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# Mineralogy in the Waste Isolation Pilot Plant (WIPP) Facility Stratigraphic Horizon



Carol L. Stein

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# Mineralogy in the Waste Isolation Pilot Plant (WIPP) Facility Stratigraphic Horizon

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### Abstract

Forty-six samples were selected for this study from two cores, one extending 50 ft up through the roof of the WIPP facility and the other penetrating 50 ft below the facility floor. These samples, selected from approximately every other foot of core length, represent the major lithologies present in the immediate vicinity of the WIPP facility horizon: "clean" halite, polyhalitic halite, argillaceous halite, and mixed polyhalitic-argillaceous halite. Samples were analyzed for non-NaCl mineralogy by determining weight percents of water- and EDTA-insoluble residues, which were then identified by x-ray diffraction. In general, WIPP halite contains at most 5 wt% non-NaCl residue. The major mineral constituents are quartz, magnesite, anhydrite, gypsum, polyhalite, and clays. Results of this study confirm that, in previous descriptions of WIPP core, trace mineral quantities have been visually overestimated by approximately an order of magnitude.

## **Acknowledgments**

Gautam Sarkar, a graduate student in the Geology Department at the University of New Mexico, provided the weight-percent data and the x-ray diffraction analyses of the non-NaCl mineral residues. His attention to detail throughout both phases of this timeconsuming project is gratefully acknowledged. In addition, I thank Klaus Keil of the Institute of Meteoritics, the UNM Geology Department, for his assistance and for many helpful discussions. The manuscript was much improved by the reviews provided by David J. Borns of Division 6331 and by Klaus Keil.

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# Mineralogy in the Waste Isolation Pilot Plant (WIPP) Facility Stratigraphic Horizon

## Introduction

As part of the Waste Isolation Pilot Plant (WIPP) continued geotechnical studies, Sandia National Laboratories (SNL) has been charged with the task of producing a detailed characterization of the geologic interval in the immediate vicinity of the WIPP facility. Preliminary work toward this end was begun in 1983 with the Site and Preliminary Design Validation (SPDV) report. The SPDV work includes a comprehensive mineralogical analysis of 43 rock samples taken at or near the facility horizon. These samples consisted primarily of grab samples taken from the excavation face as mining proceeded, and of selected samples from cores taken from some of the excavated rooms. These samples were processed for their mineralogical components by a method described in detail in the final SPDV report (Stein, 1983) and summarized elsewhere in this report. The end result was a compilation of data that quantitatively describe the non-NaCl portion of the host halite in the general stratigraphic vicinity of the WIPP facility horizon.

An important conclusion from this set of data is that, up until the time of the SPDV report, visual estimates of the non-NaCl components were incorrect by as much as an order of magnitude or more. The appearance of the Salado halite can be deceptive, especially when it contains small quantities of very fine-grained, disseminated polyhalite or clay minerals. In summary, from the SPDV work it was learned that WIPP halite contains an average maximum of 5 wt% non-NaCl mineralogy, except in the cases of units such as anhydrites and clay seams.

This information was, in turn, used to revise some of the ongoing work directed toward predicting longterm creep behavior of the salt around the excavated cavity. However, one shortcoming of the SPDV work concerned the distribution of the sample locations of the analyzed material. It was felt that a more comprehensive body of data could be obtained by using samples from precisely known locations and could be collected on a more tightly spaced grid. Moreover, the plan for the sampling program that was ultimately adopted also provided for samples to be taken simultaneously for rock mechanics testing by W. Wawersik (SNL, Division 1542). Lastly, executing this program provided a set of reference samples for the purpose of making more quantitative visual comparisons of rock samples and a suite of archived core that will remain on file at the WIPP site for future reference.

## Methods

The samples selected for this study were taken primarily from two cores, each  $\sim 50$  ft long and  $4\frac{1}{2}$  in. diameter, that were cut in Test Room 4 of the WIPP facility (see map, Figure 1). These cores were cut with a Longyear 38 drill rig, using no liquid lubricant. The cores used in this study are designated RM-1 and RM-3; these were cut vertically upward into the roof, and directly opposite were cut vertically downward, respectively. The lithologic logs for these cores are shown in Appendix A.

Samples were chosen from approximately every other foot along the length of the cores. Brief descriptions of the portions selected, along with sample numbers and footages, are listed in Appendix B. Samples were selected from each of the dominant lithologies observed in the core. These are: (1) "clean" halite, (2) polyhalitic halite, (3) argillaceous halite, (4) mixed argillaceous-polyhalitic halite, and (5) anhydrites and clay seams. These samples, each  $\sim 6$  in. long, were subsequently slabbed; one half was sent to Sandia for processing and analysis; the other remained in Carlsbad for the reference collection.

The technique used to process these samples has been described in detail (Stein, 1983). To summarize briefly, the samples, weighing from  $\sim 200$  g to  $\sim 1$  kg, were crushed to pieces the size of  $\leq 1$  cm<sup>3</sup>. Weighed amounts of samples were then placed in large beakers of distilled water and stirred continuously until all salt was dissolved. The beakers were then decanted and the remaining residues were collected by filtration onto preweighed Whatman #3 filter papers, allowed to air-dry, and then weighed again. The dry weights of the water-insoluble residues are reported in Table 1. Small fractions of these residues were reserved for x-ray diffraction analysis; results are shown in Table 2. The remaining portions were further processed by boiling for 4 hr (or longer, as necessary) in 0.25-M EDTA solution. This technique, developed by Bodine and Fernalld (1973), removes all divalent carbonates and sulfates from the water-insoluble residues. Following boiling, the samples were again collected onto preweighed Whatman #3 filter papers, dried, and weighed again to obtain the EDTA-insoluble residue weights (shown as weight percents) in Table 3. As before, where sample material was abundant enough, x-ray diffraction analyses were performed. These results are shown in Table 4.



