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ACTIVATION ENERGY OF THE THERMAL DECOMPOSITION OF AMMONIUM METAVANADATE. A THERMOGRAVIMETRIC STUDY

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The thermogravimetric curves of the title compound (AMV), recorded at two rates of heating (10 and 1.25 deg min^{-1}), exhibited two separate steps of weight loss (at 423–513 and 513–613 K). The TG curves were analysed by testing 18 solid-state reaction equations associated with the methods of Coats and Redfern (1964), Šatava and Škvára (1969) and Blazejowski et al. (1983). On the basis of linearization procedures, high-order reactions with $n=1.5$ and 2.0 best described the two TG steps. The activation energies of the two conversion stages are: first stage: 163.6 and 152.6 kJ mol^{-1}, and second stage: 238.1 and 167.7 kJ mol^{-1} (low and high heating rates, respectively).

The overall reaction for the thermal decomposition of ammonium metavanadate (AMV) is generally represented as

$$2\,NH_4VO_3 \rightarrow V_2O_5 + 2\,NH_3 + H_2O$$

accompanied by a total weight loss of 22.2% of the original sample weight. The decomposition does not take place in a single stage, but in several stages, and there is still little agreement as to the intermediates formed, the results depending on the method of detection and characterization. The mass losses after particular stages led some earlier investigators [1, 2] to believe that HVO_3 is one of the products. A three-stage decomposition was suggested to occur in air or oxidizing atmospheres by Brown et al. [3–5]:

$$\text{AMV} \xrightarrow{423\text{–}453\text{ K}} \text{ABV (ammonium bivanadate)} \xrightarrow{453\text{–}483\text{ K}} \text{AHV}$$
$$\text{(ammonium hexavanadate)} \xrightarrow{533\text{–}573\text{ K}} V_2O_5.$$

The formations of the intermediates are atmosphere-dependent stages. This was supplemented by differential enthalpic analysis (DEA) and IR spectroscopy [6].

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Formation of the hexavanadate intermediate was confirmed by Sas et al. [7]. A series of intermediates (M_1–M_5) were detected by X-ray and IR techniques by Furuichi et al. [8] in thermal decompositions performed in different atmospheres. The decomposition of AMV to V_2O_5 was conceived by Kunaev et al. [9] to proceed through stages involving the release of NH_3 and H_2O, but with the formation of intermediate solids different from those mentioned above. A completely different mechanism was proposed by Kalicki et al. [10], in which H_2O and NH_3 are released at 373–423 K, N_2, NO and N_2O at 423–473 K, and the remaining NH_3 at 573 K. A three-stage decomposition mechanism was deduced by Dziemba et al. [11] from redox measurements. From DTA tracings recorded in different atmospheres, two and three-stage endothermic processes have also been suggested, depending on the atmosphere involved [12].

The present study reports the thermogravimetry of AMV performed at two widely differing rates of heating in order to detect the possible separate appearance of the various decomposition processes. The TG curves are rigorously analysed with respect to the predominant decomposition mechanism and the associated activation energies.

Experimental

Material

Analytical grade ammonium metavanadate (B.D.H.) was analysed at two heating rates by means of two apparatuses. At the high rate of heating of 10 deg min^{-1}, analysis was carried out with an MOM apparatus (Hungary). At the low heating rate of 1.25 deg min^{-1}, a thermobalance produced by Gebrunder Netzsch, West Germany, was used. The obtained recordings are presented in Fig. 1.

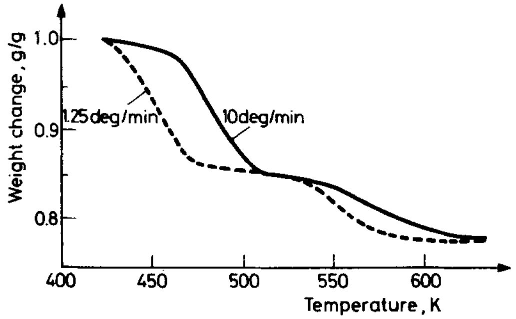

Fig. 1 Thermogravimetric curves of ammonium metavanadate, recorded at different heating rates

Analysis of TG curves

The weight loss curves were transformed into fractional decomposition plots (α *vs.* T; Fig. 2). Mathematical analysis of these curves was carried out by correlating the functions $\log [g(\alpha)/T^2]$, $\log [g(\alpha)]$ or $\log [g(\alpha)/T]$ with $1/T$.

