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VAPOR PRESSURE OF SOME VOLATILE IRIDIUM(I) COMPOUNDS WITH CARBONYL, ACETYLACETONATE AND CYCLOPENTADIENYL LIGANDS

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Volatile compounds of iridium(I): (acetylacetonato)(1,5-cyclooctadiene)iridium(I) Ir(acac)(cod), (methylcyclopentadienyl) (1,5-cyclooctadiene)iridium(I) Ir(Cp')(cod), (pentamethylcyclopentadienyl)(dicarbonyl) iridium(I) Ir(Cp*)$(CO)_2$ and (acetylacetonato)(dicarbonyl)iridium(I) Ir(acac)$(CO)_2$ were synthesized and identified by means of element analysis, NMR-spectroscopy, mass spectrometry.

Thermal properties in solid phase for synthesized iridium(I) complexes were studied by means of thermogravimetric analysis in inert atmosphere (He). By effusion Knudsen method with mass spectrometric registration of gas phase composition the temperature dependencies of saturated vapor pressure were measured for iridium(I) compounds and the thermodynamic characteristics of vaporization processes enthalpy ΔH_{T*} and entropy ΔS^0_{T*} were determined. The energy of intermolecular interaction in the crystals of complexes was calculated.

Keywords: *acetylacetonate and cyclopentadienyl ligands, carbonyl, iridium(I), mass spectrometry, thermogravimetric investigation, vapor pressure*

Introduction

Iridium metal has intensive use in different areas. Due to high melting point iridium is frequently used as protective coatings for higher temperature applications such as rocket engines [1]. Due to low chemical reactivity Ir can be used as oxidation-resistant electrodes and electric contacts [2]. Recently processes of Cu electrodeposition have been attracting growing interests and require materials with such properties as nobility, formation of soluble or conducting oxides, and insolubility in Cu. An ideal direct plating material should have good diffusion barrier properties as well as good adhesion to dielectrics. A few metal layers have been identified as candidates, which are mostly refractory metals such as iridium [3].

The thermal decomposition of organometallic compounds on heating surfaces (chemical vapor deposition) is one of the methods for forming metal thin films. For successful application of the CVD procedure, precursors possessing a definite set of properties (volatility, stability at deposition temperature, etc.) are necessary. Many classes of volatile iridium compounds are known: halides, homo- and heteroligand carbonyls, olefin, cyclopentadienyl or allyl derivatives, chelate complexes, etc. [4], however, only a limited number of compounds are used in CVD processes. This is connected both with the discrepancy between the physicochemical properties of a number of Ir precursors and the technological requirements of the deposition processes, and with the absence of the data on the thermal behavior of iridium precursors. To this moment, CVD processes involving volatile β-diketonate iridium(III) derivatives are successfully used. Thermal properties of these compounds have been studied by us previously [5, 6]. Modern technologies are aimed at new precursors with low melting point and high vapor pressure. The representatives of these compounds are volatile complexes of iridium(I): (acetylacetonato)(1,5-cyclooctadiene)iridium(I) Ir(acac)(cod), (methylcyclopentadienyl) (1,5-cyclooctadiene)iridium(I) Ir(Cp')(cod), (pentamethylcyclopenta-dienyl)(dicarbonyl)iridium(I) Ir(Cp*)$(CO)_2$ and (acetylacetonato)(dicarbonyl)iridium(I) Ir(acac)$(CO)_2$. These precursors are often used in iridium CVD, both by dosing liquid precursor in direct liquid injection CVD, and by evaporating the precursor from the solid-state [2, 7–9]; however, the data on the thermal properties of iridium(I) precursors are almost absent. Because of this, the goal of the present work is investigation of thermal behavior of iridium(I) chelates in solid and gas phases, and measurement of the thermodynamic characteristics of vaporization of iridium(I) compounds for the purposeful selection of the deposition parameters.

