from these data that gluconate is oxidized by the translucent spores. There are two known ways by which glucose is oxidized to gluconic acid. One is the Warburg-Dickens-Lipmann-Horecker pathway (5) and the other is the Stokes-Campbell route (5). The former requires the presence of phosphoric acid, but the latter does not. We found that glucose and gluconate oxidation proceeded in veronal buffer, which is phosphate-free, as efficiently as in phophate buffer. This would eliminate the Warburg-Dickens-Lipmann-Horecker pathway.

It is interesting to note that vegetative forms of this species have the cytochrome system and other oxidative enzymes (6), whereas the germinating spores can oxidize glucose or gluconate only in a phosphate-free buffer.

From this fact it may be assumed that the glucose oxidative enzyme system of B. subtilis (PCI 219) may develop in a stepwise manner as germination proceeds and as the cells change into the vegetative form.

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#### **References and Notes**

- 1. B. D. Church and H. Halvorson, Bacteriol.
- Proc. (1955), p. 41. W. A. Hardwick and J. W. Foster, J. Bacteriol. 2.
- W. A. Hardwick and J. W. Foster, J. Batteriol. 65, 355 (1952).
  Y. Hachisuka et al., ibid. 69, 399 (1955).
  Y. Hachisuka et al., ibid. 69, 407 (1955).
  C. Lamanna and M. F. Mallette, Basic Bac-teriology (Williams and Wilkins, Baltimore, Md., 1953), p. 545.
  H. Stephenson Bacterial Metabolism (Long-5.
- H. Stephenson, Bacterial Metabolism (Long-mans, Green, New York, ed. 3, 1950), p. 24. Present address: University of Texas M. D. Anderson Hospital and Tumor Institute, Texas Medical Center, Houston.
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### **Rapid Ion-Exchange Method** for Determining Total Fluoride in Impure KF · HF

As a result of the continuing need for large numbers of control analyses on electrolyte for fluorine generation cells at this plant, it became necessary to develop a rapid method of analyzing potassium acid fluoride (KF · HF) for total fluoride content (1). Accuracy comparable to that of the photofluorimetric method (2), which has been in use, was required. Consequently, an ion-exchange technique was investigated, and a procedure was developed, modifying the general methods described by Honda (3) and Roper et al. (4), in which the cations are removed from the solution, and the fluoride-containing eluant is titrated with NaOH.

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change resin (Rohm and Haas Amberlite IR-120) supported on 100-mesh nickel screen is used in the simple apparatus shown in Fig. 1. The resin is first activated by washing with 1 lit of 2-percent H<sub>2</sub>SO<sub>4</sub> at 30ml/min., the acid that remains in the tube being washed out with distilled water. The weighed electrolyte sample (impure  $KF \cdot HF$ ) is dissolved in distilled water, and an aliquot of about 10 ml is passed through the ionexchange column into a 400-ml polyethylene beaker. The column is then washed with distilled water until there is about 275 ml of eluate. This solution is then titrated to a phenolphthalein endpoint with NaOH solution prepared from CO<sub>2</sub>-free NaOH and standardized at approximately 0.1N with NaF.

Samples of known amounts of reagentgrade NaF which were dried at 110°C for 24 hours were used to check the method prior to applying it to samples of KF · HF electrolyte. Good results, which are shown in Table 1, were obtained by using the procedure outlined here. As a further check, this ion-exchange procedure was directly compared against the photofluorimetric technique previously used by analyzing parts of the same electrolyte sample (impure  $KF \cdot HF$ ) by both methods. The portion for photofluorimetric analysis was steam-distilled and titrated with thorium nitrate in a modified Klett fluorimeter, using morin as the indicator; the portion for ion-exchange analysis was handled as described in preceding paragraphs. Results, in terms of percentage of fluoride found, are shown in Table 2.

The values obtained, together with data subsequently obtained in routine use of this procedure, show that it yields results of accuracy comparable to that obtained by the photofluorimetric method in about one-fourth of the previously required time. In general, the titration is correct to 1 or 2 drops with phenolphthalein as the indicator. Simple checks showed that accuracy was not affected by varying the rate of flow of sample and wash water in the column between 5 and 30 ml per minute or by varying the depth of resin in the column from 6 in. to 12 in. It was



Fig. 1. Ion-exchange column.

Table 1. Results of analyses of samples of known fluoride content.

Sample	Fluoride present (mg)	Fluoride found (mg)
1	63.34	63.67
2	55.87	55.87
3	57.23	57.43
4	58.54	58.53
5	55.69	55.61
6	50.80	50.80
7	47.55	47.54
8	47.14	47.08
Avg.	54.52	54.57

Table 2. Comparison of percentage of fluoride found by ion-exchange and photofluorimetric methods in impure KF · HF samples.

