UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION IX LABORATORY
1337 S. 46TH STREET BLDG 201
RICHMOND, CA 94804-4698

MEMORANDUM

SUBJECT: XRF Field Analytical Support at the Iron King/Humboldt Smelter Site, Dewey-Humboldt, AZ

FROM: Peter Husby, Biologist
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THROUGH: Brenda Bettencourt, Director
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TO: Monika O'Sullivan, RPM
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Peter Husby of the US Environmental Protection Agency (EPA), Region 9 Laboratory Field and Biology Team prepared this Field Report (attached) to document the analysis of soils in the field and at the Region 9 Laboratory using XRF technology, and the collection of concurrent GPS location data for all sample locations. The work was performed near the Iron King Mine/Humboldt Smelter located near the towns of Dewey and Humboldt, AZ on January 10-11, 2012.

If there are further questions concerning this data collection effort, please contact me at (510) 412-2331.

ATTACHMENT: Field Report and CD

Iron King Mine/Humboldt Smelter Site
Humboldt, AZ
January 10-11, 2012
Field Report

Introduction:

On January 10-11, 2012, Peter Husby of the US Environmental Protection Agency (EPA) Region 9 Laboratory Field and Biology Team assisted Monika O'Sullivan, Jeff Dhont and John Hillenbrand of the US Environmental Protection Agency (EPA) Region 9 Superfund Division with the collection and analysis of surface soils for metals using XRF near the Iron King Mine/Humboldt Smelter Site in Humboldt, AZ. Samples of surface soils were collected in plastic bags and immediately analyzed by XRF. Documentation of the location included GPS readings, geologic formation information, and photos.

Participants:

Peter Husby performed XRF analyses and GPS data collection.
Monika O'Sullivan, Jeff Dhont, and John Hillenbrand selected sample locations, identified geologic formations, collected photos, and maintained field notebooks of all activities.

Site Activities:

GPS locations

GPS readings were collected for all locations sampled. Two different GPS units were used; a Trimble GeoExlorer 3 and a Trimble Geo XH 2005 Series. The data was postprocessed using GPS Pathfinder Office software. GPS readings were collected at 88 locations. At numerous locations collocated samples were collected and separate GPS readings were collected. However, at 27 collocated locations a second GPS reading was not collected. At one location, Location ID 009, no GPS position was logged. The GPS data is attached in Appendix A and mapped in Figure 1. Figures 2, 3 and 4 show data from smaller areas identified as being of interest during logistical planning. For ease in visual interpretation of the data collected, the locations have been color coded based on the concentrations of arsenic measured. Table 1 outlines the color/concentration relationship. In addition, for collocated samples, the surface sample is indicated with a balloon, and subsurface sample is represented by a dot. Sample locations where subsurface samples were not collected are indicated with a dot only. Figure 5 shows all the sample locations color coded based on the concentration of lead measured by XRF. Table 2 outlines the lead concentration/color relationship. ArcGIS files of the maps are stored at the Region 9 Laboratory. The data is also included in the attached CD under the file name IKHS XRF locations.