Figure 1. Map of underground workings (from Bechtel, 1985) showing location of Test Room 4 and location of core holes. (Note: Cores RM-3 and RM-4 are down; Cores RM-1 and RM-7 (not shown) are up.)

Sample	Sar De	nple pth ît)	Sample Weight (g)	Weight of Water- Insoluble Residue (g)	Weight % (whole rock)
	From	То			
FH-201	2	2.5	757	2.76	0.36
FH-202	4	4.7	482	165.58	34.35
FH-203	8.3	9	649	13.09	2.02
FH-204	10.8	11.3	734	6.83	0.93
FH-205	13	13.55	715	0.38	0.05
FH-206	15.55	15.85	696	27.05	3.89
FH-207	16.9	17.45	700	1.41	0.2
FH-208	18.4	19.1	670.2	2.69	0.4
FH-209	19.65	20.05	641.3	1.1	0.17
FH-210	21.5	22	742.55	13.86	1.87
FH-211	23	23.5	700	3.6	0.51
FH-212	25.1	25.7	700	5.7	0.81
FH-913	26	26.5	700	4.57	0.65
FH 914	28 25	28.85	751	1.54	0.00
F11-214 FU 915	30.5	31.05	700	0.47	0.07
FH-215 FU 916	30.0 31	38	674	9.93	1 47
FH-210 FU 917	30.95	39.85	535	13.49	9 51
FII-217	35.20 41	11 5	617 5	10.42	1 77
FIL 010	41	41.0	676 5	21 12	2 1 9
FH-219	42.0	40	700	8 79	1 945
FH-220	44	44.0	749.95	4.04	1.240
FH-221	40.0	40.00	740.00	5.04	0.04
FH-222	41	47.0	720.5	0.04	0.19
FH-223	49.05	49.00	100.0	2.2	0.3
FH-224	4	4.0	309.0	235.74	01.14
*core loss zone **From RM-4					
FH-228	0.4	0.9	1,000	1.425	0.14
FH-229	2.3	2.8	1,000	0.77	0.08
FH-230	4.75	6.75	1,000	2.38	0.24
FH-231	7.1	7.3	600	169.89	28.32
FH-232	7.75	8.15	900	3.27	0.36
FH-233	8.15	9.1	1.000	0.45	0.05
FH-234	10.5	10.9	1.000	13.46	1.35
FH-235	12.5	12.6	1.000	2.87	0.29
FH-236	14	14 45	200	174.15	87.08
FH-237	16 1	16.6	700	12.99	1 86
FH.938	29	29.5	550	34.68	6.31
FH 990	34.05	34.5	600	18 58	31
F11-200	36.0	376	700	184 11	
FII 941	38.9	38.7	1 000	3.04	20.3
ГП-241 БЦ 049	42.05	19 55	600	0.7	0.0
ГП-242 FU 049	40.00	40.00	850	J.1 G 41	1.02
F FI-243	41.2	41.1	200	0.41	0.75
FH-244	49.00	00 50 1	300	29.03	9.68
r n-240	91.3 90.05	02.1 90.45	400	31.12	9.3
FH-246T	20.05	20.45	1,000	ð	0.8
FH-247*	23.45	23.95	bUU 1.000	81.2	13.53
FH-248*	25.5	26	1,000	0.47	0.05
r H-249*	40	40.00	900	4.96	0.55
*From RM-7					

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## Table 1. Weight Percents of Water-Insoluble Residues

### Table 2. X-Ray Diffraction Results from Water-Insoluble Residues

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Sample No.	Quartz (Q)	Magnesite (M)	Anhydrite (A)	Gypsum (G)	Halite (H)	Polyhalite (P)	Bassanite (B)	Approx. Abund.	Layer 14Å	Silicate 10Å	Basal 9.3Å	Plane Peaks 7Å
FH-201	+	+			+			<b>β</b> >M>H				
FH-202		+?	+		+			$A > H > M^{2}$				
FH-203	+	+	+					Q > A > M				
FH-204	+	+			Traces			M>0>H				
FH-205	+							···				
FH-206	+	+	+		+			Q > A > M = H				
FH-207	+	+	4			+		$\dot{M} > Q > P > A$				
FH-208	+	+?	+	+	+			$G>H\geq A>Q>M$				
FH-209	+	+						M>Q				
FH-210	+	+ .		+		+		P > G > M > Q				
FH-211	+	+	+		+			Q > M > H > A				
FH-212	+	+		+				M > Q > G				
FH-213	+	+						M≥Q				
FH-214				+		+		G>>P				
FH-215	Not enoug	h sample av	ailable for x	ray diffract	tion study							
FH-216	+	+			-			M>Q				
FH-217	+	+		+				$M > Q \ge G$				
FH-218	+	+			+			M>Q≥H				
FH-219	+	+						M>Q				
FH-220	+	+						M>Q				
FH-221	+	+						M>Q				
FH-222	+	+						M>Q				
FH-223	+	+						M>Q				
FH-224			+	+		+		A>G>P				

Note: These are x-ray diffraction results of bulk water-insoluble residues; no clay mineral fractions are reported here.

