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HIGH-DENSITY CONCRETE FOR GAMMA AND NEUTRON ATTENUATION

T. E. Northup

OAK RIDGE NATIONAL LABORATORY
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GENERAL ENGINEERING AND CONSTRUCTION DIVISION

HIGH-DENSITY CONCRETE FOR GAMMA AND NEUTRON ATTENUATION

T. E. Northup

MARCH 1965

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
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PREFACE

This report was prepared at the request of the Chemical Technology Division as a record of the basic work done for the concrete shielding used in the Oak Ridge National Laboratory Transuranium Facility.

Various aggregates and concretes for shielding were investigated through the joint efforts of the Atomic Energy Commission and the Oak Ridge National Laboratory. The personnel and facilities of ORNL and the U.S. Corps of Engineers were used to conduct these investigations. The author is grateful to the personnel of these institutions for their assistance. Gratitude is also expressed to the members of the Oak Ridge National Laboratory Chemical Technology Division for their support and confidence in developing an untried concrete for use as a high-density bi-shielding concrete for the Transuranium Facility.

CONTENTS

	Page
LIST OF FIGURES	vii
ABSTRACT	1
1. INTRODUCTION	1
2. DEVELOPMENT OF DESIGN FOR TRU FACILITY CONCRETE SHIELDING	5
Material Investigation	6
Concrete Testing	13
Concrete Placement	18
3. CONCLUSIONS	21
REFERENCES	24
Appendix A. THE RUNNING WOLF MINE	27
Appendix B. SHIELDING AGGREGATE DATA	34
Appendix C. PYRITE DAMAGE TO CONCRETE	62
Appendix D. LABORATORY TESTING PROCEDURES	66

LIST OF FIGURES

Figure No.	Title	Page
1	Plan of Transuranium Facility Cell Area	3
2	General Configuration of Transuranium Facility Concrete Structure	4
3	Effect of Water-to-Cement Ratio, Period of Hydration, and Drying Temperature on Moisture Retained by Neat Portland Cement Mortars (from ref. 6)	6
4	Area Location Map for Shielding Aggregate	10
5	Split Test Cylinders FL1 through FL6	15
6	Split Concrete Block Showing FL Material Separation Under Sleeves	16
7	Split Concrete Block Showing FL Material Separation Under Formed Opening	16
8	FL7, FL8, FL9, ML10, ML11, and ML12 Split Test Cylinders	17
9	High-Density Aggregate Storage Area and Batch Plant	18
10	Wall Penetration Area Before High-Density Bi-Shielding Concrete Pour	20
11	Wall Penetration Area During High-Density Bi-Shielding Concrete Pour	20
A.1	Plan of Running Wolf Mine Site	28
A.2	Section Through Hydrous Iron Ore Vein	29
A.3	Cross Section of Running Wolf Crushing Plant	30
A.4	Production Scheme for Coarse Aggregate	31
A.5	Production Scheme for Fine Aggregate	32

HIGH-DENSITY CONCRETE FOR GAMMA AND NEUTRON ATTENUATION

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ABSTRACT

The special aggregates needed to produce concrete shields capable of attenuating high and almost equal intensities of gamma and neutron radiation at a temperature of 100°C are described in this report. Material acquisition and concrete test information on ferrophosphorus-limonite concrete and hydrous-iron-ore-limonite concrete is presented. Special appendices cover high-density aggregate mining, iron-pyrite impurities in hydrous iron ore, and shielding aggregate data tables. The basic approach to high-density concrete mix design is reviewed to show that theoretical calculations for concrete mixes do give results that are reliable. However, laboratory tests with the produced shielding aggregates are still required to confirm mix quantities, adjust workability, establish the need for additives, etc. before any concrete is actually placed.

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1. INTRODUCTION

"High-density concrete" as a general term has been used for years to describe concretes weighing more than about 150 lb/ft^3, and such concretes have been used by designers for jobs varying from foundations for towers to ballast for ships. "High-density shielding concrete" goes one step further as another general term used to describe radiation-stopping concretes installed to protect personnel from gamma and neutron radiation from nuclear reactors, cyclotrons, accelerators, radioactive isotopes, etc. These shields are usually developed specifically for gamma radiation or for neutron radiation because, theoretically, the first type of radiation is stopped by a material of high density while the second is stopped by elements of low atomic number. In certain cases where one type of radiation dominates and the other type is also present, a shield designed

for the dominate radiation type can perform both shielding jobs.

The design of multiple radiation shielding becomes more difficult when both gamma and neutron radiation are of equal importance. The development of high-flux reactors and the production of transuranium elements has increased the need for matrix shields made of high- and low-density elements that are capable of a high degree of attenuation of both gamma and neutron radiation. Various approaches to bi-shielding have been made, as reported by Munn and Pontecorno[1] in 1947, Callan[2] in 1953, Davis and Browne[3] in 1956, and Davis and Borge[4] in 1959. These approaches include the use of combinations of steel and water, concrete with boron or colemanite additives, and concretes made with iron ores or other aggregate materials containing chemically combined water. "High-density bi-shielding concrete" would be a convenient and general way of describing the concrete used for these matrix shields.

The material presented in this report was accumulated while a high-density bi-shielding concrete was being developed for use in the construction of a shielding complex to house transuranium element processing facilities (TRU) at Oak Ridge National Laboratory. A plan view of the Transuranium Facility cell area is shown in Fig. 1, and the general configuration of the concrete structure is illustrated in Fig. 2.

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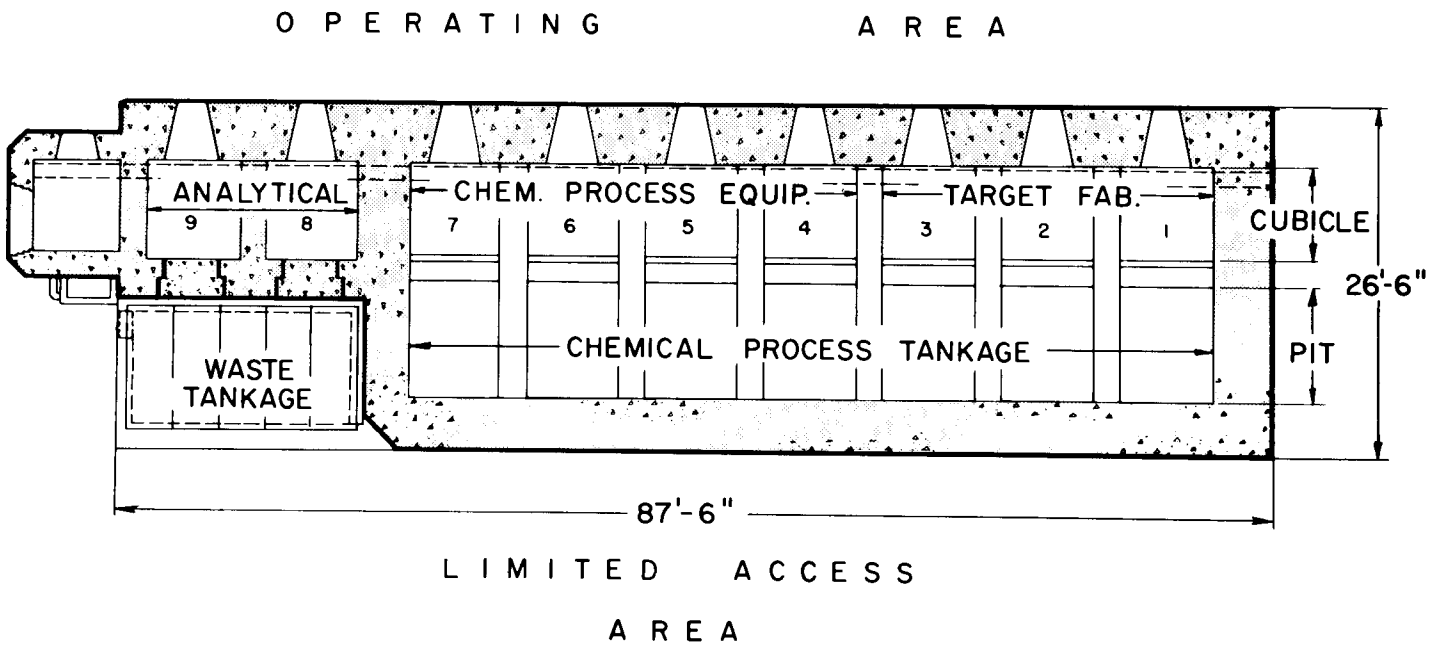

Fig. 1. Plan of Transuranium Facility Cell Area.

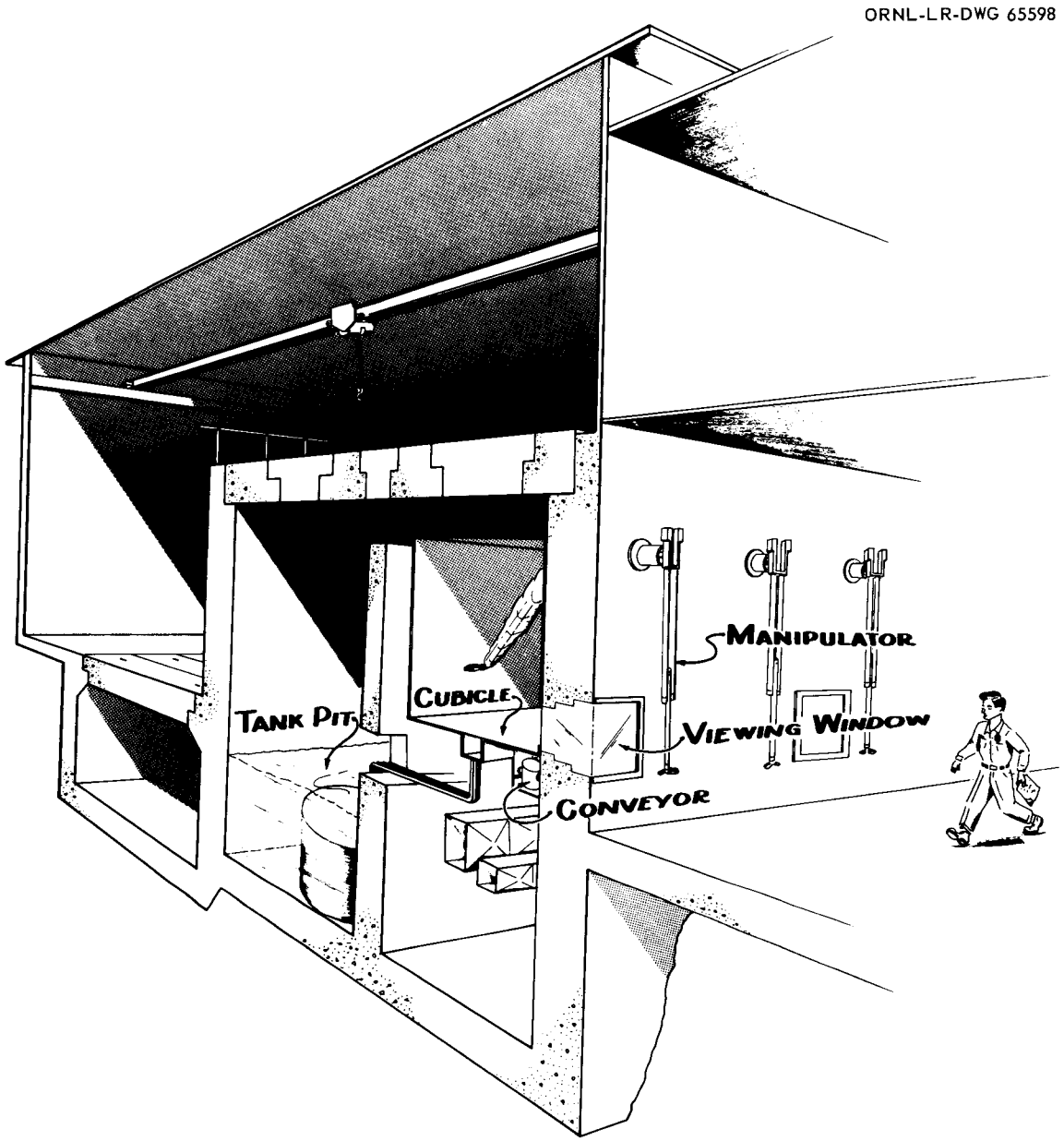

Fig. 2.
General Configuration of Transuranium Facility Concrete Structure.

2. DEVELOPMENT OF DESIGN FOR TRU FACILITY CONCRETE SHIELDING

The design for the shielding was based on sources of a solution containing 2×10^5 curies of ^{144}Ce-^{144}Pr from burnup of 10 kg of ^{239}Pu and a fifth cycle target, which is composed of residual actinides from successive High Flux Isotopes Reactor irradiations beginning with ^{242}Pu, that contains 305 mg of californium and spontaneously emits 3×10^{12} fission neutrons per second.[5] Shielding thickness was developed by using Renupak, an IBM-7090 code for the solution of the neutron transport equation by a moments method, and SDC, a gamma penetration code. It was established that a wall 54 in. thick with a total unit weight of 210 lb/ft^3 that retained 9 lb of H_2O per cubic foot at a temperature of 100°C would satisfy the dose rate criteria for personnel of less than 0.75 millirem per hour at the outer surface of the shield. An elemental iron (Fe) content of approximately 104 lb/ft^3 was desirable to increase the radiation scattering capability of the shield, thereby increasing the probability of neutron radiation attenuation.

