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Standard enthalpy of formation of copper(II) pivalate

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ABSTRACT

The combustion energy of crystalline copper(II) pivalate was determined by static-bomb isoperibolic calorimetry. The standard enthalpy of formation was calculated on basis of obtained results. The value of standard enthalpy of formation was used to calculate the enthalpy of breaking bond Cu---Piv by the reaction: $Cu(C_5H_9O_2)_2(cr) = Cu(cr) + 2C_5H_9O_2^-(g)$ and the enthalpy of formation of the trimer $Cu_3(Piv)_6$ in the gaseous state.

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1. Introduction

The development of methods for low-temperature gas-phase synthesis of thin films of metals or their oxides requires the search for suitable precursors. Carboxylates of d-transition metals are good candidates for this role due to their relatively high volatility, thermal stability, simplicity of methods of their production and availability. For the practical use of volatile complex compounds of metals, it is necessary to know the thermodynamic properties determining their applicability, in particular, the enthalpies of sublimation and formation.

Copper(II) pivalate $(Cu(C_5H_9O_2)_2, Cu(Piv)_2)$ is used in the CVD method (Chemical Vapor Deposition) as a molecular precursor for the production of high-temperature superconductors thin films of copper oxides Cu_xO, which in turn have perspective optical, electrical, thermal and magnetic properties [1,2].

In the present work, the enthalpies of combustion and formation of a crystalline copper(II) pivalate by the method of bomb calorimetry were first determined (Fig. 1).

Earlier in [3], we determined the enthalpies of combustion and formation of silver pivalate (AgPiv(cr)). It is known from the literature that the data for this class of complex compounds of copper or silver with organic ligands are scanty [4].

2. Experiment

2.1. Synthesis of sample

Crystalline sample of copper(II) pivalate, $(Cu(C_5H_9O_2)_2$, $(Cu(Piv)_2)$, Fig. 1) was synthesized by dissolution of metallic copper in nitric acid. Then to the resulting solution was added a solution of KOH. The precipitate of copper(II) hydroxide was washed with water (bidistillate) and mixed with pivalic acid. The precipitated $Cu(Piv)_2$ was purified by sublimation in a dynamic vacuum at $P \sim 1.33$ Pa and $T = 420$ K.

2.2. Characterization of sample

The composition of the sample is controlled using a laser mass-spectrometry and chemical analysis of the contents of carbon (C) and hydrogen (H). According to mass spectrometric analysis, the sample contained metals (mass fractions): Na – 0.0015 and K – 0.0014 as basic impurities and less than 0.0001 mass fractions of other metals. Elemental analysis gave the content (mass fractions) of C: (0.4532 ± 0.0020) and H: (0.0670 ± 0.0200), the theoretical calculation gives C: 0.4516 and H: 0.0683 under the assumption that the Na and K metals are in the sample in the form of pivalates (Table 1). The molar mass $Cu(Piv)_2$ (*Molar Mass* = 265.79 g·mol^{-1}) was calculated using the relative atomic masses recommended in [5]. The density of the compound ($\rho = 1.40$ g·cm^{-3}) was calculated from X-ray structural data [6].

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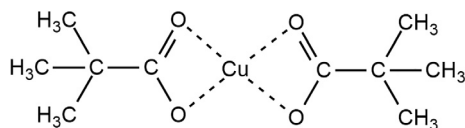 *E-mail address:* druzhinina@thermo.chem.msu.ru (A.I. Druzhinina).

Fig. 1. Schematic structure of the Cu(Piv)$_2$.

2.3. Combustion energy of Cu(Piv)$_2$

The combustion energy of crystalline Cu(Piv)$_2$ was determined in an isoperibolic calorimeter with a stationary self-sealing bomb [7]; the apparatus is similar to one described in [8]. The rise of temperature ($\sim$1 K) was measured with a copper resistance thermometer ($R_{298} \sim 50\,\Omega$) and bridge circuit [9]. A photocompensation microvolt microammeter of F-116/1 was used as a zero-device. The sensitivity of the circuit at a current in the thermometer 0.005 A was $9 \cdot 10^{-6}\,\Omega$/scale division of the device F-116/1 or $\sim$ 5×10^{-5} K. The temperature of the water shell of the calorimeter was kept constant within ±0.003 K. A capacitor discharge through a platinum wire (0.1 mm in diameter) was used to ignite the sample; the platinum wire was in contact with the sample. The ignition energy in all experiments (at calibration and with sample) was constant (2.1 ± 0.1) J and was excluded in calculating the result.

