

Keywords: *gadolinium,
oxalate, solubility, canyon*

Retention: *Permanent*

Gadolinium Oxalate Solubility Measurements in Nitric Acid Solutions

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March 2012

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Prepared for the U.S. Department of Energy under
contract number DE-AC09-08SR22470.



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Printed in the United States of America

**Prepared for
U.S. Department of Energy**

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EXECUTIVE SUMMARY

HB-Line will begin processing Pu solutions during FY2012 that will involve the recovery of Pu using oxalate precipitation and filtration. After the precipitation and filtration processes, the filtrate solution will be transferred from HB-Line to H-Canyon. The presence of excess oxalate and unfiltered Pu oxalate solids in these solutions create a criticality safety issue if they are sent to H-Canyon without controls in H-Canyon. One approach involves H-Canyon receiving the filtrate solution into a tank that is poisoned with soluble gadolinium (Gd). Decomposition of the oxalate will occur within a subsequent H-Canyon vessel.

The receipt of excess oxalate into the H-Canyon receipt tanks has the potential to precipitate a portion of the Gd poison in the receipt tanks. Because the amount of Gd in solution determines the maximum amount of Pu solids that H-Canyon can receive, H-Canyon Engineering requested that SRNL determine the solubility of Gd in aqueous solutions of 4-10 M nitric acid (HNO_3), 4-12 g/L Gd, and 0.15-0.25 M oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$) at 25 °C. The target soluble Gd concentration is 6 g/L. The data indicate that the target can be achieved above 6 M HNO_3 and below 0.25 M $\text{H}_2\text{C}_2\text{O}_4$.

At 25 °C, for 6 M HNO_3 , 11 g/L and 7 g/L Gd are soluble in 0.15 M and 0.25 M $\text{H}_2\text{C}_2\text{O}_4$, respectively. In 4 M HNO_3 , the Gd solubility drops significantly to 2.5 g/L and 0.8 g/L in 0.15 M and 0.25 M $\text{H}_2\text{C}_2\text{O}_4$, respectively. The solubility of Gd at 8-10 M HNO_3 exceeds the solubility at 6 M HNO_3 . The data for 4 M HNO_3 showed good agreement with data in the literature. To achieve a target of 6 g/L soluble Gd in solution in the presence of 0.15-0.25 M oxalate, the HNO_3 concentration must be maintained at or above 6 M HNO_3 .

The solubility of Gd in 4 M HNO_3 with 0.15 M oxalate at 10 °C is about 1.5 g/L. For 6 M HNO_3 with 0.15 M oxalate, the solubility of Gd at 10 °C is about 10 g/L.

Gadolinium nitrate is very soluble in HNO_3 . The solubility of Gd is linear as a function of HNO_3 from 343 g/L Gd in 2.88 M HNO_3 to 149 g/L in 8.16 M HNO_3 . Below 2.88 M HNO_3 , the solubility of Gd approaches a limit of about 360 g/L. However, there are no data available below 1.40 M HNO_3 , which has a Gd solubility of 353 g/L.

TABLE OF CONTENTS

LIST OF TABLES	vii
LIST OF FIGURES	vii
LIST OF ABBREVIATIONS	vii
1.0 Introduction	1
2.0 Background	1
3.0 Experimental Procedure	2
3.1 Gadolinium Nitrate – Nitric Acid – Oxalic Acid	2
3.2 Gadolinium Nitrate – Nitric Acid	3
4.0 Results and Discussion	4
4.1 Gadolinium Nitrate – Nitric Acid – Oxalic Acid	4
4.2 Gadolinium Nitrate – Nitric Acid	6
5.0 Conclusions	7
6.0 References	8

LIST OF TABLES

Table 3-1. Test Matrix Final Target Concentrations and Preparation Masses	2
Table 3-2. Calculated Starting Concentrations for Samples at 10 °C.....	3
Table 3-3. Test Conditions for HNO ₃ - GdNH Solubility at 20-22 °C.....	3
Table 4-1. Soluble Gadolinium and Oxalate Concentrations in 4-10 M HNO ₃ at 25 °C	4
Table 4-2. Soluble Gadolinium and Oxalate Concentrations in 4-8 M HNO ₃ at 10 °C	5
Table 4-3. Calculated Solution Concentrations after Addition of GdNH	6
Table 4-4. Solubility Data for GdNH in HNO ₃ at 20-22 °C.....	7

