

# Total hydrocarbon and HAP emissions from the drying of Douglas-fir lumber

Report to  
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May 23, 2013

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## Summary

Two charges of Douglas-fir 2x4 lumber were dried in a small kiln at Oregon State University. The kiln dry- and wet-bulb temperatures were based on a schedule provided by Sierra Pacific Industries. The maximum temperature was 175°F (80°C). The air velocity was 750 feet per minute (3.7 m/s). The kiln was indirectly heated with steam. The amount of air entering the kiln was regulated to control humidity.

Only the results for charge 2 are reported because of a kiln control issue during charge 1.

A JUM VE-7 total hydrocarbon analyzer was used to measure organic emissions following EPA Method 25A. The results are shown in Table 1.

**Table 1.** Summary of total hydrocarbon results to 15% moisture content. VOC units are pounds per thousand board feet as carbon.

Charge	Initial MC	Final MC	Time to 15%	VOC <sup>B</sup>
	%	%	hr:min	lb/mbf
Douglas-fir 2	32.5	12.5 <sup>A</sup>	17:49	0.185

<sup>A</sup> actual time to 10.5% MC was 25:33 hours

<sup>B</sup> VOCs are reported at 15% moisture content

NCASI Method ISS/FP-A105.01 was used to measure the MACT HAP emissions. The results for charge two are shown in Table 2. The sum of the HAPs emitted was 0.026 lb/mbf for the Douglas-fir.

**Table 2.** Summary of HAP results for moisture content and time shown in Table 1. Emissions units are pounds per thousand board feet.

	Methanol	Phenol <sup>A</sup>	Form-aldehyde	Acet-aldehyde	Propion-aldehyde	Acrolein
DF-2	0.019	0.00	0.001	0.006	0.0001	0.0004

<sup>A</sup> None detected

## 1. Description of source

The tested source is a lumber dry kiln. Lumber destined for the mill's kiln was sampled and tested in a small-scale kiln at Oregon State University.

Mill personnel reported that the logs were Inland Douglas-fir harvested on April 9, 2013. Lumber came from multiple logs but all from the same logging site on the tree farm. All trees were 51 years of age. At the mill the logs were sawed immediately after unloading from the truck. The lumber was sawed on April 29, 2013, wrapped, and loaded to be delivered to OSU on May 2, 2013.

Enough wood for three charges of lumber was delivered to Oregon State by an employee of Sierra Pacific Industries on January 25, 2013. The wood was wrapped in plastic and lumber wrap at the mill to prevent moisture loss during transport. The wood appeared to be fresh. The initial moisture content (32.5%) was slightly lower than published for Inland (Intermediate type) Douglas-fir heartwood (34% in USDA Dry Kiln Operator's Manual).

At OSU, the 8-foot 2x4s were cut to 4 feet and sorted into three 33-board units on May 2. It was wrapped in plastic and two charges were placed in a refrigerator at 35°F and the remainder in a freezer at 10°F.

## 2. Date and time of test

The first charge was dried from May 7, 2013 at 8 am until 9:30 am on May 8, 2013. The second charge was dried from May 14, 2013 at 11 am until 10:30 am on May 15, 2013. Drying was done under the supervision of Mike Milota at Oregon State University. Students were used to monitor parts of the test.

## 3. Results

### Total hydrocarbon

See Table 1, page 1, for a summary of the hydrocarbon results. Details for each sampling interval are tabulated and the hydrocarbon emissions are summarized graphically here. All emission data is presented in detail in electronic form in Appendix 2.

A summary for each sampling interval is in Table 3 for charge 2. An interval is the period between analyzer calibrations, about three to six hours of data. The interval time periods shown in the table include the calibration times and mass calculations are adjusted to account for these. Sampling occurred for approximately 95% of the drying time.

Figure 1 shows total hydrocarbon concentration (left scale) and dry gas vent rate (right scale) versus time. Concentration has a large peak at 5 hours when venting increases. The concentration then decreases until the schedules midpoint after which it increases somewhat. The latter increase occurs as the vent rate decreases.

Figure 2 shows the cumulative hydrocarbon emissions (left scale, smooth line) and the rate of emissions (right scale, jagged line) versus time. The cumulative emissions is the emissions up to any point in time in the schedule. The rate of emissions is how much is coming out per unit time. The maximum emission rate occurs at seven hours, shortly after the venting but while the concentration is still high. It then steadily decreases as the moisture loss from the wood slows.

Figure 3 shows the total hydrocarbon emissions as a function of wood moisture content. This graph would be useful for predicting emissions at various final moisture content levels.

**Table 3.** Summary of results for each sampling interval for total hydrocarbon during charge 1.

Sample Run	Time hrs	Average Humidity kg/kg	Flow rate		THC mass as C g	THC concentration		THC rate as C g/hr	THC mass as C lbs/mbf	THC rate as C lb/hr/mbf	Average		
			Dry @68 l/min	Wet @68 l/min		wet ppmv	dry ppmv				Wood MC %	Air MC %	Anal. MC %
1	2.25	0.032	5.1	5.3	0.08	68.5	73.7	0.04	0.002	0.0010	32.5	4.9	4.9
2	3.26	0.126	5.4	6.5	0.47	242.0	298.9	0.15	0.013	0.0040	32.3	16.9	7.4
3	2.40	0.211	34.0	45.6	1.93	254.1	345.5	0.80	0.053	0.0221	30.9	25.4	8.6
4	1.25	0.196	53.5	70.4	0.87	110.0	146.4	0.70	0.024	0.0192	27.6	24.0	8.2
5	1.30	0.187	49.7	64.7	0.64	83.7	110.0	0.49	0.018	0.0135	25.2	23.2	7.9
6	3.25	0.170	47.4	60.3	1.43	80.5	103.6	0.44	0.039	0.0121	21.7	21.5	7.3
7	4.11	0.150	38.2	47.4	1.29	74.5	93.4	0.32	0.036	0.0087	16.8	19.5	6.6
Sum	17.82				6.7				0.185				
Average		0.153	33.3	42.9		130.5	167.4	0.42		0.0115			



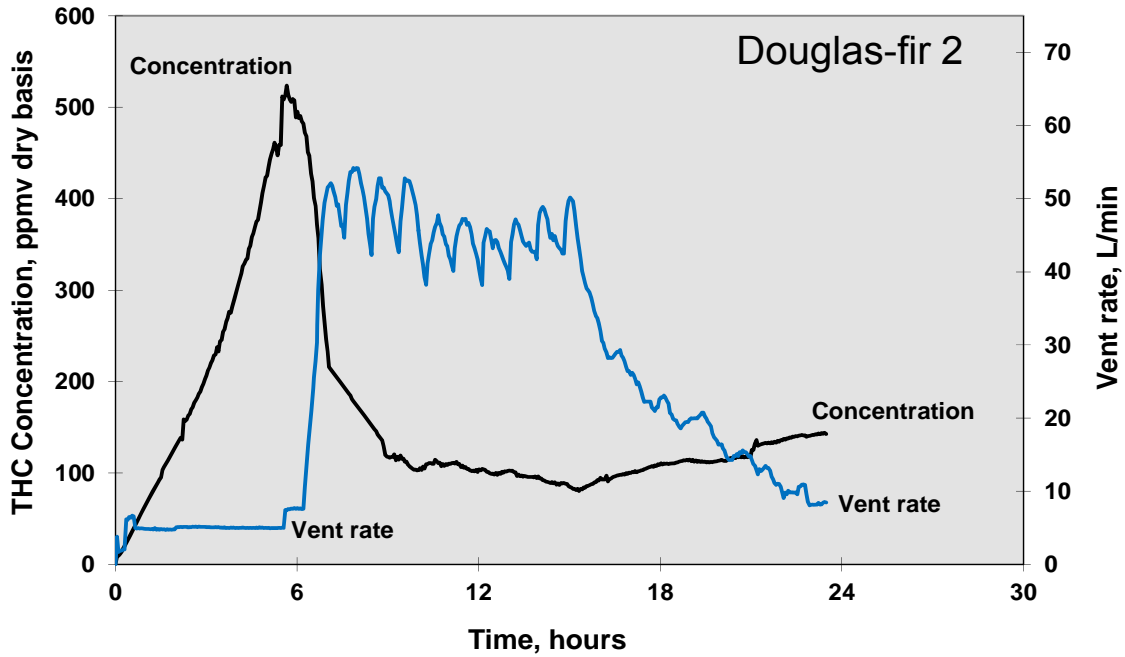


Figure 1. Hydrocarbon concentration and vent rate versus time for charge 1.

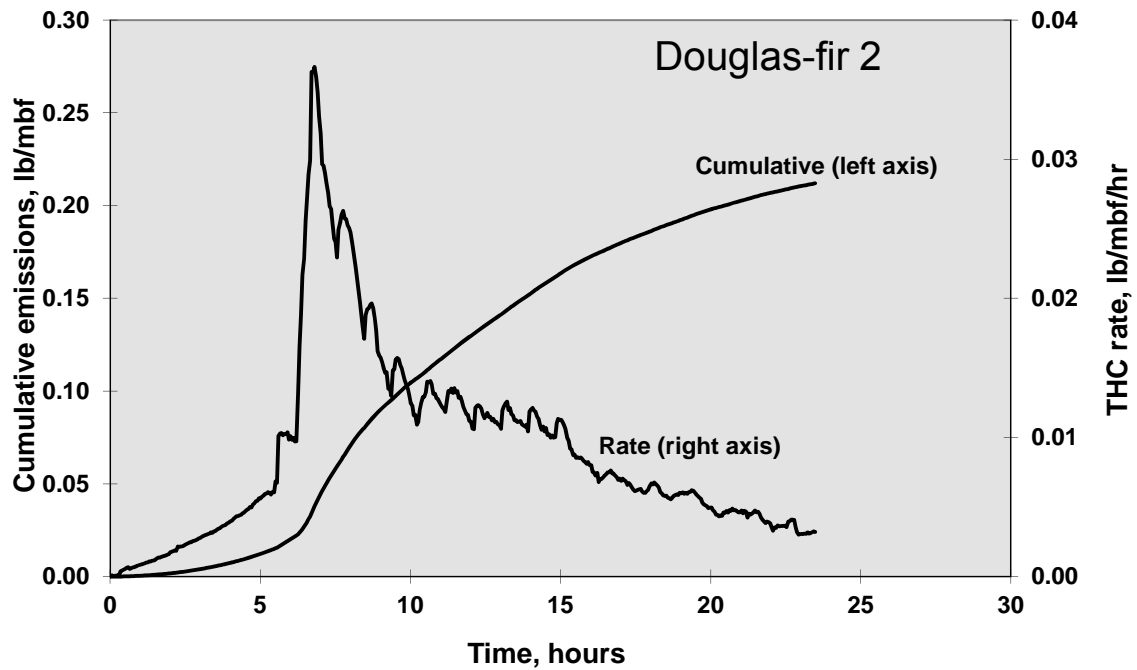
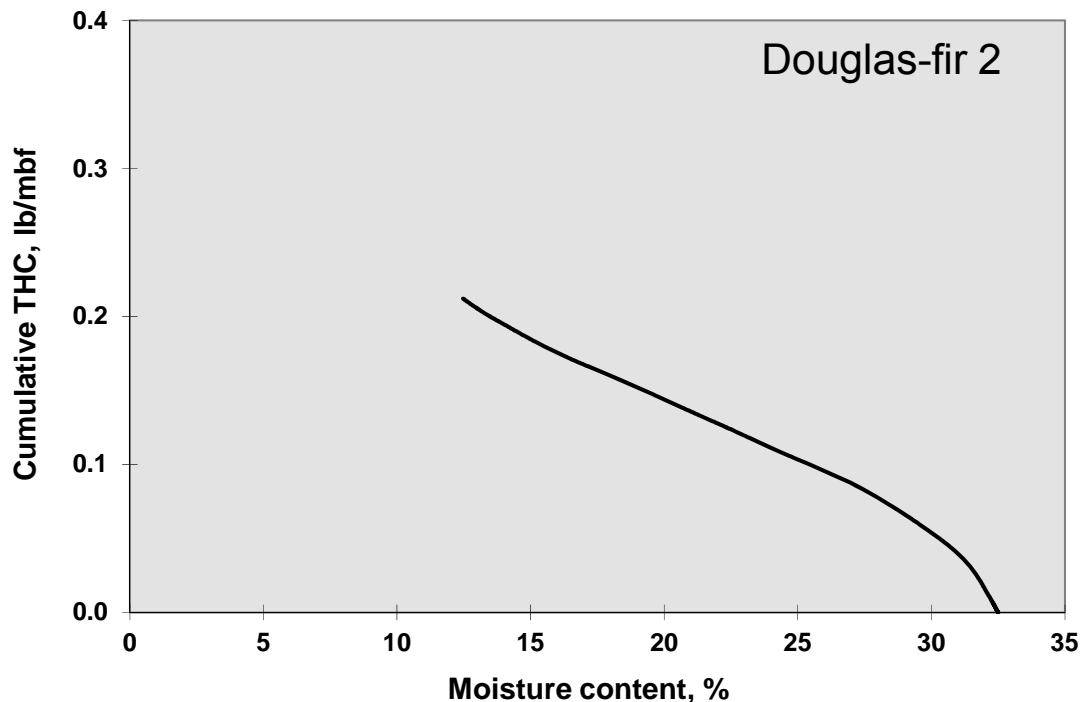


