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Field Geochemical Studies of Groundwaters in Nash Draw, Southeastern New Mexico

Steven J. Lambert, Karen L. Robinson

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550
for the United States Department of Energy
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Field Geochemical Studies of Groundwaters in Nash Draw, Southeastern New Mexico

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Abstract

Physiochemical properties of effluents from 15 pump tests in the water-producing horizons intercepted by six holes in Nash Draw east of the WIPP site in southeastern New Mexico were field-measured at intervals of a few hours. These properties were Eh, pH, specific conductance, specific gravity, bicarbonate/carbonate, chloride, divalent cations, calcium, hydrogen sulfide, and total iron. Observations were made over periods of 17 to 184 hr, during which the measurements reached varying degrees of steady state, but not necessarily equilibrium. The most useful indicators of steady state were divalent cations, chloride, and in some cases Eh. Typically, bicarbonate changed significantly during a pump test, seldom reaching steady state. This observation and the drilling history have shown that, given the nongeological sources of carbon, radiocarbon results will be difficult to interpret. Heavy-metal contamination from iron pipe in a well was not purged simply by displacing a well-bore volume, but is some complex function of "armor-coating" of iron by corrosion. This process of armor-coating is, in turn, influenced by pump rate, instantaneous solid-to-liquid ratio, dissolved gases, and aqueous thermodynamic properties. Thus, further determinations of trace constituents associated with heavy metals must be interpreted with care in view of the iron content. Reliable sampling of pumped wells requires a long observation time; detailed documentation of procedures, apparatus, and hole history; and a commitment of human and financial resources that may be much greater than those required for conventional laboratory analyses.

Acknowledgments

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Field Geochemical Studies of Groundwaters in Nash Draw, Southeastern New Mexico

1. Introduction and Scope

1.1 The Purpose of Waste Isolation Pilot Plant (WIPP) Studies

Geological studies in support of the Waste Isolation Pilot Plant (WIPP) have yielded a large body of data (Weart, 1983). Some of these studies examined issues relevant to decision points in developing the WIPP. Incompletely resolved issues include evaporite dissolution, definitions of the regional groundwater flow system, deformation of strata at or near the WIPP site, mineralogical stability, and paleoclimate. These studies have been reported in various levels of detail. Basic data reports such as this work contain considerable analytical and procedural detail that is fundamental to later interpretations. Often these basic data reports are the only documented sources of such detail. More general topical reports to follow will stress interpretations and conclusions.

1.2 The Purpose of the Nash Draw Studies

The ultimate objective of studies in Nash Draw, the "dogbone"-shaped depression west of the WIPP site (Figure 1-1), is to clarify the processes, geographic constraints, and relative chronology of shallow-seated dissolution of bedded evaporites near the WIPP site.

Analytical data from groundwater samples are meaningful only when considered in the context of the host rock. Therefore, water data alone are insupportable as the basis for geological interpretations without equal emphasis on core data from the same holes (core studies in Nash Draw are a separate activity). Geochemical analyses will be used to evaluate the nature and degree of interaction between rock and water in an area influenced by near-surface dissolution of evaporites.

1.3 Hydrological and Geochemical Studies

In 1978, six boreholes (WIPP 25 through 30) were drilled in or near Nash Draw (Figure 1-1). These boreholes were drilled into the Upper Salado Formation below the dissolution zone near the Rustler/Salado contact, and continuous core was taken from each. Each well was completed to total depth with cemented 5½-in. casing and was filled with fresh water to await further testing. Details may be found in SAND79-0279, SAND79-0280, SAND79-0281, SAND79-0282, SAND79-0283, SAND79-0284, and Seward (1982).

In 1980, Sandia National Laboratories (SNL) and the United States Geological Survey (USGS) undertook a major program of hydrological and geochemical studies in Nash Draw. In each of the six wells, three water-producing zones were to be tested: (1) the so-called "brine aquifer" of the dissolution zone near the Rustler/Salado interface; (2) the Culebra Dolomite member of the Rustler Formation; and (3) the Magenta Dolomite member of the Rustler Formation. Hydrological tests included bail, shut-in, slug-injection, and pump tests. General results of the hydrological tests are reported by Gonzalez (1983).

The wells and "aquifers" were tested (generally two wells at a time, as equipment allowed) in the following order: first, the "brine aquifer" in all the wells; second, the Culebra Dolomite in all the wells; and last, the Magenta Dolomite in all the wells except WIPP 29 where the Magenta no longer exists. Water-producing zones were perforated by using ½-in. bullet perforations at 4 shots/ft (Seward, 1982). In each well the "brine aquifer" was perforated and tested first, after which a retrievable bridge plug was set below the Culebra. The Culebra was then perforated and tested. Finally, a bridge plug was set below the Magenta, and the Magenta was perforated and tested. Pump tests were carried out with Jensen pump jacks or submersible pumps, depending on the production capacity of the well.

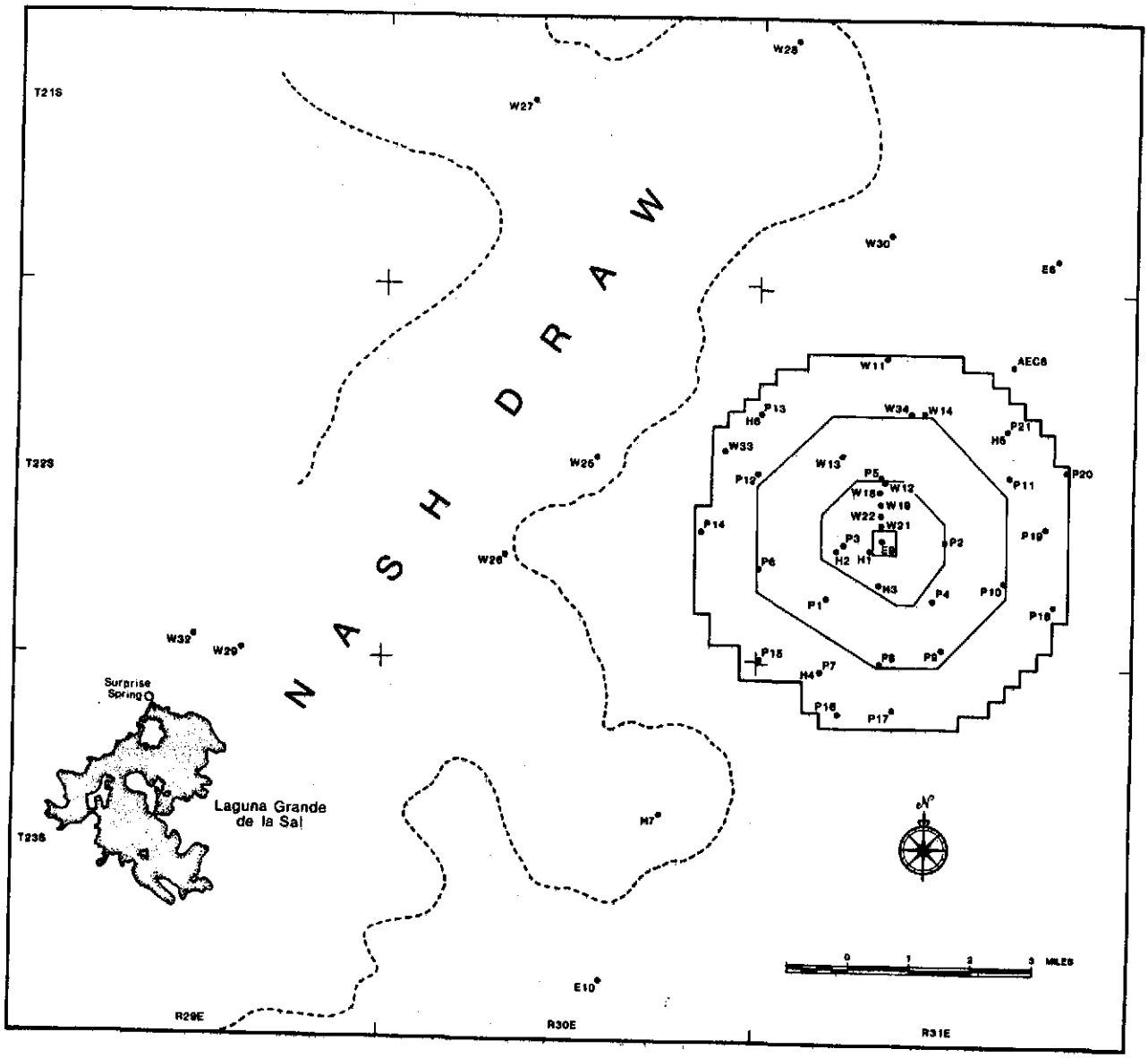


Figure 1-1 Map of Nash Draw and WIPP Nos. 25, 26, 27, 28, 29, and 30 in Relation to the WIPP Site (the octagon pattern) and to Boreholes Resulting From Other WIPP Investigations

1.4 Scope of Report

This report describes the activities and observations pertaining to the collection of *field* (wellhead and field-laboratory) data on samples recovered during extended pump tests of the "brine aquifer" (hereafter called the Rustler/Salado contact) and the Culebra and Magenta Dolomites in WIPP holes 25 through 30. Methods of preserving samples for further analyses are also described. Results of subsequent laboratory analyses are not reported here, and the analytical data reported here should be regarded as preliminary unless otherwise designated.

Previous experience in wellhead sampling of waters showed that an arbitrarily chosen water sample will, in general, not be representative of the native groundwater. The sample will instead be heavily contaminated with fluids used in drilling, cementing, slug-testing, etc (Barr et al, 1978). Consequently, scientifically defensible criteria must be adopted for selection of a wellhead sample as *bona fide* groundwater; accounts of how these criteria were applied form the substantial portion of this report. Water samples were periodically collected and analyzed for certain key constituents that were convenient to measure in the field during an extended (~24 hr or longer) pump test in which the withdrawal rate was kept as constant as practicable. This practice allows purging the well of additives and provides a means of judging a water sample as truly representative, after the analytical results have become constant within limits of precision. The results of this study or the recommendations herein should not be used to characterize pumped wells in general, but show that special concerns may arise in other groundwater systems as well.

NOTE: The use of brand names in this report is for identification only, and does not imply endorsement of specific products by Sandia National Laboratories.

2. Procedures

2.1 Wellhead Measurements (temperature, specific gravity, pH, Eh, specific conductance)

2.1.1 Sample Collection

Water samples for wellhead measurements were collected in 10-L polyethylene buckets that were rinsed with some of the water to be sampled immediately before the samples were taken. Sample volumes ranged from 2 to 8 L. The well name, "aquifer," date, and time (or time interval, when flow rates were low) were recorded in the field notebook; times were given to the nearest 5 min. Wellhead measurements were made within 5 to 10 min of sample collection.

2.1.2 Precision of Measurements

Precisions for wellhead measurements are reported as the 95% confidence limits for one replicate and were calculated according to methods given by Natrella (1963).

2.1.3 Temperature

Temperature was measured with ASTM No. 63C thermometers (−8° to +32°C, 0.1° divisions, serial Nos. 66525 and 39825, certified by SNLA Measurements Standards Laboratory; used before 25 July 1980) and ASTM No. 2C thermometers (−5° to +300°C, 1° divisions, serial Nos. A05600 and A05358, not certified at time of use; used after 25 July 1980).

The temperature of a sample was measured by placing the thermometer in the bucket immediately after the sample was collected, reading the temperature, and recording it to the nearest 0.5°C. When flow rates were low, the temperature was measured while the sample was being collected and as soon as there was enough water in the bucket to make a measurement. Although the ASTM No. 2C thermometers have not been certified, our experience with calibrated thermometers of this type indicates that they should be accurate to ±1°C. Our precision was ±1.0°C (95% confidence limits for one replicate).

