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PREPARATION OF PITCH-SOLUBLE URANYL-ORGANIC COMPOUNDS

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ABSTRACT

Batch processes on a scale of 250 to 300 g of uranium were developed for the production of uranyl oxinate (8-quinolinolate) and uranyl malonate. Both compounds are insoluble in water and were found to be suitably soluble in pitch. Uranyl oxinate was prepared by the reaction of an aqueous uranyl nitrate solution with an acetic acid solution of oxine (8-quinolinol) at about 80°C. Complete precipitation was accomplished by the addition of ammonium hydroxide. Yields of better than 99.5% were obtained. Uranyl malonate was prepared by the reaction of aqueous solutions of sodium malonate and uranyl nitrate at about 80°C in 97 to 98% yield.

Uranyl 2-ethylhexanoate was prepared by a trans-esterification reaction from uranyl acetate and 2-ethyl-hexanoic acid. Yields of 90% were obtained but the process was quite laborious and time consuming. A metathesis method of preparation was not successful.

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TABLES

1. INTRODUCTION

In development work at LASL on the fabrication of uranium-loaded graphite reactor fuel elements, the introduction of the uranium by addition of pitch-soluble uranyl-organic compounds to molten pitch, which is then coked, ground, and mixed with other ingredients, has been investigated. The preparation of a number of uranyl-organic compounds was studied, with the aim of finding pitch-soluble compounds which could be easily prepared in good yield on an adequately large scale. In this study, preparation on a scale of 250 to 300 g of uranium was set as a goal, for possible eventual use in batch-type operations with fully enriched uranium.

At the time this work was started, a laboratory procedure for the preparation of uranyl 2-ethylhexanoate, a pitch-soluble compound, by transesterification had already been developed.[1] Attempts to adapt this procedure to production scale met with only limited success, in that the procedure was laborious and yields were not as high as desired. Attempts to prepare this compound by metathesis from aqueous salt solutions were unsuccessful.

Of a number of other compounds investigated, uranyl oxinate (8-quinolinolate) and uranyl malonate, both pitch-soluble, appeared to be the most promising for production scale preparation. Satisfactory procedures were developed for the preparation of both these compounds on a scale of 250 to 300 g of uranium.

The pitches used in this work were Resin C, a pitch distillate with a 35% coking yield, and Barrett's Pitch No. 30 MHV, a pitch residue with a 55% to 65% coking yield. Both pitches melted at about 90°C and were stable to about 200°C.

2. URANYL 2-ETHYLHEXANOATE

2.1 Transesterification Method

2.1.1 Materials and Equipment

The 2-ethylhexanoic acid was obtained from the Tennessee Eastman

Company, Kingsport, Tennessee. The specifications furnished with the acid were: specific gravity – between 0.905 and. 0.909 at 20°/20°C; acidity – not less than 98.0% as 2-ethylhexanoic acid; boiling range – initial not below 223°C, dry point not above 233°C. Analysis of the acid gave the following results: specific gravity – 0.904 at 25°C; acidity – 98.5% ± 0.5%; boiling range – initial 221°C, dry point 226°C. The acid, which was a colorless, odorless liquid, was used as received. The uranyl acetate dihydrate, xylene, and hexane were all reagent grade materials.

The reactions were carried out in three-neck, round-bottom flasks of the appropriate volume. The flasks were fitted with a thermometer, a mercury-sealed stirrer, a Claisen distilling head, and a take-off condenser. Heating of the flask and the reaction mixture was done with a Glas-Col mantle.

