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Chemical Technology Division

CATALYZED REDUCTION OF NITRATE IN AQUEOUS SOLUTIONS

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EXECUTIVE SUMMARY

The presence of sodium nitrate and other nitrate salts in wastes is a major source of difficulty for permanent disposal. A chemical reduction of nitrate using aluminum metal has been demonstrated,¹ but NH_3 , hydrazine, or organic compounds containing oxygen would have important advantages for reduction of nitrate in sodium nitrate solutions. The objective of this seed money study was to determine the minimum conditions for reduction reactions. The proposed procedure was batchwise heating of aqueous solutions in closed vessels with monitoring of temperatures and pressures.

A simple and convenient apparatus and procedure was demonstrated for observation of the formation of gaseous products and the collection of samples for analyses. The test conditions were limited to 250°C and ~ 1000 psi maximum as a result of the pressure rating of system components.

We did not find any useful reduction of sodium nitrate to sodium hydroxide as the primary product. The nitrate present at pHs < 4 as HNO_3 or NH_4NO_3 is easily decomposed, and the effect of nitromethane at these low pHs (as reported in U.S. Patent 4,225,455)² was confirmed. When acetic acid or formic acid was added, 21 to 56% of the nitrate in sodium nitrate solutions was reduced by methanol or formaldehyde to give sodium acetate, sodium carbonate, or sodium formate as products. With hydrazine and acetic acid, 73% of the nitrate was decomposed to convert NaNO_3 to sodium acetate. With hydrazine and formic acid, 36% of the nitrate was decomposed. If these compounds are more acceptable for final disposal than sodium nitrate, the reagents are cheap and the conversion conditions would be practical for easy use. The use of ammonium acetate or formate salts did not give significant reductions of the nitrate in sodium nitrate solutions.

1. INTRODUCTION

The presence of nitrate salts in wastes is a major source of difficulty for permanent disposal. Inorganic nitrates are soluble and are very difficult to immobilize so that leaching will not occur. After the tank wastes at U.S. Department of Energy (DOE) plant sites are treated to remove the predominant radionuclides, then the NaNO_3 and KNO_3 are controlling requirements for disposal of the bulk of the wastes. Thermal decompositions of waste

with high NaNO_3 — KNO_3 contents require excessive temperatures ($>1000^\circ\text{C}$) and give viscous melts with poor properties for transfer or disposal.

One type of disposal treatment to lower nitrates concentration is chemical reductions. For acidic solutions, chemical reductions have been demonstrated and applied using organic compounds containing oxygen (formaldehyde, sugar, methanol, etc.), ammonia, and hydrazine. These reducing agents give high rates of reaction for high nitric acid concentrations and much lower or zero rates in caustic solutions.

One treatment currently recommended for caustic waste solutions is the reduction of NaNO_3 and KNO_3 by aluminum metal to give NH_3 and a mixture of hydrous aluminum oxides, NaOH , and sodium aluminates.¹ Experimental results show that an excess of aluminum metal is required as it also reduces water to form hydrogen. The aqueous product slurry can be calcined to convert the sodium aluminates to insoluble Al_2O_3 and NaOH . These experimental results show that 1 kg of NaNO_3 will produce about 3.5 kg of $\text{Al}_2\text{O}_3 + \text{NaOH}$. Control and safe operation of the process require the controlled removal of the large heat of reaction and disposal of NH_3 and H_2 .

For the following reasons, either NH_3 , hydrazine, or organic compounds containing oxygen would have important advantages for the reduction of nitrate in caustic solutions:

1. They are much cheaper than aluminum metal.
2. They do not reduce water to H_2 and nitrate to NH_3 ; the H_2 and NH_3 are disposal problems as well as wasted reduction capacity.
3. The amount of nonvolatile products is much smaller. They could convert 1 kg of NaNO_3 to 0.47 kg of NaOH , while aluminum metal gives 3.5 kg of $\text{NaOH} + \text{Al}_2\text{O}_3$.
4. A smaller heat of reaction is favorable to easier control of the process reactions.

