

## Appendix – Soil Sample Collection and Analysis

Composite surface soil samples were collected with a shovel forced into the soil to a depth of approximately 10 cm. Four samples collected randomly from each quadrant of a given test cell were placed on a sheet of clean plastic and mixed with a trowel. A portion of the composited material was then transferred to a polyethylene bag with Ziploc closure. The sample bag was placed in a cooler partially filled with ice for transport back to the laboratory.

The physicochemical stability of the ions of interest (chloride, sulfate, sodium, calcium and magnesium) in soil samples and leachate samples is believed to be excellent. Nevertheless, soil samples were extracted within two weeks of their arrival at the laboratory. Measurement of the conductivity and determination of the major anions and cations was then performed no more than three days after extraction. The leachate samples were analyzed at the same time as the soil extracts.

The sub-sampling and extraction of soil samples was conducted as follows. Before opening the closure of the Ziploc bag, the contents were first homogenized by kneading. A 120 g sample was then withdrawn from the bag and weighed into a pre-weighed clean, dry glass bottle (with polyethylene-lined screw-top cap). The sample was dried in an oven at 110°C for a period of at least 12 hours. At this point the sample was weighed again. A mass of high-purity water equal to the mass of the dry soil sample was then weighed into the bottle. The contents of the bottle were mixed with a thick glass stirring rod, and allowed to equilibrate for a period of at least 12 hours. The mixture was then filtered under vacuum through qualitative filter paper. The filtrate was transferred to a clean polyethylene bottle. A soil extract prepared in this manner is referred to in the literature as a “1:1 extract”. This type of extract is recommended for monitoring a change in the concentration of salt contamination in soil over the course of a remedial action (6).

To ascertain the repeatability of this sampling and extraction procedure, duplicate extractions of the first set of samples were prepared and analyzed. The agreement between duplicate samples proved to be excellent. For the second set of samples every ninth soil sample was extracted in duplicate.

The high purity water utilized in the extractions (and the dilutions noted below) was produced with a Millipore Milli-Q water purification system. This apparatus possesses a resistivity cell that measures the resistivity of the water as it is delivered.

The conductivity of samples of leachate from the pond and of soil extracts was measured using a YSI conductivity bridge and dip probe. To ensure proper operation, this apparatus was checked using a 0.0100 M solution of potassium chloride.

The concentrations of chloride and sulfate ion in the leachate sample and the soil extracts were determined by ion chromatography. Since this technique is quite sensitive, samples typically required significant dilution, which was performed quantitatively, with high purity water as the diluent. The ion chromatograph was recalibrated immediately before each set of sample solutions was analyzed, by running three different dilute standard solutions through the instrument. During each set of sample analyses the performance of the instrument was monitored by analyzing one of these standard solutions after every nine sample solutions, as well as by checking the results from duplicate samples.

The concentrations of the major cations (sodium, calcium and magnesium ion) were determined in both soil extracts and the leachate sample using inductively coupled plasma atomic emission spectrophotometry (ICP-AES). All of the leachate samples and soil extracts were diluted with 1% (volume) nitric acid to provide a uniform sample solution matrix (the same matrix employed with

the standard solutions). The **ICP-AES** was recalibrated immediately before each set of sample solutions was analyzed, by running three different dilute standard solutions. **Or** laboratory protocol calls for a calibration check (the analysis of one of the standard solutions **as** a sample) after every five sample solutions analyzed. When the measured concentration of the standard solution **was** more than  $\pm 15\%$  different from the previous calibration, a full recalibration (involving all three calibration standard solutions) was performed before the sample analysis was continued.

