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STUDIES ON THE REMOVAL OF RADIOISOTOPES FROM LIQUID WASTES BY COAGULATION

R. A. LAUDERDALE, JR.

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HEALTH PHYSICS DIVISION

STUDIES ON THE REMOVAL OF RADIOISOTOPES FROM LIQUID WASTES BY COAGULATION

by

Robert A. Lauderdale, Jr.

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Abstract

The report covers work which has been completed to date on the removal of radioisotopes from water using a calcium phosphate floc as a carrier. The work was performed as part of the Health Physics Waste Disposal Research Section work on the removal of small quantities of radioactivity from large volumes of water.

The conclusions which may be drawn from the work that has been completed are the following:

1. It has been found possible to remove from solution essentially all the cerium, yttrium, zinc, columbium, and zirconium by phosphate precipitation. The data for these elements can probably be extended to include other elements of similar chemical properties.

2. Approximately 10 per cent of the tungsten, 67 per cent of the antimony, and 95 per cent of the strontium were removed by the phosphate treatment.

3. Efficiencies of removal greater than 99 per cent have been obtained with a mixture of fission products by using clay in conjunction with the phosphate, followed by sand filtration. The efficiency of the process when used with a mixture of radio-

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active isotopes will depend on the composition of the mixture.

4. The greatest concentrations of activity in the floc were obtained under conditions of high pH and with an excess of phosphate.

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STUDIES ON THE REMOVAL OF RADIOISOTOPES FROM LIQUID WASTES BY COAGULATION

Part I.

Removal of Selected Isotopes from Distilled Water By Calcium Phosphate Precipitation

by

Robert A. Lauderdale, Jr.

Introduction

A large part of the research which has been done by various organizations on the treatment of radioactive wastes has emphasized the reduction in the volume of waste by evaporation or chemical precipitation. In the Health Physics Division, research has been directed toward removal of radioisotopes from liquid wastes by adsorption and co-precipitation of the active elements by coagulating agents such as alum, ferric hydroxide, and calcium phosphate. It is believed that coagulation as a method of waste treatment is useful and could compete with other processes for the following reasons:

1. It is suitable for the treatment of either large or small volumes of waste,
2. Adsorption is one of the most efficient methods of removing low concentration of impurities,

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3. The radioactivity can be concentrated from large volumes of waste liquid to relatively small volumes of solids,
4. Design features for coagulation, precipitation, and filtration equipment are standardized so that the development of special equipment is reduced to a minimum.
5. Minimal, rather than massive, doses of reagents are used,
6. The large quantities of sodium usually found in the wastes at atomic energy installations do not interfere in the process and can be left in solution and discharged.

The action of a coagulant in removing small quantities of impurities from liquids is not fully understood. In general it is believed that the removal is due to one or more of the following mechanisms:

1. True adsorption, due to the surface action of the floc and the charge on the adsorbed ion,
2. Co-precipitation, in which ions of the foreign element are incorporated in the floc as part of the precipitate,
3. Entrainment of microscopic particles in the waste to which foreign ions have become attached,
4. Occlusion or adsorption of particles of colloidal nature by the floc,
5. In cases in which turbidity is present in the waste, either naturally or by addition, adsorption of the foreign ion by the turbidity followed by entrainment by the floc.

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It was decided that of the three coagulants named a phosphate floc would probably produce the greatest concentration of activity. The decision to use phosphates was based on a comparison of the insolubility of phosphate compounds and hydroxides, which disclosed that the number of highly insoluble phosphates exceeds the number of insoluble hydroxides. This characteristic was considered desirable from the standpoint of Hahn's adsorption and co-precipitation rule which states that the adsorption and/or co-precipitation of an ion by a precipitant depends on the surface charge of the adsorbing precipitate and on the degree of insolubility of the adsorbed compound in the solvent involved.