Table 1
IKHS Arsenic Concentration/Color Code

Arsenic Concentration ppm	Marker Color
< 35	
35 - 41	
42 - 69	
70 - 100	
> 100	

Figure 1
IKHS XRF Sample Locations
Color coding for Arsenic Concentration

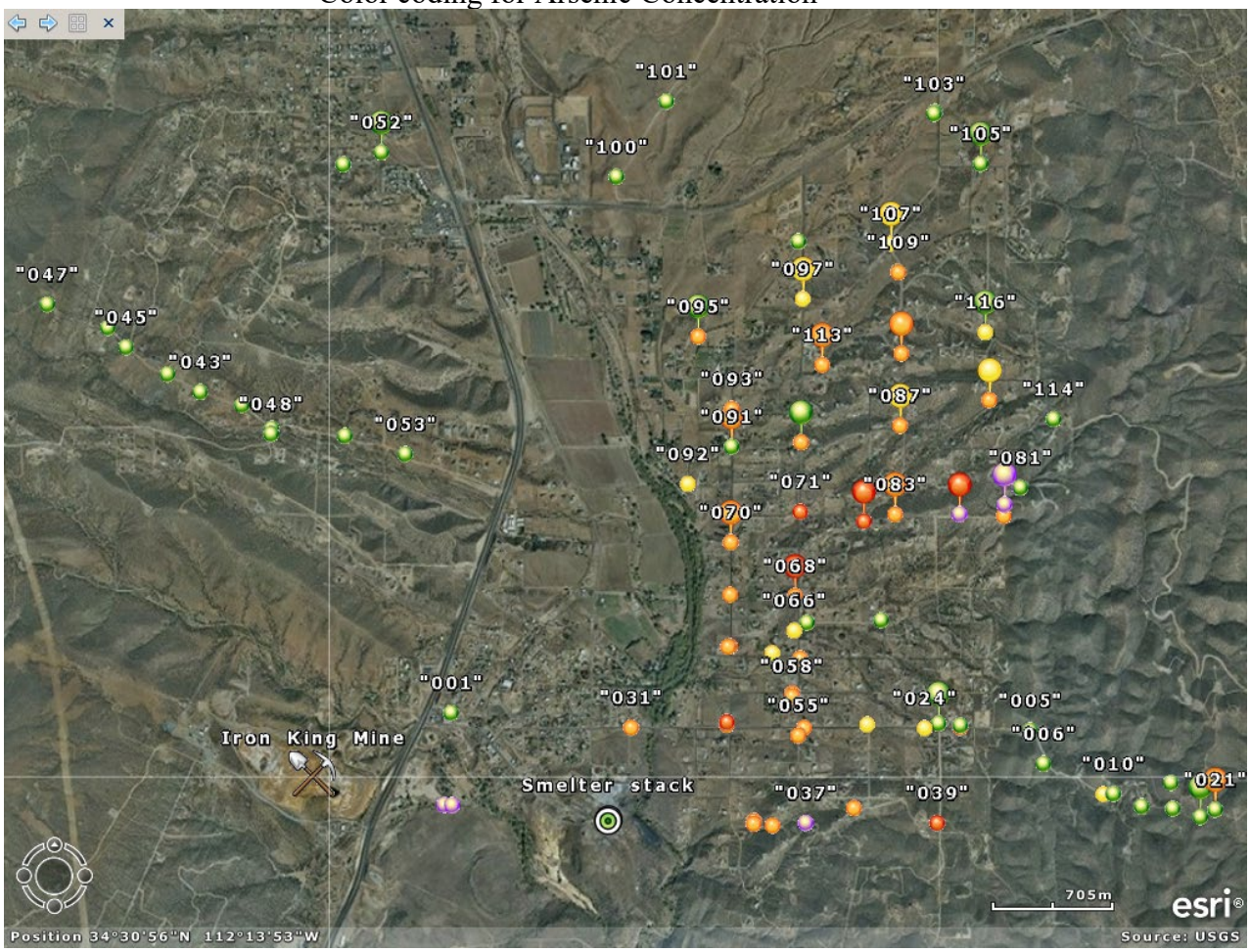


Figure 2
Third Street and Beverly Hills Drive Area