**Table 5. Soil analytical data for sampling date 9/24/00.**

<u>Sample id</u>	<u>Water Content</u>	<u>Conductivity</u>	<u>Chloride</u>	<u>Sulfate</u>	<u>Sodium</u>	<u>Calcium</u>	<u>Magnesium</u>	<u>SAR</u>
	(%)	( $\mu\text{ohms-cm-1}$ )	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	
Keifer I-1-14	8.26	7250	2.6E+03	3.6E+02	1.1E+03	4.5E+02	5.5E+01	18
Keifer I-1-Sa	8.47	7520	2.6E+03	3.1E+02	1.1E+03	4.8E+02	5.5E+01	19
Keifer I-2-4	6.78	880	3.7E+01	5.6E+01	5.3E+01	1.8E+02	0.0E+00	2
Keifer I-2-Sa	6.83	820	3.1E+01	4.4E+01	5.5E+01	1.6E+02	0.0E+00	2
Keifer I-3-S	12.20	7430	2.5E+03	6.9E+02	1.4E+03	3.1E+02	2.8E+01	30
Keifer I-3-Sa	12.61	7050	2.3E+03	7.5E+02	1.4E+03	2.8E+02	2.8E+01	30
Keifer I-4-4	6.27	1380	1.8E+02	1.5E+02	2.6E+02	1.4E+02	0.0E+00	9
Keifer I-4-Sa	6.47	1390	1.9E+02	1.6E+02	2.7E+02	1.1E+02	0.0E+00	10
Keifer I-5-S	14.40	20400	9.1E+03	2.8E+02	4.6E+03	7.6E+02	1.5E+02	57
Keifer I-5-Sa	14.02	19300	9.4E+03	2.6E+02	5.9E+03	8.9E+02	1.8E+02	66
Keifer I-6-S	12.33	1620	2.5E+02	7.4E+01	2.7E+02	1.1E+02	1.5E+01	9
Keifer I-6-Sa	11.77	1500	2.1E+02	6.3E+01	2.6E+02	1.1E+02	1.3E+01	9
Keifer I-7-S	9.81	10040	3.8E+03	7.1E+02	1.6E+03	7.5E+02	1.2E+02	20
Keifer I-7-Sa	9.42	9920	3.7E+03	5.3E+02	1.5E+03	7.2E+02	1.1E+02	20
Keifer I-8-4	8.68	2680	8.8E+02	2.9E+01	4.5E+02	1.0E+02	1.3E+01	16
Keifer I-8-Sa	8.95	3120	1.0E+03	2.4E+01	5.5E+02	1.1E+02	1.3E+01	19
Keifer I-9-4	10.53	1480	3.0E+02	6.3E+01	3.2E+02	6.5E+01	5.0E+00	14
Keifer I-9-Sa	10.54	1430	2.6E+02	5.5E+01	3.0E+02	6.5E+01	5.0E+00	14
Keifer I-10-S	9.61	8820	3.4E+03	1.9E+02	1.8E+03	2.6E+02	4.8E+01	38
Keifer I-10-Sa	9.48	9400	3.5E+03	1.1E+02	1.9E+03	2.7E+02	5.3E+01	39
Keifer I-11-S	8.57	16800	9.1E+03	9.7E+02	3.8E+03	6.8E+02	1.3E+02	50
Keifer I-11-Sa	8.68	17500	7.9E+03	1.5E+02	4.0E+03	7.1E+02	1.3E+02	51
Keifer II-1-SL	2.73	70	1.2E+00	8.5E+00	3.3E+01	3.3E+01	0.0E+00	2
Keifer II-1-Sla	2.81	120	1.2E+00	9.1E+00	3.3E+01	3.5E+01	0.0E+00	2

