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AUSTRALIAN ATOMIC ENERGY COMMISSION RESEARCH ESTABLISHMENT LUCAS HEIGHTS

THE RECOVERY OF BERYLLIUM FROM BERYLLIUM OXIDE
MATRIX FUELS THROUGH ITS OXYACETATE

Ьу

M.S. FARRELL
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P. B. YATES

Issued Sydney, July 1965



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ABSTRACT

The laboratory development of a process for the recovery and decontamination of beryllium from fuel processing solutions is described. Azeotropic distillation is used in converting aqueous beryllium solutions to beryllium oxyacetate. The beryllium oxyacetate is then purified by recrystallization. Using a feed solution containing 3β – curies of fission products per mole of beryllium, a decontamination factor of 3 to 4×10^3 can be obtained with a 90 per cent. recovery of beryllium.

Some solubility and conductance data are given for beryllium oxyacetate and its hydrolysis products. The hydrolysis reaction is briefly discussed.

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INTRODUCTION

When beryllia, an expensive material, is considered for use as a moderator in a dispersion-type fuel it is necessary to investigate the possibilities of economic recovery of the beryllium from the spent fuel.

In schemes which have been proposed for aqueous reprocessing to recover the fissile and perhaps fertile components (Cairns 1963), the bulk of the beryllium would be in the form of aqueous nitrate or sulphate solutions substantially free from actinides but contaminated with fission products.

Many methods for the purification of beryllium compounds have been developed. These have been concerned mainly with mineral ore extraction, with the preparation of high purity beryllia, or with laboratory radiochemical separations. Most of this work is covered in reviews by Moore (1960), Fairhall (1960), Whitfield (1962), Aggett (1962), Cooper (1963), and Faroche and Lecocq (1963). Of the very few tried methods for the separation of fission products from beryllium solutions, the most significant is a solvent extraction process investigated by Tabushi (1959) and further investigated by Aggett (1963) which involves the formation of beryllium acetyl—acetonate. Cook (1952) used a beryllium oxyacetate process to obtain beryllium decontaminated from fission products.

This paper describes an investigation of one process for the recovery of beryllia from fission product contaminated aqueous solutions, based on the formation of beryllium oxyacetate. This process has been studied in some detail and appears feasible at least from the chemical viewpoint.

Any process to recover beryllia via the oxyacetate must go through the following major steps (which are also illustrated in Figure 1):

- (i) conversion of the beryllium existing as the aquo-ion [Be(H₂O)₄]^{2,+}, to covalent beryllium oxyacetate, Be₄O(CH₃COO)₈
 - (ii) isolation of the beryllium oxyacetate
 - (iii) purification of the beryllium oxyacetate
 - (iv) reconversion of the beryllium oxyacetate to beryllia

The conventional process for the first step is precipitation of the beryllium hydroxide and, after filtering, washing, and drying, its repetitive digestion in glacial acetic acid, with evaporation to dryness to remove the water (Besson and Hardt 1952). Kirkpatrick et al. (1961) removed the water by distillation and Szego (1961) used an azeotropic mixture to distill the water. In the process described here, azeotropic distillation is used to convert the beryllium as the aquo—ion in aqueous solution directly without precipitation of the hydroxide into covalent beryllium oxyacetate. This report discusses methods of converting beryllium nitrate and sulphate solutions into a solution of beryllium oxyacetate in anhydrous acetic acid.

Isolation of the beryllium oxyacetate from the acetic acid is most easily effected by simple crystallization. However there was a need to obtain some solubility data and this information is also included.

Although other better methods of purification may be available, recrystallization was chosen as it is relatively simple, and it appeared to give good decontamination from fission products. An obvious choice is to make use of the covalent nature of the beryllium oxyacetate and to distil or sublime it. Beryllium oxyacetate sublimes at its melting point (285°C) in vacuum or boils at 330°C, without decomposition (Everest 1964). Pure beryllia has been produced using distillation of the beryllium oxyacetate but not in the presence of fission products (Bubes 1944; Meyerson 1955; Plocher et al. 1961).