2.1.4 Specific Gravity

Specific gravity was measured with specific gravity hydrometers (VWR catalog No. 34610-002, range 1.000 to 1.220, subdivisions 0.002, standard temperature 60°F; serial Nos. 60-1161 and 60-916; not certified at time of use).

Some of the sample was transferred into a polyethylene graduated cylinder of appropriate size (e.g., 1 L or 100 mL). The hydrometer was immersed in the sample and allowed to float freely. Because the polyethylene cylinders were not transparent, the position of the water line against the hydrometer was estimated by viewing from slightly above the water surface. The specific gravity was usually recorded to the nearest one-hundredth of a unit. The uncertainty in reading the hydrometer by the method described above is estimated as $\leq \pm 0.01$. Temperature corrections were not made on these data. Although the

hydrometers have not been certified, our experience with calibrated hydrometers of this type indicates that they should be accurate to ±0.02. Our precision was ±0.02 (95% confidence limits for one replicate).

Specific gravity was measured only on samples collected after 25 July 1980.

2.1.5 pH

Sample pH was measured with either an Orion Model 407A/F analog specific ion meter or an Orion Model 399A/F analog pH meter. Various pH-combination electrodes were used (Orion No. 91-02-00, research grade; Orion No. 91-04-00, laboratory grade; Orion No. 91-35-99, gel-filled).

The meters and electrodes were used according to the manufacturer's instructions. A pH-measuring system (meter plus electrode) was calibrated with buffer solutions of pH 7 and either pH 4 or pH 10 (chosen to bracket the sample pH) immediately before a measurement was made. Buffer solutions included both commercially prepared solutions and solutions made from Hydrion buffer capsules.

After the measuring system was calibrated, pH was measured by placing the electrode in the bucket containing the sample, waiting for the reading to stabilize, and recording the pH value (usually to the nearest tenth of a pH unit). Under field conditions, the measuring system is accurate to better than one-tenth of a pH unit, according to the manufacturer. Our precision was ±0.08 (95% confidence limits, one replicate).

2.1.6 Eh

Eh was measured with either the Orion Model 407A/F or the Orion Model 399A/F meter and combination platinum-reference electrodes (Orion No. 96-78-00). The meters and electrodes were used according to the manufacturer's instructions.

The Eh-measuring system, consisting of a meter plus an electrode, cannot be calibrated, but it can be checked with solutions of known redox potential ("Zobell" solutions). Solution A is 0.1M potassium ferrocyanide and 0.05M potassium ferricyanide. Solution B is 0.01M potassium ferrocyanide, 0.05M potassium ferricyanide, and 0.36M potassium fluoride (see Table 2-1 for details). The reagents for solutions A and B were preweighed into polyethylene vials in the Albuquerque laboratory and were dissolved and diluted to the appropriate volume in the field. Our experience showed that the prepared solutions were stable for at least 10 days; we discarded solutions more than 10 days old. Typically, fresh solutions were prepared at the beginning of the work week. The measuring system was checked daily with these solutions.

The sample Eh was measured by placing the electrode in the bucket containing the sample, allowing the reading to stabilize (typically 5 to 10 min), and recording the observed value to the nearest millivolt. Eh values are reported as oxidation potentials with respect to the standard hydrogen electrode (see Table 2-1 for correction factors). According to the manufacturer, the system is accurate to ±4 mV. We did not determine our precision.

2.1.7 Specific Conductance

Specific conductance was measured with a Labline portable Lectro Mho-Meter (Mark V, Model MC-1) and a beaker-type conductivity cell with a cell constant of 1.0.

The instrument was operated according to the manufacturer's instructions. For all Nash Draw samples, the instrument was used with the function switch in the third range of measurement (i.e., the reading was to be multiplied by 10^4). Results are reported in microsiemens per centimeter (μS/cm). Temperature corrections have not been applied to the data. Accuracy for this instrument, as determined in our Albuquerque laboratory, ranges from ±5% at midscale (0.5 to 2 μS/cm × scale factor) to ±12% at the scale ends (0.1 to 0.3 and 10 to 50 μS/cm × scale factor). Our precision was $\pm 0.3 \times 10^4$ μS/cm at midscale and $\pm 2 \times 10^4$ μS/cm at the high scale end (95% confidence limits, one replicate).

Specific conductance was measured only on samples collected after 25 July 1980.

2.2 Solute Determinations

2.2.1 Sample Collection

In addition to the polyethylene bucket (paragraph 2.1.1), 1-L narrow-mouthed polyethylene bottles (with polypropylene screw closures) were used as sample containers. Because most of the samples taken for solute determinations were not saved for more than a few days, these bottles were washed and reused. Washing consisted of tapwater rinsing (5 to 6 times), then demineralized-water rinsing (3 to 4 times), followed by air-drying. If a visible residue remained in a bottle after rinsing, the bottle was soaked in dilute acid (hydrochloric and/or nitric, as needed) for a few minutes and the rinsing was repeated. New bottles were used without pretreatment. All bottles (new and used) were rinsed with some of the sample water before filling.

Table 2-1 Eh Measurements

A. Recipes for "Zobell" solutions

	Solution A	Solution B
$K_4Fe(CN)_6 \cdot 3H_2O$	4.22 g	0.42 g
$K_3Fe(CN)_6$	1.65 g	1.65 g
$KF \cdot 2H_2O$	—	3.39 g
Final volume*	100 mL	100 mL
Expected potential	192 mV	66 mV greater than potential of Solution A

*Regents dissolved and diluted to volume with demineralized water

B. Potential relative to standard hydrogen electrode (SHE)

$$Eh(reported) = E_{SHE} = E_{MEAS} + C$$

where

E_{SHE} = redox potential of the sample relative to the standard hydrogen electrode

E_{MEAS} = potential developed by the platinum redox electrode and read off the meter

C = potential developed by the reference electrode relative to the SHE; values taken from table; values at intermediate temperatures were determined by interpolation

Temp (°C)	C* (mV)
0	257
10	251
20	244
25	241
30	238

*These C values are for a combination redox electrode (Orion No. 96-78-00) filled with Orion filling solution No. 90-00-01.

Any sample in which carbonate/bicarbonate was to be determined was collected directly from the pipe into the sample bottle to minimize contact with air. The bottle was capped as soon as it was full and was not opened until the sample was withdrawn for the carbonate/bicarbonate determination. When collection directly into a bottle was not possible because of a low flow rate (i.e., ≤1 L/5 min), the sample was collected in the bucket and *immediately* transferred to a bottle. Bottles were labeled with the well name, "aquifer," date, time (to the nearest 5 min), and "HCO_3^-."

Samples for other solutes were sometimes collected in the same manner: i.e., directly into a bottle labeled with well name, "aquifer", date, and time. However, the other solutes were more often measured in the sample left over from the wellhead measurements (paragraph 2.1). When the field-laboratory trailer was located at the well being sampled and the analyses were done within 1 to 2 hr of sample collection, the sample was simply taken as needed from the bucket. When the trailer was located away from the well being sampled or when the analyses were not done right away, a liter of sample was transferred to a labeled bottle for later analysis. (Section 3 has a discussion of time constraints.)

2.2.2 Field-Laboratory Techniques and Equipment

Filtration: At the time that the solutes (other than carbonate/bicarbonate) were being measured, ~100 to 150 mL of raw sample were filtered through filter paper (Whatman 42 before 29 August 1980; Whatman 41 after 29 August 1980) into a glass beaker from which aliquots were taken as needed.

Dilution: The volumetric glassware (pipets and volumetric flasks) used in making sample dilutions was Class A unless otherwise noted. Dilutions made for Hach determinations of iron and sulfate were sometimes done with the plasticware supplied with the Hach kit (e.g., graduated cylinders and pipets).

Titration (burets): Electrometric burets (Kimax, 50 mL, 0.1-mL subdivisions, TD 20°C) were used for the carbonate/bicarbonate and chloride titrations. We tested these burets for accuracy; they meet the tolerances specified for Class A burets. A Class A buret (Pyrex, 50 mL, 0.1-mL subdivisions, TD 20°C) was used for the calcium and total-divalent-cation titrations.

Precisions of Measurements: Precisions for carbonate and bicarbonate measurements are reported as the 95% confidence limits for one replicate and were calculated according to methods given by Natrella (1963). Because we have not reported absolute concentrations for most other solutes (and also because these measurements will be repeated in the laboratory), precisions for the other solutes are given as relative standard deviations. The relative standard deviation is the estimated standard deviation, S, divided by the concentration. S was calculated by the method of Natrella (1963).

2.2.3 Carbonate/Bicarbonate ($CO_3^=/HCO_3^-$)

Carbonate and bicarbonate were determined by an electrometric titration method based on that of Brown et al (1970). An aliquot of sample was titrated with standard H_2SO_4 (0.02N) to end points of pH 8.3 (carbonate) and pH 4.5 (bicarbonate). Carbonate/bicarbonate analyses were done as soon as possible after the sample was collected.

pH Measurement: pH was measured with either the Orion Model 407A/F or the Orion Model 399A/F meter and a research- or laboratory-grade combination electrode (par 2.1.5 pH). Immediately before the titration was begun, the measuring system, consisting of a meter plus an electrode, was calibrated with buffer solutions of pH 4 and pH 7.

Standard Acid: Standard acid solutions were prepared by diluting reagent grade sulfuric acid (98%) by a factor of 2000 with demineralized water. Solutions were stored in clean 1-L polyethylene bottles. The acid solution was standardized by titrating 25 mL of "tris" standard solution (tris(hydroxymethyl) aminomethane, NBS Standard Reference Material 723, 0.016N in demineralized water) to an endpoint of pH 4.7. This endpoint was determined graphically and agrees with that determined by Fossum et al (1951).

Sample Titration: Just before the determination, an aliquot of raw, settled sample (25 or 50 mL) was pipetted into a 100-mL glass beaker and the pH was measured. If it was <8.3, no carbonate was present and the carbonate titration was skipped. If it was >8.3, a few drops of phenolphthalein indicator were added to give visual indication of the carbonate endpoint, and the solution was titrated to an endpoint of pH 8.3. The sample and titrant were mixed by swirling the beaker by hand as the titrant was added. A few drops of methyl red indicator were then added to give visual indication of the bicarbonate endpoint (procedure started here for samples with initial pH <8.3), and the solution was titrated to an endpoint of pH 4.5.

Precision and Accuracy: We have not evaluated this method for accuracy. Our precisions (95% confidence limits, one replicate) were ±6 mg/L for bicarbonate and ±6 mg/L for carbonate.

2.2.4 Calcium (Ca^{++})

Calcium was determined by the complexometric titration method described in Brown et al (1970). An aliquot of filtered sample was titrated with standard EDTA solution (0.025N). Calcium was not determined in all samples because endpoint recognition was sometimes difficult or impossible.

Standard EDTA: Standard EDTA solutions were prepared by dissolving 4.65 g of reagent-grade disodium dihydrogen ethylenediamine tetraacetate dihydrate ($Na_2EDTA \cdot 2H_2O$) in 1 L of demineralized water. Solutions were stored in 1-L polyethylene bottles. The EDTA solution was standardized by titrating 25 mL of calcium carbonate standard solution (NBS Standard Reference Material 915, 0.02N in dilute HCl) according to the procedure described below for samples (exception: NaCN was not added).

Sample Titration: An aliquot of filtered, diluted sample was pipetted into a 250-mL glass beaker and the volume was adjusted to ~50 mL with demineralized water. The following were added: 1 mL $NH_2OH \cdot HCl$ solution (3% wt/vol), 1 mL NaOH solution (2M), 1 mL NaCN solution (2.5% wt/vol), ~0.2 g murexide indicator (0.5 wt % in sucrose), one Teflon-coated magnetic stir bar. With constant stirring, the solution was titrated with standard EDTA solution until the color changed from salmon (pink-orange) to orchid-purple. A blank correction was not made.

Precision and Accuracy: We have not evaluated this method for accuracy. Our precision (relative standard deviation) was better than ±2%.

