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AEC RESEARCH AND DEVELOPMENT REPORT

SEPARATION OF URANIUM (VI) FROM OTHER METALS BY FILTER PAPER PARTITION CHROMATOGRAPHY WITH 2-METHYLTETRAHYDROFURAN AND ITS QUANTITATIVE DETERMINATION

I. Separation Studies by Use of Radioisotopes

H. P. Raaen
P. F. Thomason

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SEPARATION OF URANIUM(VI) FROM OTHER METALS BY FILTER PAPER PARTITION CHROMATOGRAPHY WITH 2-METHYLTETRAHYDROFURAN AND ITS QUANTITATIVE DETERMINATION

I Separation Studies by Use of Radioisotopes

H P Raaen and P F Thomason

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0 ABSTRACT

Radioisotopes were used in a study of the microseparation of uranium(VI) from thirty-two metals (thirty-three radioisotopes) by filter-paper partition chromatography with 2-methyltetrahydrofuran Autoradiography of the chromatograms established the efficiency of the separations The sorption gradient of the separated U^{233} was determined by alpha-count measurements of the chromatograms, the quantitative removal of U^{233} from the paper strip was demonstrated by the same technique Maximum condensation of the U^{233} band was effected by a 1 75-hr elution with H_2O-saturated (at 25°C) 2-methyltetrahydrofuran that contained 2 5% (v/v) conc HNO_3 The average R_f value for U^{233} was 0 95 Of the radioisotopes studied, Ru^{106} - Rh^{106}, W^{185}, and Hg^{203} were not separated from U^{233} The results for Sn^{113} and Sb^{124} were not conclusive The behavior of Hg^{203} was similar to that of U^{233} The alpha-count data are presented graphically, and positive prints of the autoradiograms are shown

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1 FOREWORD

Much of the work reported herein was included in ORNL-1358, of which only fifteen copies were prepared and submitted for declassification The deletions that declassification would have necessitated were numerous and significant, also, the work reported in ORNL-1358 has been extended to include nine additional radioisotopes For these reasons it was decided to issue this complete, classified report instead of the declassified report

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2 INTRODUCTION

There is a general need for a method of separation and quantitative determination of microgram and submicrogram amounts of uranium(VI) Such a method would find direct use in the analysis of samples of high radioactivity or low uranium concentration Filter-paper partition chromatography offers a separation method of high resolving power, which is likewise rapid, sensitive, adaptable, and reliable The maximum amount of a substance that may be handled successfully by paper chromatography is of the order of 1 mg, according to Burstall et al [8] The minimum amount depends upon the sensitivity of the technique for determining the locus of the sorbed solute and of the method for its estimation either while it is in place or after it is removed from the paper

The method of filter-paper partition chromatography using 2-methyltetrahydrofuran for the separation of uranium from other metals was first reported by Arden et al [2] They selectively extracted uranyl nitrate from its solutions by use of 2-methyltetrahydrofuran that contained aqueous nitric acid, carrying out the extraction on a strip of absorbent paper The chromatograms obtained were developed with potassium ferrocyanide reagent, and the uranium was estimated by visual comparison with standard stains produced with known quantities of uranyl nitrate The method was reported to be sensitive to as little as 0 1 µg of uranium and semiquantitative for 0 25 to 200 µg with an accuracy of ± 30% Lewis and Griffiths[17] extended the work of Arden et al [2] and reported the separation of uranium from fifty-two other metals and its quantitative polarographic determination However, the description of their experimental work is meager

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The use of radioactive tracers is the most sensitive technique for identifying the locus of a chromatographically resolved substance, being sensitive to as little as a few hundred atoms or molecules [21] Autoradiography or counting techniques can be used to indicate the distribution of the resolved radioisotope within its locus The radioactive tracer technique of paper chromatography combined with autoradiography has been used by Fink and Fink in biological studies with radioisotopes[9,10] and in determinations of traces of nonradioactive or weakly radioactive derivatives [11] In the latter work they attempted to determine natural uranium quantitatively on the chromatogram by the formation of a salt of uranium that contained a beta-emitting anion and by the subsequent measurement of beta radiation The method was not satisfactory

The use of radioactive tracers in paper partition chromatography of inorganic ions and the detection of their loci by autoradiography and counting techniques is also described by Frierson and Jones [12] However, uranium(VI) was not among the cations which they studied

The work reported herein is an application of the radioactive tracer technique, together with the techniques of autoradiography and alpha radiation counting, to the separation of microgram and submicrogram quantities of uranium from other metals by filter-paper partition chromatography with 2-methyltetrahydrofuran It is an extension of the work of Arden et al [2] and of Lewis and Griffiths[17] and supplements their reports with information on the identification of the uranium(VI) locus, the sorption gradient of the uranium, solvent phase separation on the filter-paper strip, acidity differentials along the strip, effect of the age of the eluting solvent, optimum conditions for

[redacted]

isolating and condensing the uranium band on the filter-paper chromatogram, the efficiency of the method for separating uranium from other metal ions, and the quantitative elution of uranium from the chromatogram

3 SUMMARY

The use of radioisotopes in the study of separations by filter-paper chromatography makes possible the application of the highly sensitive techniques of autoradiography and activity counting The separation of uranium(VI) from other metals by filter-paper partition chromatography with 2-methyltetrahydrofuran was evaluated by the use of U^{233} and radioisotopes of other metals Autoradiography and alpha-count measurements were used to obtain more exact information than that available[2,17] regarding the optimum composition of the eluting solvent, phase separation of the eluting solvent on the strip, optimum elution time, differential migration of the uranium, distribution of uranium within the uranium band, band width, and the efficiency of the separation of uranium from other metals

Maximum condensation of the uranium band was found to occur after a 1.75-hr elution with H_2O-saturated (49 mg per ml) 2-methyltetrahydrofuran that contained 2 5% (v/v) conc HNO_3 For quantities of 0 5 µg of U^{233} that were chromatographed on 0 75-in width strips of Whatman No 1 filter paper, the average R_f value for U^{233} was 0 95 and the average band width was 0 24 cm

A summary of the separation behavior of U^{233} and other radioisotopes is given in Table 1

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Table 1

Summary of Separation Behavior of Uranium-233 and Other Radioisotopes

(By Filter-Paper Partition Chromatography with H_2O-Saturated 2-Methyltetrahydrofuran Containing 2 5% (v/v) Conc Nitric Acid)

Separated[a]				Not Separated[b]	Results Not Conclusive
Without Movement		With Movement			
Na^{22}	Zr^{95}-Nb^{95}	Sc^{46}	Cd^{115}	Ru^{106} - Rh^{106}	Sn^{113}
Na^{24}	Ag^{110}	$Fe^{55,59}$	Te^{125}	W^{185}	Sb^{124}
K^{42}	Cs^{137}	Zn^{65}	Ba^{140}	Hg^{203}	
Cr^{51}	$Ce^{141,144}$	Se^{75}	La^{140}		
Mn^{54}	Pm^{147}	$As^{73,74}$	Ir^{192}		
Co^{60}	Ta^{182}	In^{114}			
Ni^{63}	Tl^{204}				
$Sr^{89,90}$	Pa^{233}				
Y^{91}					

(a) That is, the U^{233} moves

(b) The radioisotope moves with the U^{233}

The behavior of Hg^{203} was similar to that of U^{233} This suggested the separate study that was made of the separation of Hg^{203} from other metals by the same method The results will be reported separately

The separation of Pa^{233} from U^{233} was of particular interest, it was studied somewhat extensively and applied to process-type solutions that were similar to Thorex solutions A paper that describes the work has been accepted for publication in Reactor Science and Technology, Vol 4, No 3, TID-2014 (Sept , 1954)

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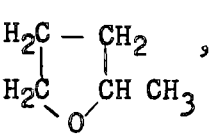
Quantitative elution of the U^{233} from the paper strip was demonstrated by alpha-count measurements Preliminary studies have been made of the polarographic method for the determination of microgram and submicrogram quantities of uranium in the eluate The method of activation analysis is being investigated for the quantitative determination of submicrogram amounts of uranium on the chromatogram

4 REAGENTS

1 2-Methyltetrahydrofuran,  , reagent 5971, Eastman Kodak Co (Supplied to Eastman by Electrochemicals Dept , E I duPont de Nemours & Co Inc) [18] Used as received

2 Nitric Acid, conc B & A , C P reagent, code 1120, General Chemical Div , Allied Chemical & Dye Corp

3 Filter Paper, Whatman No 1, ashless, 1 5-in width, roll form

4 Colorimetric Reagents

(a) Potassium Ferrocyanide, $K_4Fe(CN)_6\ 3H_2O$, analytical reagent grade, Mallinckrodt Chemical Works

(b) 8-Hydroxyquinoline, C P , reagent code 0-261, E & A , Fisher Scientific Co

(c) Ammonium Thiocyanate, NH_4CNS, B & A , A C S reagent code 1319, General Chemical Div , Allied Chemical & Dye Corp

5 Phosphoric Acid, ortho, 85%, B & A , A C S reagent, code 1149, General Chemical Div , Allied Chemical & Dye Corp

6 Sodium Fluoride, NaF, B & A , A C S reagent, code 2250, General Chemical Div , Allied Chemical & Dye Corp

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7 Radioisotopes Described in Table 2 Supplied by E I Wyatt, ORNL Radioisotope Production Analyses Group

Table 2

Radioisotope Identities

Radioisotope	Compound of Radioisotope	Half Life ($t_{1/2}$)	Activity of Original Sample on 9/12/51, mc/ml	Dilution Medium	Dilution
Na^{22}	$Na^{22}Cl$	2 6 y	0 11	HCl, 0 06 N	1 100
Na^{24}	$Na^{24}Cl$	15 1 h[a]	0 11	HNO_3, 0 1 N	1 250
K^{42}	$K^{42}Cl$	12 4 h[a]	?	HNO_3, 0 1 N	1 50
Sc^{46}	$Sc^{46}Cl_3$	85 d	0 024	HNO_3, 0 1 N	1 50
Cr^{51}	$Cr^{51}Cl_2$	26 5 d	0 77	HCl, 0 0219 N[b]	None
Mn^{54}	$Mn^{54}Cl_2$	310 d	0 0054	HCl, 0 43 N[b]	None
$Fe^{55,59}$	$Fe^{55,59}Cl_3$	2 91 y(55) 46 d(59)	0 0251	?	?
Co^{60}	$Co^{60}Cl_3$	5 3 y	1 75	HNO_3, 0 1 N	1 500
Ni^{63}	$Ni^{63}Cl_2$	85 y	1.76	HCl, 0 88 N[b]	None
Zn^{65}	$Zn^{65}Cl_2$	250 d	1 34	HNO_3, 0 1 N	1 1000
$As^{73,74}$	$As^{73,74}Cl_3$	76 d, 17 5 d	0 1, 0 089	Acid, 2 8 N	1 1
Se^{75}	$Se^{75}Cl_4$	127 d	7 48	HCl, 4 9 N	1 200
$Sr^{89,90}$	$Sr^{89,90}Cl_2$	54 d	9 5	HNO_3, 0 1 N	1 12500
Y^{91}	$Y^{91}Cl_3$	61 d	0 268	HNO_3, 0 1 N	1 500
Zr^{95}-Nb^{95}	Oxalate Complex	65 d - 37 d	3	HNO_3, 0 1 N	1 4000

