the alloyd corporation

CAMBRIDGE MASSACHUSETTS

Unclassified

Research on the Production of Ultra-Pure Molybdenum

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Submitted by:

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I. INTRODUCTION

During the current reporting period, experimental work continued with emphasis on the purification of the Molybdenum Pentachloride. The work included the design, construction and operation of an upscaled sublimation system and the continuation of the zone refining experiments coupled with new $MoCl_5$ distillation experiments.

II. EXPERIMENTAL WORK

A. Distillation of Molybdenum Pentachloride

In view of the fact that the initial distillation experiment (see prior report) indicated the quantitative removal of two elements (Ni and Cr), an improved system was designed, built and has just been placed in operation (see Fig. 1).

B. Sublimation of Molybdenum Pentachloride

The sublimation work under modified conditions was resumed.

1. Upscaling - The newly designed system is capable of handling up to 3 lbs. of $MoCl_5$ per day.

2. Operating conditions -

   a) The system operates with Cl_2 (Chlorine gas) as the carrier gas (see Fig. 2 and

2a for schematic and pictorial representation of the system).

b) The temperatures in the sublimation chamber varies in the 170 - 200°C range.

c) The tube connecting the sublimation chamber to the condenser is held at 220°C ± 10°C.

d) The condenser is kept at room temperature.

e) This is a fractionating sublimation in which only the middle 80% is used. The first fraction (10%) and the residue (10%) is discarded.

Analytical data as obtained by the Jarrell-Ash Company on the first run after establishing correct operating conditions, is presented in Table I below together with the analysis of the starting material.

TABLE I

Element:	Starting Material $MoCl_5$	Sublimation of $MoCl_5$
B	ND	ND
Si	< .0015%	< .0007%
Fe	.0005-.005%	.0002-.002%
Mn	<.001%	ND

Table I - Continued

Element:	Starting Material $MoCl_5$	Sublimation of $MoCl_5$
Mg	<.00007%	ND
Cr	<.003%	<.0007%
Ni	<.0007-.007%	<.0007%
Al	<.001%	<.001%
Cu	<.001%	<.0003%
Na	.001-.01%	.001-.01%
Ca	<.001%	<.001%

Five additional sublimations were conducted in the reporting period and some 15 lbs. of purified material was made. This material is to be used for further processing to include zone refining and reduction. The analytical data on this material has not as yet been obtained but should be of the order of purity given in Table I or better.

C. Zone Refining of Molybdenum Pentachloride

1. Horizontal zone refining

Experimental difficulties with the horizontal zone refining system appear to be extreme. Although the horizontal approach is not completely abandoned to date, indication that both operational and analytical are that this system dies not hold great promise.

The following approaches on horizontal zone refining were conducted.

a) Runs were conducted with crushed $MoCl_5$ held horizontally in closed tubes. Material transfer was reverse.to the direction of zone travel making geometry of the system unsatisfactory and causing breakage. See Fig. 3 for exact operational changes observed.

b) In order to avoid mass transfer represented above (Fig. 3) staring load was modified as seen in Fig. 4. This approach also did not prove practical as breakage still took place.

c) In order to further reduce mass transfer, a counter flow of chlorine (Cl_2) was investigated (see Fig. 5 for system). Mass transfer was not suppressed and continued in the direction contrary to zone heater motion and leading again to breakage.

2. Vertical zone refining of $MoCl_5$

This approach appears with additional experimentation to be the method which we shall utilize in the final production of high purity $MoCl_5$. The system used is a further modification of the one described in the last report. The modifications were indicated in the course of experimentation for better and trouble free operation. See Fig. 6 and 6a for schematic and pictorial representation of the system.

In the reporting period all experiments were conducted with the improved vertical zone refining system. A number of experiments were conducted using both as received starting material and sublimed material. The analytical data on these experiments is given below, Table II and III.

TABLE II

Analysis of Ingot Sections* 1 to 5

Exp. No. 210461

Vertical zone refining of $MoCl_5$ as received from Climax Molybdenum Company.

