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PILOT PLANT STUDIES OF ATMOSPHERIC AND PRESSURE LEACHING OF
URANIUM ORES IN ALKALINE SOLUTIONS

By

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ABSTRACT

During 1955 and 1956 numerous studies were made in the Alkaline Leach - Filtration Pilot Plant at Grand Junction, Colorado, comparing leach results in an autoclave and Pachuca tanks. These studies were made on several different ores including La Sal, Utex, Todilto Limestone, and Rio de Oro Dysart Shaft. The report presents the results of these studies and the effect of several variables such as contact time, temperature, and partial pressure of oxygen - a variable which is determined not only by the total pressure of the system and the vapor pressure of water, but also by the rate at which consumed oxygen is replaced. Estimated utility costs for operation of different leaching equipment used are also presented. The report concludes that increasing temperature reduces contact time appreciably, and total air addition can be reduced by use of higher pressures. Leaching under pressure requires less steam and power than at atmospheric pressure.

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INTRODUCTION

When the Alkaline Leach - Filtration Pilot Plant was constructed in Grand Junction, Colorado, Pachuca leach tanks were installed to investigate alkaline leaching under small hydrostatic pressures. During 1955 numerous leaching studies were made with the Pachuca tanks capped on top which permitted operation at 20 psig pressure. An autoclave was installed early in 1956 in order to evaluate the effect of higher pressures, up to 100 psig, on leaching ores in alkaline solution. The results of this work demonstrated the existence of and assisted in the evaluation of certain advantages and disadvantages of pressure leaching compared to atmospheric leaching.

Test work described in this report was performed to study the effects of several variables on leaching of uranium ores in carbonate solutions. Many of these variables had been investigated in laboratory equipment, but pilot plant tests were desirable to predict optimum operating conditions for future commercial mills. The results of these tests should serve as a guide in selecting the most efficient and economical method of leaching uranium ores amenable to a carbonate process.

SUMMARY

The most significant advantage of pressure leaching over leaching at atmospheric pressure is that higher pressures permit operation at higher temperatures, which appreciably reduce the required leach contact time and, accordingly, the size of vessels. Although oxygen partial pressure also affects the rate of leaching, temperature has a far more pronounced effect. When the pressure is increased from atmospheric to 20 psig and the temperature from 180°F to 220° in Pachuca tanks, leach retention times are cut roughly in half. At higher levels, in the range of 50 psig and 250°F and with mechanical agitation in an autoclave, retention time is cut from 10 to 20 times that required at atmospheric pressure, less total air addition is required, steam and power costs are lower, and space requirements are less. Autoclave pressure leaching is more versatile, permitting a range of operating conditions which best suit the ore. No significant difference in total extraction was found using atmospheric or pressure leaching.

Disadvantages of leaching under pressure are the increased cost of building a pressure vessel, somewhat more instrumentation, and the use of more complex mechanical equipment such as agitator shaft seals for an autoclave. Sodium carbonate consumption may be higher if appreciable amounts of sulfides are present in the ores. The presence of sulfides also may present a corrosion problem which should be considered in the design of the vessel.

Pilot Plant leaching costs for the utilities steam, air, and power are estimated to be \$0.75/ton for Pachucas operated at atmospheric pressure, \$0.73 for pressurized Pachucas, and \$0.52 for the autoclave. Estimated commercial mill utility costs for leaching, based on lower heat losses than experienced in the pilot plant and including heat recovery from the leached pulp are \$0.50, \$0.35, and \$0.24 per ton for Pachucas, pressurized Pachucas, and autoclaves, respectively.

CHEMISTRY OF ALKALINE LEACHING

A brief review of the chemistry of alkaline leaching will be helpful in the discussions to follow. Table I shows the important chemical reactions involved. The literature contains a number of detailed reports on the chemistry of alkaline leaching (3) (10) (11).

Table IChemistry of Alkaline Leaching

1. $2 \text{ UO}_2 + \text{O}_2 \longrightarrow 2 \text{ UO}_3$
2. $\text{UO}_3 + \text{H}_2\text{O} + 3 \text{ Na}_2\text{CO}_3 \longrightarrow \text{Na}_4\text{UO}_2(\text{CO}_3)_3 + 2 \text{ NaOH}$
3. $\text{Na}_4\text{UO}_2(\text{CO}_3)_3 + 4 \text{ NaOH} \longrightarrow \text{Na}_2\text{UO}_4 + 3 \text{ Na}_2\text{CO}_3 + 2 \text{ H}_2\text{O}$
4. $\text{NaOH} + \text{NaHCO}_3 \longrightarrow \text{Na}_2\text{CO}_3 + \text{H}_2\text{O}$
- (2 + 4) $\text{UO}_3 + \text{Na}_2\text{CO}_3 + 2 \text{ NaHCO}_3 \longrightarrow \text{Na}_4\text{UO}_2(\text{CO}_3)_3 + \text{H}_2\text{O}$
5. $\text{CaSO}_4 + \text{Na}_2\text{CO}_3 \longrightarrow \text{CaCO}_3 + \text{Na}_2\text{SO}_4$
6. $4 \text{ FeS}_2 + 15 \text{ O}_2 + 16 \text{ Na}_2\text{CO}_3 + 14 \text{ H}_2\text{O} \longrightarrow$
 $4 \text{ Fe(OH)}_3 + 8 \text{ Na}_2\text{SO}_4 + 16 \text{ NaHCO}_3$
7. $\text{SiO}_2 + 2 \text{ Na}_2\text{CO}_3 + \text{H}_2\text{O} \longrightarrow \text{Na}_2\text{SiO}_3 + 2 \text{ NaHCO}_3$

The first reaction shows the oxidation of uranium from the quadrivalent to the hexavalent form. Reaction 2 represents the dissolution of uranium by the carbonate. In the absence of bicarbonate, uranium will precipitate as a sodium uranate, reaction 3, as the hydroxide increases. Therefore, sodium bicarbonate is added, reaction 4, and the dissolution of uranium in the carbonate and bicarbonate solutions can be represented by the summation of equations 2 and 4. Reaction 5 is a carbonate consumer which usually eliminates the advantages of using alkaline leaching for ores containing appreciable amounts of gypsum. Although reactions 6 and 7 also consume Na_2CO_3 , part of the sodium ion results in NaHCO_3 which is useful to the process. The sodium sulfate is of no use and, as far as is known, has no effect on leaching. Sulfate concentrations up to 50 g/l have been experienced in the pilot plant with no detrimental effect and laboratory leach tests made with sulfate concentrations as high as 100 g/l showed no decrease in uranium extraction.