Materials and Equipment

The isotopes of immediate interest are those of the fission products, and particularly those of fairly long half-life. For the initial tests isotopes were selected which would represent the different groups of the periodic chart. It should then be possible to extrapolate the data obtained for a given element to other elements having similar chemical characteristics.

Studies of the removal of the isotopes of Ce, Sr, Zr, Nb, Zn, Y, Sb, and W have been made. The floc was formed with $Ca(OH)_2$ and either KH_2PO_4 or Na_3PO_4. Two variable speed multiple stirrers were used to agitate the liquid-floc mixture. Measurements of pH were made with a model H-2

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Beckman pH meter. All samples were counted on an end-window type counter with the exception of the Zn^{65} samples which were counted in a proportional type counter because of the very low energy of the beta radiation from this isotope.

Procedure

The phosphate floc was formed by adding the radioisotope and a solution of either KH_2PO_4 or Na_3PO_4 to a solution of $Ca(OH)_2$ in distilled water. The radioisotope was added with the phosphate in sufficient quantity to give a count of approximately 1000 c/m/ml in the final mixture. The final volume in each beaker was 500 ml. In the initial tests a variation in pH was obtained by varying the $Ca(OH)_2$ concentration. In later tests the pH was adjusted by adding NaOH or HNO_3.

During and immediately following the addition of reagents the mixture was given a five minute flash mix. This was followed by one hour of very slow stirring which allowed the floc to grow to settleable size. Following the slow mix the floc was allowed to settle for two hours.

The total radioactivity added to the water was accounted for by taking triplicate samples of the following:

1. The stock solution of the radioactive isotope,
2. The floc plus liquid mixture (taken during the slow mixing period),

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3. The settled supernatant liquid,
4. The floc, obtained by centrifuging a known volume of mixture to separate the floc and liquid,
5. The centrifuged liquid.

If a large discrepancy was found between the total count added, and the sum of the counts of the floc and the supernatant liquid, the acid used to clean the glassware was counted to determine if adsorption had taken place on the beakers used.

Data and Results

The data which have been obtained for the removal of Ce^{144}, Sr^{89}, Y^{91}, Zr^{95}, Nb^{95}, Zn^{65}, Sb^{124}, and W^{185} are presented in Tables I through VIII. The maximum removals of each which have been found are tabulated on page 11.

The figures listed are for a one-stage batch treatment only. One two-stage treatment was carried out with Ce^{144} as the active isotope. A floc was formed in a composite of the supernatant liquid from the first stage. The reduction in the activity in the water was approximately 87.5 per cent for the second stage. An approximate figure only can be given since the remaining count was not sufficiently high to be significant.

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Isotope	Coagulant	Mg/Liter Coagulant	% Removal
Ce^{144}	KH_2PO_4	200	99.8
Ce^{144}	Na_3PO_4	120	99.9
Sr^{89}	KH_2PO_4	100	81.3
Sr^{89}	Na_3PO_4	240	97.8
Y^{91}	KH_2PO_4	100	99.9
Sb^{124}	KH_3PO_4	100	66.1
Sb^{124}	Na_3PO_4	120	67.4
Nb^{95}	KH_2PO_4	100	99.2
Zn^{65}	KH_2PO_4	50	99.6
Zr^{95}	KH_2PO_4	100	99.5
W^{185}	KH_2PO_4	200	10.7

Discussion of Results

The results obtained with the isotopes Ce^{144}, Zn^{65}, Y^{91}, Nb^{95}, and Zr^{95} indicate that essentially complete removals can be obtained by phosphate coagulation. The conditions of precipitation had little effect on the removal of these isotopes. (See Tables I to VIII). However, Sb^{124} and Sr^{89} showed a marked variation in removal if the conditions of precipitation were varied. For this reason Sr^{89} was selected as the isotope to use in a study of the variables involved (pH, phosphate to calcium ratio, and quantity of floc).