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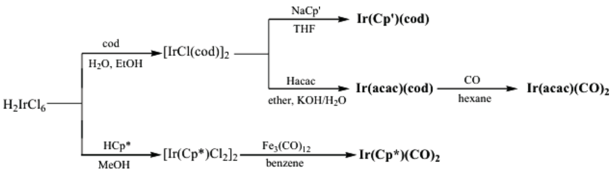
Experimental

Synthesis of Ir compounds

Synthesis of iridium(I) compounds was carried out according to the procedures modified by us [10–13], in the inert atmosphere using the standard Schlenk technology according to the scheme: All Ir(I) compounds were synthesized using the same initial reagent – hexachloroiridium acid H_2IrCl_6. The compounds were purified by recrystallization from hexane and by vacuum sublimation at p=0.01 Torr: for Ir(Cp')(cod) at t=50°C (yield: 85%); for Ir(acac)(cod) at t=90°C (yield: 75%); for $Ir(acac)(CO)_2$ at t=95°C (yield: 75%) and for $Ir(Cp^*)(CO)_2$ at t=70°C (yield: 70%). The synthesized complexes are crystal substances: white Ir(Cp')(cod) (*m.p.* 38–40°C), yellow Ir(acac)(cod) (*m.p.* 155°C), $Ir(Cp^*)(CO)_2$ (*m.p.* 110°C) and brown $Ir(acac)(CO)_2$ (*m.p.*>145°C with decomp.), insoluble in water and well soluble in organic solvents.



Instrumental methods

^1H NMR spectra were recorded for solutions in $CDCl_3$ using device of Bruker Company MSL300. $CDCl_3$ (δ_H 7.24 ppm) was used as an internal standard. The results for Ir(Cp')(cod): δ 3.56 (s, 4H, =CH, cod), 2.05 (m, 4H, exo-CH_2, cod), 1.80 (m, 4H, endo-CH_2, cod), 5.18 (m, 2H, H1, H4, Cp'), 4.97 (m, 2H, H2,H3, Cp'), 1.90 (s, 3H, CH_3, Cp'); for $Ir(Cp^*)(CO)_2$: δ 2.166 ppm; δ 2.19 Cp*; for Ir(acac)(cod): δ 3.96 (s, 4H, =CH, cod), 2.25 (m, 4H, exo-CH_2, cod), 1.63 (m, 4H, endo-CH_2, cod), 5.51 (s, 1H, C_γ–H, acac), 2.00 (s, 6H, CH_3, acac); for $Ir(acac)(CO)_2$: δ 5.78 (s, 1H, C_γ–H, acac), 2.11 (s, 6H, CH_3, acac). Obtained results are shown good agreement with available in the literature data [14, 15] and confirm the purity of synthesized compounds.

Mass spectrometry study was carried out with a high resolution mass spectrometer MI-1201 with the energy of ionizing electrons 35 eV. Temperature of ionization chamber was 120°C, source temperature was within the range 30–50°C. The limiting resolution of MI-1201 mass spectrometer within the mass number range 1200 a.m.u. was not less than 1000 at a level of 10% of the peak height. Element ion content was determined based on mass number and isotope composition.

DSC/TG data were obtained using device Netzsch STA 409 PC/PG. Experiments were conducted in He flow (20 mL min^{-1}) with heating rate 10°C min^{-1}, temperature range was 20–300°C, open crucibles were standard.

Vapor pressure

The temperature dependencies of saturated vapor pressure for iridium precursors were investigated using Knudsen's effusion method; the composition of the gas phase was analyzed with mass spectrometer (MI-1201, Russia). A detailed description of the experiment was presented in [16]. Temperature of the effusion chamber was changed stepwide with a step of 5–10°C with the help of thermoregulator 'Termodat 13M2'. Temperature and the intensity of ion current were recorded during time interval t_i. Portions of 3–5 mg were used. At each established temperature, the full mass spectrum was recorded. Measurement of the ion current intensity in the steady mode was carried out for 25–30 min. Weighed portions of the compound were vaporized at different temperatures within the required temperature range till complete disappearance of the ion peaks corresponding to the molecular forms of compound vapor. A cylindrical effusion chamber was made of molybdenum; the dimensions of the internal cavity were: d=7 mm, h=8 mm. Effusive hole was 0.3 mm in diameter; the channel length was 0.2 mm. To calibrate the mass spectrometer, we used Hertzen–Knudsen equation binding pressure in the effusion chamber with the amount of the removed matter m (g) at temperature T during time t (s) [16]:

