1. COLLECmD RADIOCHBMICAL PR.oc::HOORES

# Procedur-e 1

Source ''Procedure for the Isolation of Germanium aDd Arsenic Activities .in P.ission"

. L. **Winsberg** .

Paper 228 iu 11R.adlocbemical Studies:· Tile Fission P.roducts" edi ed by c. D. cor,ell &Ud N. Sugax:IIBD

McGraw-Rill Book Co., Inc., NewYoxlt (1951)

# A -procedure based on.the successive distillation of GeC14 AsC1 with HCl is given for the isolation of **germBDiwn aJld**

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and

arsenic activities in fission. Rffective decontamillation to produce high radioactive -p1U:ity is obtain .

* 1. INTRODUCTION

# To study the occurrence of shert-lived germanium and iu:seaic activities formed in fission. a procedure was developed for radiochemical sepuatioD of these elements fro■ the otber fission products. Because of the low fission yie ds of the iaotopes·of these elements and the necessity of working th fresbl.y irradi.ated uranium, a deeont11Dination factor of about 10- **-s** essential.

* 1. DISCUSSION

Germanium tetrachloride and arsenic·trichloride are.distilled readily from cone. HCl aolution. 1be **geniaaiua\_**and arsenic sulfides are f:iJ:st separated from uranyl nitrate solution and.

are then redissolved in NH 0H. A Zr(OB)4 seavengi12g at this • point removes many\_other HaS-insoluble ioDJJ in addition to zir­ conium, niobium (colwnbiwaJ, and the r•re eactha. Germanium tetrachloride is distilled in au atmosphere of C12 gas Wbich serves to retain the **arsenic** in the n011Volatile pentavalent

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st te. Arsenic trichloride ia distilled from a HCl solution·of\_ CUCl which keeps the arsenic in the trivalent: **state.**

In a separation of gernanium and arsenic activities-by this procedure. 40h As77 was o taiJ:led in-more than 9Q per cent purity

of

as shown by a decay curve indicating a decont&lliu tion factor more than 106• A second decay-curve of 2.lh GeC78J **showed** that this isotope •s obtained in mere than 99 per cent purity, indi­ cating a deconta.mination factor *of* more than 105 for this element.

J. PROCBD

# Step 1. To an appropriate ll!aO\Ult: of neutron-irradiated uranyl nitrate solution in a 50-llll centrifuge tube, add 2 llll each of atanda.rdized **ge manium atld** arsenic carrier solutions

(Note 1) and boil nearly to dryness to re1110Ve excess HN0 ..Take up in 15 m1 of 6N HCl, p1ace **in au** ice bath. **and paea in** H2s

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{Note 2h Centrifuge, an:l discard the supernatant ·s.o1u.tion.

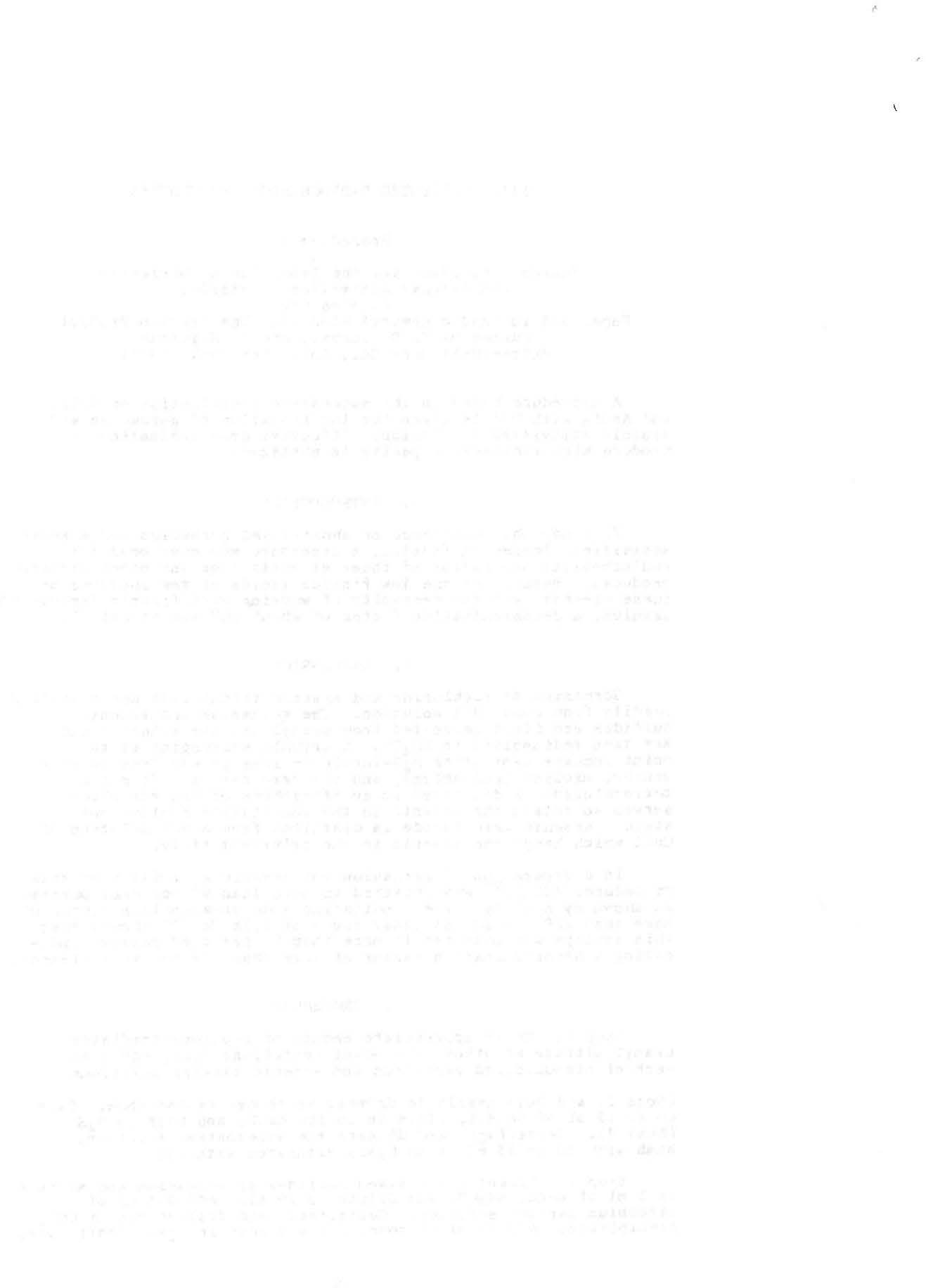
# Wash with 10 to lS ml. of 6M H2S04 saturated with H2S.

Step 2. Dissolve the mixed sulfides of **gerW1&Diwa am** arsenic in l 1111 of cone. NH40II, and dilute to 10 1111. Add o. .i of zirconium carrier solµtion. Centrifuge, and discard the Zr(OH) r:,recipitate. Add 10 :ia1 of cone. HCl and pass iu H25. cent:cifuge,

4

.s

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PROCEDURE l (Continued)

alid wash the precipitate with 10 to 1! llll of H2S04 - H2S solution.

Add .5 Jill. of R2') and heat to expel excess H2S• Add 1 ml. of cone.

NH..OH to dissolve any Ges2 or As2S3 remainiDg, anc.'I transfer to **a** stI11. Add 10 **mg** each of telluriwri, autimony, and tin carriera. (Disregard the sulfides that form in the still.)

Step 3. Add 10 ml of cone. HCJ., pass in Cl2 **gas,** and distill

all but 5 **ml** of the solution into a centrifuge tube containi:ng

o

*S* JIil of a2 cooled iD a.n i,ce bath. Set **a.sJ.de** the distillate for get'Jllaniu.m separation in step 8.

Step 4. Add 5.llll. 0£ cone. HCl aud l ml of **germanium** carrier solution, and distill.GeCl4 **·again, as** described in step·3.

Step *s.* Remove the Cla **gas and pass** in air. Add 1 .Ill of a saturated solution of CuC1 in cone. HCl and *5* ml of cone. HCl·, and ·distill to about 1 111.l: (Note 3). Add *.S* **lll1** of cone. ilCl and 1

iii.1 of CuCl - HCl solution, and distill **again** to about 1 ml (Note 3). Catch.the-distillates.iD·lO ml of H2') in. a centrifuge ube cooled in an-ice bath.. .

Step 6. Pass H2S into the combined distillate to-precipitate As2S3..Centrifuge, dissolve in 1 ml of NftiOH, add 10 mg each of tellurium, antim.ony, and tin, aDd distill as described iD steps.

