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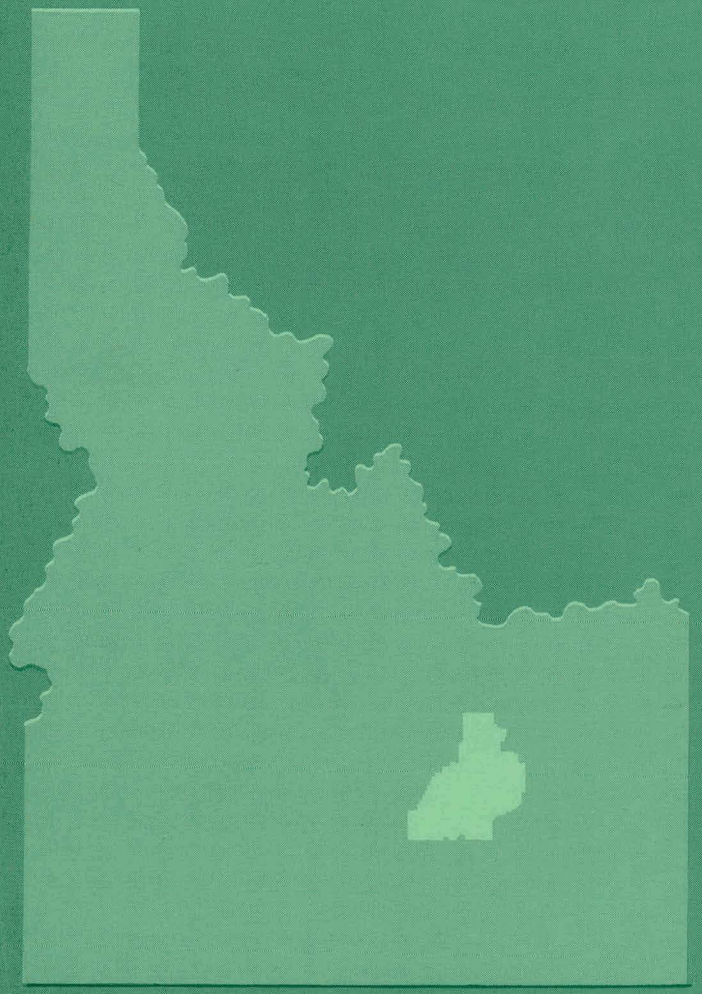
THERMAL DECOMPOSITION OF HYDRATED IRON, CHROMIUM, AND NICKEL NITRATES AND THEIR MIXTURES

E. M. Vander Wall



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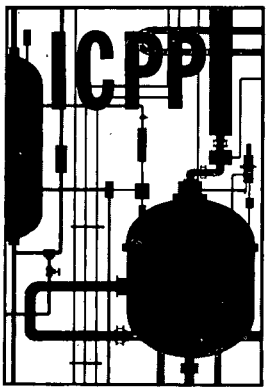

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THERMAL DECOMPOSITION OF HYDRATED IRON, CHROMIUM, AND NICKEL NITRATES AND THEIR MIXTURES

E. M. Vander Wall

A B S T R A C T

The thermal decomposition of ferric nitrate nonahydrate, chromium nitrate nonahydrate, aluminum nitrate nonahydrate, and nickel nitrate hexahydrate as well as the mixtures of these compounds in the proportions produced in the dissolution of Nichrome and stainless steel alloys was investigated. The experiments were conducted in air at two heating rates, approximately 1.1°C and 2.6°C per minute. The intermediate products and final products were analyzed by X-ray diffraction and chemically where it was appropriate.

THERMAL DECOMPOSITION OF HYDRATED IRON, CHROMIUM, AND NICKEL NITRATES AND THEIR MIXTURES

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THERMAL DECOMPOSITION OF HYDRATED IRON, CHROMIUM, AND NICKEL NITRATES AND THEIR MIXTURES

E. M. Vander Wall

I. SUMMARY

The thermal decomposition of ferric nitrate nonahydrate, chromium nitrate nonahydrate, aluminum nitrate nonahydrate, and nickel nitrate hexahydrate, as well as the mixtures of these compounds in the proportions produced in the dissolution of Nichrome and stainless steel alloys, was investigated. All the experiments were conducted in air at two heating rates, approximately 1.1°C and 2.6°C per minute. Chromium nitrate nonahydrate forms an intermediate mixed oxide with an empirical formula $CrO_{2.2}$ before decomposing completely to the chromic oxide. Nickel nitrate hexahydrate forms several intermediate products during decomposition to nickel oxide, with the principal intermediate occurring at 250°C with the above heating rates, and its empirical formula is $Ni(NO_3)(OH)\cdot\frac{1}{2}H_2O$. X-ray patterns were obtained for these intermediates.

The only crystalline material formed by the simulated Nichrome mixture heated to 450°C was nickel oxide. The stainless steel mixture was prevented from forming any crystalline material when heated to 450°C by the presence of chromium in the mixture. When heated to 1000°C, all the compounds and mixtures formed crystalline oxides.

II. INTRODUCTION

Several dissolution schemes for recovery of fissionable materials from nuclear fuels result in large quantities of aqueous waste. One possible method for reducing the waste volumes is conversion of the solutions to a solid oxide. A process for calcination of aluminum nitrate waste, using a fluidized bed operating at 400°C, has been investigated at the Idaho Chemical Processing Plant[1,2]. Since some dissolution processes for Nichrome and stainless steel alloyed nuclear fuels produce a waste stream which is essentially a nitrate solution of the alloying constituents[3,4], calcination is an attractive method of obtaining a solid form for storage. The purpose of this study was to observe the thermal decomposition behavior of mixtures containing

hydrated iron, chromium, and nickel nitrates. It has been found in the case of aluminum nitrate calcination that important process advantages can be gained by control of the degree of crystallinity of the alumina product[5,6]. The presence of a high percentage of α alumina tends to decrease the average particle size, yielding improved heat transfer to the bed, and to increase the bulk density of the product. An increased percentage of amorphous alumina results in decreased solids loading of the off-gas system. The composition can be balanced by control of the calciner operating conditions and the use of chemical additives. Therefore it is also of interest in this study to determine the extent of the crystalline characteristics of the products formed by thermal decomposition of the mixtures of iron, chromium, and nickel nitrates.

