

## CORN STOVER PRE-SAMPLING PROCEDURE

Corn stover pre-samples were harvested 14 days before the sampling event to determine target weights and percent moisture estimates. The Protocol specifies a stover moisture content of 15-20%, therefore a method for estimating percent moisture is required.

Pre-samples were collected the same way that the actual samples will be collected as follows:

For each pre-sample, 12 corn plants were selected from the TRT 01, Untreated plot. Plants were cut just above the first node. The 12 corn stalks were placed on a clean tarp-lined table. Corn ears were removed from each stalk. To reduce gross sample size, the plants were separated into 3 groups of 4 plants each. Each plant was divided into 3 approximately equal lengths with leaves attached, using clean loppers. Bottom portions were selected from one group, middle portions were selected from the next group and top portions were selected from the last group. The remaining plant portions were discarded. The selected plant portions were placed into a clean, green mesh drying bag. The bag was labeled. A total of 3 pre-samples were collected in this manner.

The pre-samples were transported to the University of Hawaii in the cab of the pick-up truck. The pre-samples were weighed and then placed into a forced air oven set to 55°C. The oven temperature was set higher than the commercial drying temperature of 35°C to speed the dry down process (for the pre-samples only). Periodically, pre-samples were removed from the oven to check the weight. Pre-samples were dried in the oven until they no longer changed in weight (0% moisture).

Final pre-sample weights were recorded and percent moisture, percent dry matter, and target dry weight ranges for the actual samples were calculated.

gc 3/7/16

gc 3/7/16

# Corn Stover Moisture Estimation Using Pre-sampling

Sample ID	Wet wt w/ bag (lbs)	Bag wt (lbs)	Wet wt w/o bag (lbs)	Final dry wt w/bag (lbs)	Final dry wt w/o bag (lbs)	% Moisture (wet wt-dry wt)÷wet wt*100	% Dry Matter (100-% Moisture)
Pre-Sample 1	2.270	0.125	2.145	0.990	0.865	59.67	40.33
Pre-Sample 2	1.645	0.125	1.520	0.795	0.670	55.92	44.08
Pre-Sample 3	1.690	0.125	1.565	0.845	0.720	53.99	46.01
Initials/Date	gc 3/7/16		X = 1.743	gc 3/10/16		Average= 56.53%	Average= 43.47%

Date/Time Into Oven: 3/7/16 10:10am Date/Time Out of Oven: 3/10/16 12:05 pm.

Target Dry Weight (80%) = (% dry matter in wet weight \* wet wt of sample) ÷ dry matter in final product (0.80)

$Target\ dry\ weight\ (80\%) = (0.4347)(1.743\ lbs) \div 0.80 = 0.947\ lbs.$

Target Dry Weight (85%) = (% dry matter in wet weight \* wet wt of sample) ÷ dry matter in final product (0.85)

$Target\ dry\ weight\ (85\%) = (0.4347)(1.743\ lbs) \div 0.85 = 0.891\ lbs.$

Target range of weights for Stover Samples (15-20% moisture, 80-85% dry matter) = 0.891 lbs to 0.947 lbs.