Figure 2. Cumulative hydrocarbon emissions (left scale, smooth line) and the rate of emissions (right scale, jagged line) versus time for charge 2.



**Figure 3.** Total hydrocarbon emissions as a function of wood moisture content for charge 2.

### HAPs

See Table 2, page 1, for a summary of the HAP results. Details for each sampling interval are tabulated and the HAP emissions are summarized graphically here. All emission data is presented in detail in electronic form in Appendix 2.

A summary of the kiln conditions for each sampling interval is in Table 4. A collection interval is the time the impingers were on and sampling occurred, approximately 1:25 to 1:35. An adjusted interval is the period spanning the midpoints between collection intervals, about three hours. For example, if the impingers were on from 2:00 to 3:30, 5:00 to 6:30, and 8:15 to 9:45, the 5:00 to 6:30 impinger set represents the adjusted interval from 4:15 to 7:22. The mass calculations are adjusted proportionally to represent emissions during the adjusted interval. For example, if a collection interval was 90 minutes and the adjusted interval was three hours, the amount of HAPs in the impinger is multiplied by two. Sampling occurred for approximately 50% of the drying time.

The MACT HAP emissions and the emissions of ethanol and acetic acid are shown in Table 5. The total HAP emissions were 0.026 lb/mbf for the Douglas-fir (does not include the non-HAPs, ethanol and acetic acid). Methanol is the HAP emitted in the greatest quantity (0.019 lb/mbf) followed by acetaldehyde (0.006 lb/mbf). The other HAPs are present, but comprise only 5% of all the HAPs. Phenol was not detected in any sample.

The HAP emissions as a function of time and wood moisture content during the cycle are shown in Figures 7 and 8, respectively. The rate of HAP emissions decreases with time while the kiln temperature is increasing (lines are concave upward in Figure 7), then decreases with time later in the schedule (lines are concave downward). The rate of HAP emissions per percent moisture content change are greatest at higher moisture content and decrease as moisture content decreases.

**Table 4.** Summary of HAP sampling intervals for charge 2.

Sample Run ID	Collection Interval	Adjusted Interval	Dry gas mass	Average Dry gas flow rate	Molar Humidity	Moisture Content	
	hours	hours	kg	kg/min	mol/mol	Mid %	End %
	1	1.50	2.25	0.825	0.006	0.062	32.5
2	1.50	3.00	1.173	0.007	0.204	32.3	32.1
3	1.50	3.10	7.783	0.042	0.353	31.0	27.9
4	1.50	3.25	11.778	0.060	0.312	24.9	22.3
5	1.50	3.36	11.535	0.057	0.274	20.0	17.6
6	13.55	2.85	6.929	0.040	0.251	15.7	15.0
SUM		17.82					

**Table 5.** Summary of the HAP, acetic acid, and ethanol emissions for charge 2..

Sample Run ID	Interval Endpoint	Wood Moisture Content	Unit mass leaving kiln							
			Methanol	Phenol	Ethanol	Acetic acid	Form-aldehyde	Acet-aldehyde	Propion-aldehyde	Acrolein
			lb/mbf	lb/mbf	lb/mbf	lb/mbf	lb/mbf	lb/mbf	lb/mbf	lb/mbf
1	2.25	32.4	0.0001	0.0000	0.0000	0.0003	0.00000	0.0001	0.00000	0.00000
2	5.26	32.1	0.0004	0.0000	0.0001	0.0015	0.00001	0.0006	0.00000	0.00001
3	8.36	27.9	0.0062	0.0000	0.0009	0.0319	0.00019	0.0032	0.00005	0.00011
4	11.61	22.3	0.0063	0.0000	0.0000	0.0316	0.00030	0.0011	0.00004	0.00013
5	14.97	17.6	0.0058	0.0000	0.0000	0.0270	0.00025	0.0006	0.00003	0.00010
6	17.82	15.0	0.0004	0.0000	0.0000	0.0017	0.00002	0.0000	0.00000	0.00001
7	17.82	0.0	0.0000	0.0000	0.0000	0.0000	0.00000	0.0000	0.00000	0.00000
8	17.82	0.0	0.0000	0.0000	0.0000	0.0000	0.00000	0.0000	0.00000	0.00000
		Sums:	0.019	0.000	0.001	0.094	0.001	0.006	0.0001	0.0004

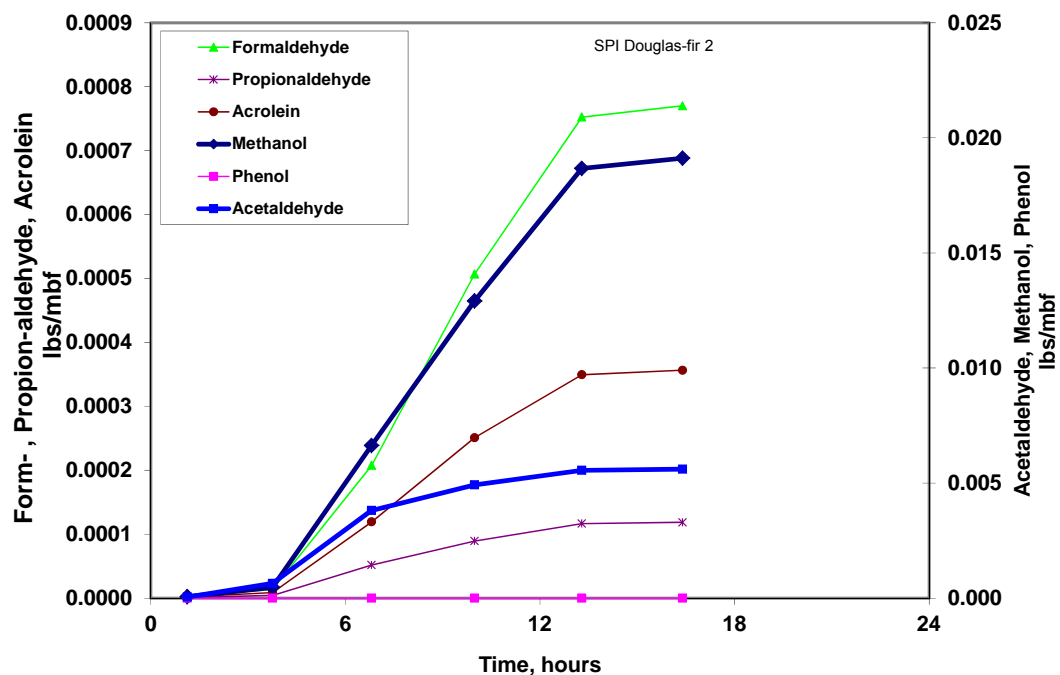


Figure 4. HAP emissions as a function of time for charge 2.

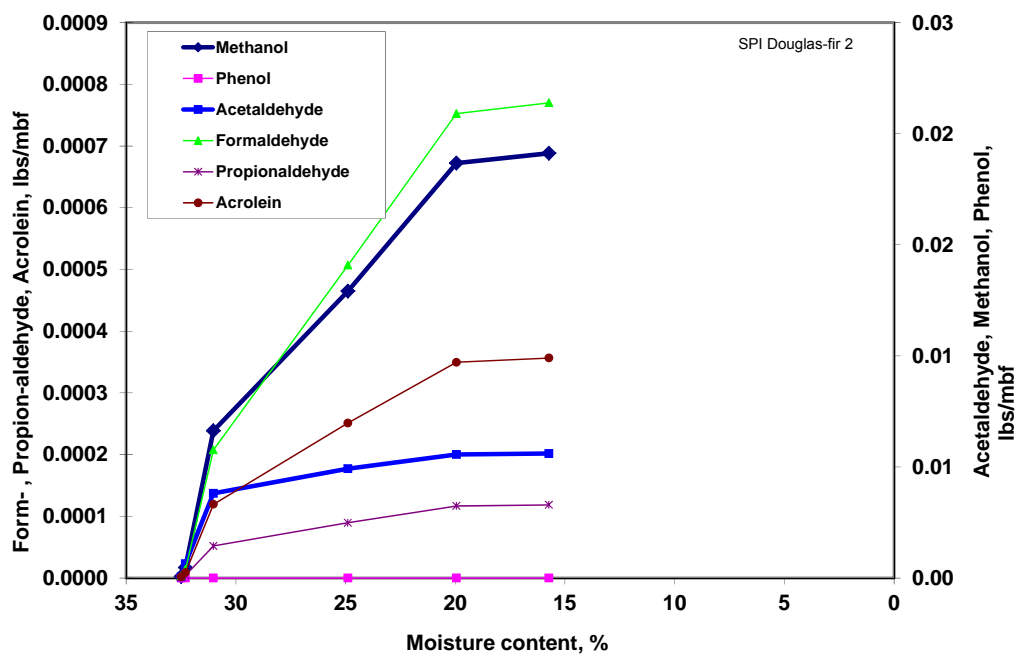


Figure 5. HAP emissions as a function of moisture content for charge 2.