**Table 6.** Soil analytical data for sampling date 1/2 1/01.

Sample id	Water Content (%)	Conductivity ( $\mu\text{ohms}\cdot\text{cm}\cdot\text{l}$ )	Chloride (ppm)	Sulfate (ppm)	Sodium (ppm)	Calcium (ppm)	Magnesium (ppm)	SAR
I-1-S	25.49	1580	2.5E+02	1.E+02	5.5E+01	1.5E+01	0.0E+00	6
I-24	25.70	9200	1.0E+01	6.E+01	3.5E+01	3.0E+01	0.0E+00	2
I-34	22.83	1220	1.6E+02	1.E+02	4.8E+01	2.5E+00	0.0E+00	12
I-43	29.79	858	2.2E+01	7.E+01	4.8E+01	5.0E+00	0.0E+00	8
I-4-Sa	30.94	601	2.5E+01	9.E+01	5.0E+01	5.0E+00	0.0E+00	9
I-54	31.37	17	4.1E+03	1.E+03	2.7E+02	4.3E+01	0.0E+00	16
I-63	30.48	850	2.0E+01	5.E+01	4.3E+01	5.0E+00	0.0E+00	7
I-73	22.32	5780	2.0E+03	2.E+02	1.2E+02	4.8E+01	0.0E+00	6
I-8-S	22.68	735	7.4E+01	4.E+01	4.8E+01	2.5E+00	0.0E+00	12
I-9-S	29.55	713	1.3E+01	4.E+01	4.3E+01	7.5E+00	0.0E+00	6
I-10-S	29.07							
I-11-S	31.97	680	1.6E+01	1.E+02	4.0E+01	5.0E+00	0.0E+00	7
I-11-Sa	25.45	668	1.8E+01	7.E+01	4.3E+01	7.5E+00	0.0E+00	6
II-1-S	29.61	13300	4.7E+03	0.E+00	2.1E+02	8.5E+01	7.5E+00	8
II-2-S	17.97	297	2.3E+01	1.E+01	3.8E+01	2.5E+00	0.0E+00	9
II-3-S	12.15	162	2.0E+00	2.E+01	3.3E+01	2.5E+00	0.0E+00	8
II-4-S	14.95	212	3.5E+00	1.E+01	3.5E+01	0.0E+00	0.0E+00	

**Table 7. Soil analytical data for sampling date 5/8/01.**

<b>Sample id</b>	<b>Water Content</b>	<b>Conductivity</b>	<b>Chloride</b>	<b>Sulfate</b>	<b>Sodium</b>	<b>Calcium</b>	<b>Magnesium</b>	<b>SAR</b>
	(%)	( $\mu\text{ohms}\cdot\text{cm}^{-1}$ )	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	
050801-I-1	6.70	5700	2.1E+03	4.E+02	8.2E+02	5.8E+02	9.3E+01	12
050801-I-2	7.54	780	3.1E+01	6.E+01	7.8E+01	2.1E+02	3.5E+01	2
050801-I-3	12.84	6700	2.7E+03	3.E+02	1.2E+03	6.1E+02	8.3E+01	16
050801-I-4	7.88	1150	1.4E+02	1.E+02	2.6E+02	8.8E+01	2.8E+01	9
050801-I-5	14.17	5600	2.1E+03	2.E+02	1.3E+03	1.6E+02	4.5E+01	33
050801-I-6a	7.29	660	5.8E+01	4.E+01	1.5E+02	8.8E+01	2.8E+01	5
050801-I-6b	7.23	650	5.3E+01	4.E+01	1.3E+02	8.3E+01	2.8E+01	4
050801-I-7	8.30	3000	9.7E+02	2.E+02	4.6E+02	2.9E+02	4.5E+01	9
050801-I-8	6.52	1400	3.6E+02	4.E+01	3.2E+02	6.0E+01	2.3E+01	12
050801-I-9	6.33	640	4.7E+01	4.E+01	1.3E+02	1.0E+02	3.0E+01	4
050801-I-10	10.06	5300	1.8E+03	8.E+01	1.1E+03	9.8E+01	1.8E+01	38
050801-I-11	9.09	2500	8.3E+02	8.E+01	4.0E+02	9.0E+01	1.5E+01	15
050801-II-1	17.51	13000	6.1E+03	7.E+01	1.9E+03	1.1E+03	2.4E+02	20
050801-II-2	12.92	12500	5.9E+03	1.E+02	2.1E+03	9.2E+02	1.6E+02	23
050801-II-3	5.00	1450	4.4E+02	1.E+02	1.5E+02	1.5E+02	2.0E+01	4
050801-II-4	6.18	1400	4.5E+02	5.E+01	1.4E+02	1.4E+02	2.3E+01	4

