



CONTACT INFORMATION

Mining Records Curator
Arizona Geological Survey
1520 West Adams St.
Phoenix, AZ 85007
602-771-1601
<http://www.azgs.az.gov>
inquiries@azgs.az.gov

The following file is part of the

Arizona Department of Mines and Mineral Resources Mining Collection

ACCESS STATEMENT

These digitized collections are accessible for purposes of education and research. We have indicated what we know about copyright and rights of privacy, publicity, or trademark. Due to the nature of archival collections, we are not always able to identify this information. We are eager to hear from any rights owners, so that we may obtain accurate information. Upon request, we will remove material from public view while we address a rights issue.

CONSTRAINTS STATEMENT

The Arizona Geological Survey does not claim to control all rights for all materials in its collection. These rights include, but are not limited to: copyright, privacy rights, and cultural protection rights. The User hereby assumes all responsibility for obtaining any rights to use the material in excess of "fair use."

The Survey makes no intellectual property claims to the products created by individual authors in the manuscript collections, except when the author deeded those rights to the Survey or when those authors were employed by the State of Arizona and created intellectual products as a function of their official duties. The Survey does maintain property rights to the physical and digital representations of the works.

QUALITY STATEMENT

The Arizona Geological Survey is not responsible for the accuracy of the records, information, or opinions that may be contained in the files. The Survey collects, catalogs, and archives data on mineral properties regardless of its views of the veracity or accuracy of those data.

PRINTED: 12/17/2002

ARIZONA DEPARTMENT OF MINES AND MINERAL RESOURCES AZMILS DATA

PRIMARY NAME: CEDAR POCKETS

ALTERNATE NAMES:

C & W GROUP
RENO
BEAVER DAM MOUNTAINS GYPSUM
CEDAR WASH
WILLSIE GYPSUM DEPOSIT
PREMIER GYPSUM

MOHAVE COUNTY MILS NUMBER: 744

LOCATION: TOWNSHIP 41 N RANGE 14 W SECTION 3 QUARTER SW
LATITUDE: N 36DEG 58MIN 42SEC LONGITUDE: W 113DEG 48MIN 15SEC
TOPO MAP NAME: LITTLEFIELD - 15 MIN

CURRENT STATUS: PAST PRODUCER

COMMODITY:

GYPSUM

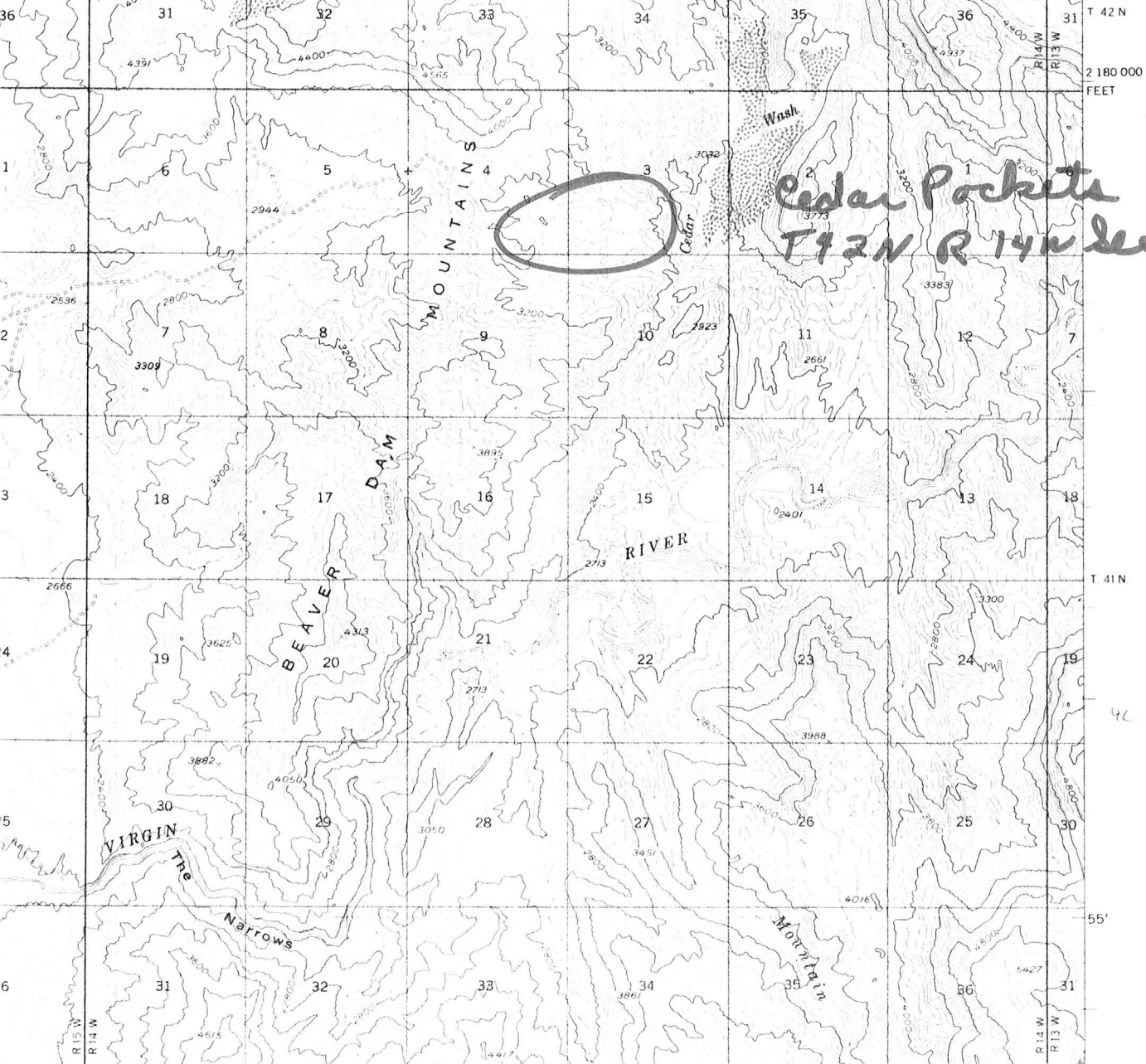
BIBLIOGRAPHY:

ADMMR CEDAR POCKETS FILE
USBM OFR MLA 21-83 BRIGGS, J. MINERAL INVEST-
GATION LIME HILLS, NARROWS, AND SAND COVE
WSA MOHAVE CO. 1983 P. 9

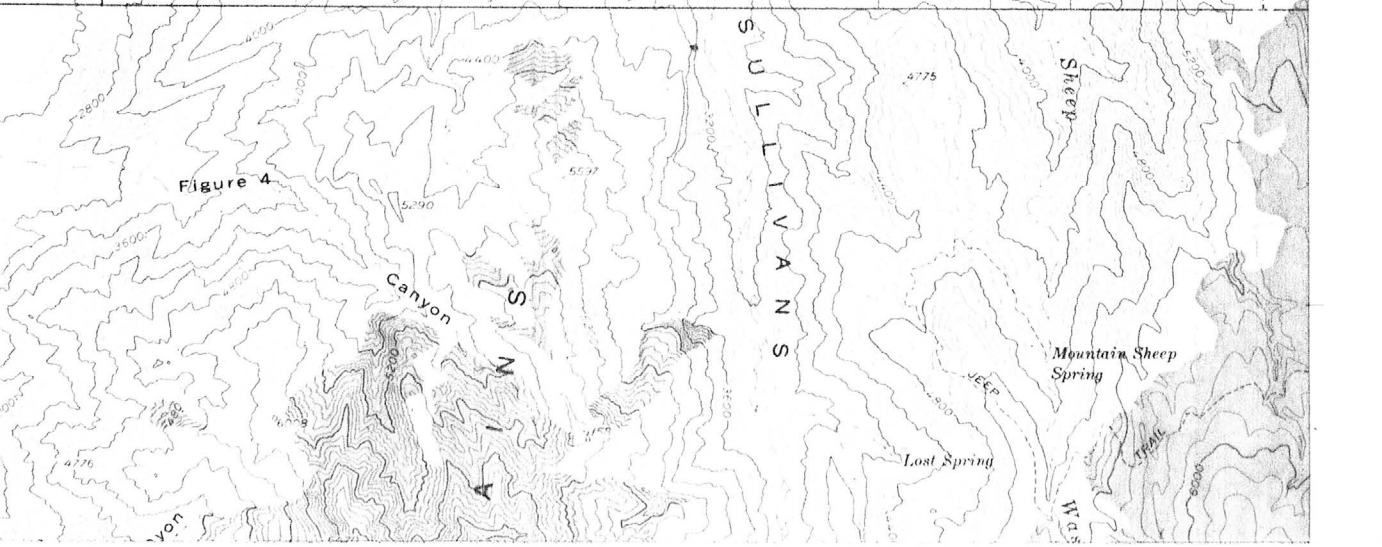
LITTLEFIELD QUADRANGLE
ARIZONA - MOHAVE CO.
15 MINUTE SERIES (TOPOGRAPHIC)

(1ST GEORGE)

UTAH 50' R 14 W 490 000 FEET WASHINGTON CO MOHAVE CO 113°45' 37°00'



Cedar Pockets
T 92 N R 14 W Sec 3 SW



A-2

The United States of America

To all to whom these presents shall come, Greeting:

Arizona 23448

WHEREAS

Curt L. Willsie

is entitled to a land patent pursuant to the general mining laws, R.S. 2329, 2331, as amended (30 U.S.C. 35), for portions of those certain claims known as C & W NO. 12 and C & W NO. 16 placer mining claims situated in the Unorganized Mining District, Mohave County, Arizona, embracing the following described land:

Gila and Salt River Meridian, Arizona

T. 41 N., R. 14 W.,

C & W NO. 12 placer mining claim embracing:
sec. 3, S $\frac{1}{2}$ NE $\frac{1}{2}$ SW $\frac{1}{2}$

C & W NO. 16 placer mining claim embracing:
sec. 3, N $\frac{1}{2}$ SE $\frac{1}{2}$ SW $\frac{1}{2}$

containing 40.00 acres.

NOW KNOW YE, that there is, therefore, granted by the UNITED STATES unto the above named claimant the land described above; TO HAVE AND TO HOLD the said land with all the rights, privileges, immunities, and appurtenances, of whatsoever nature, thereunto belonging, unto the said claimant, his heirs and assigns, forever; and

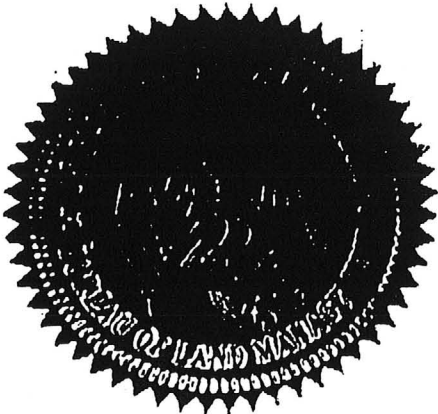
EXCEPTING AND RESERVING TO THE UNITED STATES from the land so granted a right-of-way thereon for ditches or canals constructed by the authority of the United States. Act of August 30, 1890 (43 U.S.C. 945); and

SUBJECT TO the following conditions and stipulations:

1. That the grant hereby made is restricted in its exterior limits to the boundaries of the said mining premises, and to any veins or lodes of quartz or other rock in place bearing gold, silver, cinnabar, lead, tin, copper, or other valuable deposits, which may have been discovered within said limits subsequent to and which were not known to exist on August 12, 1988;

Arizona 23448
Page 2

2. That should any vein or lode of quartz or other rock in place bearing gold, silver, cinnabar, lead, tin, copper, or other valuable deposits be known to exist within the above described premises at said last-named date, the same is expressly excepted and excluded from these presents.



IN TESTIMONY WHEREOF, the undersigned authorized officer of the Bureau of Land Management, in accordance with the provisions of the Act of June 17, 1948 (62 Stat. 476), has, in the name of the United States, caused these letters to be made Patent, and the Seal of the Bureau to be hereunto affixed.

GIVEN under my hand, in City of Washington,
the FIFTEENTH day of APRIL
in the year of our Lord one thousand nine hundred and
NINETY-SIX and of the Independence of the
United States the two hundred and TWENTIEITH

By B. Babbitt
Bruce Babbitt
Secretary of the Interior

02-96-0014

B-2

5-6-99

Serial Number
A-23448

UNITED STATES
DEPARTMENT OF THE INTERIOR
BUREAU OF LAND MANAGEMENT

MINERAL REPORT

Validity Examination
of the
C&W Nos. 1, 12, 15, 16
Mining Claims
for the
Mineral Patent Application
of
Curt L. Willsie

(Title)

Curt Willsie
705 44th Street
Bakersfield, Ca. 93312
805-324-0135

LANDS INVOLVED

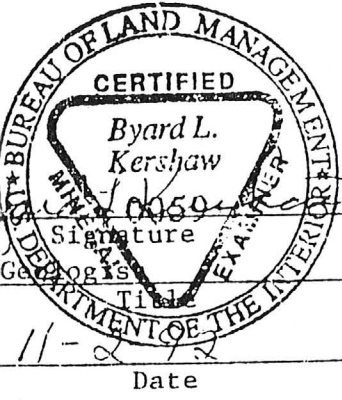
T. 41 N., R. 14 W.,
Gila Salt River Meridian
Mohave County, Arizona
Section 3, E $\frac{1}{2}$ SW $\frac{1}{4}$, E $\frac{1}{2}$ SW $\frac{1}{4}$ SW $\frac{1}{4}$, SW $\frac{1}{4}$ SW $\frac{1}{4}$ SW $\frac{1}{4}$
Section 4, S $\frac{1}{2}$ SE $\frac{1}{4}$ SE $\frac{1}{4}$
containing 130 acres

*C & W Claims
Premier System*

RECEIVED
B.L.M. AZ. STATE OFFICE

NOV 20 1989

7:45 A.M.
PHOENIX, ARIZONA



Prepared By:

John Brund

(Signature)
Geologist

(Title)

November 3, 1989

(Date)

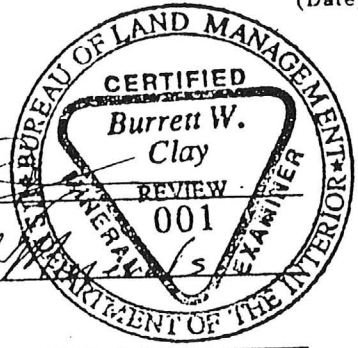
Technical Approval:

[Signature]

(Signature)
Chief, Division of Minerals

(Title)
3/11/94

(Date)



Management Acknowledgement:

[Signature]

(Signature)
Shovits Area Manager

(Title)
April 21, 1994

(Date)

B-3

TABLE OF CONTENTS

	Page
Table of Contents	i
Lists of Attachments, Tables and Plates	ii
Introductory Statement	iii
Summary, Conclusions and Recommendations	1
Introduction	1
Lands Involved	1
Status Record Data	2
Regional Geology	3
Local Geology	4
Mineral Deposits	5
Mineral Production History	5
Mineral Exploration and Development Work	6
Mills and Plants	6
Mineral Production and Marketing	7
Economic Evaluation	10
Field Work, Sampling and Analytical Methods	18
References	31

LIST OF ATTACHMENTS

<u>Attachment No.</u>	<u>Description</u>
1	Master Title Plat
2	Regional Geologic Map
3	Local Geologic Map
4	Map of Mining Claims
5	California Dept. of Food and Agriculture Short Method for Analysis of Gypsum
6	Food Chemicals Codex for Gypsum
7	Assay Analysis - BC Laboratories
8	Assay Analysis - Skyline Laboratories
9	Assay Analysis - IAS Laboratories
10	Block Diagram for Gypsum Tonnage Calculations

LIST OF TABLES

<u>Table No.</u>	<u>Description</u>
1	Analysis of Gypsum and Gypsite Samples From the Narrows Wilderness Study area, Mohave County, Arizona
2	Analysis of Samples Taken From C&W Mining Claims
3	Assays of Gypsum Samples Taken From Selected Locations, Cedar Pockets Area, Mohave County, Arizona

LIST OF PLATES

<u>Plate No.</u>	<u>Description</u>
1	Topography and Vegetation on the Western Portion of the Claims
2	Topography and Vegetation on the Eastern Portion of the Claims
3	Example of Typical Long Sample Trench
4	Example of Typical Short Sample Trench
5	Example of Typical Discovery Point
6	Example of Trench Sample at Discovery Point

INTRODUCTORY STATEMENT

The field work and original draft of this mineral report were conducted and prepared by John Branch. Mr. Branch was the District Geologist in the Arizona Strip District, Arizona at the time. Before he could finalize the report, John lost his life in an automobile accident.

In order to finalize the report, verification of the field work, personal communications and economic analysis was necessary.

That responsibility was assigned to Byard L. Kershaw, in the Division of Mineral Resources, Arizona State Office, Bureau of Land Management.

In order to verify the information, all persons in the report who were listed by John as "personal communications," were contacted by Byard Kershaw, and were asked if the information was correct. All the information was verified as correct. In some instances, the individual contacted was not the individual referenced in the report, either because the individual had died or was no longer employed by the company. In those instances, the person contacted is discussed in the report and listed in the references section of the report.

A field visit was made to the claims by Byard Kershaw on October 6, 1992 to verify the geologic and sampling information contained in the report. Not all of the sample points were found as some had been obliterated by weathering. The sample points found during the field visit were, B, C, D, E, F, I, M and N as depicted on Attachment 4 of the report. Both brass caps identified on Attachment 4 were also found.

The report was reviewed by and all calculations were verified by Byard Kershaw.

MINING CLAIM VALIDITY REPORT
MINERAL PATENT APPLICATION
A-23448

A. Summary, Conclusions and Recommendations

The subject mining claims, covering some 130 acres, contain an estimated 32.5 million tons of gypsum. The gypsum is of relatively high purity and, if selectively mined as proposed in the patent application, could meet the requirements for use as an agricultural soil amendment, as an additive in the manufacture of Portland cement, as a filler and could compete in the food and pharmaceutical grade gypsum markets. There appears to be a market of sufficient size to warrant development of gypsum from this area and, based on estimated mining, production and marketing costs, gypsum from the subject claims could be produced at a profit.

Based on the information contained in this report, it was the professional opinion of John Branch and is the opinion of Byard Kershaw that mineral has been found within the S $\frac{1}{2}$ of the C&W No. 12 and the N $\frac{1}{2}$ of the C&W No. 16 mining claims. It is evident that a person of ordinary prudence would be justified in the further expenditure of his labor and means, with reasonable prospect of success in developing a valuable mine on the forty acre parcel.

Further, it is recommended that the C&W No. 1 and C&W No. 15 mining claims be contested for lack of discovery of a valuable mineral deposit, and that the N $\frac{1}{2}$ of the C&W No. 12 and the S $\frac{1}{2}$ of the C&W No. 16 mining claims should also be contested because these ten acre subdivisions are not mineral in character.

B. Introduction

The purpose of this mining claim validity report is to determine the validity of four mining claims for which a patent application was filed. The patent application was filed on August 12, 1988. On December 28, 1988, a copy of the case file was sent to the Arizona Strip District Office for field examination and preparation of a mineral report and recommendations. This report and conclusions drawn from it are limited to that request and should not be used for purposes other than those prompting the report.

Examinations of the lands encompassed by the mining claims were conducted on June 9, 16, 19, 20, 21, and 22, 1989. Either the claimant, Curt Willsie, or Jim Adams, agent for the claimant, were present during the field examinations and collection of samples. All samples were collected by John Branch.

C. Lands Involved

The lands involved in the patent application are described as follows: T. 41 N., R. 14 W., GSRM., Arizona, section 3: E $\frac{1}{2}$ SW $\frac{1}{4}$; E $\frac{1}{2}$ SW $\frac{1}{4}$ SW $\frac{1}{4}$; SW $\frac{1}{4}$ SW $\frac{1}{4}$ SW $\frac{1}{4}$ and section 4: S $\frac{1}{2}$ SE $\frac{1}{4}$ SE $\frac{1}{4}$. Four mining claims encompassing some 130 acres are applied for in this patent application. The subject lands were identified in the field using the U.S. Geological Survey Mountain Sheep Spring 7.5 minute quadrangle, 1985

provisional edition, and U.S. Government survey monuments located along the south section line in T. 41 N., R. 14 W., section 3.

The subject lands are located in the Beaver Dam Mountains in the extreme northwest corner of Arizona. The claims are located in a shallow valley above Cedar Pockets Wash. Elevations range from approximately 2960 feet to 3200 feet. Topography in the immediate vicinity of the claims consists of rolling hills composed primarily of gypsiferous material interbedded with dolomite and cuestas capped by limestone, dolomite or other resistant rock. Vegetation is sparse (see Plates 1 and 2).

The subject claims lie some 18 miles from St. George, Utah and 96 miles from Las Vegas, Nevada. Primary access from these cities to the claims is provided by Interstate 15. Final access is provided via approximately 1.5 miles of BLM maintained graveled road. The area is accessible year long. Power is available at the site via an existing single phase 12.5 Kv power line owned by Dixie Rural Electrification Association. While the power line traverses the subject mining claims, no substation presently exists for the use of electrical energy from this line. No water is available at the site.

The main use of the land in the past has been for gypsum mining. Grazing has also been authorized on the lands. On August 28, 1984, the area covered by the mining claims was included in the Beaver Dam Wilderness area and was withdrawn from operation of the mining laws, subject to valid existing rights.

D. Status Record Data

The following is the record status data for the claims applied for.

AMC No.	Claim Name	Date Located	Date Amended	Date Recorded	County Book/Page	Subd.	Ac.
67912	C&W No.1	1/30/76		2/20/76	313/708	SE $\frac{1}{4}$ SE $\frac{1}{4}$	40
67923	C&W No.12	1/30/76	9/30/77	2/20/76 10/7/77	313/719 421/702	NE $\frac{1}{4}$ SW $\frac{1}{4}$ NE $\frac{1}{4}$ SW $\frac{1}{4}$	40 40
67926	C&W No.15	1/30/76	9/30/77	2/20/76 10/7/77	313/722 421/705	SW $\frac{1}{4}$ SE $\frac{1}{4}$ SW $\frac{1}{4}$ SW $\frac{1}{4}$	40 40
67927	C&W No.16	1/30/76	9/30/77	2/20/76 10/7/77	313/723 421/706	SE $\frac{1}{4}$ SE $\frac{1}{4}$ SE $\frac{1}{4}$ SW $\frac{1}{4}$	40 40

All mining claims are located for gypsum. No other valid existing rights have been established on the subject lands. The surface and mineral estates are in Federal ownership (see Attachment 1).

The mining claims were originally located by Curt L. Willsie and Delmar Ray Cotton. On March 2, 1982, 50% of Cottons 50% interest was conveyed to a Mr. Ray Gerawan. Between December of 1983 and August of 1985, numerous quit claim deeds and lease agreements were filed with the Mohave County Recorder. On April 3, 1985, a complaint was filed in the Superior Court of California

B-8

against Mr. Curt Willsie by Mr. Ray Gerawan. This civil dispute was settled in December of 1987. Through these court proceedings, Mr. Curt Willsie was determined to be the sole owner of the mining claims.

Assessment work has been filed for the claims every year since the date of location. The following is a listing of the assessment work record as recorded with the BLM and the Mohave County Recorder.

Year	Date Recorded- Mohave Co.	Book	Pages	Date Recorded- BLM
1977	10/07/77	421	697,698	10/10/79
1978	10/27/78	499	615,616	10/10/79
1979	10/04/79	576	554,555	10/10/79
1980	12/08/80	676	902,903	12/09/80
1981	12/28/81	770	366,367	12/29/81
1982	12/23/82	892	049,050	12/27/82
1983	12/27/83	986	181,182	12/29/83
1984	07/09/84	1037	703,704	09/28/84
1985	12/23/85	1168	410,411	01/02/86
1985	01/27/86	1176	574,578	12/31/85
1986	12/23/86	1276	462,463	12/24/86
1987	12/28/87	1386	431,432	12/30/87
1988	12/28/88	1499	231	12/29/88
1989	12/01/89	1634	604	12/05/89

Expenditures for the following work have been made for the benefit of the mining claims: road construction and improvement; stripping of overburden; trenching; drilling; geologic work; assaying; mining; crushing and the shipping of ore.

The first part of the Final Certificate was issued for the claims covered by this patent application on April 4, 1989.

E. Regional Geology

The subject mining claims lie on the extreme eastern edge of the Basin and Range physiographic province. This province extends from southern Oregon and Idaho southward into Texas, New Mexico and Mexico. The Basin and Range province in Arizona is characterized by the following general features: an irregular surface; numerous northwest trending mountain ranges with intervening, sediment filled basins; abundant igneous and metamorphic rock exposures; extensive folding and faulting; and vast exposures of Precambrian rocks.

In northern Arizona, the Precambrian crystalline basement is unconformably overlain by Paleozoic through Cenozoic rocks. The Paleozoic rocks consist of shallow marine and continental sediments deposited in the Rocky Mountain Geosyncline. This geosyncline, the edge of which was located in the vicinity of the subject claims, persisted through the end of the Paleozoic Era. During the Triassic through middle Cretaceous time, the area to the east of the claims received sediment from the Sevier Orogenic Belt which formed as a

result of eastward thrusting. The western edge of thrusting lies in the vicinity of the Beaver Dam Mountains. Further information concerning the regional geology of the area is given in Hintze (1986), Moore (1958), Wilson (1962) and Wilson and Moore (1959).

As shown on the attached geologic map (Attachment 2), sedimentary rocks are exposed at the surface in the vicinity of the subject mining claims. These rocks are composed of limestone, dolomite, sandstone, siltstone, and gypsum.

F. Local Geology

Local geology of the area is shown on Attachment 3. The geology of the area has been described in detail by Moore (1972), Briggs (1983), Harrison (1984), Swapp (1985), and Hintze (1986a). Through a compilation of the above works, the following summary of the stratigraphy and structure of the area is presented.

Rock units of relevance exposed in the immediate vicinity of the mining claims include, in ascending order, the following: Callville Limestone, Pakoon Dolomite, and Queantoweap Sandstone.

The Callville Limestone is composed of 1500 to 2000 feet of medium grey, medium to thick bedded cherty limestone interbedded with calcareous siltstones and sandstones that weather orange (Hintze, 1986; Klockenbrink, 1979). This formation outcrops immediately south of the claims. No gypsum mineralization was observed or has been reported from this formation.

Overlying the Callville Formation is the Pakoon Dolomite. This formation has been described by Hintze (1986) as a light gray, fine grained dolomite. It weathers to light-brownish-gray stair step ledges, slopes and low cliffs. The thickness of this formation ranges from 700 to 900 feet in the Beaver Dam Mountains. All of the gypsum which occurs in the immediate vicinity of the subject claims has been assigned to this unit (McNair, 1951; Hintze, 1986). The gypsum has been described as being from 60 (Hintze, 1986) to over 400 (Harrison, 1984) feet thick. In the area of the subject claims, most gypsum is exposed at the surface. Gypsum outcrops were sampled by Briggs (1983), Harrison (1984) and Swapp (1985). The results of these samples are given in tables 1, 2 and 3. In general, samples from the deposit have been reported in the range of 67% to over 99% CaSO_4 .

The Queantoweap Sandstone lies immediately above the Pakoon Dolomite. This formation is characterized as a fairly uniform, thin to thick bedded, fine to medium grained sandstone. It is from 1,500 to 2,000 feet thick in the vicinity of the Beaver Dam Mountains. This formation covers the gypsum on the northern portions of the claims.

In the vicinity of the claims, the regional strike trends northwest-southeast. Dips range from 17 to 33 degrees north. Based on the measured strikes and dips of the gypsum outcrop in the vicinity of the mining claims and the observed outcrops of both the top and bottom of the gypsum bearing strata, the

B-10

gypsum bearing unit is calculated to be approximately 615 feet thick within the mining claims.

During the field examination, approximately 666 feet of trench were dug using hand tools. The thickness of both gypsum bearing and non-gypsum bearing strata were measured. Based upon these measurements, the gypsum bearing Pakoon Dolomite within the mining claims contains approximately 50% gypsum. The remaining 50% is composed predominantly of dolomite. The true thickness of measured gypsum beds range from 1.6 to 21.4 feet and the true thickness of measured dolomite beds range from 0.7 to 15.4 feet.

G. Mineral Deposits

The gypsum occurs as a strata bound deposit in the Pakoon Dolomite. It does not appear to grade into either the overlying or underlying strata. The deposit is tabular, dipping to the north at from 17 to 33 degrees. Most dips measured were 20 degrees north.

No deposits of strategic or critical minerals are known to occur within the boundaries of the subject mining claims.

H. Mineral Production History

Initial exploration and sampling was conducted in the area in 1969. This work was conducted by Dennis Sherrel et al., on what was then called the Reno claim group. The work consisted of dozer cuts and prospect pits and the claims eventually lapsed (Swapp, 1985).

The C&W mining claims were located in January of 1976. Soon after the claims were located, several truckloads of gypsum were removed and hauled to California for testing and use as a soil conditioner. During the next few years, varying amounts of gypsum were removed from the claims for the purposes of testing the gypsum for use in agriculture, cement manufacture and food and pharmaceutical applications.

In February 1984, a mining plan was submitted for the mining of gypsum from this deposit. This plan was approved in March of 1984. In August of 1984, the area encompassed by the mining claims was designated wilderness. A mining claim validity report (Swapp, 1985), addressing ongoing operations, was completed by the BLM in October of 1985. That report concluded that the C&W Nos. 12, 15 and 16 mining claims were valid. In that report, the gypsum was evaluated for use as an additive in the manufacture of cement.

During October of 1985, production increased to several truck loads per week (Swapp, 1985). Production ceased by the end of 1985. Between 10 and 20 thousand tons of gypsum were reportedly shipped to cement companies in southern California during 1984 and 1985 (Willsie, personal communication, 1989). No mining has been conducted on the claims since the end of 1985.

I. Mineral Exploration and Development Work

Mineral exploration work to date has included trenching and the drilling of shallow exploration holes to determine the presence and quality of gypsum. The holes were drilled with a portable drill. A map showing the location of all of the exploration holes was not available to John Branch. The locations of four of these exploration holes, listed as discovery points, are described in the information submitted in the patent application. No assays were taken from samples obtained from the drill holes and the drill logs were not available to John Branch.

Development work consists of an open pit located on the C&W Nos. 12 and 16 mining claims (see Attachment 4). This pit covers approximately 15 acres and is where the production occurred in 1984 and 1985.

J. Mills and Plants

According to the information contained in the patent application, gypsum would be mined and processed for shipment at the site. The processing plant would have a capacity of 540 tons per day, 250 days per year for a total of 135,000 tons per year. Production would be directed at three markets with approximately 50,000 tons of bulk agricultural grade gypsum, 35,000 tons of bagged specification agricultural gypsum and 50,000 tons of bagged food grade gypsum being produced per year.

Mining would be conducted using open pit methods. Most of the deposit is exposed at the surface. The surface would be cleared and a bench developed with a bulldozer. The surface would be drilled with a rotary percussion drill, probably on a seven by seven foot pattern. The holes would be loaded with ammonium nitrate and detonated to break the rock. A front end loader would be used to mine and transport the gypsum to the processing facility. Waste rock, comprising approximately 50% of the material, would be hauled by the front end loader to a waste rock disposal area located on the claims. Average haul distance for both the ore and waste would be approximately 500 feet (See Attachment 4a).

At the processing facility, the gypsum would be crushed and screened to a minus $\frac{3}{4}$ inch mesh with a 12X48 jaw crusher. Gypsum would then pass through a 42X30 roll crusher where it would be crushed to minus $\frac{1}{4}$ inch. Gypsum of the size minus $\frac{1}{8}$ inch would be screened out and sent to one of six hoppers for bulk load out to the bulk agricultural and cement markets. The portion of the gypsum to be sold as a bagged product would be directed through a 32X45 hammermill where it would be crushed and screened to a minus 100 mesh. The portion of gypsum to be sold as bagged specification agricultural and filler grades would be routed through one of three bagging machines and stored on pallets for load out. The gypsum destined for the food and pharmaceutical grade markets would then be fed through a 66 inch roller mill where it would be ground to a minus 325 mesh. To assure fineness of the material and product grade, this gypsum would then pass through an air classifier. From there the food and pharmaceutical grade gypsum would be sent to a fourth bagging machine and stored on pallets for load out.

According to tests conducted by C.E. Raymond, manufacturer of the Raymond roller mill, use of the Raymond #6669 roller mill would upgrade gypsum with an average initial purity of 90% CaSO_4 to an average grade of 98% CaSO_4 ground to 325 mesh. The Raymond #6669 roller mill has the capacity to process 5.5 tons of gypsum per hour, 98% of which passes through a 325 mesh screen (Bishop, personal communication, 1989). This information was verified by Byard Kershaw after a conversation with Scott McConkey. The name of the company has since been changed to the A.B.B. Raymond Company.

K. Mineral Production and Marketing

General Market Conditions

During 1987, approximately 33,300,000 tons of crude, byproduct and calcined gypsum were produced domestically. Of this total, approximately 5,000,000 tons (15%) were used in the manufacture of portland cement; 1,028,000 tons (3%) were used in agriculture; and 301,000 (less than 1%) were used in fillers and other miscellaneous products (Davis, 1988).

The use of gypsum in portland cement is to retard the setting of concrete, and is used in manufacturing 'white cement' for use in warmer climates.

In the agricultural market, gypsum is used to neutralize alkaline soils, to improve the permeability of clay materials and to provide sulfur and catalytic support for maximum fertilizer utilization.

Filler grade gypsum is used in paint, wood fillers, adhesives, plastics, textiles, modeling clay, glazing compound, tile grouts, and body putty.

Food and pharmaceutical grade gypsum is used in many applications, including a dilutant or extender in the manufacture of pills, for mixing with colorant or pigment in food containers, in enriched flours, cereal, bread conditioners, canned vegetables, and artificially sweetened jellies and preserves.

As discussed above, mineral production from this deposit was limited to the years of 1984 and 1985. During this time, between 10 and 20 thousand tons of material were mined from the site and sold to cement manufacturers in California. Gypsum from this deposit has been found to meet the requirements for use as an additive in the manufacture of portland cement (Swapp, 1985). For the reasons stated below, sales to the bulk cement grade gypsum market are also considered in this report.

The market identified in the patent application includes 50,000 tons/year of bulk agricultural grade material, 35,000 tons per year of bagged specification agricultural grade material and 50,000 tons per year of bagged food grade gypsum. Gypsum from this deposit would be mined, ground and processed for these markets. These markets were all in existence as of August 28, 1984, the date of wilderness designation and withdrawal of the area from operation of the mining law.

DOES
NOT
HAVE
F&P

* Western Gypsum is currently mining gypsum for use in the cement, agricultural and food and pharmaceutical grade markets from a deposit located on public lands in the Arizona Strip District. This operation, which is located approximately ten miles east of the subject claims, produces approximately 50,000 tons of gypsum per year.

The mining and processing methods employed by Western Gypsum are very similar to those proposed in the patent application. Western Gypsum employs eight people in this operation. The gypsum is mined using percussion drills and explosives. It is loaded into a crusher with a front end loader. The material is crushed to minus 1.5 inches and stored in stockpiles at the mine site.

Direct mining costs for the Western Gypsum operation currently run approximately \$4.00 per ton. Transportation cost for gypsum from St. George to the southern California market area is approximately \$0.05 per ton per mile (Cecala, personal communication, 1990).

The current selling price for cement grade gypsum is \$20.00 per ton delivered in the Victorville, California area. In the Victorville area, Cecala estimated a market for approximately 200,000 tons of gypsum per year for use as an additive in the manufacture of portland cement. Additional markets exist for this product in the Riverside and Mojave areas of southern California.

Gypsum of between 92 and 95 percent purity is generally used for bulk agricultural applications. The selling price for bulk agricultural grade gypsum in the San Joaquin Valley area is currently between \$24.00 and \$28.00 per ton, depending on the amount purchased. This price appears to be set by U.S. Gypsum's operation in Hanford, California. Western Gypsum is able to enter this market by selling and delivering gypsum directly to the growers, thus eliminating the cost to the grower of loading the gypsum at the Hanford facility and transporting it to their fields. Material marketed for this use must be one eighth inch minus in size and Western Gypsum is currently marketing the fines produced by their operation for this use. Cecala estimated the market in this area to be approximately 250,000 tons per year.

The specification agricultural and filler grade gypsum markets are varied in terms of both purity requirements and associated uses. Gypsum with a purity of between 95 and 97 percent generally falls into the top grade agricultural, food grade and filler markets. Prices for this material range from \$50.00 to \$110.00 per ton. The actual price paid is determined by the specific grade of the gypsum, the size to which it is ground, whether it is sold as a bagged or bulk product, and whether any transportation charges are included (Cecala, personal communication, 1990).

Bandini Fertilizer Company, located in Los Angeles, California, is a consumer of gypsum which has a purity of between 95 and 97 percent. The gypsum they purchase is ground to approximately 100 mesh and delivered as either a bulk product or bagged in 80 pound bags. The current price paid by Bandini ranges from \$55.00 to \$60.00 per ton for delivered bagged product, and from \$25.00 to \$35.00 per ton for delivered bulk product.

B-14

Generally speaking, the food and pharmaceutical grade markets require a product of at least 98% CaSO_4 . Currently the major producer for food and pharmaceutical applications is U.S. Gypsum. Their product, food and pharmaceutical grade Terra Alba #1, is mined in Southard, Oklahoma. According to IGL Bulletin No. 801 (United States Gypsum Company, 1978), Terra Alba #1 gypsum produced from Southard, Oklahoma is ground such that 98% passes a 325 mesh. This product meets the requirements of food and pharmaceutical manufacturers (Tripathi, personal communication, 1989). The product is currently sold in 50 lb. bags for \$135.00 per ton and \$106.00 per ton bulk, FOB mine (McPherson, personal communication, 1989).