Corn Stover Pre-Sampling Procedure Followed? YES  NO

Balance Used to Weigh Stover: A+D Field Balance, Ser.No. Q2005443

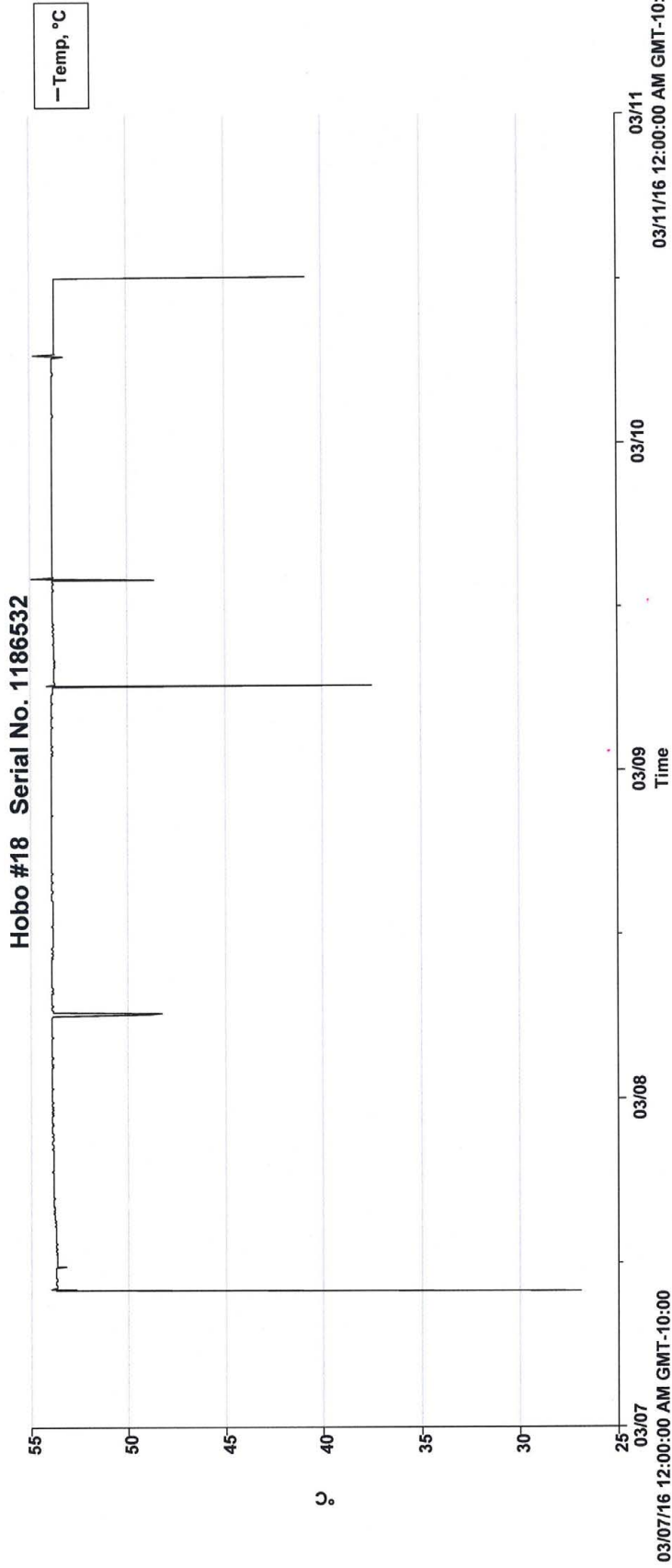
Hobo Recorder Used to Record Oven Temperature: Hobo #18 - in oven Ser.No. 605628-36.

ABOVE DATA ENTERED BY: JMmi Coughlin DATE: 3/10/16

Corn Stover Pre-Sample Drying

SAMPLE ID	DATE	TIME	WEIGHT WITH BAG (LBS)	INITIALS/DATE
1	3/7/16	10:10 am	2.270	At harvest
1	3/8/16	6:15 am	1.010	
1	3/9/16	6:15 am	0.995	
1	3/9/16	2:00 pm	0.995	
1	3/10/16	6:20 am	0.990	
1	3/10/16	12:05 pm	0.990	
1				
1				
1				
1				
1				
1				
1				
2	3/7/16	10:10 am	1.645	At harvest
2	3/8/16	6:15 am	0.810	
2	3/9/16	6:15 am	0.800	
2	3/9/16	2:00 pm	0.795	
2	3/10/16	6:20 am	0.795	
2	3/10/16	12:05 pm	0.795	
2				
2				
2				
2				
2				
2				
2				
3	3/7/16	10:10 am	1.690	At harvest
3	3/8/16	6:15 am	0.855	
3	3/9/16	6:15 am	0.845	
3	3/9/16	2:00 pm	0.845	
3	3/10/16	6:20 am	0.845	
3	3/10/16	12:05 pm	0.845	
3				
3				
3				
3				
3				
3				
3				