FH-228	+	+					+	+		+
FH-229	+	Traces	+		Traces		+	+		, -
FH-230	+	+					+	+		, -
FH-231			+				•	•		ч.
FH-232			+							
FH-233			+						<u>т</u>	
FH-234			+						T	
FH-235	+	+	+				+	+	Т	.1.
FH-236			+				I	1	I	Т
FH-237			+	+	Traces	+-				
FH-238	+	+				•	+	+		т.
FH-239	+	+					, +	+		
FH-240	+	Traces	+	Traces			r			т
FH-241			+							
FH-242	+	Traces	Traces				+	+		Т
FH-243	+	Traces	+	Traces			+	+		+ +
FH-244	+	Traces	Traces				+	, +		+ -
FH-245	+	+	Traces				+	+		+ +
FH-246	+	+	Traces				+	+		+ +
FH-247	Traces	Traces	+				I.	'		Ŧ
FH-248	+	Traces	+				+	+		+

Sample	San De	nple pth	Sample Weight	Weight of EDTA- Insoluble Residue	Weight % (water-insoluble residue)	Weight %
110.	From	<u>то</u>	(6)	(8)	1001440)	(()))))))))))))))))))))))))))))))))))))
	FIOII	10	_			
201	2	2.5	1.025	0.6	58.54	0.21
202	4	4.7	4	0.02	0.5	0.17
203	8.3	9	3	0.97	32.33	0.65
204	10.8	11.3	3	1.55	51.67	0.48
205	13	13.55	Insufficient mate	erial		
206	15.55	15.85	4	2.11	52.75	2.05
207	16.9	17.45	0.53	0.27	50.94	0.1
209	19.65	20.05	0.52	0.19	36.54	0.06
210	21.5	22	4	0.6	15	0.28
211	23	23.5	2.1	1.03	49.05	0.25
212	25.1	25.7	4	2.1	52.5	0.425
213	26	26.5	3.65	1.935	53.01	0.34
214	28.25	28.85	0.9	0.055	6.11	0.0125
215	30.5	31.05	Insufficient mate	erial		
216	31	38 *	4	2.43	60.75	0.89
217	39.25	39.85	4	1.5	37.5	0.94
218	41	41.5	4	1.95	48.75	0.86
219	42.5	43	4	2.27	56.75	1.77
220	44	44.5	4	2.61	65.25	0.81
220	45.3	45.85	2.3	1.48	64.35	0.35
221	47	47.5	4	2.3	57.5	0.45
222	49.05	49 55	1.075	0.46	42.79	0.13
224	4	4.5**	4	0.06	1.5	0.92
*Core loss z **From RM-	one -4					
000	0.4	0.9	0.7	0.255	36.4	0.05
220	0.4	28	0.7	0.15	31.6	0.00
229	2.3	2.0 6.75	2 075	0.10	16	0.025
230	4.75	0.10	2.075	0.555	40	0.11
231	7.1	9.15	2	0.000	7	0.03
232	1.10	0.10	0.00	0.21	1	0.020
233	8.15	9.1	0.30	0.09	23.7	0.01
234	10.5	10.9	ວ ຄະ	0.005	0.17	0.002
235	12.15	12.6	2.5	0.007	20	0.001
236	14	14.40	3	0.007	0.23	0.2
237	16.1	16.6	3	0.16	0.3	0.1
238	29	29.5	2.9	1.29	44.5	2.81
239	34.05	34,5	3	1.315	43.8	1.36
240	36.9	37.6	3	0.07	2.3	0.6
241	38.2	38.7	2.8	0.02	0.71	0.002
242	43.05	43.55	3	2.03	67.7	1.1
243	47.2	47.7	3	0.86	28.7	0.215
244	49.65	50	3	1.76	58.7	5.68
245	51.3	52.1	2.7	0.645	23.9	2.22
246*	20.05	20.45	3	1.09	36.3	0.29
247*	23.45	23.95	3	0.08	2.67	0.36
248*	25.5	26	0.36	0.09	25	0.0125
249*	45	45.65	3	1.37	45.7	0.25
*From core	RM-7					

## Table 3. Weight Percents of EDTA-Insoluble Residues

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Sample					Basal Planar	Spacings
No.	Quartz	Anhydrite	Magnesite	Polyhalite	14 Å	10 Å
DIL 001						
FH-201	+					
FH-202	Insufficient	material				
FH-203	+					+
FH-204	+					
FH-205	Insufficient	material				
FH-206	+					
FH-207	Insufficient	material				
FH-208	+					
FH-209	Insufficient	material				
FH-210	+	Traces		Traces		
FH-211	+				+	+
FH-212	+	Traces				
FH-213	+		Traces		+	+
FH-214	+					
FH-215	Insufficient	material				
FH-216	+					
FH-217	+					+
FH-218	+					
FH-219	+				+	+
FH-220	+					
FH-221	+				+	+
FH-222	+				+	+
FH-223	+				+	+
FH-224	Insufficient	material			,	

**Table 4. X-Ray Diffraction Results from EDTA-Insoluble Residues** 

The components of the non-NaCl mineral residues were identified by x-ray diffraction. All x-ray analyses for this study were run by using a Phillips diffractometer with Cu K $\alpha$  radiation at an operating voltage of 40 keV and 25 mA. The x-ray diffraction results obtained are at best only semiquantitative. Because of time constraints on the analyst and because of the limited amount of available sample material, it was decided to forego any attempts at more quantitative x-ray diffraction analysis at this time. Where indicated in Table 2, relative amounts of mineral species are reported on the bases of the analyst's inspection of the diffractograms and on his knowledge of the relative diffraction efficiencies of the different minerals present. Some of the water- and EDTA-insoluble residue samples were observed to contain significant quantities of minute, well-developed, euhedral quartz crystals. A few of these crystals were hand-picked from selected water-insoluble residue samples by means of a binocular microscope and surgical tweezers. These crystals were then mounted on carbon bases and examined with a scanning electron microscope (SEM), as seen in Figure 2 (a-c). In addition to this quartz, very small ( $\ll 10 \ \mu$ m) euhedral crystals of magnesite and Mg-silicate phases were also observed with the SEM and identified as to elemental content by EDAX.





(b)





(d)

### (c)

Figure 2. Scanning electron micrographs of authigenic quartz crystals found in water-insoluble residues from argillaceous halite ((a) - (c)) and thin section photomicrograph of authigenic quartz crystals in recrystallized halite (d).

