Contribution No. 992. Work was performed in the Ames Laboratory of the U.S. Atomic Energy Commission.

Absorption Spectra of the Lanthanides in Fused Salt Media

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ABSTRACT

Spectra are presented for solutions of praseodymium, neodymium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, and ytterbium fluorides in fused LiCl-KCl eutectic at 400° C. These spectra are discussed and compared with similar results which have been obtained in the same and other fused salt media and in aqueous solutions. Molar absorptivities for selected absorption bands of the rare-earth spectra which might be useful in quantitative analytical determinations are also given. The spectra were obtained with the Cary Model 12 spectrophotometer using an electrically heated cell-block furnace as the cell holder.

INTRODUCTION

The absorption spectra of fused salts have been of interest for some time as aids in studying the chemical constitution and electronic structure of these media both as pure salts (12, 14-17, 24) and as ions in fused salt solutions (3, 5-9, 18, 19, 23-26, 28-30). This investigation is concerned with the determination of the unique absorption spectra of dilute

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solutions of various lanthanide fluorides in fused LiCl-KCl eutectic solvent, and the determination of the molar absorptivities for significant absorption bands from these spectra.

The molar absorptivities of the bands of analytical interest are tabulated in the same form that Banks and Klingman (2) used for their results on aqueous solutions of rare-earth mixtures. The absorbance of the solvents used was determined at the same temperatures and in the same quartz cells used for recording the spectra of the rare-earth solutions, and then was subtracted from the absorbance of the solutions in order to obtain the absorption spectra of the solutes.

EXPERIMENTAL

Apparatus and Reagents. The high-temperature cell assembly was similar to the one used by Sundheim and Greenberg (23). The temperature fluctuated $\pm 0.5^\circ$ C. in the cell block and less than $\pm 0.1^\circ$ C. in the fused salt solutions contained in the optical cells. The optical absorption cells used were one-piece quartz cells having one-centimeter light paths. These square cells were connected to a Pyrex ball joint by means of a graded seal. The ball joint could be connected to either an outer joint fitted with a vacuum stopcock for sealing the cell or to the outer joint of the filtering apparatus used in filling the cells with molten solutions. The graded seal and tubing between the cell and the ball joint provided the necessary capacity for the LiCl-KCl eutectic when it was loaded in the solid state. The lanthanide fluorides used were prepared at the Ames Laboratory of the Atomic

Energy Commission and analyzed for fluoride and rare-earth metal content. The fluoride determination was made by the pyrohydrolysis technique of Banks, Burke, and O'Laughlin (1). The oxides resulting from these determinations were weighed and the rare-earth metal content of each fluoride calculated. These oxides were used to prepare 0.1M solutions of each rare earth in 1N perchloric acid. The lithium chloride, reagent grade, used was vacuum oven-dried at 110°C. with correction for residual water content being applied from the data of Burkhard and Corbett (4) in weighing the reagent for the preparation of the eutectic mixture. The potassium chloride, reagent grade, used was vacuum over-dried at 110° C. No residual water correction was necessary.

Procedure. The LiCl and KCl were combined to yield a 59 mole per cent LiCl eutectic mixture and then fused and purified, using an apparatus similar to the one used by Gruen and McBeth (8). The small droplets of solidified eutectic obtained were stored under carbon tetrachloride until needed. The eutectic droplets did not adhere to one another, thus varying quantities could easily be withdrawn from the storage flask. Because the eutectic was quite hygroscopic, it had to be handled and weighed in a dry, inert atmosphere. The latter was obtained by using a dry box containing a nitrogen atmosphere.

Because the droplets of eutectic contained three contaminants: dissolved HCl, dissolved He and occluded CCl_4 , a filtering apparatus similar to the one used by Boston and Smith (3) was utilized. By maintaining a pressure differential across the "fine" porosity fritted disk of this apparatus, the melt

could be supported above the disk or filtered through it. This apparatus, shown in Figure 1, was wrapped with heating tape from below the tapered joint to just above the outer ball joint. The optical cell or equivalent receiving tube was attached at the ball joint and enclosed in an electric furnace.

In order to make the spectrophotometric measurements as quantitative as possible, two procedures were followed using the filtering apparatus for purifying the eutectic droplets and filling the optical cells.