2.2.5 Chloride (Cl^-)

Chloride was determined by the Mohr titration method of Brown et al (1970). An aliquot of filtered sample was titrated with standard $AgNO_3$ (0.14N).

Standard $AgNO_3$: Standard silver nitrate solutions were prepared by dissolving 24 g of reagent grade $AgNO_3$ in 1 L of demineralized water. Solutions were stored in 1-L acid-washed brown (opaque) polyethylene bottles. The silver nitrate solution was standardized by titrating 25 mL of sodium chloride standard solution (NBS Standard Reference Material 919,

0.034 N in demineralized water) according to the procedure described below for samples.

Sample Titration: An aliquot of filtered, diluted (if necessary) sample was pipetted into a 250-mL glass beaker, and the volume was adjusted to ~50 mL with demineralized water. Ten drops of K_2CrO_4 indicator solution and a Teflon-coated magnetic stir bar were added. With constant stirring, the solution was titrated with standard $AgNO_3$ solution until the red-orange Ag_2CrO_4 persisted for 10 to 15 s. A blank correction was not made.

Precision and Accuracy: We have not evaluated this method for accuracy. Our precision (relative standard deviation) was better than ±1.5%.

2.2.6 Divalent Cations (X^{++})

Divalent cations (predominantly alkaline earths) were determined by the complexometric titration method described in Brown et al (1970). An aliquot of sample was titrated with standard EDTA solution (0.025N). Divalent cations were not measured in samples taken on 16 July 1980 because of lack of ammonium hydroxide.

Standard EDTA: The standard EDTA solution was the same as that used in calcium determinations and was described in paragraph 2.2.4.

Sample Titration: An aliquot of filtered diluted sample was pipetted into a 250-mL glass beaker and the volume was adjusted to ~50 mL with demineralized water. The following were added: 1 mL $NH_2OH \cdot HCl$ solution (3% wt/vol), 1 mL concentrated NH_4OH, 2 mL NaCN solution (2.5% wt/vol), 2 mL Eriochrome Black T indicator solution (0.4% wt/vol in 85% ethanol), one Teflon-coated magnetic stir bar. (Because manganese was never present in concentrations great enough to interfere, the $K_4Fe(CN)_6 \cdot 3H_2O$ addition of Brown et al (1970) was not necessary.) With constant stirring, the solution was titrated with standard EDTA solution until the color changed from red-purple to clear blue. A blank correction was not made.

Precision and Accuracy: We have not evaluated this method for accuracy. Our precision (relative standard deviation) was better than ±2%.

2.2.7 Hydrogen Sulfide (H_2S)

Hydrogen sulfide was determined in some samples (where its presence was indicated by odor) with a Hach DR-EL/2 portable engineer's laboratory according to the method given in the Hach instruction manual. The method involves dissolving an Alka-Seltzer tablet in an aliquot of sample, reacting the evolved gases with lead-acetate test paper, and comparing the resulting color (darkness) of the test paper to a color chart in the Hach instruction manual. Hydrogen sulfide analyses were done as soon as the samples were collected. We have not evaluated this method for precision or accuracy; numbers reported should be viewed as rough approximations.

2.2.8 Total Iron (Fe)

Iron was determined colorimetrically with a Hach DR-EL/2 portable engineer's laboratory according to the method given in the Hach instruction manual. Aliquots of filtered, diluted (if necessary) sample were transferred to two 25-mL cuvettes. One "pillow" of FerroVer reagent (orthophenanthrolene) was added to one cuvette and the color (orange) allowed to develop for 2 to 5 min; the second cuvette was used as a blank. Absorbance was measured at 510 nm. We have not evaluated this method for accuracy. Our precision (relative standard deviation) was ±10%.

2.2.9 Sulfate ($SO_4^=$)

Sulfate was determined by turbidimetry with a Hach DR-EL/2 portable engineer's laboratory according to the method given in the Hach instruction manual. Aliquots of filtered diluted sample were transferred to two 25-mL cuvettes. One "pillow" of SulfaVer reagent (barium chloride) was added to one cuvette and the turbidity allowed to develop 5 to 10 min; the second cuvette was used as a blank. Absorbance was measured at 450 nm. We have not evaluated this method for accuracy. Our precision (relative standard deviation) was ±10% for samples with up to 5000 mg/L sulfate and ±20% for samples with 10 000 to 20 000 mg/L sulfate.

2.3 Geochemical Samples for Future Analysis

2.3.1 Sample Containers

The ubiquitous polyethylene bucket (see par 2.1.1) was used to collect most samples, although raw samples (i.e., those not to be filtered) were sometimes collected directly in the sample bottles.

The glass bottles used were flint glass, 4-oz (125-mL) capacity, with screw caps with polyethylene cone liners. The bottles and caps were rinsed with some of the sample water before filling.

Polyethylene bottles were new bottles of 1-L capacity with polypropylene screw caps. These bottles

were acid-washed (i.e., soaked several hours to days in 2N nitric acid), rinsed 5 to 6 times with tap water, rinsed 3 to 4 times with demineralized water, and air-dried. These bottles were not usually rinsed with sample water before filling. Acid-washed bottles were sometimes not available when samples were being collected. In such cases, the new bottles were used as-is but were rinsed with some of the sample water before being filled.

In addition to the 1-L bottles, we also used 125-mL polyethylene bottles with polypropylene screw caps. These were new bottles and were rinsed with some of the sample water before filling.

2.3.2 Sample Treatment

Samples were collected and preserved in three ways: "raw" (i.e., not filtered, not acidified, and not treated in any other way); filtered; and filtered and acidified.

Filtering was done under pressure by using a device constructed of plastic (lucite?) with a fritted-glass disk on which a filter paper or membrane was placed and to which copper drain tube with stainless steel fittings was attached. Pressure was supplied by a hand-operated bicycle pump. Samples collected before 17 August 1980 were filtered through Whatman 42 filter paper; samples collected after 17 August 1980 were filtered through Nuclepore 0.4-μm filter membranes.

Samples were acidified with concentrated reagent-grade nitric acid to a pH of <2 (as indicated by pH paper). About 1 mL of acid was added, the sample mixed, and the pH checked. If the pH was ≥ 2, a second milliliter of acid was added, the solution mixed, and the pH rechecked.

2.3.3 Sample Labels

Samples were labeled with the following information: well name, "aquifer," date, time (to the nearest 5 min), details of sample treatment (filtration, acidification) or lack thereof ("raw"), the initials of the collector(s), and a possible use for the sample (e.g., major solutes and stable isotopes). These possible uses are suggestions only; they were written on the labels for the convenience of the WIPP geochemists but are not binding.

2.3.4 Possible Uses of Samples

Raw samples in glass bottles are suitable for analyzing stable isotopes (e.g., D/H, $^{18}O/^{16}O$) and tritium.

Raw samples in polyethylene bottles may be used for analyzing some major solutes.

Filtered samples in polyethylene bottles are suitable for analyzing major solutes (e.g., K^+, Na^+, X^{++}, Cl^-, SO^-_4, etc) and natural radioactivities (excluding tritium).

Filtered, acidified samples in polyethylene bottles are suitable for analyzing various major, minor, and/or trace solutes (e.g., Na^+, K^+, Li^+, Mg^{++}, Ca^{++}, etc) and for attempts at dating by the uranium isotope disequilibrium method.

Table 2-2 is an inventory of the samples taken for future analyses.

2.4 Samples for Radiocarbon Analysis

Samples were collected for attempted carbon-14 dating. Hydro Geo Chem, Inc. (Tucson, AZ) and the Radiocarbon Dating Laboratory at the University of Arizona are doing the radiocarbon studies.

2.4.1 Sample Collection

The Radiocarbon Dating Laboratory researchers requested that each sample contain at least 2 g of inorganic carbon. The minimum volume of sample needed to meet this requirement was calculated from the measured bicarbonate and carbonate ion contents of the sample (par 2.2.3 and Section 6). In cases where the carbon was to be precipitated as barium carbonate (par 2.4.2), 90% recovery of the inorganic carbonate was assumed.

Samples from which the carbon was to be precipitated (par 2.4.2) were collected in polyethylene carboys of 50-L capacity. Before their first use, the carboys were rinsed with tapwater and demineralized water. After each use, they were rinsed several times with tapwater, soaked overnight in dilute hydrochloric acid (0.2 to 0.5 N in tapwater), rinsed again several times with tapwater, and finally rinsed 3 to 4 times with demineralized water. (Neither scrubbing with a brush and Alconox solution nor soaking in dilute acid removed a white residue on parts of the container walls. This was assumed to be barium sulfate and was ignored thereafter.) The carboys were always rinsed with some of the sample water before the samples were collected. They were filled nearly full, with just enough air space left to allow for adding and mixing reagents.

Table 2-2 Samples for Future Geochemical Analyses

Sample Name	Collection Date (1980)	Samples: Number of Bottles/Time Collected				
		Raw 125 mL Glass	Raw 125 mL Poly.	Raw 1 L Poly.	Filtered 1 L Poly.	Filtered Acidified 1 L Poly.
WIPP 25 Rustler/Salado	17 July	3/22:20	1/22:20	1/22:20	1/22:20	1/22:20
WIPP 26 Rustler/Salado	23 July	3/19:00	1/19:00	1/19:00	1/19:15	1/19:15
WIPP 27 Rustler/Salado	7 Aug	1/08:20	—	—	—	—
WIPP 28 Rustler/Salado	31 July	3/08:55	1/08:55	1/08:55	2/08:55	*3/08:55
WIPP 29 Rustler/Salado	24 July	1/10:15 2/12:35	1/10:15	1/10:15	2/10:25	3/10:25
WIPP 30 Rustler/Salado	17 July	2/16:30	1/16:20	1/16:00	1/16:00	1/16:40
WIPP 25 Culebra	20 Aug	3/16:25	1/16:25	1/16:30	2/16:30	3/16:30
WIPP 26 Culebra	24 Aug	3/17:15	1/17:15	1/17:15	2/17:25	3/17:25
WIPP 27 Culebra	3, 5 Sep	2/17:20 (3 Sep) 2/13:50 (5 Sep)	1/13:50 (5 Sep)	1/17:20 (3 Sep) 1/13:50 (5 Sep)	2/17:20 (3 Sep)	3/17:20 (3 Sep)
WIPP 28 Culebra	11 Sep	3/17:30	1/17:30	1/17:25	2/17:40	3/17:40
WIPP 29 Culebra	28 Aug	3/10:15	1/10:15	1/10:15	2/10:20	3/10:20
WIPP 30 Culebra	6 Sep	3/05:45	1/05:45	1/05:40	2/05:45-06:00	3/05:45-06:00
WIPP 25 Magenta	17 Sep	3/16:15	1/16:15	1/16:15	2/16:15	3/16:15
WIPP 27 Magenta	25 Sep	3/13:15	1/13:15	1/13:15	2/13:20	3/13:20
WIPP 30 Magenta	9, 10 Dec	1/10:55-11:00 (9 Dec) 2/09:50-10:15 (10 Dec)	—	—	—	—

*Third bottle contains only 300 mL of sample

Samples to be shipped untreated (par 2.4.3) were collected in red plastic jerrycans (the type suitable for storing gasoline) of 19-L (5-gal) or 10-L (2-1/2-gal) capacity. (Exception: The first such samples collected were from the WIPP 26 Rustler/Salado contact. These were collected in *green* plastic jerrycans of the type suitable for storing aqueous solutions.) All jerrycans were rinsed with some of the sample water before the samples were collected. They were filled to the top and the caps were sealed in place with paraffin.

Usually the sample containers (both carboys and jerrycans) were filled directly from the pipe at the wellhead, without filtering the samples. When flow rates were low, however, the samples were filtered through a coarse cloth (e.g., a piece of "T-shirt" fabric) to keep dust and insects out of the containers.

