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OFFICE OF PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

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HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
EPA SERIES 361

MEMORANDUM

Date: 4/17/2008

SUBJECT: **Ingredient:** Tribenuron Methyl **Title:** Label Amendments and Petition for Tolerances on Barley Hay, Oat Forage and Hay, and Wheat Forage and Hay. Summary of Analytical Chemistry and Residue Data.

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Decision No.: 379517
Petition No.: 7F7220
Risk Assessment Type: None
TXR No.: None
MRID No.: 47138301, 47138302, 47138303

DP Barcode: 342085
Registration No.: 352-632, 352-714, 352-610
Regulatory Action: Section 3
Case No.: None
CAS No.: 101200-48-0
40 CFR: 180.451

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MAY 1 REC'D, 2008 RCC 514

Executive Summary

Tribenuron methyl (methyl-2-[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)methylamino] carbonyl]amino]sulfonyl]benzoate) is a sulfonylurea herbicide (Group 2) registered for postemergence application to barley, canola, cotton, flax, grasses (grown for seed), oats, sunflowers, and wheat for selective control of broadleaf weeds. It is also registered for use as a preemergence burndown broadcast application for wheat and barley, and as a preplant or at-planting burndown application for cotton, field corn, rice, grain sorghum, and soybeans.

There are currently ten end-use products (EPs) containing tribenuron methyl registered to E.I. DuPont de Nemours and Company (DuPont) for pre- and postemergence uses on barley, oats, and wheat. These EPs are formulated as either dry flowables (DF) or water soluble granules (SG) and contain 2.4-75% tribenuron methyl. Two of these EPs, a 75% DF and 50% SG, contain only tribenuron methyl, while the remaining EPs are mixtures of tribenuron methyl with thifensulfuron methyl, metsulfuron methyl, and/or dicamba. DuPont has proposed amending the current use directions for barley, oats, and wheat to allow for the harvest of hay and forage. Three example labels were provided with the proposed revisions, including a 50% SG (EPA Reg. No 352-632) and two multiple active ingredient formulations, a 16.67% SG (EPA Reg. No 352-714) and a 18.75% DF (EPA Reg. No 352-610). The registered uses allow for up to two postemergence broadcast applications to barley and wheat prior to flag leaf emergence at rates totaling 0.016 lb ai/A, and for a single broadcast postemergence application to oats prior to flag leaf emergence at up to 0.006 lb ai/A. Preemergence applications are also allowed on all three crops, but the maximum total seasonal rate is 0.016 lb ai/A. Applications may be made using ground or aerial equipment and may include the use of either a non-ionic surfactant (NIS) or a crop-oil concentrate (COC). For all three crops, the proposed pre-harvest interval (PHI) is 7 days for forage and 30 days for hay. In conjunction with these label amendments, DuPont is proposing the following permanent tolerances for tribenuron methyl:

Barley, hay	0.3 ppm
Oat, forage	0.3 ppm
Oat, hay	0.8 ppm
Wheat, forage	0.3 ppm
Wheat, hay	0.3 ppm

Permanent tolerances are established for residues of tribenuron methyl in/on barley, canola, field corn, cotton, flax, grass, oat, rice, sorghum, soybean, sunflower, and wheat commodities at levels ranging from 0.02 to 0.10 ppm [40 CFR§ 180.451 (a) and (c)]. No tolerances are established for residues in either animal commodities or rotational crops.

The nature of the residue in plants is adequately understood based on metabolism studies with wheat, canola, and cotton. The residue of concern in plant commodities is tribenuron methyl *per se*. The nature of the residue in livestock is also adequately understood based on the goat metabolism study. HED has previously determined that a poultry metabolism study is not required to support the existing uses, as residues in/on poultry feedstuffs are nondetectable, even at exaggerated rates.

An HPLC photo-conductivity detection method (Method AMR 337-85, Revision A) is available for enforcement of tolerances for residues of tribenuron methyl in/on cereal grains and straw. An LC/MS method (DuPont Method 1381) is also available for enforcing tolerances for residues of tribenuron methyl in/on canola, cotton, and flax commodities, and the validated limit of quantitation (LOQ) for this method is 0.02 ppm. The LC/MS method is also suitable for enforcing tolerances in/on corn grain, sorghum grain, and soybean seed. The FDA multiresidue methods are not suitable for tolerance enforcement because tribenuron methyl is not recovered through any of the FDA Multiresidue Method Testing protocols.

In the barley, oat, and wheat field trials, forage and hay samples were analyzed for residues of tribenuron methyl using an LC/MS/MS method (DuPont Method 13412, Revision No. 1). An earlier version of this method (DuPont Method 13412) was validated for the analysis of tribenuron methyl in oily crop matrices, and has undergone a successful independent laboratory trial. For this method, residues are extracted from forage and hay with acetonitrile:potassium phosphate buffer (pH 7), and cleaned up by solvent partitioning and elution through an ENV SPE cartridge. Residues are determined by LC/MS/MS using the m/z 396→ 155 transition for quantitation. The method was adequately validated for data collection in conjunction with the analysis of field trial samples. The validated LOQ is 0.01 ppm for tribenuron methyl in/on forage and hay.

Adequate storage stability data are available indicating that tribenuron methyl is stable in frozen corn forage for up to 6 months and in frozen wheat grain and straw for up to 21 months. These data support the 4-5.8 month frozen storage durations incurred for forage and hay samples in the barley, oat, and wheat field trials.

Using the established and recommended tolerances for tribenuron methyl in livestock feedstuffs, the maximum dietary burden (MDB) of livestock for tribenuron methyl residues was calculated to be 0.12 ppm for beef cattle, 0.54 ppm for dairy cattle, and 0.05 ppm for both poultry and swine. As the proposed tolerances will have no impact on the dietary burden for poultry, HED's previous conclusion regarding residues in poultry commodities remains unchanged. A poultry feeding study and tolerances on poultry commodities are not required, as there is no reasonable expectation of finite residues occurring in poultry commodities. Based on the results of the goat metabolism study, in which residues of tribenuron methyl were not detected in milk and tissues following dosing at 6.7 ppm (12x the MDB for dairy cattle and 56x the MDB for beef cattle), there is also no expectation of quantifiable residues occurring in milk, cattle meat, or cattle meat byproducts. Therefore, a cattle feeding study is not required for this petition, and tolerances are not required for residues in cattle commodities.

Although several deficiencies were noted in the submitted field trials, the available field trial data are acceptable, and support the existing use patterns for tribenuron methyl on barley, oats, and wheat. Adequate numbers of tests were conducted on each crop in the appropriate geographical regions using an SG formulation. Adequate numbers of oat and wheat forage samples were collected around the proposed 7-day PHI. Although several of the hay samples were not collected at the appropriate PHI, sufficient numbers of hay samples are available for barley (20 samples), oats (24 samples), and wheat (30 samples) harvested around the proposed 30-day PHI.

The oat field trial data will be used to assess tolerances on oat forage and hay; however, the oat field trials were conducted at 2.6x the maximum postemergence rate for oats. Although the maximum total preplant rate on oats is 0.016 lb ai/A, the available labels allow for only one postemergence application to oats at up to 0.006 lb ai/A. Following a single broadcast foliar application of tribenuron methyl (SG) to oats at 0.015-0.016 lb ai/A (2.6x postemergence rate), residues were <0.01-0.043 ppm in/on forage harvested at 5-8 DAT and <0.01 ppm in/on hay harvested at 25-33 DAT. Average residues were 0.014 ppm in/on forage at 5-8 DAT and 0.01 ppm in/on hay at 25-35 DAT.

Following a single broadcast foliar application of tribenuron methyl (SG) to barley at 0.015-0.016 lb ai/A (1x rate), residues were <0.01-0.36 ppm in/on hay harvested at 22-31 days after treatment (DAT), and residues in/on hay averaged 0.036 ppm.

Following a single broadcast foliar application of tribenuron methyl (SG) to wheat at 0.015-0.016 lb ai/A (1x rate), residues were <0.01-0.25 ppm in/on forage harvested at 6-8 DAT and <0.01-0.42 ppm in/on hay harvested at 25-35 DAT. Average residues were 0.049 ppm in/on wheat forage at 6-8 DAT and 0.029 ppm in/on wheat hay at 25-35 DAT.

In residue decline tests on barley, oats, and wheat, tribenuron methyl residues in/on both forage and hay declined rapidly from 0 to 7 DAT and then were either non-detectable or remained relatively steady from 14 to 45 DAT.

Requirements for residue data on processed commodities of barley, oats, and wheat have been previously satisfied. HED has concluded that wheat, barley, and oat processing data are not required because residues of tribenuron methyl were not detected in/on wheat grain following applications up to 4x the maximum seasonal rate, and applications at higher rates would be phytotoxic.

Adequate confined rotational crop studies are available reflecting soil applications of [^{14}C]tribenuron methyl at 0.028-0.031 lb ai/A (1-1.8x maximum seasonal rates). As residues of tribenuron methyl were not detected in any commodities from rotational crops planted 30 days after application, the data support a minimum 30-day plantback interval for all crops without registered uses. Labels for tribenuron methyl currently specify minimum plantback intervals of no less than 60 days for canola, rape, and sugar beets and 45 days for all other crops not listed on the label; therefore, additional rotational crop data and tolerances are not required.

Regulatory Recommendations and Residue Chemistry Deficiencies

No major deficiencies were noted in the subject petition that would preclude establishing permanent tolerances for residues of tribenuron methyl on the proposed commodities. The available field trial data are adequate and support the 7 and 30 day PHIs being proposed for forage and hay. HED recommends in favor of establishing permanent tolerances for tribenuron methyl at 0.4 ppm on barley hay, 0.05 ppm on oat forage and hay, 0.3 ppm on wheat forage, and 0.5 ppm on wheat hay. However, the petitioner should resolve the minor deficiencies listed below.

- Labels for all EPs containing postemergence uses of tribenuron methyl on wheat, barley, or oats need to be amended to specify minimum PHIs of 7 days for forage and 30 days for hay. In addition, the labels for the 50% SG (EPA Reg. No. 352-632) and the 18.75% DF (EPA Reg. No. 352-610) should be clarified to indicate minimum PHIs of 7 days for forage, 30 days for hay, and 45 days for the mature crop.

- A revised Section F is needed. This Section F should propose the following revised tolerances: barley hay (0.4 ppm), oat forage (0.05 ppm), oat hay (0.05 ppm), and wheat hay (0.5 ppm). The proposed 0.3 ppm tolerance for wheat forage is appropriate.

Background

Tribenuron methyl is a sulfonylurea herbicide (Group 2) registered for postemergence application(s) to barley, canola, cotton, flax, oats, sunflowers, wheat, and grasses grown for seed, for selective control of broadleaf weeds. It is also registered for use as a preemergence burndown broadcast application for wheat and barley, and as a preplant or at-planting burndown application for cotton, field corn, rice, grain sorghum, and soybeans. A summary of the status of residue chemistry data requirements for tribenuron methyl was issued 6/24/04 (Memo, D304059, R. Griffin).

For the existing postemergence uses on barley, oats, and wheat, DuPont is now proposing PHIs for forage and hay along with new tolerances (PP#7F7220). The chemical structure and nomenclature of tribenuron methyl are presented in Table 1. The physicochemical properties of the technical grade of tribenuron methyl are presented in Table 2.

Table 1. Tribenuron Methyl Nomenclature.

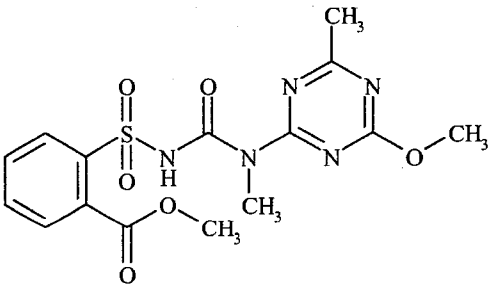
Chemical structure	
Common name	Tribenuron methyl
Company experimental name	DPX-L5300
IUPAC name	methyl-2-[4-methoxy-6-methyl-1,3,5-triazin-2-yl(methyl)carbamoyl-sulfamoyl]benzoate
CAS name	methyl-2-[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)methylamino]carbonyl]amino]sulfonyl]benzoate
CAS registry number	101200-48-0
End-use product (EP)	50% SG (DuPont™ Express® Herbicide; EPA Reg. No. 352-632); 37.5% DF (DuPont™ Ally® Extra; EPA Reg. No. 352-610); 33.3% SG (DuPont™ Harmony® Extra; EPA Reg. No. 352-714)

Table 2. Physicochemical Properties of Tribenuron Methyl.		
Parameter	Value	Reference
Melting point/range	142°C	MRID 47138301
pH (20°C)	4.64	
Density (g/cm^3 at 19.6°C)	1.46	
Water solubility (g/L at 20°C)	pH 5 0.0489 pH 7 2.04 pH 9 18.3	
Solvent solubility (g/L at 20°C)	Acetone 39.1 Acetonitrile 46.4 Dichloromethane >250 Dimethylformamide 98.2 Ethyl acetate 16.3 n-Heptane 0.02 Methanol 2.59 n-Octanol 0.383 Xylene 13.1	
Vapor pressure (25°C)	2.7×10^{-7} mm Hg	
Dissociation constant (pK_a)	5.0	
Octanol/water partition coefficient Log(K_{OW}) at 20°C	pH 5 2.60 pH 7 0.78 pH 9 0.30	
UV/visible absorption (max., λ)	pH 1.66 200, 231 nm pH 7 201, 256 nm pH 11.72 208, 256 nm	

860.1200 Directions for Use

There are currently 10 active EPs containing tribenuron methyl that are registered to DuPont for postemergence uses on wheat, barley, and oats (Table 3). These EPs are formulated as either DFs or SGs. Two of the EPs contain only tribenuron methyl at 50% or 75% a.i., and five of the EPs contain combinations of tribenuron methyl (10-37.5% a.i.) and thifensulfuron methyl (25-50% a.i.). Two formulations contain combinations of tribenuron methyl (13.6-18.75% a.i.), thifensulfuron methyl (27.3-37.5% a.i.), and metsulfuron methyl (10.9-15% a.i.). The final formulation is comprised primarily of dicamba (63.6% a.i.), with ≤4.7% each of thifensulfuron methyl, tribenuron methyl, and metsulfuron methyl.

Table 3. Tribenuron Methyl End-Use Products Registered to DuPont for Postemergence Uses on Wheat, Barley and Oats.

EPA Reg. No.	Trade Name	Form.	% Active Ingredients			
			Tribenuron methyl	Thifensulfuron methyl	Metsulfuron methyl	Dicamba (sodium salt)
352-509	DuPont™ Express® XP	DF	75%	--	--	--
352-610	DuPont™ Ally® Extra	DF	18.75%	37.5%	15.0%	--
352-611	DuPont™ Harmony® Extra XP	DF	25%	50%	--	--
352-617	DuPont™ HER11	DF	37.5%	37.5%	--	--
352-632	DuPont™ Express® SG	SG	50%	--	--	--
352-641	DuPont™ Affinity™	SG	10%	40%	--	--
352-661	DuPont™ Affinity™ GBF92	SG	25%	25%	--	--
352-714	DuPont™ Harmony® Extra	SG	16.67%	33.33%	--	--

Table 3. Tribenuron Methyl End-Use Products Registered to DuPont for Postemergence Uses on Wheat, Barley and Oats.

EPA Reg. No.	Trade Name	Form.	% Active Ingredients			
			Tribenuron methyl	Thifensulfuron methyl	Metsulfuron methyl	Dicamba (sodium salt)
352-715	DuPont™ Ally® Extra	SG	13.6%	27.3%	10.9	--
352-751	DuPont™ Agility™ SG	SG	2.4%	4.7%	1.9%	63.6%

With this petition, DuPont provided amended labels for three of these EPs: the 50% SG (EPA Reg. No. 352-632), an 18.75% DF (EPA Reg. No. 352-610), and a 16.7% SG (EPA Reg. No. 352-714). The amended use directions on barley, oats, and wheat for these EPs are summarized in Table 4. The amended use directions do not alter the current use patterns or application rates for tribenuron methyl on wheat, barley, and oats, but amend the harvest and grazing restrictions for forage and hay. The prohibition against the grazing or feeding of treated forage and hay has been replaced with minimum PHIs for the grazing or feeding of forage (7-day PHI) and hay (30-day PHI) from wheat, barley and oats.

Table 4. Summary of Directions for Use of Tribenuron Methyl.

Applic. Timing, Type, and Equip. [1]	Formulation [EPA Reg. No.]	Applic. Rate (lb ai/A)	Max. No. Applic. per Season	Max. Seasonal Applic. Rate (lb ai/A)	PHI (days)	Use Directions and Limitations [2,3]
Barley						
Preplant or preemergence burndown application, Ground or air equipment	50% SG [352-632]	0.0016	1	0.016	NA	Apply in a minimum of 5 gal/A by ground and 2-5 gal/A by air.
	16.7% SG [352-714]	0.005-0.009	2	0.016	NA	Apply in a minimum of 5 gal/A by ground and 2-5 gal/A by air.
Broadcast foliar application(s) after 2- leaf stage but prior to flag leaf emergence. Ground or air equipment	50% SG [352-632]	0.008-0.0016	2	0.016	7 (forage) 30 (hay) 45 (harvest) [4]	Apply in a minimum of 5 gal/A by ground and 2-5 gal/A by air.
	16.7% SG [352-714]	0.005-0.009	2	0.016	7 (forage) 30 (hay)	Apply in a minimum of 5 gal/A by ground and 2-5 gal/A by air.
	18.75%DF [352-610]	0.002-0.005	NS	NS	7 (forage) 30 (hay) 45 (harvest) [4]	Apply in a minimum of 5 gal/A by ground and 1-5 gal/A by air.
Oats						
Preplant burndown application Ground or Air equipment	50% SG [352-632]	0.016	1	0.016	NA	Apply in a minimum of 5 gal/A by ground and 2-5 gal/A by air.
	16.7% SG [352-714]	0.005-0.009	2	0.016	NA	Apply in a minimum of 5 gal/A by ground and 2-5 gal/A by air.
Broadcast foliar application after 2-leaf stage but before flag leaf emergence for winter oats, and after 3-leaf stage but prior to jointing for spring oats. Ground or air equipment	16.7% SG [352-714]	0.005-0.006	1	0.006	7 (forage) 30 (hay)	Apply in a minimum of 5 gal/A by ground and 2-5 gal/A by air.

Table 4. Summary of Directions for Use of Tribenuron Methyl.

Applic. Timing, Type, and Equip. [1]	Formulation [EPA Reg. No.]	Applic. Rate (lb ai/A)	Max. No. Applic. per Season	Max. Seasonal Applic. Rate (lb ai/A)	PHI (days)	Use Directions and Limitations [2,3]
Wheat (including Durum and Triticale)						
Preplant or preemergence burndown application, Ground or air equipment	50% SG [352-632]	0.0016	1	0.016	NA	Apply in a minimum of 5 gal/A by ground and 2-5 gal/A by air.
	16.7% SG [352-714]	0.005-0.009	2	0.016	NA	Apply in a minimum of 5 gal/A by ground and 2-5 gal/A by air.
Broadcast foliar application(s) after 2- leaf stage but prior to flag leaf emergence. Ground or air equipment	50% SG [352-632]	0.008-0.0016	2	0.016	7 (forage) 30 (hay) 45 (harvest) [4]	Apply in a minimum of 5 gal/A by ground and 2-5 gal/A by air.
	16.7% SG [352-714]	0.005-0.009	2	0.016	7 (forage) 30 (hay)	Apply in a minimum of 5 gal/A by ground and 2-5 gal/A by air.
	18.75%DF [352-610]	0.002-0.005	NS	NS	7 (forage) 30 (hay) 45 (harvest) [4]	Apply in a minimum of 5 gal/A by ground and 1-5 gal/A by air.

[1] The Label for the 18.75% DF prohibits application through any type of irrigation equipment, but the labels for the 50% SG and 16.7% SG allow for application through center pivot, lateral move, side (wheel) roll, solid set, or hand move irrigation systems.

[2] Applications may include a non-ionic surfactant (NIS) at 0.06-0.5% v/v or a crop oil concentrate (COC) or modified seed oil (MSO) at 0.5-2% v/v.

[3] Rotational crop restrictions varied between the three labels. The label for the 50% SG (which contains only tribenuron methyl) listed the following restrictions: wheat, barley, and triticale may be planted at anytime after application; canola, rape, and sugar beets may be planted at 60 days after application; and all other crops may be planted 45 days after application. Plant back intervals on the labels for the two MAI EPs were more restrictive than for the EP containing only tribenuron methyl.

[4] The labels for the 50% SG and 18.75% DF state "Do not harvest sooner than 45 days after the last application," however, these labels also include a 7-day PHI for forage and a 30-day PHI for hay.

