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Date Nov. 14, 1942

To: Dr. Ernest O. Lawrence

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OFFICE FOR EMERGENCY MANAGEMENT
OFFICE OF SCIENTIFIC RESEARCH AND DEVELOPMENT

1530 P STREET NW.

WASHINGTON, D. C.

VANNEVAR BUSH
Director

November 17, 1942.

Dr. E. O. Lawrence,
Radiation Laboratory,
University of California,
Berkeley, California.

Dear Dr. Lawrence:

I am enclosing herewith a Progress Report
by Harold C. Urey dated November 14, 1942, which
Dr. Urey gave you last Saturday and which you asked
have mailed to you.

Sincerely yours,



H. T. Wensel,
Technical Aide, Section S-1.

Enclosure

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ENGINEERING DIVISION

MONTHLY REPORT

File 1.4

on
FRACTIONATION OF LIQUID HYDROGEN

Prepared by J. R. Huffman

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November 11, 1942

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Summary

The design of a plant for the distillation of liquid hydrogen for the recovery of deuterium has reached the initial cost estimation stages. To produce approximately five tons of ninety mol percent HDO as final product from a nitrogen-hydrogen mixture will consume about 15000 H.P. Preliminary layouts indicate a plant covering an area of 40,000 square feet.

The distillation column will require about 200 hours of steady operation to reach maximum concentration. In case of a shut-down, it is estimated that 300 gallons of 90 mol percent HD and 1190 gallons of 21 mol percent HD can be burned to HDO. The water resulting from the latter batch will be shipped for concentration by electrolytic means.

Present plans call for the adaptation of standard carrier refrigeration equipment, producing 200 tons refrigeration at the 1000K level in auxiliary cycles. However, a large percentage of refrigeration is obtained by expansion of the hydrogen gas and proper heat exchange. The main liquifaction of hydrogen is produced by Joule Thomson expansion.

At present, the designers see no difficulties in the use of rock cork insulation, thus replacing the cumbersome vacuum jackets originally proposed.

Sufficient detail of design will be available in about three weeks to permit consideration of design and purchase of high priority equipment.

A program of investigation and a contract questionnaire are recommended for use of the liquid air equipment at Ohio State University.

Report

Object: The design of a full scale commercial plant to process approximately 5050 lbs./hour of hydrogen gas and produce about one ton a month of 90 to 100% HD by fractional distillation of liquid hydrogen; a design sufficiently adequate

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to permit negotiations for a contract with the du Pont Company for the construction and operation of the plant as soon as possible.

The basis of the calculations is the treatment of sufficient gas of one part nitrogen and three parts hydrogen to provide 5080 lbs./hour of hydrogen for distillation. Actually, about one and one half pounds of hydrogen will be liquified per pound distilled in order to provide the refrigeration required. On this initial basis a flow sheet and cost estimate will be presented to permit negotiations for purchase and construction. Simultaneously, refinements and changes of a constructional design type will be made.

This initial basis may be changed, due to the possibility of using "purified" gas (500,000 cu.ft./hr. of 94% H_2 and 5% N_2 available. The du Pont Company has recently been requested to estimate the relative times to reach plant operation on (1) 1,000,000 cu.ft./hr. & (2) 500,000 cu.ft./hr. of 1 N_2 /3 H_2 and (3) 500,000 cu.ft./hr. of purified gas. At present, there is a difference of opinion as to the advisability of using purified gas, compared to the 1 N_2 /3 H_2 mixture.

For the purposes of this report, the work will be covered in three phases: (I) The design of the distillation cycle including final Joule-Thomson expansion; (II) The design of the refrigeration and purification cycle down to final expansion, and (III) Research problems.

I DISTILLATION CYCLE

Personnel of the du Pont Company and of the Columbia group are now agreed on the section of the plant, both as to the philosophy and the basic design and engineering concepts. The design figures on this part of the plant have been checked and rechecked.

