US Environmental Protection Agency
Office of Pesticide Programs

Rinsing Procedures for Dilutable Pesticide Products in Rigid Containers – basic testing recommendations

June 25, 2008

OPPTS 8XX.XXXX Rinsing Procedures for Dilutable Pesticide Products in Rigid Containers – basic testing recommendations

EPA's test procedure, as referenced in the container regulations under §165.25(f)(1).

(a) Scope.

(1) Applicability. This guideline describes test protocols that EPA believes will generally satisfy residue removal performance testing requirements of the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) (7 U.S.C. 136, et seq.). It addresses testing to demonstrate that rigid nonrefillable containers that hold dilutable pesticide products can be effectively rinsed using triple rinsing procedures, such that a fourth rinse shows 99.99% product removal. The guideline also includes testing for pressure rinsing containers, although pressure rinsing testing is not required by the pesticide container regulations.

(2) Background. The source material used in developing this OPPTS test guideline is a rinsing protocol developed by Formulogics of Princeton, New Jersey. The requirement for dilutable pesticide products in rigid containers to achieve 99.99% product removal is required by 40 CFR Subpart B §165.25. In 1990, Formulogics conducted a container rinsing study at the request of EPA. The study was designed to examine pressure and triple rinsing for a number of containers and formulation types. Some container rinsing studies had been conducted prior to the Formulogics study, although each study had a different purpose and utilized a variety of collecting, rinsing and reporting procedures. Therefore, comparisons between these studies were difficult to make. To eliminate some of the problems of previous studies, EPA requested that a protocol be developed for triple and pressure rinsing. Formulogics developed a draft protocol and circulated the draft to interested parties for comment. The comments were considered and the document was revised and released on September 6, 1990.

(b) Introduction. This guideline addresses testing of nonrefillable pesticide containers for their ease of residue removal using triple rinse or pressure rinse procedures prior to container disposal or recycling. This guideline applies specifically to rigid containers that are small enough to be shaken, which is designated in the regulations (40 CFR §165.25(f)(1)) as containers with capacities less than or equal to 5 gallons (18.9 liters) for liquid formulations and 50 pounds (22.7 kilograms) for solid formulations. In addition, this guideline applies specifically to dilutable pesticides, which are defined in the regulations (40 CFR §165.25(f)(1)) as pesticides with labeling that allows or requires the pesticide product to be mixed with a liquid diluent prior to application. This guideline recommends tests to be conducted and data to be generated and held by the registrant. This testing protocol can be used to determine whether a container and formulation achieve 99.99 percent residue removal.

(c) Test Procedures – General.

(1) Selection of containers. For a specific pesticide product, three unopened containers of that pesticide product with the same production lot number should be selected.

(2) Selection of collection vessels. Clean, non-contaminated collection vessels should be available for containing undiluted product and rinsate prior to analysis.

(3) Rinse water. The source of rinse water is not restricted; it may be tap, city, well or any other commonly-used water. Prior to the test, the rinse water should be analyzed for the specific pesticide active ingredient or ingredients that are being tested in the study. The water should not contain the pesticide active ingredient or ingredients being tested or the background level of the active ingredient should be such that it does not interfere with the rinsing study. The analysis of the water should be conducted prior to the test. It is not necessary to analyze the water each time a sample of water is drawn. The purpose of this provision is to ensure that the test results are not biased due to the presence of the pesticide active ingredient or ingredients in the rinse water.

(4) Temperature of samples. When tested, the temperature of the samples should be 23 degrees Centigrade ± 3 degrees C.

(5) Temperature of water. The water used in the rinse study should be 23 degrees Centigrade ± 3 degrees C.

(6) Volume measurement for rinse water. The volume of rinse water that is added to the containers and the volume of rinsate collected should be determined. The rinse water volume and rinsate volume may be determined by either volumetric or gravimetric procedures and should be accurate to ± 0.1 percent.

(7) Sampling plans. Three different sampling plans are described. The plan labeled as "minimum" is an option requiring the minimum amount of analytical effort. The plan labeled as "acceptable" is somewhat more rigorous, and the plan labeled as "good" increases the analytical burden but generates the best overview of the rinsing process. Any of the sampling plans may be used.

(d) Triple Rinsing Test Procedure.

