

TEXT SEARCHABLE DOCUMENT

Data Evaluation Report on the aquatic field dissipation of penoxsulam

PMRA Submission Number {.....}

EPA MRID Number 46930302

Data Requirement: PMRA Data Code:
EPA DP Barcode: D333302
OECD Data Point:
EPA Guideline: Non-Guideline Composting Study

Test material: Penoxsulam **Concentration of a.i.:** not provided

End Use Product name: not provided **Formulation type:** not provided

Test material:

Common name: Penoxsulam.

Chemical name:

IUPAC name: 3-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)- α,α,α -trifluorotoluene-sulfonamide.
2-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide.
6-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy-s-triazolo[1,5-c]pyrimidin-2-yl)- α,α,α -trifluoro-o-toluenesulfonamide.

CAS name: 2-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide.

CAS No.: 219714-96-2.


Synonyms: XDE-638; DE-638; TSN101649; SP1019 (SePRO).

Smiles string: FC(c1cccc(c1S(=O)(=O)N(c1nn2c(n1)ccnc2))OCC(F)F)(F)F
(ISIS v2.3/Universal SMILES).
No EPI Suite, v3.12 SMILES String found as of 6/27/06.
n1c(nc2n1c(ncc2OC)OC)NS(=O)(=O)c3c(ccc3C(F)(F)F)OCC(F)F

Reviewer: Lucy Shanaman
EPA Reviewer

Signature: 
Date: May 8, 2007

Peer Reviewer: James Hetrick, Ph.D.
EPA Reviewer

Signature: 
Date: 5/8/07

Company Code

Active Code

Use Site Category

EPA PC Code: 119031

CITATION: Roberts, D. W., Brinton, W.F., Evans, W. F. Penoxsulam in Turfgrass, Compost and Compost/Peat Growth Media: Environmental Fate and Non-Target Plant Effects. Unpublished study performed by Regulatory Laboratories – Indianapolis Lab, Dow AgroSciences LLC, 9330 Zionsville Road, Indianapolis, Indiana 46268-1054. Laboratory Study



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EPA MRID Number 46703502

AgroSciences LLC, 9330 Zionsville Road, Indianapolis, Indiana 46268-1054. Laboratory Study ID: 050015. Experiment initiation March 24, 2005 and completion July 14, 2006 (p. 4). Final report issued July 14, 2006.

EXECUTIVE SUMMARY

Penoxsulam (3-(2,2-difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)- α,α,α -trifluorotoluene-2-sulfonamide) was applied once by either fertilizer-granular or soluble concentrate formulations at 0.022 or 0.067 lbs. (a.i.)/acre to mature Kentucky bluegrass (*Poa pratensis*) in Pennsylvania. Turfgrass clippings were collected 21 days post treatment to determine the rate of decline of penoxsulam in turfgrass. The two formulations were sampled at 5 days post treatment. These turf clippings were mixed with deciduous leaves to generate a typical yard waste mixture, and composted at an average temperature of about 40° C. In the low fertilizer-granular treatment rate compost, fertilizer-granular penoxsulam residues declined from 3,063 ppb to 4.63 ppb over 21 days. In the high rate fertilizer-granular treatment formulation compost, penoxsulam declined from 3,814 ppb to 7.92 ppb. For the low rate soluble concentrate formulation compost, fertilizer-granular penoxsulam residues declined from 14,974 ppb to 6.99 ppb over 21 days. For the high rate soluble concentrate formulation compost, penoxsulam declined from 30,434 ppb to 20.47 ppb, over 21 days. For all treatments, penoxsulam declined on the leaf blade portion of growing turf grass with a first-order foliar dissipation half-life of 13 to 14 days. The main route of dissipation in turf appears to be associated with foliar wash-off

Turfgrass clippings from the penoxsulam fertilizer-granular treatment formulation and the soluble concentrate were utilized for the compost dissipation study. Over 160 days of composting penoxsulam, dissipation was first-order with a dissipation half-life of 39 to 42 days. Small differences in observed dissipation rates can be attributed to experimental design. Initially, small, bench size composting chambers were used. Later in the experiment, larger 120 liter units were used.

Carbon to nitrogen ratios (C:N) varied between 15 and 42, indicating an acceptable level of microbial activity. After 90 days, the composts were mixed with loam soil at volumes of 12% and 33% to test adverse effects by measuring fresh plant weight of sugar beets, onions, snap beans, tomatoes, petunias and dwarf sunflowers at 28 day post planting. The bioassay data indicated the presence of adverse effects for sugar beets, the most sensitive species tested. Additionally, onion weight was at 91% of the control weight at one sampling interval, and sugar beets were at an average of 88.4% of the controls at two sampling intervals. Sugar beets also displayed a reduced percent of germination compared to the control at some test concentrations.

Study Acceptability: This non-guideline study is classified supplemental. While there were no significant deviations from good scientific practices, tabulated data for the bioassay studies would have assisted in analyzing the extent of adverse effects to sugar beets.

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Application of the liquid formulation was made using a CO₂ powered boom sprayer equipped with 8003 flat fan nozzles at a pressure of 30 psi and a total spray volume of 407 liters/hectare.

The control sample was collected by mowing at a 5 cm height, and transferring the clippings to plastic buckets for use in feedstock, before penoxsulam was applied. The control sample was shipped the same day, at ambient temperatures, to Woods End Research Lab. All samples that were used to test for penoxsulam residues were frozen until shipment to Carbon Dynamics Institute LLC.

Test plots were mowed at a 5 cm height, and the clippings collected for sampling in paper catch bags. The mower was cleaned of clippings, rinsed and sprayed with Nutri-Sol spray system neutralizer (to neutralize any traces of pesticides) then rinsed again and allowed to dry between collections. A new paper catch bags were used for each collection.

Day 0 samples were collected about 6 hours after application¹ in order to allow time for the herbicide to dry on the leaf surfaces. Between day 0 and day 3, 2.5 cm of irrigation was applied.

Sample Handling and Analysis

After sample collection, a fresh weight mass was obtained in a field laboratory. One 50 gram sample was collected in a zip lock bag and held under frozen storage until residue analysis. A second gram sample was collected, and dried at 65° C for 72 hours. Frozen samples were packed in dry ice, and shipped to Carbon Dynamics Institute LLC for residue analysis. Penoxsulam concentration was converted to dry weight concentration, and the recovery was then calculated on a percentage basis of concentration. The limit of detection was 0.30 µg/kg (ppb). The limit of quantification was 1.00 µg/kg (ppb).

Composting and Bioassay Phases

Compost was prepared by blending fresh clippings from the day 5 sampling of the field phase with dry leaves, from local municipal collection.

Preliminary Bioassays on Ingredients

A preliminary analysis to check for background contamination was conducted with untreated grass clippings and fresh leaves. Corn, soybeans and sugar beets were grown for 14 days in Fafard® Germination Mix amended with either grass or leaves at rates of 10% and 30%, by volume. Adverse effects to some component of the control test system were reported for sugar beets in the form of an unexplained high variability in beet growth. Slight, but not statistically significant variations in seedling growth were also seen for corn and soybeans.

¹ Product labels do not recommend that penoxsulam be watering in after application.