2.1.2 Procedure and Results

Best results were obtained with a modification of a procedure which had been developed by W. H. Stein of this Laboratory.[1] Uranyl 2-ethylhexanoate was prepared by the reaction:

$$2CH_3(CH_2)_3\underset{\displaystyle C_2H_5}{\underset{|}{C}}HCOOH + UO_2(C_2H_3O_2)_2\cdot 2H_2O \longrightarrow [CH_3(CH_2)_3\underset{\displaystyle C_2H_5}{\underset{|}{C}}HCOO]_2UO_2 + 2CH_3COOH + 2H_2O$$

The uranyl acetate, xylene, and a 2 to 5% excess of the acid were placed in the flask. To hasten the reaction, the mixture was distilled with stirring at atmospheric pressure at 95° to 110°C to remove the water and the acetic acid as it was formed. Since xylene was also distilled off during this time, additional xylene was added to the reaction mixture, either intermittently or continuously, to maintain a relatively constant volume. On completion of the reaction as determined by analysis for acid in the distillate, the solution was cooled to room temperature and mixed with decolorizing charcoal for 1 to 2 hours. The mixture was then filtered and the filtrate was distilled under vacuum to remove the solvent and excess 2-ethylhexanoic acid. The presence of excess acid interferes with the crystallization of the uranyl 2-ethylhexanoate.

Care had to be taken during distillation both at atmospheric pressure and under vacuum to keep the temperature below 110°C or excessive decomposition took place. The residue obtained after vacuum distillation was a crystalline, semisolid mass. This residue was triturated with hexane to

yield the product as a pale yellow crystalline solid melting at 234° to 235°C. It was soluble in warm benzene, toluene, and xylene, partially soluble in warm hexane, and insoluble in water.

Runs made on a scale of 0.1 to 0.2 mole of uranium gave first crop yields of about 90%. One run made on a scale of 0.1 mole of uranium gave a first crop yield of 89.4% and an over-all yield of 98.7% after two recrystallizations. It was not necessary to decolorize the solution on this scale.

However, when runs on a 1 mole scale were made using 2 liters of solvent per mole of uranium, the time necessary to bring the reaction to 95% completion rose from 7 hours for a 0.1 mole run to 10 to 15 hours, and the over-all yield dropped to 90%. A complete run, including two recrystallizations, required 2 to 3 days.

Because of the length of time required to complete a run on a 1 mole scale, the excessive care required in the operation to prevent decomposition, the fairly high loss of uranium (about 10%), the difficulty in recovering this uranium, and the large amounts of solvent (7 to 8 liters) used during a run, it was decided that this process was not adaptable to production. Consequently, a different method for the possible preparation of uranyl 2-ethylhexanoate was investigated.

2.2 Metathesis Method

2.2.1 Materials and Equipment

The 2-ethylhexanoic acid and solvents used were the same as those used in the transesterification method. The sodium hydroxide was prepared from carbonate-free 1M Stansol solutions and carbonate-free distilled water. The uranyl nitrate solutions were prepared by dissolving weighed amounts of reagent grade $UO_2(NO_3)_2 \cdot 6H_2O$ in distilled water.

All precipitations were carried out in open beakers. Stirring was done with either Mag-Mix stirrers and Teflon-covered magnetic stirring bars or by an air-driven stirrer motor with a glass stirring rod.

2.2.2 Procedure and Results

Since uranyl 2-ethylhexanoate is insoluble in water, it was hoped that it could be prepared by the metathesis of uranyl nitrate and sodium or ammonium 2-ethylhexanoate in aqueous solution according to the reaction:

$$UO_2(NO_3)_2 + 2CH_3(CH_2)_3\underset{\displaystyle C_2H_5}{\underset{|}{C}}HCOONa \longrightarrow [CH_3(CH_2)_3\underset{\displaystyle C_2H_5}{\underset{|}{C}}HCOO]_2UO_2 + 2NaNO_3$$

The sodium and ammonium salt solutions were prepared by the addition of the stoichiometric quantity of sodium or ammonium hydroxide to 2-ethylhexanoic acid with warming and stirring. The precipitations were carried out by the slow addition of either the uranyl nitrate solution to the sodium or ammonium salt solution with vigorous stirring or the reverse. Precipitations were carried out at 0°, 25°, and 60°C in aqueous solutions only and also at 25° and 60°C in the presence of benzene. The precipitates obtained were purified by washing with water and hexane or by recrystallization from toluene.