III. EXPERIMENTAL PROCEDURES

The thermogravimetric analyses were obtained using a Chevenard thermobalance equipped with a transducer system which converted weight changes to a D. C. voltage, which was then recorded using a potentiometric recorder. The sensitivity limit of the system was approximately two milligrams. The temperature was determined using a platinum vs platinum + 10 per cent rhodium thermocouple which had been calibrated using the boiling point of water and the freezing points of lead and zinc; the temperature was also recorded with a potentiometric recorder. Errors in the reported temperatures are within $\pm$ 5°C limits.

The thermal decompositions were conducted in air at atmospheric pressure (approximately 640 mm Hg); two heating rates were used, approximately 1.2 and 2.6°C per minute. The chemicals used were commercially available reagent-grade compounds. Porcelain crucibles were used throughout these decomposition studies after it had been established that they were satisfactory. Possible intermediate products resulting from thermal decomposition were converted to their oxides by heating to 500°C in a vacuum system. The volatile products were collected and fractionated, then submitted for analyses. Analyses were performed by the CPP Analytical Branch.

IV. EXPERIMENTAL RESULTS

A. Thermal Decomposition of Compounds

1. Ferric nitrate nonahydrate

The thermal decomposition curves for ferric nitrate nonahydrate are given in Appendix II, Figure 1. Constant weight is apparently achieved at 250°C using the lower heating rate. The final product is alpha ferric oxide. The sample heated to 925°C showed an 80.4 per cent weight loss and exhibited a higher degree of crystallinity than that heated to 450°C which had an 80.2 per cent weight loss. Since the decomposition rate decreases between 100 and 125°C, products from this region were analyzed. They contained from 2.4 to 2.6 nitrogen atoms and from 4.9 to 6.7 molecules of water per iron atom. X-ray analysis of material heated to 125°C at 1.1°C per minute produced a pattern (Appendix I, Table 1) which could not be identified from the ASTM X-ray Powder Data File. This indicates that ferric nitrate nonahydrate is not present, nor any basic oxides or alpha ferric oxide.

2. Chromic nitrate nonahydrate

Thermal decomposition curves for chromic nitrate nonahydrate are given in Appendix II, Figure 2. Constant weight appears to occur at 425°C using the lower heating rate. The final product is chromic oxide. The weight loss was 81.1 per cent for the sample heated to 465°C compared to 81.2 per cent for the sample heated to 1000°C. An intermediate product exists between 270 and 390°C. This material is amorphous according to X-ray analysis. This product was decomposed under vacuum and found to have the empirical formula $CrO_{2.2}$, indicating that it is a mixed oxide.

3. Aluminum nitrate nonahydrate

Since calcination of aluminum nitrate is already being investigated on a demonstration scale at the ICPP, decomposition curves for aluminum nitrate nonahydrate were obtained for comparison purposes. The curves are presented in Appendix II, Figure 3. Constant weight is apparently attained at 340°C with the lower heating rate. However,

heating was continued to 460°C; the final product was amorphous and was not completely converted to alumina as established by an 85.3 per cent weight loss. The sample heated to 1035°C formed gamma alumina and underwent an 86.1 per cent weight loss.

4. Nickel nitrate hexahydrate

The curves for the thermal decomposition of nickel nitrate hexahydrate are shown in Appendix II, Figure 4. The ultimate product is nickel oxide. At the lower heating rate, no weight change was observed above 340°C; the weight loss was 73.9 per cent when heated to 470°C compared to 74.8 per cent for a sample heated to 960°C. The decomposition curves exhibit a plateau between 250 and 275°C. If the heating rate is decreased to 0.5°C per minute, this plateau region begins at 235°C. The chemical analysis of this intermediate product corresponds approximately to the formula $Ni(OH)NO_3 \cdot \frac{1}{2}H_2O$ and possesses a very definite X-ray pattern (Appendix I, Table 3). However, on prolonged exposure to the atmosphere this X-ray pattern reverts to that of nickel nitrate hexahydrate. The amounts of nitrite and peroxide detected in an aqueous solution of this intermediate product were negligible.

There are also slight indications of formation of intermediate compounds near 125°C and possibly near 200°C. A sample heated to 125°C was analyzed; the composition was 1.9 atoms of nitrogen and 4.7 molecules of water per nickel atom. The X-ray pattern established that nickel nitrate hexahydrate was the principal constituent of this product.

Samples heated to approximately 200°C at 1.1°C per minute had an average composition of 1.8 nitrogen atoms and three molecules of water per nickel atom. The X-ray pattern of this material could not be identified from the ASTM X-ray Powder Data File. The pattern (Appendix I, Table 2) also did not correspond to that obtained from the product heated to 250°C. The intermediate formed by heating to 200°C also reverts to nickel nitrate hexahydrate upon exposure to the atmosphere at room temperature.

B. Thermal Decomposition of Nitrate Mixtures

1. Thermal decomposition of synthetic Nichrome mixtures

Since one possible nuclear fuel cladding alloy material may be

Nichrome, synthetic mixtures of nickel and chromium nitrates were prepared to simulate approximately an 80 weight per cent nickel-20 weight per cent chromium alloy. The thermal decomposition curves are given in Appendix II, Figure 5. From the thermal decomposition curves, it is apparent that the intermediate nickel compound formed near 250°C during the decomposition of pure nickel nitrate hexahydrate is also formed during the decomposition of these mixtures. X-ray analysis of the product heated to 925°C at 2.4°C per minute showed that both nickel oxide and nickel chromite ($NiCr_2O_4$) were formed. This material attained constant weight at 510°C. The product heated to 460°C at 1.0°C per minute appeared to attain constant weight at approximately 340°C, and according to the X-ray analysis the product was primarily nickel oxide, although one extra unidentified line was present. This product should have undergone a 76.3 per cent weight loss if it were completely converted to the ultimate oxides; the observed weight loss was 74.6 per cent.

