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### Title

THE ABSORPTION SPECTRUM OF  $Cf^{+3}$

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THE ABSORPTION SPECTRUM OF  $\text{Cf}^{+3}$ 

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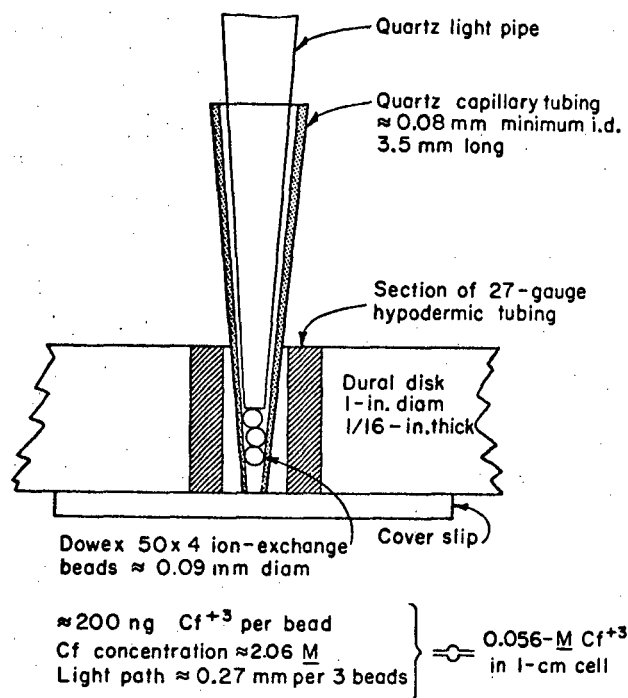
Absorption spectra have proven to be of great value in chemical studies of the 5f elements and represent a logical early objective in the systematic investigation of the chemistry of the higher actinides.

We report here observations of the absorption spectrum of  $\text{Cf}^{+3}$  in a single crystal of anhydrous  $\text{CfCl}_3$  and in a polystyrene-sulfonic acid matrix (Dowex 50). The work was done with submicrogram quantities of  ${}_{98}\text{Cf}^{249}$ ; the techniques developed would appear to be applicable to other actinide elements of limited availability and are described briefly.

The utilization of a single bead of cation ion exchange resin as an ultramicro absorption cell has been reported previously.<sup>(2)</sup> This technique consists of saturating an appropriate bead of Dowex 50 ion exchange resin with the cation of interest and visually observing the transmitted spectrum using a hand spectroscope and microscope optics. Experiments of this type performed with  $\text{Cf}^{+3}$  were encouraging enough to prompt the development of a technique in which multibead stacks were used to increase the effective path length of the cell. The configuration of the cell used is shown in Fig. 1. To obtain permanent records the apparatus

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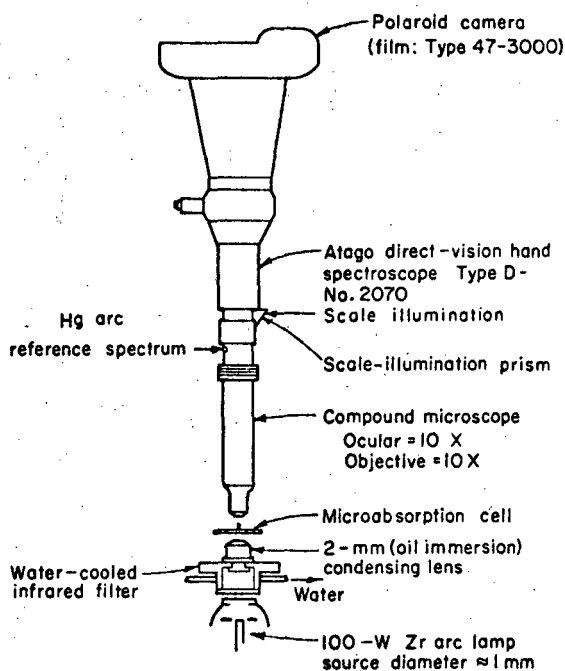
\* Present address: Los Alamos Scientific Laboratory, University of California, Los Alamos, New Mexico



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FIG. 1 Stacked bead microabsorption cell.