In the first procedure, the eutectic was removed from the storage flask in the dry box and approximately 6.7 grams weighed into a small bottle. A Pyrex receiving tube was attached to the filtering apparatus and the entire apparatus was dried by heating it to 150°C. and passing dry helium gas through it. The eutectic was transferred from the small bottle to the filtering apparatus by quickly opening and closing the chamber at the upper tapered joint. A vacuum was applied above the disk and anhydrous HCl gas was allowed to enter below it as the apparatus was slowly heated to melt the eutectic. The HCl gas, dispersed as fine bubbles by the fritted disk, was passed through the melt as the temperature was raised to 500° C. and for about ten minutes thereafter. Helium gas was then passed through the melt for about thirty minutes while the melt temperature was maintained at 500° C. Next, a vacuum was applied below the fritted disk and helium gas pressure above the melt. The melt was thereby filtered through the disk and flowed into the receiving tube below. After the transfer was completed, the filtering chamber

Figure 1. Butectic purification and filtering apparatus.

was allowed to cool slowly to 200° C. while the receiving tube temperature was maintained at 500° C. with a vacuum applied to the entire apparatus. Helium was allowed to enter the apparatus. The receiving tube was then disconnected and resealed with an outer ball joint and stopcock connection while a vacuum was applied. The tube was then removed from the furnace and cooled rapidly. Another receiving tube was connected to the apparatus and the process repeated for another quantity of eutectic. The cooled eutectic could then be accurately weighed in these sealed tubes. In the dry box the eutectic easily slipped out of the tubes and could be loaded into dry optical cells.

The second procedure used was much the same as the one mentioned above except that the special optical cells were attached to the filtering apparatus and weighed amounts of the lanthanide fluoride were added to the purified eutectic, thoroughly mixed and the mixture filtered directly into the optical cell. In order that quantitative measurements could be made, the previously filtered and accurately weighed eutectic from the first procedure was replaced above the fritted disk, instead of the eutectic weighed approximately from the storage flask. Naturally, the entire filtering apparatus had to be cooled and thoroughly cleaned between the preparation of each lanthanide fused salt solution.

The absence of water and oxygen from the eutectic solvent and solutions prepared in this manner was evidenced not only by the complete absence of etch on the highly polished quartz cells even after 48 hours at temperatures in excess of 400° C., but also

by the reproducibility of the absorption spectra.

The pure eutectic was used to determine the base line for the spectral measurements of the solutions. The lanthanide fluorides could be accurately weighed and added to the eutectic while the cells containing the fused eutectic were at temperature in the cell-block furnace. Several hours were required, however, to be certain that all of the added salt had dissolved in the eutectic and that the solution was homogeneous. The spectra of solutions prepared in the cell block furnace by addition of the lanthanide fluorides yielded the same reproducible spectra and molar absorptivities as the solutions mixed and filtered into the optical cells.

Molar concentrations were calculated using the density values for the LiCl-KCl eutectic determined by Van Artsdalen and Yaffee (27). Their value of 1.6741 grams per cubic centimeter at 400° C. was used in calculating the molarity of each solution from the weight of the rare-earth fluoride and the weight of the eutectic used. It was assumed that the density of the eutectic solvent was that of the solution. Because only the conventional cell holder was in the reference cell compartment of the spectrophotometer, the molar absorptivities are versus "air" as the reference standard.

After obtaining the weight of the eutectic solvent placed either directly into the optical cell, or into the filtering apparatus for further transfer to the optical cell, the amount of lanthanide fluoride needed to yield a solution 0.1, 0.2, 0.3 or 0.5M in lanthanide concentration was calculated, weighed, and

transferred to the optical cell or filtering apparatus. By making the molar concentrations multiples of 0.1M concentration, the molar absorptivities were easily determined directly from the absorption spectra recorded for each solute concentration at the selected wavelengths.

RESULTS AND DISCUSSION

The absorption bands occurring in the infrared region of the spectrum were not resolved due to the large slit widths required. A comparison of wavelengths and molar absorptivities observed in the LiCl-KCl eutectic and HClO_4 solutions is presented for the principal absorption bands occurring in the spectral region studied. The narrow bands so characteristic of aqueous solution spectra of the lanthanides are seen to be largely preserved in the fused salt solution spectra.