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Bench Scale Composting

While the study was designed to achieve a starting target C:N ratio of 30:1, the actual ratios normally varied between 25:1 and 40:1. A target rate for percent of grass clipping in the starting compost mixture was established at not more than 28% volume, or not more than 19% total dry matter. Five liter batches were mixed on a weight basis, and then adjusted to 65% moisture by the addition of water. Preliminary laboratory analysis was used to determine consistency of the starting mixtures. The compost was allowed to heat in the 5 liter, super insulated Dewar flasks through microbial action. Oxygen demand was monitored by periodically inserting an O₂ sensing wand into the center of the composting material. Oxygen content varied between 3% and 17% during the study. "Turning events" were simulated by dumping the contents of the Dewar, stirring with a large spoon, and returning the mixture to the Dewar. Data-loggers were placed in the center of the compost in the Dewar flasks in order to measure temperature. After composting had begun, the temperature of the composting remained over 45° C for the first 60 days, until the temperature in all test vessels reached the ambient range after 125 days. Some statistically insignificant variation was observed between treatment, and within treatments, and was attributed to variations in microbial populations. Compost pH values were initial acidic, rose steadily through the composting process to as high as 8.0, then gradually dropped to pH 7 as the temperature dropped.

Residue Analysis Phase

Samples of turfgrass clippings and compost were analyzed using a DAS method, GRM 01.25, high performance liquid chromatography in tandem with mass spectroscopy. The analytical method protocol used for the grass clippings was modified for the compost analysis through external calibration using linear regression. The limit of detection for this new method was 0.050 µg/kg.

RESULTS AND DISCUSSION

Decline in Turfgrass

Weekly clipping yield peaked at one week after treatment, and remained constant for the remainder of the six week study duration. Penoxsulam residues in turf grass clippings averaged 51,000 µg/kg on day 0, dropped to 3480 µg/kg by 0.4 weeks after treatment and continued to decline until residues were no longer detected at 6 weeks after treatment. Irrigation at 0.4 weeks after application may have washed residues from the turf grass leaves. The first order penoxsulam foliar dissipation half-life in the clippings collected during the field portion of this study was 2.3 days. Wash off of penoxsulam appears to be a significant contributing factor in the reported dissipation half-life for grass clippings. The total percent recovery of penoxsulam from turf grass for the total six week portion of the filed study duration was 12.2%, with 88% recovered on the day of application.

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minimum of nine 4.5 liter vessels were maintained for the duration of the study. Fifty grams were removed at each sampling interval, frozen and shipped by overnight service to Carbon Dynamics Institute LLC, in Springfield, IL for analysis. Parallel samples were tested at Woods End Research Lab. Analysis of variance, regression and LDS data analysis was performed using the STAT Data Analysis Program by G. Periman (1980 Data Analysis Programs for the UNIX Operating System, UCSD).

Utilization and Bioassay

Compost resulting from clippings treated at two treatment levels with penoxsulam was collected at days 95, 123 and 160. The samples were analyzed for penoxsulam residues and evaluated for effects plants using short-term seeding bioassays at two different application rates. Test species include: sugar beets, onion, tomato, bush snap beans, dwarf sunflower and petunias. The loam soil mixed with the compost, and used for controls for these tests was obtained from Seasons Downeast Landscaping, Camden, Maine. A bioassay with the 160 day compost was conducted with the most sensitive species, sugar beets and onions only. All test cultivars were tested with day 90 and day 123 compost.

Quantitative Residue Analysis Phase

Samples of turfgrass clippings and compost were analyzed by Carbon Dynamics Institute, LLC using a DAS method, GRM 01.25, high performance liquid chromatography in tandem with mass spectroscopy, Positive Electrospray Ionization (ESI⁺). Individual recovery samples were between 77 and 123% with a standard deviation of 15.4%. The limit of detection was 0.059 µg/kg.

RESULTS AND DISCUSSION

Decline in Turfgrass

Mean clipping yield peaked at day 21, and moisture was adequate throughout the sampling period. Penoxsulam residues in turf grass clippings (dry weight) ranged from 3,063 to 30,434 µg/kg on day 0. While by day 3 penoxsulam in all samples were below 8.2% of the treatment rate, this could have been due to combination of rainfall and irrigation. The first order penoxsulam foliar dissipation half-life in the clippings collected during the field portion of this study was 13 to 14 days. Recovery of penoxsulam from turf treated with the liquid formulation ranged from 20% to 12% through day 21. Recovery of the granular penoxsulam formulation was less than 2%, and might be attributed to placement of the granular formulation in the thatch layer of the turf. The main route of dissipation in turf appears to be associated with foliar wash-off.

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Seeding Bioassay Results

Short term seed growth was studied with sugar beets for all composted samples, with corn and soybeans were also used for the composting day 0 and day 7 samples. The effects of 10% treated and untreated compost were studied in 3 or 4 replicates over 21 days. Plants were kept at a 16 hour/day cycle under fluorescent lighting, and maintained at 24° to 26° C. No observed statistical differences between treated and untreated samples of corn and soy beans could be detected.

With the exception of the day 234 sample, the bioassays for the sugar beets were significantly smaller than the control samples at all sampling intervals. Two days after harvest, onions and curly cress were planted in the used test media. Germination and the first 12 days growth of the onions and curly cress were no statistically significant different than in the control media.

The day 234 samples were bioassayed with dwarf sunflowers and sugar beets with 33% treated compost in parallel with the 10% treated samples. The dwarf sunflowers were not affected by the treated compost, while the sugar beet was inhibited even more strongly by the 33% treated compost than the other test samples.

Results of Quantitative Analysis During Composting

Samples were sent to Carbon Dynamics Institute LLC, in Springfield, IL for penoxsulam analysis by Carbon Dynamics Institute, LLC using a DAS method, GRM 01.25, high performance liquid chromatography in tandem with mass spectroscopy, Positive Electrospray Ionization (ESI⁺). Parallel testing was also done at Woods End Research Lab for total solids by drying at 105°C, and total volatile solids by ignition at 550° C.

Penoxsulam concentrations were initially measured at 150 µg/kg, and declined to 1.6 ppb after 9 months of composting. The 150 µg/kg value represents compost made from 100% treated clippings treated at the maximum application rate.

Discussion of Bioassay Results

Sugar beet growth appears to correlate with penoxsulam residue concentrations (Figure 22). By day 200, sugar beets grown in a mixture of 10% composted material were 84% the weight of the untreated control samples. This suggests a NOAC level from 1 to 2 ppb, which is at, or slightly above, the 1.0 µg/kg (ppb) limit of quantification. Testing beyond the NOAC was not conducted because the study's supply was depleted before the value was determined. At a blend of 10% compost to peat, sugar beets can not be safely started from seeds until after between 150 and 234 days of composting. Observed monthly penoxsulam concentrations in compost through 190 days were extrapolated to determine the application rate threshold at which the 0.2 ppb NOEC would not be exceeded. Ninety day compost did not exceed the 0.2 ppb NOEC at an application rate of 10 tons of compost/acre, or less. The 150 day compost produces concentrations below the NOEC at up to 20 tons/acre. The 120 day compost produces concentrations below the NOEC at

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up to 50 tons of compost/acre. Therefore, the application rate would have to be at or below 50 tons of compost/acre, and composted for 150 days. Planting media mixtures containing 33% compost produced from treated turfgrass clippings is only suitable for horticultural uses not involving direct seeding.

Conclusions

The first order penoxsulam foliar dissipation half-life in the clippings collected during the field portion of this study was 2.3 days. The total average percent recovery of penoxsulam from turf grass for the total six week portion of the field study duration was 12.2%. Recovery on the day of application was 88%. The main route of dissipation in turf appears to be associated with foliar wash-off.