Sample	Photo- fluorimetric method	Ion- exchange method
1	58.81	58.73
2	58.85	58.72
3	58.80	58.77
4	58.86	58.80
Avg.	58.83	58.76

found, however, that the higher rates of flow with a 6-in. resin bed gave slightly erratic results. A single 12-in. bed has been used intermittently for 3 weeks (approximately 50 determinations) without regeneration. Since regeneration is easily accomplished with 2-percent  $H_2SO_4$ , the resin may be used indefinitely.

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#### **References** and Notes

- 1. This work was performed under contract AT-(33-2)-1 with the U.S. Atomic Energy Commission. H. H. Willard and C. A. Horton, Anal. Chem.
- 2.
- A. H. Willard and C. A. Horton, Anal. Chem. 22, 1190 (1950).
   M. Honda, J. Chem. Soc. Japan 71, 59 (1950).
   E. C. Roper and E. B. R. Prideaux, J. Soc. Chem. Ind. (London) 45, 109 (1926).
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## **Ratio of Ionium to Uranium** in Coral Limestone

Samples of coral limestone cuttings taken over the first 200 feet in drilling on Elugelab Island during the summer of 1952 (1) have been analyzed for uranium by the sodium fluoride fluorometric method and for ionium by a radiochemical procedure which involves carrier-free separation of the total thorium in the

Table 1. Experimental results. The sample numbers indicate the depth range in feet over which the sample was collected. The numbers in column 4 were calculated from the ionium and uranium contents of the samples, taking the values  $8.0 \times 10^4$  years and  $4.49 \times 10^{\circ}$  years as the half-lives of ionium and uranium, respectively. The uncertainties indicated are the expected standard deviations, based on the number of counts taken and on the estimate that the standard deviation in the uranium determination is  $\pm 5$  percent.

Sample No.	Uranium	Ionium	$N_{Io}\lambda_{Io}$
Sample No.	(ppm)	$(10^{-5}  \mathrm{ppm})$	$\overline{N_{\mathtt{U}_{\mathbf{I}}}\lambda_{\mathtt{U}_{\mathbf{I}}}}$
Y*† (surface)	0.83	< 0.1	< 0.07
		< 0.1	< 0.07
F <sub>1</sub> 20-45	3.24, 3.14, 2.95 2.93, 3.00	< 0.2	< 0.04
F <sub>1</sub> 45-55	3.73, 3.74	« 0.2	« 0.03
	3.64, 3.56		
F <sub>1</sub> 55–60	4.68, 4.91	« 0.1	« 0.01
		« 0.1	« 0.01
F <sub>1</sub> 60–70	5.22, 4.91	$1.36 (\pm 0.05)$	$0.16 (\pm 0.01)$
		$1.48 (\pm 0.09)$	$0.17 (\pm 0.01)$
F1 70-80†	5.52, 5.18, 5.35	$2.5 (\pm 0.2)$	$0.27 (\pm 0.03)$
F1 80-90	4.41, 4.59	$3.8 (\pm 0.1)$	$0.49(\pm 0.03)$
F1 90-100	4.07, 4.20	$3.5(\pm 0.2)$	$0.49(\pm 0.04)$
F <sub>1</sub> 100-110	3.53, 3.64	$4.3 (\pm 0.2)$	$0.70(\pm 0.05)$
		$4.3 (\pm 0.2)$	$0.70(\pm 0.05)$
F1 110-120†	4.08	$2.6(\pm 0.1)$	$0.37(\pm 0.03)$
		2.7 $(\pm 0.1)$	$0.38(\pm 0.03)$
		2.8 $(\pm 0.1)$	$0.40(\pm 0.03)$
F <sub>1</sub> 120-130†	4.10	$3.1 (\pm 0.2)$	$0.44(\pm 0.04)$
F <sub>1</sub> 130–140†	4.04, 4.04	2.9 $(\pm 0.1)$	$0.42(\pm 0.03)$
F <sub>1</sub> 140–150	3.72	2.7 $(\pm 0.1)$	$0.42(\pm 0.03)$
		2.6 $(\pm 0.1)$	$0.41 (\pm 0.03)$
F <sub>1</sub> 150-160 <sup>†</sup>	3.12	3.4 (± 0.2)	$0.63(\pm 0.05)$
		$3.5(\pm 0.2)$	$0.65(\pm 0.05)$
F <sub>1</sub> 160–170†	4.00	$4.38(\pm 0.05)$	$0.64(\pm 0.03)$
		$4.29 (\pm 0.05)$	$0.62 (\pm 0.03)$
F <sub>1</sub> 170-180	3.08	$3.7 (\pm 0.1)$	$0.70 (\pm 0.04)$
F <sub>1</sub> 180-190	2.90	$4.3 (\pm 0.2)$	$0.86 (\pm 0.06)$
		$4.0 (\pm 0.2)$	$0.80 (\pm 0.06)$
		4.4 $(\pm 0.1)$	$0.88(\pm 0.06)$
		$4.4 (\pm 0.1)$	$0.88 (\pm 0.06)$