The precipitates obtained at room temperature were all waxy gels. When the solid was formed by the addition of the uranyl nitrate solution to the sodium salt solution, the gel agglomerated into balls as large as 1 inch or more in diameter. When the precipitations were made at 60°C or in the presence of benzene at 25° or 60°C, stable emulsions which did not break spontaneously in 24 hours resulted.

The precipitate formed at 0°C was fairly powdery and easily filtered. However, additional solid formed in the filtrate as it warmed to room temperature.

In almost all cases the solid obtained after the original precipitate had been treated with water and cold hexane either melted at 90° to 100°C or did not melt up to 250°C. Analyses of these materials indicated that they were mixtures, usually of $Na_2U_2O_7$ and probably some combination of uranyl 2-ethylhexanoate with other components present.

3. URANYL OXINATE

Uranyl oxinate is the common name of the red compound formed by the reaction between an aqueous solution of uranyl nitrate and a solution of oxine (8-quinolinol) at controlled pH according to the reaction:

$$UO_2(NO_3)_2 + 3C_9H_6NOH \longrightarrow UO_2(C_9H_6NO)_2 \cdot C_9H_6NOH + 2HNO_3$$

Uranyl oxinate (8-quinolinolate) has been reported to be an addition compound.[2,3] However, recent work[4] has shown it to be the acid $H[UO_2(C_9H_6NO)_3]$. The compound, which is somewhat soluble in organic solvents, is reported to

be stable to 157°C, where it starts to lose one molecule of oxine. Uranyl oxinate was found to be suitably soluble in pitch.

3.1 Materials and Equipment

The uranyl nitrate, glacial acetic acid, ammonium hydroxide, and oxine (8-quinolinol) used were all reagent grade materials.

The precipitations were carried out in a 5-liter flask fitted with a thermometer and an air-driven stirrer and heated with a Glas-Col mantle. The solutions were added through the top of the flask from 500-ml dropping funnels.

3.2 Procedures

Two satisfactory methods of preparation were developed. The preferred method is given first.

Method 1. Dissolve 527 g (1.05 moles) of uranyl nitrate hexahydrate (UNH) in water and dilute to 2 liters. Place in a 5-liter flask fitted with a mechanical stirrer and heat to 80° to 85°C with a Glas-Col mantle with stirring. Dissolve 526 g (3.15 moles plus 15% excess) of oxine in 600 ml of warm (50° to 60°C) glacial acetic acid and dilute to 1500 ml with warm water. This dilution serves only to reduce the viscosity of the solution and would not be necessary in production provided the solution is not added too rapidly and efficient stirring is maintained. The oxine solution at 50° to 60°C is added with good stirring to the UNH solution, with the temperature of the reaction mixture being maintained at 80° to 85°C. After the addition of the oxine solution is complete, 450 ml of concentrated NH_4OH is slowly added with vigorous stirring. Caution: This part of the reaction is highly exothermic. Filter the hot mixture with suction on a No. 2 Whatman paper in a Buchner funnel. Wash thoroughly with five or six 1-liter portions of ice water. Dry at 120° to 130°C.

Method 2. Make up the UNH and oxine solutions as in Method 1. Add 300 ml of the oxine solution at 50° to 60°C to the UNH solution with vigorous stirring, maintaining the temperature at 80° to 85°C. Add concentrated NH_4OH to raise the pH to 3. Add the rest of the oxine solution with stirring, maintaining the pH at 3 by addition of NH_4OH and the temperature at 80° to 85°C. After addition of the oxine is complete, add NH_4OH to bring the pH up to 4.1 or slightly higher. Filter, wash, and dry as above.

3.3 Results

Nine full scale (250 g of uranium) precipitations of uranyl oxinate were

made using Method 1. The per cent yield (based on the weight of dried material obtained), losses in the filtrate and washes, and analyses of the product are given in Table 1.