A simple method of purifying the oxyacetate is counter-current aqueous scrubbing of an immiscible solution of the beryllium oxyacetate in a suitable solvent. Some preliminary experiments proved

ineffective and beryllium oxyacetate is readily hydrolysed, but the method was not fully explored. Further solubility data and some conductivity data related to beryllium oxyacetate hydrolysis were obtained and are presented in this report.

The last major step in the overall process, conversion from beryllium oxyacetate to beryllia has been developed by Kirkpatrick et al. (1961) for the production of beryllia from beryl via the acetate. Their process has been used for the oxyacetate to oxide step, but other processes such as direct pyrolysis to the oxide are possible.

2. EXPERIMENTAL AND RESULTS

2.1 Preparation of Beryllium Oxyacetate

Beryllium oxyacetate was prepared by azeotropic distillation from three different starting materials:

- (a) moist beryllium hydroxide,
- (b) beryllium as an aqueous nitrate solution, and
- (c) beryllium as an aqueous sulphate solution.

The methods of preparation were as follows.

- (a) From beryllium hydroxide: Beryllium hydroxide (21.5 g), which may contain additional moisture, was placed in a 1 litre round bottom flask fitted with a water trap (Koelsch 1955), as shown in Figure 3, and with a reflux condenser. Glacial acetic acid (350 ml) and n propyl acetate (300 ml) were added. On refluxing, the water collected in the trap and was removed. When no more water came over 350 ml of the organic solvent was distilled off. The solution was then allowed to cool and the beryllium oxyacetate crystallized out. The yield was 45 g, or 90 per cent. The crude product was recrystallized from either chloroform or acetic acid.
- (b) From aqueous beryllium nitrate solution: Using (a) as a basis, beryllium oxyacetate was prepared in the following manner: An aqueous solution 30 (ml) containing a total of 0.25 moles of beryllium and 0.35 moles of nitrate was mixed with 29 g (0.35 moles) of sodium acetate in 50 ml water. Acetic acid (300 ml) and n-propyl acetate (300 ml) were then added. The mixture was refluxed as in (a). After removal of all the water sodium nitrate was crystallized out, and the hot solution was filtered off. The sodium nitrate was washed with two lots of (50 ml) hot acetic acid. The combined solutions were then evaporated down and the crystalline beryllium oxyacetate isolated as in (a).

The yield was similar to that obtained with the beryllium hydroxide, but losses on crystallization were greater owing to the sodium nitrate contamination in the crude product. With care, a maximum yield of 80 per cent. was obtained in successive single batch runs.

(c) From aqueous beryllium sulphate solution: The beryllium sulphate in aqueous solution was first reacted with an equivalent quantity of barium acetate to give a beryllium acetate solution and barium sulphate precipitate (Steinmetz 1907). In later runs calcium acetate was used instead of the barium salt because it is cheaper and the sulphate crystallized better. After filtering off and washing the gypsum the beryllium solution was converted to beryllium oxyacetate as in (a) or (b).

A typical run used the following quantities:

44 g Be SO₄ · 4H₂O 44 g Ca(CH₃COO)₂ · H₂O 200 ml n-propyl acetate 150 ml glacial acetic acid The gypsum was washed with three lots (20 ml each) of 1 per cent. acetic acid. After removal of the water by azeotropic distillation the organic solution was decanted from the small crop of Ca SO₄ crystals before evaporation to 150 ml. The yield was 23 grams of beryllium oxyacetate, corresponding to 92 per cent.

An alternative method for converting aqueous beryllium nitrate solution is possible by first converting the nitrate to sulphate by distillation of the nitric acid followed by procedure (c).

The elimination of the anion as a sulphate precipitate before azeotropic distillation tends to simplify the process.

2.2 Recovery of Beryllium from Fission Product Solutions

2.2.1 Low activity level experiments

Aqueous beryllium solutions were spiked with mixed fission products. For low level work 0.1 to 100 mc of the mixed fission products were used per run.

The procedures for recovering the beryllium oxyacetate from these solutions were similar to those described in Section 2.1, and all three methods were found to be satisfactory. Yields varied from 80 to 90 per cent. and both β and γ decontamination factors ranged from 2000 to 4000. With two recrystallizations from acetic acid and by counter – current re—use of the second and third mother liquors in subsequent runs, both a higher yield and higher decontamination can be obtained. The major loss of beryllium occurs in the first mother liquor. The main source of residual activity in the product was ruthenium, with a smaller activity from zirconium contributing. Table 1 gives yields and decontamination factors for these low level experiments.

