WATER CHEMISTRY EFFECTS ON THE CRYSTALLIZATION TEMPERATURE OF LIQUID FERTILIZER

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Abstract. Liquid (solution) fertilizers are commonly used to fertilize Florida citrus and vegetable crops. Crystallization (salt-out) of these liquids in storage tanks can be a problem during the cool season (late December through early March). The objective of this research was to determine the effects of water quality, micronutrient addition, and polyphosphate addition on the salt-out temperature of 10-0-10, 8-0-8, and 6-0-6 liquid fertilizers made with NH₄NO₃ plus KCI or NH₄NO₃ plus KNO₃. Three water sources were used to make the fertilizers: Deionized (3 ppm TDS), Immokalee well water (560 ppm TDS), and LaBelle well water (2550 ppm TDS). Micronutrient addition included 0.25% Mg, 0.05% Mn, 0.03% Zn, and 0.01% B. Polyphosphate was added to the liquid at rates of 100, 700, or 1250 ppm, either before or after mixing the fertilizer salts. The salt-out temperature of 10-0-10 fertilizer was reduced approximately 3.5° F when mixed with deionized water as compared to the other waters. There was little difference in salt-out temperature between the well waters. Average salt-out temperatures were 26.5, 41, and 59° F for the 6-0-6, 8-0-8, and 10-0-10 fertilizer grades, respectively. Blends made with KNO₃ salted-out 2 to 6° F lower than blends made with KCI. Addition of micronutrients had minimal effect on salt-out temperature. Polyphosphate lowered salt-out temperature from 2 to 7° F, and was more effective when added last. Fertilizer salt-out can be avoided by lowering the analysis, using KNO₃ in place of KCI, or adding polyphosphate to the blend.

Liquid (solution) fertilizers are manufactured by dissolving solid fertilizer salts in water. Use of these fertilizers is regarded as a relatively new practice, beginning in the mid-20th century (Young and Hargett, 1984). Benefits of their use include ease of handling, versatility of application, and uniformity of blends. Liquid fertilizers have become popular in Florida due to the increase in fertigation of citrus and vegetable crops (application of fertilizer with irrigation water). Nutrients commonly applied through fertigation include nitrogen (N) and potassium (K). Phosphorus is less commonly applied through fertigation due to potential precipitation with calcium (Broder, 1984). Micronutrients are sometimes added to liquid blends in small concentrations. Common N sources used in solution fertilizers in Florida include urea, ammonium nitrate, and potassium nitrate. Sources of K include potassium chloride and potassium nitrate. These materials are often formulated in a 1:1 ratio in blends such as 6-0-6, 8-0-8, and 10-0-10, since many crops require similar rates of N and K₂O.

In order to maintain uniform physical and chemical characteristics, the dissolved components of liquid fertilizers must remain in solution. Crystallization of salts can occur if the temperature of the fertilizer solution drops below a level known as the salt-out temperature (Jones and Balay, 1984). Liquid fertilizers are usually stored in outdoor tanks, and salt-out can be a concern if a material purchased during warmer months remains in a tank as winter approaches. Cold fronts can decrease the solution temperature enough to cause salt-out of the fertilizer. Crystallized fertilizer salts are not easily redissolved in storage tanks, and present a difficult clean-up problem.

Salt-out temperatures are controlled by a number of factors. Knowledge of the most influential of these factors would aid liquid fertilizer users in preventing a crystallization problem. The objectives of this study were to investigate the effects on the salt-out temperature of liquid fertilizer of 1) fertilizer grade (concentration), 2) water quality used to manufacture the blend, 3) K source, and 4) the addition of micronutrients to the blend. The potential of two polyphosphate water conditioners to lower the salt-out temperature was also investigated.

Materials and Methods

Experiment 1 investigated the effect of water quality on the salt-out temperature of two 10-0-10 fertilizers. Three water sources were used: 1) deionized water; 2) Immokalee well water (designated the “good” source); and 3) LaBelle well water (designated the “poor” source). These sources differed considerably in total dissolved solids (TDS) content (Table 1). The 10-0-10 fertilizer was made using either potassium chloride (KCI) or potassium nitrate (KNO₃). Fertilizer-grade ammonium nitrate (NH₄NO₃) plus either KCI or KNO₃ were dissolved in water and agitated with a heat source to accelerate dissolution. The results obtained were as follows.