2.4.2 Sample Treatment

The preferred method of treating samples involves precipitating the inorganic carbon as barium carbonate immediately after sample collection. The precipitate is then collected and sent to the laboratory for dissolution and analysis.

The method, as dictated by personnel of the Radiocarbon Dating Laboratory (A. B. Muller, personal communication, 1980), is:

- Collect the sample in a large carboy(s)
- Adjust the sample pH to >11 with sodium hydroxide pellets (adding ~1 "capful" at a time from a 1-lb bottle and monitoring the pH with pH paper)
- Add barium chloride
- Add a flocculant to aid in settling the precipitate
- Mix the solution well
- Allow to settle overnight
- Decant the supernate, saving 1 to 2 L and discarding the rest
- Transfer the precipitate to a smaller, clean polyethylene bottle or bottles (1-L capacity) using the retained supernate to rinse the carboy
- Repeat the settling and decanting steps, as necessary, to remove as much supernate as possible and to reduce the final sample volume.

Normally, the amount of barium chloride used in precipitation would be about four times the calculated amount needed to bring down all the inorganic carbon. This works fine for low-sulfate waters but not for brines with relatively low carbonate/bicarbonate contents and high (3000 to 10 000 mg/L) sulfate contents. Instead, we calculated the amount of barium chloride needed to bring down all the sulfate and used approximately twice that amount.

2.4.3 Alternate Method

The chemical procedure described above did not work for samples with very high amounts of total dissolved solids, especially those with high divalent cation and sulfate ion contents. In such cases, the samples were collected in jerrycans, whose caps were then sealed in place with paraffin. These bulk samples were shipped directly to the radiocarbon laboratory for analysis.

2.4.4 Samples

Samples taken and details of their collection are given in Table 2-3. At some sites two samples were taken—one chemically treated and one bulk—for a comparison of results.

3. General Observations

Each pumping episode is treated in detail below, but some general observations that prevailed in many of the pumping episodes may be useful. Many of these apply to the *field* measurements and, in some cases, to intermediate or final sample collection. A discussion follows of the limitations imposed by field conditions upon any analyses undertaken at future times.

3.1 Time and Distance Constraints

Conscientious attempts were made during daylight (and even sometimes evening) hours to sample a wellhead effluent stream every 3 to 4 hr. Given the transient nature of some of the thermodynamic properties of solutions (e.g., pH, Eh), we tried to measure certain properties as quickly as possible. During much of the field season, two wells were pumped simultaneously, commonly 7 to 22 mi (one-way) apart on roads of various quality ranging from graded, to unimproved, to flooded, etc. Whereas certain measurements (e.g., temperature, pH, Eh, specific conductance, and specific gravity) were made at the wellhead, other measurements (particularly bicarbonate) were delayed 15 to 90 min, with the samples agitated on bumpy roads for certain intervals. It is unlikely that subsaturation concentrations of inorganic solutes changed during that period. Bicarbonate/carbonate was measured usually within 30 min of wellhead sampling if the field-laboratory trailer was located at that wellhead. We cannot evaluate the gain or loss of carbonaceous ions as carbon dioxide, which may have been caused by agitation in transit.

Table 2-3 Samples for Radiocarbon Analysis

A. Chemically Treated Samples

Well	Aquifer	Date (1980)	Time	Volume Collected (L)	Reagents Added to Each 50 L* NaOH (capfuls)	$BaCl_2 \cdot 2H_2O$ (kg)	Flocculant (mL)	Final Packaging (1-L bottles)
WIPP 25	Culebra	20 Aug	17:05 – 17:10	50	~5	1.00	20 – 30	2
WIPP 25	Magenta	18 Sep	09:05	100	~5	0.90	20 – 30	4
WIPP 26	Culebra	24 Aug	20:25 – 20:35	100	~6	1.00	20 – 30	4
WIPP 27	Magenta	25 Sep	14:25	100	~15-16	1.00	20 – 30	7
WIPP 30	Culebra	5-6 Sep	21:35 – 04:05	200	~5	0.85	20 – 30	8

*NaOH: Baker reagent grade, low in carbonate
$BaCl_2 \cdot 2H_2O$: Baker reagent grade
Flocculant: Prestol K225FL

B. Bulk Samples

Well	Aquifer	Date (1980)	Time	Volume Collected (L)	Containers (jerrycans)
WIPP 25	Culebra	20 Aug	19:25 – 19:35	60	6 ea of 10 L
WIPP 25	Magenta	18 Sep	09:35	57	3 ea of 19 L
WIPP 26	Rustler/Salado	23 Jul	20:00 – 21:00	57	3 ea of 19 L
WIPP 26	Culebra	24 Aug	20:35 – 20:40	69	1 ea of 19 L and 5 ea of 10 L
WIPP 27	Culebra	5 Sep	13:00 – 13:10	76	4 ea of 19 L
WIPP 27	Magenta	25 Sep	15:00	57	3 ea of 19 L
WIPP 28	Rustler/Salado	31 Jul	17:50 – 18:00	60	6 ea of 10 L
WIPP 29	Rustler/Salado	24 Jul	11:00 – 11:20	57	3 ea of 19 L
WIPP 29	Culebra	28 Aug	10:40 – 10:45	57	3 ea of 19 L

Sufficient sample usually was collected within 5 min (par 2.1.1) for all interim analyses. However, for certain very low flow rates (<100 mL/min) sampling was done for up to 30 min. In one case, a sample for carbonaceous ions was collected first and separately from the sample for all other measurements; hence, different sampling intervals are reported for WIPP 30-Magenta.

If no entry is given for an item in the tables in Section 4, that measurement was not made at that time.

3.2 Interim Measurements and Samples

Field measurements (as described in Section 2) included: date and time of day, temperature, specific gravity, specific conductance, pH, Eh, bicarbonate/carbonate, chloride, divalent cations, calcium, hydrogen sulfide, sulfate, and iron. Measurements that would lose their significance if not made immediately after sample collection are reported here as absolute numbers (e.g., temperature, pH, Eh, and bicarbonate/carbonate). Other measurements (with the exceptions noted in Section 4) are reported as approximations or as ratios relative to the first measurement, not as absolute values. These other measurements, discussed in Section 5, were used *only* to help evaluate the approach to steady-state of solute contents. A complete series of interim samples was not maintained. Final samples are documented in Table 2. Interim samples that were not discarded are identified in the tables in Section 4.

3.3 Black Particulates

At the beginning of some pumping tests it was noted that some of the first water was discolored and/or odoriferous. In most cases, the water cleared up as pumping proceeded. However, sometimes water that began milky remained so and was associated (based on odor) with dissolved hydrogen sulfide. Such waters were also slightly effervescent. Much of the initial but transient discoloration and odor is tentatively attributable to contamination of the local reservoir by slug-injection tests immediately preceding the pump tests. The slug-injection tests introduced a water whose composition was not precisely known, and which could have contained a modest degree of microbial activity, for example. One reason for long pumping times was to purge the local reservoir of surface-induced and/or testing-induced perturbations. It cannot be demonstrated that these perturbations were overcome in all cases, but the method of periodic partial analysis in the field provides criteria for determining a steady state (even if transient) for the local reservoir.

Almost all waters associated with hydrogen-sulfide odor darkened upon standing for an hour in a sealed polyethylene bottle. Some formed black precipitates. Most other waters also darkened, but over several hours. Since the waters usually left the wellhead without the black precipitate, the precursor to the precipitate was most likely originally in solution or colloidal suspension. All final water samples that were treated with nitric acid cleared up.

Because of the association of black precipitates with hydrogen sulfide, it was decided to test waters semiquantitatively for total iron and H_2S. In most cases, the H_2S concentration exceeded the limits of the lead-acetate test-paper method (5 mg/L). In two cases (WIPP 25 Culebra and Magenta), H_2S appeared crudely inversely proportional to Fe. Most of the waters had <1 mg/L Fe during much of the pumping. Since there is a large amount of iron equipment in a well during a pump test, a moderately saline water should acquire oxidized iron from the equipment. Consequently, any analysis of iron as a trace element is not meaningful as a characteristic of native groundwater. Rather, the iron is thought to have dissolved owing to the small amount of acidity interacting with conduit pipe, for example. Either reduction by H_2S or oxidation by atmospheric O_2 could then give rise to the slow formation of a black ferriferous precipitate (perhaps pyrrhotite or magnetite). No evidence exists for the derivation of black particulate directly from the rock. Significantly, at pH values indicative of slight acidity (<7) the Fe content was $\sim$0.5 to 1 mg/L, but the more alkaline waters ($\geq$ pH 8) contained Fe at typically <0.1 mg/L, indicating the tendency of slightly acid solutions to dissolve iron.

3.4 Potential Microbial Action

During many of the runs described in Section 4, certain waters were described as "effervescent," "colored," or "rank-smelling." Something described as "smelling rank" has an odor distinct from that of hydrogen sulfide. We use "rank" to describe a "rotten" smell that might result from peculiar mixtures of ketones, aldehydes, esters, and mercaptans, but *not* from H_2S. Some have described the "rank" smell as "essence of cow yard," whereas hydrogen sulfide is commonly described as smelling like rotten eggs.

Organic metabolites, such as those suggested above, have long been associated with microbial activity, which is not unknown in groundwaters. Indeed, H_2S is commonly attributed to the bacterium *Desulfovibrio desulfuricans*. The milky-white color of some H_2S-bearing waters might be ascribed to the atmospheric oxidation of H_2S, resulting in a finely divided (white) suspension of elemental sulfur.

Reddish-brown mats were accumulated on the surface of some waters collected in metal stock tanks or in earthen pits at some wellheads. These mats resembled the "iron slimes" prevalent in some commercial wells and could be colonies of oxidizing bacteria such as *Thiobacillus* or *Leptothrix*.

When some of the gaseous waters were titrated for bicarbonate/carbonate with H_2SO_4, gases were seen (CO_2?) and smelled (H_2S!) to exsolve as acid was added. Thus, the possibility that much bicarbonate/carbonate in the groundwaters arose from biological activity and not from atmospheric CO_2 at the time of recharge cannot be discounted. This may invalidate certain assumptions made in radiocarbon-dating of groundwaters. In addition, the time required to collect large samples for radiocarbon measurements could have allowed the introduction of significant amounts of very modern carbon. Even when field measurements of inorganic constituents had apparently stabilized, the bicarbonate commonly changed slowly, but perceptibly and monotonically downward. This in itself suggests that the origin of much of the CO_2 in these groundwaters was *not* atmospheric at the time of recharge to the aquifer.

Biological agents may have been native to the reservoir rock, or they may have been introduced during drilling, casing, cementing, or perforating. They may even have been transferred as downhole equipment was moved from one hole to another. For

example, J. W. Mercer (personal communication) observed that, after perforation of each hole, the fluid was forced out (except for WIPP 30) presumably by gas pressure in the "aquifer". No such "unloading" was noted during drilling. In any case, there is evidence of microbial action in the data described herein. Nutrients in large supply were introduced and not retrieved during the drilling, in order to control lost circulation in the advancing corehole. These are typically in the form of paper, cottonseed hulls, peanut shells, and various proprietary organic additives to drilling mud. Their biodegradation will most likely proceed for many years, thus providing a nongeological source of carbon that will be difficult to purge from the local groundwater system regardless of pumping time.

4. Specific Results and Observations

This section systematically reviews each pump test. Each review contains

- A description of appearances and smells of the effluents at various times
- General trends in the analyses (noted in Tables 4-1 through 4-15)
- Duration of each test
- Irregularities (measurements not made, equipment failures, etc) that may relate to any subsequent analyses or interpretations of these field data or of any subsequent laboratory data.

4.1 WIPP 25—Rustler/Salado Contact (Table 4-1)

The effluent at the beginning of pumping (16 July 1980, ~2000 hr) was clear, but at 2200 hr had turned "muddy, rusty, and odoriferous" according to observer L. Kracko. By 1000 hr on 17 July, the solution had clarified and a red-brown particulate had settled in the discharge pipe. This condition persisted throughout the test.