$$p(\mathrm{torr})=\frac{m}{(S_{\mathrm{ef}}\,t)(2\pi RT/M)^{1/2}}=17.14\left(\frac{T}{M}\right)^{1/2}\left(\frac{m}{S_{\mathrm{ef}}\,t}\right) \qquad (1)$$

here S_{ef} is the product of Clausius coefficient (K), depending on the ratio of the length of effusion channel to its diameter, and the area of effusion hole (s), M is molecular mass, R is gas constant.

Vapor pressure p in the mass spectrometric experiment in Knudsen's chamber is connected with the measured ion current intensity through a known equation:

$$p=CIT \qquad (2)$$

where C is the constant of mass spectrometer sensitivity to a given kind of ions. At each temperature point T_i, within time interval t_i the amount of the substance removed is determined, taking into account Eq. (1), by the relation:

$$m_{\mathrm{i}}=\left(S_{\mathrm{ef}}M^{1/2}C\int_0^t T_{\mathrm{i}}^{1/2}I_{\mathrm{i}}\,\mathrm{d}t\right)/17.14 \qquad (3)$$

The integral in Eq. (3) defines the amount of the substance removed at temperature T_i during time t_i. Total amount of the evaporated substance is $m(g)=\Sigma m_i$. So, sensitivity coefficient C is determined by equation:

$$C = 17.14 m / S_{\mathrm{ef}} M^{1/2} \sum \int_0^t T_i^{1/2} I_i \, \mathrm{d}t \qquad (4)$$

Having substituted coefficient C into Eq. (2), we obtain the equation combining pressure in Knudsen's effusion chamber with the values measured in the experiment:

$$P_i = \frac{17.14 W I_i T_i}{S_{\mathrm{eff}} M^{1/2} \sum_i \int_0^t I_i T_i^{1/2} \mathrm{d}t} \qquad (5)$$

here I_i in the numerator is the intensity of ion peak stable with time, measured at the established temperature T_i; measurements were carried out over these peaks.

The errors included into the calculation of pressure error were: intensity (I – 1%), temperature (T – 2 K) and mass of the weighed portion (m – 3%). The calculated limiting error of measurement of the partial pressure of saturated vapor was ~10%.

Experimental data are presented as the equation lg(p, Torr)=A–B/(T, K), where $A=\Delta S^0_{T*}/4.575+2.88$ and $B=\Delta H_{T*}/4.575$, T^* is mean temperature in the examined interval. Ir(I) compounds after two times of sublimation in the vacuum were used for vapor pressure measurements.

Calculations of the energy of intermolecular interaction

To calculate the energy of intermolecular interaction in the crystals of complexes, we used the structural data published in [17, 18] for Ir(acac)(cod) and Ir(Cp*)$(CO)_2$, and the data of X-ray structural investigation obtained by us for Ir(Cp')(cod) and Ir(acac)$(CO)_2$ [19, 20]. Packing of molecules in the crystal for Ir(acac)(cod), Ir(Cp*)$(CO)_2$, Ir(Cp')(cod) and Ir(acac)$(CO)_2$ are shown in Fig. 1.