	(1)	(2)	(3)	(4)	(5)
B	ND	ND	ND	ND	ND
Si	<.0001%	<.001%	<.0005%	<.0007%	<.0007%
Fe	.00001-.0001%	.0002-.002%	.0002-.002%	.0002-.002%	.0003-.003%
Mg	ND	<.00007%	<.00007%	ND	<.0002%
Cr	<.0007%	<.002%	<.0005%	<.0003%	<.001%
Ni	<.0003-.003%	<.0007-.007%	<.0007-.007%	<.0005-.005%	<.0007-.007%
Al	<.0005%	<.0005%	<.0007%	<.001%	<.0007%
Cu	<.00001%	<.0007%	<.0005%	<.001%	<.0015%
Na	.001-.01%	.001-.01%	.001-.01%	.001-.01%	.001-.01%
Mn	ND	<.0001%	ND	ND	<.0002%
Ca	<.0003%	<.0015%	<.001	<.002	<.001

* 1 is top section

TABLE III

Analysis of Ingot Sections* 1 to 4

Exp. No. 260461

Vertical zone refining of sublimed $MoCl_5$.

	(1)	(2)	(3)	(4)
B	ND	ND	ND	ND
Si	<.001%	<.002%	<.001%	<.001%
Fe	.00001-.0001%	.0002-.002%	.0005-.005%	.00001-.0001%
Mg	<.00007%	<.00007%	<.00007%	<.000001%
Cr	<.002%	<.001%	ND	ND
Ni	<.0007-.007%	<.0007-.007%	<.0007-.007%	ND
Al	<.001%	<.001%	<.001%	<.001%
Cu	<.0003%	<.0007%	<.001%	ND
Na	ND	ND	ND	ND
Mn	ND	ND	ND	ND
Ca	<.0005%	<.001%	<.00001%	<.0005%

* Section 1 top

III. DISCUSSION

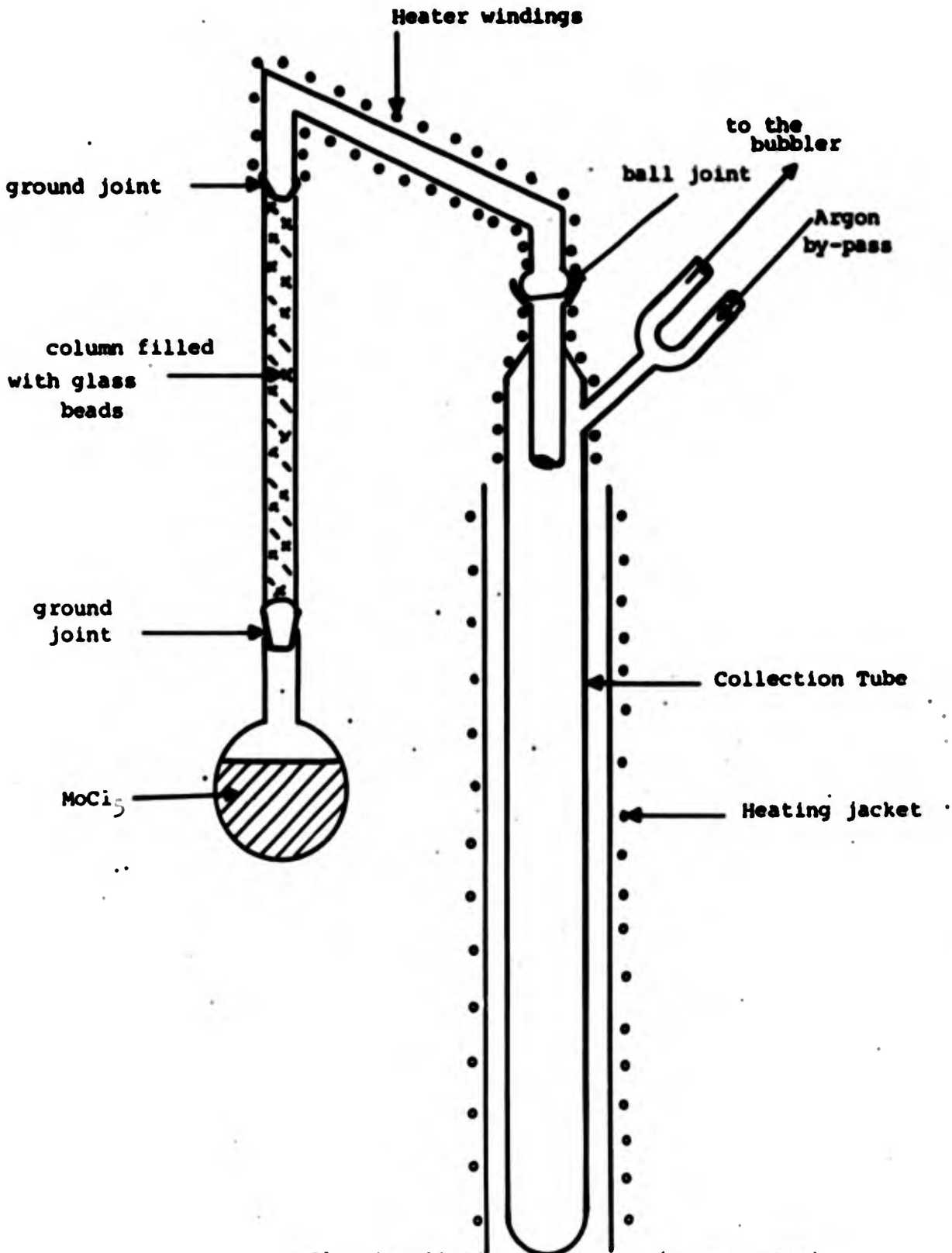
The analytical data obtained since the last reporting period together with all information obtained to date indicates the following most likely feasible production scheme (see Fig. 7).

