

DTA STUDY OF THE REACTIONS OF NON-METAL FLUORIDES WITH INORGANIC COMPOUNDS

A. A. OPALOVSKY, E. U. LABKOV, S. S. TOROSYAN and A. A. DZHAMBK

Department of Inorganic Chemistry, Odessa State University, Chemical Faculty, Odessa, USSR

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On the basis of the calculated Gibbs energy change for the standard state, the possibility of carbon tetrafluoride, silicon tetrafluoride, dichlorodifluoromethane and sulfur hexafluoride reacting with a number of elements and their compounds was evaluated. The reactions were studied by differential thermal analysis. Fluorination reactions of metals and oxides with dichlorodifluoride were investigated, and the temperature conditions of the syntheses were determined by measuring the "temperature at the start of interaction" ($t_{s.i.}$).

The existence of a correlation between $t_{s.i.}$ and $\Delta H_{\text{formation}}^{\circ}$ for the fluorinating agents was assumed, and a sequence of the fluorinating power of the non-metal fluorides was established. The perspectives of using non metal fluorides in the synthesis of inorganic fluorides are discussed.

One of the most important problems in inorganic synthesis is the correct choice of the reagent. This problem is particularly important when choosing the fluorinating agent for the synthesis of inorganic fluorides, compounds finding broad fields of application in widely differing branches of technology.

At present a large number of fluorinating agents used in inorganic synthesis are known [1-13], among them elemental fluorine [1-3], halogen fluorides [4-6] and hydrogen fluoride [8], the latter in the gaseous and liquid states, as well as in the form of aqueous solutions, being the most widespread in application.

The major advantage of these fluorinating agents consists in their extraordinary chemical activity, allowing them to convert practically any compound into fluoride. However, this high chemical activity causes great difficulties in the carrying out of the process in practice.

On the other hand, the use of fluorine or halogen fluorides leads to exhaustive fluorination, and hence does not allow one to obtain fluorides with lower oxidation stages.

It therefore appears desirable to use as fluorinating agents fluorides of non-metals, such as boron trifluoride, carbon tetrafluoride (and its derivatives), silicon tetrafluoride, nitrogen trifluoride, sulfur tetrafluoride and sulfur hexafluoride. Their use would substantially simplify equipment requirements for the synthesis, since the fluorides of boron, carbon (and its derivatives, freons), silicon, nitrogen and sulfur are inert under normal conditions. In addition, the fluorides of carbon, nitrogen and sulfur (the hexafluoride) are non-toxic, while the fluorides of boron,

the derivatives of carbon fluoride (freons), and the tetrafluorides of silicon and sulfur have the additional advantage that they can be obtained without the use of elemental fluorine.

None the less, the application of the listed compounds for fluorination, with the exception perhaps of sulfur tetrafluoride, is very limited, and practically none for carbon tetrafluoride. As noted earlier [14, 15], this fact is due to the traditional, far from always justified notion regarding the great inertness of the compounds in question.

In the present work, based on thermodynamic analysis and experimental data, the perspectives of using silicon and carbon tetrafluorides, dichlorodifluoromethane and sulfur hexafluoride as fluorinating agents in syntheses of inorganic fluorides will be discussed.

An important characteristic determining the conditions of synthesis is the establishment of the temperature conditions. This is true for any synthesis, and all the more important in the case of fluorination processes.

For this reason, differential thermal analysis was chosen as the basic method for the study, since it allows one to determine the character and the conditions of the interaction and to establish optimum temperature conditions for the synthesis [9, 16–18].

Thermodynamic analysis

Thermodynamic analysis of the interactions of CF_4 , CF_2Cl_2 , SiF_4 and SF_6 with elements and compounds of groups I–VIII of the periodic system demonstrated that the reactions listed in Table 1 are in most cases thermodynamically feasible. A loss of free energy characterizes the reactions

(i) $\text{El} + \text{CF}_4$ and $\text{El} + \text{SF}_6$ for practically all elements considered, the reactions $\text{El} + \text{SiF}_4$ for alkali metals and alkaline earth metals, aluminium, yttrium, lanthanides and zirconium;

(ii) $\text{El}_2\text{O}_n + \text{CF}_4$, $\text{El}_2\text{O}_n + \text{CF}_2\text{Cl}_2$ and $\text{El}_2\text{O}_n + \text{SF}_6$ for practically all oxides considered, the reactions $\text{El}_2\text{O}_n + \text{SiF}_4$ for the oxides of groups I, II, III (with the exception of In_2O_2) and VIII, and for PbO and MnO ;

(iii) $\text{ElCl}_n + \text{CF}_4$ or SiF_4 or SF_6 for the chlorides of alkaline earthmetals and the aluminium subgroup;

(iv) $\text{El}_2\text{S}_n + \text{CF}_4$ and $\text{El}_2\text{S}_n + \text{SF}_6$ for practically all sulfides considered.

