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Enrichment of Tin Activity Through the Szilard-Chalmers Separation

Herta W. Spano

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ENRICHMENT OF TIN ACTIVITY THROUGH THE SZILARD-CHALMERS
SEPARATION

By

Herta W. Spano

A Thesis

In Partial Fulfillment of the
Requirements for the Degree of
Master of Science in Chemistry

The University of New Mexico
1951

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MASTER OF SCIENCE

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This thesis directed and approved by the Graduate Council
has been accepted by the Graduate Council of the
University of New Mexico in partial fulfillment of the requirements
for the degree of

MASTER OF SCIENCE

IN THE
DEPARTMENT OF
BIOLOGY

BY
WILLIAM C. COTTELL

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The author wishes to express her appreciation to Dr. Milton Kahn for his aid and encouragement, and to the Los Alamos Scientific Laboratory for performing the irradiations.

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EXERCISE BOOK

EFFICIENCY

CHAPTER I

INTRODUCTION

General Discussion of the Szilard-Chalmers Reaction

The (n, γ) reaction is the nuclear reaction most commonly used for the preparation of radioactive isotopes. When an element is bombarded with slow neutrons and neutron capture takes place, the compound nucleus thus formed has gained energy equivalent to the kinetic energy and to the binding energy of the incident neutron. The compound nucleus then goes to a more stable state by the emission of some of its excess energy. When the bombarding particles are slow neutrons, the release of this excess energy takes place through gamma ray emission. The isotope thus formed, i.e. the product nucleus, may itself be radioactive.

When working with radioactive isotopes in the laboratory it is often desirable to have a preparation of high specific activity. By definition, the specific activity of an element is the ratio of the number of radioactive atoms to the total number of atoms of the element in the sample. The specific activity of the sample should be high in order to minimize the effects of isotopic dilution when the sample is used as a tracer and must undergo several fractionations.

Since the radioactive isotope produced by slow neutron bombardment is isotopic with the target material, the two cannot be

 

separated by ordinary chemical means, and consequently, the specific activity of the sample will be low. However, it has been found that a separation can be affected in some instances by utilizing the recoil energy resulting from the emission of the gamma ray.

This principle was first applied in 1934 by Szilard and Chalmers¹ when they found that, after irradiating ethyl iodide with slow neutrons, they were able to extract most of the iodine activity with water. They used a small amount of iodine carrier, reduced it to iodide, and then precipitated silver iodide. Apparently the carbon-iodine bond had been broken when I^{127} was transformed to I^{128} by capture of a slow neutron. This type of process has been used since then to concentrate the various isotopes formed by the (n, γ) reaction and has become known as the Szilard-Chalmers process.

In order that the Szilard-Chalmers reaction may be successfully applied, several conditions must be met:

1. The recoil energy component in the direction of the bond must be large enough to break the atom away from the rest of the molecule.

Since the bombarding particles are slow neutrons, their capture alone does not provide sufficient energy to break the bond. However, capture is followed by the emission of one or more gamma rays, and during this process the nucleus receives some recoil energy. Since momentum must be conserved, the momentum of the emitted gamma

reported by ordinary chemical means, and consequently, the chemical activity of the sample will be low. However, it has been found that a separation can be effected in some instances by utilizing the recoil energy resulting from the emission of the gamma ray.

This principle was first applied in 1934 by G. B. B. and G. B. B. when they found that a few percent of the initial activity of a sample, after separation, was found to be in the recoil product. They used a small amount of thorium dioxide, ThO_2 , as a source of alpha particles, and they placed a thin layer of thorium dioxide on a carbon-dioxide film and then exposed it to the alpha particles. The recoil energy of the alpha particles was used to separate the various isotopes formed by the decay of the thorium and was found to be at the initial-thorium product.

In order that the recoil-energy separation may be successfully applied, several conditions must be met:

1. The recoil energy component in the chemical bond must be large enough to break the bond away from the rest of the molecule.

Since the bonding particles are also particles, they capture alpha dose and provide nuclear energy to break the bond. However, capture is followed by the emission of one or more gamma rays, and during this process the nucleus receives some recoil energy. Since momentum must be conserved, the amount of the recoil energy

ray must equal the momentum of the recoil atom. From this condition it can be shown that if the recoil energy imparted to the product nucleus is E_r , then

$$E_r = \frac{E_\gamma^2}{1862 M} \text{ Mev}$$

where E is the energy of the emitted gamma ray in Mev and M is the mass of the product atom in atomic mass units. For the hypothetical case where E is 6 Mev and M is 100, E_r has a value of 193 ev. Since chemical bond energies are of the order of 1 to 5 ev, it is highly probable that the bond will be ruptured.

2. The atom and the remaining fragment of the molecule must not recombine.

3. Any exchange between ruptured active atoms and inactive atoms in the target molecule must be slow.

4. A chemical separation of the active atoms from the inactive atoms in the target molecule must be possible.

If the above conditions are satisfied, it may be feasible to separate the radioactive isotope produced by an (n, γ) reaction from its isotopic target, and thereby substantially increase the specific activity of the radioactive isotope.

Scope of the Investigation

It was the purpose of this investigation to utilize the Szilard-Chalmers reaction in order to prepare tin solutions of high specific activity.

Tetraphenyl tin was chosen for the target compound. It is a white, crystalline solid of excellent thermal stability. No data have been reported in the literature on the rate of exchange between inorganic forms of tin and tetraphenyl tin but it seemed highly probable that this exchange would be very slow. Tetraphenyl tin is soluble in various organic solvents but insoluble in water, and this suggested solvent extraction as a possible means of separation of the activated tin recoil atoms from the inactive tin in the target molecule.

Benzene was used as the organic solvent for the tetraphenyl tin. At room temperature, it dissolves more tetraphenyl tin per 100 grams of solvent than any of the other common organic solvents² and its volatility is not so high that it would be inconvenient to work with.

Several aqueous solvents, namely 3f hydrochloric acid, 6f hydrochloric acid, 1f sodium hydroxide, and distilled water, were used to extract the active tin from solutions of tetraphenyl tin in benzene.

It was the purpose of this investigation to determine the relative solubility of the various salts of the various acids in water and to determine the relative solubility of the various salts of the various acids in water.

For this purpose, the various salts of the various acids were dissolved in water and the relative solubility of the various salts of the various acids in water was determined.

EXPERIMENTAL

The various salts of the various acids were dissolved in water and the relative solubility of the various salts of the various acids in water was determined. The results of the investigation are given in the following table.