The detection limits for the GC instrument were

- Methanol – 0.54 µg/mL in the aqueous phase
- Phenol – 0.42 µg/mL in the aqueous phase
- Ethanol – 0.35 µg/mL in the aqueous phase
- Acetic acid – 13.5 µg/mL in the aqueous phase
- Formaldehyde - 0.04 µg/mL in the hexane phase
- Acetaldehyde – 0.08 µg/mL in the hexane phase
- Propionaldehyde – 0.08 µg/mL in the hexane phase
- Acrolein – 0.24 µg/mL in the hexane phase

The method detection limit varies with gas flow through the impingers and the amount of solution in the impingers. Typical (based on the flow conditions and impinger volumes for sample S2-4A) method detection limits in the sampled gas (wet kiln exhaust) are

- Methanol -0.04 ppm
- Phenol -0.10 ppm
- Ethanol – 0.17 ppm
- Acetic acid – 5.0 ppm
- Formaldehyde -0.01 ppm
- Acetaldehyde - 0.02 ppm
- Propionaldehyde - 0.01 ppm
- Acrolein - mean = 0.04 ppm

All samples were above the detection limits except for the acrolein in the first sample (0.22 µg/mL with a detection limit of 0.24 µg/mL). The table below shows the HAP amounts if one-half the detection limit is substituted for all samples below the detection limit, the total HAPs remain unchanged (with rounding) at 0.026 lb/mbf.

Methanol	Phenol	Ethanol	Acetic acid	Form-aldehyde	Acet-aldehyde	Propion-aldehyde	Acrolein
lb/mbf	lb/mbf	lb/mbf	lb/mbf	lb/mbf	lb/mbf	lb/mbf	lb/mbf
0.019	0.000	0.001	0.094	0.001	0.006	0.0001	0.0004

Field spikes were run by operating two impinger trains simultaneously. An aliquot of the compounds was added to one impinger train. Spike recovery percentage is the mass of a compound detected in the lab compared to mass added to the impinger. Table 6 shows the field spike recoveries. The method requires between 70% and 130% recovery if the concentration of the analyte in the gas phase is greater than 1.5 ppm in the dry gas. On some of the spikes we exceeded the spike amounts specified in the method (which depends on the amount in the impinger). Unfortunately, one does not know the amounts in the impinger until after the fact. It is hard to spike correctly because the amounts collected in the samples are not

known until drying is completed and the sample are tested in the lab. In addition, the ratios of the amounts change from interval to interval making a single spike solution not suitable for multiple intervals. Therefore, we do two or three spikes at various levels to try to meet the method requirements.

The results for a field blank collected are shown in Table 8. None of the target compounds were detected in the blank above the detection limits except acetic acid. We experienced problems with this compound. Despite being able to create a good standard curve in water, when testing in BHA we often (but not always) see a peak at the time when acetic acid appears on the chromatogram.

Duplicate samples were run by operating two impinger trains simultaneously. The results of duplicates are shown in Table 7. The percentage is the difference between the gas concentrations detected by each impinger. Phenol was not detected so duplicates could not be compared. Differences ranged from 1.2 to 28%, all within the limits of the method.

**Table 6.** Example of spike test results.

Alcohol Spike									
Run	Mass in impinger				Impinger flow mL/min	Mass corrected for flow			
	Methanol µg	Phenol µg	Ethanol µg	Acetic µg		Methanol µg	Phenol µg	Ethanol µg	Acetic µg
6	1003.8	0.0	28.9	3865.9	405.5	537.1	0.0	15.5	2068.5
603	7787.9	672.9	1550.6	7184.3	217.0	7787.9	672.9	1550.6	7184.3
Spike mass g	Spike concentrations					Spike recoveries			
	Methanol µg/mL	Phenol µg/mL	Ethanol µg/mL	Acetic µg/mL		Methanol %	Phenol %	Ethanol %	Acetic %
1.87	4300.2	328.1	800.7	2247.4		90.2	109.7	102.5	121.7
Aldehyde Spike									
Run	Mass in impinger				Impinger flow mL/min	Mass corrected for flow			
	Form-aldehyde µg	Acet-aldehyde µg	Propion-aldehyde µg	Acrolein µg		Form-aldehyde µg	Acet-aldehyde µg	Propion-aldehyde µg	Acrolein µg
6	39.3	98.3	4.2	15.6	405.5	43.3	108.3	4.6	17.2
602	998.9	955.0	308.4	240.4	446.7	998.9	955.0	308.4	240.4
Spike mass g	Spike concentrations					Spike recoveries			
	Form-aldehyde µg/mL	Acet-aldehyde µg/mL	Propion-aldehyde µg/mL	Acrolein µg/mL		Form-aldehyde %	Acet-aldehyde %	Propion-aldehyde %	Acrolein %
1.73	409.2	293.6	114.3	87.4		89.0	109.8	101.2	97.3

Table 6. Continued

Alcohol Spike									
Run	Mass in impinger				Impinger flow	Mass corrected for flow			
	Methanol	Phenol	Ethanol	Acetic		Methanol	Phenol	Ethanol	Acetic
	µg	µg	µg	µg	mL/min	µg	µg	µg	µg
7	1099.0	0.0	8.5	4048.2	404.5	1200.9	0.0	9.2	4423.6
702	10931.3	866.2	1921.0	8900.7	442.0	10931.3	866.2	1921.0	8900.7
Spike									
Spike mass	Spike concentrations					Spike recoveries			
	Methanol	Phenol	Ethanol	Acetic		Methanol	Phenol	Ethanol	Acetic
g	µg/mL	µg/mL	µg/mL	µg/mL		%	%	%	%
2.43	4300.2	328.1	800.7	2247.4		93.1	108.6	98.3	82.0

Aldehyde Spike									
Run	Mass in impinger				Impinger flow	Mass corrected for flow			
	Form-aldehyde	Acet-aldehyde	Propion-aldehyde	Acrolein		Form-aldehyde	Acet-aldehyde	Propion-aldehyde	Acrolein
	µg	µg	µg	µg	mL/min	µg	µg	µg	µg
7	52.1	153.9	5.6	21.3	404.5	27.6	81.4	3.0	11.3
703	1145.7	1221.9	495.0	366.8	214.1	1145.7	1221.9	495.0	366.8
Spike									
Spike mass	Spike concentrations					Spike recoveries			
	Form-aldehyde	Acet-aldehyde	Propion-aldehyde	Acrolein		Form-aldehyde	Acet-aldehyde	Propion-aldehyde	Acrolein
g	µg/mL	µg/mL	µg/mL	µg/mL		%	%	%	%
2.51	409.2	293.6	114.3	87.4		71.7	102.0	113.0	106.8

Table 7. Results for the field blank.

Field blank							S2-8C
Methanol	Phenol	Ethanol	Acetic acid	Form-aldehyde	Acet-aldehyde	Propion-aldehyde	Acrolein
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
0.19	0.00	0.00	22.92	0.02	0.00	0.00	0.00

Table 8. Results for duplicate runs.

Duplicate									
Run	Mass in impinger								Impinger flow
	Methanol	Phenol	Ethanol	Acetic acid	Form-aldehyde	Acet-aldehyde	Propion-aldehyde	Acrolein	
	µg	µg	µg	µg	µg	µg	µg	µg	mL/min
3	1340.9	0.0	193.1	6935.4	42.2	690.7	10.3	24.1	400.9
302	1240.5	0.0	167.8	6466.2	46.8	804.7	10.9	27.3	438.7
Difference, %	16.8	#DIV/0!	22.9	16.0	1.2	6.3	3.9	3.4	

Duplicate									
Run	Mass in impinger								Impinger flow
	Methanol	Phenol	Ethanol	Acetic acid	Form-aldehyde	Acet-aldehyde	Propion-aldehyde	Acrolein	
	µg	µg	µg	µg	µg	µg	µg	µg	mL/min
8	1911.0	0.0	0.0	4352.4	58.1	170.2	8.8	25.4	403.6
802	2070.8	0.0	0.0	4492.2	57.3	168.8	7.3	24.6	446.2
Difference, %	2.0	#DIV/0!	#DIV/0!	6.9	11.4	10.9	28.7	13.3	

## 4. Control system and operating conditions

A schematic of the kiln is shown in Figure 6(top). The kiln box is approximately 4' by 4' by 4'. It is indirectly heated by steam. Four dry-bulb thermocouples and two wet-bulb thermocouples are located on the entering-air side of the load. The dry-bulb thermocouples are spaced in a grid. The two wet-bulb thermocouples are under a single sock at the center of the entering-air side of the load.

### Humidity control

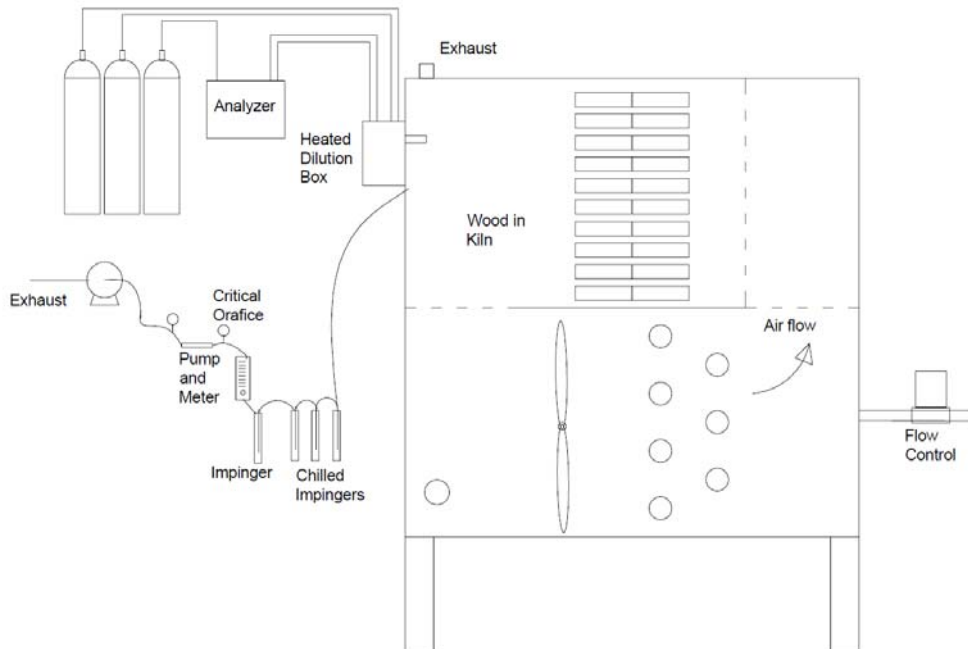
A 200 L/min MKS mass flow meter controlled the amount of air entering the kiln. It was factory calibrated and checked using a bubble meter. The amount of air entering the kiln is based on the wet-bulb temperature - if it is above setpoint, the airflow is increased and if it is below setpoint the airflow is decreased. This is analogous to venting for a commercial kiln. A minimum of 5 L/min entered the kiln at all times, more than removed by the analyzer (1.6 L/min). Putting air into the kiln at a rate of 100 L/min causes the pressure in the kiln to be 60 to 130 Pa above ambient, depending on location in the kiln (high-pressure or low-pressure side). Thus, any fugitive leakage should be out of the kiln. Two additional flow meters can be manually set to provide additional airflow. Flow meter two or three was not used during the second Douglas-fir charge.

