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# The Boron Trifluoride/Nitromethane Ratio of the $BF_3 \cdot CH_3NO_2$ Adduct

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## ABSTRACT

In recent years the boron trifluoride–nitromethane adduct has received attention in a number of different fields. It functions as a catalyst in cyclization reactions, in substitutional reactions involving the cracking of ring compounds, and in the creation of stereospecifically pure enantiomers. It has also been suggested as an effective means of separating the isotopes of boron. In all of the research involving this adduct, the ratio of $BF_3$ to nitromethane has been assumed to be somewhere in the range of 1.4/1 to 1.6/1, as reported by Herbst. However, the present study has found the ratio to be significantly lower, 0.158/1. This ratio was obtained by reacting dilute solutions of the adduct with water and titrating the resulting hydrofluoric acid. Gravimetric analysis obtained a very similar result. An independent confirmation was made by the Eagle Picher Boron Laboratory where the adduct was titrated for total boron, and the same ratio was found. A pilot plant was constructed to investigate commercial use of the adduct, and the ratio of 0.158/1 was also confirmed there. Based on this ratio, the heat of association was calculated to be $-8.51$ kJ/mol. The specific heat was measured to be 0.52 cal/g°C, and the room temperature density was determined to be 1.31 $g/cm^3$.

## INTRODUCTION

Harring and Livinghouse published a series of three papers describing the use of $BF_3 \cdot CH_3NO_2$ as a catalyst in cyclization reactions. The first, published

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in 1992, described the use of this adduct in preparing annulations of trienoate derivatives (1), and the second, published the same year, described using the adduct in the preparation of (±)-taxodione (2). The third article, published in 1994, was a follow-up of the second, describing the use of $BF_3{\cdot}CH_3NO_2$ in preparing (±)-taxodione (3). All three papers were based on Harring's 1992 PhD dissertation (4) from the University of Minnesota. Solutions of gaseous $BF_3$ in nitromethane had proven effective as catalysts for sulfenylative and selenenylative cationic cyclizations, and Harring was interested in using the same material for "$H^+$ initiated cascade cyclizations" (3). It was found to be extremely effective, yielding a 95% pure sample of (±)-taxodione in 1 hour, significantly more efficient than any of the other catalysts tested.

Other groups have also published works describing the effectiveness of various $BF_3$ adducts in promoting cyclization reactions (5–7). These adducts have been shown to be useful in stereoselective synthesis (8–10). $BF_3$ adducts are also effective catalysts in opening ring structures, allowing substitutions or the creation of linear compounds (11–13). Other studies were conducted to deduce the structure of various $BF_3$/donor complexes (14, 15). Most of the structural information came from FTIR studies (16–18), and research is still being conducted in this area.

At the same time that $BF_3$ was being studied as a catalyst, the nitromethane–boron trifluoride system was being studied in another area of research—isotopic separation. Boron-10 has a neutron capture cross section of 3837 barns, 20 times that of lead and more than 767,000 times that of boron-11, making it a valuable shielding material in the nuclear industry. Isotopic separation can be achieved by a reaction between gaseous boron trifluoride and a liquid adduct of $BF_3$ with one of several organic donor compounds. The net reaction is

$$n{}^{10}BF_{3(g)} + ({}^{11}BF_3)_n{\cdot}CH_3NO_{2(l)} = n{}^{11}BF_{3(g)} + ({}^{10}BF_3)_n{\cdot}CH_3NO_{2(l)}$$

The isotopic separation factor, $\alpha$, is simply the equilibrium constant of this equation, and typically varies from 1 to 1.04. Ever since the Manhattan Project there has been ongoing research to discover more efficient adducts to use in this reaction. Palko conducted a series of experiments with a wide variety of donors, including anisole (19), *n*-butyl sulfide (20), methyl selenide (21), ethyl ether (22), dimethyl ether (23), and dimethyl sulfide (23). Results from these experiments are summarized in a comprehensive paper published in 1969 (24).

More recently, Herbst and McCandless (25) published a paper describing several new donor possibilities, including nitromethane. This paper was based on Herbst's Ph.D. thesis (26) from Montana State University. They found the nitromethane adduct to be extraordinarily effective at isotopic separation and unlike anything previously tested. In fact, Herbst found the isotopic separation factor, α, for this adduct exceeded the theoretical maximum. Figure 1 shows α as a function of reciprocal temperature for several of the adducts tested, as well as the theoretical maximum calculated from statistical partition functions. After this unusual property was discovered, a series of experiments was begun to explain what made this adduct so unique.