Calibration of the calorimeter was carried out by burning the reference benzoic acid (K-1 brand D.I. Mendeleev Research Institute of Metrology) with the specific energy of combustion in standardized conditions $\Delta_B U = -(26432.5 \pm 1.9)$ J·g^{-1}. A slight deviation from these conditions was corrected by the amendment of Jessup [10]. The energy equivalent of a calorimeter with an empty bomb (without taking into account the specific heat capacity of the contents of the bomb after burning benzoic acid) was determined in a series of 9 experiments, $\varepsilon(\text{Calor}) = (58256.4 \pm 9.1)$ J·Ω^{-1}.

Since Cu(Piv)$_2$ poorly burns, a lot of attention was paid to selecting conditions of the burning sample: the search of an optimal ratio of the quantities of basic and auxiliary substances was carried out; the value of the initial oxygen pressure in the bomb was found.

The tablet of the sample ($\sim$0.12 g) was prepared with the help of a hydraulic press ("Carver firm", USA). Then it was sealed in a Terylene-film bag and placed in a quartz crucible over a tablet of benzoic acid ($\sim$0.41 g). Water (1 ml) was added into the bomb (at the bottom) to dissolve NO$_2$(g) formed by the oxidation of nitrogen present as an impurity in commercial oxygen. The initial oxygen pressure in the bomb was equaled 4.0 MPa. About 80% of the all amount of liberated heat in the experiment is belonging to auxiliary substances (film and benzoic acid). Under these conditions, it was possible to achieve the combustion of a sample with an insignificant amount of formation of soot in a quartz crucible (less than 1×10^{-4} g). The weighing was carried out on a Mettler

balance (Type M58 A Max Bel. 20, uncertainty is 2×10^{-6} g). The initial temperature in all experiments was (298.15 ± 0.03) K.

The analysis of gaseous products of combustion for the content of CO$_2$(g) was carried out by the Rossini gravimetric method [10] with uncertainty of 2×10^{-4} mass fractions. The theoretical mass of CO$_2$(g) was calculated taking into account impurities (NaPiv and KPiv). The absence of CO was controlled by means of indicator tubes (TU.12.43.20–76, sensitivity 6×10^{-6} g CO).

In all experiments in the quartz crucibles, the main solid residue was microcrystalline CuO with a negligible amount of Cu and soot. The CuO/Cu ratio was determined by X-ray photoelectron spectroscopy, XPS. The mixture of CuO/Cu separated from soot by annealing of the quartz crucible in air at $\sim$1500 °C after calorimetric experience. The constancy of the CuO/Cu ratio before and after annealing was proved by comparing the values obtained in calorimetric experiments (1–6, Table 2) with the values obtained in specially conducted preliminary experiments. In these experiments, the bomb after burning the substance immediately was opened and was determined the ratio Cuo/Cu without annealing the crucible. After the calorimetric experiment, the bomb was opened in a day and the crucible with CuO/Cu and soot was annealed. The ratio CuO/Cu in the crucible in calorimetric experiments was the same as in the preliminary experiments. Consequently, all the soot after annealing of the crucible burned up, and the ratio of CuO/Cu without annealing and with annealing remained constant. On the inside surface of the bomb, only microcrystalline CuO was detected using the same analysis method (XPS); Cu and Cu$_2$O were not found. The content of copper in the crucible was less than 0.01 mass fraction (determined by chemical analysis with relative standard uncertainty 0.001), which corresponds to 0.01 mol Cu. The correction on the oxidation of Cu to CuO was inputted to the average value of specific energy of combustion compound ($-\Delta_c u°$). The soot content (less than 1×10^{-4} g) was determined after burning the sample by weighing the crucible before and after it's annealed in the presence of CuO and Cu.