The silica reaction (No. 7) which is favored by high temperatures, above approximately 250°F, may decrease filtration rates and the silicate upon cooling may precipitate and occlude some uranium. However, in pilot plant autoclave leaching operations, no noticeable effect from the precipitation of silicates and occlusion of uranium has been experienced even at temperatures of 275°F. Filtration rates on residues from high temperature leaching have been somewhat lower than those from leach tests at 180°F (1). Although the exact reason for the lower filterability has not been established, the formation of silicates probably is contributory.

PROCESS EQUIPMENT AND FLOWSHEET DESCRIPTION

The flowsheet of the Alkaline Leach - Filtration Pilot Plant is shown in Figure 1 and has been described in detail in other reports (1) (9).

The ore, after being crushed to minus 3/16-in., is fed to a 3 x 5-ft ball mill operating in closed circuit with an 18-in. rake classifier. Recycled mill solution is used for grinding solution. The classifier overflow is pumped to a 12 x 8-ft thickener to thicken the pulp and to maintain a constant pulp density to the leach circuit.

Pachuca Tanks

The leach circuit consists of either four 4 x 47-ft Pachuca tanks in series or a 4.5 x 12-ft autoclave. A diagram of one of the Pachuca tanks is shown in Figure 2, and Figure 3 is a photograph of the Pachuca tanks. The four Pachucas provide a leach contact time of 96 hours with a 50 per cent solids slurry at a feed rate of 12 tons of ore per day. Each Pachuca has a 3-in. internal air lift for pulp circulation, two 1/4-in. pipes in the cone for adding aeration air, and four 1-in. steam pipes to maintain desired pulp temperatures. The vessels are insulated with one-inch Fiberglas and a corrugated aluminum covering. The 45-ft elevation to the discharge level provides a pressure of approximately 30 psig in the bottom of the tank when filled with a 50 per cent solids pulp. Although the Pachucas are usually operated at atmospheric pressure on top, the installation of a pressure regulating valve on the top of each tank has permitted operating under pressure up to 20 psig on top which results in a pressure of 50 psig at the bottom. The Pachucas have been operated continuously and batch-wise.

Figure 2
PACHUCA TANK

Figure 3

Pilot plant Pachuca leach tanks

Autoclave

To investigate leaching at higher temperatures and pressures than were possible in the Pachucas, an autoclave was installed in the pilot plant early in 1956. Figure 4 is a sketch of the pilot plant autoclave and Figure 5 is a photograph of the installation. When the autoclave is in operation, it replaces the Pachucas in the circuit. Pulp is heated by steam in a double pipe heat exchanger. Steam flows through the internal $1\frac{1}{2}$ -in. pipe and pulp flows through the external 3-in. pipe. Six passes approximately 20 ft long are used although the capacity of the heat exchanger is considerably greater than is necessary for the 14-ton-per-day feed rate used to date. At this rate, retention time in the autoclave is approximately 6.5 hours at 50 per cent solids. The pulp is maintained at the desired leaching temperature by automatically controlled electrical strip heaters fastened to the bottom of the autoclave between the shell and the insulation. However, only a small amount of heat is required or used for this purpose.

The horizontal autoclave is $4\frac{1}{2}$ ft in diameter, 12 ft long, with $1\frac{1}{2}$ -ft dished heads, and is divided into three sections by double baffle plates. Each section is agitated with a 5 hp turbine-type agitator equipped with a 5-bladed 16-in. turbine. Capacity of the vessel is approximately 1200 gallons. Air is added through a $\frac{1}{2}$ -in. line discharging beneath each agitator. The quantity of air input is regulated by an automatic control valve. A spring-loaded reducing valve controls the pressure in the autoclave. Pulp is discharged by the internal pressure through an intermittently operated plug valve. The discharge valve is operated automatically by a pulp level control and is open approximately one minute at 20-minute intervals. Temperature and pressure losses in the discharge line between the autoclave and the top of a Pachuca which is used as a surge vessel dissipate sufficient heat and pressure that very little flashing of steam takes place at the discharge. As yet, no attempt has been made to utilize the heat in the discharging pulp for pre-heating the incoming pulp. It is interesting to note that the Canadians at the Beaverlodge mill estimate a savings in heating costs of 20 cents per ton by this method of heat recovery from autoclave leach pulp (7).

The leached pulp is discharged from the Pachucas or autoclave to the first of three 4 x 8-ft cloth-covered vacuum drum filters operating in series. Provisions are made to add filtering aids to the pulp feed for each filter. The filtrate from the first filter is pregnant liquor containing the solubilized uranium. This liquor is pumped through a plate and frame clarification press and then to the precipitation tank. The solids residue is washed on the second and third filters and is pumped to tailings. The repulp solution and wash to the second filter is barren filtrate from the regeneration tank. The solids fed to the third filter are repulped and washed

Figure 4
AUTOClave

Figure 5

Pilot plant 1200-gallon autoclave

with water. Filtrate from the second filter is returned to the grinding circuit, and filtrate from the third filter is used as wash on the first filter.

Uranium is precipitated as sodium diuranate by adding an excess of caustic soda to the clarified pregnant liquor. This slurry is then filtered in a press and the cake, after being washed, is dried, barreled, sampled, and shipped. The barren filtrate is regenerated to the desired bicarbonate concentration with carbon dioxide from a submerged oil burner. It is then used for washing on the second filter, and is pumped to a mill solution tank where the carbonate concentration is brought up to the desired level by the addition of soda ash. The solution has now completed its cycle and is ready to be used again in the grinding circuit.