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Table 2 (Cont'd)

Radioisotope	Compound of Radioisotope	Half Life ($t_{1/2}$)	Activity of Original Sample on 9/12/51, mc/ml	Dilution Medium	Dilution
Ru^{106}-Rh^{106}	$Ru^{106}Cl_3$ - $Rh^{106}Cl_3$	1 y	3 0	HNO_3, 0 1 N	1 1000
Ag^{110}	Ag^{110} NO_3	270 d	2 83	HNO_3, 1 7 N	1 1000
Sn^{113}	$Sn^{113}Cl_2$	105 d	6 x 10^{-4}	5 9 N HCl(b)	None
In^{114}	$In^{114}Cl_2$	50 d	1 22	HNO_3, 0 1 N	1 1000
Cd^{115}	$Cd^{115}(NO_3)_2$	43 d	0 085	HNO_3, 0 1 N	1 100
Sb^{124}	$Sb^{124}Cl_3$	60 d	0 01	Acid(b)	None
Te^{125}	?	58 d	3 x 10^{-4}	Acid(?),1 27N(b)	None
Cs^{137}	$Cs^{137}Cl$	33 y	7 x 10^6(cpm/ml)	HNO_3,~0 1 N	?
Ba^{140}	$Ba^{140}(NO_3)_2$	12 8 d	0 77	HNO_3, 0 1 N	1 1000
La^{140}	$La^{140}Cl_3$	40 h(a)	0 25	HNO_3, 0 1 N	1 20
$Ce^{141,144}$	$Ce^{141,144}Cl_3$	275 d	1 06	HNO_3, 0 1 N	1 2000
Pm^{147}	---	3 7 y	?	HNO_3, 2 N	None
Ta^{182}	$K_8Ta_6O_{19}$ $25H_2O$	117 d	2	HCl, 6 N	1 250
W^{185}	$K_2W^{185}O_4$	73 2 d	2 53	Base, 0 571 N	1 100
Ir^{192}	$Ir^{192}Cl_3$	70 d	0 125	HCl, 1 62 N	1 100
Hg^{203}	$Hg^{203}(NO_3)_2$	51 d	0 148	HNO_3, 0 1 N	1 250
Tl^{204}	$Tl^{204}(NO_3)_2$	3 y	1 86	HNO_3, 0 1 N	1 4000
Pa^{233}	----	27 4 d	1 85 x 10^5 cpm/ml	HCl, conc	None
U^{233}	$U^{233}O_2(NO_3)_2$	1 6 x 10^5 y	0 549	HNO_3, 0 1 N	1 13 2

(a) Used immediately

(b) Medium of the undiluted radioisotope solution.

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5. APPARATUS

1 For Chromatography

The apparatus was the type described by Burstall et al [7] See Fig 1.

2 For Autoradiography

(a) X-ray film, Kodak medical No-Screen, Eastman Kodak Co , Rochester, N Y

(b) Dark box, provided with Lucite plate and light-tight double lid, for use in exposing X-ray films to chromatograms of radioisotopes See Fig 2

3 For Alpha-Counting

Proportional Alpha Counter, ORNL Instrument Dept , Model Q-721-A, Serial No 3, or Model 1C

6 EXPERIMENTAL TECHNIQUES

6 1 Preparation of Eluting Solvent

Two procedures were used for the preparation of the eluting solvent The first used was that of Arden et al [2] whereby the 2-methyltetrahydrofuran was saturated with water, then concentrated HNO_3 was added to give the desired concentration, and finally more water added to resaturate the mixture Because of the possibility of variation in the last step, the procedure was changed slightly The 2-methyltetrahydrofuran was shaken vigorously with a large excess of water and allowed to remain in contact with the water at 25 ± 2°C after phase separation An aliquot of the H_2O-saturated solvent was removed, and the desired amount of concentrated HNO_3 pipeted into it, the resulting solution

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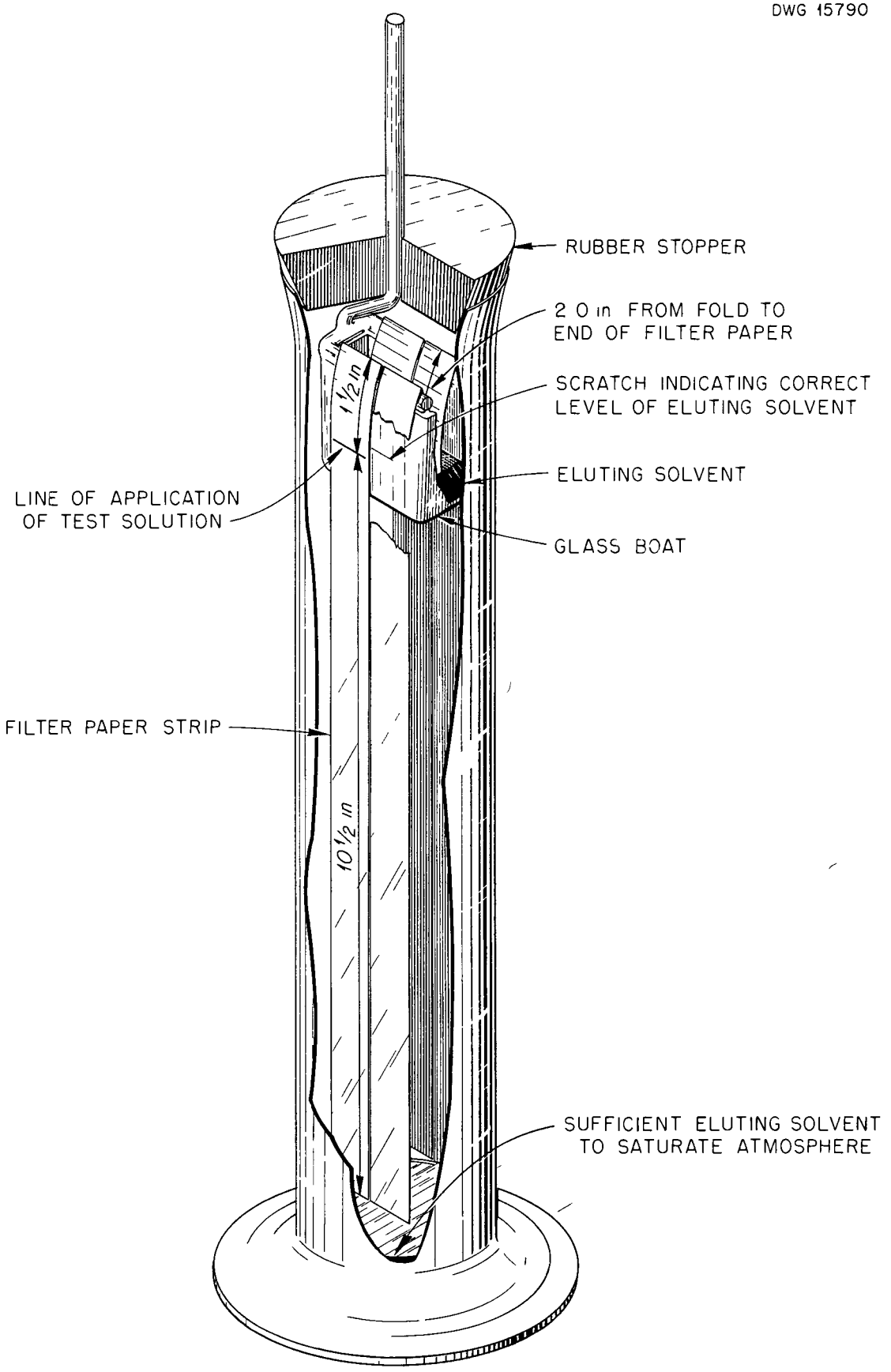

Fig 1 Apparatus for Chromatography

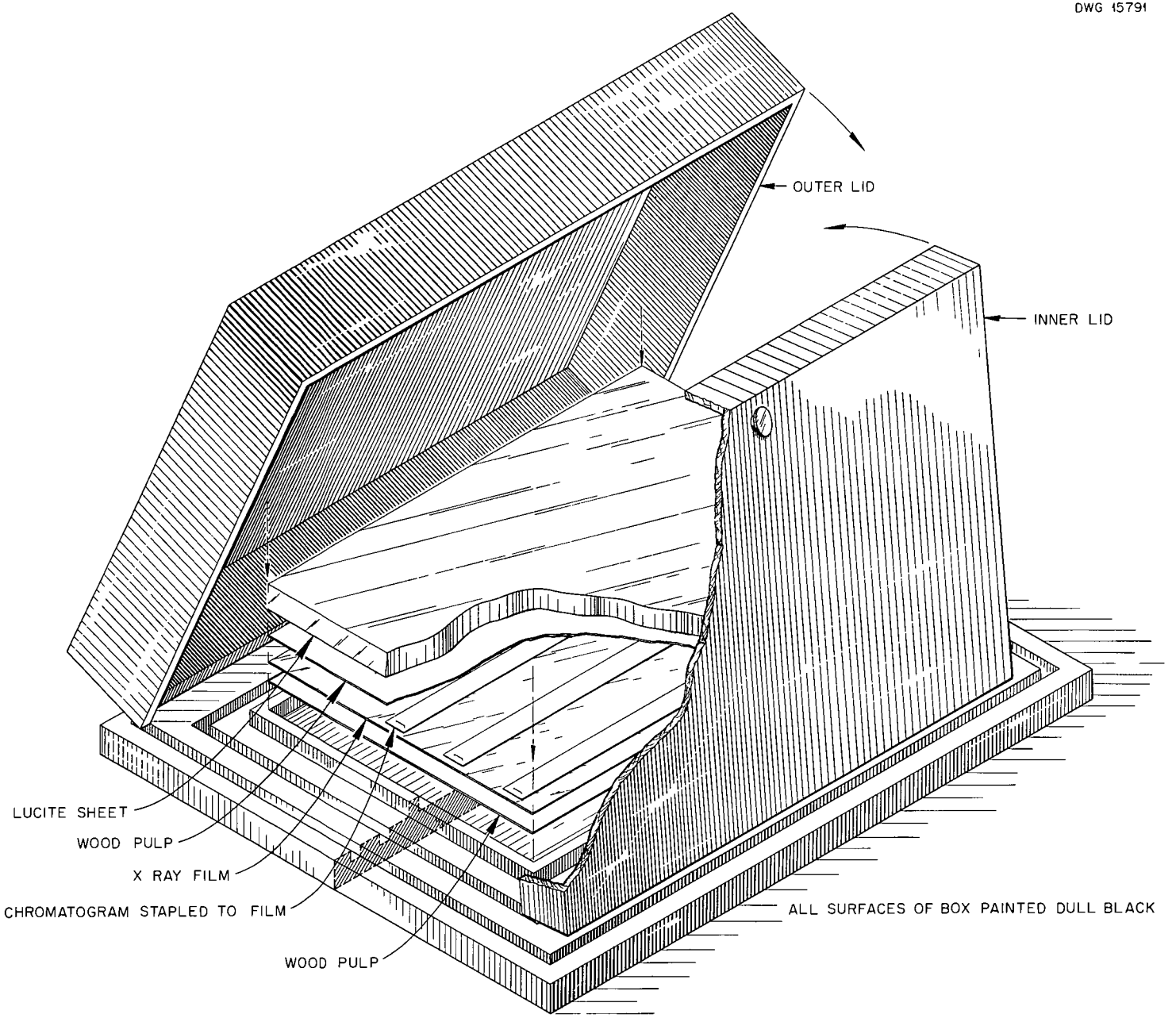

Fig 2 Dark Box for Autoradiography

being used as the eluting solvent and also for obtaining an equilibrium atmosphere in the chromatographic cylinder