The requirement for a concrete unit weight of 210 lb/ft^3 at a temperature of 100°C could be fulfilled by using one of the proven high-density aggregates, such as barytes, magnetite, or ilmenite. The 9 lb of H_2O per cubic foot would be difficult to retain even at normal room temperatures since a standard concrete mix will have only 9 to 13 lb of H_2O per cubic foot at pour. Gradual concrete drying will reduce the water content to that quantity chemically united with the cement. According to research data prepared by Wilson and Martin,[6] the amount of water retained is a function of the curing period and drying temperature. Their graph illustrating the effect of water-to-cement ratio on the water retained for various curing periods and drying temperatures is shown in Fig. 3. A mixture with a water-to-cement ratio of 0.60 by weight or a mix using 11.3 lb of H_2O per cubic foot at pour would retain only 3.57 lb of H_2O per cubic foot if subjected to a temperature of 100°C after it cured for one year. One year curing duration was considered to be reasonable for the TRU Facility. Process operation would not start until installation of all equipment was completed, and that would be accomplished at least one

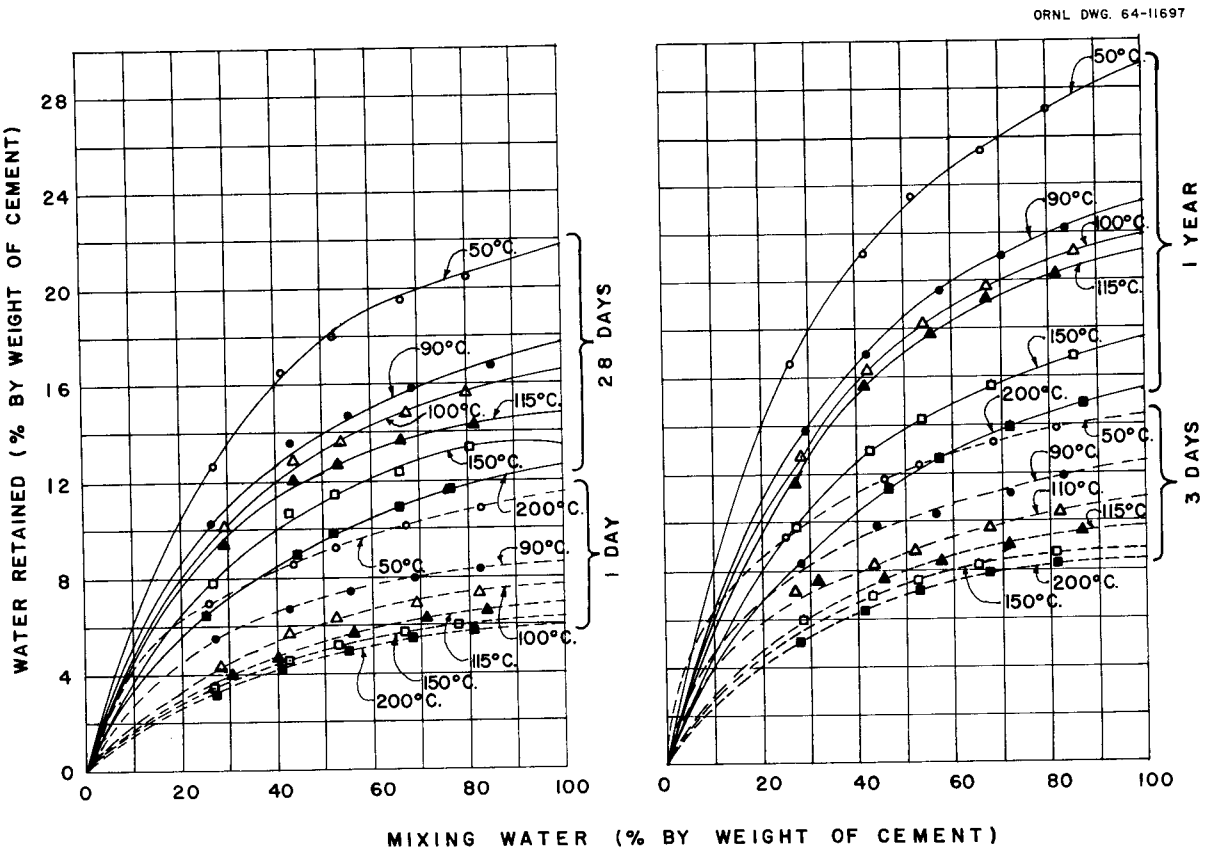

Fig. 3. Effect of Water-to-Cement Ratio, Period of Hydration, and Drying Temperature on Moisture Retained by Neat Portland Cement Mortars (from ref. 6).

year after the pouring of the shielding structure. The deficiency of water at a temperature of 100°C became the main design problem. Use of a water-bearing-material additive or special aggregate appeared to be the appropriate solution.

Material Investigation

Reviews of material did not offer any simple solution. Theoretical concrete mixes were calculated by using absolute volume procedures and high-density shielding concrete mixes reported in technical reports. As

given in Table 1, aggregate materials with a minimum specific gravity of 4.4 and containing a minimum of 4.2% combined water would, theoretically, produce a suitable radiation shield for the TRU Facility. The only known source of such quality material was the nonoperating Running Wolf Mine located near Stanford, Montana. High-density concrete had been made with ore from this mine for placement at Hanford, Washington in 1961 after laboratory testing had been performed by the U.S. Corps of Engineers at Troutdale, Oregon in 1958.[7] These laboratory tests provided sufficient evidence that concrete of adequate unit weight, H_2O content, and compressive strength could be made.

The hydrous iron ore was judged to be the only economical single aggregate material suitable for the TRU Facility. Multiple material combinations suitable for shielding were found but they were estimated to be more expensive. The materials investigated are given in Table 2, and their properties are compared with the criteria specified for the project. These various materials are located in different parts of the country; therefore, the estimated transportation expenses usually represent a major cost item. Shipping costs vary for several reasons, such as whether (1) the material is classified by the rail agency as chemical, ore, aggregate, or waste; (2) the material is to be moved over land grant sections of railroad (class 22 consideration involves haulage over Governmental Land Grant Railways); (3) the material can be hauled in bulk form or is subject to degradation and therefore will require bagging or boxing; (4) several rail companies will be involved requiring excessive switching. Truck and barge transportation modes have their own special problems, such as loading and unloading facilities, interstate shipment regulations, etc.

Appendix A has been included to explain the mining technique and equipment used at the Running Wolf Mine. Shielding Aggregate Data Tables B.1 through B.27 of Appendix B have been included as references for material properties, estimated costs, and personnel contacts. Figure 4 provides a quick reference to the approximate location of materials except limestone aggregates and steel punchings, which are to be found in almost any local area.

Table 1. Theoretical Composition of Hydrous Iron Ore Concrete with 3.52 Sp Gr at 219.5 lb/ft^3

Mix	Lb/yd^3	Sp Gr	Abs Vol	% by Weight	Cost at ORNL $/ton	Cost at ORNL $/yd
Cement[a]	636	3.15	3.24	10.62		7.30
Fine aggregate iron ore	2310	4.4	8.43	38.70	34.50[b]	39.90
Coarse aggregate iron ore	2675	4.3	9.98	44.80	34.50[b]	46.20
Water	352		5.63	5.88[c]		
Total	5973		27.28	100.00		93.40

Element	Portland II LA Cement A[e]	Portland II LA Cement B[f]	Coarse Aggregate[d] A[e]	Coarse Aggregate[d] B[f]	Fine Aggregate[d] A[e]	Fine Aggregate[d] B[f]	Water A[e]	Water B[f]	Concrete Composition C[g]	Concrete Composition D[h]
H_2O[j]			4.21	1.88	4.21	1.61	100.00	0.90	4.39	0.154
O	35.52	3.77	30.55	13.67	30.50	11.82			29.26	1.030
Fe[k]	3.01	0.32	64.00	28.68	64.00	24.77			53.77	1.893
Si	10.40	1.10	1.08	0.48	1.08	0.42			2.00	0.070
Al	2.49	0.26							0.26	0.009
Ca	45.00	4.79							4.79	0.169
Mg	1.50	0.16							0.16	0.006
S	0.68	0.07							0.07	0.002
Ti			0.03	0.01	0.03	0.01			0.02	0.001
Loss and insolubles	1.40	0.15	0.18	0.08	0.18	0.07			0.30	0.010
									95.02	3.345[m]
H_2O loss								4.98	4.98	0.175
Total								5.88	100.00	3.52

[a] Water-to-cement weight ratio = 0.486 (5.47 gal/sack).
[b] Estimated cost includes $16.20/ton shipping charge. (See Table 5 for actual cost to government.)
[c] Two pounds of H_2O per cubic foot or 0.90% by weight assumed retained by cement at 100°C.
[d] Chemical composition based on ORNL test August 23, 1962.
[e] Percent by weight in compound.
[f] Percent by weight of element in mix.
[g] Percent by weight of total elements in mix.
[h] Grams per cubic centimeter at 100°C.
[j] H_2O at 100°C, 0.154 g/cm^3 = 9.6 lb of H_2O per cubic foot.
[k] Fe = 118.1 lb/ft^3.
[m] Unit weight at a temperature of 100°C.

Table 2. Tabular Summary of Concrete Investigated

	Room Temperature				100°C						Total Materials Cost[c]	
	Unit Weight		H_2O		Fe[a]		Unit Weight		H_2O[b]			
	(lb/ft³)	(g/cm³)	(lb/ft³)	(g/cm³)	(lb/ft³)	(g/cm³)	(lb/ft³)	(g/cm³)	(lb/ft³)	(g/cm³)	($/yd³)	Basis
Specified criteria	220	3.53	12.2	0.196	104.0	1.67	210	3.37	9.1	0.146		
1. Hydrous iron ore	219.5	3.52	13.4	0.215	118.0	1.89	207[d]	3.32[d]	9.6	0.154	93.40	Tests[e]
2. Ferrophos-serpentine	227.5	3.65	10.2[d]	0.163[d]	102.5	1.63	219	3.52	7.0[d]	0.112[d]	119.58[d]	Theory
3. Ferrophos-serpentine	203.0[d]	3.26[d]	12.2	0.195	69.8[d]	1.12[d]	194.5[d]	3.12[d]	9.0	0.145	85.67	Theory
4. Cranberry magnetite			H_2O and Fe too low to consider									Tests
5. Wyoming magnetite			too low to consider									Tests
6. Wyoming magnetite-serpentine			H_2O and Fe too low to consider									Theory
7. Wyoming magnetite-MO cement	223	3.58	15.3	0.245	119.0	1.907	217.5	3.49	9.3	0.150	101.48[d,f]	Tests plus Theory
8. Ferrophos-Missouri-limonite	226	3.62	20.2	0.324	116.0	1.86	215.0	3.45	9.2	0.147	117.57[d]	Theory
9. Wyoming-magnetite Missouri-limonite	195.5[d]	3.14[d]	20.1	0.322	86.0[d]	1.30[d]	187.5[d]	3.01[d]	7.8[d]	0.125[d]	54.20	Theory plus tests[e]

[a] Fe quantities also include any Ti present.

[b] H_2O at 100°C held by cement assumed to be 2 lb/ft³ for all concretes investigated except No. 7. Water retained in No. 7 based on A. E. Pavlish and J. C. Wynd, "Concrete for Pile Shielding," USAEC Report BMI-3007, Batelle Memorial Institute, August 1948.

[c] Total material cost includes aggregates and cement F.O.B. Oak Ridge, Tennessee. (Estimated costs only)

[d] Major deficiencies.

[e] American Concrete Institute, Concrete for Radiation Shielding, 2nd edition, compilation No. 1, 1962.

[f] Total materials cost does not reflect additional cooling costs required during curing process.

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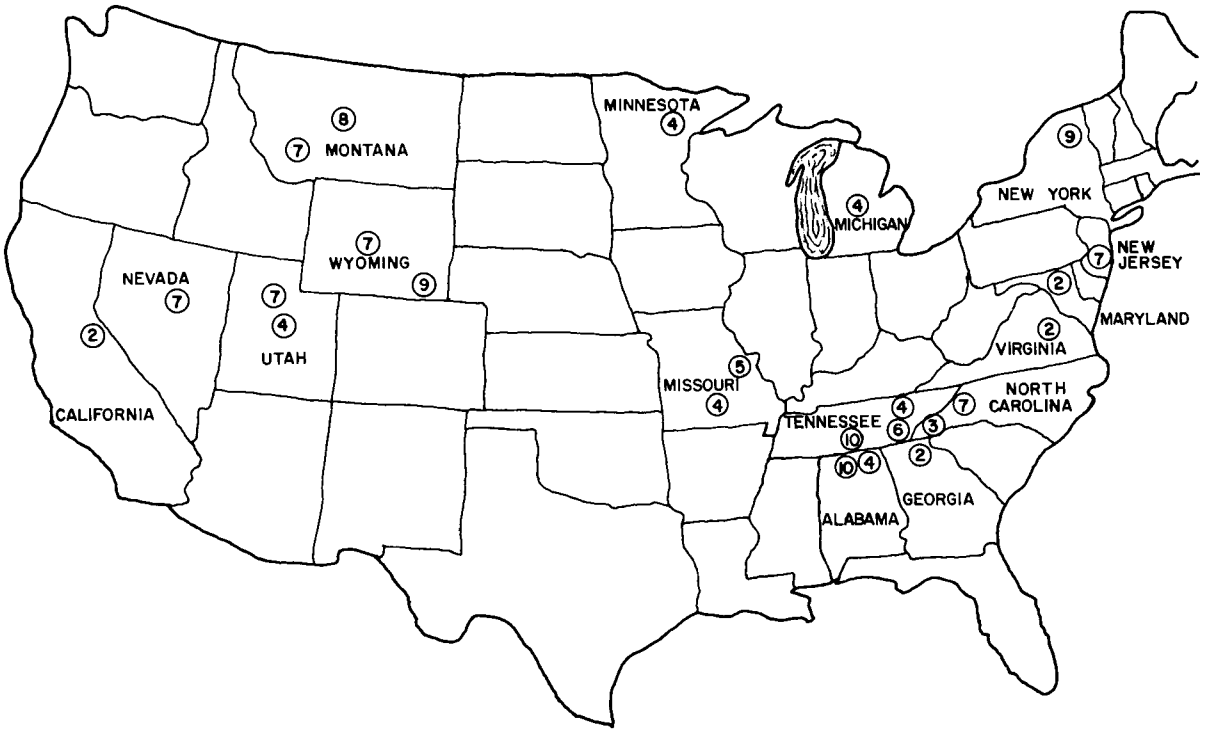

Aggregate		Concrete Unit Weight
Type	Sp Gr	(lb/ft^3)
1. Limestone	2.7	145-150
2. Serpentine, $H_4Mg_3Si_2O_9$	2.7-2.9	145-160
3. Olivine, $(M_gFe)_2SiO_4$	3.1	160
4. Limonite, $2Fe_2O_3 \cdot 3H_2O$	3.3-3.7	175-190
5. Waste Lead Slag	3.1	185
6. Barytes, $BaSO_4$	4.3	210
7. Magnetite, Fe_3O_4	4.3-4.7	210-225
8. Hydrous Iron Ore	4.5	220
9. Ilmenite	4.5-5.5	220-240
10. Ferrophosphorus	5.7-6.5	290-315
11. Steel Punchings	7.4-7.8	340-375

Fig. 4. Area Location Map for Shielding Aggregate

The hydrous iron ore was ordered to the specifications listed below.

1. Iron Content. The minimum iron content for sizes coarse and fine shall be as follows:

Average of entire shipment	Coarse, 60% Fine, 63%
Any individual carload	Coarse, 55% Fine, 58%

2. Water of Crystallization. The minimum water of crystallization shall be as follows:

Average of entire shipment	3.2%
Any individual carload	2.3%

3. Absorption. The absorption shall not exceed 2.0%

4. Specific Gravity. The minimum specific gravity of the aggregate shall be as follows:

Average of entire shipment	Coarse, 4.3
	Fine, 4.4
Any individual carload	Coarse, 4.2
	Fine, 4.3

5. Sulfur Content. The maximum sulfur content for any carload shall be 0.1%. (Increased to 1.0% in an effort to obtain adequate quantities of aggregate during one year).

This type of specification was used to minimize the material rejection rate for those carloads that did not quite meet the total shipment average. Lower water of crystallization percentages (3.2% versus 4.21%) were specified by taking advantage of the actual amount of water associated with the cement rather than the arbitrary 2 lb of H_2O per cubic foot used in Table 1.

As with any concrete aggregate, the problem of impurities must be considered. Alkali-reactivity is less likely to occur with iron ore materials, but a check for this should not be neglected. Other impurities, such as lignite, zinc, copper and lead oxide, clay, coal, excessive fines, sulfur, chlorine, etc. must be investigated. Zinc, copper, and lead oxides will cause a severe increase in concrete setting time even when found in small percentages.[8] Cracking, spalling, deterioration, or rapid decay are known to occur when sulfur is present in an aggregate used for concrete production. Initial samples of the hydrous iron ore checked by ORNL and the tests reports on the material used at the NPR Hanford shield did not show evidence of sulfur, so it was no little surprise when prebid samples from the mine site exhibited high quantities of iron pyrite (up to 5.3% FeS_2). Immediate checks for sulfur were made on material left over from the Hanford job, and they showed iron pyrite quantities of 1.91%.[9] Additional samples from the ore vein to be mined were obtained and analyzed. The results showed a pyrite content of 0.02 to 0.04% (sulfur percentage is about half of the pyrite value). After an extensive review of the deleterious effects of sulfur in concrete was conducted by the Atomic Energy Commission, as outlined in Appendix C, a maximum limit

of 0.1% sulfur content was originally set for the ore material to be purchased. This would mean that mine areas containing material with a high percentage of sulfur would have to be avoided. Because of the restrictive nature of the mine, poor material would be removed and wasted while the good material would be removed and processed through the crushing plant. Due to contractor's production problems the sulfur content limit was later raised to 1.0% by the AEC.