All above steps and operational material problems with the chloride purification are believed resolved. However, additional work is needed in the establishment of optimum reduction conditions.

IV. WORK PLANNED IN NEXT REPORTING PERIOD

The experimental scheme established as the apparent final production approach will be run throught in the next period. It is felt that additional operating experience is needed and it is also hoped that some further improvements of the material would result. As the best reduction conditions have not as yet been established, we intend to study this phase more actively in the next reporting period.

FIGURE 1



$MoCl_5$ Distillation Apparatus (Pyrex glass)

FIGURE 2

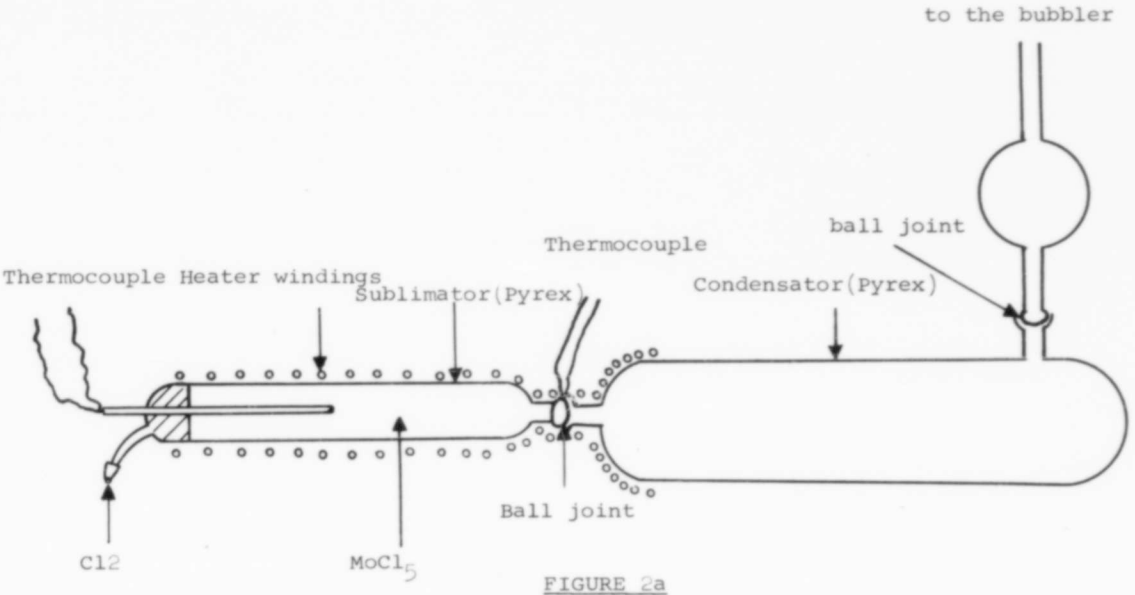


FIGURE 2a

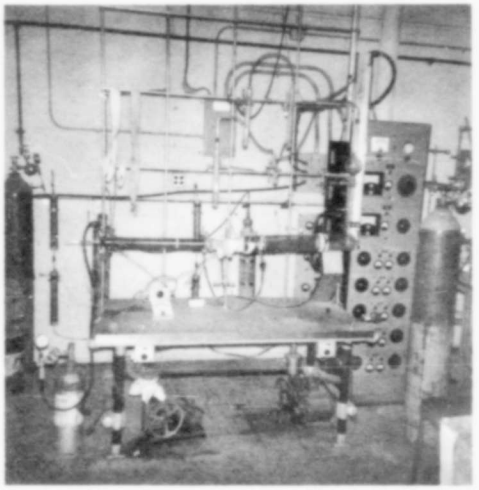
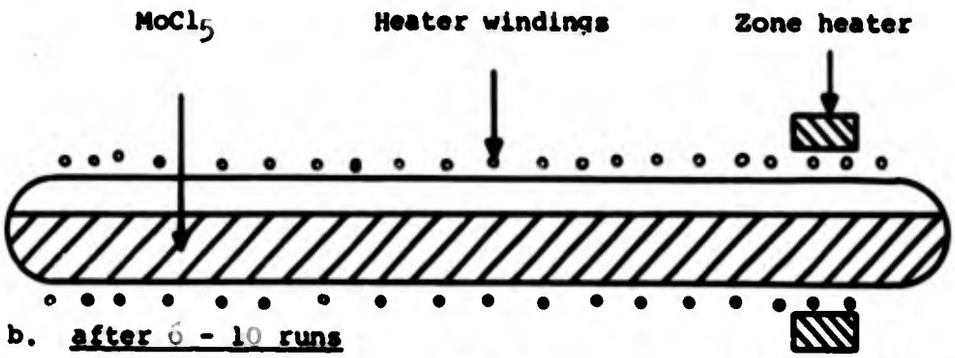