Penoxsulam concentrations in the composting mixture were initially measured at 150 ppb, and declined to 1.6 ppb after 9 months of composting. During the composting phase of the study, penoxsulam dissipated with a first-order dissipation half-life of 29 to 31 days.

During the bioassay phase of the study, penoxsulam was found to have an adverse effect to sugar beet seedlings planted in a media containing as little as 10% composted treated turf grass clippings/composted deciduous leaves.

STUDY DEFICIENCIES

1. Insufficient raw data, in tabulated form, was provided for the bioassay portion of this study. As a result, it is not possible to evaluate either the cause or the extent of the adverse effects reported for the sugar beet portion of the bioassay.
2. The stability of penoxsulam in turf clippings could not be confirmed because the storage stability study was not conducted using control samples from the test site fortified at a known concentration and stored frozen for the maximum length of storage of the test samples. Separate stability studies were conducted with penoxsulam and its transformation products in water samples (MRID 46433901) and soil samples (MRID 46433902) obtained from California, but not in either fresh turf clippings or in compost. While penoxsulam stability has been confirmed while stored in frozen soil and frozen water samples, stability has been implied, but not confirmed, in either fresh or composted grass clippings.
3. Degradation products of penoxsulam were not looked for in either the turf clippings or in the resulting compost.
4. A material balance was not determined for penoxsulam and penoxsulam degradation product residues. As a result, while the bioassay does provide useful information concerning the herbicidal activity of composted penoxsulam, the foliar dissipation half-

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life calculated in turf clippings is of questionable quantitative value beyond the use as a lower bounding value for foliar dissipation in turfgrass clippings.

REVIEWER'S COMMENTS

1. Submitted data from the bioassay portion of this study indicated an adverse effect for sprouting sugar beets which were exposed to the penoxsulam treated composted turfgrass clippings. However, insufficient data were provided to evaluate the extent of the adverse effects reported in the bioassay portion of the study.
2. A complete pesticide use history for the test site was not provided.
3. No attempt was made to determine the formation or decline of penoxsulam degradation products in either the turf grass clippings or the resulting compost.
4. The authors stated that irrigation applied during the initial sampling intervals of turf grass clippings may have contributed to the rapid drop in penoxsulam residues measured one week after the first treatment sampling interval. Since penoxsulam residues were only measured in grass clippings from the test plots, it is difficult to say exactly where penoxsulam residues will be found in the environment. Additionally, the calculated, first order half-life derived from data provided by this study report is a foliar wash-off value and not a field dissipation half life.

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Attachment 1: Penoxsulam Molecular Structure

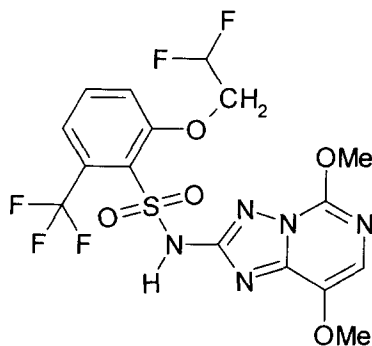
Penoxsulam [XDE-638; DE-638; TSN101649; SP1019 (SePRO)]

IUPAC Name: 3-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)- α,α,α -trifluorotoluene-sulfonamide.
2-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide.
6-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy-s-triazolo[1,5-c]pyrimidin-2-yl)- α,α,α -trifluoro-o-toluenesulfonamide.

CAS Name: 2-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide.

CAS Number: 219714-96-2.

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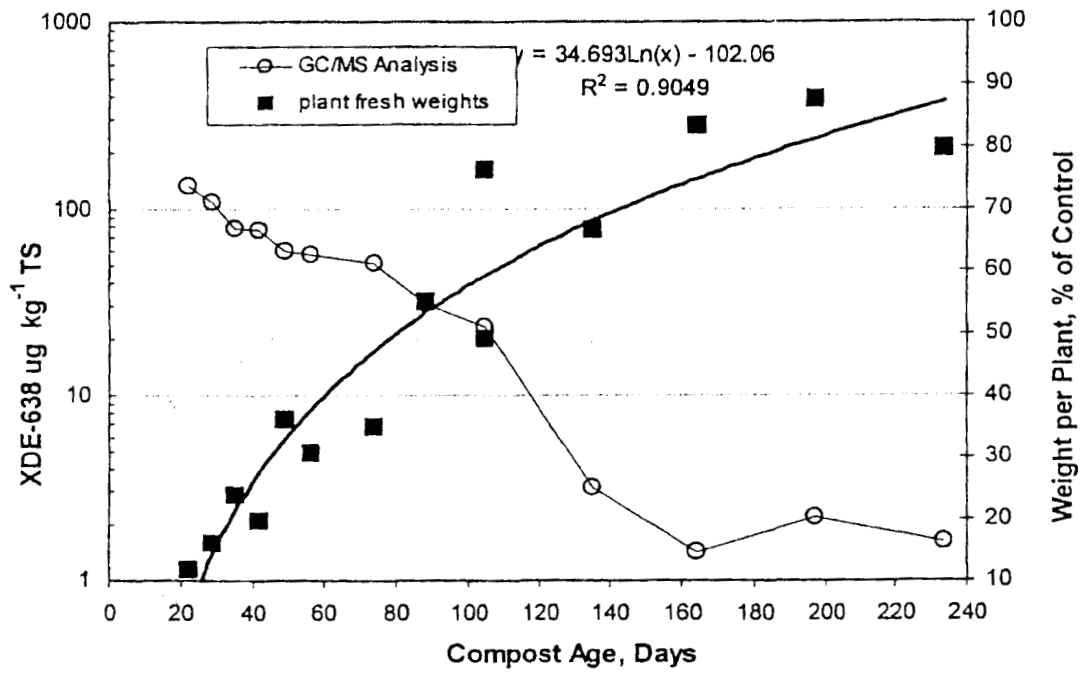
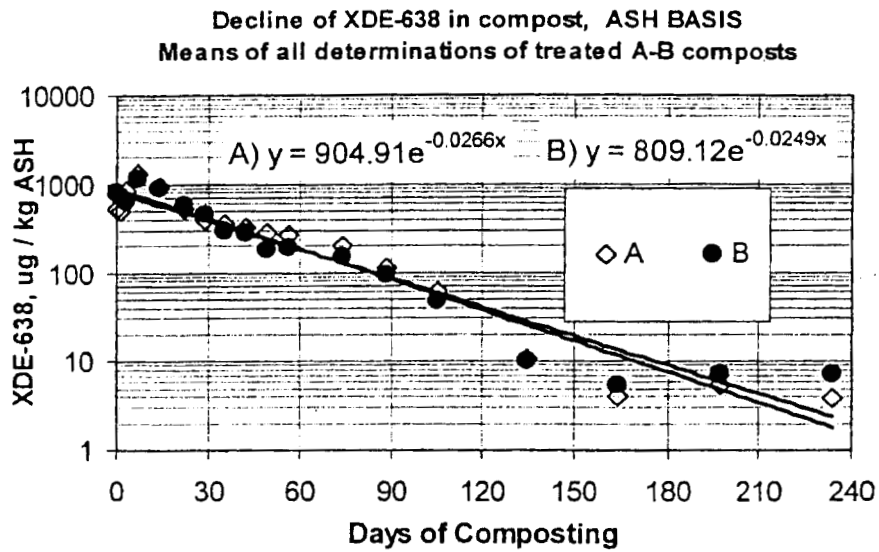
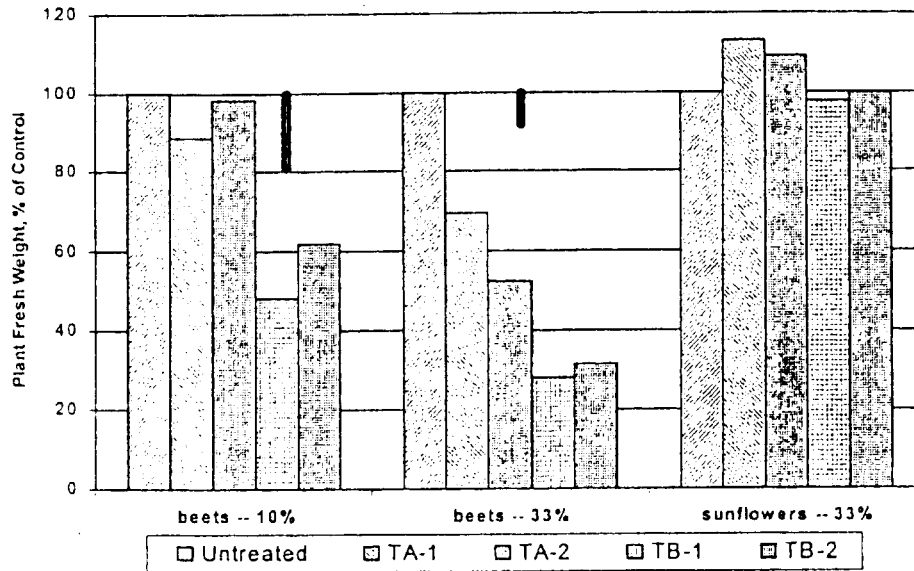


