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BDX-613-2415

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Density Separation of Boron Particles

By R. M. Smith

Published April 1980

Final Report

Prepared for the United States Department of Energy
Under Contract Number DE-AC04-76-DP00613.

**Kansas City
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Printed in the United States of America

Available From the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161.

Price:	Microfiche	\$3.00
	Paper Copy	\$4.50

DENSITY SEPARATION OF BORON PARTICLES

By R. M. Smith

Published April 1980

Final Report
W. L. Hudson, Project Leader

Project Team:

C. J. Cook
W. R. Davis
L. L. Lantz
R. M. Smith

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DENSITY SEPARATION OF BORON PARTICLES

BDX-613-2415, Final Report, Published April 1980

Prepared by R. M. Smith

A technique has been developed to isolate boron particles in narrow density ranges. This separation process allows removing particles with gross amounts of impurities to improve overall purity of the remaining material. The developed process probably can be used to separate boron with alpha and beta rhombohedral crystal structures as well; however, additional testing is required to confirm this possibility.

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The Bendix Corporation
Kansas City Division
P. O. Box 1159
Kansas City, Missouri 64141

A prime contractor with the United States
Department of Energy under Contract Number
DE-AC04-76-DP00613

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SUMMARY

A density distribution much broader than expected was observed in lots of natural boron powder supplied by two different sources. The material in both lots was found to have a rhombohedral crystal structure, and the only other parameters which seemed to account for such a distribution were impurities within the crystal structure and varying isotopic ratios. A separation technique was established to isolate boron particles in narrow density ranges. The isolated fractions were subsequently analyzed for B^{10} and total boron content in an effort to determine whether selective isotopic enrichment and nonhomogeneous impurity distribution were the causes for the broad density distribution of the boron powders.

It was found that although the B^{10} content remained nearly constant around 18 percent, the total boron content varied from 37.5 to 98.7 percent. One of the lots also was found to contain an apparently high level of alpha rhombohedral boron which broadened the density distribution considerably. During this work, a capability for removing boron particles containing gross amounts of impurities and, thereby, improving the overall purity of the remaining material was developed. In addition, the separation technique used in this study apparently isolated particles with alpha and beta rhombohedral crystal structures, although the only supporting evidence is density data.

DISCUSSION

SCOPE AND PURPOSE

The purpose of this study was to determine if there is selective isotopic enrichment in crystalline natural boron and if there is a nonhomogeneous distribution of impurities within the crystalline material. The first area is appealing because, if the separation of particles enriched in the B^{10} isotope could be accomplished at sufficiently low cost, a significant cost savings associated with the procurement of enriched boron could be implemented. Also, separation of boron particles containing gross impurities would increase the overall purity of the remaining material.

ACTIVITY

To investigate the possibility of selective isotopic enrichment or nonhomogeneous impurity distribution within boron particles, selection of a technique to separate the boron particles into fractions with narrow density ranges was necessary. The technique selected involves the use of high density liquids and mixtures of these liquids to obtain the desired densities for separation.

This technique has been shown to be effective in separating natural boron, with a density of 2.35 g/cm^3 , from enriched boron containing 90 percent B^{10} , with a density of 2.19 g/cm^3 . The method also was used to separate low density contaminants from the enriched boron.

The first step in the procedure involves loading enriched boron contaminated with low density material and natural boron into a pear-shaped separatory flask and pumping bromochloromethane into the funnel with a variable speed peristaltic pump. Bromochloromethane was chosen because its density (1.99 g/cm^3) is such that any material less dense than enriched boron would be floated away. Figure 1 shows this type of material floating on the surface of the low density liquid.

After all low density material has been floated, iodomethane is added to the funnel, which forces the low density liquid and trash into a cylindrical separatory flask (Figure 2). The bromochloromethane can then be drained back into its reservoir for reuse. The boron then is agitated thoroughly in the iodomethane, and high-density impurities including natural boron are allowed to settle to the bottom of the pear-shaped separatory funnel (Figure 3). The natural boron is drawn off the bottom, and the iodomethane is filtered back into its reservoir.