<table>
<thead>
<tr>
<th>Source</th>
<th>pH</th>
<th>Conductivity µmhos cm⁻¹</th>
<th>TDS† ppm</th>
<th>Ca</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>De-ionized</td>
<td>7.7</td>
<td>6</td>
<td>4</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Immokalee (Good)</td>
<td>7.7</td>
<td>872</td>
<td>560</td>
<td>39</td>
<td>7</td>
</tr>
<tr>
<td>LaBelle (Poor)</td>
<td>6.7</td>
<td>3990</td>
<td>2550</td>
<td>405</td>
<td>45</td>
</tr>
</tbody>
</table>

†Total dissolved solids.

resulting liquid fertilizers were filtered to remove undissolved particulate matter. Aliquots (75-ml) were poured into a 125-ml Erlenmeyer flask. A thermometer was placed in the solution, which was then slowly cooled in an ice bath. The salt-out temperature was recorded at the first sign of crystallization. This procedure was done in triplicate for each fertilizer solution.

Experiment 2 investigated the effect of fertilizer grade (concentration of N and K2O) on salt-out temperature. Two grades (8-0-8 and 6-0-6) were made using the same material combinations as in Experiment 1. The Immokalee well water (“good” quality) was used for this and all subsequent experiments. Methodology of dissolution and salt-out temperature measurement were the same as in Experiment 1. Results were compared to the salt-out temperature measured for the 10-0-10 fertilizer in “good” water from Experiment 1.

Theoretical salt-out temperatures for each of the three grades were calculated using the National Fertilizer and Environmental Research Center Form-U-Share program (Nevins, 1991), and the results were compared to the temperatures measured in the laboratory.

Experiment 3 investigated the effect on salt-out temperature of micronutrients added to the three grades of fertilizer made previously. Fertilizer solutions were made as in Experiments 1 and 2 (using the “good” water only), with the following micronutrient concentrations: 1) 0.25% Mg from magnesium sulfate, 2) 0.05% Mn from manganese sulfate, 3) 0.03% Zn from zinc sulfate, and 4) 0.01% B from boric acid. These represent typical micronutrient levels in blends used to fertilize citrus and vegetables. Salt-out temperatures were determined as above, and were compared to salt-out temperatures of corresponding blends without added micronutrients.

Experiment 4 investigated the effect of two water-conditioning additives on the salt-out temperature of 10-0-10, 8-0-8, and 6-0-6 grades made with KCl or KNO3. Additives were 1) a long-chain linear polyphosphate blend (designated Poly 1), and 2) Poly 1 with an anti-crystallization agent added (designated Poly 2). Both materials are proprietary products of Chem Craft Corp., Oklahoma City, OK, and were obtained from UAP-Florida, Immokalee, FL. Fertilizer solutions were made as in experiments 1 and 2. Poly 1 or Poly 2 was added to these solutions, each at three rates: 100, 700, and 1250 ppm by volume. The sequence of mixing was: fertilizer salts dissolved in water first, polyphosphate added last.

Experiment 5 investigated the effect of reversed sequence of mixing of the polyphosphate on salt-out temperature. Polyphosphate-fertilizer solutions were prepared as in Experiment 4, except the polyphosphate was added to the water before the solid fertilizer materials were dissolved in it. Results from this experiment were compared to those of experiment 4.

In cases where fertilizer solutions crystallized below 32°F, the ice bath was amended with sodium chloride, which lowered its temperature to approximately −4°F.

The following comparisons of salt-out temperature were made: 1) 10-0-10 fertilizers made with different water quality; 2) fertilizers of different grade; 3) fertilizers made with KNO3 vs. those made with KCI; 4) fertilizers with and without added micronutrients; 5) fertilizers with and without added polyphosphate; and 6) fertilizers with polyphosphate added last vs. those with polyphosphate added first.

Results

Experiment 1. The salt-out temperature of the 10-0-10 fertilizer made with deionized water was 4°F or less below the fertilizers made with either “good” or “poor” quality water (Fig. 1). A 10% N, 10% K2O fertilizer solution contains over 500,000 ppm TDS from the N and K fertilizer salts. The addition of 500 to 2600 ppm TDS from a water source represents less than 1% of the total TDS in solution. Thus, the salt contribution from the water source was not significant in the overall fertilizer solution.