Treatment Variables <b>test plot id</b>
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<p><b>11-4</b> subsurface drainage limestone gravel sulfur sandy loam topsoil</p>	<p><b>11-3</b> subsurface drainage limestone gravel sandy loam topsoil</p>
<p><b>11-1</b> control</p>	<p><b>11-2</b> subsurface drainage sandy loam topsoil</p>

<p><b>1-1</b> control</p>	<p><b>1-11</b> control</p>	<p><b>1-8</b> gravel</p>
<p><b>1-7</b> gravel</p>	<p><b>1-4</b> drain pipe sock</p>	<p><b>1-9</b> gravel sulfur</p>
<p><b>1-3</b> drain pipe sock</p>	<p><b>1-10</b> gravel sulfur</p>	<p><b>1-6</b> drain pipe gravel</p>
<p><b>1-2</b> control</p>	<p><b>1-5</b> drain pipe gravel</p>	

**Fig. 1** Spatial display of treatments applied to each test section.

Sodium absorption ratio
<u>test</u> plot id
09/24/00
01/21/01
05/08/01

<u>11-4</u> 2 X 4	<u>11-3</u> 2 8 4
<u>11-1</u> ... 8 20	<u>11-2</u> 2 9 23

<u>I-1</u> 18 6 12	<u>I-11</u> 50 6 15	<u>I-8</u> 18 12 12
<u>I-7</u> 20 6 9	<u>I-4</u> 10 8 9	<u>I-9</u> 14 6 4
<u>I-3</u> 30 12 16	<u>I-10</u> 38 ... 38	<u>I-6</u> 9 7 4
<u>I-2</u> 2 2 2	<u>I-5</u> 62 16 33	/

Fig. 5 Spatial display of sodium absorption ratio values for each test section.

Conductivity ( $\mu$ mhos/cm)
<u>test plot id</u>
09/24/00
01/21/01
05/08/01

<u><b>11-4</b></u> 95 212 1400	<u><b>11-3</b></u> 95 162 1450
<u><b>11-1</b></u> ... 13,300 13,000	<u><b>11-2</b></u> 95 297 12,500

<u><b>1-1</b></u> 7400 1580 5700	<u><b>1-11</b></u> 17,200 674 2500	<u><b>1-8</b></u> 2900 735 1400
<u><b>1-7</b></u> 9980 5780 3000	<u><b>1-4</b></u> 1380 730 1150	<u><b>1-9</b></u> 1450 713 640
<u><b>1-3</b></u> 7240 1220 6700	<u><b>1-10</b></u> 9110 ... 5300	<u><b>1-6</b></u> 1560 850 655
<u><b>1-2</b></u> 850 9200 780	<u><b>1-5</b></u> 19,800 17 5600	

**Fig. 2** Spatial display of conductivity values for each test section.

<b>Chloride ion</b> (ppm or mg/kg)
<b>test plot id</b>
09/24/00
01/21/01
05/08/01

<u><b>II-4</b></u> 1.2 3.5 450	<u><b>II-3</b></u> 1.2 2.0 440
<u><b>II-1</b></u> ... 4700 6100	<u><b>II-2</b></u> 1.2 23 5900

<u><b>I-1</b></u> 2600 250 2100	<u><b>I-11</b></u> 8500 17 830	<u><b>I-8</b></u> 940 74 360
<u><b>I-7</b></u> 3800 2000 970	<u><b>I-4</b></u> 180 24 140	<u><b>I-9</b></u> 280 13 47
<u><b>I-3</b></u> 2400 160 2700	<u><b>I-10</b></u> 3400 ... 1800	<u><b>I-6</b></u> 230 20 56
<u><b>I-2</b></u> 34 10 31	<u><b>I-5</b></u> 9200 4100 2100	

**Fig. 3** Spatial display of chloride ion values for each test section.

Sodium ion (ppm or mg/kg)
<b>test plot id</b>
09/24/00
01/21/01
05/08/01

<b><u>II-4</u></b> 33 35 140	<b><u>II-3</u></b> 33 33 150
<b><u>II-1</u></b> ... 210 1900	<b><u>II-2</u></b> 33 38 2100