LIST OF FIGURES

Figure 2-1. Gadolinium Oxalate Solubility in Nitric Acid.....	1
Figure 4-1. Gadolinium Solubility as Functions of HNO ₃ and Oxalate at 25 °C.....	5
Figure 4-2. Gadolinium Solubility as Functions of HNO ₃ and Oxalate at 10 and 25 °C	6
Figure 4-3. GdNH Solubility as Functions of HNO ₃ at 20-22 °C	7

LIST OF ABBREVIATIONS

AD	Analytical Development
DI	de-ionized
GdNH	gadolinium nitrate hexahydrate
IC	ion chromatography
ICPES	inductively coupled plasma emission spectroscopy
NP>4	no precipitate after 4 days
NP>11	no precipitate after 11 days
NM	not measured
SRNL	Savannah River National Laboratory

1.0 Introduction

HB-Line will begin processing Pu solutions during FY2012 that will involve the recovery of Pu using oxalate precipitation and filtration. After the precipitation and filtration processes, the presence of excess oxalate and unfiltered Pu oxalate solids pose a criticality safety issue downstream of HB-Line. In previous campaigns, HB-Line performed an oxalate decomposition (or oxalate “kill”) step to prevent the transfer of excess oxalate and Pu oxalate solids to the non-geometrically favorable tanks within H-Canyon. The current flowsheet has H-Canyon receiving the filtrate solution with possible Pu solids into a tank that is poisoned with Gd in solution; decomposition of the oxalate will occur within a subsequent H-Canyon vessel.

The receipt of excess oxalate into the H-Canyon receipt tank has the potential to precipitate a portion of the Gd poison in the receipt tanks.^[1] The amount of Gd in solution determines the maximum amount of Pu solids that H-Canyon can receive. The current H-Canyon target is a minimum of 6 g/L Gd in solution at nitric acid (HNO₃) concentrations of 6-10 M. H-Canyon Engineering requested that SRNL determine the solubility of Gd in aqueous solutions of HNO₃ and oxalic acid (H₂C₂O₄) at 25 °C.^[2] A subsequent request was made to measure Gd oxalate solubility at 10 °C and Gd nitrate solubility at ambient temperature (~20 °C).^[3]

2.0 Background

The precipitation of lanthanides, such as Gd, has been studied at SRNL^[4] and solubility data are available in the literature.^[1,5] The research by Sarver and Brinton^[1] is of particular interest because it was conducted in 2-4 M HNO₃ and 0-0.25 M H₂C₂O₄. Figure 2-1 contains the data pertinent to this study. The SRNL data were collected at 4-10 M HNO₃ and 0.15-0.25 M H₂C₂O₄ to address the operating range for H-Canyon. Although the data at 4 M HNO₃ and 0.25 M H₂C₂O₄ do not meet the H-Canyon objectives, the trends as functions of HNO₃ and H₂C₂O₄ concentrations indicate that adequate solubility of Gd may occur at higher HNO₃ concentrations and lower H₂C₂O₄ concentrations.