Step 7. Precipitate the arsenic in the distillate aa Aa2S3,

dissolve in 1 :m.1 of cone. m40H, -dilute to 10 Jill (Note 4), am add 10 ml of cone. HCl. Pass in H2s to complete the precipita­ tion and then filter through a weighed filter-paper disk. Wash

several tim.es with HtJ·, ethyl alcohol, am ether. Heat in &D

oven at 110cc for 10:min•.Weigh·as As2s3•

. . . .

Step 8. Pass H2s gas into the distillate obtaine4.in step

* 1. Centrifuge, and.·then dissolTe the GeS2 iD 1 ml ·of cone. NH40H. Transfer to a still,, add 10 **mg** each of arsenic, tellurium, antimony, a.n4 tin carriers, clistill as outlined ia step 3.·

Step 9.

10 ml of Ha()

co111Plete tlie

e.s described

P.recipitate GeS2, dissolve in cone. NH40H1 add (Note 4) **ancS 8** llll of cone. HCl, **and pass** in Has to precipitation *0£* GeS2. ·Centrifuge, **wash, alld** mouat in step 7 for ar enic. lfeish GeSa·· . •

.• Notes: 1, The genumium carrier solution **(germanium** con­ centration of 10 •rr/ml.) is prepared by.dissolving an appropriate uiount of **ae,manilllll** metal, **in a** solution of !i parts c . HCJ. -to

1 pa.rt cone. HN03 wider reflux. 'l'he e.rsenic carrier solution (arsenic concentration of 10 JIS/mi) is prepared by disso1viug As.2o3 in 6N JN)3.

2. ·several crystals of 111141 may aid.precipitation *of* the

sulfides. • •

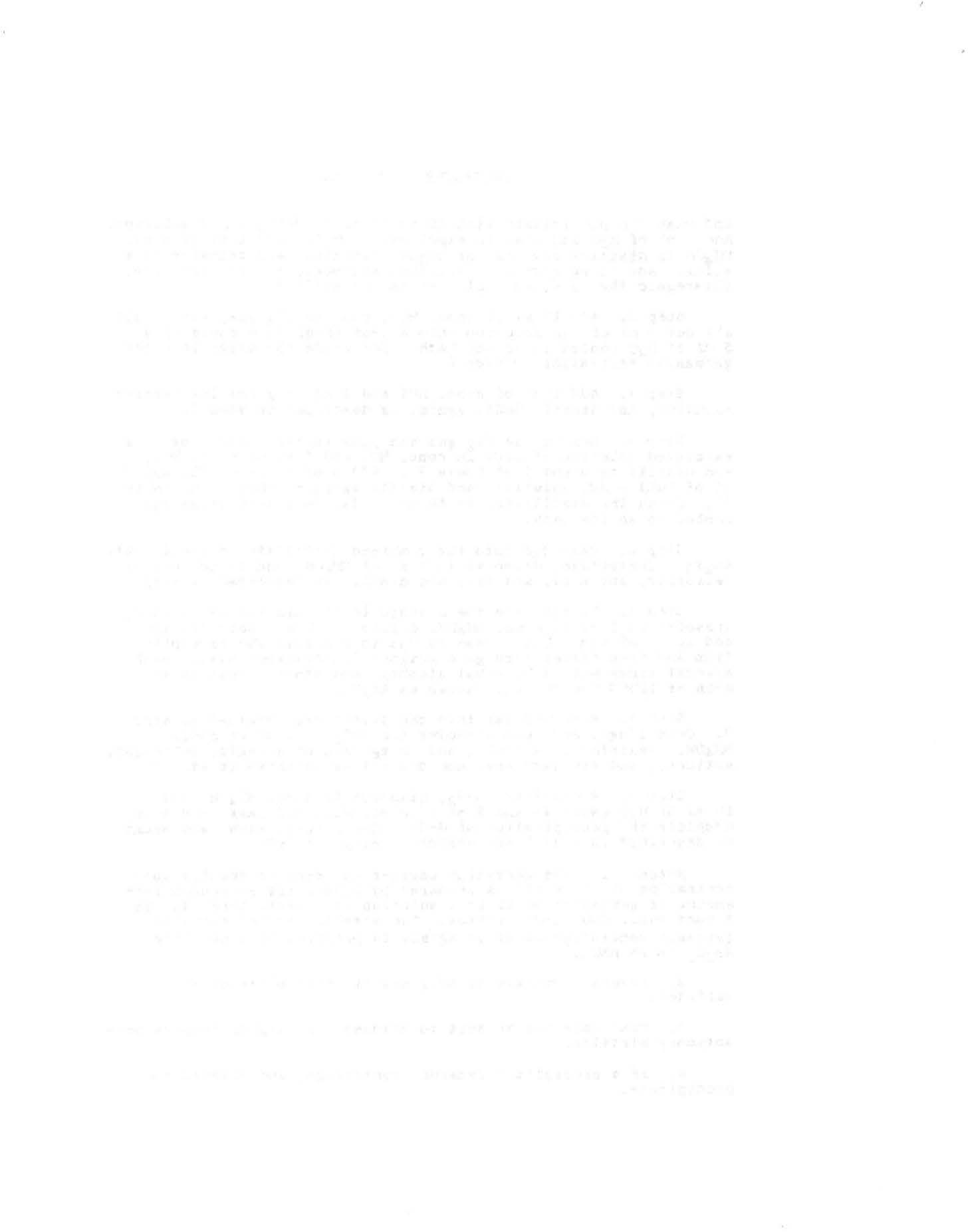
..

J. Take care not to heat to dryness. At higher temperatures

antimony distills. . · • · • •

1. *if* **a** precipitate remains, centrifuge, and discard the precipita.te.

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PROCEDURE 2

Source 11Tbe Separa1;ion of Germ&niWII. a.nd A.rs. \_ ic from a. Pission-Product Solution"

# R. J. Prestwood

AEC-IA 1721 (December 1954)

* 1. l;N'l'RODUCTIOH

It is scmetuies necessary to isolate both ra4ioarsenic and radiogermauium from the S8Jlle sample.. The.separation and isolation of these elements is readily accomplished by utilizing tb 4if­ ference ui \_ease of ex-traction of. **germiluium** CIV) a.iid ar&ellic (IIJ)• iodides into chlorofo:rm ·in the presence of h-,,tiriodi\_c acid.. •

As(V) carrier: Ge(IV) carriec: HCL: cone. lf2,so4- l.M

Hp04: 4M

RI: 47%

# Chloroform

* 1. REAGENTS

10 mg. As/ml. {added as Na so4•12H20 in H;aO)

# see Procedure 4.

J. EQUIPMBNT

# 125-1111 separa.tory funnels (four per sample)

40-:ml conical centrifuge tube : Pyrex 8320 {one per sample)

* 1. PROCBDlJRE

# Step 1. To the su,ple (Note 1) in **a** 12S-JIJl. sepa.ratory fu1J11e1. add 2.0 n1 of As(V) carrier (Note 2) nd 2.0 JIJl. of Ge(IV) carrier. (See germanium procedure 4 for the preparation of carrler.J Make the solution **3M in** HC1 **in.a** total volume· ot

10 to ·15 m1. Add J to 4 ml of n, HI and ·20 ml of CHci3• and

shake thoroughlr• (lbe As is c011plet:ely extracted into the CHCJ.a and the Ge **reu na** in the aqueous phase.) Drain the CRC13 layer into a clean separatory fwme1. To the aqueous **layer add an** additio:qal·1D.m1 of CHC13 am extract **again.** COllbine the atC13 extracts. 'l'he-CHC13 extracts containillg AsI3 **are saved** for analysis of arsenic.. •

Step 2. To the original aqueous layer **(Wllich** contains the Ge) add-20 1111 of 47% HI aid extract with 20 ml of CRCl3. Drain the CHCl3 layer (llhich-now contains tbe:Ge as G I4) **into a** clean separatory fuoael:. Extract the aqueous layer again..with .,J.o m1 of CHC13 and combine the ex-tracts. · • • • •