Conclusions. The label directions are adequate to allow evaluation of the residue data relative to the labeled uses. The available wheat (forage and hay), barley (hay), and oat (forage and hay) data, support the proposed 7-day PHI for forage and 30-day PHI for hay. However, the following label amendments are required.

- Labels of all EPs containing uses of tribenuron methyl on wheat, barley, or oats should be amended to specify minimum PHIs of 7 days for forage and 30 days for hay.

- The labels for the 50% SG (EPA Reg. No. 352-632) and the 18.75% DF (EPA Reg. No. 352-610) should be clarified to indicate minimum PHIs of 7 days for forage, 30 days for hay, and 45 days for the mature crop.

860.1300 Nature of the Residue - Plants

DP# D304059, R. Griffin, 6/24/04
PP#0F6135; DP# D266130, C. Swartz, 7/25/06

The nature of the residue in plants is adequately understood based on metabolism studies with wheat, canola, and cotton. The residue of concern in plant commodities is tribenuron methyl *per se*.

In the wheat metabolism study, [phenyl-U-^{14}C]tribenuron methyl or [triazine-2-^{14}C]tribenuron methyl was applied foliarly to wheat plants at 0.063 lb ai/A (4x the maximum seasonal rate) when wheat plants were at the tiller stage. Samples were collected 0, 4, 8, 14, 21, and 28 DAT, and at maturity (63 DAT). Total radioactive residues (TRR) were highest in wheat forage collected on the day of treatment (4.23 and 5.49 ppm) and decreased with increasing sampling intervals to 0.51 and 0.75 ppm at 28 DAT. TRR were 0.37 and 0.55 ppm in straw and 0.01 and 0.05 ppm in grain harvested at maturity. In wheat forage and straw, the major residues were glucose-conjugated saccharin, glucose-conjugated metsulfuron methyl, hydroxylated triazine amine, O-demethyl triazine amine, and hydroxylated saccharin. In wheat grain, sulfonamide urea was the major residue (45% TRR; only phenyl-label samples were analyzed); saccharin, hydroxylated saccharin, sulfonamide, and glucose-conjugated saccharin were also identified.

In a canola metabolism study, [phenyl-U-^{14}C]tribenuron methyl or [triazine-2-^{14}C]tribenuron methyl was foliarly applied to ALS-tolerant canola plants at ~0.35 oz ai/A (5.5x the maximum seasonal rate) when canola plants were at the 2 true leaf or bolting stage. Samples were collected 0, 2, and 35 DAT, and at maturity (78 DAT). TRR declined in canola foliage from 0.24-0.31 ppm at 0 DAT to 0.04 ppm at 35 DAT. TRR in immature canola seed pods were 0.04 ppm at 35 DAT. TRR in mature canola seed samples, the raw agricultural commodity of interest, were 0.02 ppm at 78 DAT. In immature canola forage collected 0 and 2 DAT, tribenuron methyl was the major residue, at 88-99% TRR. Tribenuron methyl declined to 11-25% TRR in 35-DAT immature foliage. All other residues identified in canola seed and foliage were found to be present at $\leq$0.02 ppm. The major metabolic processes in canola include hydrolytic cleavage and N-demethylation of tribenuron methyl with subsequent hydroxylation. Formation of the conjugates of the hydroxylated metabolites may also occur.

In a cotton metabolism study, [phenyl-U-^{14}C]tribenuron methyl or [triazine-2-^{14}C]tribenuron methyl was applied to cotton plants as a soil treatment at ~0.3 oz ai/A (2x the maximum seasonal rate) or 1.5 oz ai/A (9x). Application was made immediately after planting. Immature samples were collected 89 DAT (minimal boll formation) and 119 DAT (appearance of mature boll), and mature samples were collected 174 DAT. TRR were <0.01 ppm in samples of mature undelinted cotton seed (174 DAT) treated with either label at ~0.3 oz ai/A. TRR in cotton seed treated at the 1.5 oz ai/A rate were not reported. TRR in mature cotton gin byproducts (174 DAT) treated with [phenyl-U-^{14}C] tribenuron methyl were 0.028 ppm from the 0.3 oz ai/A rate and were 0.047 ppm from the 1.5 oz ai/A rate. TRR in mature cotton gin byproducts treated with [triazine-2-^{14}C]tribenuron methyl were <0.01 ppm from the lower rate. TRR in samples from the higher rate were not reported.

Chromatographic analysis of the aqueous-soluble residues in gin byproducts (~53% TRR, 0.0149 ppm) did not detect tribenuron methyl or its known metabolites. The major polar peak in the aqueous extract was characterized as a glucose conjugate based on enzyme hydrolysis. Enzyme hydrolysis of the aqueous extract of mature phenyl-labeled gin trash from the exaggerated rate study indicated that the polar peak was readily converted to a less polar unknown metabolite following treatment with β-glucosidase. The results of the cotton metabolism study suggest that tribenuron methyl is not readily translocated into the cotton plant when applied as a soil treatment immediately after planting. The metabolic pathway of tribenuron methyl in soil involves cleavage of the sulfonylurea bridge of the parent compound, forming metabolites containing either the phenyl or triazine ring. In cotton, the 2-(aminosulfonyl)benzoate and saccharin soil residues from the phenyl ring degradation yielded a phenyl ring polar glucose conjugate.

860.1300 Nature of the Residue - Livestock

DP# D304059, R. Griffin, 6/24/04

The nature of the residue in livestock is adequately understood based on an acceptable goat metabolism study. HED has previously determined that a poultry metabolism study is not required to support the existing uses on wheat and barley, as residues in/on wheat and barley grain were nondetectable, even at exaggerated application rates. Tolerances for animal commodities are not needed as a result of the proposed and established tolerances for animal feed items. As a result, the residue of concern in animal commodities is not an issue for this tolerance petition.

In the available goat metabolism studies, lactating goats were dosed orally for 5 days with either [triazine-2-^{14}C]- or [phenyl-U-^{14}C]-tribenuron methyl at levels equivalent to ~6.7 ppm in the diet (12x the TDB for dairy cattle and 56x the MDB for beef cattle). For the goat dosed with [triazine-2-^{14}C]tribenuron methyl, TRRs were 0.03-0.09 ppm in milk, 0.029 ppm in liver, 0.023 ppm in kidney, 0.013 ppm in muscle, and <LOD in fat. For the goat dosed with [phenyl-U-^{14}C]tribenuron methyl, TRRs were 0.002-0.006 ppm in milk, 0.084 ppm in liver, 0.023 ppm in kidney, and <LOD in muscle and fat. The majority of the dosed radioactivity (81-86% administered dose) was excreted.

Milk samples were separated into skim milk and cream, with 85% of the radioactivity partitioning into the skim milk. Skim milk from the goat dosed with [triazine-2-^{14}C]tribenuron methyl was subjected to characterization/identification procedures. The major residue found was N-demethyltriazine amine, at 0.01-0.02 ppm (14-29% TRR). Other identified metabolites were found at <0.01 ppm. Tribenuron methyl, metsulfuron methyl, and tribenuron acid were not detected. Liver and kidney tissues were subjected to extraction procedures, with up to 35% TRR found to be nonextractable via solvents, enzyme incubation, or acid hydrolysis. Extracts were not analyzed.

860.1340 Residue Analytical Methods

DP# D311607, S. Ary, 1/04/05
DP# D304059, R. Griffin, 6/24/04
DP# D266130, C. Swartz, 7/25/06
DP# D330633, S. Hummel, 8/8/06

Enforcement methods: An HPLC/photo-conductivity method (Method AMR 337-85, Revision A) is available for enforcement of tolerances for residues of tribenuron methyl *per se* in grain, forage, and straw commodities. For this method, residues are extracted with acetonitrile, cleaned up using silica gel chromatography, and analyzed by HPLC with a photo-conductivity detector. The method was validated by ACL in wheat grain at 0.057 and 0.113 ppm and in wheat straw at 0.113 and 0.226 ppm. The petitioner validated the method at 0.01-0.02 ppm in wheat and barley grain, at 0.02-0.04 ppm in wheat and barley straw, and at 0.01-0.1 ppm in wheat forage.

An LC/MS method (DuPont Method 1381) is also available for enforcement of tolerances for residues of tribenuron methyl in/on canola, cotton, and flax commodities. For this method, samples are extracted with an acetonitrile/ammonium carbonate buffer solution, and the concentrated residues are reconstituted in methanol for analysis. If required, a hexane wash step or cleanup step using a strong anion exchange SPE column may be incorporated. Analysis is performed by column-switching liquid chromatography. Extracts in methanol are applied to a size exclusion chromatography column, and the eluate is diverted onto a reversed phase C_8 column for MS analysis. The validated LOQ was 0.020 ppm. This method may also be used for enforcing tolerances in/on corn grain, sorghum grain, and soybean seed.

An enforcement method for animal commodities is not currently required as there are no tolerances for tribenuron methyl residues in livestock commodities.

Data collection method: Forage and hay samples from the barley, oat, and wheat field trials were analyzed for residues of tribenuron methyl using an LC/MS/MS method (DuPont Method 13412, Revision No. 1). DuPont Method 13412 was previously validated for the analysis of tribenuron methyl in oily crop matrices (46352003.de2, S. Hummel, 8/08/06). The method has undergone a successful independent laboratory trial, and is also capable of determining other sulfonylurea herbicides (ethametsulfuron methyl, rimsulfuron, trifensulfuron methyl, and chlorimuron ethyl). The modifications made to the method for the analysis of residues in forage and hay included: (1) addition of a hydration step for dry matrices; (2) replacing the second centrifugation step with filtration; and (3) modification of the hexane partition step for hay to include extra cleanup with BONDESIL-SAX.

Following hydration of hay samples, residues are extracted from forage and hay with acetonitrile:K_2HPO_4 buffer at pH 7 (75:25, v:v). For forage samples, the aqueous extract is partitioned directly with hexane, the hexane phase is discarded. For hay samples, the aqueous extract is acidified, mixed with BONDESIL-SAX, and then partitioned with hexane. The hexane fraction is discarded and the remaining aqueous fraction is neutralized. Residues from both forage and hay samples are then cleaned up using an ENV SPE cartridge eluted with 25 mM NH_4OH:methanol (1:99, v:v). Residues are concentrated, redissolved in acetonitrile:50 mM

ammonium acetate (1:9, v:v) and analyzed by LC/MS/MS, using external standards. Residues of tribenuron methyl are quantified using the m/z 396→155 transition and confirmed using the m/z 396→181 transition. The method has a validated LOQ of 0.01 ppm and a reported LOD of 0.003 ppm.

The above LC/MS/MS method was adequately validated in conjunction with the analysis of field trial samples, using control samples of forage and hay fortified with tribenuron methyl at 0.01-10.0 ppm.

Conclusions. The available residue analytical method data are adequate to satisfy data requirements. The existing tolerance enforcement methods, AMR 337-85 and DuPont Method 1381, are adequate to enforce the proposed tolerances for forage and hay, and DuPont Method 13412 (Revision 1) is adequate for data collection purposes.

860.1360 Multiresidue Methods

The FDA PESTDATA database dated 06/05 (PAM Volume I, Appendix I) does not contain any information regarding the recovery of tribenuron methyl using multiresidue methods. Data investigating the behavior of tribenuron methyl using the FDA Multiresidue Methods have been submitted by the registrant (MRID 40927202). The data were apparently not received by FDA for evaluation. The available data indicate that residues of tribenuron methyl are not recovered by the FDA multiresidue methods.

860.1380 Storage Stability

Adequate data are available indicating that tribenuron methyl is stable under frozen storage conditions for at least 4 months in corn stover and soybean seeds, 6 months in corn forage, 8.6 months in sunflower seeds, 14 months in cottonseeds and cotton gin byproducts, and 21 months in wheat grain and straw grain and forage and wheat straw, and 36 months in wheat grain.

The storage durations and conditions of samples from the barley, oat, and wheat field trials submitted to support this petition are presented in Table 5.

Table 5. Summary of Storage Conditions and Durations of Samples from Crop Field Trials.

Matrix	Storage Temperature (°C)	Actual Storage Duration (months)	Interval of Demonstrated Storage Stability (months)
Barley hay	-20 ± 5	4.0	6 (corn forage) 21 (wheat straw)
Oat forage and hay	-20 ± 5	4.8	
Wheat forage and hay	-20 ± 5	5.8	

Conclusion. The available storage stability data adequately support the sample storage durations used in the barley, oat, and wheat field trials.

860.1400 Water, Fish, and Irrigated Crops

There are no proposed or registered uses that are relevant to this guideline topic.

860.1460 Food Handling

There are no proposed or registered uses that are relevant to this guideline topic.

860.1480 Meat, Milk, Poultry, and Eggs

No livestock feeding studies were submitted as part of the subject tolerance petition. HED previously concluded that tribenuron methyl residues in livestock commodities may be classified under 40 CFR §180.6(a)(3), as there is no expectation of finite residues of tribenuron methyl in milk, eggs, meat, and poultry. This decision was based on potential dietary exposures of 0.26 ppm for beef and dairy cattle and 0.04 ppm for poultry and swine.

Because the current petition contains no new poultry or swine feedstuffs, there is no change in HED's previous conclusions regarding the need for a poultry feeding study. However, the addition of tolerances for forage and hay commodities changes the dietary burden of ruminants for tribenuron methyl residues. Based on the recommended tolerances for barley, oat, and wheat commodities and the established tolerances on other cattle feedstuffs, the MDBs for livestock were calculated using the most recent guidance from HED (October 2006) concerning revisions of feedstuffs in Table 1 (OPPTS 860.1000) and constructing reasonably balanced diets for livestock. The calculated MDBs are 0.12 ppm for beef cattle, 0.54 ppm for dairy cattle, and 0.05 ppm for poultry and swine (Table 6).

In the available goat metabolism study, residues of tribenuron methyl were not detected (<0.01 ppm) in milk or tissues following 5 days of dosing with [^{14}C-triazine] or [^{14}C-phenyl]-label tribenuron methyl at levels equivalent to 6.7 ppm in the diet, which is 12.4x the calculated TDB for dairy cattle and 57x the MDB for beef cattle. Based on the data from the goat metabolism study, HED concludes that a cattle feeding study is not required for this petition.

Table 6. Calculation of Dietary Burdens of Tribenuron-Methyl Residues to Livestock.

Feedstuff	Type[1]	% Dry Matter[2]	% Diet[2]	Recommended Tolerance (ppm)	Dietary Contribution (ppm)[3]
Beef Cattle					
Barley, hay	R	88	15	0.4	0.068
Sorghum, grain	CC	86	80	0.05	0.0465
Soybean, seed	PC	89	5	0.05	0.0028
TOTAL BURDEN			100		**0.117**
Dairy Cattle					
Wheat, forage	R	25	40	0.3	0.480
Wheat, hay	R	88	5	0.5	0.028
Soybean, seed	PC	89	15	0.05	0.008
Sorghum, grain	CC	86	40	0.05	0.023
TOTAL BURDEN			100		**0.540**
Poultry					
Soybean, seed	NA	--	20	0.050	0.010

Barley, grain	NA	--	70	0.050	0.035
Field corn grain	NA	--	10	0.050	0.005
TOTAL BURDEN			100		**0.050**
Swine					
Soybean seed	NA	--	20	0.050	0.010
Field corn grain	NA	--	60	0.050	0.030
Barley grain	NA	--	20	0.050	0.010
TOTAL BURDEN			100		**0.050**

[1] R - Roughage; CC - Carbohydrate concentrate; PC - Protein concentrate.
[2] OPPTS 860.1000 Table 1 Feedstuffs (October 2006).
[3] Contribution = ([tolerance /% DM] X % diet) for beef and dairy cattle; contribution = ([tolerance] X % diet) for poultry and swine.

Conclusions. Cattle and poultry feeding studies are not required as there is no reasonable expectation of finite residues occurring in milk, meat, eggs, or poultry [40 CFR 180.6(a)(3)].

860.1500 Crop Field Trials

47138301.der (Barley)
47138302.der (Oats)
47138303.der (Wheat)

DuPont submitted field trial data reflecting the use of tribenuron methyl (50% SG) on barley, oats, and wheat as a single early season postemergence application totaling 0.015-0.016 lb ai/A. These data were submitted to support the proposed tolerances on barley hay, oat forage and hay, and wheat forage and hay. The proposed PHIs for forage and hay are 7 and 30 days, respectively. The results from these field trials are discussed below, and the residue data are summarized in Table 7.

Table 7. Summary of Residue Data from Crop Field Trials with Tribenuron Methyl (SG).

Crop matrix	Total Applic. Rate (lb ai/A)	PHI (days)	Residue Levels (ppm) [1]						
			n	Min.	Max.	HAFT [2]	Median	Mean	Std. Dev.
Barley (proposed use = 0.016 lb ai/A total application rate, 30-day PHI for hay)									
Barley Hay	0.015-0.016	5-14	8	<0.01	0.056	0.041	0.01	0.018	0.016
		22-31	20	<0.01	0.360	0.265	0.01	0.036	0.084
Oats [3] (proposed use = 0.006 lb ai/A total application rate, 7-day and 30-day PHI for forage and hay)									
Oat Forage	0.015-0.016	0	34 [4]	0.22	1.50	1.400	0.780	0.739	0.355
		5-8	32	<0.01	0.043	0.041	0.010	0.014	0.009
Oat Hay	0.015-0.016	5-12	10	<0.01	0.028	0.021	0.010	0.012	0.006
		25-33	24 [4]	<0.01	<0.01	<0.01	0.010	0.010	NA

Table 7. Summary of Residue Data from Crop Field Trials with Tribenuron Methyl (SG).									
Crop matrix	Total Applic. Rate (lb ai/A)	PHI (days)	Residue Levels (ppm) [1]						
			n	Min.	Max.	HAFT [2]	Median	Mean	Std. Dev.
Wheat (Proposed use = 0.016 lb ai/A total application rate, 7-day and 30-day PHI for forage and hay)									
Wheat Forage	0.015-0.016	0	44	<0.01	2.80	2.60	0.71	0.855	0.661
		6-8	42	<0.01	0.25	0.24	0.01	0.049	0.071
Wheat Hay	0.015-0.016	5	6	<0.01	<0.01	<0.01	0.01	0.01	NA
		11-16	8	<0.01	<0.01	<0.01	0.01	0.01	NA
		25-35	30	<0.01	0.42	0.29	0.01	0.029	0.079

[1] The validated method LOQ is 0.01 ppm for tribenuron methyl in each commodity. For calculating the median, mean and standard deviation, the method LOQ was used for values <LOQ.

[2] HAFT = Highest Average Field Trial.

[3] The maximum use rates on oats are 0.016 lb ai/A for a preplant use, but only 0.006 lb ai/A for a postemergence use.

[4] Residue data for oat forage and hay samples from the Paynesville, MN field trial were not included as these samples were apparently mislabeled or switched.

Forage, Fodder, and Straw of Cereal Grains

Barley. In 14 barley field trials conducted during 2005-2006 in EPA Growing Zones 2, 5, 10, and 11, tribenuron methyl (50% SG) was applied to winter barley (3 tests) and spring barley (11 tests) as a single broadcast foliar application during vegetative development (Zadoks Growth Stages 36 through 50) at 0.015-0.016 lb ai/A. Applications were made using ground equipment at rates of 13-25 gal/A, and included the use of an NIS adjuvant. Single control and duplicate treated samples of hay were cut at 22-31 DAT at 10 sites, but were cut at 5-14 DAT at the remaining 4 sites. The target PHI was 30 days. To assess residue decline, hay samples were collected at 0, 3, 7, 14, 28, and 45 DAT at one test site. After harvest, hay samples were field-dried for 0-23 days before collection. Hay samples were stored at -20°C for up to 4 months prior to analysis. This time interval is supported by the available storage stability data.

The LC/MS/MS method (DuPont Method 13412, Revision No. 1) used for determining residues of tribenuron methyl in/on barley hay was adequately validated in conjunction with the analysis of field trial samples. The validated LOQ is 0.01 ppm, and the LOD is 0.003 ppm.

Following a single broadcast foliar application of tribenuron methyl (SG) to barley around flag leaf emergence at 0.015-0.016 lb ai/A, residues in/on hay were <0.01-0.056 ppm for the 8 samples harvested at 5-14 DAT and <0.01-0.36 ppm for the 20 samples harvested at 22-31 DAT. Residues were <LOQ (<0.01 ppm) in 18 out of 20 samples from the 22-31 DAT interval. Average residues in/on hay were 0.018 ppm at 5-14 DAT and 0.036 ppm at 22-31 DAT. In the residue decline test, residue levels in/on hay declined rapidly from 0 to 7 DAT, and were <0.01 ppm from 14 to 45 DAT.