The charge transfer band studied quite extensively by numerous investigators and discussed theoretically by Orgel (13) was observed as a "cut-off absorption edge" in the ultraviolet region of most of the fused salt solution spectra.

The red shift in wavelength caused by the increased temperature and concentration of chloride ion was also observed. The shift toward longer wavelengths of the absorption bands between 400° C. and 500° C. was too small to be calculated accurately, but was observed to some extent in the spectra of all of the lanthanides.

Praseodymium Fluoride. The wavelengths and molar absorptivities of the principal absorption bands found in the

spectrum of praseodymium fluoride in the fused eutectic and of praseodymium oxide in perchloric acid, are presented in Table I (see also Figure 2), along with the results which have been reported in other solvents. The absorption band occurring at 522 m μ in the fused eutectic has been previously reported (30). However, the band at 486 m μ and the shoulder band at 483 m μ have not been reported by other investigators. The wavelengths of the absorption bands in the spectrum shown agree closely with those reported by Gruen (6) in LiNO₃-KNO₃ eutectic. Gruen added KCl to the fused nitrate eutectic solutions studied until the solubility limit of the chloride ion was reached and observed only minimal changes in the spectra of trivalent praseodymium and trivalent neodymium.

Table I. Spectrum of Praseodymium in Various Solvents

LiCl-KCl 400° C.		LiF (30) 900° C.		LiF-NaF-KF 550° C. (29)		LiNO ₃ -KNO ₃ 184° C. (6)		HClO ₄ 25° C.	
λ , m μ	ϵ	λ , m μ	ϵ	λ , m μ	ϵ	λ , m μ	λ , m μ	ϵ	ϵ
448	1.47	444	1.2	444	1.4	447	444	9.49	
473	1.12	468	-	467	-	471	469	4.30	
483	1.30	479	1.1	479	1.3	-	482	3.95	
486	1.45	-	-	-	-	485	-	-	
522	-	522	-	(522)	-	-	-	-	
597	0.35	585	-	588	-	590	589	1.90	

Neodymium Fluoride. The wavelengths and molar absorptivities of the principal absorption bands found in the spectrum of neodymium fluoride in fused LiCl-KCl eutectic and of neodymium oxide in HClO₄ are presented in Table II (see also Figure 3), along with the selected results which have been reported in other solvents. The neodymium fluoride fused salt solutions were a blue-green color.

Figure 2. A. 0.2 M Praseodymium (III) in LiCl-KCl at 400°C.
B. 0.1 M Praseodymium (III) in 1 N HClO₄ at 25°C.

Table II. Spectrum of Neodymium in Various Solvents

LiCl-KCl 400° C.		LiCl-KCl 373° C. (25)		LiF (30) 900° C.		LiNO ₃ -KNO ₃ 184° C. (6)		HClO ₄ 25° C.	
λ	ε	λ	ε	λ	ε	λ	λ	ε	
-	-	-	-	-	-	-	253	2.10	
262	1.40	-	-	-	-	-	259	1.60	
353	2.70	-	-	350	-	-	347	2.70	
358	1.90	-	-	356	-	-	351	2.40	
360	1.89	360	-	-	-	-	354	3.70	
532	1.87	535	-	532	-	526	522	4.10	
582	6.90	-	-	582	1.70	-	575	6.83	
589	11.90	590	1.62	-	-	585	578	5.00	
597	7.00	-	-	-	-	-	-	-	
751.5	2.30	775	0.31	747	1.00	739	740	6.50	
808	2.55	810	0.27	803	1.10	801	794	8.08	
-	-	-	-	-	-	-	800	5.60	
881	0.87	885	-	874	-	871	862	2.90	

Samarium Fluoride. The wavelengths and molar absorptivities of the principal absorption bands found in the spectrum of samarium fluoride in fused LiCl-KCl eutectic and of samarium oxide in HClO₄ are presented in Table III (see also Figure 4), along with the results which have been reported in other solvents.