In spite of this change, only about 610 tons of coarse hydrous iron ore were produced and shipped during the summer and fall months of 1963. None of the 1,450 tons of fine material needed for the project was produced under the contract, which was ultimately terminated by the Government.

Since the required quantity of hydrous iron ore was not obtained, the suitable but expensive matrix materials of either Wyoming Magnetite and MO (magnesium-oxychloride) cement or ferrophosphorus-limonite, given as nos. 7 and 8 in Table 2, were reconsidered for the project. Neither combination had been tested thoroughly to show that these materials could be used together to make good shielding concrete. Some work on concretes made with MO-cement was reported by Pavlish and Wynd[10] in 1948, Harlow and Matthews[11] in 1953, and Creed[12] in 1963. The MO-cement approach was not considered to be feasible because of the added costs, cooling required during the concrete curing period, and field experience with steel corrosion where MO-cements have been used. Steel containers filled with steel shot and magnesium-oxychloride cement were used for shield tests in the ORNL Graphite Pile in the late 1940's. Following a year of testing, the containers were removed and stored outside in the weather. Corrosion started immediately and progressed until the steel containers split open. Although ferrophosphosphorus-MO concretes have been tested with successful results, a MO reaction with magnetite ores and the numerous embedded steel sleeves and reinforcements required in shielding walls can cause damaging expansion, especially in areas subjected to water.

Mt. Pleasant ferrophosphorus, Table B.15, and Blackburn limonite, Table B.7 of Appendix B, were selected as possible material substitutes for use in place of the hydrous iron ore material. The ferrophosphorus was selected to provide the required high density, and the limonite was

chosen to provide the high chemically bound water content. Because of the extreme difference in the specific gravity of the two materials (6.0 and 3.5), the suitability of a resulting concrete would depend upon the degree of material separation experienced during batching, mixing, transportation, and placement operations. Material proportioning and concrete batch tests were needed to properly evaluate the workability and homogeneity of a concrete made with ferrophosphorus-limonite aggregates. Laboratory testing procedures used for determining physical properties and the chemical and fixed water content of the various shielding aggregates investigated have been included for reference in Appendix D.

Concrete Testing

Laboratory tests on the two concretes developed for the TRU project were performed by the U. S. Army Engineering Division Laboratory, South Atlantic Corps of Engineers, Marietta, Georgia. A ferrophosphorus-limonite (FL) matrix and a hydrous iron ore-limonite (ML) matrix (to make use of acquired Montana material) concrete were prepared and tested, and the results are shown in Table 3. Mixes FL1 through FL6 used two aggregate gradations numerically proportioned to provide approximately 9 lb of H_2O per cubic foot at a temperature of 100°C. As shown in Fig. 5, test cylinders FL1 through FL6 from the mixes listed in Table 3 were cut in half to see if excessive material separation had occurred. Although no separation was evident (the high density work by the Corps of Engineers was much better than the cracked block wall indicates), the results were inconclusive because such a small amount of material was involved. More investigation into the problem of material separation was desired before further mix optimizing was attempted.

In order to make a better evaluation of material separation of the FL mixes, several cubic feet of 5-in. slump (mix No. FL4) concrete was mixed in a rotating mixer and then poured from a 3-ft height into a form containing metal sleeves, electrical boxes, reinforcing, and a rectangular formed opening simulating the frame of a viewing window. An internal vibrator with a constant reciprocal motion was used in the concrete to

Table 3. Composition of Bi-Shielding Concretes

	Mix Number[a]															
	FL-1	FL-2	FL-3	FL-4	FL-5	FL-6	FL-7F	FL-8F	ML-9F	ML-10F	ML-11	ML-12	M-M01	M-M02	M-M03	M-M04
Aggregate																
Coarse	Ferrophosphorus	Ferrophosphorus	Ferrophosphorus	Ferrophosphorus	Ferrophosphorus	Ferrophosphorus	Ferrophosphorus	Ferrophosphorus	Hydrous iron ore	Hydrous iron ore	Hydrous iron ore	Hydrous iron ore	Magnetite	Magnetite	Magnetite	Magnetite
Medium							Limonite	Limonite								
Fine	Limonite	Limonite	Limonite	Limonite	Limonite	Limonite	Limonite	Limonite	Limonite	Limonite	Limonite	Limonite	Magnetite	Magnetite	Magnetite	Magnetite
Cement factor, sacks per yd^3	7.25	7.25	7.00	7.00	7.00	7.00	7.00	7.00	7.50	7.50	7.50	7.50				
Water-to-cement ratio																
By wt	0.45	0.46	0.42	0.44	0.39	0.40	0.43	0.45	0.44	0.49	0.40	0.45				
By gal per sack	5.10	5.25	4.75	4.95	4.40	4.55	4.88	5.09	5.0	5.55	4.53	5.10				
Slump, in.	3	5	3	5	3	5	3	5	3	3	3	3				
Air in concrete[a], %			5.0	5.0	5.0	5.0	5.0	5.0								
Unit weight of concrete[b], lb/ft^3																
Mix	242.5	241.5	233.3	231.7	233.5	232.1	222.9	221.3	209.8	202.4	210.5	203.7	196	199	180	152
7 days	242.5	241.2	236.6	234.3	233.5	231.4	222.5	220.7	209.8	202.4	209.5	203.7	184	182	174	
14 days	239.1	238.1	232.9	230.6	230.7	228.5	220.6	218.5	207.7	199.6	207.5	201.8	184	182	174	132
28 days	238.2	237.0	232.0	229.7	229.6	228.2	219.3	217.3	206.4	198.4	206.9	201.3	184	182	174	
28 days at 105°C	233.3	232.3	227.4	224.8	223.8	223.2	214.0	211.7	201.3	192.6	201.4	195.4				
Combined water content at 100°C at 28 days[c], lb/ft^3							11.8	11.2	13.5	14.4	14.2	14.8	7.4	7.95	3.7	12.4
Compressive strength, psi																
7 days (6-in.-diam cyl)	2830	2530	2730	2370	2910	2770	2890	2620	4600	3850	5240	4230				
14 days (6-in.-diam cyl)	3825	3365	3580	3170	3820	3590	3430	3130	6220	5030	6200	5270				
28 days (6-in.-diam cyl)	4830	4340	4300	3970	4440	4060	3770	3610	6930	6320	7500	6580				
7 weeks (2-in. cube)													10,000	8000	5600	
Mix, lb/yd^3																
Type II cement[d]	682	682	658	658	658	658	658	658	705	705	705	705				
Coarse aggregate	3578	3550	3503	3466	3388	3341	2875	2844	2826	2292	2645	2162	3080	3010	3795	1660
Medium aggregate							719	711								
Fine aggregate	1979	1964	1862	1842	2002	2002	1482	1466	1822	2120	2051	2313	1078	1290	424	754
Water	308	317	277	289	257	265	285	297	312	347	283	319	(543)	(665)	(339)	(870)
Plastiment[e]	36 1/4 oz	36 1/4 oz	35 oz	35 oz	35 oz	35 oz	35 oz	35 oz	37 1/2 oz	37 1/2 oz	37 1/2 oz	37 1/2 oz				
Air entraining			4 3/4 oz[h]	4 3/4 oz[h]	5 1/2 oz[f]	5 3/4 oz[f]	10 1/2 oz[g]	10 1/2 oz[g]								
MgO													462	226	212	603
22° Be $MgCl_2$ solution													680	833	424	1100
Total weight	6547	6513	6300	6255	6305	6266	6019	5976	5665	5464	5684	5499	5300	5359	4865	4117
Remarks	Good workability, excessive bleeding.	Good workability, excessive bleeding.	Good workability, slight bleeding.	Good workability, slight bleeding.	Good workability, slight bleeding. Mix is oversanded.	Good workability, slight bleeding. Mix is oversanded.	Good workability, slight bleeding.	Good workability, slight bleeding.	Good workability, moderate bleeding.	Good workability, moderate bleeding.	Fair workability, moderate bleeding. Mix was harsher and not as plastic as Mix No. ML-9F.	Fair workability, moderate bleeding. Mix was harsher and not as plastic as Mix No. ML-10F.				
Aggregate gradation	1	1	1	1	2	2	3	3	4	5	6	7	8	8	8	8
Sieve size	Coarse / Fine				Coarse / Fine		Coarse / Medium / Fine		Coarse / Fine	Medium / Fine	Coarse / Fine	Medium / Fine	Fine aggregate minus 65 mesh, coarse aggregate 1/4 in. to 20 mesh	Fine aggregate minus 65 mesh, coarse aggregate 1/4 in. to 20 mesh	Fine aggregate minus 65 mesh, coarse aggregate 1/4 in. to 20 mesh	Fine aggregate minus 65 mesh, coarse aggregate 1/4 in. to 20 mesh
1 1/2 in.	100				100		100		100		100					
1 in.	70				70		63		69		84					
3/4 in.	48				48		22		37	100	62	100				
1/2 in.	29				29		7 / 100		22	59	44	71				
3/8 in.	18 / 100				18 / 100		5 / 65		14	37	30 / 100	48 / 100				
No. 4	3 / 98				3 / 88		10 / 100		3 / 100	7 / 100	4 / 88	88				
No. 8	90				67		3 / 78		78	78	67	67				
No. 16	68				47		43		43	43	47	47				
No. 30	43				26		26		26	26	26	26				
No. 50	20				10		11		11	11	10	10				
No. 100	6				2		5		5	5	2	2				
Material source table number	B.15 / B.7				B.15 / B.7		B.15 / B.7		B.16 / B.7	B.16 / B.7	B.16 / B.7	B.16 / B.7	B.4, B.17, B.18	B.4, B.17, B.18	B.4, B.17, B.18	B.4, B.17, B.18

[a]Conventional placement method.
[b]Corrected (minus 3.0%) for air in aggregate.
[c]Curing schedule provided seven days of wet cure with remaining days of air curing. For MO concretes, 2-inch cube samples were placed in temperature oven at 100°C after seven days curing at ambient. Cubes were removed from oven and weighed at the end of each period reported.
[d]The nonevaporable water is the amount retained after sample is subjected to oven drying at 100°C to remove free water. Mixes 1 through 12 were then heated to 850°C in inert atmosphere. Mixes MO1 through MO4 were only weighed at 100°C to determine weight loss and water retained.
[e]Alpha, Phoenixville, Alabama; Lone Star, Birmingham, Alabama, for Mixes 7F through 10F, 11 and 12.
[f]Sika Chemical Company.
[g]NVX (calculated on the basis of approximately 0.8 oz per bag of cement).
[h]NVX (calculated on the basis of 1.5 oz per bag of cement).
[i]NVX (calculated on the basis of approximately 2/3 oz per bag of cement).

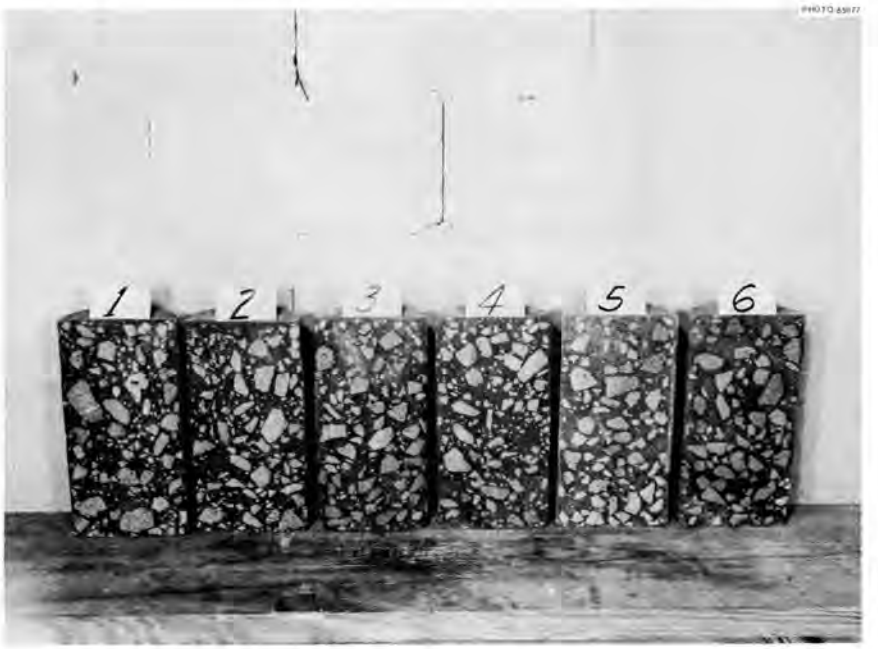

Fig. 5. Split Test Cylinders FL1 Through FL6.

keep a pool of grout from being drawn to the top. The vibrator was applied at points uniformly spaced and not farther apart than the radius over which it was visibly effective. As shown in Figs. 6 and 7, separation did occur in spite of the extreme care taken in the use of the vibrator and with the limited height of material free-fall. The lack of heavy ferrophosphorus near the top, under the sleeves, and under the formed opening would be detrimental to any shielding structure; therefore, mixes of 3-in. slump or less were specified for all high-density pours for the TRU Facility unless special approval was obtained for the use of high slumps.

Since mixes FL1 through FL6 provided only the minimum amount of H_2O desired, two more mixes, FL7 and FL8, were prepared using three aggregate gradations to optimize the water content and yet maintain a unit weight above 210 lb/ft^3 at a temperature of $100^{\circ}C$. Mixes ML9 through ML12 used two aggregate gradations proportioned to provide the maximum density possible at a temperature of $100^{\circ}C$. ML10 and ML12 contained 3/4-in. maximum size coarse aggregate because the resulting concrete was to be

Fig. 6. Split Concrete Block Showing FL Material Separation Under Sleeves.

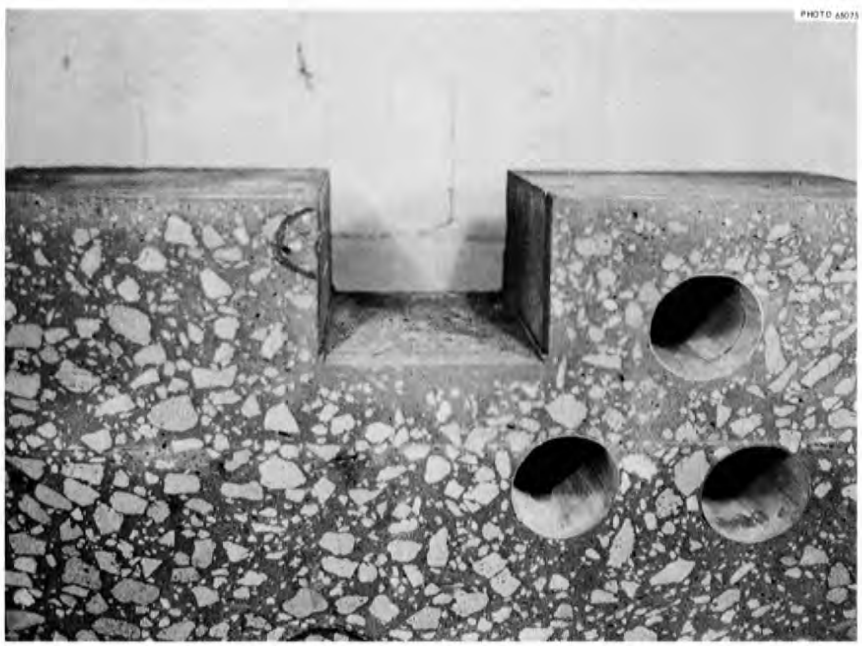

Fig. 7. Split Concrete Block Showing FL Material Separation Under Formed Opening.

used only in low-radiation areas and where complete replacement could be performed. These split test cylinders are shown in Fig. 8.

Fig. 8. FL7, FL8, FL9, ML10, ML11, and ML12 Split Test Cylinders.

