

Serial AB-10
Nov. 18, 1963
Copy 4 of 6

MONTHLY PROGRESS REPORT No. 8

PO 13-5966

to

Sandia Corporation

from

Callery Chemical Company

INVENTORIED
MAR 8 '65
3421-3

RECEIVED
2714/433
NOV 21 1963

Submitted by:

R. B. Cruikshank
R. B. Cruikshank
Project Leader

Approved by:

3421-3

H. S. Uchida
H. S. Uchida
Assistant Research Director

INVENTORIED

MAR 1 1966

SANDIA SYSTEMATIC DECLASSIFICATION REVIEW	
DOWNGRADING OR DECLASSIFICATION STAMP	
CLASSIFICATION CHANGED TO: <u>U</u>	AUTHORITY: <u>W.C. Lopez</u>
PERSON CHANGING MARKING & DATE: <u>Carmela Salgado 11/2/97</u>	RECORD ID: <u>98 SW 428</u>
PERSON VERIFYING MARKING & DATE: <u>Carmela Salgado 11/12/97</u>	DATED: <u>10/28/97</u>

George F. Huff
George F. Huff
Vice President
Research and Development

3428-3

RECORD COPY

Do Not Take From This Room

SANDIA SYSTEMATIC DECLASSIFICATION REVIEW	
Review Date: <u>10/21/97</u>	Classification (Circle Numbers)
Name: <u>P. B. Chaner</u>	1. Classification Retained
Review Date: <u>10-28-97</u>	2. Classification Changed to <u>U, E, C, C</u>
Authority: <u>W.C. Lopez</u>	3. Contains No DOE Classified Information
Name: <u>W.C. Lopez</u>	4. Contains WHA
	5. Contains UCAIT
	6. Contains:
	<u>DECLASSIFY</u>

[REDACTED]

Paul Patterson
3519/66

UNCLASSIFIED



ALLERY CHEMICAL COMPANY

RESEARCH & DEVELOPMENT DIVISION • CALLERY, PA. • EVANS CITY 3510 • TWX • EVANS CITY 136

Serial AB-10
Nov. 18, 1963

Page 1

Sandia Corporation
Post Office Box 5800
Albuquerque, New Mexico

Attn: Mr. E. B. Bergquist, 4321

Ref. Sym: 4333/142, P. O. 13-5966

Dear Mr. Bergquist:

The following constitutes the eighth monthly report for the period Sept. 1 to Sept. 30, 1963, as required under Purchase Order 13-5966.

Experimental investigation has continued on Phase I, Part C of the program. Presently our efforts have been devoted to the preparation of aminoborane decamer, $(BH_2NH_2)_{10}$, and the determination of the thermal stability of aminoborane trimer, $(BH_2NH_2)_3$, recovered from attempts to produce the decamer.

Three attempts to prepare aminoborane decamer have been unsuccessful in that no ammonia insoluble material was recovered. In one run using sodium acetylide, a large amount of ammonia insoluble material proved to be impurities carried into the reaction with the sodium acetylide. The thermal stability of a sample of impure aminoborane trimer was found to be unsatisfactory for this program. A rate of decomposition of 760 standard cc of H_2 per gram mole contained hydrogen per year was determined for this

UNCLASSIFIED

UNCLASSIFIED

Sandia Corporation
MPR No. 8
Page 2 of 4

Serial AB-10
Nov. 18, 1963

sample between 450 and 1000 hours at 165°F. It must be pointed out that the effect of the impurities on the stability of aminoborane trimer is unknown. Attempts to obtain a sample of pure aminoborane trimer are continuing.

EXPERIMENTAL

Aminoborane Decamer Preparation

Three runs were made in an attempt to produce enough aminoborane decamer for thermal stability studies. In the first run (2705-9), sodium amide (9.72 grams, 95.5% based on ammonia evolution) was charged in a dry box to a one liter, three neck flask. The flask was then attached to a special vacuum system and 900 ml of ammonia condensed in at -78°C. Diborane (7.22 grams) diluted with nitrogen was fed under the liquid level over a period of twelve hours. The reaction mixture was aged for 18 hours after which the ammonia was evaporated and the solids dried in vacuo. The solids were then redissolved in liquid ammonia to recover the insoluble aminoborane decamer by filtration.

In the second run (2705-10), sodium amide (19.8 grams) and diborane (14.1 grams) were reacted in 900 ml of ammonia to determine if the formation of aminoborane decamer would be favored by an increase in concentration of reactants. In other respects, the run was identical to the first run.

