UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

JAN 30 1990

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP#5F1639 Paraquat on Forage Grasses and Legumes. Paraquat Registration Standard - Response to Data Call-In. Evaluation of January 10, and June 23, 1989 Amendments. (MRID #s 411515-15, -16, -26, -42, -01, -09, -11, and 409437-01 through -04)[DEB #5561](HED Project #9-1799A)

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THRU: Robert S. Quick, Section Head
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TO: V. Walters/R. J. Taylor, PM Team 25
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ICI Americas requests reactivation of the petition presenting new data in response to deficiencies noted in the Paraquat Registration Standard (RS) issued on June 18, 1987. RD requests review of data to ascertain if deficiencies noted in the Paraquat RS have been resolved.

This petition was originally submitted by Chevron Chemical Company. ICI has neither proposed new tolerances, nor submitted new ICI labels in this data package.

EXECUTIVE SUMMARY OF CHEMISTRY DEFICIENCIES REMAINING TO BE RESOLVED

Directions for Use	- present new labels
Residue Analytical Method	- present new method - complete PMVs
Magnitude of Residue Crop Field Trial	- need additional data
Proposed Tolerance	- revise tolerance levels

CONCLUSIONS

DEB Conclusion on **Directions for Use**

1. No labels for Cyclone® and Gramoxone® Super were presented to show directions for paraquat use on the crops in this petition. The petitioner needs to present directions for paraquat use on non-grass and grass crop groups which match the pattern used to generate the crop field trial data. Alternately, the petitioner could present additional crop field trial data from any new proposed use(s) of paraquat on these crop groups. The absence of labels showing paraquat uses on crops in this petition is a data gap.

DEB Conclusion on **Analytical Residue Methods**

2 a. It has been previously concluded that the residue to be regulated is paraquat, **per se**.

b. At present there are adequate residue analytical methods available in FDA's PAM II to enforce the proposed and established tolerances for paraquat in forage grasses and legumes.

3. The new method 4B is suitable to gather paraquat residue data in poultry tissues and eggs assuming the following deficiencies are resolved:

   a. Define a satisfactory time period for stability of the paraquat working standard and type of glass for satisfactory storage of working standards.

   b. Define the fat level in tissues at which the hexane partitioning is or is not necessary.

   c. Present interference data to show that compounds similar to paraquat; e.g., diquat do not interfere; or present confirmatory step(s) or a procedure to confirm the identity and quantity of paraquat residues.

   d. Present additional method validation data plus supporting chromatographic data for paraquat residue at the limit of detection, intermediate levels, and highest levels found in chicken muscle, liver, fat, skin plus subcutaneous fat, and eggs.

4. Method 4B is not suitable to be an enforcement procedure until it completes a PMV and deficiencies noted above are resolved. Thus, part of the residue analytical method deficiency remains outstanding and continues unresolved.

5. The summary of method 1B, the Kennedy and Laws method of 1986 is not adequate. DEB needs a complete step by step procedure for review to decide if a valid analytical method

was used to gather the paraquat on plants residue data, and if the method is adequate for a PMV to satisfy the analytical method deficiency noted in the Paraquat RS, Table A, footnote 4, page 69. This part of the deficiency continues unresolved and remains outstanding. This is a data gap.

6. DEB defers judgment on the adequacy of all method validation data presented until we first review the detailed step by step procedure. The validation data represented in this petition may or may not be adequate to support the crop field trial data. Thus, the crop field trial data presented only tentatively resolves the Paraquat RS crop field trial data deficiencies.

DEB Conclusion on Storage Stability

7. There are tentatively adequate storage stability data on birdsfoot trefoil, corn and corn commodities that can be translated to support the forage grasses and legumes crop field trial residue data submitted in this petition. DEB concludes that residues of paraquat in corn grain, silage, forage, and fodder are stable in frozen storage at -20°C for at least 12 months, and in birdsfoot trefoil for 6 months, assuming concerns for the paraquat in plant analytical method are resolved.

8. DEB concludes that there are adequate data to show that residues of paraquat in eggs and chicken muscle are stable for at least 6 months of frozen storage, assuming analytical method deficiencies noted above are satisfactorily resolved.

9. The data are adequate assuming method concerns are resolved to support the residue data submitted in this amendment and resolve the storage stability RS deficiency noted for grass forage and legume forage.

DEB Conclusion on Magnitude of the Residue - Crop Field Trials

10a. The petitioner has tentatively presented adequate varietal and geographical representative crop field trial data from postemergence application at the suggested use rate on alfalfa forage, to show that paraquat residues should not exceed 60 ppm on alfalfa forage, not to exceed 100 ppm on alfalfa hay. The deficiency as described in footnote 50, Table A is tentatively resolved. No further data are required for this topic, assuming adequate directions for use are proposed and methods concerns are resolved.

b. For alfalfa meal the petitioner dried alfalfa hay to have dry matter greater than 90%. Thus, residues of paraquat on/in alfalfa meal are not expected to exceed 150 ppm.

c. In a revised Section F the petitioner needs to propose a feed additive tolerance for paraquat on alfalfa meal at 150 ppm once directions for paraquat use on alfalfa are presented and method concerns are resolved.

d. One of the petitioner's options is to propose revised tolerances in a new Section F for paraquat on alfalfa forage and alfalfa hay at the levels described in 10a above after adequate directions for use are presented and method concerns resolved.

11a. The petitioner has tentatively presented adequate varietal and geographically representative crop field trial data from postemergence application at the proposed use rate on clover to show that paraquat residue are not expected to exceed 75 ppm on clover forage, and not expected to exceed 150 ppm on clover hay. The deficiency as described in footnote 53, Table A, is tentatively resolved. No further data are required for this topic assuming adequate directions for use are presented and analytical method concerns noted are resolved.

b. One of the petitioner's options is to propose revised tolerances in a new Section F for paraquat on clover hay and forage at the levels described above after resolving directions for use and analytical method concerns.

12a. The petitioner has tentatively presented adequate varietal and geographically representative crop field trial data from postemergence application at the suggested use rate on birdsfoot trefoil to show that paraquat residues are not expected to exceed 60 ppm on birdsfoot trefoil forage and not expected to exceed 210 ppm on birdsfoot trefoil hay. The deficiency as described in footnote 52, Table A, is tentatively resolved. No further data are required for this topic assuming adequate directions for use are presented and analytical method concerns noted are resolved.

b. One of the petitioner's options is to propose revised tolerances in a new Section F for paraquat on birdsfoot trefoil forage and hay at the levels described above after resolving directions for use and analytical method concerns.

13a. There are adequate varietal and geographical representative data on the representative commodities and other commodities of the non-grass animal feeds crop group, with maximum residues not varying more than 5X from the maximum value observed for any crop, to support a crop group tolerance.

b. In a revised Section F, DEB suggests the petitioner consider proposing the following crop group tolerances in lieu of individual rac tolerances:

Non-grass Animal Feeds Forage Crop Group at 75 ppm and Non-grass Animal Feeds Hay Crop Group at 210 ppm.

14a. The varieties of grass used in western rangeland, western pasture, and eastern pasture paraquat field trials were not adequately identified. The petitioner needs to define the variety of grass used in each field trial in terms of the representative commodities of the grass forage and hay crop group.

b. The use of the term "mixed grasses" is not acceptable. For "mixed grasses" the petitioner needs to identify what varieties are present and in what approximate percentage each is present.

