

Thermodynamic Characteristics of Ethyl-2-cyano-3-(furan-2-yl)-prop-2-enoate Derivatives

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Abstract. Using bomb calorimetry and Knudsen effusion methods the enthalpic characteristics in condensed state of ethyl-2-cyano-3-(furan-2-yl) prop-2-enoate derivatives were experimentally determined. The values of the enthalpies of formation in the gas state of investigated esters were calculated taking into account the values of the enthalpies of sublimation adjusted to 298K. From obtained thermochemical values we defined new group contributions which can be added to the Benson's additive scheme. Using these increments the enthalpies of formation of investigated compounds in gas state were theoretically calculated and their differences from the experimentally determined were analyzed.

Keywords: Bomb calorimetry, effusion method, Benson's scheme

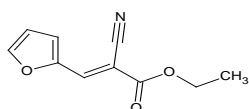
1 Introduction

Heterocyclic compounds are widely distributed in nature [1]. Many of them are particularly important for living organisms and they serve as a key components of biological processes. Many of drugs are derived from natural products, but their number is much greater than those synthesized from natural or synthetic compounds [2]. In particular, furan derivatives are commonly used in the synthesis of biologically active compounds that exhibit antibacterial, antitumor, and tuberculostatic action [3]. Determination of thermodynamic properties of substances with widespread use will help to optimize the processes of their synthesis and processing, as well as providing an opportunity to explore the energetic properties of molecules of this class. Considering these diverse information the need of a greater number of values of thermochemical parameters for esters of furanacrylic acid becomes evident. Despite this there are no reported in literature values of the standard molar enthalpies of formation in the crystalline and gas phase, as well as the standard molar enthalpies of sublimation of investigated compounds.

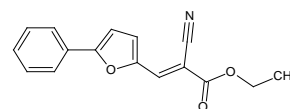
The present study provides results of the purification, standard molar energy of combustion, standard molar enthalpy of sublimation, and standard molar enthalpy of formation in both crystalline and gaseous states, $T = 298.15$ K, for the five compounds. The experimental determination of the standard energies of combustion of the five esters in oxygen at $T = 298.15$ K was conducted using static-bomb combustion calorimetry. From the temperature dependence of the vapour pressure using linear form of the Clausius-Clapeyron equation, the standard molar enthalpies of sublimation, at the average temperature were derived. Standard molar enthalpies and entropies of sublimation at the temperature of 298.15 K were calculated using estimated values for the heat capacity differences between the gas and the crystal phases of each compound. From the experimental values of the standard molar enthalpies of formation in the condensed phase and the values of the standard molar enthalpies of sublimation at $T = 298.15$ K the standard molar enthalpies of formation in the gaseous phase were calculated for the five ethyl esters of furanacrylic acid derivatives.

Due to the wide use of thermochemical characteristics of organic compounds, it is important to find the best theoretical approach for determining these properties, when experimental determination is impossible. Therefore we compared in this work the experimental data with Benson's additive scheme [4].

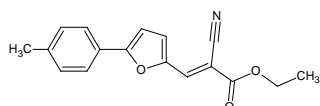
For thermochemical studies we selected number of ethyl esters of furan acrylic acid derivatives:



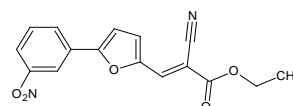
1. ethyl-2-cyano-3-(furan-2-yl)prop-2-enoate (I)



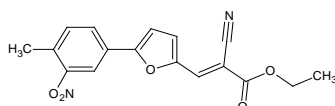
2. ethyl-2-cyano-3-(5-phenylfuran-2-yl)prop-2-enoate (II)



3. ethyl-2-cyano-3-[5-(4-methylphenyl)furan-2-yl]prop-2-enoate (III)



4. ethyl-2-cyano-3-[5-(3-nitrophenyl)furan-2-yl]prop-2-enoate (IV)



5. ethyl-2-cyano-3-[5-(4-methyl-3-nitrophenyl)furan-2-yl]prop-2-enoate (V)

2 Experimental

Ethyl-2-cyano-3-(furan-2-yl)-prop-2-enoate and its derivatives are obtained by the reaction shown, in Fig. 1.