2-Methyltetrahydrofuran reacts extremely vigorously with concentrated HNO_3, and great care was required in adding the HNO_3 to it The solvent was placed in an Erlenmeyer flask of considerably greater volume (e g , a 250-ml flask for 50 ml of solvent), and the HNO_3 added rapidly to it from a pipet, the tip of the pipet being under the surface of the solvent before HNO_3 addition was begun The solvent was swirled gently during the addition

6 2 Preparation of Chromatograms

A strip of Whatman No 1 ashless filter paper, obtained in 1 5-in roll form, was cut to a 12- or 14-in length and usually a 0 75-in width A line was penciled lightly across the strip 3 5 in from one end, and the strip was folded 2 in from the same end to facilitate its hanging in the solvent boat A measured volume of the solution to be tested was delivered from a pipet along this line, the paper strip was held in a horizontal position and care was taken to distribute the solution evenly Although the solution spread over the width of the strip, no pronounced edge effects were observed on the chromatograms The moist spot was dried in air

The cylinder and solvent boat were prepared just prior to receiving the paper strip (Fig 1) The eluting solvent was added to the boat up to a permanent mark at about half its height Ten milliliters of the same solvent was placed in the bottom of the cylinder For some experiments, water was placed in a beaker which rested on the bottom of the cylinder

The end of the paper strip nearest the test spot was placed in the solvent boat, and the boat and strip placed in the cylinder so that the strip hung

vertically and did not touch the sides of the boat or the cylinder wall The elution was allowed to proceed at 25 ± 2°C until the solvent had diffused down the paper sufficiently far to effect a separation The strip was removed from the cylinder and allowed to air-dry A chromatogram thus prepared was ready for analysis

6 3 Preparation of Autoradiograms

Autoradiograms of the chromatograms of the radioisotopes were prepared by stapling the chromatograms to sheets of Kodak medical x-ray No-Screen film, placing this between sheets of wood pulp, weighting the sheets with a Lucite block, and enclosing the assembly in a special light-tight box (Fig 2) The chromatograms were allowed to remain in contact with the film for periods ranging from about 16 to 65 5 hours The minimum time required for the fogging of the film by the chromatographed radioisotopes was not determined The film was developed, and the loci of the radioisotopes determined from the fogging produced on the film

6 4 Measurement of Alpha Counts on Chromatograms of Uranium-233

The chromatograms of uranium-233 were prepared for alpha count measurements by cutting the solvent-free chromatogram crosswise into half-inch segments The counting was done on a proportional alpha counter at 52% geometry Attention was given to constancy of counter, counting time, counter operator, position of the chromatogram segment in the counter chamber, methane flow through the chamber, and background correction

[redacted]

7 RESULTS

7 1 Determination of the Uranium Locus on the Chromatogram

7 1 1 With Colorimetric Reagents

The following colorimetric reagents were considered for identification of the uranium locus potassium ferrocyanide, 8-hydroxyquinoline, and ammonium thiocyanate No one of these was found to be sufficiently sensitive to detect microgram quantities of uranium(VI) adsorbed on the filter paper, as indicated by spot tests The lower limit of sensitivity was found to lie between 1 x 10^{-3} and 1 x 10^{-4} g of uranium(VI) per ml of test solution The 8-hydroxyquinoline was the most sensitive reagent, but it formed a yellow color difficult to detect against the yellow color imparted to the paper by the eluting solvent These results are not in agreement with those of Arden et al [2]

7 1 2 By Fluorescence Measurement

The work of Sill and Peterson[20] suggested the treatment of the adsorbed uranium(VI) with phosphoric acid followed by visual observation of the enhanced uranium fluorescence under ultraviolet light Spot tests showed that a spot made with 200 µl of a solution that contained 1 x 10^{-3} g of uranium(VI) per ml of 0 1 N HNO_3 gave excellent fluorescence after treatment with 25 and 50% phosphoric acid solutions The test was negative at 1 x 10^{-4} g of uranium(VI) per ml

Another fluorescence method considered was the ashing of sections of the chromatogram beneath a sodium fluoride pellet, fusion, and measurement of the fluorescence of the melt by the fluorophotometer A chromatogram was prepared from 500 µl of a solution of 5 0 µg of uranium(VI) per ml of 0 1 M HNO_3 The fluorophotometric measurement of the uranium in segments of the

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chromatogram indicated a sorption gradient of uranium terminating abruptly in a section of the chromatogram coincident with a narrow dark band of brownish color, which was located about 1 inch behind the solvent front This band marks the boundary between two solvent phases that exist on the paper strip, i e , an upper, strongly acidic and H_2O-saturated phase and a lower, dry-solvent phase that is almost free from acid, as indicated by spraying the chromatograms with indicator solutions The brown coloration occurred both in the presence and absence of uranium The existence of the wet and dry solvent fronts is discussed below The fluorescence method was of qualitative value only in indicating the distribution of uranium(VI) along the chromatogram

7 1 3 By Use of Uranium-233, Autoradiography, and Alpha Counting

The inadequacy of colorimetric methods and the slowness of the fluorophotometric procedure in detecting the locus of natural uranium on the chromatogram led to the substitution of the radioactive isotope, uranium-233, for natural uranium in the study of the sorption gradient of uranium on the chromatogram Autoradiograms and alpha count measurements of the uranium-233 chromatograms were made The analysis of reagent blanks by both techniques gave negative results The sorption gradient of uranium-233 in the chromatograms was indicated by both techniques for a solution that contained 1 0 μg of uranium-233(VI) per ml The counting technique was the more sensitive This is shown by Fig 3 in which the alpha-count data are presented graphically for a chromatogram of a solution of 0 1 μg of uranium-233(VI) per ml No autoradiogram of this chromatogram was detectable even after it had been in contact with the x-ray film for about 65 hr

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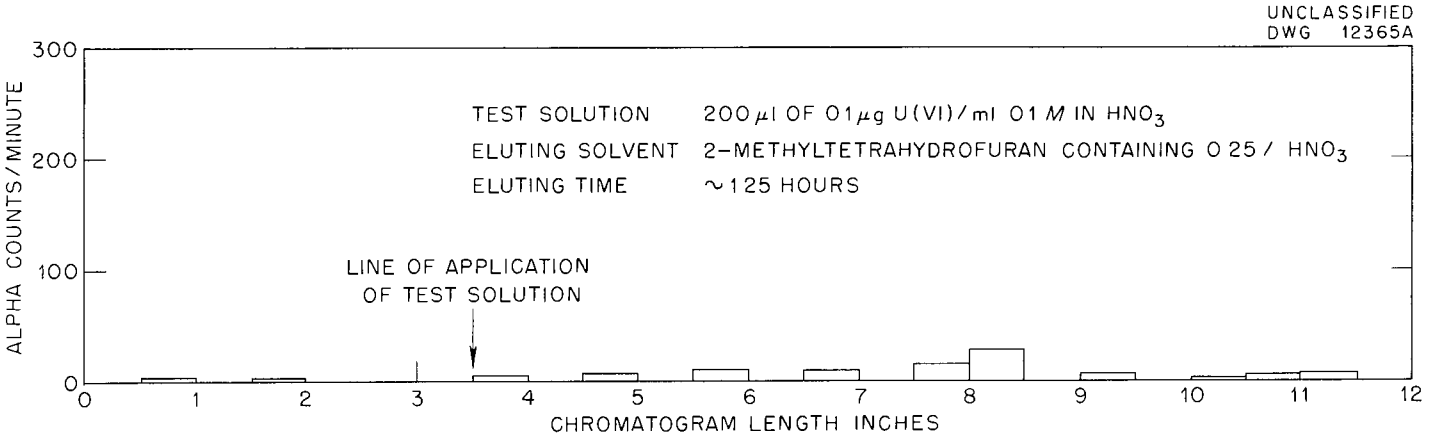

Fig 3 Distribution of Uranium (VI) in [redacted]am of 0.1 μg U(VI)/ml Solution

[redacted]

Autoradiography and alpha counting were the techniques chosen for this radioisotopic study of the separation of uranium from other metals by filter-paper partition chromatography with 2-methyltetrahydrofuran

7 2 Investigation of Eluting Solvents

The extensive work of Arden and MacWilliam[3] precluded work on any solvents other than 2-methyltetrahydrofuran

7 3 Optimum Conditions for Separating Uranium by Filter-Paper Partition Chromatography with 2-Methyltetrahydrofuran

Since the combined use of radioisotopes, autoradiography, and radiation counting permits the determination of the loci and distribution therein of chromatographed substances, these techniques are ideal for establishing the optimum conditions for a chromatographic separation They were employed in determining the effect on the chromatography of uranium(VI) of the acid content, water content, and age of the eluting solvent and also of the elution time

7 3 1 Acid Content of Eluting Solvent

Aronoff[4] has shown that the sorption gradient of a solute may be a function of the acid content of the solvent, and also that the presence of more than one locus may still be identified with a single substance Since Arden et al [2] report the use of 2-methyltetrahydrofuran that contained both 0 25 and 2 5% (v/v) HNO_3 without indicating what acid concentration is optimum, it was desirable to determine this Aliquots of 100 µl-volume of a solution that contained 5 µg of U^{233}(VI) per ml of 0 1 N HNO_3 were chromatographed with eluting solvents of H_2O-saturated 2-methyltetrahydrofuran that contained the following volume per cents of conc HNO_3 0 25, 0 5, 1 0, 2 0, 2 5, 3 0, 3 5, 4 0, and 5 0 Elution was allowed to progress approximately 1 5 hr at 25 ± 2°C

[redacted]

[redacted]

for all the chromatograms The alpha-count data and prints of the autoradiograms for a representative group of these chromatograms are presented graphically in Fig 4 The results indicate that the optimum nitric acid content of the H_2O-saturated solvent for producing the least trailing of the uranium(VI) band is 2 5% (v/v) Further increase in nitric acid concentration up to 5% (v/v) did not improve the sharpness of the band Arden and MacWilliam[3] found that the extraction of uranium(VI) into 2-methyltetrahydrofuran increases very rapidly with increase in the nitric acid concentration of the aqueous phase, containing 25% NH_4NO_3, up to 2% and thereafter much more slowly