1. Flow Sheet

The gas is delivered from purification at 30 atmospheres pressure and $37^\circ K$ at the rate of 84.2 lbs./min. of essentially pure hydrogen. A trace of nitrogen remains in the gas which will amount to about 0.28/lbs./day introduced into the liquid hydrogen cycle. It is felt that the system can handle this amount of solids with no difficulty. This gas is expanded by the Joule-Thomson to 2 atmospheres and $23^\circ K$, giving about 40% liquid. It is condensed to all liquid at 2 atmospheres and $23^\circ K$ in a 4000 sq.ft. heat exchanger, using for cooling the heat of evaporation in a portion of the boiling liquid at the base of the distillation column. The incoming hydrogen is then expanded from 2 atmospheres and $23^\circ K$

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to 1 atmosphere and 20.4°K, giving 95% liquid and is fed into a 60 (actual) plate rectification column, 8 ft. in diameter and 70 ft. in overall height, operating at an external reflux ratio of 1.5.

To provide this reflux, 134.5 lbs./min. of gas from the top of the column is compressed to 2 atmospheres and 28° K using 114 H.P. (theoretical) and condensed in a 6000 sq.ft. heat exchanger cooled by the evaporation of another portion of the boiling liquid at the base of the column. It is then expanded to 1 atmosphere and 20.4°K, giving 128.8 lbs./min. of liquid reflux and 7.6 lbs./min. of gas.

The two heat exchangers, therefore, serve to provide the "boil-up" in the column and, together with an estimated heat leak of 27,800 P.e.u./hr., furnish an evaporation rate of 223.5 lbs./min., almost exactly the amount required by the distillation itself. The heat exchanger on the feed will be duplicated to provide means of warming up and removing any possible plugs of solid nitrogen.

Rock cork (gas sealed) is planned for insulation purposes and it is felt this will be adequate, thus eliminating the vacuum jackets originally considered.

The effect of the rare gases on these calculations is now being considered. Little change is anticipated.

The effect of other-para conversion with its attendant heat effect still remains to be checked.

Attention is called to the fact that expansion turbines have been dropped in favor of a Joule-Thomson expansion valve, constituting a change from previous plans and correlating the designer's opinions with those of the University cryogenic people.

2. Distillation Calculations

For 1,000,000 cu.ft./hr. of hydrogen gas (0°G and 760 mm.Hg.), several calculations of the number of plates and reflux ratio for various percent recovery and concentration of HD at the heavy end have been made. Table I presents the important results for 90 mol % HD in the heavy product. Further details appear in Appendix I.

TABLE IDistillation of HD
(90% HD in heavy product)

<u>% Recovery</u>	<u>Reflex Ratio</u>	<u>Plates (theoretical)</u>	<u>Mole HD/month in the heavy product</u>
90	1.2	40	516
70	0.75	30	397
50	0.20	27.5	287
42.6	(No enriching section)	24	220

Since the column costs will amount to only 5% or less of the plant, the use of a shorter column or of no enriching section at a sacrifice of recovery and concentration is not warranted. At present design calculations are based on a 60 (actual) plate column, 8 feet in diameter, 70 feet high and external reflux ratio of 1.5 to give 90% recovery of the deuterium and about 1800 lbs./month of heavy product containing 90 mol % HD.

3. Attainment of Chemical Equilibrium

The working hold-up for the column proper on the basis of 8' diameter, 3/4" liquid level on plates and allowing 15% of plate volume for bubble caps, amounts to 2.67 cu.ft. (20 gallons) of liquid per plate, corresponding to 8.1 lb.mole at an average molecular weight of 2.4 pounds and a specific gravity of 0.0875. This amount of material has to be brought up to the working concentration of HD plate by plate in order for the column to deliver the desired product. In addition, there is a recirculating hold-up beyond the working hold-up of the column, which will be at 90 mol % HD.

The working hold-up in the column of 60 actual plates will amount to 1190 gallons containing 76.5 moles of HD and will require approximately 110 hours to reach equilibrium. At present, about 300 gallons of 90 mol % HD are in the recirculating system at the heavy end of the column. This will require an additional 100 hours to reach its concentration. Consequently, within 10% the total estimated time to reach equilibrium will be 200 hours.

For further details see Appendix II.

The question arises: Supposing equilibrium has been reached and it becomes necessary to shut down the column, should the contents of the still be compressed into a gasometer to be fed back at the resumption of operation, or should the contents be burned to H₂O for concentration in electrolytic cells?