As a part of these experiments various auxiliary steps were tried in an attempt to improve decontamination. Washing of the beryllium oxyacetate product with water was attempted (Kirkpatrick et al. 1961) and some increase in zirconium decontamination was achieved. The beryllium oxyacetate was dissolved in chloroform and the solution scrubbed with water, without improving the decontamination. Using an aqueous scrub solution containing 2.5 per cent. E.D.T.A a slightly improved decontamination was obtained but with lower yield due to hydrolysis of the beryllium oxyacetate. Acidifying the scrub solution increased the degree of hydrolysis and further decreased the yield.

Attempts to improve the overall decontamination by incorporating a "head-end" step before azeotropic distillation were not successful, although partial decontamination was achieved in some instances.

The following such steps were tested;

- (a) Distillation of the ruthenium as the tetroxide (RuO₄) with potassium permanganate and nitric acid. (Koda 1963). Half the ruthenium activity was removed with the distillate.
- (b) Co-precipitation of fission products with ferric hydroxide and manganese dioxide (Cook 1952; Lavrukhina 1957; Cooper 1963). The method used was as follows: The pH of the solution was adjusted to 5.0 5.5 with calcium hydroxide (about 0.13 mole per 0.25 mole of sulphate) and after filtration 50 mg of ferric nitrate and 10 mg of potassium permanganate were added. The solution was heated to 90 °C for one hour. After filtration a decontamination factor of ten (10) was consistently obtained for this step.
 - (c) Recrystallization of the beryllium sulphate from its solution, which gave no decontamination.

Towards the end of this work considerable adsorption of fission products on beryllium oxyacetate crystals was noticed when the crystals were allowed to stand under cold acetic acid for prolonged periods. Large differences in decontamination were caused by variations of time allowed for crystallization of otherwise identical beryllium oxyacetate solutions.

2.2.2 Medium activity level experiments

The process, as used in run N (Table 1), was tested with fission products present. Five batches of beryllium nitrate solutions containing up to 3β - curies of 18 months old mixed fission products per mole of beryllium were processed in a 4 inch lead - shielded glove box. Facilities in this cell were just adequate to conduct the experiments and imposed limitations. The equipment, consisting of suitably adapted laboratory glassware for tong handling, is shown in Figure 4.

All results confirmed the decontaminations obtained at lower levels of activity, but the maximum yield was only 53 per cent. The decontamination factor was 3500. The balance of the beryllium was accounted for, mainly in the calcium sulphate precipitate which had been insufficiently separated and washed owing to operational difficulty in the cell. It was demonstrated in a separate experiment, however, that the beryllium can be washed from the calcium sulphate.

2.3 Determination of Beryllium Oxyacetate Solubilities

The latest and most complete data on the solubility of beryllium oxyacetate have been given by Besson & Hardt (1952). During the present work some doubt arose on the validity of some of these figures, and no solubilities were available for some other solvents. The purest beryllium oxyacetate was used for solubility measurements after analysis had shown reasonable agreement with formula weight. [Be: 8.75% analysis, 8.87% theoretical; acetate: 85.8% analysis, 87.2% theoretical]

In the solubility experiments, beryllium oxyacetate crystals were shaken with various solvents at 25 °C for three days. Similar sample solutions were heated to 60 °C for one day and then re-equilibrated at 25 °C. The samples were contained in sealed glass ampoules and shaken in a thermostatically controlled water bath (Bishop et al. 1960). After complete sedimentation at 25 °C the liquid phase was carefully removed and analysed. The solid phase was kept to a minimum, consistent with obtaining a saturated solution. Table 2 lists the results obtained.

