MEMORANDUM

SUBJECT: Captan - Storage Stability Studies for the Captan Registration Standard

FROM: Linda S. Propst, Chemist
Dietary Exposure Branch
Health Effects Division (H7509C)

TO: Eugene M. Wilson, PM Team 23
Fungicide-Herbicide Branch
Registration Division (H7505C)

THRU: Andrew R. Rathman, Section Head
Special Registration Section I
Dietary Exposure Branch
Health Effects Division (H7509C)

Background

The Residue Chemistry Chapter of the Captan Registration Standard, published August 15, 1985, concluded that no data were available regarding the storage stability of residues of captan in or on plant commodities or in animal commodities. The following data were requested:

The storage intervals and conditions of storage of samples used to support all established tolerances for residues must be submitted. These data must be accompanied by data depicting the percent decline in residues at the times and under the conditions specified. On receipt of these data, the adequacy of the aforementioned tolerances will be reevaluated.

All residue data requested in this Registration Standard must be accompanied
by data regarding storage length and conditions of storage of samples analyzed. These data must be accompanied by data depicting the stability of residues under the conditions and for the time intervals specified.

This Submission

ICI Americas, Inc. has submitted three studies intended to evaluate the stability of captan residues after storage for up to 2 years under controlled conditions in various raw agricultural commodities (RACs) in which initial captan and tetrahydrophthalimide (THPI) residue levels have been determined. THPI is a principal plant metabolite and also a degradation product of captan. Study 1 is complete. Interim results are given for studies 2 and 3.

Analytical Method

The analytical method used to quantitate captan and THPI residues is entitled "Determination of Captan and THPI Residues in Crops." This method is designated Method RM-IK-2 by its supplier, Chevron Chemical Company. Briefly, samples are macerated, immediately acidified with phosphoric acid, then extracted three times with ethyl acetate. The extracts are combined, dried with sodium sulfate, filtered, washed three times with dilute phosphoric acid, then filtered through sodium sulfate and evaporated to dryness. The residue is dissolved in dichloromethane and passed through a nuchar-silica column to remove interfering coextractives; the residue from oily crops first passes through a standard acetonitrile-hexane partition step and is further cleaned up by gel permeation chromatography prior to the nuchar-silica cleanup. The final eluate is evaporated to dryness, and the residue is redissolved in a measured volume of solvent prior to analysis by gas chromatography.

Captan and THPI are each quantitated by gas chromatography using element-selective detection. For captan, a Coulson electrolytic conductivity detector operated in the chlorine-selective mode or an electron-capture detector may be used. For THPI, a Coulson detector operated in the nitrogen-selective mode, or a nitrogen-phosphorous detector may be used. This method is intended for determinations of captan and THPI at levels greater than 0.05 ppm.

Total residue concentrations expressed as captan were calculated from the GC determinations of captan and THPI by adding twice the concentration of THPI to that of captan. This procedure was followed because a given amount of THPI
requires as precursor twice this amount of captan, on a 
weight/weight basis. One gram of captan produces, upon 
decomposition or metabolism, almost exactly 0.5 g of THPI.

Study 1

Field treated commodities were macerated, analyzed for 
initial residue levels of captan and THPI, and stored in the 
dark at -20 ± 10°C in glass bottles with polyethylene-lined 
lids. At intervals of 3, 6, and 14 months, the samples were 
chosen randomly from storage and reanalyzed. At each 
withdrawal two untreated samples were removed. The following 
table presents the results of this study.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Interval (Months)</th>
<th>Captan ppm</th>
<th>THPI ppm</th>
<th>Total ppm*</th>
<th>Total Recovery %</th>
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</thead>
<tbody>
<tr>
<td>Apple</td>
<td>Initial</td>
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<td>0.095</td>
<td>2.84</td>
<td>-</td>
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<td>0.13</td>
<td>3.16</td>
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<td>1.29</td>
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<td>0.43</td>
<td>0.22</td>
<td>0.87</td>
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<td>-</td>
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<td></td>
<td>3</td>
<td>34.4</td>
<td>9.91</td>
<td>54.2</td>
<td>145</td>
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<td>6</td>
<td>29.9</td>
<td>7.56</td>
<td>45.0</td>
<td>121</td>
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<td>Initial</td>
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<td>0.24</td>
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<td>7.40</td>
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</tbody>
</table>

*Expressed as captan.