Since a calorimeter with a static bomb was used in the work, the main amount of the HNO$_3$ solution was at the bottom of the bomb. On the walls of the crucible, this solution can only be found in trace amounts. If we assume that it is able to dissolve the microcontent of CuO and Cu, then the correction for the dissolution energy of it will be much less than the error in determining the enthalpy of combustion of the base compound. The amount of HNO$_3$(aq) produced in the experiment was determined by the titration of washing waters $\sim$0.1 mol·dm^{-3} NaOH(aq).

The energy corrections on the auxiliary substances (benzoic acid and Terylene-film) were made. The value $\Delta_c u°$ for Terylene-film was (-22927.9 ± 6.3) J·g^{-1} [12]; $\Delta_c u°$ for benzoic acid was (-26412.0 ± 1.9) J·g^{-1}, these were obtained from certificate value by introducing amendment for reduction to the standard state

Table 1
Provenance and purity of the copper(II) pivalate.

Compound	Source	Purification method	Method of analysis	Element	Mass fraction
Cu(C$_5$H$_9$O$_2$)$_2$; Cu(Piv)$_2$	synthesis	sublimation	laser mass-spectrometry[a]	Cu	0.9970
				Na	0.0015
				K	0.0014
			elemental	C	0.4532[b]
			analysis	H	0.0670
			analysis CO$_2$(g):		
			[m(CO$_2$exp)/m(CO$_2$theor)]		0.9999[c]

[a] The relative standard uncertainty of the device is 0.1.

[b] Practical content of elements, the standard uncertainties are u(C) = 0.002 mass fraction and u(H) = 0.02 mass fraction;

[c] m(CO$_2$exp)/m(CO$_2$theor)] is value obtained from analysis of the gaseous products of combustion. (mCO$_2$expt)/(mCO$_2$theor) is the ratio of the mass of CO$_2$ determined experimentally to that calculated theoretically including impurities; the combined expanded uncertainty is U_c([m(CO$_2$exp)/m(CO$_2$theor)]) = 2×10^{-4} mass fraction (0.95 level of confidence).

Table 2
Combustion energy of crystalline copper(II) pivalate at T = 298.15 K (Molar Mass = 265.79 g·mol^{-1}; ρ = 1.40 g·cm^{-3} [6]; P^o = 101.3 kPa).[a]

№ run	1	2	3	4	5	6
m/g	0.137836	0.120639	0.120238	0.121832	0.121836	0.120086
m(b.a)/g	0.406103	0.416306	0.412222	0.411919	0.413721	0.421463
m(f)/g	0.013133	0.013217	0.014021	0.013549	0.012673	0.014203
ΔRcorr/Ω	0.238962	0.237401	0.235682	0.235943	0.236457	0.239877
ε_f (Cont.)/J·Ω^{-1}	75.9	75.8	75.8	75.8	75.8	75.9
$-\Delta U$(IBP)/J	13939.2	13848.1	13747.9	13763.1	13793.0	13992.6
ΔU(b.a.)/J	10726.0	10995.5	10887.6	10879.6	10927.2	11131.7
ΔU(f)/J	301.1	303.0	321.5	310.7	290.6	325.6
ΔU(HNO$_3$)/J	3.0	3.0	2.4	3.3	3.3	3.3
ΔU(s)/J	1.1	1.1	5.1	5.1	4.4	3.0
$\Delta U_{\sum}$/J	11.9	11.9	11.9	11.8	11.9	12.1
$(-\Delta_c u^o)/(J·g^{-1})$	21027.2	21019.7	21038.3	21035.5	21048.0	21009.1
mean $(-\Delta_c u^o)/(J·g^{-1})$	21030					
Corrections for impurities[b]					NaPiv, KPiv	11
					Cu	−6
Mean $(-\Delta_c u^o$ Cu(Piv)$_2$,cr)/(J·g^{-1})	21025					
m(CO$_2$expt)/m(CO$_2$theor)	0.9997	1.0000	1.0000	0.9997	1.0002	0.9999
Mean m(CO$_2$exp)/m(CO$_2$theor)	0.9999[c]					