FIGURE 3

a. Starting load



b. after 6 - 10 runs

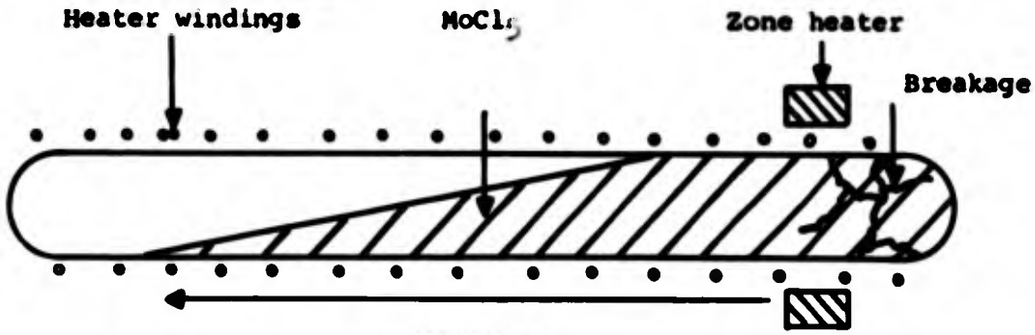


FIGURE 4

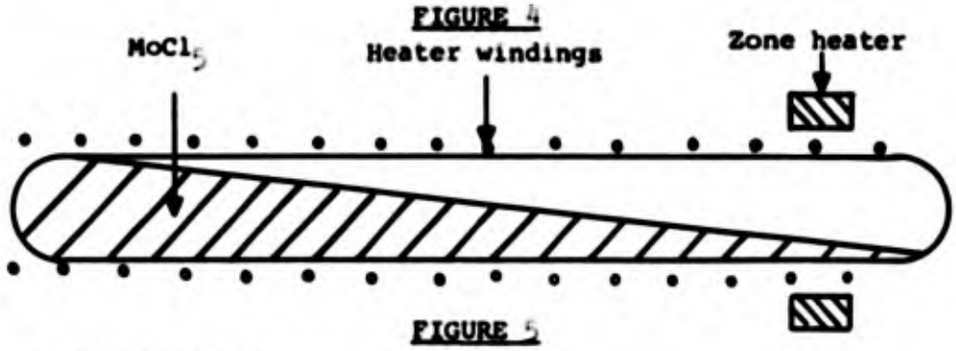


FIGURE 5

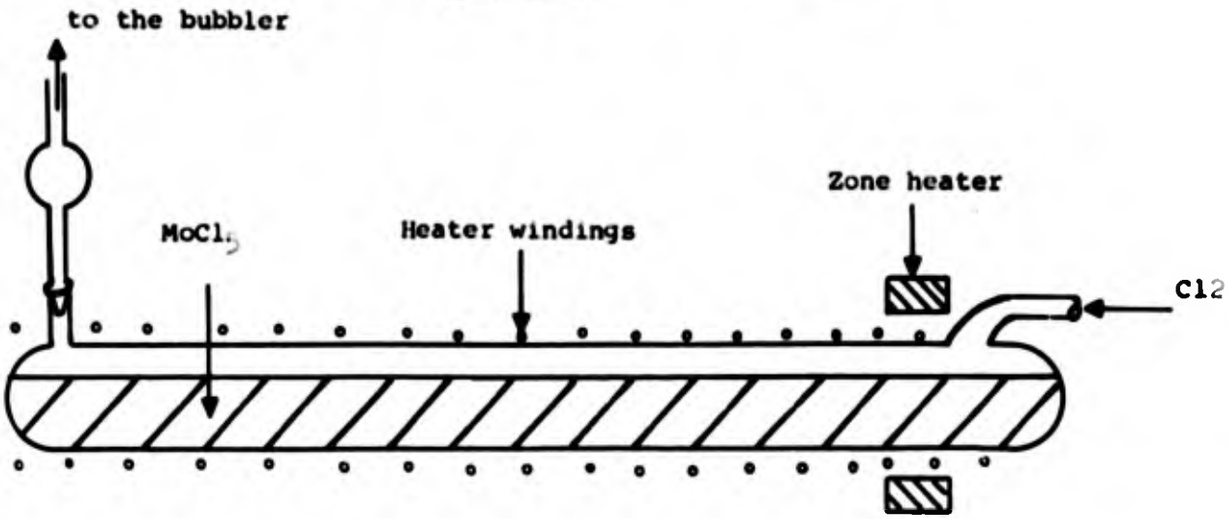