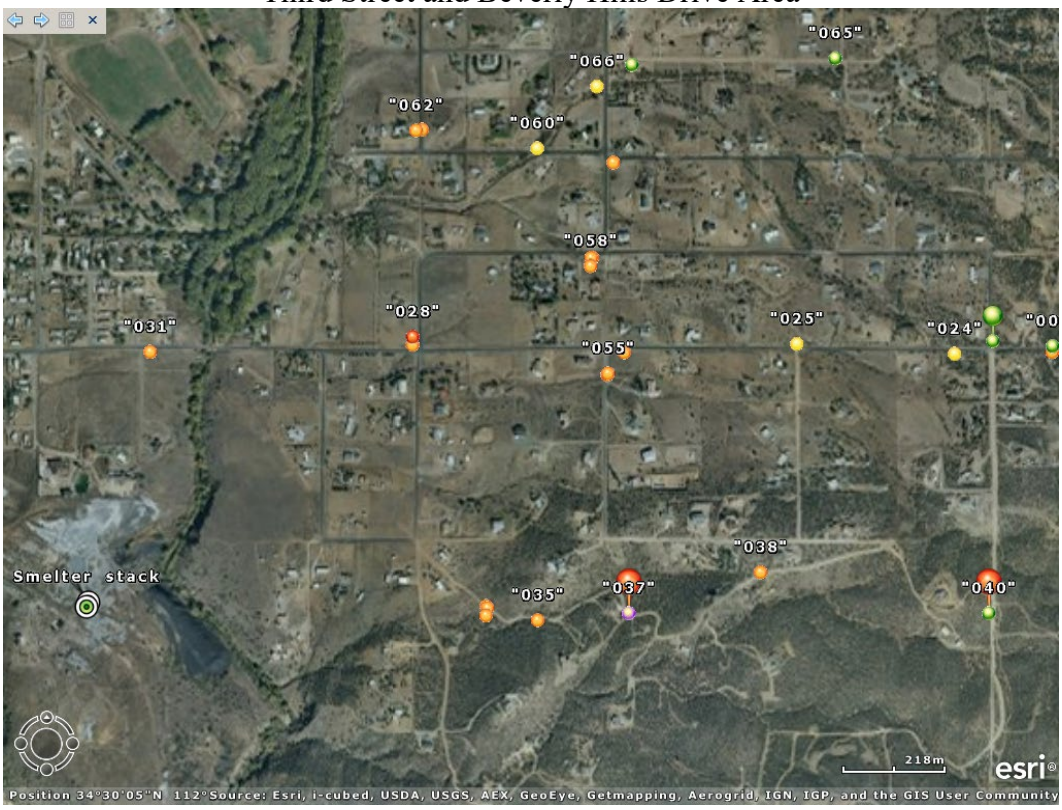


Figure 3
East Side of Highway & Donut Hole Areas

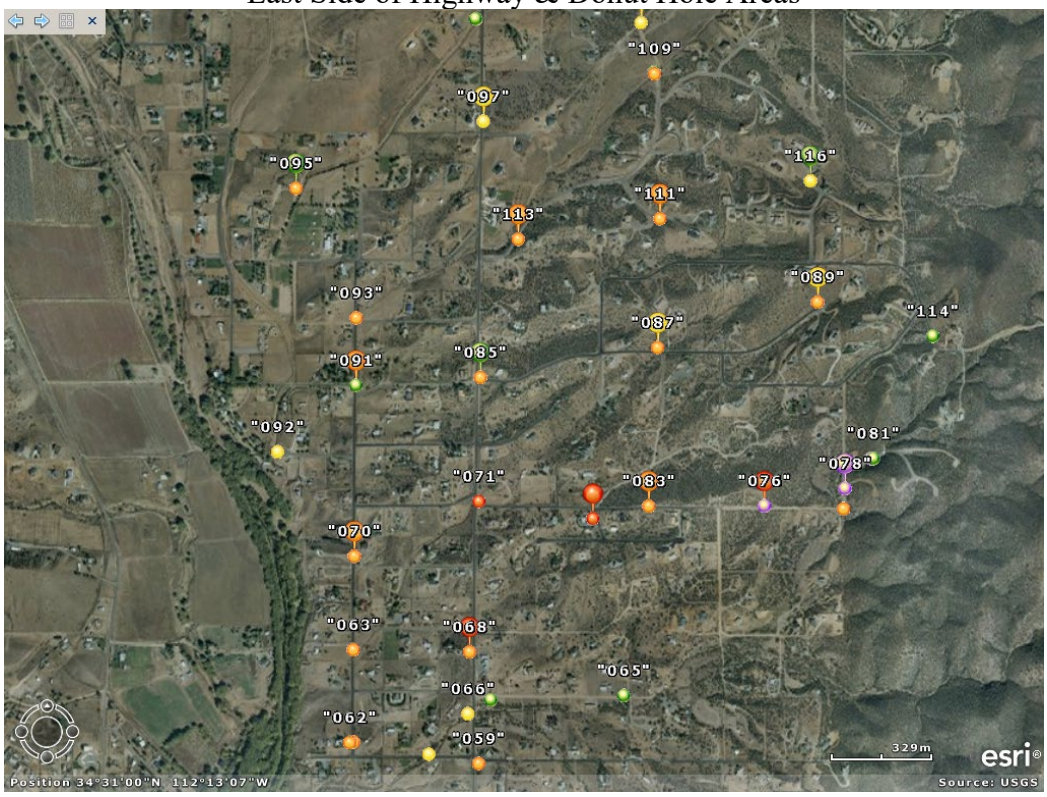