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The relation of the removal of strontium to the pH at the time of precipitation, and to the ratio of Na_3PO_4 to $Ca(OH)_2$ was determined by employing the following procedure. Six beakers were set up, each containing 55 ppm $Ca(OH)_2$ and 24 ppm Na_3PO_4 $\left(\frac{0.43 \text{ mg } Na_3PO_4}{1.0 \text{ mg } Ca(OH)_2}\right)$, but with the pH varying from approximately 9.0 through 12.0. After mixing and settling the supernatant liquid was counted as before. Five additional tests were made using the same concentration of $Ca(OH)_2$ but with quantities of phosphate sufficient to give additional ratios of 0.8:1, 2.2:1, 4.3:1, 12.9:1 and 25.8:1 mg Na_3PO_4 per mg $Ca(OH)_2$. The data thus obtained have been plotted in Figure 1 as a family of curves, with each curve showing the effect of pH on the removal of strontium for a particular ratio of phosphate to calcium.

From Figure 1 it can be seen that the pH has a marked effect on the per cent strontium removed until a pH of approximately 11.3 is reached. At pH values above 11.3, little effect due to pH was observed. In Figure 2 are shown data obtained by maintaining the pH of each sample above 11.3 and varying the ratio of Na_3PO_4 to $Ca(OH)_2$, between the values 0.43 and 25.8 mg Na_3PO_4 to 1.0 mg $Ca(OH)_2$. The per cent strontium removed increases rapidly as the phosphate to calcium ratio increases. A sharp break occurs at a ratio of 2.2:1, which corresponds to a 46 per cent excess of phosphate. Above the ratio of 2.2:1 only a slight increase in the removal of strontium was noted.

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For the isotopes studied (with the possible exception of Sb^{124} and W^{185}) it is probably true that surface adsorption was not the predominating mechanism of removal. The high removals which were found suggest that either co-precipitation was responsible or that the radioactive constituent was present in a colloidal form which could be entrained by the floc. The increased removal of Sr^{89} by Na_3PO_4 may be due to the formation of $Sr_3(PO_4)_2$ instead of $SrHPO_4$ which would be expected when KH_2PO_4 was used as the source of phosphate. In general compounds of the first form are more insoluble than those of the latter; a condition which would be expected to cause an increase in the quantity of strontium carried by the floc.

In the case of antimony (Table V) co-precipitation should not be one of the modes of removal, as compounds of antimony and the phosphoric acids are not known. The removal of antimony could be due to adsorption alone, or to the presence or formation of insoluble oxides.

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Part II

Removal of Mixed Fission Products from Tap Water

In order to determine the efficiency of phosphate flocculation on a waste containing mixed fission products, a solution was obtained which was thought to be representative of an actual waste. Radiochemical analyses showed that the following isotopes were present.

Tri-valent rare earths, other than cerium	43.5%
Cerium	27.0
Strontium	17.4
Barium	5.1
Ruthenium	2.9
Cesium	1.1
Total	97.0%

The remaining three per cent would contain traces of a large number of other isotopes with a low fission yield. The tracer runs which had been made (see Part I) indicated that essentially all the rare earths would be removed, and about 95 per cent of the strontium. It would also be expected that barium would act similarly to strontium. The ruthenium and cesium would not be expected to precipitate and hence, removal of these two would be by either adsorption on the surface of the floc,

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adsorption on an inert material such as clay or carbon, or in the case of ruthenium, occlusion as a radiocolloid.

The procedure used in the tests with mixed fission products was essentially the same as in the tracer experiments, except that tap water was used for dilution instead of distilled water. This was done to give a solution which should be more nearly representative of an actual waste. In the tests in which a two-stage treatment was used, activated carbon, ferric chloride and a kaolinitic clay were sometimes used in addition to the phosphate. Typical data for this series of experiments are shown in the following table.