There are no experimental data to show practical reductions of NaNO_3 to NaOH by ammonia, hydrazine, or organic compounds. When acidic solutions were reacted, the reductions stopped before the boiling solutions became caustic.

One important experimental result (U.S. Patent 4,225,455) shows much faster and more complete decompositions of NH_4NO_3 solutions or $\text{UO}_2(\text{NO}_3)_2$ solutions when nitromethane was added.² The demonstrated reactions were $>99\%$ of the NH_4 in NH_4NO_3 — HNO_3 and 98 to 99% reduction of the nitrate in $\text{UO}_2(\text{NO}_3)_2$ — NH_4NO_3 solution. The first solution remained acidic (from excess HNO_3), and the second showed a final pH of 3.1.

The minimum temperatures to start the reactions decreased about 80°C (from ~190 to ~110°C). One possible explanation for this now efficient reaction is that the nitromethane might be a catalyst. Another explanation would be that it initiated a chain reaction, but this does not fully explain why the reactions were much less complete without nitromethane.

Thermochemical considerations. Thermodynamic data can be applied for reduction reactions to determine the thermodynamic feasibility. The reductions of nitrate and the oxidations of organics can give many different products; a failure to consider the more favorable reactions can lead to false conclusions. Thirteen reactions of interest to this discussion are listed along with the free energies of reaction at 25°C in kJ/mol NaNO_3 (Table 1). The NAC process reduction of NaNO_3 in caustic solution¹ can be represented by Eq. (1). The nonproductive reduction of water is less energetic for the same amount of Al [Eq. (2)], but is also a very exothermic reaction. For the chemical reducing compounds considered in this study, the free energies of reaction are more favorable (more negative) in the order of ammonium salts, methyl alcohol, formaldehyde, and hydrazine. The difference in free energies for Na_2CO_3 vs sodium formate as products is small [see Eqs. (8) and (8A)]. Reactions (6) and (7) are not very favorable, and the high vapor pressure of NH_3 over hot caustic solutions is unfavorable for reaction (3).

2. EXPERIMENTAL APPARATUS

The experimental apparatus and procedures were designed to contain small batches of reactants and products under pressure with indications of the pressures and temperatures during heatup in a furnace to 250°C maximum. The system components were selected to allow pressures up to 1800 psi. The system included a rupture disk rated for 1200 psi. The vapor pressure of water at 250°C is 580 psia. The amounts of solution and reactants as compared to the total volume were selected to ensure a residue of solution at all test conditions and pressures <1000psi for complete reduction of all nitrate to gaseous products.

The apparatus is shown as a schematic diagram (Fig. 1) and a photograph (Fig. 2). The reactor was a 304 stainless steel gas cylinder rated at 1800 psi at -320 to 600°C. This cylinder had a capacity of 150 mL and 1/4-in. NPS female pipe threads in each end. An