FACTORS AFFECTING LEACH

The following factors or variables are of significant importance in alkaline leaching (2) (6) (11), most of which have been studied rather extensively in the pilot plant and are discussed below:

1. Leach contact time
2. Leach temperature
3. Partial pressure of oxygen
4. Sodium carbonate and bicarbonate concentration
5. Agitation of pulp
6. Fineness of ore grind
7. Pulp density

At atmospheric pressure relatively long contact times are required for alkaline leaching - 50 to 100 hours. For good uranium extraction, a hot leach is required and increasing temperatures above 180°F, up to 250 to 275, decreases the contact time required to 6 to 10 hours. Oxidation of the reduced uranium in the ore can be accomplished chemically, usually by KMnO_4 , or by air. Higher pressures increase the solubility of oxygen in the leaching solution through increased partial pressure and permit the use of less total air addition. Reagent concentrations are usually maintained in the range of 40 - 60 g Na_2CO_3 and 10 - 20 g NaHCO_3 per liter. Agitation should be adequate to keep the pulp in a constant and thorough fluid regime. The dispersion of air throughout the pulp is also dependent on the mixing device. Most ores require grinding to grain size in order to permit contact of the uranium particles with the leach solution. Grinds usually average 1 to 5 per cent plus 65 mesh and 50 to 75% minus 200 mesh. Pulp densities are usually

controlled between 40 and 55 per cent solids, a range which permits adequate mixing and prevents rapid settling of the coarser sands.

1. Leach Contact Time

The required contact or retention time of the ore in the leach solution is dependent upon temperature, efficiency of oxidation and agitation, and, to an extent, on the other variables. Figure 6 shows the results of alkaline leach studies conducted on La Sal ore, from the Big Indian Wash area of Utah (4), in an autoclave (250°F), pressurized Pachucas (220°F), and Pachucas at atmospheric pressure (180°F). Contact times required to reach equivalent extractions, about 97 per cent, were approximately 7, 35, and 70 hours. Of the total leach extraction, a large part takes place in the first few hours providing sufficient oxidation and agitation are present, but a much longer time is required to raise the extraction to a leveling-off point. Pilot plant results at higher pressures have shown even a more pronounced effect with 95 per cent of the total leaching taking place within the first one or, at most, two hours.

2. Leach Temperature

Figure 7 shows the comparison of autoclave and Pachuca leaching and the relationship between increased temperatures and pressures on extraction and contact time. These studies were made while processing Todilto Black Limestone from the Grants, New Mexico, area (5) and Rio de Oro Dysart Shaft ore from the Ambrosia Lake Area of New Mexico (1). Todilto Limestone contained approximately 81% CaCO_3 and Rio de Oro ore treated in the pilot plant assayed 7.5% CaCO_3 . Leaching at atmospheric pressure and 180°F required 10 to 20 times the contact time necessary at elevated pressures and at temperatures in the range of 255 to 265°F. Similar relationships are plotted in Figure 8 where results from tests in the autoclave are compared with tests in mechanically agitated leach tanks operating at atmospheric pressure (Alkaline Leach - Resin-In-Pulp Pilot Plant leach circuit) (8). These leaching studies, on Utex ore (a Big Indian Wash ore), and a somewhat different Todilto Limestone ore than is shown in Figure 7, indicate a reduction of 40 to 45 hours leach time by increased pressures and temperatures.

In nearly every study cited above, approximately the same extraction on a given ore is obtained providing the leach time is long enough. In summary, by using 20 psig top pressure on Pachuca tanks and increasing the temperature to 220°F, the contact time is cut in half. The use of 250 to 275°F and higher pressures obtainable in autoclaves reduced the required leach time 10 to 20 times from that required at atmospheric pressure and 180°F. As long as oxidation is adequate, temperature rather than pressure is the major factor which affects extraction in a given time. Increased pressures, of course, are

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Figure 6
Effect of Temperature and Pressure on Contact Time
La Sal Ore

Figure 7

Contact Time vs. Temperature and Pressure
Pachucas and Autoclave

Figure 8

Contact Time vs. Temperature and Pressure
Autoclave and Open Tanks

necessary for operation at temperature over the boiling point.

3. Partial Pressure of Oxygen

Some effects of pressure have been discussed already as it is impossible to evaluate any one of the several variables without considering others. During the latter months of 1956, a reasonably thorough study was made in the Alkaline Leach - Filtration Pilot Plant on the effect of temperature, pressure, and quantity of air on uranium extraction. The pertinent data from a series of autoclave tests are shown in Table II and the more important results are shown graphically in Figures 9 and 10. Daily operating data for these tests are shown in Appendix Tables 1 and 2.

In examining these leach studies, a few theoretical considerations should be kept in mind. Effectively no uranium in the reduced state will be extracted from the ore without oxidation. Oxidation can be accomplished by chemical means, usually KMnO_4 in alkaline solutions, or by air as was done in all tests in the pilot plant. The solubility of oxygen increases approximately directly proportional to its partial pressure, i.e.

$$X_{O_2} = \frac{P_{O_2}}{H} \quad \text{where} \quad \begin{array}{l} X = \text{solubility} \\ P_{O_2} = \text{partial pressure of oxygen} \\ H = \text{proportionality constant} \end{array}$$

As temperature increases, the vapor pressure of water increases, which effectively reduces the oxygen partial pressure for any given total pressure on the system.

If air were not added continuously, the partial pressure of oxygen would not remain constant throughout the leach because a significant quantity of oxygen is consumed. Consequently, air is flowed through the system to replenish this oxygen. The most suitable flow would depend only on the rate at which oxygen reacts if pure oxygen gas were being supplied, but since air is used, the whole gaseous phase must be replaced frequently in order to keep the partial pressure of oxygen at its maximum for the given conditions of temperature and total pressure.

During the autoclave studies the lowest extractions (92 to 93 per cent) were obtained at a total pressure of 30 psig even though the temperature was increased from 225 to 250°F and the air was also increased. When the pressure was raised to 50 psig and the temperature to 250°F, extraction increased to over 96 per cent. Higher air rates at these conditions gave no appreciable improvement. Increasing the temperature to 270 to 275°F dropped extraction to 93 per cent but higher air rates at these temperatures showed a significant improvement. Comparing Tests 5 to 7 with 8 to 10 in Table II clearly shows the effect from increasing temperatures without a corresponding increase in pressure. Extraction at a total pressure of 70 psig and a temperature of 250 to 270°F was no better than at 50 psig and 250°F.

The optimum temperature and pressure for leaching La Sal ore in the autoclave can be determined from Figures 9 and 10. A temperature of 250°F, a pressure of 50 psig, and air addition between 800 and 1000 scf/ton of ore resulted in highest extractions. It should be noted, however, that in Test 12 of Table II where air addition was reduced to 183 scf/ton and with 70 psig pressure and 250°F, extraction was reduced only slightly, to 96 per cent.