Mr. Scott Gubler, an independent processor, from St. George, Utah, prepares gypsum from the Western Gypsum operation for use in the food and pharmaceutical markets. The product Mr. Gubler produces is comparable to U.S. Gypsum's Terra Alba #1 product. The gypsum is crushed locally under contract to 1.5 inch minus. Mr. Gubler then crushes and grinds it further to an unspecified size. The gypsum is sold by Mr. Gubler for \$140.00 per ton, bagged and palletized, FOB, St. George. Mr. Gubler estimated that the total west coast market for this product is between 150 and 250 tons per month. This equates to between 1,800 and 3,000 tons annually (Gubler, personal communication, 1990).

Western Gypsum also produces gypsum suitable for the food and pharmaceutical grade markets. The price of gypsum of this grade sold by Western Gypsum is approximately \$130.00 per ton, FOB mine. Cecala estimated a market for food and pharmaceutical grade gypsum from the St. George area to be between 10,000 and 20,000 tons per year (Cecala, personal communication, 1990).

Marketing

In the application for patent, it is stated that 50,000 tons bulk agricultural grade gypsum would be produced per year. Given the lack of the applicants ability to produce sale contracts or letters of intent to purchase this type of material, it is doubtful that the 50,000 tons of gypsum could be successfully marketed initially. It should be pointed out that, as with the Western Gypsum operation, it is reasonable to assume that gypsum from this area could be successfully marketed in the cement manufacturing market of southern California. Gypsum from the subject deposit has been found to meet the requirements as an additive to portland cement (Swapp, 1985). It is reasonable to assume that by marketing gypsum in a combination of both the bulk agricultural and cement markets, the stated level of 50,000 tons of gypsum could be sold per year. These markets are not closed markets. Therefore, the applicant should be able to break into the markets.

It is also stated in the application for patent that 35,000 tons per year would be marketed in the specification grade market. This market includes the specification agricultural grade and filler grade markets. There appears to be a ready market for gypsum meeting the requirements for these uses and it is reasonable to assume that the stated level of production could be marketed.

According to information submitted in the patent application, approximately 50,000 tons per year would be marketed in the food grade gypsum market. At this time, however, no sales contracts or letters of intent to purchase have been obtained by the claimant for this product. Estimates as to the size of this market in the St. George area range from 1,800 to 20,000 tons annually. It is extremely doubtful that the level of marketing proposed by the claimant could be attained. Based on the relatively small size of this market, it is estimated that, at most, only 10,000 tons of food and pharmaceutical grade gypsum could be marketed from this deposit annually. This amount totals approximately two percent of the total market (Cecala, 1990 and U.S. Bureau of Mines, 1992).

In summary, even though information contained in the patent application projects some 135,000 tons of gypsum to be marketed annually, there appears to be only an annual market in this area for 95,000 tons. This projected market includes a portion of the cement grade gypsum market which was not identified in the patent application but appears to be large enough to handle shortfalls which are apparent in the bulk agricultural market.

L. Economic Evaluation

Based on the mining and processing scenario presented in the patent application, the mining would be conducted using conventional drilling and blasting techniques. The claimant does not currently own any mining or milling equipment. Equipment needed to mine 95,000 tons annually from this deposit would incur the following capital costs:

Equipment	No. Needed	\$ Ea.	Total \$
D-8 Cat Dozer (used, good condition)	1	60,000	60,000
Air Track Percussion Drill w/ Compressor (used, good condition)	1	20,000	20,000
988 Cat Loader (used, good condition)	1	145,000	145,000
J-D 410 Breaker Hoe (used, good condition)	1	30,000	30,000
Total			\$255,000

Processing equipment would include the following:

Equipment	No. Needed	\$ Ea.	Total \$
450 Kw Power Plant (new, installed)	1	75,000	75,000

Crushing/Screening Plant (used, very good condition)	1	135,000	135,000
Hammermill (reconditioned)	1	22,500	22,500
Roller Mill (used, very good condition)	1	375,000	375,000
Air Classifier (used, good condition)	1	30,000	30,000
Bagging Machine (new)	4	19,750	79,000
Finished Product Loading Equip. (new)	-	180,300	180,300
Hoppers (used, good condition)	6	5,000	30,000
Truck Scale (used, excellent condition)	1	11,500	11,500
Misc. Equipment	-	50,000	50,000
Total			\$988,300

Unless stated otherwise, prices listed for the above equipment reflect a current average price advertised in trade publications for used equipment. In the case of the bagging machine and finished product loading equipment, the price quoted is for new equipment and was obtained from Mr. Bill Brennan of Bemis Packaging Machinery Company (Brennan, personal communication, 1989). Mr. Brennan died on December 12, 1989. This information was verified as correct through personal communications between Byard Kershaw and Steve Laudwig, with Bemis Packaging Machinery Company. The miscellaneous equipment was listed in the patent application and appear to be reasonable for this operation. The above mining and production equipment costs would be amortized over a seven year period.

Approximately \$150,000.00 would be required to cover the costs of equipment mobilization and installation. These costs would be amortized over a seven year period.

The bagging machines are proposed to be housed in a 6,000 square foot metal building set on a concrete slab. In addition, a 1,200 square foot modular office was proposed in the patent application to accommodate administrative functions at the mine. Due to the decrease in production from that proposed

in the patent application, less equipment would need to be housed in the metal building. The space available in the metal building could be used to accommodate administrative functions at the mine, thus eliminating the need for the modular office. The estimated cost for construction of the 6,000 square foot metal building alone is \$288,300.00. The building would be amortized over a 30 year period. The cost per year attributable to the amortization of these buildings would be \$9610.00.

It is assumed that the operation would employ 7 individuals. Three individuals would be employed at the mine, one would operate the crushing and grinding equipment, one would operate the bagging machine, one would be employed in sales and marketing, and one would be employed in management and book keeping. For an eight hour shift, 56 hours of labor would be required. It is assumed that the average wage for the work force would be \$11.00/hr. The average cost of labor including benefits would be approximately \$13.30 per hour (Cecala, personal communication, 1992).

Approximately 360 gallons of diesel fuel would be required for the mine and mill equipment per day. Fuel costs would total approximately \$325.00 per day.

Approximately \$112,550.00 per year would be required for the maintenance of the mining equipment and primary crusher per year. For the hammer mill and three bagging machines used in the production of specification agricultural grade filler gypsum, approximately \$46,450.00 would be required per year. For the roller mill, approximately \$29,700.00 would be required annually.

Approximately \$175.00 per day would be required for explosives.

It is estimated that administrative costs including telephone, office utility, and insurance would not exceed \$500.00/day.

Approximately 10% of the gypsum would be marketed in the food and pharmaceutical grade markets. Gypsum sold in these markets must be at have a content of at least 98% CaSO_4 . It is reasonable to assume that some cost for assay work would be incurred in the production of gypsum for this market to assure that the product meets the specifications.

According to Tripathi (personal communication, 1989) U.S. Gypsum spends \$70.00 per sample for assays to assure quality control of their food and pharmaceutical grade Terra Alba. The assays performed by U.S. Gypsum include not only those specified in the Food Chemicals Codex (FCC), but also salmonella and other unspecified tests. U.S. Gypsum employs three full time chemists in their lab to conduct the assays. Assays conducted by U.S. Gypsum are performed on a lot basis. The lot size is variable, however. According to Tripathi, the cost per ton attributable to assays is a significant portion of the production cost for the food and pharmaceutical grade Terra Alba product.

Western Gypsum currently contracts for their assay work. Assays are performed on a batch basis, with each batch containing 100 tons. The cost to perform these assays currently runs between \$150.00 and \$200.00 per assay.

Assuming an assay cost of \$175.00 for one assay for each 100 ton batch of food and pharmaceutical grade gypsum produced, the assay cost per ton would be approximately \$1.75.

According to information contained in the patent application, approximately \$6.88 would be required per ton to cover the cost of bags, pallets and labels. Bags suitable for the packaging of food and pharmaceutical grade gypsum currently run \$0.20 each (Cecala, personal communication, 1990). The cost per ton attributable to bags for food and pharmaceutical grade gypsum would be \$8.00 alone. The cost for pallets and labels are not expected to add more than \$3.00 per ton to the bag cost for a total of \$11.00 per ton for bags, pallets and labels.

Based on the above figures, production costs for each commodity were estimated using the following formula and example calculation:

Formula

$$\frac{\text{Item Cost/Day or Year} \times \text{Factor}^1}{\# \text{ of Tons/Day or Year}} = \$/\text{Ton}$$

Example

The example calculation will be the mining equipment costs listed below.

$$\frac{\$36,430/\text{yr.} \times .53}{50,000 \text{ Tons/Year}} = \$0.39/\text{Ton}$$

Using the above figures and formula, the following production cost estimates may be made:

Bulk Cement and Agricultural Grade Gypsum 50,000 tons/year, 200 tons/day, 53% of total production.

Item	\$	Factor	\$/ton
<u>CAPITAL COSTS</u>			
Mining Equipment	36,430/yr.	0.53	0.39
Production Equipment			
generator	10,710/yr.	0.53	0.11
primary crusher	19,290/yr.	0.53	0.20
hoppers	4,290/yr.	1.00	0.09
scales	1,640/yr.	0.53	0.02
miscellaneous equipment	7,140/yr.	0.53	0.08

Equipment Mobilization and Installation of

¹The factor reflects the ratio between the quantity of the particular product and the total production from the deposit in daily or yearly terms.

Mining Equipment and Primary Crusher	5,910/yr.	0.53	0.06
Mill Building	9.610/yr.	0.53	0.10

OPERATING COSTS²

Labor (Six individuals including three at the mine, one in sales and marketing, and one in management and book keeping)	638/day	0.53	1.69
Fuel	325/day	0.53	0.86
Maintenance			
Mining Equipment and Primary Crusher	112,550/yr.	0.53	1.19
Explosives	175/day	0.53	0.46
Administrative Costs	500/day	0.53	1.33
Total			6.58
Transportation			
Cement Market (24 ton loads, 255 miles from mine to Victorville, California cement market)	0.05/ton/mile	1.00	12.75
Bulk Agriculture Market (24 ton loads, 430 miles from mine to Hanford, California bulk agricultural market)	0.05/ton/mile	1.00	21.50

Total production and transportation costs

Cement Rock	19.33
Agricultural Rock	28.08

Bagged Specification Agricultural and Filler Grade Gypsum - 35,000 tons/year, 140 tons/day, 37% of total production, 78% of bagged production.

Item	\$	Factor	\$/ton
------	----	--------	--------

² Operating costs were determined using the 20th Edition of the Caterpillar Performance Handbook, and the 1989 issue of the Dataquest Contractors' Equipment Cost Guide.

CAPITAL COSTS

Mining Equipment	36,430/yr.	0.37	0.39
Production Equipment			
generator	10,710/yr.	0.37	0.11
primary crusher	19,290/yr.	0.37	0.20
hammermill	3,210/yr.	0.78	0.07
bagging machines	8,460/yr.	1.00	0.24
finished product	25,760/yr.	0.78	0.57
loading equipment			
truck scale	1,640/yr.	0.37	0.02
miscellaneous equipment	7,140/yr.	0.37	0.08
Equipment Mobilization and Installation of			
Mining Equipment and	5,910/yr.	0.37	0.06
Primary Crusher			
Hammermill	2,850/yr.	0.78	0.06
Three Bagging Machines	1,690/yr.	1.00	0.05
Mill Building	9,610/yr.	0.37	0.10
<u>OPERATING COSTS³</u>			
Labor			
six individuals	638/day	0.37	1.69
one individual	106/day	0.78	0.59
Fuel	325/day	0.37	0.86
Maintenance			
Mining Equipment and	112,550/yr.	0.37	1.19
Primary Crusher			
Hammermill	9,280/yr.	0.78	0.21
Three Bagging Machines	28,000/yr.	1.00	0.80
Explosives	175/day	0.37	0.46
Administration	500/day	0.37	1.32
Assays	245/day	1.00	1.75
Bags and Pallets	1,540/day	1.00	11.00
Total			21.82
Transportation			
(24 ton loads, 360 miles)			

³ Operating costs were determined using the 20th Edition of the Caterpillar Performance Handbook, and the 1989 issue of the Dataquest Contractors' Equipment Cost Guide.

from mine to Los Angeles area) 0.05/ton/mile 1.00 18.00

Total production and transportation costs 39.82
for bagged specification agricultural and filler
grade gypsum.

Bagged Specification Food and Pharmaceutical Grade Gypsum - 10,000 tons/year,
40 tons/day, 10% of total production, 22% of bagged production.

Item	\$	Factor	\$/ton
<u>CAPITAL COSTS</u>			
Mining Equipment	36,430/yr.	0.10	0.36
Production Equipment			
generator	10,710/yr.	0.10	0.11
primary crusher	19,290/yr.	0.10	0.19
hammermill	3,210/yr.	0.22	0.07
roller mill	53,570/yr.	1.00	5.36
air classifier	4,290/yr.	1.00	0.43
bagging machine	2,820/yr.	1.00	0.28
finished product	25,760/yr.	0.22	0.57
loading equipment			
truck scale	1,640/yr.	0.10	0.02
miscellaneous equipment	7,140/yr.	0.10	0.07
Equipment Mobilization and Installation of			
Mining Equipment and	5,910/yr.	0.10	0.06
Primary Crusher			
Hammermill	2,850/yr.	0.22	0.06
Roller Mill	10,400/yr.	1.00	1.04
Bagging Machine	560/yr.	1.00	0.06
Mill Building	9,610/yr.	0.10	0.10
<u>OPERATING COSTS⁴</u>			
Labor			
six individuals	638/day	0.10	1.60
one individual	106/day	0.22	0.58
Fuel	325/day	0.10	0.81
Maintenance			
Mining Equipment and	112,550/yr.	0.10	1.13

⁴ Operating costs were determined using the 20th Edition of the Caterpillar Performance Handbook, and the 1989 issue of the Dataquest Contractors' Equipment Cost Guide.

B-22

Primary Crusher			
Hammermill	9,280/yr.	0.22	0.20
Roller Mill	29,430/yr.	1.00	2.94
Bagging Machine	7,915/yr.	1.00	0.79
Explosives	175/day	0.10	0.44
Administration	500/day	0.10	1.25
Assays	70/day	1.00	1.75
Bags and Pallets	440/day	1.00	11.00
Total			28.32

Sales of this product are FOB mine.

For the purposes of this analysis, it is assumed that 25,000 tons of gypsum would be sold annually in the cement market, 25,000 tons would be sold annually in the bulk agricultural market, 35,000 tons would be sold annually in the bagged specification agricultural grade market, and 10,000 tons would be sold annually in the food and pharmaceutical grade gypsum market. The respective revenues per ton sold are \$20.00, \$28.00, \$55.00, and \$130.00. Based on the above assumptions, the following net revenues (gross revenues minus capital, operating, and transportation) would be generated the first year of operation:

25,000 tons cement rock X \$0.67/ton	= \$	16,750
25,000 tons bulk agricultural grade rock X -\$0.08	= \$	-2,000
35,000 tons bagged specification grade rock X \$15.18	= \$	531,300
10,000 tons bagged F&P grade rock X \$101.67/ton	= \$	1,016,700
Total	= \$	1,562,750
Reclamation ⁵		-5,000
Total after reclamation		\$1,557,750

⁵ The applicant estimated that reclamation costs would total \$5,000.00. Reclamation would include ripping all existing roads outside the area proposed for patent, and filling in all dozer cuts which were excavated to expose possible gypsum deposits. In the estimation of Byard Kershaw, this cost is adequate.

Taxes	
Property	-15,759 ⁶
Sales and Use	-138,281 ⁷
Estimated 35% Income Tax	-491,300
Total after taxes	\$ 912,410

While the calculations show that the bulk agricultural product is being produced at a small loss, there is a significant profit from the production of the remaining products and the bulk agricultural production pays for about 25% of the mine's capital and operating costs. Without this production the cost of the other products would increase.

M. Field Work, Sampling And Analytical Methods

Sampling of the gypsum on the mining claims was conducted in June of 1989. During the six days of sample collection, some 44 samples were collected by John Branch. A continuous channel sample obtained from a trench was collected from each of the claims. In addition to the continuous channel sample, a shorter channel sample was taken from each of the remaining ten acre subdivisions. The long, continuous channel sample method was used in order to obtain information concerning the thickness of individual beds, both gypsum bearing and dolomite, and information concerning the grade of the gypsum within individual beds. This information was needed in order to determine the

⁶ Property taxes are calculated by the following formula obtained from the Assessor's Office, Mohave County, Arizona, and using the straight line depreciation value contained in the Arizona State Department of Revenue's Personal Property Manual, Valuation Table 7. The calculation is assuming that all equipment has a 14 year life, and was acquired in 1989.

Tax Amount = Depreciated Value X Assessed Value X Local Tax Rate

Depreciated Value = 0.71 X Purchase Price
 Assessed Value = 0.25 X Depreciated Value
 Local Tax Rate = 0.071415 of Assessed Value

Example for Dozer: Tax Amount = (\$60,000)(0.71) X 0.25 X 0.071515 = \$760.00

⁷ Sales and Use Tax is 3-1/8% (3.125%) of Gross Sales.

Gross Sales = \$4,425,000 X 0.03125 = \$138,281

Cement Grade = 25,000 tons X \$20.00/ton = \$500,000
 Bulk Ag. Grade = 25,000 tons X \$28.00/ton = \$700,000
 Bagged Ag. Grade = 35,000 tons X \$55.00/ton = \$1,925,000
 Food and Pharm. Grade = 10,000 tons X \$130.00/ton = 1,300,000

Total = \$4,425,000

ore to waste ratio and purity of the gypsum should selective mining be employed.

The continuous channel samples were all located within previously existing dozer cuts (see Attachment 4 and Plate 3). These sites were chosen because they offered exposures of the section where some of the weathered surface had been removed. The shorter channel sample sites (see Attachment 4 and Plates 4, 5 and 6) were chosen based on the exposure of gypsum outcrop within the ten acre subdivision.

In all cases, the trench sites were cleaned with a pulaski type of tool to a depth of two to four inches below the existing ground surface, swept with a broom and the channel cut with a chisel edged rock pick. The channel samples were all approximately one inch wide, the width of the rock pick, and from one half to one inch deep. The depth of the channel sample was constant for the interval sampled.

In all cases, the direction and slope of the channel sample was measured along with the sample interval length. A strike and dip measurement was also made at each sample site. Using this information, the true thickness of the beds was calculated using the following formula:

$$\text{True Thickness} = [\text{measured thickness}] [\text{Cos}(|90 - \text{angle between bed strike and trench strike}|)] [\text{Sin}(\text{dip angle} + \text{trench angle})]$$

The following is a summary of the measured length of the channel sample, trend and slope of the channel, strike and dip of the beds, calculated true bed thickness and rock type for interval.

All thicknesses and lengths are reported in units of feet.

C&W No.1 Mining Claim

Trend of channel: Sample 1-1 through 1-5 (including interbedded dolomite)- N5E.
Sample 1-6 through 1-9 (including interbedded dolomite)- N5W

Slope of Channel: Sample 1-1 through 1-5 (including interbedded dolomite)- 10S
Sample 1-6 through 1-9 (including interbedded dolomite)- 18S

Strike of beds: N80W

Dip of beds: 20N

<u>Sample No.</u>	<u>Measured Thickness</u>	<u>Calculated Thickness</u>	<u>Rock Type</u>
1-1	11.0	5.5	gypsum
-	14.8	7.4	dolomite
1-2	3.5	1.7	gypsum
1-3	8.5	4.2	gypsum
-	8.0	4.0	dolomite
1-4	7.2	3.6	gypsum

-	14.5	7.2	dolomite
1-5	10.0	5.0	gypsum
-	9.0	4.5	dolomite
1-6	9.8	5.8	gypsum
-	13.5	8.0	dolomite
1-7	10.0	5.9	gypsum
-	14.0	8.3	dolomite
1-8	25.6	15.2	gypsum
-	16.0	9.5	dolomite
1-9	14.0	8.3	gypsum

(END OF TRENCH)

Corrected thickness of section measured in trench: 104.1 feet

Corrected thickness of gypsum measured in trench: 55.2 feet

Corrected thickness of dolomite measured in trench: 48.9 feet

1-10 .

Trend of channel: N/A
Slope of Channel: 90
Strike of beds: N80W
Dip of beds: 20N

1-10	3.5	3.3	gypsum
------	-----	-----	--------

C&W No.12 Mining Claim

Trend of channel: Sample 12-4 (including interbedded dolomite)- N10E
Samples 12-5 and 12-6 (including interbedded dolomite)- N28E
Samples 12-7 through 12-10 (including interbedded dolomite) N15E

Slope of Channel: Sample 12-4 (gypsum bearing unit only) - 27S Samples 12-5 and 12-6 (including dolomite unit above 12-5 and two dolomite units within interval)- 7S Samples 12-7 through 12-10 (including interbedded dolomite)- 18S

Strike of beds: N64W
Dip of beds: 20N

<u>Sample No.</u>	<u>Measured Thickness</u>	<u>Calculated Thickness</u>	<u>Rock Type</u>
12-4	12.0	8.4	gypsum
-	1.5	0.7	dolomite
12-5	11.5	5.2	gypsum
-	4.7	2.1	dolomite
12-6	8.5	3.9	gypsum
-	12.5	5.7	dolomite
12-7	7.0	4.2	gypsum
-	19.7	11.9	dolomite
12-8	9.5	5.7	gypsum

-	8.5	5.1	dolomite
12-9	8.5	5.1	gypsum
-	18.5	11.2	dolomite
12-10	29.5	17.8	gypsum
-	25.5	15.4	dolomite

(END OF TRENCH)

Corrected thickness of section measured in trench: 102.4 feet
 Corrected thickness of gypsum measured in trench: 50.3 feet
 Corrected thickness of dolomite measured in trench: 52.1 feet

Trend of channel: N/A
Slope of Channel: 90
Strike of beds: N30W
Dip of beds: 17E

12-11	7.5	7.5	gypsum
-------	-----	-----	--------

Trend of channel: N55E
Slope of Channel: 28W
Strike of beds: N58W
Dip of beds: 33N

12-13	14.5	11.7	gypsum
-------	------	------	--------

Trend of channel: N44E
Slope of Channel: 28S
Strike of beds: N40W
Dip of beds: 24E

12-14	14.5	11.4	gypsum
-------	------	------	--------

C&W No.15 Mining Claim

Trend of channel: N35E
Slope of Channel: 19S
Strike of beds: N55W
Dip of beds: 33N

<u>Sample No.</u>	<u>Measured Thickness</u>	<u>Calculated Thickness</u>	<u>Rock Type</u>
15-1	16.5	13.0	gypsum
-	14.5	11.4	dolomite
15-2	26.0	20.5	gypsum
-	11.5	9.1	dolomite
15-3	10.0	7.9	gypsum
-	22.0	17.3	dolomite
15-4	29.3	23.1	gypsum
-	11.3	8.9	dolomite
15-5	11.8	9.3	gypsum
-	19.3	15.2	dolomite
15-6	6.0	4.7	gypsum
-	10.1	7.9	dolomite

B-27

15-7 10.0 7.9
 (END OF TRENCH) gypsum

Corrected thickness of section measured in trench: 156.2 feet
 Corrected thickness of gypsum measured in trench: 86.4 feet
 Corrected thickness of dolomite measured in trench: 69.8 feet

Trend of channel: N/A
Slope of Channel: 90
Strike of beds: N55W
Dip of beds: 33N

15-8 3.5 1.9 gypsum

Trend of channel: N/A
Slope of Channel: 90
Strike of beds: N55W
Dip of beds: 33N

15-9 3.5 1.9 gypsum

Trend of channel: N10E
Slope of Channel: 30S
Strike of beds: N55W
Dip of beds: 33N

15-10 15.0 12.1 gypsum

C&W No.16 Mining Claim

Trend of channel: N18W
Slope of Channel: 12S
Strike of beds: N73W
Dip of beds: 20E

<u>Sample No.</u>	<u>Measured Thickness</u>	<u>Calculated Thickness</u>	<u>Rock Type</u>
16-1	8.5	3.7	gypsum
16-2	6.0	2.6	dolomite
16-3	9.5	4.1	gypsum
16-4	10.4	4.5	dolomite
16-5	8.5	3.7	gypsum
16-6	9.5	4.1	dolomite
16-7	8.0	3.5	gypsum
16-8	19.25	8.4	dolomite
16-9	21.25	9.2	gypsum

(END OF TRENCH)

Corrected thickness of section measured in trench: 43.8 feet
 Corrected thickness of gypsum measured in trench: 19.6 feet
 Corrected thickness of dolomite measured in trench: 24.2 feet

Trend of channel: N24E

B-28

Slope of Channel: 26S
Strike of beds: N80W
Dip of beds: 20N

16-10 10.5 7.3 gypsum

Trend of channel: N/A
Slope of Channel: N/A
Strike of beds: N/A
Dip of beds: N/A

16-11 N/A Grab gypsum

Trend of channel: N16E
Slope of Channel: 40S
Strike of beds: N73E
Dip of beds: 20N

16-12 3.8 3.2 gypsum

Trend of channel: N/A
Slope of Channel: 90
Strike of beds: N73W
Dip of beds: 20N

16-13 1.7 1.7 gypsum

With the exception of samples 16-1 through 16-9, samples were taken from gypsum beds only. In the case of samples 16-1 through 16-9, samples were obtained from all intervals including the dolomite units. Only the samples containing gypsum, however, were assayed. Dolomite determination was made by John Branch. Dolomite beds were not assayed as they were judged by John Branch and claimants not to contain material suitable for the intended use.

Assay methods employed for this report were based on the intended use of the gypsum in agriculture and as a food additive. Since the intended agriculture market is located primarily in California, the California Department of Food and Agriculture, Feed and Fertilizer Section, was contacted and a copy of their test procedures obtained. The State of California, does not set minimum standards for gypsum grade used in agriculture. Standards are set, however, for testing procedures to determine the purity of gypsum sold. These test procedures were used to determine the percentage of gypsum in the samples collected from the claims. A copy of these procedures is enclosed as Attachment 5.

To determine if the samples met food grade standards, test procedures contained in the FCC, 3rd. ed. (National Academy Press, 1981) were used. These standards and test procedures are required for calcium sulfate used as a food additive by Food and Drug Administration regulations 21 CFR Ch. 1 at 184.1230 (b). A copy of the FCC standards and testing procedures is enclosed as Attachment 6.

Samples were sent to two laboratories for analysis. One of the labs, BC Laboratories, Inc. located in Bakersfield, California, was requested by the claimant. This lab had been used in the past by the claimant for sample analysis for gypsum from this deposit. The State of California does not certify testing labs but a check of other BLM offices in California indicated that the lab is reputable and does a lot of agricultural product and EPA testing (Weiwood, personal communication, 1989). Samples 1-1 through 1-10, 16-1, 16-3, 16-5, 16-7, 16-8, 16-10, 16-11 and 16-12 were sent to BC Laboratories for analysis. Samples 1-5, 1-6 and 16-5 were analyzed according to the FCC for calcium sulfate. The remainder of the samples were analyzed for gypsum content according to the California State Department of Agriculture test procedures. A copy of the analysis performed by BC Laboratories is included as Attachment 7.

A second lab was chosen to perform sample analyses as a check of lab accuracy. The second lab chosen was Skyline Laboratories located in Tucson, Arizona. This lab is certified by the State of Arizona to perform assays. Skyline Laboratory does some testing of gypsum for agricultural purposes. They had not, however, performed any tests for calcium sulfate according to the FCC. The test procedures were discussed with Mr. Jim Martin of Skyline lab and a copy of both the FCC and the State of California Department of Agriculture test procedures were sent to him. These procedures were used in the sample analysis. Skyline Laboratories analyzed samples 12-6, 12-10, 15-3, 15-7 and 16-9 according to the FCC procedures for food grade gypsum. Samples 12-4, 12-5, 12-7 through 12-11, 12-13, 12-14, 15-1, 15-2, 15-4 through 15-6, 15-8, 15-9, 16-10 and 16-13 were analyzed according to the State of California Department of Agriculture test procedures for agricultural gypsum. A copy of the test results is enclosed as Attachment 8.

All samples were collected by John Branch and remained in his possession or in a secure area until sent to the respective labs. The samples were sent to the labs via U. S. Postal Service Express Mail, Return Receipt Requested.

The results of the assays indicate that high purity gypsum does lie within the claim boundaries. Gypsum content ranges from 64.60% to 99.37%. Two of the samples contained over 99% gypsum, 31 were between 90% and 99% pure, six were between 80% and 90% pure and one was between 60% and 70% pure. None of the samples analyzed according to the FCC exceeded the limits of arsenic, selenium or heavy metals reported as lead. In the case of fluoride, four of the samples analyzed by Skyline Laboratories, samples 12-10, 15-3, 15-7 and 16-9, exceed the limits for fluoride set in the FCC.

All samples analyzed according to the FCC were sent IAS Laboratories located in Phoenix, Arizona, for a second assay to determine fluoride content. IAS Laboratories was chosen because they have the capabilities to perform the assay as required by the FCC, method II for fluoride and they do not perform assays in their lab which require the use of hydrogen fluoride. The lack of hydrogen fluoride in IAS's laboratory significantly decreased the chance of error being introduced in the assay from residual fluoride in the glass ware.

A copy of the test results from IAS Laboratories, Inc. is enclosed as

13-30

Attachment 9. The fluoride limit for food grade gypsum established by the FCC is 30 ppm. No samples were found to exceed this limit in the second test and it is assumed that the fluoride reported by Skyline Laboratories was in error due to fluoride contamination in the glass ware.

Given the nature of the gypsum deposit and the requirement for high purity gypsum in the identified markets, selective mining of gypsum from this deposit would be required. All of the gypsum beds measured during the field examination except one were greater than two feet in thickness. At the Blue Diamond deposit, Nevada, the upper two feet of the gypsum beds are routinely drilled to assure that the drillhole has entered the gypsum deposit. The bottom two feet are then backfilled and the holes are blasted to remove overburden (Rogers, personal communication, 1989). It is assumed that the minimum true bed thickness of gypsum which would be mined at this deposit is two feet. Given that, all but one of the beds measured met or exceeded this thickness requirement, it is assumed that all of the gypsum within the gypsum bearing beds would be mined using selective mining techniques.

Tonnage of the deposit lying within the boundaries of the subject mining claims was calculated in blocks as shown in Attachment 10. The tonnage calculations assume a flat upper surface and do not take into account any gypsum contained in the small hills protruding above an elevation of approximately 2800 feet. It is assumed that mining will begin on the southern end of the claim group and progress in a northerly direction. Benches would be developed based on gypsum bed thickness. It is further assumed that, since the southern end of the claims lies in the vicinity of the contact between the gypsum bearing Pakoon Dolomite and the underlying Callville Limestone, the bottom of the pit would slope 20N along the bedding planes to a maximum depth of approximately 200 feet. Once this depth was attained, it would remain constant for the remainder of the pit to the north. The maximum assumed pit depth is based on the geology of the deposit and observed mining practices at the Blue Diamond deposit. At Blue Diamond, the high wall created by mining is currently 160 feet (Rogers, personal communication, 1989). The highwall there is almost vertical but will be reduced as mining progresses. For the subject deposit, it is assumed that a 1:1 final slope would be left along all highwalls.

In calculating the tonnage of the deposit, it was assumed that the ore to waste ratio is 1:1 and each cubic foot of solid gypsum weighs 175 lbs. The tonnage calculations reflect tonnage of gypsum in place. The 50% purity in the following calculations is to account for the 1:1 stripping ratio.

Given these parameters, the following tonnage of the deposit is calculated below.

Block A

N-S Distance = 1295 feet
E-W Distance = 920 feet
Thickness = 200 feet

$1295 \times 920 \times 200 = 238,280,000$ cubic feet

238,280,000 cubic feet X 50% purity = 119,140,000 cu. ft. gypsum
119,140,000 cu. ft. gypsum X 175 lbs./cu. ft. = 20,084,950,000 lbs. gypsum
20,084,950,000 lbs. gypsum X 1 ton/2000 lbs. = 10,424,750 tons gypsum

Block B

N-S Distance = 560 feet
E-W Distance = 1580 feet
Thickness = 200 feet

560 X 1580 X 200 = 176,960,000 cubic feet
176,960,000 cubic feet X 50% purity = 88,480,000 cu. ft. gypsum
88,480,000 cu. ft. gypsum X 175 lbs./cu. ft. = 15,484,000,000 lbs. gypsum
15,484,000,000 lbs. gypsum X 1 ton/2000 lbs. = 7,742,000 tons gypsum

Block C

N-S Distance = 125 feet
E-W Distance = 1580 feet
Thickness = 179 feet

125 X 1580 X 179 = 35,352,500 cubic feet
35,352,500 cubic feet X 50% purity = 17,676,250 cu. ft. gypsum
17,676,250 cu. ft. gypsum X 175 lbs./cu. ft. = 3,093,343,800 lbs. gypsum
3,093,343,800 lbs. gypsum X 1 ton/2000 lbs. = 1,546,672 tons gypsum

Block D

N-S Distance = 460 feet
E-W Distance = 3760 feet
Thickness = 79 feet

460 X 3760 X 79 = 136,638,400 cubic feet
136,638,400 cubic feet X 50% purity = 68,319,200 cu. ft. gypsum
68,319,200 cu. ft. gypsum X 175 lbs./cu. ft. = 11,955,860,000 lbs. gypsum
11,955,860,000 lbs. gypsum X 1 ton/2000 lbs. = 5,977,930 tons gypsum

Block E1

N-S Distance = 200 feet
E-W Distance = 1980 feet
Thickness = 79 feet

200 X 1980 X 79 = 31,284,000 cubic feet
31,284,000 cubic feet X 50% purity = 15,642,000 cubic feet gypsum
15,642,000 cu. ft. gypsum X 175 lbs./cu. ft. = 2,737,350,000 lbs. gypsum
2,737,350,000 lbs. gypsum X 1 ton/2000 lbs. = 1,368,675 tons gypsum

Block E2 (2 ea.)

N-S Distance = 200 feet
E-W Distance = 660 feet
Thickness = 100 feet

200 X 660 X 100 = 13,200,000 cubic feet
13,200,000 cubic feet X 50% purity = 6,600,000 cubic feet gypsum
6,600,000 cu. ft. gypsum X 175 lbs./cu. ft. = 1,155,000,000 lbs. gypsum
1,155,000,000 lbs. gypsum X 1 ton/2000 lbs. = 577,500 tons gypsum

E2 X 2 = 1,155,000 tons gypsum total

Block E3

N-S Distance = 1320 feet
E-W Distance = 200 feet
Thickness = 100 feet

1320 X 200 X 100 = 26,400,000 cubic feet
26,400,000 cubic feet X 50% purity = 13,200,000 cubic feet gypsum
13,200,000 cu. ft. gypsum X 175 lbs./cu. ft. = 2,310,000,000 lbs. gypsum
2,310,000,000 lbs. gypsum X 1 ton/2000 lbs. = 1,155,000 tons gypsum

Block E4

N-S Distance = 200 feet
E-W Distance = 1120 feet
Thickness = 100 feet

200 X 1120 X 100 = 22,400,000 cubic feet
22,400,000 cubic feet X 50% purity = 11,200,000 cubic feet gypsum
11,200,000 cu. ft. gypsum X 175 lbs./cu. ft. = 1,960,000,000 lbs. gypsum
1,960,000,000 lbs. gypsum X 1 ton/2000 lbs. = 980,000 tons gypsum

Block E5

N-S Distance = 1980 feet
E-W Distance = 200 feet
Thickness = 100 feet

1980 X 200 X 100 = 39,600,000 cubic feet
39,600,000 cubic feet X 50% purity = 19,800,000 cubic feet gypsum
19,800,000 cu. ft. gypsum X 175 lbs./cu. ft. = 3,465,000,000 lbs. gypsum
3,465,000,000 lbs. gypsum X 1 ton/2000 lbs. = 1,732,500 tons gypsum

B-34

Block E6

N-S Distance = 660 feet
E-W Distance = 200 feet
Thickness = 79 feet

660 X 200 X 79 = 10,428,000 cubic feet
10,428,000 cubic feet X 50% purity = 5,214,000 cubic feet gypsum
5,214,000 cu. ft. gypsum X 175 lbs./cu. ft. = 912,450,000 lbs. gypsum
912,450,000 lbs. gypsum X 1 ton/2000 lbs. = 456,225 tons gypsum

Summary:

Block	Gypsum Tonnage
A	10,424,750
B	7,742,000
C	1,546,672
D	5,977,930
E1	1,368,675
E2	1,155,000
E3	1,155,000
E4	980,000
E5	1,732,500
E6	456,225
Total	<u>32,538,752</u>

The projected level of production stated in the patent application is 135,000 tons of gypsum per year. At this rate, gypsum mining could continue for approximately 240 years. At the annual rate of production for which there is a demonstrated market, 95,000 tons per year, mining on the claims could continue for some 340 years.

The main pit area developed by mining in 1984 is located within the S½ of the C&W No. 12 and the N½ of the C&W No. 16 mining claims. If mining were to be conducted on these 40 acres in the same manner as that described above for the entire block, approximately 9,917,250 tons of gypsum could be produced. At a production rate of 95,000 tons per year, this volume represents some 104 years worth of reserves.

Gypsum within the remaining 90 acres under application for patent are without current or prospective value within the foreseeable future. This gypsum is without value as a person of ordinary prudence would not be justified in expending labor and means with a reasonable anticipation of developing a valuable mine. As such, the discovery of a valuable mineral has not been made on the C&W Nos. 1 and 15 mining claims. In addition, valuable minerals do not exist within the N½ of the C&W No. 12 mining claim and the S½ of the C&W No. 16 mining claim.

B-35

In conclusion, it was the opinion of John Branch and is the opinion of Byard Kershaw that the gypsum contained within the $S\frac{1}{2}NE\frac{1}{4}SW\frac{1}{4}$ and the $N\frac{1}{2}SE\frac{1}{4}SW\frac{1}{4}$ of section 3, T. 41 N., R. 14 W., GSRM, Arizona constitutes a discovery of a valuable mineral on the C&W Nos. 12 and 16 mining claims. The reserves on the portions of the claims proposed for patent are enough for 100 years of production at the stated levels in this report.