This question can be answered purely on cost and time bases. The gasometer and compressors will cost about \$150,000 as compared to nominal costs for burners. The time for establishing equilibrium is relatively short. The average composition of the 1190 gallons of working hold-up is about 21 (mol) %, and of the 300 gallons of recirculating hold-up 90%.

At the present time, it is expected that the liquid will be blown to receivers and burned to 90% H₂O to be combined with the normal product and the column hold-up will be burned and stored separately to give 21% H₂O for electrolytic concentration elsewhere.

II REFRIGERATION AND PURIFICATION

Agreement between the two groups has been reached on the general philosophy and broad details of design for this portion of the plant. The actual flow sheet has not quite reached the stage of design that exists for the distillation cycle. A tentative flow sheet only is presented at this time.

1. Flow sheet

Incoming gas at 273°K and 30 atmospheres is cooled by heat exchange and an additional 62 tons of refrigeration (413,000 P.c.u./hr.) to 200° K; further heat exchange and another additional 40 tons of refrigeration (267,000 P.c.u./hr.) to 70° K, where liquid nitrogen will fall out; and further cooling in another heat exchanger to about 65° K. This train is followed by a heat exchanger which is duplicated where it is expected the solid nitrogen will freeze out, as the temperature reaches a value of 49° K. The free volume on the inlet side of this exchanger is spaced to hold the maximum amount of frozen nitrogen (appearing at a rate of 6 lbs./min.) before plugging, which should occur in eight hours. At this time, the alternate heat exchanger is cut into the system while the first one is warmed up. In the process at this point, an expansion engine of proven performance at 55° K will reduce the temperature to 37° K, 84.2 lbs./min. of the effluent from which will go to the Joule-Thomson expansion, and 10 lbs./min. to recirculation in order to provide sufficient refrigeration on the previously mentioned heat exchanger.

This cycle should be sufficient for the job. However, due to warming up periods for removing solid nitrogen and as an insurance of sufficient refrigeration to deliver gas to Joule-Thomson expansion always at a fixed temperature of 37° K, a thermal "flywheel" is floated on the line. This consists of two heat exchangers of about 1250 sq. ft. surface each with some recycle refrigeration followed by a three stage compressor (850 H.P.) with intercoolers operating on 90° K to 135° K temperature and 2 to 30 atmosphere pressure ranges. The cooling for the interstage coolers of this compressor will be furnished by a Carrier refrigerating cycle operating on refrigerant F-13, requiring about 170 tons refrigeration (1,132,000 P.c.u./hr.)

This "flywheel" will stabilize the system, handle any small additional load, and serve to start up the system. It will probably be the key to the successful operation of the plant.

For the delivery of the hydrogen and nitrogen back to the ammonia plant, it will be necessary to provide five or six 2700 H. P. compressors with accessories and two storage gas spheres with mixing facilities. These items will be the major costs in the plant, each compressor running to about \$125,000.

Facilities for burning the HD product to water, for storage and shipment and safety provisions are under consideration but no actual design has been completed.

General

The construction "bottlenecks" in the present contemplated design appear to be the large compressors for the return of the gas at 30 atmospheres, the Carrier refrigeration unit (200 tons of refrigeration total) used in three places, and the twenty heat exchangers (1000-3500 sq.ft.each). In two or three weeks, specifications on this equipment will permit consideration of contract questionnaires for the purchase of the compressors by du Pont and for the design of refrigeration units by Carrier.

The present power requirements total about 15,000 H.P. of which 10,000 H.P. are required to return the gas at 30 atmospheres pressure and 5000 H. P. for refrigeration. This amounts to about 12,500 kw./ton 90 % HD.

In order to obtain the necessary refrigeration, about one and one half pounds of hydrogen per pound reaching the distillation column will have to be used. This means that thirty percent of the gas will not be stripped of deuterium.

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Preliminary plant layouts are continually being constructed. At present, the total area will cover a plot 200' x 200' of which only about one fourth is necessary for the liquid hydrogen section of the plant, or that part below 49° K.