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Table II

Autoclave Leach Studies on La Sal Ore

Pulp Density: 50% solids
Average Grind: 1.1% +65 mesh, 71.5% -200 mesh
Reagent Concentration (Grinding Solution): 49 g Na₂CO₃/l, 8 g NaHCO₃/l
Ore Feed Rate: 11.8 tons/day

Test No.	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>	<u>13</u>	<u>14</u>	<u>15</u>
Ore head assay, % U ₃ O ₈	.350	.307	.294	.354	.328	.323	.332	.309	.346	.358	.349	.352	.402	.437	.376

Leach Conditions^{1/}

Total pressure, psig	30	33	30	<u>50</u>	53	54	51	55	53	53	<u>70</u>	70	70	70	70
Temperature, °F	225	<u>250</u>	250	<u>225</u>	<u>251</u>	250	250	<u>274</u>	271	273	<u>225</u>	<u>250</u>	250	250	<u>267</u>
Air, scf/ton ore	952	<u>665</u>	<u>1060</u>	<u>990</u>	<u>738</u>	<u>1110</u>	<u>1475</u>	<u>738</u>	<u>1110</u>	<u>1475</u>	<u>990</u>	<u>183</u>	<u>1100</u>	<u>2080</u>	<u>990</u>

Extraction

Final residue, % U ₃ O ₈	.025	.023	.021	.018	.012	.011	.013	.022	.017	.018	.016	.014	.014	.014	.014
U ₃ O ₈ extraction, %															
after 2.4 hr	88.3	88.9	89.5	88.7	93.9	94.1	93.4	89.6	92.2	92.7	90.8	93.5	94.8	94.7	94.1
after 4.8 hr	91.1	91.2	91.8	92.9	95.3	96.0	95.5	91.6	94.2	94.4	94.6	95.2	96.0	96.1	95.7
after 7.2 hr	92.9	92.5	92.9	94.9	96.3	96.6	96.1	92.9	94.5	95.0	95.4	96.0	96.5	96.8	96.3

Terminal Reagent Concentration

g Na ₂ CO ₃ /l	39.1	40.3	39.0	35.0	39.7	38.3	41.1	36.1	39.7	41.9	37.5	40.1	40.5	37.6	35.0
g NaHCO ₃ /l	10.5	12.6	12.0	13.5	13.2	13.3	13.1	16.7	15.2	13.8	14.0	20.8	19.3	17.9	16.3

^{1/} Underline indicates major variable changed from previous test.

Figure 9

Effect of Temperature and Pressure, Autoclave Studies
La Sal Ore

Figure 10

Effect of Air Addition at 50 psig Total Pressure,
Autoclave Studies on La Sal Ore

The Pachuca studies, shown in Table III and Figure 11, were made primarily to determine the effect of varying quantities of aeration air at temperature levels of 180° and 220°F. Aeration air is usually held in the range of 20 scfm per Pachuca (1.6 scfm/sq ft cross sectional area). Between 3.0 and 5.0 scfm is used in each Pachuca air lift. Major changes in aeration air rates do not change the degree of agitation in the Pachuca appreciably as this is affected primarily by the air supplied to the air lift. As can be noted in Table III and Figure 11, reducing the aeration air below 20 scfm per Pachuca lowered extraction. However, 3320 scf of air per ton with a 32-hour leach at 20 psig and 220°F was as effective as 5000 scf per ton with a 48-hour leach at atmospheric pressure and 180°F. For any given level of extraction, it is apparent from Figure 11 that increasing the total air decreased the required contact time.

In general, uranium recovery decreased as the aeration air rate was decreased. It is evident from these data that extraction rates increased with higher temperature and pressure. As in the autoclave, the dissolution rate increases with temperature and oxygen partial pressure. However, temperature changes were not enough to significantly affect the oxygen solubility. Daily grinding and leach data are shown in Appendix Table 3 and 4, respectively.

4. Sodium Carbonate and Bicarbonate Concentration

Reagent concentrations in alkaline leach solutions are usually controlled in the range of 40 to 60 g Na_2CO_3 and 10 to 20 g NaHCO_3 per liter. They may be varied over a wider range, particularly much higher in carbonate strength, but pilot plant studies have shown no major effect on extraction between 25 and 60 g Na_2CO_3 /l and 5 to 25 g NaHCO_3 /l. In one study the concentrations were reduced to 21 g Na_2CO_3 and 6 g NaHCO_3 per liter and no decrease in extraction was experienced. However, at these low concentrations filtration rates were reduced appreciably. The bicarbonate concentration up to a limit has been shown to effect extraction, though it is present primarily to prevent precipitation of the uranium. The optimum amount of NaHCO_3 should be determined and the concentration held at that level because the higher the NaHCO_3 concentration in the pregnant liquor, the more caustic soda will be required for precipitation.

In the pilot plant, it has been noted that with carbonate consuming ores, more carbonate is consumed - forming Na_2SO_4 and NaHCO_3 - in pressure leaching than at atmospheric pressure. On some ores the bicarbonate strength in the grinding solution has been as low as 5 g/l, yet the solution will leave the leaching step at 15 g/l or higher. Other ores will result in a complete reversal of this chemistry and produce some carbonate at the expense of the bicarbonate.

The amount of uranium in the leaching solution did not have an effect on extraction in the pilot plant. During one period of operation, a large portion of pregnant liquor was recycle to the grinding circuit and a uranium concentration of 15 g/l had no detrimental effect on leaching.

Table IIIPachuca Leach Studies on La Sal Ore

Pulp Density: 50% solids
 Average Grind: 2.3% +65 mesh, 68.1% -200 mesh
 Reagent Concentration (Grinding Solution): 52 g Na₂CO₃/l, 9 g NaHCO₃/l
 Ore Feed Rate: 17.4 tons/day

Test No.	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
Ore head assay, % U ₃ O ₈	.429	.434	.379	.350	.394

Leach Conditions^{1/}

Top pressure, psig	0	0	<u>20</u>	20	20
Temperature, °F	183	182	<u>217</u>	222	222
Air ^{2/} , scf/ton ore	2500	<u>5000</u>	<u>830</u>	<u>1660</u>	<u>3320</u>
Number of Pachuca	3	<u>3</u>	<u>2</u>	<u>2</u>	<u>2</u>

Extraction

Final residue, % U ₃ O ₈	.018	.009	.023	.011	.009
U ₃ O ₈ extraction, %					
after 16 hr	77.9	88.7	82.3	87.4	92.9
after 32 hr	91.6	95.9	93.9	96.9	97.7
after 48 hr	95.8	97.9	-	-	-

Terminal Reagent Concentration

g Na ₂ CO ₃ /l	48.6	49.9	48.7	45.0	44.6
g NaHCO ₃ /l	13.9	12.6	11.4	12.0	14.6

1/ Underline indicates major variable changed from previous test.