It was found that the relative solubility of the various salts of the various acids in water is as follows: The relative solubility of the various salts of the various acids in water is as follows: The relative solubility of the various salts of the various acids in water is as follows:

The relative solubility of the various salts of the various acids in water is as follows: The relative solubility of the various salts of the various acids in water is as follows: The relative solubility of the various salts of the various acids in water is as follows:

With the exception of distilled water, the above mentioned aqueous solutions extracted approximately fifty per cent of the total tin activity. The specific activity of the extracted tin was found to be about three thousand times greater than the specific activity of the irradiated tetraphenyl tin before extraction.

With the exception of distilled water, the above mentioned
specimen relations extended approximately 15% per cent of the total
the activity. The specific activity of the extracted tin was found
to be about three thousand times greater than the specific activity
of the irradiated background tin before extraction.

CONFIDENTIAL
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CHAPTER II

EXPERIMENTAL

Chemicals

Tetraphenyl tin and phenylarsonic acid were obtained from the Eastman Kodak Company and used without further purification. The phenylarsonic acid was used in the form of a saturated solution containing about 3.3 grams per 100 milliliters of water.

All other reagents used were of c.p. grade.

Radioactivity

When tetraphenyl tin is irradiated with slow neutrons, the radioactive products formed are all the result of neutron capture by the tin. Naturally occurring tin is a mixture of several isotopes. These isotopes are shown in Table I, together with their relative abundances, their cross sections for thermal neutron capture³ and their respective product isotopes.

TABLE I

Products of Slow Neutron Capture by Tin

Stable isotope, mass number	Abundance, per cent	Thermal neutron cross section, $\text{cm}^2 \times 10^{24}$	Product isotope, mass number
112	0.90	1.1	113
114	0.61	unknown	-
115	0.35	"	-
116	14.07	"	-
117	7.54	"	-
118	23.98	"	-
119	8.62	"	-
120	33.03	0.25	121
122	4.78	0.16	123
124	6.11	0.57	125

The four product isotopes shown in Table I are all radioactive, and consequently they all contribute to the decay observed in the sample after irradiation. The activity associated with each isotope at the end of irradiation can be calculated from the following equation:

$$dn/dt = (N)(\sigma)(I)(1 - e^{-\lambda t})$$

where dn/dt = the disintegration rate

N = the number of atoms of the isotope in question

σ = the thermal neutron cross section

I = the neutron flux

λ = the decay constant of the isotope

t = the time of irradiation.

Table II lists the active isotopes that would be found in a 36 milligram sample of tetraphenyl tin (containing 10 milligrams of tin), and shows

Isotopes of the Neutron Group by the

Sample Isotope, mass number	Abundance, per cent	Thermal neutron cross section, cm ² /atom	Product Isotope, mass number
112	0.90	1.1	112
113	0.01	maximum	-
114	0.35	"	-
115	14.07	"	-
116	7.54	"	-
117	22.84	"	-
118	8.02	"	-
119	23.03	0.25	120
120	1.18	0.16	119
121	0.11	0.27	121

The four product isotopes shown in Table I are all radio-
active, and consequently they all contribute to the decay observed
in the sample after irradiation. The activity measured with each
isotope at the end of irradiation can be calculated from the following
equation:

$$A_{\text{prod}} = (N)(\sigma)(I_0 - A_{\text{prod}})$$

where A_{prod} = the disintegration rate

N = the number of atoms of the isotope in question

σ = the thermal neutron cross section

I_0 = the neutron flux

λ = the decay constant of the isotope

t = the time of irradiation.

Table II lists the active isotopes that would be found in a 30 milligram
sample of tellurium (containing 10 milligrams of tin), and gives

their respective modes of decay, half-lives, and disintegration rates in disintegrations per minute following a two-hour irradiation in a flux of 5×10^{10} neutrons/cm²/second.

TABLE II

Activities Present in a 36 mg Sample of Tetraphenyl Tin
After a Two Hour Irradiation in a Neutron Beam of 5×10^{10}
Neutrons/cm²/second

Product Isotope	Type of decay ^{1,5}	Half-life	Disintegrations/minute
113	α , In- α α^- 0.055, 0.080 γ 0.085	105 days	7.3
121	β^- 0.35 No gamma	1.1 days	5.1×10^3
123	β^- 1.3 No gamma	130 days	4.2
125	β^- 1.3	10 min.	8.4×10^4

It can be seen from Table II that the activity due to tin¹¹³ and tin¹²³ is negligible compared to that of tin¹²¹. Tin¹²⁵ decays with a ten-minute half-life to antimony¹²⁵, which in turn is a beta emitter having a 2.7-year half-life. Therefore, at the end of a two-hour irradiation practically all tin¹²⁵ will have decayed. The activity due to the daughter, antimony¹²⁵, would be only 2.5 disintegrations per minute. This is again negligible in comparison to the tin¹²¹ activity.

In all experiments the radioactivity detected was due primarily to tin¹²¹. Figure I shows the observed decay of a 36 milligram sample of tetraphenyl tin versus time in days. The decay follows a straight line for four days after irradiation, and it was during this time that all experiments were carried out. Thereafter, the longer lived activity becomes more pronounced. When the longer lived activity, as represented by the tail of the curve, is subtracted from the original curve, a half-life of 1.1 days, corresponding to tin¹²¹, is found. The graph also shows a ratio of tin¹²¹ to tin¹²³ activity which is smaller than that indicated by Table II, but this is to be expected in view of the relative strengths of the betas emitted. The beta from tin¹²¹ has a maximum energy approximately only one-fourth that of the beta from tin¹²³, and consequently, because of greater absorption, a relatively smaller amount of the tin¹²¹ beta would reach the counter compared to that from tin¹²³.

Although some of the activity in the sample is due to antimony which would not necessarily follow the tin activity during the experiments, its effects can be considered negligible. First, the sample was used only as long as the observed rate of decay corresponded to tin¹²¹. Second, a check was made of the decay rate of the various fractions after a Szilard-Chalmers separation had been carried out, and all the fractions decayed with the same rate as is shown in Figure II. Third, a chemical separation⁶ was carried out to isolate the antimony activity from the tin activity, and the antimony activity

In all experiments the radioactivity detected was due
 entirely to ^{131}I . Figure 1 shows the observed decay of a 30 mCi-
 gram sample of technetium in various time in days. The decay follows
 a straight line for four days after irradiation, and it is during
 this time that all experiments were carried out. Thereafter, the
 longer lived activity becomes more pronounced. When the longer lived
 activity, as represented by the tail of the curve, is subtracted from
 the original curve, a half-life of 1.1 days, corresponding to ^{131}I ,
 is found. The graph also shows a ratio of ^{131}I to ^{132}I activity
 which in earlier work has been indicated by Table II, and this is to be
 expected in view of the relative strengths of the beta radiations. The
 data from this ^{131}I has a maximum energy approximately only one-fourth
 that of the beta from ^{132}I , and consequently, because of greater
 absorption, a relatively smaller amount of the ^{131}I could reach
 the counter compared to that from ^{132}I .