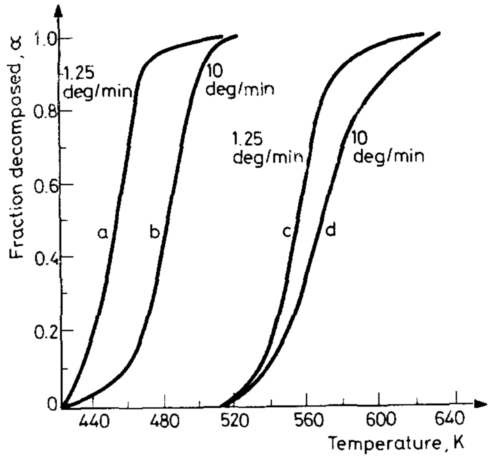

Fig. 2 Fractional decomposition curves representing the first (a, b) and second (c, d) stages of weight loss, derived from TG curves at two heating rates

These correspond to the linearization expressions suggested by Coats and Redfern (modified) (CR) [13–15], Šatava and Škvára (SS) [16] and Blazejowski et al. (Blz) [17]. Eighteen solid-state reaction mechanism equations were tested in order to investigate the most probable mechanism governing the present decomposition. These equations are listed in Table 1 [18–19].

The applicability of the different equations was tested by linear regression analysis associated with the method of least squares. The linearity of each mechanism is represented by its correlation coefficient, r, and the standard error of the estimate, S_e. From the slope of every linear relationship, the activation energy, E_a, associated with each stage was evaluated. A 32 bit M/C computer produced by Perkin–Elmer was employed to facilitate the calculations. The obtained data are listed in Tables 2–7.

Table 1 Kinetic functions describing the thermal decomposition of the solids used in the present work

Equation no.	Notation	$g(\alpha)$
1		α
2	D_1	α^2
3		$\alpha^{1/2}$
4		$\alpha^{1/3}$
5		$\alpha^{1/4}$
6	R_2	$1-(1-\alpha)^{1/2}$
7	R_3	$1-(1-\alpha)^{1/3}$
8	F_1	$-\ln(1-\alpha)$
9		$[-\ln(1-\alpha)]^{2/3}$
10	A_2	$[-\ln(1-\alpha)]^{1/2}$
11	A_3	$[-\ln(1-\alpha)]^{1/3}$
12		$[-\ln(1-\alpha)]^{1/4}$
13	D_2	$\alpha+(1-\alpha)\ln(1-\alpha)$
14	D_4	$1-2/3\alpha-(1-\alpha)^{2/3}$
15	D_3	$[1-(1-\alpha)^{1/3}]^2$
16		$e^{(\alpha-1)}-e^{-1}$
17	SO	$(1-\alpha)^{-1}-1$
18		$(1-\alpha)^{-1/2}-1$

Table 2 Linear regression parameters and activation energies, calculated by the method of Coats and Redfern (first step at two heating rates)

Eq. no.	10 deg min^{-1}			1.25 deg min^{-1}		
	E, kJ·mol^{-1}	$-r$	$S_e \times 10^2$	E, kJ·mol^{-1}	$-r$	$S_e \times 10^2$
1	96.7	0.966816	8.52	96.8	0.960833	9.33
2	189.2	0.968266	17.35	200.8	0.962372	18.96
3	41.5	0.962534	4.15	44.8	0.955791	4.60
4	25.1	0.956304	2.73	27.4	0.949419	3.03
5	16.9	0.949047	1.99	18.7	0.940892	2.25
6	107.0	0.982886	7.13	114.4	0.979649	7.83
7	113.4	0.987392	6.46	121.2	0.984836	7.14
8	127.6	0.993019	5.39	136.5	0.992572	5.59
9	82.5	0.993273	3.42	88.6	0.992420	3.66
10	59.9	0.993831	2.38	64.6	0.993172	2.54
11	37.4	0.994723	1.37	40.7	0.993831	1.52
12	26.1	0.994658	0.96	28.7	0.992375	1.19
13	208.8	0.977120	15.93	221.9	0.973543	17.41
14	217.2	0.981204	15.17	230.9	0.977504	16.57
15	234.5	0.987020	13.56	249.7	0.984852	14.69
16	109.3	0.977417	8.39	116.7	0.974521	8.98
17	181.9	0.989054	9.64	195.1	0.993344	7.56
18	152.6	0.994704	5.61	163.6	0.996806	4.38