Figure 4
Westside of Highway and Donut Hole Areas

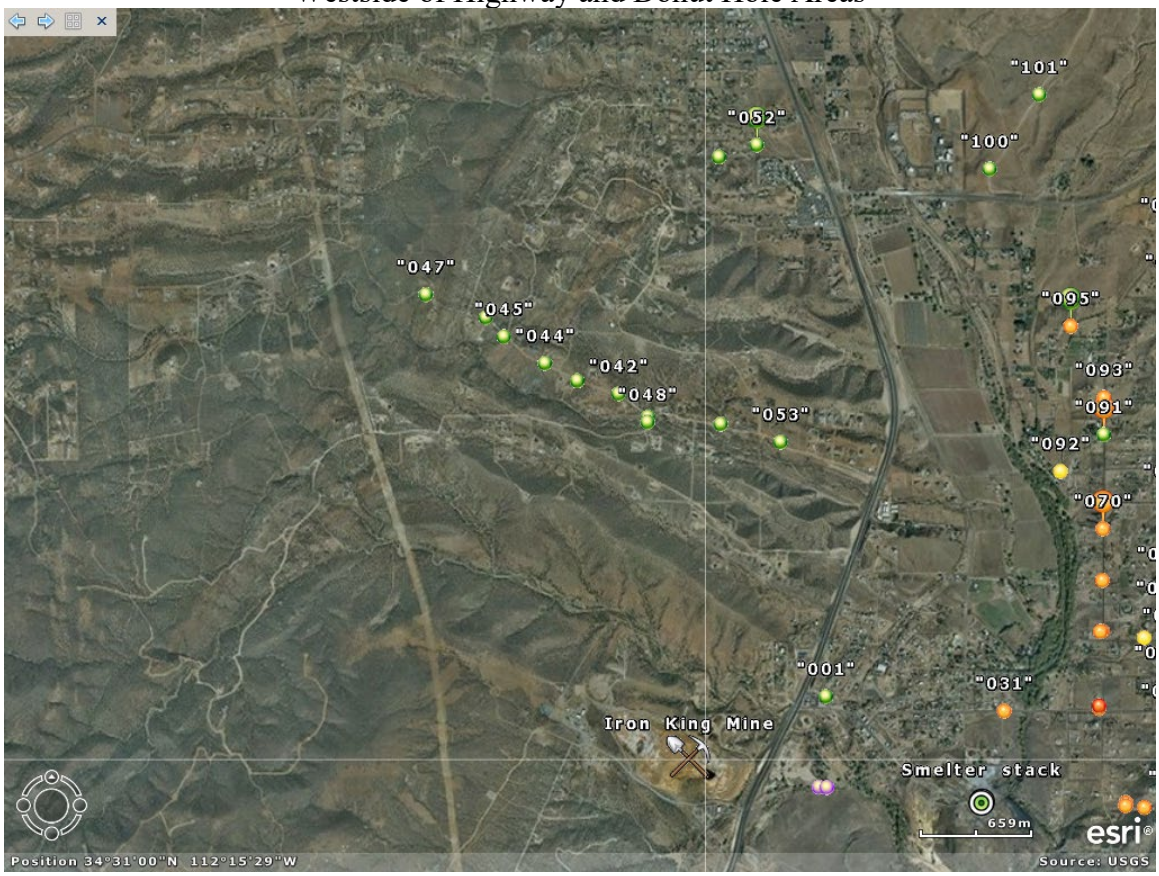
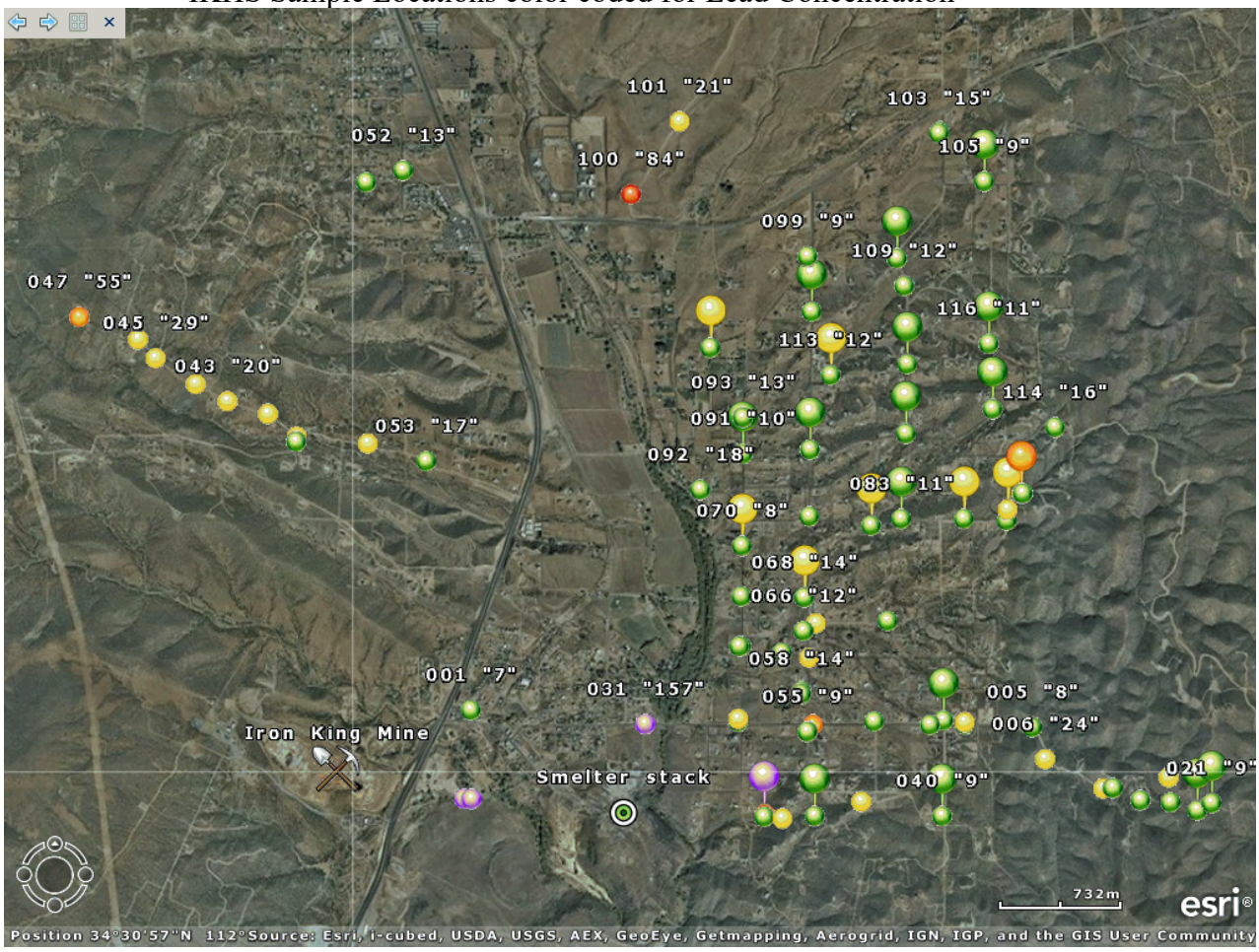