Field measurements stabilized after 24 hr of pumping. Final geochemical samples were collected after ~27 hr of pumping.

Specific conductance and specific gravity were not determined because of unavailability of equipment. Calcium was not determined because the titrimetric endpoint was indistinct. Divalent cations were not determined on 16 July because of a lack of reagents.

A radiocarbon sample was not collected here. This was one of the first wells tested (along with WIPP 30 – Rustler/Salado) and was very high in total dissolved solids. Chemical precipitation did not work; at that time, we were not prepared to collect bulk samples.

Table 4-1 WIPP 25—Rustler/Salado Contact*

Date (1980)	Time	Temp (°C)	pH	Eh (V)	HCO_3^- (mg/L)	Cl^- **	X^{++} **
#16 Jul	22:35	22.0	7.1	−0.237	127		
#17 Jul	10:00	23.0	7.55	−0.238	133	1	1
17 Jul	18:55	22.0	7.0	−0.027	135	1.01	1.09
17 Jul	22:05	22.0	7.4	−0.037	130***	0.99***	1.09***

*Pump test started on 16 July at 19:00 hr. Average flow was 1.1 gpm.

**Ratio relative to measurement 17 July, 10:00. Chloride and divalents were not measured in first sample because of a lack of time and reagents.

***Sample for solute analysis collected at 22:25.

#1 L raw sample saved.

Table 4-2 WIPP 25—Culebra Member*

Date (1980)	Time	Temp (°C)	Sp Grav	pH	Eh (V)	Sp Cond (μS/cm)	mg/L HCO_3^-	mg/L H_2S	mg/L Fe	$SO_4^{=}$**	Cl^- **	X^{++} **	Ca^{++} **
#19 Aug	14:45	22.0	1.01	6.8	−0.027	2.2×10^4	303	>5	0.3	1	1	1	1
19 Aug	18:55	22.0	1.01	6.8	+0.013	1.7×10^4	258	>5	0.5	1.0	0.92	1.01	1.00
20 Aug	10:45	23.0	1.01	6.8	+0.060	1.75×10^4	213	2 – 5	0.6	0.9	0.85	1.02	1.00
20 Aug	15:00	23.0	1.01	6.9	+0.064	1.95×10^4	206	~2	0.6	0.9	0.84	1.02	1.00

*Pump test started on 19 Aug at 12:35 hr. Average flow was 33 gpm.
**Ratio relative to first measurement.
#1 L raw sample saved.

Table 4-3 WIPP 25—Magenta Member*

Date (1980)	Time	Temp (°C)	Sp Grav	pH	Eh (V)	Sp Cond (μS/cm)	mg/L HCO_3^-	mg/L H_2S***	mg/L Fe	$SO_4^{=}$**	Cl^- **	X^{++} **	Ca^{++} **
16 Sep	17:00	22.5	1.007	6.9	−0.037	1.7×10^4	231	>5	0.2	1	1	1	1
17 Sep	08:00	21.5	1.004	6.9	+0.025	1.46×10^4	190	2 – 5	0.6	0.8	0.97	1.02	1.03
17 Sep	11:05	22.0	1.004	6.9	+0.029	1.46×10^4	188	2 – 5	0.6	0.8	0.99	1.00	1.03
17 Sep	14:05	22.0	1.004	6.9	+0.073	1.55×10^4	187	2 – 5	0.6	0.8	0.98	1.04	1.05
18 Sep	08:05	21.5		7.0			178						

*Pump test started on 16 Sept at 15:00 hr. Average flow was 34 gpm.
**Ratio relative to first measurement.
***Measurements made on samples taken 20 to 50 min after times indicated.

Table 4-4 WIPP 26—Rustler/Salado Contact*

Date (1980)	Time	Temp (°C)	pH	Eh (V)	HCO_3^- (mg/L)	Cl^- **	X^{++} **
22 Jul	11:45	24.5	7.85	−0.011	272	1	1
22 Jul	15:00	25.0	7.70	−0.009	283	0.98	1.07
22 Jul	19:40	23.0	7.70	−0.008	283	0.96	1.05
23 Jul	14:10	23.5	7.49	−0.017	278	0.97	1.11
23 Jul	17:30	25.0	7.70	+0.021	273	0.98	1.10

*Pump initially on at 13:00 hr on 20 July and off at 08:14 on 21 July (average flow = 0.92 gpm). Pump test started on 22 July at 08:14 hr. Average flow was 0.42 gpm.

**Ratio relative to first measurement.

4.2 WIPP 25—Culebra (Table 4-2; Figure 4-1)

The water contained copious H_2S, decreasing from >5 mg/L at the beginning to ~2 mg/L near the end of the pump test. During the same time, the iron doubled from ~0.3 mg/L to ~0.6 mg/L. Bicarbonate dropped and Eh rose. Near the end of the run, the water no longer turned black after standing.

Field measurements stabilized after 24 hr of pumping; final geochemical samples were collected after ~28 hr of pumping and radiocarbon samples 2 or 3 hr later.

4.3 WIPP 25—Magenta (Table 4-3)

Initially the water smelled rank and of H_2S, and turned gray after standing. Late in the run, H_2S and darkening were no longer noticed, but the rank odor persisted.

The water contained >5 mg/L H_2S and ~0.3 mg/L Fe at the beginning, and 2 to 5 mg/L H_2S and ~0.6 mg/L Fe at the end of the run. Bicarbonate dropped and Eh rose.

Field measurements stabilized after 15 hr of pumping. Final geochemical samples were collected after ~25 hr of pumping, and radiocarbon samples were collected after ~42 hr of pumping.

4.4 WIPP 26—Rustler/Salado Contact (Table 4-4)

The water appeared red to dark reddish-brown throughout the run.

Divalent cations rose ~10%.

This well was pumped initially for ~19 hr at a rate of 0.92 gpm. After 24 hr of recovery, a second test at 0.42 gpm was begun. Field measurements stabilized ~30 hr into this second test. Final geochemical samples were collected after ~36 hr of pumping (second test), and a radiocarbon sample was taken 1 hr later.

Specific conductance and specific gravity were not measured because of unavailability of equipment. Calcium was not measured due to insufficient time and personnel. WIPP 29 was also being monitored.

4.5 WIPP 26—Culebra (Table 4-5)

Some H_2S (0.3 to 0.5 mg/L) was initially present, but dissipated toward the end of the run. No darkening was noted.

Bicarbonate dropped somewhat.

Field measurements stabilized after ~20 hr of pumping. Final geochemical samples were collected after ~26 hr of pumping and radiocarbon samples were taken ~3 hr later.

Water in this well had to be recirculated in part to maintain an optimum flow for the pump.

Table 4-5 WIPP 26—Culebra Member*

Date (1980)	Time	Temp (°C)	Sp Grav	pH	Eh (V)	Sp Cond (μS/cm)	mg/L HCO_3^-	mg/L H_2S	mg/L Fe	$SO_4^{=}$ **	Cl^- **	X^{++} **	Ca^{++} **
23 Aug	13:40	23.0	~1.01	6.9	+0.194	2.6×10^4	149	~0.3-0.5	0.4	1	1	1	1
23 Aug	17:10	21.5	1.005	6.9	+0.128	$\sim2.5\times10^4$	144		0.4	1.0	1.00	0.99	0.99
24 Aug	09:15	21.5	1.005	6.9	+0.147	2.1×10^4	140		0.4		0.99	0.98	0.98
24 Aug	12:20	22.0	1.005	6.9	+0.213	$\sim2.6\times10^4$	141		0.3	1.0	0.98	0.98	0.99
24 Aug	15:15	22.0	1.005	6.9	+0.163	$\sim2.8\times10^4$	141		0.3	1.0	0.98	0.97	0.98

*Pump initially on at 12:10 on 23 Aug and off at ~14:30 hr; pump test started on 23 Aug at 15:30 hr. By-pass incorporated in system. Average flow was 34 gpm.

**Ratio relative to first measurement.

Table 4-6 WIPP 27—Rustler/Salado Contact*

Date (1980)	Time	Temp (°C)	Sp Grav	pH	Eh (V)	Sp Cond (μS/cm)	mg/L HCO_3^-	mg/L Fe	$SO_4^{=}$ **	Cl^- **	X^{++} **
5 Aug	15:15	25.5	1.07	~8.3	−0.082	13×10^4				1	1
6 Aug	07:45	22.5	1.07	7.4	−0.087	12×10^4	81.0		1	0.98	0.98
6 Aug	12:45										
	−13:00	31.0	1.07	7.15	−0.015	14×10^4	70.1			1.01	1.01
6 Aug	16:50										
	−17:05	29.5	1.07	7.1	+0.028	14×10^4	73.3			1.00	1.01
7 Aug	07:45										
	−08:15	25.5	1.07	6.4	+0.121	12×10^4	66.0	18	1.1	0.99	0.99

*Pump test started on 5 Aug at 11:00 hr. Flow was ~0.13 gpm at start of test and decreased to ~0.02 gpm on 7 Aug (average was ~0.07 gpm). 2 L raw sample was saved from each sampling period.

**Ratio relative to first measurement.

Table 4-7 WIPP 27—Culebra Member*

Date (1980)	Time	Temp (°C)	Sp Grav	pH	Eh (V)	Sp Cond (μS/cm)	mg/L HCO_3^-	mg/L Fe	$SO_4^{=}$ **	Cl^- **	X^{++} **	Ca^{++} **
3 Sep	07:20	21.5	1.09	~6.1	+0.248	14×10^4	142	0.5	1	1	1	1
3 Sep	12:20	22.0	1.09	6.2	+0.173	16×10^4	130	0.5	0.9	1.00	1.01	1.01
3 Sep	17:20	22.0	1.09	6.25	+0.163	16×10^4	126	0.4	1.1	0.99	1.00	0.99
# 4 Sep	07:50	24.5	1.09	6.5	+0.051	15×10^4	147	1.8	1.0	1.01	1.01	1.01
## 5 Sep	12.35	22.0	1.09	6.4	+0.208	15×10^4	121	0.4	1.0	0.99	0.99	1.00
5 Sep	13:50						119					

*Pump test started on 2 Sept at 19:15 hr. Flow rate dropped to ~1 gpm sometime soon after 17:20 on 3 Sept. The pump was shut down at 12:00 on 4 Sept and was restarted at 17:40 on 4 Sept. Average flow during test was 25 gpm.

**Ratio relative to first measurement.

#Flow rate was ~1 gpm. 1 L raw sample saved.

##Pump restarted. 1 L raw sample saved.

Table 4-8 WIPP 27—Magenta Member*

Date (1980)	Time	Temp (°C)	Sp Grav	pH	Eh (V)	Sp Cond (μS/cm)	mg/L HCO_3^-	mg/L Fe	$SO_4^{=}$ **	Cl^- **	X^{++} **	Ca^{++} **
24 Sep	13:35	21.0	1.09	6.3	+0.283	14×10^4	242	4	1	1	1	1
24 Sep	16:45	21.5	1.09	6.3	+0.288	14×10^4	223	3	0.9	0.98	1.05	0.99
25 Sep	08:05	21.0	1.09	6.3	+0.184	14×10^4	206	2	1.0	1.01	1.06	1.01
25 Sep	11:10	21.5	1.09	6.4	+0.153	14×10^4	210	2	1.0	1.01	1.03	1.01
25 Sep	14:05	21.0		6.4			213					

*Pump test started on 24 Sept at 11:01 hr. Average flow was 9.4 gpm.