Sodium acetylide (8.5 grams, 73.2% by acetylene evolution) was used instead of sodium amide for the third run (2705-11). In other respects, the run was the same as the previous runs.

Aminoborane Trimer Stability

A preliminary evaluation of the stability of aminoborane trimer was made using the sublimate collected at room temperature from Run 2705-5. Infrared and x-ray analyses indicated the sample to be aminoborane trimer. Chemical analysis for this sample gave values of B, 31.3 mats/g; C, 3.14 mats/g; H, 125.5 mats/g; N, 2.3 mats/g compared to theoretical for BH_2NH_2 of B, 30.6 mats/g; H, 138.5 mats/g; N, 24.6 mats/g. The analytical accountability was only 91.8 percent indicating the presence of impurities. The sample (0.5743 gram) was charged to a 170 ml glass thermal stability bomb equipped with three break-off tips to allow for periodic measurements of the gas evolved. The experimental techniques are the same as described in MPR No. 4 for the hydrazine diborane thermal stability studies in glass bombs.

UNCLASSIFIED

UNCLASSIFIED

Sandia Corporation
 MPR No. 8
 Page 3 of 4

Serial AB-10
 Nov. 18, 1963

RESULTSAminoborane Decamer Preparation

The attempts to produce aminoborane decamer using sodium amide were not successful in that no ammonia insoluble material was recovered. The reaction using sodium acetylide gave 1.8 grams of insoluble material but this material would not sublime at temperatures up to + 196°C. Chemical analysis of the starting sodium acetylide showed it to be very impure so it is concluded that the ammonium insoluble fraction recovered was impurities brought in with the sodium acetylide.

Aminoborane Trimer Stability

The results of the thermal stability studies at 165°F of an impure sample of aminoborane trimer are presented below.

Time (Hours)		H ₂ Evolved		Std. cc H ₂ evolved per Gram mole contained H ₂ per year
From	To	Mmole	Std. cc	
0	93.9	0.188	4.21	16,700
93.9	446.6	0.199	4.46	4,720
446.6	945	0.045	1.01	760

The hydrogen evolved is based on non-condensable gases evolved from the sample and analyzed by mass spectrophotometer to be essentially hydrogen. The decomposition rate is calculated from the hydrogen originally contained in the sample (125.5 mats/g), the hydrogen evolved, and the time increment.

DISCUSSIONAminoborane Decamer Preparation

The preparation of aminoborane decamer has not yet been accomplished. Attempts to contact Dr. Shore at The Ohio State University for a discussion of the problems in the preparation of the aminoborane decamer have not been successful. We have found that he has difficulties in preparing the

UNCLASSIFIED

001733

GALLERY CHEMICAL COMPANY

UNCLASSIFIED

Sandia Corporation
MPR No. 8
Page 4 of 4

Serial AB-10
Nov. 18, 1963

aminoborane decamer on any scale larger than one gram. We have been attempting to produce five to ten grams of aminoborane decamer to obtain enough material for stability studies. A reduction in scale may allow the preparation of at least some aminoborane decamer.

Another possible solution to the preparation problem is the use of sodium acetylide rather than sodium amide. The one attempt was with a commercial grade of sodium acetylide which we did not analyze until the run was complete. The addition of the diborane to liquid ammonia first to form the diammoniate of diborane and then the addition of a solution of sodium acetylide in liquid amine is reported (1) to cause the instantaneous precipitation of the aminoborane decamer. We will try the reaction after first making pure sodium acetylide.

Aminoborane Trimer Stability

The thermal stability of the impure aminoborane trimer at 165°F is not satisfactory for this program. The effects of the impurity on the stability can not be predicted. Attempts are presently underway to recover some pure aminoborane trimer from the decamer reactions by ammonia, ether, water and methanol extractions and by sublimation. If a pure sample cannot be recovered by these methods, attempts will be made to synthesize the aminoborane trimer directly by reported methods.

FUTURE WORK

1. Prepare aminoborane decamer for thermal stability studies.
2. Recover or prepare aminoborane trimer for thermal stability studies.

Very truly yours,

GALLERY CHEMICAL COMPANY



R. B. Cruikshank
Project Leader

/mb

- (1) Shore, Sheldon G., et al, The Preparation and Self-Association of BH_2NH_2 Units, Department of Chemistry, The Ohio State University, Columbus 10, Ohio. Presented at the International BN Symposium, Durham, N.C., April 1963.

UNCLASSIFIED