Oats. In 18 oat field trials conducted during 2005-2006 in EPA Growing Zones 2, 5, 6, 7, and 8, tribenuron methyl (50% SG) was applied to winter oats (3 tests) and spring oats (15 tests) as a single broadcast foliar application during vegetative development at 0.015-0.016 lb ai/A. Each field trial included separate treated plots for the harvest of forage and hay. The application was made to the forage plots between Zadoks Growth Stages 23 and 39 and to the hay plots between

Zadoks Growth Stages 37 and 45. All applications were made using ground equipment at rates of 12-26 gal/A, and included the use of an NIS adjuvant.

Forage samples were collected from each site at 0 and 5-8 DAT. Hay samples were collected at 5-12 DAT from 5 sites and at 25-33 DAT from 13 sites (the target PHI for hay was 30 days). Single control and duplicate treated samples were collected for both matrices. To assess residue decline, forage and hay samples were collected at approximately 0, 3, 7, 14, 28, and 45 DAT at two test sites. After harvest, hay samples were field-dried for 1-13 days before collection. Forage and hay samples were stored at -20°C for up to 4.8 months prior to analysis. This interval is supported by the available storage stability data.

The LC/MS/MS method (DuPont Method 13412, Revision No. 1) used to determine residues of tribenuron methyl in/on oat forage and hay was adequately validated in conjunction with the analysis of field trial samples. The validated LOQ is 0.01 ppm, and the LOD is 0.003 ppm.

The residue data from the Paynesville, MN test site were not included when calculating overall residues because the forage (0-day) and hay samples from this site were apparently either switched or mislabeled. The 0-day forage samples from this site had residues (<0.003 ppm) well below the residues in all other 0-day forage samples (0.22-1.50 ppm), but similar to the residues in the 30-day hay samples from the other sites. Concomitantly, the 30-day hay samples from this site had residues (0.57, 0.88 ppm) well above the residues in all other 30-day hay samples (<0.003 ppm), but similar to the residues in the 0-day forage samples from the other sites.

Following a single broadcast foliar application of tribenuron methyl (SG) at 0.015-0.016 lb ai/A, residues in/on oat forage were 0.22-1.50 ppm for the 34 samples harvested at 0 DAT and <0.01-0.043 ppm for 32 samples harvested at 5-8 DAT. Residues in/on hay were <0.01-0.028 ppm for the 10 samples harvested at 5-12 DAT and ND (<0.003 ppm) for the 24 samples harvested at 25-33 DAT. Average residues in/on forage were 0.739 ppm at 0 DAT and 0.014 ppm at 5-12 DAT. Average residues in/on hay were 0.012 ppm at 5-12 DAT and <0.01 ppm at 25-33 DAT. In the two residue decline tests, residue levels in/on both forage and hay declined rapidly from 0 to 7 DAT, and were <0.01 ppm from 7 to 45 DAT in both forage and hay.

Wheat. In 22 wheat field trials conducted during 2005-2006 in EPA Growing Zones 2, 4, 5, 6, 7, 8, and 11, tribenuron methyl (50% SG) was applied to winter wheat (14 tests) and spring wheat (8 tests) as a single broadcast foliar application during vegetative development at 0.015-0.016 lb ai/A. Each field trial included separate treated plots for the harvest of forage and hay. The application was made to the forage plots between Zadoks Growth Stages 21 and 39 and to the hay plots between Zadoks Growth Stages 23 and 49. All applications were made using ground equipment at rates of 12-25 gal/A, and included the use of an NIS adjuvant.

Forage samples were collected from each site at 0 and 6-8 DAT. Hay samples were collected at 5 DAT from 3 sites, at 11-16 DAT from 4 sites, and at 25-35 DAT from 15 sites (the target PHI for hay was 30 days). Single control and duplicate treated samples were collected for both matrices. To assess residue decline, forage and hay samples were collected at approximately 0, 3, 7, 14, 28, and 45 DAT at three test sites. After harvest, hay samples were field-dried for 0-8

days before collection. Forage and hay samples were stored at -20°C for up to 5.8 months prior to analysis. This interval is supported by the available storage stability data.

The LC/MS/MS method (DuPont Method 13412, Revision No. 1) used to determine residues of tribenuron methyl in/on wheat forage and hay was adequately validated in conjunction with the analysis of field trial samples. The validated LOQ is 0.01 ppm, and the LOD is 0.003 ppm.

Following a single broadcast foliar application of tribenuron methyl (SG) to wheat during vegetative development at 0.015-0.016 lb ai/A, residues in/on forage were <0.01-2.8 ppm in/on 44 samples harvested at 0 DAT and <0.01-0.25 ppm in/on 42 samples harvested at 6-8 DAT. Average residues in/on forage were 0.86 ppm at 0 DAT and 0.049 ppm by ~7 DAT. For hay, residues were <LOQ (<0.01 ppm) in/on all 14 samples collected at either 5 or 11-16 DAT, and residues were <0.01-0.42 ppm in/on the 30 samples harvested at 25-35 DAT, with 26 of these samples having residues <LOQ. Average residues in/on hay were <0.01 ppm at 5 and 11-16 DAT, and 0.029 ppm at 25-35 DAT. Data from the three residue decline tests indicate that residue levels in/on both forage and hay declined rapidly from 0 to 7 DAT, and then remained relatively steady from 14 to 45 DAT.

Conclusions. Although several deficiencies were noted in the submitted field trials, the available barley, oat and wheat field trial data are acceptable and support the existing use patterns for tribenuron methyl on barley, oats, and wheat. Adequate numbers of tests were conducted on each crop in the appropriate geographical regions using a water soluble granule formulation. Adequate numbers of oat and wheat forage samples were collected around the proposed 7-day PHI. Although several hay samples were not collected at the appropriate PHI, sufficient numbers of hay samples are available for barley (20 samples), oats (24 samples), and wheat (30 samples) harvested around the proposed 30-day PHI (22-35 DAT). All samples were analyzed for the residue of concern using an adequate method, and sample storage conditions and intervals were supported by the available storage stability data.

The oat field trial data will be used to assess tolerances on oat forage and hay; however, the oat field trials were conducted at 2.6x the maximum postemergence rate for oats. Although the maximum total preplant rate for oats is ~0.016 lb ai/A, the available labels allow for only one postemergence application to oats at up to 0.006 lb ai/A.

The available field trial data support PHIs of 7 and 30 days for forage and hay, respectively, and tolerances for residues of tribenuron methyl at 0.4 ppm on barley hay, 0.05 ppm on oat forage and hay, 0.3 ppm on wheat forage, and 0.5 ppm on wheat hay.

860.1520 Processed Food and Feed

Requirements for residue data on processed commodities of barley, oats, and wheat have been previously satisfied. Because no residues of tribenuron methyl were detected in/on wheat grain samples treated at up to 4x the maximum seasonal rate, and in consideration of the registrant's claim that phytotoxic effects are observed at application rates greater than 4x, HED concluded

that wheat processing data would not be required. By translation, processing data for barley and oats are not required.

860.1650 Submittal of Analytical Reference Standards

An analytical standard for tribenuron methyl is currently available in the National Pesticide Standards Repository. It has an expiration date of 8/31/2008 (electronic communication, D. Wright, 3/18/2008).

860.1850 and 860.1900 Confined and Field Accumulation in Rotational Crops

Two confined rotational crop studies are available reflecting treatment of sandy loam soils with [^{14}C-triazine]- and [^{14}C-phenyl]-tribenuron methyl. These studies were originally reviewed and deemed acceptable by EFED. In these tests, tribenuron methyl was not detected in any commodities from rotational crops planted 30 days after a soil application of [^{14}C]tribenuron methyl at 0.028-0.031 lb ai/A (1-1.8x the maximum seasonal rates).

In the phenyl-label study, TRR accumulated at levels greater than 0.01 ppm in/on wheat forage (0.010-0.020 ppm), wheat straw (0.054 ppm), soybean forage (0.008-0.017 ppm), soybean hay (0.052 ppm), sorghum forage (0.007-0.016 ppm), and sorghum stover (0.082 ppm) planted 30 days post-treatment. TRR were <0.01 ppm in/on cabbage heads and foliage, beet roots and tops, wheat grain, soybean seed, and sorghum grain from the same plantback interval. At the 120-day plantback interval, TRR were 0.010 ppm in soybean forage and 0.019 ppm in sorghum stover. In addition, TRR were <0.01 ppm in/on cabbage heads and foliage; beet roots and tops; wheat forage, straw, and grain; soybean hay and seed; and sorghum grain. Samples of 30-day PBI wheat straw, soybean hay, and sorghum stover, as well as 120-day PBI sorghum stover were subjected to extraction/characterization of residues, which identified saccharin as the major degradate (0.007-0.021 ppm). Tribenuron methyl was not detected in any sample.

In the triazine-labeled study, TRR accumulated at levels greater than 0.01 ppm in/on beet tops (0.075-0.156 ppm), cabbage foliage (0.018-0.135 ppm), sorghum forage (0.031-0.177 ppm), sorghum straw (0.281 ppm), and sorghum grain (0.016 ppm) planted 30 days post-treatment: TRR were <0.01 ppm in/on beet roots and cabbage heads from the same plantback interval. At the 120-day plantback interval, TRR were 0.013-0.031 ppm in beet tops, 0.010-0.047 ppm in cabbage foliage, 0.013-0.086 ppm in sorghum forage, 0.148 ppm in sorghum straw, and 0.010 ppm in sorghum grain. TRR were <0.01 ppm in/on beet roots and cabbage heads from the 120-day plantback interval. Samples of 30-day beet tops, cabbage foliage, and sorghum straw, and 120-day PBI beet tops, cabbage foliage, and sorghum straw and grain were subjected to extraction/characterization of residues. The following metabolites were detected in 30-day PBI samples: triazine amine (beet tops only), N-demethyl triazine amine (beet tops, cabbage foliage, and sorghum straw), O-demethyl triazine amine (beet tops only), and α-hydroxy triazine amine (beet tops only). Tribenuron methyl was not detected in any sample.

Conclusions. The available confined rotational crop data are adequate and support a minimum 30-day plantback interval for all crops without registered uses. As labels for tribenuron methyl currently specify minimum plantback intervals of no less than 60 days for canola, rape, and sugar beets, and 45 days for all other crops not listed on the label, data requirements for rotational crops are fulfilled, and tolerances are not required for rotational crops.

860.1550 Proposed Tolerances

Tolerances for residues of tribenuron methyl are currently expressed in terms of tribenuron methyl *per se.* Permanent tolerances are established for residues of tribenuron methyl in/on barley, canola, field corn, cotton, flax, grass, oat, rice, sorghum, soybean, sunflower and wheat commodities at levels ranging from 0.02 to 0.10 ppm [40 CFR§ 180.451 (a) and (c)]. No tolerances are established for residues in either animal commodities or rotational crops.

As the majority (55-100%) of residue values were <LOQ in/on samples of barley hay, oat forage and hay, and wheat forage and hay collected at the appropriate PHIs, tolerances for these commodities could not be calculated using HED's spreadsheet for NAFTA-harmonized tolerances (Appendix I). Therefore, the recommended tolerances for these commodities were determined based on the maximum residue values. In addition, the Agency notes that the highest residues reported for oat hay (0.57 and 0.88 ppm) are likely the result of switched or mislabeled samples from one field trial; therefore, these values were not considered in assessing the tolerance for oat hay. The recommended tolerances are presented in Table 8.

There are no established or proposed Codex Maximum residue limits (MRLs) for residues of tribenuron methyl (Appendix II). Canada and Mexico have established MRLs for tribenuron methyl on several plant commodities. However, no Canadian or Mexican MRLs for tribenuron methyl have been proposed or established for the commodities being considered under this petition. Therefore, there are no questions about compatibility of the proposed tolerances.

Table 8. Tolerance Summary for Tribenuron Methyl.

Commodity	Proposed Tolerance (ppm)	Recommended Tolerance (ppm)	Comments; *Correct Commodity Definition*
Barley, hay	0.3	0.4	Adequate residue data are available. Maximum residues in/on barley hay were 0.36 ppm
Oat, forage	0.3	0.05	Adequate residue data are available. Maximum residues in/on oat forage at ~7 DAT were 0.04 ppm.
Oat, hay	0.8	0.05	Adequate residue data are available. Maximum residues in/on oat hay were <0.01 ppm.
Wheat, forage	0.3	0.3	Adequate residue data are available. Maximum residues in/on wheat forage at ~7 DAT were 0.25 ppm.
Wheat, hay	0.3	0.5	Adequate residue data are available. Maximum residues in/on wheat hay were 0.42 ppm.

References

D304059, Tribenuron methyl Residue Chemistry Considerations, R. Griffin, 6/24/04.

D311607, Thifensulfuron Methyl. HED's Response to E.I. du Pont de Nemours and Company's Comments to the Summary of Residue Chemistry Data Evaluation Records for the Establishment of Tolerances for New Uses of Thifensulfuron Methyl on Canola, Flax, and Cotton, S. Ary, 1/4/05.

D266130, Tribenuron Methyl. PP#0F6135. Summary of Residue Chemistry Data Evaluation Records for the Establishment of Permanent Tolerances for New Uses of Tribenuron Methyl on Canola, Cotton, and Flax, C. Swartz, 7/25/06.

330663, 330814, Tribenuron Methyl. Addition of Preplant Burndown Uses on Field Corn, Rice, Sorghum and soybeans (PRIA R19 - 352-611; PP#4F6890) and Postemergence Uses on Sunflower (IR-4 Request; PP# 4E6855). Summary of Analytical Chemistry and Residue Data, S. Hummel, 8/8/2006

Attachments:
Appendix I - Tolerance Assessment Calculations
Appendix II - International Residue Limit Status sheet

Appendix I - Tolerance Assessment Calculations

The Agency's *Guidance for Setting Pesticide Tolerances Based on Field Trial Data* was utilized for determining the appropriate tolerance levels for oat and wheat forage harvested at 0 DAT. However, the tolerance spreadsheet was not used to calculate the tolerance for barley hay, oat forage (~7 DAT), oat hay, and wheat forage (~7 DAT), and wheat hay as the datasets for these commodities contained high percentages of values below the LOQ. For barley, oat and wheat hay harvested at ~30 DAT, quantifiable residues were observed in only 0-13% of the samples. For oat and wheat forage at ~7 DAT, quantifiable residues were observed in only 34-45% of the samples. The datasets for barley, oats and wheat are presented in Tables I-1 through I-3.

The dataset used to assess a possible tolerance for tribenuron methyl residues in/on wheat forage at 0 DAT consists of field trial data representing applications of the appropriate formulation at ~1x the maximum proposed use rates. As specified by the *Guidance for Setting Pesticide Tolerances Based on Field Trial Data* SOP, the field trial application rates were within 25% of the maximum label application rates. However, the dataset used to assess a possible tolerance for tribenuron methyl residues in/on oats at 0 DAT consists of field trial data representing applications at 2.6x the maximum postemergence use rate.

The datasets for tribenuron methyl residues in/on forage from oats and wheat harvested at 0 DAT were entered into the tolerance spreadsheet. Visual inspection of the lognormal probability plots (Figures I-1 and I-3) indicates that the datasets are reasonably lognormal. The results of the approximate Shapiro-Francia test statistic confirmed this assumption for oat forage, but rejected the assumption of lognormality for wheat forage (Figures I-2 and I-4).

The recommended tolerances for oat forage and wheat forage harvested at 0 DAT are 2.5 and 3.0 ppm, respectively. As the petitioner is proposing a 7-day PHI for forage, these calculations are for informational purposes only.

Table I-1. Tribenuron Methyl Residues in Barley Hay.	
Regulator:	**EPA**
Chemical:	**Tribenuron-Methyl**
Crop:	**Barley hay**
PHI:	**22-31 days**
App. Rate:	**0.015-0.016 lb ai/A**
Submitter:	**Du Pont**
MRID Citation:	**MRID 47138301**
	Residues
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.01
	0.17
	0.36
Values <LOQ (0.01 ppm) are listed in **bold.**	

Table I-2. Tribenuron Methyl Residues in/on Oat Forage and Hay.			
Regulator:	**EPA**		
Chemical:	**Tribenuron Methyl**		
Crop:	**Oat Forage**	**Oat Forage**	**Oat Hay**
PHI:	**0 days**	**7-8 days**	**28-35 days**
App. Rate:	**0.015-0.016 lb ai/A**	**0.015-0.016 lb ai/A**	**0.015-0.016 lb ai/A**
Submitter:	**Du Pont**		
MRID Citation:	**MRID 47138302**		
	Residues	**Residues**	**Residues**
	0.22	**0.01**	**0.01**
	0.24	**0.01**	**0.01**
	0.28	**0.01**	**0.01**
	0.31	**0.01**	**0.01**
	0.35	**0.01**	**0.01**
	0.39	**0.01**	**0.01**
	0.41	**0.01**	**0.01**
	0.42	**0.01**	**0.01**
	0.43	**0.01**	**0.01**
	0.44	**0.01**	**0.01**
	0.45	**0.01**	**0.01**
	0.46	**0.01**	**0.01**
	0.46	**0.01**	**0.01**
	0.52	**0.01**	**0.01**
	0.66	**0.01**	**0.01**
	0.75	**0.01**	**0.01**
	0.78	**0.01**	**0.01**
	0.78	**0.01**	**0.01**
	0.82	**0.01**	**0.01**
	0.84	**0.01**	**0.01**
	0.86	**0.01**	**0.01**
	0.86	**0.01**	**0.01**
	0.89	**0.01**	**0.01**
	0.90	0.01	**0.01**
	0.94	0.01	
	0.98	0.01	
	1.00	0.01	
	1.00	0.01	
	1.10	0.01	
	1.10	0.02	
	1.20	0.03	
	1.30	0.03	
	1.50	0.04	
	1.50	0.04	
Values <LOQ (0.01 ppm) are listed in **bold.**			

Figure I-1. Lognormal Probability Plot for Residues of Tribenuron Methyl in Oat Forage Harvested at 0 DAT.

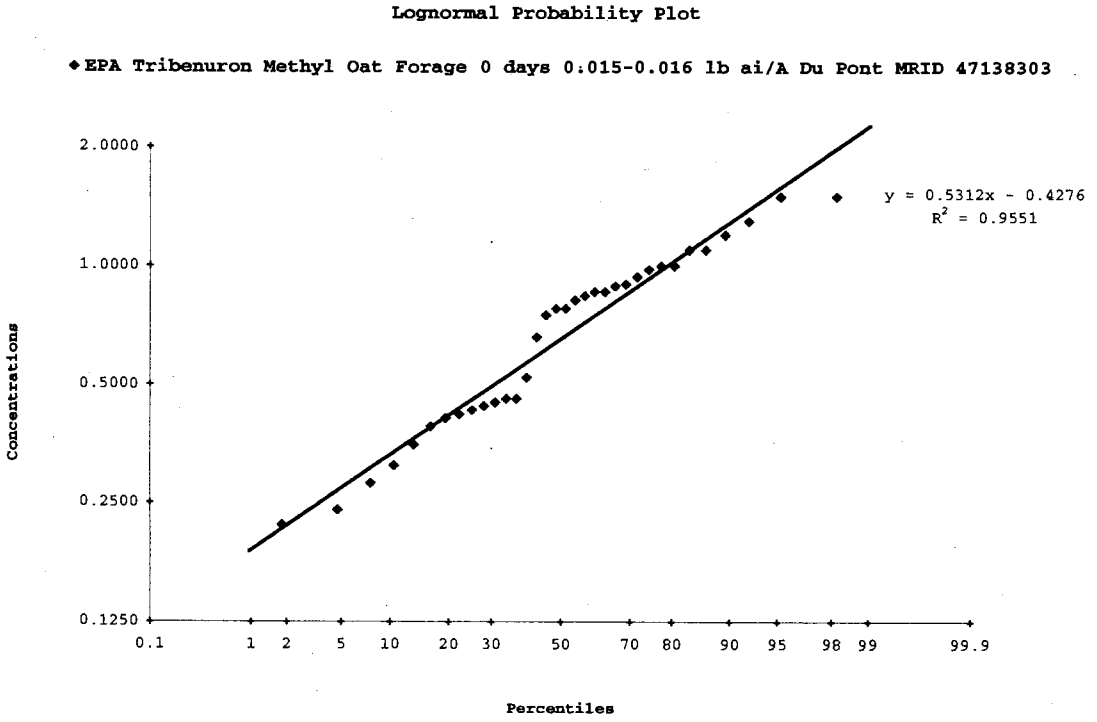


Figure I-2. Data Summary Table for Residues of Tribenuron Methyl in Oat Forage Harvested at 0 DAT.