M. References

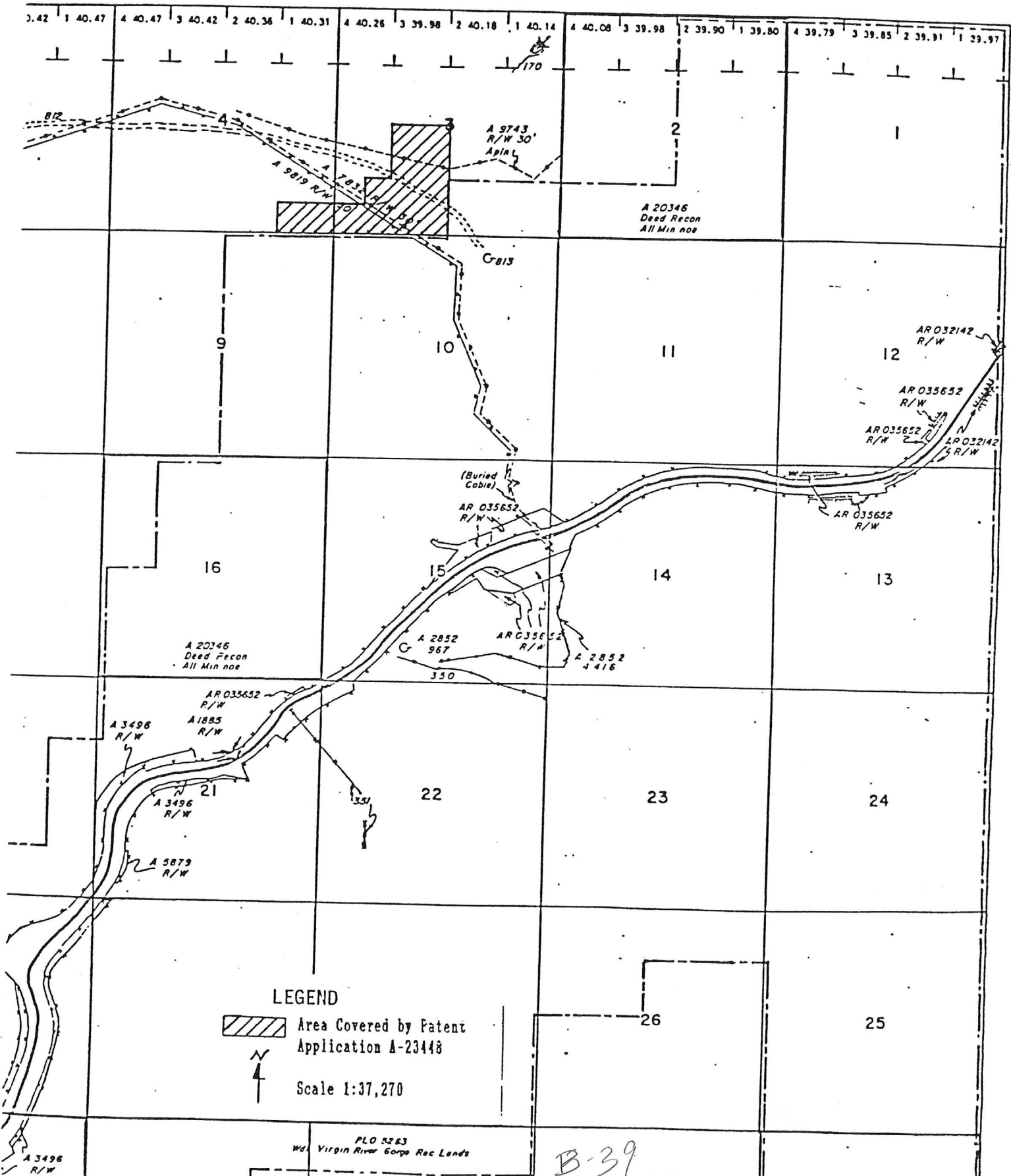
- Bishop, B., 1989, C.E. Raymond Company: personal communication.
- Brennan, B., 1989, Bemis Packaging Machinery Company: personal communication.
- Briggs, J.P., 1983, Mineral investigation of the Lime Hills, Narrows and Sand Cove wilderness study areas, Mohave County, Arizona: U.S. Bureau of Mines Open File Report 80-1056, 28 p.
- Caterpillar, Inc., 1989, Caterpillar Performance Handbook, 20th Edition: Peoria, Illinois, Caterpillar, Inc.
- Cecala, D., 1990, 1992, President, Western Gypsum Corporation, personal communication.
- Dataquest, 1989, Contractors' Equipment Cost Guide: San Jose, California, Dataquest.
- Davis, L.W., 1988, Gypsum in 1987 Minerals Yearbook: U.S. Bureau of Mines, 10 p.
- Gubler, S., 1990, 1992, personal communication.
- Harrison, R.L., 1984, Evaluation of a gypsum property, Cedar Wash, Northwestern Arizona Strip: Harrison, Bacon & Harrison, Geological Consultants, unpublished report, 14 p.
- Hintze, L.F., 1986, Stratigraphy and structure of the Beaver Dam Mountains in Griffen, D.T. and Phillips, R.W. eds., Thrusting and Extensional Structures and Mineralization in the Beaver Dam Mountains, Southwestern Utah: Utah Geological Association Publication 15, p. 1-36.
- - , 1986a, Geology of the Mountain Sheep Spring quadrangle, Mohave County, Arizona in Griffen, D.T. and Phillips, R.W. eds., Thrusting and Extensional Structures and Mineralization in the Beaver Dam Mountains, Southwestern Utah: Utah Geological Association Publication 15, scale 1:24,000.
- Klockenbrink, L.T., 1979, Lexicon of Paleozoic Stratigraphic Names used in Northwestern Arizona and Southwestern Nevada in Beus, S.S. and Rawson, R.R., eds., Carboniferous stratigraphy in the Grand Canyon country, northern Arizona and southern Nevada: American Geological Institute Guidebook Series No. 2, p. 133-138.
- Laudwig, S., 1992, Bemis Packaging Machinery Company: personal communication.
- McConkey, S., 1992, A.B.B. Raymond Company: personal communication 1992
- McNair, A.H., 1951, Paleozoic Stratigraphy of Part of Northwestern Arizona: American Association of Petroleum Geologists Bulletin v. 35, p. 503-541.

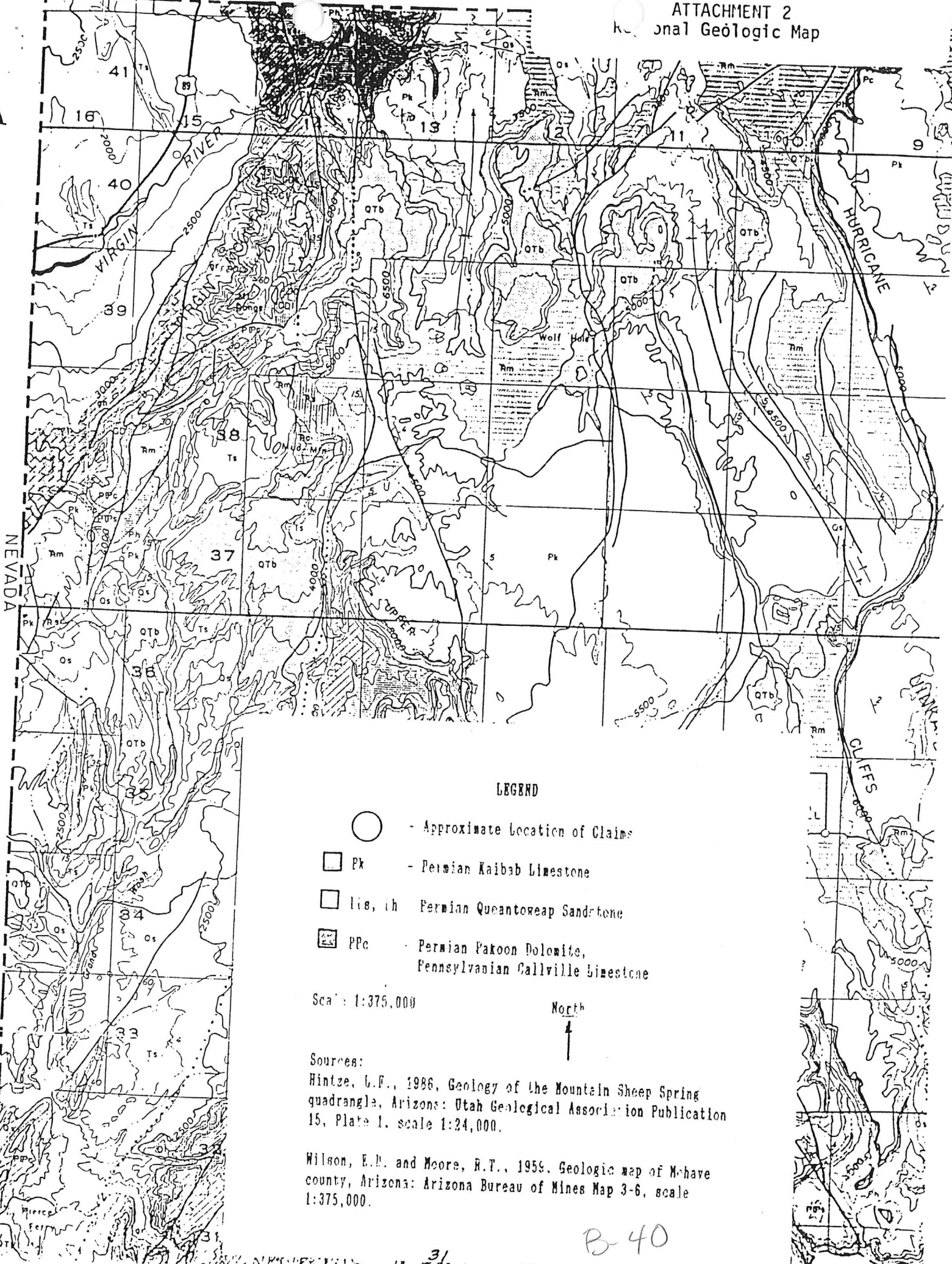
- McPherson, D., 1989, 1992, U.S. Gypsum Corporation: personal communication.
- Moore, R.T., 1958, Geology of Northwestern Mohave County, Arizona: Masters Thesis, University of Arizona, 81 p.
- -, 1972, Geology of the Virgin and Beaver Dam Mountains, Arizona: Arizona Bureau of Mines Bulletin 186, 65 p.
- Rogers, B., 1989, 1992, James Hardy Gypsum Company, Blue Diamond, Nevada: personal communication.
- Swapp, C.W., 1985, Mineral report, Cedar Pockets Gypsum, Southwest Minerals, Inc., Mohave County, Arizona: U.S. Bureau of Land Management, unpublished report, 33 p.
- Tripathi, V., 1989, U.S. Gypsum Corporation: personal communication.
- United States Bureau of Mines, 1992, Mineral Commodity Summaries, 204 pp.
- United States Gypsum Company, 1978, USG Calcium Sulfate Fillers, Industrial Applications: IGL Bulletin No. 801, pp. 85 and 86.
- Willsie, C.L., 1989, 1992, mining claimant: personal communication.
- Wilson, E.D., 1962, A Resume of the Geology of Arizona: Arizona Bureau of Mines Bulletin 171, 140 p.
- Wilson, E.D. and Moore, R.T., 1959, Geologic Map of Mohave County, Arizona: Arizona Bureau of Mines Map 3-6, scale 1:37,500.
- Weiwood, R., 1989, 1992, Bureau of Land Management, California Desert District, personal communication.

WNESHIP 41 NORTH RANGE 14 WEST OF THE GILA AND SALT RIVER

ATTACHMENT 1

Master Title Plat
Aug, 7, 1985





LEGEND

- - Approximate Location of Claims
- Pk - Permian Kaibab Limestone
- ls, sh - Permian Queantowep Sandstone
- Ppc - Permian Pukoon Dolomite,
Pennsylvanian Callville Limestone

Scale: 1:375,000

North



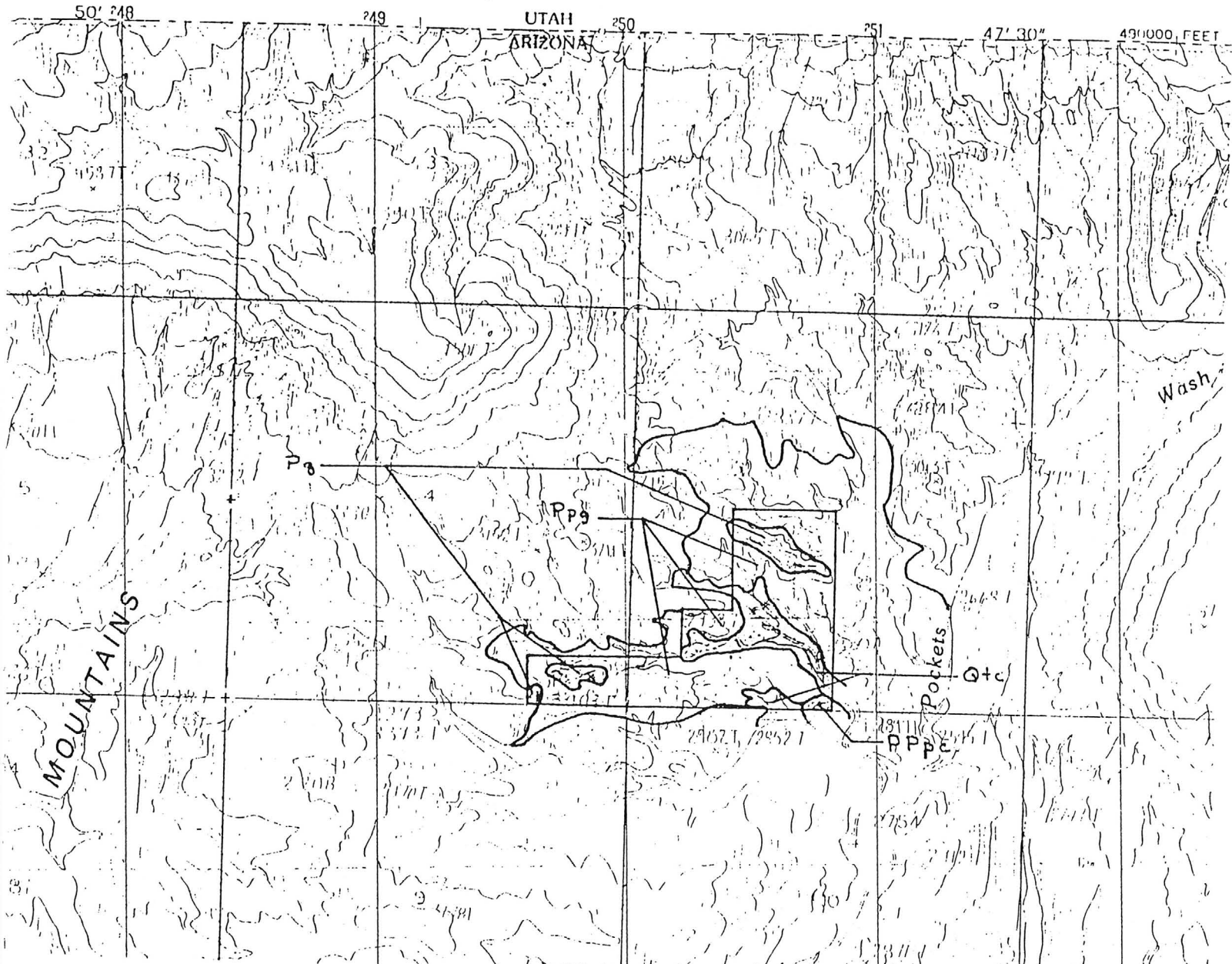
Sources:

Hintze, G.F., 1986, Geology of the Mountain Sheep Spring quadrangle, Arizona: Utah Geological Association Publication 15, Plate 1, scale 1:24,000.

Wilson, E.D. and Moore, R.T., 1959, Geologic map of Mohave county, Arizona: Arizona Bureau of Mines Map 3-6, scale 1:375,000.

B-40

ATTACHMENT 3
Local Geologic Map



LEGEND

- - Patent Application A-23148
 - Qtc - Quaternary colluvium and pediment deposits.
 - Pq - Permian Queantoweap Sandstone.
 - Pp9 - Permian Pakoon Dolomite, gypsum bearing.
 - PP9c - Pennsylvanian - Permian Pakoon Dolomite and Callville Limestone, undivided.
- Geology based on Hintze (1986a), modified by the author.

Base Map: Mountain Sheep Spring 7.5 minute topographic quadrangle, Mohave County, Arizona, provisional edition, 1985. Scale 1:24,000. Contour Interval=40 feet.

Map of Mining Claims

Branch

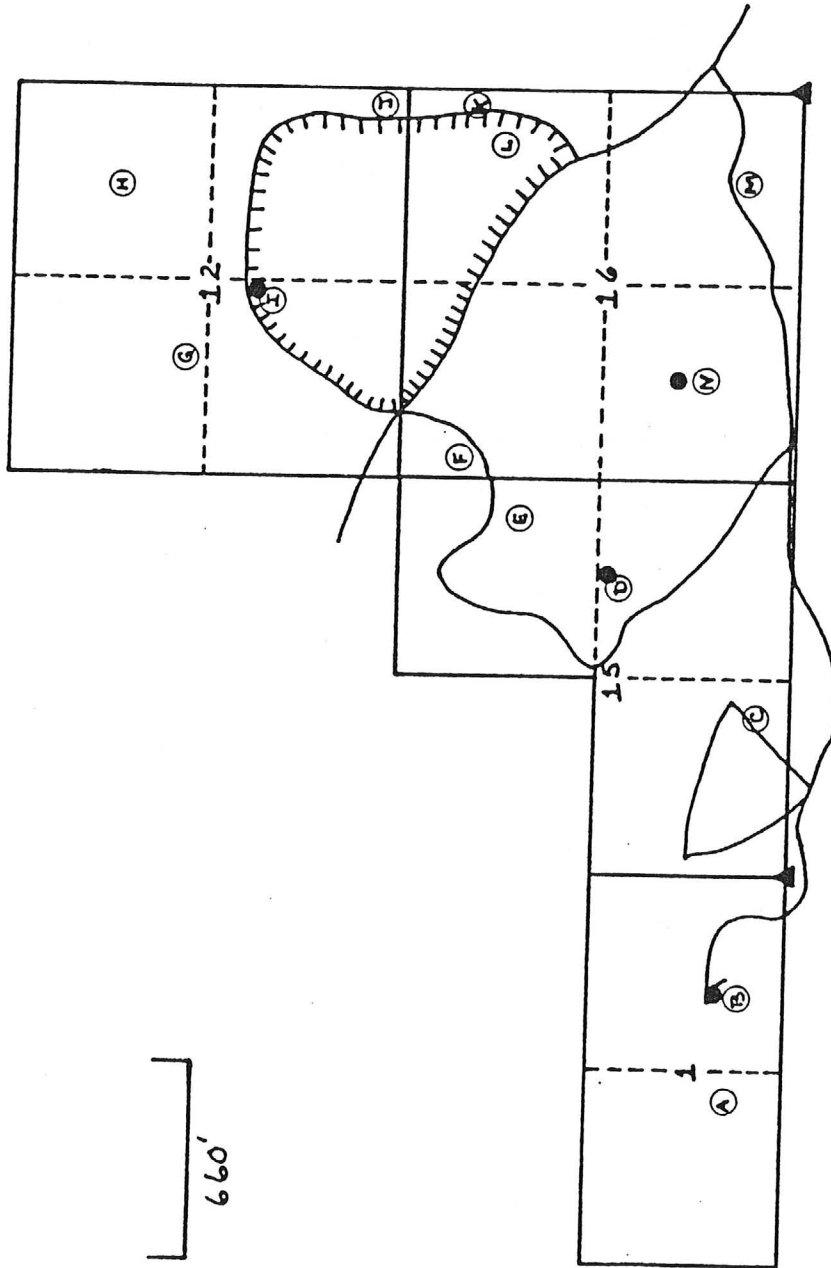
10-24-89

A-23448

Mining Claim Map

1 2

AZ-010



LEGEND

- (A) Sample 1-10
 - (B) Samples 1-1 through 1-9 (trench)
 - (C) Samples 15-1 through 15-7 (trench)
 - (D) Sample 15-10
 - (E) Samples 15-8, 16-9
 - (F) Sample 16-3
 - (G) Sample 12-14
 - (H) Sample 12-11
 - (I) Samples 12-4 through 12-10 (trench)
 - (J) Sample 12-13
 - (K) Sample 16-10
 - (L) Sample 16-11
 - (M) Samples 16-1 through 16-9 (trench)
 - (N) Samples 16-12, 16-13
 - (O) Discovery Point
 - (P) Survey Monument (brass cap)
 - (Q) Mine Area
 - (R) Road
 - (S) Ten Acre Subdivision Line
- Scale 1:7920

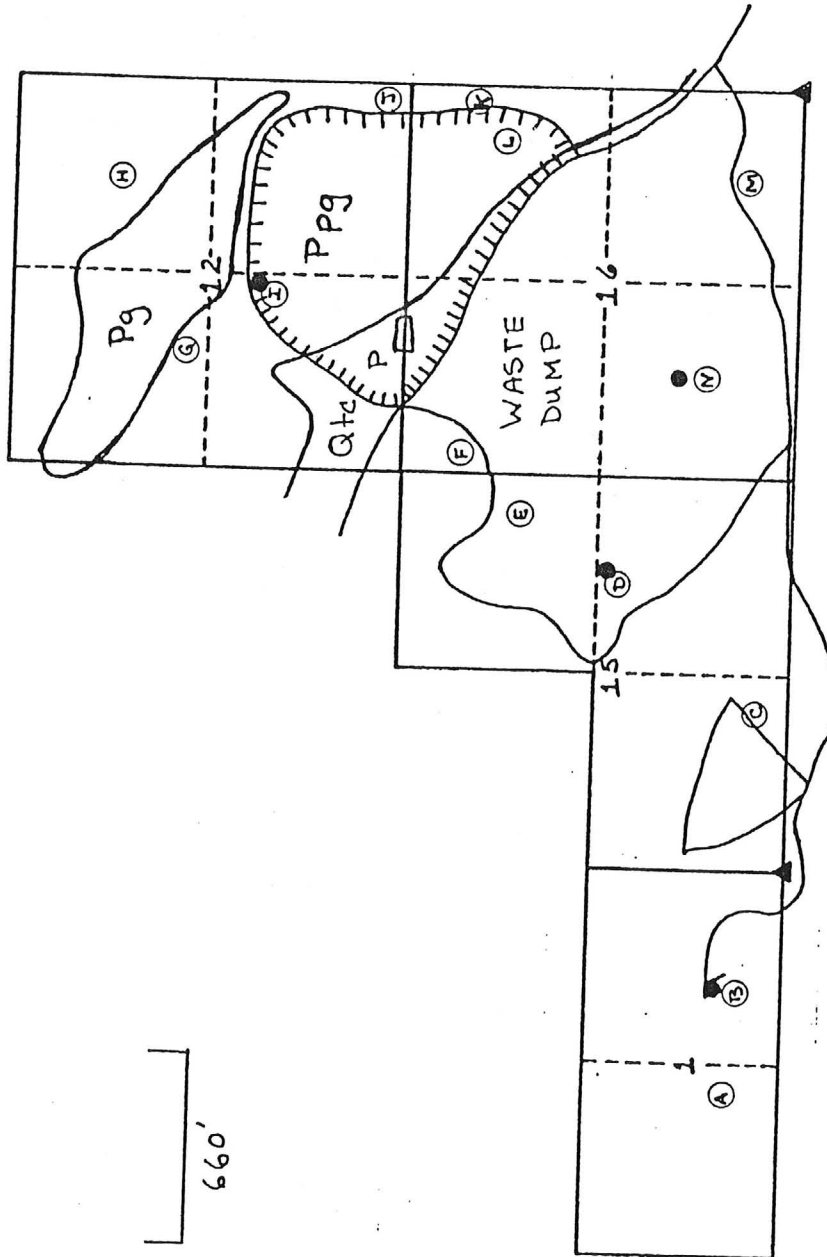
B-42

ATTACHMENT 4a
Map of Mining Claims

Branch 10-24-89 A-23448

Mining Claim Map

AZ-010



— GEOLOGIC BOUNDARIES (SEE ATTACHMENT 3)

P PROCESSING FACILITY

● Discovery Point

▲ Survey Monument (brass cap)

⬢ Mine Area

— Road

--- Ten Acre Subdivision Line

Scale: 1:7920

LEGEND

- (A) Sample 1-10
- (B) Samples 1-1 through 1-9 (trench)
- (C) Samples 15-1 through 15-7 (trench)
- (D) Sample 15-10
- (E) Samples 15-8, 16-9
- (F) Sample 16-3
- (G) Sample 12-14
- (H) Sample 12-11
- (I) Samples 12-4 through 12-10 (trench)
- (J) Sample 12-13
- (K) Sample 16-10
- (L) Sample 16-11
- (M) Samples 16-1 through 16-9 (trench)
- (N) Samples 16-12, 16-13

2

2

THIRD EDITION

FOOD
CHEMICALS
CODEX

COMMITTEE ON CODEX SPECIFICATIONS

Food and Nutrition Board
Division of Biological Sciences
Assembly of Life Sciences
National Research Council

53

1

NATIONAL ACADEMY PRESS
Washington, D.C. 1981

B-44

water, attach an air condenser, and reflux gently for 45 min. Wash the sides of the flask and the condenser with about 40 ml of water, and heat on a steam bath until no odor of alcohol remains. Add 6 ml of dilute sulfuric acid (1 in 2), heat until the fatty acids are melted, then cool to about 60°, and add 25 ml of petroleum ether. Swirl the mixture gently, and transfer quantitatively to a separator. Collect the water layer in a 100-ml volumetric flask, and wash the petroleum ether layer with two 20-ml portions of water, adding the washings to the volumetric flask. Dilute to volume with water, and mix. Transfer 1.0 ml of this solution into a second 100-ml volumetric flask, dilute to volume with water, and mix.

Procedure Transfer 1.0 ml of the *Test Preparation* into a test tube, and transfer 1.0 ml of water to a second test tube to serve as the blank. Treat each tube as follows: Add 1 drop of cupric sulfate TS, swirl gently, and then add rapidly from a buret 9.0 ml of sulfuric acid. Loosely stopper the tube, and heat in a water bath at 90° for exactly 5 min. Cool immediately to below 20° in an ice bath for 5 min, add 3 drops of *p*-phenylphenol TS, shake immediately, and heat in a water bath at 30° for 30 min, shaking the tube twice during this time to disperse the reagent. Heat the tube in a water bath at 90° for exactly 90 s, and then cool immediately to room temperature in an ice water bath. Determine the absorbance of the solution in a 1-cm cell, at 570 nm, with a suitable spectrophotometer, using the blank to set the instrument. Obtain the weight, in µg, of lactic acid in the portion of the *Test Preparation* taken for the *Procedure* by means of the *Standard Curve*.

Packaging and Storage Store in tight containers in a cool dry place.

Functional Use in Foods Dough conditioner; stabilizer; whipping agent.

Calcium Sulfate

$\text{CaSO}_4 \cdot x\text{H}_2\text{O}$

Mol wt (anhydrous) 136.14

DESCRIPTION

Calcium sulfate is anhydrous or contains two molecules of water of hydration. It occurs as a fine, white to slightly yellow white, odorless powder.

REQUIREMENTS

Identification

Dissolve about 200 mg by warming with a mixture of 4 ml of diluted hydrochloric acid TS and 16 ml of water. A white precipitate forms when 5 ml of ammonium oxalate TS is added to 10 ml of the solution. Upon the addition of barium chloride TS to the remaining 10 ml, a white precipitate forms that is insoluble in hydrochloric and nitric acids.

Assay Not less than 99.0% of CaSO_4 , calculated on the dried basis.

Arsenic (as As) Not more than 3 ppm.

Fluoride Not more than 0.003%.

Heavy Metals (as Pb) Not more than 10 ppm.

Loss on Drying CaSO_4 (anhydrous): not more than 1.5%; $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (dihydrate): between 19% and 23%.

Selenium Not more than 0.003%.

TESTS

Assay Dissolve 250 mg, accurately weighed, in 100 ml of water and 4 ml of diluted hydrochloric acid TS, boil to effect solution, and cool. While stirring, preferably with a magnetic stirrer, add about 30 ml of 0.05 *M* disodium EDTA from a 50-ml buret, then add 25 ml of sodium hydroxide TS and 300 mg of hydroxy naphthol blue indicator, and continue the titration to a blue endpoint. Each ml of 0.05 *M* disodium EDTA is equivalent to 6.807 mg of CaSO_4 .

Arsenic Mix 1 g with 10 ml of water, add 12 ml of diluted hydrochloric acid TS, and heat to boiling to dissolve the sample. Cool, filter, and dilute the filtrate to 35 ml with water. This solution meets the requirements of the *Arsenic Test*, page 464.

Fluoride Weigh accurately 1.67 g, and proceed as directed in the *Fluoride Limit Test*, page 510.

Heavy Metals Mix 2 g with 20 ml of water, add 25 ml of diluted hydrochloric acid TS, and heat to boiling to dissolve the sample. Cool, and add ammonium hydroxide to a pH of 7. Filter, evaporate to a volume of about 25 ml, and refilter if necessary to obtain a clear solution. This solution meets the requirements of the *Heavy Metals Test*, page 512, using 20 µg of lead ion (Pb) in the control (*Solution A*).

Loss on Drying, page 518 Dry at 250° to constant weight.

Selenium Determine as directed in *Method II* under the *Selenium Limit Test*, page 537, using 200 mg of sample.

Packaging and Storage Store in well-closed containers.

Functional Use in Foods Nutrient; dietary supplement; yeast food; dough conditioner; firming agent; sequestrant.

Cananga Oil

DESCRIPTION

The oil obtained by distillation from the flowers of the tree *Cananga odorata* Hook f. et Thoms., (Fam. *Anonaceae*). It is a light to deep yellow liquid having a harsh floral odor suggestive of ylang ylang. It is soluble in most fixed oils and in mineral oil, but it is practically insoluble in glycerin and in propylene glycol.

REQUIREMENTS

Identification

The infrared absorption spectrum of the sample exhibits relative

B-45

Jack Parman
916-427-4595

CALIFORNIA DEPT. OF FOOD & AGRICULTURE
FEED & FERTILIZER SECTION
CHEMISTRY LABORATORY SERVICES
3292 Meadowview Road
Sacramento, CA 95832

Received
Strip District
JULY 1988
3D: GYPSUM.MET

JUL 03 1989

St. George, Utah
10:00 AM

SHORT METHOD FOR ANALYSIS OF GYPSUM

SCOPE:

This method is suitable for analysis of natural gypsum or gypsite, which may contain a small amount of calcium carbonate. It is not suitable for analyzing by-product gypsums that contain free sulfuric acid or other sulfates. The ground sample is dissolved in dilute HCl and the sulfate is precipitated as $BaSO_4$. Percent gypsum is calculated from the weight of $BaSO_4$. All analyses are reported on the basis of the material as received and not on a dry basis. To account for the moisture lost in preparation of the sample, this method determines the moisture before and after grinding.

EQUIPMENT:

1. Aluminum weighing dish
2. Drying oven, 45°C. and 105°C.
3. 400 ml beaker
4. Hot plate or steam bath
5. Whatman glass fiber filter GF/F
6. Porcelain Gooch crucible

REAGENTS:

1. Hydrochloric acid, conc.
2. 10% Barium Chloride solution

SAMPLE PREPARATION:

1. Open the sample package and take several representative cuts through the material with a "Scoopula" trier.
2. Mix this removed portion and weigh 10.00 g into a small aluminum dish. Dry 4 hours or overnight at 45°C. Cool in a desiccator. Weigh again and calculate weight loss as moisture (or "free water") in the material as received. This does not include the water of crystallization in gypsum, $CaSO_4 \cdot 2H_2O$.
3. Grind the remainder of the sample to pass a 60-mesh sieve and mix it thoroughly.
4. Determine the moisture in the ground material in the same way as was done for the unground material.

ANALYSIS:

1. Weigh 0.5 grams of the ground sample into a 400 ml beaker.
2. Add 10 ml HCl and 50 ml H₂O. Boil gently 15 to 30 minutes or until all gypsum appears to be dissolved.
3. Filter hot solution with vacuum thru glass fiber filter (GF/F) in gooch crucible. Wash beaker and filter with hot water, police beaker to be sure all material is washed into filter. Quantitatively transfer filtrate back to beaker, volume should be about 250 ml.
4. Bring to a boil. Add excess of 10% BaCl₂ solution (15 to 20 mls). BaSO₄ precipitation in proper crystal form depends on how this is added. Add the first few milliliters dropwise until solution is well clouded. Remainder may be added more rapidly. Keep hot on steam bath or low temperature hot plate for at least one hour. Allow to cool several hours or overnight.
5. Filter cool solution with vacuum thru tared gooch crucible with glass fiber filter (GF/F) previously dried at 105°C. Police the beaker if necessary to transfer all the precipitate to the filter. Wash with at least 10 small portions of water.
6. Dry crucible and contents 2 hours at 105°C, cool and weigh.

CALCULATIONS:

% Gypsum (CaSO₄·2H₂O) in ground material

$$\% \text{ Gypsum} = \frac{\text{weight BaSO}_4 \times .7376}{\text{weight sample}} \times 100$$

$$\% \text{ Sulfur} = \% \text{ Gypsum} \times .1862$$

$$\% \text{ Calcium} = \% \text{ Gypsum} \times .2328$$

Correction for loss of moisture in grinding

$$\% \text{ Gypsum as received} = \frac{100 - \% \text{ moisture (unground)}}{100 - \% \text{ moisture (ground)}} \times \% \text{ Gypsum (ground)}$$

If there is any question about the presence of sulfates other than gypsum, both total sulfate and total calcium should be determined and the percentage of gypsum calculated from the lower component.

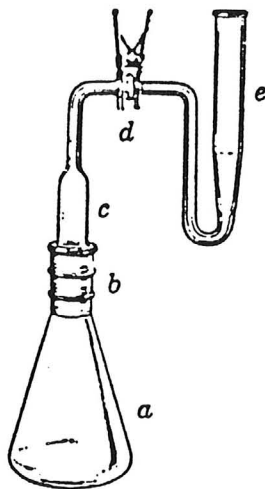


FIGURE 2 General Apparatus for Arsenic Test (Courtesy of the Fisher Scientific Co., Pittsburgh, Pa.)

accurately weighed, to a porcelain crucible, and carefully ignite until the material is completely charred.

Caution: The ignited salt should be protected from contact with the free flame at all times.

After cooling, place the crucible in a beaker, add 50 ml of water and 50.0 ml of 0.5 *N* sulfuric acid, and disperse the carbonized mass with a glass rod. Cover the beaker, and boil the mixture for 30 min. Filter, wash the residue with hot water until the washings are neutral to litmus, and cool. To the combined filtrate and washings add phenolphthalein TS, and titrate the excess acid with 0.5 *N* sodium hydroxide. The weight of the alkali salt is obtained by multiplying the volume of the acid consumed by the equivalence factor of the salt being analyzed.

A 400-mg sample and 0.1 *N* acid and sodium hydroxide may be employed satisfactorily in this assay.

Arsenic Test

Silver Diethyldithiocarbamate Colorimetric Method

NOTE: All reagents used in this test should be very low in arsenic content.

Apparatus The general apparatus, shown in Fig. 2, is to be used unless otherwise specified in an individual monograph. It consists of a 125-ml arsine generator flask (a) fitted with a scrubber unit (c) and an absorber tube (e), with a 24/40 standard-taper joint (b) and a ball-and-socket joint (d), secured with a No. 12 clamp, connecting the units. The tubing between d and e and between d and c is a capillary having an inside diameter of 2 mm and an outside diameter of 8 mm. Alternatively, an apparatus embodying the principle of the general assembly described and illustrated may be used.

NOTE: The special assemblies shown in Figs. 3, 4, and 5 are to be used only when specified in certain monographs.

Standard Arsenic Solution Weigh accurately 132.0 mg of arsenic trioxide that has been previously dried at 105° for 1 h, and dissolve it in 5 ml of sodium hydroxide solution (1 in 5). Neutralize the solution with diluted sulfuric acid TS, add 10 ml in excess, and dilute to 1000.0 ml with recently boiled water. Transfer 10.0 ml of this solution into a 1000-ml volumetric flask, add 10 ml of diluted sulfuric acid TS, dilute to volume with recently boiled water, and mix. Use this final solution, which contains 1 μg of arsenic (As) in each ml, within 3 days.

Silver Diethyldithiocarbamate Solution Dissolve 1 g of recrystallized silver diethyldithiocarbamate, $(C_2H_5)_2NCSSAg$, in 200 ml of reagent-grade pyridine. Store this solution in a light-resistant container and use within 1 month.

Silver diethyldithiocarbamate is available commercially or may be prepared as follows: Dissolve 1.7 g of reagent grade silver nitrate in 100 ml of water. In a separate container, dissolve 2.3 g of sodium diethyldithiocarbamate, $(C_2H_5)_2NCSSNa \cdot 3H_2O$, in 100 ml of water, and filter. Cool both solutions to about 15°, mix the two solutions, while stirring, collect the yellow precipitate in a medium-porosity sintered-glass crucible or funnel, and wash with about 200 ml of cold water.

Recrystallize the reagent, whether prepared as directed above or obtained commercially, as follows: Dissolve in freshly distilled pyridine, using about 100 ml of solvent for each g of reagent, and filter. Add an equal volume of cold water to the pyridine solution, while stirring. Filter off the precipitate, using suction, wash with cold water, and dry in vacuum at room temperature for 2 to 3 h. The dry salt is pure yellow in color and should show no change in character after 1 month when stored in a light-resistant container. Discard any material that changes in color or develops a strong odor.

Stannous Chloride Solution Dissolve 40 g of reagent-grade stannous chloride dihydrate, $SnCl_2 \cdot 2H_2O$, in 100 ml of hydrochloric acid. Store the solution in glass containers and use within 3 months.