shown in Fig. 2 was used to photograph the spectra. Poloroid Type 3000 film was used in the visible region and Type 47 in the infrared region. The wavelength scale in the hand spectroscope (Atago hand spectroscope, Type D) was calibrated on each exposure by simultaneously photographing a Hg arc spectrum adjacent to the absorption spectrum and the superimposed image of the scale. Observed spectral features are listed in Table I. Parallel experiments on  $\text{Pr}^{+3}$  indicated that line positions found using this technique were not noticeably shifted from those observed in solution. Further, the relative intensities of the absorption lines, as judged by visual inspection of the photographic films were the same as those found in solution. Less precise comparisons using  $\text{Nd}^{+3}$  and  $\text{Pu}^{+3}$  again indicated that there were no pronounced differences in relative intensities or line positions between the aqueous and bead spectra; accordingly we have estimated molar extinction coefficients for aqueous  $\text{Cf}^{+3}$  by comparison of photographs of the spectra of  $\text{Cf}^{+3}$  and  $\text{Pr}^{+3}$  in cation exchange resin. The estimated coefficients may be in error by a factor of two.



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FIG. 2 Apparatus for photographing spectra through a hand spectroscope.

In preparing samples of  $\text{CfCl}_3$  for crystallographic studies, one sample accidentally was converted to a single crystal of  $\text{CfCl}_3$  which contained approximately 150 ng of Cf. The sample was a clear, emerald green, roughly spherical particle approximately 35 microns in diameter, contained in a thin quartz x-ray capillary.

A major obstacle to obtaining the spectrum was the construction of a suitable light mask. The system developed for this purpose is shown in Fig. 3; it consisted of a pair of razor blades positioned in such a way as to constitute a combination positioning trough and a one-dimensional slit mask with a separation slightly less than 35 microns. Additional masking was achieved by the application of an opaque graphite-grease mixture along the length of the capillary. The general configuration of the apparatus used for recording the spectrum is shown in Fig. 4. The spectrograph was a 3 meter, f6.3, Model 75-000 Jarrel Ash fitted with a 15,000 line per inch grating blazed for 7500 Å. It was possible to cover the

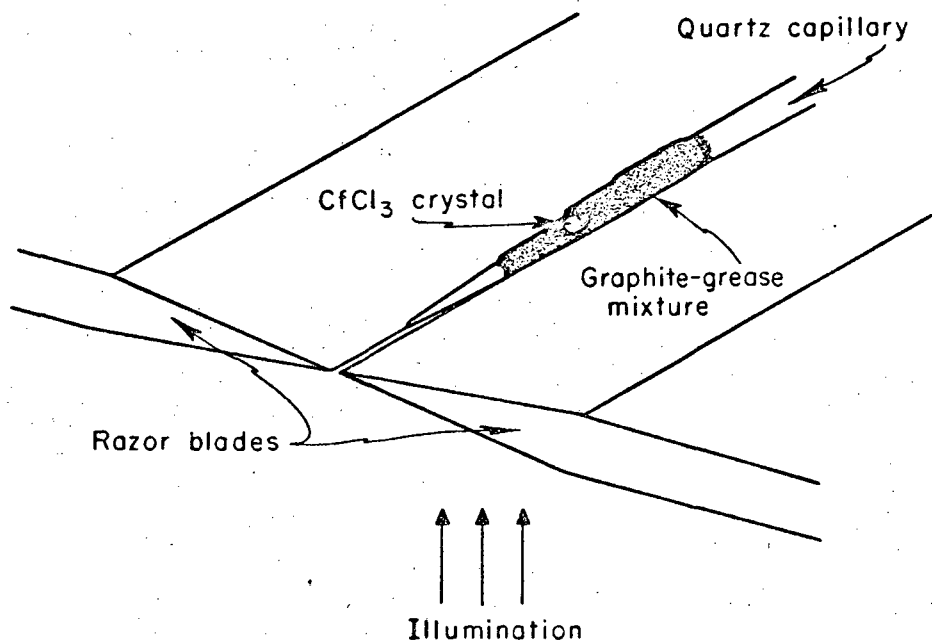
TABLE I

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Absorption Spectrum of  $\text{Cf}^{+3}$  Absorbed on Cation Exchange Resin.

Peak Position ( $\text{m}\mu$ )	Extent ( $\text{m}\mu$ )	Qualitative Description	Estimated $\epsilon$
$473 \pm 2$	469 - 475	strong	5 - 10
$491 \pm 2$	489 - 493	weak and relatively sharp	1 - 2
$591 \pm 2$	590 - 618	moderately strong	2 - 4
$595 \pm 2$			
$605 \pm 2$			
$620 \pm 3$	--	very weak (uncertain)	--
$645 \pm 3$	638 - 650		1 - 2
$675 \pm 3$	670 - 680		2 - 4
no structure	720 - 760		5 - 10

\* Visual observation indicated two lines; however, photographically, often only one moderately strong line at  $593 \text{ m}\mu$  was apparent.