The routine analytical procedure used in this study to determine weight percents of non-NaCl minerals, as described above, has two limitations: (1) it is time-consuming and labor-intensive; (2) this process yields weight percents of non-NaCl minerals as an average for a bulk sample; no information is obtained regarding the spatial distribution of these minerals within that sample. Therefore, two possible alternative techniques, gamma-beam densitometry and x-radiography, were selected on the basis of the following criteria:

- <u>Rapid data acquisition</u>: relative to the conventional analytical method described above, which requires weeks or months to process up to several meters of core, gamma-beam densitometry and x-radiography can analyze the same amount of core in hours or minutes, respectively.
- <u>Continuous data collection</u>: the information output is obtained from a continuous scan of the core, yielding far more data than integrating bulk mineralogy over a 500-g sample, while simultaneously showing the desired spatial distribution of non-NaCl components.
- <u>Sample handling and preparation</u>: whole core was used for x-radiography, and only surface milling in a lathe was required for the gamma beam densitometer. Because both of these techniques are nondestructive, the information thus obtained may then be supplemented by thin sections or other mineralogical analyses.

A "trial" halite core (actually, a scrap core of unspecified location within the facility horizon) was selected on the basis of the apparent presence of large and variable amounts of clays and other mineral impurities as ascertained by visual inspection. This core was first subjected to scanning by a gamma-beam attenuation system accountable to Sandia's Department 1510. For further details of this procedure, see Reda and Hadley (1983). This technique uses a Cs<sup>137</sup> source to produce a photon beam. The attenuation coefficient,  $\mu$ , of this beam through the core material is compared to the measured value for a reference sample of pure salt ( $\mu_{ref}$ ). The results of the gamma-beam densitometer scan are shown in Figure 3(a).

The same piece of core was also subjected to xradiography and the film then read by microdensitometry; results are shown in Figure 3(b). A large fracture in the core, which showed up on the x-radiograph as a dark line, is offset from the position of the same fracture on the gamma-beam densitometer plot (Figures 3(a) and 3(b)) as a result of the slight rotation of the core from its position in the x-radiographs.

Finally, the core was sliced into  $\sim 1$ -in. pieces and dissolved in distilled water according to the routine procedure. The total dry weight percent of mineral residue is plotted in Figure 3(b) and represents an average value over every 1-in. length of core.

Gamma-beam densitometry appears to be the least suitable of these techniques for detecting mineral impurities in halite core. No obvious correlation exists between the data in Figure 3(a) and in Figure 3(c). The x-radiograph results, however, may be fit by a smooth curve that reasonably approximates the conventional analytical data (Figures 3(b) and 3(c), respectively) exclusive of such features as peaks corresponding to obvious fractures.



Figure 3. Comparison of three different methods of detection of non-NaCl mineralogy by using surplus core from the WIPP facility horizon: (a) gamma-beam densitometry, (b) x-radiography, and (c) conventional dissolution technique.

## Discussion

The primary purpose of this study was to examine closely the non-NaCl constituents of halite in the lower Salado Formation as a means of characterizing the lithology of the WIPP host rock in terms of abundance, distribution, and identification of trace mineral components.

By way of addressing the first of these, the data presented in Tables 1 and 3 are consistent with similar analyses performed for the SPDV program. For the samples analyzed in the study reported here, weight percents of water-insoluble residues ranged from 0.05 to 87.08 wt%. For the EDTA-insoluble residues, weight percents ranged from 0.001 to 5.68 wt% of the total weights. The mean values for the water- and EDTA-insoluble residues are 5.56 wt% and 0.64 wt%, respectively. The occasional excursions from the average numbers were attributed to unusually clay-rich layers or seams and/or to distinct anhydrite units. Linear statistics for these data are found in Table 5.

One of the most significant conclusions gained from the SPDV work is that the measurements of the non-NaCl mineralogy, as weight percents, are smaller by at least an order of magnitude than previous estimates made by visual observations. Many of the WIPP lithologic logs that predate the SPDV mineralogy study report the presence of polyhalite and clays in amounts ranging from 30 to 50 wt% or more. The SPDV results show that only a very small amount of non-NaCl material can impart such color and opacity to the salt so as to create the visual impression of a much larger quantity. For this reason, it was decided to put portions of the samples used in this study together with the data from the water- and EDTAinsoluble residue analyses into a reference collection in order to better "calibrate" visual estimates and therefore enable visual core descriptions to be made more quantitatively.

It is much more difficult, however, to describe and quantify the distribution of non-NaCl minerals dispersed in a halite core. For example, consider a halite sample weighing 500 g and containing 5% clay by weight. The analytical techniques used in this study (the results of which are shown in Tables 1 and 3) do not distinguish the distribution of this amount of clay in this particular sample interval. It could be present as fine particles evenly disseminated throughout the halite; it may occur as intergranular in-fillings or as large blebs; it may be present as a single discrete layer or seam in otherwise "clean" halite; or it may be some combination of all of these. While clay seams or layers are, for the most part, visually conspicuous enough to be noted as such in the lithologic logs, the distribution of trace minerals in most halitic core is obscure enough

that an accurate characterization would best be obtained by detailed examination of many thin sections. This approach is out of the question, given the volume of core required for this study and time constraints on the analyst. An attempt was therefore made to develop a technique that would be both rapid and nondestructive, yielding an accurate determination of the distribution of trace minerals and at the same time preserving the core intact for future use. Variations in gamma-beam densitometry as a function of mineralogy are slight. Measurements of film density of the xradiographs appear to be significantly more sensitive and to parallel the results of the mineralogical determinations made by using the conventional technique described in this report.

