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SOLUBILITY OF BISMUTH
IN
LIQUID SODIUM

AEC Research and Development Report

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN ROCKWELL CORPORATION

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Errata

to

SOLUBILITY OF BISMUTH IN LIQUID SODIUM

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Page 4

The equations in the abstract should read:

<u>Temperature Range (°C)</u>	<u>Expression</u>	<u>ΔH (Solution) kcal/mole</u>
125 - 290	$\log_{10}(\text{at. \% Bi}) = 1.188 - 2100/T(^{\circ}\text{K})$	9.6
290 - 650	$\log_{10}(\text{at. \% Bi}) = 4.900 - 4178/T(^{\circ}\text{K})$	19.1

Atomics International

North American Rockwell Corp.

AI-AEC-12770

SOLUBILITY OF BISMUTH
IN
LIQUID SODIUM

By
D. C. GEHRI
R. J. SULLIVAN

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ABSTRACT

The solubility of bismuth in liquid sodium has been studied in the temperature range of 200 to 650°C, using a specially prepared Na_3Bi solute and very-high-purity sodium. A capsule technique, involving a physical separation of the excess solute from the solution, was used for the experiments. Chemical analyses were performed, using atomic absorption spectrophotometry.

The results from this work have been combined with those of two other laboratories, to yield two separate solubility expressions from which heats of solution have been individually calculated. These expressions are recommended for describing the solubility of bismuth in liquid sodium in specific temperature ranges, as given in the following list.

<u>Temperature Range (°C)</u>	<u>Expression</u>	<u>$\Delta H(\text{solution})$ (kcal/mole)</u>
125 to 290	$\log_{10}(\text{at. \% Bi}) = 4.900 - 4178/T(^{\circ}\text{K})$	19.1
290 to 650	$\log_{10}(\text{at. \% Bi}) = 1.188 - 2100/T(^{\circ}\text{K})$	9.6

The first of these expressions was derived from data which may reflect the influence of Na-Bi-O interactions; and it should be used only with this qualification understood. No such restriction is placed on the second expression, which is therefore recommended without qualification for describing the solubility of elemental bismuth in sodium.

I. INTRODUCTION

The fundamental purpose of this report is to provide a summary of the solubility of bismuth in liquid sodium, as it relates to the coolant system of a fast breeder reactor. Concern about the transport of soluble bismuth arose when detectable amounts of this element were found in the coolant of the EBR-II. At that time, little was known about the solubility of bismuth in sodium, particularly for the temperature range of interest in reactor systems. The Na-Bi phase diagram given by Hansen and Anderko,⁽¹⁾ from which only crude solubility estimates could be made, was the source of such information as was then available. The present work was initiated to provide a more adequate and suitable basis for the needed solubility information.

In keeping with the fundamental purpose of this report, solubility experiments have been conducted in the 200 to 650°C temperature range. The experimental approach involved the equilibration of purified sodium with a specially prepared Na₃Bi solute, separation of the resulting Na-Bi solution from the bulk solute, and analysis of the solution by atomic absorption spectrophotometry (AAS). Details of the experimental methods are discussed in Section II, and the results are summarized in Section III.

Additional measurements relating to the solubility of bismuth in sodium have recently been carried out at Birmingham University (UK) by Pratt and Walker,⁽²⁾ and at Argonne National Laboratory by Johnson⁽³⁾ and by Foster.⁽⁴⁾ Selected data from these laboratories are discussed in Section IV, and have been included in the derivation of the solubility equations in Section V. These investigators have graciously consented to the use of their data, prior to formal publication, in order to provide a supplement to, and a comparison for, the data obtained by the authors of this report. Similarly, the data obtained from the present work have been released to these investigators, for use in their forthcoming publications.

The experimental approach of Pratt and Walker⁽²⁾ involved the equilibration of purified sodium with bismuth metal, sampling at temperature with a dip-cup technique, and a gravimetric analysis of the resulting sample. Some of the ANL data⁽³⁾ were derived by determining the break in the solubility curve for known

Na-Bi compositions, using thermal analysis. The remainder⁽⁴⁾ involved an equilibration of reagent grade sodium with bismuth metal, sampling through a glass frit, and analysis by AAS. These approaches are distinctly different from that of the present work, but the data obtained at the three laboratories are in general agreement. This fact suggests that any one of the experimental approaches may be adequate, subject, of course, to certain precautions required by the specific techniques employed.