7 3 2 Elution Time

The minimum elution time required to give maximum sharpness of the uranium band when the chromatogram was formed by elution with H_2O-saturated 2-methyltetrahydrofuran that contained 2 5% (v/v) conc HNO_3 at 25 ± 1°C was found to be 1 75 hr The data are presented graphically in Fig 5 They show that the sorption zone does not migrate at a uniform rate, but that the lower portions of the zone, which contain the larger amount of adsorbed solute, migrate at a slower rate than the upper, a condition also characteristic of some column chromatograms[21]

7 3 3 Water Content of Eluting Solvent

No systematic study of the effect of water content of the eluting solvent on the sorption gradient of uranium(VI) was made because a satisfactory condensation of the uranium band was achieved with H_2O-saturated (at 25 ± 2°C) 2-methyltetrahydrofuran that contained 2 5% (v/v) conc HNO_3 when the elution proceded for 1 75 hr Analysis of a representative sample of the eluting solvent for water by the Karl Fischer method gave 49 0, 49 8, and

[redacted]

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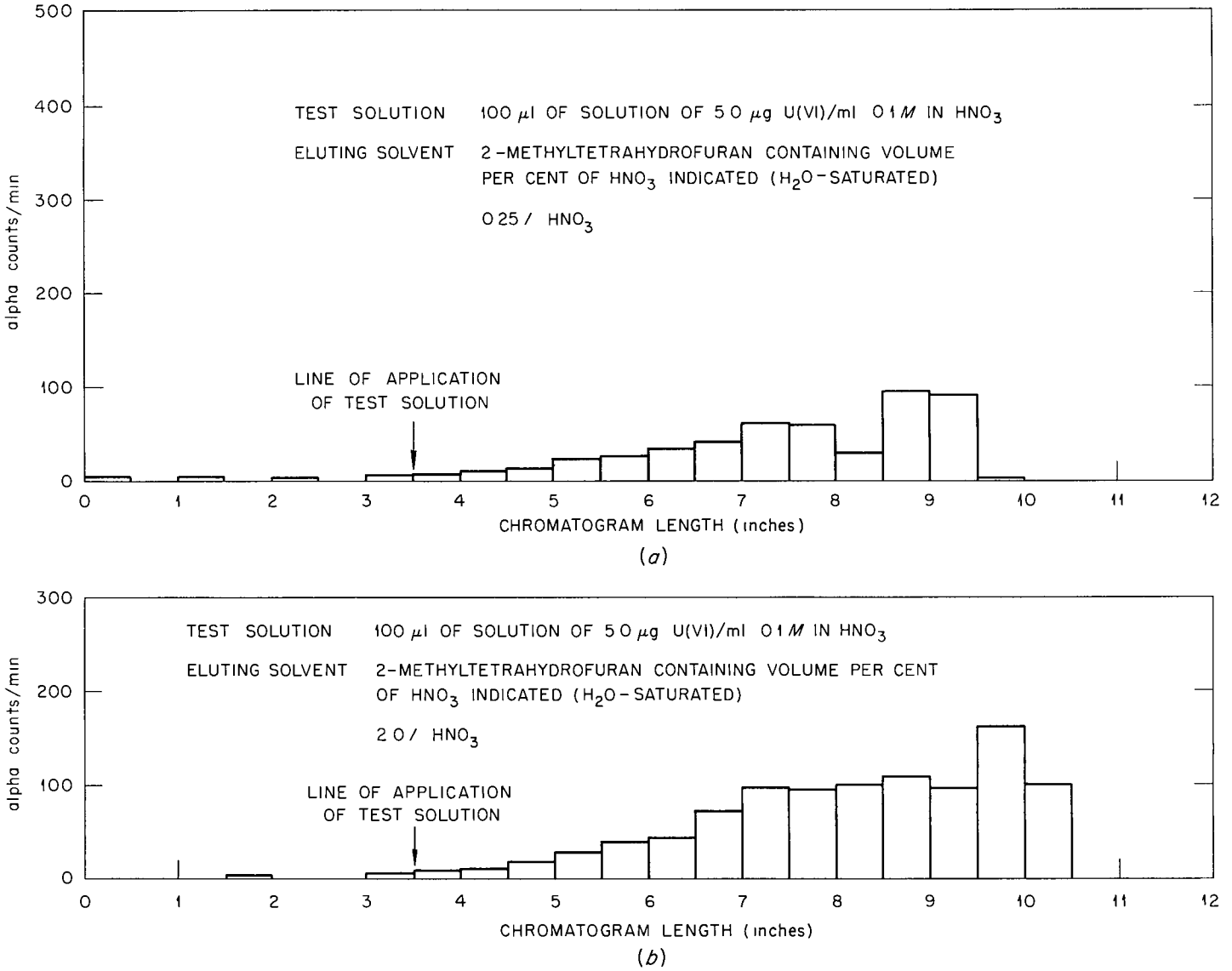

Fig 4 (*a* and *b*) — Effect of Acid Content of Eluting Solvent on Distribution of Uranium(VI) in Paper Chromatogram

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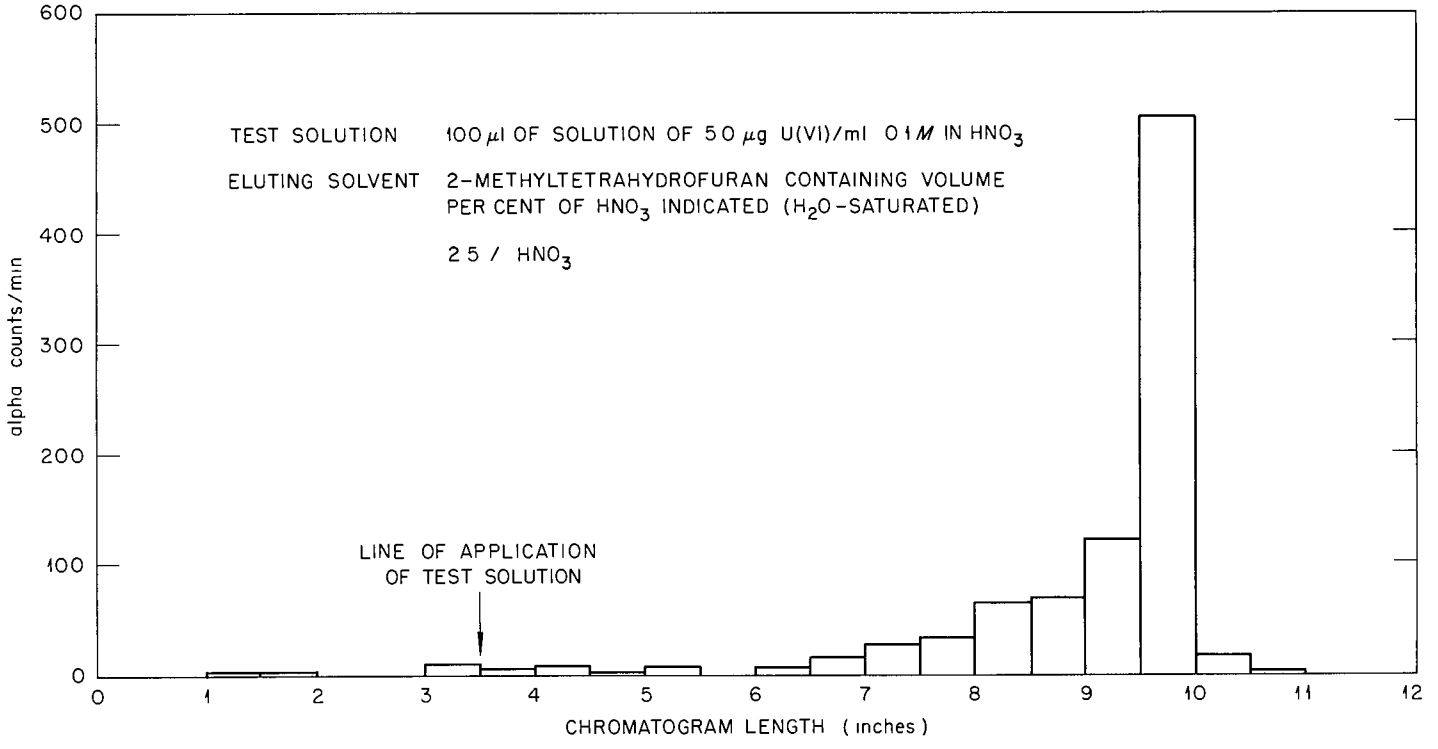
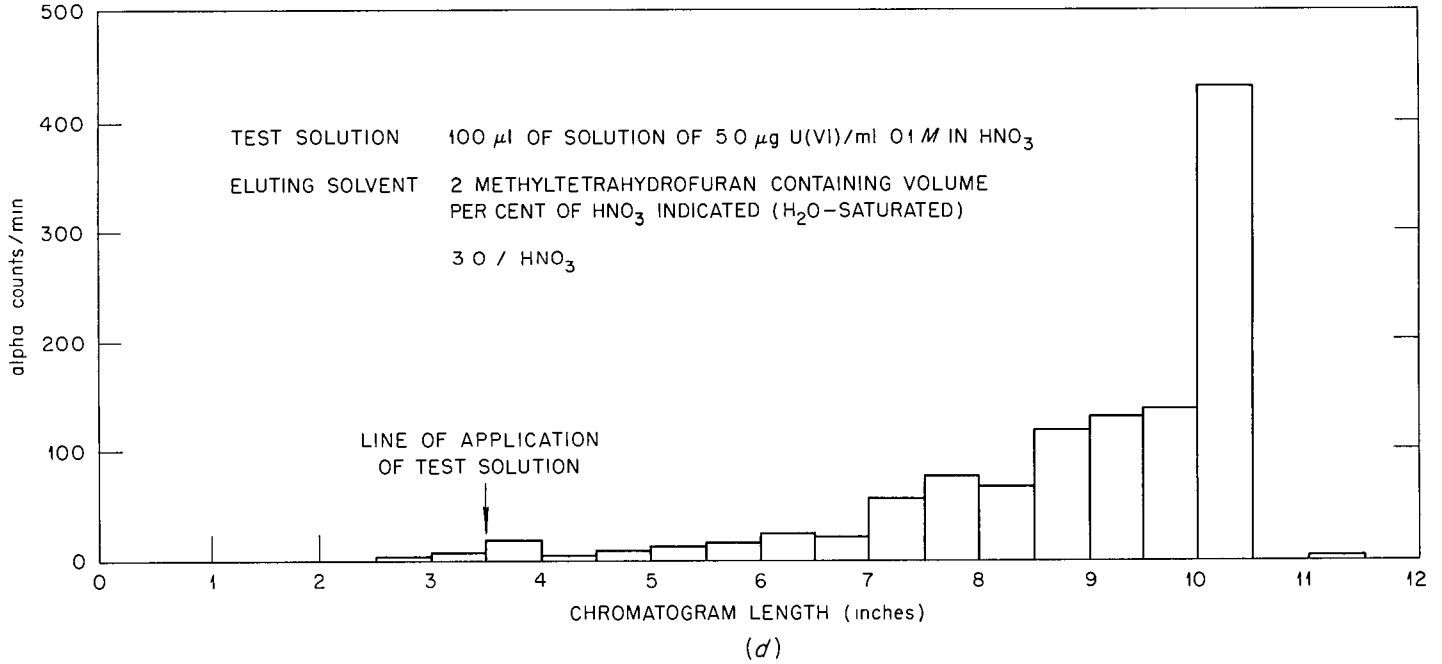