Four concretes developed by Creed[12] using magnesium-oxychloride cement are reported as M-M01 through M-M04 in Table 3. The values for the water retained and the unit weight at a temperature of 100°C were much less than those estimated for item no. 7 in Table 2.

A final review of all the test results, including those made with the procured graded aggregates, led to the use of four basic mixes for TRU Facility shielding. These were FL7F, FL8F, ML9F, and ML10F, as given in Table 3.

A plastiment additive can be used to advantage in all high-density shielding concrete work. Air entraining additives must be used with care since their use usually reduces the final concrete unit weight from 5 to 15%. Where workability is a problem and excessive water bleeding occurs, air entraining agents are very beneficial. Also, if an air-entrained

concrete is placed at a 2-in. slump, the reduction in shrinkage and the ease in placement may be worth the loss in unit weight. Closer attention to gradation of materials can actually increase the final weight of the concrete more than the loss contributed by an air entrainment additive. Grantham[13] gives additional data on the optimum gradation of aggregate material to obtain maximum density. In general, the greater the fineness modulus (FM) of the fine material, between 3.0 and 4.0, the greater the unit weight of the concrete.

Concrete Placement

The ability to continuously produce high quality shielding concrete will be a direct function of the quality of the batch plant operation. The storage of fine aggregates on concrete pads under cover, as shown in Fig. 9, is a must where mix variations are to be held to a minimum. The high-density aggregates also deserve protection because of their current high price. Once set, mix quantities for the concrete for the TRU Facility remained very constant throughout the pours. This would not have been the case if unprotected materials with widely varying amounts of water content had been used.

Fig. 9. High-Density
Aggregate Storage Area and Batch Plant.

Mixing and agitation limits were established for the concrete containing ferrophosphorus aggregates as a degradation control because the aggregate has a 98% L.A. abrasion loss factor. As the materials were loaded into the transit mixer, the mixer drum was rotated 60 to 70 revolutions at a rate of between 15 and 20 rpm. The amount of water added at the batch plant was limited to that quantity required to obtain a slump of approximately 3 in. Gentle mixer agitation not exceeding 10 rpm was permitted as the 3- to 4-yd^3 loads of material were moved from the batch plant to the job site. When needed to obtain the required slump, additional water was added just prior to concrete dumping. The mix consistently arrived at the job site about 1/2 in. lower in slump than that measured at the batch plant. Attention was directed to mixer speed, free-fall of the concrete, etc., which served to make the contractor aware of the critical nature of the material he was handling. Once this was understood, he exercised good material handling procedures.

At a slump of approximately 2 1/2 in., internal vibration was very successful in consolidating the material without causing separation. Large ferrophosphorus aggregate always remained in the top surface of each pour; a condition quite different from the first test results shown in Figs. 6 and 7. Care was taken to dump the concrete where it was needed rather than trying to use the vibrator to move it around.

It was the opinion of all those involved with the pouring operations that the FL bi-shielding concrete handled and placed well. Figure 10 shows a wall penetration area before a pour, and Fig. 11 shows that area during the pour. The only problem encountered was the surface texture of the concrete after the forms were stripped. The surface texture might be classified as fair because it consistently contained small air-bubble pits. Concrete shrinkage around steel embedments and surface drying cracks were not discernible although a high cement factor (7.0) was used for the mixes.

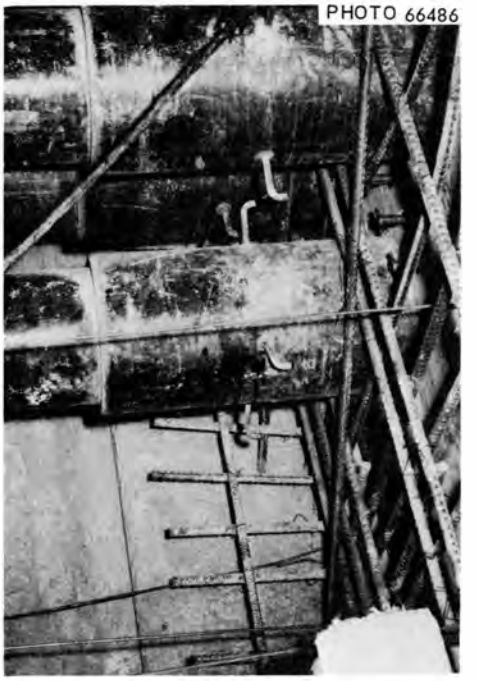

Fig. 10. Wall Penetration Area Before High-Density Bi-Shielding Concrete Pour.

Fig. 11. Wall Penetration Area During High-Density Bi-Shielding Concrete Pour.

3. CONCLUSIONS

"Can Theoretical concrete mix calculations give results that are reliable enough to permit other project planning and design to proceed with confidence without first resorting to laboratory confirmation or refinement?" Although this is a loaded question, a reasonable answer can be given in two parts since a project is usually concerned with the questions "Will the concrete give the desired shielding?" and "How much will it cost?".

For the first part, the practice of using the "absolute volume method" for proportioning the materials of concrete mixes has been proven to be acceptable when all of the various material properties are known. These properties include gradation, surface texture, absorption, saturated surface dry specific gravity, per cent voids, dry rodded unit weight, chemical analysis, fineness modulus, etc. All of these values can be obtained, or assumed where necessary, without extensive laboratory testing. Several theoretical concrete mixes are compared with actual concrete mixes developed by laboratory tests in Table 4.

The small percentage of variation shown does not mean laboratory tests can be omitted altogether because the refinement for concrete workability, additives, bleeding, permissible slump variations, and material segregation can be determined only by tests and observations. However, this phase of the work can be accomplished as the materials are being obtained, and testing should not delay any properly planned project.

The second part of the question concerns estimated costs, and this part cannot be answered with as much reliability. A comparison of estimated costs versus actual costs of three aggregate materials for high-density bi-shielding concretes is shown in Table 5. The large percentage of variation shown certainly tends to undermine confidence placed in estimated values for mixes and their cost. However, since material costs account for only about one-third of the final cost of in-place high-density concrete, the errors in material-cost estimating should be put in proper perspective. Material cost estimates are difficult to make for several reasons, such as

1. undeveloped source,
2. the uncertain cost of materials until bids are received,

Table 4. Theoretical Versus Actual Concrete Mixes

		Theoretical	Actual[a]	Variation %
Ferrophosphorus-limonite (FL-8F)	Mix, lb/yd^3,			
	Cement	636	658	
	Coarse ferro	2990	2844	
	Medium limonite	330	711[b]	
	Fine limonite	1790	1466[b]	
	Water	352	297	
	Unit wt @ mix, lb/ft^3	226	221.3	-2.1
	Unit wt @ 100°C @ 28 days, lb/ft^3	215	211.7	-1.5
	Water @ 100°C @ 28 days, lb/ft^3	11.35	11.2	-1.3
	Fe content, lb/ft^3	113	108	-4.4
Hydrous iron ore-limonite (ML-10F)	Mix, lb/yd^3			
	Cement	636	705	
	Coarse iron ore	2330	2292	
	Fine limonite	2275	2120	
	Water	352	347	
	Unit wt @ mix, lb/ft^3	207	202.4	-2.2
	Unit wt @ 100°C @ 28 days, lb/ft^3	194	192.6	-0.5
	Water @ 100°C @ 28 days, lb/ft^3	13.7	14.4	+0.7
	Fe content, lb/ft^3	100	97	-3.0

[a]Mixes FL-8F and ML-10F developed by the U. S. Army Engineering Division Laboratory, South Atlantic Corps of Engineers, Marietta, Georgia.
[b]Caused by gradation modification.

3. lack of established freight rates or special negotiated rates,
4. railhead-to-job-site handling,
5. supplementary crushing and grading that may be required.

One can answer "yes" to both parts of the posed question by diligently performing mix calculations, analyzing aggregate properties, analyzing various alternatives, and thoroughly investigating the mechanics of material acquisition, transportation, and grading. The "yes" must be followed by recommendation to start laboratory tests on the concrete to confirm mix quantities, adjust workability, establish the need for additives, etc. before any concrete is actually batched and placed.

Table 5. Estimated Versus Actual Concrete Material Costs

	Estimated Cost ($/ton)	Final Price ($/ton)	Variation (%)
Ferrophosphorus			
Graded coarse material	55.00	55.00	0
Transportation	6.80	9.66[a]	+42
F.O.B. job site	61.80	64.66	+ 4.8
Hydrous iron ore			
Graded coarse material	18.90	22.70[b]	+20
Graded fine material	18.90	30.30[b]	+60
Transportation	16.30	18.20	+12
F.O.B. job site			
Coarse	34.20	40.90	+20
Fine	34.20		
Limonite			
Mine material	7.50	9.50[c]	+27
Transportation	7.50	4.44	-41
Crushing and hauling to batch plant	9.00	6.82	-24
F.O.B. job site	24.00	20.76	-14

[a]Includes $2.66/ton for crating charge.

[b]Contract price of fine material although none was produced, and contract price of the coarse material although the total quantity required was not produced.

[c]Price includes quality control at mine of 31¢ per ton.

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7. American Concrete Institute, pp. 101-107 in Concrete for Radiation Shielding, 2nd edition, Compilation No. 1, 1962.

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12. R. A. Creed, FMC Corporation, personal communication to T. E. Northup, Oak Ridge National Laboratory, September 1963.

13. W. J. Grantham, "Barytes Concrete for Radioactive Shielding: Mix Criteria and Attenuation Characteristics," USAEC Report ORNL-3130, Oak Ridge National Laboratory, July 1961.

APPENDICES

Appendix A

THE RUNNING WOLF MINE

Hydrous Iron Ore Deposit

The hydrous iron deposit shown in Fig. A.1 is about 17 miles from Stanford, Montana, which provides the nearest railhead for shipment of ore material. The ore loading facilities at the railhead include two elevated truck ramps with tilting platform extensions for top loading of gondola cars. The railhead is normally used as a loading point for farm grains, and three grain elevators provide weighing facilities for all ore shipments.

Access to the mine is provided by about 14 miles of graveled country road and approximately two to three miles of rough mountain road. The country road grades are very mild with the only severe road grade occurring about one-half mile from the mine where the slopes may approach 10 to 14%.

The hydrous iron ore is a replacement of Madison limestone at the contact with the porphyry. At least five deposits occur along this contact that can be traced for about six miles. The ore is a fine-grained mixture of magnetite and hematite with varying amounts of limonite. The principal gauge material is calcite. The vein dips about 53 to 68 degrees from horizontal, as shown in Fig. A.2. The exposed surface of the vein varies considerably in elevation and its quality also varies from one end of the mining area to the other. The highest percentage of pyrite (iron sulfide, FeS_2) is located at the west end of the vein. Magnetite and limonite content vary throughout the area.

The pyrite content was of prime importance because of its possible deleterious effect on cured concrete. The sulfur varied at some places from 0.01 to almost 3%. A maximum limit of 0.1% was initially placed on the sulfur content of the aggregate material purchased by the AEC.

The iron content of almost all of the ore samples tested by the Corps of Engineers for Hanford and the AEC[1] was above that required for the TRU

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[1]Corps of Engineers, Troutdale, Oregon, Lab. Nos. 12223 and 12222 Test Results, April 30, 1963 (W.O. 63-cpch-40).

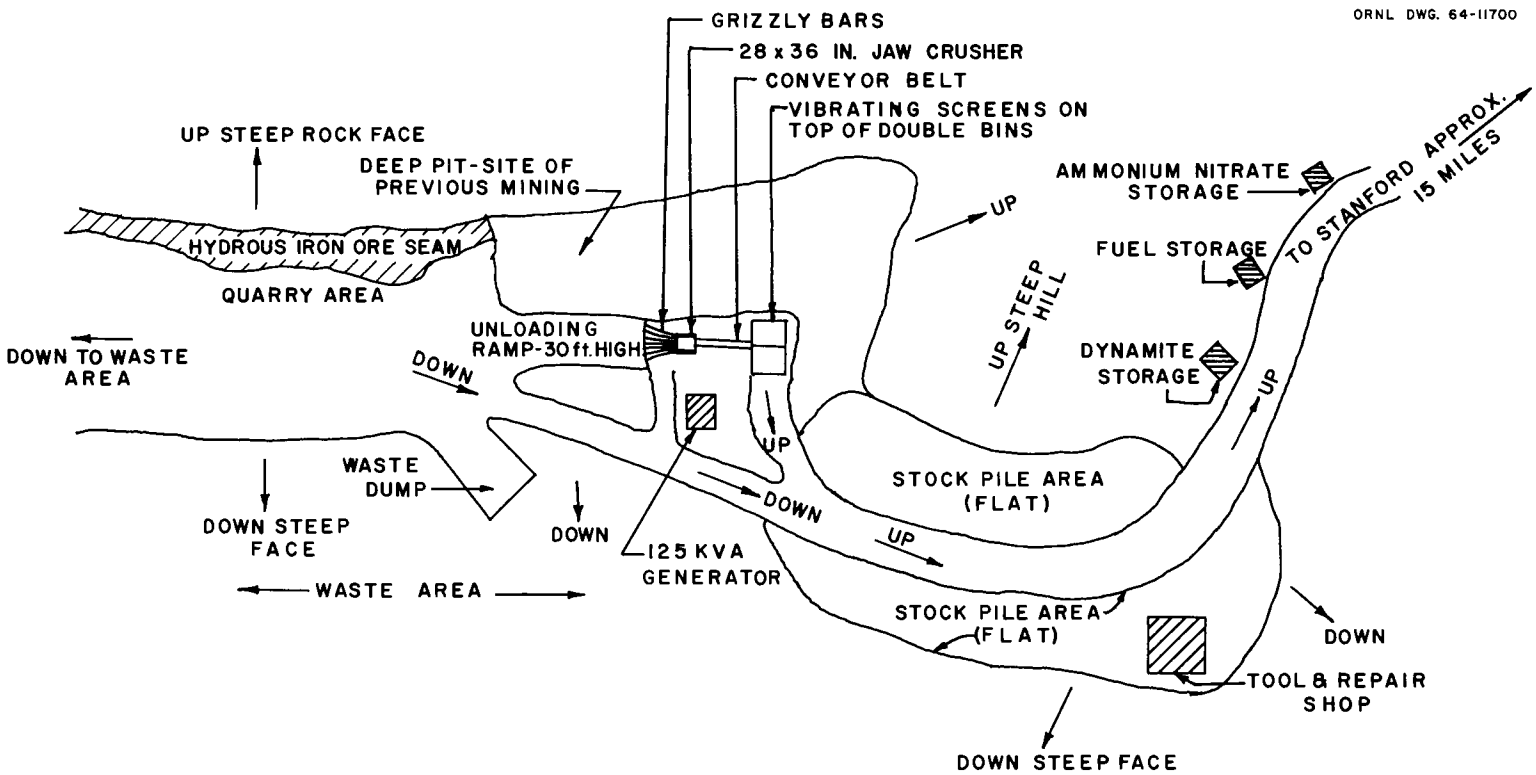


Fig. A.1. Plan of Running Wolf Mine Site.