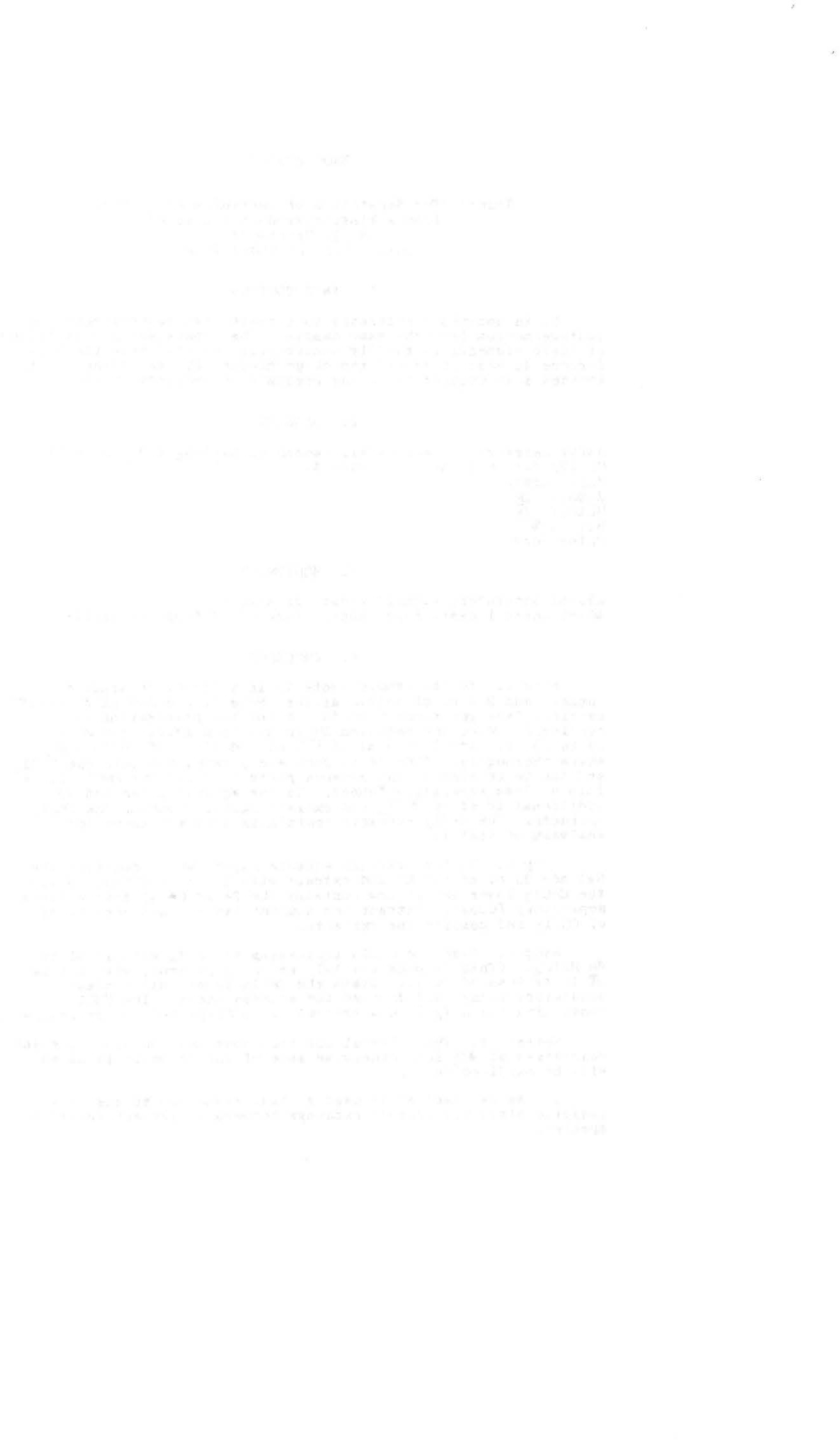
Step 3. Wash the CHC13 contaiDin« the GeI.4 with 10 ml of 4M HCl04. (This re.1DOYes a.Irf AsI3 which is p!:esent. with a loas o"f1%or less of.·Ge14.) Drain the CllCl.3 ·1ayer into a clean· sepa.ratory funnel and discar.d the **aqueous phase.** - **'lbe** .CHCl.3 •

# containing the GeI4 is now·treated as in Steps 6-10 of procedure 4.

Notes: l. The original solution **must.** not contain **appreciable** quantities·of NO - ion. otherwise some of the.HI W'.b.icb is·aMed Will be oxidized to-12. '

1. As CV) carrier is used so that reduction to the t.ri-: positive state ma promote exeha.nge between active &rid inactive species.

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PROCBDURB 3

sou.rce "The sepa:ra:tion of GeraaniW!I **·and** Arsenic

from a Pission-Product Solution"

R. J. Prestwood

ABC-LA. 1721 (Dece■ber 1954)

* 1. IN'l.'RODUCTION

Arsenic (III) is separated from **germanium** (IV) by preeipita­ tion asthe sulfide in hydrochloric acid medium containing fluoride ion, the latter strongly complexiq germanium (IV) **as** GeP = and preventing its precipitation. Prior to the sulfid precipitation arsenic (V) is reduced to the tripositiYe state

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by means of iodide iOJ1.

* 1. REAGENI'S

As (V) carrier: 10 mg.As/ml.. (added a.a Na s0 12B o in H tl)

4 2 2

Ge (IV) carrier: see Procedure 4. HCl:. 6M

HF: coiic.

83003: saturatedsolution.

H2S: gas

* Nal : .solid

Aerosol; o.• l'JL in Ha()

* 1. \_ E IPMENT

# 40"ml colrl.cal centrifuge tubes: Pyrex 8320·(two per **sanple)**

2'', 60° filter funnel (one per s8Illple) Centrifuge

Fisher burner

**Pipeta:** assotted sizes

No. 40 Whatman filter paper (9 cm)

# PROCEDURB

Step J.. To the fission-product aolutio **ina** 40-ml conical· centrifuge tube, add 2.0 ml each of As CV) and Ge (IV)· curiers. Make the solution 3 1:o SM in BC1 and the volume to 10 to 15 ml. • (Ni ti:ate .ion should be a'6sent, or present. only in small amant.) . Md SO to 100 mg of Nal and warm the solution gentl.y. Add 10 drops of cone. HP ILQd. saturate the solut:icni with H2s UJ1ti1 -the As2S3 precipitate has coagulated (time required is 3 to 5 min). centrifuge and pour the superua.te through **a** No. 40 Whatman filter in **a** 211, 60° funnel into **a** cl.eati celitri.fu,:e tube. The Aa2S3

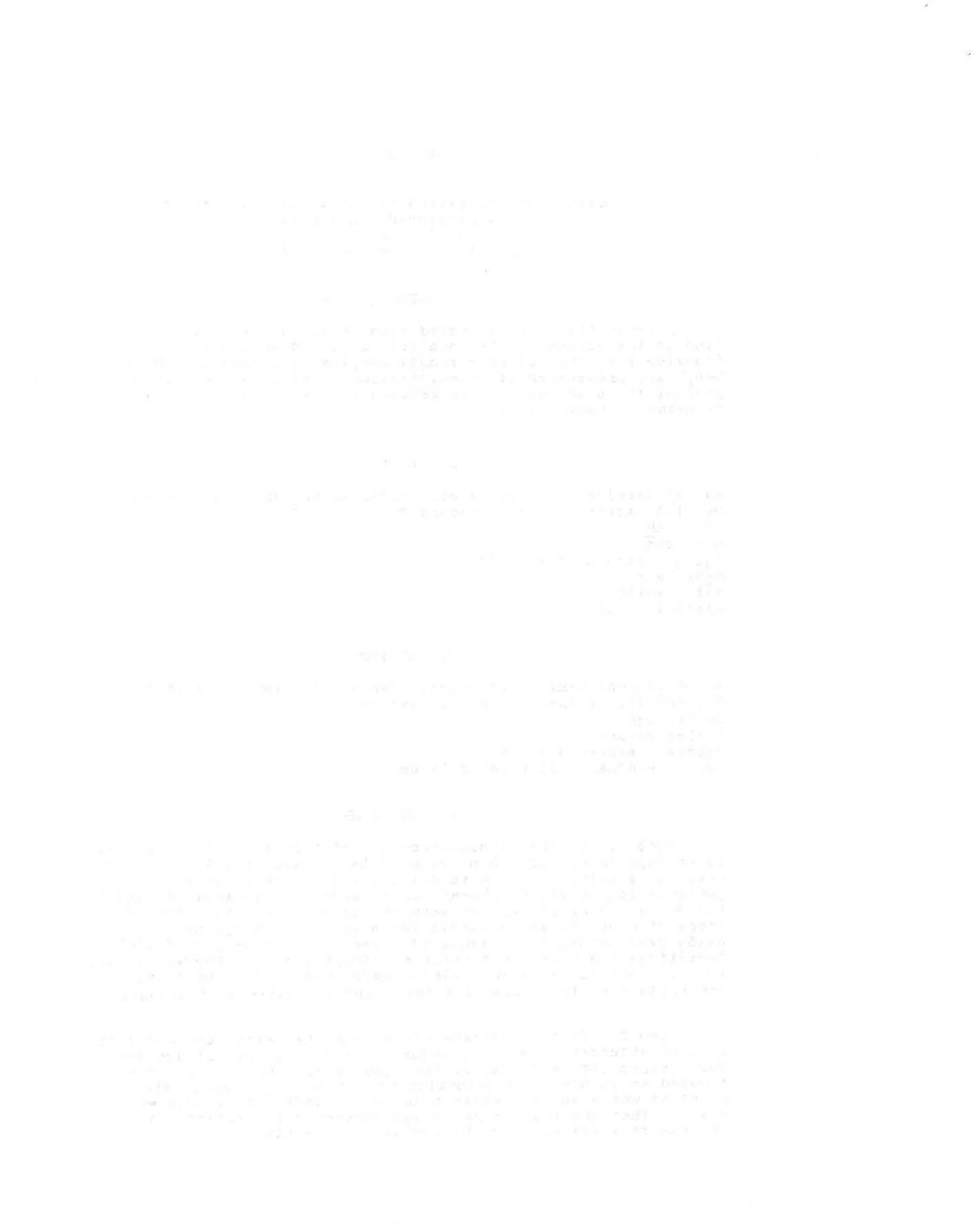
. precipitate is then!laved for tile·even-tua1 **a:a&1'fS!•** of **ueeaio.**