Figure 1. Density Separation Apparatus
Containing Bromochloromethane
and Boron

Using the same liquids, this technique also has been successful in removing high density particles in virgin boron powder. The radiographic requirement limits the number of high density inclusions to a total of 20 in the size range of 0.38 to 0.99 mm in each kilogram of boron powder; no foreign particles larger than 1.02 mm are permissible. The first attempt to remove foreign particles from virgin boron powder by density separation used boron which had been previously pyrolyzed to remove organic

Figure 2. Density Separation Apparatus
Containing Bromochloromethane,
Boron, and Iodomethane

materials, screened, and then leached in hydrofluoric acid. Progress in removing the foreign particles by screening and leaching is shown in Table 1.

As shown by these data, leaching was quite effective in removing the smaller particles, but actually increased the number of large particles somewhat. To provide a worst case test of the density separation technique, bag 5 of the leached material was selected.

Figure 3. Removal of Natural Boron Contaminant From Enriched Boron Powder.

A total of 967.0 g of boron powder was treated in four equal fractions and then recombined. A total of 10.7 g of material was removed during the process, and the remaining 956.3 g of boron

Table 1. Foreign Particle Removal by Screening and Leaching

Bag	After Screening*		After Leaching*	
	+1.02 mm	0.38 to 0.99 mm	+1.02 mm	0.38 to 0.99 mm
1	3	125+	6	9
2	7	125+	5	10
3	5	125+	6	7
4	5	125+	1	12
5	5	125+	14	9
Total	25	625+	32	47

*Values given are the number of high-density particles larger than 1.02 mm and the number between 0.38 and 0.99 mm.

was X-rayed again. The density separation technique had removed all high density particles in both categories.

In another test, the remaining four bags of leached material were screened to concentrate the foreign particles in the coarse fraction (+40 mesh). A total of 521.0 g of this coarse material was processed, and all but two of the particles in the small particle size range were removed. These tests proved that the density separation technique could be used successfully to remove high density particles from virgin boron powder.

In later attempts to process more material, problems arose with the apparatus. As the equipment was used more, the abrasive nature of the boron began chipping the glassware and introducing glass fragments into the boron. The limited work accomplished was helpful; however, more development work on the apparatus would have permitted the entire 173 kg of contaminated virgin boron powder to be processed in this manner.

Although the difference in densities between the natural and enriched boron was only 0.16 g/cm^3 , the selection of the proper liquid made separation of the two materials a simple task, especially when the particles were larger than about $40 \text{ }\mu\text{m}$. However, when trying to separate fractions of natural boron whose density differences are an order of magnitude less, selection or preparation of the correct liquid is more critical.

A list of halogenated hydrocarbon liquids being considered for this study was supplied to the Bendix Environmental Protection Department for evaluation of toxicity and flammability. All potential materials were found to be moderately to highly toxic, but were nonflammable (Table 2). From the 11 possible materials, the first five listed in Table 2 were selected on the basis of density, cost, polarity, and safety.

Two techniques were investigated for obtaining very precise densities over a given range: using a single liquid and varying its density by changing the temperature of the liquid, and mixing liquids of different densities to obtain the desired intermediate density. The first method has the advantage of using one liquid throughout, which reduces the handling and toxicity problems but requires very precise and stable temperature control. The second method permits operation over a broader density range while still using only two liquids for each desired density; however, during agitation, unequal volatilization of the two-component solutions can noticeably change the density.

An environmental chamber was acquired to provide the necessary temperature control for density adjustment. This instrument was designed to operate between -73 and 315°C . The density/temperature relationship was measured for all five liquids, and the data are given in Table 3 and Figure 4.

These liquids exhibited excellent linearity over the temperature ranges observed; however, there appears to be an error in the material purchased as 1,2-dibromoethane. This material exhibits a density very nearly that of dibromomethane at all temperatures. At 20°C , the density of dibromomethane was measured at 2.491 g/cm^3 , while the density of 1,2-dibromoethane was found to be 2.470 g/cm^3 . The literature values for these two materials are 2.4921 and 2.180 g/cm^3 , respectively. In addition, the slope of the density/temperature curves from Figure 4 are almost identical at 2.58×10^{-3} and $2.57 \times 10^{-3}\text{ g/cm}^3\cdot^{\circ}\text{C}^{-1}$ respectively. From these data, it can be concluded that the material was actually dibromomethane. The differences in measured and literature density values probably are caused by varying degrees of photodecomposition, as all liquids required filtering to remove brown solids.