TABLE 1

YIELDS AND LOSSES IN PRECIPITATION OF URANYL OXINATE

		Uranium Loss, %			Composition by Analysis,[b] wt. %			
Run	Yield, %	In Filtrate	In Wash[a]	Total	U	C	H	N
O-9	99.9	-	-	0	34.6	46.3	2.9	5.7
O-10	99.9	-	-	-	36.3	46.2	2.9	6.0
O-11	99.4	0.03	0.01	0.04	34.7	46.2	3.0	5.8
O-12	100.3	0.02	0.02	0.04	35.4	46.2	2.8	5.8
O-13	100.0	0.02	0.01	0.03	34.8	46.0	2.9	5.7
O-14	100.5	0.02	0.002	0.02	33.8	46.4	3.0	5.9
O-15	99.9	0.008	0.003[c]	0.01	33.9	46.5	3.0	5.9
O-16	99.9	0.02	0.002	0.02	33.9	46.3	3.0	5.8
O-17	100.2	0.014	0.004	0.02	33.9	46.5	3.0	5.8
Average	100.0	0.02	0.007	0.03	34.6	46.3	2.9	5.8

(a) Five 1-liter portions

(b) Theoretical for $UO_2(C_9H_6NO)_2 \cdot C_9H_6NOH$ is: U, 33.8%; C, 46.1%; H, 2.7%; N, 6.0%.

(c) Six 1-liter portions

4. URANYL MALONATE

Uranyl malonate is reported to be easily formed from aqueous solution as a bright yellow crystalline powder according to the reaction:

$$3H_2O + CH_2(COO)_2Na_2 + UO_2(NO_3)_2 \longrightarrow CH_2(COO)_2UO_2 \cdot 3H_2O + 2NaNO_3$$

Mueller[5] reports the malonate as a dihydrate. However, Fay[6] says it is a trihydrate, losing two molecules of water at 110°C and the third at 180°C. Our work as shown below agrees with that of Fay. Uranyl malonate trihydrate was found to be suitably soluble in pitch.

4.1 Materials and Equipment

The sodium hydroxide, malonic acid, and uranyl nitrate hexahydrate (UNH) used in this work were all reagent grade.

The precipitations were carried out in open beakers. Stirring was done with air-driven stirrers using glass stirring rods. Heating was done either on a steam plate or on an electric hot plate.

4.2 Procedure

The procedure developed for making large scale (300 g of uranium) precipitations of uranyl malonate is as follows:

Dissolve 100.7 g of NaOH (2.52 moles) in 700 ml of H_2O. Add this solution slowly to 131 g of malonic acid (1.26 moles) and heat to about 80°C. Dissolve 633 g of UNH (1.26 moles) in 1 liter of H_2O. (Volumes of 750, 1000, and 1300 ml were used with little, if any, effect on the yield, the largest yield being obtained with a volume of 1000 ml.) Heat the solution to about 80°C. Add the UNH solution to the sodium malonate solution with a little stirring. This may be done quite rapidly. Digest the precipitate about 2 hours at 70° to 80°C. Allow the precipitate to stand at room temperature overnight. Chill 4 to 5 hours. Filter with suction and wash well with ice water. Dry at 110°C to obtain the monohydrate.

4.3 Results

The question of whether a dihydrate or a trihydrate that decomposed to a monohydrate at 110°C was formed was resolved by dividing a fresh precipitate of uranyl malonate in half, drying one portion at 110°C and the other by aspiration and then analyzing and comparing the analyses with the theoretical compositions of the mono-, di-, and trihydrates. The results of these experiments, given in Table 2, show quite clearly that the trihydrate is formed on precipitation and that it decomposes to the monohydrate on heating at 110°C.

TABLE 2

COMPOSITION OF URANYL MALONATE AS PRECIPITATED AND ON DRYING AT 110°C

	Theoretical Composition, wt. %			Analyses of Precipitate, wt. %	
	$C_3H_2O_6U \cdot 3H_2O$	$C_3H_2O_6U \cdot 2H_2O$	$C_3H_2O_6U \cdot H_2O$	Aspirated	Dried at 110°C
U	55.86	58.32	61.0	55.85	60.8
C	8.45	8.83	9.2	8.40	9.15
H (org. + $1H_2O$)	0.95	0.98	1.0	1.0	1.1
H (total)	1.89	1.48	1.0	1.95	1.1
H_2O (at 110°C)	8.45	4.41	-	8.7	-

Precipitations made to study the effect of addition procedure and of excess sodium malonate indicated that the mode of addition is immaterial but that the use of an excess of sodium malonate reduces the yield. Use of the stoichiometric amount of sodium malonate normally gave yields of 97%, whereas a 15% excess of malonate gave a yield of 91%.