Thus, our calculations indicated that a large number of elements and their compounds are capable of reacting with the discussed fluorides of non-metals to yield the corresponding inorganic fluorides.

For practical use, it is convenient to plot the results of the thermodynamic analysis graphically. By way of example, Fig. 1 shows the results for the reactions of elements, oxides, sulfides and chlorides with carbon tetrafluoride, demonstrating that all the elements considered, their oxides, the majority of their sulfides and some of their chlorides should be suitable for fluorination with carbon tetrafluoride. It must be noted, however, that the accuracy of the $\Delta G_{\text{formation}}^\circ$ values (Table 1,

Fig. 1) taken from the literature may affect the sign of ΔG_p° , this being of particular importance in our case for ΔG_p° values from zero to + 5 kcal/mole since a reaction indicated as thermodynamically impossible by calculation may experimentally prove feasible. It therefore appeared reasonable to study not only those reactions for which $\Delta G_p^\circ < 0$, but also some others with small positive values of ΔG_p° .

Returning to the data of Table 1, it should also be noted that the effect of temperature, the possibility that the reactions take place according to another scheme, and kinetic factors may change the picture to a certain extent. Undoubtedly, however, the general conclusion remains that the non-metal fluorides considered can be used as fluorinating agents for a broad range of substances.

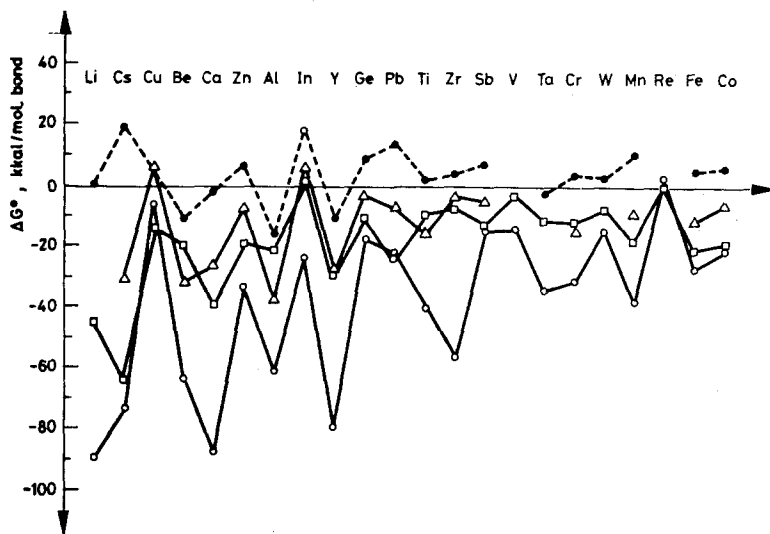


Fig. 1. Diagram of ΔG_p° values for the reactions of carbon tetrafluoride with various metals, oxides, sulfides and chlorides: \circ — $EI + CF_4$; \square — $EI_2O_n + CF_4$; — · — $EICl_n + CF_4$; \triangle — $EI_2S_n + CF_4$

Experimental

Materials: dichlorodifluoromethane (Freon 12); silicon tetrafluoride obtained by the method described in [21]; sulfur hexafluoride (Elegas); powdered metals in pure, analytically pure and chemically pure grades.

Differential thermal analysis of the gas — solid reaction was carried out in a specially constructed quartz vial allowing runs in a stream of the previously purified gaseous fluorinating agent (0.2–0.3 litres/min). The mass of the sample was 0.05 to 0.3 g. An EPP-09 M3 potentiometer was used for recording. The heating rate over the 400–800° range was close to linear; its value under the conditions of thermal analysis was 20–25°/min. A Pt–Pt/Rh thermocouple was used for temperature measurement. Ignited alumina was used as reference standard.