<b><u>I-1</u></b> 1100 55 820	<b><u>I-11</u></b> 3900 42 400	<b><u>I-8</u></b> 500 48 320
<b><u>I-7</u></b> 1600 120 460	<b><u>I-4</u></b> 260 49 260	<b><u>I-9</u></b> 310 43 130
<b><u>I-3</u></b> 1400 48 1200	<b><u>I-10</u></b> 1800 ... 1100	<b><u>I-6</u></b> 260 43 140
<b><u>I-2</u></b> 54 35 78	<b><u>I-5</u></b> 5200 270 1300	/

**Fig. 4** Spatial display of sodium ion values for each test section.

## Appendix - Microbiological Testing Details

Sterile, TGY agar plates (100X15mm) were prepared and the soil bacteria extractions performed using a sodium pyrophosphate solution (0.2% solution) were also prepared. The soil bacteria extraction was done as quickly as possible, using the procedure describes as follows. A 10-gram portion of the soil is aseptically added into an autoclaved 250 ml erlenmeyer flask. The sodium pyrophosphate solution (100ml) was aseptically added to the soil. The beaker was shaken on a rotational shaker at 2000 rpm for 30 minutes. Serial dilutions were prepared by adding 0.1pl of the previous dilution to 0.9μl of 0.9% NaCl solution in an array of 1.5μl labeled eppendorf microcentrifuge tubes. After the serial dilutions from  $10^{-2}$  to  $10^{-7}$  were prepared, 0.1μl of each dilution was plated and spread with a sterile spreader. All dilutions, plate preparations, plating, and spreading occurred in a laminar flow hood with a pathogen filter. The plates were stored at room temperature for 72 hours and counts were performed. Plate containing between 30 and 300 CFUs are included in the calculated geometric mean of the populations indicated by the dilution corrected plate counts.

Plate counts are averages of duplicate plates, multiplied to account for the dilution factor. Calculations were performed to the CFU per **gram** of soil. The moisture content of the soil samples are included to adjust the data to *dry* basis, CFU per **gram** of *dry* soil. The results of the summary of the plate counts are included in Table 8.

**Table 8.** Summary table of data collected from TGY plate counts.

ARE A	Sample Name	Sulfur Added	Type of Sample (core or soil)	Average Bacterial Population per gram of soil from dilution=10 <sup>-2</sup>	Average Bacterial Population per gram of soil from dilution=10 <sup>-3</sup>	Average Bacterial Population per gram of soil from dilution=10 <sup>-4</sup>	Average Bacterial Population per gram of soil from dilution=10 <sup>-5</sup>	Average Bacterial Population per gram of soil from dilution=10 <sup>-6</sup>	Geometric Mean of Bacterial Population per gram of soil
2	NE Cell X	NO	core		1.65E+05	2.99E+04			7.01E+04
1	Site 7 Y	NO	core		2.13E+05	4.54E+04			9.83E+04
1	Site 8 Y	NO	core		1.18E+05				1.18E+05
1	Site 8 X	NO	core	1.79E+05					1.79E+05
2	NE Cell Y	NO	core		1.83E+05				1.83E+05
1	Site 7 X	NO	core						NA
2	NE Cell A	NO	soil			3.04E+05	6.03E+04	1.19E+03	2.80E+04
1	Cell 7 B	NO	soil		3.56E+05	1.62E+05	4.72E+04	5.97E+03	6.35E+04
1	Cell 7 A	NO	soil		2.21E+05	8.24E+04			1.35E+05
1	Cell 8 B	NO	soil			1.59E+05			1.59E+05
1	Cell 8 A	NO	soil			1.78E+05			1.78E+05
2	NE Cell B	NO	soil		1.90E+05				1.90E+05
1	Site 9 X	YES	core		2.63E+05	3.94E+04			1.02E+05
1	Site 10 Y	YES	core		2.61E+05	4.06E+04			1.03E+05
2	NW Cell Y	YES	core		1.27E+05				1.27E+05
1	Site 9 Y	YES	core		1.31E+05				1.31E+05
1	Site 10 Y	YES	core	4.43E+05	5.07E+04				1.50E+05
2	NW Cell X	YES	core		2.63E+05	8.72E+04			1.51E+05
1	Cell 10 B	YES	soil			1.86E+05	3.82E+04		8.44E+04
1	Cell 10 A	YES	soil			1.02E+05			1.02E+05
1	Cell 9 A	YES	soil			1.07E+05			1.07E+05
1	Cell 9 w/ Sulfur on Gravel	YES	soil		2.38E+05	1.73E+05	7.28E+04	4.78E+04	1.09E+05
1	Cell 9 B	YES	soil			1.24E+05			1.24E+05
2	NW Cell A	YES	soil		2.63E+05	1.50E+05			1.98E+05
2	NW Cell E w/ Sulfur	YES	soil		2.27E+05				2.27E+05