Density Distribution of Boron Powders

To determine the density distribution of the virgin natural boron powders, two density gradient columns were prepared by carefully mixing dibromomethane and bromochloromethane. The linearity was checked with accurate density floats. Figure 5 represents the density gradients in the two columns, and Figure 6 shows photographs of the columns with approximately 2 g of boron in each. The first column contained boron supplied by vendor 1 and the second contained boron from vendor 2. All boron was in the particle size range of 250 to $105\text{ }\mu\text{m}$ (-60 , $+140$ mesh).

Table 2. High Density Liquids

Material	Density (g/cm ³)	Toxicity	Flammability	Approximate Cost (\$/kg)
Bromochloromethane	1.991 at 19°C	High	Nonflammable	6
1,2-Dibromoethane	2.180 at 20°C	High	Nonflammable	5
Iodomethane	2.28 at 20°C	High*	Nonflammable	35
Dibromomethane	2.4921	Slight	Nonflammable	10
1,1,2,2-Tetrabromoethane	2.9672 at 20°C	High	Nonflammable**	5
Tetrachloromethane	1.5942 at 20°C	Poisonous	Nonflammable	1
Tribromomethane	2.8899 at 20°C	High	Nonflammable	20
Diiodomethane	3.3254 at 20°C	Irritant*	No Information	50
cis-Dibromoethene	2.2464 at 20°C	Moderate	No Information	6
Tetrachloroethene	1.623 at 20°C	High	Nonflammable	5
1,1,2-Trichloro- 1,2,2-trifluoroethane	1.5635 at 20°C	High	Nonflammable	5

*Also produces narcotic effects.
 **Decomposes 65°C below its boiling point, liberating flammable and highly toxic vapors.

Table 3. Liquid Densities as a Function of Temperature

Temperature (°C)	Density*(g/cm ³)				
	1	2	3	4	5
-50			2.451	2.668	
-40			2.424	2.644	
-30			2.401	2.621	
-20			2.369	2.592	
-10	1.995	2.550	2.340	2.564	
0	1.971	2.525	2.311	2.537	
10	1.950	2.501	2.286	2.514	2.971
20	1.929	2.470	2.254**	2.486**	2.945**
30	1.905	2.442		2.467	2.926
40	1.887	2.417		2.438	2.905
50	1.856	2.391		2.410	2.879
60	1.834	2.366		2.385	2.859
70		2.338		2.359	2.838
80		2.318		2.332	2.816
90		2.293			
Measured	1.930 at 19°C	2.470 at 20°C	2.260 at 20°C	2.491 at 20°C	2.950 at 20°C
Literature ¹	1.991 at 19°C	2.180 at 20°C	2.28 at 20°C	2.4921 at 20°C	2.9672 at 20°C

*Material identification: 1 is Bromochloromethane; 2 is 1,2-Dibromoethane (identified by density data as dibromomethane); 3 is Iodomethane; 4 is Dibromomethane; and 5 is 1,1,2,2-Tetrabromoethane.

**Measured at 21°C.

The broad distribution of boron in column 1 was interpreted as being caused by the boron particles having different isotopic ratios or varying amounts of impurities which would affect the density of the particles. Several densities were selected for the separation and isolation of boron particles, based on the distribution of boron in column 1.

Prior to the preparation of column 1, it was assumed the boron would have a fairly narrow distribution around a density of 2.35 g/cm³; however, the actual distribution was much broader and was skewed toward the higher densities. Various points on column 1 were selected as fractions to be isolated and the points were converted to densities by using the calibration curve in Figure 5. The densities selected are given in Table 4.

Figure 4. Liquid Density as a Function of Temperature

Apparatus similar to that shown in Figure 1 was assembled and used in the fractionization of the two boron lots. The basic procedure was the same as in the previous work involving the separation of natural and enriched boron, that is, addition of the boron to the separatory funnel, pumping in the liquid of the desired density, thorough agitation with air bubbling, settling of the material into two fractions, collection of the denser fraction by drawing off the material which sank and filtering through a filter paper, and collection of the less dense fraction by a similar filtration.

The density ranges were selected so that the middle fractions would contain the most material; therefore, the first cut was made at the middle fraction, and each half was processed individually. The material from vendor 2 was processed through the

Figure 5. Density Gradient Calibration for Bromochloromethane and Dibromomethane

entire scheme in duplicate (Runs A and B), and the six fractions were analyzed for total boron and B^{10} content. These data are listed in Table 5.