2. Thermal decomposition of a synthetic stainless steel mixture

A sample of 347 stainless steel, which was analyzed before being used for dissolution studies in this laboratory, contained iron, chromium, and nickel in the following weight ratios, respectively: 6.18: 1.91: 1.00. Accordingly, binary nitrate mixtures of the various components were prepared in approximately these ratios, as well as the ternary nitrate mixture. These were decomposed at two heating rates and the decomposition curves are given in Appendix II, Figures 6-9.

X-ray analysis and other pertinent data concerning these mixtures are summarized in Table 1. Chromic oxide and alpha ferric oxide form a solid solution in which chromic oxide causes only a slight shift in the "d spacings" of the α ferric oxide X-ray pattern[7]. The theoretical per cent weight loss was based on the weight loss of the individual reagents when they were heated to 900°C or higher. This approach makes allowance for any extra water or acid which is present in the original reagents.

V. DISCUSSION

In considering the thermal decomposition data compiled in this report,

it must be kept in mind that the kinetics of a system are being observed, rather than an equilibrium system. Weight changes occur only as the decomposition reaction becomes rapid enough to be readily detected. Keeping this in mind, it is obvious why the values for the temperature at apparent constant weight decreases as the heating rate is decreased. At very slow heating rates one may expect reasonably constant weight to be attained at temperatures lower than those reported here.

Another observation which should be made concerning the data is that even though constant weight is observed, a product is not necessarily completely converted to the final oxide form. The exact temperature at which apparent constant weight is observed is also difficult to determine. A small change in weight was difficult to detect with the apparatus used because of the bouyancy effects at these temperatures and the sensitivity limit of a couple of milligrams. Therefore, actual final constant weight may not have been attained until the temperature was a hundred degrees higher than the reported apparent constant weight.

Other studies have been made of the thermal decomposition of the individual nitrates which are components of the mixtures considered in this study. The studies of Wendlandt[8] on aluminum, iron, chromium, and nickel nitrate decomposition using a heating rate of 5.4°C per minute agree in general with the results of this study. Two exceptions are: first, due to a lower heating rate, a noticeable break in the decomposition of ferric nitrate was observed in this study; secondly, Wendlandt observed that the plateau region in the nickel nitrate decomposition curve began at 205°C rather than 250°C. He did not report any analyses of the products which were produced during the decompositions.

During the decomposition of aluminum nitrate nonahydrate above 100°C, dehydration and loss of nitrate occur simultaneously as evidenced by the rapid decomposition of the salt with no apparent break in the curve. The same phenomenon occurs with chromic nitrate nonahydrate except that a mixed oxide system exists over a hundred degree temperature range after the nitrate and water have been removed.

Table 1

THERMAL DECOMPOSITION OF HYDRATED IRON, CHROMIUM, AND NICKEL NITRATE MIXTURES IN AIR

Mixture (Weight Per Cent)	Average Heating Rate (°C/Min)	Apparent Constant Weight Above (°C)	Maximum Temperature Sample Reached (°C)	X-Ray Analysis of Final Product	Per Cent Weight Loss Observed	Per Cent Weight Loss Theoretical
65.5 Cr, 34.5 Ni	2.6	545	1050	$NiCr_2O_4$, Cr_2O_3, unidentified trace	79.7	79.2
65.4 Cr, 34.6 Ni	1.2	415	460	Cr_2O_3 unidentified lines	77.2	79.2
86.4 Fe, 13.6 Ni	2.7	380	1045	α Fe_2O_3, $NiFe_2O_4$	79.7	79.7
86.1 Fe, 13.9 Ni	1.2	280	460	α Fe_2O_3	79.2	79.6
75.7 Fe, 24.3 Cr	2.5	445	1035	α Fe_2O_3*	80.5	80.6
76.0 Fe, 24.0 Cr	1.1	285	470	amorphous	79.3	80.6
68.0 Fe, 21.0 Cr, 11.0 Ni	2.5	385	1060	α Fe_2O_3*, $NiFe_2O_4$	80.2	80.0
68.0 Fe, 21.0 Cr, 11.0 Ni	1.2	410	450	amorphous	79.4	80.0

* Slight shift in d spacings indicates that Cr_2O_3 is probably present.

Analysis of the product prepared by heating ferric nitrate nonahydrate to 125°C at 1.1°C per minute shows that both dehydration and loss of nitrate have occurred. Up to this point, the dehydration reaction has been the predominant one. The product at this temperature is a liquid which solidifies gradually on cooling. The product may be a mixture of a lower hydrate of ferric nitrate and a basic ferric nitrate. Of the four compounds studied, ferric nitrate nonahydrate reached an apparent constant weight at the lowest temperature, 250°C.

The thermal decomposition of the nickel nitrate hexahydrate is principally a dehydration reaction up to 200°C, as evidenced by composition analyses. Above 200°C the loss of nitrate becomes an appreciable portion of the decomposition. From the fact that the intermediate product formed at 250°C reverts to nickel nitrate hexahydrate on exposure to the atmosphere at room temperature, one might conclude that the product is actually a mixture of nickel nitrate and nickel hydroxide and that one of these is further hydrated. Attempts to prepare various pure nickel nitrate hydrates and to obtain their X-ray patterns have been unsuccessful so far; however, the work is continuing. Indications are that at 200°C, there are at least two crystalline compounds present and that one of these is possibly nickel nitrate tetrahydrate.