Table 2
IKHS Lead Concentration/Color Code

Lead Concentration ppm	Marker Color
< 20	
20 - 39	
40 - 59	
60 - 99	
> 100	

Figure 5
IKHS Sample Locations color coded for Lead Concentration



XRF Analyses

At each location, a surface sample was collected by using a trowel to place approximately 2 ounces of the top 2.5 cm of soil in a plastic bag. At a number of locations, a cut face slope was located. When a cut face was available, a second sample was collected from approximately 1 to 2 feet below grade on the face of the cut slope, by removing the face soil and collecting approximately 2 ounces of the underlying soil. The samples were hand mixed in the plastic bags by manually breaking up large particles and kneeding the soil to create a sample of the finest, most uniform particles easily achieved in the field.

The samples were analyzed immediately using a Niton Model XL3t XRF analyzer. The sample run was performed using the Standard Range Filter with a 60 second analysis time. The elements detected included: Arsenic, Lead, Selenium, Copper, Zinc, Nickel, Mercury iron, Manganese, Uranium, Molybdenum, Zirconium, Strontium, Rubidium, Thorium, Tungsten and Cobalt. The main elements of interest at the site were arsenic and lead. All calibrations were evaluated for those elements only. Each sample was analyzed in duplicate by turning the bag to expose a different surface and performing an identical XRF analysis. The average Relative Percent Difference (RPD) for all the

replicate analyses performed in the field was 17.2% for Arsenic and 19.4% for Lead. This includes all samples where the metal of interest was detected in both replicates. The XRF data collected in the field is attached in Appendix B and can be found in the attached CD in the file IKHS_Field XRF Data_January2012.xls.

Data quality of the XRF results also was assessed using standard reference materials. The XRF system was challenged with these reference materials, as well as a blank soil, before, during and after the analyses performed. The data are tabulated below in Table 2 below. The SOP for XRF calls for the recovery of standards to be ±20% of the true value. All the recoveries for arsenic and lead met those criteria. In addition, arsenic and lead were not detected in the reference blank material.

Table 2
QC Sample Results

Date/Time	Units	QC SAMPLE	True Value Pb	True Value As	%R Pb	%R As	Pb	Pb Error	As	As Error
1/9/2012 18:47	ppm	BLANK SiO2	0	0	NA	NA	< LOD	5.39	< LOD	4.22
1/9/2012 18:50	ppm	NCS 73308	27	25	103	96	27.85	6.17	24.09	6.03
1/9/2012 18:57	ppm	TILL-4	50	111	89	98	44.71	8.33	108.83	10.23
1/10/2012 14:33	ppm	NCS73308	27	25	103	85	27.76	6.12	21.21	5.86
1/11/2012 7:36	ppm	BLANK	0	0	NA	NA	< LOD	5.13	< LOD	3.73
1/11/2012 7:38	ppm	NCS-73308	27	25	105	91	28.42	6.15	22.63	5.92
1/11/2012 7:40	ppm	RCRA Standard	500	500	100	115	500.02	26.69	573.22	27.81
1/11/2012 15:55	ppm	BLANK	0	0	NA	NA	< LOD	5.35	< LOD	4
1/11/2012 15:57	ppm	NCS 73308	27	25	103	102	27.87	6.22	25.4	6.1
1/11/2012 16:01	ppm	RCRA Standard	500	500	102	111	509.77	26.87	555.84	27.77

Upon returning to the laboratory, a number of the samples were re-analyzed using all three energy filters of the XRF. The XRF was set to analyze each filter for 60 seconds for a total of 180 seconds, per sample. All analyses were run in duplicate. The following elements are detected with these three energy filters: Arsenic, Lead, Selenium, Copper, Zinc, Nickel, Cadmium, Mercury, Silver, Chromium, Iron, Manganese, Calcium, Potassium, Sulphur, Titanium, Uranium, Molybdenum, Zirconium, Rubidium, Paladium, Thorium, Strontium, Tungsten, Vanadium, Cobalt, Scandium, Barium, Cesium, Tellurium, Antimony and Tin.

First, several paired (surface/subsurface) locations were analyzed for the complete list of elements. In a second group of analyses, locations from various geologic formations, recommended by John Hillenbrand, were analyzed for the complete set of elements. The data is contained in the Appendix C and the attached CD in two files: IKHS_Lab XRF

paired reruns_January 2012.xls and IKHS_Lab XRF JH geological reruns_January2012.xls. The data includes the sample results as well as the quality control sample results for standard reference materials.