(c)



(d)

Fig 4 (c and d) — Effect of Acid Content of Eluting Solvent on Distribution of Uranium(VI) in Paper Chromatogram

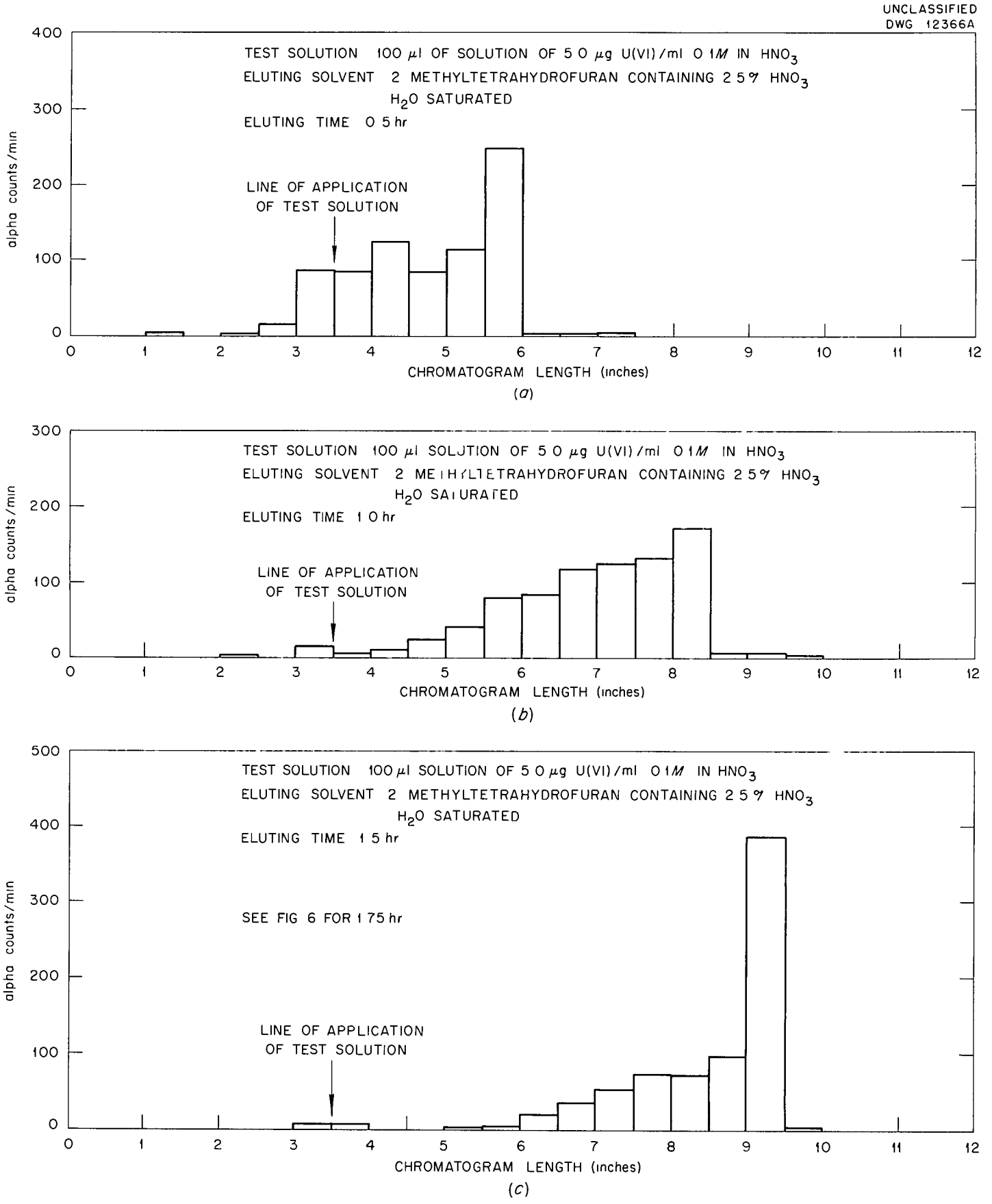


Fig 5 Effect of Elution Time on Distribution of Uranium(VI) in Paper Chromatogram

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49 9 mg/ml For the purpose of rough comparison, the values of Bennett and Philip[5] for the weight per cents of water in 2-methyltetrahydrofuran (no HNO_3 present) at the temperatures indicated follow

15°C	20°C	25°C
7 05	6 65	6 08

7 3.4 Age of Eluting Solvent

When concentrated HNO_3 is added to H_2O-saturated 2-methyltetrahydrofuran at room temperature and in the manner described above, the reaction is vigorous, and the solution undergoes an immediate color change from almost colorless to yellow On standing (approximately 1 hr), the solution becomes a deep reddish-brown color, which color fades until the solution is again yellow The nature of the reaction is not known This color change suggested experiments with eluting solvents of different ages Alpha count data from experiments with 10 min- and 1 hr-old eluting solvents are shown graphically in Fig 6 There is an indication that the uranium(VI) sorption gradient is less with the older solvent This factor warrants further study

7 4 Separation of Uranium(VI) from Other Metals Studies with Radioisotopes

The effectiveness of the separation of uranium from thirty-two other metals (thirty-three radioisotopes) by filter-paper partition chromatography with 2-methyltetrahydrofuran under the optimum conditions described above was studied with radioisotopes The number of metals tested was limited by the availability of the radioisotopes, all of which were obtained from E I Wyatt, ORNL Radioisotope Production Analyses Group They are described in Table 1

The chromatograms were prepared on 0 75-in strips of paper according to the procedure given in Section 6 2 Separate chromatograms were prepared for a blank of reagents, U^{233}(VI), the other radioisotope, and U^{233}(VI) plus the

[redacted]

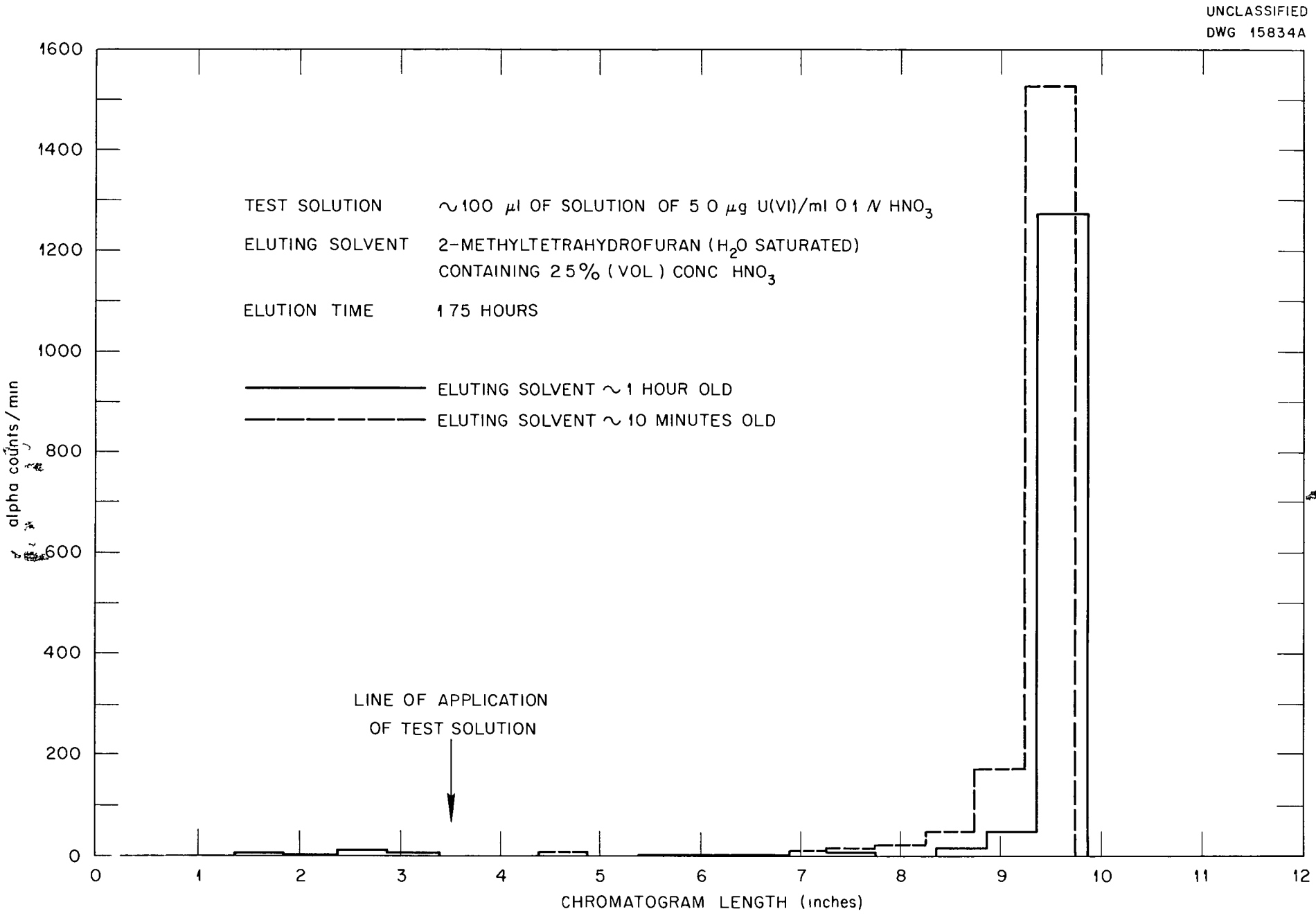

Fig 6 Effect of Age of Eluting Solvent on Distribution of Uranium(VI) in Paper Chromatogram

[redacted]

other radioisotope In the last case the two metal radioisotopes were applied to the paper strip in separate solutions, the strip being air-dried after each deposition

The loci of the U^{233}(VI) and other radioisotope bands were determined by autoradiography, and the sorption gradient of the U^{233}(VI) by alpha counting as described in Section 6 4 The method of measuring the R_f values is subject to variation and requires some explanation In the case of the U^{233}, the distance moved by the band was considered to be the distance between the line on which the solution was applied and the center of the U^{233} band (always coincident with the wet-solvent front) The distance moved by the dry-solvent front was that from the same line to a line that marked the fartherest advance of the dry-solvent front Thus

$$R_f \text{ for } U^{233} = \frac{\text{distance moved by } U^{233} \text{ band, cm}}{\text{distance moved by dry-solvent front, cm}}$$

For radioisotopes that showed diffuse bands, R_f values were estimated only roughly or not at all An R_f value of zero indicates no movement of the band, and a value of 1 00 indicates movement of the metal in the dry-solvent front