	Regulator: EPA **Chemical:** Tribenuron Methyl **Crop:** Oat Forage **PHI:** 0 days **App. Rate:** 0.015-0.016 lb ai/A **Submitter:** Du Pont **MRID Citation:** MRID 47138303 **n:** 34 **min:** 0.22 **max:** 1.50 **median;** 0.78 **average:** 0.74		
	95th Percentile	**99th Percentile**	**99.9th Percentile**
EU Method I	1.4	1.6	1.9
Normal	(1.6)	(1.9)	(--)
EU Method I	1.6	**2.5**	3.5
Log Normal	**(2.5)**	(3.5)	(--)
EU Method II Distribution-Free		2.0	
California Method μ + 3σ		**1.9**	
UPLMedian95th		**4.5**	
Approximate Shapiro-Francia Normality Test	0.9551 p-value > 0.05 : Do not reject lognormality assumption		

Table I-3. Tribenuron Methyl Residues in Wheat Forage and Hay.			
Regulator:	**EPA**		
Chemical:	**Tribenuron-Methyl**		
Crop:	**Wheat Forage**	**Wheat Forage**	**Wheat Hay**
PHI:	**0 days**	**6-8 days**	**28-35 days**
App. Rate:	**0.015-0.016 lb ai/A**	**0.015-0.016 lb ai/A**	**0.015-0.016 lb ai/A**
Submitter:	**Du Pont**		
MRID Citation:	**MRID 47138303**		
	Residues	**Residues**	**Residues**
	0.01	**0.01**	**0.01**
	0.01	**0.01**	**0.01**
	0.35	**0.01**	**0.01**
	0.35	**0.01**	**0.01**
	0.36	**0.01**	**0.01**
	0.39	**0.01**	**0.01**
	0.39	**0.01**	**0.01**
	0.43	**0.01**	**0.01**
	0.44	**0.01**	**0.01**
	0.44	**0.01**	**0.01**
	0.46	**0.01**	**0.01**
	0.46	**0.01**	**0.01**
	0.47	**0.01**	**0.01**
	0.48	**0.01**	**0.01**
	0.49	**0.01**	**0.01**
	0.52	**0.01**	**0.01**
	0.54	**0.01**	**0.01**
	0.54	**0.01**	**0.01**
	0.55	**0.01**	**0.01**
	0.60	**0.01**	**0.01**
	0.63	**0.01**	**0.01**
	0.70	**0.01**	**0.01**
	0.72	**0.01**	**0.01**
	0.72	0.013	**0.01**
	0.74	0.013	**0.01**
	0.75	0.014	**0.01**
	0.80	0.017	0.01
	0.81	0.046	0.02
	0.84	0.046	0.16
	0.86	0.051	0.42
	0.87	0.070	
	0.90	0.074	
	0.91	0.074	
	0.96	0.077	
	0.98	0.086	
	1.00	0.110	
	1.20	0.150	
	1.30	0.150	
	1.40	0.220	
	1.70	0.230	
	2.60	0.250	
	2.60	0.250	
	2.60		
	2.80		

Values <LOQ (0.01 ppm) are listed in **bold.**

Figure I-3. Lognormal Probability Plot for Residues of Tribenuron Methyl in Wheat Forage Harvested at 0 DAT.

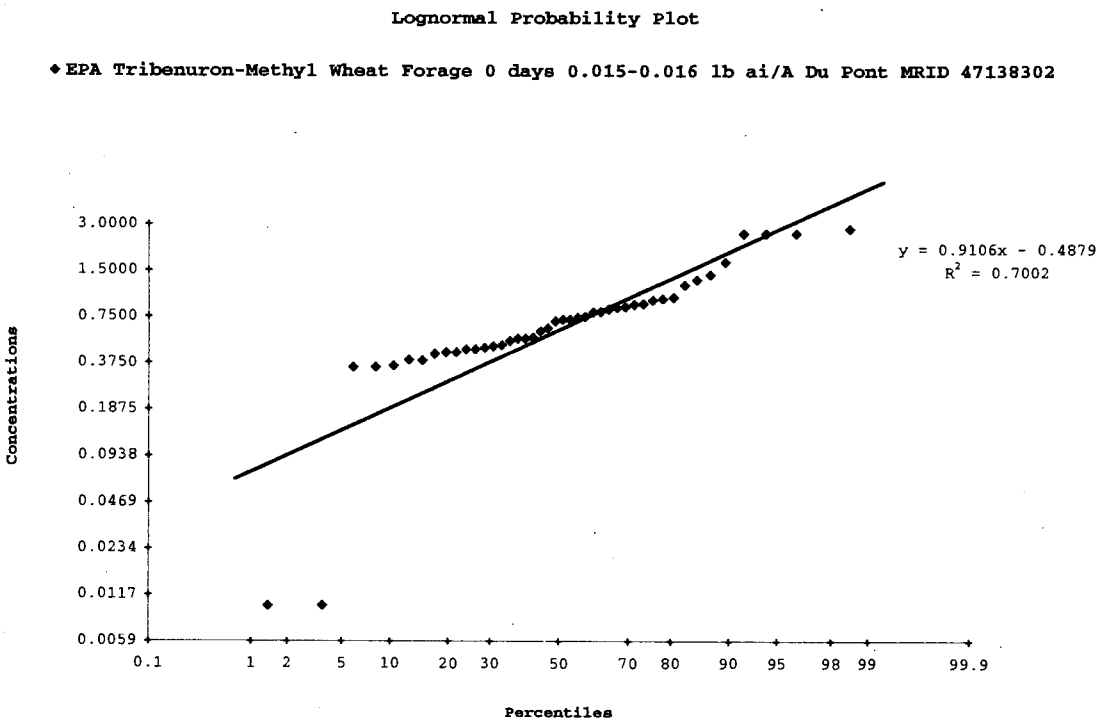


Figure I-4. Data Summary for Residues of Tribenuron Methyl in Wheat Forage Harvested at 0 DAT.

Regulator:	EPA
Chemical:	Tribenuron-Methyl
Crop:	Wheat Forage
PHI:	0 days
App. Rate:	0.015-0.016 lb ai/A
Submitter:	Du Pont
MRID Citation:	MRID 47138302
n:	44
min:	0.01
max:	2.80
median;	0.71
average:	0.86

	95th Percentile	99th Percentile	99.9th Percentile
EU Method I Normal	2.0 (2.5)	2.5 (3.0)	3.0 (--)
EU Method I Log Normal	4.0 **(6.0)**	**8.0** (14)	17 (--)
EU Method II Distribution-Free		1.9	
California Method $\mu + 3\sigma$		3.0	
UPLMedian95th		**4.0**	
Approximate Shapiro-Francia Normality Test		0.7002 p-value <= 0.01: Reject lognormality assumption	

Appendix II - International Residue Limit Status Sheet

INTERNATIONAL RESIDUE LIMIT STATUS

Chemical Name: methyl-2-[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)methylamino]carbonyl]amino]sulfonyl]benzoate	Common Name: Tribenuron methyl	√ Proposed tolerance Reevaluated tolerance Other	Date: 10/22/2007

Codex Status (Maximum Residue Limits)	**U. S. Tolerances**
√ No Codex proposal step 6 or above No Codex proposal step 6 or above for the crops requested	Petition Number: 7F7220 DP Number: 342085 Other Identifier:
Residue definition (step 8/CXL):	Reviewer/Branch: C. Swartz/ RAB2
	Residue definition: Tribenuron methyl

Crop (s)	MRL (mg/kg)	Crop(s)	Tolerance (ppm)
		Barley, hay	0.4
		Oat, forage	0.05
		Oat, hay	0.05
		Wheat, forage	0.3
		Wheat, hay	0.5

Limits for Canada	**Limits for Mexico**
No Limits √ No Limits for the crops requested	No Limits √ No Limits for the crops requested
Residue definition: methyl-2-[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)methylamino]carbonyl]amino]sulfonyl]benzoate	Residue definition: NA

Crop(s)	MRL (mg/kg)	Crop(s)	MRL (mg/kg)
Barley	0.05		
Oats	0.05		
Wheat	0.05		
Flax	0.02		
Rapeseed (canola)	0.02		
Milk	0.01		

Notes/Special Instructions:

Tribenuron Methyl (DPX-L5300)/352-632/PC Code 128887/E.I. du Pont de Nemours and Company/352
DACO 7.4.1/7.4.2/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3
Crop Field Trial/Residue Decline – Barley Hay

Primary Evaluator D. Dotson Date: 4/17/2008
Douglas Dotson, Ph.D., Chemist, RAB2

Peer Reviewer Date: 4/17/2008
William Drew, Chemist, RAB2

This DER was originally prepared under contract by Dynamac Corporation (1910 Sedwick Road, Building 100 Suite B, Durham NC 27713; submitted 10/28/2007). The DER has been reviewed by the Health Effects Division (HED) and revised to reflect current Office of Pesticide Programs (OPP) policies.

STUDY REPORT:

47138301 Thiel, A, Brookey, F. (2007) Determination of Decline and Magnitude of Residues of Tribenuron Methyl and Thifensulfuron Methyl in Barley (Hay) Following One Application of DPX-L5300 50 SG Herbicide and Two Applications of DPX-M6316 75 WG Herbicide Under Field Conditions (USA Season, 2006): Lab Project Number: DuPont-17574. ABC Laboratories Study No. 50016. Unpublished study prepared by Dupont de Nemours and Company. 301 pages.

EXECUTIVE SUMMARY:

E. I. DuPont de Nemours and Company submitted field trial data for tribenuron methyl on barley hay. In three winter barley field trials and 11 spring barley field trials conducted during 2005-2006 in EPA Growing Zones 2, 5, 10, and 11, a 50% water soluble granular (SG) formulation of tribenuron methyl was applied to barley as a single broadcast foliar application during vegetative development (Zadoks Growth Stage 36-50) at 0.015-0.016 lb ai/A. Applications were made using ground equipment at rates of 13-25 gal/A, and included the use of a non-ionic surfactant (NIS). Single control and duplicate treated samples of hay were cut at 22-31 days after treatment (DAT) at 10 sites, but were cut at 5-14 DAT at the remaining 4 sites. The target PHI was 30 days. To assess residue decline, hay samples were collected at 0, 3, 7, 14, 28 and 45 DAT at one test site. After harvest, hay samples were field-dried for 0-23 days before collection. Hay samples were stored at -20°C for up to 4 months prior to analysis. This interval is supported by the available storage stability data.

The LC/MS/MS method (DuPont Method 13412, Revision No. 1) used for determining residues of tribenuron methyl in/on barley hay was adequately validated in conjunction with the analysis of field trial samples. After hay samples were hydrated, residues were extracted twice with acetonitrile:K_2HPO_4 buffer at pH 7 (75:25, v:v) and initially cleaned up by partitioning against hexane. Aqueous soluble residues were further purified by elution through an ENV solid phase extraction (SPE) cartridge. Residues were then determined by LC/MS/MS using external standards and the m/z 396→155 ion transition for quantitation. The validated method limit of quantitation (LOQ) is 0.01 ppm, and the limit of detection (LOD) is 0.003 ppm.

Following a single broadcast foliar application of tribenuron methyl to barley around flag leaf emergence at 0.015-0.016 lb ai/A, residues in/on hay were <0.01-0.056 ppm for the 8 samples harvested at 5-14 DAT and <0.01-0.36 ppm for the 20 samples harvested at 22-31 DAT. Residues were <LOQ (<0.01 ppm) in 18 out of 20 samples from the 22-31 DAT interval. Average residues in/on hay were 0.018 ppm at 5-14 DAT and 0.036 ppm at 22-31 DAT. In the residue decline test, residue levels in/on hay declined rapidly from 0 to 7 DAT, and were <0.01 ppm from 14 to 45 DAT.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in the study, the barley field trial data are classified as scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the U.S. EPA Residue Chemistry Summary Document, Memo, D342085, D. Dotson, 4/17/2008.

COMPLIANCE:

Signed and dated Good Laboratory Practice (GLP), Quality Assurance and Data Confidentiality statements were provided. No deviations from regulatory requirements were reported which would have an adverse impact on the validity of the study.

A. BACKGROUND INFORMATION

Tribenuron methyl is a sulfonylurea herbicide (Group 2) registered for postemergence application to barley, canola, cotton, flax, field corn, oats, soybeans, sunflowers, and wheat for selective control of broadleaf weeds. Permanent tolerances are established for tribenuron methyl *per se* in/on barley, canola, corn, cotton, flax, grass, oats, rice, sorghum, soybean, sunflower, and wheat commodities at levels ranging from 0.02 to 0.10 ppm [40 CFR §180.451(a) and (c)].

For the existing postemergence uses on barley, oats, and wheat, DuPont is proposing new PHIs for forage and hay along with tolerances on these commodities (PP#7F7220). The chemical structure and nomenclature and the physicochemical properties of tribenuron methyl are presented in Tables A.1 and A.2.

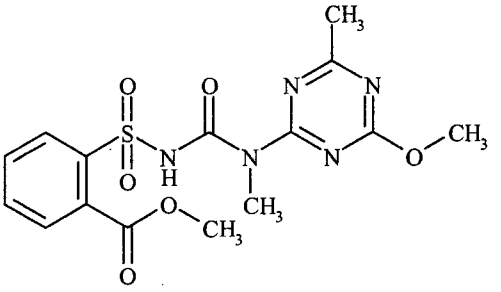
TABLE A.1. Tribenuron Methyl Nomenclature.	
Chemical structure	
Common name	Tribenuron methyl
Company experimental name	DPX-L5300
IUPAC name	methyl-2-[4-methoxy-6-methyl-1,3,5-triazin-2-yl(methyl)carbamoyl-sulfamoyl]benzoate
CAS name	methyl-2-[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)methylamino]carbonyl]amino]sulfonyl]benzoate
CAS registry number	101200-48-0
End-use product (EP)	50% SG (DuPont™ Express® Herbicide; EPA Reg. No. 352-632)

TABLE A.2. Physicochemical Properties of Tribenuron Methyl.		
Parameter	Value	Reference
Melting point/range	142°C	Provided in MRID
pH (20°C)	4.64	
Density (g/cm^3 at 19.6°C)	1.4594 ± 0.001	
Water solubility (g/L at 20°C)	pH 5 0.0489 pH 7 2.04 pH 9 18.3	
Solvent solubility (g/L at 20°C)	Acetone 39.1 Acetonitrile 46.4 Dichloromethane >250 Dimethylformamide 98.2 Ethyl acetate 16.3 n-Heptane 0.02 Methanol 2.59 n-Octanol 0.383 Xylene 13.1	
Vapor pressure (25°C)	2.7×10^{-7} mm Hg	
Dissociation constant (pK_a)	5.0	
Octanol/water partition coefficient Log(K_{OW}) at 20°C	pH 5 2.60 pH 7 0.78 pH 9 0.30	
UV/visible absorption (λ max)	pH 1.66 200, 231 nm pH 7 201, 256 nm pH 11.72 208, 256 nm	

30

B. EXPERIMENTAL DESIGN

B.1. Study Site Information

Fourteen barley field trials were conducted in EPA Growing Zones 2, 5, 10, and 11 during 2005-2006 (Tables B.1.1 and B.1.3). Winter barley was planted at 3 sites, and spring barley was planted at the other 11 sites. In each test, tribenuron methyl (50% SG) was applied to barley as a single broadcast foliar application during vegetative development (Zadoks Stage 36-50) at 0.015-0.016 lb ai/A (Table B.1.2.). Applications were made using ground equipment at rates of 13-25 gal/A, and included the use of a NIS adjuvant at 0.25-0.50% of the spray volume.

TABLE B.1.1. Trial Site Conditions.

Trial Identification (City, State; Year)	Soil characteristics [1]			
	Type	%OM	pH	CEC (meq/g)
Glen Allen, VA 2006	Sandy Loam	0.8	6.2	NR
Sycamore, GA 2006	Loamy Sand	NR	5.7	4.5
Richland, IA 2006	Silty Clay Loam	3.8	6.8	NR
Paynesville, MN 2006	Sandy Loam	3.2	6.2	19.6
York, NE 2006	Silty Clay Loam	2.8	6.4	NR
Northwood, ND 2006	Sand	2.7	7.1	NR
Northwood, ND 2006	Loam	4.0-8.0	6.6-7.8	NR
Britton, SD 2006	Loam	3.1	7.5	21.8
Northwood, ND 2006	Loam	3.0-5.0	6.1-7.3	NR
Jerome, ID 2006	Loam	2.1	8.1	21.8
Sanger, CA 2006	Sandy Loam	0.7	6.2	10.5
Ephrata, WA 2006	Loamy Sand	1.0	7.4	12.4
Payette, ID 2006	Loam	2.07	6.9	NR
York, NE 2006	Silt Loam	2.0	6.1	NR

NR=not reported

Average monthly minimum and maximum temperatures and monthly precipitation and irrigation were reported for each site during the test period, along with the 10-year monthly averages for temperatures (min and max) and precipitation. Weather conditions were generally within normal historical ranges at all test sites. The tests were conducted according to normal agricultural practices for the different regions, and information was provided on maintenance pesticides and fertilizers used at each location.

TABLE B.1.2. Study Use Pattern.

Location (City, State; Year) Trial ID	End-use Product	Application Information					Tank Mix/ Adjuvants
		Method; Timing	Volume (gal/A)	Single Rate (lb ai/A)	RTI [1] (days)	Total Rate (lb ai/A)	
Glen Allen, VA 2006 1	50% SG	One broadcast foliar application at Zadoks 42	20	0.016	--	0.016	0.25% NIS
Sycamore, GA 2006 2	50% SG	One broadcast foliar application at Zadoks 40	22	0.015	--	0.015	0.5% NIS
Richland, IA 2006 3	50% SG	One broadcast foliar application at Zadoks 40	16	0.016	--	0.016	0.26% NIS
Paynesville, MN 2006 4	50% SG	One broadcast foliar application at Zadoks 40	15	0.015	--	0.015	0.26% NIS

TABLE B.1.2. Study Use Pattern.

Location (City, State; Year) Trial ID	End-use Product	Application Information					Tank Mix/ Adjuvants
		Method; Timing	Volume (gal/A)	Single Rate (lb ai/A)	RTI [1] (days)	Total Rate (lb ai/A)	
York, NE 2006 5	50% SG	One broadcast foliar application at BBCH 39	20	0.016	--	0.016	0.5% NIS
Northwood, ND 2006 6	50% SG	One broadcast foliar application at Zadoks 38	15	0.015	--	0.015	0.3% NIS
Northwood, ND 2006 7	50% SG	One broadcast foliar application at Zadoks 38	15	0.016	--	0.016	0.3% NIS
Britton, SD 2006 8	50% SG	One broadcast foliar application at Zadoks 37	13	0.016	--	0.016	0.5% NIS
Northwood, ND 2006 9	50% SG	One broadcast foliar application at Zadoks 38	15	0.015	--	0.015	0.3% NIS
Jerome, ID 2006 10	50% SG	One broadcast foliar application at Zadoks 40	15	0.015	--	0.015	0.25% NIS
Sanger, CA 2006 11	50% SG	One broadcast foliar application at Zadoks 37	25	0.015	--	0.015	0.3% NIS
Ephrata, WA 2006 12	50% SG	One broadcast foliar application at Zadoks 39-50	15	0.016	--	0.016	0.38% NIS
Payette, ID 2006 13	50% SG	One broadcast foliar application at Zadoks 36	25	0.016	--	0.016	0.3% NIS
York, NE 2006 14	50% SG	One broadcast foliar application at BBCH 37	20	0.015	--	0.015	0.5% NIS

[1] RTI = Retreatment Interval.

TABLE B.1.3. Trial Numbers and Geographical Locations.

NAFTA Growing Zones	Barley		
	Submitted	Requested	
		Canada	U.S.
1	--	--	1 [1]
1A	--	--	--
2	2	--	1 [1]
3	--	--	--
4	--	--	--
5	8 [2]	1	3
5A	--	--	--
5B	--	1	--
6	--	--	--
7	--	2	4
7A	--	--	--
8	--	--	--
9	--	--	1
10	1	--	1
11	3 [3]	--	2
12	--	--	--
13	--	--	--
14	--	12	--
Total	14	16	12

[1] One barley field trial is required in Zone 1 or 2.
[2] Six of the field trials in Zone 5 were near the border with Zone 7.
[3] One of the field trials in Zone 11 was near the border with Zone 9.

B.2. Sample Handling and Preparation

Single control and duplicate treated samples of hay were cut at 22-31 DAT at ten field sites, but were cut earlier than the targeted 30-day interval at 4 field sites (5 or 14 DAT). Hay samples (≥0.25 kg/sample) were cut between Zadoks Growth Stages 43 and 83. To examine residue decline, hay samples from one site were harvested at 0, 3, 7, 14, 28, and 45 DAT. Hay samples were field-dried for 0-9 days after harvest, except at one site (23 days), and were placed in frozen storage within 0.5 hours of collection. Samples were stored frozen at the field sites for 4-44 days prior to frozen shipment by ACDS freezer truck to the analytical laboratory, Morse Laboratories, Inc, Sacramento, CA. At the analytical laboratory, samples were ground with dry ice and stored at -20 ± 5°C until extraction for analysis.

