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EFFECT OF STRUCTURAL FACTORS ON THE
CATALYTIC ACTIVITY OF BETA-DIKETONATES
OF 3d-ELEMENTS IN THE THERMAL
DECOMPOSITION REACTION OF AMMONIUM
PERCHLORATE

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Foreign Technology Division
Wright-Patterson Air Force Base, Ohio

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PERCHLORATE

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Block	Italic	Transliteration	Block	Italic	Transliteration
А а	<i>А а</i>	A, a	Р р	<i>Р р</i>	R, r
Б б	<i>Б б</i>	B, b	С с	<i>С с</i>	S, s
В в	<i>В в</i>	V, v	Т т	<i>Т т</i>	T, t
Г г	<i>Г г</i>	G, g	У у	<i>У у</i>	U, u
Д д	<i>Д д</i>	D, d	Ф ф	<i>Ф ф</i>	F, f
Е е	<i>Е е</i>	Ye, ye; E, e*	Х х	<i>Х х</i>	Kh, kh
Ж ж	<i>Ж ж</i>	Zh, zh	Ц ц	<i>Ц ц</i>	Ts, ts
З з	<i>З з</i>	Z, z	Ч ч	<i>Ч ч</i>	Ch, ch
И и	<i>И и</i>	I, i	Ш ш	<i>Ш ш</i>	Sh, sh
Й й	<i>Й й</i>	Y, y	Щ щ	<i>Щ щ</i>	Shch, shch
К к	<i>К к</i>	K, k	Ъ ъ	<i>Ъ ъ</i>	"
Л л	<i>Л л</i>	L, l	Ы ы	<i>Ы ы</i>	Y, y
М м	<i>М м</i>	M, m	Ь ь	<i>Ь ь</i>	'
Н н	<i>Н н</i>	N, n	Э э	<i>Э э</i>	E, e
О о	<i>О о</i>	O, o	Ю ю	<i>Ю ю</i>	Yu, yu
П п	<i>П п</i>	P, p	Я я	<i>Я я</i>	Ya, ya

*ye initially, after vowels, and after ъ, ь; e elsew nere.
 When written as ě in Russian, transliterate as yě or ě.
 The use of diacritical marks is preferred, but such marks
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RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English
sin	sin
cos	cos
tg	tan
ctg	cot
sec	sec
cosec	csc
sh	sinh
ch	cosh
th	tanh
cth	coth
sch	sech
csch	csch
arc sin	$\sin^{-1}$
arc cos	$\cos^{-1}$
arc tg	$\tan^{-1}$
arc ctg	$\cot^{-1}$
arc sec	$\sec^{-1}$
arc cosec	$\csc^{-1}$
arc sh	$\sinh^{-1}$
arc ch	$\cosh^{-1}$
arc th	$\tanh^{-1}$
arc cth	$\coth^{-1}$
arc sch	sech^{-1}
arc csch	csch^{-1}
rot	curl
lg	log

GREEK ALPHABET

Alpha	A	α	•	Nu	N	ν
Beta	B	β		Xi	Ξ	ξ
Gamma	Γ	γ		Omicron	O	o
Delta	Δ	δ		Pi	Π	π
Epsilon	E	ϵ	•	Rho	P	ρ •
Zeta	Z	ζ		Sigma	Σ	σ •
Eta	H	η		Tau	T	τ
Theta	Θ	θ	•	Upsilon	T	υ
Iota	I	ι		Phi	Φ	ϕ •
Kappa	K	κ	•	Chi	X	χ
Lambda	Λ	λ		Psi	Ψ	ψ
Mu	M	μ		Omega	Ω	ω

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Effect of ^{structural} ~~structure~~ factors on the catalytic activity of ~~the~~ β -
~~diketone~~ ^{diketone} of 3d-elements ⁱⁿ ~~the reaction of the~~
~~diketone of electrolysis elements~~ ^(reaction)
thermal decomposition of ammonium perchlorate.