Present Status

1. Rechecking of the refrigeration section is now under way.
2. The Carrier people will present a definite answer within two weeks on the adaptability of their standard refrigeration equipment to reach the 100° K level.
3. Specifications and cost estimations on the various pieces of equipment were started November 9th. While it is not safe to predict, a fairly complete preliminary estimate should be available within one month and a half.
4. Investigations of the effect of the rare gases and of the ortho-para conversion are planned.
5. Time and man-power schedules on the design part of the problem will be presented shortly.
6. Consideration of the proper quantity and the proper source of the gases available at du Font has been requested and should be available shortly.

III RESEARCH

Two phases of the problem have been assigned to other groups. First, the determination of the thermal conductivity and heat transfer coefficients for boiling hydrogen have been undertaken by Dr. Brickwedde at the Bureau of Standards. No data have been reported to date. Second, a contract questionnaire is in process for the establishment of a liquefaction system under Professor H. L. Johnston at Ohio State University.

The importance of some research at Ohio State is becoming more apparent every week. As at present outlined, the program consists of the following, to which additional items are constantly being added.

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1. The operation of the unit on recycle pure hydrogen gas to which a known percentage of nitrogen is fed to determine:
 - a) the time to plug the Giauque purifier
 - b) the percent removal of nitrogen, and
 - c) the mechanical properties of solid nitrogen.
2. Similar data to 1(a) above on replica units of a design to be used in the large plant, should this be possible.
3. The determination of the thermal conductivity of test sections of insulation to be used in the large plant.
4. The determination of properties of materials of construction such as hardness, thermal conductivity, etc., of steels, bakelite, etc.
5. Problems of liquid level and temperature control
6. Ortho-para hydrogen conversion
7. Investigation of the importance of any other thermal effects, such as transitions in the solid state, etc.

10 JRH/ms

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APPENDIX I



DISTILLATION

HD - H₂

Data and Procedure:

Vapor Pressure: H₂ $\log P_{\text{mm}} = 4.80204 + 0.0167338 T - \frac{46.1045}{T}$
 HD $\log P_{\text{mm}} = 5.04964 + 0.01479 T - \frac{55.2495}{T}$

Relative Volatility (calculated)
 at 20.40°K = 1.740
 at 22°K = 1.613

So average α is 1.68 for column operating at one atmosphere.

However y and x values were calculated from vapor pressure data rather than using an average alpha for the stripping section. Plates were stepped off on McCabe-Thiele diagram. For the enriching section, alpha was taken as 1.74 and the following formula was used:

$$\Delta n = \frac{2.303}{A} \log \frac{A X_0 + \beta}{A X_f + \beta}$$

when $A = \frac{1}{\alpha} - 1 + \frac{1}{\alpha R}$

$$\beta = \frac{-X_0}{R}$$

X₀ and X_f = mol fractions of HD

The basis of 1,000,000 cu. ft./hr. of hydrogen (0°K and 760 mm. Hg) to column was used.

TABLE II

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% Rec.	XHD Waste	R_{10}	Theoretical Plates			Waste mols./hr.	Waste mola./mon.	Transport HD/mon.	
			R.	Above Feed	Below Feed				Total
90	0.90	1.11				∞	0.780	561	505
			2.0	7.8	21.5	29.5			
			1.5	10.4	22.1	32.5			
			1.3	13.8	22.7	36.5			
			1.2	17.9	24.6	42.5			
		∞			24.5				
90	0.75	1.11				∞	0.956	689	516
			2.0	7.8	21.3	29.1			
			1.5	10.6	22.3	32.9			
			1.3	13.1	23.3	36.4			
			1.2	15.7	25.1	40.8			
		∞			20.9				
70	0.90	0.64				∞	0.612	441	397
			2.0	2.8	20.8	23.6			
			1.5	3.1	21.0	24.1			
			1.3	3.3	21.3	24.6			
			0.9	3.8	23.3	27.1			
		0.75	4.7	25.3	30.0				
		∞			22.2				
70	0.75	0.64				∞	0.744	535	401
			1.5	3.1	19	22.1			
			1.2	3.3	20	23.3			
			0.9	3.8	21	24.8			
			0.75	4.8	22	26.8			
		∞			18.9				
50	0.90	0.152				∞	0.443	319	287
			0.3	1.1	23	24.1			
			0.2	1.3	26.3	27.6			
		∞			21.4				
50	0.75	.172				∞	0.532	383	287
			0.3	1.1	22.2	23.3			
			0.2	1.3	24.4	25.7			
		∞			17.9				
50	0.25	.172				∞	1.592	1147	287
			0.3	1.1	17	18.1			
			0.2	1.3	20.2	21.5			
		∞			15.0				
50	0.10	.172				∞	3.98	2870	287
			0.3	1.1	10.2	11.3			
			0.2	1.3	12.3	13.6			
		∞			12.7				
42.8	0.90	No enriching section					0.350	244.5	220.0