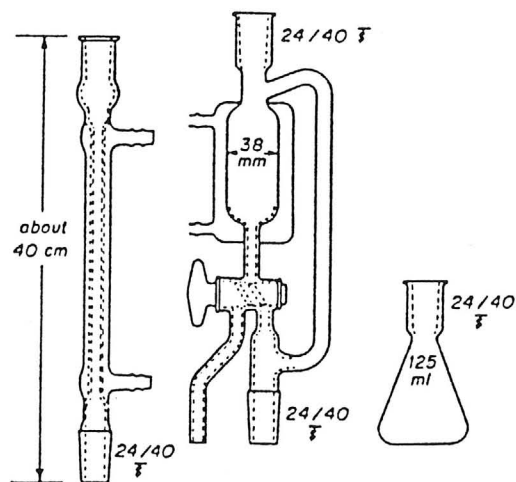


FIGURE 3 Modified Bethge Apparatus for the Distillation of Arsenic Tribromide

B-48

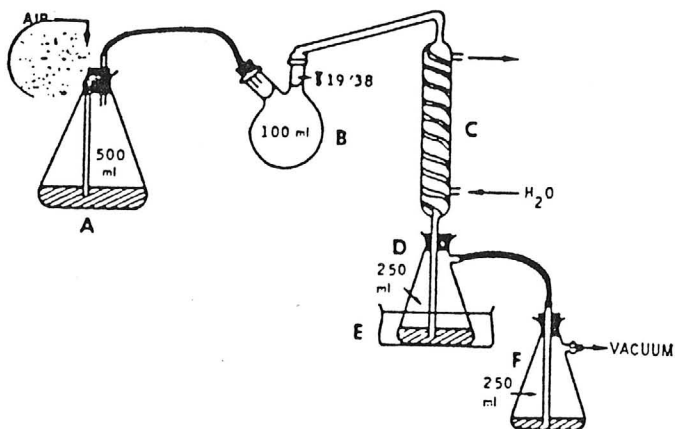


FIGURE 4 Special Apparatus for the Distillation of Arsenic Trichloride (Flask A contains 150 ml of hydrochloric acid; flasks D and F contain 20 ml of water. Flask D is placed in an ice water bath, E.)

Lead Acetate-Impregnated Cotton Soak cotton in a saturated solution of reagent-grade lead acetate, squeeze out the excess solution, and dry in a vacuum at room temperature.

Sample Solution The solution obtained by treating the sample as directed in an individual monograph is used directly as the *Sample Solution* in the *Procedure*. Sample solutions of organic compounds are prepared in the generator flask (a), unless otherwise directed, according to the following general procedure:

Caution: Some substances may react unexpectedly with explosive violence when digested with hydrogen peroxide. Appropriate safety precautions must be employed at all times.

NOTE: If halogen-containing compounds are present, use a lower temperature while heating the sample with sulfuric acid, do not boil the mixture, and add the peroxide, with caution, before charring begins, to prevent loss of trivalent arsenic.

Transfer 1.0 g of the sample into the generator flask, add 5 ml of sulfuric acid and a few glass beads, and digest at a temperature not exceeding 120° until charring begins, using preferably a hot plate in a fume hood. (Additional sulfuric acid may be necessary to completely wet some samples, but the total volume added should not exceed about 10 ml.) After the sample has been initially decomposed by the acid, add with caution, dropwise, 30% hydrogen peroxide, allowing the reaction to subside and reheating between drops. The first few drops must be added very slowly with sufficient mixing to prevent a rapid reaction, and heating should be discontinued if foaming becomes excessive. Swirl the solution in the flask to prevent unreacted substance from caking on the walls or bottom of the flask during digestion. *Maintain oxidizing conditions at all times during the digestion by adding small quantities of the peroxide*

whenever the mixture turns brown or darkens. Continue the digestion until the organic matter is destroyed, gradually raising the temperature of the hot plate to 250°–300° until fumes of sulfur trioxide are copiously evolved and the solution becomes colorless or retains only a light straw color. Cool, add cautiously 10 ml of water, again evaporate to strong fuming, and cool. Add cautiously 10 ml of water, mix, wash the sides of the flask with a few ml of water, and dilute to 35 ml.

Procedure If the *Sample Solution* was not prepared in the generator flask, transfer to the flask a volume of the solution, prepared as directed, equivalent to 1.0 g of the substance being tested, and add water to make 35 ml. Add 20 ml of dilute sulfuric acid (1 in 5), 2 ml of potassium iodide TS, and 0.5 ml of *Stannous Chloride Solution*, and mix. Allow the mixture to stand for 30 min at room temperature. Pack the scrubber tube (c) with two plugs of *Lead Acetate-Impregnated Cotton*, leaving a small air space between the two plugs, lubricate joints b and d with stopcock grease, if necessary, and connect the scrubber unit with the absorber tube (e). Transfer 3.0 ml of *Silver Diethyldithiocarbamate Solution* to the absorber tube, add 3.0 g of granular zinc (20-mesh) to the mixture in the flask, and immediately insert the standard-taper joint in the flask. Allow the evolution of hydrogen and color development to proceed at room temperature ($25^{\circ} \pm 3^{\circ}$) for 45 min, swirling the flask gently at 10-min intervals. (The addition of a small amount of isopropanol to the generator flask may improve the uniformity of the rate of gas evolution.) Disconnect the absorber tube from the generator and scrubber units, and transfer the *Silver Diethyldithiocarbamate Solution* to a 1-cm absorption cell. Determine the absorbance at the wavelength of maximum absorption between 535 nm and 540 nm, with a suitable spectrophotometer or colorimeter, using *Silver Diethyldithiocarbamate Solution* as the blank. The absorbance due to any red color from the solution of the sample does not exceed that produced by 3.0 ml of *Standard Arsenic Solution* (3 μg As) when treated in the same manner and under the same conditions as the sample. The room temperature during the

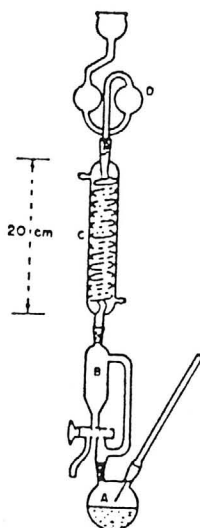


FIGURE 5 Special Apparatus for the Determination of Inorganic Arsenic (A, 250-ml distillation flask; B, receiver chamber, approximately 50-ml capacity; C, reflux condenser; D, splash head.)

B-49

generation of arsine from the standard should be held to within $\pm 2^\circ$ of that observed during the determination of the sample.

Interferences Metals or salts of metals such as chromium, cobalt, copper, mercury, molybdenum, nickel, palladium, and silver are said to interfere with the evolution of arsine. Antimony, which forms stibine, is the only metal likely to produce a positive interference in the color development with the silver diethyldithiocarbamate. Stibine forms a red color that has a maximum absorbance at 510 nm, but at 535 to 540 nm the absorbance of the antimony complex is so diminished that the results of the determination would not be altered significantly.

Ash (Acid-Insoluble)

Boil the ash obtained as directed under *Total Ash*, below, with 25 ml of diluted hydrochloric acid TS for 5 min, collect the insoluble matter on a tared Gooch crucible or ashless filter, wash with hot water, ignite, and weigh. Calculate the percentage of acid-insoluble ash from the weight of sample taken.

Ash (Total)

Unless otherwise directed, weigh accurately about 3 g of the sample in a tared crucible, ignite at a low temperature (about 550°), not to exceed very dull redness, until free from carbon, cool in a desiccator, and weigh. If a carbon-free ash is not obtained, wet the charred mass with hot water, collect the insoluble residue on an ashless filter paper, and ignite the residue and filter paper until the ash is white or nearly so. Finally, add the filtrate, evaporate it to dryness, and heat the whole to a dull redness. If a carbon-free ash is still not obtained, cool the crucible, add 15 ml of alcohol, break up the ash with a glass rod, then burn off the alcohol, again heat the whole to a dull redness, cool, and weigh.

Calcium Pantothenate Assay

Standard Stock Solution of Calcium Pantothenate Dissolve 50 mg of USP Calcium Pantothenate Reference Standard, previously dried and stored in the dark over phosphorus pentoxide and accurately weighed while protected from absorption of moisture during the weighing, in about 500 ml of water in a 1000-ml volumetric flask. Add 10 ml of 0.2 *N* acetic acid and 100 ml of sodium acetate solution (1 in 60), then add water to volume. Each ml represents 50 μ g of USP Calcium

Pantothenate Reference Standard. Store under toluene in a refrigerator.

Standard Preparation On the day of the assay, dilute a measured volume of *Standard Stock Solution of Calcium Pantothenate* with sufficient water to contain, in each ml, between 0.01 and 0.04 μ g of calcium pantothenate, the exact concentration being such that the responses obtained as directed under *Procedure*, using 2.0 and 4.0 ml of the *Standard Preparation*, are within the linear portion of the log-concentration response curve.

Assay Preparation Prepare a solution with water containing approximately the equivalent of the calcium pantothenate concentration in the *Standard Preparation*.

Basal Medium Stock Solution

Acid-Hydrolyzed Casein Solution	25	ml
Cystine-Tryptophan Solution	25	ml
Polysorbate 80 Solution	0.25	ml
Dextrose, Anhydrous	10	g
Sodium Acetate, Anhydrous	5	g
Adenine-Guanine-Uracil Solution	5	ml
Riboflavin-Thiamin Hydrochloride-Biotin Solution	5	ml
<i>para</i> -Aminobenzoic Acid-Niacin-Pyridoxine Hydrochloride Solution	5	ml
Salt Solution A	5	ml
Salt Solution B	5	ml

Dissolve the anhydrous dextrose and sodium acetate in the solutions previously mixed, and adjust to a pH of 6.8 with sodium hydroxide TS. Finally, add water to make 250 ml.

Acid-Hydrolyzed Casein Solution Mix 100 g of vitamin-free casein with 500 ml of dilute hydrochloric acid (1 in 2), and reflux the mixture for 8 to 12 h. Remove the hydrochloric acid from the mixture by distillation under reduced pressure until a thick paste remains. Redissolve the resulting paste in water, adjust the solution to a pH of 3.5 ± 0.1 with sodium hydroxide solution, and add water to make 1000 ml. Add 20 g of activated charcoal, stir for 1 h, and filter. Repeat the treatment with activated charcoal. Store under toluene in a refrigerator at a temperature not below 10°. Filter the solution if a precipitate forms under storage.

Cystine-Tryptophan Solution Suspend 4.0 g of L-cystine and 1.0 g of L-tryptophan (or 2.0 g of DL-tryptophan) in 700 to 800 ml of water, heat to 70° to 80°, and add dilute hydrochloric acid (1 in 2) dropwise, with stirring, until the solids are dissolved. Cool, and add water to make 1000 ml. Store under toluene in a refrigerator at a temperature not below 10°.

Adenine-Guanine-Uracil Solution Dissolve 200 mg each of adenine sulfate, guanine hydrochloride, and uracil, with the aid of heat, in 10 ml of dilute hydrochloric acid (1 in 3), cool, and add water to make 200 ml. Store under toluene in a refrigerator.

Salt Solution A Dissolve 25 g of monobasic potassium phosphate and 25 g of dibasic potassium phosphate in water to make 500 ml. Add 5 drops of hydrochloric acid, and store under toluene.

B-50

length, 3.5 cm in diameter, and graduated at 40, 80, and 130 ml). Wash the flask with sufficient alcohol to make a volume of 40 ml in the cylinder, and complete the transfer with warm and then cold water until the total volume is 80 ml. Finally, wash the flask with a few ml of petroleum ether, add the washings to the cylinder, cool the contents of the cylinder to room temperature, and add 50 ml of petroleum ether.

Insert the stopper, shake the cylinder vigorously for at least 1 min, and allow both layers to become clear. Siphon the upper layer as completely as possible without removing any of the lower layer, collecting the ether fraction in a 500-ml separator. Repeat the extraction and siphoning at least six times with 50-ml portions of petroleum ether, shaking vigorously each time. Wash the combined extracts, with vigorous shaking, with 25-ml portions of 10% alcohol until the wash water is neutral to phenolphthalein, and discard the washings. Transfer the ether extract to a tared beaker, and rinse the separator with 10 ml of ether, adding the rinsings to the beaker. Evaporate the ether on a steam bath just to dryness, and dry the residue to constant weight, preferably at 75° to 80° under a vacuum of not more than 200 mm of Hg, or at 100° for 30 min. Cool in a desiccator, and weigh to obtain the uncorrected weight of unsaponifiable matter.

Determine the quantity of fatty acids in the residue as follows: Dissolve the residue in 50 ml of warm alcohol (containing phenolphthalein TS and previously neutralized with sodium hydroxide to a faint pink color), and titrate with 0.02 *N* sodium hydroxide to the same color. Each ml of 0.02 *N* sodium hydroxide is equivalent to 5.659 mg of fatty acids, calculated as oleic acid.

Subtract the calculated weight of fatty acids from the weight of the residue to obtain the corrected weight of unsaponifiable matter in the sample.

VOLATILE ACIDITY

Modified Hortvet-Sellier Method

Apparatus Assemble a modified Hortvet-Sellier distillation apparatus as shown in Fig. 12, using a sufficiently large (approximately 38- × 203-mm) inner Sellier tube and large distillation trap.

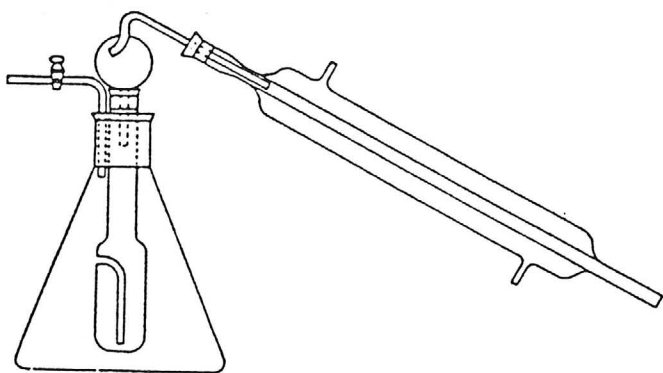


FIGURE 12 Modified Hortvet-Sellier Distillation Apparatus

Procedure Transfer the amount of sample, accurately weighed, specified in the monograph, into the inner tube of the assembly, and insert the tube in the outer flask containing about 300 ml of recently boiled hot water. To the sample add 10 ml of approximately 4 *N* perchloric acid [35 ml (60 g) of 70% perchloric acid in 100 ml of water], and connect the inner tube to a water-cooled condenser through the distillation trap. Distil by heating the outer flask so that 100 ml of distillate is collected within 20 to 25 min. Collect the distillate in 100-ml portions, add phenolphthalein TS to each portion, and titrate with 0.5 *N* sodium hydroxide. Continue the distillation until a 100-ml portion of the distillate requires no more than 0.5 ml of 0.5 *N* sodium hydroxide for neutralization. (*Caution:* Do not distil to dryness.) Calculate the weight, in mg, of volatile acids in the sample taken by the formula $V \times e$, in which *V* is the total volume, in ml, of 0.5 *N* sodium hydroxide consumed in the series of titrations and *e* is the equivalence factor given in the monograph.

Fluoride Limit Test

Method I (Thorium Nitrate Colorimetric Method)

This method should be used unless otherwise directed in the individual monograph.

Caution: When applying this test to organic compounds, the temperature at which the distillation is conducted must be rigidly controlled at all times to the recommended range of 135° to 140° to avoid the possibility of explosion.

NOTE: To minimize the distillation blank resulting from fluoride leached from the glassware, the distillation apparatus should be treated as follows: Treat the glassware with hot 10% sodium hydroxide solution, followed by flushing with tap water and rinsing with distilled water. At least once daily, treat in addition by boiling down 15 to 20 ml of dilute sulfuric acid (1 in 2) until the still is filled with fumes; cool, pour off the acid, treat again with 10% sodium hydroxide solution, and rinse thoroughly. For further details, see sections 25.050 and 25.054 in *Official Methods of Analysis of the A.O.A.C.*, Thirteenth Edition, 1980.

Unless otherwise directed, place a 5.0-g sample and 30 ml of water in a 125-ml distillation flask having a side arm and trap. The flask is connected with a condenser and carries a thermometer and a capillary tube, both of which must extend into the liquid. Slowly add, with continuous stirring, 10 ml of perchloric acid, and then add 2 or 3 drops of silver nitrate solution (1 in 2) and a few glass beads. Connect a small dropping funnel or a steam generator to the capillary tube. Support the flask on an asbestos mat with a hole that exposes about one third of the flask to the flame. Distil until the temperature reaches 135°. Add water from the funnel or introduce steam through the capillary, maintaining the temperature between 135° and 140° at

B-51

all times. Continue the distillation until 100 ml of distillate has collected. After the 100-ml portion (*Distillate A*) is collected, collect an additional 50-ml portion of distillate (*Distillate B*) to ensure that all of the fluorine has been volatilized.

Place 50 ml of *Distillate A* in a 50-ml Nessler tube. In another similar Nessler tube place 50 ml of water distilled through the apparatus as a control. Add to each tube 0.1 ml of a filtered solution of sodium alizarinsulfonate (1 in 1000) and 1 ml of freshly prepared hydroxylamine hydrochloride solution (1 in 4000), and mix well. Add, dropwise and with stirring, either 1 *N* or 0.05 *N* sodium hydroxide, depending upon the expected volume of volatile acid distilling over, to the tube containing the distillate until its color just matches that of the control, which is faintly pink. Then add to each tube 1.0 ml of 0.1 *N* hydrochloric acid, and mix well. From a buret, graduated in 0.05 ml, add slowly to the tube containing the distillate enough thorium nitrate solution (1 in 4000) so that, after mixing, the color of the liquid just changes to a faint pink. Note the volume of the solution added, then add exactly the same volume to the control, and mix. Now add to the control solution sodium fluoride TS (10 µg F per ml) from a buret to make the colors of the two tubes match after dilution to the same volume. Mix well, and allow all air bubbles to escape before making the final color comparison. Check the endpoint by adding 1 or 2 drops of sodium fluoride TS to the control. A distinct change in color should take place. Note the volume of sodium fluoride TS added.

Dilute *Distillate B* to 100 ml, and mix well. Place 50 ml of this solution in a 50-ml Nessler tube, and follow the procedure for *Distillate A*. The total volume of sodium fluoride TS added for the solutions from both *Distillate A* and *Distillate B* should not exceed 2.5 ml.

Method II (Ion-Selective Electrode Method A)

Buffer Solution Dissolve 36 g of cyclohexylenedinitrilotetraacetic acid (CDTA) in sufficient 1 *M* sodium hydroxide to make 200 ml. Transfer 20 ml of this solution (equivalent to 4 g of disodium CDTA) into a 1000-ml beaker containing 500 ml of water, 57 ml of glacial acetic acid, and 58 g of sodium chloride, and stir to dissolve. Adjust the pH of the solution to between 5.0 and 5.5 by the addition of 5 *M* sodium hydroxide, then cool to room temperature, dilute to 1000 ml with water, and mix.

Procedure Unless otherwise directed in the individual monograph, place an 8.0-g sample and 20 ml of water in a 250-ml distilling flask, cautiously add 20 ml of perchloric acid, and then add 2 or 3 drops of silver nitrate solution (1 in 2) and a few glass beads. Following the directions, and observing the *Caution* and *Note*, as given under *Method I*, distil the solution until 200 ml of distillate has been collected.

Transfer a 25.0-ml aliquot of the distillate into a 250-ml plastic beaker, and dilute to 100 ml with the *Buffer Solution*. Place the fluoride ion and reference electrodes (or a combination fluoride electrode) of a suitable ion-selective electrode apparatus (such as the Orion Model 407) in the solution, and adjust the calibration control until the indicator needle points to zero on the logarithmic concentration scale, allowing

sufficient time for equilibration (about 20 min) and stirring constantly during the equilibration period and throughout the remainder of the procedure. Pipet 1.0 ml of a solution containing 100 µg of fluoride ion (F) per ml (prepared by dissolving 22.2 mg of sodium fluoride, previously dried at 200° for 4 h, in sufficient water to make 100.0 ml) into the beaker, allow the electrode to come to equilibrium, and record the final reading on the logarithmic concentration scale. (NOTE: Follow the instrument manufacturer's instructions regarding precautions and interferences, electrode filling and check, temperature compensation, and calibration.)

Calculations Calculate the fluoride content, in ppm, of the sample taken by the formula

$$[IA/(R - I)] \times 100 \times [200/25W],$$

in which *I* is the initial scale reading before the addition of the sodium fluoride solution; *A* is the concentration, in µg per ml, of fluoride in the sodium fluoride solution added to the sample solution; *R* is the final scale reading, after addition of the sodium fluoride solution; and *W* is the original weight of the sample, in g.

Method III (Ion-Selective Electrode Method B)

Sodium Fluoride Solution (5 µg F per ml) Transfer 2.210 g of sodium fluoride, previously dried at 110° for 2 h and accurately weighed, into a 400-ml plastic beaker, add 200 ml of water, and stir until dissolved. Quantitatively transfer this solution into a 1000-ml volumetric flask with the aid of water, dilute to volume with water, and mix. Store this stock solution in a plastic bottle. On the day of use, transfer 5.0 ml of the stock solution into a 1000-ml volumetric flask, dilute to volume with water, and mix.

Calibration Curve Transfer into separate 250-ml plastic beakers 1.0, 2.0, 3.0, 5.0, 10.0, and 15.0 ml of the *Sodium Fluoride Solution*, add 50 ml of water, 5 ml of 1 *N* hydrochloric acid, 10 ml of 1 *M* sodium citrate, and 10 ml of 0.2 *M* disodium EDTA to each beaker, and mix. Transfer each solution into a 100-ml volumetric flask, dilute to volume with water, and mix. Transfer a 50-ml portion of each solution into a 125-ml plastic beaker, and measure the potential of each solution with a suitable ion-selective electrode apparatus (such as the Orion Model No. 94-09, with solid-state membrane), using a suitable reference electrode (such as the Orion Model No. 90-01, with single junction). Plot the calibration curve on two-cycle semilogarithmic paper (such as K & E No. 465130), with µg F per 100 ml solution on the logarithmic scale.

Procedure Transfer 1.00 g of the sample into a 150-ml glass beaker, add 10 ml of water, and, while stirring continuously, add 20 ml of 1 *N* hydrochloric acid slowly to dissolve the sample. Boil rapidly for 1 min, then transfer into a 250-ml plastic beaker, and cool rapidly in ice water. Add 15 ml of 1 *M* sodium citrate and 10 ml of 0.2 *M* disodium EDTA, and mix. Adjust the pH to 5.5 ± 0.1 with 1 *N* hydrochloric acid or 1 *M* sodium hydroxide, if necessary, then transfer into a 100-ml volumetric flask, dilute to volume with water, and mix. Transfer a 50-ml portion of this solution into a 125-ml plastic beaker,

B-52

and measure the potential of the solution with the apparatus described under *Calibration Curve*. Determine the fluoride content, in μg , of the sample from the *Calibration Curve*.

Method IV (Ion-Selective Electrode Method C)

Buffer Solution A Add 2 volumes of 6 *N* acetic acid to 1 volume of water, and adjust the pH to 5.0 with 50% potassium hydroxide solution.

Buffer Solution B Dissolve 150 g of sodium citrate dihydrate and 10.3 g of disodium EDTA dihydrate in 800 ml of water, adjust the pH to 8.0 with 50% sodium hydroxide solution, and dilute to 1000 ml with water.

Buffer Solution C Dissolve 36 g of cyclohexylene dinitrilotetraacetic acid (CDTA) in sufficient 1 *N* sodium hydroxide to make 200 ml by boiling, then cool, and filter through glass-fiber filter paper. Pipet 30 ml of this solution into a mixture consisting of 750 ml of water, 87 g of sodium chloride, and 85.5 ml of glacial acetic acid. Adjust the pH to between 5.0 and 5.5 by the addition of 50% sodium hydroxide solution, then cool, and dilute to 3000 ml with water.

Fluoride Standard Solution Use a solution containing 100 μg of fluoride ion (F) per ml (100 ppm), obtained commercially or prepared by dissolving 22.2 mg of sodium fluoride, previously dried at 200° for 4 h, in sufficient water to make 100.0 ml.

Sample Preparation Weigh accurately the amount of sample specified in the monograph, transfer it into a 100-ml volumetric flask, and dissolve it in a minimum amount of water or in the volume of hydrochloric acid solution specified in the monograph. Add 50.0 ml of the appropriate buffer solution, *A*, *B*, or *C*, as specified in the monograph, dilute to volume with water, and mix.

Procedure Pipet a 50-ml aliquot of the *Sample Preparation* into a plastic beaker, and place in the solution the fluoride ion and reference electrodes (or a combination fluoride electrode) of a suitable ion-selective electrode apparatus with magnetic stirrer (Orion Model 407 or equivalent). Begin stirring slowly, and set the slope of the meter to 100% and the temperature control to room temperature, which should be the temperature of the solution. Adjust the calibration control to read infinity on the increment logarithmic scale, and allow the instrument to equilibrate. (NOTE: The ion-selective electrode responds much slower than does a pH electrode, and a stable reading may not be obtained until 2 or 3 min. The reading should not change for 30 to 60 s.) Add the volume, accurately measured, of *Fluoride Standard Solution* specified in the monograph, allow the electrode to equilibrate with continued stirring, and take the final reading on the increment logarithmic scale, recording the value obtained as *S*. Perform a blank determination using 50 ml of the same buffer solution as used for the sample under analysis, and record the value obtained as *B*.

Calculation Determine the Δ value by the formula ($V \times$

$C/50$, in which *V* is the volume of *Fluoride Standard Solution* added, in ml; *C* is the exact concentration of the *Fluoride Standard Solution*, in ppm; and 50 is the ml of *Sample Preparation* used. Calculate the ppm of fluoride (F) in the sample by the formula

$$[(S \times \Delta) - B] \times (100/W),$$

in which *W* is the weight of sample taken, in g.

Heavy Metals Test

This test is designed to limit the content of common metallic impurities that are colored by sulfide ion (Ag, As, Bi, Cd, Cu, Hg, Pb, Sb, Sn) under the specified test conditions. It demonstrates that the test substance is not grossly contaminated by such heavy metals and, within the precision of the test, that it does not exceed the *Heavy Metals* limit given in the individual monograph, as determined by concomitant visual comparison with a control solution. It has been found that, in the specified pH range, the optimum concentration of lead ion (Pb) for matching purposes by this method is 20 μg in 50 ml of solution.

Determine the amount of heavy metals by *Method I*, unless otherwise specified in the individual monograph. *Method I* is used for substances that yield clear, colorless solutions prior to addition of sulfide ion. *Method II* is used for those substances that do not yield clear, colorless solutions under the test conditions specified for *Method I*, or for substances that, by virtue of their complex nature, interfere with the precipitation of metals by sulfide ion. *Method III*, a wet digestion method, is used only in those cases where neither *Method I* nor *Method II* can be used.

Special Reagents

Ammonia TS Dilute 400 ml of ACS reagent-grade ammonium hydroxide to 1000 ml with water.

Hydrochloric Acid, Sulfuric Acid, Nitric Acid, 30% Hydrogen Peroxide Use ACS reagent-grade chemicals.

Lead Nitrate Stock Solution Dissolve 159.8 mg of ACS reagent-grade lead nitrate, $\text{Pb}(\text{NO}_3)_2$, in 100 ml of water containing 1 ml of nitric acid, dilute with water to 1000.0 ml, and mix. Prepare and store this solution in glass containers that are free from lead salts.

Standard Lead Solution On the day of use, dilute 10.0 ml of *Lead Nitrate Stock Solution* with water to 100.0 ml. Each ml of *Standard Lead Solution* contains the equivalent of 10 μg of lead ion (Pb).

Procedure

NOTE: In the following procedures, failure to adjust accurately the pH of the solution within the specified limits may result in a significant loss of test sensitivity.

B-53

Method I

Solution A Pipet 2.0 ml of *Standard Lead Solution* (20 µg of lead) into a 50-ml color-comparison tube, and add water to make 25 ml. Adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) by addition of diluted acetic acid TS or ammonia TS, dilute with water to 40 ml, and mix.

Solution B Place 25 ml of the solution prepared as directed in the individual monograph in a 50-ml color-comparison tube that matches the one used for *Solution A*, adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) by addition of diluted acetic acid TS or ammonia TS, dilute with water to 40 ml, and mix.

Solution C Into a third color-comparison tube that matches those used for *Solutions A* and *B*, place 25 ml of the solution prepared as directed in the individual monograph, and add 2.0 ml of *Standard Lead Solution*. Adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) by addition of diluted acetic acid TS or ammonia TS, dilute with water to 40 ml, and mix.

To each tube add 10 ml of freshly prepared hydrogen sulfide TS, mix, allow to stand for 5 min, and view downward over a white surface. The color of *Solution B* is not darker than that of *Solution A*, and the intensity of the color of *Solution C* is equal to or greater than that of *Solution A*. If the color of *Solution C* is lighter than that of *Solution A*, the test substance is providing an interference with the test procedure and *Method II* must be used for the substance under examination.

Method II

Solution A Prepare as directed under *Method I*.

Solution B Place the quantity, accurately weighed, of sample specified in the individual monograph in a suitable crucible, add sufficient sulfuric acid to wet the sample, and carefully ignite at a low temperature until thoroughly charred, covering the crucible loosely with a suitable lid during the ignition. After the substance is thoroughly carbonized, add 2 ml of nitric acid and 5 drops of sulfuric acid, cautiously heat until white fumes are evolved, then ignite, preferably in a muffle furnace, at 500° to 600° until all of the carbon is burned off. Cool, add 4 ml of dilute hydrochloric acid (1 in 2), cover, and digest on a steam bath for 10 to 15 min. Uncover, and slowly evaporate on a steam bath to dryness. Moisten the residue with 1 drop of hydrochloric acid, add 10 ml of hot water, and digest for 2 min. Add ammonia TS dropwise until the solution is just alkaline to litmus paper, dilute with water to 25 ml, and adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) by addition of diluted acetic acid TS. Filter if necessary, rinse the crucible and the filter with 10 ml of water, transfer the solution and rinsings to a 50-ml color-comparison tube, dilute with water to 40 ml, and mix.

To each tube add 10 ml of freshly prepared hydrogen sulfide TS, mix, allow to stand for 5 min, and view downward over a white surface. The color of *Solution B* is not darker than that of *Solution A*.

Method III

Solution A Transfer a mixture of 8 ml of sulfuric acid and 10 ml of nitric acid into a 100-ml Kjeldahl flask, clamp the flask

at an angle of 45°, and then add, in small increments, an additional volume of nitric acid equal to that added in the preparation of *Solution B*, below. Heat the solution to dense, white fumes, cool, and cautiously add 10 ml of water. Add a volume of 30% hydrogen peroxide equal to that added in the preparation of *Solution B*, below, then boil gently to dense, white fumes, and cool. Cautiously add 5 ml of water, mix, and boil gently to dense, white fumes. Continue boiling until the volume is reduced to about 2 or 3 ml, then cool, and dilute cautiously with a few ml of water. Into this solution pipet 2.0 ml of *Standard Lead Solution*, and mix. Transfer into a 50-ml color-comparison tube, rinse the flask with water, adding the rinsings to the tube until the volume is 25 ml, and mix. Adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) with stronger ammonia TS initially, and then with ammonia TS as the desired range is neared, dilute with water to 40 ml, and mix.

Solution B Transfer into a 100-ml Kjeldahl flask (or into a 300-ml flask if the reaction foams excessively) the quantity, accurately weighed, of sample specified in the individual monograph, clamp the flask at an angle of 45°, and then add sufficient of a mixture of 8 ml of sulfuric acid and 10 ml of nitric acid to moisten the sample thoroughly. (NOTE: For liquid samples use 3 ml of the acid mixture.) Warm gently until the reaction commences, allow the reaction to subside, and then add additional portions of the acid mixture, heating after each addition, until all of the 18 ml of acid mixture has been added. Increase the heat, and boil gently until the reaction mixture darkens. Remove the flask from the heat, add 2 ml of nitric acid, and heat to boiling again. Continue the intermittent heating and addition of 2-ml portions of nitric acid until no further darkening occurs, then heat strongly to dense, white fumes, and cool. Cautiously add 5 ml of water, mix, boil gently to dense, white fumes, and continue heating until the volume is reduced to about 2 or 3 ml. Cool, cautiously add 5 ml of water, and examine. If the solution is yellow-colored, cautiously add 1 ml of 30% hydrogen peroxide, and again evaporate to dense, white fumes and to a volume of about 2 or 3 ml. Cool, dilute cautiously with a few ml of water, and mix. Transfer into a 50-ml color-comparison tube, rinse the flask with water, adding the rinsings to the tube until the volume is 25 ml, and mix. Adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) with stronger ammonia TS initially, and then with ammonia TS as the desired range is neared, dilute with water to 40 ml, and mix.

To each tube add 10 ml of freshly prepared hydrogen sulfide TS, mix, allow to stand for 5 min, and view downward over a white surface. The color of *Solution B* is not darker than that of *Solution A*.

B-54

Lead Limit Test

Special Reagents Select reagents having as low a lead content as practicable, and store all solutions in containers of borosilicate glass. Rinse all glassware thoroughly with warm dilute nitric acid (1 in 2) followed by water.

Ammonia-Cyanide Solution Dissolve 2 g of potassium cyanide in 15 ml of stronger ammonia TS, and dilute with water to 100 ml.

Ammonium Citrate Solution Dissolve 40 g of citric acid in 90 ml of water, add 2 or 3 drops of phenol red TS, then cautiously add stronger ammonia TS until the solution acquires a reddish color. Extract it with 20-ml portions of *Dithizone Extraction Solution* until the dithizone solution retains its green color or remains unchanged.

Diluted Standard Lead Solution (1 µg Pb in 1 ml) Immediately before use, transfer 10.0 ml of *Standard Lead Solution*, page 512, containing 10 µg of lead per ml, to a 100-ml volumetric flask, dilute to volume with dilute nitric acid (1 in 100), and mix.

Dithizone Extraction Solution Dissolve 30 mg of dithizone in 1000 ml of chloroform, add 5 ml of alcohol, and mix. Store in a refrigerator. Before use, shake a suitable volume of the solution with about half its volume of dilute nitric acid (1 in 100), discarding the nitric acid. Do not use if more than 1 month old.

Hydroxylamine Hydrochloride Solution Dissolve 20 g of hydroxylamine hydrochloride in sufficient water to make about 65 ml, transfer the solution to a separator, add a few drops of thymol blue TS, then add stronger ammonia TS until the solution assumes a yellow color. Add 10 ml of sodium diethyldithiocarbamate solution (1 in 25), mix, and allow to stand for 5 min. Extract the solution with successive 10- to 15-ml portions of chloroform until a 5-ml test portion of the chloroform extract does not assume a yellow color when shaken with a dilute cupric sulfate solution. Add diluted hydrochloric acid TS until the extracted solution is pink, adding 1 or 2 drops more of thymol blue TS if necessary, then dilute with water to 100 ml, and mix.

Potassium Cyanide Solution Dissolve 50 g of potassium cyanide in sufficient water to make 100 ml. Remove the lead from the solution by extraction with successive portions of *Dithizone Extraction Solution* as described under *Ammonium Citrate Solution*, then extract any dithizone remaining in the cyanide solution by shaking with chloroform. Finally, dilute the cyanide solution with sufficient water so that each 100 ml contains 10 g of potassium cyanide.

Standard Dithizone Solution Dissolve 10 mg of dithizone in 1000 ml of chloroform, keeping the solution in a glass-stoppered lead-free bottle suitably wrapped to protect it from light and stored in a refrigerator.

Sample Solution The solution obtained by treating the sample as directed in an individual monograph is used directly as the *Sample Solution* in the *Procedure*. Sample solutions of organic compounds are prepared, unless otherwise directed, according to the following general method:

Caution: Some substances may react unexpectedly with

explosive violence when digested with hydrogen peroxide. Appropriate safety precautions must be employed at all times.

Transfer 1.0 g of the sample into a suitable flask, add 5 ml of sulfuric acid and a few glass beads, and digest at a temperature not exceeding 120° until charring begins, using preferably a hot plate in a fume hood. (Additional sulfuric acid may be necessary to completely wet some samples, but the total volume added should not exceed about 10 ml.) After the sample has been initially decomposed by the acid, add with caution, dropwise, 30% hydrogen peroxide, allowing the reaction to subside and reheating between drops. The first few drops must be added very slowly with sufficient mixing to prevent a rapid reaction, and heating should be discontinued if foaming becomes excessive. Swirl the solution in the flask to prevent unreacted substance from caking on the walls or bottom of the flask during the digestion. Add small quantities of the peroxide when the solution begins to darken, and continue the digestion until the organic matter is destroyed, gradually raising the temperature of the hot plate to 250°–300° until fumes of sulfur trioxide are copiously evolved and the solution becomes colorless or retains only a light straw color. Cool, add cautiously 10 ml of water, again evaporate to strong fuming, and cool. Quantitatively transfer the solution into a separator with the aid of small quantities of water.

Procedure Transfer the *Sample Solution*, prepared as directed in the individual monograph, into a separator, and, unless otherwise directed, add 6 ml of *Ammonium Citrate Solution* and 2 ml of *Hydroxylamine Hydrochloride Solution*. (Use 10 ml of the citrate solution when determining lead in iron salts.) To the separator add 2 drops of phenol red TS, and make the solution just alkaline (red in color) by the addition of stronger ammonia TS. Cool the solution, if necessary, under a stream of tap water, then add 2 ml of *Potassium Cyanide Solution*. Immediately extract the solution with 5-ml portions of *Dithizone Extraction Solution*, draining each extract into another separator, until the dithizone solution retains its green color. Shake the combined dithizone solutions for 30 s with 20 ml of dilute nitric acid (1 in 100), discard the chloroform layer, add to the acid solution 5.0 ml of *Standard Dithizone Solution* and 4 ml of *Ammonia-Cyanide Solution*, and shake for 30 s. The purplish hue in the chloroform solution of the sample due to any lead dithizonate present does not exceed that in a control, containing the volume of *Diluted Standard Lead Solution* equivalent to the amount of lead specified in the monograph, when treated in the same manner as the sample.