^a The standard uncertainties are $u(T)$ = 0.01 K, u(Molar Mass) = 0.01 g·mol^{-1}, $u(P^o)$ = 0.5 kPa; the combined expanded uncertainty is $U_c(\rho)$ = 0.005 g·cm^{-3}; m is mass of the sample Cu(Piv)$_2$ in the vacuum; m(b.a) is mass of the benzoic acid in the vacuum (ρ = 1.32 g·cm^{-3} [10]); m(f) is mass of the Terylene-film in the vacuum (ρ = 1.38 g·cm^{-3} [11]), weighing was performed with precision of ±2·10^{-6} g on a Mettler balance (Type M58A Max. Bel. 20 g); ΔR_{corr} is the increase of the thermometer resistance corrected for heat exchange; the standard uncertainty $u(\Delta R_{corr})$ = 1·10^{-5} Ω; ε_f (Cont.) is the energy equivalent of contents in the final state; the combined expanded uncertainty is $U_c(\varepsilon_f$(Cont.)) = 0.1 J·Ω^{-1}; $-\Delta U$(IBP) is the energy change for the isothermal combustion reaction under actual bomb conditions, the combined expanded uncertainty is U_c(IBP) = 2.7 J (0.95% level of confidence); ΔU (b.a.) is the correction for the energy combustion of the benzoic acid. It is calculated using value $\Delta_c u^o$ = (−26412.0 ± 1.9) J·g^{-1}, which is obtained from certificate value by introducing amendment for reduction to the standard state [10]; the combined expanded uncertainty is $U_c(\Delta U$(b.a.)) = 0.8 J (0.95% level of confidence); ΔU(f) is the correction for the energy combustion of film. The energy of combustion of Terylene-film is $\Delta_c u^o$ = (−22927.9 ± 6.3) J·g^{-1} [12]; the combined expanded uncertainty is $U_c(\Delta U$(f)) = 0.1 J (0.95% level of confidence); ΔU(HNO$_3$) is the correction for the energy formation of solution HNO$_3$(aq) from N$_2$(g), O$_2$(g) and H$_2$O(liq) (based on −59.5 kJ·mol^{-1} the molar energy of formation of aqueous nitric acid [13,14]); the combined expanded uncertainty is $U_c(\Delta U$(HNO$_3$)) = 0.002 J (0.95% level of confidence); ΔU(s) is the correction for the presence of soot in the crucible. The energy of combustion of soot (−32762 ± 11 J·g^{-1}) is calculated from the standard enthalpy of formation of CO$_2$ [14]. The combined expanded uncertainty is $U_c(\Delta U$(s)) = 0.003 J (0.95 level of confidence); $\Delta U_{\sum}$ is the correction to standard state [10]; the standard uncertainty is $u(\Delta U_{\sum})$ = 0.08 J; $(-\Delta_c u^o)$ is the standard specific energy of combustion of sample; the combined expanded uncertainty is U_c(mean$(-\Delta_c u^o)$) = 15 J·g^{-1} (0.95% level of confidence).

^b Corrections for impurities of NaPiv, KPiv and oxidation Cu до CuO; Mean $(-\Delta_c u^o$Cu(Piv)$_2$,cr) is energy combustion of pure Cu(Piv)$_2$, the combined expanded uncertainty is $U_c(-\Delta_c u^o$Cu(Piv)$_2$,cr) = 15 J·g^{-1} (0.95% level of confidence).

^c (mCO$_2$expt)/(mCO$_2$theor) is the ratio of the mass of CO$_2$ determined experimentally to that calculated theoretically including impurities; the combined expanded uncertainty is U_c (m(CO$_2$exp)/m(CO$_2$theor)) = 2 × 10^{-4} mass fraction (0.95 level of confidence).

[10]. The results of 6 experiments on the determination of $(-\Delta_c u^o)$ copper(II) pivalate are given in Table 2.

The change of internal energy for the isothermal bomb process ΔU(IBP) was calculated in accordance with [10]:

$$-\Delta U(IBP) = [\varepsilon(Calor.) + \varepsilon_f(Cont.)] \cdot \Delta Rcorr \quad (1)$$

The value, $\Delta_c u^o$, was calculated on based the sample mass using the following formula:

$$-\Delta_c u^o = [-\Delta U(IBP) - \Delta U(b.a.) - \Delta U(f) - \Delta U(HNO_3) + q(s) - \Delta U_{\sum}(st)] \cdot m^{-1} \quad (2)$$

The combined standard uncertainties of the mean values of the energy equivalent of the calorimeter, ε (Calor.), and of the standard energy of combustion, $(-\Delta_c u^o)$, corresponds to the 95% confidence interval for normal distribution (k = 1.96).