ORNL DWG. 64-11699

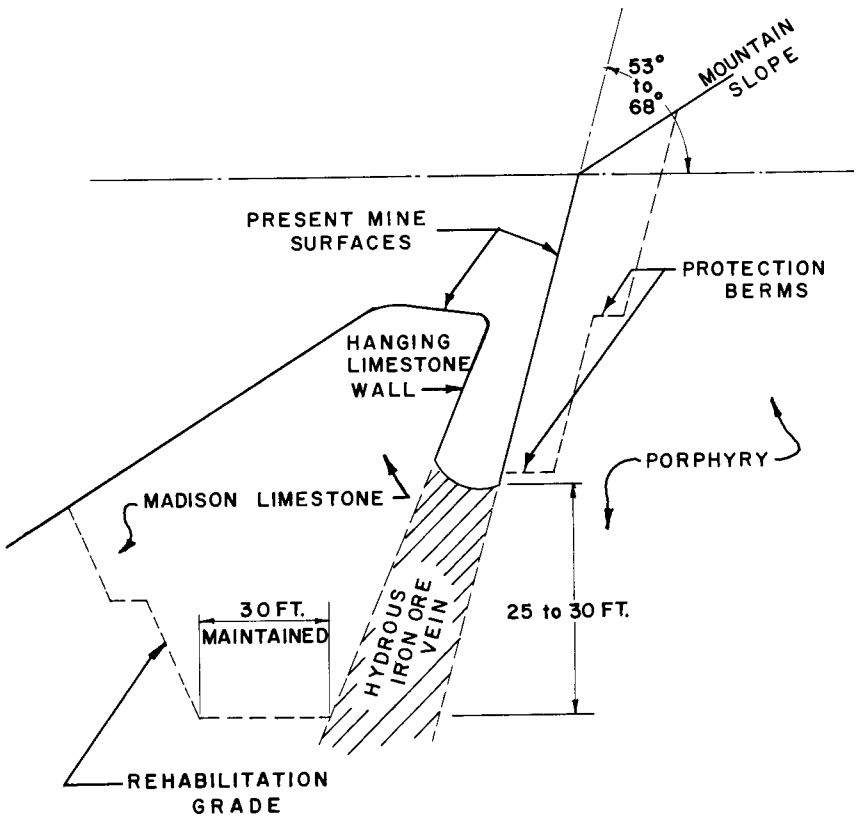


Fig. A.2.
Section Through Hydrous Iron Ore Vein.

Project. The high water of crystallization areas of the vein were not easily discernible by visual inspection, but predictions could be made based on the tests discussed below.

1. Scratch Test:

   Red-brown to red mark on ore, metallic crystal appearance, and slightly magnetic indicated magnetite-hematite. Good density, low sulfur, and low water content.

   Brown-red to dark brown mark on ore, rusty appearance, and non-magnetic indicated limonite-magnetite. Good water content, variable sulfur, and low density.

2. Visual Inspection:

   Visible metallic yellow flecks indicated high sulfur content. It was also evident from the chemical analysis reports that as the sulfur content increased the water of crystallization increased. The specific gravity of the pyrite containing ore was also high.

Aggregate Production

As shown in Fig. A.3, the rough ore was dumped into a feedhopper located over the main crusher. (The weight of the heavy iron ore must be considered when the slope of a hopper is established.) As the rough ore left the feedhopper, it passed over a set of grisly bars to remove the fine material made up of clay and limestone. Large amounts of hydrous iron ore fell through the bars because of the tendency of the ore to break in long, narrow pieces.

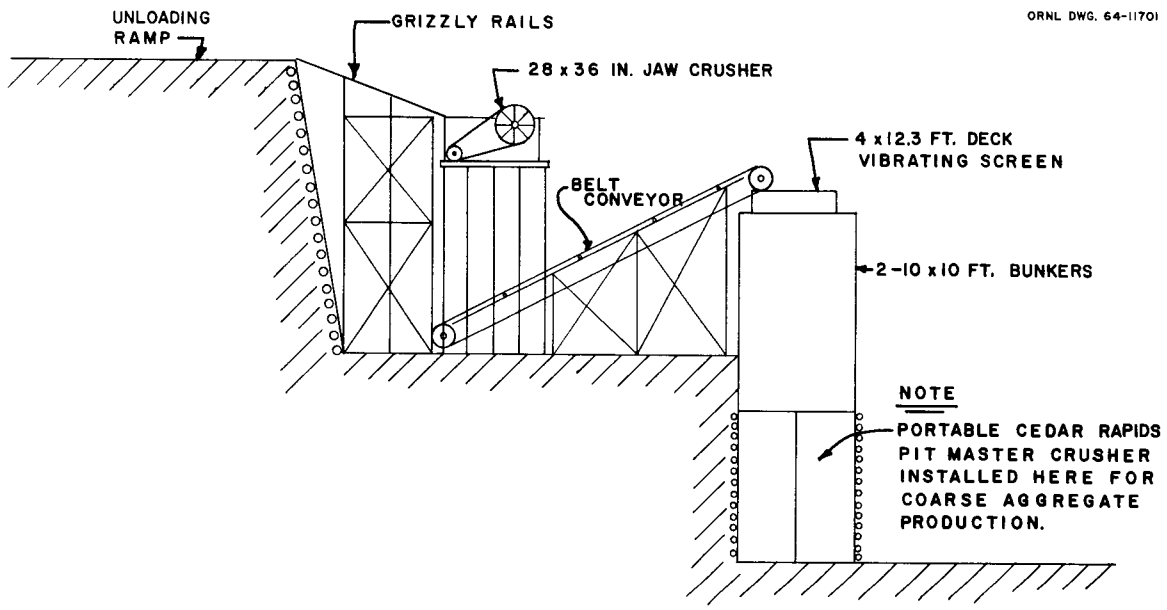


Fig. A.3. Cross Section of Running Wolf Crushing Plant.

The main crusher ejected ore onto a long belt running to a storage bin. Ore from the storage bin was then dropped into the hopper of a portable Cedar Rapids crushing and grading machine. In order to maintain high quality aggregate production, the material passing along the belt was checked closely for limestone and high pyrite ore. Although this handwork was essential, it was very time consuming.

A twin compartment storage bin was built to hold about 60 tons of 5-in. maximum size ore in each bin. This storage capacity was beneficial when either the main crusher or the Cedar Rapids machine broke down. The

5-in. ore from the storage bin was crushed to the final gradation in the Cedar Rapids machine, and the periodic control samples were taken from the final belt. Figures A.4 and A.5 are schematic illustrations of the total operation.

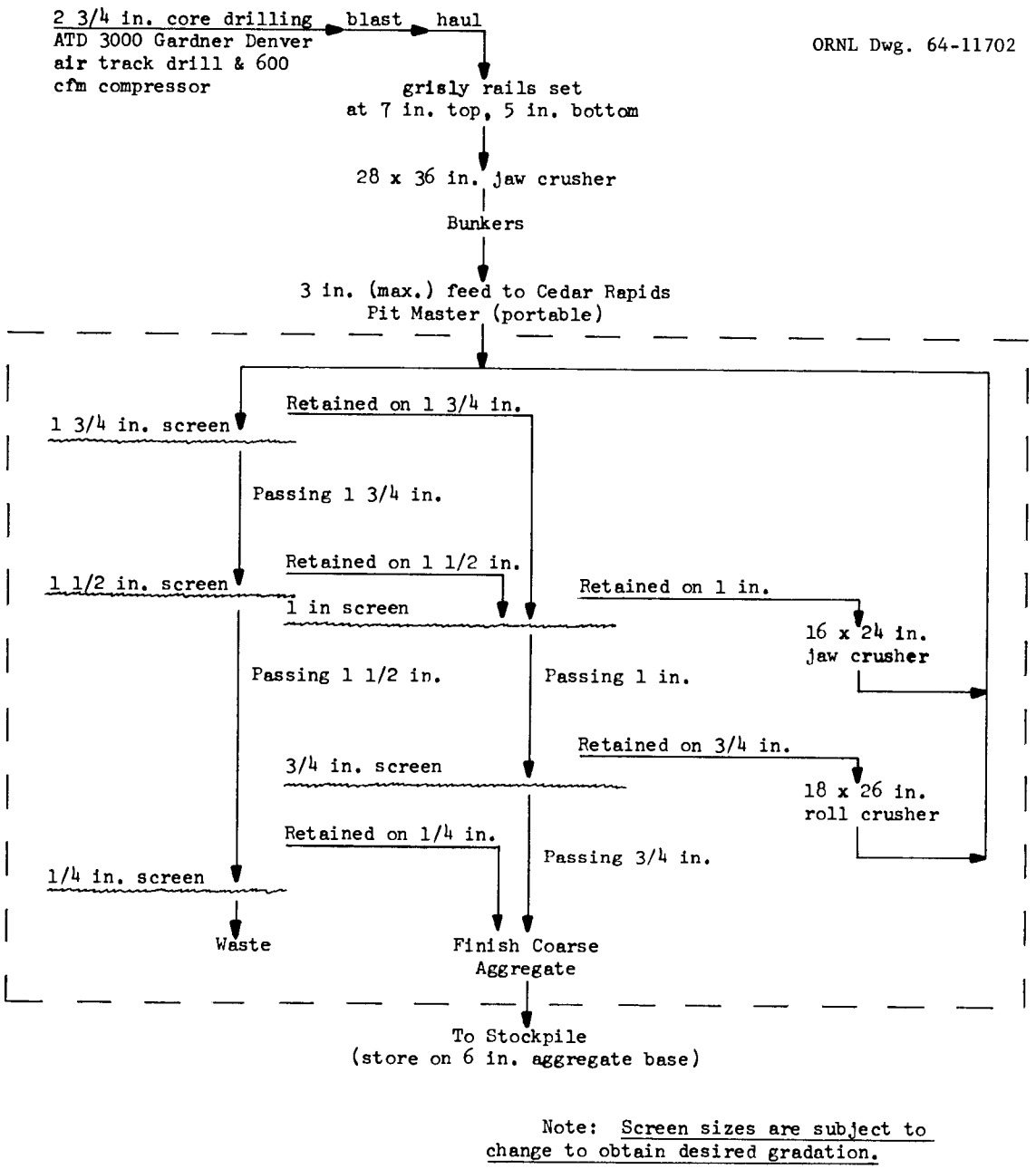
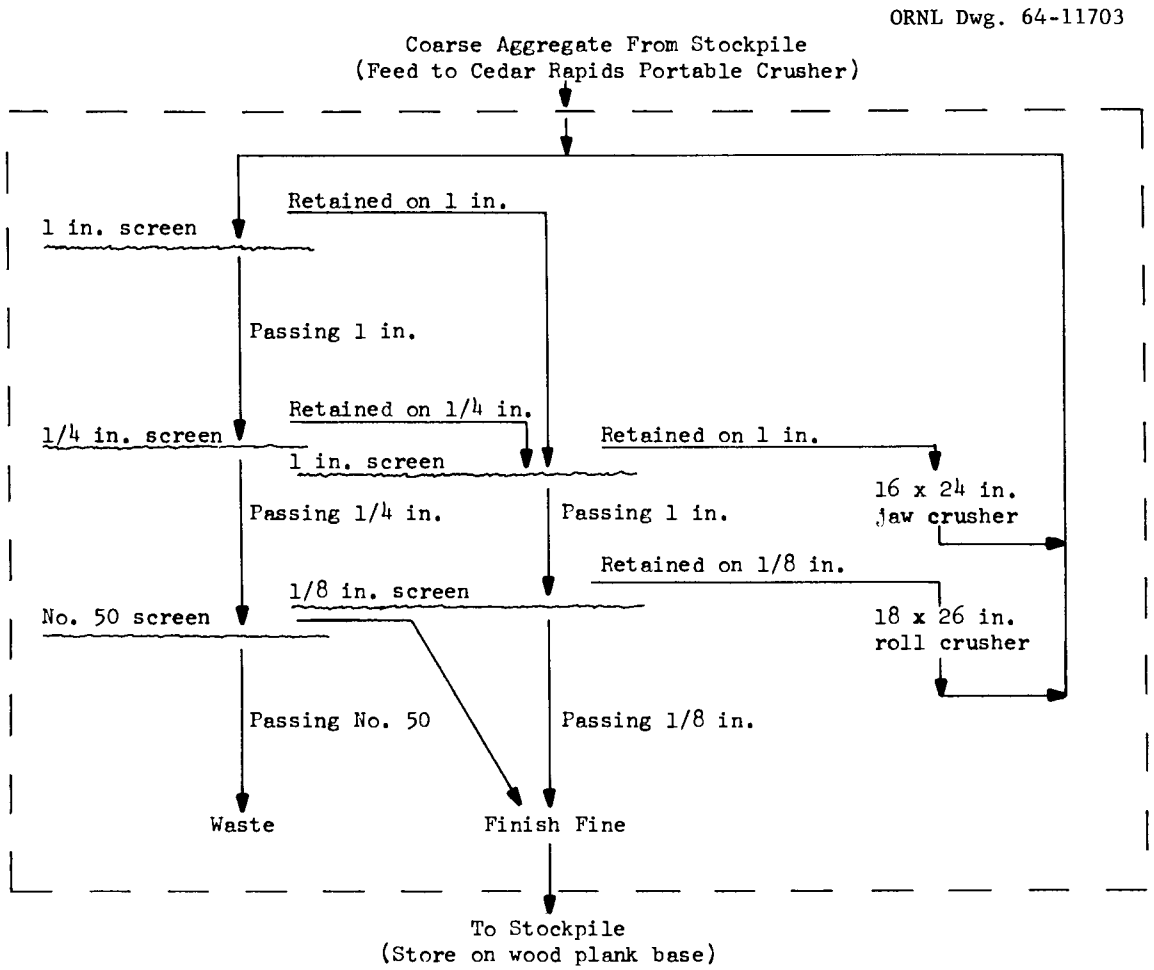


Fig. A.4.
Production Scheme for Coarse Aggregate.



Note: Screen sizes are subject to change to obtain desired gradation.

Fig. A.5. Production Scheme for Fine Aggregate.

Quality Control

The ore sampling techniques of ASTM C-33 and associated testing procedures were followed. ASTM D-75 provides several methods of ore sampling that include vein and stockpile sampling. The stockpile sampling method was decided upon as being more realistic since selective mining of the vein was employed. The coarse aggregate was accumulated in low piles (truck dump) where stockpile techniques could be used to obtain a representative sample for each 50 tons of material produced. Acceptance or rejection of

each pile of material was based on the test results. Since it was proposed that the fine aggregate be produced from the coarse aggregate, additional chemical testing was not required. Only mechanical gradation tests of the fine material were required.

The aggregate was broken into long, flat pieces, which was attributed to the nature of the material and the fact that it was being crushed from 5 in. to 1 1/2 in. pieces in one stage. A piece-by-piece count of several samples showed that the percentages of material exhibiting dimensions over twice the least dimension desired were as follows:

Material Size (in.)	Percentage by Weight
1	16
3/4	25
1/2	25
3/8	38

These elongated pieces of aggregate would tend to bridge around the reinforcing, sleeves, corners, etc., thereby reducing the unit weight of the concrete and its effectiveness as a shield.

Appendix B

SHIELDING AGGREGATE DATA

The shielding aggregate data tables in this section should provide a starting point for future searches for material, as well as a systematic method of recording and retaining the information that is accumulated. The information given in Tables B.1 through B.27 is not complete, and some of the data are already outdated because costs cannot be held constant.

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
1964	B.1	Barytes

SPECIAL INFORMATION

CONTACT: 1. B. C. Woods, Ten Mile Stone Co., Sweetwater, Tenn.
2. Earl Bolinger, Del Rio, Tenn.
3. David Groves, Knoxville, Tenn. (Tp: 525-6494)
4. New Riverside Ochre Co., Cartersville, Ga.

PER 2000 Lb./Ton.