B.3. Analytical Methodology

Samples of barley hay were analyzed for residues of tribenuron methyl using an LC/MS/MS method (DuPont Method 13412, Revision No. 1). DuPont Method 13412 was previously validated for the analysis of tribenuron methyl in oily crop matrices and cereal grains (Memo, D330633 and 46352003.de2, S. Hummel, 8/08/06). The method has undergone a successful independent laboratory trial, and is also capable of determining other sulfonylurea herbicides (ethametsulfuron methyl, rimsulfuron, thifensulfuron methyl, and chlorimuron ethyl). The modifications made to the method for the analysis of residues in hay included: (1) addition of a hydration step for dry matrices, (2) replacing the second centrifugation step with filtration, and (3) modification of the hexane partition step for hay to include extra cleanup with BONDESIL-SAX.

Hay samples were initially hydrated for 5 minutes in 20 mM K_2HPO_4 buffer (pH 7) and then soaked for 30 minutes in the extraction solvent. Residues were extracted twice by homogenization with acetonitrile:K_2HPO_4 buffer at pH 7 (75:25, v:v), and the extracts were centrifuged and filtered. The aqueous extract was acidified with glacial acetic acid, mixed with 1g of BONDESIL-SAX, partitioned with hexane, and centrifuged after mixing. The hexane fraction was discarded and the aqueous fraction was neutralized with concentrated NaOH. Residues were then cleaned up using an ENV SPE cartridge. After loading residues onto the ENV SPE cartridge, the cartridge was washed with hexane, and residues were eluted with 25 mM NH_4OH:methanol (1:99, v:v), mixed with 5 mM ammonium acetate, and concentrated to remove the methanol. Residue were redissolved in acetonitrile:50 mM ammonium acetate (1:9, v:v) and analyzed by LC/MS/MS. The HPLC system utilized a phenyl-hexyl column with a mobile phase gradient of water to methanol, each containing 0.05% formic acid, and a MS/MS detector. Tribenuron methyl residues were quantified using the m/z 396→155 transition and confirmed using the m/z 396→181 transition. External standards were used for quantitation. The method has a validated LOQ of 0.01 ppm and a reported LOD of 0.003 ppm.

The above method was validated in conjunction with the analysis of field trial samples, using control samples of barley hay fortified with tribenuron methyl at 0.01-5.0 ppm.

3)

C. RESULTS AND DISCUSSION

Barley hay samples were stored at -20 ± 5°C for up to 4 months prior to extraction for analysis. Storage stability data are available indicating that tribenuron methyl is stable in frozen storage for up to 4 and 6 months in corn stover and forage, respectively (Memo, D330633, S. Hummel, 8/08/06). These data will adequately support the storage intervals and conditions for hay from the current barley field trials.

The LC/MS/MS method (Method 13412, Rev. 1) used for determining residues of tribenuron methyl in/on barley hay was adequately validated in conjunction with the analysis of field trial samples. The average concurrent recovery of tribenuron methyl (± the standard deviation) was 75 ± 9% from barley hay. The validated method LOQ for tribenuron methyl is 0.01 ppm and the LOD is 0.003 ppm in both matrices. Apparent residues of tribenuron methyl were <LOD in/on all control samples of hay. Adequate sample calculations and example chromatograms were provided, and the fortification levels used for the concurrent recoveries bracketed the residue levels observed in treated samples.

Following a single broadcast foliar application of tribenuron methyl to barley around flag leaf emergence at 0.015-0.016 lb ai/A, residues in/on hay were <0.01-0.056 ppm for the 8 samples harvested at 5-14 DAT and <0.01-0.36 ppm for the 20 samples harvested at 22-31 DAT (Table C.3). Residues were <0.01 ppm in/on 18 out of 20 samples at the 22-31 DAT interval. Average residues in/on hay were 0.018 ppm at 5-14 DAT and 0.036 ppm at 22-31 DAT (Table C.4). In the residue decline test, residue levels in/on hay declined rapidly from 0.14 ppm at 0 DAT to 0.02 ppm by 7 DAT, and were <0.01 ppm from 14 to 45 DAT (Figure C.1).

Common cultural practices were used to maintain plants. The weather conditions, maintenance chemicals, and fertilizer used in the study did not have a notable impact on the residue data.

TABLE C.1. Summary of Concurrent Recoveries of Tribenuron Methyl from Barley Hay.

Matrix	Spike Level (ppm)	Sample Size (n)	Recoveries (%)	Mean ± Std. Dev. (%)
Barley Hay	0.01	9	68, 90, 74, 70, 76, 89, 74, 85, 68	77 ± 9
	0.05	6	61, 86, 78, 86, 68, 63	74 ± 11
	0.10	1	68	68
	0.50	1	65	65
	5.0	1	80	80
	Total	**18**	**61-90**	**75 ± 9**

TABLE C.2. Summary of Storage Conditions

Matrix	Storage Temperature (°C)	Actual Storage Duration (months)	Interval of Demonstrated Storage Stability (months)
Barley Hay	-20 ± 5	4	4 (corn stover) [1] 6 (corn forage) [1]

[1] D330633, S. Hummel, 8/08/06.

TABLE C.3. Residue Data from Barley Field Trials with Tribenuron Methyl.

Trial ID (City, State; Year)	Zone	Type; Variety	Matrix	Total Rate (lb ai/A)	PHI (days)	Residues (ppm)[1]	
Glen Allen, VA 2006 1	1	Winter; Thoroughbred	Hay	0.016	28	ND [2]	ND
Sycamore, GA 2006 2	2	Winter; Callad	Hay	0.015	30	(0.006)	(0.005)
Richland, IA 2006 3	5	Spring; Robust	Hay	0.016	22	ND	ND
Paynesville, MN 2006 4	5	Spring; Conton	Hay	0.015	31	0.17	0.36
York, NE 2006 5	5	Spring; Robust	Hay	0.016	30	ND	ND
Northwood, ND 2006 6	7	Spring; Lacey	Hay	0.015	5	0.026	0.056
Northwood, ND 2006 7	7	Spring; Lacey	Hay	0.016	5	(0.008)	(0.006)
Britton, SD 2006 8	7	Spring; Stander	Hay	0.016	0	0.14	0.14
					3	0.045	0.043
					7	0.02	0.018
					14	ND	ND
					28	ND	ND
					45	ND	ND
Northwood, ND 2006 9	7	Spring; Lacey	Hay	0.015	5	(0.004)	(0.003)
Jerome, ID 2006 10	11	Spring; Merit	Hay	0.015	28	ND	ND
Sanger, CA 2006 11	10	Winter; UC 937	Hay	0.015	30	ND	ND
Ephrata, WA 2006 12	11	Spring; Baronesse	Hay	0.016	30	ND	ND
Payette, ID 2006 13	11	Spring; Baronesse	Hay	0.016	14	ND	ND
York, NE 2006 14	7	Spring; Robust	Hay	0.015	28	ND	ND

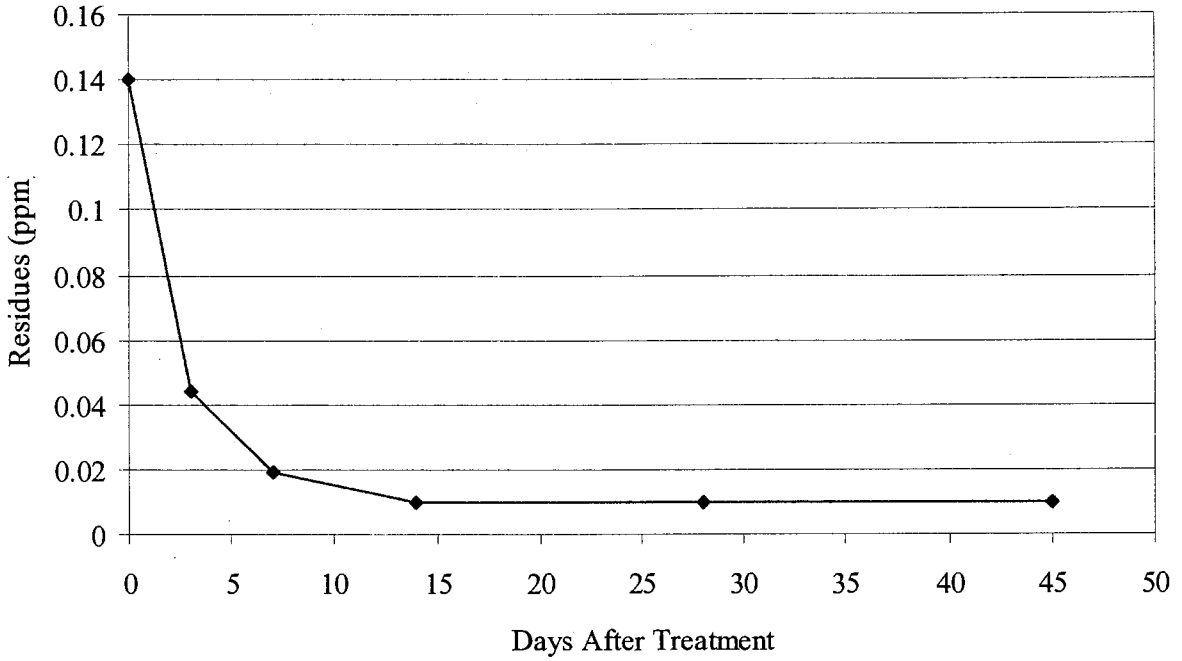
[1] The method LOQ and LOD are 0.01 and 0.003 ppm, respectively. Values <LOQ but ≥LOD are reported in parentheses.
[2] ND=non-detectable (<LOD).

TABLE C.4. Summary of Residue Data from Barley Field Trials with Tribenuron Methyl.

Commodity	Total Applic. Rate (lb ai/A)	PHI (days)	Residue Levels (ppm) [1]						
			n	Min.	Max.	HAFT[1]	Median (STMdR)	Mean (STMR)	Std. Dev.
Barley Hay	0.015-0.016	5-14	8	<0.01	0.056	0.041	0.01	0.018	0.016
		22-31	20	<0.01	0.360	0.265	0.01	0.036	0.084

[1] The method LOQ is 0.01 ppm. For calculating the median, mean, and standard deviation, the method LOQ was used for values <LOQ.
[2] HAFT = Highest Average Field Trial.

Figure C.1. Decline Data for Residues of Tribenuron-methyl in/on Barley Hay.



D. CONCLUSION

Although 8 of the 28 hay samples were not harvested at the targeted PHI, the barley field trial data are adequate and support the use of a single postemergence application of tribenuron methyl to barley at 0.015 lb ai/A. The available residue data support a minimum PHI of 30 days for hay.

E. REFERENCES

D330633, Tribenuron Methyl. Addition of Preplant Burndown Uses on Field Corn, Rice, Sorghum, and Soybeans (PRIA R19; PP#4F6890) and Postemergence Uses on Sunflower (IR-4 Request; PP#4E6855). Summary of Analytical Chemistry and Residue Data., S. Hummel, 8/08/2006

F. DOCUMENT TRACKING

Petition Number: 7F7220
DP Number: 342085
PC Code: 128887

Tribenuron Methyl(DPX-L5300)/352-632/PC Code 128845/E.I. duPont de Nemours and Co./352
DACO 7.4.1/7.4.2/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3
Crop Field Trial/Residue Decline – Oat Forage and Hay

Primary Evaluator D. Dotson Date: 4/17/2008
Douglas Dotson, Ph.D., Chemist, RAB2

Peer Reviewer Date: 4/17/2008
William Drew, Chemist, RAB2

This DER was originally prepared under contract by Dynamac Corporation (1910 Sedwick Road, Building 100 Suite B, Durham NC 27713; submitted 10/26/2007). The DER has been reviewed by the Health Effects Division (HED) and revised to reflect current Office of Pesticide Programs (OPP) policies.

STUDY REPORT:

47138302 Thiel, A, Brookey, F. (2007) Determination of Decline and Magnitude of Residues of Tribenuron Methyl and Thifensulfuron Methyl in Oat (Forage and Hay) Following One Application of DPX-L5300 50SG Herbicide and Two Applications of DPX-M6316 75WG Herbicide Under Field Conditions (USA Season, 2006): Lab Project Number: DuPont-17575. ABC Laboratories Study No. 50019. Unpublished study prepared by DuPont de Nemours and Company. 464 pages.

EXECUTIVE SUMMARY:

E. I. DuPont de Nemours and Company submitted field trial data for tribenuron methyl on oat forage and hay. In three winter oat field trials and 15 spring oat field trials conducted during 2005-2006 in EPA Growing Zones 2, 5, 6, 7, and 8, a 50% soluble granular (SG) formulation of tribenuron methyl was applied to oats as a single broadcast foliar application during vegetative development at 0.015-0.016 lb ai/A. Each field trial included separate treated plots for the harvest of forage and hay. The application was made to the forage plots during Zadoks Growth Stages 23 through 39, and to the hay plots during Zadoks Growth Stages 37 through 45. All applications were made using ground equipment at rates of 12-26 gal/A, and included the use of a non-ionic surfactant (NIS).

Forage samples were collected from each site at 0 and 5-8 days after treatment (DAT). Hay samples were collected at 5-12 DAT from 5 sites and at 25-33 DAT from 13 sites (the target PHI for hay was 30 days). Single control and duplicate treated samples were collected for both matrices. To assess residue decline, forage and hay samples were collected at approximately 0, 3, 7, 14, 28, and 45 DAT at two test sites. After harvest, hay samples were field-dried for 1-13 days before collection. Forage and hay samples were stored at -20°C for up to 4.8 months prior to analysis. This interval is supported by the available storage stability data.

The LC/MS/MS method (DuPont Method 13412, Revision No. 1) used to determine residues of tribenuron methyl in/on oat forage and hay was adequately validated in conjunction with the analysis of field trial samples. After the hay samples were hydrated, residues were extracted twice from forage and hay with acetonitrile:K_2HPO_4 buffer at pH 7 (75:25, v:v) and initially cleaned up by partitioning against hexane. Aqueous soluble residues were further purified by

elution through an ENV solid phase extraction (SPE) cartridge. Residues were then determined by LC/MS/MS using external standards and the m/z 396→155 ion transition for quantitation. The validated method limit of quantitation (LOQ) is 0.01 ppm, and the limit of detection (LOD) is 0.003 ppm.

The residue data from the Paynesville, MN test site were not included when calculating overall residues because the forage (0-day) and hay samples from this site were apparently either switched or mislabeled. The 0-day forage samples from this site had residues (<0.003 ppm) well below the residues in all other 0-day forage samples (0.22-1.50 ppm), but similar to the residues in the other 30-day hay samples. Concomitantly, the 30-day hay samples from this site had residues (0.57, 0.88 ppm) well above the residues in all other 30-day hay samples (<0.003 ppm), but similar to the residue levels in the 0-day forage samples from the other sites.

Following a single broadcast foliar application of tribenuron methyl (SG) at 0.015-0.016 lb ai/A, residues in/on oat forage were 0.22-1.50 ppm for the 34 samples harvested at 0 DAT and <0.01-0.043 ppm for 32 samples harvested at 5-8 DAT. Residues in/on hay were <0.01-0.028 ppm for the 10 samples harvested at 5-12 DAT and ND (<0.003 ppm) for the 24 samples harvested at 25-33 DAT. Average residues in/on forage were 0.739 ppm at 0 DAT and 0.014 ppm at 5-12 DAT. Average residues in/on hay were 0.012 ppm at 5-12 DAT and <0.01 ppm at 25-33 DAT. In the two residue decline tests, residue levels in/on both forage and hay declined rapidly from 0 to 7 DAT, and were <0.01 ppm from 7 to 45 DAT in both forage and hay.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in the study, the oat field trial data are classified as scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the U.S. EPA Residue Chemistry Summary Document, Memo, D342085, D. Dotson, 4/17/2008.

COMPLIANCE:

Signed and dated Good Laboratory Practice (GLP), Quality Assurance and Data Confidentiality statements were provided. No deviations from regulatory requirements were reported which would have an adverse impact on the validity of the study.

A. BACKGROUND INFORMATION

Tribenuron methyl is a sulfonylurea herbicide (Group 2) registered for postemergence application to barley, canola, cotton, flax, field corn, oats, soybeans, sunflowers, and wheat for selective control of broadleaf weeds. Permanent tolerances are established for tribenuron methyl *per se* in/on barley, canola, corn, cotton, flax, grass, oat, rice, sorghum, soybean, sunflower, and wheat commodities at levels ranging from 0.02 to 0.10 ppm [40 CFR §180.451(a) and (c)].

For the existing postemergence uses on barley, oats and wheat, DuPont is proposing new PHIs for forage and hay along with tolerances on these commodities (PP#7F7220). The chemical

structure and nomenclature and the physicochemical properties of tribenuron methyl are presented in Tables A.1 and A.2.

TABLE A.1. Tribenuron Methyl Nomenclature.

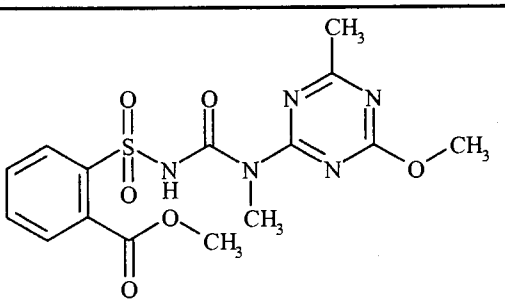
Chemical structure	
Common name	Tribenuron methyl
Company experimental name	DPX-L5300
IUPAC name	methyl-2-[4-methoxy-6-methyl-1,3,5-triazin-2-yl(methyl)carbamoyl-sulfamoyl]benzoate
CAS name	methyl-2-[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)methylamino]carbonyl]amino]sulfonyl]benzoate
CAS registry number	101200-48-0
End-use product (EP)	50% SG (DuPont™ Express® Herbicide; EPA Reg. No. 352-632)

TABLE A.2. Physicochemical Properties of Tribenuron Methyl.

Parameter	Value	Reference
Melting point/range	142°C	Provided in MRID
pH (20°C)	4.64	
Density (g/cm^3 at 19.6°C)	1.4594 ± 0.001	
Water solubility (g/L at 20°C)	pH 5 0.0489 pH 7 2.04 pH 9 18.3	
Solvent solubility (g/L at 20°C)	Acetone 39.1 Acetonitrile 46.4 Dichloromethane >250 Dimethylformamide 98.2 Ethyl acetate 16.3 n-Heptane 0.02 Methanol 2.59 n-Octanol 0.383 Xylene 13.1	
Vapor pressure (25°C)	2.7×10^{-7} mm Hg	
Dissociation constant (pK_a)	5.0	
Octanol/water partition coefficient Log(K_{OW}) at 20°C	pH 5 2.60 pH 7 0.78 pH 9 0.30	
UV/visible absorption (λ max)	pH 1.66 200, 231 nm pH 7 201, 256 nm pH 11.72 208, 256 nm	

B. EXPERIMENTAL DESIGN

B.1. Study Site Information

Eighteen oat field trials were conducted in EPA Growing Zones 2, 5, 6, 7, and 8 during 2005-2006 (Tables B.1.1 and B.1.3). Winter oats were planted at 3 sites and spring oats were planted at the other 15 sites. Each trial consisted of a control and two treated plots, one for the harvest of forage and another for the harvest of hay. In each test, tribenuron methyl (50% SG) was applied to oats as a single broadcast foliar application during vegetative development at 0.015-0.016 lb ai/A (Table B.1.2.). The application was made to the forage plots during Zadoks Growth Stages 23 through 39, and to the hay plots during Zadoks Growth Stages 37 through 45. All applications were made using ground equipment at rates of 12-26 gal/A, and included the use of a NIS adjuvant at 0.24-0.51% of the spray volume.

TABLE B.1.1. Trial Site Conditions.