The decomposition curves of the binary mixtures have essentially the same characteristics as the individual components themselves. In the ternary stainless steel mixture only the ferric nitrate decomposition curve is recognizable.

There is a significant difference in the crystalline characteristics of the products from the mixtures and from the individual salts when they are heated to approximately 450°C. Hydrated iron, chromium, and nickel nitrates all form crystalline products if heated to 450°C. In all the mixtures heated to 450°C, which contain chromium as a minor component, crystalline chromic oxide is not present in sufficient quantity to be detected by X-ray analysis. Heating these mixtures to 1000°C does produce chromic oxide as a crystalline component of some system, although its presence is not readily detected in the presence of a α ferric oxide with which it forms a solid solution[7].

The presence of chromium in iron-chromium nitrate mixtures actually prevents the formation of crystalline ferric oxide even though the mixture is heated to 470°C. α-Ferric oxide can be formed, however, if the mixture is heated to 570°C. It is evidently the chromium component in the stainless steel mixtures which prevents any product crystallization at 450°C. From the decomposition data, it appears that Nichrome waste solutions calcined at 450°C will produce a product containing crystalline nickel oxide along with amorphous chromium oxide. Calcination of stainless steel waste solutions at 450°C produces an amorphous product. These preceding observations are based on decomposition in an atmosphere of air and when the mixtures are heated gradually to the desired final temperature. The system may behave quite differently in an atmosphere of nitrogen oxides and in a fluidized bed. Gradual thermal decomposition of aluminum nitrate in air produces amorphous alumina at 450°C, while thermal decomposition of aluminum nitrate in an atmosphere of nitrogen oxides produces some α-alumina[6]. The formation of α-alumina can be prevented by addition of other reagents[5,6]. Recently some experiments were conducted at Stanford Research Institute under a sub-contract to Phillips Petroleum Company in which synthetic stainless steel waste solutions were calcined in a fluidized bed[9]. At 400°C, in these experiments, an appreciable amount of α-Fe_2O_3 did form.

VI. CONCLUSIONS

1. Thermal decomposition of chromium nitrate nonahydrate results in the formation of a mixed chromium oxide. This material is stable to 390°C where it begins to decompose to crystalline chromic oxide.

2. Thermal decomposition of nickel nitrate hexahydrate results in the formation of an intermediate crystalline material which has the empirical formula $Ni(NO_3)(OH)\cdot\frac{1}{2}H_2O$ which decomposes on further heating to crystalline nickel oxide.

3. During thermal decomposition of ferric nitrate nonahydrate an unidentified crystalline species occurs before the formation of α-ferric oxide.

4. The presence of chromium prevents the formation of crystalline products during the thermal decomposition of stainless steel nitrates when gradually heated in air up to 450°C.

5. Calcination to the oxides of Nichrome and stainless nitrates appears to be feasible in the 400 to 500°C temperature range.

VII. ACKNOWLEDGEMENTS

The author wishes to acknowledge Dr. D. W. Rhodes for suggesting this study and for his advice during the course of the work, as well as Mr. W. A. Ryder of the CPP Analytical Branch, who was responsible for the X-ray diffraction analyses.

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IX. APPENDIX I
X-RAY PATTERNS

Table 1

X-RAY PATTERN FOR THE IRON DECOMPOSITION PRODUCT FORMED BY HEATING FERRIC NITRATE NONAHYDRATE TO 125°C

Heating rate = 1.1°C/min
Component ratio $Fe:NO_3:H_2O$ = 1.0:2.4 ⟶ 2.6:4.9 ⟶ 6.7

d(°A)	I/I°
9.4	100
6.8	50
4.29	85
3.14	35
2.73	20
2.54	20
2.30	20
1.150	25

Table 2

X-RAY PATTERN FOR NICKEL DECOMPOSITION PRODUCT FORMED BY HEATING NICKEL NITRATE HEXAHYDRATE TO 200°C

Heating rate = 1.1°C/min
Component ratio $Ni:(NO_3):(H_2O)$ = 1:1.8:3

d(°A)	I/I°
6.8	29
6.4	26
5.75	37
4.88	100
4.73	52
4.55	29
4.40	66
4.10	31
3.92	38
3.71	42
3.38	38
3.32	38
2.90	45
2.81	32
2.58	35
2.36	27
2.06	22
2.04	26
2.01	29
1.96	22

Table 3

X-RAY PATTERN FOR THE NICKEL DECOMPOSITION PRODUCT FORMED BY HEATING NICKEL NITRATE HEXAHYDRATE TO 250°C

Heating rate = 1.1°C/min
Empirical formula = $Ni(NO_3)(OH)\cdot\frac{1}{2}H_2O$

d(°A)	I/I°
6.86	100
4.82	51
4.74	16
4.55	16
4.39	27
4.21	95
3.95	16
3.64	19
3.46	30
3.38	8
3.25	80
2.98	58
2.90	27
2.80	11
2.71	22
2.65	14
2.44	31
2.31	23
2.20	76
2.11	47
2.06	9
2.03	18
1.96	27
1.90	11
1.83	19
1.68	23
1.64	34
1.57	11
1.49	24
1.41	38
1.24	19
1.22	16