Results and discussion

1. Stepwise weight loss

Although recorded at two widely differing rates of heating, the original TG tracings (Fig. 1) display only two distinctly separate weight loss steps. These appear at 423–513 K and 513–613 K, accompanied by weight losses of 15.0% and 7.2%, respectively. According to Brown et al. [5], two weight loss steps take place at 423–453 K and 453–483 K, with weight losses of 11.1% and 3.7% (original weight basis). These two steps involve the decomposition of AMV to ABV, and of ABV to AHV. A somewhat different explanation was given by Taniguchi et al. [20], who stated that the end-product at 517 K has the formula $(NH_4)_{1.5}V_3O_8$. Nevertheless, the total weight loss recorded at 513 K in the present case (15.0%) is in general comparable with the values given both by Brown et al. [5] (14.8%) and by Taniguchi et al. [20] (15.4%). The second weight loss step (between 513 and 613 K) corresponds approximately to the decomposition of either of the compounds $NH_4V_3O_8$ [5] or $(NH_4)_{1.5}V_3O_8$ [20].

It is evident that recording at the lower heating rate (1.25 deg min^{-1}) leads to a shift to lower temperature for both steps. Thus, at $\alpha = 0.5$ ($T_{0.5}$), the two occur at $T_{0.5}$ = 454 and 555 K, as compared with 482 and 568 K in the fast-heating curve (10 deg min^{-1}).

Table 3 Linear regression parameters and activation energies, calculated by the method of Šatava and Škvára (first step at two heating rates)

Eq. no.	10 deg·min^{-1}			1.25 deg·min^{-1}		
	E, kJ·mol^{-1}	$-r$	$S_e \times 10^2$	E, kJ·mol^{-1}	$-r$	$S_e \times 10^2$
1	106.6	0.969751	8.80	111.9	0.964195	9.57
2	206.1	0.969628	17.61	216.9	0.964161	19.15
3	56.6	0.969786	4.40	59.1	0.964353	4.77
4	39.7	0.969810	2.93	41.4	0.964382	3.19
5	31.2	0.969784	2.21	32.3	0.964337	2.39
6	123.1	0.983361	7.54	129.7	0.980463	8.16
7	129.5	0.987033	7.02	136.6	0.984996	7.53
8	143.9	0.992187	6.05	152.0	0.991859	6.17
9	98.3	0.992181	4.03	103.6	0.991858	4.11
10	75.4	0.992192	3.03	79.4	0.991873	3.09
11	52.4	0.992192	2.01	54.9	0.991872	2.06
12	40.8	0.992190	1.51	42.6	0.991856	1.54
13	225.8	0.978430	16.24	238.2	0.974618	17.60
14	234.4	0.981946	15.40	247.4	0.978682	16.72
15	252.5	0.986553	13.90	260.8	0.976694	15.03
16	125.3	0.978428	8.77	132.0	0.975704	9.30
17	198.7	0.988643	10.24	211.1	0.992763	8.18
18	169.2	0.993740	6.41	179.3	0.975831	5.23