II. EXPERIMENTAL METHOD - PRESENT WORK

A. PREPARATION AND PROPERTIES OF THE SOLUTE MATERIAL

The choice of Na_3Bi as a solute material was suggested by the fact that solid Na_3Bi is the phase in equilibrium with an Na-Bi solution, for compositions of <25 at.% Bi. This choice also allowed convenient separation of the solute from the solution, even at temperatures above the melting point of bismuth (271°C). Preliminary experiments indicated that Na_3Bi , which is a purple crystalline solid, can be readily oxidized by trace amounts of oxygen or moisture. This oxidation manifests itself by a blackening of the surface. Both pure Na_3Bi and the oxidized material react violently with water, forming a spongy black precipitate which is soluble in hot dilute HNO_3 . Such behavior suggests that the precipitate is probably Bi_2O_3 , which is insoluble in water but will dissolve in acid.

In order to prepare a suitable solute, it was therefore necessary to use high-purity starting materials, and to protect these materials and the product (Na_3Bi) from exposure to oxidizing or contaminating environments. The preparation was begun by adding a weighed amount of high-purity bismuth metal to a stainless steel cup contained within a cylindrical stainless steel capsule. A typical analysis of the bismuth metal showed <0.001% for each of four major impurities (Fe, Sb, As, and Zn), and a total impurity level of 0.001% for the sum of four others (Ag, Pb, Cu, and Te). The metal was washed with dilute HCl, prior to use, in order to remove any surface contamination, and was loaded into the preparation capsules in a recirculating argon glove box.

The capsule assembly and an appropriate cap were then weighed and transferred to a high-vacuum system, where very-high-purity sodium was added and the cap welded in place with an electron-beam welder. This sodium had been purified by hot gettering, distilled, and collected in an extruder, which was subsequently attached to the sodium loading chamber of the high-vacuum system. Pressure in this chamber was maintained in the low 10^{-8} mm range by large ion pumps, thus preventing gas-phase contamination of the extruded sodium. Details of the sodium purification apparatus can be found in NAA-SR-12444,⁽⁵⁾ and a description of the high-vacuum system is given in AI-AEC-12671.⁽⁶⁾

Analysis of the sodium used in the preparation of Na_3Bi (and also in subsequent solubility experiments) showed 9 wppm C, <3 wppm O, and <10 wppm of detectable metallic contaminants.

The exact amount of sodium added, and therefore the composition of the Na-Bi mixture, was determined by a final weighing of the sealed capsule after removal from the chamber, because it is difficult to measure the exact weight of an extruded piece of sodium inside the chamber. An effort was made to dispense enough sodium to produce a Na-Bi composition close to, but slightly above, 25 at.% Bi (75.19 wt %). The success of this effort is attested to by the compositions given in Table 1 for the five capsules which were loaded.

TABLE 1
 Na_3Bi PREPARATION

Capsule Number	Composition (wt % Bi)*
1	76.64
2	76.72
3	76.68
4	81.58
5	75.40

* Na_3Bi contains 75.19% Bi

Although preliminary experiments indicated that Na_3Bi would form from a Na-Bi mixture at temperatures as low as 500°C, the product was too friable for use as a convenient solute material. The five capsules were therefore heated to ~900°C in a Marshall furnace contained within an argon glove box. The fused Na_3Bi , prepared in this way, proved to be a more satisfactory solute, and the contents of Capsules 1, 2, and 3 were used in subsequent solubility experiments. A cooling curve was run on the contents of Capsule 5, in order to test the purity of the Na_3Bi and the accuracy of the weighing methods.

The cooling curve showed two inflections, one at 845°C and the other at 725°C. The higher of these was ascribed to the Na_3Bi melting process, and

agrees well with the melting point of Na_3Bi (848.1°C) as determined at ANL.⁽³⁾ The preparation method was therefore deemed satisfactory, and the contents of the cooling-curve capsule were recovered for use in solubility experiments. A 725°C inflection is not readily explainable for the Na-Bi system, but it may have resulted from the austenite-ferrite phase transformation of the heating element support, whose cooling curve is superimposed on that of the Na-Bi phase.