Appendix A

Locations for January 10, 2012

Longitude	Latitude	Location ID
-112.242308	34.50329887	001
-112.2101352	34.50253653	002
-112.2101228	34.50264937	003
-112.2057231	34.50242428	004
-112.205715	34.50237981	005
-112.2048737	34.50066577	006
-112.2010936	34.4990921	007
-112.2010933	34.4990918	008
No location info		009
-112.2004528	34.49910715	010
-112.1986902	34.49846771	011
-112.1986675	34.49845231	012
-112.1967026	34.49834166	013
-112.1967026	34.49834166	014
-112.1967026	34.49834166	015
-112.1968212	34.4996818	016
-112.1968212	34.4996818	017
-112.1949611	34.49791024	018
-112.1949611	34.49791024	019
-112.1940188	34.49829539	020
-112.1940188	34.49829539	021
-112.2115039	34.50274517	022
-112.2115039	34.50274517	023
-112.2123852	34.50250433	024
-112.2160218	34.50269222	025
-112.2200024	34.50253102	026
-112.2248862	34.50267445	027
-112.2248818	34.50282775	028
-112.2427347	34.49853962	029
-112.2422458	34.49851961	030
-112.2309352	34.50254096	031
-112.2231848	34.4977388	032
-112.2231582	34.49770473	033
-112.223192	34.49756279	034
-112.2220006	34.49747408	035
-112.2198977	34.49760528	036
-112.2198977	34.49760528	037
-112.2168604	34.49837951	038
-112.2115962	34.49760485	039
-112.2115962	34.49760485	040
-112.2536513	34.51799734	041
-112.255533	34.51921705	042
-112.2581668	34.51989532	043
-112.2602256	34.52082043	044
-112.262847	34.52221131	045
-112.2639938	34.5232242	046
-112.267823	34.52443006	047
-112.2536857	34.51772308	048
-112.2490187	34.51762414	049
-112.2491263	34.53165969	050
-112.2467138	34.53229034	051
-112.2467138	34.53229034	052
-112.2452024	34.51667892	053

Locations for January 11, 2012

Longitude	Latitude	Location ID
-112.2203949	34.50210433	054
-112.2203778	34.50213604	055
-112.220731	34.50432155	056
-112.2207656	34.5043197	057
-112.2207806	34.5041622	058
-112.2202552	34.50611623	059
-112.2220104	34.5063846	060
-112.2246655	34.50673859	061
-112.2248039	34.50672417	062
-112.2246788	34.50941095	063
-112.2198256	34.50796124	064
-112.2151351	34.50808539	065
-112.2206324	34.50755414	066
-112.2205756	34.50934894	067
-112.2205756	34.50934894	068
-112.2246399	34.51212041	069
-112.2246399	34.51212041	070
-112.2202468	34.51370797	071
-112.2162256	34.51321424	072
-112.2162256	34.51321424	073
-112.2073842	34.5134917	074
-112.2101748	34.51357756	075
-112.2101748	34.51357756	076
-112.2073305	34.51407838	077
-112.2073305	34.51407838	078
-112.2064191	34.51495116	079
-112.2064191	34.51495116	080
-112.2063317	34.51494827	081
-112.2142538	34.51357135	082
-112.2142538	34.51357135	083
-112.2201942	34.51729632	084
-112.2201942	34.51729632	085
-112.2139439	34.51815955	086
-112.2139439	34.51815955	087
-112.2083008	34.51947674	088
-112.2083008	34.51947674	089
-112.2245823	34.51706685	090
-112.2245823	34.51706685	091
-112.2273477	34.51513991	092
-112.2245725	34.5190199	093
-112.2266994	34.52277145	094
-112.2266994	34.52277145	095
-112.2200902	34.52470968	096
-112.2200902	34.52470968	097
-112.2203446	34.52766282	098
-112.220364	34.52768127	099
-112.2318612	34.53102358	100
-112.2287037	34.53492721	101
-112.2117291	34.53439283	102
-112.211791	34.53428569	103
-112.2088448	34.53169938	104
-112.2088448	34.53169938	105
-112.2145305	34.5275671	106
-112.2145305	34.5275671	107
-112.2140668	34.52614695	108
-112.214052	34.52608864	109
-112.2138619	34.52189065	110
-112.2138619	34.52189065	111
-112.2188607	34.52129919	112
-112.2188607	34.52129919	113
-112.2042303	34.51849327	114
-112.2085516	34.52298724	115
-112.2085516	34.52298724	116