X. APPENDIX II
THERMAL DECOMPOSITION CURVES

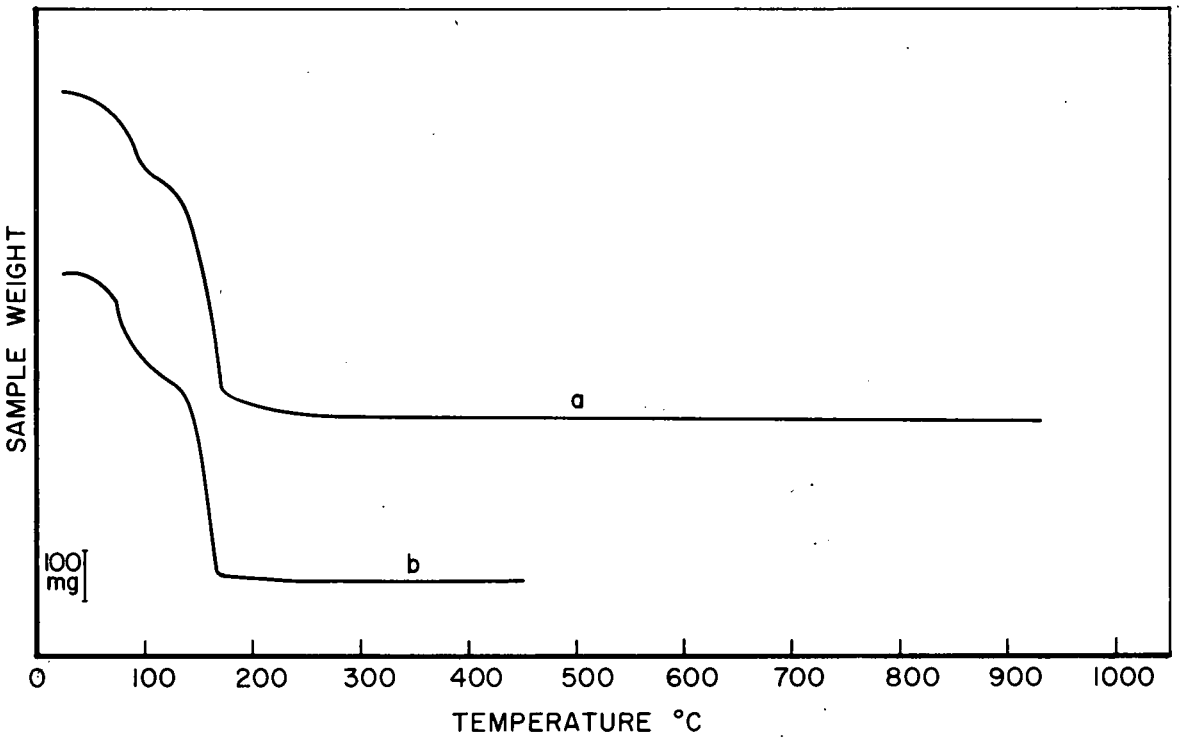

Fig. 1 Thermal Decomposition of Ferric Nitrate Nonahydrate
a) Heating rate = 2.7°C/min; weight loss = 80.4 per cent
b) Heating rate = 1.1°C/min; weight loss = 80.2 per cent

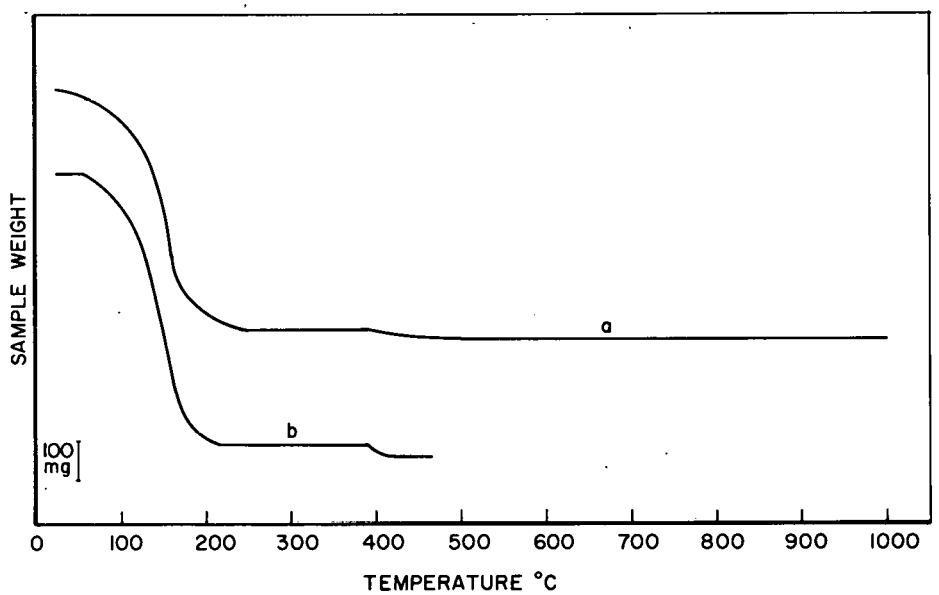

Fig. 2 Thermal Decomposition of Chromium Nitrate Nonahydrate
a) Heating rate = 2.6°C/min; weight loss = 81.2 per cent
b) Heating rate = 1.0°C/min; weight loss = 81.1 per cent