2. *First decomposition stage*

Recording of this stage (423–513 K) at rates of 10 and 1.25 deg min^{-1} gave the data presented in Tables 2–4. Table 2 (CR method) reveals that at a heating rate of 10 deg min^{-1} the highest r values correspond to the series of equations derived by Avrami and Erofeev (Eqs 8–12) and to the equation with order 1.5 (Eq. 18). The first group of equations exhibit exactly the same r values with a successive decrease of both E and S_e as observed in many cases before [22, 23]. Some of the evaluated activation energies in this group are unreasonably low (Eqs 11 and 12), i.e. lower than the latent heat of vaporization of water (<40 kJ mol^{-1}). Apparent reaction orders higher than one have previously been reported for kaolinite [21] and nontronites [22, 23]. A reaction order of 1 or 1.5 would represent the first decomposition stage with an activation energy of 127.6 or 152.6 kJ mol^{-1}. Due to the complexity of the solid-state reaction considered, the latter appears to be the more probable ($E = 152.6$ kJ mol^{-1}).

Decrease of the rate of heating to 1.25 deg min^{-1} results in the values given in Table 2. The best reaction equations describing this decomposition are similarly the Avrami–Erofeev expressions plus the equation with order 1.5. The latter yields a value of 163.6 kJ mol^{-1}, about 7% higher than that estimated at 10 deg min^{-1}.

Table 4 Linear regression parameters and activation energies, calculated by the method of Blazejowski et al. (first step at two heating rates)

Eq. no.	10 deg·min^{-1}			1.25 deg·min^{-1}		
	E, kJ·mol^{-1}	$-r$	$S_e \times 10^2$	E, kJ·mol^{-1}	$-r$	$S_e \times 10^2$
1	94.6	0.968265	8.68	100.4	0.962665	9.44
2	193.0	0.969123	17.46	204.5	0.963529	18.99
3	45.4	0.966178	4.30	48.4	0.960767	4.67
4	28.9	0.963644	2.85	31.1	0.958038	3.11
5	20.8	0.960565	2.14	22.4	0.954156	2.34
6	116.9	0.983381	7.28	119.3	0.980205	7.97
7	117.3	0.987389	6.68	124.9	0.985034	7.31
8	131.5	0.992490	5.76	140.2	0.992226	5.88
9	86.4	0.992778	3.71	92.2	0.992058	3.91
10	63.8	0.992836	2.73	68.3	0.992479	2.81
11	41.2	0.992831	1.76	44.3	0.992155	1.87
12	30.0	0.993008	1.27	32.3	0.992338	1.34
13	212.7	0.978156	16.06	225.6	0.974106	17.50
14	221.1	0.981679	15.25	234.7	0.978410	16.58
15	238.5	0.987277	13.56	253.4	0.985196	14.75
16	113.1	0.977991	8.57	120.3	0.975201	9.13
17	185.8	0.988854	9.94	198.8	0.993166	7.82
18	156.5	0.994275	5.98	167.2	0.996178	4.83

Table 5 Linear regression parameters and activation energies, calculated by the method of Coats and Redfern (second step at two heating rates)

Eq. no.	10 $\mathrm{deg \cdot min^{-1}}$			1.25 $\mathrm{deg \cdot min^{-1}}$		
	E, $\mathrm{kJ \cdot mol^{-1}}$	$-r$	$S_e \times 10^2$	E, $\mathrm{kJ \cdot mol^{-1}}$	$-r$	$S_e \times 10^2$
1	78.4	0.919090	12.90	113.8	0.924143	12.89
2	166.1	0.927010	25.81	236.6	0.928464	25.94
3	34.5	0.899845	6.42	52.5	0.914031	6.38
4	19.9	0.869402	4.34	32.0	0.900195	4.24
5	12.6	0.831284	3.23	22.0	0.880931	3.20
6	94.1	0.950690	11.79	135.7	0.951260	12.05
7	100.3	0.960177	11.21	144.3	0.959585	11.59
8	114.1	0.975923	9.80	163.5	0.973609	10.50
9	73.0	0.974102	6.51	106.0	0.972847	6.91
10	52.4	0.972462	4.83	77.3	0.972115	5.11
11	31.8	0.966684	3.24	48.6	0.970419	3.31
12	21.5	0.960590	2.40	34.2	0.968169	2.42
13	184.9	0.944011	24.82	263.0	0.943602	25.25
14	193.1	0.951090	24.09	274.0	0.950098	24.65
15	210.0	0.963114	22.53	297.5	0.961096	23.43
16	95.8	0.941933	13.11	137.9	0.942538	13.39
17	167.7	0.998071	4.07	238.1	0.993763	7.32
18	138.8	0.991875	6.84	197.8	0.988019	8.47