Figure 22. Sugar Beet Performance Averaged for All Samples (A & B combined) vs. Concentration of Penoxsulam in Compost.



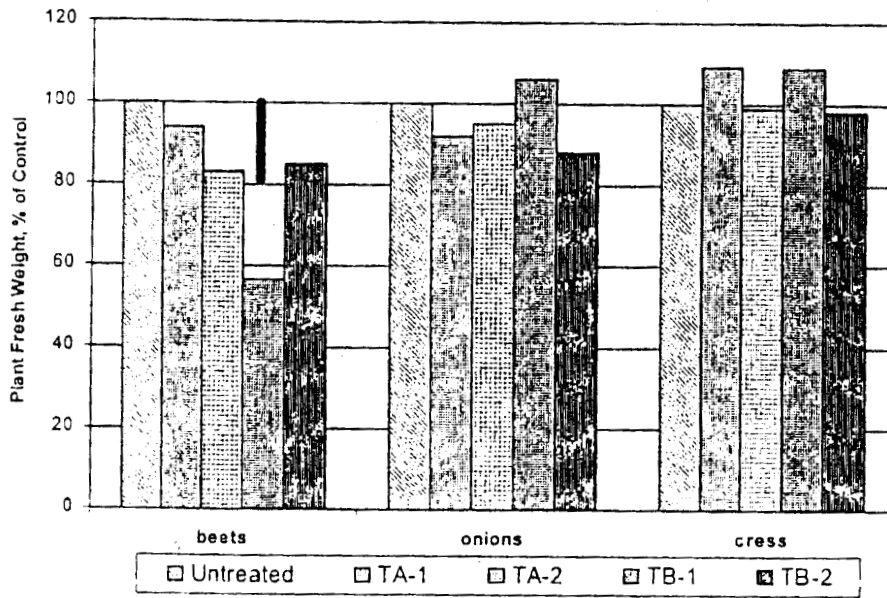
Batches A and B (Ash or TS basis)	Half-life (days)
A and B, (Ash Basis)	26.1, 27.8
A and B, (TS Basis)	29.0, 31.1

Figure 20. Ash and TS-Basis Regression Analysis and Half-life of Penoxsulam in Compost vs. Time of Compost, A vs. B Compost Batches.



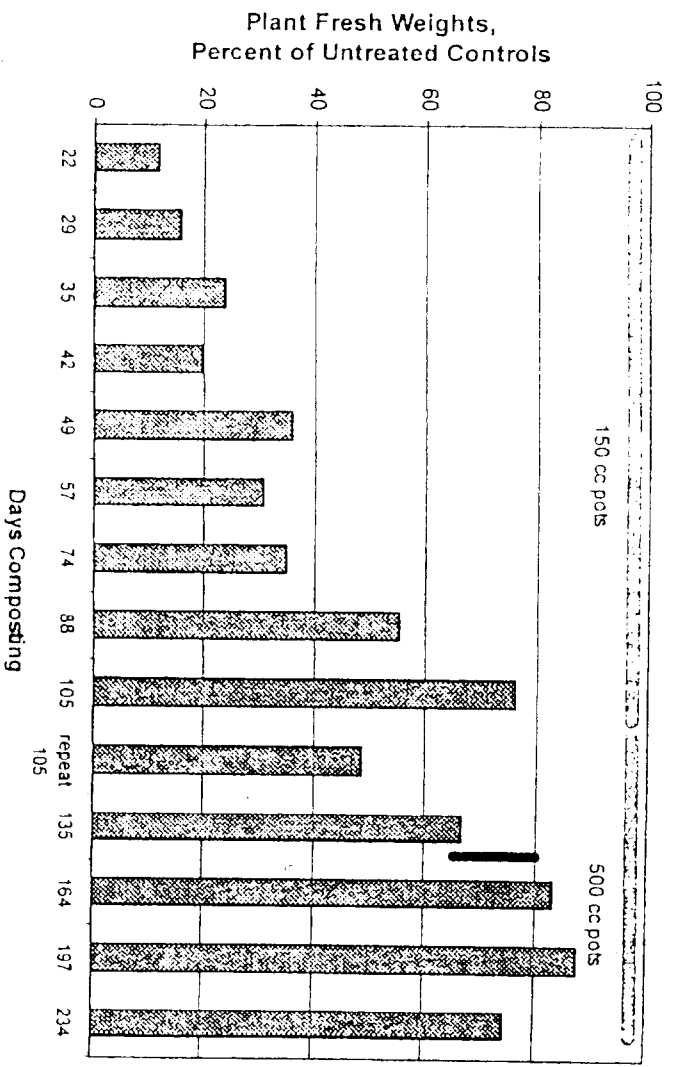
Note: Vertical black bars gives LSD values for p = 0.05 (for beets).

Figure 16. Comparison of Sugar Beet and Sunflower Seedling Bioassay Results for day-235 Compost Samples, Incorporated in the Test Media at 10 and 33% by Volume.