Table 1

Thermodynamic analysis of the reactions of CF_4 , SiF_4 , SF_6 and CF_2Cl_2 with various

Group, Elements, Stage of oxidation	$\Delta G^\circ_{\text{reaction}}$, kcal/mole·bond								
	I		II				III		
	Li	Cs	Cu	Be	Ca	Zn	Al	In	Y
	1	2	2	2	2	2	3	3	3
1 $1/n \text{El} + 1/4 \text{CF}_4 =$ $= 1/n \text{ElF}_n + 1/4 \text{C}$	-87	-73	-5	-62	-86	-32	-60	-23	-87
2 $1/n \text{El} + 1/4 \text{SiF}_4 =$ $= 1/n \text{ElF}_n + 1/4 \text{Si}$	-46	-32	36	-21	-45	9	-19	18	-37
3 $1/n \text{El} + 1/6 \text{SF}_6 =$ $= 1/n \text{ElF}_n + 1/6 \text{S}$ (g)	-86	-72	-4	-61	-85	-31	-59	-22	-77
$\Delta G^\circ = 1/n \Delta G^\circ_{\text{ElF}_n} + A$									
4 $1/2n \text{El}_2\text{O}_n + 1/4$ $\text{CF}_4 = 1/n \text{ElF}_n +$ $+ 1/4 \text{CO}_2$	-43	-63	-13	-18	-38	-18	-20	2	-29
5 $1/2n \text{El}_2\text{O}_n + 1/4$ $\text{SiF}_4 = 1/n \text{ElF}_n +$ $+ 1/4 \text{SiO}_2$	-30	-50	0	-5	-25	-5	-7	15	-16
6 $1/2n \text{El}_2\text{O}_n + 1/6$ $\text{SF}_6 = 1/n \text{ElF}_n + 1/6$ $\text{SO}_2 + 1/12 \text{O}_2$	-40	-60	-10	-15	-35	-15	-17	5	-26
7 $1/2n \text{El}_2\text{O}_n + 1/2$ $\text{CF}_2\text{Cl}_2 = 1/n \text{ElF}_n +$ $+ 1/2 \text{COCl}_2$	-45	-65	-15	-20	-40	-20	-22	0	-31
$\Delta G^\circ = 1/n(\Delta G^\circ_{\text{ElF}_n} - 1/2\Delta G^\circ_{\text{El}_2\text{O}_n}) + A$									
8 $1/n \text{ElCl}_n + 1/4$ $\text{CF}_4 = 1/n \text{ElF}_n +$ $+ 1/4 \text{CCl}_4$	1	20	7	-10	-0.5	8	-15	20	-10
9 $1/n \text{ElCl}_n + 1/4$ $\text{SiF}_4 = 1/n \text{ElF}_n +$ $+ 1/4 \text{SiCl}_4$	9	28	15	-2	7.5	16	-7	28	-2
10 $1/n \text{ElCl}_n + 1/6$ $\text{SF}_6 = 1/n \text{ElF}_n +$ $+ 1/12 \text{S}_2\text{Cl}_2(\text{g}) +$ $+ 5/12 \text{Cl}_2$	-4	15	2	-15	-6	3	-20	15	-15
$\Delta G^\circ = 1/n(\Delta G^\circ_{\text{ElF}_n} - \Delta G^\circ_{\text{ElCl}_n}) + A$									
11 $1/2n \text{El}_2\text{S}_n + 1/4$ $\text{CF}_4 = 1/n \text{ElF}_n +$ $+ 1/4 \text{CS}_2(\text{g})$		-30	7	-31	-25	-6	-36	7	-27
12 $1/2n \text{El}_2\text{S}_n + 1/4$ $\text{SiF}_4 = 1/n \text{ElF}_n +$ $+ 1/4 \text{SiS}_2(\text{s})$		-3	34	-4	2	21	-11	34	-0.5
13 $1/2n \text{El}_2\text{S}_n + 3/2$ $\text{SF}_6 = 1/n \text{ElF}_n +$ $+ 2 \text{SF}_4$		-33	4	-34	-28	-9	-41	-4	-31
$\Delta G^\circ = 1/n(\Delta G^\circ_{\text{ElF}_n} - 1/2 \Delta G^\circ_{\text{El}_2\text{S}_n}) + A$									