One of the most interesting results from these experiments pertains to the $BF_3$/donor ratio. All of the adducts investigated by Palko had a ratio of approximately 1/1, and all of the adducts in Herbst's experiments were also assumed to be 1/1. Even Harring's work with the adduct as a catalyst referred to it as $BF_3 \cdot CH_3NO_2$, a 1/1 ratio. It was known that some of the adducts varied slightly from a perfect 1/1 ratio, but the variations were small enough to be negligible in most calculations. Most publications continued to refer to the adducts simply as 1/1; for example, see Nxumalo and Ford (18). However, in a private communication to Herbst, Harring reported finding a ratio for the nitromethane adduct between 1.4/1 and 1.6/1, significantly larger than expected, and too large a variation to be overlooked. In his thesis, Herbst wrote, "One mole of nitromethane was reported to absorb 1.4 to 1.6 moles of $BF_3$ gas at 0°C." He further stated that, "These properties were confirmed in this laboratory. . . ." (26). He speculated that the excess $BF_3$, beyond that necessary for a 1/1 ratio, was weakly bonding with the second oxygen available on nitromethane. All of the other adducts studied were single site donors, but nitromethane had two sites available. Herbst believed this explained why it can

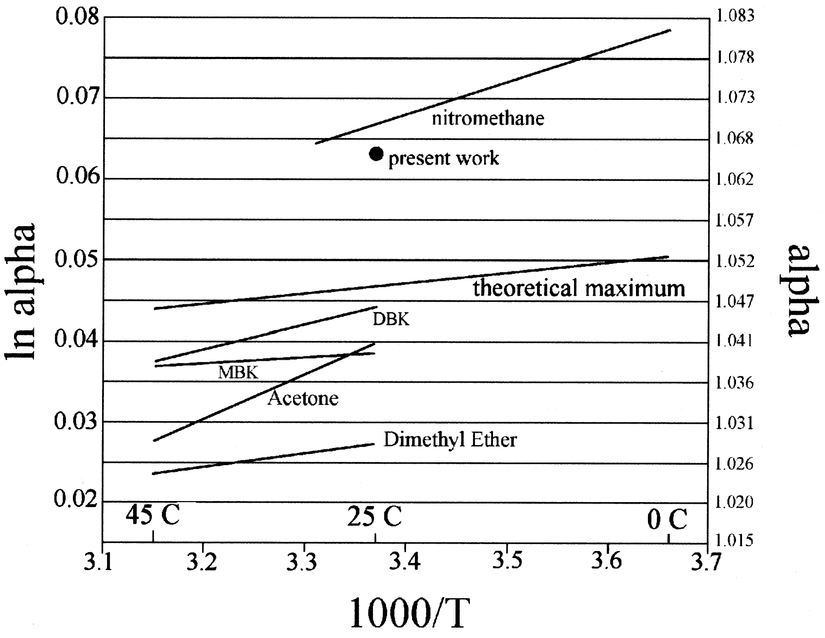

FIG. 1 Separation coefficient vs reciprocal temperature, after Herbst (26).

exceed the theoretical maximum for single site donors. Other than this unique report, very little was known about the properties of this potentially valuable adduct.

The present investigation was conducted in order to measure such basic properties as the heat capacity, the density, the heat of association, the heat of formation, and the temperature range of stability. These values are required for the engineering design of an isotopic separation plant in sizing the heat transfer and mass transfer equipment. The primary objective, however, was a conclusive determination of the $BF_3$/nitromethane ratio. This ratio was necessary to determine the heat of association, and is a key to understanding what makes the adduct so effective as a catalyst and an isotopic separator. Moreover, the molar ratio is important because in an isotopic separation plant the size of the gas–liquid contact equipment is inversely related to the concentration of the boron in the complex. Any gain in the reduction of the column length resulting from the increased separation factor could be negated by the need for an increased column diameter from the decreased boron content of the adduct.