Loss on Drying

This procedure is used to determine the amount of volatile matter expelled under the conditions specified in the monograph. Since the volatile matter may include material other than adsorbed moisture, this test is designed for compounds in which the loss on drying may not definitely be attributable to water

B-55

one. For substances appearing to contain water as the only volatile constituent, the *Karl Fischer Titrimetric Method* provided under *Water*, page 552, is usually appropriate.

Procedure Unless otherwise directed in the monograph, conduct the determination on 1 to 2 g of the substance, previously mixed and accurately weighed. If the sample is in the form of large crystals, reduce the particle size to about 2 mm, quickly crushing to avoid absorption or loss of moisture. Tare a glass-stoppered shallow weighing bottle that has been dried for 30 min under the same conditions to be observed in the determination. Transfer the sample to the bottle, replace the cover, and weigh the bottle and its contents. By gentle sidewise shaking distribute the sample as evenly as possible to a depth of about 5 mm for most substances and not over 10 mm in the case of bulky materials. Place the loaded bottle in the drying chamber, removing the stopper and leaving it also in the chamber, and dry at the temperature and for the length of time specified. Upon opening the chamber, close the bottle promptly and allow it to come to room temperature, preferably in a desiccator, before weighing.

If the test substance melts at a temperature lower than that specified for the determination, preheat the bottle and its contents for 1 to 2 h at a temperature 5° to 10° below the melting range, then continue drying at the specified temperature for the determination. When drying in a desiccator, exercise particular care to ensure that the desiccant is kept fully effective by frequent replacement.

Melting Range or Temperature

For purposes of this Codex, the melting range or temperature of a solid is defined as those points of temperature within which or the point at which the solid coalesces and is completely melted, when determined as directed below. Any apparatus or method capable of equal accuracy may be used. The accuracy should be checked frequently by the use of one or more of the six USP Melting Point Reference Standards, preferably the one that melts nearest the melting temperature of the compound to be tested.

Five procedures for the determination of melting range or temperature are given herein, varying in accordance with the nature of the substance. When no class is designated in the monograph, use the procedure for *Class I*.

The procedure known as the mixed melting point determination, whereby the melting range of a solid under test is compared with that of an intimate mixture of equal parts of the solid and an authentic specimen of it, may be used as a confirmatory identification test. Agreement of the observations on the original and the mixture usually constitutes reliable evidence of chemical identity.

Apparatus The melting range apparatus consists of a glass container for a bath of colorless fluid, a suitable stirring device, an accurate thermometer (see page 547), and a controlled

source of heat. The bath fluid is selected with a view to the temperature required, but light paraffin is used generally, and certain liquid silicones are well adapted to the higher temperature ranges. The fluid is deep enough to permit immersion of the thermometer to its specified immersion depth so that the bulb is still about 2 cm above the bottom of the bath. The heat may be supplied by an open flame or electrically. The capillary tube is about 10 cm long and 0.8 to 1.2 mm in internal diameter with walls 0.2 to 0.3 mm in thickness.

The thermometer is preferably one that conforms to the specifications provided under *Thermometers*, page 547, selected for the desired accuracy and range of temperature.

Procedure for Class I Reduce the sample to a very fine powder, and, unless otherwise directed, render it anhydrous when it contains water of hydration by drying it at the temperature specified in the monograph, or, when the substance contains no water of hydration, dry it over a suitable desiccant for 16 to 24 h.

Charge a capillary glass tube, one end of which is sealed, with sufficient of the dry powder to form a column in the bottom of the tube 2.5 to 3.5 mm high when packed down as closely as possible by moderate tapping on a solid surface.

Heat the bath until a temperature approximately 30° below the expected melting point is reached, attach the capillary tube to the thermometer, and adjust its height so that the material in the capillary is level with the thermometer bulb. Return the thermometer to the bath, continue the heating, with constant stirring, at a rate of rise of approximately 3° per min until a temperature 3° below the expected melting point is attained, then carefully regulate the rate to about 1° to 2° per min until melting is complete.

The temperature at which the column of the sample is observed to collapse definitely against the side of the tube at any point is defined as the beginning of melting, and the temperature at which the sample becomes liquid throughout is defined as the end of melting. The two temperatures fall within the limits of the melting range.

Procedure for Class Ia Prepare the sample and charge the capillary glass tube as directed for *Class I*. Heat the bath until a temperature 10° ± 1° below the expected melting range is reached, then introduce the charged tube, and heat at a rate of rise of 3° ± 0.5° per min until melting is complete. Record the melting range as for *Class I*.

Procedure for Class Ib Place the sample in a closed container, and cool to 10°, or lower, for at least 2 h. Without previous powdering, charge the cooled material into the capillary tube as directed for *Class I*, then immediately place the charged tube in a vacuum desiccator and dry at a pressure not exceeding 20 mm of Hg for 3 h. Immediately upon removal from the desiccator, fire-seal the open end of the tube, and as soon as practicable proceed with the determination of the melting range as follows: Heat the bath until a temperature of 10° ± 1° below the expected melting range is reached, then introduce the charged tube, and heat at a rate of rise of 3° ± 0.5° per min until melting is complete. Record the melting range as directed in *Class I*.

If the particle size of the material is too large for the capillary, precool the sample as directed above, then with as

VISCOSITY

Unless otherwise directed in the individual monograph, transfer the sample into an 8-oz wide-mouth glass jar, 10.8 cm (4.25 in.) high and 5.7 cm (2.75 in.) in inside diameter, equipped with a screw lid. Condition the sample in a water bath at $25^{\circ} \pm 0.2^{\circ}$ for 2 h (± 5 min), taking care to prevent water from coming into contact with the sample. Insert a No. 4 spindle in a Brookfield Model RVF viscometer,* or equivalent, and move the jar into place under the spindle, adjusting the elevation of the jar so that the upper surface of the sample is in the center of the shaft indentation and the spindle is in the center of the jar. (NOTE: The viscometer must be kept level at all times during the test procedure.) Set the viscometer to rotate at 20 rpm, and allow the spindle to rotate until a constant dial reading is obtained. The viscosity, in poises, is the dial reading on the 100 scale, or the dial reading on the 500 scale divided by 5.

Selenium Limit Test

Reagents and Solutions

2,3-Diaminonaphthalene Solution On the day of use, dissolve 100 mg of 2,3-diaminonaphthalene ($C_{10}H_{10}N_2$) and 500 mg of hydroxylamine hydrochloride ($NH_2OH \cdot HCl$) in sufficient 0.1 N hydrochloric acid to make 100 ml.

Selenium Stock Solution Transfer 40.0 mg of powdered metallic selenium into a 1000-ml volumetric flask, and dissolve 100 ml of dilute nitric acid (1 in 2), warming gently on a steam bath to effect solution. Cool, dilute with water to volume, and mix.

Selenium Standard Solution Pipet 5.0 ml of *Selenium Stock Solution* into a 200-ml volumetric flask, dilute to volume with water, and mix. Each ml of this solution contains the equivalent of 1 μ g of selenium (Se).

Method I

Standard Preparation Pipet 6.0 ml of *Selenium Standard Solution* into a 150-ml beaker, add 50 ml of 0.25 N nitric acid, and mix.

Sample Preparation Using a 1000-ml combustion flask and 25 ml of 0.5 N nitric acid as the absorbing liquid, proceed as directed under *Oxygen Flask Combustion*, page 531, using the amount of sample specified in the individual monograph (and the magnesium oxide or other reagent, where specified). (NOTE: If the sample contains water of hydration or more than 1% of moisture, dry it at 140° for 2 h before combustion, unless otherwise directed.) Upon completion of combustion, place a few ml of water in the cup or lip of the combustion flask, loosen the stopper of the flask, and rinse the stopper, sample holder, and sides of the flask with about 10 ml of water. Transfer the

solution, with the aid of about 20 ml of water, into a 150-ml beaker. Heat gently to boiling, boil for 10 min, and cool.

Procedure Treat the *Sample Preparation*, the *Standard Preparation*, and 50 ml of 0.25 N nitric acid, to serve as the blank, similarly and in parallel as follows: Add dilute ammonium hydroxide (1 in 2) to adjust the pH of the solution to 2.0 ± 0.2 . Dilute with water to 60.0 ml, and transfer to a low-actinic separator with the aid of 10.0 ml of water, adding the 10.0 ml of rinsings to the separator. Add 200 mg of hydroxylamine hydrochloride, swirl to dissolve, immediately add 5.0 ml of 2,3-Diaminonaphthalene Solution, insert the stopper, and swirl to mix. Allow the solution to stand at room temperature for 100 min. Add 5.0 ml of cyclohexane, shake vigorously for 2 min, and allow the layers to separate. Discard the aqueous phases, and centrifuge the cyclohexane extracts to remove any traces of water. Determine the absorbance of each extract in a 1-cm cell at the maximum at about 380 nm with a suitable spectrophotometer, using the blank to set the instrument. The absorbance of the extract from the *Sample Preparation* is not greater than that from the *Standard Preparation* when a 200-mg sample is tested, or not greater than one-half the absorbance of the extract from the *Standard Preparation* when a 100-mg sample is tested.

Method II

Standard Preparation Pipet 6.0 ml of *Selenium Standard Solution* into a 150-ml beaker, add 50 ml of 2 N hydrochloric acid, and mix.

Sample Preparation Transfer the amount of sample specified in the individual monograph into a 150-ml beaker, dissolve in 25 ml of 4 N hydrochloric acid, swirling if necessary to effect solution, heat gently to boiling, and digest on a steam bath for 15 min. Remove from heat, add 25 ml of water, and allow to cool to room temperature.

Procedure Place the beakers containing the *Standard Preparation* and the *Sample Preparation* in a fume hood, and to a third beaker add 50 ml of 2 N hydrochloric acid to serve as the blank. Cautiously add 5 ml of ammonium hydroxide to each beaker, mix, and allow the solution to cool. Treat each solution, similarly and in parallel, as directed under *Procedure in Method I*, beginning with "Add dilute ammonium hydroxide (1 in 2). . . ."

Sieve Analysis of Granular Metal Powders

(Based on ASTM Designation: B 214)

Apparatus

Sieves Use a set of standard sieves, ranging from +80 mesh to -325 mesh, conforming to the specifications in ASTM Designation: E 11 (Sieves for Testing Purposes).

Sieve Shaker Use a mechanically operated sieve shaker that imparts to the set of sieves a horizontal rotary motion of

Standard Solutions for the Preparation of Controls and Standards

The following solutions are used in tests for impurities that require the comparison of the color or turbidity produced in a solution of the test substance with that produced by a known amount of the impurity in a control. Directions for the preparation of other standard solutions are given in the monographs or under the general tests in which they are required (see also *Index*).

Ammonium Standard Solution (10 μg NH_4 in 1 ml) Dissolve 296.0 mg of ammonium chloride, NH_4Cl , in sufficient water to make 100.0 ml, and mix. Transfer 10.0 ml of this solution into a 1000-ml volumetric flask, dilute to volume with water, and mix.

Barium Standard Solution (100 μg Ba in 1 ml) Dissolve 177.9 mg of barium chloride, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, in water in a 1000-ml volumetric flask, dilute to volume with water, and mix.

Iron Standard Solution (10 μg Fe in 1 ml) Dissolve 702.2 mg of ferrous ammonium sulfate, $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$, in 10 ml of diluted sulfuric acid TS in a 100-ml volumetric flask, dilute to volume with water, and mix. Transfer 10.0 ml of this solution into a 1000-ml volumetric flask, add 10 ml of diluted sulfuric acid TS, dilute to volume with water, and mix.

Magnesium Standard Solution (50 μg Mg in 1 ml) Dissolve 50.0 mg of magnesium metal, Mg, in 1 ml of hydrochloric acid in a 1000-ml volumetric flask, dilute to volume with water, and mix.

Phosphate Standard Solution (10 μg PO_4 in 1 ml) Dissolve 143.3 mg of monobasic potassium phosphate, KH_2PO_4 , in water in a 100-ml volumetric flask, dilute to volume with water, and mix. Transfer 10.0 ml of this solution into a 1000-ml volumetric flask, dilute to volume with water, and mix.

Test Solutions (TS) and Other Reagents

Certain of the following test solutions are intended for use as acid-base indicators in volumetric analyses. Such solutions should be so adjusted that when 0.15 ml of the indicator solution is added to 25 ml of carbon dioxide-free water, 0.25 ml of 0.02 *N* acid or alkali, respectively, will produce the characteristic color change.

In general, the directive to prepare a solution "fresh" indicates that the solution is of limited stability and must be prepared on the day of use.

Acetic Acid TS, Diluted A solution containing about 6% (w/v) of CH_3COOH . Prepare by diluting 60.0 ml of glacial

acetic acid, or 166.6 ml of 36% acetic acid (6 *N*), with sufficient water to make 1000 ml.

Alcohol (*Ethanol; Ethyl Alcohol; $\text{C}_2\text{H}_5\text{OH}$*) Use ACS reagent-grade *Ethyl Alcohol* (not less than 95.0%, by volume, of $\text{C}_2\text{H}_5\text{OH}$). (NOTE: For use in assays and tests involving ultraviolet spectrophotometry, use ACS reagent-grade *Ethyl Alcohol Suitable for Use in Ultraviolet Spectrophotometry*.)

Alcohol, Absolute (*Anhydrous Alcohol; Dehydrated Alcohol*) Use ACS reagent-grade *Ethyl Alcohol, Absolute* (not less than 99.5%, by volume, of $\text{C}_2\text{H}_5\text{OH}$).

Alcohol, Diluted A solution containing 41.0% to 42.0%, by weight, corresponding to 48.4% to 49.5%, by volume, at 15.56°, of $\text{C}_2\text{H}_5\text{OH}$.

Alcohol, 70% (at 15.56°) A 38.6:15 mixture (v/v) of 95% alcohol and water, having a specific gravity of 0.884 at 25°. To prepare 100 ml, dilute 73.7 ml of alcohol to 100 ml with water at 25°.

Alcohol, 80% (at 15.56°) A 45.5:9.5 mixture (v/v) of 95% alcohol and water, having a specific gravity of 0.857 at 25°. To prepare 100 ml, dilute 84.3 ml of alcohol to 100 ml with water at 25°.

Alcohol, 90% (at 15.56°) A 51:3 mixture (v/v) of 95% alcohol and water, having a specific gravity of 0.827 at 25°. To prepare 100 ml, dilute 94.8 ml of alcohol to 100 ml with water at 25°.

Alcohol, Aldehyde-Free Dissolve 2.5 g of lead acetate in 5 ml of water, add the solution to 1000 ml of alcohol contained in a glass-stoppered bottle, and mix. Dissolve 5 g of potassium hydroxide in 25 ml of warm alcohol, cool, and add slowly, without stirring, to the alcoholic solution of lead acetate. Allow to stand for 1 h, then shake the mixture vigorously, allow to stand overnight, decant the clear liquid, and recover the alcohol by distillation. Ethyl Alcohol FCC, Alcohol USP, or USSD #3A or #30 may be used. If the titration of a 250-ml sample of the alcohol by *Hydroxylamine Hydrochloride Solution* (see page 561) does not exceed 0.25 ml of 0.5 *N* alcoholic potassium hydroxide, the above treatment may be omitted.

Alcoholic Potassium Hydroxide TS See *Potassium Hydroxide TS, Alcoholic*.

Alkaline Cupric Tartrate TS (*Fehling's Solution*) See *Cupric Tartrate TS, Alkaline*.

Alkaline Mercuric-Potassium Iodide TS (*Nessler's Reagent*) See *Mercuric-Potassium Iodide TS, Alkaline*.

Ammonia-Ammonium Chloride Buffer TS (approximately pH 10) Dissolve 67.5 g of ammonium chloride, NH_4Cl , in water, add 570 ml of ammonium hydroxide (28%), and dilute with water to 1000 ml.

Ammonia TS A solution containing between 9.5% and 10.5%

of NH_3 . Prepare by diluting 400 ml of ammonium hydroxide (28%) with sufficient water to make 1000 ml.

Ammonia TS, Stronger (*Ammonium Hydroxide, 28%, Stronger Ammonia Water*) A practically saturated solution of ammonia in water, containing between 28% and 30% of NH_3 .

Ammoniacal Silver Nitrate TS Add ammonia TS, dropwise, to a 1 in 20 solution of silver nitrate until the precipitate that first forms is almost, but not entirely, dissolved. Filter the solution, and store in a dark bottle.

Caution: Ammoniacal silver nitrate TS forms explosive compounds on standing. Do not store this solution, but prepare a fresh quantity for each series of determinations. Neutralize the excess reagent and rinse all glassware with hydrochloric acid immediately after completing a test.

Ammonium Acetate TS Dissolve 10 g of ammonium acetate, $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$, in sufficient water to make 100 ml.

Ammonium Carbonate TS Dissolve 20 g of ammonium carbonate and 20 ml of ammonia TS in sufficient water to make 100 ml.

Ammonium Chloride TS Dissolve 10.5 g of ammonium chloride, NH_4Cl , in sufficient water to make 100 ml.

Ammonium Molybdate TS Dissolve 6.5 g of finely powdered molybdic acid (85%) in a mixture of 14 ml of water and 14.5 ml stronger ammonia TS. Cool the solution, and add it slowly, with stirring, to a well-cooled mixture of 32 ml of nitric acid and 40 ml of water. Allow to stand for 48 h, and filter through a fine-porosity sintered-glass crucible lined at the bottom with a layer of glass wool. This solution deteriorates upon standing and is unsuitable for use if, upon the addition of 2 ml of sodium phosphate TS to 5 ml of the solution, an abundant yellow precipitate does not form at once or after slight warming. Store it in the dark. If a precipitate forms during storage, use only the clear, supernatant solution.

Ammonium Oxalate TS Dissolve 3.5 g of ammonium oxalate, $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$, in sufficient water to make 100 ml.

Ammonium Sulfanilate TS To 2.5 g of sulfanilic acid add 15 ml of water and 3 ml of ammonia TS, and mix. Add, with stirring, more ammonia TS, if necessary, until the acid dissolves, adjust the pH of the solution to about 4.5 with diluted hydrochloric acid TS, using bromocresol green TS as an outside indicator, and dilute to 25 ml.

Ammonium Sulfide TS Saturate ammonia TS with hydrogen sulfide, H_2S , and add two thirds of its volume of ammonia TS. Residue upon ignition: not more than 0.05%. The solution is not rendered turbid either by magnesium sulfate TS or by calcium chloride TS (*carbonate*). This solution is unsuitable for use if an abundant precipitate of sulfur is present. Store it in small, well-filled, dark amber-colored bottles in a cold, dark place.

Ammonium Thiocyanate TS Dissolve 8 g of ammonium thiocyanate, NH_4SCN , in sufficient water to make 100 ml.

Antimony Trichloride TS Dissolve 20 g of antimony trichloride, SbCl_3 , in chloroform to make 100 ml. Filter if necessary.

Barium Chloride TS Dissolve 12 g of barium chloride, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, in sufficient water to make 100 ml.

Barium Diphenylamine Sulfonate TS Dissolve 300 mg of *p*-diphenylamine sulfonic acid barium salt in 100 ml of water.

Benedict's Qualitative Reagent See *Cupric Citrate TS, Alkaline*.

Benzidine TS Dissolve 50 mg of benzidine in 10 ml of glacial acetic acid, dilute to 100 ml with water, and mix.

Bismuth Nitrate, TS Reflux 5 g of bismuth nitrate, $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, with 7.5 ml of nitric acid and 10 ml of water until dissolved, cool, filter, and dilute to 250 ml with water.

Bromine TS (*Bromine Water*) A saturated solution of bromine, prepared by agitating 2 to 3 ml of bromine, Br_2 , with 100 ml of cold water in a glass-stoppered bottle, the stopper of which should be lubricated with petrolatum. Store it in a cold place protected from light.

Bromocresol Blue TS Use *Bromocresol Green TS*.

Bromocresol Green TS Dissolve 50 mg of bromocresol green in 100 ml of alcohol, and filter if necessary.

Bromocresol Purple TS Dissolve 250 mg of bromocresol purple in 20 ml of 0.5 *N* sodium hydroxide, and dilute with water to 250 ml.

Bromophenol Blue TS Dissolve 100 mg of bromophenol blue in 100 ml of dilute alcohol (1 in 2), and filter if necessary.

Bromothymol Blue TS Dissolve 100 mg of bromothymol blue in 100 ml of dilute alcohol (1 in 2), and filter if necessary.

Calcium Chloride TS Dissolve 7.5 g of calcium chloride, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, in sufficient water to make 100 ml.

Calcium Hydroxide TS A solution containing approximately 140 mg of $\text{Ca}(\text{OH})_2$ in each 100 ml. To prepare, add 3 g of calcium hydroxide, $\text{Ca}(\text{OH})_2$, to 1000 ml of water, and agitate the mixture vigorously and repeatedly during 1 h. Allow the excess calcium hydroxide to settle, and decant or draw off the clear, supernatant liquid.

Carr-Price Reagent See *Antimony Trichloride TS*.

Ceric Ammonium Nitrate TS Dissolve 6.25 g of ceric ammonium nitrate, $(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6$, in 100 ml of 0.25 *N* nitric acid. Prepare the solution fresh every third day.

Chlorine TS (Chlorine Water) A saturated solution of chlorine in water. Place the solution in small, completely filled, light-resistant containers. Chlorine TS, even when kept from light and air, is apt to deteriorate. Store it in a cold, dark place. For full strength, prepare this solution fresh.

Chromotropic Acid TS Dissolve 50 mg of chromotropic acid or its sodium salt in 100 ml of 75% sulfuric acid (made by adding cautiously 75 ml of 95% to 98% sulfuric acid to 33.3 ml of water).

Cobaltous Chloride TS Dissolve 2 g of cobaltous chloride, $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, in 1 ml of hydrochloric acid and sufficient water to make 100 ml.

Cobalt-Uranyl Acetate TS Dissolve, with warming, 40 g of uranyl acetate, $\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$, in a mixture of 30 g of glacial acetic acid and sufficient water to make 500 ml. Similarly, prepare a solution containing 200 g of cobaltous acetate, $\text{Co}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$, in a mixture of 30 g of glacial acetic acid and sufficient water to make 500 ml. Mix the two solutions while still warm, and cool to 20°. Maintain the temperature at 20° for about 2 h to separate the excess salts from solution, and then filter through a dry filter.

Congo Red TS Dissolve 500 mg of congo red in a mixture of 10 ml of alcohol and 90 ml of water.

Cresol Red TS Triturate 100 mg of cresol red in a mortar with 26.2 ml of 0.01 *N* sodium hydroxide until solution is complete, then dilute the solution with water to 250 ml.

Cresol Red-Thymol Blue TS Add 15 ml of thymol blue TS to 5 ml of cresol red TS, and mix.

Crystal Violet TS Dissolve 100 mg of crystal violet in 10 ml of glacial acetic acid.

Cupric Citrate TS, Alkaline (Benedict's Qualitative Reagent) With the aid of heat, dissolve 173 g of sodium citrate, $\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$, and 117 g of sodium carbonate, $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$, in about 100 ml of water, and filter through paper, if necessary. In a separate container dissolve 17.3 g of cupric sulfate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, in about 700 ml of water, and slowly add this solution, with constant stirring, to the first solution. Cool the mixture, dilute to 1000 ml, and mix.

Cupric Nitrate TS Dissolve 2.4 g of cupric nitrate, $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$, in sufficient water to make 100 ml.

Cupric Sulfate TS Dissolve 12.5 g of cupric sulfate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, in sufficient water to make 100 ml, and mix.

Cupric Tartrate TS, Alkaline (Fehling's Solution) *The Copper Solution (A):* Dissolve 34.66 g of carefully selected, small crystals of cupric sulfate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, showing no trace of efflorescence or of adhering moisture, in sufficient water to make 500 ml. Store this solution in small, tight containers. *The Alkaline Tartrate Solution (B):* Dissolve 173 g of crystallized

potassium sodium tartrate, $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$, and 50 g of sodium hydroxide, NaOH , in sufficient water to make 500 ml. Store this solution in small, alkali-resistant containers. For use, mix exactly equal volumes of solutions *A* and *B* at the time required.

Cyanogen Bromide TS Dissolve 5 g of cyanogen bromide in water to make 50 ml.

Caution: Prepare this solution under a hood, as cyanogen bromide volatilizes at room temperature and the vapor is highly irritating and poisonous.

Denigès' Reagent See *Mercuric Sulfate TS*.

2,7-Dihydroxynaphthalene TS Dissolve 100 mg of 2,7-dihydroxynaphthalene in 1000 ml of sulfuric acid, and allow the solution to stand until the initial color disappears. If the solution is very dark, discard it and prepare a new solution from a different supply of sulfuric acid. This solution is stable for approximately 1 month if stored in a dark bottle.

Diphenylamine TS Dissolve 1 g of diphenylamine in 100 ml of sulfuric acid. The solution should be colorless.

Diphenylcarbazone TS Dissolve about 1 g of diphenylcarbazone ($\text{C}_{13}\text{H}_{12}\text{N}_4\text{O}$) in sufficient alcohol to make 100 ml. Store this solution in a brown bottle.

α, α' -Dipyridyl TS Dissolve 100 mg of α, α' -dipyridyl, $\text{C}_{10}\text{H}_8\text{N}_2$, in 50 ml of absolute alcohol.

Dithizone TS Dissolve 25.6 mg of dithizone in 100 ml of alcohol.

Eosin Y TS (adsorption indicator) Dissolve 50 mg of eosin Y in 10 ml of water.

Eriochrome Black TS Dissolve 200 mg of eriochrome black T and 2 g of hydroxylamine hydrochloride, $\text{NH}_2\text{OH} \cdot \text{HCl}$, in sufficient methanol to make 50 ml, and filter. Store the solution in a light-resistant container and use within 2 weeks.

***p*-Ethoxychrysoidin TS** Dissolve 50 mg of *p*-ethoxychrysoidin monohydrochloride in a mixture of 25 ml of water and 25 ml of alcohol, add 3 drops of hydrochloric acid, stir vigorously, and filter if necessary to obtain a clear solution.

Fehling's Solution See *Cupric Tartrate TS, Alkaline*.

Ferric Ammonium Sulfate TS Dissolve 8 g of ferric ammonium sulfate, $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$, in sufficient water to make 100 ml.

Ferric Chloride TS Dissolve 9 g of ferric chloride, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, in sufficient water to make 100 ml.

Ferric Chloride TS, Alcoholic Dissolve 100 mg of ferric

chloride, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, in 50 ml of absolute alcohol. Prepare this fresh.

Ferric Sulfate TS, Acid Add 7.5 ml of sulfuric acid to 100 ml of water, and dissolve 80 g of ferrous sulfate in the mixture with the aid of heat. Mix 7.5 ml of nitric acid and 20 ml of water, warm, and add to this the ferrous sulfate solution. Concentrate the mixture until, upon the sudden disengagement of ruddy vapors, the black color of the liquid changes to red. Test for the absence of ferrous iron, and, if necessary, add a few drops of nitric acid and boil again. When the solution is cold, add sufficient water to make 110 ml.

Ferrous Sulfate TS Dissolve 8 g of clear crystals of ferrous sulfate, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, in about 100 ml of recently boiled and thoroughly cooled water. Prepare this solution fresh.

Formaldehyde TS A solution containing approximately 37.0% (w/v) of HCHO . It may contain methanol to prevent polymerization.

Hydrochloric Acid Use ACS reagent-grade *Hydrochloric Acid* (36.5% to 38.0% of HCl).

Hydrochloric Acid TS, Diluted A solution containing 10% (w/v) of HCl . Prepare by diluting 226 ml of hydrochloric acid (36%) with sufficient water to make 1000 ml.

Hydrogen Peroxide TS A solution containing between 2.5 and 3.0 g of H_2O_2 in each 100 ml. It may contain suitable stabilizers, totaling not more than 0.05%.

Hydrogen Sulfide TS A saturated solution of hydrogen sulfide made by passing H_2S into cold water. Store it in small, dark amber-colored bottles, filled nearly to the top. It is unsuitable unless it possesses a strong odor of H_2S , and unless it produces at once a copious precipitate of sulfur when added to an equal volume of ferric chloride TS. Store in a cold, dark place.

Hydroxylamine Hydrochloride TS Dissolve 3.5 g of hydroxylamine hydrochloride, $\text{NH}_2\text{OH} \cdot \text{HCl}$, in 95 ml of 60% alcohol, and add 0.5 ml of bromophenol blue solution (1 in 1000) and 0.5 *N* alcoholic potassium hydroxide until a greenish tint develops in the solution. Then add sufficient 60% alcohol to make 100 ml.

8-Hydroxyquinoline TS Dissolve 5 g of 8-hydroxyquinoline (oxine) in sufficient alcohol to make 100 ml.

Indigo Carmine TS (Sodium Indigotindisulfonate TS) Dissolve a quantity of sodium indigotindisulfonate, equivalent to 180 mg of $\text{C}_{16}\text{H}_8\text{N}_2\text{O}_2(\text{SO}_3\text{Na})_2$, in sufficient water to make 100 ml. Use within 60 days.

Iodine TS Dissolve 14 g of iodine, I_2 , in a solution of 36 g of potassium iodide, KI , in 100 ml of water, add 3 drops of hydrochloric acid, dilute with water to 1000 ml, and mix.

Isopropanol [Isopropyl Alcohol; 2-Propanol; $(\text{CH}_3)_2\text{CHOH}$]

Use ACS reagent-grade *Isopropyl Alcohol*. (NOTE: For use in assays and tests involving ultraviolet spectrophotometry, use ACS reagent-grade *Isopropyl Alcohol Suitable for Use in Ultraviolet Spectrophotometry*.)

Isopropanol, Anhydrous (Dehydrated Isopropanol) Use *Isopropanol* that has been previously dried by shaking with anhydrous calcium chloride, followed by filtering.

Lead Acetate TS Dissolve 9.5 g of clear, transparent crystals of lead acetate, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$, in sufficient recently boiled water to make 100 ml. Store in well-stoppered bottles.

Lead Subacetate TS Triturate 14 g of lead monoxide, PbO , to a smooth paste with 10 ml of water, and transfer the mixture to a bottle, using an additional 10 ml of water for rinsing. Dissolve 22 g of lead acetate, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$, in 70 ml of water, and add the solution to the lead oxide mixture. Shake it vigorously for 5 min, then set it aside, shaking it frequently during 7 days. Finally filter, and add enough recently boiled water through the filter to make 100 ml.

Lead Subacetate TS, Diluted Dilute 3.25 ml of lead subacetate TS with sufficient water, recently boiled and cooled, to make 100 ml. Store in small, well-fitted, tight containers.

Litmus TS Digest 25 g of powdered litmus with three successive 100-ml portions of boiling alcohol, continuing each extraction for about 1 h. Filter, wash with alcohol, and discard the alcohol filtrate. Macerate the residue with about 25 ml of cold water for 4 h, filter, and discard the filtrate. Finally, digest the residue with 125 ml of boiling water for 1 h, cool, and filter.

Magnesia Mixture TS Dissolve 5.5 g of magnesium chloride, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, and 7 g of ammonium chloride, NH_4Cl , in 65 ml of water, add 35 ml of ammonia TS, set the mixture aside for a few days in a well-stoppered bottle, and filter. If the solution is not perfectly clear, filter it before using.

Magnesium Sulfate TS Dissolve 12 g of crystals of magnesium sulfate, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, selected for freedom from efflorescence, in water to make 100 ml.

Malachite Green TS Dissolve 1 g of malachite green oxalate in 100 ml of glacial acetic acid.

Mayer's Reagent See *Mercuric-Potassium Iodide TS*.

Mercuric Acetate TS Dissolve 6 g of mercuric acetate, $\text{Hg}(\text{C}_2\text{H}_3\text{O}_2)_2$, in sufficient glacial acetic acid to make 100 ml. Store in tight containers protected from direct sunlight.

Mercuric Chloride TS Dissolve 6.5 g of mercuric chloride, HgCl_2 , in water to make 100 ml.

Mercuric-Potassium Iodide TS (Mayer's Reagent) Dissolve 1.358 g of mercuric chloride, HgCl_2 , in 60 ml of water. Dissolve 5 g of potassium iodide, KI , in 10 ml of water. Mix the two solutions, and add water to make 100 ml.

Mercuric-Potassium Iodide TS, Alkaline (Nessler's Reagent)

Dissolve 10 g of potassium iodide, KI, in 10 ml of water, and add slowly, with stirring, a saturated solution of mercuric chloride until a slight red precipitate remains undissolved. To this mixture add an ice-cold solution of 30 g of potassium hydroxide, KOH, in 60 ml of water, then add 1 ml more of the saturated solution of mercuric chloride. Dilute with water to 200 ml. Allow the precipitate to settle, and draw off the clear liquid. A 2-ml portion of this reagent, when added to 100 ml of a 1 in 300,000 solution of ammonium chloride in ammonia-free water, produces at once a yellowish brown color.

Mercuric Sulfate TS (Denigès' Reagent) Mix 5 g of yellow mercuric oxide, HgO, with 40 ml of water, and while stirring slowly add 20 ml of sulfuric acid, then add another 40 ml of water, and stir until completely dissolved.

Methanol (Methyl Alcohol) Use ACS reagent-grade *Methanol*.

Methanol, Anhydrous (Dehydrated Methanol) Use *Methanol*.

p-Methylaminophenol Sulfate TS Dissolve 2 g of p-methylaminophenol sulfate, $(\text{HO.C}_6\text{H}_4\text{NHCH}_3)_2\text{H}_2\text{SO}_4$, in 100 ml of water. To 10 ml of this solution add 90 ml of water and 20 g of sodium bisulfite. Confirm the suitability of this solution by the following test: Add 1 ml of the solution to each of four tubes containing 25 ml of 0.5 N sulfuric acid and 1 ml of ammonium molybdate TS. Add 5 μg of phosphate (PO_4) to one tube, 10 μg to a second, and 20 μg to a third, using 0.5, 1.0, and 2.0 ml, respectively, of *Phosphate Standard Solution*, and allow to stand for 2 h. The solutions in the three tubes should show readily perceptible differences in blue color corresponding to the relative amounts of phosphate added, and the one to which 5 μg of phosphate was added should be perceptibly bluer than the blank.

Methylene Blue TS Dissolve 125 mg of methylene blue in 100 ml of alcohol, and dilute with alcohol to 250 ml.

Methyl Orange TS Dissolve 100 mg of methyl orange in 100 ml of water, and filter if necessary.

Methyl Red TS Dissolve 100 mg of methyl red in 100 ml of alcohol, and filter if necessary.

Methyl Red-Methylene Blue TS Add 10 ml of methyl red TS to 10 ml of methylene blue TS, and mix.

Methylrosaniline Chloride TS See *Crystal Violet TS*.

Methyl Violet TS See *Crystal Violet TS*.

Millon's Reagent To 2 ml of mercury in an Erlenmeyer flask add 20 ml of nitric acid. Shake the flask under a hood to break up the mercury into small globules. After about 10 min add 35 ml of water, and, if a precipitate or crystals appear, add sufficient dilute nitric acid (1 in 5, prepared from nitric acid from which the oxides have been removed by blowing air

through it until it is colorless) to dissolve the separated solid. Add sodium hydroxide solution (1 in 10), dropwise, with thorough mixing, until the curdy precipitate that forms after the addition of each drop no longer redissolves but is dispersed to form a suspension. Add 5 ml more of the dilute nitric acid, and mix well. Prepare this solution fresh.

Naphthol Green TS Dissolve 500 mg of naphthol green B in water to make 1000 ml.

Nessler's Reagent See *Mercuric-Potassium Iodide TS, Alkaline*.

Neutral Red TS Dissolve 100 mg of neutral red in 100 ml of 50% alcohol.

Ninhydrin TS See *Triketohydrindene Hydrate TS*.

Nitric Acid Use ACS reagent-grade *Nitric Acid* (69.0% to 71.0% of HNO_3).

Nitric Acid TS, Diluted A solution containing about 10% (w/v) of HNO_3 . Prepare by diluting 105 ml of nitric acid (70%) with water to make 1000 ml.

Orthophenanthroline TS Dissolve 150 mg of orthophenanthroline, $\text{C}_{12}\text{H}_8\text{N}_2\text{H}_2\text{O}$, in 10 ml of a solution of ferrous sulfate, prepared by dissolving 700 mg of clear crystals of ferrous sulfate, $\text{FeSO}_4\cdot 7\text{H}_2\text{O}$, in 100 ml of water. The ferrous sulfate solution must be prepared immediately before dissolving the orthophenanthroline. Store the solution in well-closed containers.

Oxalic Acid TS Dissolve 6.3 g of oxalic acid, $\text{H}_2\text{C}_2\text{O}_4\cdot 2\text{H}_2\text{O}$, in water to make 100 ml.

Phenol Red TS (Phenolsulfonphthalein TS) Dissolve 100 mg of phenolsulfonphthalein in 100 ml of alcohol, and filter if necessary.

Phenolphthalein TS Dissolve 1 g of phenolphthalein in 100 ml of alcohol.

Phenolsulfonphthalein TS See *Phenol Red TS*.

p-Phenylphenol TS On the day of use, dissolve 750 mg of p-phenylphenol in 50 ml of sodium hydroxide TS.

Phosphoric Acid Use ACS reagent-grade *Phosphoric Acid* (not less than 85.0% of H_3PO_4).

Phosphotungstic Acid TS Dissolve 1 g of phosphotungstic acid (approximately $24\text{WO}_3\cdot 2\text{H}_3\text{PO}_4\cdot 48\text{H}_2\text{O}$) in water to make 100 ml.

Picric Acid TS See *Trinitrophenol TS*.

Potassium Acetate TS Dissolve 10 g of potassium acetate, $\text{KC}_2\text{H}_3\text{O}_2$, in water to make 100 ml.

B-62

Potassium Chromate TS Dissolve 10 g of potassium chromate, K_2CrO_4 , in water to make 100 ml.

Potassium Dichromate TS Dissolve 7.5 g of potassium dichromate, $K_2Cr_2O_7$, in water to make 100 ml.

