

activation greatly increased by a hydrogenic environment.

Since the Ba^{134} resulting from the decomposition is not found by mass spectrum analysis, it may be presumed to be unstable. However, we have added a small amount of barium to cesium nitrate activated by neutrons and upon precipitating the barium chemically, have found that the barium shows no activity of half-lives between ten minutes and three days. Chemical separations also show the absence of radio-iodine or radio-xenon.

We are indebted to Dr. L. R. Taussig and the University of California Hospital for 150 milligrams of radon used in the experiments.

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA

W. M. LATIMER
D. E. HULL
W. F. LIBBY

RECEIVED MARCH 25, 1935

THE REACTION OF FLUORINE WITH NITRIC ACID AND WITH SOLID POTASSIUM NITRATE TO FORM NO_3F

Sir:

Cady [THIS JOURNAL, 56, 2635 (1934)] has recently discovered that when fluorine is bubbled through 3 *N* nitric acid, the remarkable compound NO_3F is formed. Through Dr. Cady's kindness his apparatus was made available to us and we have repeated the preparation. The resulting material has all the properties described by him. It boils at about -42° , it liberates iodine from iodides and, in the gaseous form, it explodes when heated to some 200 or 300° .

We have found that NO_3F reacts very slowly with VO^{++} to give vanadic acid, and that no appreciable reaction takes place with Mn^{++} in 6 *N* H_2SO_4 , as judged by color changes. When, however, the gas is brought in contact with solutions of silver nitrate, a black precipitate (Ag_2O_3) forms at once. Mixtures of Mn^{++} and Ag^+ in 6 *N* H_2SO_4 reacted with the NO_3F to give permanganate. The reaction with silver ion is convenient for testing purposes if ozone and OF_2 are known to be absent.

We have also found that fluorine reacts readily and efficiently with solid potassium nitrate to form a substance that appears to be NO_3F . The gas explodes on heating, it reacts with iodide ion and silver ion in the manner described above, and the boiling point agrees, qualitatively, with that given by Cady for NO_3F . Twenty cc. of the substance in the liquid state (at -79°) was prepared.

By allowing it to boil at atmospheric pressure for a time it was freed from any of the more volatile impurities. The vapor, at atmospheric pressure, was then passed through dilute nitric acid to remove any hydrolyzable impurities. It was then distilled from a trap cooled to -78° into a trap immersed in liquid air. The resulting material had, when molten, and unlike the material prepared from nitric acid, a very slightly yellowish color. Due to some other substances, or to a slow decomposition, an apparently constant pressure of from 5 to 10 mm. developed above the material cooled with liquid air, even after repeated pumping. The solid material, while under vacuum in the trap cooled with liquid air, eventually exploded very violently (danger!), and for no obvious reason.

CONTRIBUTION FROM
GATES CHEMICAL LAB.
CALIFORNIA INST. OF TECH.
PASADENA, CALIFORNIA

DON M. YOST
ALAN BEERBOWER

RECEIVED MARCH 25, 1935

1,2-BENZOPYRENE

Sir:

In preparing a quantity of 1,2-benzopyrene required in a study which is being carried out in collaboration with the Office of Cancer Investigations, U. S. Public Health Service, the synthesis of Cook and Hewett [*J. Chem. Soc.*, 398 (1933)] has been modified to advantage in certain details, the over-all yield from pyrene being 36%.

The condensation of pyrene (0.6 mole) with succinic anhydride (0.72 mole) in nitrobenzene solution (600 cc.) with aluminum chloride (190 g.) was carried out as described [THIS JOURNAL, 54, 4351 (1932)] for the similar condensation of acenaphthene. Sodium β -1-pyrenoylpropionate was collected after one crystallization and washed free of tars with alcohol and ether. The material was directly pure and suitable for reduction; yield 90–94%. The method of reduction of Cook and Hewett was not improved in yield (71–75%, pure), but the process was simplified. After acidifying the solution of γ -1-pyrenylbutyric acid, the suspension was heated at the boiling point for one hour to dehydrate the hydroxy acid and give a granular product. This was extracted with hot sodium bicarbonate solution, then with cold soda solution, and the combined filtrates were acidified and digested. The crude acid (m. p. 181–183°) was crystallized once from xylene (m. p. 187–188°).