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APPENDIX II
 ATTAINMENT OF CHEMICAL EQUILIBRIUM

Formula

$$N_t \log \frac{N_t}{N_b} + (1-N_t) \log \frac{1-N_t}{1-N_b} = \frac{WN_t(\alpha-1)k \log \alpha}{H} t$$

- N_t = mol fraction HD at top
- N_b = mol fraction HD at base
- α = 1.70
- H = 6.1 mols per plate
- k = No. of theoretical plates, 24
- W = Rate of feed, 2785 mols/hour
- t = 47.5 hours

For 60 actual plates, H becomes 6.1 x 60 rather than 6.1 x 30.6 as calculated above.

For holdup beyond the actual working holdup the following additional term must be added to the left side of the equation.

$$\left(\frac{h k}{H} \log \alpha \right) (N_b - N_t) \qquad h = \text{holdup, 55.5 mols.}$$

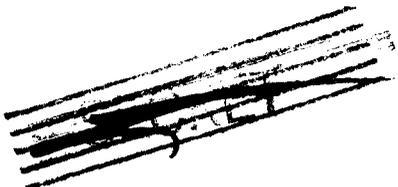


TABLE III

Concentrations on Plates at Total Reflux

Plate No.	Mol. frac. H ₂	Recovery	Average Conc. X _{H₂}	Percent Total Holdup
Still	0.10			17.9
1	0.154			34.8
2	0.228			50.1
3	0.328			63.5
4	0.448			74.5
5	0.575			83.0
6	0.693			89.1
7	0.792			93.1
8	0.858			96.0
9	0.918		0.5094	97.6
10	0.9512			98.6
11	0.9717			99.2
12	0.9840			99.5
13	0.9912			99.6
14	0.99492			99.9
15	0.99706			
16	0.99831			
17	0.999040			
18	0.999450			
19	0.999690			
20	0.999820	(50% or (No enricher)	0.76097	
21	0.999895			
22	0.999939	70%	0.78149	
23	0.999965			
24	0.999998	90%	0.79921	

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The time to reach chemical equilibrium has been calculated first at total reflux when twenty-four theoretical plates are required for almost any chosen end conditions. About 475 gallons of total liquid, corresponding to 30.6 lb. mols of HD constitute the working hold up. 99 mol % of this amount of HD is on the first ten plates above the still (See Table III).

On the basis of 90% recovery the transport into the column is 0.716 mols HD/hour, so that 42.7 hours (30.6/.716) are necessary to reach chemical equilibrium, assuming no hold up beyond the bottom plate. Using the formula developed by Urey and Huffman⁽¹⁾, 47.5 hours were obtained.

If in addition 300 gallons of 90 mol % HD are in the recirculating system at the heavy end of the column an additional 77 to 100 hours are required to reach maximum concentration (90 mol % HD).

⁽¹⁾ See Urey, The Physical Review (London) VI, 57 (1938)

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File 1.8

ENGINEERING DIVISION
MONTHLY REPORT
ON
A NEW METHOD FOR THE
CONCENTRATION OF DEUTERIUM

CLASSIFICATION CONTROLLED
BY AUTHORITY OF THE ARMY
DOCUMENT REVIEW SC 1111

Prepared by Jerome Spevack

ON _____ DATE _____ SIGNATURE _____

November 11, 1942

A new method for the concentration of isotopes is based on the principle that the equilibrium constant for an exchange reaction is a function of the temperature. By suitable design, a countercurrent system of two or more towers operating at different temperatures, can be set up, whereby, concentration of a desired constituent can be effected in the region between the different temperatures.

Application of this method to the concentration of deuterium permits the choice of a wide variety of hydrogen exchange reactions. Use of a system involving the exchange of one component with water provides a means for the concentration of deuterium using water as the raw material.