(1) Preparation of container. The selected container should be opened and inverted over a suitable collection vessel. The container should be drained according to the process described in section (d)(2). After draining, the container should be recapped to prevent the residue from evaporating. If possible, the rinsing study should be initiated immediately after the container has been drained. The study should not be initiated if the drained container is allowed to remain capped and standing for longer than thirty (30) minutes.

(2) Draining the container. The container should be drained in an inverted position for thirty (30) seconds when removing the original contents or rinsate from the container.

> **(i) Timing.** The thirty (30) second period begins once the flow of liquid from the mouth of the container can no longer be described as a continuous stream.
>
> **(ii) Inverted.** The word inverted should be interpreted as meaning that the container is held in a position that will maximize the amount of liquid that will drain from the container in the minimum amount of time.
>
> **(iii) Specific instructions for draining material from containers which do not have hollow handles.** The container should be inverted during draining. The opening of the

container defines the top. The container should be held so the face of the container opening is directed towards and parallel to the floor at the start of the drain period. At 5 seconds into the drain period, the container should be rotated clockwise 90 degrees so the face of the opening is perpendicular to the floor. The container should be held in this position for 5 seconds and then rotated back to the original inverted position. At 15 seconds into the drain period, the container should again be rotated clockwise 90 degrees so the face of the opening is perpendicular to the floor. At 20 seconds into the drain period, the container should be rotated back to its original inverted position and held for 10 more seconds.

(iv) Specific instructions for draining material from containers with a hollow handle on the side. The container should be inverted during draining. The opening of the container defines the top. At the start of the drain period, the container should be held so the face of the opening is directed towards and parallel to the floor and the handle is on the left hand side as you look at the container. At 5 seconds into the drain period, the container should be rotated clockwise 90 degrees so the face of the opening is perpendicular to the floor. The handle should be parallel to the floor and on the upper surface of container. The container should be held in this position for 5 seconds and then rotated back to the original inverted position. At 15 seconds into the drain period, the container should again be rotated clockwise 90 degrees so the face of the opening is perpendicular to the floor. At 20 seconds into the drain period, the container should be rotated back to its original inverted position and held for 10 more seconds.

(v) Specific instructions for draining material from containers with a hollow handle on the top. The container should be inverted during draining. The opening of the container defines the top. At the start of the drain period, the container should be held so the face of the opening is directed towards and is parallel to the floor. The opening should be on the right hand side as you face the container, with the handle oriented along a line running left to right and parallel to the floor. At 5 seconds into the drain period, the container should be rotated clockwise 90 degrees so the face of the opening is perpendicular to the floor. The handle should be on the left hand side and perpendicular to the floor. The container should be held in this position for 5 seconds and then rotated back to the original inverted position. At 15 seconds into the drain period, the container should again be rotated clockwise 90 degrees so the face of the opening is perpendicular to the floor. At 20 seconds into the drain period, the container should be rotated back to its original inverted position and held for 10 more seconds.

(3) Adding rinse water. Clean rinse water should be added to the drained container. The volume of rinse water should be equivalent to 25% (1/4) of the stated volume of the container for each rinse cycle. The volume of the clean rinse water that is added to the container should be recorded. After the water is added, the cap should be placed back on the container before shaking the container according to the instructions in section (d)(4).

(4) Shaking the container. The container should be shaken by hand for thirty (30) seconds per rinse. No mechanical devices should be used.

(i) Initial position. To start, the container should be held in the horizontal plane with the cap or top portion of the container being held in your left hand.

(ii) Container facing left. For the first ten seconds, the container should be shaken vigorously in a back and forth (left to right) manner, maintaining the container parallel to the floor. The container should be shaken at a rate of approximately two strokes per second with the distance of the stroke between 4 and 6 inches.

(iii) Container facing down. After ten seconds and in a continuous motion, the container should be rotated 90 degrees so the cap or upper portion of the container is now oriented towards the floor. The container should be shaken vigorously in an up and down manner, maintaining the container perpendicular to the floor. The container should be shaken at a rate of approximately two strokes per second with the distance of the stroke between 4 and 6 inches.

(iv) Container facing up. At 20 seconds, the container should be rotated so the cap or upper portion is now oriented towards the ceiling. The container should be shaken vigorously in an up and down manner, maintaining the container perpendicular to the floor. The container should be shaken at a rate of approximately two strokes per second with the distance of the stroke between 4 and 6 inches until the 30 seconds have been consumed.