2.4 Measurement of Conductivities of Beryllium Acetate Solutions

Conductances were measured using a Philips conductivity bridge PR 9500 and a PR 9512/00 cell, held in a thermostatically controlled water bath. The acetic acid used in this work was distilled twice over potassium permanganate, then kept in a polythene bottle. Freshly distilled acetic acid had a conductance which decreased with time. Specific conductances of beryllium oxyacetate in anhydrous acetic acid and in acetic acid containing 1 mole of water per litre are shown in Table 3. Comparative measurements for barium acetate are shown also. The specific conductances of the hydrolysed beryllium oxyacetate in aqueous solutions of 0.01 to 2.0 mole equivalent concentrations are shown in Figure 5. For comparison, measurements were also made with the addition of 0.5 moles of acetic acid per mole of beryllium, making the final beryllium to acetate ratio 2:1. This is the ratio used by Sidgwick and Lewis (1926) whose results are plotted in Figure 5.

3. DISCUSSION

Although the experimental work is incomplete, a fairly detailed chemical flowsheet (Figure 2) can be constructed from the work to date, which will assist the testing of this particular process at higher levels of radioactivity. Further research is necessary into other methods of purifying beryllium oxyacetate, on the determination of the kinetics of hydrolysis of beryllium oxyacetate, and on the chemistry of the fission products in this system. However, some understanding of the chemistry has been obtained and is discussed below.

3.1 Azeotropic Water Removal

The novel and most important aspect of the process is the conversion of the aqueous beryllium acetate solution to an anhydrous solvent system by azeotropic distillation. Azeotropic removal of water from dilute solutions has been used in acetic acid manufacture for many years. Othmer (1936a, 1936b, 1942) describes suitable "entrainers" (that is, auxiliary solvents which form azeotropic mixtures with water) which are useful for this purpose

It was necessary to choose an entrainer suited for this slightly different process, and to determine its suitability in the presence of a large quantity of dissolved solids. n-Propyl acetate was found to be satisfactory, while iso-propyl acetate, n-butyl acetate, chloroform, and benzene were less successful. In experiments using benzene too much acetic acid went over with the water but, in a technical process when a fractionating column is included before the water trap, benzene could possibly prove preferable.

This particular method of converting aqueous to non-aqueous solutions avoids evaporation to dryness and may be of general interest. The economy of the procedure may be enhanced by partial evaporation before the entrainer, or even before the main organic solvent is added.

3.2 Solubility of Beryllium Oxyacetate

Generally speaking, solubilities of inorganic compounds are lower in the acetic acid solvent system than in aqueous solutions, since dissociation is lower and solvation of the ions is also less pronounced. With regard to acid-base equilibria, a hydrogen ion — acetate ion balance exists where the latter shows a close analogy to the hydroxyl ion in aqueous systems. References to investigation of this solvent system are given by Audrieth and Kleinberg (1953). Almost all sulphates are insoluble in acetic acid, while sodium nitrate is not very soluble and barium nitrate is completely insoluble.

Beryllium oxyacetate solubilities listed in Table 2 are much lower than those reported in the literature. This is believed to be due to a purer product and to longer crystallizing times used in our determinations. The increased solubility in the presence of sodium acetato shows that an anionic acetato — beryllium species must be postulated in acetic acid solutions of basic character. This is analogous to the beryllate ion in aqueous solutions. A similar analogy has been noted for zinc by Davidson (1931).

It follows, then, that in the process an excess of calcium acetate is to be avoided. It can also be seen that small quantities of water in the acetic acid will not affect the solubility greatly so that beryllium oxyacetate may be recrystallized from 98 per cent. acid without loss. For the measurement of the acidity function (Ho) in acetic acid a glass electrode was used and an ordinary HgCl reference electrode in which the KCl solution was replaced by a saturated solution of LiCl in acetic acid. This method was found to be useful for achieving a balance between calcium or sodium acetate on the one hand and nitric or sulphuric acid on the other. Such a balance was important in view of the soluble anionic acetato — beryllium complex reported here.