The results of the separation studies are summarized in Table 1 and given in detail in Table 3 Positive prints of the autoradiograms are given in Fig 7 It should be pointed out that consideration of the band width is essential to the proper interpretation of band resolution

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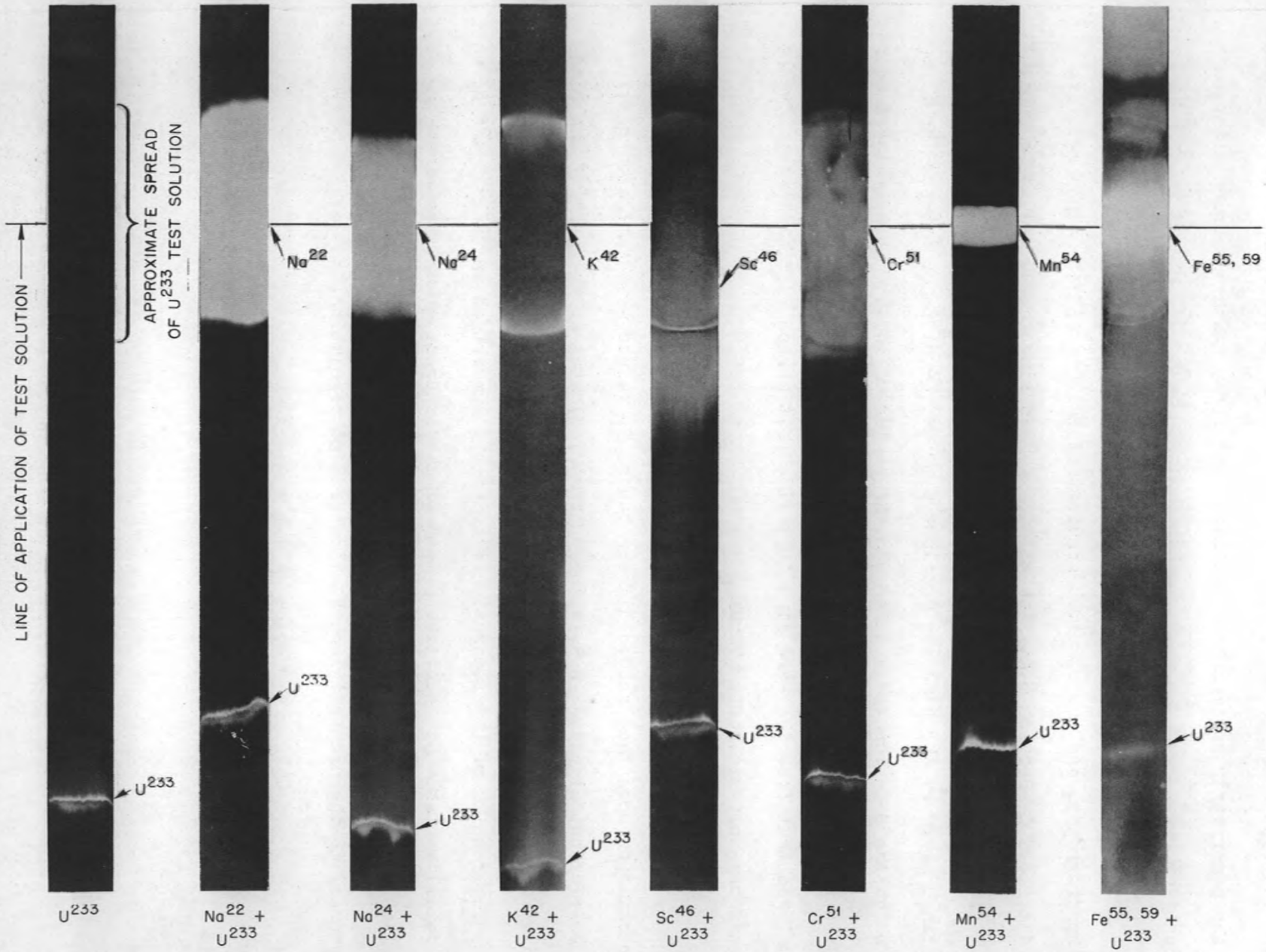

Fig. 7. Prints of Autoradiograms of Chromatograms Showing Separation of Uranium-233 from the Radioisotope Indicated.

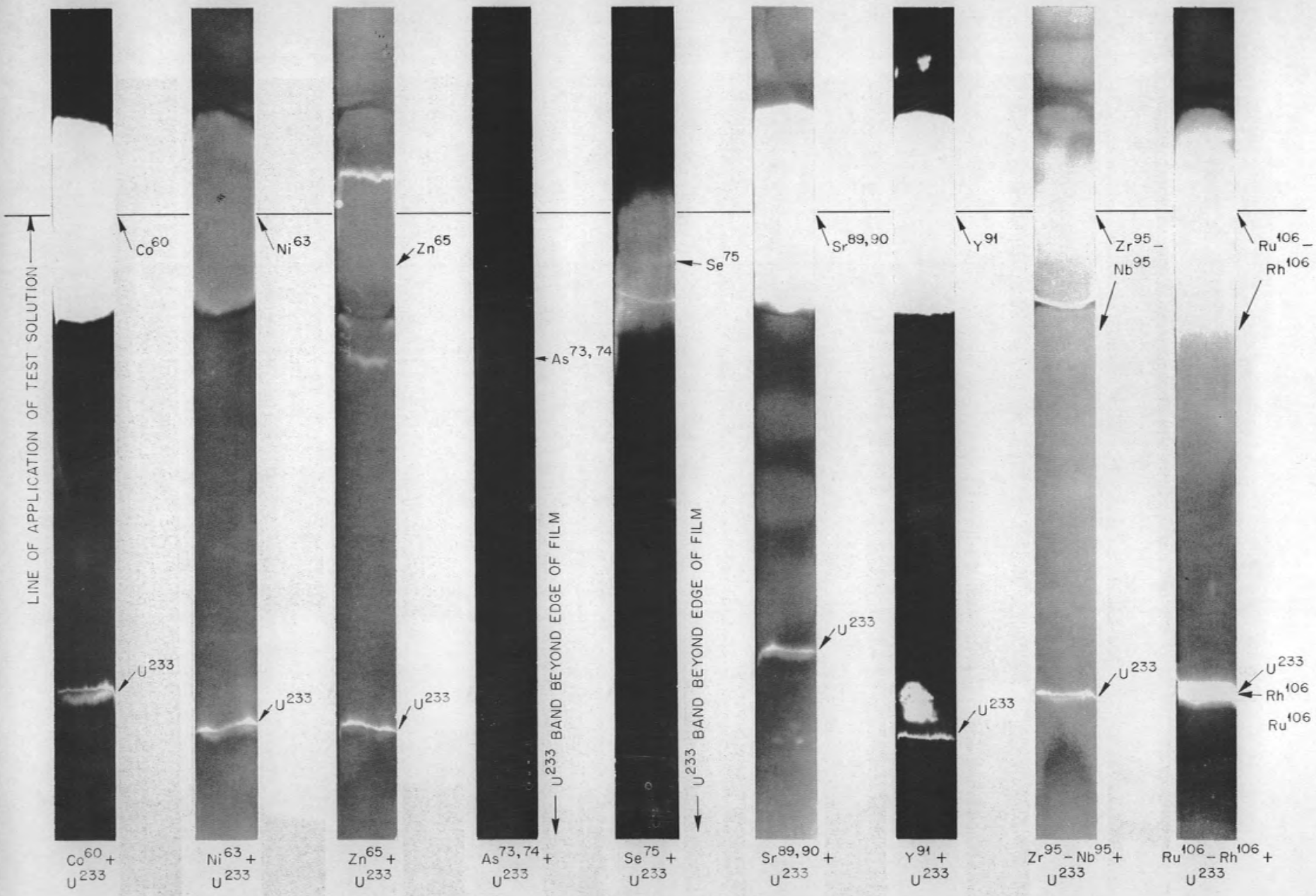

Fig. 7. (Continued)

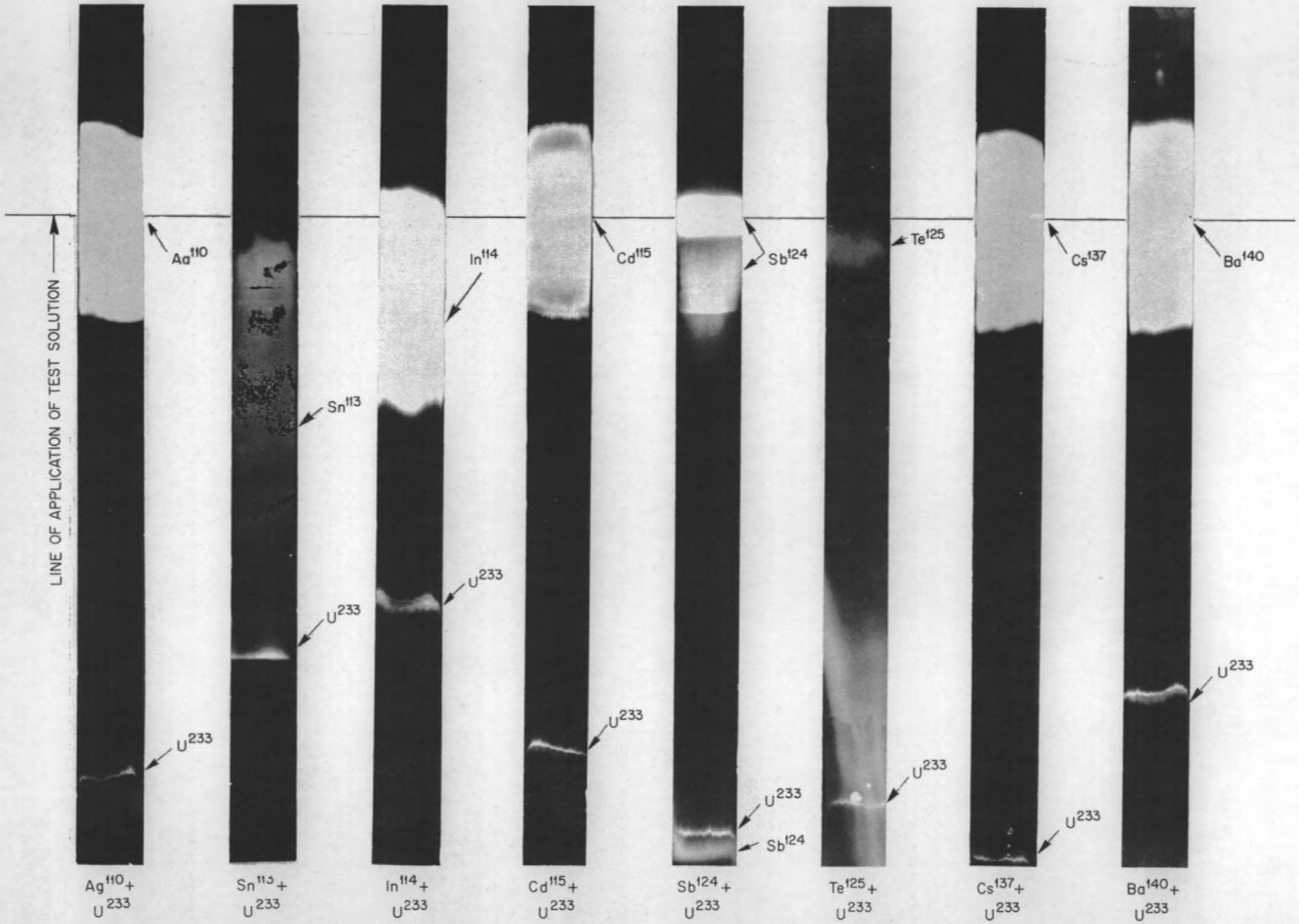

Fig. 7. (Continued)