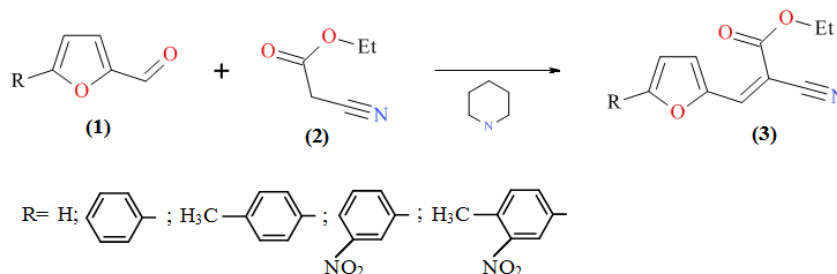


Figure 1. Synthesis scheme of ethyl-2-cyano-3-(furan-2-yl)prop-2-enoate and its derivatives

2-3 drops of piperidine were added to equimolar solution (0.1 mol) of the aldehyde (1) and cyanoacetic ester (2) in 30 ml of ethanol during boiling and mixing stage. The mixture was heated until the formation of precipitate. After cooling the reaction mixture, the precipitate was filtered, washed with ethanol and recrystallized from an ethanol-dimethylformamide mixture [5].

The structures of the substances were confirmed by IR-spectroscopy using a spectrophotometer «Specord». In the spectra of the samples absorption bands that were found that were not inherent substances of this class. The identity of the compound was confirmed by thin layer of chromatography plates Silufol UV-254.

We used samples of substances for thermochemical studies obtained after the fourth and fifth sequential re-crystallization from solvent mixture of ethanol-dimethylformamide.

The temperature dependence of the vapor pressure and enthalpies of sublimation were determined by Knudsen integral effusion method. Features of integral effusion installation are similar to those described

in [6]. The design of the camera and membranes taken from [7] Methodology of experiments was selected in accordance with the recommendations [6].

Vacuum system of the installation has reached the pressure of 0.1 Pa for 45 ± 15 s. The weight of the efunded substance m was determined using analytical scales VLR-20 ($\pm 5 \cdot 10^{-6}$ g) as the difference of the efusion camera weight before and after the experiment. To eliminate the adsorbed moisture and volatile impurities of the sample a series of preliminary experiments were carried out at the same temperature. The experiment was finished, when the evaporation rate m/τ became constant within 1 % deviation.

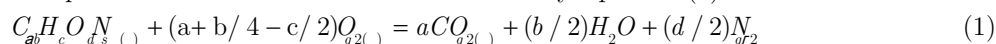
Reliability of the installation was tested in a series of experiments determining the temperature dependence of the vapor pressure of standard benzoic acid K-1 in the temperature range of 322.7-354.1 K using two membranes with the following diameters of holes of 2.050 mm (A) and 2.100 mm (B). Thickness of these membranes was 0.09 mm. The results of experimental determination of the temperature dependence of the vapor pressure of benzoic acid was processed by least squares method and approximated as a linear equation: $\ln P$ (Pa) = $(33.13 \pm 0.92) - (10535 \pm 89) \cdot 1/T$; the correlation coefficient $\rho = 0.998$. The calculated value of the enthalpy of sublimation of benzoic acid was 87.6 ± 0.7 kJ/mol at an average temperature $T_m = 338.4$ K. This value is in concordance with the value obtained by Tomez Gomez by colorimetric method at $T = 335.0$ K $\Delta_{sub}H = 87.5 \pm 0.3$ kJ/mol [8].

Measure vapor pressure implementation of the experiments of investigated substances is similar to the experiments with benzoic acid. The results of the effusion measurements are presented in Table 1, including vapor pressure (P) of the substance in the solid aggregate state and linear equations temperature dependence of vapor pressure in the Clapeyron-Clausius equation coordinates, the value of the enthalpy of sublimation and the temperature ranges (T_m) that were researched. More information about the methodology and calculation of the experiment is presented in [9]

The energy of combustion of investigated substances was determined using precision calorimeter V-08-MA with an isothermal shell (± 0.003 K) and static calorimeter bomb [10]. The energy equivalent of the calorimetric system ($W = 15300 \pm 9$ J/B) with accuracy ± 0.06 % was estimated by combustion of the reference benzoic acid grade K-1 (the major component content—99.995 % mol). Heat of combustion, considers the Jessup factor $\Delta U_b = -26434.4$ J/g.

