

# APPENDIX III

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X-Ray Diffraction

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## X-RAY DIFFRACTION DATA

The interpreted x-ray diffraction (XRD) data for samples distributed throughout the Rustler from WIPP 19 and H-12 are presented in tables attached to this section. The data are only generally semi-quantitative and are limited by procedures (SOP for XRD at end of this Appendix) to major evaporite minerals. Clays have not been included in this investigation.

Two features of this XRD study are somewhat unusual. Quartz occurs more frequently and more abundantly than expected in purer chemical sediments, and dolomite and high magnesium calcite are more ubiquitous than expected. The carbonate phases are apparent through petrography, and XRD confirms the Mg content. Further petrographic study might be necessary to evaluate the significance of carbonate where a dolomite as well as probable high magnesium calcite occur.

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X-RAY DIFFRACTION - H-12

NO*	GYPSUM	ANHYDRITE	QUARTZ	HALITE	DOLOMITE	CALCITE	POLYHALITE	OTHER
1				XX				
5	x	?tr	X	tr	x	? Mg calcite		? feldspar
8			X	x	x			? feldspar
10			X	X	x	? Mg calcite		? feldspar
12			X		x	? Mg calcite		
13			X	X	x	? Mg calcite		? feldspar
15		x	X	X	x	x Mg calcite		? feldspar
19		x	X	X				
23			? tr	X				
25		x	tr	tr				
28			x	X				
31		x		X				
33		XX						
36			X	x				
39			X	x	X	x		
41	X				X			
42	X			x	x?			
46		XX						
50		X		x				prob. elec. err
51		X		X				
52		x		X				? magnesite t
54		tr		X				
58		tr	x	X				
59		tr	x	X				
61		tr	x	X				
62		X		?tr				X magnesite
63		X				x?		?tr
64		XX						?tr
65		XX		?				
67		X				x		?
68		X		x				
71	X		x	tr?		x		?
73	X		tr	tr?		x	x?	
74	tr	X						
76			x			X		
79		x	X	x		x		
80			X	X		x		
81		X		tr?				
82		X				x		

KEY: XX - sole or overwhelming mineral  
 X - major  
 x - minor  
 tr - trace  
 ? - questionable  
 Mg calcite - magnesium-rich calcite, generally >20% Mg  
 \*Sample Number



X-RAY DIFFRACTION - WIPP 19

NO*	GYPSUM	ANHYDRITE	QUARTZ	HALITE	DOLOMITE	CALCITE	POLYHALITE	OTHER
1		X	X	X				
2		tr	X	X		? Mg calcite		unidentified
4	x		X		x	? Mg calcite		unidentified
5		tr	X	x	x	?		unidentified
6	tr	tr	X	x	x			
10			X	X	x			
11		x	X	X				
12		X		X				
14		x	x	X				
20	X		x					
23				?	X			
24			x	x?	X			
25			tr		X			
38	X	tr				x		
39	X					x		
40	X	X				tr		
41		XX						
42		X				tr		
43		X	x					
44	x	X	tr		x			
46	X	tr	X		X			
47	?	X			X			
49	X	x						unidentified
50	XX							
55			X		x	?tr		? Feldspars
57	x	tr	X	X		? Mg calcite		
60		XX						
62		XX		tr?	x			
63	x	X			?tr			

KEY: XX - sole or overwhelming mineral

X - major

x - minor

tr - trace

? - questionable

Mg calcite - magnesium-rich calcite, generally >20% Mg

unidentified - unidentified minor peaks

\*Sample Number



STANDARD OPERATING PROCEDURE

X-RAY DIFFRACTION ANALYSIS

RUSTLER FORMATION

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## GENERAL

This procedure is developed for use on samples from the Waste Isolation Pilot Plant (WIPP) project to determine mineralogy and qualitative to semi-quantitative mineral proportions from samples from the Rustler Formation. The techniques presented here are collectively based on experience of the authors and well-known sources such as Mueller (1967) and more recent literature (e.g. Fang and Zevin, 1985).

## OBJECTIVES

There are two principal objectives for the X-ray diffraction work on samples from the Rustler Formation:

- a) provide a basic comparative mineralogy for the Rustler based on regular sampling procedure from available core, and
- b) provide specialized mineral identification (e.g. clays or rare mineral phases) for further interpretive work.

The first objective is met through a sampling program from two boreholes (WIPP 19 and H-12) which provide a general sampling from both near the site center and in an area near the depocenter for parts of the Rustler. Samples were collected at regular spacings to provide a general representation of the mineralogy of the formation. The second objective is met through some initial specialized sampling of minerals and features of particular interest and through separation and/or concentration of mineral phases of interest from these special samples or from regularly spaced samples.