COST:	Graded at Sweetwater	$19.00
FREIGHT:	By truck	2.50
TOTAL: F.O.B. OAK RIDGE, TENN.		$21.50

MINE DATA Ochre Co: Fine Agg. is blend of:
(1) Two parts "Float Jig Ore" (High Grade)
(2) One part "Separator Concentrate" (Chem. Grade)

REMARKS:
a. ORNL Report 3130, July 10, 1961
b. Max. 3/4 in. coarse and fine mixed together produced dry rodded unit weight of 176 lb per ft^3

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 4.04[a]

UNIT WT. (#/Ft.^{3}) C. 134 F. 148[b]

ABSORPTION =

GRADATION: ASTM-C 33

SIZE	% PASSING	SIZE	% PASSING
3/8	100	1 1/2	100
No.4	90-100	1	90-100
8	65-85	3/4	80-95
16	35-60	1/2	40-65
30	20-40	3/8	24-40
50	10-35	No.4	0-10
100	12-24	8	0-2
200	6-12		

COMPOSITION

COMPOUND	% BY WT. REF.	ELEMENT	% BY WT. REF.	SPECTRO REF.
$BaSO_4$	89-93			
Fe_2O_3	5.0			

SPECTRO SYMBOLS (PERCENT)
A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
December 1962	B.2	Tahawus Magnetite

SPECIAL INFORMATION

CONTACT:

National Lead Company
111 Broadway
New York 6, New York
Mr. John M. Ciborski, Mgr.

PER 2000 Lb./Ton.

COST:	$ 8.80
FREIGHT:	8.09
TOTAL: F.O.B. OAK RIDGE, TENN.	$16.89
MINE DATA	Tahawus, New York

REMARKS:

a. Furnished by National Lead Company, 8-1-61

b. Ilmenite mining operations produce the magnetic concentrate as a co-product

PHYSICAL AND CHEMICAL DATA

Sp. Gr.

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION:

SIZE	% PASSING	SIZE	% PASSING
No.28	87.0		
35	68.1		
48	50.8		
65	37.0		
100	26.3		
150	18.2		
200	12.1		
-200	0		

COMPOSITION

COMPOUND	% BY WT. REF. a	ELEMENT	% BY WT. REF. a	SPECTRO REF.
		Fe	55-58	
P_2O_5	0.02-0.03			
SiO_2	1.0-3.0			
MnO	0.1-0.3			
Al_2O_3	4.0-5.0			
CaO	0.3-1.0			
MgO	1.5-2.0			
Cr_2O_3	0.2-0.4			
V_2O_5	0.7			
TiO_2	10-11	S	0.28	

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
December 1963	B.3	Dover Magnetite

SPECIAL INFORMATION

CONTACT:

Warcrete Marine Corp.
99 Norman Ave.
Brooklyn 22, New York or
Dover, New Jersey

PER 2000 Lb./Ton.

COST:	Graded	$40.00
FREIGHT:	Carload Lot (Estimated)	8.00
TOTAL: F.O.B. OAK RIDGE, TENN.		$48.00

MINE DATA Morris County, New Jersey
(Near Dover, New Jersey)

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 4.51[a]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION = 0.1

GRADATION:

SIZE	% PASSING	SIZE	% PASSING
1	100		
3/4	78		
1/2	35		
3/8	23		
No.4	13		
8	10		

REMARKS:

a. South Atlantic Corps of Engineers, Report 1891, 12-6-63
b. ORNL Report 8533, 11-21-63
c. Non Evaporable Water (850°C), % by Weight (100°C Dry Basis)
d. Supplied by Warcrete, December 1961

COMPOSITION

COMPOUND	% BY WT. REF.[a,c]	ELEMENT	% BY WT. REF.[a,b,d]	SPECTRO REF.
H_2O	0.3	Fe	58.6	
		S	<0.01	
		Fe	59.96	
		S	58 ppm	
H_2O[b]	0.2	Fe	64.16	
		Si	6.54	
		P	0.7	
		Al	2.15	
		Mg	0.79	
		Ca	1.92	
		Ti	0.67	
		S	0.006	

SPECTRO SYMBOLS (PERCENT)
A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
1962	B.4	Cranberry Magnetite

SPECIAL INFORMATION

CONTACT:

Cranberry Magnetite Corporation
Cranberry, North Carolina
Mr. N. T. Dixson
Tp: Newland, N. C., 704-733-2115

PER 2000 Lb./Ton.

COST:	Graded at mine	$12.00
FREIGHT:	25-ton trucks	8.00
TOTAL: F.O.B. OAK RIDGE, TENN.		$20.00

MINE DATA

Crushed and graded at mine site, Cranberry, N. C.

REMARKS:

a. Composition Ref: U. S. Dept. of Interior Report of Investigations, RI 3980, December 1946, by F. D. Lamb and D. A. Woodard

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 4.0 to 4.3

UNIT WT. (#/Ft.^{3}) C. 142 F. 189

ABSORPTION = 0.3

GRADATION: Close to ASTM C-33

SIZE	% PASSING	SIZE	% PASSING

COMPOSITION

COMPOUND	% BY WT. REF. a	ELEMENT	% BY WT. REF. a	SPECTRO REF.
		Fe	48.1	
SiO_2	17.8	S	0.14	
CaO	8.4	P	0.005	
TiO_2	0.17	O	25.385	(Assumed)

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 27, 1962	B.5	Wyoming Magnetite

SPECIAL INFORMATION

CONTACT: Mr. F. C. Burton

Nuclear Shielding Supplies and Services, Inc.

175 Main Street

White Plains, New York

Tp: Rockwell 1-1214

PER 2000 Lb./Ton.

COST:	All Grades at Cheyenne, Wyo.	$10.80
FREIGHT:	Hopper Bottom Gondolas (October 1961)	13.95
TOTAL:	F.O.B. OAK RIDGE, TENN.	24.75

MINE DATA

Wyoming - Shipments made from stockpile all year.

REMARKS:

a. Supplier's Data Sheet

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 4.50[a]

UNIT WT. (#/Ft.^{3}) C. 170 F. 186

ABSORPTION =

GRADATION:[a] F.M. Fines 2.66, Coarse 7.35

SIZE	% PASSING		SIZE	% PASSING
2	100		3/8	100
1 1/2	93		No.4	94.6
1	36	100	8	66
3/4	10	98.5	16	47.3
1/2	2.5	57.2	30	27
3/8	0.2	28.4	50	17.7
No.4	0.1	2.2	100	10
			200	2

COMPOSITION

	COMPOUND	% BY WT. REF.a	ELEMENT	% BY WT. REF.a	SPECTRO REF.
	MnO	0.33	Fe	50.25	
Magnetite	Fe_3O_4	49.8	Ti	12.19	
Ilmenite	FeO TiO_2	38.5	O	31.07	
	SiO_2	2.7	Si	1.26	
	Al_2O_3	4.4	Al	2.33	
	CaO	0.54	Ca	.38	
	MgO	2.9	Mg	1.75	
	Cr_2O_3	0.09	Cr	.06	
			V	.32	
	V_2O_5	0.57	Mn	.26	
	P_2O_5	0.07	P	.03	
	S	0.10	S	.10	

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
October 1963	B.6	Mahoning Limonite

SPECIAL INFORMATION

CONTACT:

Mahoning Mine

Hibbing, Minnesota

Ed Tyler, Mine Manager

PER 2000 Lb./Ton.

COST:

FREIGHT:

TOTAL: F.O.B. OAK RIDGE, TENN.

MINE DATA

Hibbing, Minnesota (mine closes for winter)

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 4.22[f], 3.945[a], 3.878[e]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION: As Mined - 28% Passing No. 100

SIZE	% PASSING [g]	SIZE	% PASSING
No.4	98.36		
8	80.56		
16	65.44		
30	49.44		
50	30.24		
100	13.32		

REMARKS:

a. ORNL Report 5249, 10-4-63

b. Combined Water by Train Method

c. Free Water

d. ORNL Report Film 8313, 10-24-63

e. ORNL Report 6115, 10-17-63

f. Obtained from Mine Records, September 1963

g. Normal plant production gradation

COMPOSITION

COMPOUND	% BY WT. REF. a	ELEMENT	% BY WT. REF.	SPECTRO REF. d
H_2O[b]	9.51	Al		D
H_2O[c]	0.43	Cu		E
		Fe		A
		Mg		E
Ignition Loss	9.90	Si		C-B
			f	
	e	Fe	58.83	
H_2O[b]	9.91	Si	4.25	
H_3O[c]	0.38	S	<0.007	
	Ignition	Loss	10.10	(1500°F)

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 30, 1963	B.7	Blackburn Limonite

SPECIAL INFORMATION

CONTACT:

Mr. A. M. Shook III or Mr. H. O. Thomas
Shook & Fletcher Supply Co.
Birmingham, Ala.
814 First Ave., N. Tp: 322-6721

PER 2000 Lb./Ton.

COST:	Blast furnace gradation	$ 7.50
	Quality control	0.31
FREIGHT:	Hopper cars	3.65
TOTAL: F.O.B. OAK RIDGE, TENN.		$11.46

MINE DATA[e] 3 miles west on Hwy 24
Russellville, Route No. 3, Ala.
Mr. E. H. Craddock. Highly developed pit mine

REMARKS:

a. ORNL Lab Test 4018, 9-9-63
b. Additional crushing and grading is required for concrete work. Approximately 1500 tons of 3 to 1/16 in. material was crushed and graded for AEC at 6.82/ton at Oak Ridge, Tenn. Total material cost using 18% loss experience was 11.46(1.22)=13.94+6.82= $20.76/ton at batch plant.
c. Combined water by train method
d. Free water
e. One shift, 6-day week - 10,000 tons/month, 150,000 ton stockpile as of 10-1-63. Year-round operation - operated since 1949.
f. Shook Field Office Analysis - 9-24-63
g. After crushing & grading (see b. above)
h. Ignition loss at 1950°F on 10-1-63 at Shook Field Office.

NOTE: Woodward Iron Co. Daily Report, 9-21-63 on ore: Fe=52.54, Al_2O_3=2.7, S=.02, P=.61, Mn=.57, H_2O^d=3.97, SiO_2= 6.52

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 3.669[a]
UNIT WT. (#/Ft.³) C. 139[g] F. 141[g]
ABSORPTION = 2.2
GRADATION:[b] 3 in. to 1/16 in. & 3/8 in. to No. 30

SIZE	% PASSING	SIZE	% PASSING
3 in.	93.0	3/8	91.7
2	87.5	No.4	54.5
1 1/2	77.0	8	26.7
1	56.0	16	10.0
3/4	39.0	30	5.0
1/2	17.5	50	3.0
3/8	6.5	100	1.0
		200	0.5

COMPOSITION

COMPOUND	% BY WT. REF. a	ELEMENT	% BY WT. REF. f	SPECTRO REF.
H_2O^c	11.16	Fe	51.30	
H_2O^d	1.17	Mn	0.67	Fine
Fe	55.2	P	0.57	
S	5 ppm	SiO_2	10.30	
		Fe	54.40	
		Mn	0.54	
		P	0.59	
		SiO_2	7.12	
		Fe	53.3	
		Mn	0.49	Mine Run
		P	0.62	
		SiO_2	9.04	

SPECTRO SYMBOLS (PERCENT)
A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 25, 1963	B.8	Georgia Iron Ore

SPECIAL INFORMATION

CONTACT:

Mr. Harry Pohlem

Tenn. Products Co.

Rockwood, Tenn.

Tp: 354-9362

PER 2000 Lb./Ton.

COST:

FREIGHT:

TOTAL: F.O.B. OAK RIDGE, TENN.

MINE DATA

Material purchased by Tenn. Products.

Originating source unknown.

REMARKS:

a. ORNL Lab. Test 3173 (9-13-63)

b. Combined Water by Train Method

c. Free Water

d. Additional crushing and grading is required for concrete work.

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 3.3[a]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION: [d]For Blast Furnace use(3 in. max size)

SIZE	% PASSING	SIZE	% PASSING

COMPOSITION

COMPOUND	% BY WT. REF.a	ELE-MENT	% BY WT. REF.	SPECTRO REF.a
H_2O[b]	9.17	Fe		A
H_2O[c]	4.50	Si		B
S	0.015	Al		C
		Co		E
		Cr		F
		Cu		E
		Mn		D
		Ni		E
		Zn		D

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 30, 1963	B.9	Tranham Iron Ore

SPECIAL INFORMATION

CONTACT:

Mr. Merril Fraizer

Plateau Iron Ore Corp.

West Plains, Mo.

Tp: 417-256-485

PER 2000 Lb./Ton.

COST:

FREIGHT:

TOTAL: F.O.B. OAK RIDGE, TENN.

MINE DATA

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 2.684[a]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION:[d]

SIZE	% PASSING	SIZE	% PASSING

2 Grades for Blast Furnace Use

3 in. to fines are available

COMPOSITION

COMPOUND	% BY WT. REF.a	ELE-MENT	% BY WT. REF.	SPECTRO REF.
H_2O[b]	7.92			
H_2O[c]	10.5			

REMARKS:

a. ORNL Lab. Tests, 4308 (9-23-63)

b. Combined Water by Train Method

c. Free Water

d. Additional grading for concrete work anticipated. Material is washed at mine then again at Plateau Iron Ore Corp. along with classification to eliminate unwanted materials.

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 30, 1963	3.10	Willard Iron Ore

SPECIAL INFORMATION

CONTACT:

Mr. Merril Fraizer
Plateau Iron Ore Corp.
West Plains, Mo.
Tp: 417-256-485

PER 2000 Lb./Ton.

COST:	F.O.B. Rockwood, Tenn.[d]	$13.65
FREIGHT:	Additional grading at West Plains (Estimated)	3.35
TOTAL:	F.O.B. OAK RIDGE, TENN.	$17.00

MINE DATA

20 miles southwest of West Plains, Mo.

REMARKS:

a. ORNL Lab Test 4306 (9-23-65)
b. Combined water by train method
c. Free water
d. Includes $4 or $4/2000 lb ton shipping
e. Additional grading for concrete work anticipated. Material is washed at mine then again at Plateau Iron Ore Corp. along with classification to eliminate unwanted materials.

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 2.89[a]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION:

SIZE	% PASSING	SIZE	% PASSING

2 grades for blast furnace use
3 in. to fines are available

COMPOSITION

COMPOUND	% BY WT. REF. a	ELEMENT	% BY WT. REF.	SPECTRO REF.
H_2O[b]	8.13			
H_2O[c]	8.01			

SPECTRO SYMBOLS (PERCENT)
A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 30, 1963	B.11	Howard Iron Ore

SPECIAL INFORMATION

CONTACT:

Mr. Merril Fraizer

Plateau Iron Ore Corp.

West Plains, Mo.

Tp: 417-256-485

PER 2000 Lb./Ton.

COST:

FREIGHT:

TOTAL: F.O.B. OAK RIDGE, TENN.

MINE DATA

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 3.202[a]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION:[d]

SIZE	% PASSING	SIZE	% PASSING

2 grades for blast furnace use

3 in. to fines are available

REMARKS:

a. ORNL Lab Test 4307 (9-23-63)

b. Combined water by train method

c. Free water

d. Additional grading for concrete work anticipated; material is washed at mine then again at Plateau Iron Ore Corp. along with classification to eliminate unwanted materials.

COMPOSITION

COMPOUND	% BY WT. REF. a	ELEMENT	% BY WT. REF.	SPECTRO REF.
H_2O[b]	7.15			
H_2O[c]	4.79			

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 25, 1963	B.12	Wilson Iron Ore

SPECIAL INFORMATION

CONTACT:

Mr. Charles Wilson

Rockwood, Tenn.

Tp: 354-0430 (Home)

354-9362 (Tenn. Products Co.)

PER 2000 Lb./Ton.

COST:

FREIGHT:

TOTAL: F.O.B. OAK RIDGE, TENN.

MINE DATA [a]

Union County near LaFollette, Tenn.

PHYSICAL AND CHEMICAL DATA

Sp. Gr.

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION:

SIZE	% PASSING	SIZE	% PASSING

REMARKS:

a. Mine has been shut down since first of 1963 due to high phosphorus content of ore.

b. Fron Rockwood Blast Furnace Report of Ores Analyzed November 1962.

c. Owner will provide samples for analysis at a fee of approximately $100.