Table 1. Reactions for chemical reductions of nitrate in NaNO₃ solutions

Equation No.	Chemical equation	Free energy of reaction at 298 K (kJ/mol NaNO ₃)
(1)	$6\text{NaNO}_3 + 16\text{Al} + 12\text{H}_2\text{O} \rightarrow 6\text{NaOH} + 8\text{Al}_2\text{O}_3 + 6\text{NH}_3$	-1673
(2)	$2\text{Al} + 3\text{H}_2\text{O} \rightarrow \text{Al}_2\text{O}_3 + 3\text{H}_2$	-1134 (per 16/6 Al)
(3)	$\text{NaNO}_3 + \text{NH}_4\text{OH} \rightarrow \text{NaOH} + \text{N}_2\text{O} + \text{H}_2\text{O}$	-162
(4)	$2\text{NaNO}_3 + \text{CH}_3\text{OH} \rightarrow \text{Na}_2\text{CO}_3 + 2\text{NO} + 2\text{H}_2\text{O}$	-216
(5)	$2\text{NaNO}_3 + 2\text{HCHO} \rightarrow \text{Na}_2\text{CO}_3 + \text{N}_2\text{O} + 2\text{H}_2\text{O} + \text{CO}_2$	-425
(6)	$\text{NaNO}_3 + \text{NH}_4(\text{CHO}_2) \rightarrow \text{Na}(\text{CHO}_2) + \text{N}_2\text{O} + 2\text{H}_2\text{O}$	-110
(7)	$\text{NaNO}_3 + \text{NH}_4(\text{C}_2\text{H}_3\text{O}_2) \rightarrow \text{Na}(\text{C}_2\text{H}_3\text{O}_2) + \text{N}_2\text{O} + 2\text{H}_2\text{O}$	-128
(8)	$2\text{NaNO}_3 + \text{CH}_3\text{OH} + 2\text{HCOOH} \rightarrow \text{Na}_2\text{CO}_3 + 2\text{NO} + 4\text{H}_2\text{O} + 2\text{CO}$	-244
(8A)	$2\text{NaNO}_3 + \text{CH}_3\text{OH} + 2\text{HCOOH} \rightarrow 2\text{Na}(\text{CHO}_2) + 2\text{NO} + 3\text{H}_2\text{O} + \text{CO}_2$	-268
(9)	$2\text{NaNO}_3 + \text{CH}_3\text{OH} + 2\text{CH}_3\text{COOH} \rightarrow 2\text{Na}(\text{C}_2\text{H}_3\text{O}_2) + 2\text{NO} + 3\text{H}_2\text{O} + \text{CO}_2$	-267
(10)	$2\text{NaNO}_3 + 2\text{HCHO} + 2\text{HCOOH} \rightarrow \text{Na}_2\text{CO}_3 + \text{N}_2\text{O} + 4\text{H}_2\text{O} + \text{CO}_2 + 2\text{CO}$	-477
(11)	$2\text{NaNO}_3 + 2\text{HCHO} + 2\text{CH}_3\text{COOH} \rightarrow 2\text{Na}(\text{C}_2\text{H}_3\text{O}_2) + \text{N}_2\text{O} + 3\text{H}_2\text{O} + 2\text{CO}_2$	-477
(12)	$4\text{NaNO}_3 + 5\text{N}_2\text{H}_5\text{OH} \rightarrow 4\text{NaOH} + 7\text{N}_2 + 13\text{H}_2\text{O}$	-680
(13)	$4\text{NaNO}_3 + 5\text{N}_2\text{H}_5\text{OH} + 4\text{CH}_3\text{COOH} \rightarrow 4\text{Na}(\text{C}_2\text{H}_3\text{O}_2) + 7\text{N}_2 + 17\text{H}_2\text{O}$	-760

- a GAS CYLINDER (304 STAINLESS STEEL)
MATHESON MODEL 4HD150 (RATED FOR 1800 psi)
- b RUPTURE DISK (1200 psi)
- c PRESSURE GAGE (0-1000)
- d PRESSURE TRANSDUCER
- e THERMOCOUPLE
- f TWO-PEN RECORDER (STRIP CHART)
- g INSULATION (TOP-SPLIT; BOTTOM-ATTACHED WITH HOLE)

Fig. 1. Equipment for studies of reduction of nitrate in solutions.

Fig. 2. A photograph of the CRON apparatus.

initial aqueous solution volume of 30 to 40 mL and 80 to 100 mmol maximum of nitrate were selected to meet the criteria given above. A pressurization to 60 to 90 psig with N_2 was selected to give a low partial pressure of O_2 and to fill the connections outside the furnace with noncondensable gases at the maximum test pressures. The 30 to 40 mL of solution was adequate for easy material balances and samples for analyses or titration.