As shown in Table 2, the water-insoluble mineralogy of these samples consists of quartz, magnesite, anhydrite, gypsum, polyhalite, alkali feldspar, and clays. Because of the complexity of the analytical process required to distinguish clay mineral species (separation of the clay fraction, heat treatment, exposure to ethylene glycol, etc), we report here only the basal planar spacings observed in the diffractograms of these samples. Major peaks belonging to clay minerals were observed at 14 Å, 10 Å, 9.3 Å, and 7 Å. Detailed clay mineralogical analyses on these and other WIPP samples are in progress; results will be presented in a later report. The EDTA-insoluble mineralogy (Table 4) consists entirely of quartz and clays except for a few samples showing traces of anhydrite, magnesite, and polyhalite, where EDTA digestion was obviously incomplete. Again, as described in the preceding paragraph, the clay minerals have not been identified as particular individual species, but only by the presence or absence of major peaks.

Most of the quartz (>90%), based on binocular microscope inspection of the samples in which it was found) appears to consist of well-developed, doubly terminated euhedral crystals. This morphology is highly suggestive of an authigenic origin. First observed in the SPDV samples, these quartz crystals appear to be associated exclusively with clay-bearing or "argillaceous" halite, as described in the lithologic logs. This association implies an origin of authigenic quartz through diagenetic alteration of the clay minerals; one possible mechanism is (from Siever, 1962):

montmorillonite  $\longrightarrow$  illite + SiO<sub>2</sub>

The authigenic morphologies (Figures 2(a) - (c)) together with the textural relationships as seen in thin section (Figure 2(d)) also suggest that clay diagenesis and quartz precipitation preceded halite recrystallization.

Water-Insoluble	Residues	EDTA-Insoluble Residues				
М	0.45000D + 02	М	0.42000D + 02			
Mean	0.55620D + 01	Mean	0.63524D + 00			
Std. Dev.	0.14567 D + 02	Std. Dev.	0.10344D + 01			
Variance	0.21218D + 03	Variance	0.10701D + 01			
Max. X	0.87080D + 02	Max. X	0.56600D + 01			
Min. X	0.50000D - 01	Min. X	0.10000D - 02			
Range	0.07030D + 02	Range	0.56790D + 01			
Sum. X	0.25029D + 03	Sum. X	0.26680D + 02			
Sum. X Square	0.10728D + 05	Sum. X Square	0.60821D + 02			
Correction Term	0.13921D + 04	Correction Term	0.16948D + 02			
Sum. Small X Square	0.93361D + 04	Sum. Small X Square	0.43873D + 02			
Std. Dev. (Mean)	0.21715D + 01	Std. Dev. (Mean)	0.15962D + 00			
Variance (Mean)	0.47152D + 01	Variance (Mean)	0.25478D - 01			
Normality Test		Normality Test				
Mean Dev.	0.75722D + 01	Mean Dev.	0.64367D + 00			
Third Moment	0.12952D + 05	Third Moment	0.34221D + 01			
Fourth Moment	0.10072D + 07	Fourth Moment	0.16297D + 02			
Α	0.52571D + 00	Α	0.62978D + 00			
Sort B Sub1	0.43342D + 01	Sort B Sub1	0.32053D + 01			
B Sub2	0.23399D + 02	B Sub2	0.14935D + 02			
D-Test Statistic	0.98294D + 01	<b>D-Test Statistic</b>	0.49859D + 01			

### Table 5. Statistics on Weight-Percent Data

A simple mass balance calculation (Stein, 1984) clearly indicates that the volume of authigenic quartz present in these halite samples is, on the average, greater by almost two orders of magnitude than that which could have been produced by simple evaporation of seawater alone. Further studies of authigenic silicate formation and clay diagenesis are in progress. In addition to the quartz, other authigenic minerals may be present; authigenic feldspars and pyrite and possibly a zeolite have been identified by the author in thin-section, x-ray diffraction, and SEM work on some of the samples in this study. Because these results are preliminary, further details are not included here but will be reported on at a later date.

Figures 4 and 5 illustrate the relationship between weight percents of water- and EDTA-insoluble residues and lithology, as seen in the cores from which these samples were taken. No obvious correlation exists between the non-NaCl mineralogy and distance from the repository in either the up or down direction. However, these figures indicate that samples from anhydrite-bearing units contain the largest amounts of insoluble residues. Perhaps not surprisingly, samples from the visually designated "clean" halites contain the least. Moreover, in Figures 4 and 5, two lines are used for each sample interval to represent weight percents of water- and EDTA-insoluble residues. The larger the difference between these two lines in any one sample, the greater the relative abundance of the EDTA-soluble component (mainly the sulfate minerals polyhalite and anhydrite). This is clearly seen in samples such as FH-231, FH-236, and FH-240, taken from distinct anhydrite units in core RM-1 at depths of 7.1 to 7.3 ft, 14 to 14.45 ft, and 36.9 to 37.6 ft, respectively. Conversely, where the difference between each pair of lines is small (for example, FH-239, FH-242, or FH-217 through FH-224), the amounts of water- and EDTA-insoluble residues are nearly equal and thus composed primarily of insoluble silicate residues (quartz and clays) as is typically seen in the zones of argillaceous halite.

It is beyond the scope of this report to discuss the effects of trace mineralogy in WIPP salt on material properties or on creep behavior in rock salt. It is intended that the data reported here will be used to further modify the structural computations as discussed in Krieg et al (1980). This report is intended for use as a supplement to Krieg et al (1982) and Krieg (1984), by providing a more complete reference stratigraphy that includes detailed analyses of the halite mineralogy.

#### CORE: RM-1 DIRECTION OF DRILLING: VERTICAL UP LOCATION: TEST ROOM 4 (ROOF)



\* SAMPLES ARE FROM RM-7

Figure 4. Core RM-1, showing lithologies and amounts of water- and EDTA-insoluble residues. (RM-7 samples are from another core, taken in the up direction, in close proximity to RM-1 (see Figure 1).)

#### CORE: RM-3 DIRECTION OF DRILLING: VERTICAL DOWN LOCATION: TEST ROOM 4 (FLOOR)



Figure 5. Core RM-3, showing lithologies and amounts of water- and EDTA-insoluble residues.