TREATMENT OF MIXED FISSION PRODUCTS BY FLOCCULATION

Test	Initial c/m/ml	Stage	Treatment	pH	% Removed	Method of Separation
I	14,050	1	220 ppm clay 220 ppm Na_3PO_4	11.5	96.4	Centrifuged
		2	100 ppm $Ca(OH)_2$	11.5	98.1	
II	13,300	1	220 ppm Na_3PO_4 50 ppm carbon	11.5	94.1	Centrifuged
		2	100 ppm $Ca(OH)_2$	11.5	97.0	
III	13,485	1	200 ppm Fullers earth	8.3	75.0	Centrifuged
		2	125 ppm Na_3PO_4	11.5	96.8	Centrifuged
IV	1,295	1	50 ppm carbon 100 ppm $FeCl_3$	8.0	73.0	Centrifuged
		2	200 ppm clay 200 ppm Na_3PO_4	11.5	98.5	
V	3,758	1	200 ppm Na_3PO_4	11.5	95.8	Centrifuged
		2	100 ppm $Ca(OH)_2$ 100 ppm Na_3PO_4	11.5	97.3	
		3	200 ppm clay 50 ppm $Ca(OH)_2$	11.5	99.0	

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On the basis of these and similar data the conclusion was reached that pH was one of the most important factors in the treatment of radioactive wastes by this method, that carbon and fuller's earth were no more effective than clay in supplementing the phosphate treatment, and that a calcium phosphate floc was superior to ferric hydroxide.

In the remainder of the tests that were made, the following procedure was used. The pH of the waste was raised with NaOH to between 11.5 and 12.0. After a short stirring time clay was added in quantities up to 200 ppm. After approximately ten minutes mixing the sodium phosphate was added to produce the calcium phosphate floc. It was unnecessary to add calcium since it was present as a natural constituent of the tap water used for dilution. After twenty to thirty minutes of slow stirring the floc was allowed to settle and the liquid was filtered through a sand filter made of a 5/8" glass tube filled to a depth of 21 inches with a commercial grade sand. A portion of the data obtained using this procedure is shown below.

The removals listed below can be applied only to the particular mixture of isotopes used in these tests. Any change in the composition of the mixture will be reflected in residual radioactivity left in the solution. For example, if cesium were present in greater quantities, the efficiency of the precipitation process would probably be lowered. If the mixture were composed entirely of the rare earth isotopes, the efficiency would probably be higher.

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TREATMENT OF MIXED FISSION PRODUCTS BY PHOSPHATE PRECIPITATION

Test	Stage	Treatment	Initial c/m/ml	Final c/m/ml	% Removed
VI	1	100 ppm clay 100 ppm Na_3PO_4	4810	31	99.4
VII	1	100 ppm clay 100 ppm Na_3PO_4	2150	27	98.7
	2	100 ppm $Ca(OH)_2$ 100 ppm Na_3PO_4		18	99.2
VIII	1	100 ppm clay 100 ppm Na_3PO_4	2935	12	99.6

Conclusions

The conclusions which may be drawn from the work that has been completed are the following:

1. It has been found possible to remove from solution essentially all the cerium, yttrium zinc, columbium, and zirconium by phosphate precipitation. The data for these elements can probably be extended to include other elements of similar chemical properties.

2. Approximately 10 per cent of the tungsten, 67 per cent of the antimony, and 95 per cent of the strontium were removed by the phosphate treatment.

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3. Efficiencies of removal greater than 99 per cent have been obtained with a mixture of fission products by using clay in conjunction with the phosphate, followed by sand filtration. The efficiency of the process when used with a mixture of radioactive isotopes will depend on the composition of the mixture.

4. The greatest concentrations of activity in the floc were obtained under conditions of high pH and with an excess of phosphate.

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Mr. T.W. Brockett and Mr. A.H. Emmons, both of Oak Ridge National Laboratory.