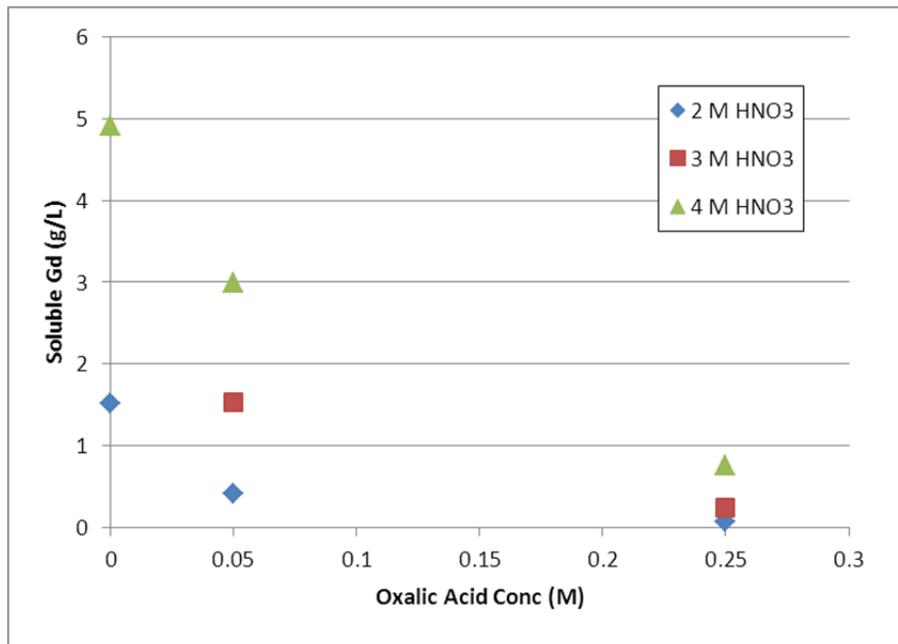


Figure 2-1. Gadolinium Oxalate Solubility in Nitric Acid^[1]

3.0 Experimental Procedure

3.1 Gadolinium Nitrate – Nitric Acid – Oxalic Acid

A solubility test matrix was established that varied the HNO₃ (4-10 M), H₂C₂O₄ (0.15-0.25 M), and Gd (4-12 g/L) concentrations. The target concentrations are listed in Table 3-1. Samples were prepared by adding the chemicals of Table 3-1 into 60-mL polyethylene bottles and diluting with de-ionized (DI) water to 25 mL. The 1 M H₂C₂O₄ was prepared by combining 55.046 g of DI water with 7.4427 g of H₂C₂O₄·2H₂O in a polyethylene bottle, adding a Teflon™-coated stir bar, and stirring at ambient temperature for 24 h (the final volume was 59 mL).

The bottles were capped and placed into a controlled-temperature water bath with a shaker table. The samples were maintained at 25±1 °C for the duration of the test and shaken periodically. The samples were regularly inspected for the formation of a precipitate.

After 30 h at temperature, only Sample 2 formed a precipitate. Consequently, 0.287 g of gadolinium nitrate hexahydrate (the equivalent of 4 g/L Gd) were added to the remaining nine samples. Therefore, Samples 1, 3, and 4 contained ~0.574 g of Gd(NO₃)₃·6H₂O [GdNH] (~8 g/L Gd), and Samples 5-10 contained ~0.861 g of GdNH (~12 g/L Gd). The final GdNH additions and Gd concentrations are shown in Table 3-1.

Table 3-1. Test Matrix Final Target Concentrations and Preparation Masses

Sample	Target Concentrations			Added and Diluted to 25 mL		
	HNO ₃ (M)	H ₂ C ₂ O ₄ (M)	Gd (g/L)	15.7 M HNO ₃ (mL)	1 M H ₂ C ₂ O ₄ (mL)	GdNH* (g)
1	4	0.15	8	6.37	3.75	0.574 [#]
2	4	0.25	4	6.37	6.25	0.287
3	6	0.15	8	9.55	3.75	0.574 [#]
4	6	0.25	8	9.55	6.25	0.574 [#]
5	6	0.15	12	9.55	3.75	0.861 [#]
6	6	0.25	12	9.55	6.25	0.861 [#]
7	8	0.15	12	12.74	3.75	0.861 [#]
8	8	0.25	12	12.74	6.25	0.861 [#]
9	10	0.15	12	15.92	3.75	0.861 [#]
10	10	0.25	12	15.92	6.25	0.861 [#]

* GdNH = gadolinium nitrate hexahydrate [Gd(NO₃)₃·6H₂O]
[#] An addition of 0.287 g of GdNH (~4 g/L Gd) occurred after 30 h

After 5 days, liquid subsamples were submitted to Analytical Development (AD) from Samples 1, 2, 4, 6, and 8 for analysis by inductively coupled plasma emission spectroscopy (ICPES) and ion chromatography (IC). After 7 days, a few crystals of H₂C₂O₄·2H₂O were added to Samples 3, 5, 7, and 9 to facilitate precipitation. After 11 days, liquid subsamples were withdrawn from Samples 3, 5, 7, 9, and 10 for analysis by ICPES and IC. The samples for each analysis contained ~1.8 mL.

Samples 1, 3, 5, and 7 were placed into a water bath at 10 °C for 4 days. After four days, liquid subsamples were withdrawn for analyses by ICPES and IC. Accounting for the removal of 3.6 mL of subsample of known concentration (Table 4-1) from Samples 1, 3, 5, and 7, the starting concentrations of those four samples were calculated (Table 3-2).