3. INTERPRETATION OF RESULTS TO SHOW REACTIONS

The chemical reactions are indicated by several different results. The experimental measurements included the amounts of material charged to the test, the amounts of material removed after cooldown to room temperatures, the power setting for the furnace, the temperatures and the pressures throughout the test, and chemical analyses of the product solution. The most useful indications of chemical reactions were as follows:

1. The product solutions were analyzed for nitrate and sodium. The nitrate-to-sodium ratio, as compared to that in the charge, indicated what fraction of the nitrate was unreacted. This comparison was better than a nitrate material balance because the ratio would be independent of the weight or volume losses as gaseous products, leaks, or incomplete recovery. The sodium analyses were compared with material balance results as a check for gross errors.
2. The formation of gaseous products was indicated by the pressure in excess of the pressure for a test without reaction. The best indications were for test conditions that were otherwise as identical as possible. The most common conditions used were about 45 g of solution charge, N_2 to 82 psig at $22^\circ C$, furnace power setting of 29%, placement of insulation to give reproducible openings or gaps, and temperatures and pressures read from indicators (a pressure gage and a digital temperature indicator for the thermocouple in the well exposed to solution).

The most accurate and sensitive excess pressures were from comparisons at the same temperature (Figs. 3 and 4). Comparisons at the same run time after the start of furnace power was less accurate because the heat from chemical reactions and the heat losses through gaps in the insulation were variable.

Fig. 3. Excess pressures from gaseous products of reactions.

Fig. 4. Additional excess pressures from gaseous products of reactions.

3. The solution weight loss was a measure of the weight of volatile products. This indication had two serious errors: losses as leaks or incomplete recovery and reactions to give volatile products without reaction of nitrate. For example, sodium formate can decompose to form sodium carbonate, carbon dioxide, and hydrogen without any reduction of nitrate.
4. The amount of gas minus the gas charged was a measure of the amount of reaction. Calculations to correct for vapor pressures and temperature would be difficult. The excess pressure as described above was equivalent to an empirical vapor pressure and temperature correction. The pressure after cooldown to room temperature did not require such corrections, but small leaks were a serious source of errors. As mentioned above, decomposition of sodium formate or other reactions can also give gaseous products.

Some other possible indications of chemical reactions were not very useful. The heat effects of the chemical reactions were usually small compared to the heat input from the furnace and the heat capacity of the reactor vessel. Very high rates of reaction were detectable as blips on the temperature recorder chart, but temperature effects were otherwise not useful. For test conditions intended to convert sodium nitrate to sodium hydroxide, product solution samples were titrated with HCl solutions. Tests with blanks indicated that graphs of pH vs amounts of HCl would give measures of the amounts of NaOH, Na_2CO_3 , and NaHCO_3 present. The product pHs and the titrations both showed negligible conversions of NaNO_3 to NaOH or Na_2CO_3 . The test conditions with mixtures of organic acids, ammonium compounds, and methyl alcohol or formaldehyde introduced too many variables to allow identification of products by titration.

4. DISCUSSION OF RESULTS

The charge variables for the tests were the reducing constituents (CH_3OH , HCHO , sugar, NH_4^+ , and $\text{N}_2\text{H}_5\text{OH}$); the absence or presence of CH_3NO_2 or a platinum gauze as catalysts; and the presence or absence of acetate, formate, or phosphate to form their sodium salts as products. In the absence of the acetate, formate, or phosphate, the sodium products after reduction of nitrate would be caustic (either NaOH or Na_2CO_3) solutions.

The chemical analyses of product solutions were the most important verification of nitrate destruction (Table 2). The excess pressure was a good measure of the formation of gaseous products (Table 3), but the amount of gases varied with the products of nitrate reduction (N_2 , N_2O , NO , etc.) and with other reactions, such as the decomposition of sodium formate or ammonium salts. Near room temperature, high pressures of CO_2 gave large solubilities in the solution. The weight losses commonly showed about 25% more reaction of nitrate than the chemical analyses or excess gas pressures (Tables 2 and 3). These differences also occurred for tests with no reaction and were probably the losses (~ 1 g/test) as vapor, leaks, and unrecovered solution.

Most of the test results that showed reductions of nitrate by chemical analyses (Table 2) gave product solution pHs of 3.62 to 4.99 (Table 3). These reductions of nitrate were by CH_3OH , $HCHO$, or N_2H_5OH with some acetic or formic acid in solutions. Test results showed that sodium acetate was stable and that there was no reduction of nitrate by ammonium acetate—acetic acid. For $NaNO_3$ —ammonium formate—formic acid solution, chemical analyses showed a small (perhaps not significant) loss of nitrate, but gaseous products were formed — probably by decomposition of formate.