## Conclusions

In summary, we report here the quantities, distribution, and mineralogical species of the non-NaCl components of halite and anydrites in the immediate vicinity (e.g., within 100 vertical feet) of the WIPP facility horizon. These components, as determined by x-ray diffraction, consist of quartz, anhydrite, gyp-sum, magnesite, polyhalite, and clays, with traces of alkali feldspar and possible zeolites. The quartz is primarily authigenic and is probably derived from alteration of the clay minerals. Textural relations indicate that this alteration and precipitation of the authigentic quartz either preceded or perhaps occurred simultaneously with halite recrystallization.

The results of the analyses presented herein confirm previous conclusions regarding discrepancies between visual estimates of mineral impurities in WIPP core and accurate measurements, as obtained by the precedure defined during the SPDV investigation. It has been shown that visual estimates of trace minerals may be too high by as much as an order of magnitude. WIPP salt contains, on the average, <5 wt% mineral impurities, except in areas of well-defined anhydrite or clay bands or layers. These impurities occur as either finely divided particles dispersed throughout the halite, as intergranular coatings, or as discrete blebs, lenses, laminae, or seams.

The analytical procedure used to produce the results reported here is time-consuming and provides no detailed information as to the distribution of non-NaCl minerals. Alternative methods of sample analysis are being investigated. It appears that x-radiography of halite core is rapid, nondestructive, and accurately reflects mineral impurities in the salt. More work is necessary before this technique can be considered a quantitative tool for this type of examination. However, the data obtained from the samples analyzed in this study will be used as points for calibration.

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# Appendix A

# **Lithologic Logs**

Core: RM-1

Direction of drilling: Vertical up Location: Test Room 4 (roof) Mine Coordinates: N1364 W631 approx. Depth of Borehole: 52.1 ft Logged by: J. E. Gallerani

Elev. above Ceiling (ft)		_	Elev. ab Ceiling	ove (ft)			
From	То	_	From	То	-		
0	7.05	Halite: clear to medium reddish- orange to light gray, coarsely crystalline, hard. Fine to medium	18.8	19.3	Halite: clear, coarsely crystal- line, hard. Scattered polyhalite blebs ( $<1\%$ ).		
		( $<1/2\%$ ) polyhalite. Trace ( $<1/2\%$ ) gray clay.	19.3	19.3	19.3	24.05	Argillaceous halite: clear to me- dium grayish-brown, fine to coarsely crystalline, medium
7.05	7.2	Anhydrite (anhydrite "b"): very light gray to medium gray, mi- crocrystalline; hard. Some patches of halite within. Trace gray clay at 7.05 ft. Irregular upper surface at 7.2 ft.			hard to hard. Contains gray and brown clay, generally <2%. Trace medium reddish-orange color. Gray anhydrite layer at 23.85 to 24 ft, thin gray lamina immediately beneath.		
7.2	13.9	Halite: clear to medium reddish- orange, medium to coarsely crys- talline, hard. White anhydrite stringers and laminae found within core. Beginning at 12.3 ft trace gray clay ( $<1\%$ ). Core me-	24.05	32.4	Halite: clear, coarsely crystal- line, hard. Several irregular white patches at 25 to 25.7 ft. Clear to medium brown from 28.3 to 29.5 ft; brown clay $(1\%$ to 2%) breaks at 29 ft and 29.25 ft.		
		dium grading to fine-medium crystalline toward anhydrite.	32.4	36.65	Argillaceous halite: clear to me- dium reddish-brown and gravish-		
13.9	14.4	Anhydrite: very light gray to medium gray, microcrystalline, hard. Scattered halite. Trace gray clay at 13.9 ft. Upper con- tact at 14.4 ft is irregular, tightly			brown, coarsely crystalline, me- dium hard to hard. Dispersed argillaceous, predominantly brown. Avg. $<1\%$ to $3\%$ , locally >5%.		
14.44	18.8	weided. Polyhalitic halite: clear to me- dium reddish-brown-orange, coarsely crystalline, moderately hard to hard. Contains blebs and patches of polyhalite (1% to 5%), locally >5%.	36.65	37.6	Anhydrite: very light gray to me- dium gray, microcrystalline hard anhydrite with irregular zone of halite at $\sim$ 37.15 to 37.35 ft. Up- per contact irregular, tightly welded. Gray clay at 36.7 ft.		