Although none of the activity in the sample is due to antimony
 which would not necessarily follow the tin activity during the experi-
 ments, the effects can be considered negligible. First, the sample
 was used only as long as the observed rate of decay corresponded to
 the ^{131}I . Second, a check was made of the decay rate of the various
 fractions after a 24-hour-24-hour separation had been carried out,
 and all the fractions decayed with the same rate as in other runs.
 Figure II. Third, a chemical separation⁶ was carried out to separate
 the antimony activity from the tin activity, and the antimony activity

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FIGURE 1

DECAY CURVE

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FIGURE II

was found to contribute only 8 counts per minute to the total activity of a 36 milligram sample of tetraphenyl tin. Due to the 2.7-year half-life of antimony-¹²⁵, its activity remains constant throughout the experiments. Since the lowest counting rate observed was 600 counts per minute, (with the exception of one of 200 counts per minute), the maximum contribution of 8 counts per minute due to the antimony would represent at most only 1.3 per cent of the total detectable activity and can therefore be neglected.

Tin¹²¹ being a beta emitter, the activity in the various samples must be determined by beta counting. This requires that samples be prepared with great care so that physical reproducibility is obtained. Otherwise, the counting rate of each sample would have to be corrected for geometry, self-absorption, and back-scattering.

In order to obtain all samples with uniform weight, thickness, and geometry, the tin was precipitated as stannic phenylarsonate.⁷ In order to precipitate the tin in tetraphenyl tin as the phenylarsonate, the organic matter had to be destroyed. The procedure is outlined below, and shown in abbreviated form in Figure III. Tetraphenyl tin was ashed with a mixture of concentrated sulfuric acid and 30% hydrogen peroxide until all organic matter was destroyed and the tin obtained in the stannic state. The sample was then heated until sulfur trioxide fumes appeared in order to remove any remaining traces of hydrogen peroxide. After cooling and diluting

FIGURE III

Preparation of Samples for Counting

PLATE III

Illustration of samples for counting

(a) (b) (c)

100 mg. of sample

20

Sample, 100 mg. of sample
and 100 mg. of sample

(d) (e) (f)

100 mg. of sample

100 mg. of sample

100 mg. of sample

(g) (h) (i)

the sample, stannic hydroxide was precipitated by the addition of concentrated ammonium hydroxide to the methyl red end-point. The sample was then centrifuged, the supernatant liquid removed, and the stannic hydroxide dissolved in the minimum amount of 6*f* hydrochloric acid. One-half milliliter of 6*f* hydrochloric acid was added in excess, and the sample made up to a volume of five milliliters. The sample was warmed in a steambath for a few minutes, and then five milliliters of a saturated solution of phenylarsonic acid was added to precipitate the tin phenylarsonate. After a digestion period of ten minutes in the steambath, the sample was cooled and allowed to stand another fifteen minutes. It was then centrifuged, the tin phenylarsonate was washed with five milliliters of 95% ethanol, and the wash liquid discarded. Next, the precipitate was suspended in 4 milliliters of 95% ethanol, and the resulting slurry was filtered on a tared, 24 mm diameter, Whatman #50 filter paper. The precipitate was dried for thirty minutes at 110° C, weighed, and mounted on a 2x2½ inch cardboard with Scotch tape.

Starting initially with aliquots containing 10 milligrams of tin, it was found on the basis of six runs that the recovery of tin in the final precipitate was 100.6 ± 1.3%. Thus under the conditions of precipitation used tin was precipitated quantitatively as stannic phenylarsonate. When samples were not of uniform weight, absorption corrections were applied amounting to a 1.15% increase in counts per minute for each milligram increase in sample weight.⁷

The sample, after being dried, was precipitated by the addition of concentrated ammonia hydroxide to the boiling red end-point. The sample was then centrifuged, the supernatant liquid removed, and the ammonia dissolved in the solution amount of 0.1 percent of the total. One-half milliliter of 0.1 percent ammonia solution was added, and the sample made up to a volume of 100 milliliters. The sample was warmed in a waterbath for a few minutes, and then five milliliters of a saturated solution of potassium acid was added to precipitate the tin phosphates. After a digestion period of ten minutes in the waterbath, the sample was cooled and allowed to stand overnight fifteen minutes. It was then centrifuged, the tin phosphates washed with five milliliters of 95% ethanol, and the wash liquid discarded. Next, the precipitate was suspended in 10 milliliters of 95% ethanol, and the resulting slurry was filtered on a filter, the filtrate, washed with 95% ethanol. The precipitate was dried for thirty minutes at 110°C, weighed, and counted on a 2000 gram scale board with Geiger tube.

Starting initially with aliquots containing 10 milligrams

of tin, it was found on the basis of six runs that the recovery of tin in the final precipitate was 100.0 ± 1.5%. Thus under the conditions of precipitation used tin was precipitated quantitatively as tin phosphates. When samples were not of uniform weight, count rate corrections were applied amounting to a 1.1% increase in counts per minute for each milligram increase in sample weight.

The sample was always mounted in the same position relative to the detection device, a Geiger-Müller end-window tube. The tube used was an Amperex #100C model, having a 3.5 mg/cm^2 mica window. The impulses were recorded by a Nuclear Instrument and Chemical Company Model #161 scaler, which was operated at 1340 volts. This was in the middle of a 75 volt plateau, having a rise of 1.5% in this range.

The counter was always checked before use with a uranium oxide standard which, under the conditions used, had a counting rate of 3550 counts per minute.

The observed counting rates were not corrected for coincidence. This correction was considered to be unnecessary on the basis of the decay curve shown in Figure I, which is linear up to 6000 counts per minutes.

Also, all samples from any one experiment were counted within the space of one hour, and therefore no corrections were applied for decay during the counting period.

Procedure for Szilard-Chalmers Separation

Eight hundred-milligram samples of tetraphenyl tin were irradiated by the Los Alamos Scientific Laboratory with slow neutrons for two hours. On receipt of the active sample, it was dissolved in

The sample was also prepared in the same manner relative to the detection device, a Gammex-4100 model, having a 3.5 inch active volume. The impurities were removed by a standard procedure and the Gammex model 4100 series, which was operated at 1500 volts, was in the middle of a 12 volt plateau, having a rise of 100 in this range.