When the precipitation was made at room temperature, the solid was slow to form and adhered quite strongly to the sides and bottom of the container.

Four precipitations were made to study the effect of digestion and chilling on the yield. The results are shown in Table 3 and indicate that a slight increase in yield is to be gained by both digestion at 70° to 80°C and chilling prior to filtration.

Four full-scale precipitations were made as described in Section 4.2. Preparations 13 and 14 were washed with six 500-ml portions of ice water, and preparations 15 and 16 were washed with six 500-ml portions of water at 25°C. All were chilled 4 hours prior to filtration except 16, which was chilled 2 hours. The yields and losses in the filtrate and wash are given in Table 4.

The primary purpose of the water wash is to remove sodium ion from the uranyl malonate. In these runs the amount of sodium present in the dried material was 0.04 to 0.05%.

TABLE 3

EFFECT OF DIGESTION AT 70° TO 80°C AND SUBSEQUENT CHILLING ON YIELD OF URANYL MALONATE

Run	Stand Overnight	Digest	Chill	Yield, %
5	No	4 hr	No	96.1
6	No	No	No	94.7
7	Yes	4 hr	15 hr	96.3
8	No	No	5 hr	95.2

TABLE 4

YIELDS AND LOSSES IN PRECIPITATION OF URANYL MALONATE

	Uranium Loss, %		
Run	In Filtrate	In Wash	Yield, %
13	2.0	0.30	97.1
14	1.8	0.36	97.8
15	1.6	0.49	97.6
16	2.2	0.49	96.5

5. OTHER COMPOUNDS

The uranyl salts of succinic, tannic, benzylmalonic, cinnamic, α-naphthoic, and β-naphthoic acids, and of phenol, α-naphthol, and β-naphthol were prepared by a method given by Mueller.[5] This consists of adding NaOH to the organic compound in water to prepare a solution of the sodium salt and then adding an aqueous solution of uranyl nitrate to precipitate the uranyl compound.

All the precipitations were made hot. The precipitates of uranyl α-naphtholate and uranyl β-naphtholate were chilled and then filtered. All the others were allowed to stand at least overnight and in some cases over a week end before filtering. The uranyl salts of benzylmalonic, cinnamic,

α-naphthoic and β-naphthoic acids were digested on a steam plate for 1 to 4 hours.

The uranyl salt of naphthenic acid (a mixture of acids from petroleum distillates) was also prepared in a manner similar to that given above, except that the precipitation was carried out at 0°C. The directions for preparation were supplied by R. P. Hammond of this Laboratory.[7]

All the salts were dried by aspiration on a filter for at least 18 hours after having been washed with water and in some cases with ethanol and ether.

Table 5 lists these compounds with their theoretical compositions, the analyses of each, per cent yield, and loss of uranium in the combined filtrate and water wash. All were either completely or partially insoluble in water and soluble in the usual organic solvents such as ether, ethanol, and benzene, except uranyl succinate which was insoluble in ethanol and ether. Uranyl benzylmalonate and uranyl β-naphthoate were found to be suitably soluble in pitch. The solubility of the other compounds in pitch was not tested.

The percentage compositions were based on the weight of the air-dried salt. Water was determined by measuring the weight loss incurred by heating for 2 hours at 100°C. The hydrogen was measured first by burning the air-dried sample to obtain the total hydrogen and second by burning the material dried at 100°C to obtain the hydrogen from the organic part of the molecule plus that from any water of hydration which was not driven off at 100°C. Uranium in the filtrates was determined colorimetrically.

