

Studies of Acetate Interaction Reactions Manganese and copper with N-methylurea

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ABSTRACT

The kinetic laws of the condensation reaction between metal acetates are investigated: - manganese and copper $\text{Me}[\text{OC}(\text{O})\text{CH}_3]_2$ ($\text{Me}(\text{Ac})_2$) with N-methylolurea. The numerical values of the rate constant at various temperatures and the activation energy of the condensation process between these compounds are estimated. A conclusion is drawn regarding the activity of the acetates of the studied metals in the reactions of their interaction with N-methylolurea.

Keywords: N-Methylolurea; Manganese and Copper Acetates; Rate Constant; Activation Energy

Introduction

The synthesis and study of the properties of alcoholates of metals of variable valence has been the subject of many works, of which the authors' work deserves special attention [1-3]. Alcoholates of metals of variable valency are interesting in that they exhibit catalytic activity both in the synthesis of individual organic compounds and in the complex radical polymerisation of vinyl monomers [4,5]. So, in the aforementioned work [4], in particular, a high catalytic activity of metal alcoholates-manganese and copper was noted. Meanwhile, in the scientific literature there is practically no data on a quantitative assessment of the formation of alcoholates from acetates of the above metals. This report is devoted to a detailed study of the reaction between metal acetates: -manganese and copper with N-methylurea at various temperatures, followed by determination of both the rate constant and the activation energy of their interaction.

Experimental Part

$\text{Mn}[\text{OC}(\text{O})\text{CH}_3]_2 \cdot 4\text{H}_2\text{O}$ and $\text{Cu}[\text{OC}(\text{O})\text{CH}_3]_2 \cdot \text{H}_2\text{O}$ were used in the studies "chda" brand, N - methylolurea was synthesized according to the method described in detail in [6] and identified by IR spectroscopy on a NIKOLET / FT-IRNEXUS spectrophotometer. Getting MMch. 15 g (0.25 mol) of urea and 20 g of a 38% aqueous formaldehyde solution (0.25 mol) are loaded into the reactor and stirred at a temperature of (35 ± 0.5) OC for 30 minutes.

Then under low pressure (15-20) mm. Hg. Art. at a temperature of $(45-50)^\circ\text{C}$ water is distilled off. The white precipitate (MMh) is repeatedly washed and recrystallized with ethanol and dried under pressure (15-20) mm Hg. at $50-60^\circ\text{C}$ until a constant mass is achieved. The yield of MMh is 93%, the melting point (T_m) is $(111 \pm 0.5)^\circ\text{C}$. The elemental composition in (%) was established: C-26.6(26.67); H6.8(6.66); N-31.2(31.11). In our studies, a mixture of dimethylformamide (DMF) with water in a volume ratio (1:1) was used as an MMh solvent. Acetic acid obtained during the reaction was determined by volumetric analysis i.e. titration of 4 ml of the sample with 0.1 N aqueous alkali solution (NaOH) prepared from fixanal. The indicator was phenolphthalein. The total volume of the reaction mixture was 50 ml.

The Discussion of The Results

The kinetic regularities of the accumulation of acetic acid formed in the reaction between $\text{Mn}[\text{OC}(\text{O})\text{CH}_3]_2$ ($\text{Mn}(\text{Ac})_2$) and N-methylolurea (MMh) at various temperatures studied, depending on the reaction time, are given in Table 1. From the data in Table 1 it follows that the reaction between $\text{Mn}(\text{Ac})_2$ and MMh is a second-order reaction, and the rate constant is described [7] by the equation below

$$k = \frac{1}{t} \cdot \frac{1}{A_o - B_o} \ln \frac{B_o(A_o - x)}{A_o(B_o - x)} \quad (1)$$

Using the data in Table 1 and plotting the dependencies $\frac{1}{A_0 - B_0} \ln \frac{B_0(A_0 - x)}{A_0(B_0 - x)}$ from t (reaction time) are estimated and presented in Table 2 values of the reaction rate constant between Mn (Ac)₂ and MMh at different temperatures. Induction periods shown in Figure 1 regularities can be attributed to the lifetime of the intermediate compound formed upon the interaction of MMh with Mn (Ac)₂. The

probable structure of the intermediate compound and its further decay can be represented as:

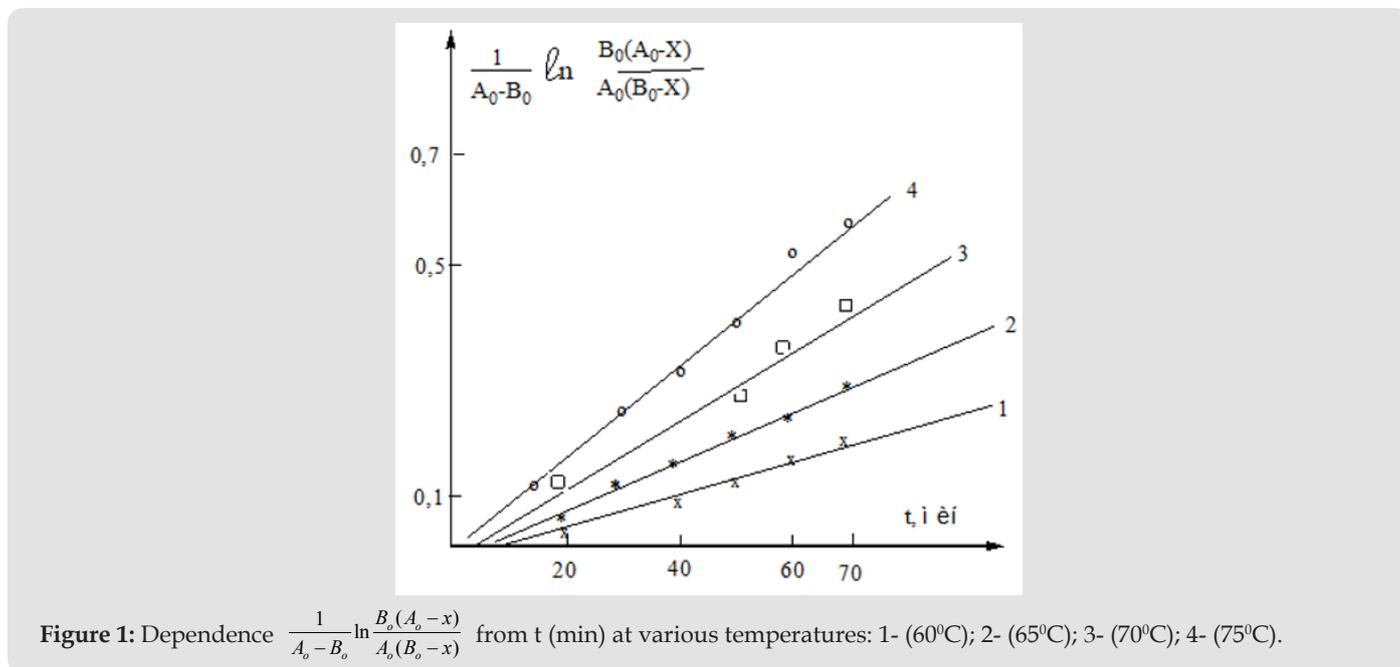
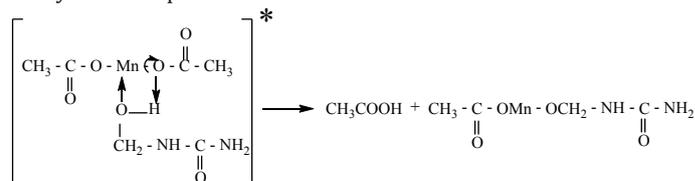


Figure 1: Dependence $\frac{1}{A_0 - B_0} \ln \frac{B_0(A_0 - x)}{A_0(B_0 - x)}$ from t (min) at various temperatures: 1- (60°C); 2- (65°C); 3- (70°C); 4- (75°C).

Table 1: Kinetics of the reaction between Mn (Ac)₂ and MMh at different temperatures [MMh]₀ = (A)₀=0.6 mol / l; [Mn (Ac)₂]₀ = (V)₀=0.3 mol/L.

$\frac{N_0}{N}$ $\frac{n}{n}$	The concentration of acetic acid (x) during the reaction time Mn (AC) 2 with MMh at temperatures:							
	600C		650C		700C		750C	
	t.min	$X \cdot \frac{M}{\pi}$	t.min	$X \cdot \frac{M}{\pi}$	t.min	$X \cdot \frac{M}{\pi}$	t.min	$X \cdot \frac{M}{\pi}$
1	8,0	0,004	15	0,001	20	0,014	15	0,018
2	30,0	0,008	20	0,008	30	0,018	20	0,02
3	40,0	0,014	25	0,012	40	0,024	30	0,032
4	50,0	0,022	30	0,014	50	0,042	40	0,044
5	60,0	0,025	40	0,022	60	0,052	50	0,062
6	70,0	0,032	50	0,032	70	0,067	60	0,08
7	-	-	60	0,038	-	-	70	0,088
8	-	-	70	0,045	-	-	-	-

Table 2: The values of the reaction rate constant between MMh and Mn (Ac)₂ at various temperatures.

T (°K)	333	338	343	348
k x 10 ⁵ (l / mol sec)	5,3	6,0	10,02	14,6

Using the Arrhenius equation $k = Ae^{-E_a/RT}$ (2) [7] and constructing the dependence of $\ln k$ or $\ln \left(\frac{1}{T}\right)$ (Figure 2) based on the numerical values of k of (Table 2), the activation energy (E_a) the reaction of the interaction of MMC with Mn (Ac)₂, which turned out to be equal to (23±2) kcal / mol. The data in Table 3 indicate that the reaction between Cu (Ac)₂ and MMh, like the reaction of