* Initial data for the calculation were taken from [19 and [20]

elements and their compounds*

IV				V			VI		VII		VIII	
Ge	Pb	Ti	Zr	Sb	V	Ta	Cr	W	Mn	Re	Fe	Co
4	2	4	4	3	5	5	3	6	2	6	2	2
-16	-21	-38	-55	-14	-13	-33	-30	-14	-37	3	-26	-20
25	20	3	-14	27	28	8	11	27	4	44	15	21
-15	-20	-37	-54	-13	-12	-32	-29	-13	-36	4	-25	-19
[A = 53.08 (); 93.95 (2); 53.84 (3)]												
-9	-22	-8	-6	-12	-2	-10	-11	-7	-17	0	-20	-19
4	-9	5	7	1	11	3	2	6	-4	-13	-7	-6
-6	-19	-5	-3	-9	1	-7	-8	-4	-14	3	-17	-16
-11	-24	-10	-8	-14	-4	-12	-13	-9	-19	-2	-22	-21
[A = 29.51 (4); 42.82 (5); 32.5 (6); 27.39 (7)]												
10	15	3	5	8		-0.5	5	4	12		6	7
18	23	11	13	16		7.5	13	12	20		14	15
5	10	-2	0	3		-6	0	-1	7		1	2
+ A [A = 49.12 (8); 57.06 (9); 43.48 (10):												
-2	-6	-14	-3	-4			-14		-8		-10	-5
25	21	13	24	23			13		19		17	22
-5	-9	-17	-6	-7			-17		-11		-13	-8
[A = 56.96 (11); 83.47 (12); 53.64 (13):												

Results and discussion

The elements and compounds to be studied by DTA were selected on the basis of thermodynamic calculations (Table 1, Fig. 1).

It was assumed that the solid — gas reaction takes place under the conditions of thermal analysis if sharp exoothermic effects can be observed in the heating curves. In this sense, those thermodynamically possible reactions took place for which the value of ΔG_p° did not exceed $-20 \dots -30$ kcal/mole · bond. As may be seen from Table 2, the reactions with metals and oxides of the fluorinating agents considered satisfy this condition.

Table 2 lists the results of thermal analysis for the reactions of a number of metals and oxides with dichlorodifluoromethane, silicon tetrafluoride and sulfur hexafluoride

Table 2

Results of the thermoanalytical study of the reaction conditions between dichlorodifluoromethane, silicon tetrafluoride and sulfur hexafluoride, respectively, and various metals and oxides

No.	Metal or oxide	CF_2Cl_2	SiF_4	SF_6
		$\Delta H_{\text{formation}} =$ = -113 kcal/mole	$\Delta H_{\text{formation}} =$ = -386 kcal/mole	$\Delta H_{\text{formation}} =$ = -292 kcal/mole
T, °C				
1	Zn	440	550	613
2	Al	470	910	560
3	Ti	490	520	494
4	Sb	—	none	none
5	W	none	none	684
6	CuO	none	none	none
7	CaO	740	815	727
8	CdO	none	none	none
9	Al_2O_3	—	835	530
10	Y_2O_3	470	805	657
11	Pr_6O_{11}	—	620	584
12	Nd_2O_3	530	780	647
13	Sm_2O_3	520	830	670
14	Gd_2O_3	—	880	594
15	Ho_2O_3	—	850	642
16	Er_2O_3	—	860	597
17	PbO	500	380	500
18	TiO_2	none	none	none
19	Sb_2O_3	none	—	none
20	V_2O_5	—	none	none
21	MoO_3	none	440—570	none
22	Tm_2O_3	—	805	670
23	Yb_2O_3	—	840	700
24	Lu_2O_3	—	830	590
25	Tb_4O_7	—	555	637

"None" = no thermal effect, or only a blurred effect, was recorded in the DTA curve

hexafluoride. Figures 2 and 3 present typical DTA curves for metals and oxides belonging to various groups of the periodic system, demonstrating that the reactions are accompanied by high rates of heat evolution, as indicated in the curves by sharp-ended peaks.