3.3 Hydrolysis of Beryllium Oxyacetate

According to Quinet (1944) the hydrolysis of beryllium oxyacetate proceeds through a neutral water-soluble complex Be 4O 3 (CH 3 COO) 2 which results from the reaction:

$$Be_4O(CH_3COO)_6 + 2H_2O \longrightarrow Be_4O_3(CH_3COO)_2 + 4 CH_5COOH(1)$$

The conductance data in Figure 5 show that the soluble hydrolysis product is practically identical with the metathetically prepared "beryllium acetate" solutions used by Sidgwick and Lewis (1926). In fact, the present process is based on this identity of metathetically prepared basic beryllium acetate solutions with the corresponding hydrolysates of beryllium oxcyacetate. This view was reinforced when Be was extracted from an aqueous solution containing 0.25 M Be, 1.5 M acetic acid, and 6 M total acetate into chloroform. However the quantity extracted amounted to only 12 per cent.

Comparison of the conductance values shown in Figure 5 with those of other acetates (Landolt-Bornstein 1960) shows quite pronounced ionic association already in 0.01N solution. At this concentration the equivalent conductance is only about half that of corresponding Mg, Ca, Ba, Sr, or Zn values. At higher concentration the difference becomes even more definite. Only Zn shows somewhat similar behaviour at these higher concentrations. The similarity seems significant, since both metals can form a tetrahedral oxygen complex. Whilst some ionic association is evident in beryllium

acetate solutions, the measured conductances are much too high for accordance with Equation 1. In this equation only acetic acid is capable of conducting. The very small difference in conductance made by the addition of acetic acid shows clearly that acetic acid is not responsible for the main portion of current transport. We conclude then that if the complex Be₄O₃(CH₃COO)₂ exists at all, it must have been hydrolysed further under the conditions used.

The earlier stages of hydrolysis can be discussed by reference to Table 3. The figures clearly show that substantial hydrolysis takes place in the presence of 1 molar water in acetic acid. These are very mild conditions. If Equation 1 was valid for this system, the net effect of the reaction on conductance would be equal to that of water removal, since the two neutral beryllium complexes are non-conductive. The very strong increase in specific conductance with beryllium concentrations listed in the last column of the table shows clearly that the "soluble neutral complex" is again considerably hydrolysed. If such a complex hydrolyses readily in almost pure acetic acid, then surely it cannot be an important species in the much more radical hydrolysis in pure water.

These views gain further support from distribution measurements made in this laboratory. The rate of increase of the γ activity in water was measured, after shaking with a chloroform solution of ${}^7\text{Be}_4\text{O(CH}_3\text{COO)}_6$ plus inactive ${}^8\text{Be}_4\text{O(CH}_3\text{COO)}_6$ carrier at 25 ${}^\circ\text{C}$. Addition of acetic acid increased the reaction rate, while an acetate buffer of pH = 4.5 slowed it down considerably.

It has been established that the hydrolysis of beryllium oxyacetate leads to ionic species, as one would expect, and that no neutral complex is in evidence. The anionic hydrolysis product is almost certainly the acetate ion. The composition of the first cationic hydrolysis product has not been fully established, but the form:

$$Be_4O(CH_3COO)_6 + H_2O \rightleftharpoons 2 Be_2(OH) CH_3COO^{2+} + 4 CH_3COO^{-}$$

for Equation 1 would be more in accordance with our findings.

3.4 Fission Product Chemistry

For a full investigation of this process it would be necessary to follow the fate of each fission product in the process and to learn something about its acetate chemistry. The present study has been confined to identification of the main residual fission products (106 Ru and 95 Zr) by their γ spectrum, as measured by a 256 – channel analyser. Some 144 Ce was also present, but by far the highest contamination stemmed from the ruthenium. Decontamination factors were at first calculated from β – counts, but as the less cumbersome and more reproducible γ – counting gave so closely similar values it was adopted for the calculations of gross decontamination factors in the remainder of the experiments.

Preliminary work with zirconium nitrate put through a similar process gave products which dissolved readily in water. However, the products were not well characterized and they appeared to lose acetic acid in both moist and dry air. It seems doubtful that a well-defined zirconyl oxyacetate exists.