### Temperature control

Temperature in the kiln is controlled by indirect steam heating. When the dry-bulb temperature is below setpoint, the steam pressure in the coil is increased. When it is above setpoint, steam flow to the coil is reduced.

The dry- and wet-bulb temperatures recorded for each charge are shown in Figure 6. The schedule provided by the mill is also shown. The poor kiln control in charge one is evident and this is the main reason we do not include data from this charge. The agreement between the recorded temperatures and the drying schedule is very close for charge 2.





**Figure 6.** Schematic of kiln and sampling system (top) and photo of charge 2 in kiln (bottom).

## 5. Production-related parameters

### Wood quantity

The wood properties were determined using the nominal wood dimensions (2x4 in this case) which provides for 0.66 board feet per lineal foot. There were 33 pieces in the kiln at 44" in length. The board footage was therefore 80 board feet. This quantity was used to express the emissions from the drying cycle on a production basis of lb/mbf (pounds per thousand board feet).

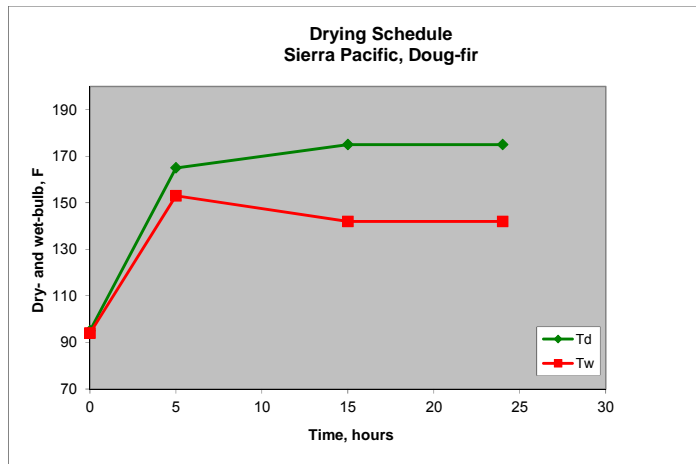
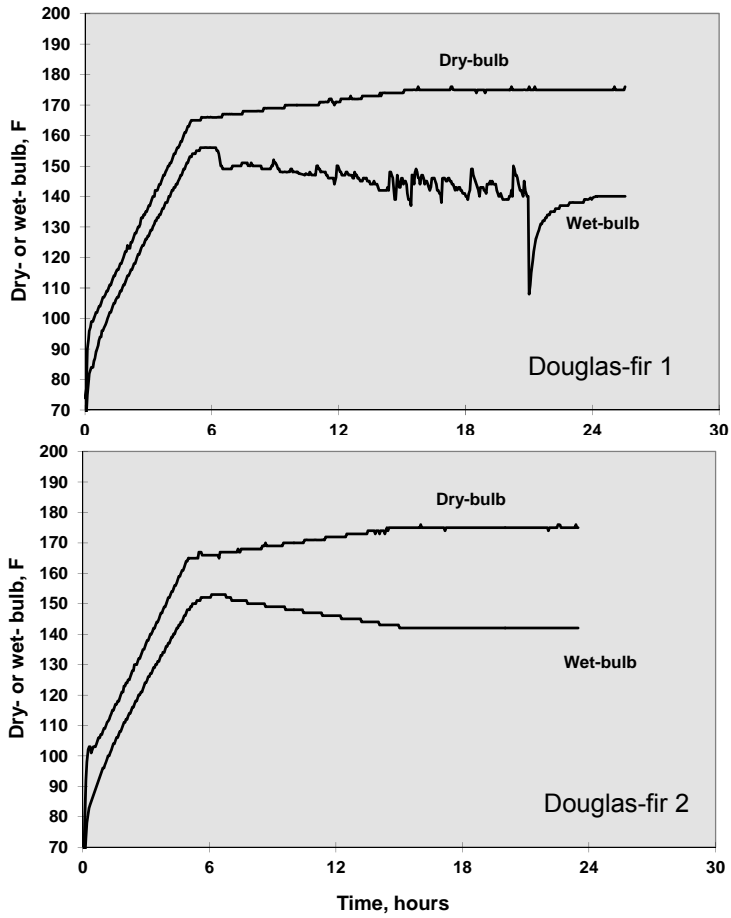
### Wood properties

The wood property measurements are shown in Table 10. Individual measurements can be found in the Excel file "Weights, SPI, Run \*.XLS" in Appendix 2.

Heartwood percentage was determined by estimating the heartwood percent at each end of the board and averaging all pieces.

The average ring count was determined by counting the rings over a 1" radial distance and averaging for all boards.

The knots were counted on the top face of each board and averaged. This was a count of all knots. Knot diameter is an average of the knots present. The knots occupied approximately 0.7% of the boards' faces.



**Figure 7.** Schedules followed (top) and schedule provided by mill (bottom).

**Table 9.** Wood properties.

Charge	Knots		Heartwood	Ring count	Pith In
	Number	Size			
	#	In.	%	#/in	# with pith
Douglas-fir 2	5.5	0.5	100	3.3	10

## 6. Test methods

### Charge Sequence

The lumber was unwrapped and 2" were trimmed from each end of each board to give 44" samples. These were then weighed, placed in the kiln and dried. At the end of drying the wood was weighed, oven dried, and reweighed so initial and final moisture contents could be determined by ASTM D4442 (oven-dry method).

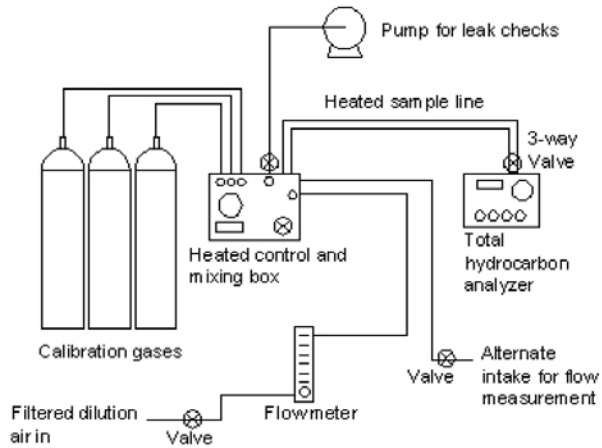
### Sampling Methodologies

#### *Hydrocarbon*

Sampling for total hydrocarbon is done directly from the kiln as shown in Figure 6 (top). The concentration obtained from the hydrocarbon analyzer and the amount of air entering the kiln allow the total hydrocarbon emissions to be calculated.

Figure 8 shows the hydrocarbon sampling system. Unlike stack testing, all necessary equipment is located in a lab and flows are controlled with valves. The sample is withdrawn from the kiln under the assumption that the gas in the kiln is well-mixed and that the composition in the kiln near the exhaust is the same as the composition of the exhaust. The THC sample was drawn from the kiln through a heated sampling line (239-249°F) and into a heated dilution/filter box. The box was heated to 257°F. Heated dilution gas can be added to the hydrocarbon sample gas to lower the gas moisture content to the detector. Dilution air would have been used when the gas moisture content in the kiln was greater than 15% so that the air moisture content to the detector remained less than 15%. The sample line from the box to the analyzer was heated to 266°F. The 3-way valve at the back of the analyzer was heated to 275°F.

The fuel gas was hydrogen. The span gas was EPA Protocol 197 ppm propane in air, the mid-gas was EPA Protocol 50 ppm propane in air. The zero gas was <0.1 ppm air. Detailed sampling procedures are in Appendix 1.



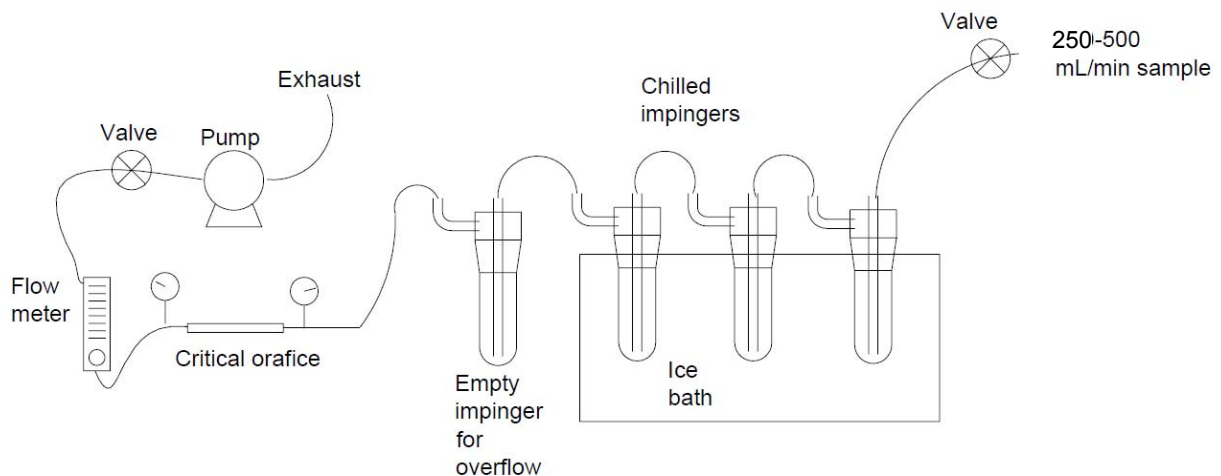
**Figure 8.** Schematic of heated filter box with air dilution system, heated sample line, and analyzer (top). Sample enters heated box from back of drawing through a heated sampling line. Photo (left) of heated box and (right) of calibration gas valves and dilution air valves. Line to analyzer is the green line on the left in the left photo.

## HAPs

The sampling train for NCASI Method 105 is shown in Figure 9. The impingers were in a glycol solution maintained at -1 C. Prior to each sampling interval, the impingers were laboratory-washed and 10 to 15 mL of BHA solution were added to the first and second impingers. The third impinger was left empty. The fourth impinger was present in the system to prevent any overflow from reaching the critical orifice. The system was then assembled and a vacuum check was performed with the valves at each end closed. Less than 1" Hg of pressure change over 2 minutes was acceptable. This was met for each interval. The flow rate through the system was then measured using a Gilibrator flow meter to take four flow readings at the probe tip. This was approximately 240-500 mL/min, depending on the sampling train. A valve at the probe tip was then turned to sample from the kiln and the sampling interval begun. The collection interval time was approximately 1:30 and an interval was started approximately every three hours.

The flow rate was measured after each sampling interval. The fluid in the three impingers was weighed and placed in a glass bottle. The impingers were then rinsed with 10 mL of water followed by 3 to 5 mL of hexane. The rinses were also placed in the bottle and it was sealed. Samples were kept refrigerated and in the dark until lab analysis was done. Lab analysis was done within one week of sample collection.

The local airport altimeter setting and the lab temperature were recorded at the beginning and end of each interval so the flow rates could be adjusted to standard conditions.