FIGURE 6

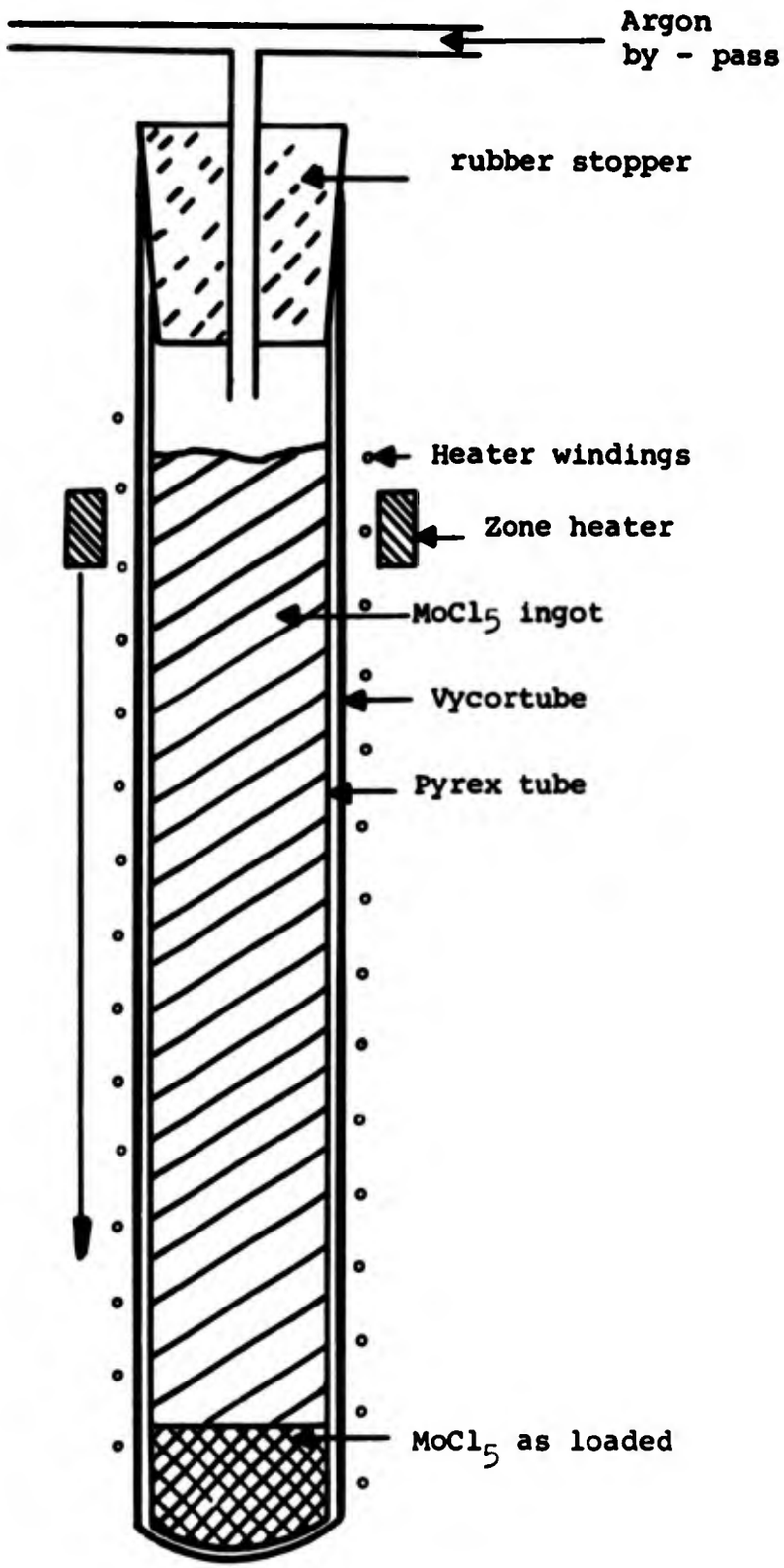
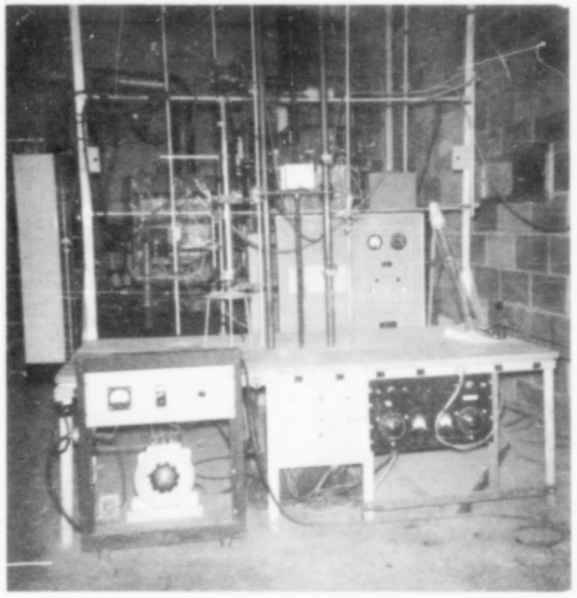

Argon
by - pass
rubber stopper
Heater windings
Zone heater
$MoCl_5$ ingot
Vycortube
Pyrex tube
$MoCl_5$ as loaded