4. CONCLUSIONS

- (i) The work reported shows the chemical feasibility of a process on the laboratory scale for the recovery and decontamination, via the oxyacetate, of beryllium from process solutions containing fission products. In batch wise counter-current operation, yields of over 90 per cent. and decontamination factors better than 3000 were obtained at activity levels up to 3 β-curies/mole BeO.
- (ii) The use of azeotropic distillation to produce the beryllium oxyacetate directly from aqueous solutions without the need of precipitation, separation, and drying of the beryllium hydroxide may be advantageous for the acetate route used in the production of beryllia from ore.
- (iii) It has been shown that the hydrolysis of beryllium oxyacetate does not give rise to a water soluble neutral complex, but forms ionic species. Evidence of an anionic acetato beryllium complex

was found in acetic acid solution. In aqueous solutions a cationic acetato - beryllium complex is postulated, but not positively identified.

8. ACKNOWLEDGEMENTS

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TABLE 1

LOW ACTIVITY LEVEL EXPERIMENTS - SUMMARY OF RESULTS

	ſ		LAT ACTIVITY LEVEL BAFERIMEN IS - SUMMARY OF RESULTS	10 - 50 M	MARIO	KESOLIS
Experiment	Run	Procedure (see Section 2.1)	Recrystallization	D.F.	Yield	Remarks
RECOVERY FROM	V	Addition of sodium acetate and removal of sodium nitrate. Thence crystallization from an anhydrous system after azeotropic distillation with n-butyl acetate.	Twice from benzene.	3,000%	70%	Oxyacetate products in all runs were washed with acetic acid, chilled water and ether.
Be(NO 3) 2	æ	As above, except that the azeotropic distillation was	Twice from chloroform.	3,500%	74%	
	С		Twice from acetic acid.	3,8007	80%	
RECOVERY	Q	Addition of barium acetate and removal of barium sulphate. Thence crystallization from acetic acid after azeotropic distillation with n - propyl acetate.	None	4007	206	
FROM BeSO 4	珂	Addition of calcium acetate and removal of calcium sulphate. Thence as in D,	Мопе	6007	86%	
	ſĿ,	•	None	500%	85%	A preliminary decontamination with ferric
	Ö	In runs F and G the azeotropic distilla-Once from acetic acid	Once from acetic acid and once from chloroform.	2,700%	80%	nifate and potassium permanganate was used for rups E to H. In run H the HNOs distilled for slowly due to insufficient
RECOVERY FROM Be(NO 3)2 RY EVA-PORATION WITH HALF MOLE EQUIVALENT OF H2SO 4		Addition of calcium acetate and removal of calcium sulphate. Thence crystallization from acetic acid after azeotropic distillation with n - propyl acetate.	None	8607	81%	H ₂ SO ₄ .
	5	ម្រី	None	4807 520 <i>8</i>	82%	
,		of carcium supporte. Inches crystalliza- tion from accetic acid after azeotropic	Twice from acetic acid	2,200%	%08	Batch countercurrent use of recrystalliza-
Be(NO ₃) ₂ BY CON	1	distillation with n-propyl acetate.	Twice from acetic acid	2,2007	2598	A preliminary decontamination was used
VERSION TO	٤		Twice from acetic acid	2,8607	806	in tuns L and M.
BeSO 4	z	Addition of calcium acetate and removal	Twice from acetic acid	3,100%	83%	Batch countercurrent, use of recrystalliza-
	Д	or calcium sulphate tollowed by addi- tion of barium acetate and removal of	Twice from acetic acid	3,2007	88%	tion mother liquors in runs N. P and Q. A preliminary decontamination with potassium
	Q!	barium sulphate. Thence crystallization from acetic acid as above.	Twice from acetic acid	3,8007	92%	permanganate only was used in run N. No preliminary decontamination used in runs P, and Q.

TABLE 2

SOLUBILITY OF BERYLLIUM OXYACETATE IN

VARIOUS SOLVENTS AT 25 °C

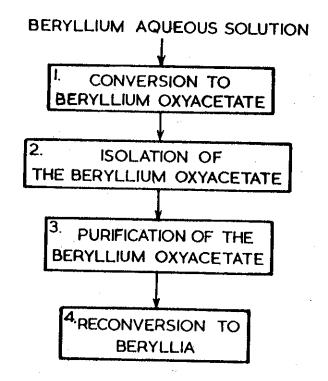
Salman Carrier	Solubility in g/100g Solution_		
Solvent Composition	on heating to 25°C	on cooling to 25°C	
Acetic acid	0.81	0.82	
Acetic acid containing 8.2% sodium acetate	2.41	2.58	
Acetic acid containing 1M H 2O	0.84	0.85	
Acetic acid containing 10MH ₂ O	3.9	4.6	
Acetic acid containing 30MH 20	20	20	
Acetic anhydride	1.0	1.0	
Water (after hydrolysis)	35.2	36.5	
n – Propyl acetate	0.76	0.80	
n – Hexane	0.008	0.008	
Kerosene	0.006	0.006	