Table 3-2. Calculated Starting Concentrations for Samples at 10 °C

Sample	Starting Concentrations		
	HNO ₃ (M)	H ₂ C ₂ O ₄ (M)	Gd (g/L)
1	4	0.157	8.76
3	6	0.150	8.00
5	6	0.151	12.1
7	8	0.150	12.0

3.2 Gadolinium Nitrate – Nitric Acid

Four 20-mL solutions were prepared in 30-mL polyethylene bottles using calibrated pipettes – 4 M HNO₃, 6 M HNO₃, 8 M HNO₃, and 10 M HNO₃. At ambient temperature (20-22 °C), incremental additions of Gd(NO₃)₃·6H₂O were made to the bottles. The bottles were capped and shook for at least 30 min or until the Gd(NO₃)₃·6H₂O dissolved. Additions of Gd(NO₃)₃·6H₂O to the 4 M, 6 M, and 8 M HNO₃ solutions were discontinued before saturation was achieved. The amount of Gd(NO₃)₃·6H₂O added to each bottle is shown in Table 3-3. After additions were discontinued, the final solution volumes were measured for each bottle.

Table 3-3. Test Conditions for HNO₃- GdNH Solubility at 20-22 °C

HNO ₃ (M)	Initial Volume (mL)	GdNH Added (g)	Final Volume (mL)
4	20.0	16.40*	26.1*
6	20.0	16.13*	26.1*
8	20.0	13.71*	25.0*
10	20.0	12.49	24.8
* Did not achieve saturation			

The solutions prepared in 4 M, 6 M, and 8 M HNO₃ were uncapped, placed on a hot plate at 90 °C and gradually evaporated. The bottles were periodically removed from the hot plate, cooled to ambient temperature, and observed for the formation of a precipitate. If no precipitate was observed, the bottle was returned to the hot plate for further evaporation until precipitation occurred. The solutions were stored overnight at ambient temperature to confirm that saturation occurred. After all samples were saturated with GdNH, subsamples were collected from each solution for ICPES and total acid/free acid analyses.

4.0 Results and Discussion

4.1 Gadolinium Nitrate – Nitric Acid – Oxalic Acid

Of the ten samples prepared, only seven precipitated (determined by visual observation) with the presence of precipitate in Sample 5 considered questionable. The supernatant liquids were analyzed for Gd concentration by ICPEs. Those data are listed in Table 4-1. Based on the Gd data, the initial $\text{H}_2\text{C}_2\text{O}_4$ concentration, and the stoichiometry for the formation of gadolinium oxalate [$\text{Gd}_2(\text{C}_2\text{O}_4)_3$], the soluble concentrations of $\text{C}_2\text{O}_4^{2-}$ were calculated and are provided in Table 4-1. The soluble Gd and the calculated $\text{C}_2\text{O}_4^{2-}$ concentrations for 4-8 M HNO_3 are graphed along with the 1927 data from Sarver and Brinton^[1] in Figure 4-1.

The 4 M HNO_3 data for the current study exhibit good agreement with the data from the literature. The trend for the 6 M HNO_3 samples appears to follow that of the 2-4 M HNO_3 samples. Consequently, the formation of precipitate in Sample 5 would be borderline because it is near the projected solubility limit. Similarly, precipitates in Samples 3, 7, and 9 would not be expected. Analyses of the oxalate concentrations (Table 4-1) agree with the calculated values within analytical uncertainty for all samples except Sample 10.

Table 4-1. Soluble Gadolinium and Oxalate Concentrations in 4-10 M HNO_3 at 25 °C

Sample	HNO_3 (M)	$\text{H}_2\text{C}_2\text{O}_4$ (M)	Gd (g/L)	Days to Precipitate	Soluble Gd (g/L)*	Soluble $\text{C}_2\text{O}_4^{2-}$ (M) [Calc / Measure*]
1	4	0.15	8	4	3.47	0.107 / 0.109
2	4	0.25	4	1	1.03	0.222 / NM
3	6	0.15	8	NP>11	8.05	0.150 / 0.151
4	6	0.25	8	4	7.18	0.242 / 0.232
5	6	0.15	12	11	11.5	0.144 / 0.131
6	6	0.25	12	4	7.86	0.211 / 0.207
7	8	0.15	12	NP>11	12.0	0.150 / NM
8	8	0.25	12	4	11.5	0.245 / NM
9	10	0.15	12	NP>11	11.9	0.150 / NM
10	10	0.25	12	5	12.2	0.252 / 0.144