Table III. Spectrum of Samarium in Various Solvents

LiCl-KCl 400° C.		LiF (30) 900° C.		HClO ₄ (20) 25° C.		HClO ₄ 25° C.	
λ	ϵ	λ	ϵ	λ	ϵ	λ	ϵ
303	cut-off	-	-	306	0.15	306	0.30
348	0.95	340	-	344	0.52	345	0.52
364	0.93	358	-	362	0.69	361	0.55
380	0.90	373	-	375	0.66	375	0.48
407.5	2.65	399	0.63	401.6	2.98	402	2.31
426	0.65	-	-	416	0.43	416	0.48

Figure 3. A. 0.1 M Neodymium (III) in LiCl-KCl at 400°C.
B. 0.1 M Neodymium (III) in 1 N HClO₄ at 25°C.

Table III (concluded)

LiCl-KCl 400° C.		LiF (30) 900° C.		HClO ₄ (20) 25° C.		HClO ₄ 25° C.	
λ	ϵ	λ	ϵ	λ	ϵ	λ	ϵ
427	0.69	419	-	418	0.38	418	0.43
451	0.10	441	-	442	0.16	444	0.15
471	0.15	460	-	464	0.50	464	0.30
477	0.12	468	-	479	0.55	479	0.40
940-60	0.10	-	-	955	0.35	940-8	0.15

Europium Fluoride. The wavelengths and molar absorptivities of the principal absorption bands found in the spectrum of europium fluoride in fused LiCl-KCl eutectic and of europium oxide in HClO₄ are presented in Table IV (see also Figure 5), along with the results of Stewart (20). The spectrum of europium chloride in LiCl-KCl eutectic has been studied qualitatively (25), but only the ultraviolet cut-off at 240 m μ with no superimposed fine structure was reported.

The concentration of europium was made 0.01M in order to reduce the effect of the charge transfer band or ultraviolet cut-off as shown by 0.1M concentrations of europium in LiCl-KCl eutectic. The molar absorptivities calculated from the fused salt spectrum are not linear with respect to concentration unless the contour of the charge transfer band is used as the baseline absorbance value. The wavelengths of the fused salt solution bands and the aqueous solution bands compare very well.

Figure 4. A. 0.1 M Samarium (III) in LiCl-KCl at 400°C.
B. 0.1 M Samarium (III) in 1 N HClO₄ at 25°C.

Table IV. Spectrum of Other Lanthanides in Two Solvents

Lanthanide	LiCl-KCl 400° C.		HClO ₄ 25° C.		HClO ₄ 25° C.	
	λ	ϵ	λ	ϵ	λ	ϵ
(20)						
Eu	397	0.33	394	2.36	394	2.80
	465	0.04	465	0.052	465	0.05
	526	-	526	0.078	526	-
	536	0.008	536	0.029	536	-
(20)						
Gd	243	1.60	244	0.145	243	1.10
	246	1.45	246	0.158	246	1.10
	248	1.48	252	0.147	252	0.95
	254	1.50	-	-	-	-
	255	1.40	-	-	-	-
	274	1.30	272.7	2.04	273	3.55
	275	1.15	-	-	274	1.63
	277	1.10	276	1.22	276	2.30
	307	0.45	305	0.042	305	0.15
	313	0.52	311	0.086	311	0.22
	340	0.12	-	-	-	-
(22)						
Tb	255	cut-off	258	0.49	-	-
	265	0.35	264	0.74	-	-
	285	0.10	284	0.23	284	0.20
	304	0.02	303	0.09	310	0.10
	320	0.12	318	0.05	-	-
	340	0.04	342	0.11	-	-
	379	0.10	378	0.19	377	0.15
	489	0.02	488	0.03	488	0.05

Table IV (continued)