B. SOLUBILITY EXPERIMENTS

The capsules containing the Na_3Bi solute were opened in an argon glove box, and the solute removed for transfer to a stainless steel solubility capsule. A typical solubility capsule is pictured in Figure 1. It consists of a screen assembly, a cylindrical capsule with a shoulder at mid-length to hold the screen in place, and a cap. The screen was 100-mesh (150μ) stainless steel, which was designed to allow the Na-Bi solution to pass through while collecting the Na_3Bi particles. Only those particles of Na_3Bi which would not pass through an 18-mesh (1 mm) screen were used in the experiments. In addition, enough excess Na_3Bi was added to insure that the solution process would be unlikely to reduce the size of the particles to $<500\mu$. In one experiment at 720°C , it was found that the Na-Bi solution itself would not pass through the 150μ screen, and the screen size was therefore increased to 300μ for a 650°C solubility experiment. No evidence for solution holdup was found in the 650°C experiment, or in any other experiment below 500°C , even with the 150μ screen.

After the solute was added to a solubility capsule, the screen assembly was inserted by spreading the legs and forcing the assembly into position. It was held in place by the shoulder, and prevented from falling out of the capsule by the legs. The capsule and its cap were then placed in an O-ring sealed transfer vessel, removed from the glove box, and placed immediately in the antechamber of the high-vacuum system. From there, the vessel was moved into the turbo-molecular-pumped vacuum chamber, where the vessel was opened and the argon pumped out. The capsule and cap were then taken into the loading chamber, where ~ 2 gm of high-purity sodium was added and the cap welded in place. The loaded capsules were removed from the high-vacuum system, equilibrated at the desired temperature for ~ 20 hr, inverted at temperature to separate the

Figure 1. Solubility Capsule

Figure 2. Marshall Furnace

undissolved Na_3Bi from the solution, and given to the Analytical Chemistry Group for analysis. Both the preparation capsules and the solubility capsules were heated in the same Marshall furnace, a drawing of which is shown in Figure 2.

Bismuth analysis was accomplished by cutting the capsule open in a glove box, stepwise dissolution of the Na-Bi in alcohol, water, and finally dilute HNO_3 , and measuring the bismuth with AAS. Standard samples of bismuth were prepared, using the same high-purity bismuth metal that was used in the solute preparation. The analysis consisted of measuring the absorption of a series of these standards, and relating the amount of bismuth in the sample, as indicated by its absorption, to the calibration curve provided by the standards. Sample size was determined by weighing the capsule end, before and after dissolution of the sample.

III. EXPERIMENTAL RESULTS - PRESENT WORK

Ten solubility experiments were conducted, using the methods and procedures described in the preceding section. Eight of these were deemed reliable, on the basis of pertinent observations during the course of the experiments, and the results are given in Table 2. Two were rejected, one at 300°C because of an unsatisfactory temperature equilibration, and the other because of problems in the flow of the solution phase through the screen. This flow problem, as mentioned before, was only observed in a 720°C experiment, where the bismuth concentration presumably was very high, and the Na-Bi solution apparently was too viscous to flow freely through the screen when the capsule was inverted.

Although a statistically valid linear solubility expression can be derived from these eight experiments, the 200°C results suggest that such a linear fit, and the implication of a constant ΔH (solution), may not be the best representation of bismuth solubility over the entire temperature range involved. These data, which are in good agreement with the British and the ANL data, have therefore been included in a composite treatment of all available bismuth solubility results. Such a treatment is given in Section V of this report.

TABLE 2
RESULTS OF BISMUTH-IN-SODIUM SOLUBILITY
EXPERIMENTS - Na₃Bi SOLUTE

Run No.	Temperature (°C)	Bi in Na (wppm)
664-8	203	38
664-11	302	185
664-12	402	2,500 (0.250 wt %)
665-2	500.5	23,400 (2.34 wt %)
665-3	500	23,600 (2.36 wt %)
665-8	401	5,250 (0.525 wt %)
665-12	201.5	42
666-2	648.5	130,400 (13.04 wt %)

IV. EXPERIMENTAL METHODS AND RESULTS - OTHER LABORATORIES

A. BRITISH WORK - PRATT AND WALKER⁽²⁾

Ten bismuth solubility experiments have been reported by Pratt and Walker. Their experimental method consisted in the equilibration of 230 cc of sodium, containing 15 ppm oxygen, with an excess of bismuth metal. The Na-Bi solution was contained in an electroformed nickel crucible, which was heated within a dry box and stirred with a nickel rod driven by an external motor. Samples were taken at the equilibration temperature, by means of smaller nickel crucibles which held 4 cc of solution. These samples were removed from the dry box in sealed weighing bottles, and analyzed for bismuth by a gravimetric technique involving the formation of BiPO_4 . A total of twelve samples were taken, but the two at 164 and 210°C were not amenable to gravimetric analysis because of the low bismuth concentrations involved. Results for the remaining ten are given in Table 3.