Calculations of the energy of van der Waals intermolecular interaction were carried out within the atom-atomic potential method (AAP). Buckingham's potential was used in calculations:

$$E_{ij} = -A_{nm} r_{ij}^6 + B_{nm} \exp(-\alpha_{nm} r_{ij}) \qquad (6)$$

here r_{ij} is the distance between atoms i and j; the kinds of atoms are designated with indexes n and m; A, B and α are empirical parameters determined with the following equations:

$$A = 1.79 \varepsilon_0 r_0^6,\ B = 6.364 \cdot 10^2 \varepsilon_0,\ \alpha = 13.6/r_0 \qquad (7)$$

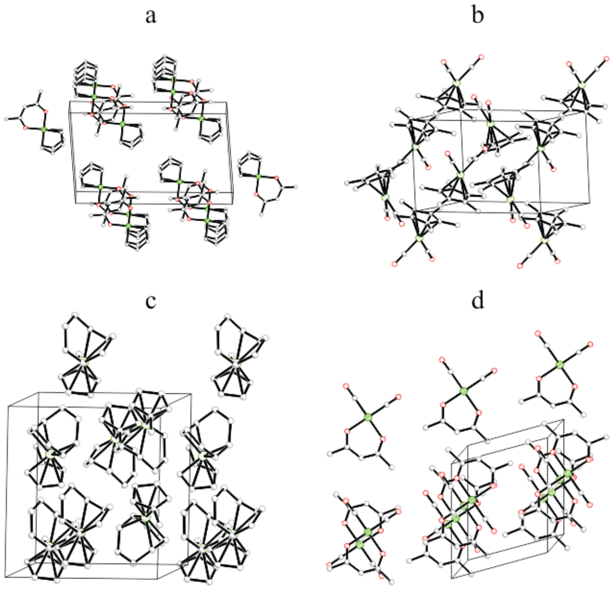


Fig. 1 Molecule packing in the crystal: a – Ir(acac)(cod), b – Ir(Cp*)$(CO)_2$, c – Ir(Cp')(cod) and d – Ir(acac)$(CO)_2$

where ε_0 and r_0 are the depth of the potential hole and the equilibrium distance between the atoms.

The parameters of interaction potentials A, B and α for light atoms were taken from the works of Zorky [10]; for the metal-metal interaction, ε_0 and r_0 were estimated graphically using the dependence $\varepsilon_0(r_0)=f(N)$, built up on the basis of the known values of parameters for inert gases [11, 12], where N is element number.

The parameters of potentials of the intermolecular interaction between the atoms of different kinds A_{nm}, B_{nm}, α_{nm} were determined as a geometric mean:

$$A_{nm} = \sqrt{A_{nn} A_{mm}},\ B_{nm} = \sqrt{B_{nn} B_{mm}},\ \alpha_{nm} = \sqrt{\alpha_{nn} \alpha_{mm}} \qquad (8)$$

Results and discussion

Mass spectra

The mass spectra of the synthesized Ir(I) compounds are presented in Table 1. Intensive peaks of molecular ions are observed in all the mass spectra. All studied compounds transfer to gas phase as monomers at corresponding temperatures. Within the investigated temperature range, relative intensities of ion peaks in the mass spectrum do not change, which is an evidence of thermal stability of complex vapors within this temperature range.

TG study

The TG curves of mass loss for Ir(I) precursors in the inert atmosphere during heating are shown in Fig. 2. Under the recording conditions, all the complexes pass