The catalyzed decomposition of NH_4NO_3 to water and N_2O in acidic solutions was confirmed. But ammonium acetate—acetic acid or ammonium formate—formic acid did not reduce $NaNO_3$ to form the acetate or formate salts. Combinations of CH_3OH or $HCHO$ with acetic acid or formic acid gave 21 to 56% reductions of the nitrate in sodium nitrate solutions (Table 2). Both CH_3OH and $HCHO$ showed detectable rates of reaction at starting temperatures of 120 to 150°C and high rates at 150 to 160°C. The catalytic effects of CH_3NO_2 or Pt were not observed but were only tested for one set (CH_3OH —acetic acid) of these charge compositions.

The tests with hydrazine show more reduction of the nitrate in sodium nitrate solutions than the reductions by CH_3OH or $HCHO$. Hydrazine alone gave 29% reductions of the nitrate. The product was probably $NaOH$. Hydrazine and acetic acid gave 73% reduction of nitrate and a product solution pH of 6.70. Hydrazine and formic acid gave similar formation of gaseous products, but only 36% reduction of the nitrate. Most of the gas was probably from decomposition of sodium formate.

The sugar and phosphates have undesirable characteristics that make them less promising than the alternate reducing compounds. The sugar easily decomposes or

Table 2. A summary of major test results for reductions of nitrate in aqueous solutions

Test No.	Equation from Table 1	Charge NO_3^-/Na ratio (mol/mol)	Charge pH	Reducing compound	Intended sodium product	Catalyst present	NO_3^- reduced from analyses (%)	Comments
16		2.09	6.6	CH_3OH	Na_2CO_3	CH_3NO_2	46	HNO_3 and NH_4NO_3 decomposed
40	(13)	1.00	6.14	$\text{N}_2\text{H}_5\text{OH}$	$\text{NaC}_2\text{H}_3\text{O}_2$	CH_3NO_2	73	
32	(5)	0.88	3.74	HCHO	Na_2CO_3	None	56	
26	(9)	1.00	1.78	CH_3OH	$\text{NaC}_2\text{H}_3\text{O}_2$	Pt, CH_3NO_2	40	
41		1.00	5.56	$\text{N}_2\text{H}_5\text{OH}$	Na_2CO_3	CH_3NO_2 , Pt	36	
35	(11)	1.00	1.67	HCHO	$\text{NaC}_2\text{H}_3\text{O}_2$	CH_3NO_2	32	
39	(12)	1.00	10.0	$\text{N}_2\text{H}_5\text{OH}$	NaOH	CH_3NO_2	29	
27	(9)	1.00	1.81	CH_3OH	$\text{NaC}_2\text{H}_3\text{O}_2$	None	26	
34	(8A)	1.00	1.11	CH_3OH	NaCHO_2	None	22	
37	(10)	0.94	1.84	HCHO	Na_2CO_3	None	21	
33	(6)	1.00	4.12	NH_4CHO_2	NaCHO_2	CH_3NO_2	≤ 5	
31	(5)	1.00	1.76	HCHO	Na_2CO_3	None	0	NaCHO_2 decomposed
30	(7)	1.00	5.8	$\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$	$\text{NaC}_2\text{H}_3\text{O}_2$	CH_3NO_2	0	No gas formation
36		0	6.16	None	$\text{NaC}_2\text{H}_3\text{O}_2$			No NaNO_3
29		1.00	6.55	None	NaNO_3	None	0	NaNO_3 only
28	(7)	1.00	5.55	$\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$	$\text{NaC}_2\text{H}_3\text{O}_2$	Pt	0	No gas formation