Comparison of the results obtained by applying the three methods (Tables 2–4) indicates that Eqs 8–12, 17 and 18 provide the best data optimization for the first stage. However, they display an increase in the sequence CR, Blz, SS for both E and S_e. For the best equations, r increases insignificantly in the same sequence. Accordingly, on the basis of the best equation, the CR method seems better than either SS or Blz, irrespective of the criterion chosen, r or S_e.

3. Second decomposition stage

Comparison of the data in Tables 5–7 reveals that this stage generally displays a poor fit to a straight line, except for the high-order equations [17, 18]. This is true irrespective of the data linearization method. It is very clear that the high-order equations best describe the second stage of decomposition. However, the estimated activation energies are considerably (ca. -40%) higher when evaluated from the slow-heating TG recording. The three methods of data optimization give almost exactly the same r values at both rates, and only slightly different S_e values.

Accordingly, from the lowest S_e values (CR method) the values of E estimated from the fast and slow-heating curves are 138.8 and 197.8 $\mathrm{kJ\ mol^{-1}}$ (Eq. 18). From

Table 6 Linear regression parameters and activation energies, calculated by the method of Šatava and Škvára (second step at two heating rates)

Eq. no.	10 deg·min^{-1}			1.25 deg·min^{-1}		
	E, kJ·mol^{-1}	$-r$	$S_e \times 10^2$	E, kJ·mol^{-1}	$-r$	$S_e \times 10^2$
1	97.0	0.933710	12.92	132.3	0.932329	13.05
2	185.8	0.933582	25.86	256.2	0.932228	26.12
3	52.2	0.933723	6.46	70.0	0.932356	6.52
4	37.0	0.933724	4.31	49.2	0.932381	4.35
5	29.3	0.933709	3.23	38.4	0.932349	3.26
6	113.0	0.958201	11.87	154.4	0.955139	12.28
7	119.3	0.965662	11.33	163.0	0.962224	11.88
8	133.3	0.978848	9.92	182.5	0.974808	10.81
9	91.6	0.978843	6.61	124.4	0.974790	7.21
10	70.6	0.978849	4.95	95.3	0.974803	5.41
11	49.4	0.978858	3.30	66.6	0.974808	3.61
12	38.7	0.978844	2.48	51.3	0.974806	2.70
13	204.8	0.948728	24.86	282.5	0.946263	25.43
14	213.0	0.955007	24.15	293.9	0.952258	24.86
15	227.1	0.963663	21.11	306.8	0.960036	22.88
16	114.6	0.950645	13.18	156.6	0.947087	13.63
17	187.4	0.997610	4.71	257.8	0.992983	8.06
18	158.2	0.992183	7.16	271.1	0.987740	8.95

the second-order equation (Eq. 17), the corresponding values are 167.7 and 238.1 kJ mol^{-1}.

It is noteworthy that change in the order from 1.0 to 1.5 to 2.0 brings about a successive increase in E, from 163.5 to 197.8 to 238.1 kJ mol^{-1} (slow-heating curve). The fast-heating recording yields respective values of 114.1, 138.8 and 167.7 kJ mol^{-1}. There are more or less corresponding increase in r and decreases in S_e. This might justify a second-order reaction as the most probable for a description of the second stage of decomposition of ammonium metavanadate.