15a. The petitioner has tentatively presented adequate geographically representative crop field trial data from either preemergence or postemergence application at the suggested use rate of paraquat on western rangeland grass, eastern and western grass pastures to show that residues of paraquat on grass rangeland forage should not exceed 90 ppm, and should not exceed 30 ppm on grass pasture forage. Likewise when these grass forages are dried to make grass hay paraquat residues in grass rangeland hay should not exceed 175 ppm, and should not exceed 40 ppm on grass pasture. The deficiencies as described in footnotes 48 and 52, Table A, are tentatively resolved. No further data are required for these topics, assuming adequate directions for use are proposed and analytical method concerns noted are resolved.

b. Once the petitioner has adequately identified all varieties of grass used in the crop field trial in the submission, adequate directions for use are proposed and analytical method concerns noted are resolved, then the petitioner could propose in a revised Section F, new paraquat tolerance on grass rangeland forage and hay, and grass pasture forage and hay at the levels described above.

c. Once the varieties of grass are adequately identified in each of these crop field trials and there are adequate varietal representative data for the grass forage and hay crop group representative commodities present plus adequate directions for paraquat use and analytical method concerns are resolved, then the petitioner could propose a paraquat on grass forage crop group tolerance and a paraquat on grass hay crop group tolerance.

DEB Conclusion on **Magnitude of the Residue - Meat/Milk/Poultry/Eggs**

16a. DEB concludes the petitioner has conducted an adequate paraquat poultry feeding study. The feeding of a poultry diet containing alfalfa meal, soybean, corn, wheat and/or

sorghum grain with paraquat residues at suggested tolerances contributing up to 8 ppm paraquat has demonstrated the presence of low levels of paraquat in eggs and poultry tissue. DEB concludes the various proposed paraquat uses are categorized as 40 CFR §180.6(a)(1). The Paraquat RS deficiency is resolved. No further data are required for this topic.

b. In a revised Section F the petitioner now needs to propose secondary paraquat tolerance in eggs and poultry tissue as follows:

eggs	0.02 ppm
poultry meat, fat, and meat byproducts	0.05 ppm

17a. DEB concludes the petitioner's original ruminant paraquat feeding study is adequate. The feeding of a ruminant diet containing alfalfa/clover hay or forage, grass hay or forage and including corn, wheat, sorghum, soybean grains plus their straw, forage, or hay contributing from 175-180 ppm paraquat has demonstrated the presence of low levels of paraquat in milk and ruminant tissues. DEB concludes the various proposed paraquat uses are categorized as 40 CFR §180.6(a)(1).

b. In a revised Section F the petitioner now needs to amend the existing paraquat tolerances in meat as follows:

cattle, goats, hogs, horses & sheep - meat and fat	0.05 ppm
cattle, goats, hogs, horses & sheep - meat byproducts	0.3 ppm

RECOMMENDATION

For reasons cited in Conclusions 1, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, and 17 above DEB recommends against the establishment of revised paraquat tolerances on rangeland and pasture grass and hay at this time.

DETAILED CONSIDERATIONS

INTRODUCTION AND BACKGROUND

The Paraquat Registration Standard Product Chemistry and Residue Chemistry Chapters were completed in November, 1985, and forwarded to PM-25. The final RS document was sent to the registrant on June 18, 1987. Table A: Generic Data Requirements for Paraquat Dichloride attached to the Residue Chemistry Chapter listed a number of data gaps for forage grasses and legumes.

The Agency initially received a request on May 29, 1975, from the Ortho Agricultural Chemical Division of the Chevron

Chemical Company to establish tolerances for residues of paraquat on/in pasture and range grasses; grasses grown for hay or silage; small grains grown for hay, grazing, or silage; alfalfa; clover; birdsfoot trefoil; and crown vetch all at 60 ppm. The data submitted were adequately reviewed by J. Worthington in his October 17, 1975 memo in PP#5F1639. Deficiencies were noted in the nature of the residue in livestock, directions for use, and proposed tolerances. In a subsequent amendment reviewed by J. Worthington on September 10, 1976, DEB (a.k.a. RCB) recommended for tolerances of paraquat at 60 ppm in the forage and hay of alfalfa, birdsfoot trefoil, clover, grasses (pasture and rangeland), oats, rye, sorghum, vetch, and wheat.

Numerous tolerances have been established for paraquat. Some of these tolerances are on sorghum grain and forage at 0.05 ppm (N), corn grain, fodder, forage, fresh (K+CWHR) at 0.05 ppm (N), wheat grain at 0.05 ppm (N), and on soybeans and soybean forage at 0.05ppm (N). Paraquat tolerances range on the crops noted above from 0.05 ppm to 5 ppm on alfalfa, clover, birdsfoot trefoil, and grass pasture and range. Paraquat tolerances are also established for cattle, goats, hogs, horses, poultry, and sheep meat, fat, and meat byproducts (kidney and liver) ranging from 0.01 ppm (N) in all poultry products to 0.05 ppm in other animal tissues and to 0.3 ppm in kidney. Paraquat tolerances are established in milk and eggs at 0.01 ppm (N) (see 40 CFR §180.205).

Alfalfa and clover are the representative commodities of the non-grass animal feeds (forage, fodder, straw, and hay) crop group. Birdsfoot trefoil is one of the commodities listed in paragraph A of this commodity definition (see 40 CFR §180.34(f)(9)(xviii)(A and B). The Paraquat RS noted that a non-grass animal feed crop grouping tolerance was inappropriate as additional data are needed for alfalfa and clover.

The representative commodities of the grass forage, fodder, and hay crop group are Bermuda grass, bluegrass, and bromegrass or fescue. Any grass that is a member of the gramincae family, except sugarcane, is a commodity in this definition (see 40 CFR §180.34(f)(9)(xvii)(A and B). The Paraquat RS did not address the issue of a grass forage, fodder, hay crop grouping tolerance.

Tolerances with regional registration [24(c)] are established for paraquat at 0.05 ppm in cassava, pigeon peas, taniers, tyfon, and yams.

Food additive tolerances for paraquat are established on dried hops at 0.02 ppm (see 40 CFR §185.4700). Feed additive tolerances are established for paraquat on spent mint hay at 3.0 ppm and on sunflower seed hulls at 6.0 ppm (see 40 CFR §186.4700).

The most recent emergency exemptions (Section 18) for paraquat were on dry edible beans in Maine at 0.3 ppm (see memo

by W. L. Anthony on July 18, 1988 for 88-ME-02), and on grain sorghum in Louisiana at 5 ppm (see memo by L. Cheng on September 1, 1987 for 87-LA-08).

In his letter of January 10, 1989, the registrant requests reactivation of several petitions; eg, 9F2222 (wheat straw), 2F2672/2H5345 (soybeans and soybean hulls), 5F1591/6H5120 (sorghum and sorghum grain milling fractions), 5F1625/5H5088 (corn and corn gluten) and 5F1639 (forage grasses and legumes) to establish paraquat tolerances on various raw agricultural commodities (rac's) and their processed commodities. The registrant points out that the Toxicology Branch's Peer Review Committee has now reclassified paraquat to Category E (see Tox Branch memo of July 28, 1989). The request to reactivate these petitions was held back until TOX completed its second peer review.

DIRECTIONS FOR USE

The petitioner has presented no labels for Cyclone® and Gramoxone® Super to show directions for paraquat use on the crops in this petition. The petitioner needs to present directions for paraquat use on non-grass and grass crop groups which match the pattern used to generate the crop field trial data. Alternatively the petitioner could present additional crop field trial data from any new proposed use(s) of paraquat on non-grass and grass crop groups. The absence of labels showing a paraquat use(s) on the crops in this petition is a data gap.

RESIDUE ANALYTICAL METHODS

At present there are adequate residue analytical methods available to enforce the proposed and established tolerances for paraquat in forage grasses and legumes in PAM-II as of November 1, 1975.

In summary these methods involve acid reflux (H_2SO_4), cation exchange cleanup with NH_4Cl elution, reduction with dithionite to an unstable free radical that is measured spectrophotometrically at 394 nm. These methods are excessively long in their analysis time (5 samples in 5 work days). The paraquat RS for the residue analytical method concluded that (see page 32 of the Residue Chemistry Chapter):

> "The analytical method is acceptable for obtaining residue data but is only minimally adequate for enforcement/ monitoring purposes due to the fact that it is too long and cumbersome (ca. 5 work days per 6 determinations). The length of the procedure is caused by long digestion times, use of ion-exchange columns and the fact that the determination step is colorimetric. Therefore new methodology is required which will allow faster determination of paraquat in r.a.c.'s and food. Development

of an hplc procedure may provide a less lengthy method for quantitation of paraquat."