The action of the impurities NaPiv and KPiv on the combustion energy of sample Cu(Piv)$_2$ was evaluated on the assumption that during combustion a solution of acid salts of Na and K in the form (MHCO3 (sol-n)) is formed:

$$MC_5H_9O_2(cr) + 6.5 \ O_2(g) + (aq)$$
$$= MHCO_3(sol \ 1 - n) + 4CO_2(g) + 4H_2O(liq) \quad (3)$$
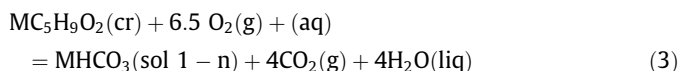
(where M is Na or K), also the enthalpies of formation of NaPiv and KPiv are equal to the enthalpy of silver pivalate $\Delta_f H^0$(AgPiv,cr) = −(466.9 ± 5.6) kJ·mol^{-1} [3].

The content of these impurities in 1 g of the test sample was m(NaPiv) = 0.00194 g and m(KPiv) = 0.00116 g, in a burnt tablet this amounts to ~2·10^{-6} and 1·10^{-6} mol, respectively. The enthalpies of

formation of NaHCO$_3$ and KHCO$_3$ solutions depend very little on the concentration of the solutions and equal to $\Delta_f H^0$(NaHCO$_3$, sol-n) = −930.1 kJ·mol^{-1} and $\Delta_f H^0$(KHCO$_3$, sol-n) = −940.9 kJ·mol^{-1} at T = 298.15 K [13].

The molar values of enthalpy, energy and specific energy of combustion of NaPiv and KPiv were calculated from values given above and amount to:

$\Delta_c H_m^0$NaPiv (cr) = −3180.5 kJ·mol^{-1}; $\Delta_c U_m^0$(cr) = −3174.45 kJ·mol^{-1}; $\Delta_c u^o$ = −25574 J·g^{-1}

$\Delta_c H_m^0$KPiv (cr) = −3191.4 kJ·mol^{-1}; $\Delta_c U_m^0$(cr) = −3185.2 kJ·mol^{-1}; $\Delta_c u^o$ = −22715 J·g^{-1}. Hence,

$$\Delta_c u_{pure}^0 = \frac{\Delta_c u^o - (\Delta_c u^o(NaPiv)·m(NaPiv) + \Delta_c u^o(KPiv)·m(KPiv))}{1 - (m(NaPiv) - m(KPiv))} =$$
$$\frac{-21030 - (-25574·0.00194 - 22715·0.00116)}{1 - (0.00194 + 0.00116)} = -21019 J \cdot g^{-1} \quad (4)$$

The correction to the specific combustion energy of the sample Cu(Piv)$_2$ was 11 ± 0.1 J·g^{-1}; and the combustion energy of Cu(Piv)$_2$ is (−21019 J·g^{-1}) or (−5586.6 kJ·mol^{-1}).

The correction for oxidation of 0.01 mol Cu to CuO(cr) ($\Delta_f H^0$(CuO,cr) = −157.03 ± 0.89 kJ·mol^{-1} [13]) is (−1.6 kJ·mol^{-1}) in the molar combustion energy of Cu(Piv)$_2$ or (−6 J·g^{-1}) in the standard specific energy of combustion of the sample, $\Delta_c u^o$. Taking into account both corrections, the combustion energy of pure Cu(Piv)$_2$ is (−21025 J·mol^{-1}).

3. Results and discussion

The standard mole energy, $\Delta_c U_m^o$, and the enthalpy, $\Delta_c H_m^o$, of combustion and the enthalpy of formation, $\Delta_f H_m^o$, of the pure Cu(Piv)$_2$ are calculated according to reaction:

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Table 3
Standard energy of combustion, $\Delta_c U_m^o$ (cr), enthalpies of combustion, $\Delta_c H_m^o$ (cr), and formation, $\Delta_f H_m^o$ (cr), of the Cu(Piv)$_2$ at T = 298.15 K and P^o = 101.3 kPa, (kJ·mol^{-1}).[a]