Trial Identification (City, State; Year)	Soil characteristics [1]			
	Type	%OM	pH	CEC (meq/g)
Glen Allen, VA 2006	Sandy Loam	1.5	5.1	7.4
Sycamore, GA 2006	Loamy Sand	NR	5.7	NR
Richland, IA 2006	Silt Loam	4.0	6.3	NR
Richland, IA 2006	Silty Clay Loam	4.0	6.9	NR
Delavan, WI 2006	Silt Loam	2.0-4.0	4.5-7.8	NR
Paynesville, MN 2006	Sandy Loam	3.2	6.2	NR
Clarence, MO 2006	Silty Clay Loam	2.5	5.5	18.4
Northwood, ND 2006	Sand	5.3	6.6	NR
Fargo, ND 2006	Clay Loam	3.6	7.8	30.5
York, NE 2006	Silty Clay Loam	2.8	6.4	NR
Britton, SD 2006	Clay Loam	3.0	7.8	33.8
East Bernard, TX 2006	Sandy Clay	0.4	7.4	15.6
Northwood, ND 2006	Sand	2.7	7.1	NR
Northwood, ND 2006	Loam	4.0-8.0	6.6-7.8	NR
Velva, ND 2006	Loam	4.0	7.6	27.2
Levelland, TX 2006	Sandy Loam	0.5	8.1	NR
Clarence, MO 2006	Silt Loam	4.2	5.8	12.9
St. Joseph, MO 2006	Silt Loam	1.4	6.8	13.1

[1] These parameters are optional except in cases where their value affects the use pattern for the chemical.
NR = not reported.

Average monthly minimum and maximum temperatures and monthly precipitation and irrigation were reported for each site during the test period, along with the 10-year monthly averages for temperatures (min and max) and precipitation. Weather conditions were within normal historical ranges at all test sites. The tests were conducted according to normal agricultural practices for the different regions and information was provided on maintenance pesticides and fertilizers used at each location.

TABLE B.1.2. Study Use Pattern.

Location (City, State; Year) Trial ID	End-use Product	Application Information					Tank Mix/ Adjuvants
		Method; Timing	Volume (gal/A)	Single Rate (lb ai/A)	RTI [1] (days)	Total Rate (lb ai/A)	
Glen Allen, VA 2006 1	50% SG	One broadcast foliar application, Zadoks 37	20	0.015	--	0.015	0.25% NIS
		One broadcast foliar application, Zadoks 40	20	0.016	--	0.016	0.25% NIS
Sycamore, GA 2006 2	50% SG	One broadcast foliar application, Zadoks 39	24	0.016	--	0.016	0.5% NIS
		One broadcast foliar application, Zadoks 39-40	25	0.015	--	0.015	0.51% NIS
Richland, IA 2006 3	50% SG	One broadcast foliar application, Zadoks 23-24	16	0.015	--	0.015	0.24% NIS
		One broadcast foliar application, Zadoks 39-40	16	0.016	--	0.016	0.26% NIS
Richland, IA 2006 4	50% SG	One broadcast foliar application, Zadoks 24-25	17	0.015	--	0.015	0.28% NIS
		One broadcast foliar application, Zadoks 40	16	0.016	--	0.016	0.26% NIS
Delavan, WI 2006 5	50% SG	One broadcast foliar application, Zadoks 30	19	0.015	--	0.015	0.25% NIS
		One broadcast foliar application, Zadoks 45	19	0.016	--	0.016	0.25% NIS
Paynesville, MN 2006 6	50% SG	One broadcast foliar application, Zadoks 30	15	0.015	--	0.015	0.26% NIS
		One broadcast foliar application, Zadoks 38	15	0.015	--	0.015	0.26% NIS
Clarence, MO 2006 7	50% SG	One broadcast foliar application, Zadoks 37-39	20	0.016	--	0.016	0.27% NIS
		One broadcast foliar application, Zadoks 40	20	0.015	--	0.015	0.30% NIS
Northwood, ND 2006 8	50% SG	One broadcast foliar application, Zadoks 24	15	0.015	--	0.015	0.30% NIS
		One broadcast foliar application, Zadoks 38	15	0.015	--	0.015	0.30% NIS
Fargo, ND 2006 9	50% SG	One broadcast foliar application, BBCH 32	15	0.016	--	0.016	0.50% NIS
		One broadcast foliar application, BBCH 39	15	0.016	--	0.016	0.50% NIS
York, NE 2006 10	50% SG	One broadcast foliar application, BBCH 30	20	0.015	--	0.015	0.50% NIS
		One broadcast foliar application, BBCH 37	20	0.016	--	0.016	0.50% NIS
Britton, SD 2006 11	50% SG	One broadcast foliar application, Zadoks 32-33	13	0.016	--	0.016	0.50% NIS
		One broadcast foliar application, Zadoks 39	13	0.016	--	0.016	0.50% NIS
East Bernard, TX 2006 12	50% SG	One broadcast foliar application, Zadoks 36	25	0.016	--	0.016	0.50% NIS
		One broadcast foliar application, Zadoks 40	26	0.016	--	0.016	0.50% NIS
Northwood, ND 2006 13	50% SG	One broadcast foliar application, Zadoks 24	15	0.015	--	0.015	0.30% NIS
		One broadcast foliar application, Zadoks 38	15	0.015	--	0.015	0.30% NIS

A1

TABLE B.1.2. Study Use Pattern.

Location (City, State; Year) Trial ID	End-use Product	Application Information					Tank Mix/ Adjuvants
		Method; Timing	Volume (gal/A)	Single Rate (lb ai/A)	RTI [1] (days)	Total Rate (lb ai/A)	
Northwood, ND 2006 14	50% SG	One broadcast foliar application, Zadoks 24	15	0.015	--	0.015	0.30% NIS
		One broadcast foliar application, Zadoks 38	15	0.015	--	0.015	0.30% NIS
Velva, ND 2006 15	50% SG	One broadcast foliar application, Zadoks 30	12	0.016	--	0.016	0.25% NIS
		One broadcast foliar application, Zadoks 37	12	0.016	--	0.016	0.25% NIS
Levelland, TX 2006 16	50% SG	One broadcast foliar application, Zadoks 30	15	0.016	--	0.016	0.40% NIS
		One broadcast foliar application, Zadoks 39	15	0.015	--	0.015	0.40% NIS
Clarence, MO 2006 17	50% SG	One broadcast foliar application, Zadoks 37	20	0.016	--	0.016	0.29% NIS
		One broadcast foliar application, Zadoks 39	20	0.016	--	0.016	0.30% NIS
St. Joseph, MO 2006 18	50% SG	One broadcast foliar application, Zadoks 31-32	14	0.015	--	0.015	0.25% NIS
		One broadcast foliar application, Zadoks 39-40	14	0.015	--	0.015	0.25% NIS

[1] RTI = Retreatment Interval.

TABLE B.1.3. Trial Numbers and Geographical Locations.

NAFTA Growing Zones	Oats		
	Submitted	Requested	
		Canada	U.S.
1	--	1	1
1A	--	--	--
2	2	--	1
3	--	--	--
4	--	--	--
5	11	1	9
5A	--	1	--
5B	--	1	--
6	1	--	1
7	3	2	3
7A	--	--	--
8	1	--	1
9	--	--	--
10	--	--	--
11	--	--	--
12	--	--	--
13	--	--	--
14	--	10	--
Total	18	16	16

42

B.2. Sample Handling and Preparation

Single control and duplicate treated samples of oat forage and hay were harvested from each test site mechanically or by hand. Forage samples (≥1 kg/sample) were collected from each site at 0 and 5-8 DAT. At two sites, forage samples were collected immediately before the second application (-0 DAT) and at 0, 3, 7, 14, 28-30, and 44-45 DAT to assess residue decline. Harvest intervals for hay were earlier than the targeted 30-day interval at 5 field sites (5-12 DAT), but were 25-33 DAT at the remaining 13 field sites. Hay samples (≥0.35 kg/sample) were generally cut around Zadoks Stage 50-70, except at a few sites where hay samples were collected earlier (Zadoks Stage 43). To examine residue decline, hay samples from two sites were harvested at 0, 3, 7, 14, 28, and 42-45 DAT. Hay samples were field-dried after harvest for 1-13 days prior to collection. Both forage and hay samples were placed in frozen storage within 5.5 hours of collection. Samples were stored frozen at the field sites for 1-51 days prior to frozen shipment by ACDS freezer truck to the analytical laboratory, Morse Laboratories, Inc, Sacramento, CA. At the analytical laboratory, samples were ground with dry ice and stored at -20 ± 5°C until extraction for analysis.

B.3. Analytical Methodology

Forage and hay samples were analyzed for residues of tribenuron methyl using an LC/MS/MS method (DuPont Method 13412, Revision No. 1). DuPont Method 13412 was previously validated for the analysis of tribenuron methyl in oily crop matrices and cereal grains (Memo, D330633, and 46352003.de2, S. Hummel 8/8/2006). The method has undergone a successful independent laboratory trial, and is also capable of determining other sulfonylurea herbicides (ethametsulfuron methyl, rimsulfuron, thifensulfuron methyl, and chlorimuron ethyl). The modifications made to the method for the analysis of residues in forage and hay included: (1) addition of a hydration step for dry matrices; (2) replacing the second centrifugation step with filtration; and (3) modification of the hexane partition step for hay to include extra cleanup with BONDESIL-SAX.

Hay samples were initially hydrated for 5 minutes in 20 mM K_2HPO_4 buffer (pH 7) and then soaked for 30 minutes in the extraction solvent. Residues were extracted twice from forage and hay by homogenization with acetonitrile:K_2HPO_4 buffer at pH 7 (75:25, v:v), and the extracts were centrifuged and filtered. For forage, the aqueous extract was partitioned directly with hexane, and the hexane phase was discarded. For hay samples, the aqueous extract was acidified with glacial acetic acid, mixed with 1g of BONDESIL-SAX, partitioned with hexane, then centrifuged after mixing. The hexane fraction was discarded and the aqueous fraction was neutralized with concentrated NaOH. Residues from both forage and hay samples were then cleaned up using an ENV SPE cartridge. After residues were loaded onto the ENV SPE cartridge, the cartridge was washed with hexane, and residues were eluted with 25 mM NH_4OH:methanol (1:99, v:v), mixed with 5 mM ammonium acetate, and concentrated to remove the methanol. Residues were redissolved in acetonitrile:50 mM ammonium acetate (1:9, v:v) and analyzed by LC/MS/MS. The HPLC system utilized a phenyl-hexyl column with a mobile phase gradient of water to methanol, each containing 0.05% formic acid, and a MS/MS detector. Residues were quantified using the m/z 396→155 transition and confirmed using the m/z

396→181 transition. External standards were used for quantitation. The method has a validated LOQ of 0.01 ppm and a reported LOD of 0.003 ppm.

The above method was validated in conjunction with the analysis of field trial samples, using control samples of oat forage and hay fortified with tribenuron methyl at 0.01-5.0 ppm.

C. RESULTS AND DISCUSSION

Oat forage and hay samples were stored at -20 ± 5°C for up to 4.8 months prior to extraction for analysis. Storage stability data are available indicating that tribenuron methyl is stable in frozen storage for up to 4 and 6 months in corn stover and forage, respectively (Memo, D330633, S. Hummel, 8/08/06). These data will adequately support the storage intervals and conditions for forage and hay from the current oat field trials.

The LC/MS/MS method (Method 13412, Rev. 1) used for determining residues of tribenuron methyl in/on oat forage and hay was adequately validated in conjunction with the analysis of field trial samples. The average concurrent recovery of tribenuron methyl (± the standard deviation) was 88 ± 10% from forage and 81 ± 7% from hay. The validated method LOQ for tribenuron methyl is 0.01 ppm and the LOD is 0.003 ppm in both matrices. Apparent residues of tribenuron methyl were <LOD in/on control samples of forage and hay. Adequate sample calculations and example chromatograms were provided, and the fortification levels used for the concurrent recoveries bracketed the residue levels observed in treated samples.

Although no explanation was provided by the study authors, the reviewer noted that the 0-day forage samples and 30-day hay samples from the Paynesville, MN test site appear to have been switched or mislabeled. The 0-day forage samples from this site had nondetectable residues (<0.003 ppm) well below the residues in all other 0-day forage samples (0.22-1.50 ppm), but similar to the residues in the 30-day hay samples from the other sites. Concomitantly, the 30-day hay samples from the Paynesville test had residues (0.57, 0.88 ppm) well above the residues in all other 30-day hay samples (ND, <0.003 ppm), but well within the range of values for the 0-day forage samples from other sites. In addition, residues of thifensulfuron methyl, which were also determined in these samples (47138302.del, D342084, D. Dotson, 4/17/2008), showed the same inverted pattern for residues in the 0-day forage and 30-day hay. Therefore, the residue data from this field trial were not included when calculating overall residues.

Following a single broadcast foliar application of tribenuron methyl at 0.015-0.016 lb ai/A, residues in/on forage were 0.22-1.50 ppm for the 34 samples harvested at 0 DAT and <0.01-0.043 ppm for 32 samples harvested at 5-8 DAT (Table C.3). Residues in/on hay were <0.01-0.028 ppm for the 10 samples harvested at 5-12 DAT and <0.003 ppm for the 24 samples harvested around the targeted 30-day PHI (25-33 DAT). Average residues in/on forage were 0.739 ppm at 0 DAT and 0.014 ppm at 5-12 DAT (Table C.4.1). Average residues in/on hay were 0.012 ppm at 5-12 DAT and <0.01 ppm at 25-33 DAT.

In the two residue decline tests, residue levels in/on both forage and hay declined rapidly from 0 to 7 DAT, and were <0.01 ppm from 7 to 45 DAT in both forage and hay (Figure C.1; Table C.4.2).

Common cultural practices were used to maintain plants, and the weather conditions, maintenance chemicals, and fertilizer used in the study did not have a notable impact on the residue data.

TABLE C.1. Summary of Concurrent Recoveries of Tribenuron Methyl from Oat Forage and Hay.

Matrix	Spike Level (ppm)	Sample Size (n)	Recoveries (%)	Mean ± Std. Dev. (%)
Oat Forage	0.01	15	79, 88, 82, 81, 79, 97, 84, 90, 77, 116, 89, 78, 90, 109, 97	89 ± 12
	0.05	1	79	79
	0.10	2	84, 89	87 ± 4
	1.0	1	105	105
	2.0	4	88, 78, 85, 94	86 ± 7
	5.0	7	83, 82, 89, 86, 87, 75, 91	85 ± 5
	Total	**30**	**75-116**	**88 ± 10**
Oat Hay	0.01	14	89, 71, 84, 90, 85, 68, 75, 93, 82, 74, 77, 82, 94, 84	82 ± 8
	0.05	11	73, 82, 70, 82, 82, 74, 84, 74, 93, 82, 83	80 ± 7
	0.10	1	83	83
	0.5	1	70	70
	5.0	1	83	83
	Total	**28**	**68-94**	**81 ± 7**

TABLE C.2. Summary of Storage Conditions.

Matrix	Storage Temperature (°C)	Actual Storage Duration (months)	Interval of Demonstrated Storage Stability (months)
Oat Forage and Hay	-20 ± 5	4.8	4 (corn stover) [1] 6 (corn forage) [1]

[1] D330633, S. Hummel, 8/08/06

TABLE C.3. Residue Data from Crop Field Trials with Tribenuron Methyl (SG).

Trial ID (City, State; Year)	Zone	Type; Variety	Matrix	Total Rate (lb ai/A)	PHI (days)	Residues (ppm)[1]	
Glen Allen, VA 2006 1	2	Spring; 76-30	Forage	0.015	0	0.94	1.1
					7	Not sampled (NS)	NS
			Hay	0.016	33	ND[2]	ND
Sycamore, GA 2006 2	2	Winter; Nk-Coker 820	Forage	0.016	0	1.1	0.86
					7	0.01	(0.007)
			Hay	0.015	30	ND	ND
Richland, IA 2006 3	5	Spring; Jerry	Forage	0.015	0	0.66	0.84
					8	ND	ND
			Hay	0.016	30	ND	ND
Richland, IA 2006 4	5	Spring; Jerry	Forage	0.015	0	0.42	0.44
					8	ND	ND
			Hay	0.016	30	ND	ND
Delavan, WI 2006 5	5	Spring; Esker	Forage	0.015	0	0.28	0.22
					7	ND	ND
			Hay	0.016	25	ND	ND
Paynesville, MN 2006 6	5	Spring; Wabasha	Forage	0.015	0	ND[3]	ND[3]
					7	NS	NS
			Hay	0.015	30	0.57[3]	0.88[3]
Clarence, MO 2006 7	5	Spring; Jerry	Forage	0.016	0	1.0	0.82
					7	0.031	0.025
			Hay	0.016	7	ND	(0.003)
Northwood, ND 2006 8	5	Spring; Jerry	Forage	0.015	0	0.89	0.98
					7	(0.004)	(0.004)
			Hay	0.015	8	(0.004)	(0.003)
Fargo, ND 2006 9	5	Spring; Morton	Forage	0.016	0	1.5	1.3
					7	0.039	0.043
			Hay	0.016	30	ND	ND
York, NE 2006 10	5	Spring; Jerry	Forage	0.015	0	0.75	0.78
					7	ND	ND
			Hay	0.016	30	ND	ND
Britton, SD 2006 11	5	Spring; Reeves	Forage	0.016	-0[4]	ND	ND
					0	0.46	0.45
					3	ND	ND
					7	ND	ND
					14	ND	ND
					28	ND	ND
					45	ND	ND
			Hay	0.016	0	0.30	0.27
					3	0.026	0.027
					7	(0.006)	(0.005)
					14	ND	ND
					28	ND	ND
					45	ND	ND

TABLE C.3. Residue Data from Crop Field Trials with Tribenuron Methyl (SG).

Trial ID (City, State; Year)	Zone	Type; Variety	Matrix	Total Rate (lb ai/A)	PHI (days)	Residues (ppm)[1]	
East Bernard, TX 2006 12	6	Winter; Bob	Forage	0.016	0	0.90	0.78
					6	0.014	0.010
			Hay	0.016	9	(0.009)	0.010
Northwood, ND 2006 13	7	Spring; Jerry	Forage	0.015	-0[4]	ND	ND
					0	0.24	0.31
					3	0.17	0.16
					7	ND	ND
					14	ND	ND
					30	ND	ND
					44	ND	ND
			Hay	0.015	0	0.39	0.50
					3	0.019	0.017
					7	(0.006)	(0.007)
					14	ND	ND
					28	ND	ND
					42	ND	ND
Northwood, ND 2006 14	7	Spring; Jerry	Forage	0.015	0	0.39	0.52
					7	(0.004)	(0.005)
			Hay	0.015	5	0.013	0.028
Velva, ND 2006 15	7	Spring; Morton	Forage	0.016	0	0.86	1.0
					7	(0.004)	(0.005)
			Hay	0.016	30	ND	ND
Levelland, TX 2006 16	8	Winter; Walken	Forage	0.016	0	1.5	1.2
					7	0.017	0.014
			Hay	0.015	27	ND	ND
Clarence, MO 2006 17	5	Spring; Jerry	Forage	0.016	0	0.35	0.46
					5	0.01	0.01
			Hay	0.016	12	ND	ND
St. Joseph, MO 2006 18	5	Spring; Jerry	Forage	0.015	0	0.43	0.41
					7	ND	ND
			Hay	0.015	29	ND	ND

[1] The method LOQ and LOD are 0.01 and 0.003 ppm, respectively. Values <LOQ but ≥LOD are reported in parentheses.
[2] ND=non-detectable (<LOD).
[3] The residue data from the Paynesville, MN field trial were not used to calculate overall residues in forage and hay. Based on all the residue data from the other oat field trials, the forage (0 DAT) and hay sample in this trial appear to have been inadvertently switched.
[4] -0: Forage samples were collected immediately before the second application.

TABLE C.4.1. Summary of Residue Data from Oat Field Trials with Tribenuron Methyl (SG).