## MATERIALS

The $BF_3$ used in all experiments was purchased from the Matheson Company and was a minimum of 99.99% pure. The nitromethane was HPLC grade, a minimum of 96% pure. The boron trifluoride diethyletherate was CP grade, ≤96% pure. The diethyl ether was HPLC solvent grade, 99.9% pure. All three of these were purchased from Sigma Aldrich. The boron trifluoride·nitromethane adduct was synthesized in the lab, as described hereafter.

## METHODS

### Reaction Cell

Because of the rapid reaction of $BF_3$ with water vapor, all the procedures described henceforth occurred inside of a dry box with ambient water vapor maintained at ≤20 ppm. The adduct was formed by reacting $BF_3$ gas with nitromethane inside of a custom-made stainless-steel reaction cell, following Herbst (25). The lid of the cell contained an inlet port for the $BF_3$, a pressure sensor that allowed the internal pressure to be monitored, and a valved outlet port which allowed samples of gas to be taken from the cell. A Teflon O-ring sealed the lid tightly to the main body of the cell. Liquid was drawn from a port at the bottom of the cell and circulated via Teflon tubing through a peristaltic pump and returned to a sparger port at the top of the cell, above the level of the liquid. This allowed more of the liquid to come in contact with the gas, allowing equilibrium to be established much faster. A threeway valve at the lower port allowed liquid samples to be taken without losing pressure in the cell. Copper coils welded to the outside of the cell allowed liquid nitrogen or ice water to be circulated without contaminating the dry box, permitting samples of the adduct to be created below room temperature. Samples above room temperature were created by simply placing the entire cell on a hot plate inside of the dry box. Whenever a sample was prepared above or below room temperature, the fact is noted in the procedures for that particular experiment.

## Density

The first step in characterizing the adduct was a simple determination of its density. Volumetric vials, precise to 0.01 mL, filled with the adduct were weighed on a digital balance, precise to 0.001 g. Before measuring the density of the adduct, the accuracy of the system was verified using distilled water as a reference; the density of the water was measured accurately to within 0.003 g/cm$^3$. Three separate measurements of the adduct yielded an average density of 1.31 g/cm$^3$, a 15% increase from the density of pure nitromethane (1.137 g/cm$^3$).

## Temperature Stability

The temperature stability of the adduct was determined by direct observation of a sample heated in a glass beaker in the dry box. The adduct was held in the cell in equilibrium with 5 psig overpressure of $BF_3$ overnight in order to ensure complete saturation, then transferred to the beaker via the liquid sampling port. The ambient moisture was roughly 10 ppm, and no fuming, indicative of a reaction between $BF_3$ and water, was observed at any time during the experiment. The heating rate was slightly over 1°C/min, the lowest possible with the hot plate used.

## $BF_3/CH_3NO_2$ Ratio by Titration at UMR

The primary objective was determination of the $BF_3$/nitromethane ratio. Harring's report of 1.4 to 1.6 $BF_3$'s per nitromethane appeared to explain the unusual isotopic separation effectively, but this number had only been confirmed once, by Herbst, and the procedure used was not published. The first method used in this investigation was titration of HF formed by reacting the adduct with water. Since it is known that the adduct decomposes at high temperatures, the cell was cooled to $-40$°C and pressurized to ~3.6 psig with $BF_3$, overnight, in order to obtain the highest possible concentration of $BF_3$. It should be noted that the freezing point of nitromethane is $-29$°C; the fact that the liquid in the cell did not freeze at $-40$°C indicates that all of the nitromethane was in the form of the adduct. In the dry box a sample of roughly

1 mL of adduct was weighed into a known quantity of pure nitromethane (25–30 mL) to dilute it to the point where it would not react quickly with moisture in the air. A measured sample of the mixture was then quickly mixed into 100 mL of distilled water outside of the dry box, forming a weak solution of HF. The acid was then titrated to the phenolphthalein end point with NaOH, and the $BF_3/CH_3NO_2$ ratio was backcalculated from the results.

To verify the accuracy of this technique it was first tested on the diethyl ether–boron trifluoride adduct, which has a published ether/$BF_3$ ratio of 1/1 (23). Three consecutive titrations produced an average ratio of 1.09/1 These results confirmed the published ratio, providing confidence in this technique. Titration of the ether adduct was reproducible, requiring only a careful, slow approach to the end point, allowing time for the reaction to take place.