**Ratio relative to first measurement.

Table 4-9 WIPP 28—Rustler/Salado Contact*

Date (1980)	Time	Temp (°C)	Sp Grav	pH	Eh (V)	Sp Cond (μS/cm)	mg/L HCO_3^-	mg/L Fe	mg/L $SO_4^{=}$ **	mg/L Cl^- **	mg/L X^{++} **
29 Jul	11:00	24.0	~1.17	7.4	−0.263	25×10^4	232			(1.83)	(1.22)
# 30 Jul	11:40	23.0	1.10	7.7	−0.073	17×10^4	170	0.1	1	1	1
# 30 Jul	15:15	24.5	~1.18	6.7	−0.054	25×10^4	162			1.88	1.23
30 Jul	15:30	24.5	1.18	6.95	+0.106	25×10^4	184			1.87	1.22
30 Jul	18:35	24.5	1.18	6.9	+0.096	24×10^4	173			1.88	1.24
31 Jul	08:45	24.5	1.18	6.95	+0.091	21×10^4	141		1.4	1.88	1.23
# 31 Jul	16:25	24.0	1.18	7.4	+0.060	24×10^4	166				

*Pump initially on at 14:00 hr on 28 July and off at 08:50 hr on 29 July; turned on briefly at 11:00 on 29 July. Pump test started at 11:00 on 30 July. By-pass started at 14:38 on 30 July. After ~09:00 on 31 July, flow was intermittent. Average flow was 3.1 gpm.

**Ratio relative to "first" measurement (30 Jul 1980, 11:40).

#1 L raw sample saved.

Table 4-10 WIPP 28—Culebra Member*

Date (1980)	Time	Temp (°C)	Sp Grav	pH	Eh (V)	Sp Cond (μS/cm)	mg/L HCO_3^-	mg/L H_2S***	mg/L Fe	mg/L $SO_4^{=}$**	mg/L Cl^- **	mg/L X^{++} **	mg/L Ca^{++} **
9 Sep	16:15	22.0	1.04	6.3	−0.083		849	>5	0.2	(.8)	(.95)	(.82)	(.91)
# 10 Sep	08:15	22.0	1.03	6.4	−0.027	4.85×10^4	409	>5	0.8	1	1	1	1
# 10 Sep	11:15	22.0	1.03	6.4	−0.027	5.6×10^4	441	>5	0.6	1.2	1.16	1.04	1.03
10 Sep	14:55	22.5	1.03	6.4	−0.019	5.7×10^4	431		0.7	1.2	1.15	1.04	1.04
# 10 Sep	18:05	22.0	1.03	6.4	−0.019	5.9×10^4	422		0.8	1.4	1.20	1.05	1.03
11 Sep	09:00	22.5	1.03	6.45	−0.019	5.9×10^4	388	>5	0.8	1.3	1.21	1.07	1.04
# 11 Sep	12:10	22.5	1.03	6.45	−0.025	5.9×10^4	379	>5	0.8	1.4	1.16	1.06	1.02
# 11 Sep	15:00	22.5	1.03	6.45	−0.021	5.9×10^4	374		0.8	1.0	1.18	1.07	1.03
# 11 Sep	17:05			6.5			373						
12 Sep	08:35			6.5			344			1.4			

*Pump initially on at 16:00 on 9 September; pump off at 17:30; pump test started at 21:30 hr. Average flow was 17 gpm.

**Ratio relative to "first" measurement (10 Sep 1980, 08:15).

***Measurement made on sample taken 1 to 3 hr after time indicated.

#1 L raw sample saved.

Table 4-11 WIPP 29—Rustler/Salado Contact*

Date (1980)	Time	Temp (°C)	pH	Eh (V)	HCO_3^- (mg/L)	$SO_4^{=}$**	Cl^- **	X^{++} **
22 Jul	16:20	22.0	6.71	+0.178	278		1	1
# 22 Jul	21:05	20.5	~6.9	+0.190		1		
23 Jul	12:00	22.0	6.9	+0.073	217		1.07	1.03
24 Jul	07:25	20.5	7.15	+0.103	201		1.10	1.04
24 Jul	10:00	22.0	7.15	+0.083				

*Pump on at 12:30 on 22 July. Average flow was 1.5 gpm.
**Ratio relative to first measurement.
#1 L raw sample saved.

Table 4-12 WIPP 29—Culebra Member*

Date (1980)	Time	Temp (°C)	Sp Grav	pH	Eh (V)	Sp Cond (μS/cm)	mg/L HCO_3^-	mg/L Fe	$SO_4^{=}$ **	Cl^- **	X^{++} **	Ca^{++} **
26 Aug	17:15	21.0	1.15	5.9	+0.114	20×10^4	241	1.2	1	1	1	1
27 Aug	07:30	20.0	1.16	6.0	+0.144	18×10^4	220	0.9	0.7	1.00	1.05	1.00
27 Aug	10:35	20.5	1.16	6.0	+0.115	20×10^4	218	0.9	0.8	1.02	1.02	0.99
27 Aug	15:00	20.0	1.16	6.0	+0.124	19×10^4	216	0.9	0.8	1.01	1.03	—#
28 Aug	07:55	20.0	1.16	6.1	+0.139	18×10^4	213	0.8	0.7	1.01	1.05	—#

*Pump test started on 26 August at 14:30 hr. Average flow was 37 gpm.
**Ratio relative to first measurement.
#Titrimetric endpoint indistinct.

Table 4-13 WIPP 30—Rustler/Salado Contact*

Date (1980)	Time	Temp (°C)	pH	Eh (V)	HCO_3^- (mg/L)	Cl^- **	X^{++} ***
16 Jul	10:25	25.5	7.3	−0.372	532	1	
16 Jul	14:40	26.5	7.4	−0.377	576	1.00	
16 Jul	17:35	25.0	7.5	−0.379	544	0.99	
16 Jul	20:50	22.5	7.5	−0.362	560	0.99	
17 Jul	12:25	25.5	7.45	−0.364	624	1.04	1
17 Jul	14:30	26.0	7.45	−0.357	617	1.02	1.0

*Pump test started on 15 July at 19:00 hr. Average flow was 0.22 gpm. 1 L raw sample saved from each sampling period.

**Ratio relative to first measurement.

***Divalent cations were not determined in the first four samples because of a lack of reagents. Ratio relative to measurement of 17 July, 12:25 hr.

4.6 WIPP 26—Magenta

The Magenta member of the Rustler Formation did not contain sufficient quantities of water to pump.

4.7 WIPP 27—Rustler/Salado Contact (Table 4-6)

Effluent water was clear but turned yellowish upon standing.

Bicarbonate dropped ~20% and Eh rose to slightly oxidizing conditions during the run. Calcium was not measured because of time constraints.

After 24 hr, the field measurements showed evidence of stability. However, the continually dropping water level, dropping pump rate, and high Fe content (18 mg/L) indicated that the water had been in prolonged contact with the pipe and was no longer representative of the reservoir. Except for one raw sample in a glass bottle, no samples for future analysis were collected (geochemical or radiocarbon).

4.8 WIPP 27—Culebra (Table 4-7)

The water was mostly clear, but turned murky just before the "pump failure" in the middle of the run.

Bicarbonate dropped ~15% during the run.

After ~22 hr of pumping at >22 gpm, the flow rate suddenly dropped to ~1 gpm. The pump was shut down ~19 hr later and after ~6 hr of recovery was restarted. Field measurements had stabilized after 17 hr. Some geochemical samples were taken just before the change in flow rate; others were taken ~20 hr after restart, as was the radiocarbon sample.

This hole was almost inaccessible throughout the run because of poor drainage of the road after a flash flood.

4.9 WIPP 27—Magenta (Table 4-8)

Throughout the run the water was frothy white and milky-looking, with a rank smell.

Bicarbonate dropped ~15% during the run.

Field measurements stabilized after ~21 hr; final geochemical samples were taken after ~26 hr of pumping and radiocarbon samples were taken 1 to 2 hr later.

4.10 WIPP 28—Rustler/Salado Contact (Table 4-9)

Shortly after pumping began, the pump was turned off. When it was briefly restarted to allow sampling, milky water resulted that turned dark brown after standing. Throughout the final run, the water remained milky white and became tinged with a rose-brown precipitate of sediment after standing.

Table 4-14 WIPP 30—Culebra Member

Date (1980)	Time	Temp (°C)	Sp Grav	pH	Eh (V)	Sp Cond (μS/cm)	mg/L HCO_3^-	mg/L CO_3^-	mg/L Fe	$SO_4^{=}$***	Cl^- ***	X^{++} ***	Ca^{++} ***
*A. Contaminated With Leak From Rustler/Salado Contact**													
19 Aug	09:50	24.0	1.04	8.62	+0.054	7.5×10^4	59.0	14.1		1	1	1	1
19 Aug	17:05	23.0	1.07	7.7	−0.403	13×10^4	219		6.5	1.3	1.90	1.46	1.16
20 Aug	08:35	21.5	1.11	7.55	−0.388	16×10^4	339		5.2	1.4	2.92	1.80	1.03
20 Aug	13:00	24.0	1.11	7.55	−0.413	18×10^4	330		3.8	1.4	2.98	1.78	1.02
20 Aug	18:10	23.5	1.115	7.5	−0.369	18×10^4	346		0.7	1.4	3.09	1.72	0.99
21 Aug	09:50	23.0	1.12	7.55	−0.423	17.5×10^4	362		0.1	1.3	3.39	1.94	0.93
21 Aug	14:20	25.0	1.12	7.6	−0.374	20×10^4	358		0.1	1.4	3.39	1.93	0.93
21 Aug	17:20	23.0	1.12	7.55	−0.35#	20×10^4	342		0.2	1.2	3.29	1.90	0.92
22 Aug	07:20	21.5	1.125	7.45	−0.377	17.5×10^4	350		0.2	1.2	3.48	1.97	0.90
*B. Retest After Packer Leak Remedied***													
2 Sep	19:15	21.5	1.06	7.9	+0.038	$\sim11\times10^4$			0.24	1	1	1	1
3 Sep	10:05	23.5	1.05	7.75	+0.162	$\sim8.5\times10^4$	93.4		1.0	0.9	0.76	0.89	1.03
3 Sep	15:05	24.5	1.03	7.9	+0.094	$\sim6.2\times10^4$	66.1		0.24	0.7	0.42	0.82	1.05
3 Sep	20:05												
	−20:10	21.5	1.02	8.2	+0.153	$\sim4.4\times10^4$	55.7		0.12	0.7	0.34	0.78	1.04
4 Sep	09:50												
	−10:00	23.5	1.02	8.5	+0.197	$\sim4.2\times10^4$	50.3	4.2	0.06	0.9	0.30	0.77	1.03
4 Sep	19:10												
	−19:20	22.5	1.02	8.7	+0.173	4.3×10^4	44.7	8.8	0.04	0.9	0.30	0.76	1.03
5 Sep	08:30												
	−08:40	22.5	1.02	8.8	+0.153	3.8×10^4	40.2	15.2	0.04	0.9	0.29	0.76	1.02
5 Sep	17:10												
	−17:20	22.5	1.02	8.85	+0.178	4.1×10^4	34.3	16.9	0.05	0.9	0.30	0.77	1.03
5 Sep	20:15												
	−20:25	22.0	1.02	8.9	+0.183	3.7×10^4	29.5	18.0					
6 Sep	04:25	21.0		~8.8			40.2	16.9					

*Average flow was 0.26 gpm. 1 L raw sample saved from each sampling period.

**Pump test started on 2 September at 13:00 hr. Flow initially was 0.3 gpm and decreased to 0.1 gpm; average flow was 0.2 gpm. 1 L raw sample saved from each sampling period.

***Ratio relative to first measurement.

#Unstable reading.