TABLE 3
RESULTS OF BISMUTH-IN-SODIUM
SOLUBILITY EXPERIMENTS
PRATT AND WALKER⁽²⁾

Temperature (°C)	Bi in Na (wt %)	Bi in Na (at. %)
563	7.71	0.911
514	10.24	1.24
493	3.25	0.368
475	1.23	0.137
434	0.810	0.0898
402	0.300	0.0331
370	0.361	0.0398
332	0.135	0.0149
293	0.050	0.00546
253	0.008	0.00092

TABLE 4
ANL PHASE DIAGRAM DATA -
JOHNSON et al.^(3, 7)

Liquid Phase Composition (at. % Na)	Break in Solubility Curve (°C)	Reference
95.0	650.0	3
90.0	714.9	3
85.0	751.4	3
82.5	769.8	3
80.0	792.6	3
80.0	797	7
77.5	820	7
75.0 (Na ₃ Bi)	848.1	3

TABLE 5
ANL BISMUTH-IN-SODIUM
SOLUBILITY DATA -
FOSTER⁽⁴⁾

Temperature (°C)	Bismuth (at. % x 10 ³)
125	0.071
125	0.078
150	0.267
175	0.229
175	0.331
200	0.784
225	1.35
250	2.28

B. ANL WORK — JOHNSON⁽³⁾ AND FOSTER⁽⁴⁾

Two types of bismuth solubility experiments have been conducted at ANL. An extensive series of thermal analyses has been conducted, in order to define the Na-Bi phase diagram. Results have been obtained for compositions ranging from 9.8 to 95 at.% Na. In these experiments, the break in the solubility curve and/or the peritectic and eutectic halt has been measured for fixed Na-Bi compositions. Of interest to the present discussion are the five thermal analyses conducted with compositions in the 80 to 95 at.% Na range. Two additional points have been extracted from some liquid-vapor phase diagram work that had previously been published by ANL workers.⁽⁷⁾ These seven results, together with a thermal analysis of 75.0 at.% Na (Na_3Bi), are given in Table 4.

The second type of experiment involved low-temperature solubility measurements, using techniques similar to those of the British workers. Eight such experiments were conducted, in which reagent grade sodium was equilibrated with bismuth metal in an appropriate container within an argon glove box. The Na-Bi mixture was stirred and maintained at temperature for 4 to 24 hr, after which a sample was taken through a fine porosity glass frit. Analysis was accomplished using AAS techniques similar to those previously described. These data are given in Table 5, but it should be emphasized that they result from an equilibration with reagent grade sodium, for which the oxygen levels may be relatively high. An estimate of this level, as provided by numerous analyses performed on this type of sodium at our laboratory, indicates a nominal value of ~50 ppm oxygen. Oxygen interactions with the Na-Bi solution could therefore be important in such experiments, particularly at the low bismuth concentrations measured. The implications of Na-Bi-O interactions, and their potential effect on bismuth solubility, are discussed in more detail in the following section.

TABLE 6
BISMUTH-IN-SODIUM SOLUBILITY - ALL DATA

Temperature (°C)	Bismuth (at.%)	Method of Analysis	Oxygen Level in Na (ppm)	Reference
125	0.000071	AAS	~50	4
125	0.000078	AAS	~50	4
150	0.000267	AAS	~50	4
175	0.000229	AAS	~50	4
175	0.000331	AAS	~50	4
200	0.000784	AAS	~50	4
201.5	0.00046	AAS	<3	This work
203	0.00042	AAS	<3	This work
225	0.00135	AAS	~50	4
250	0.00228	AAS	~50	4
253	0.00092	Gravimetric	15	2
293	0.00546	Gravimetric	15	2
302	0.00204	AAS	<3	This work
332	0.0149	Gravimetric	15	2
370	0.0398	Gravimetric	15	2
401	0.0580	AAS	<3	This work
402	0.0276	AAS	<3	This work
402	0.0331	Gravimetric	15	2
434	0.0898	Gravimetric	15	2
475	0.137	Gravimetric	15	2
493	0.368	Gravimetric	15	2
500	0.265	AAS	<3	This work
500.5	0.263	AAS	<3	This work
514	1.24	Gravimetric	15	2
563	0.911	Gravimetric	15	2
648.5	1.623	AAS	<3	This work
650.0	5.0	Thermal	?	3
714.9	10.0	Thermal	?	3
751.4	15.0	Thermal	?	3
769.8	17.5	Thermal	?	3
792.6	20.0	Thermal	?	3
797	20.0	Vapor-Liquid	~50	4
820	22.5	Vapor-Liquid	~50	4
848.1	25.0	Thermal	?	3