COMPOSITION

COMPOUND	% BY WT. REF.	ELEMENT	% BY WT. REF. [b]	SPECTRO REF.
		Fe	52.52	
		Insol	10.30	
		P	0.398	
		Mn	0.39	

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 25, 1963	B.13	Smith Iron Ore

SPECIAL INFORMATION

CONTACT:

E. B. Smith

LaFollette, Tenn.

Tp: 562-2895

PER 2000 Lb./Ton.

COST:

FREIGHT:

TOTAL: F.O.B. OAK RIDGE, TENN.

MINE DATA

Northeast of LaFollette on Flat Hollow Road

Private property; not developed[d]

REMARKS:

a. 9-12-63, ORNL Lab Tests, 8161, 3169, 3170

b. Combined water by train method

c. Free water

d. The ore breaks surface on E. B. Smith's property. Quantity and quality has not been established.

Note: Hydrous Iron ($2Fe_2O_3 \cdot 3H_2O$ and $Fe_2O_3 \cdot H_2O$) Limonite or Geothite

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 3.57, 3.62, 3.1, 3.4[a]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION:

SIZE	% PASSING	SIZE	% PASSING

COMPOSITION

COMPOUND	% BY WT. REF. a	ELEMENT	% BY WT. REF.	SPECTRO REF. a
H_2O[b]	9.12	Fe		A
H_2O[c]	1.62	Cu		F-E
S	0.009	Al		D-C
H_2O[b]	9.00	Co		E
H_2O[c]	1.69	Mn		C
S	0.015	Ni		E
H_2O[b]	9.20	Si		B
H_2O[c]	1.65			
S	17 ppm			

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
October 1963	B.14	Ferrophosphorus

SPECIAL INFORMATION

CONTACT:

Tennessee Valley Authority (T.V.A.)

Muscle Shoals, Alabama

Mr. Craotree

PER 2000 Lb./Ton.

COST:		
	Coarse	$60.00
	Fine	30.00
	Ungraded (3 to 10 in.)	32.00

FREIGHT:

TOTAL: F.O.B. OAK RIDGE, TENN.

MINE DATA

Chemical Plant byproduct at Muscle Shoals.

Crushing equipment is makeshift and antiquated.

REMARKS:

a. Report by T.V.A., July 1960

b. Each ton of coarse (10 in. max) material products 3/4 ton of fines and 1/4 ton of coarse during crushing operations. Approximately 50% of the fines are wasted as too fine. Large variation in material structure. Small stockpile of large chunks.

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 6.2

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION:[b] ASTM C-33

SIZE	% PASSING	SIZE	% PASSING

COMPOSITION

COMPOUND	% BY WT. REF.	ELEMENT	% BY WT. REF. [a]	SPECTRO REF.
		P	23.7	
		Fe	68.1	
		Mn	2.8	
		Ti	1.7	
		Si	1.7	
		Lost	2.0	
			100.0	

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
1964	B.15	Ferrophosphorus

SPECIAL INFORMATION

CONTACT:

Mr. Dan Andrews
Mt. Pleasant
Tennessee
615-379-3251

PER 2000 Lb./Ton.

COST: [c]	Graded (in bulk)	$55.00
FREIGHT: [d]	Truck (includes $2.66 per ton crate charge)	9.66
TOTAL: F.O.B. OAK RIDGE, TENN.	at job site	$64.66

MINE DATA Chemical Plant is at Mt. Pleasant, Tennessee, Mgr. Lex Hutchinson (Tp: 3793251). Adequate crushing and grading equipment.

REMARKS:

a. Sp. Gr. (SSD) from Prepakt Concrete Co. Report D. CC. Job No. Du 62-4, 6-14-61 Tests on High Density Prepakt Concrete, Ferrophosphorus and Colemanite Aggregates.
b. KAPL-901
c. Purchase price paid for approximately 1000 tons of coarse material, January 1964.
d. 638-3000 lb crates of coarse material hauled by truck.
e. U. S. South Atlantic Corps of Engineers Report on W. O. 1891, 1-20-64.
f. ORNL Report 6113, 10-17-63, and Film 8321, 8329.
g. Shipments must be strictly controlled to minimize aggregate degradation. (L. A. Abrasion (A) 98% loss at 500 Rev.) 3000 lb crates are recommended.

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 5.72[a], 6.50[b], 5.96[e], 6.48[f]

UNIT WT. (#/Ft.³) C. 215, 226[e] F.

ABSORPTION = 0.9

GRADATION:[g] ASTM C-33 or other

SIZE	% PASSING	SIZE	% PASSING

COMPOSITION

COMPOUND	% BY WT. REF. b	ELEMENT	% BY WT. REF. f	SPECTRO. REF. i
Fe	70	Ti		C
P	25	Cr		D
Mn	3	Cu		D
Si	1	Ca		E
Ti	1	Ni		D
		V		D
		C	0.035	
		S	<0.002	
		Fe	68.59	
		Mn	2.9	
		P	23.3	
		Si	0.36	
Av. of 15 Samples		Fe	68.1 Jan. 20, 1964[e]	

SPECTRO SYMBOLS (PERCENT)
A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE: 1963

TABLE: B.16

SHIELDING AGGREGATE: Hydrous Iron Ore

SPECIAL INFORMATION

CONTACT:

Running Wolf Mine

Stanford, Montana

Mr. Dewey Whittaker

PER 2000 Lb./Ton.

COST: Coarse Material (Contract Price)	$22.70
FREIGHT: Open 50-ton hopper cars	16.20
TOTAL: F.O.B. OAK RIDGE, TENN.[b] ORNL job site	$40.90

MINE DATA

15 miles from Stanford in the Gibson Mountain Range

REMARKS:

a. Average of 600 tons of coarse aggregate purchased by AEC for ORNL during 1963.

b. Combined water by train method.

c. 1 1/2 in. max. and 3/4 in. max., Coarse Gradation.

d. Includes $2.00/ton cost for unloading hopper cars and trucking material to batch plant.

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 4.41[a]

UNIT WT. (#/Ft.³) C. 159.6[c], 149 F.

ABSORPTION = 0.68

GRADATION: Mined ore - Requires crushing and grading

SIZE	% PASSING	SIZE	% PASSING

COMPOSITION

COMPOUND	% BY WT. REF. a	ELEMENT	% BY WT. REF.	SPECTRO REF.
H_2O[b]	3.65			
Fe	63.33			
S	0.40			

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
November 1962	B.17	Calcined Magnesite

SPECIAL INFORMATION

CONTACT:

Mr. R. A. Creed or W. C. Marshall
Inorganic Chemicals Division
633 Third Ave., New York 17, New York
212 Murray Hill 7-7400

PER 2000 Lb./Ton.

COST: [a]	75 lb bags	$100.00
FREIGHT: [a]	40-ton cars	24.70
TOTAL: F.O.B. OAK RIDGE, TENN.		$124.70

MINE DATA [b]

PHYSICAL AND CHEMICAL DATA

Sp. Gr. MgO=3.1

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION:

SIZE	% PASSING	SIZE	% PASSING

REMARKS:

a. Letter from John Manville, dated 11-6-62 (Mr. W. C. Marshall)

b. $MgCO_3$ is calcined to drive off CO_3 to leave MgO

c. Composition of Magnesium Oxychloride Cement. See Table E.18 for $MgCl_2$ source.

d. FMC Tech Bulletin on MgO Cements

COMPOSITION [c]

COMPOUND	% BY WT. REF. d	ELEMENT	% BY WT. REF.	SPECTRO REF.
3MgO	29.2	Mg	23.48	
$MgCL_2$	23.0	Cl	17.12	
$11H_2O$	47.8	O	54.03	
		H	5.37	
or				
5MgO	37.9	Mg	27.39	
$MgCL_2$	18.0	CL	13.41	
$13H_2O$	44.1	O	54.25	
		H	4.95	

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
November 1962	B.18	Magnesium Chloride

SPECIAL INFORMATION

CONTACT:

Mr. R. A. Creed or W. C. Marshall
Inorganic Chemicals Division
633 Third Ave., New York 17, New York
212 Murray Hill 7-7400

PER 2000 Lb./Ton.

COST: [b]	100 lb bags	$60.00
FREIGHT: [b]	40-ton cars	22.80
TOTAL: F.O.B. OAK RIDGE, TENN.		$82.80

MINE DATA

PHYSICAL AND CHEMICAL DATA

Sp. Gr. $MgCl_2$=1.18[a]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION: Flake Magnesium Chloride

SIZE	% PASSING	SIZE	% PASSING

REMARKS:

a. 22° Be $MgCl_2$ solution. $MgCl_2$ only has an approximate specific gravity of 2.33

b. Letter from John Manville, dated 11-6-62 (Mr. W. C. Marshall)

c. For composition of Magnesium Oxychloride Cement see Table E.17

COMPOSITION [c]

COMPOUND	% BY WT. REF.	ELEMENT	% BY WT. REF.	SPECTRO REF.

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
1959	B.19	Ilmenite

SPECIAL INFORMATION

CONTACT:

Continental Iron and Titanium Mining Co.
St. Paul, Quebec
Nuclear Shielding Supplies and Services, Inc.
175 Main Street, White Plains, N. Y.

PER 2000 Lb./Ton.

COST:

FREIGHT:

TOTAL: F.O.B. OAK RIDGE, TENN.

MINE DATA Open pit mined by crane. Crushed at plant near mine. Shipments made from stockpile all year.

REMARKS:

a. Furnished by Prepakt Concrete Co., Cleveland 14, Ohio
b. Nuclear Shielding Supplies and Services, Inc.

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 4.6

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION = Negligible

GRADATION: b

SIZE	% PASSING	SIZE	% PASSING
Coarse: 1 1/2 to 1/4" F.M. 7.5			
Fine: No. 100 to No. 4 F.M. 3.0			

COMPOSITION

COMPOUND	% BY WT. REF. b	ELEMENT	% BY WT. REF. a	SPECTRO REF.
TiO_2	42.9	Ti	25.69	
Fe	33.7	Fe	37.67	
Fe_2O_3	16.8	O	32.61	
SiO_2	1.1	Si	0.49	
Al_2O_3	1.4	Al	0.74	
CaO	0.15	Ca	0.11	
MgO	3.3	Mg	1.96	
Cr_2O_3	0.22	Cr	0.15	
V_2O_5	0.32	V	0.17	
MnO	0.32	S	0.04	
P_2O_5	0.028	Mn	0.25	
S	0.04	P	0.12	

SPECTRO SYMBOLS (PERCENT)
A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 11, 1963	B .20	Ilmenite

SPECIAL INFORMATION

CONTACT:

Quebec Iron and Titanium Mining Co.
1510 Drummond Street
Montreal, Canada

PER 2000 Lb./Ton.

COST:

FREIGHT:

TOTAL: F.O.B. OAK RIDGE, TENN.

MINE DATA

Stockpile at Sorel, Quebec (Mr. H. F. Bartan, 514 Univ. 61592)

REMARKS:

PHYSICAL AND CHEMICAL DATA

Sp. Gr.

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION:

SIZE	% PASSING	SIZE	% PASSING

COMPOSITION

COMPOUND	% BY WT. REF.	ELE-MENT	% BY WT. REF.	SPECTRO REF.

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 25, 1963	B.21	Balsam Gap Olivine[e]

SPECIAL INFORMATION

CONTACT:

Mr. Jacks Haley — Mr. Jesse Miller
Balsam Gap Co. — 310 Penn Center Plaza
Waynesville, N. C. — Phil. 2, Pa.
704-456-9303 — Locust 8-2000, Ext 346

PER 2000 Lb./Ton.

COST:

FREIGHT:

TOTAL: F.O.B. OAK RIDGE, TENN.

MINE DATA[d] Waynesville, N. C.

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 3.08, 3.12[a]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION:

SIZE	% PASSING	SIZE	% PASSING

REMARKS:

a. ORNL Lab Test 3816, 3817 (9-16-63)
b. Combined water by train method (850°C)
c. Free water
d. Samples obtained from Chet Morris of ORINS who visited the mine in the spring of 1963 to obtain material with low natural radiation background.
e. Chrysolite $(MgFe)_2 SiO_4$

COMPOSITION

COMPOUND	% BY WT. REF. a	ELEMENT	% BY WT. REF. a	SPECTRO REF. a
H_2O[b]	1.17	Mg		A
H_2O[c]	0.13	Mn		C
		Si		B-A
H_2O[b]	1.43	Al		C-B
H_2O[c]	0.12	Ni		D
		Ca		D
		Cr		E
		Cu		E
		Fe		B

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
November 12, 1963	B.22	Balsam Gap Serpentine

SPECIAL INFORMATION

CONTACT:

Mr. Jesse Miller

Three Penn Center Plaza

Philadelphia 2, Pa.

Locust 8-2000, Ext. 346

PER 2000 Lb./Ton.

COST:

FREIGHT:

TOTAL: F.O.B. OAK RIDGE, TENN.

MINE DATA[d] Waynesville, N. C.

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 2.72[a]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION

GRADATION:

SIZE	% PASSING	SIZE	% PASSING

REMARKS:

a. ORNL Lab Test 6754 (10-28-63)

b. Combined water by train method (850°C)

c. Free water

d. Samples submitted by Mr. Miller and classified as a special sample (adjacent to olivine area) containing high water content

COMPOSITION

COMPOUND	% BY WT. REF. a	ELEMENT	% BY WT. REF. a	SPECTRO REF. a
H_2O[b]	9.26	Fe	5.05	
H_2O[c]	0.51	Al		Moderate
		Ca		Weak
		Cr		Weak
		Mg		Strong
		Mn		Weak
		Ni		Strong
		Si		Strong

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 25, 1963	B.23	Cardiff Serpentine[g]

SPECIAL INFORMATION

CONTACT:

Mr. Robert G. Hughes
Maryland Green Marble Corp.
Box 1198, Roanoke, Va.
Tp: 704-344-3253

PER 2000 Lb./Ton.

	Bags	Bulk
COST: Graded[d]	$25.00	$21.50
FREIGHT: 40-ton cars[f]	8.13	7.43
TOTAL: F.O.B. OAK RIDGE, TENN.	$33.13	$28.93

MINE DATA Cardiff, Harford County, Maryland.
Tp: Glendale 2-2111. 40 miles No. of Baltimore.
200' deep mine. All season work. 1000 tons/mo.

REMARKS: No problem

a. ORNL Lab Test 3368 (9-13-63)
b. Combined water by train method
c. Free water
d. Furnished by telephone from Hughes on 5-25-62, unless noted otherwise.
e. Furnished by letter from Hughes 6-12-62 as water of crystallization per Dr. Byron N. Cooper of V.P.I. at Blacksburg, Va., 703-PR-28-261, Ext 279
f. Furnished by letter from Hughes 5-25-62
g. Hydrous Magnesium silicate ($H_4Mg_3Si_2O_9$)

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 2.69[a]
UNIT WT. (#/Ft.^{3}) C. F.
ABSORPTION =
GRADATION: d

SIZE	% PASSING	SIZE	% PASSING
1 1/8-7/8:	7/8-5/8:	5/8-3/8:	
3/8-1/4:	1/4-3/32:	3/32-20:	20-Dust

COMPOSITION

COMPOUND	% BY WT. REF. d	ELE. MENT	% BY WT. REF. d	SPECTRO REF.
H_2O[b]	10.8[a]	Si	16.6	
H_2O[c]	0.74[a]	Fe	5.97	
		Al	0.40	
SiO_2	35.50	Ca	0.14	
Fe_2O_3	8.53	Mg	26.00	
Al_2O_3	0.75	H	1.38	
CaO	0.20	O	49.95	
MgO	43.05			
H_2O	12.41			
H_2O	12.86[e]			
			100.44	

SPECTRO SYMBOLS (PERCENT)
A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 25, 1963	B.24	Sonord Serpentine[f]

SPECIAL INFORMATION

CONTACT:

Sample for Test Supplied
By H. S. Davis of Hanford
Material from Sonord, California

PER 2000 Lb./Ton.	
COST:[d] (Estimated)	$20.00
FREIGHT:[e] From California	35.00
TOTAL: F.O.B. OAK RIDGE, TENN.	$55.00

MINE DATA

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 2.6[a]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION:

SIZE	% PASSING	SIZE	% PASSING

REMARKS:

a. Determined by ORNL 9-16-63 Lab No. 3818 and 3819 (Two samples)
b. Combined water by train method
c. Free water
d. Based on quotes from Maryland Green Marble Co., Roanoke, Va.
e. Based on quoted freight rates on borax, colemanite, trona, ulexite, rasorite, etc.
f. Hydrous magnesium silicate ($H_4Mg_3Si_2O_9$)

COMPOSITION

COMPOUND	% BY WT. REF. a	ELEMENT	% BY WT. REF.	SPECTRO REF.
H_2O[b]	11.7			
H_2O[c]	1.0			

SPECTRO SYMBOLS (PERCENT)
A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 9, 1963	B.25	Georgia - Serpentine

SPECIAL INFORMATION

CONTACT:

Tennessee Marble Co. or Georgia Marble Co.

