

VAPORIZATION CHARACTERISTICS OF HEAVY METAL COMPOUNDS AT ELEVATED TEMPERATURES

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Abstract—Volatilities and conversions of heavy metal compounds under oxidation conditions at elevated temperatures up to 1,000°C are analyzed using a thermogravimetric furnace to evaluate their behaviors in an incinerator. Most pure metals and their oxides are relatively stable without releasing metal substances into the atmosphere, except for several metals such as As and Hg. However, some chlorides are vaporized in the combustion temperatures and for these volatile chlorides, vaporization fluxes are obtained based on the measurement of their weight losses with increasing temperatures in the furnace. Experimental vaporization fluxes are compared to maximum theoretical fluxes from Herz-Knudsen's kinetic theory of gas. Results of comparison show that evaporation coefficient, α , for each volatile heavy metal compound appears to be a characteristic value of the evaporating substance, not varying with increasing temperatures and the obtained coefficients are ranged in 10^{-6} to 10^{-8} , which could be explained by a vaporization-condensation model of liquids. With some theoretical consideration on interfacial resistance, mass transfer coefficients, K_c for evaporation, are determined as a function of absolute temperatures, and ranged in 10^{-6} to 10^{-7} .

INTRODUCTION

Incineration is a well-known historically applied thermo-chemical process for volume reduction and stabilization of hazardous and radioactive waste materials. Through the incineration of complex mixtures of waste materials, contaminants in the waste are entrained in the off-gas with gaseous forms such as HCl, HF, SO₂, NO_x, etc. and particulate such as fly ash and unburned carbon. The effective removal of these undesirable materials of gaseous and particulate forms has been successfully demonstrated [1-4].

There are also, however, contaminants which can be presented in off-gas as gaseous, liquid or solid forms depending on temperature, pressure, chemical reaction and gas composition. These elements are referred to as "semi-volatiles", which readily vaporize under normal incineration temperatures, and they may not be trapped easily even with currently utilized air pollution control devices. Furthermore, it is difficult to measure these semi-volatile heavy metals in stack

emission [5-8].

Since vaporization is a physical phenomena governed by a kinetic theory of gas, the higher temperature and the longer residence time normally required for the destruction of organic compounds results in greater vaporization. Therefore, when hazardous and/or radioactive metals are contained in the waste materials it is important to consider emissions of those metals which will occur as a result of vaporization at high-temperature conditions of incineration. Recognizing the inability to control or accurately measure stack emissions, the concentration of semi-volatile metals in the waste has to be limited according to the vaporization rates.

It is known that semi-volatile compounds of heavy elements up to 1,000°C as a normal incineration temperature are chlorides of Cd, Hg, In, Tl, Pb, As, Sb, Bi, Se, Te, fluorides of Hg, As, Sb, Bi, Se, Te, hydrate of As, Se, Pb, Te, Sb, Bi, and free elements of Se, Te, As, Sb, Bi, Ti, Cd, Hg [9]. Among these semi-volatiles As, Cd, Pb, Zn and Hg are strictly regulated in stack emission because of their toxicity. Among important radionuclides, Cs and Ru are also known to be

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volatile in high temperature conditions [10, 11]. Therefore, vaporization characteristics of such elements should be known to both the regulatory body and the workers in the field of hazardous and/or radioactive incineration.

Several groups in the field of thermal treatment of hazardous and radioactive materials have recently studied the vaporization of semi-volatile heavy metal compounds. Most of the previous reports focussed on how semi-volatiles were distributed in the incineration process based on the examination of the heavy element found in fly ash, bottom ash, and scrubber residues [12-17]. In contrast to those works, theoretical analyses on maximum vaporization fluxes of arsenic compounds and some chlorides based on the classical kinetic theory of gas were dealt with by M. Trichon and J. Feldman [8, 18]. However, there has been no correlation between maximum and real vaporization flux, which will be dealt with in this paper.

In this study a correlation between maximum and real vaporization flux of some semi-volatile heavy metal compounds was developed with a theoretical analysis on gas-liquid interfacial resistance in mass transfer phenomena. In order to evaluate interfacial resistance on vaporization, experimentally obtained vaporization fluxes are compared to a kinetic theory of gas and finally, mass transfer coefficients are obtained.