This method possesses a decided advantage over others, inasmuch as the energy requirements are small and the raw material is readily available. The unlimited supply of water, which is the only necessary source of deuterium, enables a volume of production heretofore economically impractical. Effectively, an unlimited and unrestricted production of deuterium is possible anywhere.

At the present time, two systems have been selected to be the most practical. The simpler of the two makes use of the deuterium exchange between hydrogen sulfide and water. Here the reaction mechanism is ionic and takes place rapidly in the liquid without a catalyst. Calculations indicate that recovery and concentration of 20% of the total deuterium in the feed water can be achieved with relative ease. The other system is more complex and makes use of a combination of three exchange reactions. These are the ammonia-hydrogen exchange, the water-hydrogen exchange and the water-ammonia exchange. Here the two exchange reactions involving hydrogen require catalysts. However, recovery and concentration of at least 40% of the total deuterium in the feed water can be effected by this system.

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6511971

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MALCOLM THEISEN, EAS
Name (ADD) - Organization

Date 18 Aug 95

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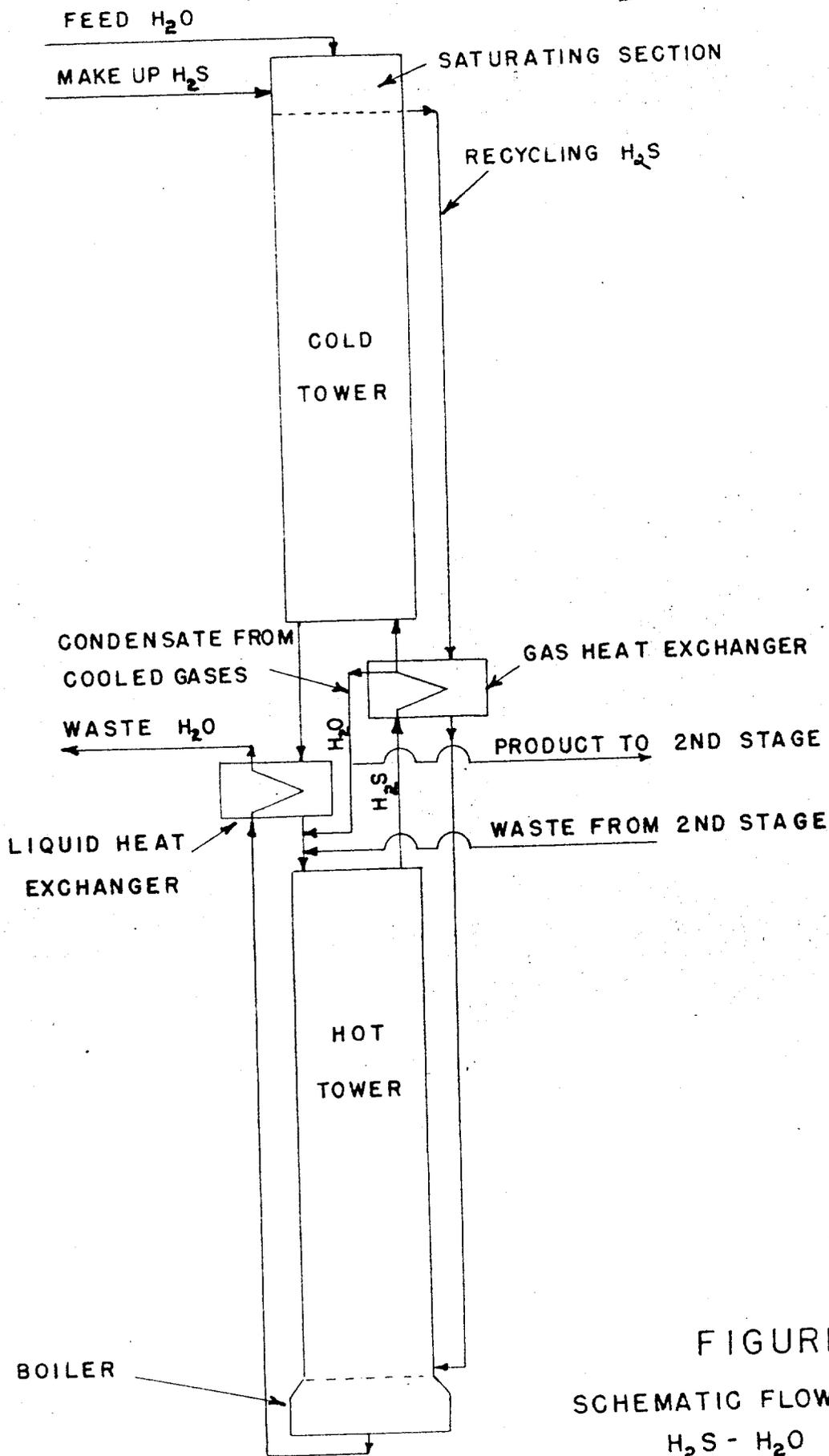