Potassium Ferricyanide TS Dissolve 1 g of potassium ferricyanide, $K_3Fe(CN)_6$, in 10 ml of water. Prepare this solution fresh.

Potassium Ferrocyanide TS Dissolve 1 g of potassium ferrocyanide, $K_4Fe(CN)_6 \cdot 3H_2O$, in 10 ml of water. Prepare this solution fresh.

Potassium Hydroxide TS Dissolve 6.5 g of potassium hydroxide, KOH, in water to make 100 ml.

Potassium Hydroxide TS, Alcoholic Use 0.5 *N* Alcoholic Potassium Hydroxide, page 566.

Potassium Iodide TS Dissolve 16.5 g of potassium iodide, KI, in water to make 100 ml. Store in light-resistant containers.

Potassium Permanganate TS Use 0.1 *N* Potassium Permanganate, page 566.

Potassium Sulfate TS Dissolve 1 g of potassium sulfate, K_2SO_4 , in sufficient water to make 100 ml.

Stannous Molybdate TS Dissolve 70 g of sodium molybdate, $Na_2MoO_4 \cdot 2H_2O$ in 150 ml of water (*Solution A*). Dissolve 60 g of citric acid in a mixture of 85 ml of nitric acid and 150 ml of water, and cool (*Solution B*). Gradually add *Solution A* to *Solution B*, with stirring, to produce *Solution C*. Dissolve 5.0 ml of synthetic quinoline in a mixture of 35 ml of nitric acid and 100 ml of water (*Solution D*). Gradually add *Solution D* to *Solution C*, mix well, and allow to stand overnight. Filter the mixture, add 280 ml of acetone to the filtrate, dilute to 1000 ml with water, and mix. Store in a polyethylene bottle.

Caution: This reagent contains acetone. Do not use it near an open flame. Operations involving heating or boiling should be conducted in a well-ventilated hood.

Quinaldine Red TS Dissolve 100 mg of quinaldine red in 100 ml of glacial acetic acid.

Schiff's Reagent, Modified Dissolve 200 mg of rosaniline hydrochloride, $C_{20}H_{20}ClN_3$, in 120 ml of hot water. Cool, add 2 g of sodium bisulfite, $NaHSO_3$, followed by 2 ml of hydrochloric acid, and dilute to 200 ml with water. Store in a brown bottle at 15° or lower.

Silver Nitrate TS Use 0.1 *N* Silver Nitrate, page 567.

Sodium Bisulfite TS Dissolve 10 g of sodium bisulfite, $NaHSO_3$, in water to make 30 ml. Prepare this solution fresh.

Sodium Bitartrate TS Dissolve 1 g of sodium bitartrate,

$NaHC_4H_4O_6 \cdot H_2O$, in water to make 10 ml. Prepare this solution fresh.

Sodium Borate TS Dissolve 2 g of sodium borate, $Na_2B_4O_7 \cdot 10H_2O$, in water to make 100 ml.

Sodium Carbonate TS Dissolve 10.6 g of anhydrous sodium carbonate, Na_2CO_3 , in water to make 100 ml.

Sodium Cobaltinitrite TS Dissolve 10 g of sodium cobaltinitrite, $Na_3Co(NO_2)_6$, in water to make 50 ml, and filter if necessary.

Sodium Fluoride TS Dry about 500 mg of sodium fluoride, NaF, at 200° for 4 h. Weigh accurately 222 mg of the dried sodium fluoride, and dissolve it in sufficient water to make exactly 100 ml. Transfer 10.0 ml of this solution into a 1000-ml volumetric flask, dilute to volume with water, and mix. Each ml of this final solution corresponds to 10 µg of fluorine (F).

Sodium Hydroxide TS Dissolve 4.3 g of sodium hydroxide, NaOH, in water to make 100 ml.

Sodium Indigotindisulfonate TS See *Indigo Carmine TS*.

Sodium Nitroferrocyanide TS Dissolve 1 g of sodium nitroferrocyanide, $Na_2Fe(NO)(CN)_5 \cdot 2H_2O$, in water to make 20 ml. Prepare this solution fresh.

Sodium Phosphate TS Dissolve 12 g of clear crystals of dibasic sodium phosphate, $Na_2HPO_4 \cdot 7H_2O$, in water to make 100 ml.

Sodium Sulfide TS Dissolve 1 g of sodium sulfide, $Na_2S \cdot 9H_2O$, in water to make 10 ml. Prepare this solution fresh.

Sodium Thiosulfate TS Use 0.1 *N* Sodium Thiosulfate, page 567.

Stannous Chloride TS Dissolve 40 g of reagent-grade stannous chloride dihydrate, $SnCl_2 \cdot 2H_2O$, in 100 ml of hydrochloric acid.

Starch TS Mix 1 g of a suitable starch with 10 mg of red mercuric oxide and sufficient cold water to make a thin paste. Add 20 ml of boiling water, boil for 1 min with continuous stirring, and cool. Use only the clear solution.

Starch Iodide Paste TS Heat 100 mg of water in a 250-ml beaker to boiling, add a solution of 750 mg of potassium iodide, KI, in 5 ml of water, then add 2 g of zinc chloride, $ZnCl_2$, dissolved in 10 ml of water, and, while the solution is boiling, add with stirring a smooth suspension of 5 g of potato starch in 30 ml of cold water. Continue to boil for 2 min, then cool. Store in well-closed containers in a cool place. This mixture must show a definite blue streak when a glass rod dipped in a mixture of 1 ml of 0.1 *M* sodium nitrite, 500 ml of water, and 10 ml of hydrochloric acid is streaked on a smear of the paste.

Sulfanilic Acid TS Dissolve 800 mg of sulfanilic acid, p - $\text{NH}_2\text{C}_6\text{H}_4\text{SO}_3\text{H}\cdot\text{H}_2\text{O}$, in 100 ml of acetic acid. Store in tight containers.

Sulfuric Acid Use ACS reagent-grade *Sulfuric Acid* (95.0% to 98.0% of H_2SO_4).

Sulfuric Acid TS See page 532.

Sulfuric Acid TS, Diluted A solution containing 10% (w/v) of H_2SO_4 . Prepare by cautiously adding 57 ml of sulfuric acid (95% to 98%) or sulfuric acid TS to about 100 ml of water, then cool to room temperature, and dilute with water to 1000 ml.

Tannic Acid TS Dissolve 1 g of tannic acid (tannin) in 1 ml of alcohol, and add water to make 10 ml. Prepare this solution fresh.

Thymol Blue TS Dissolve 100 mg of thymol blue in 100 ml of alcohol, and filter if necessary.

Thymolphthalein TS Dissolve 100 mg of thymolphthalein in 100 ml of alcohol, and filter if necessary.

Triketohydrindene Hydrate TS (Ninhydrin TS) Dissolve 200 mg of triketohydrindene hydrate, $\text{C}_9\text{H}_4\text{O}_3\cdot\text{H}_2\text{O}$, in water to make 100 ml. Prepare this solution fresh.

Trinitrophenol TS (Picric Acid TS) Dissolve the equivalent of 1 g of anhydrous trinitrophenol in 100 ml of hot water. Cool the solution, and filter if necessary.

Xylenol Orange TS Dissolve 100 mg of xylenol orange in 100 ml of alcohol.

Volumetric Solutions

Normal Solutions A normal solution contains 1 g equivalent weight of the solute per liter of solution. The normalities of solutions used in volumetric determinations are designated as 1 N , 0.1 N , 0.05 N , etc., in this Codex.

Molar Solutions A molar solution contains 1 g molecular weight of the solute per liter of solution. The molarities of such solutions are designated as 1 M , 0.1 M , 0.05 M , etc., in this Codex.

Preparation and Methods of Standardization

The details for the preparation and standardization of solutions used in several normalities are usually given only for the one most frequently required. Solutions of other normalities are prepared and standardized in the same general manner as

described. Solutions of lower normalities may be prepared accurately by making an exact dilution of a stronger solution, but solutions prepared in this way should be restandardized before use.

Dilute solutions that are not stable, such as 0.01 N potassium permanganate and sodium thiosulfate, are preferably prepared by diluting exactly the higher normality with thoroughly boiled and cooled water on the same day they are to be used.

All volumetric solutions should be prepared, standardized, and used at the standard temperature of 25°, if practicable. When a titration must be carried out at a markedly different temperature, the volumetric solution should be standardized at that same temperature, or a suitable temperature correction should be made. Since the strength of a standard solution may change upon standing, the normality or molarity factor should be redetermined frequently.

Although the directions provide only one method of standardization, other methods of equal or greater accuracy may be used. For substances available as certified primary standards, or of comparable quality, the final standard solution may be prepared by weighing accurately a suitable quantity of the substance and dissolving it to produce a specific volume solution of known concentration. Hydrochloric and sulfuric acids may be standardized against a certified primary standard.

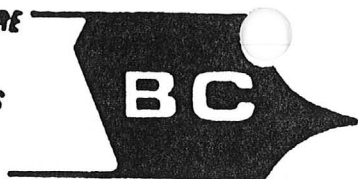
In volumetric assays described in this Codex, the number of mg of the test substance equivalent to 1 ml of the primary volumetric solution is given. In general, these equivalents may be derived by simple calculation (see also *Solutions*, page 3).

Ammonium Thiocyanate, 0.1 N (7.612 g NH_4SCN per 1000 ml) Dissolve about 8 g of ammonium thiocyanate, NH_4SCN , in 1000 ml of water, and standardize by titrating the solution against 0.1 N silver nitrate as follows: Transfer about 30 ml of 0.1 N silver nitrate, accurately measured, into a glass-stoppered flask. Dilute with 50 ml of water, then add 2 ml of ferric ammonium sulfate TS and 2 ml of nitric acid, and titrate with the ammonium thiocyanate solution to the first appearance of a red brown color. Calculate the normality, and, if desired, adjust the solution to exactly 0.1 N . If desired, 0.1 N ammonium thiocyanate may be replaced by 0.1 N potassium thiocyanate where the former is directed in various tests and assays.

Bromine, 0.1 N (7.990 g Br per 1000 ml) Dissolve 3 g of potassium bromate, KBrO_3 , and 15 g of potassium bromide, KBr , in sufficient water to make 1000 ml, and standardize the solution as follows: Transfer about 25 ml of the solution, accurately measured, into a 500-ml iodine flask, and dilute with 120 ml of water. Add 5 ml of hydrochloric acid, stopper the flask, and shake it gently. Then add 5 ml of potassium iodide TS, restopper, shake the mixture, allow it to stand for 5 min, and titrate the liberated iodine with 0.1 N sodium thiosulfate, adding starch TS near the end of the titration. Calculate the normality. Store this solution in dark amber-colored, glass-stoppered bottles.

Ceric Sulfate, 0.1 N [33.22 g $\text{Ce}(\text{SO}_4)_2$ per 1000 ml] Transfer 59 g of ceric ammonium nitrate, $\text{Ce}(\text{NO}_3)_4\cdot 2\text{NH}_4\text{NO}_3\cdot 2\text{H}_2\text{O}$, to a beaker, add 31 ml of sulfuric acid, mix, and cautiously add water, in 20-ml portions, until solution is complete. Cover the

AGRICULTURE
CHEMICAL ANALYSIS
PETROLEUM



LABORATORIES INC.

J. J. EGLIN, REG. CHEM. ENGR.

3016 UNION AVE. BAKERSFIELD, CALIFORNIA 93305 PHONE 324-1815
MAIN OFFICE: 4100 PIERCE ROAD, BAKERSFIELD, CA. 93308 PHONE 327-4911

Submitted by: United States Dept. of the Interior
Bureau of Land Management
Arizona Strip District Office
390 North 3050 East
St. George, Utah 84770
Attn: Mr. John Branch

Date Reported: 7/11/89
Date Received: 6/30/89
Laboratory No.: 5181-1 to 5181-18

GYPSSUM ANALYSIS

P.O. #AZ-010-PH9-189

Sample Description	Dry Basis		As Received Basis		
	Gypsum Percent	*Combined Sulfur Percent	Gypsum CaSO ₄ · 2H ₂ O Equivalent Percent	*Combined Sulfur Percent	Moisture Weight Loss @ 114°F Percent
3800 (010) 1-5	94.98	17.68	94.94	17.67	0.06
3800 (010) 1-6	98.58	18.35	98.54	18.34	0.06
3800 (010) 16-5	94.32	17.55	94.28	17.55	0.05
3800 (010) 1-1	99.42	18.50	99.37	18.49	0.05
3800 (010) 1-2	99.36	18.49	99.30	18.48	0.06
3800 (010) 1-3	96.80	18.00	96.76	18.00	0.04
3800 (010) 1-4	93.22	17.35	93.14	17.33	0.08
3800 (010) 1-7	98.85	18.40	98.82	18.39	0.03
3800 (010) 1-8	86.11	16.03	86.02	16.01	0.11
3800 (010) 1-9	94.62	17.61	94.56	17.60	0.06
3800 (010) 1-10	98.19	18.27	98.12	18.26	0.08
3800 (010) 16-1	88.95	16.55	88.87	16.54	0.09
3800 (010) 16-3	91.04	16.94	90.99	16.93	0.05
3800 (010) 16-7	64.66	12.03	64.60	12.02	0.09
3800 (010) 16-8	87.63	16.31	87.55	16.29	0.09
3800 (010) 16-10	92.31	17.18	92.26	17.17	0.06
3800 (010) 16-11	98.60	18.35	98.54	18.34	0.06
3800 (010) 16-12	96.39	17.94	96.29	17.92	0.10

BLM Received
Arizona Strip District Office

JUL 12 1989

St. George, Utah
10:00 A.M.

LABORATORIES, INC.

J. J. Eglin
J. J. Eglin

B-65

*Sulfur combined with calcium to form gypsum

AGRICULTURE

CHEMICAL ANALYSIS

PETROLEUM

BC

LABORATORIES, INC.

J. J. EGLIN, REG. CHEM. ENGR.

4100 PIERCE RD., BAKERSFIELD, CALIFORNIA 93308 PHONE 327-4911

United States Dept. of the Interior
Bureau of Land Management
Arizona Strip District Office
390 North 3050 East
St. George, Utah 84770

Date Reported: 7/11/89
Date Received: 6/30/89
Laboratory No.: 5181-1 to 5181-3

P.O. #AZ-010-PH9-189

Attn: Mr. John Branch

Sample Description: 3800 (010) 1-5

Selenium:	less than 0.25 mg/kg
Arsenic:	less than 0.25 mg/kg
Fluoride:	less than 1.0 mg/kg
Lead:	1.34 mg/kg

Sample Description: 3800 (010) 1-6

Selenium:	less than 0.25 mg/kg
Arsenic:	less than 0.25 mg/kg
Fluoride:	less than 1.0 mg/kg
Lead:	2.35 mg/kg

Sample Description: 3800 (010) 16-5

Selenium:	less than 0.25 mg/kg
Arsenic:	less than 0.25 mg/kg
Fluoride:	less than 1.0 mg/kg
Lead:	2.53 mg/kg

B C LABORATORIES, INC.

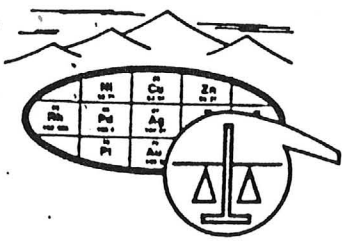
BY J. J. Eglin
J. J. Eglin

BLM Received
Arizona Strip District Office

JUL 12 1989

St. George, Utah
10:00 A.M.

56 B-66



SKONE LABS, INC.
1775 W. Sahuaro Dr. • P.O. Box 50106
Tucson, Arizona 85703
(602) 622-4836

REPORT OF ANALYSIS

JOB NO. VQW 002
July 24, 1989
AZ-010-PH9-217
12-4 TO 16-13
PAGE 1 OF 1

U.S. DEPARTMENT OF THE INTERIOR
Attn: Mr. S. William Lamb
Bureau of Land Management
390 North 3050 East
St. George, UT 84770

Receiving
Columbia Strip District Office

JUL 26 1989

St. George, Utah
-- 10:00 A.M.

Analysis of 17 Rock Samples

ITEM	SAMPLE NO.	S03 (%)	GYPSUM* (%)
1	12-4	38.2	82.1
2	12-5	45.4	97.8
3	12-7	44.6	96.0
4	12-8	41.4	89.0
5	12-9	44.2	95.1
6	12-11	44.2	95.0
7	12-13	43.6	93.7
8	12-14	45.1	97.0
9	15-1	43.1	92.6
10	15-2	42.4	91.1
11	15-4	43.8	94.3
12	15-5	42.2	90.6
13	15-6	45.9	98.7
14	15-8	45.1	97.0
15	15-9	44.6	95.8
16	15-10	44.0	94.6
17	16-13	45.9	98.6

*NOTE: Total S03(%) calculated as Gypsum.

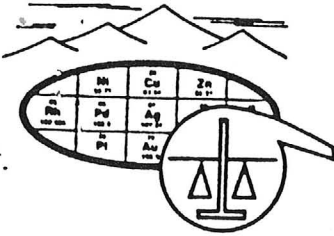
cc: Mr. John Branch

REGISTERED ASSAYER
 CERTIFICATE NO. 9425
 WILLIAM L. LEHMBECK
 WILLIAM L. Lehmbek
 Manager
 Arizona U.S.A.
 7/25/89

Charles E. Thompson
Arizona Registered Assayer No. 9427

William L. Lehmbek
Arizona Registered Assayer No. 9425

James A. Martin
Arizona Registered Assayer No. 11122



SKLINE LABS, INC.
 1775 W. Sahuaro Dr. • P.O. Box 50106
 Tucson, Arizona 85703
 (602) 622-4836

REPORT OF ANALYSIS

JOB NO. VQW 001
 August 15, 1989
 P.O. NO. AZ-010-PH9-192
 12-6 TO 16-9
 PAGE 1 OF 2

U.S. DEPARTMENT OF THE INTERIOR
 Attn: Mr. S. William Lamb
 Bureau of Land Management
 Arizona Strip District Office
 390 North 3050 East
 St. George, UT 84770

BLM Received
 Arizona Strip District Office

AUG 18 1989

St. George, Utah
 10:00 A.M.

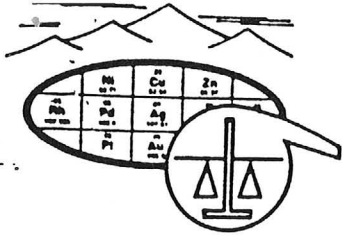
Analysis of 5 Rock Samples

ITEM	SAMPLE NO.	CaSO4* (%)	SO3 (%)	As (ppm)	F (ppm)
1	12-6	97.4	45.8	<1.	20.
2	12-10	89.7	43.1	<1.	150.
3	15-3	96.5	45.8	<1.	45.
4	15-7	96.4	45.7	<1.	45.
5	16-9	94.0	44.8	<1.	130.

Charles E. Thompson
 Arizona Registered Assayer No. 8427

B-60
 William L. Lehmbeck
 Arizona Registered Assayer No. 8425

James A. Martin
 Arizona Registered Assayer No. 11122



SKYSTONE LABS, INC.
 1775 W. Sahuaro Dr. • P.O. Box 50106
 Tucson, Arizona 85703
 (602) 622-4836

JOB NO. VQW 001
 August 15, 1989
 PAGE 2 OF 2

ITEM	SAMPLE NO.	Se (ppm)	Heavy Metals (ppm)	H2O (45C) (%)	H2O (220C) (%)
1	12-6	<1.	<10.	<.05	20.0
2	12-10	<1.	<10.	.11	18.2
3	15-3	<1.	<10.	.11	19.2
4	15-7	<1.	<10.	.20	19.0
5	16-9	<1.	<10.	.17	18.8

BLM Received
 Arizona Strip District Office

*NOTE: Based on dry weight.

AUG 18 1989

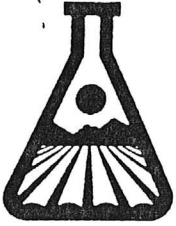
St. George, Utah
 10:00 A.M.

REGISTERED ASSAYER
 CERTIFICATE NO.
 9427
 CHARLES E.
 THOMPSON
 Charles E. Thompson
 Chief Assayer
 Arizona U. S. A. 7/17/89

Charles E. Thompson
 Arizona Registered Assayer No. 9427

William L. Lehmbek
 Arizona Registered Assayer No. 9425

James A. Martin
 Arizona Registered Assayer No. 11122



Inter Ag Services, Inc.

2643 East University Drive
 Suite 113
 Phoenix, Arizona
 85034

(602) 273-7248

BLM Received
 Arizona Strip District Office

OCT 27 1989

St. George, Utah
 10:00 A.M.

October 25, 1989

GROWER: BLM of AZ

REPORT TO: G. William Lamb

REPORT # 9296003

GYPSUM ANALYSIS REPORT

<u>SAMPLE ID</u>	<u>LAB #</u>	<u>FLUORIDE (ppm)*</u>
12-6	591	1.2
12-10	592	9.0
15-3	593	1.6
15-7	594	3.0
16-9	595	8.8
5181-1	596	3.1
5181-2	597	1.0
5181-3	598	2.2

* Method III (Ion-Selective Electrode Method B) page 511 FCC
 111/General Test and Apparatus.

60 B-70

Branch

9-26-89

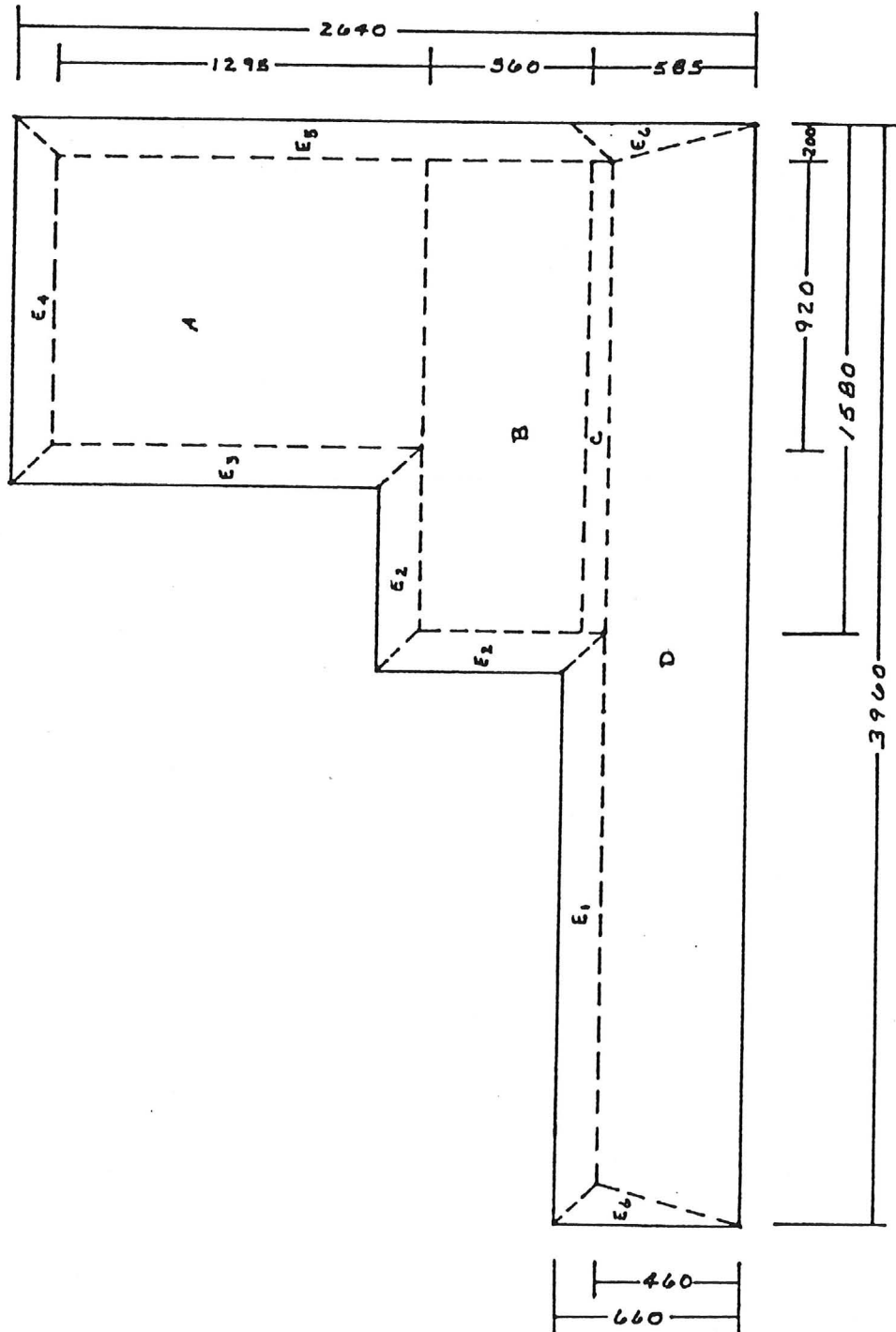
A-2344B

1 21

C+W Nos. 1, 12, 15, 16

AZ-010

Block Diagram for Tonnage Calculations



61

B-71

TABLE 1
 Analyses of Gypsum and Gypsite Samples From the Narrows
 Wilderness Study Area, Mohave County, Arizona
 (Briggs, 1983)

[Components determined by chemical analysis; values reported in percent;
 n.a., not analyzed]

Sample No.	Sample Type	%CaSO ₄	%SO ₃	H ₂ O Free & Combined	Insol. Residue	Fe	Na	Mg	Mn
NA0100	Chip. Vert., (4')	78.4	-	20.51	2.00	.04	.02	.03	.001
NA0101	Chip. Vert., (2.0')	67.3	-	21.31	5.90	.18	.13	.65	.002
NA0102	Chip. Vert. (4')	72.4	-	19.34	n.a.	.31	.04	5.10	.006
NA0103	Chip. Vert., (10')	71.3	-	20.61	n.a.	.14	.03	.63	.002
NA0104	Select	77.8	-	26.86	0.010	.02	.01	.02	.001
NA0105	Select	76.1	-	20.69	1.4	.03	.01	.02	.001
NA0106	Select	77.4	-	20.83	1.70	.005	.01	.12	.001

TABLE 2
Analyses of Samples Taken From C&W Mining Claims
(Harrison, 1984)

Minimum detection limit for As and Se = 0.50 ppm.

Fluoride extracted with water.

Heavy Metals: Pass = <10 ppm heavy metals reported as lead

Fail = >10 ppm heavy metals reported as lead

As, Se, F, heavy metals, and CaSO₄ reported on dry basis.

Loss on drying @ 250°F.

ND = element not detected.

Sample No.	Sample Type	%CaSO ₄	%SO ₃	H ₂ O Free & Combined	As (ppm)	Se (ppm)	F (ppm)	Heavy Metals
1	-	97.07	-	20.1	ND	ND	<3	Pass
2	-	96.66	-	20.2	ND	ND	<3	Pass
3	-	96.39	-	20.1	ND	ND	<3	Pass
4	-	97.88	-	20.3	ND	ND	<3	Pass
5	-	96.11	-	20.2	ND	ND	<3	Fail*
6	-	96.66	-	20.1	ND	ND	<3	Pass
7	-	96.93	-	20.1	ND	ND	<3	Pass
8	-	97.72	-	20.1	ND	ND	<3	Pass
9	-	96.66	-	20.1	ND	ND	<3	Pass
10	-	96.80	-	20.2	ND	ND	<3	Pass
11	-	96.39	-	20.2	ND	ND	<3	Pass
12	-	96.39	-	20.1	ND	ND	<3	Pass
13	-	96.66	-	20.2	ND	ND	<3	Pass
14	-	97.34	-	20.2	ND	ND	<3	Pass
15	-	96.39	-	20.2	ND	ND	<3	Pass
16	-	97.34	-	20.0	ND	ND	<3	Pass
17	-	97.72	-	20.1	ND	ND	<3	Pass
18	-	97.61	-	20.1	ND	ND	<3	Pass
19	-	97.34	-	20.0	ND	ND	<3	Pass
20	-	96.93	-	20.1	ND	ND	<3	Pass
21	-	96.66	-	20.2	ND	ND	<3	Pass
22	-	96.80	-	20.1	ND	ND	<3	Pass
23	-	97.72	-	20.1	ND	ND	<3	Pass
24	-	97.93	-	20.1	ND	ND	<3	Pass
25	-	97.20	-	20.1	ND	ND	<3	Pass
26	-	96.80	-	20.2	ND	ND	<3	Pass
27	-	96.66	-	20.2	ND	ND	<3	Pass
28	-	95.43	-	20.1	ND	ND	<3	Pass

* Sample had approximately 20 ppm Cu as determined by atomic absorption.

B-73

TABLE 3
 Assays of Gypsum Samples Taken From Selected Locations, Cedar Pockets Area,
 Mohave County, Arizona
 (Swapp, 1985)

Sample No.	Sample Type	%CaSO ₄	%SO ₃	H ₂ O Free & Combined	Insol. Residue	Fe	Na	Mg	Mn
1	Vert., Chip, (3')	98.92	46.00	-	0.20	-	-	-	-
2	Vert., Chip, (2')	98.47	45.79	-	0.22	-	-	-	-
3	Vert., Chip, (2')	98.08	45.61	-	0.22	-	-	-	-
4	Vert., Chip, (2')	98.54	46.29	-	0.12	-	-	-	-
5	Vert., Chip, (3')	98.98	46.03	-	0.16	-	-	-	-
6	Vert., Chip, (2')	99.15	46.11	-	0.12	-	-	-	-
7	Vert., Chip, (2')	99.35	46.20	-	0.12	-	-	-	-
8	Random Grab	97.00	45.11	-	1.64	-	-	-	-

B-74

50'

R 14 W

490 000 FEET

WASHINGTON CO

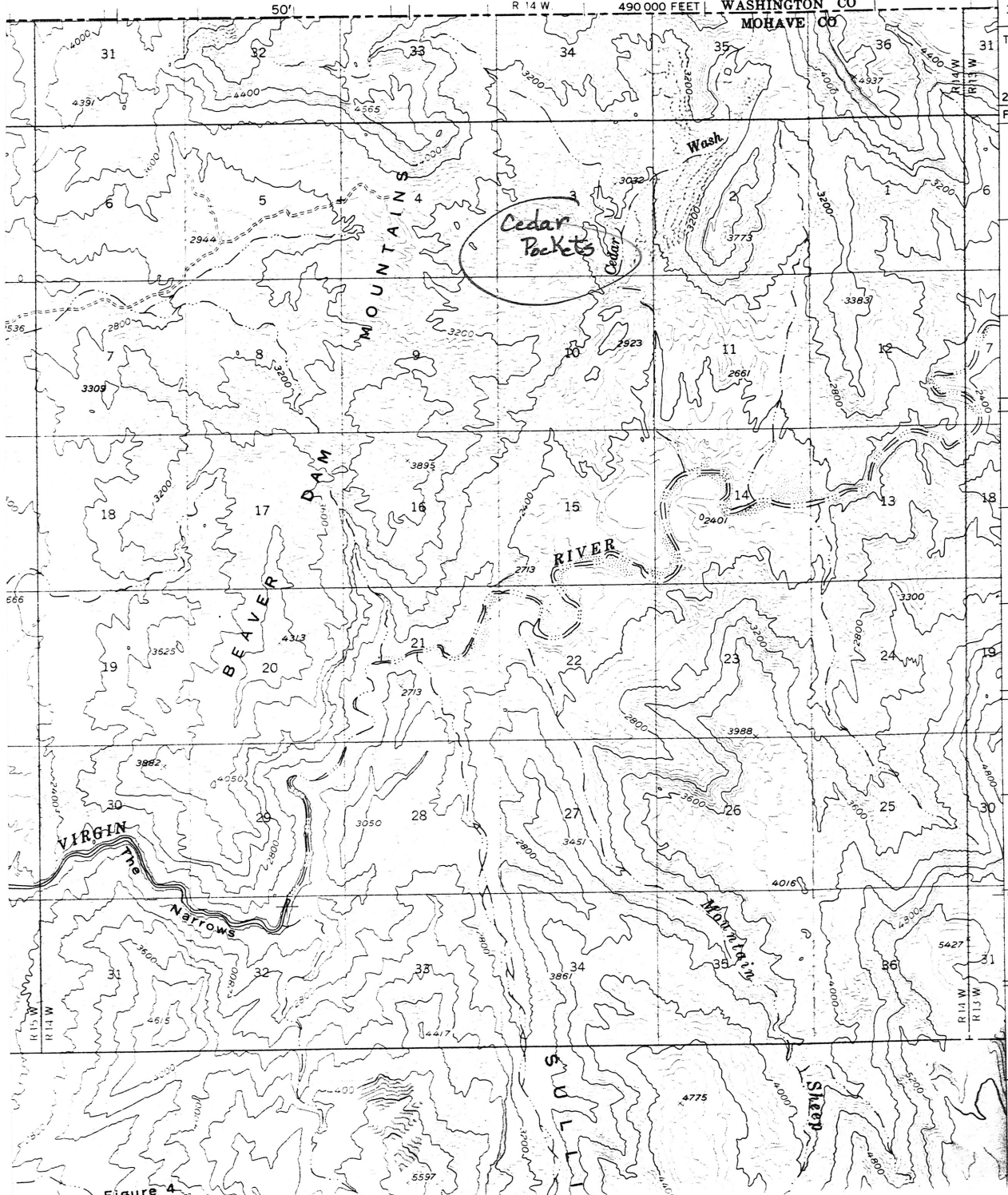
MOHAVE CO

113°45'

37°00'

T 42 N

2 180 000
FEET



A-12

Figure 4

Littlefield 15' Quad.

Date Printed: 12/02/97

ARIZONA DEPARTMENT OF MINES AND MINERAL RESOURCES

INFORMATION SUMMARY

Information from: Curt Willsie

Company: Premier Gypsum

Address:

City, State ZIP:

Phone:

MINE: C & W

ADMMR Mine File: Cedar Pockets

County: Mohave

AzMILS Number: 744

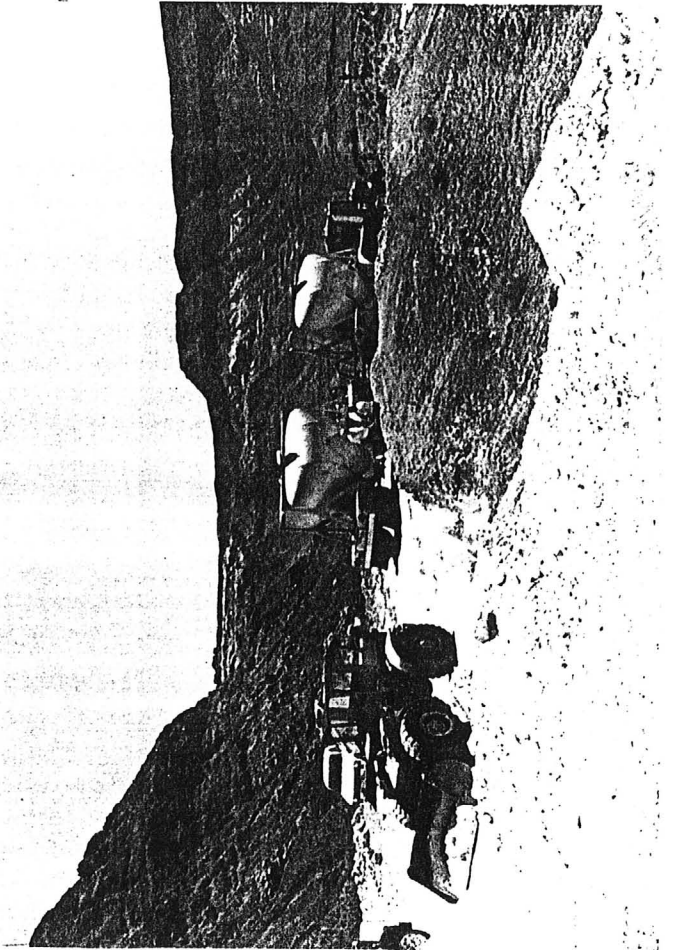
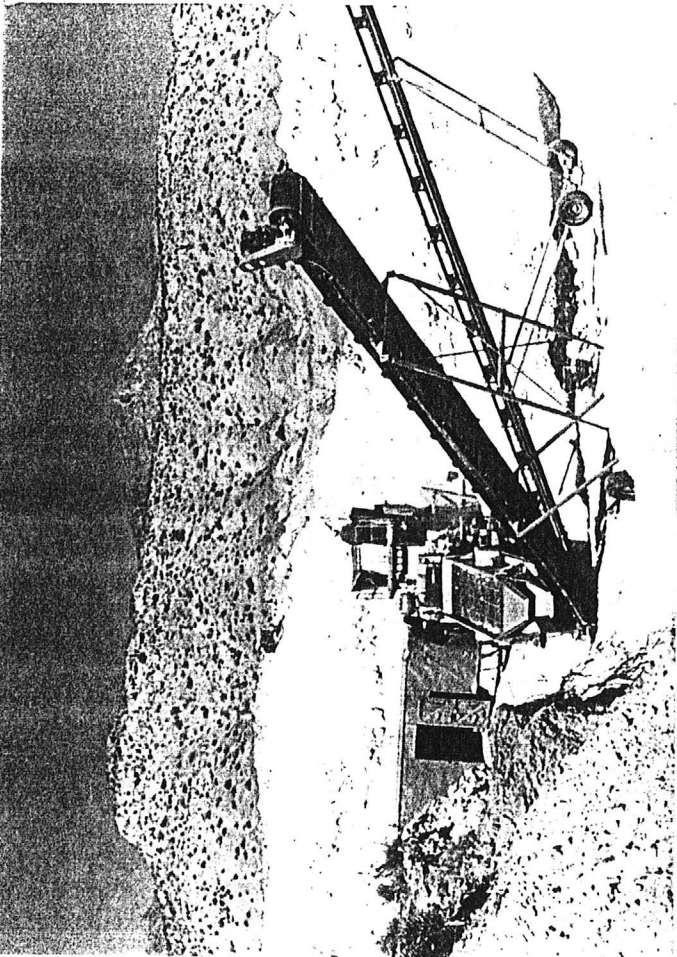
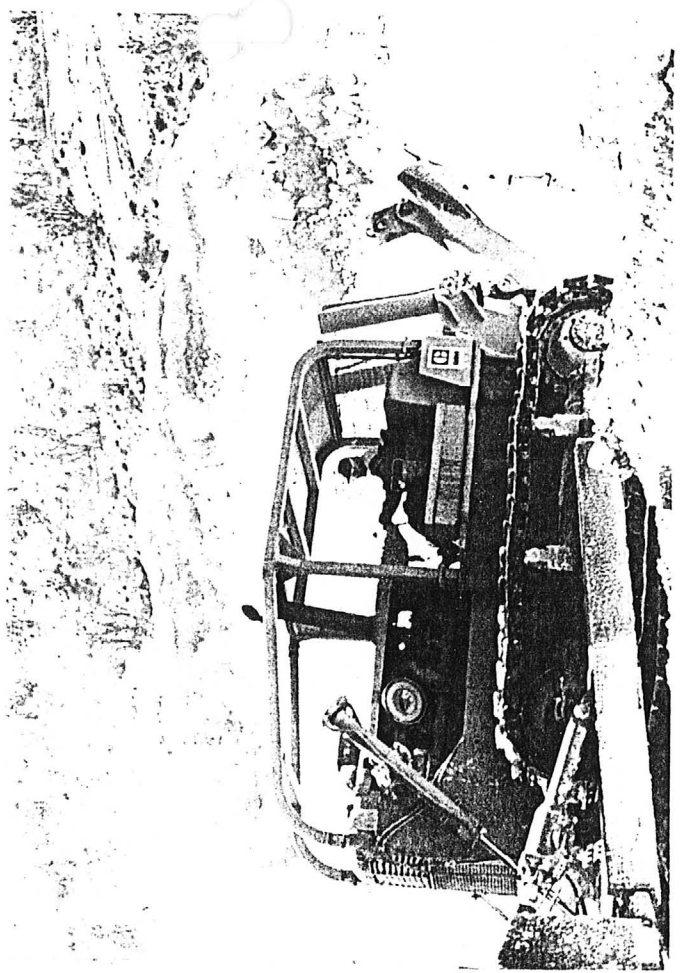
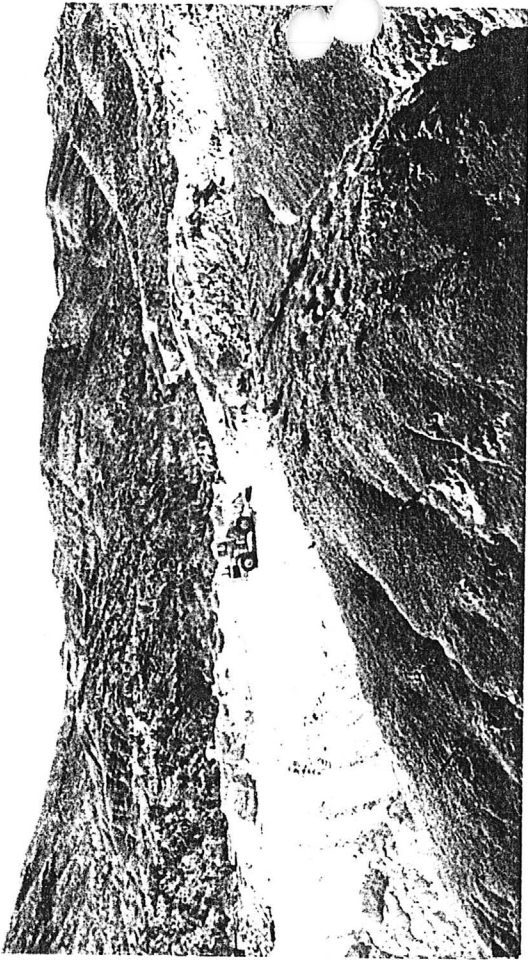
SUMMARY

A complete copy of the BLM file was provided by Curt Willsie and thus it did not have to be obtained through a Freedom of Information Act request as was reported in my August 1, 1996 VIS.

