

**N-Ethyltrifluoroacetanilide.**—Trifluoroacetyl chloride<sup>3</sup> was prepared; however, it was not collected but run directly into a pyridine solution of ethylaniline. The amine mixture was cooled in an ice-bath and stirred constantly. When the acyl chloride addition was complete, the reaction mixture was put on a steam-bath for one-half hour, cooled and poured into water. On acidification to litmus, an oily layer developed on the bottom of the container. An ether extract was washed three times with water, once with an aqueous sodium bicarbonate solution, and three times with water. The extract was dried over anhydrous sodium sulfate. On distillation a 48.8% yield of product was collected distilling at 80–82° (3 mm.);  $n_D^{20}$  1.4680;  $d_{20}^{20}$  1.2227;  $MR$  calcd., 48.29; found, 49.14. The liquid was redistilled and a colorless middle fraction taken for the infrared spectrogram. Nitrogen analyses were checked by Miss Zerwo of the University of Colorado.

*Anal.* Calcd. for  $C_{10}H_{10}F_3NO$ : N, 6.45. Found: N, 6.17, 6.22. Found: N, 6.47.

**Ethyl N-Isoamyl-N-phenylcarbamate.**—This compound was prepared by the method of Hartman and Brethen<sup>4</sup> for ethyl N-methylcarbamate. On distillation a 65.2% yield of a liquid was collected distilling at 130–134° (1.5 mm.);  $n_D^{20}$  1.4971;  $d_{20}^{20}$  0.9898;  $MR$  calcd., 69.28; found, 69.49. The liquid was redistilled and a yellow-colored middle fraction taken for the infrared spectrogram.

*Anal.* Calcd. for  $C_{14}H_{21}NO_2$ : N, 5.96. Found: N, 6.05, 5.96.

**Infrared Absorption Spectra.**—To resolve all the peaks of complete absorption cell thicknesses of 0.10 and 0.025 mm. were used as well as a 10% concentration of the compounds in *n*-heptane in the 0.025-mm. cell.

**Insecticide Investigation.**—The candidate insecticides dissolved in deobase (5% w./v.) were aspirated into a Lucite chamber which enclosed the roaches. After a 15-minute contact period, the roaches were removed to clean petri dishes and observed. The results are summarized in Table I.

TABLE I  
SUMMARY OF INSECTICIDE TESTING

Compound	Knock-down in 15 min., %	Kill in 24 hr., %	Comment
N-Ethyl- <i>p</i> -ethoxyacetanilide	0	0	
N-Ethyl- <i>o</i> -ethoxyacetanilide	40	100	Warm day
	0	0	Cool day
N-Ethylphenoxyacetanilide			Insoluble in deobase
N-Isoamylphenoxyacetanilide	0	60	
N-Ethyltrifluoroacetanilide	0	0	
Ethyl N,N-isoamyl phenylcarbamate	0	60	

(3) Henne, Alm and Smook, *THIS JOURNAL*, **70**, 1968 (1948).

(4) Hartman and Brethen, "Organic Syntheses," Coll. Vol. II, John Wiley and Sons, Inc., New York, N. Y., 1943, p. 278.

CHEMICAL FOUNDATION LABORATORY AND  
CHEMISTRY DEPARTMENT OF THE  
UNIVERSITY OF COLORADO  
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## Ion-Exchange Separation of Hafnium and Zirconium

BY I. E. NEWNHAM

In the course of an investigation on the properties of the cation exchange resin "Dowex 50" Street and Seaborg<sup>1</sup> separated 10 mg. of hafnium oxide from a mixture containing 35 mg. of  $ZrO_2$  and 15 mg. of  $HfO_2$ . In the light of their suggestion that gram samples could probably be handled effectively their method was applied to a 2-g. oxide mixture

(1) K. Street and G. T. Seaborg, *THIS JOURNAL*, **70**, 4268 (1948).

containing 20%  $HfO_2$ . This mixture had been prepared from a 30-g. sample of Australian zircon in the course of an investigation on suitable methods for concentration of the low hafnium content of this material.

"Dowex 50" of 100–200 mesh, kindly supplied by the Dow Chemical Company, was packed in a column 150 cm. high  $\times$  3.5 cm. diameter. In accordance with the technique suggested by Street and Seaborg the oxide mixture was converted to oxychloride crystals (2.8 g.) which were slowly added to 1200 cc. of 2 *M* perchloric acid containing 40 cc. of "Dowex 50." After 30 minutes the supernatant liquid was siphoned off and the resin slurry was added to the top of the exchange column. Elution with 6 *M* hydrochloric acid at the rate of 0.5 cc./min. followed. The hafnium content of successive fractions listed below in their order of collection indicates the possibilities of this separation method.

Fraction no.	Total $HfO_2$ recovered, %	$HfO_2$ content of fraction, %
1	42	99.9
2	18	90
3	10	75
4	10	52
5	10	34

DIVISION OF INDUSTRIAL CHEMISTRY  
COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH  
ORGANIZATION  
MELBOURNE, AUSTRALIA

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## Quinoxaline Studies. III. The Preparation and Physical Properties of Some 2,3-Dialkoxyquinoxalines

BY ROBERT PATTON<sup>1</sup> AND HARRY P. SCHULTZ

A series of 2,3-dialkoxyquinoxalines have been prepared and their physical properties have been determined. Only two 2,3-dialkoxyquinolines have previously been reported.<sup>2</sup>

The 2,3-dialkoxyquinoxalines were prepared by the reaction of 2,3-dichloroquinoxaline with sodium, or preferably potassium, alkoxides.

Table I lists the physical properties of the compounds. As is also shown in Table I, all compounds of this series show similar ultraviolet absorption characteristics: a maximum absorption at 241 and 312 millimicrons, a small plateau between 300–302  $m\mu$ .

### Experimental Procedures

**2,3-Dichloroquinoxaline.**—This material was prepared from 2,3-dihydroxyquinoxaline<sup>3</sup> according to the procedure of Hinsberg and Pollak.<sup>4</sup>

**2,3-Dialkoxyquinoxalines, General Procedure.**—Two grams (0.01 mole) of 2,3-dichloroquinoxaline was added to a solution of 0.78 g. (0.02 mole) of potassium dissolved in 10 ml. of the requisite dry alcohol. The solution was stirred and heated on a steam-bath until neutral to moist pH paper.

(1) Abstracted from a thesis by Robert Patton, presented to the Graduate Faculty of the University of Miami, in partial fulfillment of the requirements for the degree of Master of Science in chemistry, June, 1951.

(2) (a) J. Stevens, K. Pfister, III, and F. Wolf, *THIS JOURNAL*, **68**, 1035 (1946); (b) A. Gowenlock, G. Newbold and F. Spring, *J. Chem. Soc.*, 622 (1945).

(3) R. Meyer and A. Seelinger, *Ber.*, **29**, 2641 (1896).

(4) O. Hinsberg and J. Pollak, *ibid.*, **29**, 784 (1896).