MATERIALS AND METHODS

The basis of the present investigation is the vaporization at high temperatures under oxidizing condition. Some heavy metal compounds, regulated in stack emission, which are listed in the Table 1 were heated up to 1,000°C under oxidizing condition in the thermogravimetric furnace. The melting, subliming and boiling temperatures of tested sample metal compounds are also shown in Table 1.

The experimental apparatus (thermogravimetric analyzing device, MAC-500, LECO Co.) mainly consists of a furnace, a control and data acquisition system and a conditioning system for the furnace [19]. A schematic diagram of the apparatus is shown in Fig. 1. Temperature of the furnace could be controlled with any mode of heating by a programmable microprocessor up to 1,000°C.

Temperature, weight loss of samples and time were recorded and stored in the computer. The detailed structure of the furnace is shown in Fig. 2. Up to 20 samples in crucibles can be tested at once using a turntable. Initially empty crucibles were weighed using a balance. The experiment was performed under

Table 1. Melting points and boiling points of sample metal compounds [20]

Hazardous metal	m.p., °C	b.p., °C
Pure cadmium (Cd)	321	767
Oxide (CdO)	Sb	1497
Chloride (CdCl ₂)	569	960
Pure lead (Pb)	327	1750
Oxide (PbO ₂)	-	-
Chloride (PbCl ₂)	501	-
Pure chromium (Cr)	1857	2672
Oxide (CrO ₂)	-	-
Chloride (CrCl ₂ ·6H ₂ O)	815	1300
Pure copper (Cu)	1083	2560
Oxide (Cu ₂ O)	1236	-
Chloride (CuCl ₂ ·2H ₂ O)	-	-
Pure zinc (Zn)	419.6	907
Oxide (ZnO)	1975	-
Chloride (ZnCl ₂)	317	732
Pure nickel (Ni)	1453	2910
Oxide (Ni ₂ O ₃)	-	-
Chloride (NiCl ₂ ·6H ₂ O)	1009	-
Pure arsenic (As)	Sb	603
Oxide (As ₂ O ₃)	141	371
Pure cobalt (Co)	1453	2910
Oxide (Co ₃ O ₄)	-	-
Chloride (CoCl ₂ ·6H ₂ O)	2870	1049
Pure manganese (Mn)	1244	1962
Oxide (MnO ₂)	-	-
Chloride (MnCl ₂ ·4H ₂ O)	650	1232
Mercury chloride (HgCl ₂)	280	302
Cesium chloride (CsCl)	645	1324

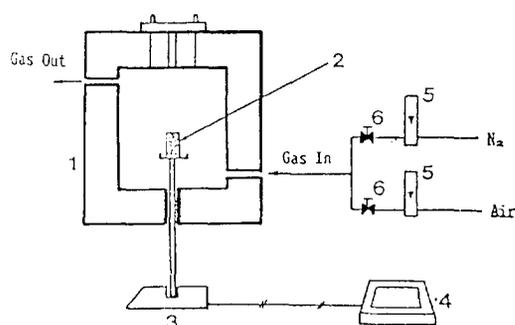


Fig. 1. A Schematic diagram of TGA system.

- | | |
|------------|-------------------|
| 1. Furnace | 4. Computer |
| 2. Sample | 5. Flowmeter |
| 3. Balance | 6. Flow regulator |

oxidizing condition to observe oxidation of experimental metal compounds. Oxygen was fed to have

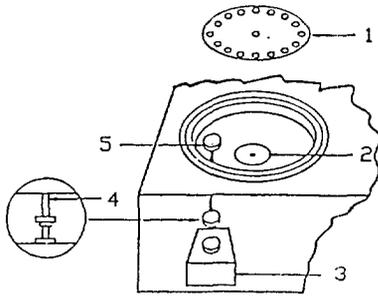


Fig. 2. Detailed structure of the furnace.

- 1. Turntable
- 2. Turntable spindle
- 3. Balance
- 4. Balance post assembly
- 5. Balance post assembly

Table 2. Program of thermogravimetric analysis (TGA)

Final temperature	1,000°C				
Initial temperature	10°C				
Method	2 Step TGA				
Step	Ramp rate	Final temp.	Mode	Step and mode	Time out
1	6°C/min	1,000°C	O ₂	Time out	60 min

such condition and the temperature was ramped with the rate of 6°C/min from the room temperature, then reached and sustained at 1,000°C for one hour as shown in Table 2. The weight of each sample in turning crucibles were continuously measured by microbalance with a load-carrying capacity of 5 g of each sample and sensitivity of 1 µg while temperature elevation and the data were automatically acquired and stored with time and temperature.