Two major conclusions can be drawn from the data in Table 5: there are definite density ranges in which the higher purity boron fractions can be collected, and the density distribution exhibited by this material was not caused by selective isotopic enrichment. In the case of the total boron values, reasonably good agreement was found between the two separate runs. Differences between the two samples at each density are consistently less than the differences between values at different densities. As the data indicate, boron powders with a purity as high as 97.8 percent and as low as 88.6 percent were separated from virgin boron powder with an average total boron content of 93.0 percent prior to separation.

**COLUMN 1
VENDOR 1
NATURAL
BORON**

**COLUMN 1
VENDOR 2
NATURAL
BORON**

Figure 6. Natural Boron in Density Gradient Columns Using Mixtures of Bromochloromethane and Dibromoethane

Table 4. Density Selection Data

Area	Height on Column 1 (cm)	Density (g/cm ³)	Density Range (g/cm ³)
1	67.7	2.341	
2	71.4	2.356	0.015
3	74.2	2.367	0.011
4	82.6	2.400	0.033
5	99.8	2.470	0.070

Table 5. Separation of Vendor 2 Natural Boron

Sample	Density Range (g/cm ³)	Boron Weight (g)	Boron* (Percent)	
			Total	B ¹⁰
1A	<u><2.341</u>	3.782	91.2	18.40
1B		3.550	97.7	18.38
2A	2.341 to 2.356	22.376	96.7	18.17
2B		13.146	97.8	18.40
3A	2.356 to 2.372	5.243	92.3	18.22
3B		9.300	93.2	18.42
4A	2.372 to 2.400	5.857	95.8	18.08
4B		5.010	95.1	18.28
5A	2.400 to 2.470	3.117	90.3	18.31
5B		5.184	88.6	19.41
6A	<u>≥2.470</u>	0.324	**	18.04
6B		0.257	**	18.06
Virgin Powder			93.0	18.04

*Both analyses have a relative precision of ± 0.1 percent.

**Insufficient sample.

In an effort to determine why the purity varied with density, samples 2A, 3A and 4A were analyzed for the major impurities present in the virgin powder, such as aluminum, calcium, copper, iron, magnesium, manganese, phosphorous, and silicon. The first six elements were analyzed by atomic absorption spectroscopy while the concentrations of the latter two were determined by X-ray fluorescence. The purity data and the relative percent error associated with each analysis are given in Table 6.

The data in Table 6 are of little help in explaining the relationship between density and total boron content. Although some of the minor elements (less than 100 ppm) found in the virgin powder may have been highly concentrated in certain fractions, this seems doubtful; none of the major elements showed any dramatic changes in concentration after separation. In fact, the two samples with the highest total boron content (2A and 4A) have slightly higher impurities in the elements measured than either the virgin powder or sample 3A. The overall differences in impurities between the four samples are probably within the limits of experimental error.

It was stated earlier that the density distribution of the vendor 2 material was not caused by selective enrichment; however, these data do not imply that such enrichment is not present, either. Considering the precision of the test method (± 0.03 percent), there are obviously fractions which have slightly higher B^{10} content than others, but these subtle differences exhibit no discernible pattern and certainly could not account for the broad density distribution observed in this study.

Natural boron obtained from vendor 1 was separated by the same technique and using the same liquid densities for fractionization. Although the separation was again run in duplicate, only the A samples were analyzed for total boron and B^{10} content. These data are given in Table 7.

Although the distribution of the separated boron is different, which would be expected from Figure 6, the B^{10} and total boron values of the vendor 1 material indicate the same characteristics as did the vendor 2 material. Here again, the B^{10} content is nearly constant, and the total boron values show definite density ranges in which the most pure material can be collected.