Step 2. To the filtrate contaim.ug the ge niu , add 5 to 6 .ml. of saturated H 3 and saturate with H2S•. Ceatrifage tile *GeS2* precipitate aal discard the supernate. ',l1le GeS2 is then treated a.a in Step 7 o.f. germ&niWR procedure **·4 up tlart>aO ·the** • poi.nt a.t which the ,enra.nate so1utioD is -de Just ac:C"dic with

HC1. 'l.'hen the :regul.ar wermaniu111 procedure ia carried. out i:a

# its entirety ('except·for che addition of Ge (IV) carrier).

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PROCBDURE 4

Soiu:ce "GerDLU.iuJD.11

R. J. Prestwood

AEC-LA. 1721 (December 1954)"

1. INTRODUCTION

In the separation of radiogermanium frOlll other fission products, acid sulfide, lanthanum fluoride, aud barium sulfate scavengillgs are performed in the prese ce of fluoride ion which keeps 1ermanium in solution as the GeF6 ion. Thefluo complex

is then destroyed and ser:mani11111 distilled as tbe tetracb1oride

in **a** specia1ly designed lllllltiple still. Gepn&niwa is finally precipitated and JD.ounted as the sulfide GeS2. The chemical yield is 80 to.90% and the analysis of samples in quadruplicate requires about four hours. •

1. REAGBNrS

Ge (IV) carrier! 10.00 mg. /1111. - see Preparation of Carrier As Carrier: 10 mg.As/ml (added as Na. •12H2.\_0 in H2o)

Ba Carrier: 10 **mg Ba/,-1** (added as Ba{Hl3J2 i1CH20)

La carrier: 10 **:mg La/ml (added as** La(M:>3)3 • i() inH2O)

CU Carrier: 10 **mg Cu/ml** (added as Cu(N:'l3)2•6H;aO in R20)

Zr Carrier: 10 **mg Zr/ml (added** ai!I.Z.rO(N:'13)2•21120 in J.!!\_Hti'.}3)

HC1: 4.5 • S.SM

HCl.: cone. -

HI: 47 aqueous solution

*H2S04:* co.uc.

HP: ·cone. .

H3B03: ·saturated solution

NR40lh cone.

J!2S: gas

t:HJOH: anhydrous

1. .BQUIPMB.NT

Fishe-r burner Drying oven Centrifuge

B1ock for holding centrifuge -tubes

MountiDg plates

Forceps

Pipets: assorted sizes .

**50 ml beakers (two pe·r sample)**

Ground-off Hirsch funnels: Coors OOOA (one·per, **sample)**

Filter chimneys **(one** per sample)

Filter **flasks** • •

No. 40 **Wbatma.n** filter **pe.per (9** cm) • • • • . No. 42 Whatman filter circles: 7/8" diameter-weighed 2", .60° **fwmels (fou.r per** &lllDPle) .

40-llll conical cent-ri.fuge tubea1. yrex 8320 **(two per aample)**

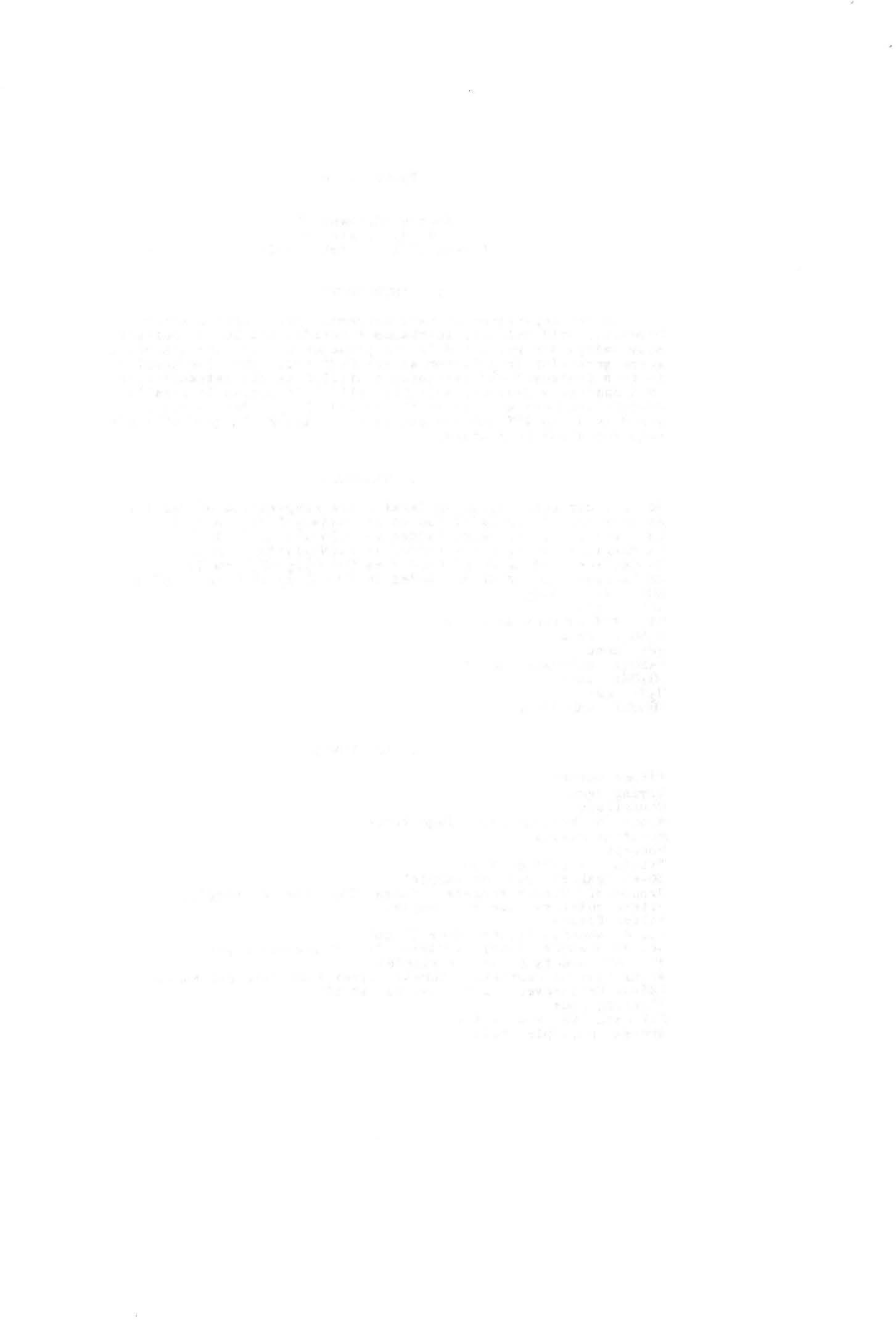
12S-lll Brienme,er flask (five per sample)

Stirring rods

Oi1 bath (J.0" x 4"x 4•t)

·special 1111111:iple still

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PROCEDURE 4 (Continued)

1. PREPARATION OF CARRIER

Fuse 14.4092 gm of c.p. GeO with 30 gm. of Na20J3. Dissolve

the 2

melt in H?<) and dilute to 1 :Citer. Permit to stand for 24 hours and filter. The solution contains 10.00 mg Ge/ml and is used as a primary stamard.

* 1. 'PROCEDURE

Step 1. To the sample in a 125-ml Erlenmeyer flask add

the following: 2.0 ml of s1:andat:d Ge carrier; 1 ml of As carrier;

1 ml of Ba carrier; 1 ml of Cu carrier; 1 ml of La carrier; 2 ml of 47% HI solution;. and 1 ml of cone. HP. Make the solution neutral by the addition of cone. NH40H, add 10-20 drops of cone. H S04, place on a steam bath, and saturate with H2S for a few m1nutes.