Above data entered by Julie Coughlin. (UE) 11/29/16



Oven temperature for corn stover pre-samples, qc 3/10/16

Oven serial no. = 605628-36 qc 3/10/16

qc 3/10/16

Deployment Info

- Launch Name: Hobo #18 Serial No. 1186532
- Launch Time: 03/07/16 09:58:45 AM GMT-10:00
- Logging Interval: 00 Hr 05 Min 00 Sec

Series Statistics

- Samples: 891
- Max: 54.906
- Min: 26.891
- Avg: 53.710
- Std Dev ( $\sigma$ ): 1.181
- First Sample Time: 03/07/16 09:58:46 AM GMT-10:00
- Last Sample Time: 03/10/16 12:08:46 PM GMT-10:00

IR-4 FIELD DATA BOOK

PART 7. SAMPLE COLLECTION AND STORAGE STOVER HARVEST

A.1. GENERAL HARVESTING INFORMATION INSTRUCTIONS: Complete a separate form for each sampling date.

HARVEST DATE<sup>1</sup> 3/21/16 SAMPLING DATE<sup>2</sup> See Part 7 B PHI<sup>3</sup> 41 days after

<sup>1</sup>Record the date of crop harvest (harvest defined as crop digging, crop cutting, picking, etc.)

<sup>2</sup>Enter the date the sampled crop items were placed in sample bags (i.e. sample collection)

<sup>3</sup>Record the number of days from last application to harvest (PHI)

Soil Application

IF THE PHI IS 0 DAYS, WAS THE SPRAY DRY BEFORE THE CROP WAS HARVESTED? YES \_\_\_ NO \_\_\_ NA \_\_\_

(Check NA if PHI > 0 days or if the test substance was not sprayed, e.g. a granular application.)

DESCRIPTION OF HARVESTED CROP STAGE

(E.g. commercially mature lettuce heads, blueberries mature in size (mostly blue in color), mature plums for drying)

mature corn stalks.

Number of (check one) Plants <input checked="" type="checkbox"/> Trees ___ Bushes ___ Areas ___ of the Plot from Which Each Sample was Collected	<u>12</u>
Number and Location of Rows from Which Each Sample Was Collected Examples: "6 middle rows" "All 3 rows" "1" (for single-row plot)	<u>TRT 01 + TRT 02 - 6 middle rows</u> <u>TRT 03 - 2 middle rows.</u>
Minimum Number of (check one) Fruit ___ Heads ___ Roots ___ Plants <input checked="" type="checkbox"/> Other ___ (describe) Actually Collected per Sample	<u>12</u> (If a minimum is required by the protocol)
Number of (check one) Plants ___ Trees ___ Bushes ___ at Each End, or (check) Length of Row Ends <input checked="" type="checkbox"/> , That Were Not Sampled	<u>5 ft. on each end of rows</u>
Was Less Than 50% of the Harvestable Crop Sampled? (May be determined by visual estimation)	YES <input checked="" type="checkbox"/> NO ___ If no is checked, contact the Study Director
Was Each Sample Collected in a Separate Run Through the Entire Plot?	YES <input checked="" type="checkbox"/> NO ___ If no is checked, contact the Study Director
HARVESTING EQUIPMENT (Provide a brief description of harvesting equipment, including make and model numbers, if appropriate. Do not include gloves, sample bags, coolers, or scales.)	
<u>Lopers</u>	
ORDER OF SAMPLE COLLECTION	<u>TRT 01, Sample M, N; TRT 02, Sample O, P; TRT 03, Sample Q, R.</u> ←

← see Part 7, page 11 for explanation.

BRIEFLY DESCRIBE PROCEDURES UTILIZED TO HARVEST CROP. Provide enough details in addition to data entered above to ensure that protocol requirements have been met and to inform a data reviewer exactly how this crop was harvested. Examples: "Hand-picked berries from one side of the row, then the other. Collected fruit from high and low, exposed and shielded areas." "Barley was cut 3-4 inches above the ground with a scythe and left on the ground to dry for hay samples. Each entire plot was cut." ATTACH A SEPARATE SHEET IF NECESSARY.