All samples of non-grass animal feeds crop group plus alfalfa meal, all samples of grass forage and hay crop group plus samples of birdsfoot trefoil were analyzed by Method 1B, the Kennedy and Laws method of 1986. The petitioner has presented only a detailed summary but not the step by step procedure. DEB needs a detailed step by step procedure to review to decide if a validated analytical method was used to gather the residue data reported and if the method is adequate for a PMV to satisfy the analytical method deficiency noted in the Paraquat RS Table A, footnote 4 on page 69.

DEB will defer judgement on all method validation data presented until we first review the detailed method. The validation data for paraquat on plants may or may not be adequate to support the crop field trial data. Thus the crop field trial data presented only tentatively resolves the crop field trial RS deficiencies. DEB suggests the petitioner present the detailed method for review along with all validation generated for this petition as one review document.

The Paraquat RS deficiency on analytical methods is not resolved and remains outstanding. This is a data gap.

One of the registrant's responses for residue analytical methods is in MRID #409437-01. The title of the method is "The Determination of Residues of Paraquat (PP148) in Animal Products; A High Performance Liquid Chromatographic Method" by M. Earl and A. D. Boseley, dated November, 1988 and coded Method 4B.

DEB Comments

The petitioner is presenting a new residue analytical method, 4B,to determine paraquat residues in animal tissues and eggs.

In summary, to a 25 gram sample of blended eggs, or finely chopped tissue add 50 ml of 10% trichloroacetic acid (TCA) and homogenize in a Silverson homogenizer for 5 minutes. After centrifuging for 10 minutes at 3000 rpm the supernatant is transferred to a flask. The TCA extraction is repeated 2X more saving each supernatant. High fat samples are partitioned with 100 ml hexane discarding the hexane. The petitioner needs to define the fat level at which this partition step is necessary. DEB feels the partitioning step will be necessary for most samples in order to insure rapid elution through the cation exchange column. The supernatant is filtered through glass fiber filter paper to remove particulates of extraction. The TCA extracts are diluted with 500 ml of deionized water and percolated through a cation exchange column at 5-10 ml/min. The cation exchange resin is 52-100 mesh, 0.15-0.3 mm particle size,

sodium form, 3.5 grams packed into a 1.0 cm(id) X50 cm glass column.

The paraquat is eluted off this column in the third wash of saturated NH_4Cl solution at a 1 ml/min flow rate in the first 50 ml. The petitioner cautions against flow rates higher than 1.0 ml/min.

The determination is by reverse phase ion pair hplc using a spherisorb S5P, 25 cm x 4.6 mm (id) column. The mobile phase is water: CH_3OH(9/1) with 3 modifiers, eg, 0.01 mol dm^{-3} Na-1-octane sulphonate, 0.8% o-H_3PO_4, and 1% diethylamine, at a flow rate of 1.5 ml/min, isocratic, pumped by a Waters 501 pimp. Samples were injected by a Waters 712 sample processor and detection was UV at 258 nm on a Waters M-481 LC-UV detector. Injections were 350 µl/0.5 gram aliquot.

Quantitation of results is by peak height, or peak area if computing integrators are available. The concentration of working standard of paraquat are 10 µg/ml and 0.1 µg/ml. A standard is rerun after a maximum of 6 sample injections. These working standards are stable in sol. NH_4Cl for extended periods provided they do not come into direct contact with sunlight. DEB considers these instructions for standard preparation too vague. The petitioner needs to define a satisfactory time period for paraquat working standards stability. Likewise the use of clear or colored glass for preparation and storage of paraquat standard should be addressed.

The petitioner claims a 0.005 ppm limit of detection (LD) for paraquat in eggs and animal tissues using this method.

DEB notes the petitioner did not present a confirmatory step(s) or procedure for paraquat. Considering the high degree of specificity of the procedure DEB will not request such, IF the registrant will provide interference study data to show that residues of closely related compounds; eg, diquat, do not interfere if they were present in similar levels. If diquat does interfere with paraquat in this procedure, then confirmatory step(s) or a procedure is necessary. DEB notes UAR's (Unidentified Analytical Responses) were present in all chromatograms. However, they are well removed from the elution point of paraquat.

The petitioner presented paraquat validation data in eggs for one sample fortified at 0.1 ppm with 0.083 ppm recovered. Chromatographic data were also presented for one standard at 0.1 µg/ml, a control egg, spiked egg and a treated egg showing 0.04 ppm. The mean recovery of paraquat from eggs is 86% ± 13%. For eggs the petitioner needs to present additional method validation data at the limit of detection 0.005 ppm, to 0.05 ppm in whole egg, and to 0.2 ppm in egg whites plus sufficient additional chromatograms showing paraquat at all of these levels. The number of determinations plus all raw recovery data that was used

to calculate the mean plus one standard deviation should be presented.

The recovery of paraquat from a 0.1 ppm spike in chicken muscle was 84%, and from a 0.1 ppm spike in chicken fat was 90%. From a 0.1 ppm paraquat spike in chicken kidney and liver paraquat recovery was 84% in liver and 88% from kidney. Paraquat spiked at 0.1 ppm in abdominal chicken fat has a 90% recovery and when spiked at 0.1 ppm into skin plus subcutaneous fat the paraquat recovery was 81%. For each of these recovery samples the petitioner provided chromatographic support showing a 0.1 µg/ml paraquat standard, an untreated control, the spike, and one actual treated sample. As with eggs the petitioner needs to present additional method validation data for paraquat in all poultry tissues at the limit of detection and at the highest level of paraquat reported. Additional supporting chromatographic data are needed for these samples. The number of determinations plus all raw recovery data that was used to calculate the reported sample means plus one standard deviation should be presented for chicken muscle, liver, abdominal fat, and skin plus subcutaneous fat. These data are not required for chicken kidney.

The petitioner presented additional method validation data plus chromatographic supporting data for paraquat recovery from ovine heart, bovine liver, and porcine kidney. Paraquat recovery from a 0.1 ppm spike in ovine heart was 90%, from a 0.1 ppm spike in bovine liver was 83%, and from a 0.1 ppm spike in porcine kidney was 87%. While these recovery data are not germane to a poultry feeding study they can be used to support the ability of the method to recover paraquat from a variety of animal tissues.

Method 4B is tentatively suitable to gather paraquat residue data in poultry tissues and eggs that are presented in this amendment, provided the deficiencies noted above can be satisfactorily resolved. The method 4B is not suitable to be an enforcement procedure until the deficiencies noted above in the partitioning step, working standard preparation, and additional validation data requirements are corrected and the method completes a successful petition method validation (PMV).

In the interest of speeding along the reregistration process DEB plans to initiate a PMV for method 4B to determine the suitability of method 4B for enforcing paraquat tolerances in eggs and tissues. DEB recognizes this method was developed in England using reagents and apparatus from English chemical supply houses. If US equivalents are not readily available for the reagents and apparatus, then the method will be returned to the petitioner for revising to list appropriate U.S. equivalent supplies. Likewise, if difficulties are encountered with any area that DEB notes a deficiency, then the PMV will be terminated and not be reinitiated until the petitioner corrects the problem.

STORAGE STABILITY

Samples of grass forage and hay (both eastern and western pasture use plus rangeland) were analyzed within 254 days (8 months). Alfalfa forage, hay, and meal samples were analyzed within 282 days (9+ months). Clover forage and hay samples were analyzed within 359 days (12 months) and birdsfoot trefoil forage and hay samples were analyzed within 83 days (3 months).