Table 3. Complete product measurements for selected CRON tests

Test No.	Chemical analyses		Product pH	Excess gas pressure			Calculated NO ₃ ⁻ reduced from excess pressure (%)	Calculated NO ₃ ⁻ reduced from weight loss (%)
	NO ₃ ⁻ /Na (mol/mol)	Na out/Na in (%)		(psi)	Initial temp (°C)	Maximum reaction (°C)		
16	1.13	Leak	4.81	305	100	130	44	Leak
26	0.60	98	4.63	> 150	140	150	33	46
27	0.74	104	4.46	> 150	130	150	49	58
28	1.01	112	5.01	0	None		0	35
29	0.98		8.45	0	None		0	<0
30	0.96	110	5.00	0	None		0	34
31	1.08	92	5.39	160	150	200	69	100
32	0.39	92	4.99	135	150	190	55	95
33	0.95	95	8.26	> 200	195	230	85	77
34	0.78	95	3.62	> 200	120	160	69	93
35	0.68	101	4.19	160	150	160	49	73
36			6.09	0	None		0	~0
37	0.74	95	3.56	350	130	180	68	84
39	0.71	100	12.70	> 200	220	240	53	100
40	0.27	99	6.70	300	160	240	79	87
41	0.64	116	9.63	255	180	240	~50	80

caramelizes to give black solids or solutions. The sodium phosphates have lower solubilities than most other sodium salts. Simple trisodium phosphate is strongly alkaline and would be a product at high pHs only. Other sodium salts like NaH_2PO_4 or salts of pyro- or triphosphoric acid have low sodium contents and are probably undesirable as components of final waste forms. Some tests with ammonium phosphates did not show any reductions of nitrate.

4.1 EXAMPLES OF RESULTS

The measurements and calculations are illustrated by a set of examples. The first 25 tests were equipment checks or tests of caustic feed with no significant reactions, or the test results were compromised by equipment leaks. The examples are, therefore, mostly from Tests 26 through 37.

Test CRON 26. The feed contained 98 mmol of NaNO_3 , 202 mmol of CH_3OH , 140 mmol of acetic acid, 0.18 g of CH_3NO_2 , and 1.8 g of Pt gauze. The feed charged was 45.02 g, pH = 1.78.

The product solution was 42.78 g and pH of 4.63. The solution analyses showed NO_3^-/Na of 0.60 mol/mol and 96 mmol of Na or a decomposition of 40% of the nitrate.

The excess pressure data (Fig. 3) show that volatile products were formed about 38 to 100 min at temperatures of 140 to 230°C. The maximum rate of gas formation was at about 150°C.

This intended primary reaction was Eq. (9) (Table 1). The observed weight loss of 2.24 g gives a calculated nitrate decomposition of 4.5 mmol or 46%.

The excess gas pressure (Fig. 4) indicated about 49 mmol of gas, or a nitrate decomposition of 33%.

Test CRON-30. The feed contained 107 mmol of NaNO_3 , 153 mmol of ammonium acetate, 30 mmol of CH_3OH , and 26 mmol of acetic acid. The total weight of feed charge was 46.6 g, and the pH was about 5.8.

The product was 45.15 g, pH = 5.00, and had a NO_3^-/Na ratio of 0.96 mol/mol. The NO_3^-/Na ratio and a nitrate material balance both show near-zero decomposition of nitrate.

The intended primary reaction was Eq. (7) (Table 1). The observed weight loss would indicate a decomposition of about 33 mmol of nitrate, or 31%.

The excess pressure data (Fig. 4) show no formation of gaseous products.

Test CRON-32. The feed contained 98 mmol of Na and 86 mmol of nitrate for a NO_3^-/Na ratio of 0.88. The feed also contained 22 mmol of formic acid, 130 mmol of HCHO, and water to a total weight of 42.06 g and a pH of 3.74.

The product solution was 38.45 g, pH = 4.99, and had a NO_3^-/Na ratio of 0.39. The solution analyses showed 90 mmol of sodium.

The excess pressure data (Fig. 3) showed that volatile products were formed from about 40 to 85 min or 150 to 210°C. The maximum rate of gas formation was at about 190°C.

The intended primary reaction was Eq. (5) (Table 1). The observed weight loss of 3.61 g gives a calculated nitrate decomposition of 82 mmol, or 95%.

The excess gas pressure indicated about 47 mmol of gas, or a nitrate decomposition of 55%.

5. REFERENCES

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