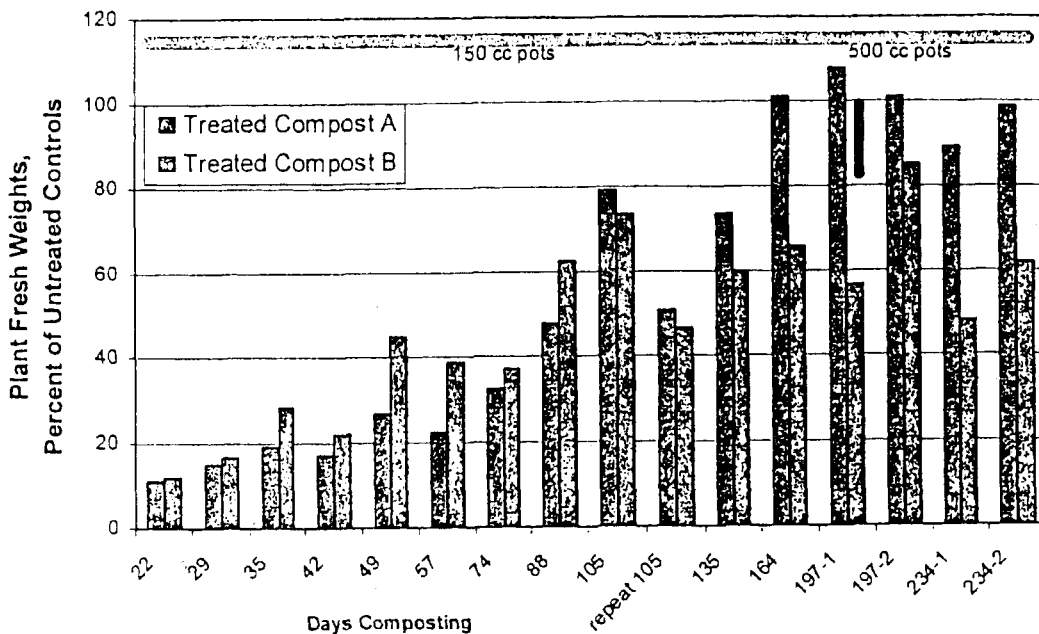
Note: After harvest of the sugar beets, onions and cress were planted in the same pots without disturbing the media. Vertical bar is LSD @ p=0.05 (for beets).

Figure 15. Comparison of Sugar Beet, Onion and Cress Seedling Bioassay Results for day-197 Compost Samples, at 10% of Media Volume.



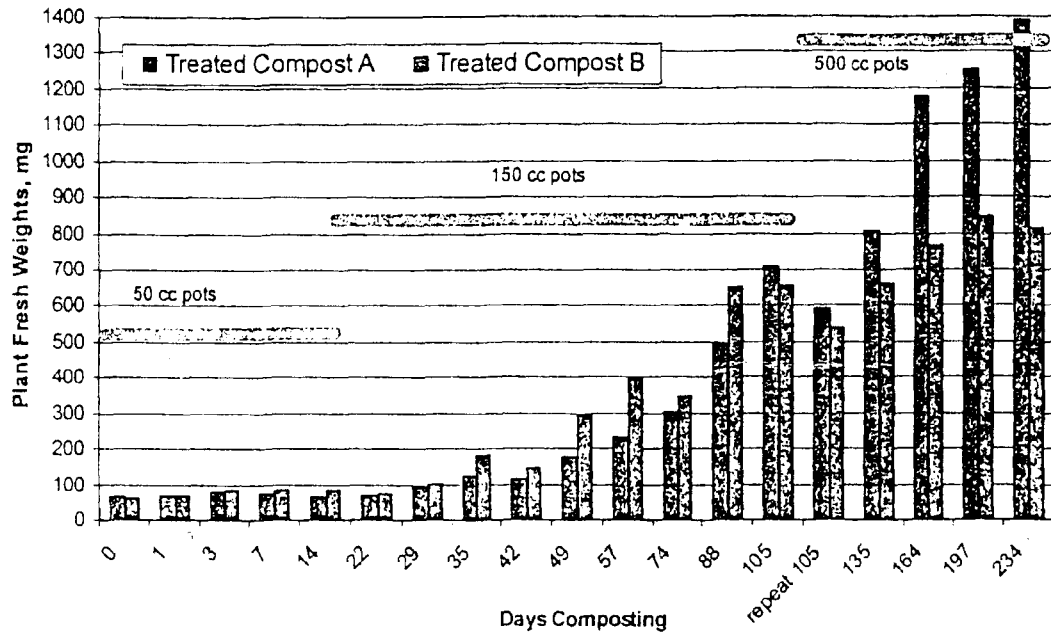
Note: Each bar represents the mean of results for compost batches A and B. Vertical bar shows the LSD value for $p = 0.05$ for differences due to compost age at days 135 to 234.

Figure 14. Mean Results of Sugar Beet Bioassay As Percent of Plant Weights Grown in



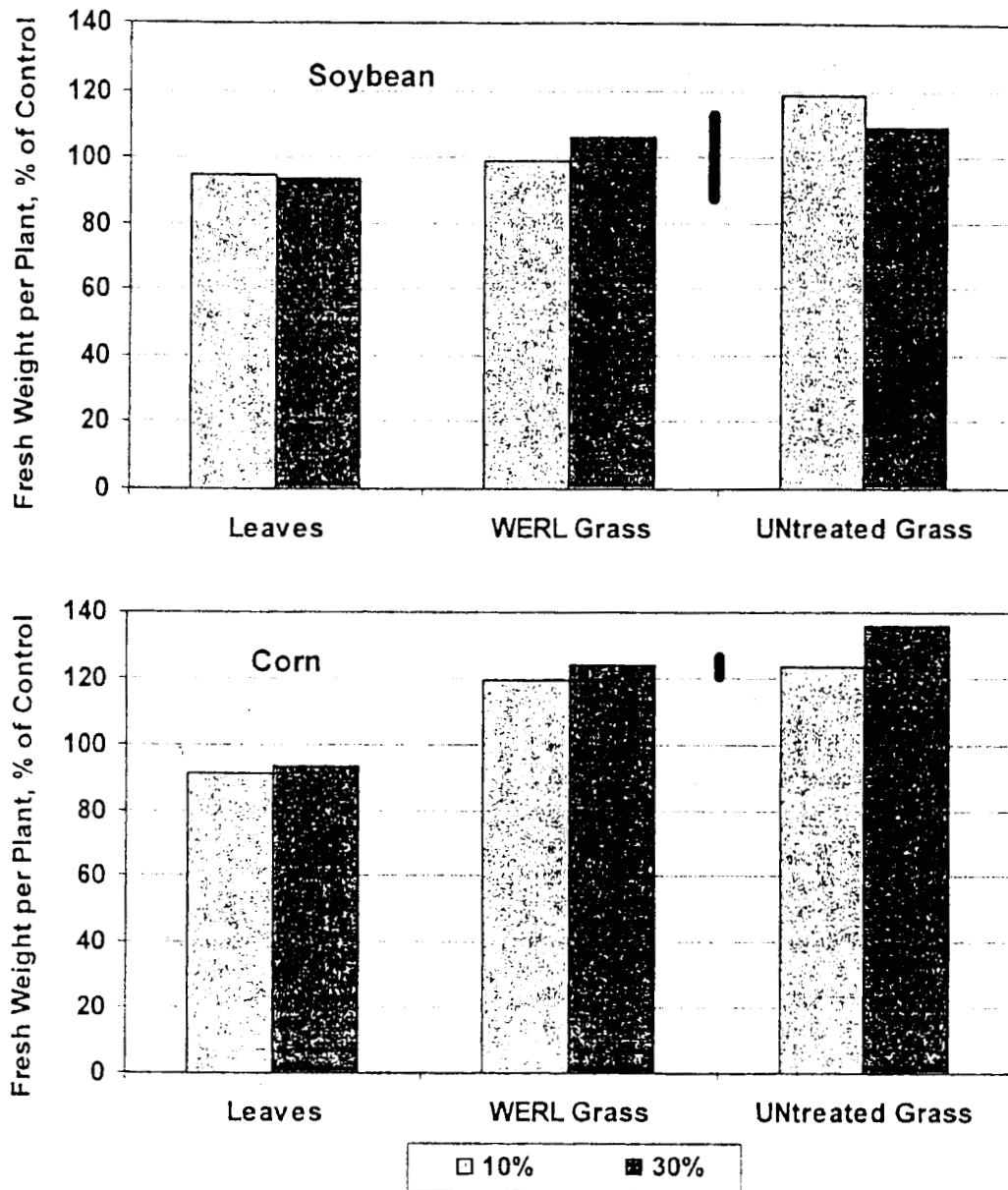
Note: Day-197 and day-234 samples taken from two separate vessels of each of the batches A and B (e.g. 197-1 and 197-2). Vertical bar shows the LSD value for $p = 0.05$.