Storage stability data for paraquat in corn grain, forage, silage, and fodder have been presented and adequately reviewed (see memo in PP#5F1625/5H5088 by F. D. Griffith, Jr., in January 1990). The data show that residues of paraquat are stable in frozen storage at 20°C for at least 12 months. Paraquat fortification levels ranged from 0.05 ppm to 0.2 ppm. The paraquat recoveries from frozen stored corn ranged from 82% to 106%. Samplings were at 1, 3, 6, and 12 months. There are tentatively adequate storage stability data that can be translated to support the forage grasses and legumes crop field trial residue data submitted in this amendment assuming analytical concerns noted are resolved.

In this amendment the registrant is presenting new data in response to a deficiency noted in the Paraquat RS Residue Chemistry Chapter (see page 62):

> "All residue data requested in this 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."

The registrant's response is in MRID #411515-11, 409437-02 and -03. The title of the first study is "Stability of Weathered Residues of Paraquat on Birdsfoot Trefoil Forage and Hay in Frozen Storage" by E. M. Roper dated March 14, 1989, and coded TMR 0025B, WRC 89-91. The title of the second study is "Paraquat: Storage Stability of Residues in Frozen Eggs" by M. Earl and A. D. Boseley, dated December, 9988 and coded M4847B. The title of the third study is "Paraquat: Storage Stability of Residue in Frozen Hen Muscle Tissue" by Earl and Boseley dated December, 1988 and coded M4846B.

Samples of birdsfoot trefoil forage were prepared from a plot in Wisconsin treated in July 1988 at a rate of 0.45 lb a.i. paraquat/acre one application, broadcast, and harvested on the date of application. The birdsfoot trefoil forage samples were frozen within 2 hours of harvest. At the same time as treated forage was harvested control birdsfoot trefoil was also harvested. Birdsfoot trefoil hay samples were prepared by allowing the forage to air dry 3 days before freezing. All of the samples both control and treated forage and control and treated hay were stored frozen at ≤15°C in plastic lined paper bags until analyzed.

Samples of forage at 0 day PHI and the hay at its 0 day PHI were analyzed. Samples of forage and hay were removed from the freezer at intervals of 12 weeks (3 months) and 25 weeks (6 months). The petitioner has prepared adequate samples for analysis at 12, 18, and 24 months. No storage stability data for paraquat on birdsfoot trefoil forage and hay were reported for 12, 18, and 24 months of storage.

At each analysis date the petitioner spiked the control samples that day to validate the results of Method 1B. The validation data at 0 day PHI were 87-120% recovery from 5 ppm fortification and from the 50 and 200 ppm spikes recoveries of paraquat ranged from 86% to 89% (n = 4). The petitioner has presented sufficient validation data and chromatographic supporting data to show the method is suitable to gather residue data for this paraquat storage stability study.

Paraquat residues in birdsfoot trefoil forage at 0 day PHI were 59 ppm. After 12 weeks the paraquat residues were 62 ppm, then at 25 weeks the paraquat residue in birdsfoot trefoil forage was 63 ppm and 58 ppm. Paraquat residues in birdsfoot trefoil hay at 0 day PHI were 206 ppm. After 12 weeks the paraquat residues were 199 and 203 ppm then at 25 weeks the paraquat residues were 178 ppm and 213 ppm. DEB tentatively concludes the petitioner has presented the requested paraquat storage stability data to show that field incurred residues of paraquat at 60 ppm on birdsfoot trefoil forage and at 200 ppm on birdsfoot trefoil hay are stable for at least 25 weeks of frozen storage, assuming analytical method concerns noted for the method use to generate the crop field trial residue data are resolved. These storage stability data can be translated to other petitions reactivated by the registrant's January 10, 1989 letter. The registrant should be encouraged to provide the 12, 18, and 24 month paraquat on birdsfoot trefoil commodities storage stability data.

The registrant has presented two paraquat storage stability studies on livestock commodities; ie, eggs and chicken muscle. Basically the two studies were conducted in the same manner and DEB will review them as one. Control egg sample and muscle from control hens were used for the paraquat storage stability study. The control eggs were removed from their shells and the egg whites and yolks were thoroughly blended prior to fortification at 0.1 ppm. 25 gram sub samples of eggs were individually fortified prior to frozen storage at ≤18°C. The hen control muscle tissues were thoroughly minced prior to fortification at 0.1 ppm. 25 gram sub samples of chicken muscle were individually fortified prior to frozen storage at ≤18°C.

A 0 day "PHI" sample of fortified eggs and chicken muscle were analyzed by Method 4B, the Earl and Boseley method reviewed above. The petitioner has presented sufficient method validation data to show recovery of paraquat greater than 80% and supporting chromatographic data to show that the method is suitable to gather residue data for these storage stability studies. Samples

of frozen fortified egg were removed for analyses at 31 days (1 month), 91 days (3 months), and 178 days (6 months). Fortified frozen chicken muscle samples were removed for analyses on a similar schedule, ie, 30 days, 92 days, and 161 days.

Residues of paraquat in eggs on the day of fortification ranged from 0.09 ppm to 0.11 ppm (n=3). Then on day 31 paraquat residues in eggs were 0.09 ppm (n=3), on 91 day paraquat residues in eggs were 0.10 ppm (n=3), and at 178 day paraquat in eggs were 0.10 ppm and 0.11 ppm. Residues of paraquat in fortified chicken muscle on the day of fortification were 0.10 ppm (n=3). Then on day 30 paraquat residues in chicken muscle were 0.12 ppm (n=3), on day 92 were 0.11 ppm and 0.10 ppm, and at day 161 paraquat residues in fortified chicken muscle were 0.08 and 0.10 ppm.

DEB concludes the registrant has presented the requested paraquat storage stability data to show that residues of paraquat fortified at 0.1 ppm in eggs and chicken muscle are stable for at least 6 months frozen storage at ≤18°C.

The paraquat storage stability deficiency for paraquat residues in tissues as noted in the RS is resolved for PP#5F1639 (grasses) only.

MAGNITUDE OF THE RESIDUE - CROP FIELD TRIALS

Non-Grass Animal Feeds (Forage, Fodder, Straw and Hay) Crop Group

The registrant is presenting new residue data in response to deficiencies noted in the Paraquat *RS* reiterated below (see footnotes 50, 51, 52, and 53 in Table A attached to the Residue Chemistry Chapter on page 75, dated June 18, 1987):

> "(50) Data depicting residues in or on alfalfa forage, seed and hay from pastures and rangeland treated broadcast with the 2 lb/gal SC/L formulation at 0.5 lb cation/A. Samples must be harvested the day of application. Tests must be conducted in the major alfalfa growing regions of the country, including WI, CA, NE, PA and NC. Tolerances must be proposed for alfalfa forage, seed and hay, based on the results of the requested studies. (Note: The pending 60 ppm tolerance level may be acceptable.)"
>
> "(51) Residue data with meal processed from alfalfa bearing measurable weathered residues. If residues are found to concentrate in meal, an appropriate food/feed additive regulation would be required. However, final disposition of food/feed additive regulations is dependent upon the Agency's position regarding Delaney Clause issues."
>
> "(52) Data depicting residues in or on birdsfoot trefoil from pasture and rangeland treated with the 2 lb/gal SC/L formulation at 0.5 lb cation/A. Samples must be harvested the day of application. Tests must be conducted in MI and

WI. Tolerances must be proposed for forage and hay based on the results of the requested studies. (Note: The pending 60 ppm tolerance level may be acceptable.)"

"(53) Data depicting residues in or on clover from rangeland treated with the 2 lb/gal SC/L formulation at 0.5 lb cation/A. Samples must be harvested the day of application. Tests must be conducted in TX (13%), MO (11%), KY, NY, and OR. Tolerances must be proposed for residues in or on clover forage and hay based on the required data. (Note: The pending 60 ppm tolerance level may be acceptable.)"