The compounds under normal conditions are in solid aggregate state. The crystalline esters were grinded in chalcedony mortar before combustion, screened and placed in platinum cup. A cotton thread, tied to the platinum wire ($d = 0.1$ mm) was used for the ignition. The initial pressure of the oxygen, which was previously purified from the combustible impurities, carbon dioxide and water, was equal to $3.02 \cdot 10^6$ Pa. 298.15 K was the initial temperature of the main period in all experiments. After each burning we conducted the quantitative analysis of the combustion products for the presence of carbon – mono and –dioxide, soot and nitric acid. The presence of carbon oxides was indicated by the Rossini method [11] within the accuracy of $\pm 1 \cdot 10^{-4}$ g. Reliability of gas analysis was confirmed by a series of experiments with a reference benzoic acid. The anticipated carbon monoxide that was supposed to be formed during the combustion of products by using detector tubes within $\pm 1 \cdot 10^{-6}$ g, was not encountered. The soot mass was determined by weighting the quartz cup before and after the combustion with the accuracy of $\pm 5 \cdot 10^{-6}$ g. The nitric acid content was detected by titration of the liquid phase in a bomb with a 0.1 M solution of NaOH

The investigated compounds' combustion reaction is described by equation (1):



The internal energy of combustion in the conditions of the experiment was calculated by the formula (2):

$$-\Delta_c U_{298.15} = \frac{W \cdot \Delta T - q_t - q_{HNO_3} + q_s}{m} \quad (2)$$

where: m – weight burned substance; W – energy equivalent of calorimetric system; ΔT – true temperature increase; q_t – amendment to the heat of combustion threads (16704.2 J/g) [12]; q_{HNO_3} – amendment to the heat of formation of nitric acid solution (59 J/g) [12]; q_s – amendment to the heat of combustion of soot (32800 J/g) [12]. More information about the methodology and calculation of the experiment is presented in [9]

Table 1. The results of effusion research of ethyl-2-cyano-3-(furan-2-yl)-prop-2-enoate and its derivatives in the crystalline state.

Sample	T, K	m, g	τ , s	P, Pa	Sample	T, K	m, g	τ , s	P, Pa
<i>1. ethyl-2-cyano-3-(furan-2-yl)-prop-2-enoate (I)</i>									
Membrane (A)					Membrane (B)				
1	327.5	0.00250	3630	1.440	1	327.5	0.00360	3630	1.448
	334.1	0.00490	3640	2.846		334.1	0.00550	3640	2.924
2	331.2	0.00360	3630	2.110	2	326.8	0.00210	3630	1.217
	335.2	0.00550	3650	3.214		331.2	0.00360	3630	2.227
$\ln P(\text{Pa}) = (35.9 \pm 1.9) - (12187 \pm 644) \cdot 1/T$; $\Delta_{\text{sub}}H = 101.3 \pm 5.3 \text{ kJ/mol}$									
<i>2. ethyl-2-cyano-3-(5-phenylfuran-2-yl)-prop-2-enoate (II)</i>									
Membrane (A)					Membrane (B)				
1	361.1	0.00080	14420	0.022	1	361.1	0.00085	14420	0.038
	367.9	0.00200	14424	0.056		367.9	0.00215	14424	0.076
2	365.2	0.00120	14420	0.033	2	365.2	0.00140	14420	0.038
	370.1	0.00190	10821	0.071		370.1	0.00210	10821	0.077
$\ln P(\text{Pa}) = (41.0 \pm 2.0) - (16201 \pm 742) \cdot 1/T$; $\Delta_{\text{sub}}H = 134.7 \pm 6.2 \text{ kJ/mol}$									
<i>3. ethyl-2-cyano-3-[5-(4-methylphenyl)-furan-2-yl]-prop-2-enoate (III)</i>									
Membrane (A)					Membrane (B)				
1	366.2	0.00115	18026	0.025	1	366.2	0.00120	18026	0.025
	372.0	0.00215	14425	0.058		372.0	0.00235	14425	0.062
2	367.8	0.00125	14429	0.033	2	369.6	0.00160	14421	0.042
	369.6	0.00165	14421	0.045		372.8	0.00125	7219	0.066
$\ln P(\text{Pa}) = (43.4 \pm 2.3) - (17239 \pm 877) \cdot 1/T$; $\Delta_{\text{sub}}H = 143.3 \pm 7.3 \text{ kJ/mol}$									
<i>4. ethyl-2-cyano-3-[5-(3-nitrophenyl)-furan-2-yl]-prop-2-enoate (IV)</i>									
Membrane (A)					Membrane (B)				
1	430.2	0.00190	12643	0.060	1	435.9	0.00090	3652	0.096
	435.9	0.00095	3652	0.105		445.2	0.00230	3648	0.249
2	434.6	0.00155	7248	0.086	2	430.9	0.00205	10847	0.074
	439.5	0.00130	3650	0.144		434.6	0.00170	7248	0.092
$\ln P(\text{Pa}) = (37.1 \pm 1.9) - (17168 \pm 855) \cdot 1/T$; $\Delta_{\text{sub}}H = 142.7 \pm 7.1 \text{ kJ/mol}$									
<i>5. ethyl-2-cyano-3-[5-(4-methyl-3-nitrophenyl)-furan-2-yl]-prop-2-enoate (V)</i>									
Membrane (A)					Membrane (B)				
1	406.1	0.00135	14422	0.036	1	406.1	0.00140	14422	0.036
	412.4	0.00130	7228	0.069		408.2	0.00045	3619	0.046
2	399.6	0.00075	14421	0.020	2	399.6	0.00080	14421	0.020
	407.6	0.00180	14419	0.048		402.8	0.00080	10842	0.027
$\ln P(\text{Pa}) = (35.4 \pm 2.0) - (15698 \pm 800) \cdot 1/T$; $\Delta_{\text{sub}}H = 130.5 \pm 6.6 \text{ kJ/mol}$									