RESULTS AND DISCUSSION

1. Reaction

The results of weight changes for solid metal elements, metal oxides and metal chlorides at elevated temperatures are shown in Fig. 3, 4 and 5 respectively. Most element metals except Arsenic were non-volatile but converted into oxide forms with increment of their weights in the experimented range of temperature. Metal oxides reacted with oxygen and were converted into other stable oxides at a certain temperature. Converted oxide metal compounds were very stable and became constant in weight at elevated temperatures. These reactions could be described as Eqs. (1), (2) and (3).

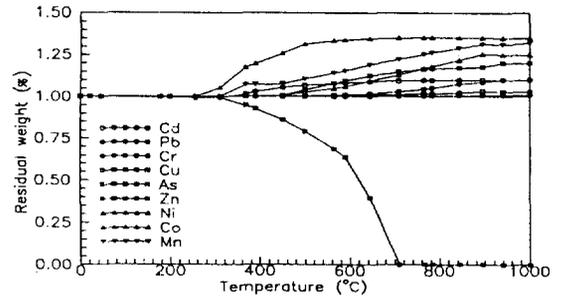
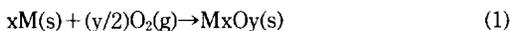


Fig. 3. Weight changes of element metals at elevated temperatures.

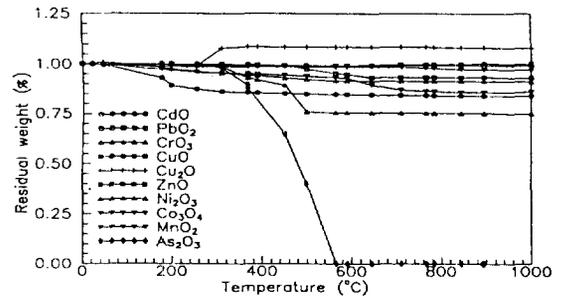


Fig. 4. Weight changes of metal oxides at elevated temperatures.

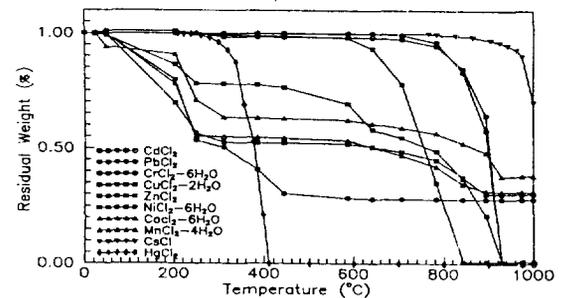
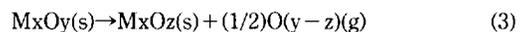
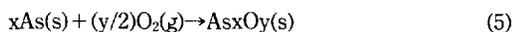


Fig. 5. Weight changes of metal chlorides at elevated temperatures.

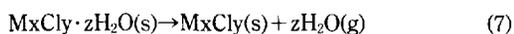


In the case of Arsenic Oxide (As₂O₃), it started to vaporize without changing into any other oxide forms at temperature above 300°C. For As, the element metal was converted into an oxide form, As_xO_y, however, it became very volatile at temperatures above 300°C. The reaction formula could be written as Eqs. (4), (5) and (6) [8]. Therefore' all the Arsenic compounds were vaporized and exhausted into the atmosphere

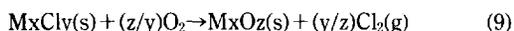
at temperature above 700°C.



Metal chlorides such as CdCl_2 , PbCl_2 , ZnCl_2 , CsCl_2 and HgCl_2 were volatile and vaporized at a certain temperature. Vaporization of these compounds follows Eqs. (7) and (8).



However, there were non-volatile chloride compounds such as CrCl_3 , NiCl_2 , CoCl_2 , MnCl_2 and SrCl_2 at the temperature up to 1,000°C as shown in Fig. 5. The weight losses shown at the temperature less than 200°C were due to the vaporization of moisture as in Eq. (7). Another conversion with weight changes after water vaporization was explained by an oxidation reaction as Eq. (9). The reaction generated chlorine gas. However, the metal itself was converted into a stable oxide form without releasing any metal components.