**Figure 9.** HAPs sampling train.

## 7. Analytical procedures

### Hydrocarbon

Leak checks of the VOC sampling train were conducted before and after the charge was dried. A valve was closed at the probe tip and a 3-way valve was closed at the back of the analyzer. All components from just behind the probe tip to the valve at the back of the analyzer were placed under a 15-20 inHg vacuum. Less than one inHg pressure change during two minutes is acceptable and this was met.

Total flow and sample flow to the analyzer were checked using an NIST-traceable flow meter. Total flow is measured with the dilution gas off and is equal to both the sample flow from the kiln when the dilution is off and the total volume drawn by the analyzer. Sample flow is measured with dilution gas on (if used for that interval) and is the volume of gas sampled from the kiln when the dilution gas is on. This was done at the beginning and end of each sampling interval. The meter was attached to the system near the probe tip within the heated box. The valves were repositioned so that the sample came from the flow meter rather than the kiln. Readings of flow were made with the dilution gas both off and on. The flow readings were verified by observing the analyzer reading for span gas with the dilution gas off and on. The dilution ratio calculated based on the analyzer readings was always within 5% of that determined by the flow meter and often within 2%. Note that dilution was not actually used for the entire test because the kiln wet-bulb was low enough initially that the gas moisture content was less than 15% until the wet-bulb temperature was greater than 130°F.

Calibration of the zero and span of the detector was done at the beginning of each run (about every three to six hours). The calibration gas was introduced by setting the valves so the calibration gas entered the system in the white heated mixing box at ambient pressure. The calibration was checked at the end of each run with no adjustments made to the instrument's zero or span during the run. A span drift less than 10% of the span value was acceptable. A zero drift of less than 3% of the span value was acceptable. A total calibration drift less than 10% was acceptable for a sampling run. These criteria were met.

### HAPs

#### *Lab analysis for aldehydes*

Aldehyde standards were prepared by the volumetric dilution of neat aldehydes in water (to 250 ppm for formaldehyde, propionaldehyde, acrolein and acetaldehyde). This stock solution was mixed a BHA solution [from ortho-benzylhydroxylamine hydrochloride (BHA) and deionized water (30g BHA per

liter of water)]. The stock solution-BHA mixture was vigorously agitated and allowed to sit for six hours to allow for derivatization of the aldehydes into aldoximes. The derivatized aldehyde solution was extracted with three aliquots of hexane to create a 400 ppm stock solution in hexane. This was volumetrically (but calculations based on mass) diluted to make standards down to 0.2 ppm. 1 mL aliquots were placed in GC autosampler vials with 20  $\mu$ L of 8800 ppm nitrobenzene added to each as an internal standard.

The samples (from the bottles collected in field) were prepared by three extractions in a separatory funnel. The first extraction was with the hexane added in the field. The second extraction was with a 7-mL aliquot of hexane after using it to rinse the sample bottle. The final extraction was done with 7 mL of clean hexane. The total hexane volume was approximately 18-20 mL. The volumes of the two phases were calculated from their weights. A 1 mL aliquot of the hexane fraction was transferred to an autosampler vial and spiked with internal standard.

The analytical instrument was a Shimadzu GC model 2010 with a flame thermionic detector (FTD), the Shimadzu equivalent of a nitrogen phosphorous detector (NPD). The column was a 105-meter Restek RTX-5 capillary with a 0.25 mm outside diameter and a stationary phase thickness of 0.25  $\mu$ m. The oven schedule was: 2 minutes at 120°C, 2°C/min ramp to 160°C, 40°C/min ramp to 220°C and 6.5 minutes at 220°C. The column flow was 25 cm/sec, with 3 mL/min septum purge, and a 1:10 split ratio with a glass wool packed split injection liner. The detector make up He was set to 20 mL/min and the H<sub>2</sub> was set to 3 mL/min. The air was set to 140 mL/min, and the source current was set to 2 pA. The He and H<sub>2</sub> gases were grade 5 and the air was grade 0.1. The injector temperature was 200°C and the detector temperature 280°C. An AOC-20i autosampler was used to perform 1  $\mu$ L injections using a 10  $\mu$ L syringe with a steel plunger.

### ***Lab analysis for alcohols***

Standards for methanol, phenol, ethanol, and acetic acid were prepared by the volumetric dilution of neat reagents in water. The mixed standard was prepared at a concentration of 1000 milligrams per liter (mg/L). Additional standards were prepared by the volumetric dilution of the mixed standard at a range from 1 mg/L to 500 mg/L. Aliquots of these were placed into autosampler vials with 20  $\mu$ L of 20,000 ppm cyclohexanol internal standard..

Samples were prepared by transferring aliquots of the previously hexane-extracted aqueous fractions into autosampler vials and adding internal standard. The analytical instrument was a Shimadzu GC model 2010 with a FID detector. The column was a 60-meter Restek Stabilwax capillary with a 0.53 mm outside diameter and a stationary phase thickness of 1.5  $\mu$ m. The oven schedule was: 3 minutes at 60°C, 10°C/min ramp to 80°C, 3 minutes at 80°C, 10°C/min ramp to 230°C, and 10 minutes at 230°C. The column flow was 30 cm/sec, with 3 mL/min septum purge, and a 1:10 split ratio with a glass wool packed split injection liner.



The detector make up He was set to 25 mL/min and the H<sub>2</sub> was set to 50 mL/min. The air was set to 500 mL/min. The He and H<sub>2</sub> gases were grade 5 and the air was grade 0.1. The injector temperature was 175°C and the detector temperature 250°C. An AOC-20i autosampler was used to perform 1 µL injections using a 10 µL syringe with a PTFE plunger.

## 8. Field data sheets and sample calculations

### Field data sheets

Samples of field data sheets are shown in Figures 10 to 13. All field data sheets are in Appendix 2 this report in electronic format (pdf).

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FIELD DATA SHEET FOR TOTAL HYDROCARBON ANALYZER - BEFORE

**BACKGROUND INFORMATION**

Event (kiln charge): Sierra Pacific - 2      Dry-bulb temperature: 175

Run: 8      Wet-bulb temperature: 140

Operator: MRM      Target Dilution Ratio (TDR): \_\_\_\_\_

Date: 5-15-03      Laboratory temperature: 75 °F

**ANALYZER CALIBRATION** (change pots to calibrate)      [ Valves 1, 2 = off; 3=on; 4=vent ]

	Range	Analyzer, reading	Computer, ppm	Within range	Pot settings
zero	3	200 (0)	000 (0)		482
span	3	197 (1.98)	197 (198.8)		390
mid	3	0.50 (0.50)	0.50 (0.50)	45 to 55	
mid	2	4.70 (0.03)			

Reset range to 3. Range: 3

**SET DILUTION FLOW BEFORE RUN** (do not change pots)

Total flow rate (TFR): 1794 mL/min [ Valves 1, 2, 3 = off; 4=meter ]

Target dilution flow rate (TDFR) \_\_\_\_\_ mL/min [ TFR x (1 - DR) ]

Set and read dilution meter: 1.8 scfh [ scfh = mL/min \* 0.00212 ]

Sample flow rate (SFR): 807.5 mL/min [ 1 = on; 2, 3 = off; 4=meter ]

**CHECK DILUTION FLOW BEFORE RUN** (do not change pots)      [ 1, 3=on; 2=off; 4=vent ]

	Analyzer	DR <sub>Span</sub> [ Span/Output/Span ]	DR <sub>Flow</sub> [ SFR / TFR ]	Difference, % 100*(DR <sub>Span</sub> - DR <sub>Flow</sub> )/DR <sub>Flow</sub>
Span <sub>Calculated</sub>	0.94	0.477	0.460	3.5

START TIME: 03:12 (clock)      [ 1, 2, 5 = on; 3, 4 = off; tank valves off ]

ANALYZER RANGE: 3      [ 0.6 < reading < 7.5 ]

ANALYZER RANGE ON COMPUTER (set to match analyzer): 3  
*To Range 2 at 03:13*

FIELD DATA SHEET FOR TOTAL HYDROCARBON ANALYZER - AFTER

Operator: MRM      Event (kiln charge): Sierra Pacific - 2

Run (sample): 6      Laboratory temperature: 74 °F

END TIME: 7:50

Range setting on Analyzer: 2      on Computer: 2

Reset range to 3. Range: 3

**CHECK DILUTION FLOW AFTER RUN** (do not change pots)      [ 1, 3=on; 2=off; 4=vent ]

	Analyzer reading	Computer, ppm
Span <sub>Calculated</sub>	0.95	0.95

Sample flow rate (SFR) : 802.8 mL/min [ 1 = on; 2, 3 = off; 4=meter ]

Read dilution meter: 1.8 scfh

Total flow rate (TFR): 1732 mL/min [ 1, 2, 3 = off; 4=meter ] (label print out)

Dilution ratio (DR<sub>Flow</sub>): 0.466 [ SFR / TFR ]

**CHECK OF ANALYZER CALIBRATION** (do not change pots)      [ 1, 2=off; 3=on; 4=vent ]

	Analyzer reading	Computer, ppm	Within range	Pot settings
span	2.02	2.01	187 to 206	390
mid	0.51	0.51	45 to 55	
zero	0.01	0.00	-5 to +5	482

Dilution ratio (DR<sub>Span</sub>): 0.470 [ Span<sub>Calculated</sub> / Span ]

Dilution ratio difference: 0.8 % [ 100\*(Abs(DR<sub>Span</sub> - DR<sub>Flow</sub>))/DR<sub>Flow</sub> ]

End time for check: 8:03

Comments: \_\_\_\_\_

Figure 10. Sample of field data sheet for hydrocarbon analyzer.

**FIELD DATA SHEET, 105 HAPS MEASUREMENT**  
**TRAIN #2 - BEFORE SAMPLE**

Operator: MM Event (kiln charge): Sierra Pacific - 2  
Date: 5-15-13 Run (sample): 6 -B

Altimeter setting: 3013 inHg  
Isopropanol rinse or lab wash:

**IMPINGER WEIGHTS** (add BHA to impingers)

	Dry Weight, g	Wet Weight, g	BHA added, g
Impinger #2A	41.92	52.30	(-15 mg)
Impinger #2B	40.88	55.36	(-15 mg)
Impinger #2C	40.55	-----	-----
Total BHA added:			

Optional Spike: 1.73 g Spike type: Alcohol  Aldehyde

(take impingers to kiln from scale)

Lab (ambient) temperature 75 °F  
Leak check: Vacuum 16.5 inHg 75 Vacuum check ok  
Sample flow rate: 50.1 mL/min [Average of 4. Label printout]  
Set probe valve to sample from kiln  
CLOCK TIME: 2:57  
ELAPSED TIME: 15:57

Is the probe valve set to sample?  
Is gas flowing (look at flow meters)?