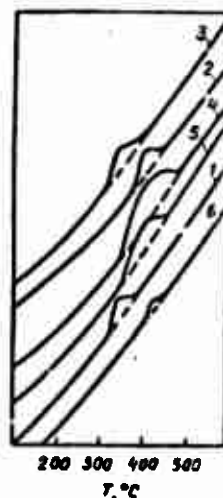
In the present work ~~is~~ investigated the effect of the structure
factors of the β -dicarbonyl of ^{3d} ~~the~~ compounds of ~~electrolysis~~

elements on the rate of the nonisothermal decomposition of ammonium perchlorate (PXA). The study of their thermal resistance under conditions of nonisothermal heating in the atmosphere, ~~concentrated by~~ ^{enriched with} oxygen, ~~it~~ showed that in the final stage of the decomposition of substance is ~~exhibited~~ ^{manifested} the sharp and intense ~~exoeffect~~ ^{exoeffect}, ~~no in the~~ ^{which is a} in inert atmosphere or in air [1]. The observed in this case value of heat release distorts the shape of the curve of temperature rise ~~on~~ ^{at} 50-60° C (Fig. 1). The presence of this effect and the temperature interval of its manifestation in conjunction with data [2-5] about the effectiveness of tris ^{(acetylacetonate) iron} ~~(acetylacetonate) glan~~ (III) in an increase in the rate of combustion of some systems ~~on the basis of~~ ^{with a} PXA and nitromethane ^{base} give grounds to assume that the investigated compounds will turn out to be catalytically active in the processes of the thermal decomposition of PXA and similar oxidizers.

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THE TABLE (FIGURE) WHICH MAY HAVE BEEN LEFT OUT*****

Fig. 1. Nature of the distortion of the shape of the curve of
 temperature rise *during formation of thermographs of acetylene*
~~at the thermograph of acetylene~~ in the
 atmosphere, *enriched* by oxygen (according to *data in* [1]): 1 - [Cu
 $(C_5H_7O_2)_2$]; 2 - [CO $(C_5H_7O_2)_2$]; 3 - [CO $(C_5H_7O_2)_3$]; 4 - [Mn
 $(C_5H_7O_2)_3$]; 5 - [Fe $(C_5H_7O_2)_3$]; 6 - [CR $(C_5H_7O_2)_3$].



In our investigations ^{we used} ~~utilized~~ a specimen/sample ^{of} the PXA of ^{manufacture} industrial ~~izgotavleniya~~ with average particle size $\sim 30 \mu$.

Page 760.

The conditions of the obtaining and property ^{the} of the investigated by us ^{is} ~~β -diketone~~ ^{dicarbonyl} compounds Mn^{3+} , Mn^{2+} , Cu^{2+} , Co^{3+} , Co^{2+} , Fe^{3+} , Cr^{3+} on the basis of acetylacetone, benzoyl acetone, ^{dibenzoylmethane,} ~~dibenzoylacetone,~~ ^{benzoyltrifluoroacetone,} ~~benzoyltrifluoroacetone,~~ ^{benzoyltrifluoroacetone} and 3-nitroacetylacetone are described earlier [6, 7].

^{To ensure uniform} ~~for the safeguard for even~~ distribution of catalyst in the bulk of the specimen/sample of PXA is used the procedure, described in work [1]. The effect of the indicated compounds on rate and nature of the decomposition of PXA was investigated by thermogravimetric method on derivatograph [8], equipped with the system of the ^{extraction} ~~suction~~ of decay products from the heated space, which largely eliminated the possible

effect of gaseous decay products on the kinetics of decomposition.

(weighed quantity)
sample - 50 mg, the average rate of climb of temperature is 19°C ~~in~~ ^{per} min, on straight portion - 22°C ~~in~~ ^{per} min, the content of catalyst is 10% of the weight of PXA).

Table 1.

*****EDITOR: PLEASE CHECK FOR ALL CYRILLIC OR UNCLEAR ITEMS ON

THE TABLE (FIGURE) WHICH MAY HAVE BEEN LEFT OUT*****

diketonates
Table 1. Effect of the β -diketonates of d-elements of the type $[M^{n+} (RCOCHCOR)_n]$ ^{on} ~~to~~ rate and nature of the thermal decomposition of PXA.