2/ In addition to air lift air which amounted to 4 scfm/Pachuca

Figure 11

Pachuca Leach Studies
La Sal Ore

Reagents consumed in the Alkaline Leach - Filtration Pilot Plant are caustic soda, soda ash, and fuel oil. Sodium bicarbonate is made from the carbonate with CO_2 generated from a submerged oil burner. For easy comparison of reagent consumptions on various ores or at different leach conditions, the sodium carbonate is calculated on an equivalent caustic basis and total reagent consumption is reported as equivalent pounds of NaOH per ton of ore. On most ores treated in the pilot plant this figure has been very close to 30 lb NaOH /ton. The caustic consumption can be broken down as an average of 16 lb Na_2CO_3 and 18 lb NaOH per ton ore. Fuel oil consumption for regeneration will vary between 0.4 and 0.6 gal No. 2 fuel oil per ton. Reagent consumption data from the amenability test on Rio de Oro Dysart Shaft ore (1), in which the ore was treated in the autoclave and the Pachucas, indicate higher reagent consumption in the autoclave than in the Pachuca tanks, from 5 to 8 lb more NaOH per ton of ore treated. This difference is under further investigation.

5. Agitation of Pulp

Agitation in atmospheric leach tanks and autoclaves is usually provided by mechanically driven agitators. Of course, a packing gland around the agitator shaft must be provided in autoclaves. Pachuca leaching utilizes internal air lifts for agitation of the pulp. Air lifts are advantageous in that no moving parts are involved. However, properly designed mechanical agitators assure far more complete mixing. Where mechanical agitators will tend to break down particle size somewhat, air lifts have little tendency to do so.

One of the more important effects of the type of mixing device used is the efficiency by which oxygen is dispersed in the pulp. An agitator which efficiently mixes the pulp will tend to break the air bubbles into small ones thus increasing the air to pulp surface contact area. The liquid film surrounding the air bubbles is reduced in thickness by thorough agitation which increases the mass transfer coefficient of oxygen through the film and into solution. Greater oxidation effect of the air is also accomplished if, through complete mixing, the air is retained in the pulp a longer time and bubbles of air do not rise immediately to the surface from their point of discharge into the pulp.

Some comparisons of leaching in Pachucas and in conventional mechanically agitated leach tanks (8) indicate contact time is reduced appreciably by more thorough agitation. Considerably more investigative work need to be done to determine the most efficient method of introducing the air and the effect of various methods of agitation on extraction.

6. Fineness of Grind

In acid leaching, a grind which reduces all the material to minus 28 or minus 35 mesh is usually sufficient to permit good uranium extraction. However, carbonate liquors do not tend to dissolve interstitial material and it is necessary to grind to the grain size of the ore to permit contact of the uranium particles with carbonate solution. Pilot plant grinding which reduces the ore to 2 per cent plus 65-mesh and 70 per cent minus 200-mesh material has been adequate for high uranium extraction.

7. Pulp Density

The degree of agitation is related to pulp density. If pulp densities are too high to permit adequate mixing, extraction will suffer. When using air agitation, such as air lifts in the Pachucas, pulp densities greater than 50 per cent solids can reduce extraction, other factors such as temperatures and contact time being equal. An example of this is one test made in the Pachuca tanks on Rio de Oro ore (1). At 51 per cent solids, extraction was 95.4 per cent in 60 hours time and 96.5 in 100 hours. When leaching at 59 per cent solids, extractions were 94.7 per cent in 60 hours and 95.4 in 100 hours, showing approximately a 1 per cent lower recovery or 0.05 lb U_3O_8 /ton.

AUTOCLAVE CORROSION

The Canadians at the Beaverlodge mill have experienced a pitting type of corrosion in their autoclaves. The corrosion takes place at the froth or splash line in the vessel and little or no corrosion has been observed elsewhere. The attack is attributed to the high sulfide content of the ore and takes place in the autoclaves near the head of the leach circuit. The reaction is thought to be from sulfurous or sulfuric acid which is formed from the sulfide as the pulp which splashes on the walls is diluted by condensed water vapor. The solution near this froth area is changed from alkaline to acidic.

An autoclave containing a clad belt of low carbon, type 316, stainless steel in the area of the pulp level could minimize this corrosion problem. The possibilities of electrolytic corrosion are also being investigated in Canada.

National Lead Company's pilot plant autoclave has been in operating service four months during which 1400 tons of ore have been leached. A recent inspection of the autoclave showed no signs of corrosion at or below the pulp level. Above the pulp level there were several deposits of hard mud-like material. When these deposits were scraped off, there were indications of pitting corrosion beneath some but not all of the deposits. The majority of the pits were so small

or shallow that they were not measured. It was noted during the inspection that the deposits were acidic, testing in the range of pH 5 to 6 with litmus paper. Sulfide content of the ores treated in the pilot plant has been considerably less than those treated in autoclaves at Beaverlodge.

UTILITY COSTS

During more recent pilot plant operation, studies have been underway to determine the quantity of steam, air, and power required. A summary of average utility consumption figures is shown in Table IV along with a rough estimate of the cost of steam and power for the autoclave and for Pachuca tanks operated with and without pressure applied on top. The leaching conditions shown resulted in essentially the same uranium extraction, 97 per cent, on La Sal ore.