Lanthanide	LiCl-KCl 400° C.		HClO ₄ 25° C.		HClO ₄ 25° C.	
	λ	ϵ	λ	ϵ	λ	ϵ
(22)						
Dy	240	cut-off	256	1.16	256	1.80
	300	0.18	295	0.92	295	0.80
	328	0.08	325	1.80	325	1.90
	353	0.28	351	2.57	351	2.60
	367	0.04	365	2.16	365	2.10
	389	0.13	388	0.89	388	0.90
	428	0.04	428	0.11	428	0.30
	453-4	0.11	453	0.11	453	0.32
	810	0.055	809	1.87	807	1.50
	911	0.08	911	2.51	907	1.70
(22)						
Ho	235	cut-off	239	0.28	239	1.75
	242	1.72	241	3.28	241	4.70
	252	0.80	250	0.45	250	1.90
	262	0.70	260	0.12	260	1.00
	280	0.80	278	1.97	278	2.15
	289	0.90	288	3.14	288	3.25
	290	0.65	289	1.20	289	1.26
	336	0.40	333	0.68	334	0.70
	363	4.05	361	2.00	362	2.10
	418	0.75	416	2.46	416	2.60
	423	1.00	422	0.81	422	0.95
	451.5	10.30	451	3.92	451-2	3.90
	456	8.10	-	-	456	1.80
	460	12.10	468	0.73	468	0.78
	475	0.50	474	0.82	474	0.82
	488	0.28	486	1.71	486	1.80
	539	0.62	537	4.44	537	4.63
	545	0.58	543	1.72	543	1.80
	644	0.38	641	2.83	641	3.10
	656	0.47	654	0.95	654	0.96

Table IV (continued)

Lanthanide	LiCl-KCl 400° C.		HClO ₄ 25° C.		HClO ₄ 25° C.	
	λ	ϵ	λ	ϵ	λ	ϵ
(22)						
Er	no cut-off					
	257	2.18	255	7.97	255	7.60
	259	2.22	259	0.67	259	0.68
	364	0.50	364	2.06	364	2.00
	368	0.90	-	-	368	0.50
	380	9.55	379	6.66	379	6.00
	409	0.20	-	-	408	0.50
	454	0.20	454	0.43	454	0.35
	489	0.40	487.5	2.15	487	1.90
	524	6.30	523.5	3.46	524	3.10
	-	-	542	0.76	542	0.80
	550	0.30	549	0.24	549	0.30
	655	0.45	654	2.12	653	2.12
	667	0.22	-	-	665	0.68
	804	0.14	807	0.29	803	0.23
(22)						
Tm	261	2.60	-	-	260	1.70
	263	2.30	262	0.95	262	1.75
	274	1.37	273.5	0.26	274	0.42
	279	0.93	-	-	276	0.48
	290	0.67	284	0.29	284	0.50
	-	-	356.5	0.75	357	0.72
	362	0.28	360	0.79	361	0.76
	-	-	464	0.46	464	0.45
	473	0.25	-	-	469	0.10
	694	0.37	682.5	2.34	683	2.52
	800	0.88	781	1.01	780	1.00
(22)						
Yb	323	cut-off	-	-	250	1.85
	950	0.50	950	0.72	945	0.77
	962	0.60	-	-	966	1.55
	975	1.25	978	2.00	970	1.50

Figure 5. A. 0.1 M Europium (III) in LiCl-KCl at 400°C.
 B. 0.1 M Europium (III) in 1 N HClO₄ at 25°C.

Gadolinium Fluoride. The wavelengths and molar absorptivities of the principal absorption bands found in the spectrum of gadolinium fluoride in fused LiCl-KCl eutectic and of gadolinium oxide in HClO_4 are presented in Table IV (see also Figure 6), along with the results of Stewart (20). The aqueous absorption band at 252 $\text{m}\mu$ splits into three absorption bands at 248 $\text{m}\mu$, 254 $\text{m}\mu$, and 255 $\text{m}\mu$ in the LiCl-KCl eutectic. The molar absorptivity value, 3.43 liters/mole-centimeter, given by Moeller and Brantley (11) for the 272.7 $\text{m}\mu$ band in HCl solution, agrees better with the 3.55 liters/mole-centimeter value obtained in HClO_4 solution in this study than the 2.04 liters/mole-centimeter value of Stewart (20).

Other Lanthanides. The wavelengths and molar absorptivities of the principal absorption bands found in the spectra of terbium, dysprosium, holmium, erbium, thulium, and ytterbium fluoride in fused LiCl-KCl eutectic and their respective oxides in HClO_4 , are presented in Table IV (see also Figures 7 through 12). These spectral measurements are compared with the results of Stewart (22) in the various sections of the table.