The ester's combustion energies determination results are listed in Table 2, which besides the above notation, $m^{\text{exp}}/m^{\text{cal}}$ also specifies the combustion completion by carbon dioxide, that was obtained experimentally.

The standard enthalpies of combustion $\Delta_c H_{298}^0$ (kJ/mol) of investigated compounds were calculated by the average values of internal energy changes in the conditions of the experiment $\Delta_c U$ considering the Washburn's correction π [13], and the extension work correction ΔnRT .

The standard enthalpy of formation $\Delta_f H_{298}^0$ was calculated using standard enthalpy of combustion and standard enthalpy of formation of combustion products (kJ/mol): $\Delta_f H_{298}^0(\text{CO}_{2(\text{gas})}) = 398.512 \pm 0.046$; $\Delta_f H_{298}^0(\text{H}_2\text{O}_{(\text{liq})}) = 285.829 \pm 0.040 \pm 1 \cdot 10^{-4}$; $\Delta_f H_{298}^0(\text{N}_{2(\text{gas})}) = 0$ [14].

The results of calorimetric determination of the standard enthalpies of combustion and formation of these substances in the crystalline state at 298 K are shown in Table 3.

Table 2. Results of the experimental determination of combustion energies of the investigated compounds at 298.15 K.

Sample №	m, g	ΔT , V	q_t	q_{HNO_3}	Q_s	$-\Delta_c U$, J · g ⁻¹	m^{exp}/m^{calc}
			J				
<i>1. ethyl-2-cyano-3-(furan-2-yl)prop-2-enoate (I)</i>							
1	0.35790	0.60428	102.0	14.4	16.7	25553	1.0001
	0.29219	0.49359	112.8	13.6	30.2	25517	0.9990
	0.29703	0.50070	74.2	14.2	26.2	25580	1.0000
2	0.29211	0.49209	97.2	13.0	32.0	25522	1.0000
	0.29484	0.49782	103.3	14.2	32.5	25545	0.9998
	0.29739	0.50340	108.1	14.6	32.8	25599	0.9999
<i>Average value $-\Delta_c U=25552\pm 35$</i>							
<i>2. ethyl-2-cyano-3-(5-phenylfuran-2-yl)prop-2-enoate (II)</i>							
1	0.29137	0.56740	89.1	11.8	26.2	29538	0.9957
	0.29343	0.56931	71.8	7.1	29.4	29515	0.9955
	0.29638	0.57713	93.6	8.3	23.6	29528	0.9950
2	0.29435	0.57230	88.0	8.4	32.4	29530	0.9945
	0.28966	0.56426	90.3	6.2	20.0	29540	0.9960
	0.29006	0.56331	75.6	7.5	25.3	29514	0.9952
	0.29865	0.58046	81.3	9.6	27.6	29525	0.9954
<i>Average value $-\Delta_c U=29527\pm 10$</i>							
<i>3. ethyl-2-cyano-3-[5-(4-methylphenyl)furan-2-yl]prop-2-enoate (III)</i>							
1	0.29570	0.58978	75.3	16.2	36.1	30329	1.0000
	0.28961	0.57877	88.1	14.8	28.0	30317	0.9994
	0.29854	0.59654	91.2	15.6	30.1	30315	0.9990
	0.29457	0.58699	69.7	11.8	23.5	30291	0.9994
2	0.29123	0.58127	74.5	14.8	21.5	30304	0.9985