Study 2

In this study, cherry and tomato samples were from 
captan-treated crops, while samples of almonds, apple juice, 
corn grain, potato tubers, soybeans, beet tops, and soybean 
forage were commercially obtained from local markets or
producers. The cherry and tomato samples were macerated, analyzed for captan and THPI, then stored. The remaining commodities were macerated and divided into several portions that were fortified with both captan and THPI ranging from 0.2 to 0.5 ppm, then stored. (The only exceptions were beet tops and soybean forage, for which separate samples were fortified with either captan or THPI). The interim results of that study are presented in the following table.

<table>
<thead>
<tr>
<th>RAC</th>
<th>Interval (Months)</th>
<th>Captan Recov. ppm</th>
<th>THPI Recov. ppm</th>
<th>Total Recov. ppm</th>
<th>Total Recov. %</th>
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<tbody>
<tr>
<td>Almond (Fortification, ppm)</td>
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<td></td>
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<tr>
<td>0</td>
<td>0.5</td>
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<tr>
<td>1</td>
<td>&lt; 0.05</td>
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<td>0.36</td>
<td>0.72</td>
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<td>Apple Juice (Fortification, ppm)</td>
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<td></td>
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<td>0.48</td>
<td>0.24</td>
<td>0.96</td>
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<td>Initial Concentration</td>
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<td>0.24</td>
<td>0.96</td>
<td></td>
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</tr>
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<td>0.47</td>
<td>0.17</td>
<td>0.81</td>
<td>84</td>
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<td>0.60</td>
<td>63</td>
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<td>RAC</td>
<td>Interval (Months)</td>
<td>Captan Recov. ppm</td>
<td>THPI Recov. ppm</td>
<td>Total Recov. ppm</td>
<td>Total Recov. %</td>
</tr>
<tr>
<td>-----</td>
<td>-----------------</td>
<td>-------------------</td>
<td>----------------</td>
<td>-----------------</td>
<td>---------------</td>
</tr>
<tr>
<td>Soybean Grain</td>
<td>Fortification, ppm</td>
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<td>0.24</td>
<td>0.96</td>
<td>0.29</td>
</tr>
<tr>
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<td>3</td>
<td>0.30</td>
<td>0.25</td>
</tr>
<tr>
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<td></td>
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<td>6</td>
<td>0.28</td>
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<td>0.15</td>
<td>0.30</td>
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<td>RAC</td>
<td>Interval (Months)</td>
<td>Captan Recov. ppm</td>
<td>THPI Recov. ppm</td>
<td>Captan Recov. %</td>
<td>THPI Recov. %</td>
</tr>
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<td>-------------------</td>
<td>----------------</td>
<td>-----------------</td>
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<td>27</td>
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<tr>
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<td>0.31</td>
<td>0</td>
</tr>
<tr>
<td>Soybean Forage</td>
<td>Fortification, ppm</td>
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<td>73</td>
<td>85</td>
</tr>
<tr>
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<td></td>
<td>15</td>
<td>0.36</td>
<td>0.38</td>
<td>69</td>
</tr>
</tbody>
</table>

In this study, a total of 47 separate captan fortifications were made at levels between 0.48 and 20 ppm. Captan recoveries ranged between 72 and 119 percent.

In the 47 fortifications made with THPI at levels from 0.24 to 1 ppm, recoveries ranged between 52 and 125 percent.
Study 3

In this study, the stability of captan and THPI in RACs were evaluated independently. The samples were ground by machine only enough to allow for representative subsampling prior to fortification. In two cases, corn and almonds, whole samples were also analyzed. Because machine grinding of spinach resulted in finely macerated material, coarsely hand-chopped spinach was also tested. The following table presents the interim results of that study.