Commodity	Total Applic. Rate (lb ai/A)	PHI (days)	Residue Levels (ppm)[1]						
			n	Min.	Max.	HAFT[2]	Median (STMdR)	Mean (STMR)	Std. Dev.
Oat Forage	0.015-0.016	0	34[3]	0.22	1.50	1.40	0.780	0.739	0.355
		5-8	32	<0.01	0.043	0.041	0.010	0.014	0.009
Oat Hay	0.015-0.016	5-12	10	<0.01	0.028	0.021	0.010	0.012	0.006
		25-33	24[3]	<0.01	<0.01	<0.01	0.010	0.010	NA

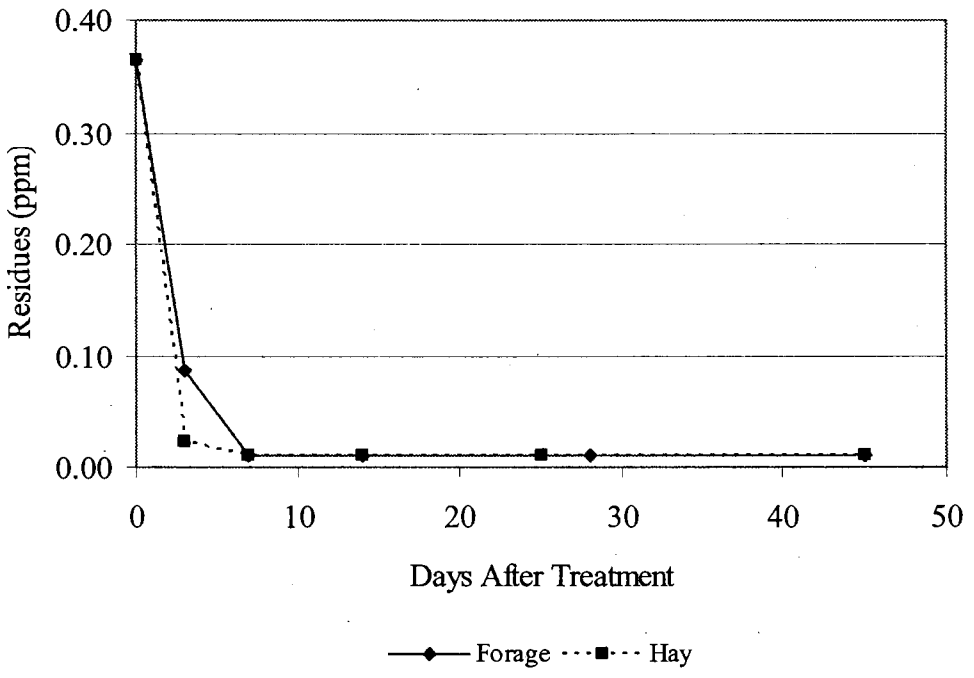
[1] The method LOQ is 0.01 ppm. For calculating the median, mean and standard deviation, the method LOQ was used for values <LOQ.
[2] HAFT = Highest Average Field Trial.
[3] Data do not include the samples from the Paynesville, MN field trial, where samples were apparently mislabeled.

TABLE C.4.2. Summary of Residue Decline Data from Oat Field Trials with Tribenuron Methyl (SG).

Commodity	Total Applic. Rate (lb ai/A)	PHI (days)	Residue Levels (ppm) [1]						
			n	Min.	Max.	HAFT [2]	Median (STMdR)	Mean (STMR)	Std. Dev.
Oat Forage	0.015-0.016	-0 [3]	4	<0.01	<0.01	<0.01	<0.01	<0.01	NA
		0	4	0.24	0.46	0.46	0.38	0.37	0.11
		3	4	<0.01	0.17	0.17	0.09	0.09	0.09
		7	4	<0.01	<0.01	<0.01	<0.01	<0.01	NA
		14	4	<0.01	<0.01	<0.01	<0.01	<0.01	NA
		28-30	4	<0.01	<0.01	<0.01	<0.01	<0.01	NA
		44-45	4	<0.01	<0.01	<0.01	<0.01	<0.01	NA
Oat Hay	0.015-0.016	0	4	0.27	0.50	0.45	0.35	0.37	0.10
		3	4	0.02	0.03	0.03	0.02	0.02	0.00
		7	4	<0.01	<0.01	<0.01	<0.01	<0.01	NA
		14	4	<0.01	<0.01	<0.01	<0.01	<0.01	NA
		28	4	<0.01	<0.01	<0.01	<0.01	<0.01	NA
		42-45	4	<0.01	<0.01	<0.01	<0.01	<0.01	NA

[1] The method LOQ is 0.01 ppm. For calculating the median, mean and standard deviation, the method LOQ was used for values <LOQ.
[2] HAFT = Highest Average Field Trial.
[3] Samples were collected immediately before the second application.

Figure C.1. Decline Data for Residues of Tribenuron Methyl in/on Oat Forage and Hay.



D. CONCLUSION

Although 10 of the 36 hay samples were not harvested at the targeted PHI, the oat field trial data are adequate and support the use of a single postemergence application of tribenuron methyl (SG) to oats up to flag leaf emergence at 0.0156 lb ai/A. The available residue data support minimum PHIs of 0 or 7 days for forage and 30 days for hay.

E. REFERENCES

D330633, Tribenuron Methyl. Addition of Preplant Burndown Uses on Field Corn, Rice, Sorghum, and Soybeans (PRIA R19; PP#4F6890) and Postemergence Uses on Sunflower (IR-4 Request; PP#4E6855). Summary of Analytical Chemistry and Residue Data., S. Hummel, 8/08/2006

F. DOCUMENT TRACKING

Petition Number: 7F7220
DP Number: 342085
PC Code: 128887

Tribenuron Methyl(DPX-L5300)/352-632/PC Code 128845/E.I. duPont de Nemours and Co./352
DACO 7.4.1/7.4.2/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3
Crop Field Trial/Residue Decline – Wheat forage and hay

Primary Evaluator D. Dotson — Date: 4/17/2008
Douglas Dotson, Ph.D., Chemist, RAB2

Peer Reviewer W T Drew — Date: 4/17/2008
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This DER was originally prepared under contract by Dynamac Corporation (1910 Sedwick Road, Building 100 Suite B, Durham NC 27713; submitted 10/28/2007). The DER has been reviewed by the Health Effects Division (HED) and revised to reflect current Office of Pesticide Programs (OPP) policies.

STUDY REPORT:

47138303 Thiel, A, Brookey, F. (2007) Determination of Decline and Magnitude of Residues of Tribenuron Methyl and Thifensulfuron Methyl in Wheat (Forage and Hay) Following One Application of DPX-L5300 50SG Herbicide and Two Applications of DPX-M6316 75WG Herbicide Under Field Conditions (U.S.A Seasons, 2005 and 2006): Lab Project Number: DuPont-17576. ABC Laboratories Study No. 50018. Unpublished study prepared by E. I. DuPont de Nemours and Company. 548 pages.

EXECUTIVE SUMMARY:

E. I. DuPont de Nemours and Company submitted field trial data for tribenuron methyl on wheat forage and hay. In 14 winter wheat field trials and 8 spring wheat field trials conducted during 2005-2006 in EPA Growing Zones 2, 4, 5, 6, 7, 8, and 11, a 50% soluble granular (SG) formulation of tribenuron methyl was applied to wheat as a single broadcast foliar application during vegetative development at 0.015-0.016 lb ai/A. Each field trial included separate treated plots for the harvest of forage and hay. The application was made between Zadoks Growth Stages 21 and 39 to the forage plots and between Zadoks Growth Stages 23 and 49 to the hay plots. All applications were made using ground equipment at rates of 12-25 gal/A, and included the use of a non-ionic surfactant (NIS).

Forage samples were collected from each site at 0 and 6-8 days after treatment (DAT), and hay samples were collected at 5 DAT from 3 sites, at 11-16 DAT from 4 sites, and at 25-35 DAT from 15 sites (the target PHI for hay was 30 days). Single control and duplicate treated samples were collected for both matrices. To assess residue decline, forage and hay samples were collected at approximately 0, 3, 7, 14, 28, and 45 DAT at three test sites. After harvest, hay samples were field dried for 0-8 days before collection. Forage and hay samples were stored at -20°C for up to 5.8 months prior to analysis. This interval is supported by the available storage stability data.

The LC/MS/MS method (DuPont Method 13412, Revision No. 1) used to determine residues of tribenuron methyl in/on wheat forage and hay was adequately validated in conjunction with the analysis of field trial samples. After the hay samples were hydrated, residues were extracted twice from forage and hay with acetonitrile:K_2HPO_4 buffer at pH 7 (75:25, v:v) and initially

cleaned up by partitioning against hexane. Aqueous soluble residues were further purified by elution through an ENV solid phase extraction (SPE) cartridge. Residues were then determined by LC/MS/MS using external standards and the m/z 396→155 ion transition for quantitation. The validated method limit of quantitation (LOQ) is 0.01 ppm, and the limit of detection (LOD) is 0.003 ppm.

Following a single broadcast foliar application of tribenuron methyl (SG) to wheat during vegetative development at 0.015-0.016 lb ai/A, residues in/on forage were <0.01-2.8 ppm in/on 44 samples harvested at 0 DAT and <0.01-0.25 ppm in/on 42 samples harvested at 6-8 DAT. Average residues in/on forage were 0.86 ppm at 0 DAT and 0.049 ppm by ~7 DAT. For hay, residues were <LOQ (<0.01 ppm) in/on all 14 samples collected at either 5 or 11-16 DAT, and residues were <0.01-0.42 ppm in/on the 30 samples harvested at 25-35 DAT, with 26 of these samples having residues <LOQ. Average residues in/on hay were <0.01 ppm at 5 and 11-16 DAT, and 0.029 ppm at 25-35 DAT. Data from the three residue decline tests indicate that residue levels in/on both forage and hay declined rapidly from 0 to 7 DAT, and then remained relatively steady from 14 to 45 DAT.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in the study, the wheat field trial data are classified as scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the U.S. EPA Residue Chemistry Summary Document, Memo, D342085, D. Dotson, 4/17/2008.

COMPLIANCE:

Signed and dated Good Laboratory Practice (GLP), Quality Assurance and Data Confidentiality statements were provided. No deviations from regulatory requirements were reported which would have an adverse impact on the validity of the study.

A. BACKGROUND INFORMATION

Tribenuron methyl is a sulfonylurea herbicide (Group 2) registered for postemergence application to barley, canola, cotton, flax, field corn, oats, soybeans, sunflowers, and wheat for selective control of broadleaf weeds. Permanent tolerances are established for tribenuron methyl *per se* in/on barley, canola, corn, cotton, flax, grass, oat, rice, sorghum, soybean, sunflower, and wheat commodities at levels ranging from 0.02 to 0.10 ppm [40 CFR §180.451(a) and (c)].

For the existing postemergence uses on barley, oats, and wheat, DuPont is proposing new PHIs for forage and hay along with tolerances on these commodities (PP#7F7220). The chemical structure and nomenclature and the physicochemical properties of tribenuron methyl are presented in Tables A.1 and A.2.

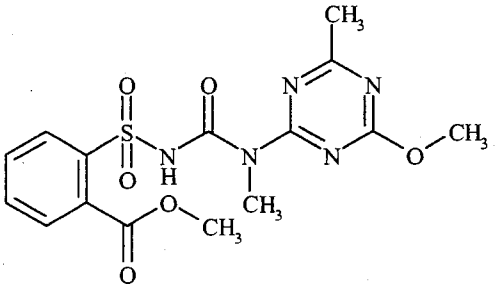
TABLE A.1. Tribenuron Methyl Nomenclature.	
Chemical structure	
Common name	Tribenuron methyl
Company experimental name	DPX-L5300
IUPAC name	methyl-2-[4-methoxy-6-methyl-1,3,5-triazin-2-yl(methyl)carbamoyl-sulfamoyl]benzoate
CAS name	methyl-2-[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)methylamino]carbonyl]amino]sulfonyl]benzoate
CAS registry number	101200-48-0
End-use product (EP)	50% SG (DuPont™ Express® Herbicide; EPA Reg. No. 352-632)

TABLE A.2. Physicochemical Properties of Tribenuron Methyl.		
Parameter	Value	Reference
Melting point/range	142°C	Provided in MRID
pH (20°C)	4.64	
Density (g/cm^3 at 19.6°C)	1.4594 ± 0.001	
Water solubility (g/L at 20°C)	pH 5 0.0489 pH 7 2.04 pH 9 18.3	
Solvent solubility (g/L at 20°C)	Acetone 39.1 Acetonitrile 46.4 Dichloromethane >250 Dimethylformamide 98.2 Ethyl acetate 16.3 n-Heptane 0.02 Methanol 2.59 n-Octanol 0.383 Xylene 13.1	
Vapor pressure (25°C)	2.7×10^{-7} mm Hg	
Dissociation constant (pK_a)	5.0	
Octanol/water partition coefficient Log(K_{OW}) at 20°C	pH 5 2.60 pH 7 0.78 pH 9 0.30	
UV/visible absorption (λ max)	pH 1.66 200, 231 nm pH 7 201, 256 nm pH 11.72 208, 256 nm	

B. EXPERIMENTAL DESIGN

B.1. Study Site Information

Twenty-two wheat field trials were conducted in Zones 2, 4, 5, 6, 7, 8, and 11 during 2005-2006 (Tables B.1.1 and B.1.3). Winter wheat was used in 14 trials, and spring wheat was used in the other 8 trials. Each trial consisted of a control and two treated plots, one for the harvest of forage and another for the harvest of hay. In each test, tribenuron methyl (50% SG) was applied to wheat as a single broadcast foliar application during vegetative development at 0.015-0.016 lb ai/A (Table B.1.2.). The application was made to the forage plots during Zadoks Growth Stages 21 through 39 and to the hay plots during Zadoks Growth Stages 23 through 49. All applications were made using ground equipment at rates of 12-25 gal/A, and included the use of a NIS adjuvant at 0.25-0.50% of the spray volume.

TABLE B.1.1. Trial Site Conditions.

Trial Identification (City, State; Year)	Soil characteristics [1]			
	Type	%OM	pH	CEC (meq/g)
Sycamore, GA 2006	Loamy Sand	NR	5.7	NR
Washington, LA 2006	Sandy Loam	1.2	5.8	NR
Richland, IA 2006	Silt Loam	3.2	6.2	NR
Paynesville, MN 2006	Sandy Loam	3.2	6.2	NR
Gardner, ND 2006	Silt Clay Loam	NR	7.4-8.4	NR
York, NE 2006	Silty Clay Loam	2.8	6.4	NR
East Bernard, TX 2005-2006	Sandy Clay	0.4	7.4	15.6
Frederick, SD 2005-2006	Loam	3.1	7.5	21.8
New Rockford, ND 2005-2006	Sand	2.7	7.1	NR
Velva, ND 2005-2006	Loam	4.0	7.6	27.2
New Rockford, ND 2005-2006	Loam	4-8	6.6-7.8	NR
Carrington, ND 2005-2006	Loam	3-5	6.1-7.3	NR
Clarence, MO 2005-2006	Silty Clay Loam	3.1	7.1	8.6
Uvalde, TX 2005-2006	Clay	2.8	7.7	NR
Hinton, OK 2005-2006	Sandy Loam	0.72	6	NR
Hart, TX 2005-2006	Loam	<1	8.0	NR
Groom, TX 2005-2006	Loam	1.9	7.1	NR
Levelland, TX 2005-2006	Sandy Loam	0.8	7.6	NR
Groom, TX 2005-2006	Loam	2.4	7.2	NR
Ephrata, WA 2005-2006	Loamy Sand	1.0	7.4	12.4
Oberon, ND 2005-2006	Loam	3-5	7.5	NR
St. Joseph, MO 2005-2006	Silt Loam	2.1	6.6	12.1

[1] These parameters are optional except in cases where their value affects the use pattern for the chemical.
NR= not reported

Average monthly minimum and maximum temperatures and monthly precipitation and irrigation were reported for each site during the test period, along with the 10-year monthly averages for temperatures (min and max) and precipitation. Weather conditions were within normal historical ranges except at the ND sites, where temperatures were up to 30% higher than the historical average. The tests were conducted according to normal agricultural practices for the different regions, and information was provided on maintenance pesticides and fertilizers used at each location.

TABLE B.1.2. Study Use Pattern.

Location (City, State; Year) Trial ID	End-use Product	Application Information					Tank Mix/ Adjuvants
		Method; Timing	Volume (gal/A)	Single Rate (lb ai/A)	RTI[1] (days)	Total Rate (lb ai/A)	
Sycamore, GA 2006 1	50% SG	One broadcast foliar application Zadoks Stage 29	22	0.015	--	0.015	0.50% NIS
		One broadcast foliar application, Zadoks Stage 39-40	25	0.015	--	0.015	0.51% NIS
Washington, LA 2006 2	50% SG	One broadcast foliar application, Zadoks Stage 30	19	0.016	--	0.016	0.25% NIS
		One broadcast foliar application, Zadoks Stage 40	19	0.016	--	0.016	0.25% NIS
Richland, IA 2006 3	50% SG	One broadcast foliar application, Zadoks Stage 34-35	16	0.015	--	0.015	0.25% NIS
		One broadcast foliar application, Zadoks Stage 39	16	0.016	--	0.016	0.24% NIS
Paynesville, MN 2006 4	50% SG	One broadcast foliar application, Zadoks Stage 39	15	0.015	--	0.015	0.26% NIS
		One broadcast foliar application, Zadoks Stage 40	15	0.015	--	0.015	0.27% NIS
Gardner, ND 2006 5	50% SG	One broadcast foliar application, Zadoks Stage 25	15	0.016	--	0.016	0.50% NIS
		One broadcast foliar application, Zadoks Stage 39	15	0.015	--	0.015	0.50% NIS
York, NE 2006 6	50% SG	One broadcast foliar application, BBCH 25	20	0.016	--	0.016	0.50% NIS
		One broadcast foliar application, BBCH 37	20	0.016	--	0.016	0.50% NIS
East Bernard, TX 2005-2006 7	50% SG	One broadcast foliar application, Zadoks Stage 30	25	0.015	--	0.015	0.50% NIS
		One broadcast foliar application, Zadoks Stage 39	25	0.016	--	0.016	0.50% NIS
Frederick, SD 2005-2006 8	50% SG	One broadcast foliar application, Zadoks Stage 30-31	12.5	0.016	--	0.016	0.50% NIS
		One broadcast foliar application, Zadoks Stage 33-34	12.5	0.016	--	0.016	0.50% NIS
New Rockford, ND 2005-2006 9	50% SG	One broadcast foliar application, Zadoks Stage 24	15	0.015	--	0.015	0.30% NIS
		One broadcast foliar application, Zadoks Stage 38	15	0.015	--	0.015	0.30% NIS
Velva, ND 2005-2006 10	50% SG	One broadcast foliar application, Zadoks Stage 30	12	0.015	--	0.015	0.25% NIS
		One broadcast foliar application, Zadoks Stage 37	12	0.015	--	0.015	0.25% NIS
New Rockford, ND 2005-2006 11	50% SG	One broadcast foliar application, Zadoks Stage 24	15	0.015	--	0.015	0.30% NIS
		One broadcast foliar application, Zadoks Stage 38	15	0.015	--	0.015	0.30% NIS
Carrington, ND 2005-2006 12	50% SG	One broadcast foliar application, Zadoks Stage 24	15	0.015	--	0.015	0.30% NIS
		One broadcast foliar application, Zadoks Stage 38	15	0.015	--	0.015	0.30% NIS
Clarence, MO 2005-2006 13	50% SG	One broadcast foliar application, Zadoks Stage 30	20	0.015	--	0.015	0.27% NIS
		One broadcast foliar application, Zadoks Stage 39	20	0.016	--	0.016	0.28% NIS

TABLE B.1.2. Study Use Pattern.

Location (City, State; Year) Trial ID	End-use Product	Application Information					Tank Mix/ Adjuvants
		Method; Timing	Volume (gal/A)	Single Rate (lb ai/A)	RTI[1] (days)	Total Rate (lb ai/A)	
Uvalde, TX 2005-2006 14	50% SG	One broadcast foliar application, Zadoks Stage 29	15	0.016	--	0.016	0.25% NIS
		One broadcast foliar application, Zadoks Stage 38	15	0.016	--	0.016	0.25% NIS
Hinton, OK 2005-2006 15	50% SG	One broadcast foliar application, Zadoks Stage 25	13	0.016	--	0.016	0.25% NIS
		One broadcast foliar application, Zadoks Stage 47-49	13	0.016	--	0.016	0.25% NIS
Hart, TX 2005-2006 16	50% SG	One broadcast foliar application, Zadoks Stage 30	18	0.016	--	0.016	0.25% NIS
		One broadcast foliar application, Zadoks Stage 39	18	0.016	--	0.016	0.25% NIS
Groom, TX 2005-2006 17	50% SG	One broadcast foliar application, Zadoks Stage 32	18	0.015	--	0.015	0.25% NIS
		One broadcast foliar application, Zadoks Stage 42	18	0.015	--	0.015	0.25% NIS
Levelland, TX 2005-2006 18	50% SG	One broadcast foliar application, Zadoks Stage 30	15	0.016	--	0.016	0.40% NIS
		One broadcast foliar application, Zadoks Stage 39-40	15	0.016	--	0.016	0.40% NIS
Groom, TX 2005-2006 19	50% SG	One broadcast foliar application, Zadoks Stage 32	18	0.016	--	0.016	0.25% NIS
		One broadcast foliar application, Zadoks Stage 42	18	0.016	--	0.016	0.25% NIS
Ephrata, WA 2005-2006 20	50% SG	One broadcast foliar application, Zadoks Stage 30	15	0.015	--	0.015	0.38% NIS
		One broadcast foliar application, Zadoks Stage 41	15	0.016	--	0.016	0.38% NIS
Oberon, ND 2005-2006 21	50% SG	One broadcast foliar application, Zadoks Stage 21	15	0.015	--	0.015	0.30% NIS
		One broadcast foliar application, Zadoks Stage 23	15	0.015	--	0.015	0.30% NIS
St. Joseph, MO 2005-2006 22	50% SG	One broadcast foliar application, Zadoks Stage 27-31	14	0.016	--	0.016	0.25% NIS
		One broadcast folia application, Zadoks Stage 37	14	0.015	--	0.015	0.25% NIS

[1] RTI = Retreatment Interval.