TABLE 3

SPECIFIC CONDUCTANCES (*) AND EQUIVALENT CONDUCTANCES (A) OF

BERYLLIUM OXYACETATE IN ANHYDROUS AND AQUEOUS ACETIC ACID

	Ba(CH ₃ COO) ₂	Be 4 O (CH 3COO) ₆		
Normality (metal)	Normality $\kappa \times 10^8 \Omega^{-1} \text{cm}^{-1}$		$\kappa \times 10^8 \Omega^{-1} \rm cm^{-1}$	
($\Omega^{-1} \operatorname{cm}^{2} \operatorname{mole}^{-n}$	anhydrous	1 molar H ₂ O
0.1	760	0.0018	19	560
0.01	100	0.0076	8.6	196
0.001	22	0.0271	3.66	87
Pure Acid	_	_	0.95	18.5



THE CHOSEN PROCESS USES

- I. AZEOTROPIC DISTILLATION
- 2. CRYSTALLIZATION OF BERYLLIUM OXYACETATE
- 3. RECRYSTALLIZATION OF BERYLLIUM OXYACETATE
- 4. HYDROLYSIS TO BERYLLIUM HYDROXIDE FOLLOWED BY CALCINATION TO OXIDE

FIGURE I THE MAIN STEPS IN THE RECOVERY OF BERYLLIUM VIA THE OXYACETATE

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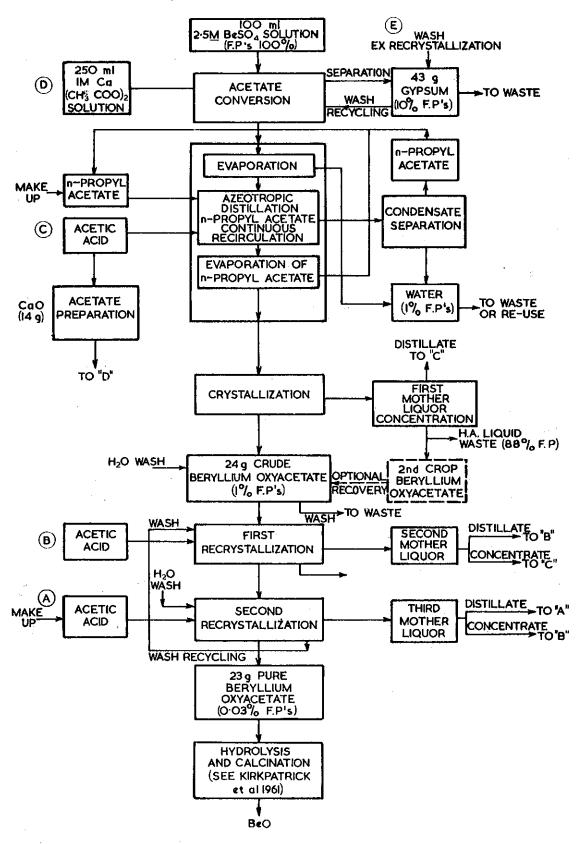