(5) Draining the container after shaking. The cap should be removed from the container. The container should be inverted over a clean collection vessel and drained for thirty (30) seconds according to the process described in section (d)(2). The volume of the rinsate collected should be measured and recorded.

(6) Cleaning the threads. After draining the rinsate from the container, the container cap and the threads on the neck of the container should be wiped clean. If a clean replacement cap is available, it may be used in place of the previous cap.

(7) Additional rinses. The procedures in sections (d)(3) through (d)(6) should be repeated at least three (3) more times, for a total of at least four (4) rinses.

(8) Sampling during the triple rinse procedure. The rinsate should be sampled using good laboratory techniques. Any pesticide residues should be maintained in suspension during the sampling process.

(i) Minimum sampling plan. The fourth rinsate should be collected, sampled and analyzed according to section (d)(9). The results of the analysis should be recorded.

(ii) Acceptable sampling plan. The third and fourth rinsates should be collected, sampled and analyzed according to section (d)(9). The results of the analyses should be recorded.

(iii) Good sampling plan. The second, third, fourth and fifth rinsates should be collected, sampled and analyzed according to section (d)(9). The results of the analysis should be recorded.

(**9) Analysis.** The samples should be analyzed for the pesticide active ingredient or ingredients using any validated analytical procedure suitable for the analysis of the active ingredient(s) in water. If no extraction or concentration procedures are employed and the water is analyzed directly, the method should have a level of sensitivity of 0.5 ppm or lower. The following information should be recorded:

(i) Whether the rinsate was chemically or physically treated to ensure compound stability prior to analysis;
(ii) The sensitivity and the detection limit of the procedure; and
(iii) Whether the water samples were extracted.

(10) Waste disposal. The rinsate and the undiluted pesticide product collected in this study should be discarded utilizing approved disposal procedures.

(11) Format of the results. The results of the rinsate analysis should be reported as: (i) the rinsate concentration (mg/ml) and (ii) percent removal. Percent removal should be calculated as follows:

$$\frac{\text{Final rinsate concentration mg/ml}}{\text{Original concentration mg/ml}} = A$$

$$\text{Percent Removal} = [\ 1.0 - A\ ] \times 100$$

(e) Pressure Rinsing Procedure.

(1) Selection of pressure rinsing device. A rinsing device that can supply a pressure of at least 40 pounds per square inch (psi), such as a Jet Rinse, Quik-Rinse or other standard pressure rinsing device should be used.

(2) Water pressure. The pressure of the water in the line coming into the lab should be 40 $\pm$ 2 pounds per square inch.

(3) Measuring the volume of water discharged. Before beginning the pressure rinse process, the amount of water that is discharged by the pressure rinsing device during a ten (10), twenty (20) and thirty (30) second discharge should be measured and recorded.

(4) Preparation of container. The selected container should be opened and inverted over a suitable collection vessel. The container should be drained according to the process described in section (e)(5). After draining, the container should be recapped to prevent the residue from evaporating. If possible, the rinsing study should be initiated immediately after the container has been drained. The study should not be initiated if the drained container is allowed to remain capped and standing for longer than thirty (30) minutes.

(5) Draining the container. The container should be drained in an inverted position for thirty

(30) seconds when removing the original contents or rinsate from the container.

(i) Timing. The thirty (30) second period begins once the flow of liquid from the mouth of the container can no longer be described as a continuous stream.

(ii) Inverted. The word inverted should be interpreted as meaning that the container is held in a position that will maximize the amount of liquid that will drain from the container in the minimum amount of time.

(iii) Specific instructions for draining material from containers which do not have hollow handles. The container should be inverted during draining. The opening of the container defines the top. The container should be held so the face of the container opening is directed towards and parallel to the floor at the start of the drain period. At 5 seconds into the drain period, the container should be rotated clockwise 90 degrees so the face of the opening is perpendicular to the floor. The container should be held in this position for 5 seconds and then rotated back to the original inverted position. At 15 seconds into the drain period, the container should again be rotated clockwise 90 degrees so the face of the opening is perpendicular to the floor. At 20 seconds into the drain period, the container should be rotated back to its original inverted position and held for 10 more seconds.

