose of the solution. Evaporating liquid off involves less solution and is therefore less costly, but it requires more space for a longer time and involves a higher concentration of chemicals in the final waste solution. Getting rid of the entire solution volume is more costly due to the quantity of material. However, the availability of this service may be limited or require additional cost, depending on location.

The solution may also be neutralized with an acid and then disposed. However, this option involves the purchase of more chemicals and development of another solution. The addition of an acid will also increase the volume of material waste. Between the chemical cost and waste disposal, neutralizing the solution may be more costly than disposing of the initial, caustic solution.

LW has a lower pH, ranging from 9 to 11.5, compared with APS, which has a pH of approximately 13. Because pH measurements are based on a logarithmic scale, the difference in pH between LW and APS is significant. As a result, the environmental and safety concerns are lower

for LW. Additionally, LW is much easier to neutralize or dilute and then dispose of.

APS DEVELOPMENT CHECKLIST

The following checklist describes the considerations to account for when using the bucket test on a larger scale, in a high-volume tank or in many small-volume buckets:

- Plan for a large volume of space to store the solution in a temperate environment.
- Plan for the labor and space requirements needed to adequately develop the solution at regular intervals.
- Review the safety data sheets for each chemical before developing solution.
- Personnel measuring out chemical amounts and in the vicinity of the solution, during development and afterward should wear a well-fitting P100 chemical respirator face mask, safety glasses, nitrile or latex gloves, long-sleeved clothing, long pants, and steel- or composite-toed safety boots.
- Accurately and precisely measure chemical and water quantities.
- Personnel should wash their skin with soap and water immediately if the solution makes direct skin contact.
- Develop the solution in a container with a lid and plan for the facility's ability to move the amount of solution created. Do not use a metal container.
- Do not introduce metal into the solution to prevent the development of hydrogen gas. Pay particular attention to be sure no metals are present in stirring implements, thermometers, temperature-monitoring equipment, or solution-heating equipment.
- Consider the fumes and caustic nature of the chemicals and solution.
- Mix the solution in a well-ventilated area under a fume hood or outdoors.
- Keep flames or other flammable items away from the solution, especially during development.
- Monitor the temperature of the solution during development, particularly when adding chemicals. Consider introducing the chemicals in parts.
- Use ice to replace a portion of the water content when developing a large tank of solution. Understand the melting point of the solution container and development tools to ensure the solution temperature does not damage the container or tools, especially in the case of PVC containers.
- Ensure a constant solution-to-sample volume ratio.

- Verify that the APS composition adequately reflects the actual composition of the pore solution within the concrete of interest.
- Minimize mixing concrete specimens of different types within one container of solution.
- Ensure that each sample remains in the solution long enough to promote pore solution equilibration.
- Do not pour the solution down any drains.
- Prepare a plan for disposing of the chemicals and solution without contaminating the environment and considering the high pH nature of the solution.
- Consider the costs associated with additional chemical purchases and special chemical waste disposal of the solution.

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