Table 1 The most intensive ion peaks in the mass spectra of Ir(I) compounds

Ir(Cp')(cod)			Ir(Cp*)$(CO)_2$		
Ion$^+$	*M*/*z*	Relativity intensity/%	Ion$^+$	*M*/*z*	Relativity intensity/%
[Ir(Cp')(cod)]	380	100	[Ir(Cp*)$(CO)_2$]	384	68.8
[Ir(Cp')(cod)-H]	379	42.9	[Ir$C_8H_{11}(CO)_2$]	356	45.7
[Ir(Cp')(cod)-H_2]	378	73.1	[Ir$C_8H_9(CO)_2$]	354	73
[Ir(Cp')(cod)-H_3]	377	58.8	[Ir$C_8H_7(CO)_2$]	352	100
[Ir(Cp')(cod)-H_4]	376	13	[IrC_8H_7CO]	324	62.5
[Ir(Cp')(cod)-CH_3]	365	14.7	[IrC_8H_5CO]	322	60
Ir(acac)(cod)			Ir(acac)$(CO)_2$		
[Ir(cod)(acac)]	400	100	[Ir(acac)$(CO)_2$]	348	100
[Ir(cod)(acac)-$CO_2C_2H_7$]	325	15.5	[Ir(acac)CO]	320	10
[IrC_8H_{10}]	299	19	[Ir(acac)]	292	87
[IrC_8H_9]	298	45	[Ir$C_4H_4O_2$]	277	13
[IrC_8H_8]	297	32.5	[IrC_4H_5O]	262	18
[IrC_8H_6]	295	50	[IrC_3H_2O]	247	13
[IrC_8H_5]	294	50	[IrC_3HO]	246	15
[IrC_6H_4]	269	25	[IrC_2H]	218	14

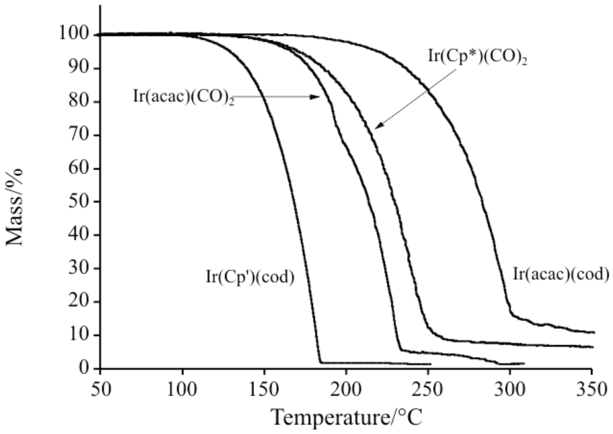


Fig. 2 TG curves for Ir(I) precursors

completely into the gas phase almost without decomposition. Among the presented set of compounds, the most volatile compound is Ir(Cp')(cod). The following volatility row was established for the 50% mass loss related to the evaporation process for the synthesized chelates under identical experimental conditions: Ir(Cp')(cod) (168°C)>Ir(acac)$(CO)_2$ (213°C)>Ir(Cp*)$(CO)_2$ (229°C)>Ir(acac)(cod) (283°C).

For Ir(Cp')(cod) and Ir(acac)(cod), endothermic peaks are observed on the DTA curve at the melting points of compounds: 40 and 155°C, respectively; they correspond with the experimental m.p. obtained using Kefler's table.

Vapor pressure

Experimental measurements of saturated vapor pressure are at present the main source of information about the volatility of organometallic compounds. The temperature dependencies of saturated vapor pressure for Ir(I) complexes studied in this work are presented in Fig. 3. Using these dependencies, we calculated standard thermodynamic characteristics: enthalpy ΔH_{T*} and entropy ΔS^0_{T*} of sublimation and evaporation (Table 2). We have published preliminarily some experimental data in conference proceedings [13].

One can see from the presented *p*/*T* dependencies for Ir(I) precursors that within the investigated

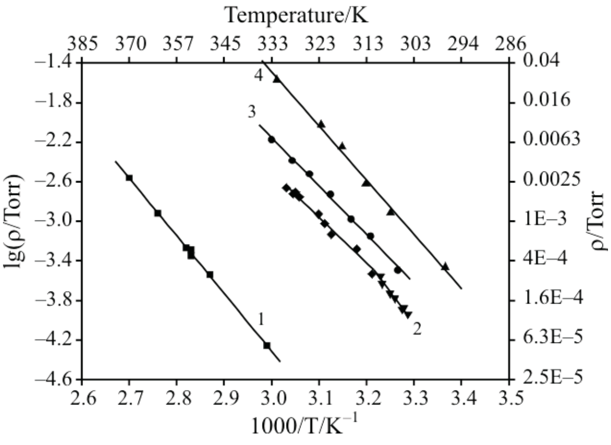


Fig. 3 Dependencies of logarithm of saturated vapor pressure on reciprocal temperature for the complexes 1 – Ir(acac)(cod), 2 – Ir(Cp')(cod), 3 – Ir(acac)$(CO)_2$, 4 – Ir(Cp*)$(CO)_2$