The counter was always checked before and after a number of days standard which, using the standard used, had a constant rate of 350 counts per minute.

The observed counting rates were not corrected for coincidence. This correction was considered to be unnecessary on the basis of the decay curve shown in figure 1, which is fitted up to 6000 counts per minute.

Also, all samples from any one experiment were counted within the space of one day, and therefore no correction was applied for decay during the counting period.

Procedures for standardization of the detector were followed by the use of standard sources of known activity. The standard sources were of known activity and were counted for two hours. In the case of the standard source, it was placed in

100 milliliters of thiophene-free benzene, this step being carried out in a 150-milliliter beaker in all the runs but one. A five-milliliter aliquot was evaporated to dryness. The organic residue was treated as mentioned in the foregoing procedure, the tin being mounted for counting as tin phenylarsonate.

The remaining 95 milliliters of benzene solution was transferred to a separatory funnel. Twenty milliliters of aqueous solvent was added, and the mixture shaken for five minutes. The phases were allowed to separate, and the aqueous layer drawn off.

Another five-milliliter aliquot was removed from the remaining benzene phase and treated as described above in order to determine the amount of activity present in the extracted benzene phase.

The aqueous phase was washed with 50 milliliters of benzene, this benzene being discarded after separation. Five milliliters of the aqueous phase was removed, warmed slightly to remove any traces of benzene, and then treated with a drop of liquid bromine so that all the tin present would be in the stannic state. The excess bromine was removed by gentle heating, and one milliliter of a solution of stannic chloride containing 10 milligrams of tin per milliliter was added. The tin was then precipitated as stannic hydroxide, and the usual procedure followed to obtain the tin finally as tin phenylarsonate which was counted.

100 milliliters of benzene-free benzene, this was being carried out in a 150-milliliter flask in all the runs and was a 150-milliliter aliquot was evaporated to dryness. The organic residue was treated as mentioned in the preceding paragraph, the filtrate was mounted for counting as the hydrocarbon.

The remaining 50 milliliters of benzene solution was transferred to a separatory funnel. Twenty milliliters of aqueous sodium was added, and the mixture shaken for five minutes. The phases were allowed to separate, and the aqueous layer drawn off.

Another five-milliliter aliquot was removed from the remaining benzene phase and treated as described above in order to determine the amount of activity present in the organic phase.

The aqueous phase was washed with 50 milliliters of benzene, this benzene being drawn off after separation. The milliliters of the aqueous phase was removed, washed with benzene, and then treated with a drop of 10% bromine so that all the bromine would be in the aqueous phase. The excess bromine was removed by gentle heating, and one milliliter of a solution of stannous chloride containing 10 milliliters of 5% per milliliter was added. The flask was then gently shaken and hydroxide, and the usual procedure followed to obtain the final as the hydrocarbon which was counted.

Another portion of the aqueous phase was taken and analysed colorimetrically for small amounts of tin according to the method described in Snell and Snell.⁸ Two stock solutions were required for this procedure. Solution I was made by dissolving 5.3 grams of ammonium molybdate in 100 milliliters of water, adding 10 milliliters of concentrated sulfuric acid, and diluting to 200 milliliters. Solution II was made by fusing 5.9 grams $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ and 1.1 grams of powdered silicic acid in a platinum crucible for half an hour, dissolving the melt in water, and diluting to one liter. An aliquot of the solution suspected of containing tin in small amounts was adjusted to a final volume of 6 milliliters, which was 6f in HCl. This solution was transferred to a 100 milliliter beaker, heated to the boiling point, and a gram pellet of zinc was added. After one minute, 20 milliliters of a solution (made by diluting 10 ml of solution I to 800 ml, adding 2.5 ml of solution II, diluting to one liter, and allowing to stand one-half hour) was added to the beaker. After ten seconds, the contents were transferred to a six-inch test tube, and the tube was stoppered. Five minutes later, the color was compared visually with a series of standards made up at the same time and in the same way as the unknown solution. The authors claim an accuracy of 15% for amounts of tin ranging from 0.02 to 0.2 milligrams by this method. In all the experiments carried out, the amount of tin found ranged between the blank and 0.01 milligrams, and consequently, the amount of tin indicated as having been extracted into the aqueous layer is always a maximum value.

Another portion of the sample was also analyzed.

Colorimetrically for small amounts of the substance in the sample.

described in detail and results. The above substance was found

for this procedure. Volume 1 was found to contain 1.5 grams of

substance and volume 2 in the laboratory of work, which is similar

of concentrated sulfuric acid, and analysis for this substance.

Volume 2 was also analyzed by the same method as above.

of powdered silica was a mixture of silica for the purpose of

dissolving the silica in water, and the liquid to be analyzed

of the solution suspected of containing the substance.

attached to a flask volume of 2 milliliters, which was then

this solution was transferred to a 100 milliliter flask, and

the boiling point, and a gram weight of silica was added, and

volume, 20 milliliters of a solution (made by dissolving 10 g of

volume 1 to 100 ml, adding 1.5 ml of solution 2), which was

then, and allowed to stand overnight, and then to be tested.

After ten seconds, the contents were transferred to a 100 ml flask

tube, and the tube was removed. The mixture, later, the color was

compared visually with a series of standards made in the same

and in the same way as the unknown solution. The results are

accuracy of 1% for amounts of the substance from 0.1 to 0.5 milligrams

by this method. In all the experiments carried out, the amount of the

found ranged between the 0.1 and 0.5 milligrams, and the results

the amount of the substance as found was compared with the known

layer is always a mixture of silica.

It was found, after the first few runs, that the activity recovered from the benzene before extraction was considerably less than the total activity obtained by separately ashing a sample of tetraphenyl tin before solution in benzene. However, when the sum of the activity in the benzene phase after extraction and the activity in the aqueous layer was compared to the activity in the benzene phase before extraction, the material balance was much better. It was thought that some of the activity might be lost when the tetraphenyl tin was first dissolved in benzene in a beaker due to radio-colloidal effects, in which case some of the activity might be left behind on the walls of the vessel used. To test this, in some of the subsequent runs an attempt was made to remove any activity that might have adhered to the walls of the beaker in which the tetraphenyl tin was originally dissolved, by washing the walls with hot concentrated sulfuric acid and 30% hydrogen peroxide. After the beaker and contents had cooled to room temperature, 10 milligrams of tin in the form of stannic chloride was added, and the walls of the beaker washed with the contents. The tin was again precipitated as tin phenylarsonate and counted in order to determine whether any of the activity had gone to the walls of the beaker when the active tetraphenyl tin was originally dissolved in benzene.