RALauderdale/r
November 28, 1950

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Table I

REMOVAL OF Ce^{144} FROM WATER BY CALCIUM PHOSPHATE FLOC

Coagulant Used	Mg/Liter Coagulant	Mg/Liter $Ca(OH)_2$	pH	% Removed
KH_2PO_4	10	7.0	9.4	no floc
		11.0	9.9	no floc
		18.0	10.3	70.9
		27.0	10.6	85.3
		36.0	10.8	82.7
		45.0	11.0	75.6
KH_2PO_4	20	18	10.0	83.5
		27	10.7	94.4
		36	11.0	97.6
		54	11.3	98.0
		72	11.4	97.9
		108	11.7	98.1
KH_2PO_4	40	11.0	7.9	no floc
		18.0	9.3	no floc
		27.0	10.1	95.5
		27.0	9.8	95.4
		45.0	10.8	98.0
		45.0	10.8	98.4
		72.0	11.2	99.1
		90.0	11.4	99.3
		108.0	11.5	97.7
		108.0	11.6	99.3
		144.0	11.8	99.3
		216.0	12.0	99.4
KH_2PO_4	100	45	8.7	86.3
		63	9.5	98.8
		90	11.0	98.6
		135	11.7	98.9
		162	11.9	99.4
		234	12.2	99.6

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Table I (Cont.)

Coagulant Used	Mg/Liter Coagulant	Mg/Liter $Ca(OH)_2$	Mg/Liter Cerium	pH	% Removed
KH_2PO_4	200	108		10.3	98.8
		135		11.0	98.7
		162		11.3	99.3
		216		11.6	99.0
		270		11.8	99.8
		315		11.9	99.8
Na_3PO_4	120	108		11.8	99.6
		135		11.85	99.7
		162		11.95	99.7
		216		12.1	99.6
		270		12.1	99.7
		315		12.1	99.6
Na_3PO_4	120	135	0.25	11.9	98.8
			0.50	11.9	99.5
			1.0	11.85	99.9
			1.75	11.7	99.7
			2.0	11.8	99.3
			2.0	11.7	99.4
			3.0	11.7	99.2
			4.0	11.7	99.2
			4.0	11.7	99.1
			5.0	11.7	99.9
			6.0	11.7	99.7
			6.0	11.7	99.7
			7.0	11.7	99.8
			7.0	11.8	99.7
			8.0	11.7	99.3
			8.0	11.8	99.8

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Table II

REMOVAL OF Zn^{65} FROM WATER BY CALCIUM PHOSPHATE FLOC

Coagulant Used	Mg/Liter Coagulant	Mg/Liter $Ca(OH)_2$	pH	% Removed
KH_2PO_4	50	65	10.5	93.8
		70	10.9	~ 100
		80	11.2	99.6
		90	11.5	99.3
		100	11.5	99.6
KH_2PO_4	100	160	8.9	97.6
		180	10.8	99.3
		225	11.5	98.4
		250	11.6	98.5
		270	11.8	98.5

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Table III

REMOVAL OF Zr^{95} FROM WATER BY CALCIUM PHOSPHATE FLOC

Coagulant	Mg/Liter Coagulant	Mg/Liter $Ca(OH)_2$	pH	% Removed
KH_2PO_4	100	180	11.2	85.1
		180	10.1	98.9
		270	11.4	98.5
		270	11.5	98.4
		320	11.6	~100
		320	11.6	98.3
		380	11.8	99.5
		380	11.7	97.9
		450	11.9	99.5
		450	11.9	98.9

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Table IV

REMOVAL OF Y^{91} FROM WATER BY CALCIUM PHOSPHATE FLOC

Coagulant Used	Mg/Liter Coagulant	Mg/Liter $Ca(OH)_2$	pH	% Removed
KH_2PO_4	20	18	10.5	no floc
		27	10.6	94.6
		36	10.9	97.0
		54	11.2	98.3
		90	11.4	99.1
KH_2PO_4	40	18	8.5	no floc
		36	9.8	96.8
		54	10.4	98.3
		90	11.0	98.9
		125	11.3	98.9
KH_2PO_4	100	72	10.3	98.8
		108	10.9	99.4
		144	11.2	99.8
		180	11.3	99.9
		225	11.4	99.7