TRT 01 + TRT 02 - I cut 2 corn stalks per row from the inner 6 rows in each plot. TRT 03 - I cut 6 corn stalks from the inner 2 rows in the plot. Corn stalks were selected throughout the length of the rows, leaving at least 5 ft. on row ends unsampled. Corn stalks were cut just above the first node using clean lopers. 12 corn stalks were collected per sample.

Were harvested crop items collected directly into residue sample bags? YES \_\_\_ NO

IF NO, PLEASE EXPLAIN After cutting, corn stalks were handed to James and he carried the stalks out of the plot and placed them on a clean, tarp-lined table.

ABOVE DATA ENTERED BY: Julie Coughlin DATE: 3/21/16

Imidacloprid/Corn (seed crop)

ID No. 11270.15-HI197

FIELD ID NO:

Coughlin

### IR-4 FIELD DATA BOOK

#### PART 7. SAMPLE COLLECTION AND STORAGE

A.2. GENERAL SAMPLING INFORMATION--*Complete a separate form for each sampling date.*

**DESCRIPTION OF SAMPLED CROP STAGE** (if different from harvested crop, such as dried plums, mint oil)

*Corn stover (corn stalks dried to approximately 15-20% moisture)*

**IF THE SAMPLING OCCURRED AFTER THE HARVEST DATE, DESCRIBE SAMPLE COLLECTION. ALSO, DESCRIBE ANY MODIFICATIONS TO THE HARVESTED CROP SUCH AS TRIMMING, CLEANING, CUTTING, DRYING AND/OR COMPOSITING SAMPLES. IF THE MODIFICATIONS ARE TOO COMPLEX TO BE DESCRIBED BELOW, ATTACH A SEPARATE SHEET THAT CLEARLY DESCRIBES THE MODIFICATION PROCEDURES**

Include a description of equipment, duration of procedure(s), temperatures, estimated moisture content, etc., as appropriate.

*See following page for modifications to harvested crop of 3/21/16*

**CHECK ALL PROCEDURES USED TO PREVENT CONTAMINATION OF RESIDUE SAMPLES**

- UNCONTAMINATED GLOVES WORN AND CHANGED BETWEEN SAMPLES
- TREATMENTS WERE SAMPLED BY DIFFERENT PEOPLE
- PHYSICALLY SEPARATED TREATED AND UNTREATED SAMPLES
- CLEANED SAMPLING EQUIPMENT BETWEEN COLLECTIONS OF EACH TREATMENT
- OTHER, EXPLAIN: *Cleaned lopers with soap and water between each sample. Cleaned tarp-lined table with soap + water, rinsed, and dried between each sample.*

**DESCRIBE HOLDING AND TRANSPORT OF SAMPLES FROM FIELD TO FREEZER**

(E.g. Sample bags placed in cooler with blue ice, then transported by pickup truck to research center for pitting. Following pit removal, sample bags were hand-carried to freezer.)

*Green mesh bags containing stover were placed into new trash bags and then transported to field lab at Univ. of HI in the cab of the pick up truck.*

*Separate trash bags were used for each sample.*

ABOVE DATA ENTERED BY:

*Julie Coughlin*

DATE:

*3/21/16*

PART 7. A.2

MODIFICATIONS TO THE HARVESTED CROP

For each sample, 12 corn stalks were placed on a clean tarp-lined table. To reduce gross sample size, the plants were separated into 3 groups of 4 plants each. Corn ears were removed by hand and discarded. Each plant was divided into 3 approximately equal lengths with leaves attached using clean loppers. Bottom portions were selected from one group, middle portions were selected from the next group and top portions were selected from the last group. The remaining plant portions were discarded. James, Mike and I worked together to complete this operation.

The selected plant portions were placed into a new, green mesh plastic bag and tied shut. The green mesh bag was labeled with the Sample ID and then the green mesh bag was placed into a new plastic trash bag and tied shut. The bags were transported to the University of Hawaii field lab in the cab of the pick-up truck.