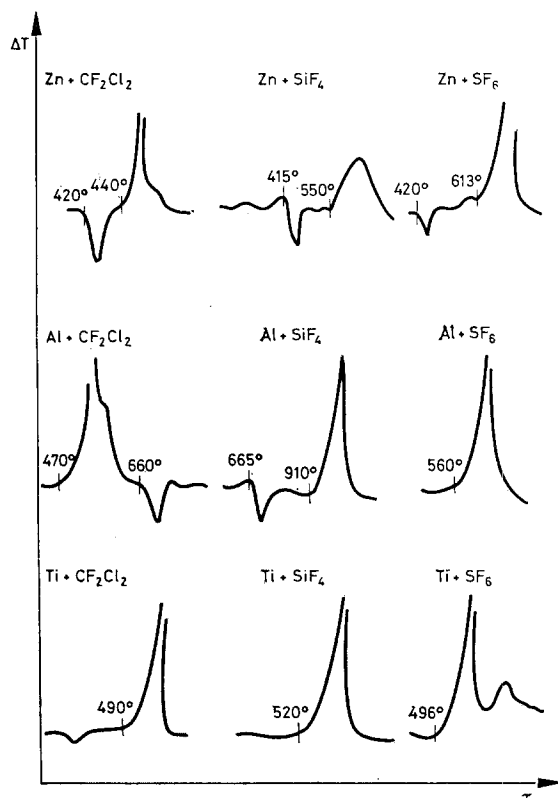


Fig. 2. Heating curves of metals in CF₂Cl₂, SiF₄ and SF₆ atmospheres

In spite of the great informative power of the DTA method for determining the conditions of synthesis of inorganic fluorides using non-metal fluorides as fluorinating agents, it should be noted that in some cases thermal effects are not recorded for reactions where $\Delta G_p^\circ < 0$, owing to the slowness of heat evolution in the course of the chemical reaction. In such cases the necessary information as to whether a reaction has taken place is supplied by the weight change of the sample and by X-ray phase analysis of the reaction product.

Turning now to the thermoanalytical data obtained, it appears that the essential difference between the processes with CF₂Cl₂ on the one hand, and with SF₆ and SiF₄ on the other, is the temperature at which the exothermic peak is recorded. Thus, aluminium and zinc react with CF₂Cl₂ before or at the moment of their

transition into the liquid state, while their reactions with SF_6 and SiF_4 require further heating of the molten metal.

A similar difference in the value of the "temperature of the start of interaction" ($t_{s,i}$) characterizes most oxides, these reacting at the lowest temperature with CF_2Cl_2 . The sequence of fluorinating power regarding metals and oxides is therefore



The differing reactivities (as regards activation energy E_a and rate constant k_v) of these fluorinating agents towards the substances to be fluorinated are due to causes determined by the nature of these components. If the substance to be fluorinated is one and the same, the reactivity will be determined by the nature of the fluorinating agent, by its structural and thermodynamic characteristics. At present it is impossible to calculate E_a and k_v using only thermodynamic and structural data. It is also rather difficult to determine E_a and k_v experimentally in heterogeneous syntheses. It is more simple and convenient to determine, by DTA, the

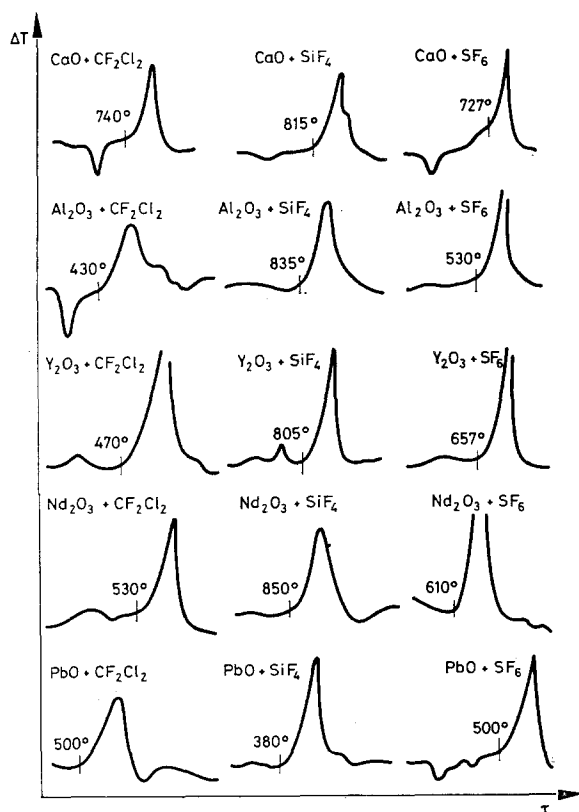


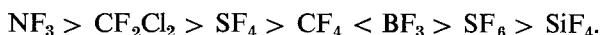
Fig. 3. Heating curves of oxides in CF_2Cl_2 , SiF_4 and SF_6 atmospheres