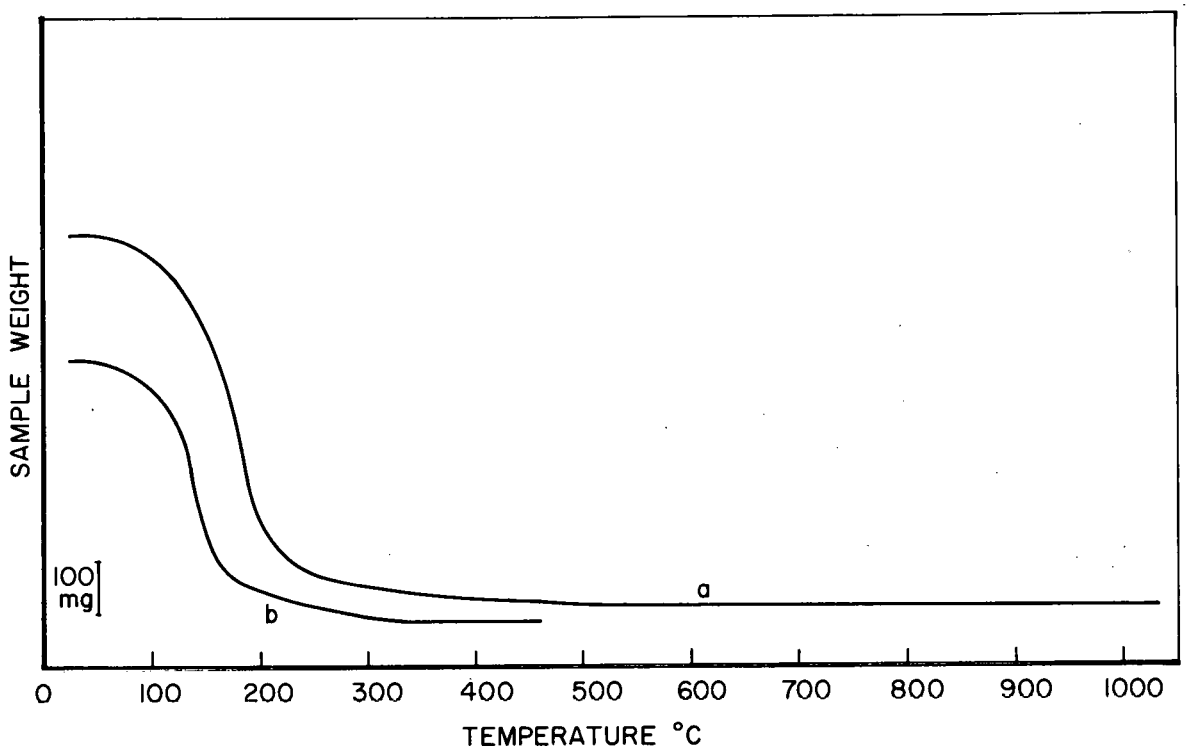

Fig. 3 Thermal Decomposition of Aluminum Nitrate Nonahydrate
a) Heating rate = 2.6°C/min; weight loss = 86.1 per cent
b) Heating rate = 1.0°C/min; weight loss = 85.3 per cent

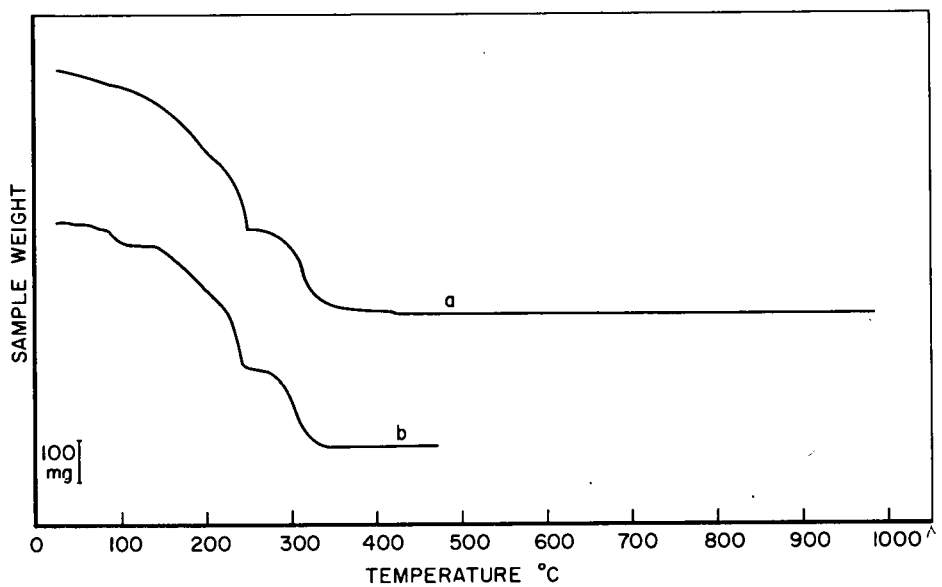

Fig. 4 Thermal Decomposition of Nickel Nitrate Hexahydrate
a) Heating rate = 2.4°C/min; weight loss = 74.8 per cent
b) Heating rate = 1.1°C/min; weight loss = 73.9 per cent