V. DISCUSSION

All the data given in Tables 2, 3, 4, and 5 have been reduced to the common basis of at.% Bi, ordered with respect to temperature, and are summarized in Table 6 and plotted in Figure 3. As a first step in the treatment of these data, an attempt was made to fit all the given data points to a simple solubility equation of the form:

$$\log_{10}(\text{at.\% Bi}) = A + B/T(^{\circ}\text{K})$$

Statistical analysis of the resulting line indicated that such a linear fit was not satisfactory at the 95% confidence level. The reasons for this are obvious from an examination of Figure 3. Two nonlinear trends are clearly indicated. The first is an apparently lower heat of solution for the data below 260°C, resulting in a systematically higher solubility than would be predicted by a linear fit of all the data. The second is an inflection in the solubility curve at a temperature $\geq 650^{\circ}\text{C}$, which is suggested by the temperature dependence of the thermal analysis data.

The experimental reason for the unusual behavior at high temperature is not immediately obvious; and, since the solubility measurement at 650°C gave a substantially lower result than did thermal analysis, it was decided to exclude the thermal analysis data from further consideration in this report. In addition, the solubility of bismuth in sodium above 650°C is of little interest in reactor technology, so that the fundamental purpose of this study is not compromised by such an exclusion.

Although eight of the eleven data points at temperatures below 260°C were derived from experiments in which the oxygen level of the sodium was rather high, the remaining three also seem to indicate a somewhat lower heat of solution than would be calculated from a linear fit of all solubility data between 125 and 650°C. If one notes that the solubility values range over nearly five decades on the log (at.%) vs 1/T plot in the 125 to 650°C temperature range, it is not unreasonable to expect a significant departure from linearity. The treatment of the data, excluding thermal analysis results, was therefore predicated on the assumption that the true solubility curve is best represented by taking this nonlinearity into account.

Figure 3. All Data Points.

At the same time, it seemed inadvisable to treat the data as a single set, since the oxygen levels of the sodium used in eight of the low-temperature experiments were significantly higher than in any of the others. Measured solubilities might include the contributions of Na-Bi-O compounds or complexes. For fixed oxygen levels, such contributions would become relatively more important as the true solubility decreased. Thus, both the measured solubility values and the slope of the solubility curve could be affected by the presence of oxygen.

The data were therefore divided into two sets, one of which included the previously-mentioned eight data points and three others in the same temperature range. The other set consisted of 15 data points, the lowest of which was taken at 293°C. Statistical analysis of each set indicated that a separate linear fit was satisfactory at the 97.5% confidence level. Least squares calculations provided the following equations for the two sets of data:

<u>Data Range</u>	<u>Solubility Equation</u>	
293 to 648.5°C	$\log_{10}(\text{at. \% Bi}) = 4.900 - 4178/T(^{\circ}\text{K})$	...(1)
125 to 253°C	$\log_{10}(\text{at. \% Bi}) = 1.188 - 2100/T(^{\circ}\text{K})$	...(2)

These lines, and the data points from which they were derived, are plotted in Figure 4. Heats of solution, calculated from these equations, are 19.1 and 9.6 kcal/mole, respectively.

It should be emphasized that the division of the data into two sets, and the resulting equations, should not be construed as an indication of a discontinuity in the true solubility curve. Rather, this was done to obviate the errors that might have arisen from the leverage that could be exerted by the low-temperature data on a single-set treatment. Furthermore, such a division may well be fortuitous in FBR design calculations.

Thus, Equation 1 is recommended for use in describing the transport of soluble bismuth in the temperature range of 290 to 650°C, which includes the normal operating range for the coolant systems of an LMFBR. At lower temperatures, which are characteristic of cold traps and similar LMFBR components,

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Figure 4. The Solubility of Bismuth in Liquid Sodium - Recommended Expressions

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Na-Bi-O interactions may become important. Equation 2, which covers the lower temperature range, was derived from data which may reflect the potential problems associated with oxygen interactions. This equation could therefore provide a useful approximation at these lower temperatures (125 to 290°C), and in those applications which must take such interactions into account.

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