* Measurement uncertainty for ICPEs and IC was 10%
 NP>11 = no precipitate after 11 d
 NM = not measured

For Sample 10, the Gd measurement of 12.2 g/L does not agree with the observation that a precipitate formed after five days. If the measured value for $\text{C}_2\text{O}_4^{2-}$ was used to calculate the corresponding Gd concentration, the calculated Gd concentration would be 1.0 g/L, which is also not consistent with the observed precipitation behavior. It is not known if the high HNO_3 concentration is having a negative impact on the sample (i.e., decomposition of $\text{H}_2\text{C}_2\text{O}_4$).

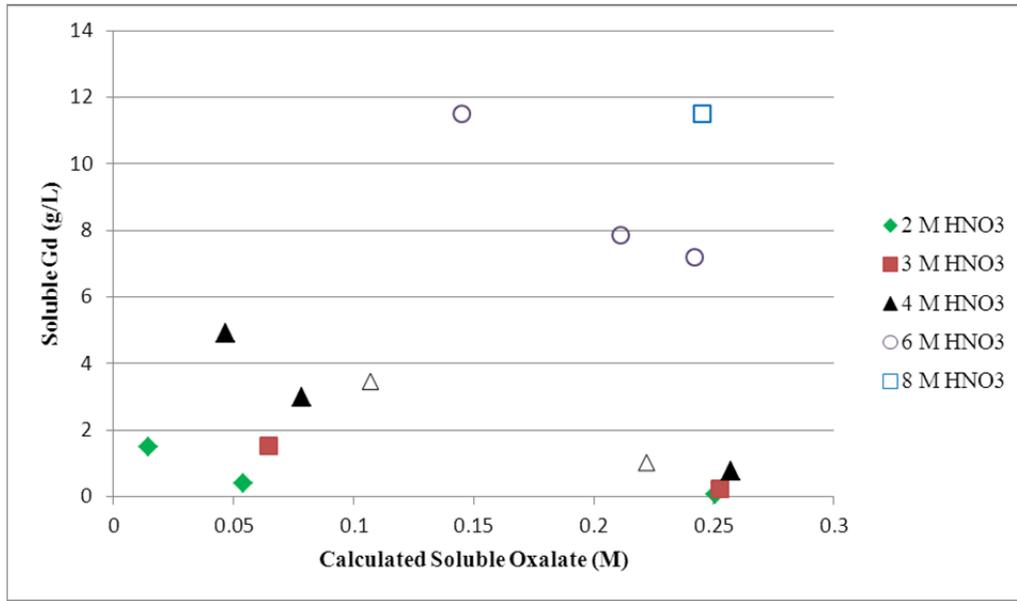


Figure 4-1. Gadolinium Solubility as Functions of HNO₃ and Oxalate at 25 °C

Interpolation of the 4 M HNO₃ data in Figure 4-1 indicates that, for the system containing only Gd, H₂C₂O₄, and HNO₃, 2.5 g/L and 0.8 g/L Gd are soluble in 0.15 M and 0.25 M oxalate, respectively. Similarly, at 6 M HNO₃, 11 g/L and 7 g/L Gd are soluble in 0.15 M and 0.25 M oxalate, respectively. The increase in solubility from 4 M to 6 M HNO₃ was expected, but the magnitude of the increase was not anticipated. The solubility of Gd at 8-10 M HNO₃ is greater than the solubility at 6 M HNO₃. It should be noted that the addition of other cations, such as iron, would create competition with Gd for oxalate and would thus likely increase Gd solubility.^[6] Plutonium(III) shows similar behavior to that of Gd, but the data are too few to draw direct comparisons.^[7]

The solubility data measured at 10 °C are listed in Table 4-2. The measured Gd data which exhibit a decrease in Gd for Samples 1 and 5 and not Sample 3 and 7 agree with the observed precipitation behavior. A comparison of the 10 °C and 25 °C data is shown in Figure 4-2. The 10 °C samples, as expected, have a lower solubility than the 25 °C samples. The difference in solubility is outside of the method uncertainty of 10%. Based on Figure 4-2, the solubility of Gd at 4 M HNO₃ with 0.15 M oxalate at 10 °C is about 1.5 g/L. Similarly, in 6 M HNO₃, the solubility of Gd with 0.15 M oxalate at 10 °C is about 10 g/L.