The most striking data in Table 7 are the total boron values for sample 5A and its duplicate 5B. These values of 39.3 and 37.5 percent, respectively, indicate there is good agreement between duplicate runs just as there was in the duplicate runs of the vendor 2 material (Tables 5 and 2). Even more important, these values prove that separation of grossly contaminated material by density fractionization can be accomplished. Table 8 provides purity data on selected fractions of vendor 1 material. Although

Table 6. Isotopic and Purity Data of Vendor 2 Natural Boron

Component	Relative Error (Percent)	Virgin Powder	Sample		
			2A	3A	4A
Total Boron (Percent)	± 0.1	93.0	96.7	92.3	95.8
B ¹⁰ (Percent)	± 0.1	18.04	18.17	18.22	18.08
Al (ppm)	±10	515	565	467	580
Ca (ppm)	± 5	133	220	252	228
Cu (ppm)	± 5	130	113	91	112
Fe (ppm)	± 5	1685	1677	2431	2092
Mg (ppm)	± 2	21800	22520	20567	23062
Mn (ppm)	± 5	329	108	87	106
P (ppm)	±10	125	58	118	90
Si (ppm)	±10	1045	773	697	677

Table 7. Separation of Vendor 1 Natural Boron

Sample	Density Range (g/cm ³)	Boron Weight (g)	Boron (Percent)	
			Total	B ¹⁰
1A	<2.341	4.400	88.3	18.41
2A	2.341 to 2.356	1.837	98.7	14.48
3A	2.356 to 2.372	0.714	95.1	18.44
4A	2.372 to 2.400	10.450	94.0	18.48
5A	2.400 to 2.470	0.667	39.3	18.51
5B			37.5	
6A	>2.470	16.359	92.4	18.49
Virgin Powder			94.8	18.24

Table 8. Purity Data of Vendor 1 Natural Boron.

Element	Sample			
	1A	2A	3B	5B
Total Boron (Percent)	88.3	98.7	*	37.5
Fe (ppm)	1336	2495	2651	219,000 (21.9 percent)
Ni (ppm)	82	229	286	19,800 (1.98 percent)
Cr (ppm)	80	135	209	17,000 (1.70 percent)
Mn (ppm)	42		237	2,700 (0.27 percent)
Mg (ppm)	220	663	422	241
Ca (ppm)	164	171		154

*Insufficient sample.

the limited data do not explain the differences between samples 1A, 2A, and 3B (assuming the total boron content of 3B is similar to the 95.1-percent value of 3A), the differences between these three and sample 5B are obvious. The extremely high levels of impurities in this sample help explain the very low total boron content.

As in nearly all the samples analyzed in this study, the sum of the impurities and the total boron content do not equal 100 percent (sample 5B is the worst example). However, in the case of sample 5B, the chemistry involved in dissolution of the sample would indicate that the majority of the missing impurities is probably silicon.

Another interesting aspect of the vendor 1 natural boron was the distribution over a broad density range and, more specifically, that 47.5 percent of the material collected has a density equal to or greater than 2.47 g/cm³. This result was particularly puzzling, because the density for beta rhombohedral boron ranges from 2.168 g/cm³ for pure B¹⁰ to 2.406 g/cm³ for pure B¹¹. A contact with the vendor provided the probable answer. The vendor 1

material used in this study was a mixture of alpha and beta rhombohedral natural boron. Although the percentages were not available, it appears from the data in Table 7 that about half was probably of the alpha crystal structure; the density of natural boron with that structure is 2.47 g/cm^3 , and the density increases to 2.51 g/cm^3 for pure B11.²

In an effort to further characterize the material in the separated fractions, scanning electron photomicrographs were obtained. The views given in Figure 7 are typical of all the fractions at magnifications of 50 and 150X, respectively. These photographs illustrate the general appearance typical of virgin boron particles: irregular shapes and usually sharp edges. Figures 8 through 12 are photographs of contaminants collected with fractions 1A, 2A, 3A, 4B, and 6A, respectively. Analysis of those particular areas by energy dispersive spectrometry indicated the major elements were iron and chlorine; aluminum, nickel, and chromium also were present at lower levels. The main exception was sample 1A. The particle analyzed in that sample contained primarily potassium and calcium, with lesser amounts of aluminum, phosphorous, sulfur, and iron. Even though the particle could not be identified further, it is worth noting that its principle constituents were the lighter elements, which corresponds with that sample having the lowest density fraction.