<table>
<thead>
<tr>
<th></th>
<th>(Months)</th>
<th>Captan ppm</th>
<th>Captan rec.</th>
<th>THPI ppm</th>
<th>THPI rec.</th>
<th>Total ppm</th>
<th>Total rec.</th>
<th>THPI ppm</th>
<th>THPI rec.</th>
</tr>
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<td>Almond Nuts,</td>
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<td>101%</td>
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<td>a</td>
<td>0.38</td>
<td>77%</td>
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</tr>
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<td>Coarsely Ground</td>
<td></td>
<td>0.21</td>
<td>41%</td>
<td>a</td>
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<td>a</td>
<td>0.38</td>
<td>76%</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.33</td>
<td>66%</td>
<td>0.07</td>
<td>46%</td>
<td>92%</td>
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<td>a</td>
<td></td>
</tr>
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<td>Almond Nuts,</td>
<td></td>
<td>0.41</td>
<td>81%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.44</td>
<td>89%</td>
<td></td>
</tr>
<tr>
<td>Whole</td>
<td></td>
<td>0.27</td>
<td>53%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.36</td>
<td>72%</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.42</td>
<td>84%</td>
<td>&lt; 0.05</td>
<td>42%</td>
<td>84%</td>
<td>a</td>
<td>a</td>
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<td></td>
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<td>82%</td>
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<td>0.44</td>
<td>87%</td>
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<td></td>
<td>0.39</td>
<td>78%</td>
<td>a</td>
<td>a</td>
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<td>0.40</td>
<td>80%</td>
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</tr>
<tr>
<td>Apple Sauce</td>
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<td>0.47</td>
<td>94%</td>
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<td>0.43</td>
<td>85%</td>
<td></td>
</tr>
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<td></td>
<td>0.40</td>
<td>80%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.37</td>
<td>74%</td>
<td></td>
</tr>
<tr>
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<td>74%</td>
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<td>a</td>
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<td>0.36</td>
<td>73%</td>
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<tr>
<td>Corn Grain,</td>
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<td>0.44</td>
<td>88%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.40</td>
<td>80%</td>
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</tr>
<tr>
<td>Coarsely Ground</td>
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<td>11%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.36</td>
<td>72%</td>
<td></td>
</tr>
<tr>
<td>Corn Grain,</td>
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<td>0.40</td>
<td>81%</td>
<td>a</td>
<td>a</td>
<td>a</td>
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<td>92%</td>
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<td>112%</td>
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<td>84%</td>
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<td>0.43</td>
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</tr>
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<td>Raisin</td>
<td></td>
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<td>a</td>
<td>a</td>
<td>0.39</td>
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</tr>
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<td></td>
<td>0.36</td>
<td>71%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.44</td>
<td>87%</td>
<td></td>
</tr>
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<td></td>
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<td>Fortified With 0.05 ppm THPI</td>
<td></td>
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<td></td>
<td></td>
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<td>Captan ppm</td>
<td>Captan rec.</td>
<td>THPI ppm</td>
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<td>Total rec.</td>
<td>THPI ppm</td>
<td>THPI rec.</td>
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<td>(Months)</td>
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<td>a</td>
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<td>99%</td>
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<td>Chopped</td>
<td>1</td>
<td>0.15</td>
<td>30%</td>
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<td>a</td>
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<td>0.37</td>
<td>73%</td>
<td></td>
</tr>
<tr>
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<td>0.48</td>
<td>95%</td>
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<td>a</td>
<td>a</td>
<td>0.44</td>
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<td>Coarsely</td>
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<td>11%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.39</td>
<td>79%</td>
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<td>2</td>
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<td>24%</td>
<td>0.10</td>
<td>0.03</td>
<td>63%</td>
<td>a</td>
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<td>0.16</td>
<td>0.45</td>
<td>90%</td>
<td>0.47</td>
<td>79%</td>
<td></td>
</tr>
<tr>
<td>Tomato Sauce</td>
<td>0</td>
<td>0.42</td>
<td>84%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.41</td>
<td>82%</td>
<td></td>
</tr>
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<td>0.51</td>
<td>102%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.38</td>
<td>77%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.44</td>
<td>87%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.53</td>
<td>79%</td>
<td></td>
</tr>
<tr>
<td>Tomato</td>
<td>0</td>
<td>0.46</td>
<td>92%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.37</td>
<td>75%</td>
<td></td>
</tr>
<tr>
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<td>1</td>
<td>0.38</td>
<td>76%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.42</td>
<td>84%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.38</td>
<td>75%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.46</td>
<td>92%</td>
<td></td>
</tr>
<tr>
<td>Tomato Dry Pomace</td>
<td>0</td>
<td>0.43</td>
<td>87%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.43</td>
<td>85%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>0.42</td>
<td>84%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.38</td>
<td>76%</td>
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</tr>
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<td>3</td>
<td>0.38</td>
<td>76%</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>0.40</td>
<td>80%</td>
<td></td>
</tr>
</tbody>
</table>