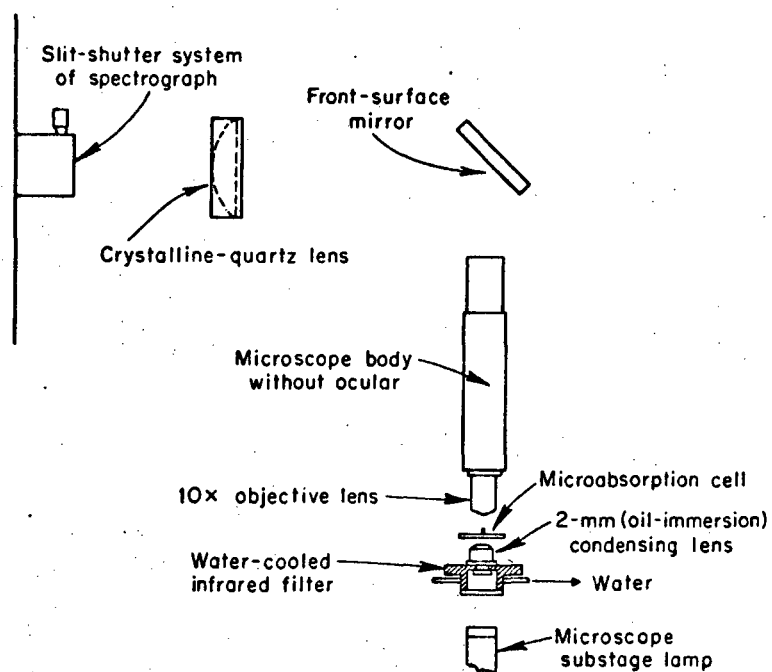


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FIG. 3. Microabsorption cell for  $\text{CfCl}_3$  crystal.



wavelength region from approximately 4000 Å to 10,000 Å. Multiple exposures of the absorption spectra with adjacent Hg arc reference spectra were taken using glass spectrographic plates of a type appropriate to the wavelength region covered. The wavelengths corresponding to peak maxima were computed from measurements taken from densitometer tracings of the absorption and reference spectra. No attempt was made to separate incompletely resolved components. The spectral features are summarized in Table II. Relative intensities of the absorption lines were estimated visually.



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FIG. 4. Apparatus used to record absorption spectra on a grating spectrograph.

Absorption spectrum of a single crystal of anhydrous  $\text{CfCl}_3$  photographed using a grating spectrograph.

Peak position (A)	Error limits (A)	Description of components	Relative intensity of band
4304	5	major peak	
4383	5	small shoulder	moderate-broad
4505	5	small shoulder	
4579	5	small shoulder	
4828	5	major peak	strong-moderately broad
5005	5	major peak	weak-sharp
5837 <sup>a</sup>	5	trace	trace-very sharp
6148	5	major peak	
6237	10	minor shoulder	moderate-broad
6295	10	medium shoulder	
6678	5	large shoulder	weak-broad
6872	5	major peak	
7539	5	major peak	strong-moderately broad
7764	5	major peak	strong-broad
8043 <sup>a</sup>	5	trace	trace-very sharp
8546 <sup>b</sup>	5	major peak	
8734 <sup>b</sup>	10	minor shoulder	weak-broad
8907 <sup>b</sup>	10	minor shoulder	

<sup>a</sup>Lines ascribed to Nd impurity. Mass analysis showed that the sample contained 0.32 atom per cent Nd.

<sup>b</sup>Only one useful exposure available; therefore, the measurement of peak positions was not done in duplicate.

A preliminary analysis of the data has been carried out by Dr. Kathryn Rajnak (3,1); however, since the spectra were recorded at room temperature, the individual crystal field components of the J levels were not resolved,

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leading to uncertainty in the assignment of J values. It is hoped that this difficulty can be overcome by refining the technique to allow the spectrum to be taken at low temperatures.

Acknowledgment: This work was performed under the auspices of the United States Atomic Energy Commission.

#### REFERENCES

- <sup>1</sup>J. L. Green, Lawrence Radiation Laboratory Report UCRL-16516 (1965).
- <sup>2</sup>B. B. Cunningham, Microchemical Journal Symposium (1961).
- <sup>3</sup>K. Rajnak, Argonne National Laboratory, Argonne, Illinois. Unpublished calculations (1965).

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