Isotope Segregation

Because the natural boron used in this study showed no significant evidence of selective isotopic enrichment and an established method of separating enriched boron from natural boron was available, the next step was to try to generate conditions which would promote segregation of the isotopes. The possibility that the two isotopes might have slightly different melting points presented a potential means of accomplishing isotopic enrichment. This is based on the assumption that a sample of boron could be maintained in a molten state, then very slowly cooled to allow the isotope with the highest melting point to crystallize first. Upon further cooling, the other isotope also would crystallize, generating localized areas of the segregated isotopes within the solid sample. This solidified material then would be crushed to yield particles in the 50-to 250- μm range, which lends itself well to the density separation technique. Complete separation of the isotopes on a first time basis is idealistic; therefore, the process would involve recycling through the melting, crushing, and separation cycle.

Although boron melting at Bendix has been investigated for other purposes,³ the equipment used in those projects is no longer available, and other sources were sought. Three different operations were considered: an electron beam welder, a heliarc welder, and a plasma spray torch. As the ultimate goal was a molten pool

0.4 mm

0.2 mm

Figure 7. SEM Photomicrographs of Vendor 1 Boron Sample 1A

Figure 8. SEM Photomicrograph and Energy Dispersive X-Ray Spectrum of Foreign Particle in Vendor 1 Sample 1A

of boron, the plasma spray technique was eliminated as a possibility.

In the first attempt, an electron beam welder, with a power output at 60 kV of 500 mA and operating at vacuums greater than 13 mPa, was used. The natural boron was loaded into a circular graphite crucible 10.2 cm ID by 5.7 cm deep and a 1.3 cm wall and bottom thickness.

Melting the boron was not difficult when the beam was focused on a small (less than about 0.75 cm) portion of the material. However, maintaining even a small pool of molten boron was difficult because of the tendency to "erupt" and spray the material around the chamber. The technique of defocusing the beam was tried in two variations, so that the focal point was either above or below the surface of the boron (Figure 13). The best results were obtained by focusing the beam beneath the surface and increasing the power of the beam until the entire surface material began to melt.

Figure 9. SEM Photomicrograph and Energy Dispersive X-Ray Spectrum of Foreign Particle in Vendor 1 Sample 2A

Because the powder in the crucible was very loose, the melted portion required less volume and began to sink into the bed of powder. As this occurred, the melt would approach the focal point of the beam and, therefore, be heated even more until it eventually would erupt. Once the initial surface melting began, this sequence proceeded very quickly so that vertical positioning of the focal point to maintain the melt was impossible. The largest pieces of melted boron obtained in the electron beam are shown in Figure 14. The piece on the left is actually a top view of the largest piece, which is about 32 mm in diameter and 38 mm deep. The hollow areas can be seen easily, indicating the penetration depth of the beam. The piece on the right shows the vertical channels created during melting. Obviously, neither piece exhibits the desired "molten pool" characteristic.

A heliarc welder was tried next. The welder was equipped with a heavy duty torch and was operated between 200 and 225 A. This technique was more successful in providing a pool of molten

Figure 10. SEM Photomicrograph of
Vendor 1 Sample 3A

boron, but the total depth of the melted portion was only about 1.5 mm (Figure 15). Because of this and the difficulty in maintaining an arc, this technique was abandoned also.

ACCOMPLISHMENTS

Grossly contaminated boron particles can be removed by a developed density separation technique, thereby improving the overall purity of the remaining material. This technique probably will apply to the separation of boron with alpha and beta rhombohedral crystal structures, although additional data are needed to verify this.

No indication of significant selective isotopic enrichment was found in either of the lots of natural boron used; however, if future work indicates that such enrichment could be generated,

Figure 11. SEM Photomicrograph
of Vendor 1 Sample 4B

Figure 12. SEM Photomicrograph
of Vendor 1 Sample 6A

FOCAL POINT ABOVE SURFACE

FOCAL POINT BELOW SURFACE

Figure 13. Electron Beam Focal Points

the powders of varying isotope contents could be separated at nearly any isotopic ratio by the technique developed in this study.

FUTURE WORK

Although the separation of natural boron from enriched boron or the removal of low purity material from either pure natural or enriched boron is significant, the real potential in this work is in the area of isotopic enrichment. Two prime areas for future work would be development and feasibility studies on the process of laser-induced boron isotope separation⁴⁻⁶ and the separation of high boron content, low molecular weight compounds based on density differential.

Figure 14. Boron Melted by Electron Beam Welder

Figure 15. Boron Melted by Heliarc Welder

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BDX-613-2415

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Smith, Final, April 1980

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SPECIAL PROJECTS: Boron Fractionization

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