Table IV

Pilot Plant Steam and Power Costs

<u>Conditions</u>	<u>Pressurized</u>		
	<u>Pachucas</u>	<u>Pachucas</u>	<u>Autoclave</u>
Ore, tons/day	17.4	17.4	11.8
Leach time, hr	48	32	7
Pressure, psig	0	20	50
Temperature, °F	180	220	250
Air, scf/ton ore	6000	3980	1110
Steam, lb (100 psig)/ton	800	820	430
Power for agitation, KWH/ton	-	-	12.9
Power for air, KWH/ton	8.7	5.7	1.62
Total power, KWH/ton	8.7	5.7	14.5

Estimated Operation Costs Per Ton

Cost of steam at 80¢/1000 lb, \$	0.64	0.66	0.35
Cost of power at 1.2¢/KWH, \$	0.10	0.07	0.17
Cost of steam and power, \$	0.74	0.73	0.52

With Pulp Heat Recovery System - calculated figures

Steam, lb (100 psig)/ton	800	660	217
Cost of steam at 80¢/1000 lb, \$	0.64	0.53	0.17
Cost of steam and power, \$	<u>0.74</u>	<u>0.60</u>	<u>0.34</u>

Steam consumption in the Pachucas was almost twice that for the autoclave, 800 to 820 lb/ton compared with 430 lb. Theoretical heat balances did not corroborate the pilot plant figures for the Pachucas. Further investigation showed that the discrepancies were due to high heat losses from the Pachucas and the quantity of heat expelled in the form of water vapor in the air leaving the top of the Pachucas. Measurements made during operation of the Pachucas at atmospheric pressure indicated as much as 200 lb water/ton ore was lost with the air leaving the four vessels. It is estimated that heat losses from the Pachucas due to radiation and convection are approximately 35 per cent of the total heat input. The radiation area per unit volume of pulp in the pilot vessels, of course, is very large (approximately 5 times) compared to commercial size tanks. In commercial mills a large amount of the heat in the pulp leaving the leach circuit operated at elevated temperatures would be recovered both for reasons of heat economy and to prevent excessive flashing of steam. The steam consumptions at the bottom of Table IV are calculated on the basis of recovery of heat from the exit pulp, 250° in the autoclave and 220° in the pressurized Pachucas, down to a temperature of 150°F. Further cooling is not desirable as it would be detrimental to filtration. Were the heat recovery equipment in use in the pilot plant, steam consumptions by calculations would be 800 lb/ton for the Pachucas, 660 lb/ton for pressurized Pachucas, and 217 lb/ton for the autoclave. At a steam cost of \$0.80 per 1000 pounds, a savings in pilot plant steam costs of approximately \$0.47 per ton of ore could thus be realized by operation of the autoclave and \$0.11 by the pressurized Pachucas. Theoretical heat balance figures for a commercial mill indicate steam use for leaching will be 500 lb/ton for Pachucas, 350 lb/ton for pressurized Pachucas, and 200 to 250 lb/ton for autoclaves.

Power requirements in the autoclave for air compression and agitation are higher than for the Pachucas. Mixing in the autoclave is, of course, very thorough compared to the Pachucas. Although no attempt has been made to determine if the agitation in the autoclave is in great excess, it is estimated that the same agitators, total of 15 connected horsepower, would be adequate for a vessel of two or three times the size of the existing one which has a capacity of 1200 gallons.

Applying reasonable commercial cost figures to the two utilities, steam and power, pilot plant operational costs would be \$0.74, \$0.73, and \$0.52 per ton for Pachucas, pressurized Pachucas, and autoclave, respectively. With heat recovery from the leached pulp, these figures are calculated to be \$0.74, \$0.60, and \$0.34.

Assuming: (1) that the previously mentioned mill steam consumption figures based on heat balances are reasonable, (2) that the power requirements from the pilot plant Pachucas are applicable for a commercial mill, and (3) that 40 per cent of the pilot plant autoclave power for agitation would be required in a commercial autoclave, then utility costs for the autoclave will be \$0.11 per ton cheaper than for pressure Pachucas and \$0.26 per ton cheaper than for Pachucas operated at atmospheric pressure. The estimated mill figures for Pachucas, pressurized Pachucas, and autoclave in this case would be \$0.50, \$0.35, and \$0.24 per ton, respectively.

To select the best leach vessel for the job, these costs of operation must be balanced against other factors such as type of ore to be treated, capital cost, maintenance, space requirements, reagent consumption, filterability of leached pulp, and the variety of ores to be treated.

Pilot plant amenability tests have shown the autoclave to be more versatile than the Pachucas in handling different ores. Where the leach time for best extraction in the Pachucas has varied from 48 to 120 hours, 6 to 7 hours retention time in the autoclave has been adequate for all ores treated. A Pachuca not under pressure has a limited maximum operating temperature. An autoclave permits a fairly wide range of operating temperatures and pressures and leaching conditions can be varied to suit the ore.

Pilot plant tests using the Pachucas under pressure have not been extensive. However, the available results indicate the advantages of operating at a small pressure on top justify any additional initial investment over Pachucas designed to operate at atmospheric pressure. Actually, pressurized Pachucas will probably be cheaper to build for although somewhat heavier plate will be required for construction, there will be fewer Pachucas required.

APPENDIX

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Table 1Daily Grinding and Thickening DataLa Sal Ore Autoclave Studies

Test No.	Date 1956	Dry Tons	Belt Feed % U_3O_8	Grind, %		Grinding Sol'n, g/l		Autoclave Feed % Solids
				+ 65 mesh	-200 mesh	Na_2CO_3	$NaHCO_3$	
1	9/27	12.0	.336	0.6	71.3	44.7	6.0	50.0
	9/28	11.8	.326	1.1	70.9	45.6	8.3	50.8
	9/29	11.8	.413	0.8	72.8	47.4	6.3	50.8
	9/30	11.8	.325	0.6	70.4	48.2	8.4	50.0
2	10/6	12.0	.324	1.0	74.5	48.5	8.0	50.0
	10/7	11.9	.325	0.1	71.9	50.1	7.6	50.0
	10/8	12.0	.320	0.6	71.3	48.8	8.0	50.0
	10/9	11.9	.257	1.0	71.5	49.0	9.3	50.8
3	10/1	10.5	.277	1.0	68.4	40.4	4.4	50.0
	10/2	12.1	.296	0.9	71.2	47.7	8.5	50.8
	10/3	7.3	.267	1.6	69.6	49.5	6.9	50.0
	10/4	12.9	.326	1.9	68.0	47.0	8.7	50.8
	10/5	16.0	.305	0.8	74.3	42.5	6.1	50.8
4	9/22	12.1	.388	0.7	71.8	44.9	7.4	50.8
	9/23	12.1	.331	1.3	71.4	45.8	7.6	50.8
	9/24	11.9	.344	1.1	73.0	44.2	7.3	50.0
5	10/11	11.8	.263	1.2	70.4	51.0	4.0	49.3
	10/12	11.8	.367	0.9	72.7	49.5	10.3	50.0
	10/13	11.8	.357	1.3	69.6	50.0	8.6	50.0
	10/14	12.1	.325	0.8	72.2	47.2	8.8	50.0