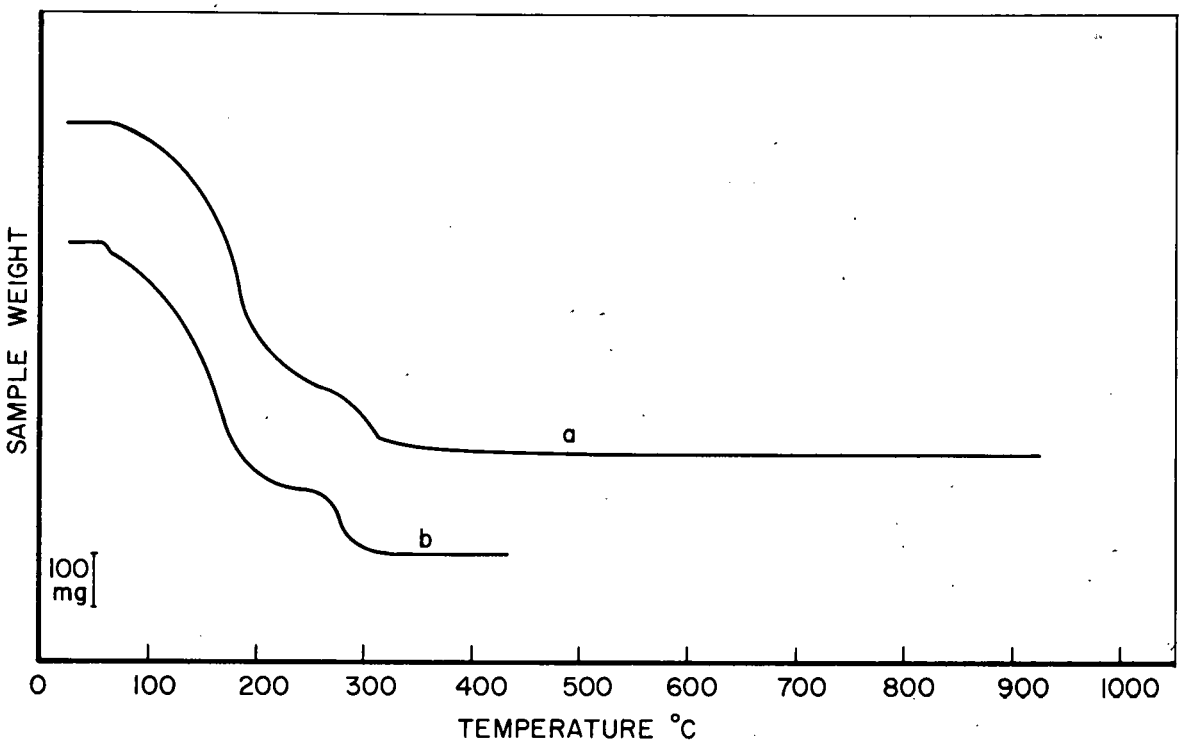

Fig. 5 Thermal Decomposition of Hydrated Nichrome Nitrate Mixture
a) Heating rate = 2.4°C; composition = 80.9 w/o Ni, 19.1 w/o Cr; weight loss = 76.2 per cent
b) Heating rate = 1.0°C; composition = 79.8 w/o Ni, 20.2 w/o Cr; weight loss = 74.6 per cent

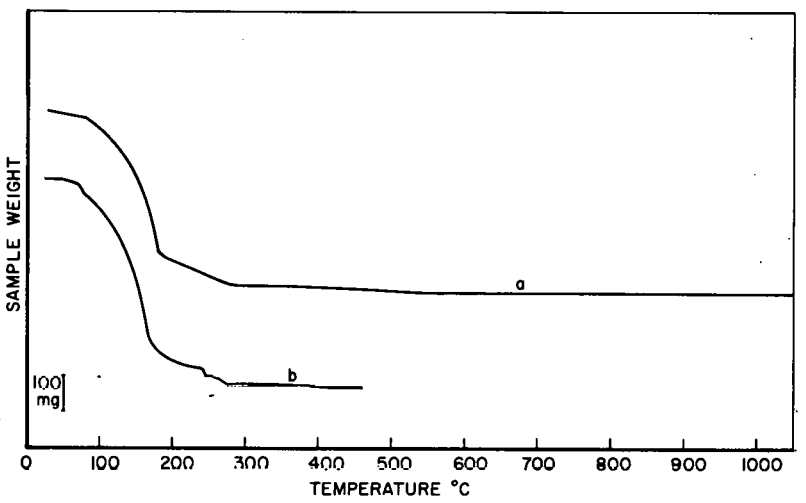

Fig. 6 Thermal Decomposition of Hydrated Chromium-Nickel Nitrate Mixture
a) Heating rate = 2.6°C/min; composition = 65.6 w/o Cr, 34.5 w/o Ni; weight loss = 79.7 per cent
b) Heating rate = 1.2°C/min; composition = 65.4 w/o Cr, 34.6 w/o Ni; weight loss = 77.2 per cent

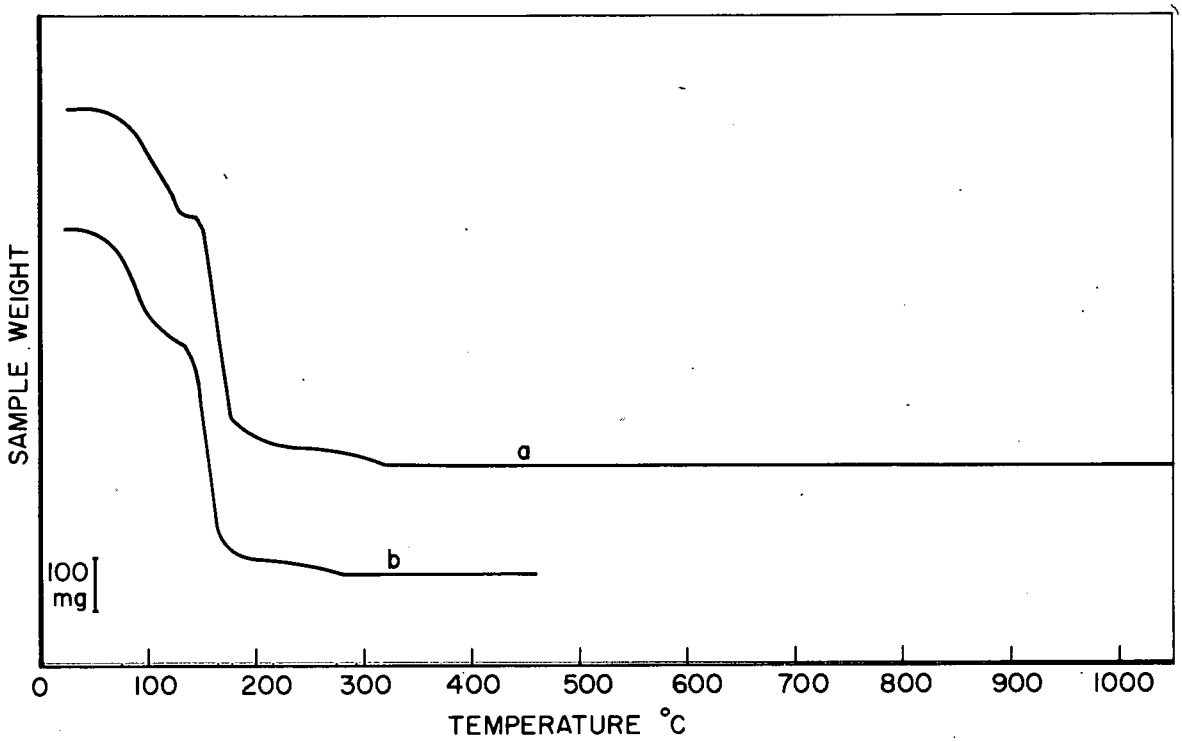

Fig. 7 Thermal Decomposition of Hydrated Iron-Nickel Nitrate Mixtures
a) Heating rate = 2.7°C/min; composition = 86.4 w/o Fe, 13.6 w/o Ni; weight loss = 79.7 per cent
b) Heating rate = 1.2°C/min; composition = 86.1 w/o Fe, 13.9 w/o Ni; weight loss = 79.2 per cent