## Appendix- Meteorological Summary for Bartlesville Site

With regards to the performance of the leachate collection system, there needs to be some comment made as to the nature of the precipitation patterns and how the soil characteristics are contributing to some behavior that was not anticipated. As seen in Figure 6, based on data taken from the Copan Mesonet station, the average daily rainfall for each month is relatively low, however, the majority of the rainfall occurs in larger precipitation events of short duration as seen in Figure 7. With the permeability of the soils in the  $10^{-6}$  cm/s range, the majority of the rainfall appears to be standing on the surface until it evaporates instead of leaching in a regular, prolonged way to the LCS. This makes monitoring the flow of the leachate and the concentration trends very difficult. Since this problem would be frequently encountered in future remediation efforts, some analysis of the costs and benefits of further soil conditioning to enhance the benefits of the LCS could be studied.

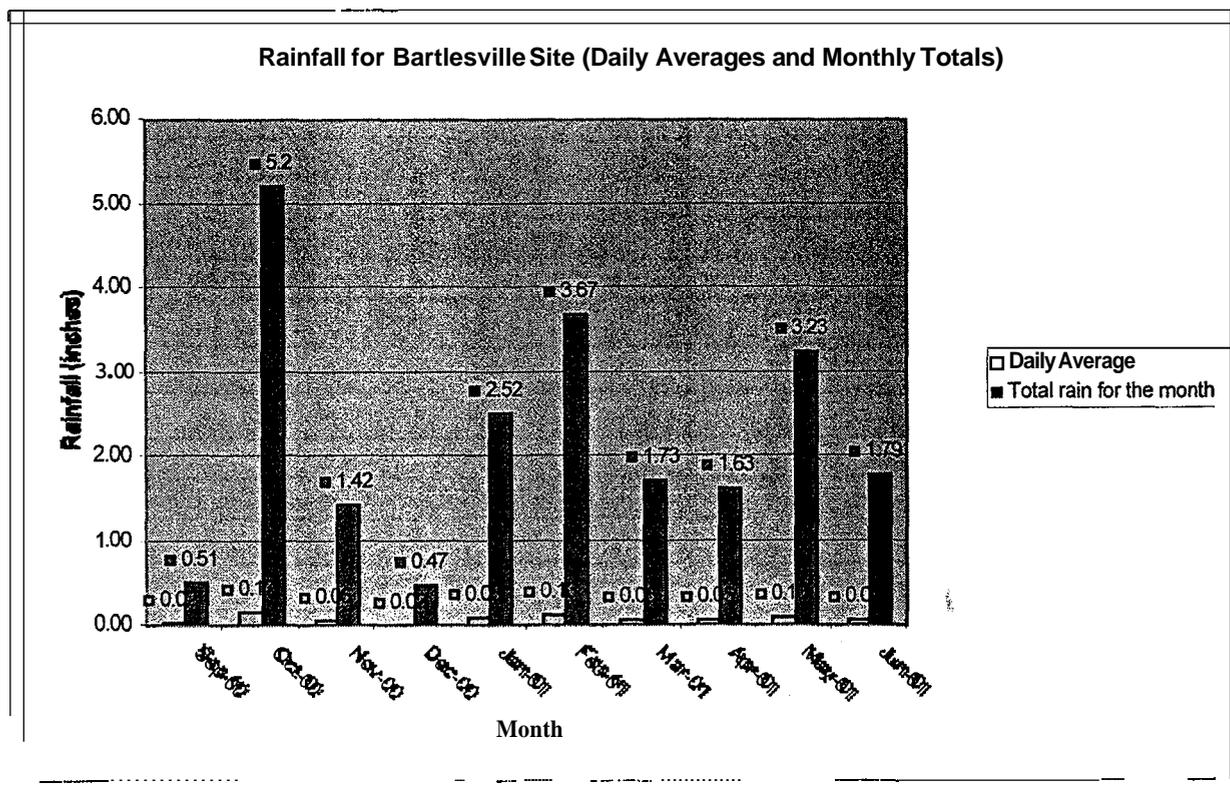
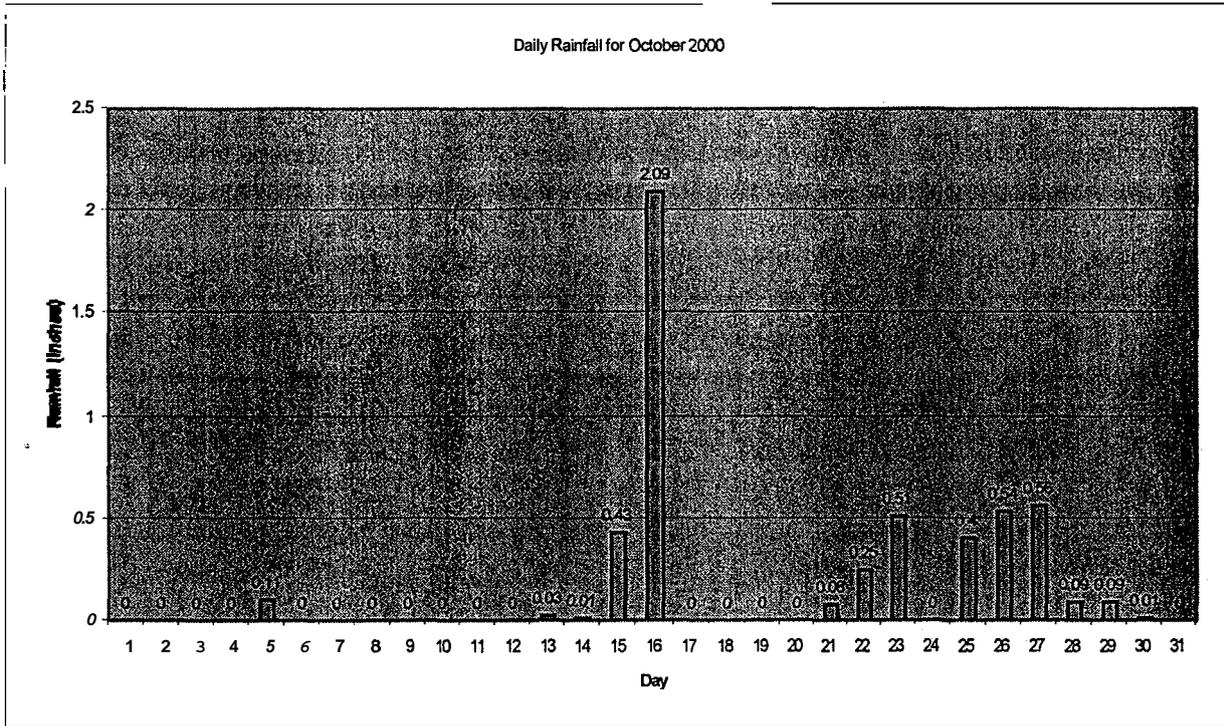


Figure 6. Rainfall for Bartlesville Site, 9/2000-6/2001.



**Figure 7.** Daily rainfall for October 2000.