Figure 13. Fresh Weights of Sugar Beets Grown in Peat-Perlite Media Amended with 10% A or B batches of Compost.



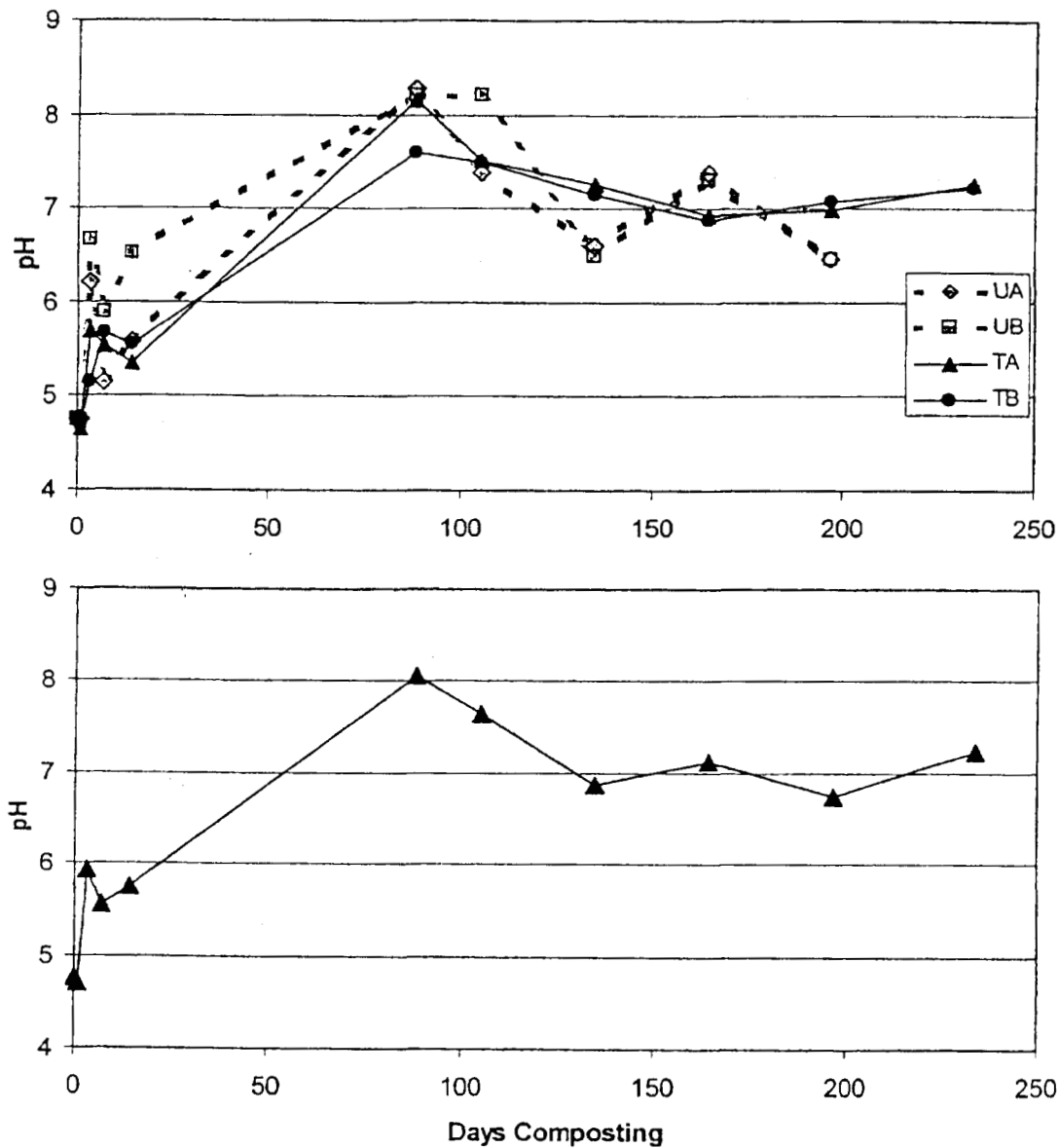
Note: The bioassay pot size was increased on day-22 and day-135 as indicated in the graph.

Figure 12. Sugar Beet Fresh Seedling Weights for T-A and T-B Composts at 10% of Test Media.



Note: Vertical black bars represent LSD for $p \leq 0.05$ for differences between ingredient means.

Figure 9. Effect of Compost Ingredients Incorporated at 10% and 30% of Peat-Perlite Media on Soybean and Corn Fresh Weights in 14-day Seedling Bioassay.



Note: Separate treatments (above) and the average for all vessels (below).

Figure 8. Changes in pH During Composting.