**FLOW METER READINGS DURING TEST** (every 20 minutes)

Clock time	3:15	3:47
Vacuum, inHg	28	29
Flow rate, mL/min	46.8	46

**FIELD DATA SHEET, 105 HAPS MEASUREMENT**  
**TRAIN #2 - AFTER SAMPLE**

Operator: MM Event (kiln charge): Sierra Pacific - 2  
Run (sample): 6 -B

Altimeter setting: 3011 inHg

Lab (ambient) temperature: 77 °F  
Change position of valve at the kiln Clock time: 4:30  
Elapsed time: 17:30

Set probe valve to measure sample flow rate. Shut off other flows using lower valves.  
Sample flow rate: 49.9 mL/min [Average of 4. Label printout]  
(take impingers to scale from kiln)

Empty bottle weight: 134.16 g (with lid)

**IMPINGER WEIGHTS** (transfer sample to bottle)

	Wet Weight, g	Dry Weight, g	Water removed, g
Impinger #2A	62.78		
Impinger #2B	55.46		
Impinger #2C	40.61		

Bottle weight without impinger rinse: 174.37 g (with lid)  
(rinse impingers with 10 mL DI water)  
Bottle weight with impinger rinse: 185.01 g (with lid)  
(rinse impingers with 5 mL hexane)  
Filled bottle weight: 189.29 g (with lid)

Fluids lost during handling: 0 mL [estimate]

Comments:

Figure 11. Sample of field data sheet for HAPs collection.

Charge: Sierra Pacific - 2 Date: 5/14/2013 Time: 2

Clock time	Elapsed time	Run #	Temperatures										Flows			Vacuum					
			T dry °F	T wet °F	Valve °C	G. line °C	Box °C	Def. Box °C	Line 2 °C	Line 1 °C	Chiller °C	Flow 1 L/min	Flow 2 L/min	Flow 3 L/min	Dilution SCFM	Line 1 m/min	Line 2 m/min	Line 3 m/min	Line 1 inHG	Line 2 inHG	Line 3 inHG
00:25	13:25	5	173	144	130	125	122	120	117	115	-1	450	---	---	2.2	450	---	---	29	---	---
01:16	14:16	5	174	143	130	125	122	120	117	115	-1	47.4	---	---	2.2	---	---	---	---	---	---
02:31	15:31	7	175	142	130	125	121	120	117	115	-1	38.6	---	---	2.2	---	---	---	---	---	---
03:15	16:15	6	175	142	131	125	125	120	117	115	-1	28.5	---	---	1.8	400	450	350	28	28	25
04:49	17:51	8	175	142	130	125	122	120	117	115	-1	21.0	---	---	1.8	---	---	---	---	---	---
06:02	19:02	8	175	142	130	125	122	120	117	115	-1	20.2	---	---	1.8	400	450	350	28	28	28
6:21	19:21	8	175	142	130	125	122	120	117	115	-1	20.9	---	---	1.8	400	450	350	28	28	28
7:08	20:08	8	175	142	130	125	122	120	117	115	-1	15.7	---	---	1.8	400	450	350	28	28	28
8:02	21:02	8	175	142	130	125	124	120	118	115	-1	14.1	---	---	---	---	---	---	---	---	---
8:56	21:56	9	175	142	131	125	122	120	117	115	-1	11.1	---	---	1.7	---	---	---	---	---	---
9:47	22:47	9	175	142	130	125	123	120	117	115	-1	10.1	---	---	1.7	400	450	---	28	28	---
10:08	23:08	9	175	142	130	125	119	120	117	115	-1	8	---	---	1.7	400	450	---	28	28	---

Figure 12. Sample of kiln log data sheet.

Section	Interval	Flow Average	# Samples
GILIBRATOR 2 MET V4.4 DATE	01	444.1	01
	02	443.4	02
	03	447.2	03
	04	452.8	04
PUMP S/N	01	495.7	01
	02	498.3	02
	03	480.0	03
	04	501.6	04
GILIBRATOR 2 MET V4.4 DATE	01	239.4	01
	02	239.4	02
	03	241.2	03
	04	241.4	04
PUMP S/N	01	495.2	01
	02	496.9	02
	03	497.6	03
	04	499.4	04
GILIBRATOR 2 MET V4.4 DATE	01	457.4	01
	02	461.1	02
	03	457.0	03
	04	459.9	04
PUMP S/N	01	500.7	01
	02	499.8	02
	03	501.2	03
	04	508.3	04
GILIBRATOR 2 MET V4.4 DATE	01	238.7	01
	02	238.5	02
	03	243.9	03
	04	243.6	04
PUMP S/N	01	757.5	01
	02	765.6	02
	03	759.4	03
	04	762.7	04
GILIBRATOR 2 MET V4.4 DATE	01	1733	01
	02	1733	02
	03	1733	03
	04	1733	04

Figure 13. Sample of flow measurement record.

## Calculations

The “FlowCalc” worksheet in the Excel files “Kiln, SPI, Run \*.XLS in Appendix 2 shows the calculations for each 3-minute interval during the charges. Column A is a reading number. Columns B and C are the clock and charge times, respectively. Columns D/E and F/G are the average dry- and wet-bulb temperatures.

## Humidity

Column H is the vapor pressure ( $P_{vp}$ , Pa) of water at the wet-bulb temperature. The absolute humidity ( $AbHum$ ,  $kg_{water} \cdot kg_{air}^{-1}$ ) is shown in column I and the molal humidity ( $mol_{water} \cdot mol_{air}^{-1}$ ) in column J. These are calculated based on the dry-bulb temperature ( $T_d$ , °C) and wet-bulb temperature ( $T_w$ , °C),

$$P_{vp} = P_{ambient} * 10^{(16.373 - 2818.6/(T_d+273.16) - 1.6908 * \text{LOG}_{10}(T_d + 273.16) - 0.0057546 * (T_d + 273.16) + 0.0000040073 * (T_d + 273.16)^2)}$$

$$AbHum = (MW_{water} / MW_{air}) * (1 / (P_{kiln}/P_{vp} - 1)) - ((T_d - T_w) * R_{psy}) / \lambda$$

$$MolHum = AbHum * MW_{air} / MW_{water}$$

where MW are molecular weights ( $kg \cdot kmol^{-1}$ ),  $R_{psy}$  is the psychrometric ratio ( $0.95 \text{ kJ} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$ ), and  $\lambda$  is the latent heat ( $2419 \text{ kJ} \cdot \text{kg}^{-1}$ ).

## Flows

The volumetric dry gas flow rate (DryGasV,  $L \cdot \text{min}^{-1}$ ) in column K is the flowmeter reading adjusted for the meter calibrations and the molal humidity of the entering

gas. This is in standard (at 0°C) liters per minute. In column L this has been converted to a mass flow rate (DryGasM, kg•min<sup>-1</sup>) and in column M is the same information is expressed as a molal flow rate (DryGas, kgmol•min<sup>-1</sup>). These values are for the dry gas vented from the kiln.

$$\text{DryGasV} = (\text{FlowMeter1} + \text{FlowMeter2} + \text{FlowMeter3}) * (1/(1+\text{MolHum}_{\text{In}}))$$

$$\text{DryGasM} = (\text{DryGasV L}\cdot\text{min}^{-1}) * 1/(22.4 \text{ m}^3\cdot\text{kgmol}^{-1}) * \text{MW}_{\text{air}} / (1000 \text{ L}\cdot\text{m}^{-3})$$

$$\text{DryGas (kgmol/min)} = \text{DryGasM} / \text{MW}_{\text{air}}$$

The water removal rate (WaterVented, g•min<sup>-1</sup>) (column N) is calculated from the humidity (column I) and the gas flow (column L). The total water (column O) is an integration of column N over time.

$$\text{WaterVented} = (\text{AbHum} - \text{AbHum}_{\text{In}}) * (\text{DryGasM} * 1000 \text{ g}\cdot\text{kg}^{-1})$$

### ***Moisture content***

The moisture content of the wood at each three-minute interval (column P) was determined by reducing the moisture content of the wood from the previous value by accounting for the amount of water leaving the kiln during the interval.

$$\text{MC} = \text{MC}_{\text{Previous}} - 100 * (\text{WaterVented} / (1000 \text{ g}\cdot\text{kg}^{-1}) / \text{ODWoodWt})$$

This amount is then adjusted by adjusting the wet-bulb temperature to make the ending moisture content match that measured by ASTM D4222.

### ***Hydrocarbon***

The original total hydrocarbon analyzer reading is shown in column Q. In column R this has been corrected to compensate for the range setting switch on the analyzer. Also in column R, the THA data between sampling runs (rows labeled “test” in column AA) has been adjusted to the average of the data during the 9-minute period before and the 9-minute period after the analyzer testing and calibration time.

The dilution THA (column S) is the corrected THA reading divided by the dilution ratio (from column AA). In column T we have the opportunity to compensate for the effect of moisture on the JUM detector. Column T equals column S because dilution was used and no compensation was made. Finally in column U, the hydrocarbon concentration is converted to a dry gas basis concentration using the molar humidity (column J).

$$\text{THC}_{\text{Dry, ppm}} = \text{THC} * (1 + \text{MolHum})$$

In column V, the hydrocarbon flow rate ( $\text{THC}_{\text{Vented}}$ ,  $\text{g}_{\text{Carbon}} \cdot \text{min}^{-1}$ ) is calculated in a manner analogous to the water flow rate using the dry gas flow rate and the hydrocarbon concentration.

$$\text{THC}_{\text{Vented}} = \text{DryGas} * (\text{THC}_{\text{Dry}} / 10^6) * \text{MW}_{\text{Propane}} * (1000 \text{ g} \cdot \text{kg}^{-1}) * (0.81818 \text{ g}_{\text{Carbon}} \cdot \text{g}_{\text{Propane}}^{-1})$$

Column W is the integral of column V over time, the cumulative hydrocarbon released up to that point in the schedule (in grams). Column X is the cumulative unit emissions, that is, column W divided by the oven-dry weight of the wood in the kiln. Column AI is the cumulative emissions in pounds per thousand board feet and column AH is the rate of emissions release ( $\text{lb} \cdot \text{mbf}^{-1} \cdot \text{hr}^{-1}$ )

Column Z indicates the hydrocarbon sampling run and column AA is the dilution ratio during that run.

The remaining columns are used not used in the hydrocarbon calculations. They are for graphing shown on other worksheets in the workbook.

At the end of the FlowCalc spreadsheet (at the bottom) are summaries by run of the flow data for the total hydrocarbon run intervals (interval summary button will reposition spreadsheet).

Moisture content and board weight data are on the “Define” worksheet and the original data are in the files named “Weights, Wood.XLS”.

## **HAPs**

Within the file “HAPs, SPI Run \*.xls”, the summary page presents the data by run interval. The data is copied from the other pages to provide a concise summary.

The “Field Data” page is data transcribed from the field data sheets (copies of the sheets are included in Appendix 2 in PDF format) and includes the ambient pressure, lab temperature, flow rate through the impingers, and run start and stop times.

The “Laboratory Data” page contains results from the lab analysis for HAPs. These values come from the files “AQU, GC Sheet, SPI Run \*.xls” and “ALD, GC Sheet, SPI Run \*.xls” in the “Lab Data” directory. The GC retention times and peak areas and the GC calibrations are in these files.