Elev. ab Ceiling (	ove ft)		Depth b floor (ft	elow )	_
From	То		From	То	
37.6	41.8	Halite: clear to medium reddish- orange, coarsely crystalline, hard. Contains $<1\%$ polyhalite (dispersed reddish-orange col- or). Core contains highest % po- lyhalite 37.6 to 38.2 ft, scattered white (magnesite) stringers.	2.7	6.85	Anhydrite (MB-139): mixture of medium reddish-orange polyha- lite, polyhalitic halite and anhy- drite from 2.7 to 4.2 ft. Coarsely crystalline polyhalitic halite mixed with polyhalitic and mi- crocrystalline anhydrite, approx.
41.8	43.9	Argillaceous halite: clear to me- dium brown, coarsely crystalline with some fine, medium hard to hard. Predominantly brown ar- gillaceous $1\%$ to $3\%$ , locally up to $5\%$ .			40% to 60%. Grades to higher % anhydrite 4.2 to 5.6 ft. From 5.6 to 6.85 ft is anhydrite. Very light gray to white microcrystalline, laminated anhydrite mixed with 10% to 30% polyhalite and po- lyhalitic halite, hard. Core bro-
43.9	48	Halite: clear to medium brown, fine to coarsely crystalline, me-			ken in small pieces at 4.4 to 4.6 ft. Trace gray clay at 6.85 ft.
48	52.1	dium hard. Trace (<1%) argil- laceous and polyhalite. Argillaceous halite: clear to me- dium brown, medium with some fine to coarsely crystalline, me-	6.85	10.3	Halite: clear to grayish to me- dium reddish-orange, medium to coarsely crystalline, hard. Gray clay $<1\%$ , scattered clay breaks. Trace ( $<1\%$ ) polyhalite.
		dium hard to hard. Brown argil- laceous (1% to 5%, avg. 1% to 2%. Clay break at 52.1 ft. 5% to $>10\%$ brown clay at 49.6 to 50.2 ft. Trace (<1%) polyhalite.	10.3	14.1	Polyhalitic halite: clear to me- dium reddish-orange, coarsely crystalline, hard. Polyhalite <1% to $3%$ , locally $>3%$ . Scat- tered gray clay breaks to 11.3 ft.
Core: RI Direction Location Mine Co Depth o Logged	M-4 n of drillin n: Test Roc oordinates: f Borehole by: J. E. G	g: Vertical down om 4 (floor) N1362.6 W627.5 : 49.5 ft callerani	14.1	16.6	Halite: clear to light-gray to me- dium reddish-orange, medium to coarsely crystalline, hard. Trace ( $<1\%$ ) gray clay. Parting at 14.3 ft. Scattered clay breaks, <1% polyhalitic.
Depth b floor (ft)	elow )		16.6	17.7	Halite: clear with trace of me- dium reddish-orange, coarsely crystalline, hard. Trace $(<1\%)$ polyhalite.
From 0	2.7	2.7 Halite: clear to medium brown, medium to coarsely crystalline, medium hard to hard. Trace (<1%) polyhalite and gray clay.		19.9	Halite: clear to medium reddish- orange-brown, coarsely crystal- line, hard. Generally <1% poly- halite with local zones to 3%. From 18.4 to 19.6 ft, up to 5% polyhalite.
			19.9	26.5	Halite: clear mottled with me- dium reddish-orange and light gray, medium to coarsely crys- talline, hard. Gray clay $<1\%$ . <1% polyhalite with local zones to 2\%.

Depth I floor (ft	below t)		Depth h floor (ft	pelow ;)	
From	То	_	From	То	
26.5	32.05	Halite: clear to medium reddish- orange-brown, coarsely crystal- line, hard, polyhalitic. Up to 5% polyhalite from $\sim 26.5$ to 27.5 ft decreasing to avg $< 1\%$ polyha- lite below 29.5 ft. Mostly anhy- drite at 31.45 to 31.65 ft. Clear halite 30.65 to 31.45 ft.	3	7.2	Anhydrite (MB-139): mixture of 50/50% anhydrite and halite from 3 to 3.9 ft. Upper contact is irregular at 20° to 25° dip. Core hard. From 3.9 to 7.2 ft, very light to medium gray, mi- crocrystalline, hard. Contains halite and polyhalitic halite
32.4	39.5	Halite: clear to medium reddish- orange and medium-dark gray, medium to coarsely gratelling	79	10.45	within. Core loss most probably in this zone.
		hard. Trace $(<1\%)$ gray clay and trace $(<1\%)$ scattered poly- halite blebs. Core broken up in	1.2	10.40	dium to coarsely crystalline, me- dium hard to hard. Gray argilla- ceous $<1\%$ .
		to 1 in.) at 33.4 to 33.75 ft.	10.45	14.8	Polyhalitic halite: clear to me-
39.5	43	Halite: clear to medium brown, medium to coarsely crystalline, medium hard to hard. Contains <1% to $3%$ predominantly brown argillaceous, clay breaks			crystalline, hard. Trace $<1/2\%$ gray clay, $<1\%$ to $3\%$ polyha- lite. $3/4$ in. irregular polyhalite layer at 14.6 ft. Dark gray clay seam up to $3/4$ in. at $\sim14.8$ ft.
43	50.2	scattered within. Trace $(<1/2\%)$ polyhalite dispersed throughout. <i>Halite</i> : clear mottled with traces (<1%) of gray clay and medium	14.8	16.5	Halite: clear to light gray, fine to coarsely crystalline, medium hard to hard. Trace ( $<1\%$ ) poly- halite and gray clay
		reddish-orange polyhalite blebs. Medium to coarsely crystalline, hard. Up to 3% gray argilla-	16.5	17.9	Halite: clear with some medium reddish-orange, coarsely crys- talline, hard. <1% polyhalite.
		ceous at 48.55 to 49.1 ft.	17.9	19.5	Polyhalitic halite: clear to me-
Core: R Directio Location Mine Co	M-3 on of drillin n: Test Ro oordinates	ng: Vertical down om 4 (floor) : N1361 W631 approx.			to 5% polyhalite, trace ( $<1/2\%$ ) gray clay.
Depth o Logged	of Borehole by: J. E. C	e: 51.05 ft Gallerani	19.5	32	Halite: clear, mottled with me- dium reddish-orange, fine to
Depth b floor (ft	)				coarsely crystalline, medium hard to hard. Trace ( $<1\%$ to 2%) gray clay and $<1%$ to $2%$
From	То	-			polyhalite with local zones
0	0.9	Halite: clear, coarsely crystal- line, hard. Trace $(<1/2\%)$ poly- halite blebs and gray clay.			within core. Polyhalitic halite at 25.2 to 27.8 ft. Avg 2% to 4% polyhalite. No clay at 27.8 to
0.9	3	Polyhalitic halite: clear to me- dium reddish-orange, coarsely crystalline hard. $<1\%$ polyha- lite increasing to $\sim3\%$ to $4\%$ toward lower part of section.			30.1 ft. >10% polyhalite at 31.55 to 31.75 ft.