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Table V

REMOVAL OF Sb^{124} FROM WATER BY CALCIUM PHOSPHATE FLOC

Coagulant Used	Mg/Liter Coagulant	Mg/Liter $Ca(OH)_2$	pH	% Removed
KH_2PO_4	100	72	8.5	5.5
		108	10.0	24.1
		108	10.9	39.5
		108	11.2	45.0
		180	11.3	56.5
		225	11.6	54.4
		225	11.5	58.1
		270	11.6	61.9
		315	11.7	65.2
		360	11.8	66.1
Na_3PO_4	120	90	11.4	18.9
		135	11.6	47.2
		180	11.7	59.7
		225	11.85	64.6
		270	11.95	67.4

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Table VI

REMOVAL OF Nb^{95} FROM WATER BY CALCIUM PHOSPHATE FLOC

Coagulant Used	Mg/Liter Coagulant	Mg/Liter $Ca(OH)_2$	pH	% Removed
KH_2PO_4	100	270	10.3	95.6
		320	11.25	98.1
		360	11.52	98.2
		450	11.80	99.0
		540	12.0	99.2

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Table VII

REMOVAL OF Sr^{89} FROM WATER BY CALCIUM PHOSPHATE FLOC

Coagulant	Mg/Liter Coagulant	Mg/Liter $Ca(OH)_2$	pH	% Sr Removed
KH_2PO_4	100	63	9.1	61.5
		63	9.0	30.4
		72	9.0	56.2
		81	9.8	73.8
		90	9.9	81.3
		90	10.4	80.1
		90	10.2	80.3
		100	10.5	81.2
		108	10.7	80.0
		117	10.6	75.3
		117	11.1	75.8
		117	10.9	74.8
		144	11.0	66.0
		180	11.2	31.2
		180	11.5	57.3
		216	11.5	56.0
		216	11.7	53.3
Na_3PO_4	24.1	56	9.0	10.5
			9.4	16.6
			10.2	21.2
			11.0	27.0
			11.5	27.6
			12.2	25.2
	48.3	56	9.0	24.9
			9.4	32.8
			10.1	47.9
			11.0	52.6
			11.6	54.8
			12.1	55.7

[redacted]

[redacted]

Table VII (Con't)

Coagulant	Mg/Liter Coagulant	Mg/Liter $Ca(OH)_2$	pH	% Sr Removed
Na_3PO_4	120.6	56	8.8	33.2
			9.3	56.7
			10.1	78.2
			10.9	91.8
			11.5	95.3
			12.0	95.9
	241.2	56	8.8	56.2
			9.3	----
			10.2	91.3
			10.9	95.7
			11.3	95.6
			11.9	97.4
	723.6	56	9.1	76.5
			9.5	84.8
			10.4	94.3
			11.2	96.9
			11.6	97.0
			12.1	96.7
	1457	56	9.0	78.2
			9.5	90.5
			10.3	95.4
			11.0	97.8
			11.6	98.1
			12.1	97.5

[redacted]

[redacted]