The petitioner's response for paraquat residues on alfalfa is in MRID #411515-01. The title of the study is "Paraquat: Magnitude of Residues in Alfalfa Hay, Forage, and Meal" by E. M. Roper, and dated April 5, 1989. The report is coded TMR-0014B, WRC 89-080.

The petitioner's response for paraquat residues on birdsfoot trefoil is in MRID #411515-42. The study title is "Gramoxone Super: Residues of Paraquat in Birdsfoot Trefoil Forage and Hay, 1988" by E. M. Roper and dated December 9, 1989. The report is coded TMU 3672/B, WRC 89-130.

The petitioner's response for paraquat residues on clover is in MRID #411515-09. The study title is "Gramoxone Super: Residues of Paraquat in Clover Forage and Hay" by E. M. Roper and dated April 14, 1989. The report is coded TMR 0023B, WRC 89-89.

DEB Comments

Alfalfa

Five crop field trials representing 5 varieties of alfalfa were planted in the crop year 1988 one trial each in Wisconsin, California, Nebraska, Pennsylvania, and North Carolina. Residue data of paraquat on alfalfa from these 5 states represents alfalfa harvested on 6,075,000 acres out of a national alfalfa harvest on 25,485,000 acres (see Agricultural Statistics, 1988). Data from these 5 states satisfies the Paraquat RS deficiency for adequate geographical representative crop field trial data.

One plot of alfalfa in each of the above listed states was treated with Gramoxone® Super at a rate of 0.5 lb a.i. paraquat/acre postemergence, broadcast, ground equipment. Control plots were planted at each test location. Samples of alfalfa forage were harvested on the day of application for a 0 day PHI from both the test plot, and control plot (for comparison purposes). Forage alfalfa samples were air dried 2 to 7 days to make alfalfa hay.

All alfalfa forage samples were frozen within 8.5 hours of harvest and shipped frozen to ICI for analyses. The amount of alfalfa forage collected for analyses ranged from 2.5 lbs to 5

lbs and the amount of alfalfa hay collected for analyses ranged from 1.5 lbs to 5 lbs.

Alfalfa meal was produced from oven drying alfalfa hay at 101°C to about 95% dry matter. DEB reiterates that alfalfa hay dried to 90% dry matter could be substituted for alfalfa meal as a sample matrix.

Paraquat residues on alfalfa forage ranged from 22 ppm (2 samples under 30 ppm) to 60 ppm (1 sample above 40 ppm). From alfalfa forage air dried to produce alfalfa hay paraquat residues ranged from 46 ppm (2 samples under 60 ppm) to 93 ppm (1 sample above 85 ppm). The percent dry matter in these hay samples ranged from 46% to 88%. With the alfalfa hay further dried at 101°C to produce alfalfa meal the % dry matter of the alfalfa at 46% rose to 82% with other alfalfa meal samples dry matter content being greater than 95%. Paraquat residues in alfalfa meal ranged from 60 ppm to 144 ppm. DEB reiterates we accept residue data on alfalfa hay with dry matter at or greater than 90%, in lieu of alfalfa meal residue data.

Alfalfa forage and hay paraquat residue data initially received in PP#5F1639 (ibid) and in the Paraquat RS were found to be adequate. Paraquat residue data on alfalfa from 12 states east of the Rocky Mountains from both ground and aerial application representing 432 determinations on alfalfa forage and hay are now incorporated herein by reference. PHI's for these trials ranged from 21 days to 183 days.

One of the petitioner's options is to propose in a revised Section F new tolerances for paraquat on alfalfa forage at 60 ppm and on alfalfa hay at 100 ppm. Also a feed additive tolerance for paraquat on alfalfa meal at 150 ppm needs to be proposed. The petitioner tentatively presented adequate varietal and geographical representative crop field trial data to resolve deficiencies 50 and 51, Table A on magnitude of paraquat residues on alfalfa forage, hay, and meal, assuming adequate directions for use are presented and analytical method concerns are resolved.

Clover

Five crop field trials representing 4 varieties of clover were planted in the crop year 1988, one field trial each in Texas, Missouri, Kentucky, New York, and Oregon. Data from these 5 states satisfy the Paraquat RS deficiency for adequate geographical representative crop field trial data.

Plots of clover in Texas, Kentucky, New York and Oregon were treated with Gramoxone® Super at a rate of 0.5 lb a.i. paraquat/acre. The Missouri field trial was treated at a 0.5X rate or 0.25 lb a.i. paraquat per acre. All plots were treated one time, broadcast, ground application. Control plots of clover were planted at each test location. Samples of clover forage

were harvested at the day of application for a 0 day PHI from both the test plots and control plots (for comparison purposes). Extra clover forage samples were air dried 4 days to make clover hay.

All clover forage samples were frozen within 3 hours of harvest and shipped to ICI for analysis. The amount of clover forage collected for analysis ranged from 2.5 lbs to 3 lbs and the amount of clover hay collected for analysis ranged from 0.5 lbs to 1.1 lbs.

Paraquat residues on clover forage ranged from 22 ppm (3 samples under 30 ppm) to 74 ppm (2 samples above 50 ppm). Paraquat residues on clover hay ranged from 51 ppm (3 samples under 80 ppm) to 148 ppm (2 samples above 130 ppm).

The percent dry matter in clover hay ranged from 62% to 80%. The percent dry matter in clover was determined from drying clover forage in an oven at 100°C.

Clover forage and hay paraquat residue data initially reviewed in PP#5F1639 (ibid) and in the Paraquat RS were found to be adequate. Paraquat residue data from these studies are now incorporated herein by reference. PHI's on the tests from Iowa (2), Kentucky (2), Ohio (9), California (9), and Oregon (1) are greater than 30 days.

One of the petitioner's options is to propose in a revised Section F new tolerances for paraquat on clover forage at 75 ppm and on clover hay at 150 ppm. The petitioner tentatively has presented adequate varietal and geographically representative crop field trial data to resolve deficiency 53, Table A, on the magnitude of the paraquat residue on clover forage and hay, assuming adequate directions for use are proposed and method concerns are resolved.

Birdsfoot Trefoil

Two crop field trials representing 1 variety of birdsfoot trefoil were planted in the crop year 1988, one field trial each in Michigan and Wisconsin. Data from these 2 states satisfy the Paraquat RS deficiency for adequate geographical representative crop field trial data.

The plots of birdsfoot trefoil in Michigan and Wisconsin were treated with Gramoxone® Super at a rate of 0.5 lb a.i. paraquat/acre in Michigan and 0.45 lb a.i. paraquat/acre in Wisconsin. Both plots were treated one time, broadcast, ground equipment. Control plots of birdsfoot trefoil were planted at each test site. Samples of birdsfoot trefoil forage were harvested at the day of application for a 0 day PHI from both test plots and both control plots (for comparison purposes). Birdsfoot trefoil forage samples were air dried 2-3 days to make birdsfoot trefoil hay.

All samples of birdsfoot trefoil forage and hay were frozen within 3 days of harvest and shipped frozen to ICI for analyses. The amount of birdsfoot trefoil collected for analyses was 2.5-3.0 lbs.

Paraquat residues on birdsfoot trefoil forage were 43 ppm and 59 ppm. Paraquat residues on birdsfoot trefoil hay were 108 ppm and 206 ppm. The percent dry matter in birdsfoot trefoil hay was 79% and 88%.

Birdsfoot trefoil forage and hay paraquat residue data initially reviewed in PP#5F1639 (ibid) and in the Paraquat RS were found to be adequate. Paraquat residue data on birdsfoot trefoil from these studies in Iowa (2) and Ohio (8) are incorporated herein by reference.

One of the petitioner's options is to propose in a revised Section F new tolerances for paraquat on birdsfoot trefoil forage at 60 ppm and on birdsfoot trefoil hay at 210 ppm.