6. SUMMARY

Uranyl 2-ethylhexanoate has been prepared by a transesterification reaction between uranyl acetate and 2-ethylhexanoic acid in xylene. The reaction was carried out at temperatures of 95° to 110°C. Although over-all yields of 90% were obtained on a 1 mole scale, the process was quite laborious and time consuming and required considerable care to prevent decomposition.

Processes have been developed for the batch production of pitch-soluble uranyl oxinate (8-quinolinolate) and uranyl malonate on a scale of 250 to 300 g of uranium. Uranyl oxinate was precipitated by the addition of an acetic acid solution of oxine (8-quinolinol) to an aqueous solution of uranyl nitrate, followed by the addition of ammonium hydroxide, all at 80°C. Yields of better than 99.5% were obtained. Uranyl malonate was precipitated with 97 to 98% yield by the addition of an aqueous solution of uranyl nitrate to an aqueous solution of sodium malonate at about 80°C.

TABLE 5

OTHFR COMPOUNDS

Compound	Wash	Color	Calc Yield %	Filtrate Loss of U %	Formula	Theoretical Composition, wt %					Composition by Analysis, wt %				
						U	C	Org H	Total H	H_2O	U	C	Org H	Total H	H_2O
Uranyl napthenate[a]	Ice water	Yellow	-	26 7	-	-	-	-	-	-	31 2	43 8	-	6 0	2 8
Uranyl succinate	Ice water ethanol ether	Light yellow	90 1	3 9	$C_4H_4O_6U\ 2H_2O$	56 4	11 4	0 96	1 9	8 5	57 9	10 3	1 7	2 0	2 7
Uranyl phenolate[b]	Hot water ethanol	Yellow	58 6	7 9	$C_{12}H_{10}O_4U\ 2H_2O$	48 4	29 3	2 1	2 9	7 3	74 9	0 1	0 7	1 3	5 0
Uranyl tannate[c]	Ice water	Brown	52 2	16 7	$C_{28}H_{18}O_{20}U\ 15H_2O$	20 1	28 4	1 5	4 1	22 8	31 0	26 5	1 9	3 1	11 0
Uranyl benzyl-malonate	Ice water	Light yellow	94 0	1 4	$C_{10}H_8O_6U$	51 5	26 0	1 7	1 7	-	51 3	26 0	1 7	1 8	-
Uranyl cinnamate	Hot water	Lemon yellow	76 7	11 3	$C_{18}H_{14}O_6U$	42 2	38 3	2 5	2 5	-	40 7	38 4	2 5	2 5	-
Uranyl α-naphtholate	Ice water	Violet-black[d]	84 4	14 5	$C_{20}H_{14}O_4U\ 3\ 5H_2O$	38 4	38 8	2 3	3 4	10 2	37 8	40 9	2 9	3 4	4 8
Uranyl β-naphtholate	Ice water	Yellow-tan[d]	91 9	6 7	$C_{20}H_{14}O_4U\ 2H_2O$	40 2	40 6	2 4	3 1	6 1	42 1	35 9	2 5	3 1	5 4
Uranyl α-naphthoate	Ice water	Yellow	84 2	4 6	$C_{22}H_{14}O_6U\ 3H_2O$	35 7	39 6	2 1	3 0	8 1	38 9	42 5	2 4	2 5	0 6
Uranyl β-naphthoate	Ice water	Yellow	98 1	0 8	$C_{22}H_{14}O_6U\ H_2O$	37 8	41 9	2 2	2 6	2 9	38 4	41 5	2 5	2 6	1 1

(a) Since naphthenic acid is a mixture of acids of unknown composition it is impossible to determine the per cent yield or the theoretical composition

(b) Red filtrate Not investigated.

(c) Mueller[5] reports this compound to be easily soluble in water but Das Gupta[8] and Schoeller and Webb[9] have used it in quantitative analysis of uranium

(d) Mueller[5] reports the colors of these compounds as just the opposite

The uranyl salts of succinic, tannic, benzylmalonic, cinnamic, α-naphthoic, and β-naphthoic acids and of phenol, α-naphthol, and β-naphthol were also prepared on a small scale.