TABLE B.1.3. Trial Numbers and Geographical Locations.

NAFTA Growing Zones	Wheat		
	Submitted	Requested	
		Canada	U.S.
1	--	--	--
1A	--	--	--
2	1	--	1
3	--	--	--
4	1	--	1
5	6	2	5
5A	--	--	--
5B	--	--	--
6	2	--	1
7	6	7	5
7A	--	1	
8	5	--	6
9	--	--	--
10	--	--	--
11	1	--	1
12	--	--	--
13	--	--	--
14	--	10	--
Total	22	20	20

B.2. Sample Handling and Preparation

Single control and duplicate treated samples of wheat forage and hay were harvested from each test site mechanically or by hand. Forage samples (≥1 kg/sample) were collected from each site at 0 and 6-8 DAT. At three sites, forage samples were collected immediately before the second application (-0 DAT) and at 0, 3, 7-8, 14-15, 28-29, and 45-47 DAT to assess residue decline. Harvest intervals for hay were 5 DAT at 3 field sites, 11-16 DAT at 4 field sites, and 25-35 DAT at the remaining 15 field sites. Hay samples (≥0.5 kg/sample) were generally cut between Zadoks Growth Stages 50 and 70, except at a few sites where hay samples were collected earlier (Zadoks Stage 43), apparently because of weather conditions. Sample sizes at these sites were also lower (0.3 kg/sample). To examine residue decline, hay samples from three sites were harvested at 0, 3-6, 7-9, 13-14, 28-35, and 45-48 DAT. Hay samples were field-dried after harvest for 0-8 days prior to collection. Both forage and hay samples were placed in frozen storage within 1-6 hours of collection. Samples were stored frozen at the field sites for 0-87 days prior to frozen shipment by overnight courier or by ACDS freezer truck to the analytical laboratory, Morse Laboratories, Inc, Sacramento, CA. At the analytical laboratory, samples were ground with dry ice and stored at -20 ± 5°C until extraction for analysis.

B.3. Analytical Methodology

Forage and hay samples were analyzed for residues of tribenuron methyl using an LC/MS/MS method (DuPont Method 13412, Revision No. 1). DuPont Method 13412 was previously validated for the analysis of tribenuron methyl in oily crop matrices and cereal grains (Memo, D330633 and 46352003.de2, S. Hummel, 8/8/2006). The method has undergone a successful

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independent laboratory trial, and is also capable of determining other sulfonylurea herbicides (ethametsulfuron methyl, rimsulfuron, thifensulfuron methyl, and chlorimuron ethyl). The modifications made to the method for the analysis of residues in forage and hay included: (1) addition of a hydration step for dry matrices; (2) replacing the second centrifugation step with filtration; and (3) modification of the hexane partition step for hay to include extra cleanup with BONDESIL-SAX.

Hay samples were initially hydrated for 5 minutes in 20 mM K_2HPO_4 buffer (pH 7) and then soaked for 30 minutes in the extraction solvent. Residues were extracted twice from forage and hay by homogenization with acetonitrile:K_2HPO_4 buffer at pH 7 (75:25, v:v), and the extracts were centrifuged and filtered. For forage, the aqueous extract was partitioned directly with hexane, discarding the hexane phase. For hay samples, the aqueous extract was acidified with glacial acetic acid, mixed with 1g of BONDESIL-SAX, partitioned with hexane, and centrifuged after mixing. The hexane fraction was discarded, and the aqueous fraction was neutralized with concentrated NaOH. Residues from both forage and hay samples were then cleaned up using an ENV SPE cartridge. After residues were loaded onto the ENV SPE cartridge, the cartridge was washed with hexane, and residues were eluted with 25 mM NH_4OH:methanol (1:99, v:v), mixed with 5 mM ammonium acetate, and concentrated to remove the methanol. Residues were redissolved in acetonitrile:50 mM ammonium acetate (1:9, v:v) and analyzed by LC/MS/MS. The HPLC system utilized a phenyl-hexyl column with a mobile phase gradient of water to methanol, each containing 0.05% formic acid, and a MS/MS detector. Residues were quantified using the m/z 396→155 transition and confirmed using the m/z 396→181 transition. External standards were used for quantitation. The method has a validated LOQ of 0.01 ppm and a reported LOD of 0.003 ppm.

The above method was validated in conjunction with the analysis of field trial samples, using control samples of wheat forage and hay fortified with tribenuron methyl at 0.01-10.0 ppm.

C. RESULTS AND DISCUSSION

Wheat forage and hay samples were stored at -20 ± 5°C for up to 5.8 months prior to extraction for analysis. Storage stability data are available indicating that tribenuron methyl is stable in frozen storage for up to 4 months in corn stover, 6 months in corn forage, and 21 months in wheat straw (Memos, D330633, S. Hummel, 8/8/2006, and D304059, R. Griffin, 6/24/04). These data will adequately support the storage intervals and conditions for forage and hay from the current oat field trials.

The LC/MS/MS method (Method 13412, Rev. 1) used for determining residues of tribenuron methyl in/on wheat forage and hay was adequately validated in conjunction with the analysis of field trial samples. The average concurrent recovery of tribenuron methyl (± the standard deviation) was 84 ± 8% from forage and 80 ± 9% from hay. The validated method LOQ for tribenuron methyl is 0.01 ppm and the LOD is 0.003 ppm in both matrices. Apparent residues of tribenuron methyl were <LOD in/on control samples of forage and hay, with the exception of one control sample of hay which had apparent residues of 0.004 ppm. Adequate sample

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calculations and example chromatograms were provided, and the fortification levels used for the concurrent recoveries bracketed the residue levels observed in treated samples.

Following a single broadcast foliar application of tribenuron methyl to wheat during vegetative development, residues in/on forage were <0.01-2.8 ppm in/on samples harvested at 0 DAT and <0.01-0.25 ppm in/on 42 samples harvested at 6-8 DAT (Table C.3). At the 6-8 DAT harvest interval, 16 of the 42 samples had residues <LOQ. Average residues in/on forage were 0.86 ppm at 0 DAT and 0.049 ppm by ~7 DAT (Table C.4.1).

For hay, most samples (30) were harvested at 25-35 DAT, around the targeted 30-day PHI; however, 6 hay samples were collected at 5 DAT, and 8 hay samples were collected at 11-16 DAT. Residues in/on hay were <LOQ (<0.01 ppm) in/on all samples collected at either 5 or 11-16 DAT. Residues were <0.01-0.42 ppm in/on the 30 samples harvested at 25-35 DAT, with 26 of these samples having residues <LOQ. Average residues in/on hay were 0.01 ppm at 5 and 11-16 DAT, and 0.029 ppm at 25-35 DAT.

In the three residue decline tests, residue levels in/on both forage and hay declined rapidly from 0 to 7 DAT, and then remained relatively steady from 7 to 45 DAT (Figure C.1). In forage, average residues declined from 0.623 ppm at 0 DAT to 0.085 ppm at ~7 DAT, and were 0.067-0.83 ppm from 14 to 45 DAT (Table C.4.2). In hay, average residues declined from 0.975 ppm at 0 DAT to 0.044 ppm at ~7 DAT, and were 0.082-0.117 ppm from 14 to 45 DAT.

Common cultural practices were used to maintain plants, and the weather conditions, maintenance chemicals, and fertilizer used in the study did not have a notable impact on the residue data.

TABLE C.1. Summary of Concurrent Recoveries of Tribenuron Methyl from Wheat Forage and Hay.

Matrix	Spike Level (ppm)	Sample Size (n)	Recoveries (%)	Mean ± Std. Dev. (%)
Wheat Forage	0.01	20	89, 81, 75, 82, 84, 76, 85, 95, 93, 80, 101, 66, 100, 71, 86, 72, 74, 76, 86, 86	83 ± 10
	0.05	4	94, 90, 74, 81	85 ± 9
	0.10	1	75	75
	0.50	4	87, 78, 85, 85	84 ± 4
	1.0	4	87, 87, 82, 74	83 ± 6
	2.0	3	82, 93, 80	85 ± 7
	5.0	5	92, 82, 91, 89, 78	86 ± 6
	10.0	1	87	87
	Total	**42**	**66-101**	**84 ± 8**
Wheat Hay	0.01	18	73, 71, 81, 77, 99, 80, 87, 70, 82, 84, 98, 88, 89, 74, 76, 74, 78, 87	82 ± 9
	0.05	12	70, 68, 70, 78, 89, 64, 85, 84, 85, 71, 70, 76	76 ± 9
	0.20	1	75	75
	2.0	1	93	93
	5.0	3	84, 96, 87	89 ± 6
	10.0	1	79	79
	Total	**36**	**64-99**	**80 ± 9**

[1] Values not included in mean calculations.

TABLE C.2. Summary of Storage Conditions.

Matrix	Storage Temperature (°C)	Actual Storage Duration (months)	Interval of Demonstrated Storage Stability (months)
Wheat Forage and Hay	-20 ± 5	5.8	4 (corn stover) [1] 6 (corn forage) [1] 21 (wheat straw) [2]

[1] D330633, 8/08/06, S. Hummel.
[2] D304059, 6/24/04, R. Griffin.

TABLE C.3. Residue Data from Crop Field Trials with Tribenuron Methyl.

Trial ID (City, State; Year)	Zone	Type; Variety	Matrix	Total Rate (lb ai/A)	PHI (days)	Residues (ppm)[1]	
Sycamore, GA 2006 1	2	Winter; Pioneer 26R61	Forage	0.015	0	0.87	1.0
					7	(0.006)	(0.005)
			Hay	0.015	30	ND[2]	ND
Washington, LA 2006 2	4	Winter; AGS 2000	Forage	0.016	0	0.52	0.60
					7	0.086	0.074
			Hay	0.016	28	ND	ND
Richland, IA 2006 3	5	Winter; Roane	Forage	0.015	0	0.55	0.54
					7	0.017	0.014
			Hay	0.016	30	ND	ND
Paynesville, MN 2006 4	5	Spring; Oxen	Forage	0.015	-0[3]	ND	ND
					0	ND	ND
					3	0.26	0.24
					7	(0.004)	(0.007)
					14	0.064	0.058
					28	0.19	0.17
					45	0.22	0.24
			Hay	0.015	0	1.3	0.88
					3	0.82	0.44
					7	0.096	0.13
					14	0.26	0.19
					28	0.16	0.42
					45	0.41	0.25
Gardner, ND 2006 5	5	Spring; Knudson	Forage	0.016	0	1.3	1.4
					7	0.070	0.046
			Hay	0.015	30	ND	ND
York, NE 2006 6	5	Winter; Wahoo HRW	Forage	0.016	0	0.86	0.72
					8	(0.008)	(0.007)
			Hay	0.016	30	ND	ND
East Bernard, TX 2005-2006 7	6	Winter; Ranger 30127	Forage	0.015	0	0.98	0.91
					6	0.051	0.046
			Hay	0.016	11	ND	ND
Frederick, SD 2005-2006 8	7	Spring; Hard Red Spring	Forage	0.016	0	0.46	0.44
					7	0.013	0.013
			Hay	0.016	30	ND	ND
New Rockford, ND 2005-2006 9	7	Spring; Alsen	Forage	0.015	0	0.35	0.39
					7	ND	ND
			Hay	0.015	5	(0.004)	(0.004)
Velva, ND 2005-2006 10	7	Spring; Alsen	Forage	0.015	0	0.80	0.72
					7	ND	ND
			Hay	0.015	30	ND	ND

TABLE C.3. Residue Data from Crop Field Trials with Tribenuron Methyl.

Trial ID (City, State; Year)	Zone	Type; Variety	Matrix	Total Rate (lb ai/A)	PHI (days)	Residues (ppm)[1]	
New Rockford, ND 2005-2006 11	7	Spring; Alsen	Forage	0.015	0	0.47	0.49
					7	(0.004)	(0.005)
			Hay	0.015	5	(0.009)	(0.007)
Carrington, ND 2005-2006 12	7	Spring; Alsen	Forage	0.015	0	0.48	0.54
					7	ND	(0.003)
			Hay	0.015	5	ND	ND
Clarence, MO 2005-2006 13	5	Winter; Pioneer 25R78	Forage	0.015	0	0.90	0.84
					8	0.074	0.077
			Hay	0.016	15	(0.006)	(0.005)
Uvalde, TX 2005-2006 14	6	Winter; Ogallala	Forage	0.016	0	0.70	0.74
					6	0.25	0.23
			Hay	0.016	30	0.015	0.01
Hinton, OK 2005-2006 15	8	Winter; Jagger	Forage	0.016	0	2.6	2.6
			Hay	0.016	25	(0.005)	(0.005)
Hart, TX 2005-2006 16	8	Winter; TAM 200	Forage	0.016	0	2.8	2.6
					7	(0.005)	(0.005)
			Hay	0.016	30	ND	ND
Groom, TX 2005-2006 17	8	Winter; TAM 200	Forage	0.015	0	0.46	0.44
					7	0.15	0.15
			Hay	0.015	14	ND	ND
Levelland, TX 2005-2006 18	8	Winter; TAM 110	Forage	0.016	0	0.81	0.96
					7	(0.007)	(0.007)
			Hay	0.016	30	ND	(0.004)
Groom, TX 2005-2006 19	8	Winter; TAM 110	Forage	0.016	-0[3]	ND	ND
					0	0.43	0.39
					3	0.23	0.26
					7	0.25	0.22
					14	0.15	0.15
					28	ND	ND
					45	ND	ND
			Hay	0.016	0	0.83	0.89
					3	0.13	0.15
					7	(0.004)	(0.004)
					14	(0.004)	(0.003)
					28	ND	ND
					45	ND	ND
Ephrata, WA 2005-2006 20	11	Winter; Stephens	Forage	0.015	0	0.36	0.35
					7	(0.008)	0.011
			Hay	0.016	30	ND	ND
Oberon, ND 2005-2006 21	7	Spring; Granite	Forage	0.015	0	0.63	0.72
					7	(0.005)	(0.005)
			Hay	0.015	16	ND	ND

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TABLE C.3. Residue Data from Crop Field Trials with Tribenuron Methyl.

Trial ID (City, State; Year)	Zone	Type; Variety	Matrix	Total Rate (lb ai/A)	PHI (days)	Residues (ppm)[1]	
St. Joseph, MO 2005-2006 22	5	Winter; Onaga	Forage	0.016	-0[3]	ND	ND
					0	1.2	1.7
					3	0.074	0.097
					8	ND	ND
					15	ND	ND
					28	ND	ND
					47	ND	ND
			Hay	0.015	6	ND	ND
					9	ND	ND
					13	ND	ND
					22	ND	ND
					35	ND	ND
					48	ND	ND

[1] The method LOQ and LOD are 0.01 and 0.003 ppm, respectively. Values <LOQ but ≥LOD are reported in parentheses.
[2] ND=non-detectable (<LOD).
[3] -0: Forage samples were collected immediately before the second application.

TABLE C.4.1. Summary of Residue Data from Wheat Field Trials with Tribenuron Methyl.

Commodity	Total Applic. Rate (lb ai/A)	PHI (days)	Residue Levels (ppm)[1]						
			n	Min.	Max.	HAFT[2]	Median (STMdR)	Mean (STMR)	Std. Dev.
Wheat Forage	0.015-0.016	0	44	<0.01	2.80	2.60	0.71	0.855	0.661
		6-8	42	<0.01	0.25	0.24	0.01	0.049	0.071
Wheat Hay	0.015-0.016	5	6	<0.01	<0.01	<0.01	0.01	0.01	NA
		11-16	8	<0.01	<0.01	<0.01	0.01	0.01	NA
		25-35	30	<0.01	0.42	0.29	0.01	0.029	0.079

[1] The method LOQ is 0.01 ppm. For calculating the median, mean and standard deviation, the method LOQ was used for values <LOQ.
[2] HAFT = Highest Average Field Trial.

TABLE C.4.2. Summary of Residue Decline Data from Wheat Field Trials with Tribenuron Methyl.

Commodity	Total Applic. Rate (lb ai/A)	PHI (days)	Residue Levels (ppm)[1]						
			n	Min.	Max.	HAFT[2]	Median (STMdR)	Mean (STMR)	Std. Dev.
Wheat Forage	0.015-0.016	-0[3]	6	<0.01	<0.01	0.01	0.010	0.010	NA
		0	6	<0.01	1.70	1.45	0.410	0.623	0.684
		3	6	0.074	0.26	0.250	0.235	0.194	0.085
		7-8	6	<0.01	0.25	0.235	0.010	0.085	0.117
		14-15	6	<0.01	0.15	0.150	0.061	0.074	0.063
		28-29	6	<0.01	0.19	0.180	0.010	0.067	0.088
		45	6	<0.01	0.24	0.230	0.010	0.083	0.114
Wheat Hay	0.015-0.016	0	4	0.83	1.30	1.09	0.885	0.975	0.218
		3-6	6	<0.01	0.82	0.630	0.14	0.260	0.316
		7-9	6	<0.01	0.13	0.113	0.010	0.044	0.054
		13-14	6	<0.01	0.26	0.225	0.010	0.082	0.113
		22-28	6	<0.01	0.42	0.290	0.010	0.103	0.166
		45-48	6	<0.01	0.41	0.330	0.010	0.117	0.173

[1] The method LOQ is 0.01 ppm. For calculating the median, mean and standard deviation, the method LOQ was used for values <LOQ.
[2] HAFT = Highest Average Field Trial.
[3] Samples were collected immediately before the second application.

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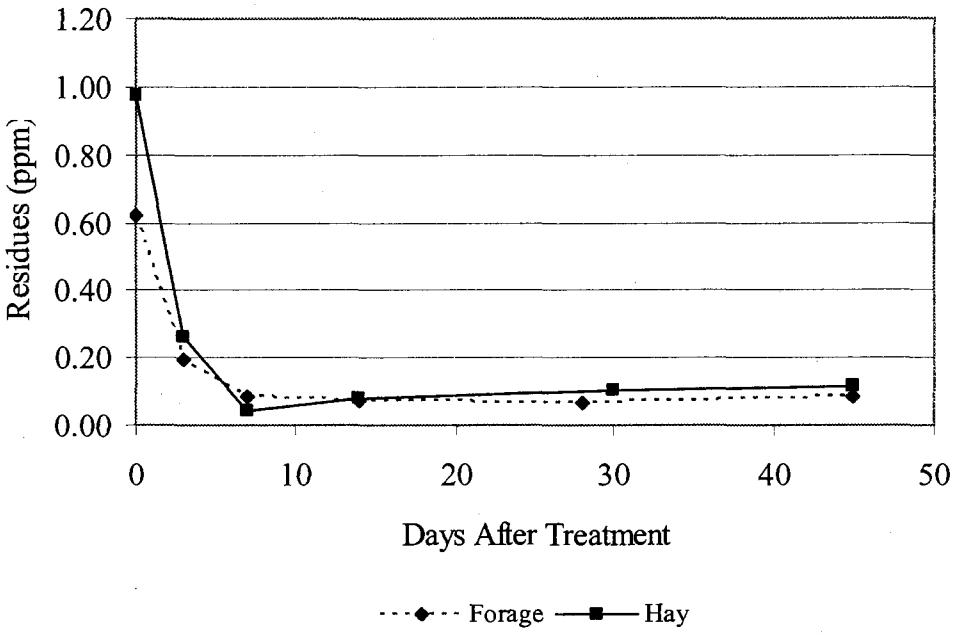
Figure C.1. Residue Decline Data for Tribenuron Methyl in/on Wheat Forage and Hay.



D. CONCLUSION

Although 14 of the 44 hay samples were not harvested at the targeted PHI, the wheat field trial data are adequate and support the use of a postemergence application of tribenuron methyl (SG) to wheat prior to flag leaf emergence at up to 0.0156 lb ai/A. The available residue data support minimum PHIs of 0 or 7 days for forage and 30 days for hay.

E. REFERENCES

D304059, Tribenuron Methyl. Residue Chemistry Considerations., R. Griffin, 6/24/2004.

D330633, Tribenuron Methyl. Addition of Preplant Burndown Uses on Field Corn, Rice, Sorghum, and Soybeans (PRIA R19; PP#4F6890) and Postemergence Uses on Sunflower (IR-4 Request; PP#4E6855). Summary of Analytical Chemistry and Residue Data., S. Hummel, 8/08/2006.

F. DOCUMENT TRACKING

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DP Number: 342085
PC Code: 128887

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R158880

Chemical: Tribenuron

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128887

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DPD304059
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DPD266130
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