Key: (1) catalyst; (2) temperature ($^{\circ}C$) and the

reduction in
~~loss/depreciation~~ of the mass (o/o) of the first and second

effects;

~~exco-effectov;~~ (3) the temperatures of beginning and end of the

reduction in t_{decomp} 80% of the weighed quantity of PXA,
~~loss/depreciation~~ of mass: (4) ~~change~~; (5) ligand.

Table 1. Effect of the β -diketonates of d-elements of the type $[M^{n+} (RCOCHCOR)_n]$ on rate and nature of the thermal decomposition of PXA.

(1) Катализатор		(2) Температура ($^{\circ}C$) и убыль массы (%) пер- вого и второго экзо- эффектов				(3) Температуры на- чала и конца убыли массы			(4) t_{decomp} 80% взвешен. PXA, мин
M^{n+}	(5) лиганд	T_1	Δm_1	T_2	Δm_2	θ_n	θ_k	$\Delta \theta$	
—	—	325	15	460	85	300	415	155	7.0
Co^{2+}	$C_6H_5COCHCOC_6H_5$	315	100	—	—	297	318	21	1.0
Co^{3+}		320	83	360	17	290	320	30	1.4
Mn^{2+}		295	24	335	76	300	335	35	1.6
Ca^{2+}		320	85	375	15	280	322	42	1.9
Fe^{2+}		335	16	400	84	300	390	90	4.1
Cr^{2+}		325	15	460	85	300	460	160	7.3
Co^{2+}	$CH_3COCHCOC_6H_5$	310	—	378	100	320	372	52	2.4
Mn^{2+}		335	100	—	—	315	355	40	1.8
Ca^{2+}		315	85	370	15	302	328	26	1.2
Fe^{2+}		340	15	425	85	326	430	101	4.7
Cr^{2+}		328	15	400	85	298	420	122	5.5
Co^{2+}	$C_6H_5COCHCOC_6H_5$	335	81	410	19	325	370	25	1.1
Mn^{2+}		311	53	385	47	320	380	60	2.7
Ca^{2+}		320	85	372	15	313	329	16	0.7
Fe^{2+}		335	15	408	85	322	410	88	4.0
Cr^{2+}		310	15	440	85	318	450	132	6.0
Ca^{2+}	$CF_3COCHCOC_6H_5$	315	80	385	20	295	325	30	1.4
Mn^{2+}		315	15	360	85	300	372	72	3.3
Ca^{2+}	$CF_3COCHCOC_6H_5S$	320	91	360	9	310	330	20	1.0
Mn^{2+}		322	15	365	85	310	370	60	2.7
Co^{2+}	$CH_3COC(NO_2)COC_6H_5$	360	100	—	—				0.7
Ca^{2+}		320	75	370	25				
Cr^{2+}		310	15	460	85				

The data
~~These~~ on effect ^{of} ~~p-diketonate~~ on the rate of nonisothermal decomposition ^{of} PXA are represented in Table 1.

Page 761.

All the investigated compounds, with the exception of the tris
(acetylacetonate)
(~~acetylacetonate~~) of chromium (III), are catalytically ~~are~~ active in
the process of the thermal decomposition of PXA under nonisothermal
conditions and accelerate this process to 10 times. Most effective
from the viewpoint of shortening ~~in~~ the duration of process as a whole
turn out to be the compounds, which affect predominantly the initial
stages of the decomposition of PXA (Fig. 2). ^{The} Most effective of the
number of examined compounds increase the degree of the conversion of
PXA from 15 to 75 - 100%. It is characteristic that the temperature
of the manifestation of the first exothermic ^{effect on the decomposition} ~~response to expansion~~
^{of} PXA does not undergo substantial changes; however, the

decomposition

temperature interval of the full expansion of PXA in the presence of

catalysts sharply is reduced in accordance with the degree of

shortening in the duration of the process of decomposition of the

weighed quantity

charge of PXA. The most effective catalysts prove to be the

derivatives Cu^{2+} and Co^{2+} , are least active - Fe^{3+} and Cr^{3+}

Intermediate position ^{are} ~~they~~ occupy ^{by diketonates} β -diketonate Co^{3+} , Mn^{3+} and Mn^{2+}