Table 4-15 WIPP 30—Magenta Member*

Date (1980)	Time #	Temp (°C)	Sp Grav	pH	Eh (V)	Sp Cond (μS/cm)	mg/L HCO_3^-	mg/L $CO_3^=$	mg/L Fe	mg/L $SO_4^=$**	mg/L Cl^- **	mg/L X^{++} **	mg/L Ca^{++} **
3 Dec	13:25-13:35						78.3	0					
	13:35-13:50	17.5	1.01	8.25	+0.259	1.37×10^4			0.12	1	1	1	1
4 Dec	08:45-08:55						67.5	8.65					
	08:55-09:10	15.0	1.005	8.6	+0.237	1.14×10^4			0.08	1.1	0.99	0.99	0.95
4 Dec	12:45-12:55						74.6	3.63					
	12:55-13:10	20.0	1.004	8.45	+0.219	1.35×10^4			0.07	1.1	1.03	1.03	1.00
## 5 Dec	12:35-12:45						54.7	18.4					
	12:45-13:05	19.0	1.01	8.55	+0.187	2.42×10^4			0.05	3.1	1.80	1.89	2.14
6 Dec	08:35-08:50						62.7	10.9					
	08:50-09:10	14.0	1.01	8.6	+0.278	1.83×10^4			0.08	2.6	1.39	1.70	1.94
6 Dec	12:30-12:45						57.9	14.5					
	12:45-13:05	20.0	1.01	8.55	+0.224	1.94×10^4			0.08	2.4	1.32	1.68	1.91
7 Dec	08:15-08:30						63.8	9.21					
	08:30-08:50	15.0	1.01	8.55	+0.257	1.65×10^4			0.09	2.6	1.09	1.59	1.82
8 Dec	08:00-08:15						48.2	15.4					
	08:15-08:45	4.0	1.01	8.55	+0.335	1.25×10^4					0.99	1.56	1.74
8 Dec	14:25-14:40						50.5	14.0					
	14:40-15:05	5.0	1.01	8.55	+0.269	1.31×10^4					0.96	1.55	1.73
9 Dec	08:15-08:30						49.4	14.8					
	08:30-09:00	7.0	1.01	8.6	+0.335	1.16×10^4					0.88	1.53	1.70
### 10 Dec	07:55-08:10						58.7	8.09					
	08:10-08:40	7.5	1.005	8.55	+0.322	1.11×10^4					0.81	1.49	1.66

*Pump test started on 2 December at 16:00 hr. Flow initially was ~100 mL/min and decreased to approximately half that value during the run.
1 L raw sample saved from each sampling period.

**Ratio relative to first measurement.

#Flow rate so low that two sampling intervals were required to collect enough water:
First interval—sample taken for $CO_3^=/HCO_3^-$;
Second interval—sample taken for all other measurements

##Attempt to measure water level with steel tape on 4 Dec; sampling conditions perturbed.

###Decision made to terminate run. Between 09:50 and 11:05 hr, 3 L of sample were collected (1 each raw, filtered, filtered and acidified); however, these are *not* considered representative samples of WIPP 30-Magenta water.

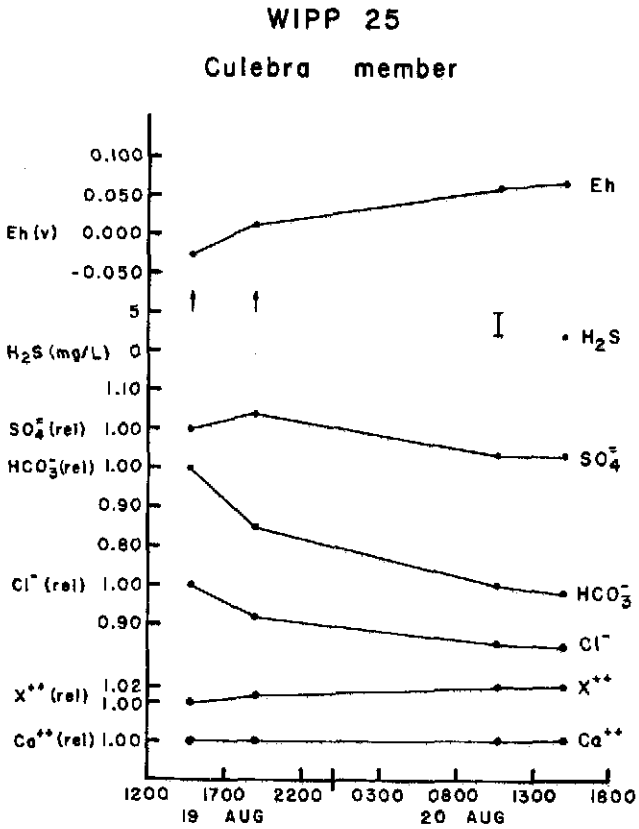


Figure 4-1 Trends in Measurements of Aqueous Species in Water From the Culebra Member, WIPP 25. (All measurements except Eh and H_2S are reported as ratios relative to the first measurements at 1445 hr, 19 August. Typical behavior of aqueous species in this and many other tests is an asymptotic approach to some steady-state value as a function of time.)

The pump rate was changed during this run. In response to the changes, the bicarbonate varied ~50%, chloride ~90%, and divalent cations ~20%. Eh changed from moderately reducing to mildly oxidizing during the run.

The well was pumped for ~19 hr and allowed to recover for ~26 hr. As mentioned above, 2 hr into the recovery period the pump was turned on for a few minutes and a sample was taken. The pump test was restarted, but the well stopped producing after ~22 hr of pumping, during the final anticipated interim sampling. However, the analyses showed that the field measurements had stabilized after a few hours. The water collected in the bucket was then used as the final sample and was insufficient to make up the full complement of final preserved samples (Table 2-2). The well started producing again and a radiocarbon sample was collected a few hours later that day; this sample may have equilibrated with atmospheric CO_2 in the bottom of the hole.

Calcium was not measured because of time constraints imposed by a long drive from field trailer to wellhead.

Water in this well had to be recirculated in part to maintain an optimum flow for the pump.

4.11 WIPP 28—Culebra (Table 4-10; Figure 4-2)

The water started as murky dark-gray, gradually lightening during the run to milky white and finally to clear, but always with H_2S (>5 mg/L). The water was always heavily effervescent. For some analyses, the murkiness was not removed by filtration through Whatman 41 filter paper.

Bicarbonate steadily dropped 20% over the entire run and never stabilized. Eh remained mildly reducing throughout the run.

After ~22 hr of pumping, the field measurements stabilized (except for bicarbonate). Final geochemical samples were taken after ~45 hr of pumping.

A radiocarbon sample was not collected here because of the continually decreasing bicarbonate values, as well as the heavy effervescence.

4.12 WIPP 28—Magenta

The Magenta member of the Rustler Formation did not contain enough water to pump.

4.13 WIPP 29—Rustler/Salado Contact (Table 4-11)

The water started out as black, clearing by the end of the run.

Bicarbonate dropped 25% during the run.

After 42 hr, field measurements apparently stabilized. Final geochemical samples were taken after ~46 hr of pumping; a radiocarbon sample was taken 1 hr later.

Specific conductance and specific gravity were not measured because of nonavailability of equipment. Calcium was not measured because of insufficient time and personnel. WIPP 26 was also being monitored.

4.14 WIPP 29—Culebra (Table 4-12)

The water remained clear throughout the run.

Bicarbonate dropped ~10% during the run.

Field measurements stabilized after ~24 hr. Final geochemical samples and a radiocarbon sample were taken after ~44 hr of pumping.

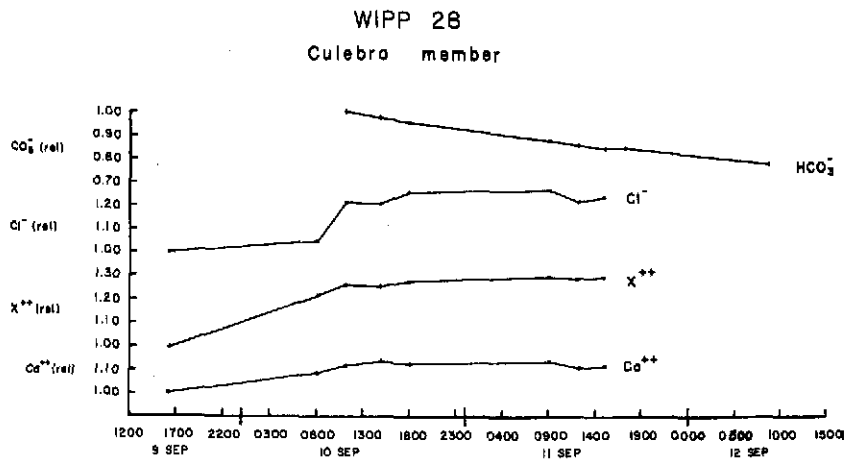


Figure 4-2 Trends in Measurements of Aqueous Species in Water From the Culebra Member, WIPP 28. (Measurements of Cl^-, X^{++}, and Ca^{++} are reported relative to the values obtained shortly after the beginning of a 90-min pumping episode, 1600 hr, 9 September. HCO^-_3 values are reported relative to the value as of 1115 hr, 10 September. Note the continued, steady, significant drop in HCO^-_3, with little leveling trend, after other solutes were judged to have attained steady-state values. Steady drop in bicarbonate with continued effervescence suggests a nongeological source of carbon dioxide.)

4.15 WIPP 29—Magenta

The Magenta member of the Rustler Formation does not occur in the subsurface of WIPP 29.

4.16 WIPP 30—Rustler/Salado Contact (Table 4-13)

The water smelled rank throughout the run and turned slightly yellowish on standing.

Bicarbonate rose ~15% during the run.

Field measurements stabilized after ~28 hr. Final geochemical samples were taken after ~45 hr of pumping.

Specific conductance and specific gravity were not determined because of unavailability of equipment. The calcium measurements were abandoned because of indistinct titrimetric endpoints. Divalent cations were not determined on 16 July because of a lack of reagents.

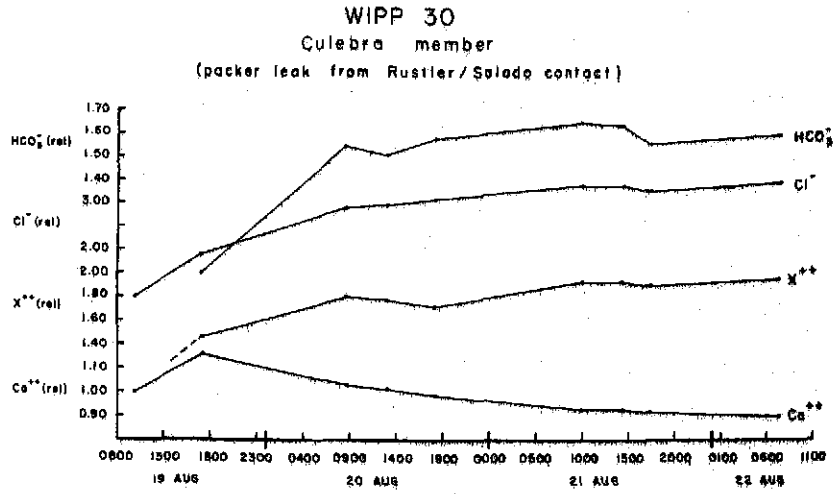
A radiocarbon sample was not collected here. This was one of the first wells tested, along with WIPP 25—Rustler/Salado; the water was very high in total dissolved solids. The chemical precipitation method did not work. We were not prepared at that time to collect bulk samples.