## METHODS



### Sample Preparation

A sample of the core will be selected for XRD analysis; general samples will usually be about 20-100 gms in weight while specialized samples may be as small as an individual crystal. The sample will be visually selected to be representative of the core specimen or to be a sample consistent with the analytical objective.

The sample will be individually stored in a plastic canister labeled by the sample identifier. The sample will be crushed in a mortar and a smaller amount, as a subsample, will be ground in an agate mortar with an agate pestle to obtain powder of the order of 10 $\mu$  in maximum diameter. A small amount of

Si metal or powdered fluorite ( $\text{CaF}_2$ ) will be added to provide an internal standard peak. General mineralogy samples will be approximately unoriented samples obtained by sprinkling powder onto a glass slide with vaseline on the surface.

Specialized samples may be prepared differently. Clay samples may be obtained after concentration of the clay fraction through dissolution of water and/or acid soluble portions (Bodine and Fernald, 1973; Bodine, 1978). Clay identification will be made on the basis of three general tests (Carroll, 1970; Millot, 1970):

- a) oriented sample
- b) oriented sample treated with ethylene glycol
- c) oriented sample heated to  $550^\circ\text{C}$ .

Oriented samples will be made in triplicate by sedimentation onto glass slide from water (with Calgon) suspension or by soaking the sample with acetone on the slide. Samples to be treated for ethylene glycol will be moistened and placed for 24+ hours in a desiccator with liquid ethylene glycol in the bottom. Heated samples will be kept at  $550^\circ\text{C}$  for a period of at least 1 hour, and will be X-rayed within four hours of heating. Samples will be kept in a desiccator with desiccant as appropriate during the interval.

Other special techniques of importance will be documented as carried out.

#### X-ray Diffraction

All XRD work for this project will be done with  $\text{Cu K}_\alpha$  radiation with a Ni filter to eliminate  $\text{Cu K}_\beta$ -radiation. The current and voltage will be set at 18 mA and 30 kV, respectively for all runs. Scaling and time factors will be established each day, and will be as uniform as is reasonably possible. These parameters will be recorded for each trace of any sample.

For general samples, a speed of  $2^\circ/\text{minute}$  for the goniometer and a chart speed of  $1''/\text{minute}$  will be used. Each sample will be examined through  $2-35^\circ 2\theta$ . The trace will be compared to an artificial pattern for common minerals. Peaks not in this set will be identified by standard methods of comparing peak intensities to patterns within ASTM card files. Peaks not identified by this methods will be noted for possible further work.

Special samples will be characterized through an appropriate range of  $2\theta$  and with modified chart and goniometer speeds as appropriate to the type of sample.

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### Interpretation

All identified minerals will be listed and note taken of unidentified or unidentifiable peaks. The relative intensities of mineral peaks will be used to assigned general abundance descriptors: major, minor, trace. As appropriate, certain mineral combinations, such as gypsum and anhydrite, may be assigned semi-quantitative proportions based on comparison of peak heights with artificial mixes. Records of these mixes will also be kept for checking.

### Safety

All operators of XRD equipment will meet Department of Geological Sciences training requirements. All operators are required to wear monitoring badges for exposure to radiation. These badges are checked periodically by the University.

### Quality Assurance

All operators are experienced in the use of XRD equipment for mineral analysis. Holt and Powers will provide supervision of the sample preparation and equipment operation periodically to ensure that record-keeping is appropriate.

All samples are marked with unique identifiers. Subsamples and all subsequent slides and traces will be immediately marked with identifying sample numbers and modifiers (e.g. G or H to denote glycolated or heated, respectively) as appropriate. Patterns will be individually marked with equipment operating parameters. Traces will be maintained in a file. An individual worksheet will be prepared with a summary of important information, including mineral identification for each sample (see attached).



#### REFERENCES CITED

- Bodine, M.W., Jr., 1978, Clay-mineral assemblages from drill core of Ochoan evaporites, Eddy County, New Mexico: Circ. 159, New Mexico Bureau of Mines and Mineral Resources, pp. 21-31.
- Bodine, M.W., Jr., and Fernald, T.H., 1973, EDTA dissolution of gypsum, anhydrite, and Ca-Mg carbonates: Jour. of Sedimentary Petrol., v. 43, pp. 1152-1156.
- Carroll, D., 1970, Clay Minerals: a guide to their X-ray identification: Spec. Paper 126, Geol. Soc. Am.
- Fang, J.H., and Zevin, L., 1985, Quantitative X-ray diffractometry of carbonate rocks: Jour. of Sedimentary Petrol., v. 55, pp. 611-613.
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