In proportion to the complication of organic ligand is ^{increased} ~~raised~~

catalytic activity in accordance with a change in the electron-donor

properties of ligands. This fact finds natural explanation under the

assumption about the initiation of the ^{examined} ~~considered~~ reaction by means of

the transfer (redistribution) of charge ^{during} ~~with~~ the coordination of

reagents with catalyst, since in this case, according to [9],

catalytic activity depends on the donor-acceptor properties of

partners, which are determined by the Fermi level and by the

oxidation-reduction (catalytic) capacities of reagents and catalyst.

The latter completely ^{definitely} ~~determined form~~ depend on the parameters of the

electronic structure of molecules. Actually (Table 2), there is a

determined interrelation between the stability of the β -diketonate

about which we ~~is~~ judge^{of the} from the value force constant connection $M -$

0 [10], and the value of the catalytic activity, which ^{increases} ~~grow/rises~~ in

proportion to an increase in the stability of catalyst. The indirect

confirmation of this conclusion ⁱⁿ ~~derivation~~ are the data Tables 1 on

^{greater,} ~~larger~~ in comparison with ^{acetylacetonates,} ~~the acetylacetonates~~ of the catalytic

^{Chelates} activity of ~~chelates~~ on the basis of the more complex ligands, which

are characterized, as this is shown in [1, 7], by larger ^{natural} ~~its own~~

thermal resistance.

12
11
10
9
8
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3

*****EDITOR: PLEASE CHECK FOR ALL CYRILLIC OR UNCLEAR ITEMS ON

THE TABLE (FIGURE) WHICH MAY HAVE BEEN LEFT OUT*****

Table 2. Dependence of the catalytic activity of the β -diketonates^{es} of d-elements of the type $[M^{n+}(\text{RCOCHCOR}^1)_n]$ in the process of the thermal decomposition of PXA ~~from~~ ^{on} the parameters of the structure of catalyst.

β -diketonate;
Key: (1) ~~β -diketonat~~; (2) ~~charges~~. σ^* in β -diketones;

(3) t_{decomp} 80% of the weighed quantity of PX

(4) $M-O, K \cdot 10^{-5}$, dyne/cm [10]

Table 2. Dependence of the catalytic activity of the β -diketonates of d-elements of the type $[M^{n+}(\text{RCOCHCOR}^1)_n]$ in the process of the thermal decomposition of PXA on the parameters of the structure of catalyst.

(1) β -Дикетонат	(4) $M-O, K \cdot 10^{-5}$, дин/см [10]	(2) σ^* в β -дикето- нах [11-12]	(3) $t_{\text{разл}} 80\%$ навес- ки ПХА, мин
$[\text{Fe}(\text{C}_6\text{H}_7\text{O}_2)_2]$	1.65	0	4.1
$[\text{Cu}(\text{C}_6\text{H}_7\text{O}_2)_2]$	2.20	0	1.9
$[\text{Co}(\text{C}_6\text{H}_7\text{O}_2)_2]$	2.40	0	1.4
$[\text{Cu}(\text{C}_{10}\text{H}_9\text{O}_2)_2]$	—	0.6	1.2
$[\text{Cu}(\text{C}_{10}\text{H}_{11}\text{O}_2)_2]$	—	1.2	0.7
$[\text{Cu}(\text{C}_6\text{H}_7\text{O}_2\text{SF}_2)_2]$	—	3.0	1.0
$[\text{Cu}(\text{C}_{10}\text{H}_9\text{O}_2\text{F}_2)_2]$	—	3.2	1.4
$[\text{Cu}[(\text{CH}_3\text{CO})_2\text{CNO}_2]_2]$	—	3.5	0.7