Table 2 Temperature dependences of the saturated vapor pressure, enthalpy (ΔH_{T*}) and entropy (ΔS^0_T) of evaporation and sublimation of Ir(I) complexes, normalized to the mean temperature (T*) in the examined interval (ΔT)

Compound	Process	ΔT/K	n	lg(p, Torr)±σ=A–B/(T, K)			ΔH_{T*}/ kJ mol^{-1}	ΔS^0_{T*}/ J mol^{-1} K^{-1}
				A	B	σ		
(Cp')Ir(cod)	sub.	304–310	7	17.4	6510	0.03	124.6±5.0	279±16
	evap.	310–330	9	11.3	4600	0.04	88.1±1.3	161.2±4.2
(Cp*)Ir(CO)$_2$	sub.	297–332	6	15.0	5490	0.05	105.0±3.4	232±11
(acac)Ir(cod)	sub.	335–370	7	13.2	5840	0.02	111.7±1.7	197.5±5.0
(acac)Ir(CO)$_2$	sub.	306–333	7	12.6	4910	0.03	94.1±2.7	186.1±7.8

n – number of points

Table 3 Calculation of the energy of intermolecular Van der Waals interaction in crystals

Compound	Space group	Z	V/Z	Packing index	$-E_{\mathrm{cryst}}$/ kJ mol^{-1}	$-\Delta H_{T*}$ (experiment)/ kJ mol^{-1}
(acac)Ir(cod)	C2/m	4	316.4	0.683	119.2	111.7±1.7
(Cp*)Ir(CO)$_2$	P2$_1$/n	4	302.1	0.677	98.6	105.0±3.4
(Cp')Ir(cod)	P2$_1$/n	4	286.5	0.722	116.5	124.6±5.0
(acac)Ir(CO)$_2$	P-1	2	216.6	0.678	39.7	94.1±2.7

temperature range the most volatile complex is Ir(Cp*)(CO)$_2$, the least volatile one is Ir(acac)(cod). Since the crystal structure of a compound has the strongest effect on the parameters of its sublimation, differences in vapor pressure are determined by the features of the crystal structure of the compounds and by the intermolecular interactions in the crystal. The main contribution into the crystal lattice energy is made by Van der Waals interactions of the neighboring molecules. According to the data of X-ray structural investigation (Fig. 1), the molecules of complexes Ir(acac)(cod), Ir(Cp')(cod) and Ir(Cp*)(CO)$_2$ have such a structure that the iridium atom is screened from the interaction with the atoms of the neighboring molecules. The molecule of Ir(acac)(CO)$_2$ has a flat structure, so iridium atom can be in contact with the atoms of the neighboring molecules. Analysis of molecule packing for the complexes Ir(acac)(cod), Ir(Cp')(cod) and Ir(Cp*)(CO)$_2$ showed that all the contacts in the crystals are Van-der-Waals ones, that is, the distances are equal to, or larger than, the sum of the Van der Waals radii of the atoms. There are close Ir–Ir contacts in the packing of Ir(acac)(CO)$_2$ complex, equal to 3.2 Å, which is smaller than the sum of Van der Waals radii of iridium (R=2,5 Å). The results of calculation of the energy of Van-der-Waals intermolecular interaction in the crystals of the compounds are shown in Table 3. One can also see there the packing indexes (the ratio of the Van-der-Waals volume of a molecule to its crystallographic volume, calculation was carried out using program www.cryst.chem.uu.nl/platon) for the compounds under investigation. For Ir(Cp*)(CO)$_2$ the packing index is the smallest, which points to a more loose packing of molecules in the crystal and therefore to a higher volatility of the complex compound; this is confirmed by the experimental p/T data.