Experiment 2. There was a 14 to 18°F decrease in salt-out temperature for each 2% decrease in fertilizer N and K concentration (Fig. 2). With the 8-0-8 and 10-0-10 grades, salt-out temperature was about 4°F less for blends made with KNO3 than those made with KCl. This was most likely due to the presence of chloride in the KCl mixtures. The chloride was not a nutrient source, but added to the TDS of the solution.
Salt-out temperatures of the 10-0-10, 8-0-8, and 6-0-6 grades as calculated by the Form-U-Share program were 56, 38, and 19° F, respectively. These were within 2 to 8° F of the salt-out temperatures measured in this experiment.

Experiment 3. Addition of micronutrients had no profound effect on the salt-out temperature of any fertilizer grade (Fig. 2). Micronutrient concentrations were 24 to 1000 times less than the N and K₂O concentrations, depending on fertilizer grade. These concentrations were too low to significantly affect the overall TDS (500,000 ppm) of the fertilizer.

Experiment 4. The addition of Poly 1 decreased salt-out temperature from 3 to 8° F, depending on the fertilizer grade and K source (Fig. 3). Poly 2 lowered the salt-out temperature of the 6-0-6 grade up to 7° F, but showed little effectiveness when added to the 8-0-8 or 10-0-10 grades. The polyphosphates were most effective at the 100 ppm concentration with the 6-0-6 blend, but 1250 ppm gave the best results with the 8-0-8 and the 10-0-10 blends.

Experiment 5. Adding the polyphosphates to water before mixing the fertilizers had less impact on salt-out temperature depression (Fig. 4). In fact, mixing the polyphosphates first increased the salt-out temperature in some cases. However, the greatest salt-out differential observed was with the 6-0-6 blend where the polyphosphate was added first, in which the salt-out temperature decreased about 9° F.

Discussion and Conclusions

Because of the very small difference in salt-out temperatures of the fertilizer blends with different water qualities, there is no need to be concerned about the source of the water being used to manufacture the fertilizer. This is significant for liquid fertilizer blenders because they need not be too concerned about the quality of their water.

Addition of micronutrients had no serious effect on the salt-out temperature of any of the fertilizer solutions. This is meaningful to Florida growers, because micronutrients often need to be applied to sandy soils of low fertility. Since Florida crops are grown during the winter, it is important to know that the addition of such key elements will not raise the temperature at which the fertilizer crystallizes.

In summary, for cool-season vegetable and citrus production in Florida, the following recommendations are
suggested to prevent problems with the crystallizing of liquid fertilizer: 1) There is no need to be concerned about the water source used to make the fertilizer; 2) Lower the grade of fertilizer being used; 3) Use KNO₃ as the K source for higher-analysis blends; 4) There is no significant consequence of adding micronutrients; and 5) The use of polyphosphate can provide an extra degree of protection if desired, but the effectiveness depends on the fertilizer grade used.

**Literature Cited**


**COLD PROTECTION MECHANISMS**¹

Cold protection methods have been developed and refined mostly by the growers who employ them. The methods are mostly art. Science has been confined to explanations of how the methods found to be effective work. Mechanisms thought of as causal processes have been quantified in models (Barfield et al., 1990; Crawford, 1964; Gerber, 1969). These mechanisms are the subject of this paper. Most of the mechanisms were either unknown or ignored in early explanations or models. This seems to suggest that there are several mechanisms yet to be understood even if they have been discovered. In understanding mechanisms underlying the methods, refinements will be suggested. One purpose is to cast doubt on the notion that sufficient knowledge of cold protection methodology already exists, e.g. additional research is not needed.

Effect of freezes on citrus yield [see Martsolf, 1990a; Fig. 1a] and on other cold sensitive horticultural crops in Florida is obvious. If even a relatively small portion of that loss could be avoided it would justify many times over the cost of proposed investigations in the field. Industry leaders become concerned when yield begins to outdistance the market. Their interest in avoiding freeze damage decreases because concern about oversupply increases. To view freezes simply as acts of God and recognize them as effective limiters of supply is to deny the trauma that accompanies them.

It is prudent to avoid any need for cold protection by confining cold sensitive crops to sites on the globe completely devoid of freeze hazards. Indeed it could be declared that perfect site selection is the best method of cold protection. But how does this position help those who for one reason or another recognize that their current site has a freeze risk. There seems to be hope that plants will be biologically engineered to endure not only cold stress but any major stress. This would make cold protection

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