*Not analyzed.*

In the above study, 80 fortifications using 0.5 ppm captan were made. Recoveries ranged between 68 and 109 percent. A total of 79 fortifications were made with 0.5 ppm THPI. Recoveries ranged between 70 and 118 percent.

DEB's Conclusions and Recommendations

The above studies indicate that residues of captan, per se, are unstable in most crops which have been macerated prior to storage. However, in those cases where captan degrades, it generates an equivalent amount of THPI. Therefore, the stability of captan is a function of the stability of THPI.

The registrant has calculated the total residue concentration expressed as captan by taking the levels of
captan and THPI determined by GC and adding twice the concentration of THPI to that of captan. It should be noted that we have some reservations as to the validity of this approach; however, on this basis, DEB concludes from the data submitted that the total residue concentrations expressed as captan are stable in the following RACs for the intervals specified:

<table>
<thead>
<tr>
<th>Produce</th>
<th>Stability</th>
</tr>
</thead>
<tbody>
<tr>
<td>Almonds</td>
<td>1 month</td>
</tr>
<tr>
<td>Apples</td>
<td>14 months</td>
</tr>
<tr>
<td>Apple Juice</td>
<td>15 months</td>
</tr>
<tr>
<td>Apple Sauce</td>
<td>3 months</td>
</tr>
<tr>
<td>Cherries</td>
<td>12 months</td>
</tr>
<tr>
<td>Corn Grain</td>
<td>1 month</td>
</tr>
<tr>
<td>Cucumber</td>
<td>14 months</td>
</tr>
<tr>
<td>Lettuce</td>
<td>6 months</td>
</tr>
<tr>
<td>Potatoes</td>
<td>6 months</td>
</tr>
<tr>
<td>Soybean Grain</td>
<td>3 months</td>
</tr>
<tr>
<td>Soybean Forage</td>
<td>15 months</td>
</tr>
<tr>
<td>Tomatoes</td>
<td>12 months</td>
</tr>
<tr>
<td>Strawberries</td>
<td>14 months</td>
</tr>
<tr>
<td>Spinach</td>
<td>3 months</td>
</tr>
<tr>
<td>Tomato Sauce</td>
<td>3 months</td>
</tr>
<tr>
<td>Dry Tomato Pomace</td>
<td>3 months</td>
</tr>
<tr>
<td>Dry Grape Pomace</td>
<td>3 months</td>
</tr>
<tr>
<td>Raisin</td>
<td>3 months</td>
</tr>
<tr>
<td>Beet Tops</td>
<td>Data Inconclusive</td>
</tr>
</tbody>
</table>

Final conclusions concerning the storage stability of total residues expressed as captan in the various RACs will be made at the time the final results are submitted for Study 2 and Study 3.

cc: Reading File, Circulation, Subject File, Captan Reg.  
    Std. File, Reviewer, PMSD/ISB, Branch Chief  
RDI: A.R. Rathman, 12/1/89; E. Zager, 12/1/89  
H7509C:DEB:LSP:lsp:CM-2:Rm803C:557-7324:12/18/89  