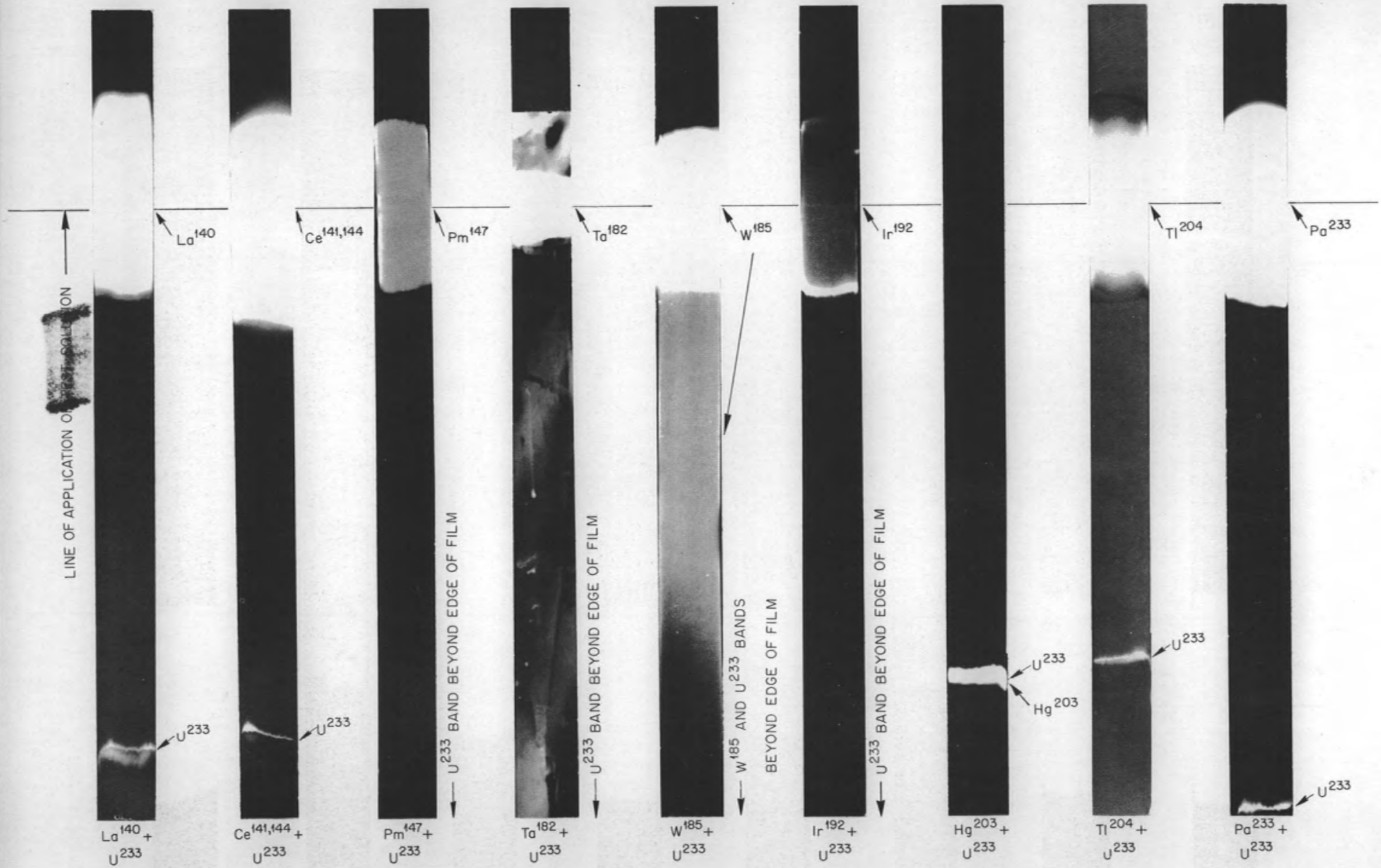

Fig. 7. (Continued)

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Table 3

Separation Behavior of Uranium-233 and Other Radioisotopes

(By Filter-Paper Partition Chromatography with H_2O-Saturated 2-Methyltetrahydrofuran Containing 2 5% (v/v) Conc Nitric Acid)

Radioisotope[a]	Distance Moved, cm			Band Width, cm[b,c]		R_f Value[d]		Behavior of Radioisotope[e]
	Dry-Solvent Front	Radioisotope	U^{233}	Radioisotope	U^{233}	Radioisotope	U^{233}	
None present	--	--	--	--	--	--	0 95[f]	4
Na^{22}	15 0	None	14 3	--	0 50	0	0 95	1
Na^{24}	18 7	None	17 9	--	0 05	0	0 95	1
K^{42}	20 0	None	19 1	--	0 05	0	0 95	1
Sc^{46}	17 5	?	--	--	0 15	--	--	2
Cr^{51}	17 0	None	16 0	--	0 10	0	0 94	1
Mn^{54}	16 4	None	15 6	--	0 30	0	0 95	1
$Fe^{55,59}$	16 7	?	15 7	--	--	?	0 94	--
Co^{60}	16 2	None	15 5	--	--	0	0 96	--
Ni^{63}	17 6	None	16 6	--	0 10	0	0 94	1
Zn^{65}	17 3	?	16 5	(6 20)	0 15	?	0 95	2
$As^{73,74}$	21 8	?	21 2	(11 5)	0 40	?	0 97	3
Se^{75}	22 5	1 5	21 5	(6)	0 20	?	0 96	2
$Sr^{89,90}$	14 8	None	14 2	--	0 15	0	0 96	1
Y^{91}	17 8	None	16 9	--	0.10	0	0 95	1
Zr^{95}- Nb^{95}	16 2	None	15 6	--	0 10	0	0 96	1
Ru^{106}-Rh^{106}	16 2	?	15 4	(19 0)	0.10	?	0 95	2
Ag^{110}	18.2	None	17 5	--	0 12	-0	0 96	1
Sn^{113}	13 5	?	13 3	(13 0)	0 12	?	(0 99)	2
In^{114}	12 6	2 8	12 0	(7 0)	0 70	0 22	0 95	2

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[redacted]

Table 3 (Cont'd)

Radioisotope[a]	Distance Moved, cm			Band Width, cm[b,c]		R_f Value[d]		Behavior of Radioisotope[e]
	Dry-Solvent Front	Radioisotope	U^{233}	Radioisotope	U^{233}	Radioisotope	U^{233}	
Cd^{115}	17 1	0 2	16 6	(6 0)	0 50	0 01	0 97	2
Sb^{124}	20 0	?	19 0	Resolved into 3 bands	0 20	?	0 95	1,3,4
Te^{125}	18 8	0 8	18 1	(1 3)	0 3	0 04	0 96	2
Cs^{137}	20 5	None	19 8	--	0 1	0	0 97	1
Ba^{140}	15 4	0 5	14 6	(6 4)	0 5	0 03	0 95	2
La^{140}	19 1	0 1	18 2	(6 6)	0 6	0 01	0 95	2
$Ce^{141,144}$	18 1	None	17 0	--	0 2	0	0 94	1
Pm^{147}	22 1	None	21 5	--	0 2	0	0 97	1
Ta^{182}	24 3	None	23 1	--	0 15	0	0 95	1
W^{185}	20 8	?	20 0	Resolved into several bands	0 1	?	0 96	1,2,3,4
Ir^{192}	22 1	?	21 3	(8)	0 2	?	0 96	2
Hg^{203}	15 8	15 4	?	0 65	?	0 98	?	4
Tl^{204}	15 7	None	15 1	--	0 20	0	0 96	1
Pa^{233}	20 1	None	19 3	--	0 15	0	0 97	1

(a) Radioisotope other than U^{233} See Table 1 for a complete description

(b) The approximate band width covered by the aliquot of radioisotope solution, an application to the filter paper strip, was ~6 0 cm for the ~100-µl aliquot, and ~1 5 cm for an ~5-µl aliquot

(c) Approximately 100 µl of the radioisotope solution was used except in the case of the Mn^{54}, Sb^{124}, Te^{125}, Ta^{182}, and Hg^{203} solutions of which approximately 5 µl was used

(d) $R_f = \frac{\text{cm traveled by radioisotope}}{\text{cm traveled by dry-solvent front}}$

(e) The exact behavior of each radioisotope in the presence of U^{233} is indicated by its autoradiograms, Fig 7 The behavior in the absence of the uranium was the same in eachcase, therefore those autoradiograms are not shown The general behavior is indicated by one of the following statements

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(1) No movement from test spot

(2) Partial movement from test spot

(3) Complete movement from test spot, band diffused

(4) Complete movement from test spot, band condensed

(f) An average value determined from 29 chromatograms

7 5 Quantitative Removal of Uranium from the Filter-Paper Strip

The techniques of Soxhlet extraction and elution were investigated for the quantitative removal of uranium from the filter paper In the former, the segment of the chromatogram that contained the uranium band was cut out and placed in the thimble of a semimicro Soxhlet extraction apparatus Extraction with ethyl ether was attempted but was not effective

Quantitative removal of the uranium was achieved by elution The paper strips were cut to the shape and dimensions shown in Fig 8 The uranium was chromatographed as described above The minimum time required for its removal was determined by use of U^{233} and measurement of the residual alpha counts on the chromatograms after various elution times The data of Table 4 indicate that quantitative removal of the amount of U^{233} indicated is achieved if the elution proceeds under the conditions designated for a minimum of 1 75 hr after the front of the U^{233} band (wet-solvent front) reaches the end of the strip

The quantitative determination of the uranium on the chromatogram by activation analysis and in the eluate from the chromatogram by polarography is currently being studied