Conversion temperatures, converted forms and volatilities of products after reaction were summarized in Table 3. These results were verified by a stoichiometric estimation as well as available data-books for melting, subliming and boiling points with Gibbs Free Energy Data for the reaction [20-23].

2. Vaporization Flux

Vaporization flux of heavy metal compounds could be obtained from the results of the experiment as follows;

$$N_{v,p} = \frac{1}{AM} \frac{dW}{dt} = - \frac{1}{AM} \frac{W_1 - W_2}{\Delta t} \quad (10)$$

here A is the surface area (cm^2), M is the molecular weight (g/mol), dW is the weight changes (g) in difference, Δt (sec). And the vaporization theory of a pure solid or liquid is based on the Herz-Knudsen's classical kinetic theory of gas [24, 25].

$$\frac{dn}{dt} = \frac{P}{(2\pi m k T)^{1/2}} \quad (11)$$

where (dn/dt) is the number of molecules per unit time passing through an arbitrary plane of 1 cm^2 area randomly placed in the gas phase, P is pressure existing in gas phase, T is absolute temperature of the

Table 3. Volatilities of sample metal compounds and their change of composition at elevated temperatures

Metal comp.	Volatilities	Vaporizing temp., °C	Converted form	Conversion temp., °C
Cd			CdO	310-550
CdO			-	
CdCl ₂	o	600-	-	
Pb			PbO	550-910
PbO ₂			PbO	440-620
PbCl ₂	o	310-	-	
Cr			-	-
CrO ₃			Cr ₂ O ₃	230-490
CrCl ₃			CrO	270-510
Cu			-	-
CuO			Cu ₂ O	100-710
CuCl	o	470-	-	380-820
Zn			ZnO	450-930
ZnO			-	
ZnCl	o	200-	-	
Ni			NiO	300-820
Ni ₂ O ₃			NiO	170-550
NiCl ₂			NiO	590-890
As	o	400-	-	
As ₂ O ₃	o	300-	-	
Co			CoO	220-710
Co ₃ O ₄			CoO	180-830
CoCl ₂			CoO	450-850
Mn			Mn ₃ O ₄	290-890
MnO ₂			Mn ₃ O ₄	100-810
MnCl ₂			Mn ₃ O ₄	510-920
HgCl ₂	o	200-		
CsCl	o	750-		

gas, m is the molecular mass and k is Boltzmann constant. From the relations of $M = mN_{av}$, $R = kN_{av}$, and $N_v = (dn/dt)/N_{av}$, where N_{av} is Avogadro's Number and N_v is mole flux, Eq. (11) can be converted into,

$$N_v = \frac{P}{(2\pi MRT)^{1/2}} \quad (12)$$

This Eq. (12) is used for the calculation of maximum vaporization flux by M. Trichon and J. Feldman [12, 18, 24, 25]. If the reference plane is placed at the gas-liquid interface, Eq. (12) with $P = P_i$ gives the number of molecules striking the liquid surface from the gas phase. In equilibrium condition vapor pressure at liquid-gas interface, P_i is equal to vapor pressure at liquid surface, P_s . Therefore $P_i/(2\pi MRT)^{1/2}$ moles of vaporized gas striking liquid surface and $P_s/(2\pi MRT)^{1/2}$

molecules leave the liquid surface to the interface. Assuming some fraction α times of moles striking liquid surface is actually condenses, $\alpha P_s/(2\pi MRT)^{1/2}$ molecules enter the liquid surface from gas phase per unit time and unit surface area. Since there is no net transfer occurring between liquid surface and interface in equilibrium condition, $\alpha P_s/(2\pi MRT)^{1/2}$ moles leave the liquid surface to interface per unit time and unit surface. Therefore $\alpha P_s/(2\pi MRT)^{1/2} = \alpha P_i/(2\pi MRT)^{1/2}$ moles enter and leave the liquid surface per unit time and unit surface area in equilibrium condition [26].