Ken A. Phillips, Chief Engineer

Date: December 2, 1997

Name of Mine or Prospect: Beaver Dam Mountains	Township 41N	Range 14W	Section 3	Priority C
Principal Minerals: Gypsum	1:250,000 Quad Grand Canyon		7.5' - 15' Quad Littlefield	
Associated Minerals: Limestone	District		Principal Product Gypsum	
Type of Operation: Surface	County Mohave	State Ar.	Type of Deposit Sedimentary	
Ownership or Controlling Interest: Consult current USBLM mining claim records.				
Access: From Littlefield, Ar. proceed north on U.S. 91 for 5.5 miles. Turn right on unimproved road and proceed easterly for 7 miles. Prospect is not located on topographic quadrangle.				
Structural Control or Geological Association: "Past production for agriculture, gypsum lenses in limestone." ¹ "Gypsum lenses at the top of the Callville limestone of late Pennsylvania Age." ²				
Age of Mineralization: Pennsylvanian				
Production History			Geochemical Analyses	
Intermittant production. ²				
References				
1) Elevatorski (1978), p. 42. 2) ABM (1969), Bull. 180, p. 368-381.				



Date Printed: 12/02/97

ARIZONA DEPARTMENT OF MINES AND MINERAL RESOURCES

INFORMATION SUMMARY

Information from: Curt Willsie

Company: Premier Gypsum

Address:

City, State ZIP:

Phone:

MINE: C & W

ADMMR Mine File: Cedar Pockets

County: Mohave

AzMILS Number: 744

SUMMARY

A complete copy of the BLM file was provided by Curt Willsie and thus it did not have to be obtained through a Freedom of Information Act request as was reported in my August 1, 1996 VIS.

Ken A. Phillips, Chief Engineer Date: December 2, 1997

Date Printed: 08/01/96

ARIZONA DEPARTMENT OF MINES AND MINERAL RESOURCES

INFORMATION SUMMARY

Information from: Byard L. Kershaw

Company: Bureau of Land Management - State Office

Address:

City, State ZIP:

Phone: 602-650-0348

MINE: C & W

ADMMR Mine File: Cedar Pockets

County: Mohave

AzMILS Number: 744

SUMMARY

Byard L. Kershaw, Bureau of Land Management, Phoenix, provided information on the C & W Group patent issuance.

Patent was issued on 40 acres of portions of the C & W 12 and C & W 16 placer claims to Curt Willsie of California. Patent issuance was based on a valid discovery of 9,900,000 tons of gypsum. The patented land is within the Beaver Dam Mountains Wilderness Area.

A complete copy of the BLM file can only be obtained through a Freedom of Information Act request.

Ken A. Phillips, Chief Engineer Date: August 1, 1996 ✓

CEDAR POCKETS

MOHAVE COUNTY

NJN WR 8/19/88: Larry Dietz (card) reported that Curtis Willsie is patening the unpatented claim group at Cedar Pockets (file) Mohave County. This property is a past gypsum producer and is now surrounded by wilderness. Mr. Dietz speculates that after receiving his patent Mr. Willsie may exchange this land with the BLM so they can remove the cherry stem from their wilderness.

ARIZONA DEPARTMENT OF MINES AND MINERAL RESOURCES

VERBAL INFORMATION SUMMARY

May be Reproduced

- 1. Information from: Mr. Cloyd Swapp
Address: Bureau of Land Management, St. George, UT, phone (801) 673-3545
- 2. Mine: CEDAR POCKETS 3. ADMMR Mine File
- 4. County Mohave 5. District
- 6. Township 41N Range 14W Sec(s) SW $\frac{1}{4}$, 3
- 7. Location: North of I-15 via Cedar Wash Canyon (Littlefield 15' Quad.)
- 8. No. of Claims - Patented Unpatented C & W group (#1-20)
- 9. Owner (if different from above) Mr. Curtis Willsie
- 10. Address: Yuma, AZ, phone 344-0624 (office 344-0616)
- 11. Operating Company:
- 12. Pertinent People and/or Firm:
- 13. Commodities: Gypsum
- 14. Operational Status: "Standby"
- 15. Summary of Information received, comments, etc.:

Mr. Swapp reports this gypsum deposit is considered by the BLM to be a valid mineral occurrence. It is about 50 to 60 feet thick and is generally high grade but with some interlayered bands of dolomite and other impurities.

There has been some minor production from the deposit, apparently by Mr. Willsie.

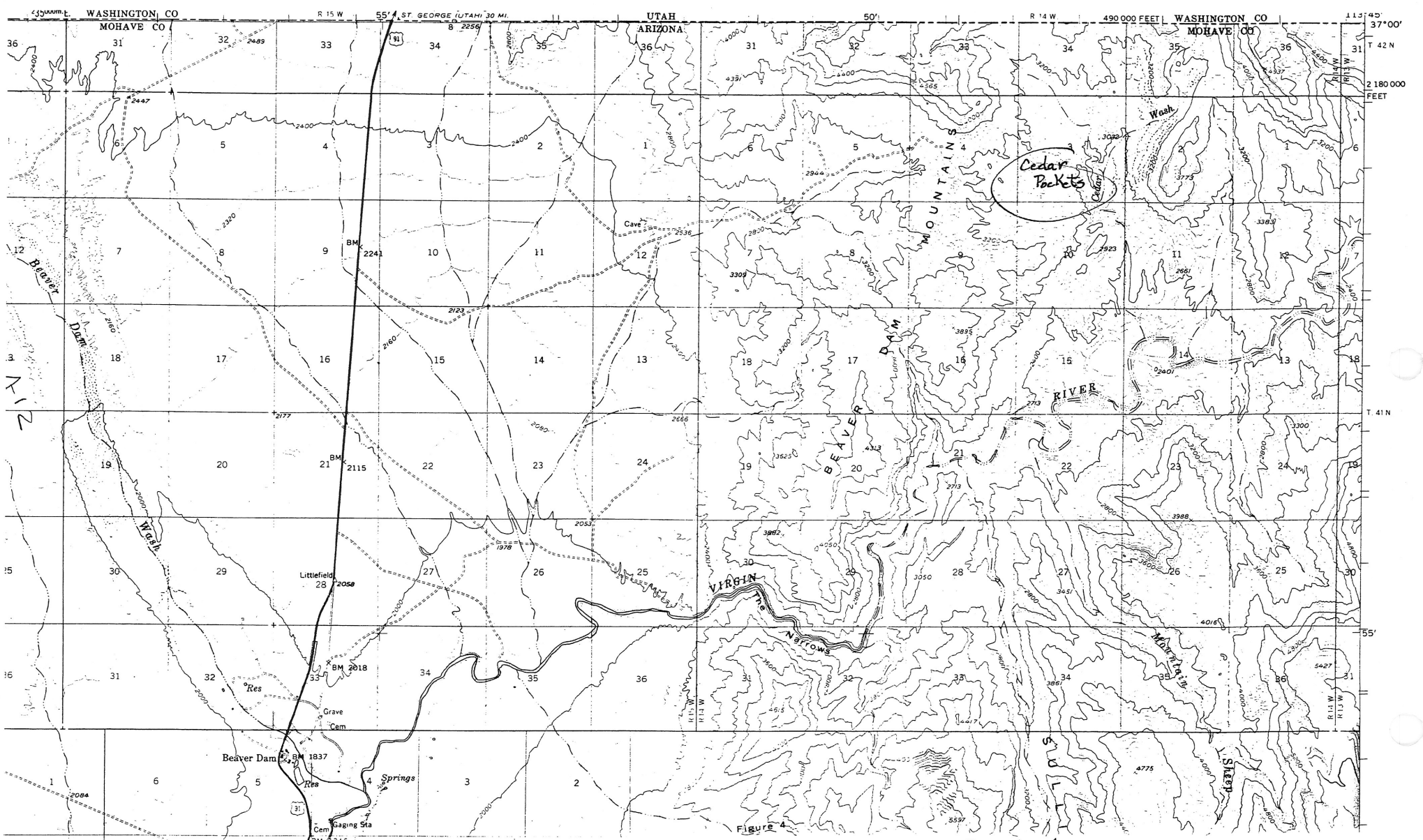
Approximately 1½ to 2 years ago (1984?), Mr. Willsie reportedly leased the property to an operator that mined the deposit for 1-2 months and shipped gypsum to a cement plant in Tehachapi, CA (between Barstow and Bakersfield). The gypsum was sold for \$25-\$30/ton. The property has not been worked since.

Since the Cedar Pockets deposit is in the Beaver Dam Wilderness Area, the BLM has requested that Mr. Willsie submit a plan of operations by Jan 1, 1987.

Date: September 11, 1986

(Signature)

Michael N. Greeley
ADMMR



Littlefield 15' Quad.

UNITED STATES
DEPARTMENT OF THE INTERIOR
BUREAU OF LAND MANAGEMENT

Serial Number
AS-010-84-22P

MINERAL REPORT

*ADmmR
Cedar Pockets file
Mohave County*

CEDAR POCKETS GYPSUM
SOUTHWEST MINERALS INC.
MOHAVE COUNTY, ARIZONA

(Title)

LANDS INVOLVED

T.41N., R.14W. G&SRM
Sec. 3: W $\frac{1}{2}$ NE SW, SE NE SW,
- E $\frac{1}{2}$ SW SW, SW SW SW,
N $\frac{1}{2}$ SE SW

Prepared By:

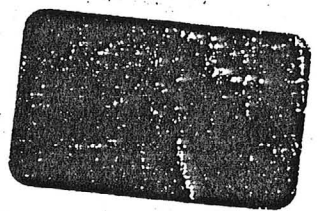
[Handwritten Signature]

(Signature)
GEOLOGIST

(Title)

JULY 1985

(Date)



Technical Approval:

Alan S. Rabinoff

(Signature)
Geologist, Arizona State Office

(Title)
October 11, 1985

Management Acknowledgement:

[Handwritten Signature]

(Signature)
SPLINTS AREA MANAGER

(Title)
October 21, 1985

A-13

Cedar Pockets Gypsum
Southwest Minerals Inc.
Minerals Report

A. Summary and Conclusions

In doing an assessment of this nature it is apparent that the most critical items relate to the quality and quantity of mineral present and its marketability. This evaluation has been done along these lines in addition to a records check to confirm the presence and apparent mining claim holdings.

The presence and to some degree the quality of the subject mineral deposits have been generally known for a number of years. The feeling in the past has been that there is an abundance of top grade gypsum here but due to the distance to market and cost factor in getting it there, the product was not considered to be economically marketable.

At this time trial runs have been made as to actual mining and shipment to market of the product for use in cement production. The actual and estimated costs for overhead, mining and shipping have been weighed against market receipts from the cement companies in Southern California. These figures show that a reasonable return or profit for the investment required can be expected at this time.

Other potential market possibilities for products such as wall board, agriculture and fillers are also being investigated and may prove to be marketable at this time.

It is this writers opinion that the following described portions of C&W Claim No's. 12, 15 and 16 were valid claims at the time the Arizona Wilderness Act, Public Law 98-406, August 28, 1984 was passed. They are also considered to be valid as of the date of this report.

<u>Claim Name</u>	<u>Ser. No.</u>	<u>Portion Considered Valid</u>	<u>Acres</u>
C & W No. 12	67923	W $\frac{1}{2}$, SE $\frac{1}{4}$	30
C & W No. 15	67926	E $\frac{1}{2}$, SW $\frac{1}{4}$	30
C & W No. 16	67927	N $\frac{1}{2}$	<u>20</u>
		Total	80

It has been determined that minerals are in evidence on the portions of claims indicated above and is of such a character that a man of ordinary prudence would be justified in the further expenditure of his labor and means with a reasonable prospect of success in developing a valuable mine.

All indications are that this mineral deposit due to its quality, quantity, accessibility, proximity to market etc., is of such value that it can be mined, removed, and disposed of at a profit.

B. Introduction:

Both the Wilderness Management Policy and Section 8560.38A1e of the Manual require a mineral examination of existing operations to verify validity of unpatented mining claims that fall within a designated wilderness area. A Plan of Operations was submitted to the Arizona Strip District Office by Southwest Minerals Inc., on February 21, 1984 to mine gypsum on a portion of C & W Claims number 12 and 16. This plan was approved on March 14, 1984.

In making a field appraisal of this operation, it has been determined that the apparent mineral for the present operation plus a logical progress for the near future is located on that portion of C&W Claims numbered 12, 15 and 16 as shown on Figure 1. For this reason this validity determination covers this area.

Southwest Minerals Inc., commenced operations soon after their plan was approved and have been working with some short shut down periods since that time.

The Arizona Wilderness Act of 1984 (PL 98-406) designated the general area of these claims as wilderness. This was with exception to an existing access road that goes through the Southwest Minerals Inc., claims. Under present guidelines, operations may continue in cases such as this, pending determination of valid existing rights.

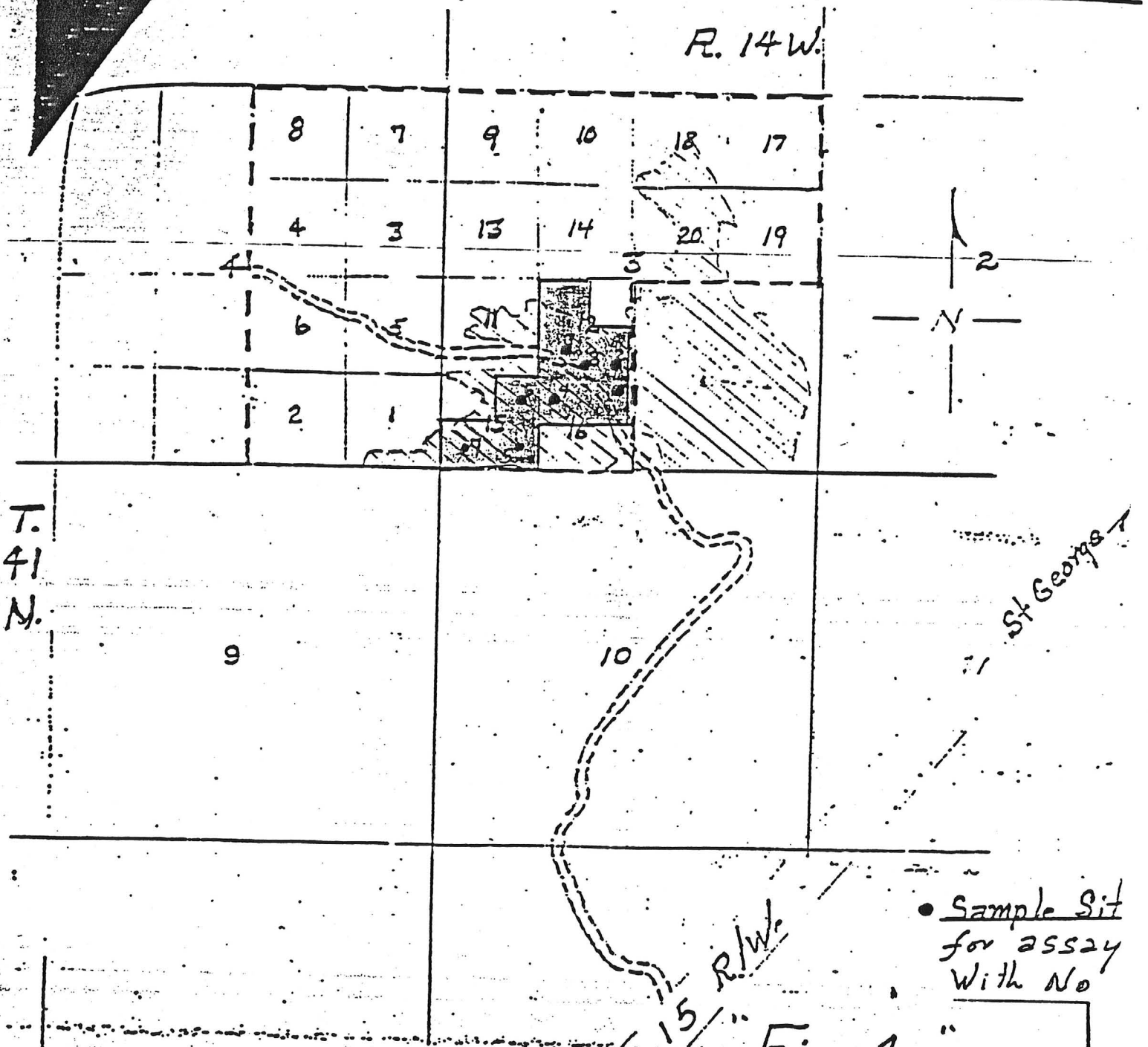
C. Land Involved

The area covered by claims of Curt Willsie (Southwest Minerals Inc.) in this vicinity is as shown in Fig. 1. This report will only consider validity of that portion of the claims colored in blue. These are 40 acre association placer claims and consequently each 10 acres is required to show mineral in character. A description of the legal subdivisions being addressed here is as shown on the title page. A portion of the MTP plat showing the area of the township involved is also attached. The terrain involved is a relatively low valley located in the upper reaches of the Cedar Pockets Wash (see photos No. 1 and No. 2).

D. Status Record Data:


The first claims of record shown in this area are the Reno group, filed in November of 1969 by Dennis Sherrell et. al. They covered the same general area as does the subject CW claims. The latest assessment year of record shown for these is 1984. There is no record however which shows that either labor or notices of intent to hold was filed on these claims for 1981, 1982 or 1983.

The CW claims which this report covers were located in January of 1976 and September of 1977 by Curt Willsie and Delmar Ray Cotton. The latest assessment year for these claims is shown on record as 1984. A copy of the mining claim recordation micro-fish listing the subject claims is attached.




• Sample Sit
for assay
With No

Fig. 1

 Area of Validity Determination
In this report

Cedar Pockets Gypsum
Southwest Minerals Inc., Claims
Scale = 1" = 2000'

MESQUITE

 Gypsum Outcrop
1, 2 etc - Placer Claim A

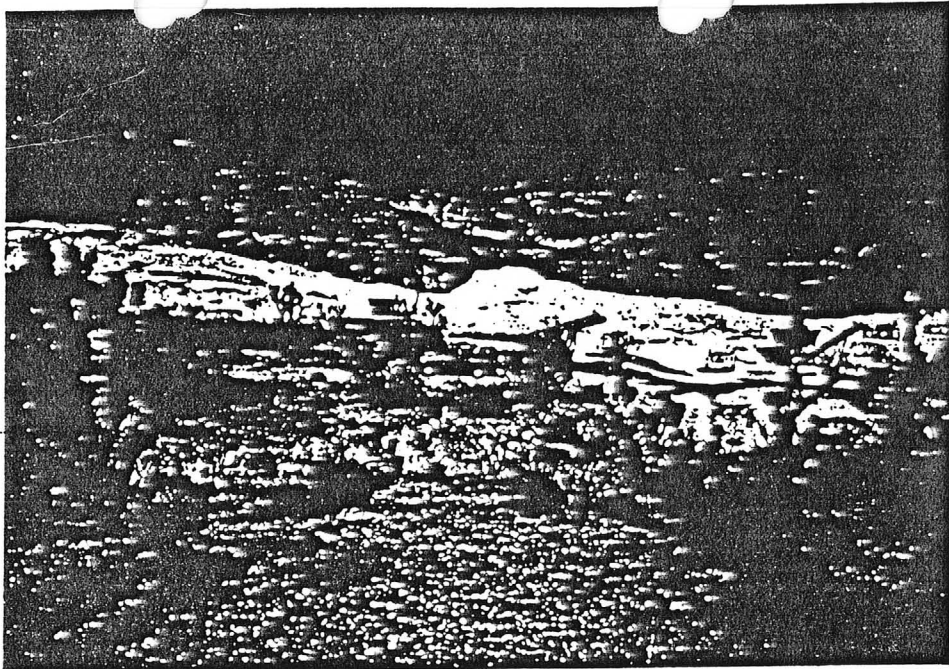


Photo No. 1 - View looking northeast across area of operation. Skyline shows gray colored Kaibab and Toroweap formations underlain by the Supai redbeds. BLM photo by C. Swapp, November 1984.

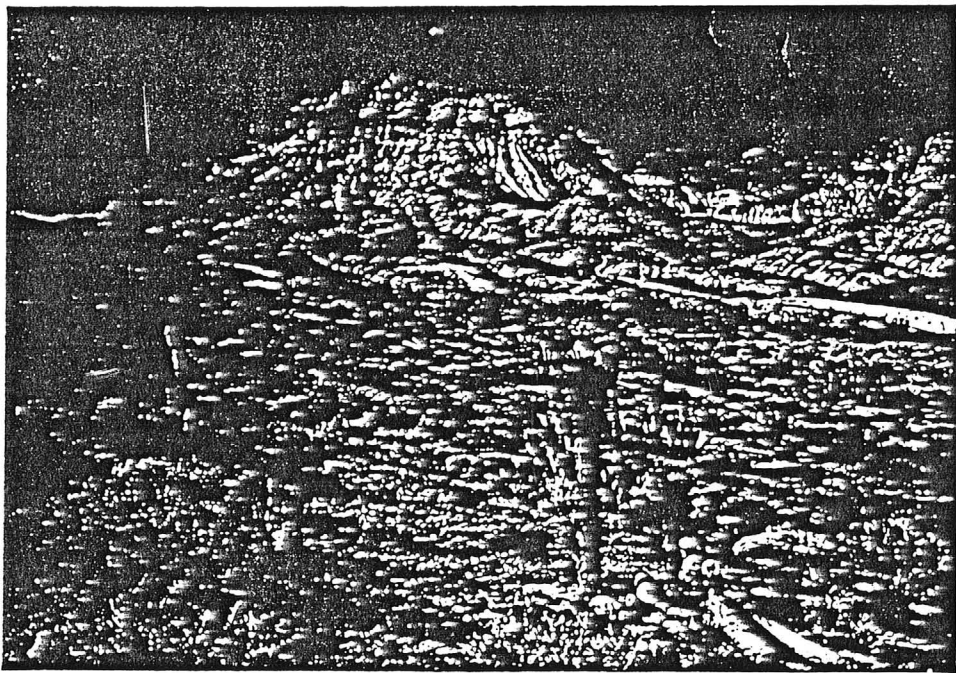


Photo No. 2. View looking northwest with gypsum beds outcropping near right center of photo. BLM photo by C. Swapp, Nov. 1984.

During May of 1979 a group including Harold Friedel et. al. staked placer claims over this same general area. According to the records their last year of recorded assessment was 1984.

On February 10th, 1984 Curt Willsie staked six lode claims which cover portions of the southwest quarter of Section 4, T.4aN., R.14W., G&SRM. These would be over a portion of the placer claims which he and Delmar Ray Cotton filed in 1976 and 1977.

E. General Geology and Mining History

1. General Geology

The Cedar Pockets area in which the subject claims are located lies in the transition zone between the Colorado Plateaus and the Basin and Range Physiographic Provinces. Just a half mile to the east of the claims, the structural conditions are relatively simple with little faulting and a gentle northeast regional dip. Some significant faulting is present in the vicinity of the claims with some thrusting also evident. Limestone beds of the Redwall and Callville formations are dipping at very high angles at various places within a few hundred yards of the CW No. 12 claim.

Stratigraphy in the vicinity of the claims is primarily the Pennsylvanian and Permian Callville limestone overlain in turn by the Supai, Toroweap and Kaibab formations all of Permian age.

The Callville limestone consists primarily of interbedded limestone and dolomitic limestone. Some cross bedded pink sandstone is also present. Locally a lense of gypsum interbedded with dolomitic limestone lies at the top of this formation. Moore¹ shows this gypsum as being up to 200 feet thick and assigns it to the Callville formation.

The Supai formation overlies the gypsum deposits and consists primarily of flat to cross bedded tan to brownish sandstone with an estimated total thickness of about 1000 feet. Above the Supai beds the combined Toroweap and Kaibab formations comprise about 750 feet of limestone, marly and gypsiferous red bed units with minor amounts of siltstones and sandstone.

2. Mining History

The first record of any minerals activity in the general area of the subject claims was on the Reno group of claims mentioned above. Some test pits and channel cuts were made by this group but little or no mining was accomplished.

In January 1976 the CW Group of claims were filed and soon thereafter several truckloads of gypsum was hauled by these operators to California for testing and use as a soil conditioner in agriculture. Several times during the following years, this group took samples in varying amounts to be tested for use in

cement, agricultural, and food and pharmaceutical purposes. During February of 1984 the group holding the CW claims submitted a Plan of Operation to the district office. This plan was approved the following March 14th and a few more truckload shipments were made for cement purposes. This has continued to the present time with the production being very limited until about October when shipments were increased to several truckloads each week. The trucks currently being used are triple trailer bottom dump type which haul about 14 tons in each unit or about 42 tons per truck. This is being shipped to a Las Vegas yard for further distribution to cement plants in southern California.

F. Mining Claim Geology and Mineralization

A geologic map taken from Moore¹ is shown as Fig. 2. Here indicated, the prominent Cedar Wash Fault trends generally northeast through the subject gypsum outcrops. Several secondary faults extend generally west from the Cedar Wash fault in the vicinity of the gypsum claims.

Moore¹ attributes this grouping of faults in the vicinity of the gypsum to the inhomogeneity in formation strain due to the presence of the gypsum.

The gypsum here is actually broken into at least two major blocks by the faulting. The blocks in the vicinity of the present mining are dipping 20-25 degrees to the north.

In order for a deposit to be considered workable or economically feasible under existing conditions, little or no overburden could be present.

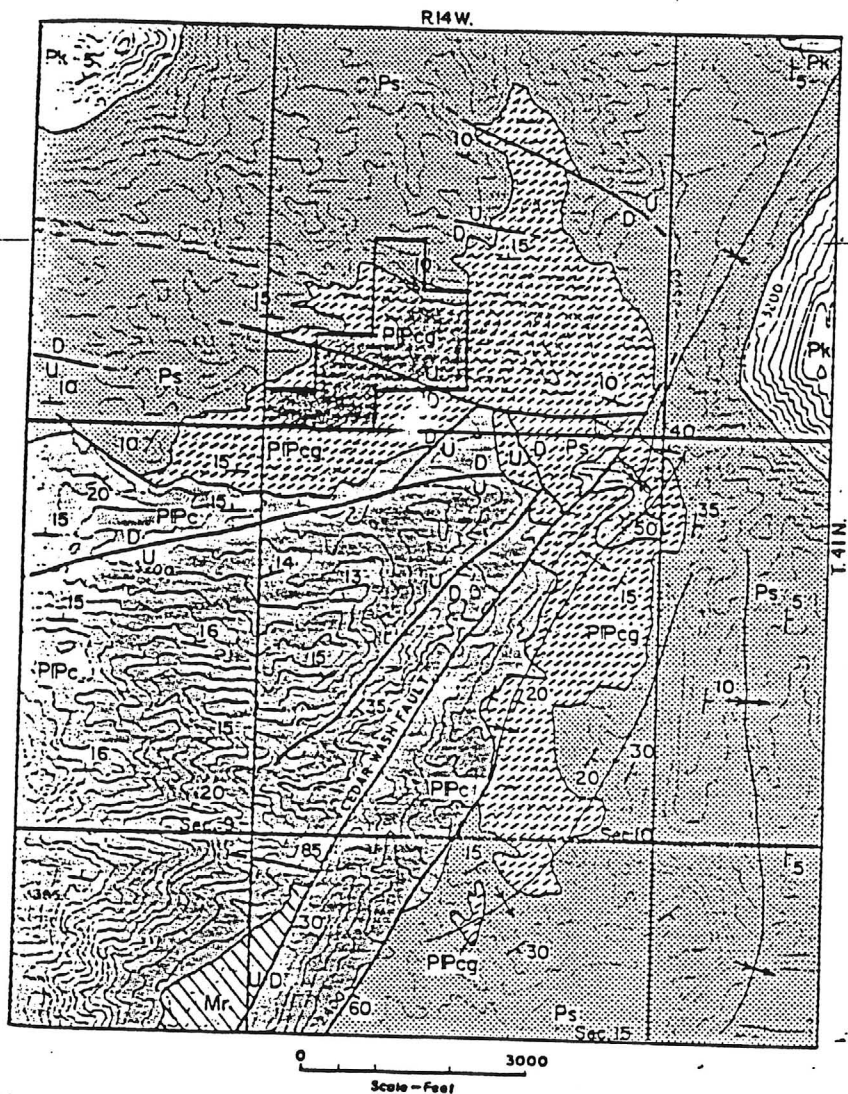



Fig. 2. Geologic map of the Cedar Wash area. Pk, Kaibab Limestone; Ps, Supai Formation; PIPc, Callville Limestone; PIPcg, gypsiferous member of Callville Limestone; Mr, Redwall Limestone.

Taken from Moore, R. T. 1

 *Validity Determination, this report.*

As is shown in Fig. 1, only claims numbered 1, 11, 12, 15, 16, 18 and 20 have significant gypsum exposed as a surface outcrop that may be considered a discovery.

The factors which might tend to make this deposit workable includes its relative purity in given beds, its lack of overburden and its accessibility. This is the only deposit in the area known to occur in this particular stratigraphic position viz., at the top of the Callville limestone. All others in the district that are presently getting some attention as to their possible development occur in the Alpha member of the Permian Kaibab formation.

G. Development & Surface Workings

The present development production being carried on by Southwest Minerals Inc., is somewhat experimental to determine the most feasible and economical methods to be used. There was initially a scalping operation followed by bench select and waste process. The dolomite or dolomitic limestone zones are being pushed off to one side and the select gypsum zones then ripped where possible and drilled and shot where this is not feasible. The broken gypsum material is then moved to an impact crusher and screening unit by a large front end loader. Presently the material is segregated into two size ranges including minus 2 inches down to 3/8 inch in one pile and minus 3/8 inch in a second pile (See Photo No. 3). There is presently a market for both sizes at different cement plants, but the finer material has less demand than does the coarser due largely to ease in handling through hoppers at the cement plants.

Equipment for mining and processing presently at the mine site is as follows:

1. Portable crushing/screening plant (250 ton/hr.) Cedar Rapids 3042.

2. Caterpillar D-8 bulldozer.
3. Front end loader.
4. Screening plant.
5. Stacking conveyors.
6. 250 KV Generator (caterpillar).
7. Air compressor and miscellaneous support materials.
8. Air track drill.
9. Back hoe

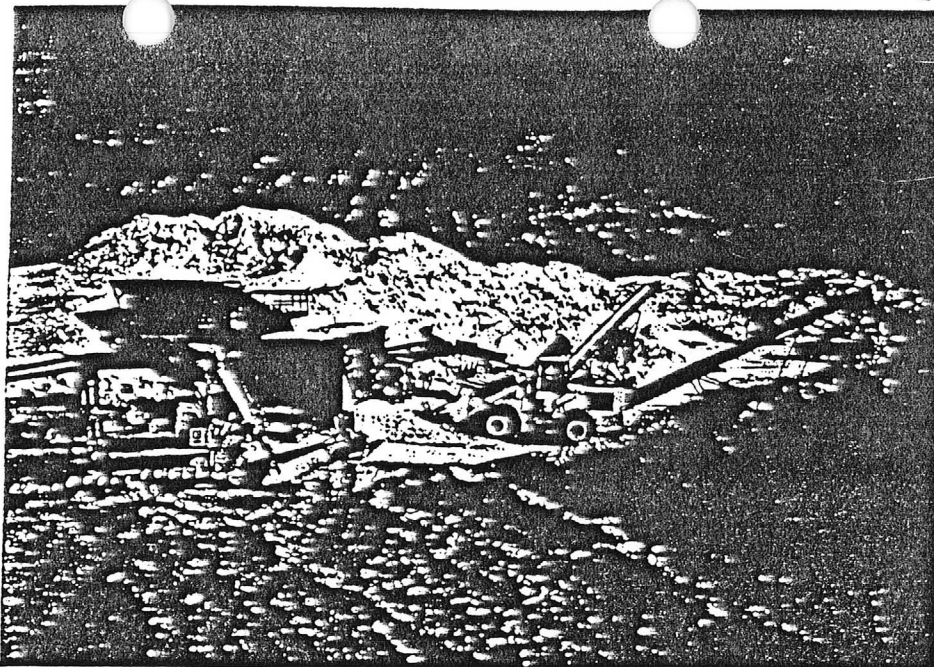


Photo No. 3. View looking northeast showing present operations on placer mining claim C&W No. 12. BLM photo by C. Swapp, Nov. 1984.

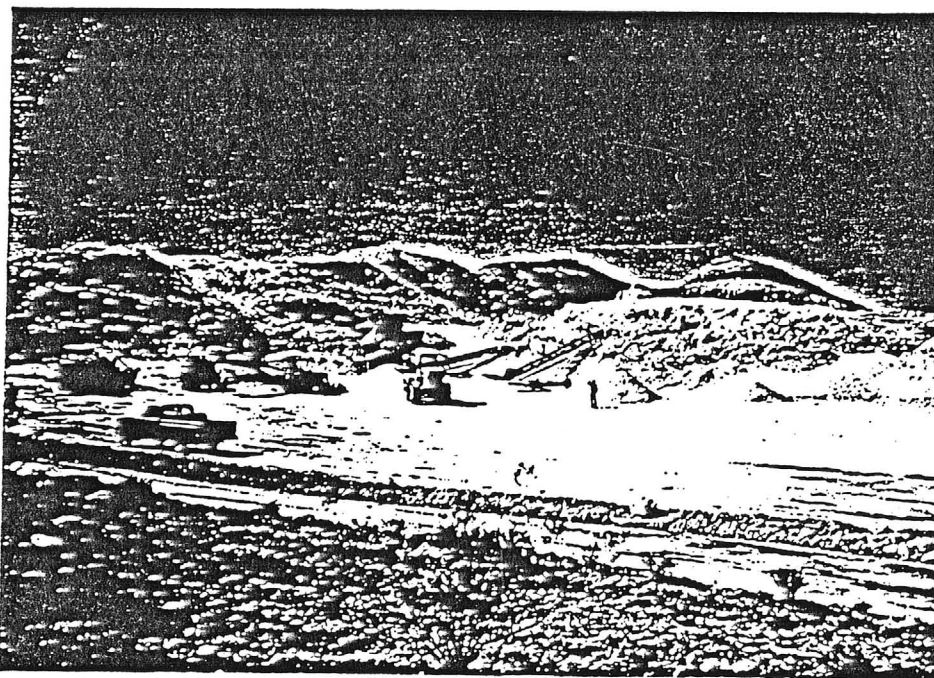


Photo No. 4. Zonah Corp. operations about 10 miles east of the Southwest Minerals properties shown in Photos 1, 2, and 3. BLM photo by C. Swapp, Mar. 1985.