Cerium, Promethium, and Lutetium. The spectra of cerium, promethium, and lutetium are not presented for various reasons. Cerium has broad, intense absorption bands which occur in the ultraviolet region of the spectrum very near the fused LiCl-KCl eutectic cut-off in quartz cells, as shown by the results of several investigators (2, 10, 11) of aqueous solutions of trivalent cerium compounds. Only an absorption edge in the ultraviolet would be expected and would be of little

Figure 6. A. 0.1 $\underline{M}$ Gadolinium (III) in $\underline{LiCl-KCl}$ at 400°C .
B. 0.1 $\underline{M}$ Gadolinium (III) in 1. $\underline{N}$ $\underline{HClO_4}$ at 25°C .

Figure 7. A. 0.5 $\overline{M}$ Terbium (III) in LiCl-KCl at 400°C.
 B. 0.1 $\overline{M}$ Terbium (III) in 1 $\underline{N}$ HClO_4 at 25°C.

Figure 8. A. 0.5 $\underline{M}$ Dysprosium (III) in LiCl-KCl at 400°C.
 B. 0.1 $\underline{M}$ Dysprosium (III) in 1 $\underline{N}$ HClO₄ at 25°C.

Figure 9. A. 0.1 M Holmium (III) in LiCl-KCl at 400°C.
B. 0.1 M Holmium (III) in 1 N HClO₄ at 25°C.

Figure 10. A. 0.1 $\overline{\text{M}}$ Erbium (III) in LiCl-KCl at 400°C .
B. 0.1 $\overline{\text{M}}$ Erbium (III) in 1 $\underline{\text{N}}$ HClO_4 at 25°C .

Figure 11. A. 0.3 M Thulium (III) in LiCl-KCl at 400°C.
 B. 0.1 M Thulium (III) in 1 N HClO₄ at 25°C.

Figure 12. A. 0.1 $\underline{\text{M}}$ Ytterbium (III) in LiCl-KCl at 400°C.
 B. 0.1 $\underline{\text{M}}$ Ytterbium (III) in 1 $\underline{\text{N}}$ HClO_4 at 25°C.

analytical value other than a possible evaluation as to cerium interference in the determinations of other rare earths in its presence.

Promethium was not available in any form to the authors for investigation in this study.

Lutetium fluoride was dissolved in the fused LiCl-KCl eutectic and, as expected, there was no absorption found in the wavelength region investigated in this study.

SUMMARY

As can be seen from the spectra presented and the molar absorptivities determined, it would not be practical to determine the concentrations of europium, gadolinium, or terbium in fused salt media by the spectrophotometric technique used in this study. Other lanthanides, with the possible exception of dysprosium, could be determined in the presence of gadolinium and terbium but not in the presence of europium by this technique.

Table V shows the main absorption bands of eight of the lanthanide fluorides in fused LiCl-KCl eutectic at 400° C., and the interference molar absorptivities of the other lanthanides listed in the table at each absorption band.

Table V. Molar Absorptivities of Analytically Significant Lanthanide Absorption Bands at 400° C.

λ	Molar Absorptivities, Liters/Mole-Centimeter							
	Pr	Nd	Sm	Dy	Ho	Er	Tm	Yb
289	-	-	cut-off	0.09	0.90	-	0.68	cut-off
353	-	2.70	0.50	0.28*	0.20	-	-	0.70
360	-	1.89*	0.50	0.08	0.80	0.25	0.25	0.38
363	-	1.10	0.93	0.04	4.05*	0.30	0.27	0.30
380	-	0.20	0.90	0.05	-	9.55*	-	0.10
407.5	-	-	2.65*	0.02	0.05	0.10	-	-
460	0.87	0.22	0.08	0.05	12.10*	0.12	0.05	-
483	1.30*	0.25	0.05	0.03	0.12	0.15	0.07	-
486	1.45*	0.16	0.06	0.02	0.20	0.25	0.04	-
524	0.07	1.02	0.10	-	-	6.30*	-	-
539	-	1.00	0.10	-	0.62*	0.40	-	-
589	0.28	11.90*	-	-	-	0.12	-	-
644	-	0.05	-	-	0.38*	0.15	-	-
694	-	0.20	-	-	-	0.06	0.37*	-
751.5	-	2.30*	-	-	-	-	-	-
808	-	2.55*	-	0.05	-	0.10	0.70	-
911	-	0.20	0.05	0.08*	0.12	-	-	0.20
975	-	-	-	-	-	-	-	1.25*

*Analytically significant absorption bands

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