In order to obtain the total tin activity in any sample, a separate 36 milligram sample of tetraphenyl tin (tin content 10 milligrams), which had been irradiated together with the 800-milligram

It was found, after the first run, that the activity

recovered from the decrease but the amount was considerably less

than the total activity obtained by repeatedly adding a small amount

of the activity to the system. However, when the system

of the activity in the system was after separation and the activity

in the system was compared to the activity in the system

phase before separation, the material balance was well satisfied. It

was thought that some of the activity was lost by the

process in the first run. In the second run, the activity

collected in the first run was used in the second run. The

defined on the basis of the initial amount. The first run was

the experiment was in which the activity was used in the first run

might have adhered to the walls of the vessel in which the activity

was originally dissolved, or because the activity was not completely

collected in the first run. After the second run, the activity

was found to be less than the activity in the first run.

activity was added, and the walls of the vessel were washed

the activity. The activity was then used in the third run.

and counted in order to determine whether any of the activity was

gone to the walls of the vessel. The activity was found to be

originally dissolved in benzene.

In order to obtain the total activity in the system,

a separate experiment was made of separating the activity in

benzene, which was then dissolved in benzene with the 100-ml. benzene

sample used in the Skillard-Chalmers separation, was ashed directly and its activity determined.

The entire procedure is presented in the form of an abbreviated flow sheet, as shown in Figure IV.

sample used in the United States is as follows:

and its activity is measured.

The entire procedure is presented in the form of a

flow chart in the appendix.

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FIGURE IV

Ssillard-Chalmers Separation

Page 11

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Procedure for Determining Form of Retained Activity

In order to determine whether or not all of the activity retained in the benzene phase after extraction was in the form of tetraphenyl tin, the following experiment was carried out.

An 800-milligram sample of tetraphenyl tin which had been irradiated with slow neutrons for two hours was dissolved in 100 milliliters of benzene. This solution was extracted with 20 milliliters 3f hydrochloric acid. A five-milliliter aliquot was removed from the benzene phase, the benzene evaporated, and the remaining organic residue was ashed. The tin in this aliquot was eventually precipitated as stannic phenylarsonate and counted to obtain a measure of the total tin activity.

Then, a twenty-milliliter aliquot was removed from the benzene phase. The tetraphenyl tin was crystallized from this solution in three successive fractions, and a fourth and final fraction was obtained by completely evaporating the remaining solvent. The apparatus used in this step is shown in Figure V. Dried air was sucked through the solution in order to keep it homogeneous and to hasten the crystallizations. The solid fractions thus obtained were ashed in the usual way, and the specific activity of tin in each fraction compared.

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FIGURE V

APPARATUS FOR THE FRACTIONAL CRYSTALLIZATION
OF TETRAPHENYL TIN FROM BENZENE

OF TETRAPHENYL LITHIUM BENZENE
 APPARATUS FOR THE EVOLUTIVE CRYSTALLIZATION

FIGURE 1

CHAPTER III

RESULTS

The data collected are listed in Tables III through X and summarized in Table XI.

It is seen that for 800-milligram samples of tetraphenyl tin, the specific activity in the aqueous solvent was about three thousand times larger than the specific activity in the benzene before extraction and that about fifty per cent of the total activity was extracted into the aqueous phase. However, this was not true when distilled water was the aqueous solvent. Further, when smaller samples of tetraphenyl tin were used, the enrichment factor was greatly decreased, but the amount of activity extracted into the aqueous phase remained about the same.

In the individual experiments, the total activity obtained by direct ashing of a separate sample irradiated under identical conditions equaled, within 10 per cent, the sum of the activity on the walls of the beaker and the total activity found in the benzene solution before extraction, (except for experiment 3).

Also, the activity in the benzene phase after extraction plus the activity in the aqueous phase equals the activity in the benzene phase before extraction, within 9 per cent. This again does

RESULTS

The data collected are listed in Table III through I and

summarized in Table II.

It is seen that for 200-milligram samples of benzene and

oil, the specific activity in the aqueous solvent was about three

thousand times larger than the specific activity in the benzene solvent

extraction and that about fifty per cent of the total activity was

extracted into the aqueous phase. However, this was not true when

distilled water was the aqueous solvent. Further, when smaller

samples of benzene were used, the enrichment factor was

greatly decreased, but the amount of activity extracted into the

aqueous phase remained about the same.

In the individual experiments, the total activity obtained

by direct washing of a separate sample irradiated water identical

conditions equaled, within 10 per cent, the sum of the activity in

the walls of the beaker and the total activity found in the benzene

solution before extraction (except for experiment 3).

Also, the activity in the benzene phase after extraction

was the activity in the aqueous phase equals the activity in the

benzene phase before extraction, within 2 per cent. This again was

not hold for experiment 3, where distilled water was used, unless wall losses are considered to occur on the separatory funnel of the order of magnitude of wall losses on the beaker.

In the experiment on the fractional crystallization of tetraphenyl tin from benzene after the solution had been extracted with 3f hydrochloric acid, it was found that the specific activity of successive fractions did not remain constant.

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will losses are ...
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In the ...
...
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of successive fractions ...

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TABLE III

Experiment 1

Aqueous phase.	0.3f HCl
Weight of tetraphenyl tin dissolved in 100 ml benzene ^a .	36 mg
Percent of total tin activity extracted into aqueous phase.	28%
Maximum amount of tin in aqueous phase	0.03 mg
Percent of total tin activity recovered ^b	83%
Enrichment factor ^c	93

<u>Source of Activity</u>	<u>Activity, counts per minute</u>
Benzene phase before extraction	---
Benzene phase after extraction	1,830
Aqueous phase	962
Tetraphenyl tin before solution in benzene (Total tin activity) ^d	3,447

^aSample was dissolved in a beaker and then transferred to a separatory funnel

^bPercent total tin activity recovered =

$$100 \times \frac{(\text{activity in benzene phase after extraction}) + (\text{activity in aqueous phase})}{\text{activity in tetraphenyl tin before solution in benzene}}$$

^cEnrichment factor = $\frac{\text{specific activity of tin in aqueous phase}}{\text{specific activity of tin before solution in benzene}}$

^dActivity determined from separately ashed 36 mg irradiated tetraphenyl tin

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Amount of activity	Amount of activity
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Amount of activity	Amount of activity