Step 2. Filter into a clean 125-.Ull Erlenmeyer **flask** through No. 40 What.man filter paper in a 2", 60° funnel. Wash *the* pre­ cipitate with a small quantity of water. Discard the precipitat.e.

*Stet>* 3. To the filtrate add 10 drops of La and 1 1111 of Cu carriers and saturate with H2S on a steam bath. Filter as in Step 2 and wash the precipitate.

Step 4. To the filtrate add 1 ml of Cu carrier a.nd saturate with H2S in the cold. Filter as in Step 2 and wash the precipitate.

Step 5. Repeat Step 4.

Step 6. To the filtrate add 10 ml of cone. HCl and 10 ml

of saturated H3003, and saturate with H2S. Transfer to 40-ml conical centrifuge tube. centrifuge the Ges2 precipitate, and discard the supernate (Note 1).

Step 7. Dissolve the GeS2 in 1 ml of cone. NH40H and

dilute to 15-20 lll1 with H20. Add 4 drops of Zr carrier, centrifuge and discard the precipitafe. Make the supernate *3M* with HCl. saturate with H2s, centrifuge, and discard the supernate.

Step 8. Slurry the GeS2 with 4.5-5.SM HCl (Note 2) and transfer the solution to the special still7 (The total volume of a·cid used should be about 15 ml.)

Step 9. Distill the GeCI4 on an oil bath at 120° into a 50-.ml beaker containing 5 ml of *4.5-5.SM* HCl saturated at room temperature with H2S and placed in an ice bath (Note 3). 15-20

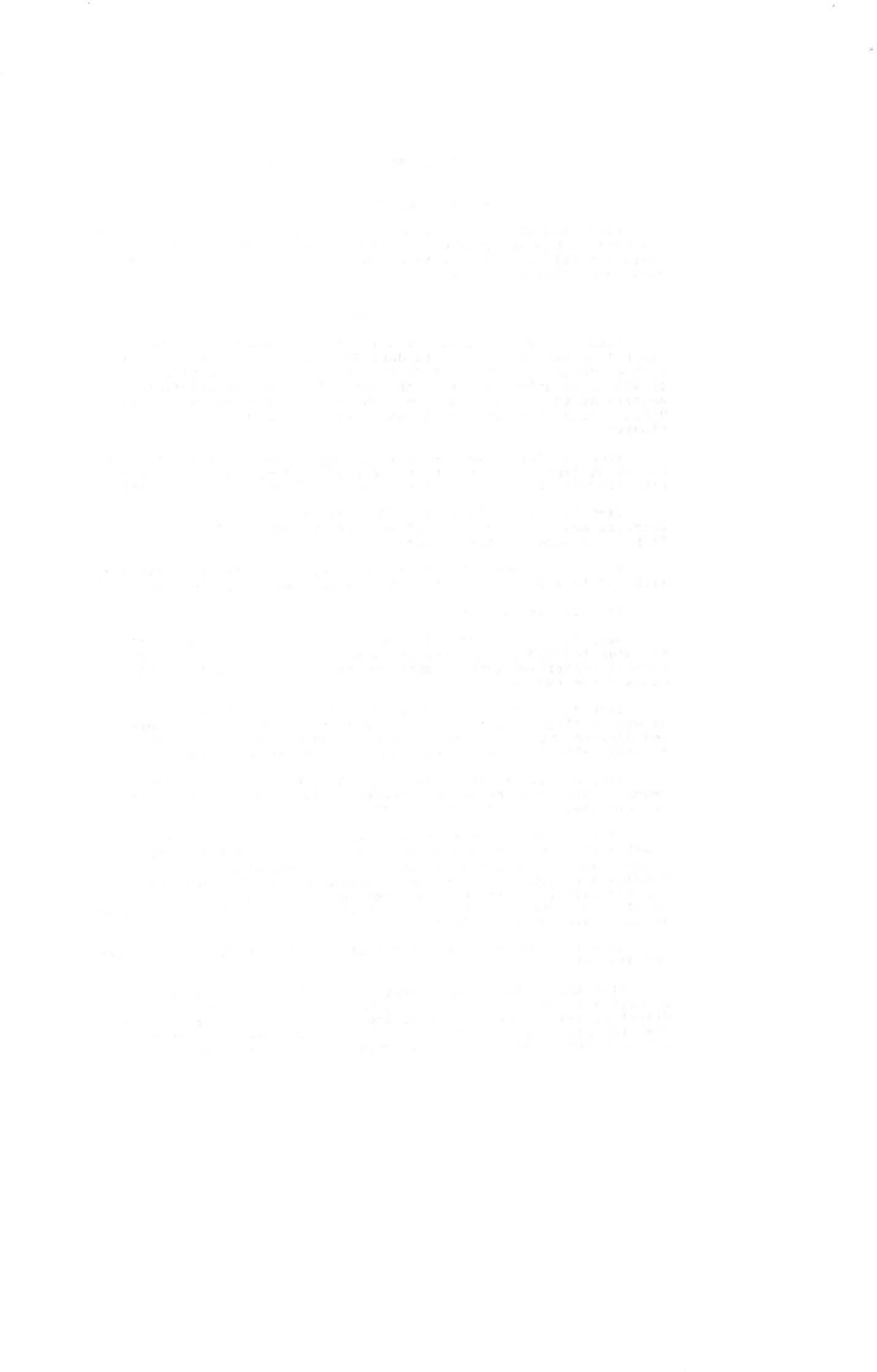
minutes are required before Gec14 begins to distill over and then the distillation must be maintained for an additional 10-15

:minutes to ensure completion. Very c1ose to 100% yield **is obtain d in** the distillation process.

Step 10. Transfer the distillate to another still and repeat the distillation.

Step .11. Saturate the receiver with His and filter the precipitate onto a weighed No. 42 Whatman f1lter circle, 7/8" diameter, using a ground-off Hirsch funnel and a filter chi ney. Wash the precipitate with anhydrous CH30H and dry in an oven at 110-120° for 10 minutes. Cool, weigh· as Ges2, and mount.

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PROCEDURE 4 (Continued)

# Notea1 1. A 'W&ter-clea.r supernate is not ordina.ril7 ob­ tained upon centrifugation of the GeS2 uuless the mixture is perlllitted to stand for several hours. Since the latter is not p%'acticable9 and unce the losses are inaipificaat, do not hesitate to discard a slightly turbid supernate **froa a** GeSa prec:ipita.tiou.

2. The concentration of HCl 'IIIIISt not exceed that of the constant boiling lllixture or GeCl4 will e cape during the distilla­ tion unless the deliTery tube is below the surface of the receiving liquid. ·so long as the HCl concentration in the still is leas than that required for the constant boiling mixture, only **water** am no GeC14 is distilled. **As soon as** the composition of the still reaches that of the constant bolling :mixture, 1!!11 tbe GeC14 com.es over rapidly. At bigher HCl coace.atratioaa the GeCl4 is immediately swept out with HCl **gas.**

J. 'lb.e lbS ls present in the receiver to show (by the formation of white GeS2,) when GeC14 begins to distU.l.

# PROCBDURB 5

Source "Preparation of Radioactive Germanium Radiochemi.:ally Pure"

**A. *N.*** B&raboshkin

Zhur. Neorg. **Xhim.** 29 2680-1 (1957)

(translated from Preach Report CBA Tr.-R-635 by Elyse R. Marinsky)

Radioactive **germaaium,1** provided as the metal by industry, is not radiochemically pure. The quantity of residual activity is· very great in certain s ci:mens and 111akes up a large pe:ccent of the total·.radioaetivity.2 A part of ttte mixed radioactivities bas more energetic radiation than that of ger111&niUJ1 and likewise a greater balf-U.fe (11.4 days). With ti.,ne the contributlon of the residual activity increases.

Por the utili2a ion of radioactive **ger.an.iu**■ **in** physical or chemical processes, it is necessary to avoid erroneous con-· clu.sions by first purify:iq the .sllllples reB>Ving the extra i:ad.ioa.ctivity.

'lbe author has teated **two methods** of purification. nie first COllSisted of a nri.ce-repeated precipitation of **germanium** disulfide **from** a & N sulfuric acid aolutioa **a-ad the** ulti•ate dissolution of the precipit&te on the filter **paper with** U1110nia.