The petitioner has tentatively presented adequate varietal and geographical representative crop field trial data to resolve deficiency 52, Table A, on magnitude of the paraquat residue on birdsfoot trefoil forage and hay, assuming adequate directions for use are presented and residue analytical method concerns are resolved.

The petitioner's other option is to propose in a revised Section F crop group tolerances. There are adequate varietal and geographical representative crop field trial data for clover and alfalfa, the representative commodities of the non-grass animal feed group to support a crop group tolerance. Additional supporting paraquat crop field trial residue data are available for the commodities birdsfoot trefoil and crown vetch. These commodities are members of the non-grass animal feeds crop group. DEB suggests the petitioner consider proposing the following crop group tolerances once directions for use are proposed and method concerns are resolved:

Non-grass animal feeds forage crop group	75 ppm
and	
Non-grass animal feeds fodder, straw, or hay crop group	210 ppm

The maximum paraquat on forage residues for the crops in the non-grass animal feeds crop group range from 60 ppm to 75 ppm (1.25X). The maximum paraquat on hay residues for the crops in the non-grass animal feeds crop group range from 100 ppm to 210 ppm (2.1X). In both cases since maximum residues do not vary more than 5X from the maximum value observed for any crop in the group then a crop group tolerance can be established. In all cases the pattern of paraquat use consists of the same amount

applied (0.5 lb) one time, a 0 day PHI, broadcast application for non-grass animal feeds.

Grass Forage, Fodder, and Hay Crop Group

The registrant is presenting new residue data in response to deficiencies noted in the Paraquat RS reiterated below (see footnotes 48 and 52 in Table A attached to the Residue Chemistry Chapter on page 75, dated June 18, 1987):

> "(48) Data depicting residues in or on forage and hay from rangeland treated broadcast with 2 lb/gal SC/L formulation at 0.5 lb cation/A. Samples must be harvested the day of application. Tests must be conducted in CA, OR or WA in areas west of the Sierra Nevada and Cascade Mountains. Tolerances must be proposed for grass forage and hay based on the requested studies."
>
> and
>
> "Data depicting residues in or on forage and hay from pastures treated broadcast with the 2 lb/gal SC/L formulation at 0.5 lb cation/A. In areas east of the Rocky Mountains, tests conducted in TX, KY, NY, TN, AL and SD, and samples must be harvested 30 days post treatment.....A tolerance must be proposed for hay based on the results of the requested data, or a label restriction against the cutting treated grass must be proposed. (Note: The pending 60 ppm tolerance level may be acceptable.)"
>
> "(52) Data depicting residues in or on forage and hay from pastures treated broadcast with the 2 lb/gal SC/L formulation at 0.5 lb cation/A.....In areas west of the Cascade and Sierra Nevada Mountains, tests must be conducted in CA and OR or WA, and samples must be taken when growth has reached 3-6" height. A tolerance must be proposed for hay based on the results of the requested data, or a label restriction against the cutting treated grass must be proposed. (Note: the pending 60 ppm tolerance level may be acceptable.)"

The petitioner's response for paraquat residue data on grasses, western use rangeland is in MRID #411515-16. The title of the study is "Paraquat: Magnitude of Residues in Grass Forage and Hay (Western Use Rangeland)" by E. M. Roper and dated March 15, 1989. The report is coded TMR 0033B, WRC 89-99.

The petitioner's response for paraquat residue data on grasses, eastern pasture is in MRID #411515-15. The title of the study is "Paraquat: Magnitude of Residues in Grass Forage and Hay (Eastern Pasture)" by E. M. Roper, and dated March 3, 1988. The report is coded TMR-0032B, WRC 89-98.

The petitioner's response for paraquat residue data on grasses, western pasture is in MRID #411515-26. The title of the study is "Paraquat: Magnitude of Residues in Pasture Grass (Western Use Pastures)" by E. M. Roper, and dated May 18, 1989. The report is coded TMR 0044B, WRC 89-110.

DEB Comments

Western Rangeland

Four crop field trials of grass were planted in the crop year 1988 in California (1), Oregon (2), and Washington (1). Varieties of grass were not adequately identified. The petitioner needs to present an adequate description of the variety of grass used in each of these four trials. The petitioner needs to define each variety in each test plot in terms of the representative commodities for the grass forage and hay crop group. Use of the term "mixed grasses" is not acceptable. The petitioner needs to identify the varieties present and in what approximate percentages present. Residue data for paraquat on grasses from these 3 states satisfies the Paraquat RS deficiency for adequate geographical representative crop field trial data.

The test plots were treated with Gramoxone® Super, at a rate of 0.5 lb a.i. paraquat/acre, postemergence, one application, broadcast, ground equipment. Control plots of grass were planted at each test location. Samples of grass forage were harvested at the day of application for a 0 day PHI from both the test and control plots (for comparison purposes). Extra grass forage was harvested at each test plot then air dried 3 to 5 days to make grass hay.

All of these grass forage samples were frozen within 3 hours of harvest and shipped frozen to ICI for analyses. 1.5 lbs of grass forage was collected for analyses and 1-2.5 lbs of grass hay were collected for paraquat analyses.

Paraquat residues on western use rangeland grass forage ranged from 59 ppm to 81 ppm. Paraquat residues on grass hay made from the above forage ranged from 84 ppm to 162 ppm. The percent dry matter in grass hay ranged from 53% to 80%. The percent dry matter in grass was determined from drying grass forage and hay in an oven at 100°.

Eastern Pastures

Eight crop field trials of grass were planted in the crop year 1988 in Texas (2), Kentucky (2), New York (1), Tennessee (1), Alabama (1), and South Dakota (1). Varieties were not adequately identified. The petitioner needs to define each variety listed in each test plot in terms of the representative commodities for the grass forage and hay crop group. Residue data for paraquat on grasses from the 6 states satisfies the

Paraquat RS deficiency for adequate geographical representative crop field trial data.

The test plots of pasture grass forage were treated with Gramoxone® Super at a rate of 0.5 lb a.i. paraquat/acre, postemergence, one application, broadcast, ground application. Control plots of grass were planted at each test location. Samples of grass forage were harvested 28 days to 61 days after paraquat application (7 trials has PHI <35 days) from both the test and control plots (for comparison purposes). Extra grass forage was harvested at each test plot then air dried to produce grass hay.

All of the grass forage samples were frozen within 1 day of harvest and shipped frozen to ICI for analyses. 1 to 3 lbs of grass forage were collected for paraquat analyses and 2.5-3.0 lbs of grass hay were collected for paraquat analyses.

Paraquat residues on eastern pasture grass forage ranged from <0.05 ppm - 0.1ppm to 28 ppm with 5 trials having ≤3ppm paraquat residue on grass. Paraquat residues on grass hay made from the above forage ranged from 0.06 ppm to 40 ppm with 5 trials having ≤7 ppm paraquat residues on grass hay.

The petitioner has tentatively presented adequate geographical (but not adequate varietal) representative crop field trial data to resolve deficiency 48, Table A on the magnitude of the paraquat residues in/on western rangeland grass and eastern pasture grass, assuming adequate directions for use are presented and analytical method concerns noted are resolved.

Western Pastures

Four crop field trials of grass were planted in the crop year 1988 in California (2), Oregon (1), and Washington (1). Varieties of grass were not adequately identified. For consideration of a crop group tolerance the petitioner needs to define each variety in each test plot in terms of the representative commodities for the grass forage and hay crop group. Use of the term "mixed grasses" is not acceptable. The petitioner needs to identify the varieties of grass present in mixed grasses and in what approximate percentage is each variety present. Residue data for paraquat on grass from these 3 states satisfies the Paraquat RS deficiency for adequate geographical representative crop field trial data.

The test plots were treated with Gramoxone® Super at a rate of 0.5 lb a.i. paraquat per acre, preemergence, one application, broadcast, ground equipment. Control plots of grass were planted at each test location. Samples of grass forage were harvested after the grass had reached 3" to 6" height. The PHI for grass to reach this height ranged from 103 days to 196 days. Samples were harvested from both test and control grass plots. Extra

grass forage was harvested from each test plot then air dried to make grass hay.