Conclusions

The thermogravimetry of ammonium metavanadate at two fairly different rates of heating demonstrates only two weight loss steps. At a low heating rate (1.25 deg min^{-1}), separation of the two stages suggested by Brown et al. [3–6] at 423–483 K was expected, but this was not observed. These stages could be overlapping consecutive or partly simultaneous processes, and thus not likely to appear in the form of distinctly separated TG steps. Initiation of the stage at

Table 7 Linear regression parameters and activation energies, calculated by the method of Blazejowski et al. (second step at two heating rates)

Eq. no.	10 deg·min^{-1}			1.25 deg·min^{-1}		
	E, kJ·mol^{-1}	$-r$	$S_e \times 10^2$	E, kJ·mol^{-1}	$-r$	$S_e \times 10^2$
1	83.1	0.926845	12.92	118.3	0.928249	12.99
2	170.8	0.930477	25.82	241.1	0.930474	26.01
3	39.2	0.918664	6.47	56.9	0.923815	6.46
4	25.5	0.908978	4.32	36.5	0.918288	4.31
5	17.2	0.897340	3.26	26.2	0.911507	3.24
6	98.8	0.954857	11.81	140.2	0.953457	12.15
7	105.0	0.963183	11.26	148.7	0.961090	11.71
8	118.9	0.977545	9.84	168.0	0.974199	10.66
9	77.7	0.976741	6.55	110.5	0.973890	7.06
10	57.1	0.975730	4.92	81.7	0.973481	5.26
11	36.7	0.973638	3.28	53.1	0.972355	3.45
12	26.2	0.971497	2.45	38.6	0.970810	2.61
13	189.6	0.946444	24.83	26.7	0.944954	25.34
14	197.8	0.953170	24.10	278.5	0.951273	24.74
15	214.7	0.964557	22.56	301.9	0.961941	23.50
16	100.5	0.946674	13.13	142.4	0.944984	13.50
17	172.4	0.997791	4.41	242.6	0.993375	7.69
18	143.5	0.992054	6.99	202.3	0.987993	8.67

453–483 K could be induced by the evolving gases, so that only a continuous curve is seen.

The activation energies of conversion for both stages increased when the heating rate was decreased (10–40%). Such behaviour has been reported by Simon [24], and confirmed by Fevre et al. [25], who noted that the activation energy increased when either the heating rate of the sample weight was decreased. The latter authors [25] strongly recommended TG at low rates of heating (down to 20 deg/h).

If the low-heating rate TG recordings are preferred, activation energies $E_a = 163.6$ and 238.1 kJ mol^{-1} are indicated for the two stages (CR method). These are appreciably different from the values reported previously [4, 5, 9, 20].

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Zusammenfassung — Die thermogravimetrische Untersuchung der Titelverbindung (AMV) ergab bei zwei Aufheizgeschwindigkeiten (10 und 1,25 deg·min^{-1}) zwei verschiedene Gewichtsverlustintervalle (423–513 und 513–613 K). Die Analyse der TG-Kurven wurden anhand der Gleichungen von 18 Festkörperreaktionen mittels der Coats–Redfern Methode (1964), der Methode von Šatava und Škvára (1969) sowie der Methode von Blazejowski und Mitarb. (1983) durchgeführt. Auf Grund des Linearisierungsverfahrens erwiesen sich höhere Reaktionsordnungen mit $n = 1{,}5$ und 2 am geeignetsten zur Beschreibung beider TG-Kurven. Die Aktivierungsenergie der beiden Konversionsschritte (für die kleinere und größere Aufheizgeschwindigkeit) beträgt: 1. Schritt: 163,6, 152,6 und 2. Schritt: 238,1 und 167,7 kJ·mol^{-1}.

Резюме — Термогравиметрические измерения метаванадата аммония, проведенные при двух скоростях нагрева (10 и 1,25 К·мин$^{-1}$), показали две различные кривые потери веса в интервалах 423–513 и 513–613 К. Анализ кривых ТГ был проведен на основе уравнений для 18 твердотельных реакций, связанных с методами Коутса–Редферна, Шатавы–Шквары, а также Блазейовски с сотр. Кажется, что высокие порядки реакции $n = 1{,}5$ и 2,0 наилучше описывают обе ТГ кривые. Энергии активации первой стадии превращения для быстрого и медленного нагрева, равны, соответственно, 163,6 и 152,6 кдж·моль$^{-1}$, а для второй стадии — 238,1 и 167,7 кдж·моль$^{-1}$.