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Complex Polyphosphates
in Neutron - Irradiated Phosphates (1)

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Progress Report

Since the first work done in this area of phosphorus oxy-anion hot-atom chemistry(2) some serious doubts have been raised as to the validity of the conclusions. Originally, paper chromatographic separation of neutron - irradiated phosphorus oxy-anions led to the conclusion, based on the appearance of a slowly-moving broad peak, that high-molecular weight polyphosphates were formed. The evidence was strengthened by an activity peak eluted only with strong acid in an anion exchange separation of the same material. It was concluded here that the activity was due to polyphosphates containing $> 14-15$ phosphorus atoms. The literature provided the quantitative estimation of the number of phosphorus atoms from experiments analyzing Graham's Salt on the same type of chromatographic material.

It seemed judicious to try still a third method of analysis since the presence or absence of polyphosphates is an important aspect of the over-all view of hot-atom chemistry in solids. A porous polyacrylamide gel (Bio-gel P available from Bio-Rad Laboratories, Richmond, California) was chosen because it behaves like a molecular sieve in separating substances in order of decreasing molecular mass and is non-reactive. Two mesh sizes were used, 100-200 mesh, and < 400 mesh in a column measuring 1 inch in diameter and

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containing 53 ml. of gel. The calibration of void volume used neutron - irradiated Graham's Salt and resulted in a value of, 35%, in agreement with the manufacture's prediction of 38%.

A series of neutron - irradiated $\text{NH}_4\text{H}_2\text{PO}_4$, $\text{NH}_4\text{H}_2\text{PO}_3$, and $\text{NH}_4\text{H}_2\text{PO}_2$ salts were run on the column with the 100-200 mesh gel giving 2, 3, and 3 peaks respectively. The average molecular mass corresponding to the first peak was approximately 400, 400, and 700 respectively. (A polyphosphate with 14 phosphorous atoms would have a molecular mass of approximately 900.)

With the finer mesh material and the same salts, we seem to be getting lower molecular masses. These results are clearly contrary to those previous reported by one of us (V.C.A.). In continuing with this study we are repeating the experiments on the finer-mesh gel in the hopes of getting better definition and resolution. Also we will separate neutron - irradiated $^{15}\text{NH}_4\text{H}_2\text{PO}_4$ in the hopes of detecting any effect due to the $^{14}\text{N}(\text{n},\text{p})^{14}\text{C}$ reaction. Also we are continuing our studies on the anion-exchange column to test column parameters as a function of the concentration of the anomalous activity.

There is no doubt that this activity exists and that it appears only in neutron - irradiated phosphates, e.g., it is not found to any appreciable degree in commercially available $\text{H}_3^{32}\text{PO}_4$, or at least it is easily removed from this source. Our present thinking is that this anomalous activity is due to some metastable forms of polyphosphates which are stronger acids than the stable polyphosphates and so bond more strongly to the anion-exchange resin. But these species are not bound by the gel and slightly affected by the paper in paper chromatography.

no

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at 1000 ft. 1000 ft. 1000 ft.

References

- (1) Work performed under Contract (AT-1) 1618 with the United States Atomic Energy Commission.
- (2)
 - a) V.C. Anselmo, Ph.D. Thesis, University of Kansas, 1961.
 - b) V.C. Anselmo and Ernest Sanchez, A.E.C. Report C00-1618-2.
 - c) V.C. Anselmo, Radiochim. Acta 4, 203 (1965).

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1. The first part of the report is a general introduction to the subject of the study. It discusses the importance of the study and the objectives of the research.