\* Sample Y was a 360-g cluster of coral collected near the surface on Yurochi Island (Bikini). The entire piece was dissolved. Aliquots of the solution were analyzed. † Samples in which ionium and uranium were determined by analysis of aliquots of the same solution.

In other samples, separate portions of ground and well-mixed solid sample were used for ionium and uranium analysis.

sample by thenoyltrifluoroacetone extraction and ion-exchange techniques, followed by pulse analysis of the alpha activity in the separated thorium (2, 3).

The ionium content of the coral was found to vary with the depth in a fairly simple manner. Near the surface, the coral contains less than  $2 \times 10^{-6}$  ppm of ionium. With increasing depth, the ionium content first increases, reaches a value of  $4 \times 10^{-5}$  ppm at 100 feet, decreases sharply to about  $2 \times 10^{-5}$  ppm, then increases again, reaching a value of  $4 \times 10^{-5}$  ppm at 160 feet (Table 1).

The uranium content of the Elugelab coral also varies somewhat, but within much narrower limits-2.9 to 5.5 ppm.

On examination of the data obtained in the analysis of 16 samples over the depth range of 20 to 190 feet, it becomes apparent that coral near the surface contains far less than the equilibrium quantity of ionium. In a sample collected at 60 to 70 feet, for example, one calculates -on the basis of the uranium content $8.7 \times 10^{-5}$  ppm of ionium for secular equilibrium. The ionium found,  $1.4 \times 10^{-5}$ ppm, is only 16 percent of the equilibrium quantity. In a sample still nearer the surface, 20 to 45 feet, there is less than 4 percent of the amount of ionium required for equilibrium. At a depth of 100 to 110 feet, however, the coral contains approximately 70 percent of the equilibrium quantity of ionium calculated on the basis of the known uranium content.

The data obtained thus far suggest that, in the absence of leaching or other processes which would lead to differential movement of uranium and ionium in the coral deposit, the magnitude of the ionium-uranium ratio in a particular specimen may indicate the age of the specimen. This statement is based on the assumption that newly formed corals contain uranium but are essentially free of ionium.

In addition to the samples reported here, ten cutting samples from the Parry

Island drilling (1) have been analyzed. It is interesting to note that among the coral samples analyzed to date none has been found which contains ionium in excess of the amount required for secular equilibrium with the uranium present. If separation of uranium from ionium occurs in the coral, one may expect to encounter such samples.

Core samples taken over the entire depth range in the Eniwetok drillings, live corals, and other calcareous marine deposits are now being analyzed.

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### References and Notes

H. S. Ladd et al., Bull. Am. Assoc. Petroleum Geol. 37, No. 10 (1953).
 This work was supported by U.S. Atomic Energy Commission contract W-7405-Eng. 36.
 J. W. Barnes, E. J. Lang, H. A. Potratz, "U.S. Atomic Forence Commission Pencet I A 1945."

Atomic Energy Commission Report LA-1845." Academic address: Department of Chemistry,

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# **Flotational Lipoproteins Extracted** from Human Atherosclerotic Aortas

In view of the interest in the relation between the lipoproteins in serum and atherosclerotic activity (1, 2), an examination of human aortas for the presence of similar lipoprotein fractions seemed mandatory. Experiments with rabbits that were force-fed cholesterol have shown a remarkable correlation between atherosclerotic lesions and the Sr 10-30 class of lipoproteins in the serum (1). In human serum any relationships are complicated by diverse, normal classes of lipoproteins, so that interpretation becomes highly statistical (3). Also, plaques cannot be evaluated because of the obvious inaccessibility of the tissues.

Accordingly, we have endeavored to extract fractions from aortas of fresh human necropsies similar to those observed in serum and to assess them quantitatively by ultracentrifugal flotation (4). These findings are compared with the degree of atherosclerosis of each extracted aorta and with the cause of death where it was classified as cardiovascular or noncardiovascular.

Aortas from both sexes were obtained from a 500-bed general hospital. Autopsies were performed 1 to 8 hours after death. Three inches of thoracic aorta just above the diaphragm were taken. They were placed in 0.9-percent NaCl and maintained at 4°C for less than 1 week.

The cause of death was determined