FIGURE I
 SCHEMATIC FLOW SHEET
 $H_2S - H_2O$
 EXCHANGE SYSTEM

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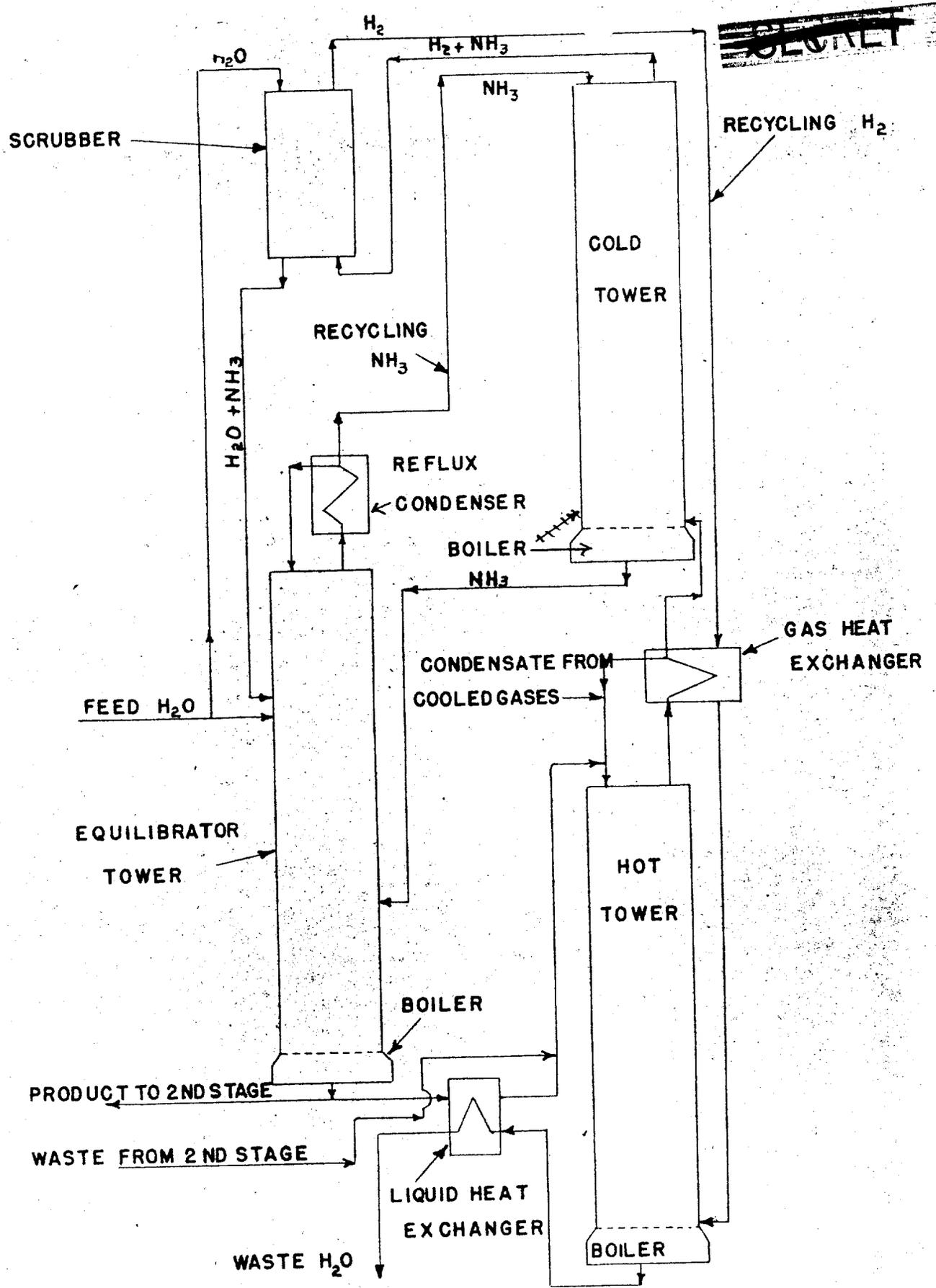