Table 1 (contd)

Daily Grinding and Thickening Data

La Sal Ore Autoclave Studies

Test No.	Date 1956	Dry Tons	Belt Feed % U_3O_8	Grind, %		Grinding Sol'n, g/l		Autoclave Feed % Solids
				+ 65 mesh	-200 mesh	Na_2CO_3	$NaHCO_3$	
6	10/19	11.9	.335	1.0	68.8	47.3	8.1	50.8
	10/20	11.9	.340	0.8	71.0	49.1	8.7	50.0
	10/21	12.2	.296	0.9	74.3	46.2	8.7	49.3
7	10/30	11.8	.332	-	-	53.0	8.0	50.8
	10/31	3.4	-	-	-	-	-	50.0
8	10/16	11.8	.305	1.0	69.2	47.2	-	50.8
	10/17	11.8	.313	1.2	70.4	45.2	5.2	49.3
9	10/23	11.9	.320	1.1	72.3	48.7	8.1	50.8
	10/24	11.8	.375	1.3	69.0	50.5	5.9	50.0
	10/25	11.9	.342	1.5	67.7	50.6	5.9	49.3
10	10/27	11.7	.347	1.4	70.2	51.0	6.1	49.3
	10/28	11.7	.380	1.2	71.8	50.5	6.6	50.0
	10/29	11.9	.346	1.6	72.3	49.1	7.6	49.3
11	9/18	11.2	.348	0.9	71.1	45.4	6.9	50.0
	9/19	11.8	.357	0.9	71.1	47.4	8.0	50.0
	9/20	11.7	.341	1.1	75.1	46.7	7.0	50.8
12	8/27	11.7	.357	0.8	74.1	47.8	13.3	50.0
	8/28	11.7	.352	1.0	73.0	55.2	14.3	49.3
	8/29	11.8	.331	0.9	76.7	49.5	12.3	50.0
	8/30	12.1	.368	2.7	71.1	50.5	11.2	49.3

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Table 1 (contd)

Daily Grinding and Thickening Data

La Sal Ore Autoclave Studies

<u>Test No.</u>	<u>Date 1956</u>	<u>Dry Tons</u>	<u>Belt Feed % U₃O₈</u>	<u>Grind, %</u>		<u>Grinding Sol'n, g/l</u>		<u>Autoclave Feed % Solids</u>
				<u>+ 65 mesh</u>	<u>-200 mesh</u>	<u>Na₂CO₃</u>	<u>NaHCO₃</u>	
13	8/31	11.8	.389	1.3	73.8	54.2	11.6	50.0
	9/1	11.9	.407	0.8	83.1	53.0	12.2	50.8
	9/2	12.0	.410	0.8	74.4	52.1	14.1	49.3
14	9/7	12.1	.408	0.9	72.8	47.8	9.7	50.0
	9/8	12.0	.494	1.1	71.9	44.8	12.1	50.0
	9/9	11.8	.408	1.0	70.7	44.0	9.2	50.0
15	9/13	12.1	.334	1.7	72.0	49.7	6.6	49.3
	9/14	11.8	.347	1.1	72.1	46.6	5.6	50.0
	9/15	11.9	.410	0.7	75.1	47.0	8.6	49.3
	9/16	12.0	.413	1.6	66.3	47.0	9.0	50.0

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Table 2

Daily Leach Conditions and Results

La Sal Ore Autoclave Studies

Test No.	Date 1956	Pressure psig	Temperature °F	Air scfm/ Section	Steam lb/ton	Residue, % U ₃ O ₈ After			Leach Disch, g/l	
						2.4 hr	4.8 hr	7.2 hr	Na ₂ CO ₃	NaHCO ₃
1	9/27	31	224	2.6	212	.043	.029	.024	39.1	9.0
	9/28	30	225	2.6	290	.043	.034	.024	39.2	8.9
	9/29	30	225	2.6	339	.037	.029	.024	39.9	13.2
	9/30	30	225	2.6	291	.042	.033	.027	38.0	11.1
2	10/6	33	250	1.8	-	.035	.030	.024	40.4	12.7
	10/7	33	250	1.8	381	.039	.033	.028	40.9	11.9
	10/8	33	250	1.8	361	.029	.023	.022	39.6	13.3
	10/9	33	250	1.8	347	.033	.022	.021	40.5	12.3
3	10/1	31	232	2.9	-	.033	.023	.021	38.5	11.0
	10/2	33	246	2.9	257 (?)	.027	.018	.017	36.5	13.6
	10/3	34	250	2.9	455	.033	.026	.022	38.2	11.5
	10/4	34	250	2.9	-	.027	.021	.018	38.7	12.8
	10/5	34	250	2.9	-	.037	.029	.024	40.5	12.3
4	9/22	48	225	2.7	234	.029	.023	.020	35.8	13.5
	9/23	48	225	2.7	283	.040	.028	.018	35.3	13.7
	9/24	48	226	2.7	290	.062	.020	.016	34.0	12.2
5	10/11	52	252	2.0	424	.020	.012	.012	38.9	16.8
	10/12	54	252	2.0	277	.018	.014	.012	39.8	14.2
	10/13	54	251	2.0	405	.019	.013	.012	41.0	11.0
	10/14	52	250	2.0	326	.023	.018	.014	39.2	11.0

Table 2 (contd)