Knoxville, Tenn. Atlanta 3, Georgia

Mr. Bayless, Tp: Jackson JA35591

PER 2000 Lb./Ton.

COST:	At plant in gradation listed	$ 8.00
FREIGHT:	90,000 lbs. open car (minimum)	2.30
TOTAL: F.O.B. OAK RIDGE, TENN.		$10.30

MINE DATA

Tate, Georgia

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 2.81[a]

UNIT WT. (#/Ft.³) C. F.

ABSORPTION =

GRADATION: 1 1/2 in. to fine

SIZE	% PASSING	SIZE	% PASSING

REMARKS:

a. Determined by ORNL 8-23-62

b. Combined train test (inert gas) 850°C

c. Determined by ORNL 8-23-63

d. Ignition loss values reported by some suppliers should not be taken as the amount of water lost because of the burn off of CO_3

e. Estimated

COMPOSITION

COMPOUND	% BY WT. REF.	ELEMENT	% BY WT. REF[c]	SPECTRO REF.
H_2O[b]	8.57	Fe	8.80	
CO_3[d]	10.85	Si	15.43	
		Mg	23.24	
		H	0.96	
		O	49.40	
		C	2.17	
			100.00	

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
October 17, 1963	B.26	Schuyler Serpentine[f]

SPECIAL INFORMATION

CONTACT:

Mr. W. Atkin

Alberine Stone Corp.

387 Park Ave., S.

New York 16, New York

PER 2000 Lb./Ton.

COST:	At plant in gradation listed (5-29-62)	$ 9.00
FREIGHT:	50,000 lb min. carload	10.40
TOTAL:	F.O.B. OAK RIDGE, TENN.	$19.40

MINE DATA Schuyler, Va.

(Quarry & mill south of Charlottesville, Va.)

No crushing equip. available as of 9-9-63.

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 2.928[a]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION: 1 1/2 in to fine

SIZE	% PASSING	SIZE	% PASSING

REMARKS:

a. Determined by ORNL 6114, 10-17-63

b. Combined H_2O by train test (inert gas) 850°C, ORNL 6114

c. Free H_2O (ignition loss varies from 8.44% to 18.36%)

d. Provided by Mr. Bayless, Atlanta, JA 35591, 5-29-62

e. ORNL Film 8313, 10-24-63

f. Hydrous Magnesium silicate ($H_4Mg_3Si_2O_9$)

COMPOSITION

COMPOUND	% BY WT. REF.	ELEMENT	% BY WT. REF. d	SPECTRO REF. e
H_2O[b]	4.32	Si	17.40	B-A
H_2O[c]	0.11	Mg	16.20	B-A
		Al	4.45	B
		Fe	7.80	A
		Ca	3.28	
		Ti+Misc	0.50	C
		V		E
		Cr		B-A
		Cu		E
		Ni		B-A
	Ignition	Loss	11.20	

SPECTRO SYMBOLS (PERCENT)

A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

SHIELDING AGGREGATE DATA

DATE	TABLE	SHIELDING AGGREGATE
Sept. 18, 1964	B.27	Shanton Magnetite

SPECIAL INFORMATION

CONTACT:

Richard L. Peterson
United States Aggregate Company
Douglas, Wyoming

PER 2000 Lb./Ton.

COST: All grades @ Bosler, Wyoming	$16.00
FREIGHT:	10.00
TOTAL: F.O.B. OAK RIDGE, TENN.[b]	$26.00

MINE DATA Jean Claims, Union Pacific Property Albany County, Wyoming

REMARKS:

a. ORNL Lab Test 9513 (8-12-64)
b. Costs furnished by Mr. Peterson by letter dated July 7, 1964
c. Aggregate gradations made on material from present stockpiles, Shanton Mine, Bosler, Wyoming

PHYSICAL AND CHEMICAL DATA

Sp. Gr. 4.664[a]

UNIT WT. (#/Ft.^{3}) C. F.

ABSORPTION =

GRADATION:[c] F. M. Fines 3.19

SIZE	% PASSING	SIZE	% PASSING
1 1/2	100.0	3/8	100.0
1	99.7	No.4	95.5
3/4	74.5	8	73.3
1/2	29.7	16	49.4
3/8	18.1	30	31.4
No.4	6.1	50	19.9
		100	11.5
		200	6.7

COMPOSITION

COMPOUND	% BY WT. REF.	ELEMENT	% BY WT. REF.	SPECTRO REF.
Fe_3O_4	72.17	Fe	52.3	
TiO_2	20.68	Ti	12.4	
V_2O_5	0.62	V	0.35	
SiO_2	2.78	Si	1.30	
Al_2O_3	3.59	Al	1.90	
MgO	0.51	Mg	0.31	
CaO	0.32	Ca	0.23	
S	0.01	S	0.01	
P	0.0013	P	0.0013	
		O	31.88	
H O	0.74			
H O	0.06			

SPECTRO SYMBOLS (PERCENT)
A - 10 - 100
B - 1 - 10
C - 0.1 - 1
D - 0.01 - 0.1
E - 0.001 - 0.1
F - 0.0001 - 0.001

UCN-5882
(3 6-64)

Appendix C

PYRITE DAMAGE TO CONCRETE

The iron pyrite content of the hydrous iron ore proposed for use in the concrete shields of the TRU cells was brought to the attention of the AEC by the Corps of Engineers at Troutdale, Oregon. The Corps stated that the pyrites could cause certain disintegration of concrete in a relatively short time (5 yrs).[1-4] The AEC sought assistance from several agencies on the pyrite problem, and other references to the problem were reviewed. The results of the short investigation are summarized below.

1. Chief Engineer, U. S. Bureau of Reclamation, Denver Federal Center, Lakewood, Colorado:[5]

   No problem with aggregates containing less than 1% pyrites (S = 0.54%).

   No experience with aggregates containing more than 1% pyrite.

2. Montana State University, Civil Engineering Department, Missoula, Montana:[6]

   Pyrite contributes to volume change in moist environments.

   Sulfur is destructive if convertible to water soluble sulfate.

   Experience limited to minor pyrite staining of normal aggregate concrete.

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[1]R. B. Somers, "Pre-Award Samples of Hydrous Iron Ore Aggregate, Oak Ridge P. O. OR-63-1203 and OR-63-1140, TRU Processing Plant," AEC Memo, April 5, 1963.

[2]Telegram to Corps of Engineers at Troutdale, Oregon from L. H. Jackson, AEC, April 9, 1963.

[3]Telegram to Treasure Belt Mining Company in Seattle, Washington from W. M. Taylor, AEC, April 15, 1963.

[4]Telegram to W. M. Taylor, AEC from D. F. Whittaker, April 16, 1963.

[5]Telegram to U. S. Bureau of Reclamation Denver Federal Center in Lakewood, Colorado from L. H. Jackson, AEC, April 25, 1963.

[6]Telegram to Montana School of Mines in Butte, Montana from L. H. Jackson, AEC, April 26, 1963.

3. Portland Cement Association, Research Department, 33 West Grand Avenue, Chicago 10, Illinois.[7]

Iron pyrites of sulfur in aggregates may have either directly or indirectly harmful effects on concrete.

Aggregate containing pyrite is not recommended.

Use Type IV or II cement if aggregate containing pyrite is used.

4. ACI, Volume 52, 1955-56, page 1000:

Pyrites in concrete may be harmful if concrete is exposed to moisture.

5. Proceedings American Concrete Institute, Volume 44, 1948, page 263:

Pyrites in concrete may be harmful if concrete is exposed to moisture.

6. Lea:[8]

Oxidation of pyrites or marcasite occasionally causes popping or spalling with some aggregates.

Pyrites occur in two forms, reactive and nonreactive, which are distinguishable by immersing the mineral in limewater. The reactive form produces brown precipitate, ferric hydroxide.

Ferric hydroxide and calcium sulphoaluminate have been found present in stained concrete.

Least severe condition of exposure occurs where structure does not set in flowing ground water.

7. Moum and Rosengvist:[9]

Concretes placed in shale formation containing small amounts of the unstable iron sulfide, pyrrhotite, are subjected to severe sulfate attack. Flowing water accelerates the concrete deterioration.

---

[7]Telegram to Portland Cement Association from L. H. Jackson, AEC, April 26, 1963.

[8]F. M. Lea, pp. 493, 558, and 588 in "The Chemistry of Cement and Concrete," St. Martins Press, Inc., New York.

[9]J. Moum and I. T. Rosengvist, "Sulfate Attack on Concrete in the Oslo Region," J. Am. Concrete Inst., 257-264 (September 1959).

8. Midgley:[10]

Mineral pyrite, FeS_2, causes brown staining of concrete made with Thames River gravels.

Not all of the pyrite is reactive.

Sulfate ions react with the calcium aluminates in the cement to form the mineral ettringite (calcium sulphoaluminate hydrate).

Two experimental samples containing nonreactive pyrite did not react after two years' exposure.

Experimental samples containing reactive form stained after only three months' exposure.

Limewater tests will identify the reactive type of pyrite.

9. Investigation of Types of Damage Caused by Pyritic Minerals:[11]

Aggregates containing pyrite and pyrrhotite were used in concrete beams.

Pyrite content ranged from 15 to 20%.

Total deterioration after about five years.

10. Material furnished by AEC:[12]

Attacks of sulfate water on concrete are recorded for several field cases of concrete failure.

Pyrites in the surrounding ground material and water cause concrete damage.

11. Report on Pyrite as Heavyweight Aggregate for Radiation Shielding:[13]

Iron Mountain massive iron pyrite aggregates have been used to produce shielding concrete weighing 230 lb/ft^3.

---

[10]H. G. Midgley, "The Staining of Concrete by Pyrite," Mag. Concrete Res. (August 1958).

[11]"Investigation of Types of Damage Caused by Pyrite Minerals," unreferenced page 783 provided by AEC.

[12]Unreferenced pp. 224-230 furnished by AEC

[13]Letter from H. S. Davis of Hanford Atomic Products Operation to H. L. McCracken of AEC at Oak Ridge, Tennessee dated September 6, 1963 with attached report, "Pyrite as Heavyweight Aggregate for Radiation Shielding," The Mountain Copper Co., 230 California Street, San Francisco 11, California.

Tests were performed on the aggregate for sodium sulfate soundness (ASTM-C88-61T), chemical reactivity (ASTM-C-289-57T - alkali reactivity), abrasive resistance (wetshot test), cobalt analysis, and gross beta-gamma activity.

Deemed suitable for use as an aggregate for concrete of below-normal background radiation.

Sulfur effects on concrete were not investigated.

On May 10, 1963, Mr. Harold S. Davis of General Electric, AEC, Hanford, Washington, provided the Corps of Engineers in Troutdale, Oregon with three concrete cylinders made with aggregates taken from the Running Wolf Mine at Stanford, Montana. These cylinders were cast in 1961. A chemical analysis of the aggregate showed a sulfur content of 1.02%. Additional observations are given below.

1. The limewater test on the aggregate containing pyrite produced no reaction.

2. The cylinders were cut and examined under the stereomicroscope at various magnifications and faint traces of reaction of the sulfides in the iron ore were discernible, no calcium sulfoaluminate deposits were found (see investigation items 6 and 8 above), and the concrete in cylinders was good.

3. A limewater test on marcasite, which is another pyrite-bearing material, produced a very positive reaction in five minutes.

Any conclusions pertaining to the ore from the Running Wolf Mine that are based on the preceding investigation must be drawn at the discretion of the reader. Qualified experimental data concerning the detrimental effects of sulfur in the ore from the Running Wolf Mine on concrete are not available.

Appendix D

LABORATORY TESTING PROCEDURES

The laboratory testing procedures used to determine various physical and chemical properties of shielding aggregates are listed below.

1. Physical properties,

Mechanical gradation	ASTM C-136-61T and ASTM C-3361T
Bulk sp gr (SSD)	ASTM C-127 and 128-59
Absorption	ASTM C-127 and 128-59
Material finer than No. 200	ASTM C-117-61T
Test for unit weight of aggregate	ASTM C-29-60
L.A. abrasion, grading A	ASTM C-131-55

2. Chemical properties,

Sulfur	Method No. 1-218180, LECO Combustion Photometric Titration Method. Oak Ridge National Laboratory Master Analytical Manual*, TID-7015, Supplement 3.
Iron	Method No. 1-214170, Volumetric Di Chromate Method. Oak Ridge National Laboratory Master Analytical Manual*, TID-7015, Section 1.
Silica	Method No. 1-217750, Gravimetric Acid Dehydration-Hydro Fluorination Method. Oak Ridge National Laboratory Master Analytical Manual*, TID-7015, Section 1.

The Train Method is used to determine the combined water in iron ore and serpentine samples. The weighed samples are heated in a dry nitrogen atmosphere at a temperature of 850°C in a tube furnace. The water evolved at this temperature is swept into a Nesbitt tower containing anhydrone

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*Available from U.S. Department of Commerce, Office of Technical Services, Washington, D. C. 20230.

(magnesium perchlorate), and the gain in weight is calculated as percent water. The amount of free water, as indicated by the loss at a temperature of 105°C, is subtracted, and the difference in weight is reported as combined water.

The combustion train apparatus consists of a cylinder of dry nitrogen, a gas drying tower, a vycor combustion tube heated by a tube furnace 1 in. in diameter and 12 in. long, a Nesbitt tower filled with anhydrone, and a sulfuric acid bubble tower.

The Train Method procedure is given below.

1. Nitrogen is passed through the combustion apparatus, and the Nesbitt tower is weighed at 30-min intervals until the gain in weight is less than 0.5 mg/hr, hereafter referred to as the "Blank".

2. A sample is ground to pass through a No. 100 mesh sieve.

3. A 1-gram sample is weighed into a preignited porcelain boat, and the boat and sample are quickly placed in the combustion tube.

4. Dry nitrogen is passed over the sample while it is heated at a temperature of 850°C for 90 min.

5. The Nesbitt tower is disconnected and weighed.

6. The tower is replaced, and the sample is again swept with nitrogen for 15 min; the tower is then reweighed.

7. Step 6 is repeated until the gain in weight is approximately equal to the blank.

8. The percent combined water is calculated by using the formula

$$\frac{(W-B) \times 100}{S} = \text{percent water*},$$

where

W = total gain in weight of tower, grams,

B = gain in weight of tower without sample from step 1 (Blank), grams,

S = weight of sample, grams.

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*If the original sample is analyzed, the percent combined water is calculated by subtracting the percent free water from the percent total water as determined above. If the sample has been dried at 105°C before starting the above procedure, the percent water as calculated by the above equation represents the combined water.

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