### Depth below

floor (ft	.)	_
From	То	-
32.0	32.1	Anhydrite: very light gray to light-gray anhydrite mixed with some halite. Microcrystalline, hard. Trace gray clay at 32.1 ft. Core loss from 30.75 to 35.75 ft. Depth of anhydrite based on boring RM-2.
32.1	38.3	Halite: clear with some medium reddish-orange and light gray. Medium to coarsely crystalline, medium hard to hard. Trace (<1%) gray clay and polyhalite. Core loss in this section.
38.3	51.05	Halite: clear to light gray mot- tled with medium reddish- orange polyhalite. Coarsely crystalline, hard. Avg $<1\%$ to 4% gray clay. Clay breaks irreg- ular and low angle, commonly scattered in core to $\sim44$ ft. <1% clay from 44 ft to bottom of hole. $<1\%$ polyhalite average with 1% to 2% polyhalite 38.3 to 40.15 ft.

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# Appendix B

# **Description of Core Samples Used In This Study**

Sample No.	Hole No.	Depth Interval (ft)		Sample Description
		From	То	_
FH-201	<b>RM-</b> 3	2	2.5	Clear with trace polyhalite
FH-202	<b>RM-</b> 3	4	4.7	Polyhalitic halite
FH-203	<b>RM</b> -3	8.3	9	8.3 to 8.5 ft – mixed clay/polyhalite
				8.5 to 9 ft – clear
FH-204	RM-3	10.8	11.3	Clear, trace polyhalite (5 pieces)
FH-205	<b>RM-</b> 3	13	13.55	Polyhalitic (4 pieces)
FH-206	<b>RM-</b> 3	15.55	15.85	Mixed clay/polyhalite
FH-207	<b>RM-</b> 3	16.9	17.45	Clear, coarsely crystalline, trace polyhalite
FH-208	<b>RM-</b> 3	18.4	19.1	Polyhalitic (3 pieces)
FH-209	<b>RM-</b> 3	19.65	20.05	Polyhalitic (3 pieces)
FH-210	<b>RM-</b> 3	21.5	22	Clear mottled with polyhalite
FH-211	<b>RM-</b> 3	23	23.5	Same as above
FH-212	<b>RM</b> -3	25.1	25.7	Clear to mod. reddish-orange, trace polyhalite
FH-213	<b>RM-</b> 3	26	26.5	Clear to mod. reddish-orange, trace polyhalite
FH-214	<b>RM-</b> 3	28.25	28.85	Same as above with some horizontal fractures
FH-215	<b>RM</b> -3	30.5	31.05	Clear, coarsely crystalline, very little polyhalite
FH-216	<b>RM-</b> 3	31	38	Clear, trace gray clay, polyhalite
		(Core lo	oss zone)	
FH-217	<b>RM</b> -3	39.25	39.85	Grayish-brown mixed clay/polyhalite
FH-218	<b>RM</b> -3	41	41.5	Mixed clay/polyhalite, coarsely crystalline, very little
FH-219	<b>RM</b> -3	42.5	43	Mixed clay/polyhalite, medium coarsely crystalline, very
FH-220	<b>RM</b> _3	14	44 5	Mixed clay/polyhalite coarsely crystalline
FH-221	RM-3	45.3	45.85	Clear coarsely crystalline
FH-222	RM-3	40.0	47.5	Clear trace gray clay
FH-223	RM-3	49.05	49.55	Clear trace gray clay/nolyhalite
FH-224	RM-3	4	4.5	Medium reddish-orange polyhalite/anhydrite
Core Sample	s for Geomecl	nanical Testi	ng	
FH-225	RM-4	40.75	42.5	Mixed clay/polyhalite
FH-226	RM-4	42.5	44	Mixed clay/polyhalite
FH-227	<b>RM-5</b>	39.05	41.6	Mixed clay/polyhalite
FH-228	RM-1	0.4	0.9	Halite, clear to medium reddish-brown to light gray, coarsely, some fine to medium crystalline. Trace polyha- lite and clay
FH-229	<b>RM-1</b>	2.3	2.8	Same as above
FH-230	<b>RM-1</b>	4.75	6.75	Same as above
FH-231	RM-1	7.1	7.3	Anhydrite "b" with halite 7.2 to 7.3 ft

Split Core Samples for Geochemistry Analysis

Sample No.	Hole No.	Depth Interval (ft)		Sample Description
		From	То	_
FH-232	RM-1	7.75	8.15	Halite, clear to medium reddish-orange, medium to coarsely crystalline, white anhydrite stringers
FH-233	<b>RM-1</b>	8.15	9.1	Same as above
FH-234	<b>RM-1</b>	10.5	10.9	Same as above
FH-235	<b>RM-1</b>	12.15	12.6	Same with trace gray clay
FH-236	<b>RM-1</b>	14	14.45	Anhydrite "a"
FH-237	<b>RM-1</b>	16.1	16.6	Polyhalite halite
FH-238	<b>RM-1</b>	29	29.5	Halite with brown clay, coarsely crystalline
FH-239	<b>RM-1</b>	34.05	34.5	Argillaceous halite
FH-240	<b>RM-1</b>	36.9	37.6	Anhydrite with some halite
FH-241	<b>RM-1</b>	38.2	38.7	Halite with trace polyhalite, magnesite stringers
FH-242	<b>RM-1</b>	43.05	43.55	Argillaceous halite
FH-243	<b>RM-1</b>	47.2	47.7	Halite, trace polyhalite and clay
FH-244	<b>RM-1</b>	49.65	50	Argillaceous halite
FH-245	<b>RM</b> -1	51.3	52.1	Argillaceous halite, break at 52.1 ft
FH-246	<b>RM-7</b>	20.05	20.45	Halite, trace of clay and polyhalite
FH-247	<b>RM-7</b>	23.45	23.95	Halite with gray anhydrite, trace clay
FH-248	<b>RM-7</b>	25.5	26	Halite – clear, coarsely crystalline
FH-249	<b>RM-7</b>	45	45.65	Halite, trace brown clay, coarsely crystalline

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