All of the grass forage samples were frozen within 2 hours of harvest and shipped frozen to ICI for analyses. 1 lb to 2.2 lbs of grass forage were collected for paraquat analyses and 1 to 2 lbs of grass hay were collected for analyses.

Paraquat residues on western pasture grass forage were ≤0.025 ppm or at the limit of method's detection. Paraquat residues on grass hay made from the above forage ranged from <0.05 ppm to 0.2 ppm.

The petitioner has tentatively presented adequate geographical (but not adequate varietal) representative crop field trial data to resolve deficiency 52, Table A, on the magnitude of the paraquat residues in/on western pasture grasses assuming adequate directions for use are presented and method concerns noted above are resolved.

Grass forage and grass hay paraquat residue data initially reviewed in PP#5F1639 (ibid) and in the Paraquat RS were found to be adequate. Paraquat residue data from application rates of 0.25 lb a.i./acre to 1.0 lb a.i./acre producing paraquat residue on grass forage and hay ranging from ≤0.01 ppm to 150 ppm are now incorporated herein by reference.

Once the petitioner has adequately identified all varieties of grass, presented adequate directions for use and resolved analytical method concerns noted, then one of the petitioner's options could be to propose in a revised Section F new tolerances for paraquat on grass rangeland forage at 90 ppm, paraquat on grass pasture forage at 30 ppm. The petitioner also needs to propose paraquat tolerance on grass rangeland hay at 175 ppm and paraquat tolerance on grass pasture hay at 40 ppm.

The petitioner's other option is to propose a crop group tolerance for paraquat on/in the grass forage, fodder and hay crop group. However, first the petitioner needs to identify the variety of grass present in each paraquat on grass crop field trial submitted in this amendment, resolve method concerns and present adequate directions for use. Then, if adequate varietal representative commodity data are present a crop group tolerance could be proposed for paraquat residues on the grass forage crop group and the grass hay crop group.

MAGNITUDE OF THE RESIDUE - MEAT/MILK/POULTRY/EGGS

The registrant is presenting the results of a new paraquat poultry feeding study in response to a deficiency noted in the paraquat RS reiterated below:

> "A feeding study depicting residues in the fat, meat, and meat byproducts, of poultry fed paraquat cation at no less

than 1.4 ppm in the diet for 28 days and killed within 24 hours after the feeding period. Eggs must be collected and analyzed for residues of paraquat at intervals throughout the feeding period. A validated analytical method capable of detecting residues of paraquat cation at 0.01 ppm must be used."

The petitioner's response for paraquat residues in poultry is in MRID #409437-04. The title of the study is "Paraquat: Residue Transfer Study with Laying Hens Fed on a Diet Containing the Herbicide" by M. Earl and A. D. Boseley dated December 5, 1988. The study is coded RJ0703B.

DEB Comments

The petitioner presented the results of a 35 day paraquat poultry feeding study done during April-June 1988 at the Huntingdon Research Center, Ltd., Cambridgeshire, United Kingdom. 30 week old domestic hens, a brown hybrid egg laying strain (ISA) were used in the study. All pullets were examined to confirm they were in egg production. A total of 130 pullets were purchased from Elemby Farm Eggs, Petersborough, Cambridgeshire. Each bird was assigned a unique number that followed her through the study. Prior to the start of the study all pullets were weighed with weights ranging from 1.75 kg to 2.47 kg. Weights of each pullet were recorded every 7 days of the test beginning at -14 days (start of acclimation) to sacrifice or 49 days.

The pullets were divided into groups of 10 as follows: 3 groups of control designated A1, A2, and A3; 2 groups of 10 hens to receive a 6 ppm paraquat dose and designated B1 and B2; 2 groups of 10 hens to receive a 13 ppm paraquat dose and designated C1 and C2; 3 groups of 10 hens designated to receive a 30 ppm paraquat dose and designated D1, D2, and D3. All hens were acclimated 14 days prior to the start of the feeding study. The hens were housed in galvanized steel wire mesh cage measuring 1.2m x 1.5m on a concrete floor covered with wood shavings as bedding. The hens were in environmentally controlled conditions of 68% relative humidity, 17 hours of light and in a temperature range of 75°F to 81°F. The base diet was a special laying hen diet containing 3.8% crude fiber, 7.2% fat, and 24.3% protein; plus minerals and vitamin A and E. The mix, batch 3475, was analyzed to confirm no extraneous chemicals such antibiotics or growth promoting agents were present. Water was allowed ad libitum.

The base feed was mixed with water solutions of paraquat to give batches containing 6 ppm paraquat for test groups B1 and B2, 13 ppm paraquat for test groups C1 and C2, and 30 ppm for test groups D1, D2, and D3. The compound use of this study was paraquat dichloride, 99.6% purity. The dose level is based on the paraquat in concentration at 72.4%. Aliquots of feed were analyzed prior to feeding to be sure each test group of hens received the correct dose, and at the end of the study to be sure

paraquat levels in feed remained stable throughout the test period. The feed was given the pullets ad libitum.

5% of poultry diets can contain alfalfa meal. Thus, based on DEB's suggested tolerance of 150 ppm for alfalfa meal poultry could receive 7.5 ppm paraquat in their diet. The petitioner has also reactivated 4 other petitions each containing a poultry feed item. DEB suggests a 2 ppm paraquat tolerance on soybeans. Soybeans can be 20% of poultry diets for a potential paraquat feeding level of 0.4 ppm. Sorghum grain, corn grain, and wheat grain can be up to 80% of a poultry diet. At 60% to 70% of poultry diets the potential paraquat feeding level is 0.03 ppm - 0.035 ppm. Grass forage and grass hay are not poultry feed items. The maximum paraquat level in a poultry diet containing 5% alfalfa meal (5% x 150 ppm), 20% soybean grain (20% x 2 ppm), and the remaining 75% consisting of corn, sorghum, or wheat (70% x 0.05 ppm) would be 8 ppm. DEB recognizes this diet is artificial but none the less maximizes potential paraquat exposure to laying hens.

Eggs from each group were collected daily. Eggs at the start of acclimation period from days -14 to -3 were discarded. From each group on day -2 to day 21 eggs were collected daily, pooled, with yolks and whites together. From day 22 to 35 eggs were collected from both 6 ppm and 13 ppm paraquat feeding groups plus 1 control and 1 30 ppm paraquat feeding group then pooled with yolks and whites together. A second control group and 30 ppm paraquat feeding group (to day 35) to day 42 had eggs collected daily, then pooled, yolks and whites together. The third control and 30 ppm paraquat feeding (to day 35) to day 49 had eggs collected daily, then pooled, yolks and whites were kept separate.

Review of the body weights throughout the study shows a decrease in body weights during the acclimation period from 6% to 20%. During the actual paraquat feeding half of the test groups recovered some weight while the other 5 groups lost some additional weight. There is no evidence of paraquat feeding having an effect on weights. Food consumption among the 10 test groups was variable, but there is no evidence of paraquat feeding having an effect on feed consumption. Egg production did not show any differences due to paraquat dose. Likewise, total egg weights per group did not show any differences due to paraquat dose.

Pullets from one control group, both groups of 6 ppm and 13 ppm paraquat feeding and 1 group from the 30 ppm paraquat feeding were sacrificed at 35/36 days. Then one group of control hens and one group 30 ppm paraquat feeding were sacrificed at 42 days (7 days depuration) and 49 days (14 days depuration). At sacrifice the hens were plucked then samples of breast and thigh muscle, all of the liver, both kidneys, abdominal fat, and skin plus subcutaneous fat were removed for analyses. All of the samples from the 3 30 ppm paraquat feeding groups and one control

group were stored individually frozen at -18°C and were analyzed within 6 months. From 2 control groups and both paraquat 6 ppm and 13 ppm feeding groups the muscle samples were stored separately, while liver, kidney, fat (abdominal), skin and subcutaneous fat samples were composited in 2 sub samples per group.