In a non-equilibrium condition where p_s is not equal to p_i and vaporization occurs, net transfer between liquid surface to interface is given by the difference in the opposing rates of mass transfer,

$$N_v = \frac{\alpha P_s}{(2\pi MRT_s)^{1/2}} - \frac{\alpha P_i}{(2\pi MRT_i)^{1/2}} \quad (13)$$

Assuming $T_s = T_i$ in Eq. (13) which the temperatures enter as the half power of the absolute temperature and introducing interfacial mass transfer coefficient, k_i , and gas phase mass transfer coefficient, k_g , gives,

$$N_v = \frac{\alpha(P_s - P_i)}{(2\pi MRT)^{1/2}} = k_i(P_s - P_i) = k_g(P_i - P_g) \quad (14)$$

Removing interfacial equilibrium vapor pressure, P_i , in Eq. (14), gives overall mass transfer coefficient between liquid surface to bulk gas phase,

$$N_v = \frac{k_i(P_s - P_g)}{1 + k_i/k_g} = K_G(P_s - P_g) \quad (15)$$

This Eq. (15) shows that when k_i is much smaller than k_g , namely, the resistance of phase transition is greater than that of gas phase mass transfer, k_i is equal to K_G and the rate of mass transfer for vaporization can be written as,

$$N_v = \frac{\alpha(P_s - P_g)}{(2\pi MRT)^{1/2}} \quad (16)$$

where, N_v is mole flux in g-mole/cm²·s, and P is in dynes/cm² for the value R in 8.317 × 10⁷ erg/mol·K. In order to allow P to be expressed in atm, R in cm³·atm/mol·K as conventional unit, Eq. (16) can be expressed,

$$N_v = \frac{44.3\alpha(P_s - P_g)}{(MT)^{1/2}} \quad (17)$$

Evaporation coefficient α in Eq. (17), also called "sticking" or "accommodation" coefficient, defined as the fraction of the gas molecules impinging on the

Table 4. Vapor pressure data for evaporating metal compounds ($\log p = A \times T^{-1} + B \log T + C \times T + D$) [20]

Substance	log P(mmHg)				Temperature range, K
	A	B	C × 10 ⁻³	D	
CdCl ₂	-9270	-5.04	-	25.907	m.p.-b.p.
PbCl ₂	-10170	-	-	8.04	m.p.-b.p.
CsCl	-9815	-3.52	-	20.38	m.p.-b.p.
HgCl ₂	-4580	-2.0	-	16.39	298-m.p.
ZnCl ₂	-8415	-5.03	-	26.42	693-883
As ₄	-6160	-	-	9.82	600-900
As ₂ O ₃	-5282	-	-	10.91	373-573

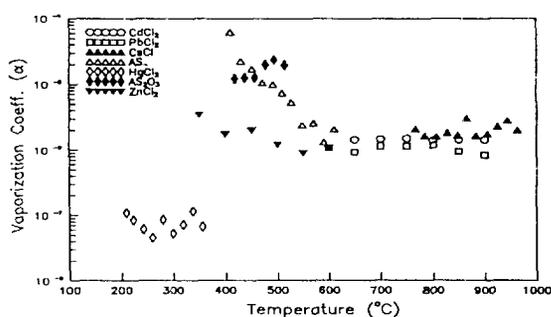


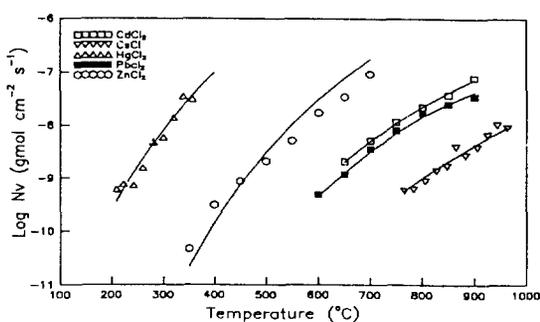
Fig. 6. Vaporization coefficient for volatile metal compounds.

condensing surface which stick and are not reflected [26], could be obtained in comparison with N_{exp} in Eq. (10). In making assumption of vapor pressure in bulk phase P_g to be zero considering continuous fresh gas inserting condition in this experiment, driving force $P_s - P_g$ in Eq. (17) is replaced by P_s in calculation of α with Eq. (10) and (17). Here P_s is vapor pressure of the surface, able to be obtained from available data from the literature as shown in Table 4 [22-25]. It has been generally known that α depends on specific properties of the evaporating substance [27]. Measured α values for vaporizing metal compounds except As as shown in Fig. 6 are almost constant with temperature increase from m.p. to b.p. and the results show the temperature effect on α could be ignored in a given temperature range. Therefore, mass transfer coefficient, K_G , could be determined as a function of temperature with constant α in the given temperature range using Eq. (14).