Upon arrival at the field lab, the sample bags were removed from the plastic trash bags, weighed on the field balance, and the weights recorded. The weights included the green mesh bags. The samples were weighed in ascending order from TRT 01 to TRT 03. A tray was used on the balance to support the bags. The tray was washed with soap and water, rinsed and dried, between each sample. The TRT 01 samples M and N were placed into drying oven Serial No. 9308-007. The TRT 02 samples O and P, and TRT 03 samples Q and R were placed on separate shelves in drying oven Serial No. 605628-36. Both ovens are located in the Field Lab (St. John, Rm. 006). Both ovens were set to 35°C with airflow setting at 8. Hobo temperature recorders were placed into each oven to record the drying temperatures.

ABOVE DATA ENTERED BY: Julie Coughlin DATE: 3/21/16

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Samples were dried to within the target weight ranges calculated in the Stover Target Weights Table. During the drying process, samples were removed from the oven periodically to check the weight. When each stover sample was dried to the appropriate target weight, the bags were removed individually from the oven and weighed, and the weight recorded. The stover was removed from the green mesh bag and cut into smaller pieces so that it would fit in the sample bag. The stover was cut on a clean counter using clean pruning shears. The stover pieces were placed into a labeled IR-4 sample bag then into the lab freezer, RF-17. The final sample weights were calculated by subtracting the bag weight from the weight recorded.