“temperature of the start of interaction” of the gas – solid process, which is indirectly related to E_a and k_v , since $t_{s,i}$ is the temperature corresponding to the minimum rate of the process (rate of heat evolution) which can already be recorded with a sensitive instrument. In this case, the lower the value of $t_{s,i}$, the higher the reactivity of the fluorinating agent. It may be seen from the data in Table 2 that $t_{s,i}$ is in correlation with the values of $\Delta H_{\text{formation}}^\circ$ of the fluorinating agents, increasing with the decrease of the latter. Thus, in the reaction $\text{EIF}_n + \text{Y}_2\text{O}_3$:

	CF_2Cl_2	SF_6	SiF_4
$\Delta H_{\text{formation}}^\circ$, kcal/mole	-113	-292	-386
$t_{s,i}$, °C	470	657	805

The picture obtained with other oxides and metals is similar.

It is possible that such a sequence could be observed for many other “inert” fluorinating agents, and that the sequence of fluorinating powers of non-metal fluorides is



The simplest and most convenient confirmation of this assumption can be achieved by differential thermal analysis.

To summarize, the DTA method allowed determination of the temperature conditions for the synthesis of inorganic fluorides when dichlorodifluoromethane, silicon tetrafluoride and sulfur hexafluoride were used as fluorinating agents. These nonmetal fluorides appear most promising for application in inorganic syntheses owing to a number of advantages: high inertness under normal conditions, high fluorine content and simplicity of manufacture.

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RÉSUMÉ — A partir des variations de l'énergie de Gibbs calculée à l'état standard, on a évalué la possibilité qu'il se produise une réaction entre le carbone et la fluorure de silicium, le dichlorodifluorométhane et l'hexafluorure de soufre ainsi que plusieurs éléments et leurs composés. Le déroulement effectif des réactions a été étudié par analyse thermique différentielle. Les réactions des métaux et oxydes avec le dichlorodifluorométhane, le tétrafluorure de silicium et l'hexafluorure de soufre ont été examinées et les températures où s'effectuent les synthèses ont été définies en mesurant la "température du début de réaction" ($t_{s,i}$).

En supposant l'existence d'une corrélation entre $t_{s,i}$ et $\Delta H_{\text{formation}}^{\circ}$ pour les agents de fluoration, une séquence du potentiel fluorant des fluorures non-métalliques a été établie. La perspective de l'utilisation des fluorures non-métalliques pour la synthèse des fluorures inorganiques est discutée.

ZUSAMMENFASSUNG — Auf Grund der berechneten Gibbsschen Energieänderungen des Standardzustandes wurde die Möglichkeit von Reaktionen zwischen Kohle und Silikontetrafluorid, Dichlordifluormethan, bzw. Schwefelhexafluorid, sowie einer Anzahl von Elementen und ihrer Verbindungen ausgewertet. Das tatsächliche Zustandekommen der Reaktionen wurde durch Differentialthermoanalyse untersucht. Fluorierungsreaktionen von Metallen und Oxiden mit Dichlordifluormethan, Silikontetrafluorid und Schwefelhexafluorid wurden untersucht und die Temperaturbedingungen der Synthesen durch Messungen der "Temperatur am Anfang der Wechselwirkung" ($t_{s,i}$) bestimmt.

Unter Annahme der Existenz einer Korrelation zwischen $t_{s,i}$ und $\Delta H_{\text{Bildung}}^{\circ}$ der fluorierenden Substanzen wurde eine Sequenz des Fluorierungsvermögens der Nicht-Metallfluoride aufgestellt. Die Perspektive der Anwendung von Nicht-Metallfluoriden bei der Synthese anorganischer Fluoride wird erörtert.

Резюме — На основании рассчитанного для стандартного состояния изменения энергии Гиббса оценена возможность протекания реакций тетрафторидов углерода и кремния, дифтордихлорметана и гексафторида серы с рядом элементов и их соединений.

Вопрос об осуществлении тех или иных реакций решался на основании дифференциально-термического анализа по кривым нагревания.

Проведено дифференциально-термическое исследование реакций фторирования металлов и окислов дифтордихлорметаном, тетрафторидом кремния и гексафторидом серы. Температурные условия синтеза определены по установленным температурам «начала взаимодействия» ($t_{н.в.}$).

Полагая возможность корреляции $t_{н.в.}$ с $\Delta H^\circ_{обр.}$ Фторирующих агентов, предлагается ряд фторидов неметаллов по их фторирующей способности. Обсуждаются перспективы использования фторидов неметаллов в синтезе неорганических фторидов.