One can see in Table 3 that the calculated lattice energies are in good agreement with the values of enthalpy of sublimation obtained from the experimental p–T curves, except for the compound Ir(acac)(CO)$_2$. Unlike all the other complexes under consideration, this complex has a flat structure. These complexes are packed in the crystal in piles parallel to each other (taking into account turnover by 180°). As a result, the central Ir atom has two neighboring iridium atoms of the other molecules at a close distance. According to Ir–Ir potentials, these contacts correspond to repulsion between metal atoms, which brings distortions into calculation results. Because of this, the AAP procedure for the calculation of the energy of Van der Waals interactions cannot be applied for flat complexes having close metal–metal contacts.

To compare the volatility of complexes under investigation and some iridium(III) beta-diketonates obtained by us previously [6], we show the dependencies of vapor pressure on reciprocal temperature in Fig. 4. One may note that the carbonyl derivatives Ir(Cp*)(CO)$_2$ and Ir(acac)(CO)$_2$ exhibit the volatility comparable with that of fluorinated iridium(III) β-diketonates; the complexes with CH_3 and $C(CH_3)_3$ substituents in β-diketonate complexes have the volatility similar to that of the cyclopentadienyl derivatives of Ir(I).

We can see that vapor pressure changes by approximately 4 orders of magnitude in the row of irid-

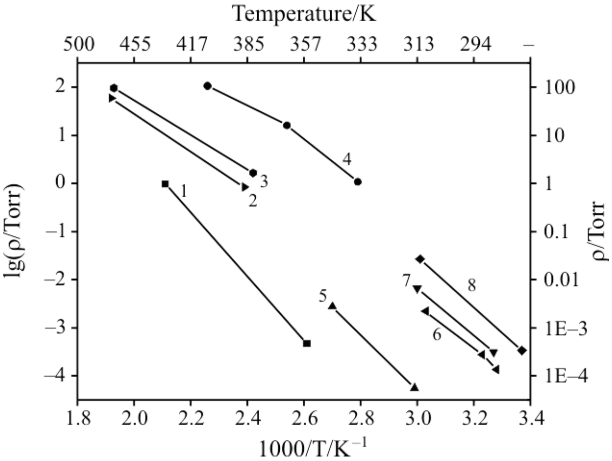


Fig. 4 Dependencies of logarithm of saturated vapor pressure on reciprocal temperature for the complexes 1 – Ir(acac)$_3$, 2 – Ir(thd)$_3$, 3 – Ir(ptac)$_3$, 4 – Ir(hfac)$_3$, 5 – (acac)Ir(cod), 6 – (Cp')Ir(cod), 7 – (acac)Ir(CO)$_2$, 8 – (Cp*)Ir(CO)$_2$

ium(III) beta-diketonates depends of end substitutes in ligand, while by approximately 3 orders of magnitude for heteroligand iridium(I) complexes. Thus influence of central atom on volatility is practically absent, and nature and structure of ligand make main contribution to vaporization processes.

Conclusions

By means of DTA/TG the mass loss curves in inert atmosphere is obtained and the qualitative row of vaporization processes is determined for Ir(I) compounds: (acetylacetonato)(1,5-cyclooctadiene)iridium(I) Ir(acac)(cod), (methylcyclopentadienyl) (1,5-cyclooctadiene)iridium(I) Ir(Cp')(cod), (pentamethylcyclopentadienyl)(dicarbonyl)iridium(I) Ir(Cp*)(CO)$_2$ and (acetylacetonato)(dicarbonyl)iridium(I) Ir(acac)(CO)$_2$. Thermodynamic parameters of vaporization processes were determined using Knudsen's method for synthesized compounds. Calculations of the energy of Van-der-Waals intermolecular interaction in the crystals were carried out by means of atom-atomic potentials. Calculation results for the molecules of complexes having non-flat structure are in good agreement in the experimental values of sublimation enthalpies. It was shown that influence of central atom on volatility is practically absent, and nature and structure of ligand make main contribution to vaporization processes.

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