Daily Leach Conditions and ResultsLa Sal Ore Autoclave Studies

Test No.	Date 1956	Pressure psig	Temperature °F	Air scfm/ Section	Steam lb/ton	Residue, % U_3O_8 After			Leach Disch, g/l	
						2.4 hr	4.8 hr	7.2 hr	Na_2CO_3	$NaHCO_3$
6	10/19	53	248	3.0	473	-	.014	.012	38.5	12.6
	10/20	54	250	3.0	408	.020	.013	.011	37.2	14.9
	10/21	54	251	3.0	380	.018	.013	.011	39.0	12.9
7	10/30	52	251	4.0	461	.022	.015	.013	40.8	13.2
	10/31	50	250	4.0	375	.022	.014	.013	41.5	12.9
8	10/16	56	273	2.0	417	.030	.024	.020	35.8	18.5
	10/17	54	274	2.0	-	.037	.030	.025	34.1	15.2
9	10/23	55	273	3.0	464	.024	.018	.017	37.9	17.6
	10/24	54	268	3.0	486	.027	.020	.016	41.0	14.1
	10/25	50	272	3.0	475	.029	.021	.018	40.0	13.9
10	10/27	53	275	4.0	626	.025	.018	.017	41.9	14.3
	10/28	54	273	4.0	648	.027	.021	.019	41.9	13.5
	10/29	53	258	4.0	-	-	-	-	41.2	13.5
11	9/18	70	225	2.7	269	.036	.019	.016	36.9	14.5
	9/19	70	226	2.7	272	.025	.017	.014	38.2	15.0
	9/20	70	226	2.7	222	.036	.022	.018	37.5	13.0
12	8/27	71	250	0.5	400	.023	.019	.015	38.7	23.8
	8/28	71	250	0.5	412	-	-	.015	39.8	20.9
	8/29	71	250	0.5	504	.023	.017	.013	40.2	20.4
	8/30	71	250	0.5	328	.023	.018	.014	42.1	18.8

Table 2 (contd)

Daily Leach Conditions and Results

La Sal Ore Autoclave Studies

Test No.	Date 1956	Pressure psig	Temperature °F	Air scfm/ Section	Steam lb/ton	Residue, % U_3O_8 After			Leach Disch, g/l	
						2.4 hr	4.8 hr	7.2 hr	Na_2CO_3	$NaHCO_3$
13	8/31	72	250	3.0	414	.021	.016	.013	40.8	18.7
	9/1	69	250	3.0	386	.022	.017	.015	39.6	19.6
	9/2	70	250	3.0	394	.021	.018	.014	-	-
14	9/7	71	250	5.7	305	.025	.017	.014	41.5	17.1
	9/8	71	250	5.7	332	.026	.018	.014	36.7	17.4
	9/9	71	250	5.7	361	.015	.015	.014	36.3	15.2
15	9/13	70	271	2.7	415	.020	.015	.014	35.4	17.0
	9/14	70	270	2.7	425	.021	.015	.014	33.7	16.4
	9/15	70	271	2.7	387	.023	.016	.013	34.9	16.9
	9/16	70	271	2.7	403	.023	.016	.013	36.7	15.8

Table 3

Daily Grinding and Thickening Data

La Sal Ore Pachuca Studies

<u>Test No.</u>	<u>Date 1957</u>	<u>Dry Tons</u>	<u>Belt Feed % U₃O₈</u>	<u>Grind, %</u>		<u>Grinding Solution, g/l</u>		<u>Pachuca Feed % Solids</u>
				<u>+ 65 mesh</u>	<u>-200 mesh</u>	<u>Na₂CO₃</u>	<u>NaHCO₃</u>	
1	11/9	16.7	.435	2.3	67.8	51.9	12.7	50.0
	11/10	17.6	.442	1.5	70.0	-	-	49.3
	11/11	17.4	.430	2.2	67.2	51.5	16.3	49.3
2	11/4	17.4	.375	2.8	65.1	53.7	8.2	50.0
	11/5	17.5	.434	2.1	68.3	53.5	8.0	49.3
	11/6	17.4	.495	2.5	67.0	52.9	11.1	50.0
	11/7	17.4	.432	1.9	68.1	50.4	10.3	50.0
3	11/21	17.3	.403	2.8	69.2	52.5	5.9	50.0
	11/22	17.6	.328	1.8	70.5	53.2	6.0	50.0
	11/23	17.2	.381	2.3	70.0	54.4	5.7	50.0
	11/24	17.4	.400	2.8	69.5	53.5	5.5	50.0
	11/25	18.0	.385	2.0	70.8	50.4	7.6	50.0
4	11/17	17.2	.398	2.2	68.4	51.5	7.8	50.0
	11/18	17.4	.345	1.7	68.0	53.1	5.7	49.3
	11/19	17.3	.288	2.8	68.0	51.4	6.8	50.8
5	11/13	17.5	.441	2.3	67.8	51.4	14.8	50.8
	11/14	17.4	.398	2.2	66.1	53.2	8.0	50.0
	11/15	17.5	.333	3.0	66.6	52.1	8.7	50.8

Table 4
Daily Leach Conditions and Results

La Sal Ore Pachuca Studies

Test No.	Date 1957	Top Pressure psig	Temperature, °F			Air		Steam lb/ton	Residue, % U ₃ O ₈ After			Leach Disch, g/l	
			No. 1	No. 2	No. 3	scfm/ Pachuca ^{1/}	Total scfm		16 hr	32 hr	48 hr	Na ₂ CO ₃	NaHCO ₃
1	11/9	0	176	184	187	10	42	872	.074	.032	.015	49.3	13.4
	11/10	0	180	182	181	10	42	875	.080	.026	.015	48.2	14.4
	11/11	0	182	182	180	10	42	865	.130	.049	.023	48.2	13.9
2	11/4	0	174	183	182	20	72	-	.047	.018	.010	49.4	13.1
	11/5	0	181	183	183	20	72	882	.042	.016	.009	50.8	12.2
	11/6	0	178	179	183	20	72	753	.046	.015	.008	51.6	12.4
	11/7	0	180	182	180	20	72	758	.061	.021	.010	47.8	12.9
3	11/21	20	202	216	-	5	18	795	.069	.019	-	46.4	12.5
	11/22	20	207	221	-	5	18	703	.067	.022	-	51.6	11.5
	11/23	20	213	221	-	5	18	663	.064	.025	-	49.1	9.2
	11/24	20	199	215	-	5	18	1100(?)	.073	.025	-	47.7	10.1
	11/25	20	194	213	-	5	18	-	.063	.023	-	48.7	13.9
4	11/17	20	220	221	-	10	28	822	.044	.010	-	44.5	14.3
	11/18	20	222	223	-	10	28	831	.044	.010	-	45.9	11.3
	11/19	20	220	223	-	10	28	825	.045	.012	-	44.5	10.5
5	11/3	20	223	223	-	20	48	975	.029	.009	-	44.6	13.7
	11/14	20	220	222	-	20	48	911	.027	.009	-	45.3	14.3
	11/15	20	220	222	-	20	48	928	.029	.009	-	44.0	15.9

^{1/} Does not include 4 scfm airlift air/Pachuca

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