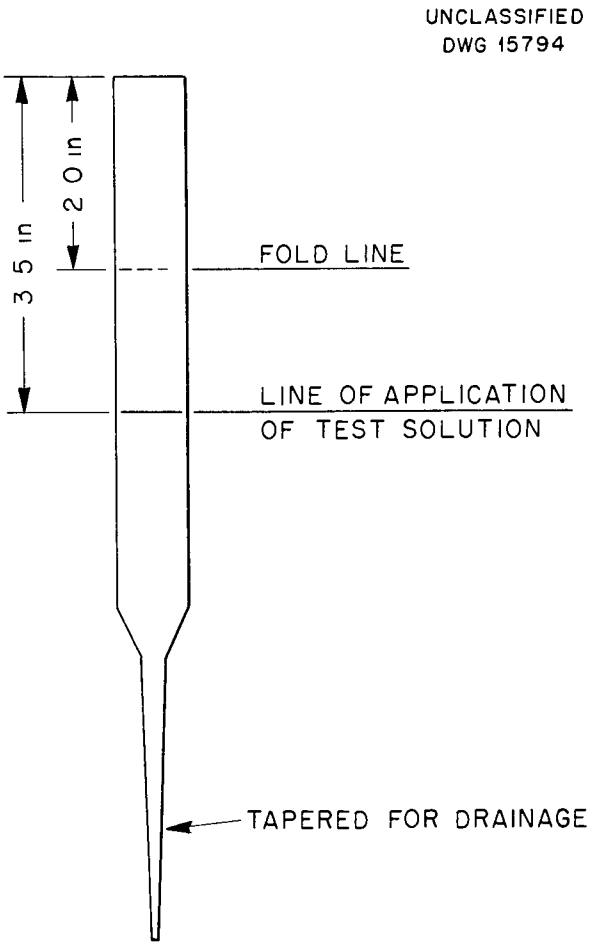

Fig 8 Filter Paper Strip Used for Quantitative Elution of Uranium

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Table 4

Alpha Count Data Showing Quantitative Elution of Uranium-233 from Filter Paper Chromatograms

Conditions

Shape and dimensions of paper strip - see Fig 8
Eluting solvent - 2-methyltetrahydrofuran (H_2O-saturated) containing 2 5% (v/v) of conc HNO_3
Size of chromatogram segments - 0 5 in x 0 75 in (except for tapered end)
Amount of U^{233} deposited on paper strip - 100 µl of a solution containing 5 µg of U^{233} per ml of 0 1 N HNO_3
Counter - ORNL Model Q-721-A Proportional Alpha Counter

	Alpha Counts per Minute												
				Eluted Uranium-233 Chromatograms[a]									
				1 5-hr Elution		2-hr Elution				3-hr Elution			
Segment No	Filter Paper Blanks 1	Filter Paper Blanks 2	Filter Paper Blank + U^{233}, not eluted[c]	1	2	1	2	3	4	1	2	3	4
1[b]	1	0	0	0	0	0	0	0	0	0	0	0	0
2	4	1	0	0	0	0	0	0	0	0	0	0	0
3	4	0	0	0	0	0	1	3	0	0	0	0	0
4	6	0	0	0	0	0	0	2	0	0	0	0	0
5	0	1	31	0	0	0	0	2	0	0	0	0	0
6	0	0	299	0	0	0	0	2	0	0	0	0	0
7	0	0	266	0	0	0	0	0	0	0	0	0	0
8	0	0	255	0	0	0	0	0	0	0	0	0	0
9	1	0	290	0	0	0	0	5	0	3	1	1	0
10	2	0	103	1	0	0	0	0	0	0	0	0	0
11	0	0	0	0	0	0	0	0	0	0	1	0	0
12	5	0	0	3	0	0	0	0	0	6	0	0	0
13	1	0	3	3	0	0	0	0	4	2	0	0	0
14	0	0	0	3	9	2	0	1	1	2	0	1	3
15	0	0	1	6	9	1	5	4	6	14	0	3	2
16	0	0	0	6	9	2	2	1	6	8	0	4	4
17	5	1	0	9	9	7	8	0	9	8	4	5	4
18	1	0	0	10	0	2	7	3	3	9	3	11	8
	30	3	1248	40	36	14	32	23	29	52	9	25	21
	Avg = 16			Avg = 38		Avg = 25				Avg = 27			

[redacted]

[redacted]

Table 4 (Cont'd)

Uranium-233 activity remaining on the filter paper after 2-hr elution, approximate per cent = 2 0 (not corrected for count on the paper blank)

(a) Elution time measured after the wet solvent front reached the end of the paper strip

(b) This end of the strip dipped into the eluting solvent

(c) The uranium-233 solution was applied on a line between segments No 7 and 8, i e , 3 5 in from the end of the strip

[redacted]

[redacted]

8 DISCUSSION

8 1 Mechanism

According to Arden et al [1,2] and Burstall et al ,[8] the factors which cause separation of inorganic salts by partition chromatography on cellulose may be (a) selective extraction of the salts by the organic solvent, (b) partition of the inorganic substances between organic solvent and aqueous layer, (c) adsorption of metallic ions by the paper, and (d) the formation of complexes with high solubility in organic media Pollard et al [19] likewise suggest that one of the main factors in paper chromatography of inorganic compounds is the distribution of the compounds between the organic solvent and water That factors (a) and (b) are the major ones causing the separation of uranium(VI) when uranyl nitrate is eluted on filter paper with 2-methyltetrahydrofuran that contains aqueous HNO_3 is indicated by the work of Arden and MacWilliam[3] and of Burstall et al [7] The former have demonstrated the power of 2-methyltetrahydrofuran "to extract uranyl nitrate even from solutions containing very little salting-out agent" by determining the partition coefficient of uranyl nitrate, contained in aqueous solutions of various concentrations of HNO_3 and of NH_4NO_3, between the aqueous phase and each of 41 organic solvents The 2-methyltetrahydrofuran is shown to be strikingly superior to the other solvents Burstall et al [7] suggested that those inorganic salts which have a high solubility in organic solvents tend to be extracted in the solvent front and to form bands narrower than the original spot This behavior was observed consistently for U^{233}(VI)

The formation of wet and dry zones and of an acid gradient on the paper strip was observed in the course of this study These conditions are discussed below, an explanation for which has been proposed by Burstall et al [7]

[redacted]

8 2 Separation of Phases on the Filter-Paper Strip

The H_2O-saturated 2-methyltetrahydrofuran that contains nitric acid undergoes a frontal analysis as it passes along the paper strip, i e , it changes composition as it moves away from the bulk of the solvent that is contained in the boat This composition change results from the sorption of water by the cellulose until an equilibrium is reached and a dry-solvent front moves ahead of the wet-solvent front The nitric acid undergoes partition between the water in the cellulose and that in the solvent, and an acid gradient is formed in the wet phase, the dry phase being only slightly acidic The mechanism for the formation of the two phases and the partition of acid between them has been postulated by Burstall et al [7] for the n-butanol-HCl-H_2O eluting solvent system Similar behavior was found for the 2-methyltetrahydrofuran-HNO_3-H_2O system The acid differential in the aqueous phase was identified by spraying the chromatograms with an indicator solution The existence of two solvent fronts on paper strip chromatograms has also been shown by other workers for the n-butanol-HCl-H_2O[16] and the n-propylketone-HCl-H_2O[6] systems

A so-called "brown front"[13] marked the advancing edge of the wet phase, both in the presence and absence of added metal ions It was especially useful in indicating the uranium(VI) band, which was always coincident with it This coloration at the wet-solvent front has been described for both nonacidic and acidic systems, i e , collidine-H_2O[13] and n-butanol-HCl[16] The nature of it is not known

8 3 Differential Migration of Uranium

The differential migration of uranium-233 along the paper strip is indicated by the alpha-count data of Fig 5 The process by which this differential migration takes place is suggested by Kowkabany and Cassidy[13], i e , the eluting solvent "operates as a moving vehicle which transports solute from the rear to the front of a zone "

[redacted]

8 4 R_f Values

R_f values are influenced by a variety of factors and should not be interpreted without full consideration of the experimental conditions Kowkabany and Cassidy[13] have described some factors that affect R_f values as determined by experiments with amino acids as solutes Distance of the initial spot from the surface of the eluting solvent and time of elution both affected R_f values, whereas the following factors were reported to have had little affect amount of solute being eluted, rate of flow of eluting solvent, temperature, long "equilibration", prewashing of the filter paper with the eluting solvent, and prefiltration of the eluting solvent through the paper From this information, some generalizations can be made for inorganic chromatography Burstall et al [7] have described other factors which affect R_f values of inorganic solutes

For the calculation of the R_f values given herein, the distance of travel of the dry-solvent front was preferred to that of the wet-solvent front because the dry-solvent front was easily marked by the yellow color of the paper over which the solvent moved, and because certain solutes moved beyond the wet-solvent front

The reproducibility of the R_f values for uranium, when experimental conditions are held constant, is indicated by the data of Table 3 Under these same experimental conditions, the R_f value could be used for the identification of uranium in an unknown

8.5 Band Width

Consideration of band width together with the R_f value is essential to the proper interpretation of the paper chromatographic separation of a solute The width of the uranium(VI) band is shown by the data herein to be a function

[redacted]

[redacted]

of the acid and water contents of the eluting solvent, of elution time, and perhaps of age of the eluting solvent The average band width obtained was 0 24 cm Maximum condensation of the band is believed to have been effected under the optimum conditions described

For the other radioisotopes tested and found to be mobile, the band width may have either increased or decreased with respect to the width of the original test spot, depending upon whether their solutes had low or high solubilities in the eluting solvent

8 6 Separation of Uranium from Other Metals

Of the thirty-three radioisotopes studied, U^{233} was satisfactorily separated from twenty-nine The autoradiographs for the separation of U^{233} from Ru^{106}-Rh^{106}, Sn^{113}, Sb^{124}, and W^{185} (Fig 7) show for the radioisotope other than U^{233} either a sorption gradient all along the strip or a resolution of the radioisotope activity into several bands In each case, separation was not effected and further study is required These interferences were not recognized by Arden et al (2) or by Lederer and Lederer (14)

The separation of mercury by use of 2-methyltetrahydrofuran as a solvent was reported by Arden et al (1) but was not discussed by Arden et al (2) in their work with uranium Lederer and Lederer(14) imply that mercury is separated from uranium The very positive nature of the interference is indicated by the autoradiogram for Hg^{203} (Fig 7), the behavior of which appears to be similar to that of U^{233}, thus the method is indicated to be equally useful for the separation of mercury from other metals A study of Hg^{203} separation similar to that reported herein for U^{233} has been made and will be reported separately

[redacted]

[redacted]

The separation of Pa^{233} and U^{233} has been studied more extensively and applied to process-type solutions This is believed to be the first filter paper chromatographic separation of Pa^{233} and U^{233} A paper that describes this work is being submitted for publication in Reactor Science and Technology

The movement of certain radioisotopes (Table 1) with the eluting solvent suggests the possibility of using the system for multiple separations if additional movement and/or condensations of the bands can be effected

The interference of thorium was recognized by Arden et al [2] The methods referred to by Lederer and Lederer[15] for the paper chromatographic separation of uranium and thorium might possibly eliminate this difficulty

8.7 Applicability of the Method to Sample Analysis

Arden et al [2] report satisfactory separations of uranium "in the presence of chloride, sulfate, oxalate, and acetate, provided nitrate ion is present in sufficient concentrations" Phosphate ion interferes[2,3] The cation interferences are discussed above Methods of preparing samples have been described by Arden et al [2], which should be equally useful under the slightly different conditions described herein

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