## Gravimetric Analysis

A second experiment was designed to determine the ratio directly by gravimetric analysis. The reaction cell was removed from the dry box, cleaned and dried, and weighed to the nearest 0.1 g. It was then returned to the box, half filled with nitromethane, and reweighed. The nitromethane was then frozen by cooling the cell to $-60$°C and evacuated with a vacuum pump to 30 torr. After evacuation, the cell was allowed to return to room temperature and pressurized with $BF_3$ to 20 psig to completely saturate the nitromethane. This pressure was maintained overnight to allow equilibration, and then the temperature was dropped to $-90$°C, freezing the adduct. (The observed melting point of the adduct was approximately $-49$°C.) The cell was again evacuated to 30 torr and sealed, leaving the $BF_3$ only in the adduct form. After warming back to room temperature, the cell with the adduct was weighed a final time. From the difference in weights the amount of $BF_3$ which reacted can be determined, and from that the $BF_3/CH_3NO_2$ ratio.

## $BF_3/CH_3NO_2$ Ratio by Titration at Eagle Picher

As a final confirmation, several samples of the adduct were sealed in airtight vials and sent to Eagle Picher Boron Laboratories for analysis. The method used there is unique for boron and has been used at the Eagle Picher Boron Isotopic Separation Plant in Quapaw, Oklahoma for the past 20 years. It is the standard practice for determining water-soluble boron and was used at the Eagle Picher Miami, Oklahoma research facility for decades before the Quapaw plant was built.

The technique consists of dissolving the adduct in water, titrating with NaOH to the neutral inflection point, and then adding excess mannitol which reacts with the boron to form a mannitoboric acid matrix proportional to the boron content. Finally, titrating to the second end point allows the boron content to be calculated. This procedure is described in ASTM C-791 Total Boron by Titrimetry and C-809. Furthermore, Eagle Picher set up a pilot plant to study the feasibility of using the nitromethane adduct for commercial separation of boron, and in the course of daily operations remeasured the ratio by means of this same procedure.

### Heat of Association

Once the $BF_3/CH_3NO_2$ ratio was established, it was possible to determine the heat of association of the adduct. Small, carefully weighed samples of $BF_3 \cdot CH_3NO_2$, diethyl ether, and diethyl ether$\cdot BF_3$ adduct were injected into glass ampules which were then sealed with a small torch in the dry box. The ampules were placed in a Tronac Model 450 Isoperibol Titration Calorimeter and broken underneath a large quantity of nitromethane. By measuring the heat of reaction of each of these with nitromethane, it was possible to calculate the heat of association of $BF_3$ with nitromethane

### Isotopic Separation Factor

Although the primary objective of this study was determination of the $BF_3$/nitromethane ratio, the isotopic separation factor was also measured to provide a comparison with published data. Three airtight, glass vials (~20 mL each) were half-filled with adduct drawn from the same batch as the titration samples (adduct formed at $-40$°C). The headspace at the top of each vial was then flushed with $BF_3$ from the reaction cell's gas port for about 30 seconds. The vials were then sealed and allowed to equilibrate at room temperature for 24 hours. Samples of the liquid and gas phases from each vial were then injected (separately) into a Hewlett-Packard Model 5988 mass spectrometer, and the $^{10}B/^{11}B$ ratio measured. From these data the separation factor of each of the three samples was calculated. The average was $\alpha = 1.060$, which agrees with Herbst and McCandless's result of 1.066 (25). Obtaining the same isotopic separation factor as Herbst and McCandless was taken as evidence that we were dealing with the same adduct, even though our data indicated a very different $BF_3$/nitromethane ratio.

## RESULTS AND DISCUSSION

### Thermal Stability

Thermal decomposition of the adduct revealed that the boron-10 enriched $BF_3$ can be easily separated from the donor. Small bubbles were observed forming at 40°C, first in the vicinity of the thermometer, then randomly over the bottom of the beaker. Since the boiling point of nitromethane is 100.8°C, this bubbling is believed to indicate the dissociation of $BF_3$ from the adduct.

The bubbling increased until ~60°C, at which point it decreased significantly, and by 65°C it had stopped completely. There was no further change in the liquid until the normal boiling point of nitromethane was reached. A sample of the liquid taken at 60°C was kept in an airtight vial for mass spectroscopy. The results of the analysis showed no $BF_3$ within the detectability limits of the equipment. Thus it is concluded that effectively all of the $BF_3$ can be reclaimed from the liquid adduct simply by heating to 60°C.