Table VIII

REMOVAL OF W^{185} FROM WATER BY CALCIUM PHOSPHATE FLOC

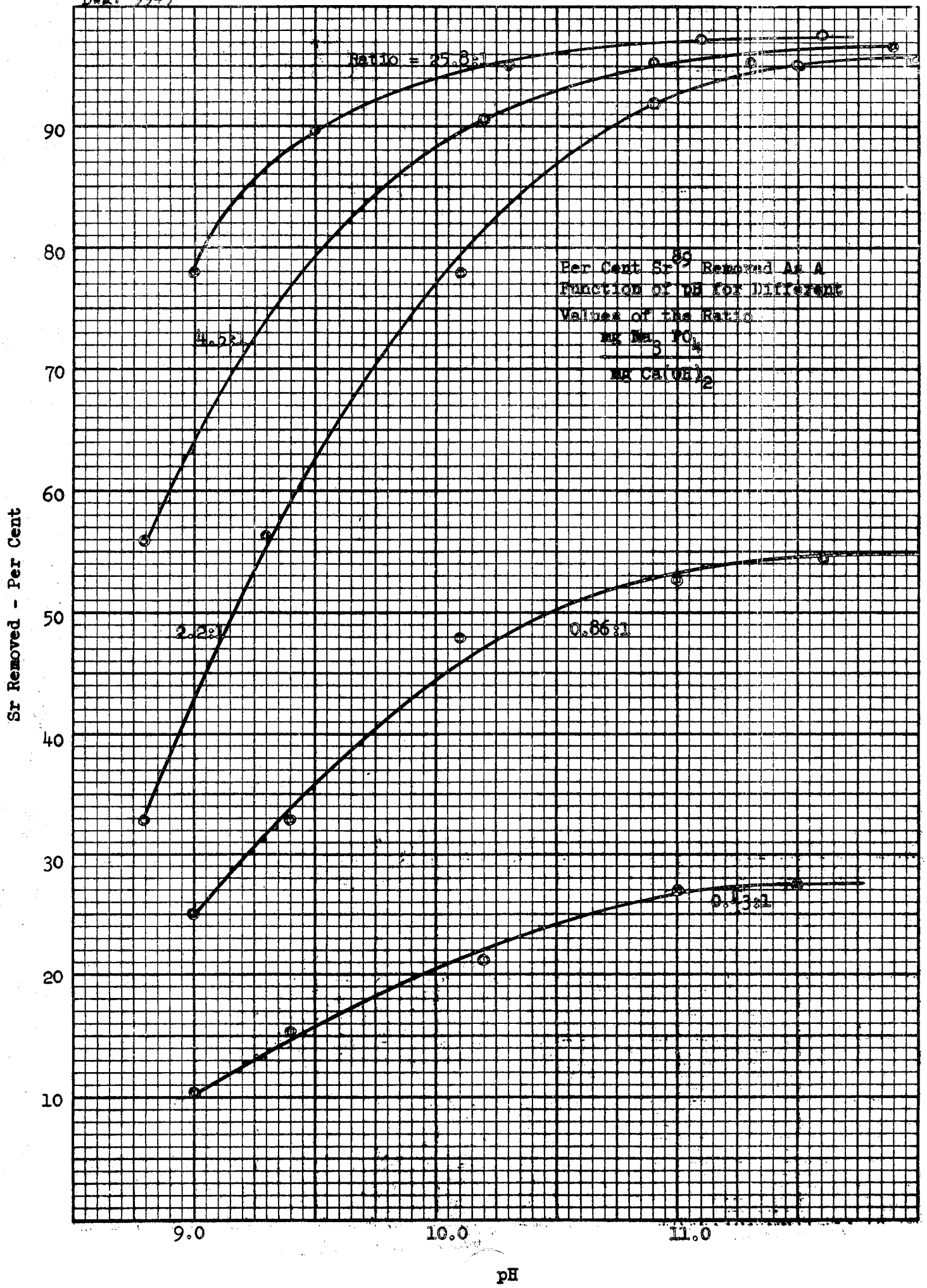
Coagulant Used	Mg/Liter Coagulant	Mg/Liter $Ca(OH)_2$	pH	% Removed
KH_2PO_4	200	72	9.3	6.5
	200	108	10.7	6.3
	200	144	11.4	10.7
	200	180	11.6	7.3
	200	216	11.8	10.5
	200	270	11.9	6.4

[redacted]