Table 4-2. Soluble Gadolinium and Oxalate Concentrations in 4-8 M HNO₃ at 10 °C

Sample	HNO ₃ (M)	H ₂ C ₂ O ₄ (M)	Gd (g/L)	Days to Precipitate	Soluble Gd (g/L)*	Soluble C ₂ O ₄ ²⁻ (M)* [Calc / Measure]
1	4	0.157	8.76	1	2.02	0.093 / 0.102
3	6	0.150	8.00	NP>4	7.97	0.150 / 0.156
5	6	0.151	12.1	1	10.6	0.137 / 0.137
7	8	0.150	12.0	NP>4	12.2	0.152 / 0.145

* measurement uncertainty for ICPES and IC was 10%
NP>4 = no precipitate after 4 d

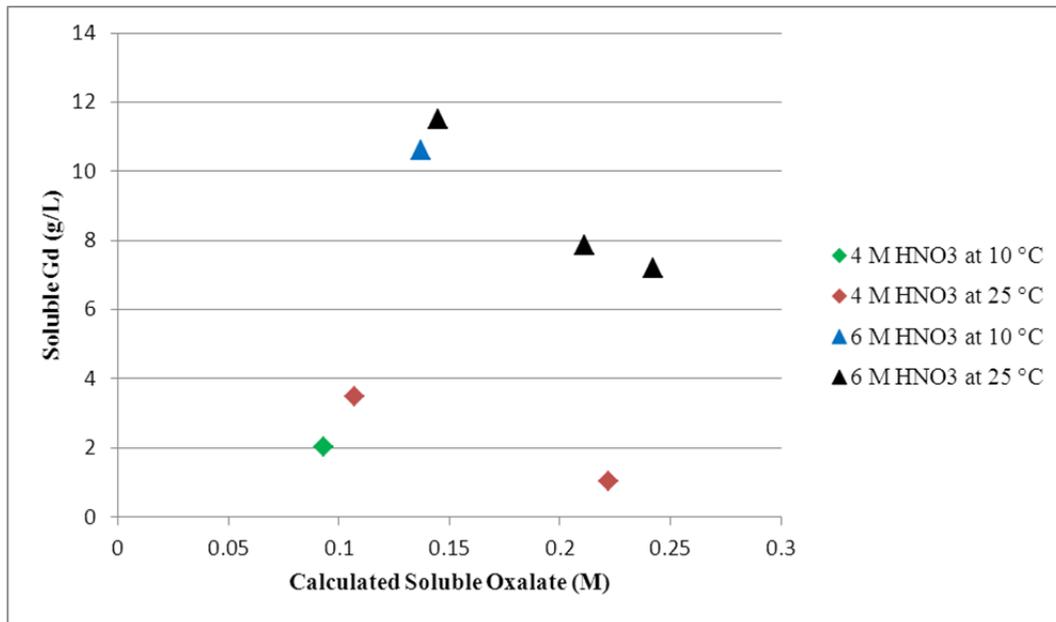


Figure 4-2. Gadolinium Solubility as Functions of HNO₃ and Oxalate at 10 and 25 °C

4.2 Gadolinium Nitrate – Nitric Acid

After the addition of GdNH to the four solution concentrations, only one sample (10 M HNO₃) contained undissolved GdNH, indicating it was saturated. Based on the initial volume, final volume, and mass of GdNH added, the final Gd and HNO₃ concentrations for each sample were calculated. The calculated values are provided in Table 4-3.