manganese acetate Mn (Ac)₂ with MMh, is a second-order reaction, and the rate constant is described by equation (1). Using the data in (Table 3) and plotting the dependencies $\frac{1}{A_0 - B_0} \ln \frac{B_0(A_0 - x)}{A_0(B_0 - x)}$ from t (reaction time) are estimated and presented in (Table 4) values of the reaction rate constant between Cu (Ac)₂ and MMh at different temperatures (Figure 3). Yeritsyan M.L. Using the data of Table 4

and plotting the dependence of $\ln k \cdot OT \left(\frac{1}{T}\right)$ (Figure 4), the value of the activation energy of the reaction between $\text{Cu}(\text{Ac})_2$ and MMh was estimated, which turned out to be $10 \pm 1.5 \text{ kcal/mol}$. Thus,

from a comparison of the numerical values of the interaction activation energy, we can conclude that copper acetate ($\text{Cu}(\text{Ac})_2$) in the reaction with MMh is more active than manganese acetate ($\text{Mn}(\text{Ac})_2$).

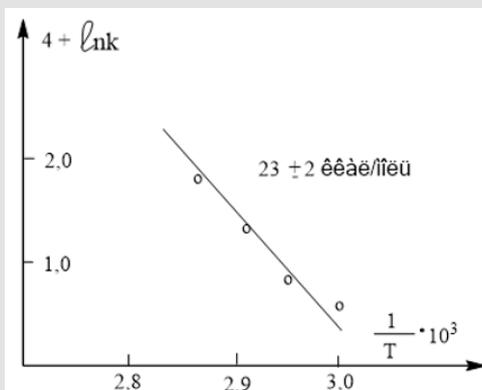


Figure 2: Dependence $k \cdot OT \left(\frac{1}{T}\right)$.

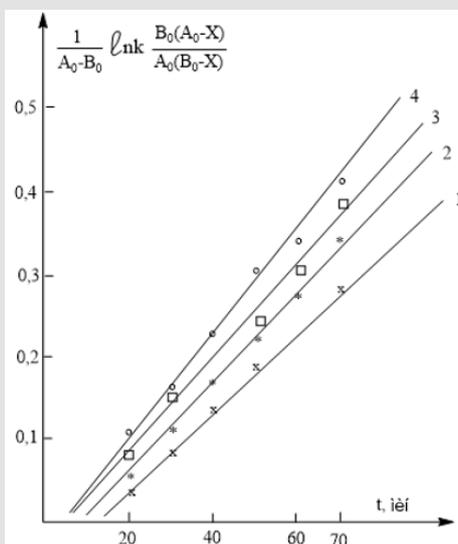


Figure 3: Dependence $\frac{1}{A_0 - B_0} \ln \frac{B_0(A_0 - x)}{A_0(B_0 - x)}$ from OT t (min) at various temperatures: 1- (600C); 2-(650C); 3- (700C); 4- (750C)..

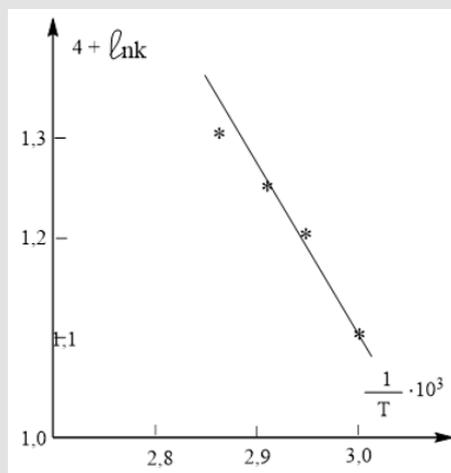


Figure 4: Dependence $\ln k \cdot OT \left(\frac{1}{T}\right)$.

Table 3: The change in the concentration of acetic acid over the course of the reaction between $[\text{Cu}(\text{Ac})_2]$ and (MMh) at various temperatures studied: $[\text{MMh}]_0 = (A)_0 = 0.6 \text{ mol/l}$; $[\text{Cu}(\text{Ac})_2]_0 = (V)_0 = 0.3 \text{ mol/L}$.

The concentration of acetic acid (x) in time the reaction of $\text{Cu}(\text{Ac})_2$ with MMh at temperatures:							
60°C		65°C		70°C		75°C	
t.min	$X \cdot \frac{M}{\pi}$	t.min	$X \cdot \frac{M}{\pi}$	t.min	$X \cdot \frac{M}{\pi}$	t.min	$X \cdot \frac{M}{\pi}$
20	0,006	9	0,04	10	0,007	10	0,009
30	0,015	20	0,01	20	0,015	20	0,015
40	0,025	30	0,019	30	0,025	30	0,034
50	0,032	40	0,027	40	0,037	40	0,041
60	0,043	50	0,036	50	0,044	50	0,047
70	0,045	60	0,044	60	0,048	60	0,052
		70	0,054	70	0,051	70	0,069

Table 4: The values of the reaction rate constant between $\text{Cu}(\text{Ac})_2$ and MMh. at various temperatures.

T (OK)	333	338	343	348
$k \times 10^5 \text{ (l/mol sec)}$	8,2	9,1	9,5	10,02

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