FIGURE 6a



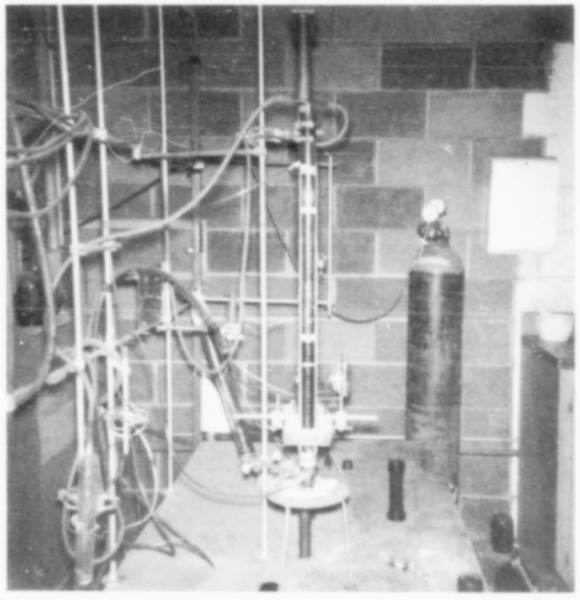
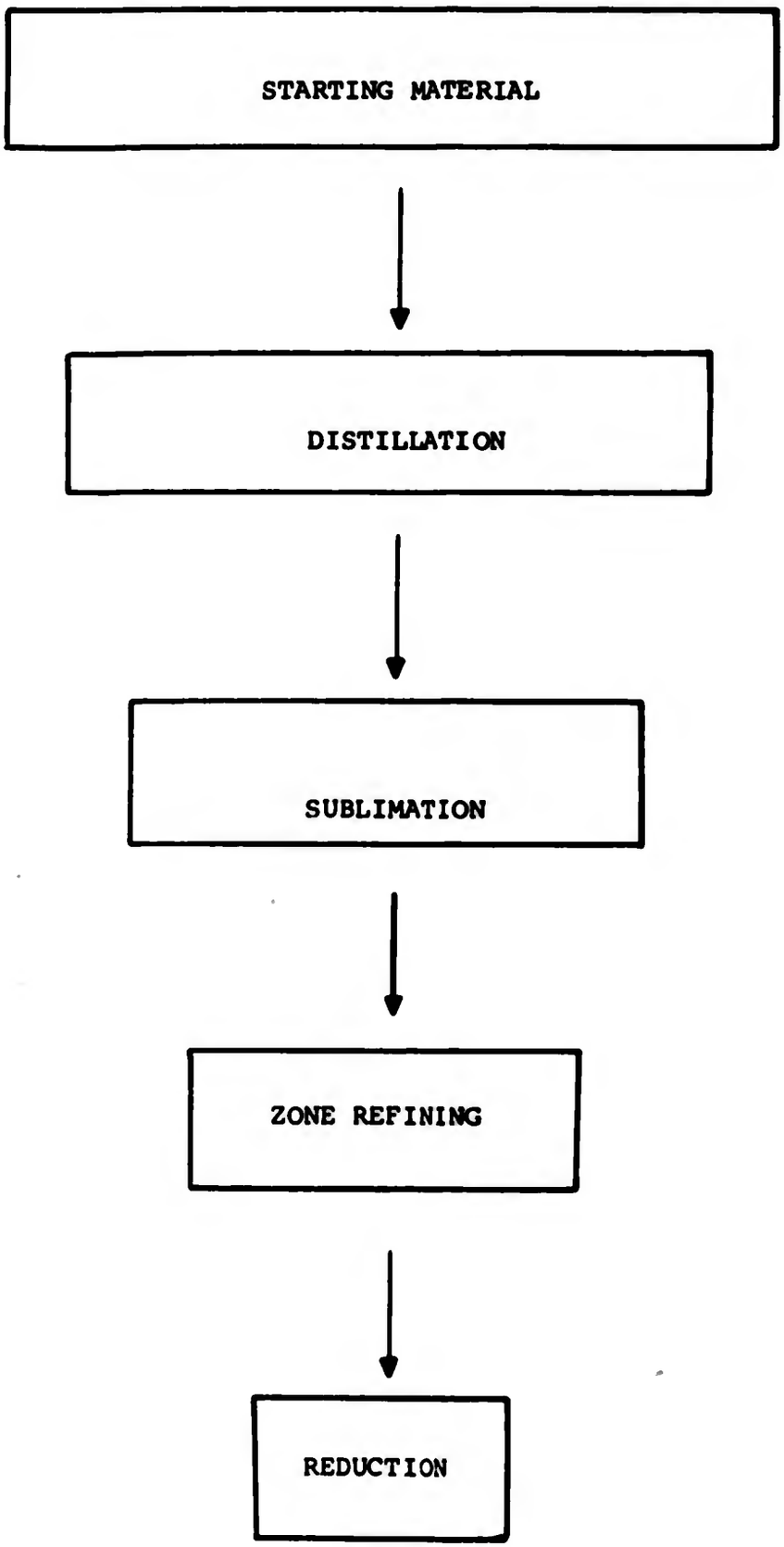


FIGURE 7

Production Scheme of High Purity $MoCl_5$



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