The aecoud !lll!!thod aonaiats of the distillatioB o.f **genaauiwa**

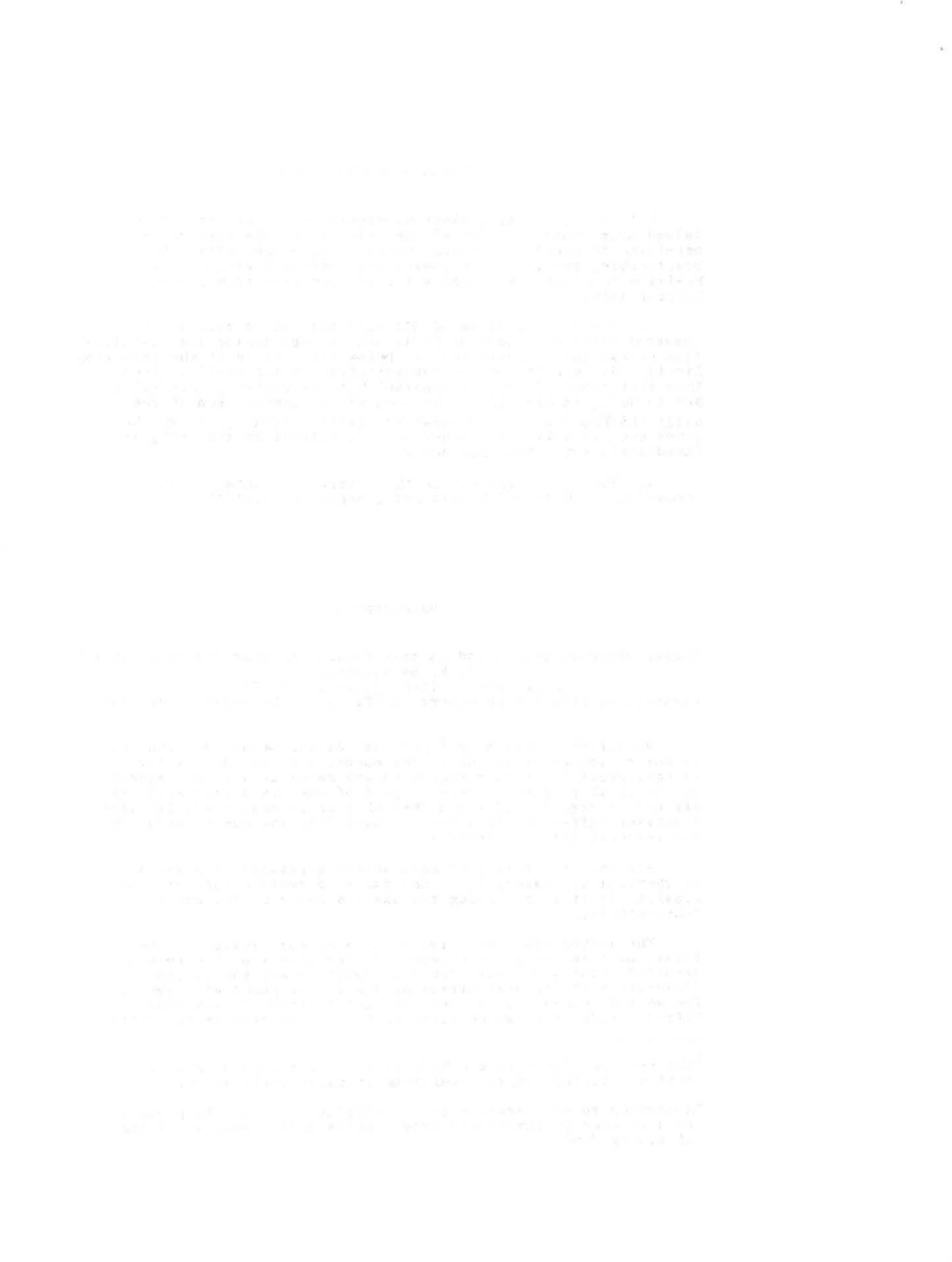
# tetrachloride fros hydrochloric acid. One d.eter•ines the effect-

1The isotope 71Ge having a half-life of 11.4 daya decays by

# orbit&l electron capture ellittlnjJ characteristic x-rays.

2According to measurements of the r&dioactivity of the products by a counter of the Met-17 type, haviq a .mica window thickness of *4.S* YJB./c-2.

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PROCEDURE 5 {Continued)

ivenesa of purification am the desree f purity fro:m alimimm absorption curves of the radiation•.

The purification by the aeco:nd 111et od is more rapid aud has been accompli ed in the following manner: a sample of radio­ active gerllllllliwa metal was oxidized. during its griuding in an agate 110rtar under a 1:i mixture of concentrated hydrogen peroxide and 3 N **potaasium.** hydroxide. After 24 hours the contents of the mortar are.placed **in a** quartz crucible and are evaporated to dryness. Tile residue is dissolved in hot wa.ter. nie solution

is neutrali-d **with** concentrated hydrochloric acid and transferred to a roull4 bottoa flask of a.distillation apparatua. l'bere one adds the same yo1Ullle of hydrochloric acid (d .10) and proceeds with the distillation. GerJll&Jl:I.Ulll disulfide is precipitated in

the distillate with hydrogen aulfide. 'Dle preci ita e is filtered, washed with 3 N sulfuric acid saturated with hydrogen su1fide am dissolved on the filter with concentrated•a-onia. Hydrogen peroxide is added dropwise to the 8.llllloniacal solution until disappearance of the ye11ow color (due to the decoaposition of germaniWII disu1fide)\_. The solution is boiled until the decQ!llposi­ tion of excess h-ydrogen peroxide and the elimination of free a1111110nia; the filtrate is trans erred into a flask adjusting the volume to the.calibration iaar .

One sees that the absorption curves of the purified products develop according *to* the absorption law for monochromatic x-rays,

I = I0e-#'d • e;uog I0 = log I0 - 0.434)4d.

This fact shows that the two methods described provide in practice a radiochemically pure sample of the radioisotope 71Ge.

lbe principal part of residual activity is separated in

the fjrst stage of purification and reJ11&ins, either in the filtrate after \_precipitation of ihe disulfide, or in the non-volatile residue from the distillation of the tetrachloride.

'the author has not succeeded in identifying the elements of lo.ug half-life. Ho,rever,·he-has spec;l\_fied that they -are not coprecipitated.with hydroxidc·s of iron and zinc and the sulfates of calcilllll and barium.

PIWCEDURB6

S.ource "Cyclotron Targets:-Preparation and Radiochemical

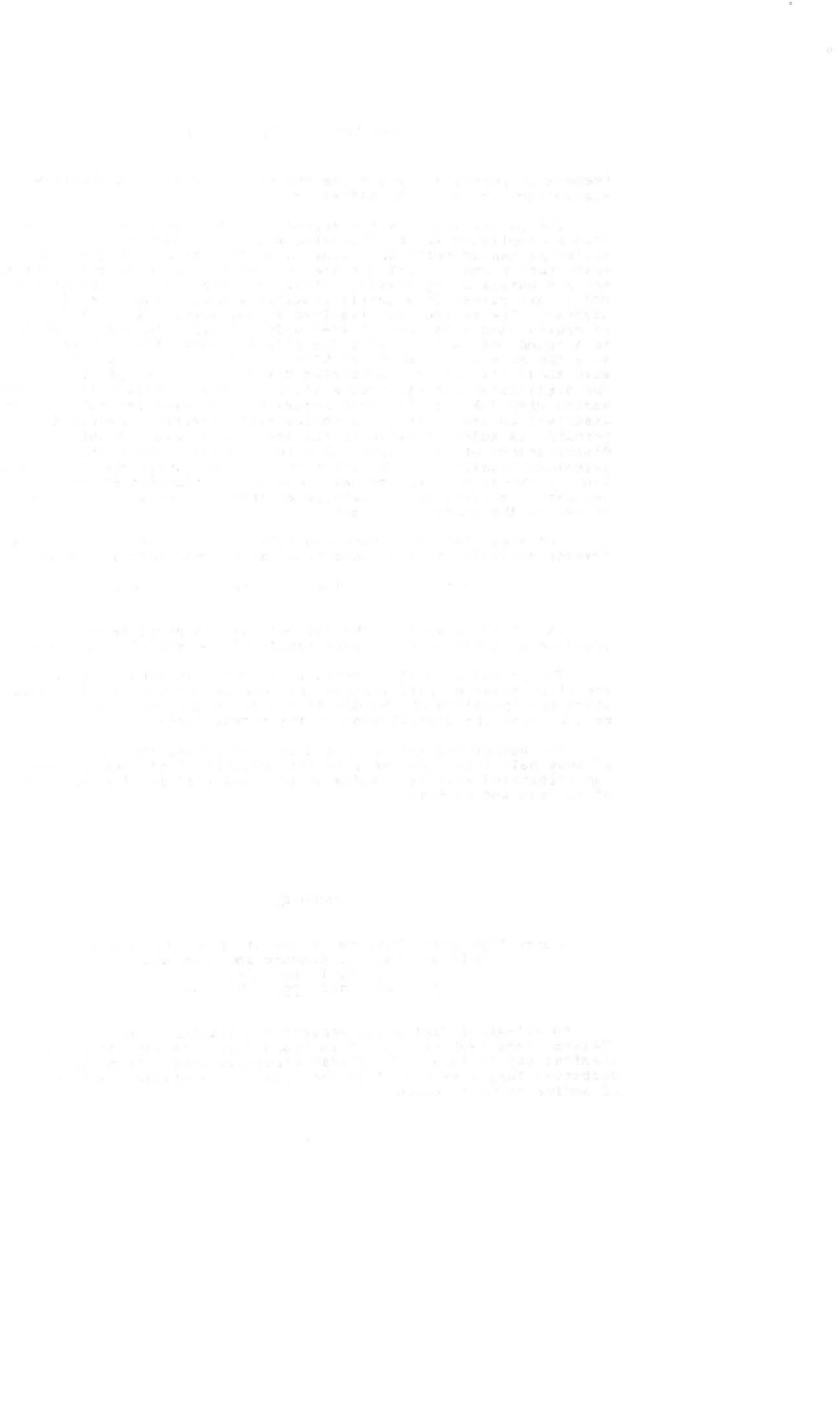
Sepa·rations. I. Arsenic and Germanium"