	0.29456	0.58852	90.4	15.9	25.8	30295	0.9999
	0.29139	0.58110	70.3	14.2	18.7	30286	0.9989
<i>Average value $-\Delta_c U=30305.3\pm 9.5$</i>							
<i>4. ethyl-2-cyano-3-[5-(3-nitrophenyl)furan-2-yl]prop-2-enoate (IV)</i>							
1	0.29498	0.48009	60.4	13.6	25.9	24738	0.9958
	0.28954	0.47231	72.9	9.4	27.4	24768	0.9970
	0.29712	0.48444	55.0	8.9	15.3	24782	0.9997
2	0.29130	0.47627	91.0	13.6	23.8	24737	0.9978
	0.32090	0.52161	68.0	13.6	34.8	24723	0.9979
	0.29198	0.47700	84.3	14.6	30.5	24761	0.9994
	0.30105	0.49123	74.6	15.3	21.9	24739	0.9990
<i>Average value: $-\Delta_c U=24750\pm 13$</i>							
<i>5. ethyl-2-cyano-3-[5-(4-methyl-3-nitrophenyl)furan-2-yl]prop-2-enoate (V)</i>							
1	0.28785	0.48716	80.9	11.2	28.5	25673	0.9935
	0.29072	0.49416	88.9	10.0	24.8	25751	0.9933
	0.29439	0.49926	91.2	14.3	35.6	25710	0.9992
	0.31747	0.53703	75.4	11.8	28.0	25694	0.9944
2	0.29863	0.50571	84.4	13.0	29.4	25681	0.9946
	0.29177	0.49432	89.1	11.2	42.8	25724	0.9986
	0.30993	0.52582	85.2	14.2	29.0	25730	0.9941
<i>Average value: $-\Delta_c U=25709\pm 18$</i>							

Table 3. Standard enthalpy of formation and combustion in the condensed and gaseous state.

Compound	$-\Delta U$	$-\pi$	ΔnRT	$\Delta_c H_{298}^0$	$-\Delta_f H_{298}^0$ (sol)
kJ/mol					
I	4885.2 ± 6.5	3.7	0.6	4889.4 ± 6.5	331.9 ± 6.5
II	7892.1 ± 2.7	5.7	-3.1	7900.1 ± 2.7	253.2 ± 2.7
III	8525.2 ± 2.7	5.9	-4.3	8535.5 ± 2.7	297.9 ± 2.7
IV	7728.8 ± 4.1	6.2	1.2	7735.0 ± 4.1	277.4 ± 4.1
V	8389.0 ± 6.0	6.4	0	8395.6 ± 6.0	295.1 ± 6.0

3 Discussion

The thermochemical properties, identified in this work were defined at different temperatures, which are caused by the conditions of the experiment. Therefore there is a need for adjusting enthalpy of sublimation from average temperature of experiment to 298K. According to the Kirchoff equation for conversion it is necessary to know the change in heat capacity of the appropriate phase transition. Since we do not have experimental data changes in heat capacity, it is possible to use the approximate methods of calculation. To adjust the value of the enthalpy of sublimation we used the equation proposed in [15]

$$\Delta_{sub} H^{298} = \Delta_{sub} H^{T_m} + (0,259 \pm 0,041) \cdot M \cdot (T_m - 298) \quad (3)$$

The values of enthalpy of formation of the compounds at 298 K in gaseous state are calculated by the equation $\Delta_f H_{298}^0$ (gas) = $\Delta_f H_{298}^0$ (sol) + $\Delta_{sub} H_{298}^0$ and are shown in table 4.