TABLE IV
Experiment 2

Aqueous phase.	3f HCl
Weight of tetraphenyl tin dissolved in 100 ml benzene ^a . .	80 mg
Percent of total tin activity extracted into aqueous phase	48%
Maximum amount of tin in aqueous phase	0.03 mg
Percent of total tin activity recovered ^b	78%
Enrichment factor ^c	.370

<u>Source of Activity</u>	<u>Activity, counts per minute</u>
Benzene phase before extraction	---
Benzene phase after extraction	1,204
Aqueous phase	2,164
Tetraphenyl tin before solution in benzene (Total tin activity) ^d	4,350

^aSample was dissolved in a beaker and then transferred to a separatory funnel

$$b\% = \frac{(\text{activity in benzene phase after extraction}) + (\text{activity in aqueous phase})}{\text{activity in tetraphenyl tin before solution in benzene}} \times 100$$

$$c \text{ Enrichment factor} = \frac{\text{specific activity of tin in aqueous phase}}{\text{specific activity of tin before solution in benzene}}$$

^dActivity determined from separately ashed 36 mg irradiated tetraphenyl tin

TABLE V
Experiment 3

Aqueous phase.	distilled H ₂ O
Weight of tetraphenyl tin dissolved in 100 ml of benzene ^a	800 mg
Percent of total tin activity extracted into aqueous phase ^b	4%
Maximum amount of tin in aqueous phase	0.03 mg
Percent of total tin activity recovered ^b	82%
Enrichment factor ^c	113

<u>Source of Activity</u>	<u>Activity, counts per minute</u>
Benzene phase before extraction	16,050
Benzene phase after extraction	7,450
Aqueous phase	808
Beaker in which tetraphenyl tin was dissolved	9,050
Tetraphenyl tin before solution in benzene (Total tin activity) ^d	21,100

^aSample was dissolved in a beaker and then transferred to a separatory funnel

$$b\% = \frac{(\text{activity in benzene phase after extraction}) + (\text{activity in aqueous phase}) + (\text{activity on beaker walls})}{\text{activity in tetraphenyl tin before solution in benzene}} \times 100$$

The activity in the benzene phase before extraction + the activity on the walls of the beaker = 25,100 c/m. whereas the total tin activity was found to be 21,000 c/m. The percentages in this experiment were calculated on the basis of 21,000 c/m and although they may be in error by as much as 20 percent, it is certainly clear that distilled water is not a good extracting agent for the tin activity.

^cEnrichment factor = $\frac{\text{specific activity of tin in aqueous phase}}{\text{specific activity of tin before solution in benzene}}$

^dActivity determined from separately ashed 36 mg irradiated tetraphenyl tin

Enrollment factor = $\frac{\text{Enrollment}}{\text{Enrollment} + \text{Enrollment} \times \text{Enrollment}}$
Enrollment factor = $\frac{\text{Enrollment}}{\text{Enrollment} + \text{Enrollment} \times \text{Enrollment}}$
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Activity determined from enrollment factor to be $\frac{\text{Enrollment}}{\text{Enrollment} + \text{Enrollment} \times \text{Enrollment}}$
Activity determined from enrollment factor to be $\frac{\text{Enrollment}}{\text{Enrollment} + \text{Enrollment} \times \text{Enrollment}}$

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TABLE VI
Experiment 4

Aqueous phase.	3f HCl
Weight of tetraphenyl tin dissolved in 100 ml of benzene ^a	800 mg
Percent of total tin activity extracted into aqueous phase.	40%
Maximum amount of tin in aqueous phase	0.04 mg
Percent of total tin activity recovered ^b	76%
Enrichment factor ^c	2200

<u>Source of activity</u>	<u>Activity, counts per minute</u>
Benzene phase before extraction	55,100
Benzene phase after extraction	22,300
Aqueous phase	28,160
Tetraphenyl tin before solution in benzene (Total tin activity) ^d	66,900

^aSample was dissolved in a beaker and then transferred to a separatory funnel

$$b\% = \frac{(\text{activity in benzene phase after extraction}) + (\text{activity in aqueous phase})}{\text{activity in tetraphenyl tin before solution in benzene}} \times 100$$

$$c \text{ Enrichment factor} = \frac{\text{specific activity of tin in aqueous phase}}{\text{specific activity of tin before solution in benzene}}$$

^dActivity determined from separately ashed 36 mg irradiated tetraphenyl tin

Table VI

Experiment II

Agarose phase 30.00

Volume of water-soluble fraction in 100 ml of
benzene 30.00

Percent of total tin activity extracted into aqueous
phase 100

Maximum amount of tin in aqueous phase
Percent of total tin activity recovered

Enrichment factor

Activity (total tin activity)	Source of activity
30.00	benzene phase before extraction
30.00	benzene phase after extraction
30.00	Aqueous phase
30.00	Initial activity in benzene solution in benzene (total tin activity)

Sample was dissolved in a solvent and then transferred to a separator
funnel

$DF = (\text{activity in benzene phase after extraction}) / (\text{activity in aqueous phase})$
activity in benzene phase after extraction in benzene

Enrichment factor = specific activity of tin in aqueous phase
specific activity of tin before extraction in benzene

Activity determined from an activity standard by comparison with
standard

TABLE VII
Experiment 5

Aqueous phase.	6f HCl
Weight of tetraphenyl tin dissolved in 100 ml of benzene ^a	800 mg
Percent of total tin activity extracted into aqueous phase.	4.8%
Maximum amount of tin in aqueous phase	0.04
Percent of total tin activity recovered ^b	96%
Enrichment factor ^d	2700

<u>Source of Activity</u>	<u>Activity, counts per minute</u>
Benzene phase before extraction	39,800
Benzene phase after extraction	16,830
Aqueous phase	26,640
Beaker in which tetraphenyl tin was dissolved	7,240
Tetraphenyl tin before solution in benzene (Total tin activity) ^d	52,900

^aSample was dissolved in a beaker and then transferred to a separatory funnel

^b% = $\frac{(\text{activity in benzene phase after extraction}) + (\text{activity in aqueous phase}) + (\text{activity on beaker walls})}{\text{activity in tetraphenyl tin before solution in benzene}} \times 100$

^cEnrichment factor = $\frac{\text{specific activity of tin in aqueous phase}}{\text{specific activity of tin in benzene phase before extraction}}$

^dActivity determined from separately ashed 36 mg irradiated tetraphenyl tin

TABLE VII
Experiment 2

Activity	Activity, counts per minute
Baseline phase before extraction	39,300
Baseline phase after extraction	38,330
Agonous phase	30,840
Baseline in which testosterone was dissolved	1,730
Testosterone in before solution in testosterone (total and active)	32,900

Sample was dissolved in a beaker and then transferred to a test tube.