The results of the analyses on tissues from method 4B (Earl and Boseley) are summarized as follows:

Tissue	Control (ppm)	6 ppm Paraquat (ppm)	13 ppm Paraquat (ppm)	30 ppm Paraquat (ppm)
whole eggs	<0.005	<0.005-0.01	<0.005-0.02	<0.005-0.05
egg whites	<0.005	--	--	<0.005
egg yolks	<0.005	--	--	<0.005-0.19
liver	<0.005	<0.005	<0.005	<0.005-0.1
kidney	<0.005	<0.005-0.02	0.04-0.06	<0.005-0.14
abdominal fat	<0.005	<0.005	<0.005	<0.005
skin plus subcutaneous fat	<0.005	<0.005	<0.005-0.02	<0.005-0.02
muscle	<0.005	<0.005	<0.005-0.01	<0.005-0.05

Comments are necessary to further amplify the values reported above. Only one sample of eggs from the 6 ppm paraquat feeding was positive and that was on day 35. Paraquat residues in eggs from the 13 ppm paraquat dose were positive at 0.01 ppm on day 5 and plateaued at 0.01 ppm-0.02 ppm to the end of the feeding study. Paraquat residues in eggs from the 30 ppm feeding also showed first at 5 days and plateaued at 0.03-0.05 ppm level. Only one egg sample was at 0.06 ppm (day 28). Paraquat did not remain in eggs after the feeding was stopped with paraquat values being <0.005 ppm on day 42, or 7 days depuration. In egg yolks paraquat residues plateaued from day 28 to day 35 at 0.12-0.13 ppm, declining to 0.1 ppm on 4 days depuration and to <0.005 ppm 7 and 14 days depuration.

Depuration of paraquat was not only rapid in eggs but also no paraquat residues were detected in liver, kidney fat, and skin plus subcutaneous fat at 7 days after feeding 30 ppm paraquat. However, depuration of paraquat from muscle was not rapid as residues were essentially the same 14 days after the last paraquat feeding.

DEB concludes the petitioner has conducted an adequate paraquat poultry feeding study. Since the poultry feeding study has demonstrated the presence of low levels of paraquat in eggs and poultry tissues, any poultry feed use of a paraquat treated raw agricultural commodity must necessarily be characterized within 40 CFR 180.6(a)(1) or (a)(2). DEB concludes the paraquat uses are 180.6(a)(1) in that the feeding study has shown that finite paraquat residues will actually be incurred in eggs and poultry from the feed use of a paraquat treated raw agricultural commodity (or its byproduct). In a revised Section F the

petitioner needs to propose the following secondary paraquat tolerances:

eggs	0.02 ppm
poultry meat, fat, and meat byproducts	0.05 ppm

The poultry paraquat feeding study deficiency as described in the Paraquat RS is resolved. No further data are required for this topic in this petition.

With the reactivation of 5 paraquat petitions each containing ruminant feed items DEB feels it is prudent to rereview the results of a paraquat ruminant feeding study (ibid). In summary, three groups of cows were fed paraquat at a 25 ppm dose, a 80 ppm dose, and a 170 ppm. The paraquat fed to these cows was from weathered residues on field dried treated grass. The results on tissues gathered within 24 hours of the last feeding were generated from existing PAM-II methods are as follows:

	25 ppm Paraquat	80 ppm Paraquat	170 ppm Paraquat
milk	<0.1-0.6 ppb	<0.1-0.6 ppb	<0.1-0.5 ppb
liver	<0.01 ppm	<0.01 ppm	<0.01-0.09 ppm
kidney	0.06 - 0.19 ppm	0.14-0.31 ppm	0.21-0.31 ppm
fat	<0.01 ppm	<0.01 ppm	<0.01-0.02 ppm
muscle	<0.01 ppm	<0.01 ppm	<0.01-0.03 ppm

DEB concludes the original paraquat ruminant feeding study is still adequate. 80% of dairy cattle diets can be alfalfa forage or hay. Thus the potential contribution of paraquat from DEB's suggested revised tolerance is 160 ppm from alfalfa/clover hay or 60 ppm from alfalfa forage. From grass forage at 75% of cattle diets the potential paraquat feeding level based on DEB's 90 ppm suggested tolerance is 67.5 ppm and from grass hay based on DEB's 175 ppm suggested tolerance is 122.5 ppm. Paraquat levels on wheat grain, corn grain, and/or sorghum grain at 0.05 ppm could contribute from 0.025 ppm (50% wheat) to 0.04 ppm (80% from corn grain or sorghum grain). Use of wheat straw at a 1 ppm paraquat level could contribute 0.1 ppm to cattle diet (10% of diet at 1 ppm). From corn forage at DEB suggested tolerance of 3 ppm cattle could receive 0.75 ppm (25% of diet at 3 ppm) and from a 6 ppm paraquat tolerance corn fodder cattle could receive 1.5 ppm (25% of diet at 6 ppm). The paraquat contribution from sorghum hay could be 0.05 ppm based on 25% in the diet from a 0.2 ppm paraquat tolerance. Sorghum silage can be 25% of cattle diets thus 25% of 0.4 ppm is 0.1 ppm potential paraquat in cattle diets sorghum silage. Soybean grain can be 25% of cattle diets, thus from a 2 ppm paraquat tolerance on soybean grain cattle can receive up to 0.5 ppm. Significant contributions of paraquat to cattle diets from soybean forage and/or hay is possible. Soybean hay or forage can be 40% of cattle diets. From DEB's suggested paraquat tolerance on soybean forage at 250 ppm cattle could receive 100 ppm paraquat in their diet from eating paraquat

treated soybean forage and from a 150 ppm paraquat tolerance on soybean hay cattle could receive up to 60 ppm from eating soybean paraquat treated hay. DEB recognizes a cattle diet from various combinations of feed items above is artificial. DEB feels a diet of 80% alfalfa/clover hay contributing 160 ppm paraquat plus 20% of the diet from grass forage is reasonable. Decreasing either alfalfa hay or grass forage with increase from corn, wheat, sorghum, or soybean grain and their forages or hays is possible, but will decrease the potential amount of paraquat ingested. DEB expects a 175-180 ppm potential paraquat feeding level in ruminants based on the levels of paraquat residues reported in the 5 reactivated petitions. While 170 ppm was the maximum paraquat fed cattle DEB concludes the original ruminant study is still adequate. DEB feels a new paraquat ruminant feeding study is not necessary as there were no significant changes in tissue levels between 80 ppm and 170 ppm paraquat feedings.

Since the ruminant paraquat feeding study has demonstrated the presence of low levels of paraquat in milk and cattle tissues, any ruminant feed use of a paraquat treated raw agricultural commodity must necessarily be characterized within 40 CFR 180.6(a)(1) or (a)(2). DEB concludes the paraquat uses are 180.6(a)(1) in that the feeding study has shown that finite paraquat residues will actually be incurred in milk and cattle tissues from the feed use of a paraquat treated raw agricultural commodity (or its byproducts). In a revised Section F the petitioner needs to propose the following secondary paraquat tolerances:

cattle, goats, hogs, horses, and sheep meat and fat	0.05 ppm
cattle, goats, hogs, horses, and sheep meat byproducts	0.3 ppm

H7509C:DEB:Reviewer(FDG):CM#2:Rm814B:5570826:mb:1/23/09:
edited:fdg:1/24/90.

cc:R.F.,Circu(7),PP#5F1639,Reviewer(FDG),Paraquat Reg. Std. File,
ISB/PMSD(Eldredge),TOX-HF Support.

RDI:Section Head:R.S.Quick:1/26/90:R.A.Loranger:1/30/90.