Table 4-3. Calculated Solution Concentrations after Addition of GdNH

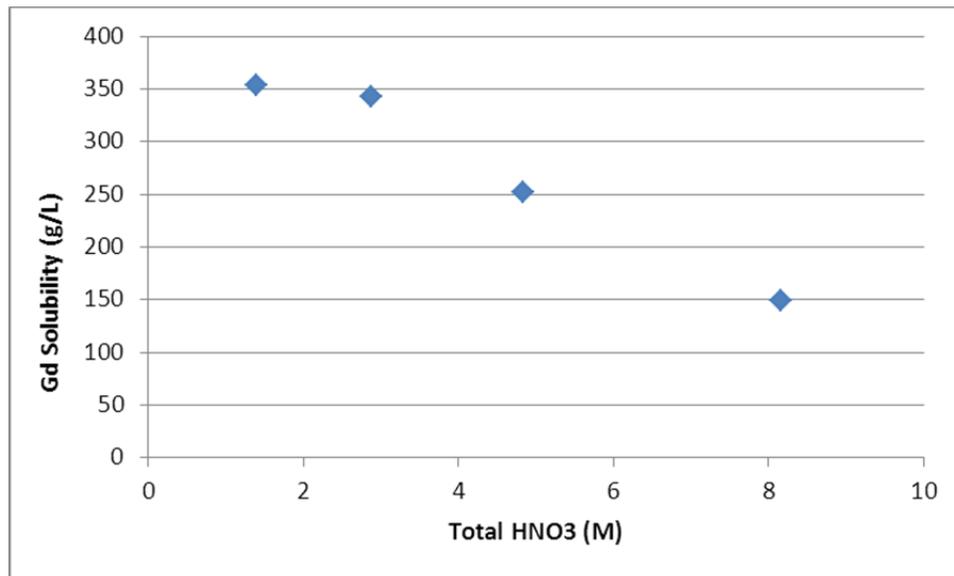
HNO ₃ (M)	Initial Volume (mL)	GdNH Added (g)	Final Volume (mL)	Final Gd (g/L)	Final HNO ₃ (M)
4	20.0	16.40	26.1	219	3.1
6	20.0	16.13	26.1	215	4.6
8	20.0	13.71	25.0	191	6.4
10	20.0	12.49	24.8	<175*	8.1

* Saturated solution

After the 4 M, 6 M, and 8 M HNO₃ solutions were evaporated to saturation, all samples were analyzed for Gd and HNO₃ concentrations. Those data are shown in Table 4-4. The uncertainty for the Gd and HNO₃ data is 10%. The total acid and Gd data are plotted in Figure 4-3. The solubility of Gd appears to be linear as a function of HNO₃ from 343 g/L Gd in 2.88 M HNO₃ to 149 g/L in 8.16 M HNO₃; Gd solubility appears to approach a limit below 2.88 M HNO₃, although there are no data below 1.40 M HNO₃. The behavior above 8.16 M is not known.

Table 4-4. Solubility Data for GdNH in HNO₃ at 20-22 °C

Initial HNO ₃ (M)	Free H ⁺ (M)	Total H ⁺ (M)	Gd at Saturation (g/L)
4	1.08	1.40	353
6	1.91	2.88	343
8	4.74	4.84	252
10	7.72	8.16	149
ICPES and acid measurement uncertainties are 10%			

**Figure 4-3. GdNH Solubility as Functions of HNO₃ at 20-22 °C**

5.0 Conclusions

SRNL measured gadolinium oxalate solubility at 4-10 M HNO₃, 4-12 g/L Gd, and 0.15-0.25 M oxalate at 10 °C and 25 °C. At 25 °C, solubility data for 4 M HNO₃ show good agreement with data in the literature. The data indicate that the target of 6 g/L soluble Gd can be achieved above 6 M HNO₃ and below 0.25 M H₂C₂O₄. For 0.15 M oxalate, 2.5 g/L and 11 g/L Gd are soluble in 4 M and 6 M HNO₃, respectively. For 0.25 M oxalate, 0.8 g/L and 7 g/L Gd are soluble in 4 M and 6 M HNO₃, respectively. The increase in Gd solubility from 4 M to 6 M HNO₃ was expected, but the magnitude of the increase in solubility was greater than expected. The solubility of Gd at 8-10 M HNO₃ exceeds the solubility at 6 M HNO₃.

The solubility of Gd in 4 M HNO₃ and 0.15 M oxalate at 10 °C is about 1.5 g/L. For 6 M HNO₃ and 0.15 M oxalate, the solubility of Gd at 10 °C is about 10 g/L.

Gadolinium nitrate is very soluble in HNO₃. The solubility of Gd exhibits linearity as a function of HNO₃ from 343 g/L Gd in 2.88 M HNO₃ to 149 g/L in 8.16 M HNO₃. Below 2.88 M HNO₃, the solubility of Gd approaches a limit of about 360 g/L, although no data exist below 1.40 M HNO₃.

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