## $BF_3$ /$CH_3NO_2$ Ratio

The $BF_3$/nitromethane ratio was first measured by titration for HF at the University of Missouri–Rolla. These initial results indicated that the ratio was less than 0.3/1, about one-fifth of the published value. In order to confirm this unexpected result, the gravimetric analysis method was devised. Using this technique, a value of 0.158/1 was obtained, which is the same order of magnitude as the titration results. This result is believed to be more accurate than the titration results because there was no gaseous $BF_3$ dissolved as there was during the titrations. Samples of the adduct prepared for gravimetric analysis were sealed in airtight bottles and sent to Eagle Picher for boron titration. Their results also indicated a ratio of 0.15/1. Over the course of several weeks, similar titrations were carried out in the pilot-scale facility, using samples from batches of adduct much larger than were previously available. The ratio calculated from these titrations was 0.158/1 the same as that obtained by gravimetric analysis.

## Heat of Association

Once the ratio was established, it was possible to calculate the heat of association as shown:

$$(C_2H_5)_2O{\cdot}BF_{3(l)} + (100 + 1/n)(CH_3NO_2)_{(l)} =$$
$$(1/n)[(BF_3)_n{\cdot}CH_3NO_2]_{(NM)} + (C_2H_5)_2O_{(NM)} + 100(CH_3NO_2)_{(l)} \quad (1)$$

where $n$ is the $BF_3$/nitromethane ratio, and (NM) refers to a nitromethane solution

$$\Delta H_1 = +0.73 \text{ kJ/mol}$$

$$(C_2H_5)_2O_{(l)} + 100(CH_3NO_2)_{(l)} = (C_2H_5)_2O_{(NM)} + 100(CH_3NO_2)_{(l)} \quad (2)$$

$$\Delta H_2 = +3.86 \text{ kJ/mol}$$

$$[(BF_3)_n{\cdot}CH_3NO_2]_{(l)} + 100(CH_3NO_2)_{(l)} =$$
$$[(BF_3)_n{\cdot}CH_3NO_2]_{(NM)} + 100(CH_3NO_2)_{(l)} \quad (3)$$

$$\Delta H_3 = -3.56[1 + 0.5262(n - 1)] \text{ kJ/mol}$$

These equations can be combined to give

$$n[(C_2H_5)_2O\cdot BF_{3(l)}] + CH_3NO_{2(l)} = [(BF_3)_n\cdot CH_3NO_2]_{(l)} + n(C_2H_5)_2O_{(l)} \qquad (4)$$

$$\Delta H_4 = n(\Delta H_1 - \Delta H_2) - \Delta H_3$$

Filling in the measured quantities:

$$\Delta H_4 = -1.26n + 1.69 \text{ kJ/mol}$$

$\Delta H_4$ can also be written as

$$\Delta H_4 = \Delta H_f[(BF_3)_n\cdot CH_3NO_2] + n\Delta H_f[(C_2H_5)_2O_{(l)}] - n\Delta H_f[(C_2H_5)_2O\cdot BF_{3\,(l)}] - \Delta H_f[CH_3NO_{2(l)}]$$

which can be rearranged to show

$$\Delta H_f[(BF_3)_n\cdot CH_3NO_2]_{(l)} = \Delta H_4 - n\Delta H_f[(C_2H_5)_2O_{(l)}] + n\Delta H_f[(C_2H_5)_2O\cdot BF_{3\,(l)}] + \Delta H_f[CH_3NO_{2(l)}]$$

Again, filling in known quantities:

$$\Delta H_f[(BF_3)_n\cdot CH_3NO_2]_{(l)} = \Delta H_4 - 1202.8n - 113.1 \text{ kJ/mol}$$

The heat of association can now be written, using the values found above and known values,

$$\Delta H_{\text{assoc}} = \Delta H_f[(BF_3)_n\cdot CH_3NO_2]_{(l)} - \Delta H_f[CH_3NO_{2(l)}] - n\Delta H_f[BF_{3(g)}]$$

$$= -1.26n + 1.69 - 1202.8n - 113.1 - (-113.1) - n(-1137.0) \text{ kJ/mol}$$

Using the ratio found in this study, $n = 0.158$, this gives a heat of association of −8.51 kJ/mol, or −12.9 kcal/mol $BF_3$. The specific heat of the adduct was measured at the same time as the heats of reaction and was found to be 0.52 ± 0.01 cal/g°C in the 0–23°C range.