ABOVE DATA ENTERED BY: Julie Coughlin DATE: 3/27/16

# STOVER HARVEST



3 groups of 4 stalks each

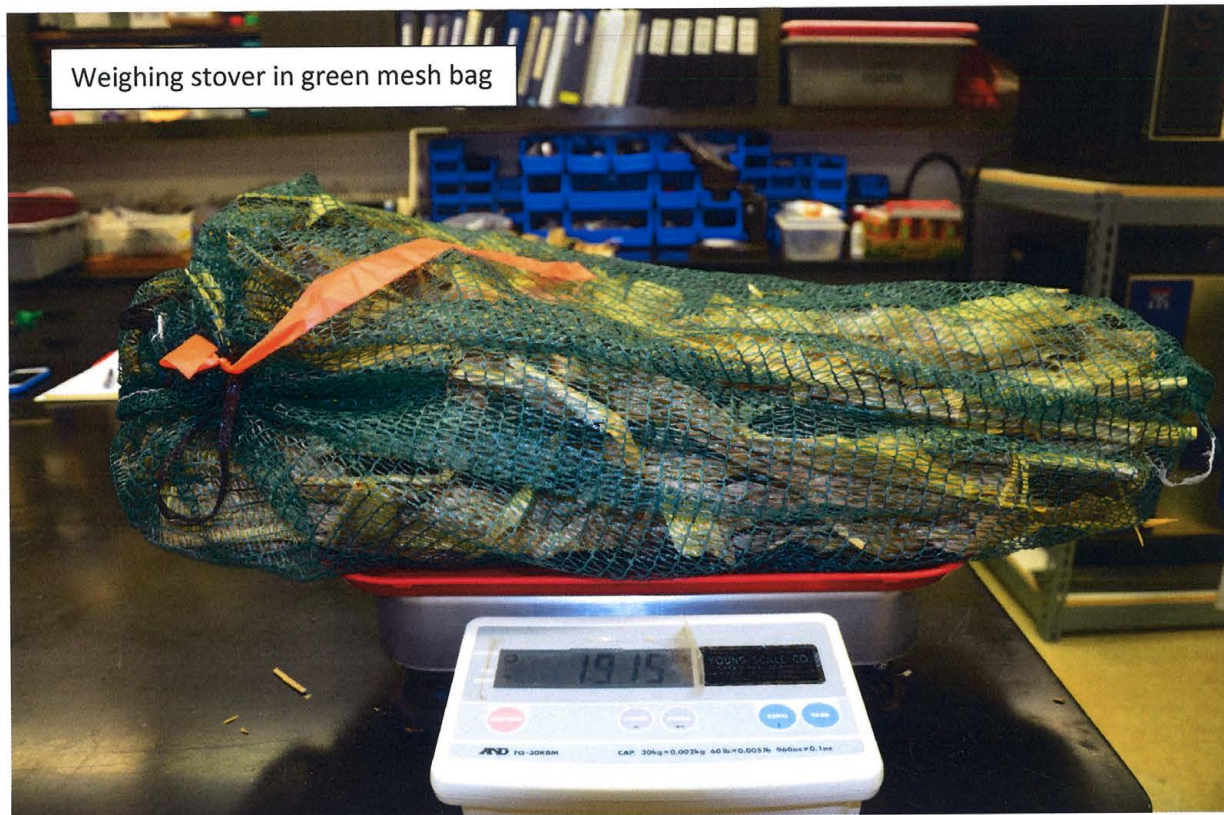


Top, middle, and bottom portions

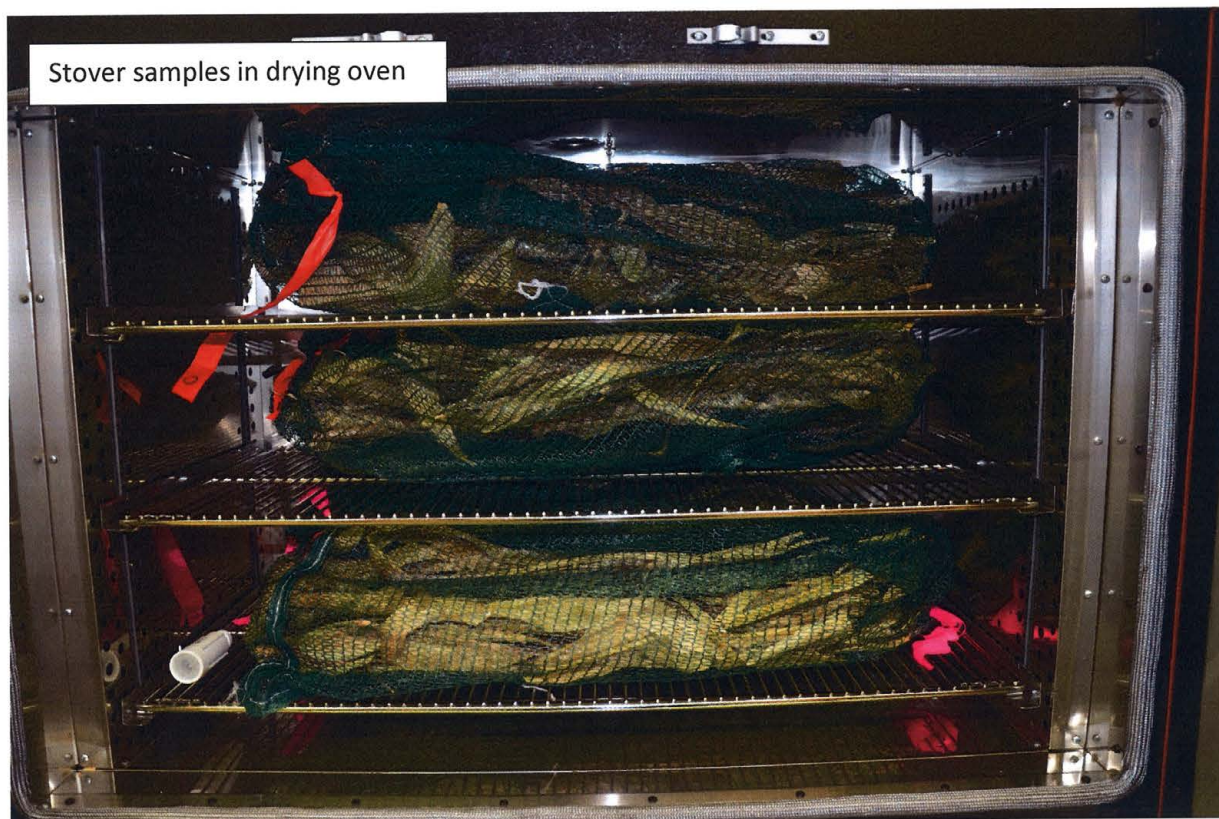


# STOVER DRYING

Weighing stover in green mesh bag



Stover samples in drying oven



### STOVER HARVEST TABLE

SAMPLE ID	DATE/TIME HARVESTED	WET WEIGHT W/BAG (LBS)	TIME SAMPLE PLACED INTO OVEN	OVEN SERIAL NUMBER
M	3/21/16 9:20 am	1.695	2:01 pm.	9308-007
N	3/21/16 9:40 am	1.675	2:02 pm	9308-007
O	3/21/16 10:20 am	1.915	2:04 pm	605628-36
P	3/21/16 10:35 am	1.450	2:04 pm	605628-36
Q	3/21/16 11:20 am	1.655	2:06 pm	605628-36
R	3/21/16 11:30 am	1.655	2:07 pm	605628-36

ABOVE DATA ENTERED BY: JM Coughlin DATE: 3/21/16

*JC* 4/22/16

STOVER TARGET WEIGHTS TABLE

SAMPLE ID	WET WEIGHT W/BAG (LBS)		WET WEIGHT W/O BAG (LBS)	% DRY MATTER <sup>1</sup>	15%	20%	15%	20%
