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# COMPOUNDS OF CALIFORNIUM*

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Techniques which were successful in preparing five new berkelium (1) compounds on the microgram scale, were applied in the preparation of californium compounds. Californium 249, the alpha emitting isotope ($T_{\frac{1}{2}}$ = 323y.) is the nuclide most suitable for chemical study. It is the daughter resulting from the beta decay of $^{249}Bk$ according to the reaction

$$^{249}Bk \xrightarrow[314\ d]{\beta^-} {}^{249}Cf$$

and may be milked from samples of $^{249}Bk$ that have been allowed to decay for appropriate lengths of time.

The separation of Cf from its parent is accomplished by taking advantage of the fact that Bk can be oxidized to the IV state by $NaBrO_3$ in strong nitric acid. Under these conditions the Bk(IV) can be extracted into di(2-ethyl-hexyl) ortho phosphoric acid (HDEHP). The Cf remaining behind is isolated from the residual solution by means of an extraction chromatography column (2) using HDEHP on diatomaceous earth. This method is effective in separating Cf from the alkali as well as the alkaline earths and aluminum. Spectroscopic analysis of a two microgram sample of the purified Cf showed the only impurity to be a small amount of europium.

*Based on work performed under the auspices of the U. S. Atomic Energy Commission.

All experiments were carried out on the 1-3 microgram scale. Powder diffraction patterns were obtained with a Norelco camera (114.59 mm diameter) and CuKα radiation ($\lambda K\bar{\alpha}$ = 1.5417Å).

Californium bromide--A small fragment of californium hydroxide was introduced into a quartz x-ray capillary, attached to a vacuum system and exposed to gaseous HBr admitted to 1/2 an atmosphere. The HBr was obtained from a sample portion of liquid HBr kept at a temperature of -75°C. The end of the capillary was heated to about 500°C for a few minutes after which the HBr was pumped off. The temperature was then raised to about 785°C and a white solid sublimed out and condensed in the cool portion of the capillary. The capillary was sealed off and submitted for x-ray diffraction analysis.

$CfBr_3$--The pattern is not of the characteristic β or orthorhombic modification of which $PuBr_3$ is a prototype. One preparation (1) of $BkBr_3$ was isostructural with the $PuBr_3$, while another preparation was similar to the $CfBr_3$ reported here. As this composition is not the α-modification ($UCl_3$-type) it remains to be determined if this form of $CfBr_3$ represents a third structure-type.

Californium Oxybromide--The sample containing the californium bromide was opened at one end and the open capillary placed inside another x-ray capillary just large enough to receive it. The assembly was attached to a manifold, pumped out and exposed to the mixture of $H_2O$ - HBr vapors from a saturated solution of HBr. It was heated for a few minutes at 550°C. The resulting brown product was sealed off in the x-ray capillary and examined by x-ray diffraction analysis.

CfOBr--The diffraction pattern shows that CfOBr is isostructural with BkOBr (1). The cell is tetragonal with a = 3.90 A and c = 8.12 A, and belongs to the PbFCl-type.

Californium Triiodide--A sample of precipitated californium hydroxide was placed at the bottom of a quartz x-ray capillary and in a manner similar to that described for the preparation of the californium bromide, was exposed to the vapors from liquid HI. After reaction the temperature was raised to approximately 800°C when a lemon yellow sublimate appeared outside the furnace zone. When this capillary was sealed off to the proper length it was submitted for x-ray diffraction analysis.

$CfI_3$--This compound is isostructural with $BkI_3$ (1) and is of the $BiI_3$-type. Cell dimensions based upon hexagonal axes are: a = 7.55 A and c = 20.8 A.

Californium Oxyiodide--When the capillary containing the $CfI_3$ was opened in the manner described above for californium bromide, and heated in an HI - $H_2O$ atmosphere from saturated aqueous HI, at 550°C for about 2 hours, a tan colored reaction product resulted.

CfOI--This compound is also of the PbFCl-type and is isostrucutral with the BkOI (1) salt. The dimensions of the tetragonal cell are: a = 3.97 A and c = 9.14 A.

Californium Sulfide--When a small amount of californium hydroxide is treated with $H_2S$ (which had been bubbled through $CS_2$) at 1100°C a black solid results. This sample was sealed in a quartz x-ray capillary for x-ray analysis.

$Cf_2S_3$--The sulphide is cubic and of the $Ce_2S_3$-type. The cell dimensions, a = 8.388 A, are measurably smaller than those of $Bk_2S_3$ (1) for which a = 8.44 A.

A comparison of the reactions of Bk and Cf when subjected to the same conditions reveals a close similarity between the two elements. This observation confirms the expectation that these actinide elements exhibit close relationship in their chemical properties.

## References

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