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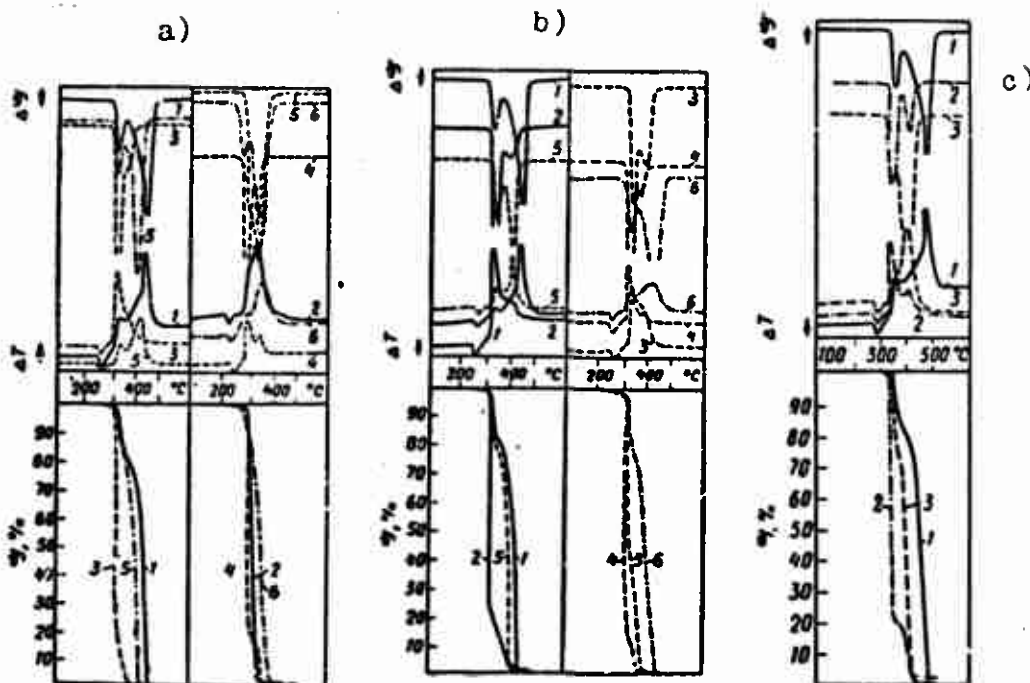
THE TABLE (FIGURE) WHICH MAY HAVE BEEN LEFT OUT*****

Fig. 2. *Derivatogram of* ~~Derivatogram of~~ PXA with addition ^{of} 10/o ~~of~~ ^{ES} ~~β-diketonates~~
(average rate of ^{rise} ~~climb~~ of the temperature 190° C ^{per} ~~in~~ min).

benzoylacetates:
a) the ~~Benzoilacetates~~ (1 - PXA; 2 - PXA + [CO (C₁₀H₉O₂)₃]; 3 -
PXA + [Mn (C₁₀H₉O₂)₃]; 4 - PXA + [Cu (C₁₀H₉O₂)₂]; 5 - PXA + [Fe
(C₁₀H₉O₂)₃]; 6 - PXA + [CR (C₁₀H₉O₂)₃]. b) the *dibenzoylmethan*
~~Benzoilacetates~~:

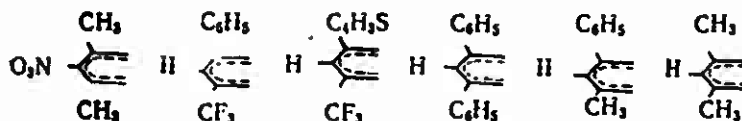
1 - PXA; 2 - PXA + [CO (C₁₅H₁₁O₂)₃]; 3 - PXA + [Mn (C₁₅H₁₁O₂)₃]; 4 -
PXA + [Cu (C₁₅H₁₁O₂)₂]; 5 - PXA + [Fe (C₁₅H₁₁O₂)₃]; 6 - PXA + [CR

benzoyltrifluoroacetates:
(C₁₅H₁₁O₂)₃]. c) the ~~Benzoiltrifluoroacetates~~: 1 - PXA; 2 - PXA +
[Cu (C₁₀H₆O₂F₃)₂]; 3 - PXA + [Mn (C₁₀H₆O₂F₃)₂].



Page 763.