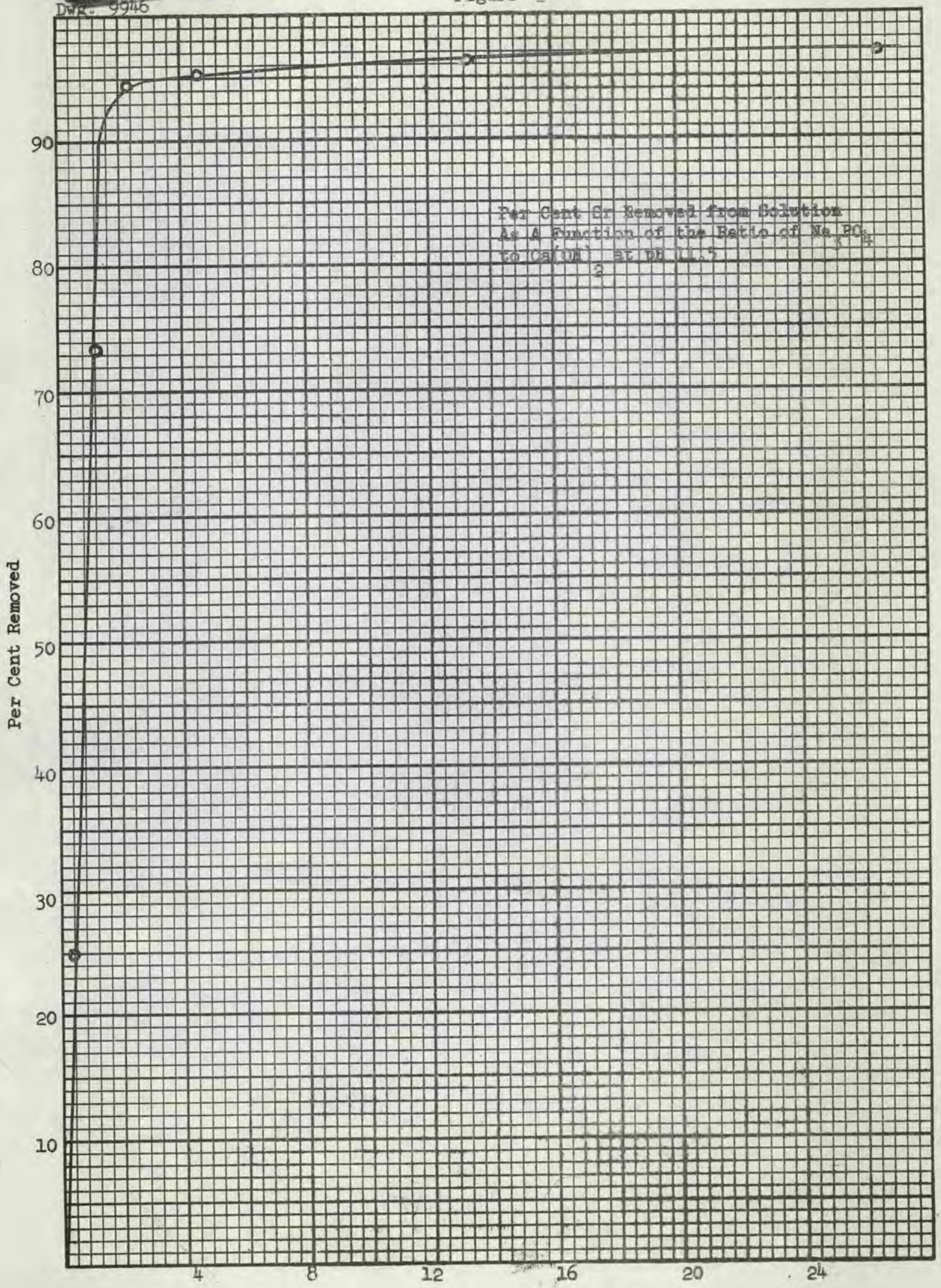
[redacted]

Dwg. 9945

Figure 1



[redacted]

Figure 2

Dwg. 9946