	WEIGHT	BAG WEIGHT			MOISTURE TARGET WEIGHT W/O BAG (LBS) <sup>2</sup>	MOISTURE TARGET WEIGHT W/O BAG (LBS) <sup>3</sup>	MOISTURE TARGET WEIGHT W/BAG (LBS)	MOISTURE TARGET WEIGHT W/BAG (LBS)
M	1.695	0.135	1.560	0.4347	0.798	0.848	0.933	0.983
N	1.675	0.135	1.540	0.4347	0.788	0.837	0.923	0.972
O	1.915	0.135	1.780	0.4347	0.910	0.967	1.045	1.102
P	1.450	0.135	1.315	0.4347	0.673	0.715	0.808	0.850
Q	1.655	0.135	1.520	0.4347	0.777	0.826	0.912	0.961
R	1.655	0.135	1.520	0.4347	0.777	0.826	0.912	0.961

<sup>1</sup> % Dry Matter estimation from pre-sample calculations.

<sup>2</sup> Target dry weight (85%) = (% dry matter X wet wt of sample) ÷ dry matter in final product (.85)

<sup>3</sup> Target dry weight (80%) = (% dry matter X wet wt of sample) ÷ dry matter in final product (.80)

*[A large blue diagonal line is drawn across the page, starting from the top left and ending near the bottom right. The initials 'JC' and the date '3/21/16' are written in blue ink near the end of the line.]*