$\Delta_c U_m^o$ (cr)/kJ·mol^{-1}	$\Delta_c H_m^o$ (cr)/kJ·mol^{-1}	$\Delta_f H_m^o$ (cr)/kJ·mol^{-1}
−5588.2	−5595.6	−1069.0

[a] The standard uncertainties are $u(T)$ = 0.01 K, $u(P^o)$ = 0.5 kPa. The combined expanded uncertainties are $U_c(\Delta_c U_m^o$ (cr)) = 4.0 kJ·mol^{-1}, U_c ($\Delta_c H_m^o$ (cr)) = 4.6 kJ·mol^{-1} (when calculating this uncertainty, the combined expanded uncertainties in the energy equivalent, auxiliary substances and the test compound takes into account), $U_c(\Delta_f H_m^o$ (cr)) = 4.9 kJ·mol^{-1} (0.95 level of confidence).

$$Cu(C_5H_9O_2)_2(cr) + 13O_2(g) = CuO(cr) + 10CO_2(g) + 9H_2O(liq)$$
(5)
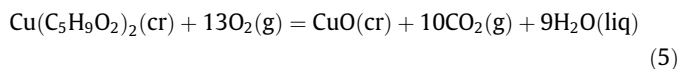
on the basis of specific combustion energy ($-\Delta_c u^\circ$) of Cu(Piv)$_2$ (Table 2), the enthalpies of formation of CuO(cr) ($\Delta_f H_m^o$ (CuO,cr) = −157.03 kJ·mol^{-1}, $U_c(\Delta_f H_m^o$ (CuO,cr)) = 0.89 kJ·mol^{-1}) [13], carbon dioxide ($\Delta_f H_m^o$ (CO$_2$, g) = −393.51 kJ·mol^{-1}, $U_c(\Delta_f H_m^o$ (CO$_2$, g)) = 0.13 kJ·mol^{-1}) and water ($\Delta_f H_m^o$ (H$_2$O, liq) = −285.83 kJ·mol^{-1}, U_c ($\Delta_f H_m^o$ (H$_2$O, liq)) = 0.04 kJ·mol^{-1}) [14]. Table 3 shows the thermodynamic characteristics obtained for pure Cu(Piv)$_2$.

The value $\Delta_f H_m^o$ (Cu(Piv)$_2$, cr) was used to calculate the enthalpy of breaking bond Cu --- Piv by the reaction: Cu(C$_5$H$_9$O$_2$)$_2$(cr) = Cu(cr) + 2 C$_5$H$_9$O$_2$·(g). The enthalpy of the formation of the radical (C$_5$H$_9$O$_2$·) (g) was estimated by us early in [3] from the assumption that the difference between the enthalpy of formation of gaseous pivalic acid and its radical is similar to the difference for n-butanoic acid and its radical [15,16], this is $\Delta_f H^o(C_5H_9O_2·)$(g) = −265 kJ·mol^{-1}. Hence the enthalpy of breaking bond Cu --- Piv is equal to 270 kJ·mol^{-1}. Recently in [17] the enthalpy of the formation of copper(I) pivalate was determined by DSC ($\Delta_f H^o$(CuPiv,cr) = −(506.5 ± 7.6) kJ·mol^{-1}). Using this data, we estimated the enthalpy of breaking the bond in copper(I) pivalate, it is 241 kJ·mol^{-1}. The enthalpy of breaking bond Ag --- Piv is equal 202 kJ mol^{-1} [3]. As seen from the values of the enthalpy of breaking bond obtained in our work and found from the data [17], the bond of Ag --- Ligand is weaker than the bond of Cu --- Ligand.

Also, on basis of $\Delta_f H^o$(Cu(Piv)$_2$, cr) = −(1069.0 ± 4.9) kJ·mol^{-1} (Table 3) and the enthalpy of sublimation of the trimer Cu$_3$(Piv)$_6$ ($\Delta_s H^o$Cu$_3$(Piv)$_6$ = (173.6 ± 4.6) kJ·mol^{-1} [18]) the enthalpy of formation of the trimer in the gaseous state was calculated, $\Delta_f H^o$(Cu$_3$(Piv)$_6$,g) = −(3033 ± 15) kJ·mol^{-1}.

Acknowledgement

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