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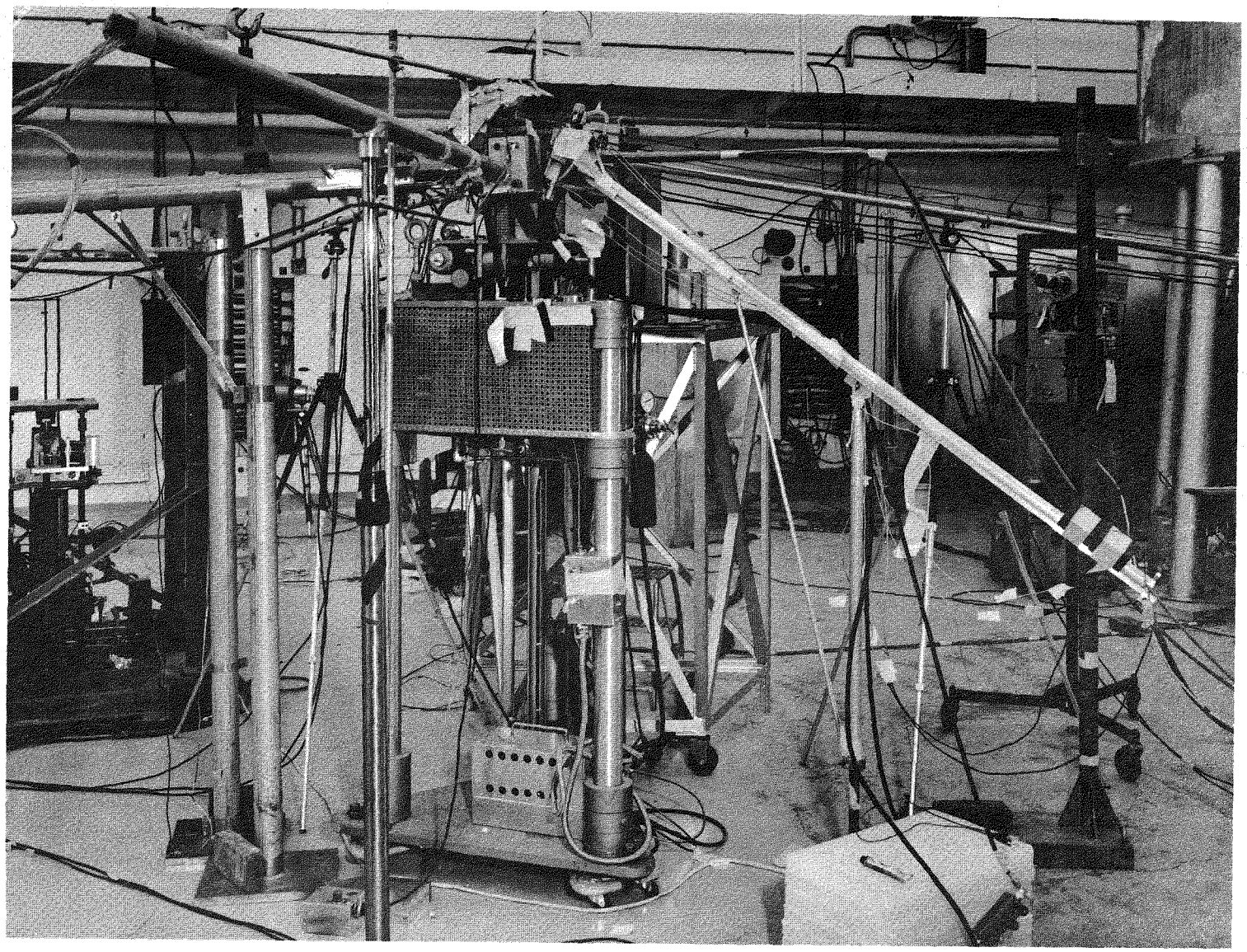

Fig. 1. General View of Setup for Radiation Effects Experiments.