ABOVE DATA ENTERED BY: Jamie Coughlin DATE: 3/21/16

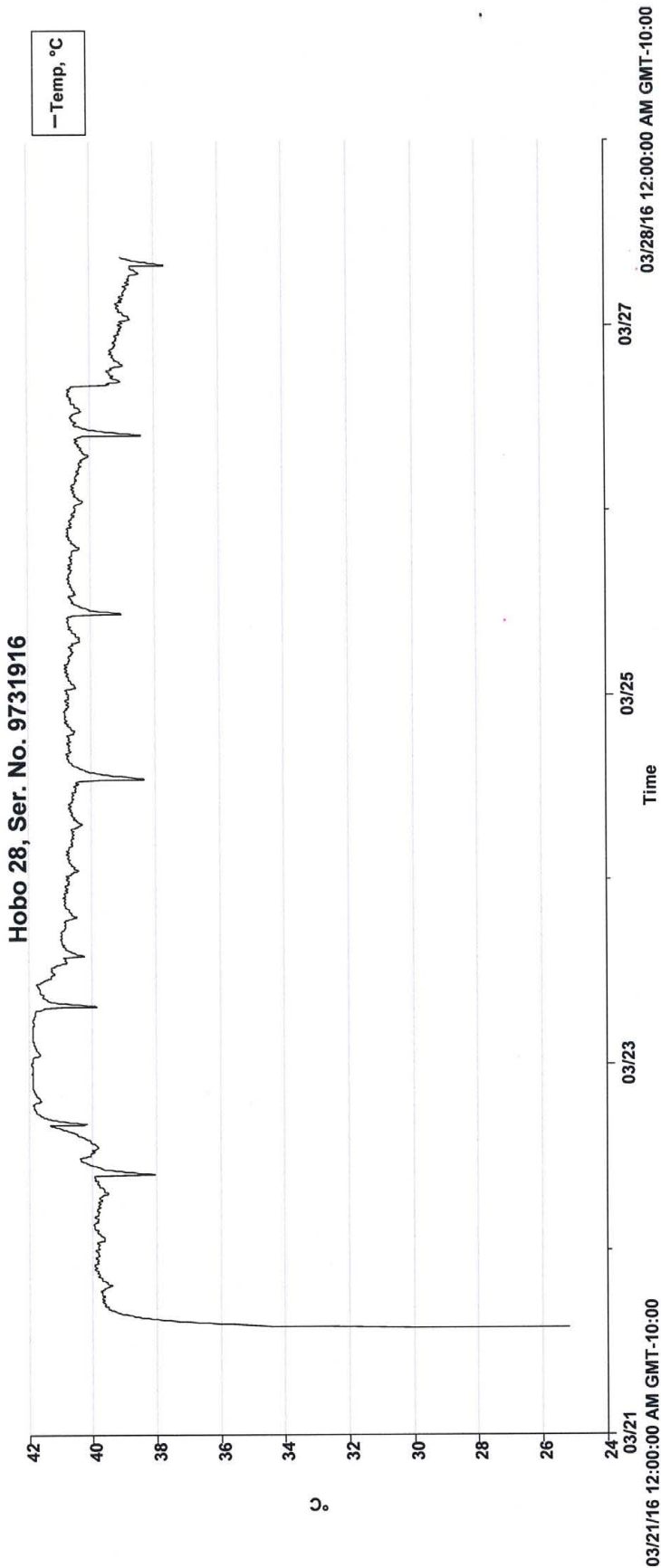
## Final Stover Sample Weights and % Moisture

Sample ID	Wet wt w/o bag (lbs)	Final dry wt w/bag (lbs)	Final dry wt w/o bag <sup>1</sup> (lbs)	Dry wt (Wet wt w/o bag x % dry matter calc. from pre-sampling)	(Final dry wt w/o bag - Dry wt) ÷ Final dry wt w/o bag x 100	% Moisture	Meets Protocol 15% -20% moisture content?	Initials / Date
M	1.560	0.980	0.845	0.678132	19.75	19.75	Yes	gc 3/26/16
N	1.540	0.965	0.830	0.669438	19.34	19.34	Yes	gc 3/27/16
O	1.780	1.070	0.935	0.773766	17.24	17.24	Yes	gc 3/27/16
P	1.315	0.850	0.715	0.5716305	20.05	20.05	Yes	gc 3/27/16
Q	1.520	0.960	0.825	0.660744	19.91	19.91	Yes	gc 3/27/16
R	1.520	0.930	0.795	0.660744	16.89	16.89	Yes	gc 3/26/16

<sup>1</sup> Bag Weight = 0.135 lbs

Comments:

Percent dry matter calculated from pre-sampling = 43.47%  
 gc 3/27/16



PART 7 PAGE 30

Deployment Info

- Launch Name: Hobo 28, Ser. No. 9731916
- Launch Time: 03/21/16 01:57:00 PM GMT-10:00
- Logging Interval: 00 Hr 05 Min 00 Sec

Series Statistics

- Samples: 1,670
- Max: 41.913
- Min: 25.162
- Avg: 40.353
- Std Dev ( $\sigma$ ): 0.994
- First Sample Time: 03/21/16 01:57:01 PM GMT-10:00
- Last Sample Time: 03/27/16 09:02:01 AM GMT-10:00

Oven temperature for Stover samples  
TKR 01, Samples M and N.

QC 3/27/16

Oven Serial No. = 9308-007.

QC 3/27/16

QC 3/27/16