On the “Impinger Calculations” page, the field data and laboratory data are used to give a dry gas flow rate through the impingers (columns J and K) and the mass of target compounds in the impingers (columns L to Q). Flow rates were adjusted to standard conditions in columns F and G.

$$\text{ImpgrFlow}_{\text{Std}_m\text{L}} = \text{ImpgrFlow} * (273.16\text{K} / T_{\text{meter}}) / (P_{\text{meter}} / 101.33 \text{ kPa})$$

A dry gas flow rate is calculated in columns H and I

$$\text{ImpgrFlow}_{\text{Dry\_mL}} = \text{ImpgrFlow}_{\text{Std\_mL}} / (1 + \text{MolHum})$$

The average of the before and after gas flow measurements through the impingers (column J) is then converted to a mass basis in column K.

$$\text{ImpgrFlow}_{\text{Dry\_g}} = \text{MWair} * \text{ImpgrFlw}_{\text{Dry\_mL}} * P / (T * R)$$

Finally, the mass of each compound recovered from the impinger is calculated in columns L to S.

$$\text{Mass}_i = (\text{Concentration}_i) / (\text{DenSolvent}) * (\text{Mass solvent})$$

The “Kiln Calculations” page uses a ratio of the dry gas flow through the kiln (calculated in the spreadsheets named “Kiln, SPI Run \*.xls” and copied to column D) to the dry gas flow rate through the impinger to scale up the quantities and obtain the mass of each compound leaving the kiln (columns I to P).

On the “Emission” page, the amount of a HAP leaving the kiln is divided by the mass (in kg) or volume of wood (in mbf) to express the emissions on a per kg of wood (columns B-I) or per mbf basis (columns J-Q). Concentrations leaving the kiln are given in columns R to AG.

The “Quality Assurance” page presents information on the spikes, duplicates and blanks. For each spike a % recovery is calculated based on the mass of a HAP recovered divided by the amount added. The difference for each duplicate is calculated as a percentage from the difference between the impingers divided by the average mass collected after adjusting for impinger flow.

The remaining pages in “HAPs, Wood.xls” are for graphing purposes.

## 9. Chain of custody information

Wood was collected by mill personnel and delivered to Oregon State by Sierra Pacific Industries. The wood was retained by Oregon State after delivery as documented in section 1. Field samples remained at Oregon State University.

## 10. Calibration documentation

**Sensidyne, LP**  
CALIBRATION CERTIFICATE

Cell S/N: 1302015-S Date: February 07, 2013

This is to certify that the above referenced Gilibrator Flow Cell was calibrated using film flowmeter MCS-102, which has been calibrated by instruments directly traceable to the National Institute of Standards and Technology, NIST Report 8361604.

Results:

REFERENCE MCS-102 cc/min	S/N 1302015-S cc/min	RELATIVE DIFF. cc/min	PERCENT DIFF.
1998	2001	3	0.15
2002	2001	-1	-0.05
1996	2004	8	0.40
2004	2005	1	0.05
1996	2004	8	0.40
2001	2002	1	0.05
2000	2004	4	0.20
2001	2002	1	0.05
1994	2004	10	0.50
2001	2004	3	0.15

---

MAX 10.0 0.5

MEAN 1999.3 2003.1

Calibrated by Eric N. Peay Date: February 07, 2013  
CODE 100

Figure 14. Flow meter calibration.

## CERTIFICATE OF ANALYSIS Grade of Product: EPA Protocol

Part Number: E02AI99E15A0456      Reference Number: 48-124285462-2  
 Cylinder Number: CC172274      Cylinder Volume: 146 Cu.Ft.  
 Laboratory: ASG - Los Angeles - CA      Cylinder Pressure: 2015 PSIG  
 PGVP Number: B32011      Valve Outlet: 590  
 Gas Code: APPVD      Analysis Date: Oct 13, 2011

Expiration Date: Oct 13, 2014

Certification performed in accordance with "EPA Traceability Protocol (Sept. 1997)" using the assay procedures listed. Analytical Methodology does not require correction for analytical interferences. This cylinder has a total analytical uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a volume/volume basis unless otherwise noted.  
 Do Not Use This Cylinder below 150 psig i.e. 1 Mega Pascal

ANALYTICAL RESULTS				
Component	Requested Concentration	Actual Concentration	Protocol Method	Total Relative Uncertainty
PROPANE	50.00 PPM	50.33 PPM	G1	+/- 1% NIST Traceable
Air	Balance			

CALIBRATION STANDARDS				
Type	Lot ID	Cylinder No	Concentration	Expiration Date
NTRM	080610	CC263076	49.62PPM PROPANE/AIR	Jul 15, 2012

ANALYTICAL EQUIPMENT		
Instrument/Make/Model	Analytical Principle	Last Multipoint Calibration
Nicolet 6700 AHR0801551 C3H8	FTIR	Sep 21, 2011

Triad Data Available Upon Request

Notes:

Signature on file

Approved for Release

**Airgas Specialty Gases**  
 11711 South Alameda Street  
 Los Angeles, CA 90059  
 (323) 568-2293 Fax: (323) 567-3686  
 www.airgas.com

## CERTIFICATE OF ANALYSIS Grade of Product: EPA Protocol

Part Number: E02AI99E15A1511      Reference Number: 48-124370716-1  
 Cylinder Number: SG9115185BAL      Cylinder Volume: 146.2 CF  
 Laboratory: ASG - Los Angeles - CA      Cylinder Pressure: 2015 PSIG  
 PGVP Number: B32013      Valve Outlet: 590  
 Gas Code: PPN,BALA      Certification Date: Apr 23, 2013

Expiration Date: Apr 23, 2021

Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (May 2012)" document: EPA 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical interference. This cylinder has a total analytical uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a volume/volume basis unless otherwise noted.  
 Do Not Use This Cylinder below 100 psig i.e. 0.7 megapascals.

ANALYTICAL RESULTS					
Component	Requested Concentration	Actual Concentration	Protocol Method	Total Relative Uncertainty	Assay Dates
PROPANE	200.0 PPM	199.3 PPM	G1	+/- 0.3% NIST Traceable	04/23/2013
AIR	Balance				

CALIBRATION STANDARDS					
Type	Lot ID	Cylinder No	Concentration	Uncertainty	Expiration Date
NTRM	08061123	CC283016	249.7 PPM PROPANE NITROGEN	+/- 0.6%	Jun 22, 2018

ANALYTICAL EQUIPMENT		
Instrument/Make/Model	Analytical Principle	Last Multipoint Calibration
Nicolet 6700 AHR0801551 C3H8	FTIR	Apr 11, 2013

Triad Data Available Upon Request

Notes:

Approved for Release

Figure 15. Certificates for calibration gases.



## 11. Anomalies

The wet bulb was erratic during charge 1. The sock was angled below the water level and liquid water dripped off the wick. This was corrected late in the charge. The kiln control computer crashed twice during charge one. Four periods of approximately 15 minutes of dry- and wet-bulb data were lost. The kiln continued to control during these periods. The data from charge one is included electronically but was not included in the text of this report.

The only anomaly in charge 2 was from 6:10 pm to 7:40 pm when the wrong valve was open and the total hydrocarbon analyzer sampled lab air instead of kiln air. The data during this period was interpolated from the hydrocarbon data before and after the interval. See Figure 1 between hours 7 and 8.5 for the impact of this. We do not believe that it materially impacted the results.

## 12. Statement of validity

The statements in this report accurately represent the testing that occurred.



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## Appendix 1. Detailed sampling procedures

### Checks of kiln to record on log

**Purpose:** Ensure kiln is operating correctly

**Clock time:** Record from computer

**Run time:** Record from computer. Check the box if the computer screen being refreshed and time is advancing.

**Box temperature:** Read from plastic electrical enclosure on wall or on computer screen. The top and bottom numbers on controller should be similar and greater than the kiln temperature, 230-250°F.

**Line temperature:** Read from plastic electrical enclosure on wall or on computer screen. The top and bottom numbers on controller should be similar and greater than the box temperature, 255-275°F.

**Valve temperature:** Read from plastic electrical enclosure on wall or on computer screen. The top and bottom numbers on controller should be similar and greater than the line temperature, 290-300°F.

**Dry-bulb temperature:** Read from computer screen. Compare to paper graph to be sure it's correct. If it's not within a degree or two of the chart, check again in a few minutes. During startup (the first 3 or so hours), it may not be able to track. If it's too high, the heat valve should be closed, too low and the heat valve should be open. If it does not appear to be working correctly, call Mike.

**Wet-bulb temperature:** Read from computer screen. Compare to graph to be sure it's correct.

If the wet-bulb is too low, it means that the kiln atmosphere is too dry. Check the flow meters. If Flow1 is about 6 L/min (its lower limit), make sure that Flow2 and Flow3 are turned off. Flow2 records automatically. Enter any Flow3 change into the computer. Otherwise, call Mike.

If it's too high, then either the kiln atmosphere is too humid or the sock is not being wetted. If Flow 1 is near 200 L/min (its upper limit) add venting by opening Flow2 and/or Flow 3. Enter any Flow3 change into the computer. The maximum for Flow2 is 50 L/min, if it reads over this value for several readings, reduce it to about 45 L/min. Don't change Flow3 often, rather set it and leave it for several hours if possible. Keep the Flow 3 reading constant by small adjustments. As Flow1 decreases or Flow2 turned down, there is more pressure behind Flow3 and the flow increased. Check for water in the wet-bulb reservoir (push the float down and make sure it's getting water).

Check both Wet-bulb1 and Wet-bulb2 and make sure they are reading about the same. If they differ by more than 2°F, call Mike

If both wet-bulbs are reading the same as the dry-bulb, check the wet-bulb water.

If these procedures do not correct the wet-bulb temperature within 30 minutes, call Mike.

**Chiller temperature:** Read the chiller temperature. It should be about -1°C.

**Flow 1:** Read from computer. The value of Flow1 changes depending on the wet-bulb. If Flow 1 is 6 L/min and the wet-bulb is too low, there's probably nothing we can do. If it's 200 L/min and the wet-bulb is too high, Flow2 and/or Flow3 can be opened. Flow2 and Flow3 should be adjusted so that Flow1 stays below 175 to 200 L/min.

**Flow 2:** Read from computer. The value of Flow2 is set by you. It will vary a little - as flow 1 goes down, flow 2 will go up. Do not set it to > 40 L/min if you think Flow1 is going to decrease or it will go off scale and not be read by the computer

**Flow 3:** Read from meter. The value of Flow3 is set by you. It will vary a little - as flow 1 goes down, flow 2 will go up. Be sure to clearly record this value and when you change it. Change it on the computer screen (click on it and type the new value).

**Dilution flow:** Read dilution flow meter. It should read the same setting as the red flag. Do not adjust. If significantly different, investigate.

**Impinger flows:** Read from rotometers. This should be about 250 to 500 cc/min.

**Line vacuum:** Read from the vacuum gauge. This should be about 20"Hg.

## Total hydrocarbon analyzer

### BACKGROUND INFORMATION

Get the dry- and wet-bulb temperatures from the kiln schedule or off the computer. Use the highest expected values for the next three to six hours.

Read absolute humidity off the psychrometric chart or table.

Calculate or read from tables -

$$\text{Percent moisture} = 100 / [ 1 + 1 / 1.61 * \text{AbHum} ]$$

$$\text{Target Dilution Ratio (TDR)} = 15 / \text{Percent Moisture}$$

Event = the name of the drying cycle.