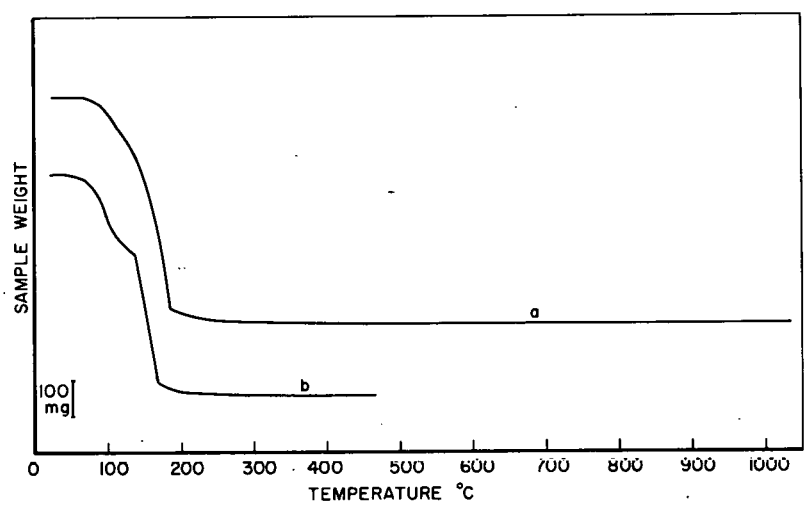

Fig. 8 Thermal Decomposition of Hydrated Iron-Chromium Nitrate Mixtures
a) Heating rate = 2.5°C/min; composition = 75.7 w/o Fe, 24.3 w/o Cr; weight loss = 80.5 per cent
b) Heating rate = 1.1°C/min; composition = 76.0 w/o Fe, 24.0 w/o Cr; weight loss = 79.3 per cent

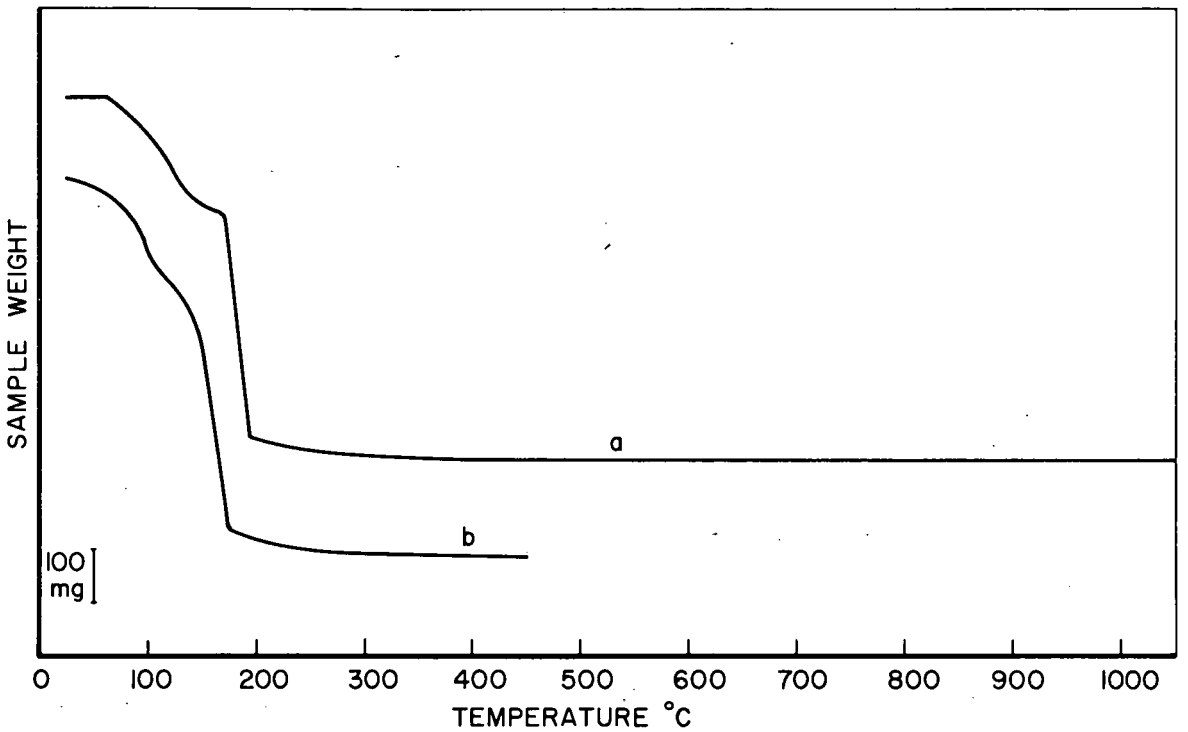

Fig. 9 Thermal Decomposition of Hydrated Iron-Chromium-Nickel Nitrate Mixtures

a) Heating rate = 2.5°C/min; composition = 68.0 w/o Fe, 21.0 w/o Cr, 11.0 w/o Ni; weight loss = 80.2 per cent

b) Heating rate = 1.2°C/min; composition 68.0 w/o Fe, 21.0 w/o Cr, 11.0 w/o Ni; weight loss = 79.4 per cent

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