$1\mu = (\text{activity in baseline phase after extraction}) + (\text{activity in agonous phase}) + (\text{activity in baseline phase})$

Activity in testosterone was before solution in testosterone

Testosterone factor = $\frac{\text{activity in baseline phase}}{\text{activity in testosterone before extraction}}$

Activity determined from equation used to be transferred to a beaker

TABLE VIII
Experiment 6

Aqueous phase.	1f NaOH
Weight of tetraphenyl tin dissolved in 100 ml of benzene ^a	800 mg
Percent of total tin activity extracted into aqueous phase.	50%
Maximum amount of tin in aqueous phase	0.04 mg
Percent of total tin activity recovered ^b	92%
Enrichment factor ^c	2800

<u>Source of Activity</u>	<u>Activity, counts per minute</u>
Benzene phase before extraction	43,300
Benzene phase after extraction	13,820
Aqueous phase	27,960
Beaker in which tetraphenyl tin was dissolved	7,040
Tetraphenyl tin before solution in benzene (Total tin activity) ^d	52,900

^aSample was dissolved in a beaker and then transferred to a separatory funnel

^b% = $\frac{(\text{activity in benzene phase after extraction}) + (\text{activity in aqueous phase}) + (\text{activity on beaker walls})}{\text{activity in tetraphenyl tin before solution in benzene}} \times 100$

^cEnrichment factor = $\frac{\text{specific activity of tin in aqueous phase}}{\text{specific activity of tin before solution in benzene}}$

^dActivity determined from separately ashed 36 mg irradiated tetraphenyl tin

Statement of Assets and Liabilities

Balance forward, 12/31/1911 \$100.00
 Receipts of contributions and other sources, 1912 50.00
 Disbursements for general expenses, 1912 (20.00)
 Balance forward, 12/31/1912 \$130.00
 Receipts of contributions and other sources, 1913 75.00
 Disbursements for general expenses, 1913 (40.00)
 Balance forward, 12/31/1913 \$165.00

Assets	Liabilities
Real estate, 100.00	Accounts payable, 20.00
Investments, 50.00	Notes payable, 10.00
Receivables, 30.00	Unpaid salaries, 5.00
Prepaid expenses, 10.00	Unpaid taxes, 5.00
Other assets, 5.00	Other liabilities, 5.00
Total, 195.00	Total, 45.00

Assets are classified as current and non-current, and liabilities as current and non-current.

The assets are classified as current and non-current, and the liabilities as current and non-current.

The assets are classified as current and non-current, and the liabilities as current and non-current.

Assets are classified as current and non-current, and liabilities as current and non-current.

TABLE IX
Experiment 7

Aqueous phase.	3f HCl
Weight of tetraphenyl tin dissolved in 100 ml of benzene ^a . 800 mg	
Percent of total tin activity extracted into aqueous phase.	61%
Maximum amount of tin in aqueous phase	0.04 mg
Percent of total tin activity recovered ^b	100%
Enrichment factor ^c	3400

<u>Source of Activity</u>	<u>Activity, counts per minute</u>
Benzene phase before extraction	43,400
Benzene phase after extraction	17,940
Aqueous phase	30,080
Tetraphenyl tin before solution in benzene (Total tin activity) ^d	47,800

^aSample was dissolved directly in a separatory funnel

$$b\% = \frac{(\text{activity in benzene phase removed before extraction}) + (\text{activity in benzene phase after extraction}) + (\text{activity in aqueous phase})}{\text{activity in tetraphenyl tin before solution in benzene}} \times 100$$

$$c \text{ Enrichment factor} = \frac{\text{specific activity of tin in aqueous phase}}{\text{specific activity of tin before solution in benzene}}$$

^dActivity determined from separately ashed 36 mg irradiated tetraphenyl tin

TABLE IX

Experiment 7

Adipose tissue..... 1.13 g
 Weight of fat removed at 110 ml of benzene..... 1.00 g
 Percent of total dry weight extracted into adipose phase..... 88.5
 Benzene amount of oil in adipose phase..... 0.43 g
 Percent of total dry weight recovered..... 100
 Extraction factor..... 3.00

Percent of activity	Activity, counts per minute
Benzene phase before extraction	15,100
Benzene phase after extraction	17,100
Adipose phase	30,000
Total activity in benzene before extraction	15,100
Benzene (Total activity)	17,100

^a Sample was dissolved directly in a separatory funnel

$df =$ (activity in benzene phase removed before extraction) + (activity in benzene phase after extraction) - (activity in adipose phase)

Extraction factor = $\frac{\text{activity in benzene phase after extraction}}{\text{activity in benzene phase before extraction}}$

^b Activity determined from scintillation counter

TABLE X

Fractional Crystallization Experiment

Sample	Weight in mg of $\text{Sn}(\text{C}_6\text{H}_5\text{AsO}_3)_2$	Specific Activity, c/m per 50.2 mg $\text{Sn}(\text{C}_6\text{H}_5\text{AsO}_3)_2$
first fraction	43.2	2,090
second fraction	31.3	1,530
third fraction	53.5	615
fourth fraction	55.7	2,283
five-ml aliquot of benzene phase ashed directly	50.2	1,816

Total Activity by direct ashing. 7,264 c/m

Total activity recovered in fractionation. 6,518 c/m

Percent of total tin activity recovered. 90%

Percent of total tin recovered^a. 92%

^a% =
$$\frac{\text{sum of the weights of stannic phenylarsonate from all four fractions}}{\text{four times the weight of stannic phenylarsonate from direct ashing}} \times 100$$

Sample	Weight in oz. of solution	Weight of solid in the solution
First fraction	10.0	2.000
Second fraction	10.0	1.330
Third fraction	10.0	0.75
Fourth fraction	10.0	2.280
Fifth fraction	10.0	1.310
Residue	10.0	1.310

Total activity by direct assay 1.610 g/e
 Total activity recovered in fractionation 6.510 g/e
 Percent of total activity recovered 40%
 Percent of total activity recovered 40%

Sum of the weights of solids precipitated from all
 four times the weight of solids precipitated from
 one fraction = 100%

TABLE XI

Summary of Results

Aqueous Solution	mg of Tetraphenyl Tin dissolved in 100 ml Benzene	Enrichment Factor ^a	% Activity in Aqueous Phase
0.3f HCl	36	93	28
3.0f HCl	80	370	48
distilled water	800	113	4
3.0f HCl ^b	800	3400	61
3.0f HCl	800	2200	40
6.0f HCl	800	2700	48
1.0f NaOH	800	2800	50

^aEnrichment factor = $\frac{\text{specific activity of tin in aqueous phase}}{\text{specific activity of tin before solution in benzene}}$

^bSample of tetraphenyl tin was dissolved directly in separatory funnel

Temperature	Time	Pressure	Volume
20	10	100	100
20	20	100	100
20	30	100	100
20	40	100	100
20	50	100	100
20	60	100	100
20	70	100	100
20	80	100	100
20	90	100	100
20	100	100	100

The following table shows the results of the experiments conducted on the 10th of June 1900. The results are given in the form of a table, the columns of which are headed as follows:—
 Temperature, Time, Pressure, Volume.