(iv) Specific instructions for draining material from containers with a hollow handle on the side. The container should be inverted during draining. The opening of the container defines the top. At the start of the drain period, the container should be held so the face of the opening is directed towards and parallel to the floor and the handle is on the left hand side as you look at the container. At 5 seconds into the drain period, the container should be rotated clockwise 90 degrees so the face of the opening is perpendicular to the floor. The handle should be parallel to the floor and on the upper surface of container. The container should be held in this position for 5 seconds and then rotated back to the original inverted position. At 15 seconds into the drain period, the container should again be rotated clockwise 90 degrees so the face of the opening is perpendicular to the floor. At 20 seconds into the drain period, the container should be rotated back to its original inverted position and held for 10 more seconds.

(v) Specific instructions for draining material from containers with a hollow handle on the top. The container should be inverted during draining. The opening of the container defines the top. At the start of the drain period, the container should be held so the face of the opening is directed towards and is parallel to the floor. The opening should be on the right hand side as you face the container, with the handle oriented along a line running left to right and parallel to the floor. At 5 seconds into the drain period, the container should be rotated clockwise 90 degrees so the face of the opening is perpendicular to the floor. The handle should be on the left hand side and perpendicular to the floor. The container should be held in this position for 5 seconds and then rotated back to the original inverted position. At 15 seconds into the drain period, the container should again be rotated clockwise 90 degrees so the face of the opening is perpendicular to the floor. At 20 seconds into the drain period, the container should be rotated back to its original inverted position and held for 10 more seconds.

(6) Rinsing the container. The container should be pressure rinsed as follows.

(i) Remove cap. The cap should be removed from the container.

(ii) Invert container. The container should be inverted over a vessel with a volume sufficient to collect at least sixty (60) seconds worth of rinsate. Sufficient room between the container and the collection vessel should be allowed to permit the sampling of the rinsate.

(iii) Clean the threads. Before inserting the pressure rinsing device, the face of the opening and the threads of the neck of the container should be wiped clean.

(iv) Insert the pressure rinsing device. The pressure rinsing device should be inserted into the container at a point in the uppermost surface or a side of the container that will maximize the rinsing of the internal surfaces of the container. The location that the pressure rinsing device is inserted into the container should be recorded.

(v) Rinse the container. The water to the pressure rinsing device should be turned on. The pressure rinsing device should be moved and twisted to ensure that all of the internal surface has been rinsed.

(vi) Length of rinse. The container should be rinsed for at least thirty (30) seconds from when the water is turned on. Measure and record the <u>total</u> volume of rinsate collected.

(7) Sampling during the triple rinse procedure. The rinsate should be sampled using good laboratory techniques. Any pesticide residues should be maintained in suspension during the sampling process.

(i) Minimum sampling plan. The last two seconds (29^{th} and 30^{th} seconds) of rinsate should be collected, sampled and analyzed according to section (e)(8). The volume of rinsate collected during this period should be measured and recorded. The results of the analysis should be recorded.

(ii) Acceptable sampling plan. The rinsate during the 19^{th} – 20^{th} second interval and the 29^{th} – 30^{th} second interval should be collected, sampled and analyzed according to section (e)(8). The volume of rinsate collected during each time period should be measured and recorded. The results of the analyses should be recorded.

(iii) Good sampling plan. The rinsate during the 9^{th} – 10^{th} second interval, the 19^{th} – 20^{th} second interval, the 29^{th} – 30^{th} second interval and the 39^{th} – 40^{th} second interval should be collected, sampled and analyzed according to section (e)(8). The volume of rinsate collected during each time period should be measured and recorded. The results of the analyses should be recorded.

(**8) Analysis.** The samples should be analyzed for the pesticide active ingredient or ingredients using any validated analytical procedure suitable for the analysis of the active ingredient(s) in water. If no extraction or concentration procedures are employed and the water is analyzed directly, the method should have a level of sensitivity of 0.5 ppm or lower. The following information should be recorded:

(i) Whether the rinsate was chemically or physically treated to insure compound stability prior to analysis;
(ii) The sensitivity and the detection limit of the procedure; and
(iii) Whether the water samples were extracted.

(9) Waste disposal. The rinsate and the undiluted pesticide product collected in this study should be discarded utilizing approved disposal procedures.

(10) Format of the results. The results of the rinsate analysis should be reported as: (i) the rinsate concentration (mg/ml) and (ii) percent removal. Percent removal should be calculated as follows:

$$\frac{\text{Final rinsate concentration mg/ml}}{\text{Original concentration mg/ml}} = A$$

$$\text{Percent Removal} = [\, 1.0 - A \,] \times 100$$