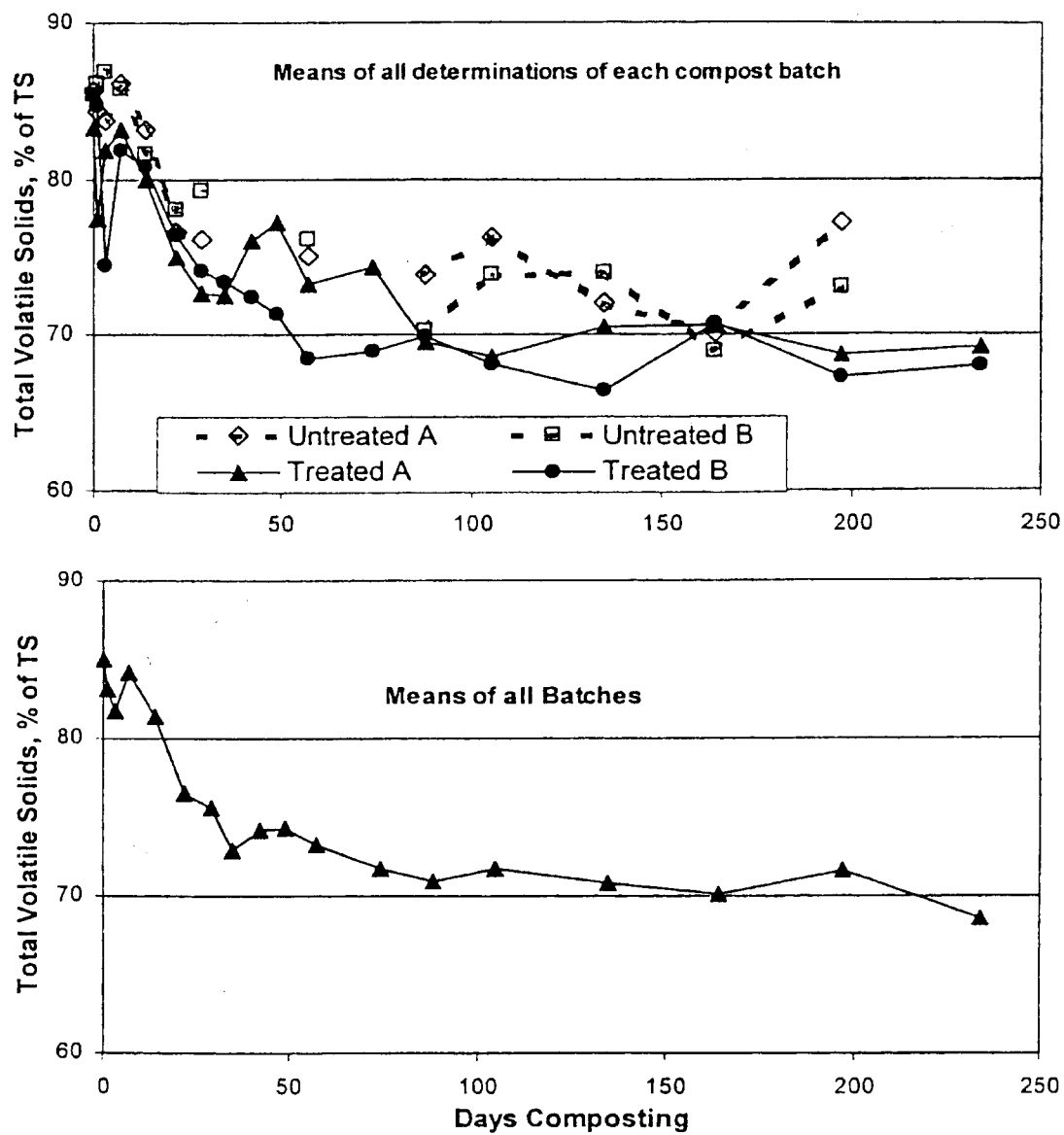


Figure 7. Changes in Total Volatile Solids of All Composts During Composting.

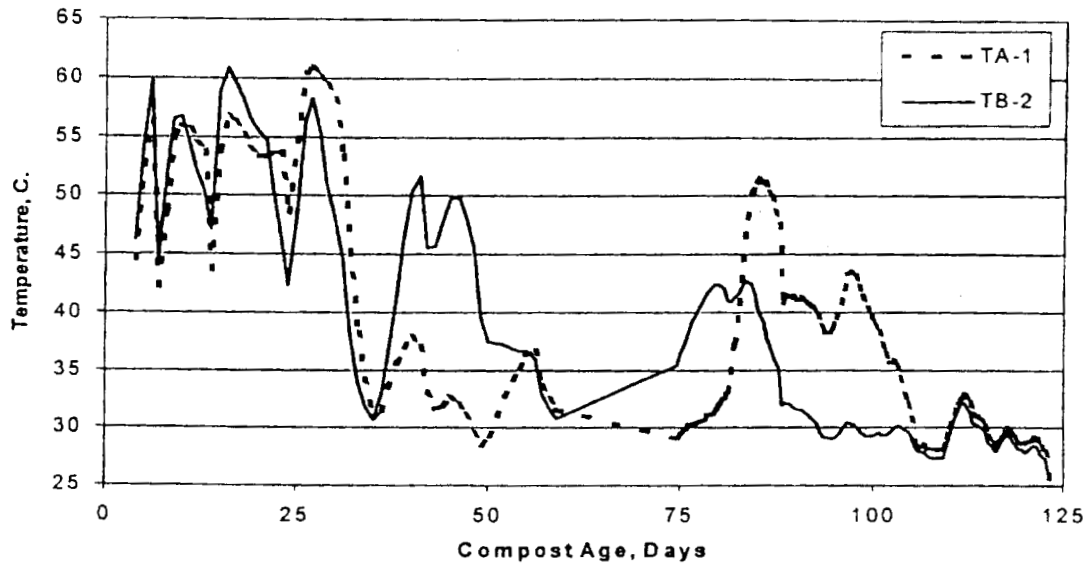
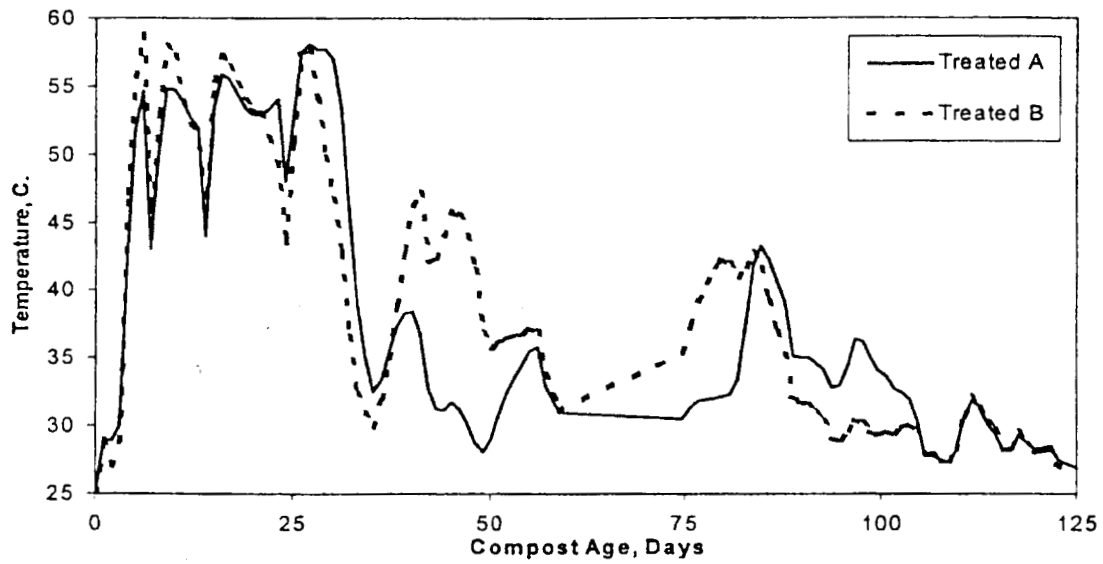
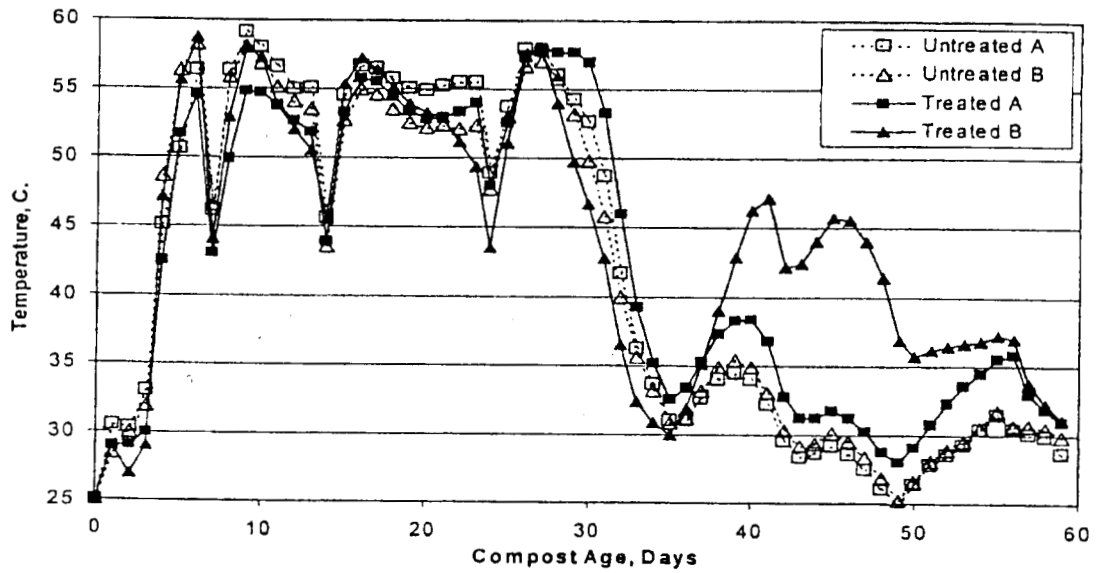


Figure 6. Temperature History of Two Sub-vessels of Treated Compost.