## CONCLUSIONS

The physical properties of the $(BF_3)_n\cdot CH_3NO_2$ adduct have been studied using a variety of techniques. The density was found to be 1.31 g/cm$^3$, the heat of association −8.51 kJ/mol, and the specific heat 0.52 cal/g°C. The adduct was found to be most stable at low temperatures and to decompose completely by 60°C. Most significantly, the ratio of $BF_3$ to nitromethane was found to be far smaller than previously thought. Where it had previously been reported to be 1.4/1 to 1.6/1, this study found a ratio of 0.158/1. This ratio was obtained from titration data, confirmed by gravimetric analysis, verified by a two-step titration for boron by Eagle Picher, and again verified in an operating pilot plant. This finding contradicts previously published data. The remarkable properties of this adduct cannot be explained by a higher $BF_3$/donor ratio than other such adducts. On the contrary, the ratio is lower than any other $BF_3$ adduct reported by a factor of 5.

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## REFERENCES

1. R. S. Harring and T. J. Livinghouse, *Chem. Soc.—Chem. Commun.*, *6*, 503 (1992).
2. R. S. Harring and T. J. Livinghouse, *Ibid.*, *6*, 502 (1992).
3. R. S. Harring and T. J. Livinghouse, *Tetrahedron*, *50*, 9229 (1994).
4. R. S. Harring, "Improved Reagents for Episulfonium Ion and Lewis Acid Mediated Cationic Cascade Annulations: Applications to Natural Product Synthesis," Ph.D. Thesis, University of Minnesota, 1992.
5. H. Florke and E. Schaumann, *Synthesis* (*Stuttgart*), *5*, 647 (1996).
6. W. K. Anderson and G. F. Lai, *Ibid.*, *10*, 1287 (1995).
7. M. Morioka, M. Kato, H. Yoshida, and T. Ogata, *Heterocycles*, *43*, 305 (1996).
8. W. Adam, C. Sahin, and M. Schneider, *J. Am. Chem. Soc.*, *117*, 1695 (1995).
9. N. Katagiri, M. Okada, Y. Morishita, and C. Kaneko, *Chem. Commun.*, *18*, 2137 (1996).
10. L. McKinstry and T. Livinghouse, *Tetrahedron*, *51*, 7655 (1995).
11. T. Imai, T. Muramoto, and T. Tsuji, *Chem. Lett.*, *5*, 355 (1995).
12. H. Matsunaga, *Biomed. Chromatogr.*, *10*, 95 (1996).
13. H. Y. Kang, B. N. Park, and H. Y. Koh, *Korean Chem. Soc. Bull.*, *18*, 1245 (1997).
14. L. M. Nxumalo, M. Andrzejak, and T. A. Ford, *J. Chem. Inf. Comput. Sci.*, *36*, 377 (1996).
15. W. A. Herrebout and B. J. Vanderveken, *J. Am. Chem. Soc.*, *119*, 10446 (1997).
16. G. P. Everaert, *Chem. Eur. J.*, *4*, 321 (1998).
17. L. M. Nxumalo and T. A. Ford, *Spectrochim. Acta A*, *53*, 2511 (1997).
18. L. M. Nxumalo and T. A. Ford, *Mikrochim. Acta*, *S14*, 383 (1997).
19. A. A. Palko, R. M. Healy, and L. Landau, *J. Chem. Phys.*, *28*, 214 (1958).
20. A. A. Palko, *Ibid.*, *30*, 1187 (1959).
21. A. A. Palko and J. S. Drury, *Ibid.*, *33*, 779 (1960).
22. A. A. Palko and J. S. Drury, *Ibid.*, *40*, 278 (1964).
23. A. A. Palko and J. S. Drury, *Ibid.*, *46*, 2297 (1967).
24. A. A. Palko and J. S. Drury, in *Advances in Chemistry Series*: *Isotope Effects in Chemical Processes*, *89*, 1969.
25. R. S. Herbst and F. P. McCandless, *Sep. Sci. Technol.*, *29*, 1293 (1994).
26. R. S. Herbst, "An Analysis of Chemical Exchange Systems for the Separation of Boron Isotopes," Ph.D. Thesis, Montana State University, Bozeman, 1992.

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