CHAPTER IV

DISCUSSION

According to the results summarized in Table XI, the Szilard-Chalmers reaction can be used to prepare tin solutions of high specific activity. When hydrochloric acid or sodium hydroxide is used as the extraction solvent, approximately fifty per cent of the total activity is extracted, and the specific activity is increased about 3000 times over what it was in the target compound.

Since the colorimetric analysis used for determining the total amount of tin extracted into the aqueous phase indicated only an upper limit, the enrichment factors are probably larger than reported.

Table XI shows that when distilled water is used for the aqueous solvent, the enrichment factor, as well as the per cent of total activity extracted, are small. This might be accounted for by the formation of radiocolloids. Generally, when substances are present in trace amounts, which under ordinary conditions form insoluble compounds, the tracer quantities tend to form colloids. When distilled water is used as the extraction solvent, any inorganic tin present would be expected to hydrolyze, and, as a result, exhibit radiocolloidal behavior.

In the experiments where the beaker, in which the tetraphenyl

In the wastewater effluent the oxygen is still not fully depleted

result, which is not unusual.

Inorganic substances which are reported to be present in the effluent are listed in the following table.

Insoluble compounds, the presence of which is indicated by the formation of a precipitate when the effluent is treated with barium chloride, are listed in the following table.

by the formation of a precipitate. Compounds which are reported to be present in the effluent are listed in the following table.

total solids reported, and which are reported to be present in the effluent are listed in the following table.

separate solvent, the effluent is treated with barium chloride, and the total solids reported, and which are reported to be present in the effluent are listed in the following table.

reported.

an upper limit, the effluent is treated with barium chloride, and the total solids reported, and which are reported to be present in the effluent are listed in the following table.

total amount of the effluent is treated with barium chloride, and the total solids reported, and which are reported to be present in the effluent are listed in the following table.

increased about 1000 times over what is reported to be present in the effluent are listed in the following table.

the total solids is much less, and the effluent is treated with barium chloride, and the total solids reported, and which are reported to be present in the effluent are listed in the following table.

is used as the effluent is treated with barium chloride, and the total solids reported, and which are reported to be present in the effluent are listed in the following table.

high specific activity, and the effluent is treated with barium chloride, and the total solids reported, and which are reported to be present in the effluent are listed in the following table.

Barford-Chalkley method, and the effluent is treated with barium chloride, and the total solids reported, and which are reported to be present in the effluent are listed in the following table.

According to the results presented in Table I, the effluent is treated with barium chloride, and the total solids reported, and which are reported to be present in the effluent are listed in the following table.

tin was originally dissolved in one hundred milliliters of benzene, was tested for activity, it was found that a considerable amount, about fifteen per cent of the total activity, went to the walls. Here again, if some of the activated tin is present in an inorganic state, it might not dissolve completely in the benzene, and some of it is adsorbed instead on the beaker walls.

Loss of activity on the walls of vessels naturally leads to an increase in the percent of activity retained, and a corresponding decrease in the percent of activity extracted into the aqueous solvent. This difficulty can be overcome by dissolving the active sample in benzene directly in a separatory funnel, for in that case, the activity which goes to the walls of the funnel may be washed off by the extraction solvent. This apparently occurred in Experiment 7 where the sample was dissolved directly in the separatory funnel. In that event, one hundred percent of the activity was accounted for, and slight increase in the enrichment factor and in the percent of activity extracted was observed.

The experiment designed to determine if the retained activity was present as tetraphenyl tin seems to indicate that, while most of the activity is present in that form, some of it is retained otherwise..

If all the retained activity were present as tetraphenyl tin, the specific activity in succeeding fractions should be constant.

the one originally proposed in the preceding chapter of the
was based on activity. It was found that a certain amount
about fifteen per cent of the total activity, and in the
very early, it was of the order of 10 per cent in the
state, it might be expected to be in the order of 10 per cent
it is assumed to be in the order of 10 per cent.

loss of activity in the early stages of the reaction is
to an increase in the amount of activity. It was found that
decrease in the amount of activity. It was found that
solvent. The activity of the solvent was found to be
sample in the early stages of the reaction. It was found
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In the early stages of the reaction, the activity was found
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The activity was found to be in the order of 10 per cent
was found as being in the order of 10 per cent. It was
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This was not the case. Instead, the specific activity decreased in the first three fractions, while the last fraction, from which all the remaining solvent was evaporated, showed a very strong increase in specific activity.

Table XII^{9,10} shows some of the known organo-metallic compounds of tin with their properties. It is possible that some of these might account for part of the activity which is retained in the benzene phase, but is not there as tetraphenyl tin.

TABLE XII

Compounds of Tin

Name	M.P.	B.P.	Solubility, g/100 ml
triphenyl tin	232.5	d.280	insoluble in water, 0.079 in alcohol, 7.82 in benzene, 18.1 in chloroform
diphenyl tin	225.7	---	insoluble in water; soluble in chloroform, benzene, and ether; insoluble in absolute alcohol
hexaphenyl ditin	232.5	d. < 280	---
triphenyl tin-hydride	---	173-174	---
diphenyltin dihydride	---	---	---
diphenyl tin oxide	dec.	---	insoluble in water, soluble in concentrated acid, insoluble in organic solvents
triphenyl tin hydroxide	118	---	---

No attempt was made to test this hypothesis as that would require the synthesis of most of these compounds as well as extensive separation experiments.

to extend the term of the lease to 1950. The lease was renewed for a period of 10 years, and the lessee was required to pay a sum of \$10,000 per annum for the term of the lease. The lease was renewed for a period of 10 years, and the lessee was required to pay a sum of \$10,000 per annum for the term of the lease.

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