* J. w. Irvine, Jr.

J. Phys. Cl1em. ,.1!, 910 (1942)

\_ An all-glass distilling apparatus utilizing a modified Cla1sen flask (250 ml ) ha.s been found useful in several radio­ che ical. separations. 'Ib.e coarse germanium polllder from the bombarded target is placed in the flask and a stlllldard solution of sodium arsenite added.

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PROCEDURE 6 (Continued)

The target material is· treated with *25* ml. of 12 M hydro­ chloric acid, heated, and disso1ved by the dropwise addition of 30 per cent hydrogen peroxide. 'this operation requires 10-20 ml, of hydrogen peroxide and takes about *S* lllin.

GermaniWII is removed as the vo1atiie tetrachloride by distillation of the hydrochloric acid solution. Prequent addi­ tions of a few drops of hydrogen peroxide minimize reduction of As-t-5 to Att+l by. the hydrochloric a :l.d and co11ta.lllina.tiou of the **germanium** by arsenic as arsenic tricbloride. **When solids pre­** cipitate **from.the** solution in the flask, 10 ml.. of 12 **M hydro­** chloric **acid is added and\_the** distillation is **repeated. Glus** beads or pieces of tile will reduce **bWlpillf!'** of the·vtixture **during** distillation.

Radiogermanium is recovered from the first two distillates Y precipitation as the sulfide. Souie radioarsenic •will be fouud

1n these distillates. Although it usually amounts to no more

than a few .microgra111s, its activity is lar\_ge so it must be removed.

Several milligrams of arsenic as sodiUDI arsenite *µe* added to the combined distillates and precipitated with ammonium hypo­ phosphite with gentle heating. The·retallic arsenic is filtered off, some more carrier added, and the precipitation repeated. Activity measurements on the precipitates i:adicate when the radioarsenic is completely removed. TWo precipitations are usually adequate.

Germanium sulfide is then precipitated by adjusting the acidity to approximately 6 Mand passing a rapid stream of hydrogen sulfide through.the hot solution. **The white ger:in.aaiu** sulfi'de coagulates well and is easily ·filtered.

PROCEDURE 7

Source 11Che ca1 Separation of Fission Products"

G. Wilkinson and W. E. Gru'llltllitt

. Nucleonics, Vol. 9, No. 3, pp. *54-55* (September, 19.51)

Fission-Product P.rocedures Germanium,·Arsenic, and Selenium

The ehemica1 procedures used were those of Noyes and Bray.

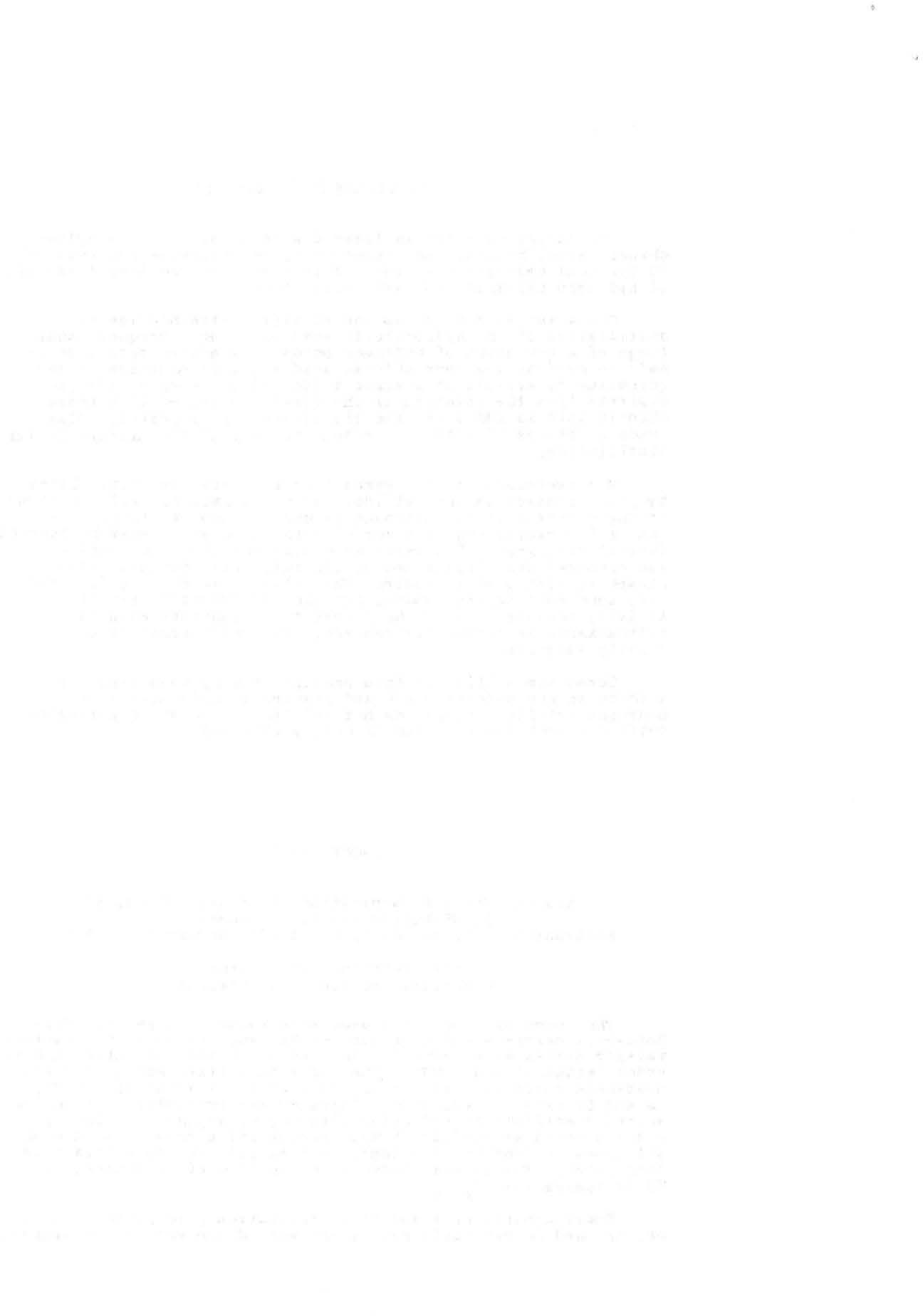
Hold-back carriers and carriers for Ge, As, and Se in the.various va1ency states were added to the.U solution aud the solution bailed under- ref1ux with 40% HBr.. ni.e first distillate· was\_co11ecte

hold-back carriers were added, and a.Fedistillation of tHe Ge, As and Se broJddes was :made. SeleDiu ·was precipitated from the second distillate *by* reauction with S02 or lffi21,JH•HCl•. 'Ibe *As* was recovered as sulfide by H2s preeipi ation.rrom SN HCl-HBr soluti.ons or from HP solutions, Ge re111&ining in\_ the filtl'ate **in ..** both cases. The Ge was recovered as sulfide fter relll.OV'.al of HP by fuming with H3SU4 • • •

Sraall amounts of I and Sn contaminat:ion were. present in the

Ge. As, and;se precipitates. 'lb.ese were dissolved in\_the minimwn

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PRO KDORE 7 (Continued)

of HNOJ 1- carrier added, and AgI precipitated. The solutions were th;n made 6N in HCl;-se was agai reduced by NH20H•HCl and As attd Ge were precipitated as sulfides from )2N HC1.