Note: Each line represents the daily means of two compost vessels.

Figure 5. Temperature History of A and B Batches of Treated Compost.



Note: Each point is the daily mean of two compost vessels.

Figure 4. Temperature History of A and B Replicate Batches of U (untreated) and T (treated) Composts.

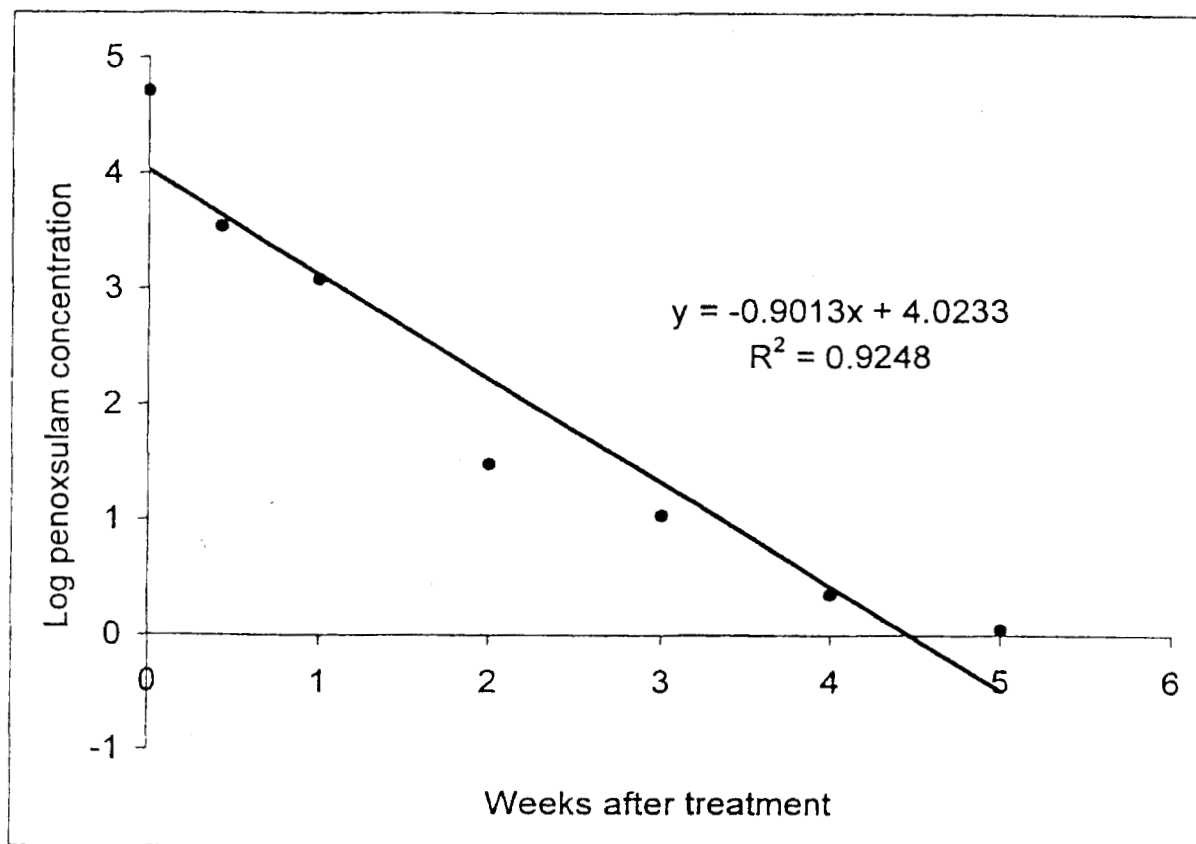


Figure 2. Penoxsulam Concentration (\log_{10}) in Turfgrass 0 - 6 Weeks After Treatment.

Table 7. Predicted Application Rate Thresholds of Compost Containing Penoxsulam at Various Compost Ages.

Compost Age (days)	Conc. of XDE-638 in Compost (ppb, TS basis)	Application Rate, Tons compost/acre		
		10	20	50
		resulting soil conc. (ppb) in 6 in. soil		
0	250	1.25	2.50	6.25
30	100	0.50	1.00	2.50
60	55	0.28	0.55	1.38
90	35	0.18	0.35	0.88
120	10	0.05	0.10	0.25
150	2	0.01	0.02	0.05
190	1	0.01	0.01	0.03

Shaded values are less than or equal to the 0.2 ppb NOEC for sugar beet.
 Calculations: Soil acre = 6 in. and 1000 tons; compost = 50% Total Solids.

Table 6. Composition of Day-234 Treated Composts and Bioassay Potting Media.

batch	compost EC	media EC	compost pH	media pH	cmpst C:N	cmpst NO3	media NO3	Cmpst nitrite	media nitrite	beet, % of U
Day 197 Compost										
UA	2.9	-	6.46	-	13.4	2453	-	225	-	100
UB	1.9	-	6.47	-	12.5	2527	-	94	-	100
TA1	2.5	-	6.91	-	22.2	1150	-	27	-	108
TA2	1.6	-	7.05	-	15.7	1053	-	63	-	102
TB1	2.4	-	7.16	-	12.3	1492	-	75	-	57
TB2	2.3	-	6.98	-	12.7	1596	-	81	-	85
Day 234 Compost and Bioassay media										
UA	-	3.1	-	6.67	-	-	4797	-	<2	100
UB	-	-	-	-	-	-	-	-	-	-
TA1	5.0	2.0	7.28	6.85	-	5775	1652	<2	<2	89
TA2	5.7	2.6	7.23	6.80	-	6309	2697	<2	<2	98
TB1	6.0	2.6	7.29	6.82	-	8409	2670	<2	<2	48
TB2	8.0	2.7	7.15	6.79	-	6607	3027	<2	<2	62

EC = electrical conductivity, dS/m as determined on saturated paste extract

NO3 = nitrate-N concentration as mg/kg from water-soluble extract

Table 2. Composition of Day-0 Compost for Treated and Untreated Duplicate Vessels.

	Treated A	Treated B	Untreated A	Untreated B
Moisture, %	59.2	59.9	60.3	59.4
Organic Matter, %	81.1	83.8	84.0	83.2
Total Nitrogen, %	1.30	1.33	1.46	1.45
C: N	33	33	30	31

Table 1. Composition of Ingredients and Blending Ratios for Composting Process.

	Grass Clippings	Leaves
Moisture, %	71	25
Organic Matter, %	86	80
Total Nitrogen, %	3.2	1.05
Carbon : Nitrogen Ratio	15	41
Volume, % of recipe	28	72
Dry Matter, % of recipe	19	81