Run = the number of the 3-hour interval.

Operator, that's you.

Date – use date VOC run will start if close to midnight

### AMBIENT DATA

Read the laboratory temperature from the computer or thermometer.

### ANALYZER CALIBRATION (BEFORE SIDE OF SHEET)

Set valves so that 1, 2 = OFF; 3=ON; 4=VENT. This allows gas to flow out of the vents from the calibration tanks and shuts off all other sources. Only calibration gas should go through the detector.

Open the zero gas tank valve

set analyzer to range 3

zero valve on, others off

set flow to 3 L/min using regulator on tank

wait for a stable reading (about 30 to 60 seconds)

use the zero dial (pot) on THA to get a zero reading

read the analyzer

read computer

note pot setting

Close valve on zero gas tank

Open span gas tank valve (may be 197 or 794 ppm gas)

span valve on, others off

set flow to 3 L/min using regulator on tank

wait for a stable reading (about 30 to 60 seconds)

use the span dial (pot) on THA to get a reading of 610ppm

read the analyzer and record, eg, record 7.96

read computer (should read about 794)

record pot setting  
Leave span tank valve open

Open mid gas tank valve (197 or 50 ppm gas)  
mid valve right on, others off  
set flow to 3 L/min using regulator on tank  
wait for a stable reading (about 30 to 60 seconds)  
read and record analyzer and computer (do not adjust pot settings)  
check for within tolerance  
switch analyzer to range 2  
read analyzer and computer  
check for within tolerance  
switch analyzer back to range 3  
Turn off mid gas tank valve

### **SET DILUTION FLOW BEFORE RUN (BEFORE SIDE OF SHEET)**

Set valves so that 1, 2, 3 = OFF; 4=meter. This allows gas to flow only from the meter to the detector.

Use the Gilibrator to take 4 readings of the total flow rate (TFR). This is the total flow drawn by the analyzer and should be about 1.6 L/min  
Make sure the average does not include any "bad" readings  
Record the average in mL/min; It should be 1500-1600 mL/min  
Write the Run # and "Pre-TFR" on the Gilibrator printout.

Calculate the next two values -  
Target dilution flow rate (TDFR) is the  $TFR \times (1 - DR)$   
Target sample flow rate (TSFR) is the  $TFR \times DR$   
Check that the sum of these is the Total Flow Rate

Set dilution flow  
Set red pointer to desired dilution flow  
Slowly open lower valve on dilution flow meter (1=ON)  
Use upper valve on dilution flow meter to adjust flow  
Do not adjust this meter after this point  
Read the meter that you just set and record the value in SCFH  
Calculate and record L/min

Use the Gilibrator to take 4 readings of the sample flow rate (SFR). This is the flow through the analyzer after dilution is set. It will vary, depending on the dilution setting.  
Make sure the average does not include any "bad" readings  
Record the average in mL/min  
Write "Pre-SFR" on the Gilibrator printout.

## **CHECK DILUTION FLOW BEFORE RUN (BEFORE SIDE OF SHEET)**

Set valves so that 1, 3 = ON; 2=OFF; 4=VENT. This allows gas to flow out of the vent from the calibration tank and shuts off all other sources. Calibration gas and dilution air will go through the detector.

Open span gas tank valve (should already be open)  
span panel valve right (on), others down (off)  
set flow to 3 L/min using regulator on tank  
set analyzer to range 3  
wait for a stable reading (about 30 to 60 seconds), record  
turn off all calibration gas tank valves  
all calibration gas panel valves off  
All tank valves off

Calculate the dilution ratio based on gas flow by dividing the Sample Flow Rate by the Total Flow Rate.  $DR = \text{Absolute value of } [ 100 * (DR \text{ Span} - DR \text{ Flow}) / DR \text{ Flow} ]$

Calculate the dilution ratio based on span gas by dividing the diluted span by the undiluted span.

If the dilution ratio calculated from the span gas and the dilution ratio calculated from the flow do not agree within 5% - DO NOT PROCEED\*\*\*\*. Check the calculations, then redo the measurements.

\*\*\*\* check calculations, check that values for ppm and flows make sense, remeasure everything. If it still does not agree, call Mike

## **START RUN (BOTTOM OF BEFORE SIDE OF SHEET)**

Set valve so that 1, 2, 5 = on; 3, 4=off; all calibration tank valves off

Record the start time. Use the computer clock or stopwatch time.

Make sure analyzer is on appropriate range, usually range 3, to keep THC reading on computer between 60 and 600.

Monitor system, as needed. Record system condition at least hourly.

End time should be no more than 3-6 hours from start time.

## **POST-SAMPLE PROCEDURE - AT END OF RUN (AFTER SIDE OF SHEET)**

Record your name as the operator.

Event = the drying cycle.  
Run = number of the 3-hour interval.  
Operator, that's you.

## **AMBIENT DATA**

Read the laboratory temperature from the thermometer.

Fill out appropriate information on Pre-sample side of data sheet for next run.  
This will save time in between runs.

## **END TIME**

Record computer time.  
DO NOT adjust dilution gas or analyzer pots until the instructions tell you to.

## **CHECK DILUTION FLOW AFTER RUN (AFTER SIDE OF SHEET)**

Measure diluted span gas: Set valves so that 1, 3 = on; 2=off; 4=vent. This allows gas to flow out of the vent from the calibration tank and shuts off all other sources. Calibration gas and dilution air will go through the detector.

Open span gas tank valve  
span panel valve ON, others OFF  
set flow to 3 L/min using regulator on tank  
set analyzer to range 3  
wait for a stable reading (about 30 -60 seconds)  
record

Sample flow rate: Set valves so that 1=on; 2, 3 = off; 4=meter. This allows gas to flow only from the meter and the dilution to the detector.

Use the Gilibrator to take 4 readings of the sample flow rate (SFR). This is the flow through the analyzer with dilution on.

Make sure the average does not include any "bad" readings

Record the average in L/min

Write Run # and "Post-SFR" on the Gilibrator printout.

Read dilution flow meter

To calculate the L/min, divide scfh by 2.12

Turn off dilution flow meter using valve 1 (lower dilution valve)

Total flow rate. Set valves so that 1, 2, 3 = off; 4=meter. This allows gas to flow only from the meter to the detector.

Use the Gilibrator to take 4 readings of the total flow rate (TFR). This is the total flow drawn by the analyzer and should be about 1.6 L/min  
Make sure the average does not include any "bad" readings  
Record the average  
Write Run # and "Post-TFR" on the Gilibrator printout.

Calculate the dilution ratio based on gas flow by dividing the Sample Flow Rate by the Total Flow Rate.

### **CHECK CALIBRATION OF ANALYZER (AFTER SIDE OF SHEET)**

Set valves so that 1, 2 = off; 3=on; 4=vent. This allows gas to flow out of the vents from the calibration tanks and shuts off all other sources. Only calibration gas should go through the detector.

Span gas tank valve should be open  
span panel valve ON, others down OFF  
set flow to 3 L/min using regulator on tank  
set analyzer to range 3  
wait for a stable reading (about 30 -60 seconds)  
read analyzer (do not adjust pot settings),  
record, for example, 7.94 as 794  
read computer (should read about the same)  
note pot setting  
check for within tolerance - between 582 and 619

Open mid gas tank valve  
mid panel valve = ON, others OFF  
set flow to 3 L/min using regulator on tank  
set analyzer to range 3  
wait for a stable reading (about 30 -60 seconds)  
read analyzer (do not adjust pot settings),  
record, for example, 1.97 as 197  
read computer (should read same as analyzer)  
check for within tolerance

Open the zero gas tank valve  
zero panel valve = ON, others OFF  
set flow to 3 L/min using regulator on tank  
wait for a stable reading (about 30 -60 seconds)  
read analyzer (do not adjust pot settings)  
read computer  
note pot setting

Close all tank valves if charge is ending



Calculate the dilution ratio based on gas concentration by dividing the Diluted span by the Span

Calculate % difference in the two dilution ratios as  $100 * \frac{\text{Absolute Value (DRSpan-DRFlow)}}{\text{DRFlow}}$

Record the time now as the end time for check.

Start Pre-Sample procedure for next run.

## **HAP 105 Collection**

### **BACKGROUND DATA**

Begin about 15 minutes before run should start  
Operator, that's you.  
Date, today or tomorrow if sample will start after midnight  
Event = Kiln Charge  
Run = sequence of M/F measurement (1-A, or 5-C, etc )

### **PRE RUN DATA**

Call 9-541-754-0081 and get altimeter setting.

### **IMPINGER WEIGHTS**

Verify that the impinger weights match the prerecorded weights on the data sheet.

Put 15 mL of BHA solution in impinger #1.  
Put 10 mL of BHA solution in impinger #2.  
Impinger #3 is not filled. It is for overflow.

Reweigh the impingers with the BHA solution.  
Place BHA stock back into cooler  
Install impingers and lower into chiller

### **LEAK CHECK**

Read the laboratory temperature.  
Close valve to sample probe.  
Turn on pump (it may already be on)  
Evacuate to 15 to 18 " Hg, record  
Close valve that is near pump  
Note pressure and start timer  
Allowable pressure change is 1" Hg in 2 minutes, if it is much more than this, find the source of the leak. Record change.  
Slowly open valve near probe tip so that pressure is slowly relieved.  
Completely open valve near probe tip  
Open valve near pump

### **SAMPLE FLOW RATE**

Attach probe tip to Gilibrator  
Take 4 readings  
Make sure all readings in average are "good" readings

Record the average

### **START TIME**

Put probe into kiln (or turn valve to sample kiln) and record time.  
Check meters to make sure gas is flowing

### **FLOW READINGS DURING TEST**

Note flow meter reading at intervals of at least 20-30 minutes  
Run test for 1:30 or less if impingers fill

### **POST RUN DATA**

Begin about 10 minutes before run should end  
Label a sample bottle with the Event and Run numbers and record the weight.  
Call 9-541-754-0081 and get altimeter setting.

### **END TIME**

Remove probe (or turn valve to meter setting) from kiln  
Record time

### **SAMPLE FLOW RATE**

Rinse probe with 5 mL of DI water  
Read the laboratory.  
Attach probe tip to Gilibrator  
Take 5 readings  
Make sure all readings in average are "good" readings  
Record the average

### **IMPINGER WEIGHTS**

Lift impingers from chiller, take to scale, and place onto rack  
Dry the outside of the impingers  
Remove U tubes connecting the impingers together  
Weigh sample bottle with lid  
Weigh the impingers (without stoppers) with the catch and record  
Transfer the impinger contents to the sample bottle  
Weigh the sample bottle with lid and record  
Rinse impingers (last to first) with 10 mL DIW (save in the sample bottle)  
Weigh the sample bottle with lid and record  
Rinse impingers (last to first) with 5 mL hexane (save in the sample bottle)  
Weigh the sample bottle with lid and record  
Place the sample bottle into cold storage  
Record the volume of any liquids lost during this procedure.  
Wash glassware with phosphate-free detergent and set out to dry.

**Appendix 2. Electronic copy of data and calculations**