Inactive samples of Ge. As, and *Se* were.obtained from samples containing over 106 disintegrations r minute of gross.fission. products, with chemical yields of 40-60%.\_ Since no act ve species were found, tbe complete ss of radiochemical exchange 1s aot known.

PROCBOURE 8

Source "Some Geochemical Determinations Usini;: Radioactive

and Stable Isotopes" •

A. A. Smales, D. Mapper, J. Morgan, R. K. Webster and A. J. ood

A7Conf.1S/P/282 •

The deter:mi'nation of copper, chromium, **germanium,** arsenic a.lid antimony in iron meteorites by neut on activation

Sample Treatment

Cubes of about 3-4 mm side were cut frolll the meteorite samples, care being taken to avoid inclusions as far as possible.· 'the

pieces were cleaned in dilute acid• washed and d.r:ied before being sealed into small si1ica ampoules. TW0 solutions, the first of 300 *pg* As/ml. and 10 jAg Sb/1111, and the second of 10 mg Cr/ml,

3 mg Cu/llll.and 6 mg Ge/ml, were used as standuds, quantities of aboat 0.1 m1 being weighed and sealed iD1o silica ampoules.

Samples and stam!ards were loaded into standard aluminium ca.ns

and irradiated .in HEPO for two weeks.

After trradiation the samples were surface cleaned· by rinsi'hg with dilute acid, and then dissolved in.a mixture of hydrochloric, nitric, hydtofluoric and perchloric acids, in the presence of

SO mg. arsenic, 40 111g. anti•ony, 20 mg. copper and germani'IJJII, and 10 mg. chromium as inactive carrier solationa. After taking to fW11eS of perch1oric acid complete dissolution of the samples had ta.ken place. 'Ihe presence of hydrofluoric acid ensured that no losses of volatile ger.mauiWII coJDPounds occurred. .

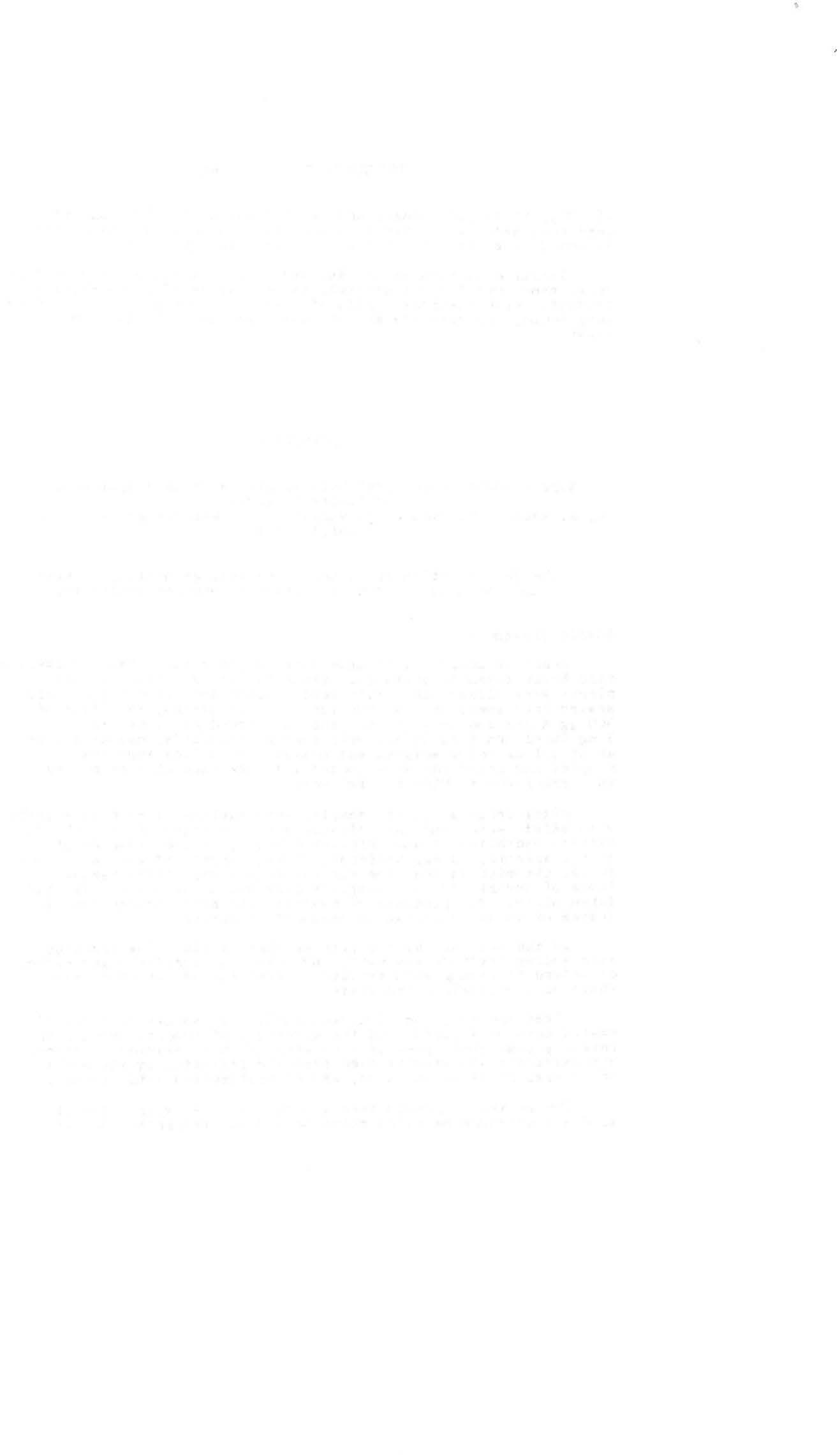
* In the case of the troi ite samples; a s.i.Dtering techai(l\le

th sodium·peroxide was used, auy imioluble residue being either d1ssol.ved in atrong acid lllix1:ure, containing HP, or elae being fused with potassiUJI bisu.lphate.

After the addition of hydrochloric acid so1ution, *5* gm. of oxalic acid were added, a.ad the sulphides of ccpper, arsenic alld antimony completely-precipitated with 2% thioacetam.ide solution. The germanium was p ecipitated from the filtrate, by the addition of 25 :mls. of 5% tannic acid; and neutralization·with amonia.

The germanium precipitate w s then treated with hydrochloric acid and hydrogen peroxide solution and the tetrachloride steam.

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PROCEDURE 8 {Cont nued)

distilled inthe pre-sence of arsenic, iron, cobalt and copper b.old-back carders. Ger'Bl&Dium sulphide was fiaa11y precipitated from the disti late and then·D.OUnted on aluminiu,n cou-nting trays.

The copper, arsenic and antimony sulphides precipitate was­ purified as previously described and similarly prepared for couoting, as cuprous thiocyanate. ele11tenta arsenic and antimODy trisulphide respectively.

The residua1 solution containing chr01T1ium was oxidized with nitric and perchloric acids, so that all the organic matter was destroyed and the chromiWII conve.rted to chromate. The latter was the.u ether extracted as the··peroxy-com.plex, purified fr0t11 contaminating iron and cobalt activities, and finally prepared· for counting a.s barium chromate.

Ali the counting was performed on automatic f,.counting equipment, using thin-em-window Geiger-Muller tubes. \'lhere advantageous, as for example for Slcr, use was made of a *Y­*

spectrometer with a 100-channel tnilse height analyser. Radio­ chemical purity of the sample sources was checked in all cases, by decay measurea!DtS; in addition, **/-energy** a6"sorption measure­ ments and Y-sp·ectrometer 111easw:e"111e.11ts were also used, where

. appropriate. Self-abso ption corrections were applied in the

am

/-counting of 51Cr 64Cu.. . . . •

'lb.e nuclides used wei:e 12.8 hi:·. 64cu(l.7 x 106), 27.8 day 5lcr(l.4 x 105) 12 hr. 77Ge(7.6 x 103), *2h.5* hr. 76As(2 x 106)

and 2.75 day 12 sb(1.l x.106), the figures in brackets being the activity due to that nuclide in units of disintegrations/minute/J,lg of elements, after two weeks irradiation inthe Harwell pile HEPC> . at a neutron flux of 1012n/cm2/sec:. In some ca.ses alternative germanium nuclides were **wsed,** 82 min. 75Ge(8.2 x 104) or 11.4 - day 71Ge(l.7 x 10S). •

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