4.17 WIPP 30—Culebra (Table 4-14; Figure 4-3)

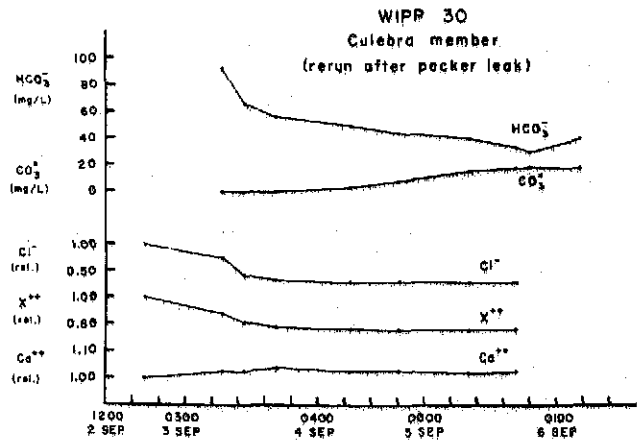
During the pumping from 19 August to 22 August 1980 (Table 4-14 (A)), these measurements rose steadily for 69-1/2 hr: specific gravity (10%), specific conductance (130%), bicarbonate (60%), chloride (350%), and divalents (100%). These values suggested that the solution contained a component of solution similar to that from the Rustler/Salado contact on 16 and 17 July 1980. A leak was suspected and the run was terminated. When the packer between the Rustler/Salado contact and the Culebra was removed for inspection, it was discovered that the shear-plug was missing, thus confirming the suspected leak. Throughout this run, the water came out clear but turned yellow-greenish upon standing, similar to water from the WIPP 30 Rustler/Salado contact. The entire test was rescheduled and repeated on 2 to 6 September 1980 (Table 4-14 (B)).

During the second run of WIPP 30 Culebra, the water first came out dirty-brown, gradually lightened to brownish-gray, and was light-gray at the end of the run.

Bicarbonate fell while carbonate and pH rose. Chloride dropped 70%, divalents 20%, and iron by a factor of 20—all indicating purgation of the "leak water" from the previous run.



A: Wide fluctuations in measurements together with no indication of approach to steady-state values suggested nonsystematic but time-dependent addition of another solution. This suspicion was confirmed on discovery of a leak in the packer that was intended to isolate the Culebra from the producing zone of the Rustler/Salado contact.



B: Moderate monotonic changes in measurements together with asymptotic approaches to steady-state values indicated that in the rerun after the packer was replaced, the local Culebra system was being purged of contaminant fluid from the Rustler/Salado contact. Note also the rise in carbonate/bicarbonate ratio in response to a rising pH (cf. Table 4-14B).

Figure 4-3 Trends in Measurements of Aqueous Species in Water From the Culebra Member, WIPP 30.

Field measurements stabilized after 54 hr. A radiocarbon sample was collected after ~81 hr of pumping, and final geochemical samples were taken after ~89 hr of pumping.

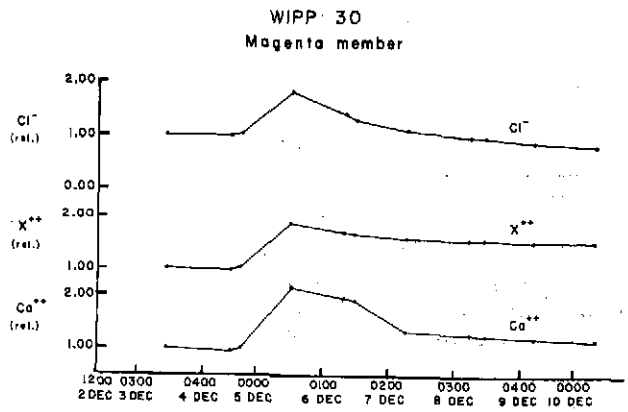


Figure 4-4 Trends in Measurements of Aqueous Species in Water From the Magenta Member, WIPP 30. Because of the low discharge rate (<100 mL/min) and the slow recovery from mechanical perturbation on 4 December, it was judged that steady state would not be achieved in a reasonable time; the test was terminated after 169-1/2 hr.)

4.18 WIPP 30—Magenta (Table 4-15; Figure 4-4)

As the run proceeded, small but increasing amounts of black sediment settled out from samples.

Throughout this run, pump-discharge rate was <100 mL/min. During the run, a field engineer attempted to measure depth-to-water with metal tape. This introduced a perturbation resulting in abrupt rise in sulfate, carbonate, chloride, divalents, and calcium as well as specific gravity and specific conductance that was exceedingly slow to dissipate.

After ~184 hr, there was no evidence of stability of field measurements and the run was terminated. Raw samples in glass bottles were taken for possible stable-isotope analyses; other samples (geochemical and radiocarbon) were not taken.

5. Discussion: Useful Stability Indicators

Two of the most useful indicators of steady-state geochemical stability are chloride and divalent cations (predominantly alkaline earths). Calcium presents some difficulty in detecting titrimetric endpoints with a dye indicator, and has proven not as useful as chloride and divalent cations (the latter probably indicate soluble magnesium in the evaporite environment). Trends in carbonaceous species are of limited value as indicators of steady state, since significant changes in bicarbonate and carbonate were observed long after the values of chloride, divalents, and Eh had stabilized.

We found that specific conductance alone was not a reliable indicator of steady state. Conductance is not a linear function of total dissolved solids, nor is it proportional to any single ionic species. Rather, it depends on temperature and concentration of several ionic species. We found (Tables 4-2 through 4-15) that during many of the pump tests, specific conductance varied after solute concentrations had stabilized. Conversely, we found that in other pump tests some of the solutes varied even with a stable specific conductance.

In short, several parameters had to be evaluated before we could determine steady state, not necessarily equilibrium. Allowances had to be made for natural variation outside experimental error, especially in Eh and bicarbonate, since in some cases all other measurements could be stable except those two.

Certain approximately determined constituents were sometimes useful as indicators of abrupt change or of system purging and stability. These are iron, sulfate, and H_2S (if within the limits of the measurement method). It is particularly desirable to achieve as low an iron level as possible in samples to be subsequently analyzed for $^{234}U/^{238}U$ ratio or any heavy metals. Barr et al (1978) showed that contamination from iron pipe introduces uranium with an isotope ratio lower than that of the steady-state value in the water.

The criteria for stability also took into account the history of measurements. For example, two consecutive sets of measurements in reasonable agreement may be used to indicate stability for a fast-flowing well whose effluent rate and properties showed little variation throughout its pump test. However, longer observation and smaller tolerances of variations may be required for a well that trickles rather than flows and that shows larger variations early in its pumping history.

6. Subsequent Analyses

Certain field measurements reported here as absolute results are taken as the most representative available values: pH, Eh, specific conductance, specific gravity, temperature, and bicarbonate/carbonate. These measurements were made soon after wellhead sampling, as recommended by Brown et al (1970). The limitations of these data were discussed above. Certain other measurements under field conditions were reported as relative values (measurements of chloride, divalent cations, calcium, etc). These measurements were made only to evaluate the steady-state solute content and are not to be used for any other purpose. These and other solute determinations will be redetermined under carefully controlled laboratory conditions. Analyses that are routinely made under the auspices of Sandia's WIPP Geochemistry Program are:

- Major and minor inorganic solutes
- $^{18}O/^{16}O$ and D/H ratios for water origin
- $^{234}U/^{238}U$ for any possible indication of time of confinement in the rock

Preliminary (approximate) solute concentrations and the best available values of the "perishable" parameters are reported in Table 6-1.

7. Conclusions

We have shown that any arbitrarily chosen sample from a pumped or bailed well cannot be shown as representative of the nearly unperturbed subsurface reservoir. Any geochemical interpretation of a random sample depends heavily on the history of the well. The well system, which includes local reservoir rock and the sampling system, must be purged of introduced material and stagnant products of the interaction of water with metal before a geologically significant water sample can be collected and analyzed. The time required for this purging may be several times greater than that required to displace a single well-bore volume of fluid.

Criteria for determining a sample's significance are not absolute, but must instead be based on long-term observations of the well that include withdrawal of water and periodic measurements. Certain of these measurements should be routinely made on any sample as soon as possible because of their transient nature: temperature, specific gravity, specific conductance, pH, Eh, and certain dissolved gases (e.g., the CO_2 species). These measurements alone are usually not sufficient stability criteria. Effluent temperature, particularly at low flow rates, reflects diurnal temperature variations of the atmosphere. Specific gravity is temperature-dependent and is only an approximate measurement of *total* dissolved solids. The case of specific conductance is similar. Eh and pH, although useful in certain instances, can change upon the exposure of water to atmosphere, and therefore represent approximations to certain intensive thermodynamic properties. Bicarbonate/carbonate may be similarly short-lived, and (as discussed above) the geological significance of that measurement and percent-modern-carbon is suspect in view of the many sources of CO_2 in the local groundwater system. These sources include atmospheric CO_2, dissolved carbonate rock, metabolic CO_2, introduced organic material, etc.

We have in some cases provided the best available estimates for field-measured solution parameters. These measurements are derived from evaluating the approach of the locally pumped well system to steady state in terms of solution properties. Measurements of transient constituents would have questionable significance if repeated in the laboratory after samples had stood for more than a day or so. However, certain measurements require both sophisticated laboratory facilities and samples specially collected and preserved according to the nature of each measurement. In such cases field measurements should not be used nor cited if laboratory measurements are to be made under more stringent laboratory standardization and instrumentation. Some laboratory measurements, regardless of sophistication, may never be geologically significant because of the nature of the local groundwater system. Such limitations are particularly important for heavy metals because of the ubiquitous iron pipe used in test holes, and for carbonaceous species because of the organic materials introduced into a hole and then permanently entrapped. Organic materials are commonly used in drilling fluid in order to control lost circulation.

Table 6-1 Best Available Field Measurements and Estimates of Some Solute Concentrations

Well/Aquifer	Temp* (°C)	pH*	Eh* (V)	Sp Grav*	Sp Cond* (μS/cm)	HCO_3^-**	$CO_3^=$**	H_2S**	Approx Cl^- #	Approx $SO_4^=$ #	Approx Ca^{++} #	Approx X^{++} ##
WIPP 25 Rustler/Salado	22	7.4	−0.04	—	—	130	—	—	2×10^5	—	—	3×10^2
WIPP 25 Culebra	23	6.9	+0.06	1.01	2×10^4	210	—	~2	5×10^3	3×10^3	9×10^2	7×10^1
WIPP 25 Magenta	22	6.9	+0.07	1.004	1.5×10^4	180	—	~2	5×10^3	3×10^3	9×10^2	7×10^1
WIPP 26 Rustler/Salado	25	7.7	+0.02	—	—	270	—	—	1×10^5	—	—	2×10^2
WIPP 26 Culebra	22	6.9	+0.16	1.005	3×10^4	140	—	—	7×10^3	3×10^3	1×10^3	9×10^1
WIPP 27 Culebra	22	6.3	+0.16	1.09	16×10^4	120	—	—	8×10^4	4×10^3	3×10^3	3×10^2
WIPP 27 Magenta	21	6.4	+0.15	1.09	14×10^4	210	—	—	8×10^4	3×10^3	4×10^3	3×10^2
WIPP 28 Rustler/Salado	24	7.0	+0.09	1.18	24×10^4	170	—	—	1×10^5	2×10^4	—	3×10^2
WIPP 28 Culebra	22	6.5	−0.02	1.03	6×10^4	—	—	>5	2×10^4	4×10^3	1×10^3	1×10^2
WIPP 29 Rustler/Salado	22	7.2	+0.08	—	—	200	—	—	6×10^4	1×10^4	—	2×10^2
WIPP 29 Culebra	20	6.1	+0.14	1.16	18×10^4	210	—	—	1×10^5	1×10^4	1×10^3	5×10^2
WIPP 30 Rustler/Salado	26	7.5	−0.36	—	—	620	—	—	2×10^5	—	—	3×10^2
WIPP 30 Culebra	21	8.8	+0.18	1.02	4×10^4	40	17	—	1×10^4	4×10^3	1×10^3	1×10^2

*Values at time of final sampling. Estimated accuracies are: Temperature, ±1°C; pH, ±0.1 pH unit; Eh, ±0.01 V; specific gravity, ±0.02; specific conductance, ±10%.

**Units are mg/L. Confidence limits (95%; based on 1 replicate) are ±6 mg/L for carbonate and ±10 mg/L for bicarbonate. The H_2S concentrations should be viewed as rough approximations.

#Determinations will be repeated in laboratory; units are mg/L.

##X^{++} = Divalent cations; units are meq/L.

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