Table 4. Enthalpic characteristics of the substances at 298K.

Enthalpy (kJ/mol)	Substance				
	I	II	III	IV	V
$-\Delta_f H_{298}^0$ (sol)	331.9 ± 6.5	331.9 ± 6.5	331.9 ± 6.5	331.9 ± 6.5	331.9 ± 6.5
$\Delta_{sub} H_{298}^0$	102.9 ± 5.6	139.8 ± 7.0	148.9 ± 8.2	154.2 ± 8.0	154.2 ± 8.0
$-\Delta_f H_{298}^0$ (gas)	230.0 ± 8.5	113.4 ± 7.5	149.0 ± 8.6	123.2 ± 9.0	155.3 ± 10

Additive Benson's method is one of the most common theoretical methods for calculating the enthalpy of formation of individual organic compounds in the gas phase [16]. Being studied in this article, compounds are the complex substances containing previously unknown group of additive contributions in Benson's scheme: $C_d - (CN)(C_d)(CO)$ and $C_d - (C_d)(O)(C_b)$. We used enthalpy of formation in the gas phase of compounds I, II and group contributions for their determination referred to in [15,16] and shown in table 5.

Table 5. Used group contributions to calculate the enthalpy of formation of investigated compounds' in gaseous state.

Group type	$\Delta_f H_{298}^0$, kJ/mol	Group type	$\Delta_f H_{298}^0$, kJ/mol
$C_d - (C_d)(O)(H)$	36.61	$CO - (C_d)(O)$	-140.16
$C_d - (C_d)_2(H)$	28.37	$O - (CO)(C)$	-172.80
$O - (C_d)_2$	-137.24	$C - (H)_2(O)(C)$	-35.56
$C_d - (C_d)(O)(C_d)$	43.40	$C - (C)(H)_3$	-42.17
$C_d - (C_d)(H)(C_d)$	28.37	Correction to furan cycle	-25.94
$C_b - (C_b)_2(H)$	13.82		
$C_b - (C_b)_2(C_d)$	23.76	$C_b - (C_b)_2(NO_2)$	-0.52
$C_b - (C_b)_2(H)$	13.82	$C_b - (C_b)_2(N)$	-2.09
$C_d - (CN)(C_d)(CO)$	158.75	$C_d - (C_d)(O)(C_b)$	59.65

To verify the definition of group contributions and their possible application for the calculation we had calculated enthalpy of formation in the gas phase of ethyl-2-cyano-3-[5-(4-methylphenyl)furan-2-yl]prop-2-enoate (III), ethyl-2-cyano-3-[5-(3-nitrophenyl)furan-2-yl]prop-2-enoate (IV), ethyl-2-cyano-3-[5-(4-methyl-3-nitrophenyl)furan-2-yl]prop-2-enoate (V) and compared them with the experimentally determined values (table 6), where Δ – is a difference between the experimental and calculated values.

Table 6. Experimentally determined and calculated by Benson's additive scheme enthalpy of formation of studied ethyl ester's in the gaseous state.

Ester	$-\Delta_f H^\circ_{\text{exp}}$, kJ/mol	$-\Delta_f H^\circ_{\text{calc}}$, kJ/mol	Δ , kJ/mol
III	149.00	146.87	2.13
IV	123.20	127.40	4.20
V	155.30	161.21	5.91

4 Conclusions

A combined experimental and theoretical study has been carried out to obtain the standard molar enthalpies of formation in the gas-phase at 298 K for five ethyl esters of furanacrylic acid derivatives. From the experimental data for ethyl-2-cyano-3-(furan-2-yl)-prop-2-enoate derivatives enthalpies of combustion, and sublimation were determined. Combining these two quantities, the standard molar enthalpies of formation in the gaseous phase at 298 K were obtained. From the obtained values two new increments filling Benson's additive-group scheme were determined. Since, the Benson group method yields estimates of standard molar enthalpies of formation in the crystalline and gas phase are very close to the experimental values, we may conclude that the new increments can be accepted with confidence for the estimation of formation enthalpies for the compounds that are not studied experimentally.

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