H. Mineral Quality and Quantity

1. Quality

The quality of gypsum in this deposit has been appraised largely for its potential for use in cement production. It does show possible potential for other uses but the proposal at this time is for cement and the market study is also along this line. A common consensus among cement companies relative to gypsum requirements is that the gypsum should assay a minimum of 90% $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. Another quality that they generally require is that the acid insoluble content be under 5%.

Tables 1, 2, and 3 as follows relating to both assays of the subject deposit and other deposits of the United States for comparison are generally self explanatory.

The results of samples taken by this writer in Table 2 are somewhat select. They were taken as vertical chip samples but were limited to the thickness of zones showing better quality. With some very selective mining, this could be achieved. Sample No. 8 however is a random grab sample from the fines stockpile ready for shipment and is probably more representative of what would be expected under normal control.

Table 1

--Analyses of gypsum and gypsite samples from the Lime Hills
Wilderness Study Areas, Mohave County, Arizona

[Components determined by chemical analysis; values reported in percent; n.a., not analyzed]

Sample No.	Sample type (feet)	CaSO ₄	Water		Iron	Sodium	Magnesium	Manganese
			Free and Combined	Insol. residue				
NARROWS WILDERNESS STUDY AREA								
NA 0100	Chip, vertical (4.0)	78.4	20.51	2.00	.04	.02	.03	.001
NA 0101	Chip, vertical (2.0)	67.3	21.31	5.90	.18	.13	.65	.002
NA 0102	Chip, vertical (4.0)	72.4	19.34	n.a.	.31	.04	5.10	.006
NA 0103	Chip, vertical (10.0)	71.3	20.61	n.a.	.14	.03	.63	.002
NA 0104	Select	77.8	26.86	0.010	.02	.01	.02	.001
NA 0105	Select	76.1	20.69	1.4	.03	.01	.02	.001
NA 0106	Select	77.4	20.83	1.70	.005	.01	.12	.001

Taken from U.S. Bureau of Mines Open File
Report by John P. Briggs - (MLA 21-83, 1983)
Note For site location of the above samples see Fig. 3

Table 2

Assay of Gypsum Samples taken from locations as shown by "Figure 1",
Cedar Pockets area, Mohave Co., Arizona, May 1985.

Sample No.	Sample Type (feet)	% Acid Insoluble	% SO ₃	% CaSO ₄ 2H ₂ O
1	Vertical Chip 3'	0.20	46.00	98.92
2	Vertical Chip 2'	0.22	45.79	98.47
3	Vertical Chip 2'	0.22	45.61	98.08
4	Vertical Chip 2'	0.12	46.29	98.54
5	Vertical Chip 3'	0.16	46.03	98.98
6	Vertical Chip 2'	0.12	46.11	99.15
7	Vertical Chip 2'	0.12	46.20	99.35
8	Random Grab Stockpile	1.64	45.11	97.00

Table 3

Commerical deposits of gypsum vary in SO₃ content and nature of impurities. The following analyses are representative of important sources of gypsum used by portland cement companies in the United States. (Hansen 3)

AREA	Gypsum CaSO ₄ 2H ₂ O	Anhydrite CaSO ₄	Dolomite		Iron & Aluminum Oxides R ₂ O ₃	Silica & Insoluable
			Calcium Carbonate CaCO ₃	Magnesium Carbonate MgCO ₃		
Western NY Ontario (Oakfeld)	88.1%	3.4%	7.0%		.3%	1.2%
Oklahoma Kansas (Southard)	97.0%	1.7%	.4%		.5%	.4%
Iowa (Ft. Dodge)	94.4%	-	.6%		.3%	4.7%
Nova Scotia (Windsor)	92.0%	2.1%	3.5%		.5%	1.9%
Michigan (Alabaster)	94.8%	-	2.2%		.5%	2.5%
California (Plaster City)	95.6%	1.2%	1.3%		.2%	1.7%

A-29

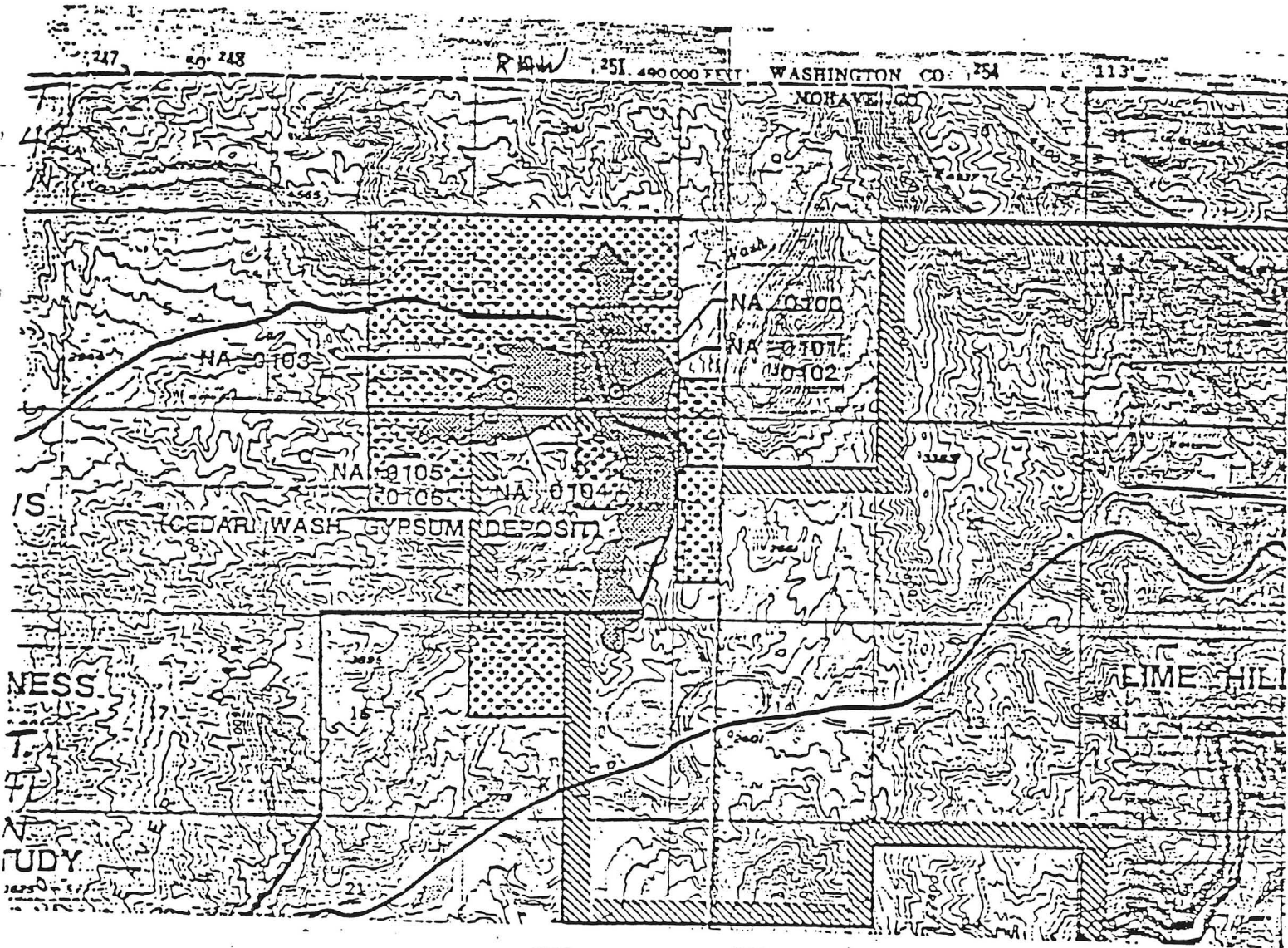


Figure 3
Plat showing sample locations
for analyses shown on "Exhibit C"

From U. S. Bureau of Mines

Open File Report MLA 21-53-1983

Several other assays made by cement companies presently buying gypsum from this deposit which shows the quality to be comparable to required standards for cement use are on file in the district office.

The overall results of these assays indicate that the grade of gypsum here is adequate for use in cement production.

2. Quantity

Briggs² reports that this deposit contains about 300 feet of massive granular gypsum, interbedded with numerous limestone beds 6 inches to 4 feet thick and crops out over an area of about 480 acres within and adjacent to the area of this report. Disregarding the dolomitic limestone interbeds and assuming a uniform thickness of 300 feet and a bulk density of 144 lbs. per cubic foot, his estimate shows the deposit contains about 450 million tons of gypsum.

A section of the gypsiferous zone was recently measured and along the east edge of claims no. 12 and 16 by the writer. The total section here comes closer to the 200 feet mentioned by Moore¹ in his report. Furthermore only the upper 100 feet of this appears to contain gypsum of a thickness and purity that would tend to make it economically feasible to mine. It is further estimated that only 50% of this 100 feet would contain mineable gypsum. In considering erosion which has removed an estimated 50% of this 100 feet from

the area being considered for validity, this would leave an average of 25 feet of mineable gypsum over an area of 80 acres. Using a figure of 150 lbs. per cubic foot for in place gypsum this gives a figure of about 6.5 million tons of mineable reserves in the area being evaluated in this report.

I. Mineral Evaluation and Marketability:

It is apparent that a relatively large quantity of high quality gypsum is present on the portion of the claims being evaluated in this report. Assays of the mineral here would indicate that it is of a quality satisfactory for such uses as cement production, agriculture, plaster, wallboard and fillers.

A mining method lending the best efficiency and economy will have to be determined and used. Market distance and transportation costs are also a prime factor in this evaluation.

The possibility of marketing this mineral for several possible uses is being investigated by the present operators. They are presently setting up a bagging plant in Las Vegas to bag for agricultural use. They feel that the profit from this outlet will be greater than the present portland cement market. Because of the more readily available production, transportation and overhead cost factors associated with the cement market and because this is the actual market being served at this time, this outlet will be used in helping to determine the marketability of the product.

The following is a cost estimate for mining, processing and shipping of the gypsum material in bulk form to a distribution yard in Las Vegas, Nevada

A. Mining and Processing

It is difficult to obtain comparative mining and processing cost estimates that would be closely analogous to the Cedar Pockets operation.

A table of surface mine model cost summaries shown by "Mining Cost Service" and published by Western mine Engineering does give some cost estimates for some open pit operations. The condition variables are so great however that it makes comparison difficult. Cost estimates on models given that might conceivably have some comparison to the Cedar Pockets operations range from \$2.84 to \$12.42 per ton for mining. The model producing ore for \$2.84 per ton is a 10,000 ton per day operation but requires waste-ore haulage of over one mile and a stripping ratio of 1:1. The model producing ore for \$12.42 per ton is a 500 ton per day operation with a stripping ratio of 1:1 and an ore-waste haul distance of 1300 feet. Both of these operations required very significant pre-production overburden removal and worked in pits from 400 to 150 feet deep.

The Cedar Pockets operation required relatively little overburden removal and has a stripping waste ratio of less than 1:1. The present distance required for ore and waste moving is 200 feet or less. With this in mind it is estimated that the ore from this operation can be mined and crushed for three to four dollars per ton.

Southwest Minerals/White Mountain Gypsum has prepared a cost estimate breakdown which includes mining, transporting and sales which is attached in "Exhibit A". This writer has reviewed this and finds no significant discrepancies with the exception of one modification as shown in it where a maintenance cost factor was added to the equipment list.

In summary of the two analysis, it is estimated that this gypsum can be placed in crushed form in Las Vegas for about six to seven dollars per ton.

B. Transportation

In trying to get some estimate of cost for transporting gypsum from the Cedar Pockets mine to Las Vegas, the best analogy available is a freight rate listing from the Iron Horse Equipment Corporation of Victorville, California (see "Exhibit B"). This listing indicates a rate of \$11.95 per ton for bulk gypsum from Apex, Nevada to the

Monolith Cement Company in California. This is a distance of about 265 miles and would break down to a rate of about .045 cents per ton mile. This rate is listed for a minimum load of 20 tons. The Southwest Minerals/White Mountain Gypsum estimates include using their own equipment and hauling 42 ton per trip. This results in a figure of .031 cents per ton mile which is considered to be within the realm of probability. This is especially true when it is realized that they are not required to make a profit on the haul but only to cover costs.

Using this estimate and a figure of 100 miles for the distance from the mine to Las Vegas it would cost \$3.10 per ton for shipping the gypsum to Las Vegas.

C. Marketability

Southwest Minerals Inc., through their mine operator White Mountain Gypsum have established a selling price for their product at \$10.00 per ton F.O.B. Las Vegas, Nevada. They are presently selling to four different cement companies of Southern California. These cement companies are shipping bulk cement into Las Vegas and then buying the gypsum there and using it as a backhaul to their respective plants. A copy of a purchase order agreement from one of these companies is attached as "Exhibit C".

In reviewing "Exhibit A" along with the information given above, the following evaluation is made.

<u>Item</u>	<u>Cost Est/Ton (Range)</u>
Mine & crush	\$3.00 - \$4.00
Transport	\$3.00 - \$3.10
Las Vegas Term Oper	\$.65 - \$.65
Las Vegas Admin. etc.	\$1.80 - \$1.80
	<u>\$8.45</u> <u>\$9.55</u>

It can be seen that a net profit of \$4455.00 to \$14,355.00 per month might be expected from this operation when it is producing at a level of 9900 ton per month as proposed.

Another company (Zonah Corp.) has set up operations for producing gypsum in the general area and are about ready to start shipping their product. Their operation is located about 10 miles east of those discussed in this report (see photo no. 4). These deposits occur in the Permian Kaibab formation but are quite comparable to the subject operation relative to quality and marketing conditions. Their present intent is to ship both into central Utah and southern California. This operation is mentioned only to show that other interests also feel that the type and quality of gypsum in this area is marketable.

References

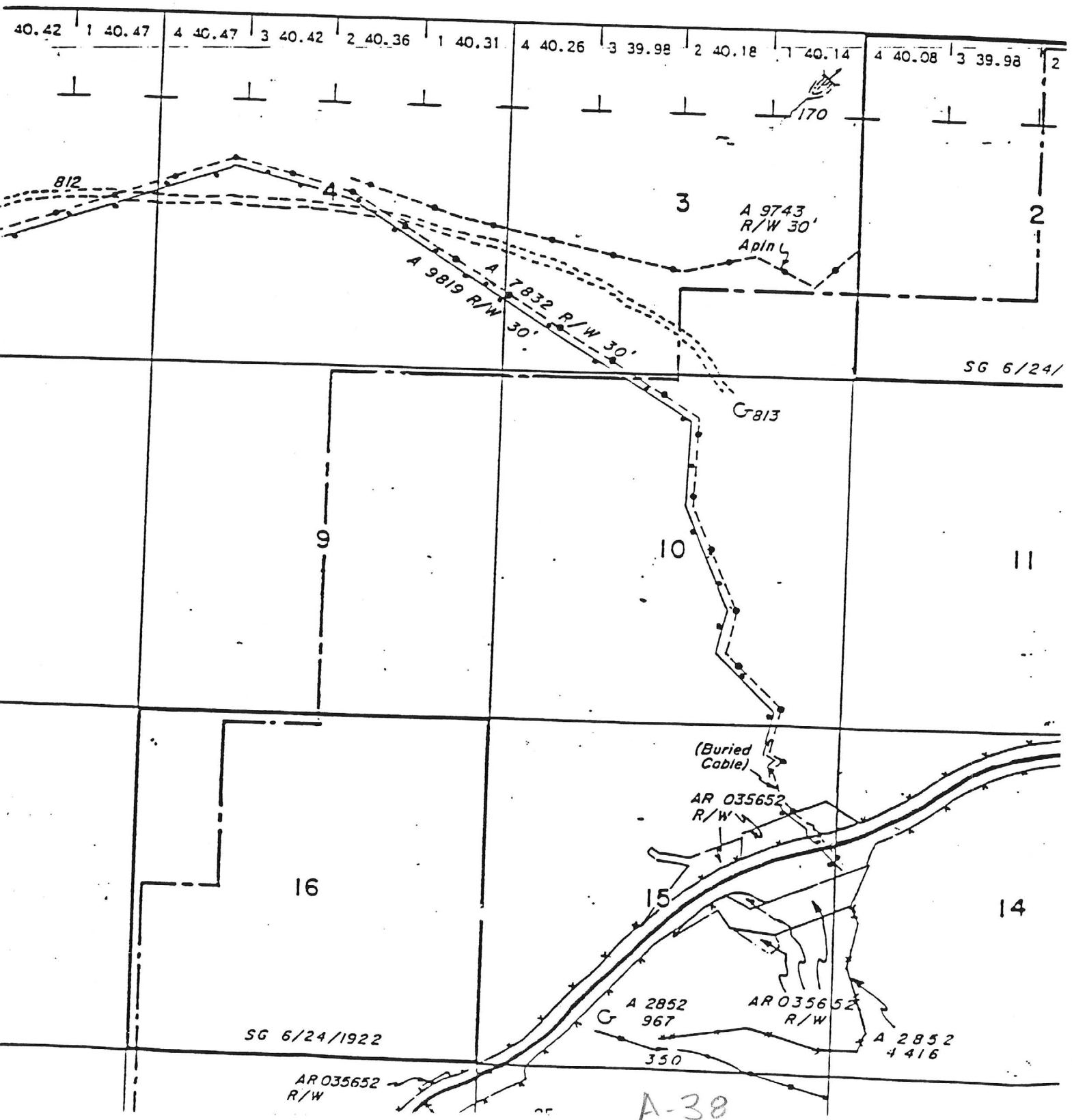
1. Moore, R. T., 1972, Geology of the Virgin and Beaver Dam Mountains, Arizona: Arizona Bureau of Mines Bulletin 186, P. 15.

2. Briggs, John P., 1983, Mineral Investigations of the Lime Hills, Narrows, and Sand Cove Wilderness Study Areas.
3. Hansen, W. C. and Offutt, J. S., Gypsum and Anhydrite in Portland Cement, 2nd Edn., published by U.S. Gypsum, (Date unknown).

TOWNSHIP 41 NORTH RANGE 14 WEST OF THE

M T P

MOHAVE COUNTY



39 N 14 W 19 NW

L 13

REPORT DATE: MAR 12, 1985
STATE: ARIZONA

PCN: LT892PP1

UNITED STATES DEPARTMENT OF THE INTERIOR
BUREAU OF LAND MANAGEMENT

PAGE NO: 4511

MERIDIAN: GILA-SALT R.

GEOGRAPHIC INDEX

- LEGAL DESCRIPTION -		- GEO BLM SERIAL CASE		CLAIM NAME/NUMBER	CLAIMANT(S)	LEAD FILE	COUNTY BOOK; PAGE	LOCATION DATE	LATEST ASSESS-YR	CASE CLOSED
TOWNSHIP	RANGE	SEC	SUBDV							
39 N	14 W	19 NW	15	1	138042 LD	GREEN ROSE #1	GILMER JIM ADVANCED METALS ADAMS CLEO	736; 849	8/06/1981	1984
		NW			138043 LD	GREEN ROSE #2	CAPLE CARVER GILMER JIM ADVANCED METALS	138042 736; 849	8/07/1981	1984
		NE			138044 LD	GREEN ROSE #3	ADAMS CLEO CAPLE CARVER GILMER JIM ADVANCED METALS	138042 736; 851	8/07/1981	1984
		S2			138045 LD	GREEN ROSE #4	ADAMS CLEO CAPLE CARVER GILMER JIM	138042 736; 852	8/06/1981	1984
		NW			138046 LD	GREEN ROSE #5	ADVANCED METALS ADAMS CLEO CAPLE CARVER GILMER JIM ADVANCED METALS	138042 736; 853	8/06/1981	1984
	20 N				72549 LD	ROADRUNNER #5	ADAMS CLEO EBY BRETT M	72545 0-0	2/24/1984	1984
	NW				72550 LD	ROADRUNNER #6		72545 0-0	2/24/1984	1984
	NW				72551 LD	ROADRUNNER #7		72545 0-0	2/24/1984	1984
	NW				72556 LD	ROADRUNNER E EXT #3		72545 6-J; 430	6/27/1984	1984
	NW				72559 LD	EAST #3		72545 6-J; 428	6/27/1984	1984
	W2				72560 LD	LEAD #1		72545 6-J; 416	7/01/1984	1984
	W2				72561 LD	LEAD #2		72545 6-J; 417	7/01/1984	1984
41 N	14 W	3 SE			52628 PL	FRIEDEL GYPSITE NO 6	FRIEDEL HAROLD FRIEDEL SHERRY FRIEDEL THOMAS FRIEDEL HAROLD M JR FRIEDEL BARBARA C DALEY DEBRA F DALEY MICHAEL O FRIEDEL JAMES R	52623 559; 43	5/10/1979	1984
		S2			52629 PL	FRIEDEL GYPSITE NO 7	FRIEDEL HAROLD FRIEDEL SHERRY FRIEDEL THOMAS FRIEDEL HAROLD M JR FRIEDEL BARBARA C DALEY DEBRA F DALEY MICHAEL O FRIEDEL JAMES R	52623 559; 45	5/10/1979	1984
		SW			52630 PL	FRIEDEL GYPSITE NO 8	FRIEDEL HAROLD FRIEDEL SHERRY FRIEDEL THOMAS FRIEDEL HAROLD M JR FRIEDEL BARBARA C	52623 559; 47	5/10/1979	1984

*** DISCLOSURE *** ALL INFORMATION RECEIVED IN THIS OFFICE MAY NOT YET BE LISTED ON THIS REPORT. NAMES AND ADDRESSES ARE ENTERED AS THEY APPEAR ON THE LOCATION NOTICE OR ARE ABBREVIATED TO FIT LIMITED SPACE; THEREFORE THEY MAY NOT APPEAR IN THE EXPECTED SEQUENCE. A BLANK LATEST ASSESSMENT YEAR IN THIS REPORT DOES NOT CONSTITUTE AN ABANDONED CLAIM.

-26-

A-39

41 N 14 W 3 SW

REPORT DATE: MAR 12, 1985
STATE: ARIZONA

PCN: LT892PP1

UNITED STATES DEPARTMENT OF THE INTERIOR
BUREAU OF LAND MANAGEMENT

M 13

PAGE NO: 4512

MERIDIAN: GILA-SALT R.

GEOGRAPHIC INDEX

LEGAL DESCRIPTION - GEO BLM SERIAL CASE
TOWNSHIP RANGE SEC SUBDV CTY DIST NO. TYPE CLAIM NAME/NUMBER CLAIMANT(S)

LEAD COUNTY LOCATION LATEST CASE
FILE BOOK;PAGE DATE ASSESS-YR CLOSED

TOWNSHIP	RANGE	SEC	SUBDV	CTY	DIST	NO.	TYPE	CLAIM NAME/NUMBER	CLAIMANT(S)	LEAD FILE	COUNTY BOOK;PAGE	LOCATION DATE	LATEST ASSESS-YR	CASE CLOSED
1 N	14 W	3 SW	15	1		52630	PL	FRIEDEL GYPSITE NO. 8	D'LEY, DEBRA F. DALZY MICHAEL O. FRIEDEL JAMES R.	52623	559;47	5/10/1979	1984	
		SE				53220	PL	REMO GYPSUM CL #1	SHERRELL DENNIS RICCONINI MICHAEL RICCONINI LUCILLE SIEMENS GARY JONES ELDRIDGE W JONES ELDRIDGE D JANAS LAURA	53219	6-S;61	11/13/1969	1980	
		SW				53221	PL	NO GYPSUM CL #2	CROWTHER TEDDEE J SHERRELL DENNIS RICCONINI MICHAEL RICCONINI LUCILLE SIEMENS GARY JONES ELDRIDGE W JONES ELDRIDGE D JANAS LAURA CROWTHER TEDDEE J	53219	6-S;62	11/13/1969	1980	
		NW				67920	PL	C & W NO 9	WILLSIE CURT	67912	313;716	1/30/1976	1984	
		NW				67921	PL	C & W NO 10	GERAWAN RAY WILLSIE CURT	67912	313;717	1/30/1976	1984	
		SW				67922	PL	C & W NO 11	GERAWAN RAY WILLSIE CURT	67912	313;718	1/30/1976	1984	
		SW				67923	PL	C & W NO 12	GERAWAN RAY WILLSIE CURT	67912	421;702	9/30/1977	1984	
		NW				67924	PL	C & W NO 13	GERAWAN RAY WILLSIE CURT	67912	313;720	1/30/1976	1984	
		NW				67925	PL	C & W NO 14	GERAWAN RAY WILLSIE CURT	67912	313;721	1/30/1976	1984	
		SW				67926	PL	C & W NO 15	GERAWAN RAY WILLSIE CURT	67912	313;722	1/30/1976	1984	
		SW				67927	PL	C & W NO 16	GERAWAN RAY WILLSIE CURT	67912	313;723	1/30/1976	1984	
		NE				67928	PL	C & W NO 17	GERAWAN RAY WILLSIE CURT	67912	313;724	1/30/1976	1984	
		NE				67929	PL	C & W NO 18	GERAWAN RAY WILLSIE CURT	67912	313;725	1/30/1976	1984	
		NE				67930	PL	C & W NO 19	GERAWAN RAY WILLSIE CURT	67912	421;709	9/30/1977	1984	
		NE				67931	PL	C & W NO 20	GERAWAN RAY WILLSIE CURT	67912	313;727	1/30/1976	1984	
		SW				215235	LD	G W #2	GERAWAN RAY	215234	998;41	2/09/1984	0000	
		SW				215236	LD	G W #3	WILLSIE CURT	215234	998;43	2/10/1984	0000	
		SW				215237	LD	G W #4	WILLSIE CURT	215234	998;45	2/10/1984	0000	

* * DISCLOSURE * * ALL INFORMATION RECEIVED IN THIS OFFICE MAY NOT YET BE LISTED ON THIS REPORT. NAMES AND ADDRESSES ARE ENTERED AS THEY APPEAR ON THE LOCATION NOTICE OR ARE ABBREVIATED TO FIT LIMITED SPACE; THEREFORE THEY MAY NOT APPEAR IN THE EXPECTED SEQUENCE. A BLANK LATEST ASSESSMENT YEAR IN THIS REPORT DOES NOT CONSTITUTE AN ABBREVIATION.

-27- A-40

41 N 14 W 3 SW

REPORT DATE: MAR 12, 1985
STATE: ARIZONA

PCN: LT892PP1

UNITED STATES DEPARTMENT OF THE INTERIOR
BUREAU OF LAND MANAGEMENT

N 13

PAGE NO: 4513

MERIDIAN: GILA-EALT R.

GEOGRAPHIC INDEX

LEGAL DESCRIPTION - GEO BLM SERIAL CASE
TOWNSHIP RANGE SEC SUBDV CTY DIST NO. TYPE CLAIM NAME/NUMBER CLAIMANT(S)

LEAD COUNTY LOCATION LATEST CASE
FILE BOOK;PAGE DATE ASSESS-YR CLOSED

TOWNSHIP	RANGE	SEC	SUBDV	CTY	DIST	NO.	TYPE	CLAIM NAME/NUMBER	CLAIMANT(S)	LEAD FILE	COUNTY BOOK;PAGE	LOCATION DATE	LATEST ASSESS-YR	CASE CLOSED
41 N	14 W	3 SW		15	1	215238	LD	G W #5	WILLSIE CURT	215236	998; 47	2/10/1984	0000	
		SW				215239	LD	G W #6	GERAWAN ROY	215237	998; 49	2/10/1984	0000	
		SW				215322	LD	G W #7			998; 673	2/11/1984	0000	
		S2				215323	LD	G W #8			998; 675	2/10/1984	0000	
		SE				215324	LD	G W #9			998; 677	2/10/1984	0000	
		S2				215325	LD	G W #10			998; 679	2/12/1984	0000	
		SE				215326	LD	G W #11			998; 681	2/12/1984	0000	
		SE				215327	LD	G W #12 FRAC			998; 683	2/12/1984	0000	
		4 SE				52630	PL	FRIEDEL GYPSITE NO 8	FRIEDEL HAROLD FRIEDEL SHERRY FRIEDEL THOMAS FRIEDEL HAROLD M JR FRIEDEL BARBARA C DALEY DEBRA F DALEY MICHAEL O FRIEDEL JAMES R	52623	559; 47	5/10/1979	1984	
		S2				52631	PL	FRIEDEL GYPSITE NO 9	FRIEDEL HAROLD FRIEDEL SHERRY FRIEDEL THOMAS FRIEDEL HAROLD M JR FRIEDEL BARBARA C DALEY DEBRA F DALEY MICHAEL O FRIEDEL JAMES R	52623	559; 49	5/10/1979	1984	
		SE				53222	PL	REMO GYPSUM CL #3	SHERRILL DENNIS RICCOMINI MICHAEL RICCOMINI LUCILLE SIEMENS GARY JONES ELDRIDGE W JONES ELDRIDGE D JANAS LAURA CROWTHER TEDDEE J	53219	6-5; 63	11/13/1969	1980	
		SE				67912	PL	C & W NO 1	WILLSIE CURT		313; 708	1/30/1976	1984	
		S2				67913	PL	C & W NO 2	GERAWAN RAY		313; 709	1/30/1976	1984	
		NE				67914	PL	C & W NO 3	WILLSIE CURT	67912	313; 709	1/30/1976	1984	
		NE				67915	PL	C & W NO 4	GERAWAN RAY	67912	313; 710	1/30/1976	1984	
		SE				67916	PL	C & W NO 5	WILLSIE CURT	67912	313; 711	1/30/1976	1984	
		SE				67917	PL	C & W NO 6	GERAWAN RAY	67912	313; 712	1/30/1976	1984	
		NE				67918	PL	C & W NO 7	WILLSIE CURT	67912	313; 713	1/30/1976	1984	
									GERAWAN RAY	67912	313; 714	1/30/1976	1984	

** DISCLOSURE ** ALL INFORMATION RECEIVED IN THIS OFFICE MAY NOT YET BE LISTED ON THIS REPORT, NAMES AND ADDRESSES ARE ENTERED AS THEY APPEAR ON THE LOCATION NOTICE OR ARE ABBREVIATED TO FIT LIMITED SPACE; THEREFORE THEY MAY NOT APPEAR IN THE EXPECTED SEQUENCE. A BLANK LATEST ASSESSMENT YEAR IN THIS REPORT DOES NOT CONSTITUTE AN ABANDONED CLAIM.

-28-

A-41

May 1985

"EXHIBIT A"

Cost estimates as supplied by Southwest Minerals/White Mtn. Gypsum for mining, transporting and selling of gypsum from the Cedar Pockets area (based on producing 450 ton per day) is as follows:

A. Mining - Processing & Loading

1. Equipment (used)

<u>ITEM</u>	<u>COST</u>
D-8 Cat. w/dozer	\$ 50,000
Air tractor drill w/ compressor	20,000
Excavator	25,000
988 Cat. loader	150,000
J-D 410 breaker hoe	30,000
Crusher/screen plant, 250 ton/hr- Cedar Rapids	170,000
*Misc. Equipment & Maintenance	75,000
TOTAL	<u>\$550,000</u>

The above equipment cost would be amortized over a 5-year period

$$\frac{\$550,000}{60 \text{ (month)}} = \$9,167/\text{month} \quad \frac{9,167}{9900 \text{ T/mo.}} = \$.93/\text{ton}$$

2. Fuel Costs

Diesel fuel - 3759/day @ \$1.00/gal. = \$375.00 / 450 T. = \$.84/T

3. Labor

4 Men @ \$10.00/hr. = 1 hr. (average) = \$40.00/hr
 - \$40.00 X 8 hrs. = \$320.00/day / 450 T. = \$.72/hr.

4. Explosives

7' X 7' pattern for drilling with 10' deep holes. This would require 64 holes for 49' X 49' area.

Cost Hole

Dynamite	=	\$1.00
Nitrate	=	.75
Cord	=	1.50
		TOTAL \$3.25/hole
\$3.25 X 64		= 208.00 / 450/ton = \$.46/ton

*\$50,000 was added to this figure by this writer to cover maintenance costs.

Total Mining & Crushing Costs

<u>Item</u>	<u>Cost</u>
Equipment & Maintenance	\$.93
Labor	.72
Fuel	.84
Explosives	.46
Misc. Cost	.13
TOTAL	<u>\$3.08</u>

* \$50,000 was added to this figure by this writer to cover maintenance costs.

B. Transportation Cost (Mine to Las Vegas) Haul units consist of two or three axle tractors with three bottom dump trailers with each unit. Total payload per truck would be 42 ton.

Two trucks @4 trips per day/truck for 6 days = 48 trips plus 2 trips each on Sunday = 54 trips X 42 ton = 2268 ton per week for approx. 450 ton/day.

Cost of Trucking Units = \$40,000 for tractor and trailers - amortized over 36 months - $\$40,000 / 36 = \$1,111/\text{month} = 112 \text{ loads/month} = \$9.92/\text{load}$.

Insurance Cost = \$3,523/yr. /12 months =
\$294/mo./112/loads=\$2.62/load

Labor Cost = \$40/load.

Tires = \$16/load

Fuel = \$45/load

Maintenance = \$8/load

Misc. = \$4.46/load

C. Las Vegas Terminal Cost

<u>Item</u>	<u>Cost/mo</u>
Yard Rent	\$1500.00
Labor for 10/hr day X22 days @8.00	1700.00
Equipment = 2-2/2 yd. loader @\$30,000, 5 yr amortization \$30,000 =	500.00
Fuel Cost	2200.00
Utilities	200.00
Misc.	200.00
TOTAL	<u>\$6,300.00</u>

grams per tonne and occurs in the alluvial gravels along the Beaver Dam Wash. Exploration for these gold deposits is taking place immediately north of the Arizona Strip District in Utah (Spooner, 1988). Based on the geologic environment, the inferred geologic processes, and reported occurrence of gold in this area, the alluvial material along Beaver Dam Wash has a moderate potential for the occurrence of gold. Gold exploration is occurring although development potential is speculative at the present time.

Breccia pipe related precious and base metal deposits are known to occur along the lower Grand Wash Cliffs and Virgin Mountains. These deposits reportedly contain copper (up to 23 percent), silver (up to 10 ounces/ton), and relatively minor amounts of lead, zinc, uranium, and gold (Keith and others, 1983). Germanium and Gallium are also known to occur in the Apex deposit in Utah (Bernstein, 1986). It is possible that these elements could occur in breccia pipes located along the lower Grand Wash Cliffs and Virgin Mountains. Based upon the geologic environment, inferred geologic processes and mines in these areas, they have been rated as having a high potential for the occurrence of metallic mineral resources (Map III-3). The available data provide abundant direct and indirect evidence to support the existence of the resource.

URANIUM

Exploration for and development of uranium resources are currently the most active mineral related operations on the district. There are two mines in operation, three in various stages of development, and three that have been closed and reclaimed. These mines lie to the north and west of the Kanab Creek drainage. The uranium occurs in collapse features known as breccia pipes (Figure III-1).

Breccia pipes in the Arizona Strip originate in Red-wall Limestone and form collapse features in overlying rocks as young as the Chinle Formation. Uranium mineralization occurs in the Supai through Toroweap Formations (Krewedl and Carisey, 1986). Eight deposits of uranium, presently economical to develop, have been identified by Energy Fuels Nuclear in the Kanab Creek area. These deposits are almost exclusively uranium bearing, though other metals are known to exist. Active exploration programs have been undertaken by several companies in search of additional deposits on the Arizona Strip.

Sandstone type uranium deposits are known to occur in the Petrified Forest and Shinarump members of the Chinle Formation. Uranium was produced from deposits

in these members in the 1950s (Keith and others, 1983; Scarborough, 1981; Baillieu and Zollinger, 1980). Approximately 1,524 tons of uranium ore averaging 0.201 percent U308 was produced from the Vermillion Cliffs deposits between 1954 and 1957 (Scarborough, 1981). These deposits are located within the present day Vermillion Cliffs Wilderness Area. Uranium was also produced from the Rainbow Hills mining district though no production figures are available. Based on the geologic environments, inferred geologic processes and numerous mines in these areas; they have been rated as having a high potential for the occurrence of uranium resources (Map III-4). This rating is supported by abundant direct and indirect evidence.

Cedar Pockets GYPSUM

On the Arizona Strip District, gypsum occurs in the Pakoon Dolomite, the Seligman and Woods Ranch members of the Toroweap Formation (Nielson, 1986; Hintze, 1986; Moore, 1972), the Harrisburg Member of the Kaibab Formation (Nielson, 1986; Cheevers and Rawson, 1979); and the Lower Red Member of the Moenkopi Formation (Stewart et al, 1972; Wilson, 1962). Gypsum in the Kaibab and Moenkopi formations appears to be of good quality. Based on the known occurrence of gypsum in these formations, areas overlain by the Toroweap, Kaibab, and Moenkopi Formations have a high favorability for containing gypsum. The thick gypsum deposit in the Pakoon Dolomite appears to be an isolated occurrence in the Cedar Pockets area and, as such, the Pakoon Dolomite has been rated highly favorable in that area (Map III-2). The certainty that gypsum occurs in these areas is also high, supported by abundant direct and indirect evidence.

Shinarump
Large gypsum deposits are found in the northwestern portion of the district around Black Rock Gulch, the north end of the Sunshine Trail and in Cedar Pockets. Operators are actively mining in two locations and another mine is currently inactive. Assays show the Cedar Pockets and Black Rock Gulch deposits to be of high quality and good potential exists for gypsum mining from these areas.

SAND AND GRAVEL

In the western portion of the district, gravel is abundant along the lower slopes of the Virgin and Beaver Dam Mountains. Here alluvial fans have formed and the gravel is expected to be unsorted but of good quality. Well sorted, good quality gravel is also expected to occur in

A-5