The experimental data make it possible to arrange the investigated β -diketonates according to the degree of the effect of substituent's nature on catalytic activity in the following series:



The obtained sequence ^{indicates} ~~testifies to~~ the presence of the correlation between the catalytic activity of β -diketonates in the reaction of the thermal decomposition of PXA and the total ^{induction} ~~inductive~~ constant ^{of Taft} ~~* the Taft~~ of the substituents [11, 12] in β -diketone (Table 2), ^{which is the} ~~by the being~~ indirect characteristic of the degree of

localization of electron density (value of effective charge) on the central ion of metal. The observed in this case certain

disturbance/breakdown of law ^{of the change} in the lower part of the Table,

apparently, is connected with ^{differences} distinctions in the effect of the

investigated compounds on the final stages of the decomposition of PXA

(Fig. 2).

Difficulties in the study of process are here connected also with the large curvature of the corresponding sections of the curves of

DTG [DTP- derivatogram].

The obtained results give grounds to assume ~~to be~~ participation ^{event} in the ~~event/report~~ of catalysis of the d-electron of metal and to

determine the direction of the transfer of charge in system ^{of} catalysts-

reagents. From them also it follows that in spite of the widespread

opinion, in accordance with which the catalysts of the thermal

decomposition of PXA take part predominantly in the high-temperature

reactions of heterogeneous decomposition of ^{perchloric} ~~chloric~~ acid and oxidation

of ammonia [13], at least, under some conditions, catalyst contributes

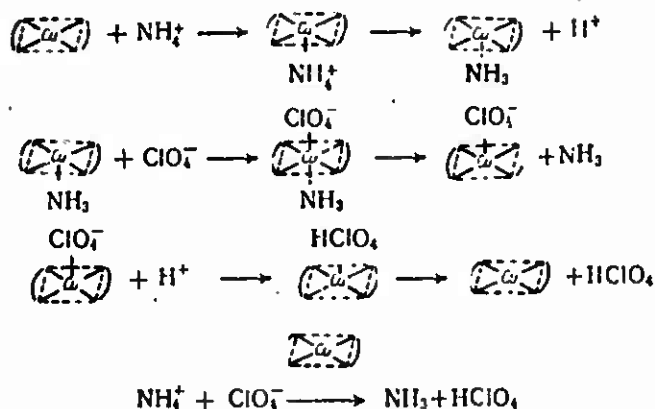
also to acceleration and the low-temperature stage of the

decomposition of PXA, which leads to formation ^{of} HClO_4 and NH_3 .

The findings make it possible to assume the following diagram of

*Chelates*the mechanism of this process (in an example of the ~~chelat~~ of copper

(II)):



The proposed mechanism agrees with known charge distribution on

the atoms bis-(~~acetylacetonate~~) of copper (II) [14]. The velocityof the coordination of ammonium ion and the ~~cleavage~~ *splitting* of proton(especially the ~~cleavage~~ *splitting* of proton) ~~is~~ depends on the value of

positive charge on the atom of transition metal. It must be noted

also that this complex can coordinate the determined atomic groupings
the 5th on ~~5th~~ or 6th place.

Page 764.

This mechanism agrees also with the established-above dependence of
catalytic activity on the value of effective charge on central atom.

This diagram of the mechanism of catalytic decomposition PXA agrees

well with the conventional concepts about the mechanism of the

~~noncatalytic~~
~~noncatalytic~~ decomposition of PXA. The ~~examined~~ diagram can be,

apparently, transferred, also, to octahedral complexes, if one

considers that during the first stage of reaction under the considered

conditions can occur the temperature ^{splitting} ~~cleavage~~ of ligands and they

become coordination-~~non~~ ^{non?} saturated, in the same way as this was

observed in work [15] during the study of the thermal resistance of

the coordination-~~non~~ saturated complexes on the basis of the β -

diketonates ^{ES} of cobalt (II). It goes without saying that the

possibility of the realization of concrete intermediate stages

requires supplementary substantiation and experimental confirmation.

Nevertheless it seems that the findings can be the basis of the

directed search for the catalysts of the ^{examined} ~~considered~~ process.

Preferable turn out to be complexes with the low value of effective

charge on the atom of metal and, therefore, with ligands with the

large positive values of the inductive ^{on} constants of the substituents.

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