For As, measured α values decreased with temperature increase from 400-600°C. It can be explained that oxidation reaction in the solid surface may simultaneously occur with the vaporization of As and the

Table 5. Measured average vaporization coefficient, α , and proposed mass transfer coefficient, K_G

Substance	Vaporization coefficient, α	Mass transfer	
		coefficient, K_G ($\times T_s^{-0.5}$), $\text{gmol s}^{-1} \text{cm}^{-2} \text{atm}^{-1}$	Temperature range, $^{\circ}\text{C}$
CdCl ₂	1.42×10^{-6}	4.65×10^{-6}	650-900
PbCl ₂	1.03×10^{-6}	2.66×10^{-6}	600-900
HgCl ₂	7.21×10^{-8}	3.63×10^{-7}	200-370
CsCl	2.01×10^{-6}	2.14×10^{-7}	750-975
ZnCl ₂	1.16×10^{-6}	1.76×10^{-6}	340-590

**Fig. 7. Comparison of experimentally obtained vaporization flux to calculated flux from proposed K_G .**

product of oxidation (As_2O_3) also vaporize with high volatility in the same temperature range. As the rate of the oxidation reaction will be higher with the temperature increase, and more As_2O_3 will be generated in the surface of As, of which vaporization coefficients are lower than that of As, the evaporation coefficients of As will decrease with the temperature increase. Therefore it could not be described real evaporation coefficient for As in this experiment since reaction kinetics could not be analyzed by evaporation of As.

From the calculated average values of α in given ranges of the temperature and the proposed estimated K_G as a function of temperature except for As are listed in Table 5. The calculated vaporization fluxes using proposed values of K_G which are noted as solid lines are compared to experimentally obtained vaporization fluxes in Fig. 7.

CONCLUSIONS

The analyses and results of the experiment were performed to observe the vaporization of hazardous and radioactive metals in oxidation atmosphere up to the temperature of $1,000^{\circ}\text{C}$ and could be concluded

as follows;

1. The pure metals such as Cd, Pb, Cr, Cu, Zn, Ni, Co and Mn except As convert into stable oxide forms and do not vaporize at elevated temperatures.

2. In case of oxide forms of the tested metals, except arsenic oxide, some are quite stable themselves and the others are converted into other stable oxide forms without vaporization of metal components.

3. There are some volatile compounds in metal chlorides with their vaporizing temperatures at about 600°C for CdCl₂, 550°C for PbCl₂, 511°C for ZnCl₂, 470°C for CuCl₂, 200°C for HgCl₂, 300°C for As_2O_3 , 750°C for CsCl, respectively. Other metal chlorides are converted into oxide forms at a certain temperature and become stable in temperatures up to $1,000^{\circ}\text{C}$.

4. Evaporation coefficients, α , for the vaporizing substances are constant with the temperature increase. With some theoretical consideration, mass transfer coefficients K_G for evaporating heavy metal compounds could be estimated as a function of half power of the temperature using the obtained values of α .

ACKNOWLEDGEMENT

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NOMENCLATURE

- A : surface area [cm^2]
- k : Boltzmann constant, 1.3805×10^{-16} erg K^{-1} molecule⁻¹
- M : molecular weight [g g-mole^{-1}]
- N_{av} : Avogadro's number, 6.023×10^{23} molecules g-mole^{-1}
- N_v : evaporation flux [$\text{g-moles cm}^{-2} \text{s}^{-1}$]
- P : pressure existing in gas phase in dynes/ cm^2
- P_G : partial pressure in bulk gas [dynes/cm^2 , atm]
- P_i : pressure existing at the interface [dynes/cm^2 , atm]
- P_s : vapor pressure at the surface of the liquid [dynes/cm^2 , atm]
- R : gas constant, $82.03 \text{ cm}^3 \text{ atm g-mole}^{-1} \text{ s}^{-1}$
- t : time [sec]
- T : absolute temperature [K]
- T_s : absolute temperature of liquid [K]
- T_i : absolute temperature of gas at the interface [K]
- W : weight of sample [g]

Greek Letter

- α : vaporization coefficient [-]

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