FIGURE 2

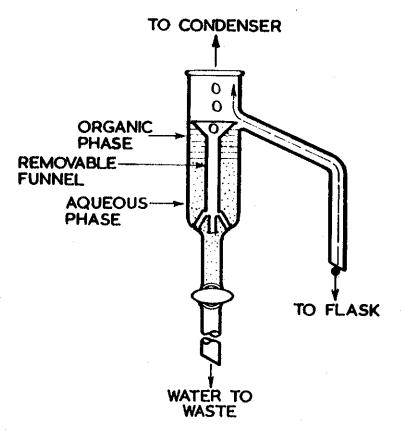


FIGURE 3 KOELSCH WATER TRAP

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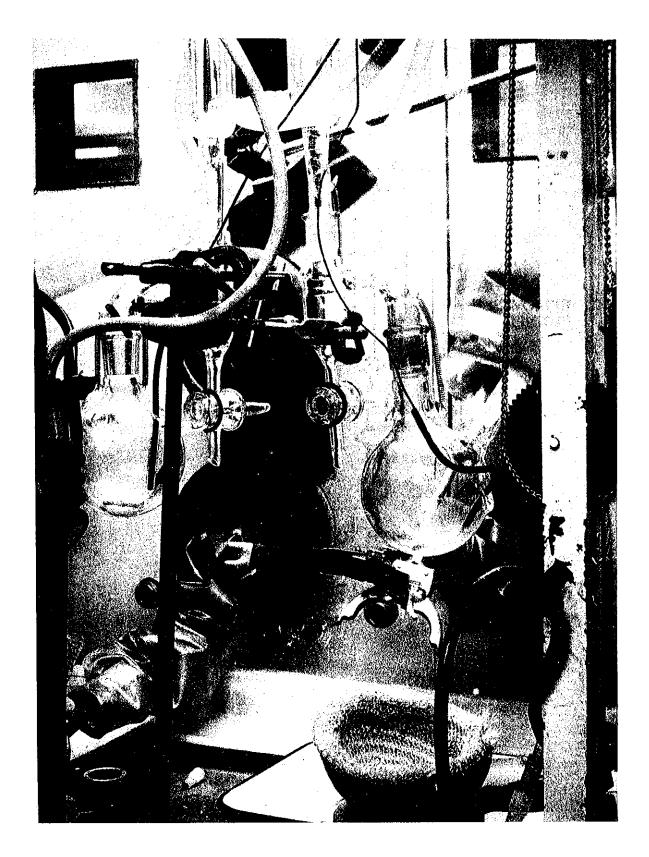
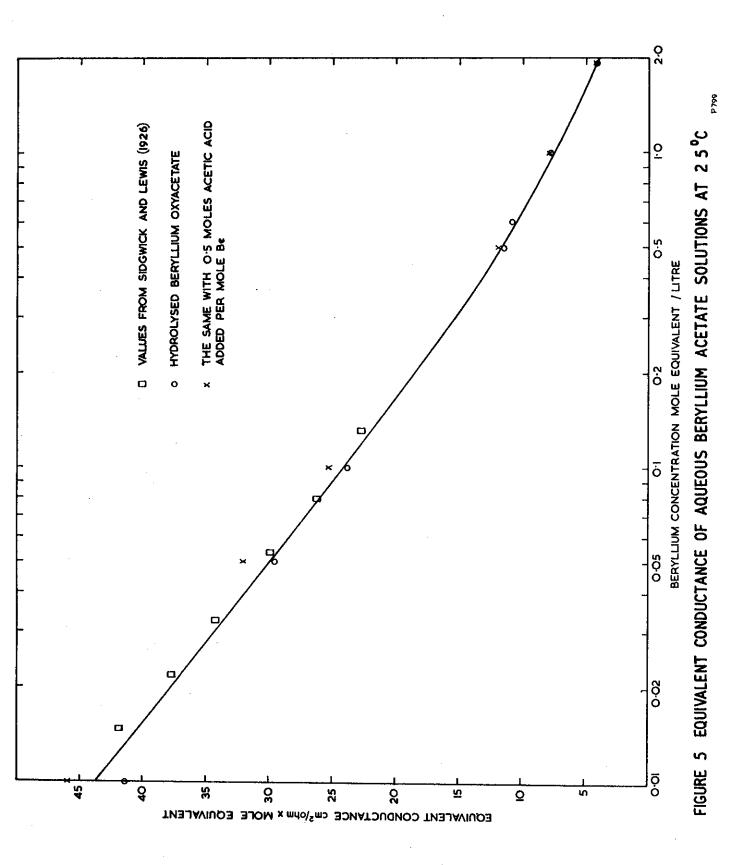


FIGURE 4. PART VIEW OF CELL



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