FIGURE 2

SCHEMATIC FLOW SHEET

NH₃ - H₂ - H₂O
EXCHANGE SYSTEM

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Jerome Spevack

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Monthly Report
11/11/42

Discussion

A comparison of the two systems indicates that the one using hydrogen sulfide requires only two towers filled with standard packing or constructed of bubble plates and built to withstand pressure and corrosion by the hydrogen sulfide solution. The ammonia system requires three towers, two of them of special construction to permit co-current gas flow over catalyst beds and an overall countercurrent liquid-gas flow through the tower. One of these towers contains catalyst for the low temperature ammonia-hydrogen exchange, the other tower contains catalyst for the high temperature water-hydrogen exchange. The third tower is for the ammonia-water exchange and requires only packing or bubble plates. The schematic diagrams in figures (1) and (2) show the operational and functional details of the proposed plants using the hydrogen sulfide-water exchange reaction and the ammonia-hydrogen-water exchange reactions respectively.

(a) Hydrogen Sulfide-Water Exchange System

The construction of the hydrogen sulfide system consists of two packed towers, one operating cold at about 35°C, the other operating hot at about 191°C. The total pressure in the system is limited to twenty-five atmospheres by the vapor pressure of hydrogen sulfide at the cold tower temperature. The temperature of the hot tower is set by the desired partial pressure of water in it. The top of the cold tower serves to saturate the feed water with hydrogen sulfide. The bottom of the hot tower contains a boiler and acts to vaporize water to humidify the incoming hydrogen sulfide. The hydrogen sulfide flows through the hot tower and cold tower, recycling continuously. The feed water enters the top of the cold tower and flows countercurrent to the hydrogen sulfide through both towers and leaves as waste from the boiler. The product is removed from the condensate that is formed from the gases that leave the hot tower. The enriched product is sent to the second stage of the cascade system for further concentration. The waste from the second stage is returned to the first stage for further stripping.

Enrichment in the first stage is determined principally by the liquid gas ratio and the allowable height of the towers. Calculations on the system operating under an optimum hydrogen sulfide-water ratio of 1.8,

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and a step-wise graphical determination of the number of theoretical plates in both towers, indicate the total height of tower required. A height equivalent to forty-two theoretical plates is necessary for a two-fold enrichment, while sixty-nine theoretical plates are necessary for a five fold, and one hundred and five are required for a twenty fold increase in concentration. It is obvious that it is desirable to construct the stage to the optimum height determined from economic considerations. The table on page (11) of the appendix shows the effect of the number of theoretical plates on the concentration of the product.

At present a report is being completed that will present a detailed analysis of the system, the methods of calculation, the theoretical considerations and the physical properties of the components involved.

Preliminary design has been made on the construction of the semi-works unit. This will test the system under operation and obtain data for the design of a plant.

(b) Ammonia-Hydrogen-Water Exchange System

The construction of the ammonia-hydrogen-water system consists of three towers. One operates cold at about 50°C and contains the catalyst for the ammonia-hydrogen exchange. Another operates hot at about 215°C and contains the catalyst for the water-hydrogen exchange. The temperature of this tower is set by the desired partial pressure of the water in it. The third is a packed tower for the ammonia-water exchange and operates essentially as a combination distillation tower and equilibrater. No catalyst is required for this exchange.

The bottom of the hot tower contains a boiler that serves to humidify the incoming hydrogen. Above the cold tower is a scrubber to remove the ammonia that is carried out by the hydrogen. At the bottom of the cold tower is a boiler that serves to saturate the incoming hydrogen with ammonia. The hydrogen flows through the hot tower and cold tower, recycling continuously. Liquid ammonia flows countercurrent to the hydrogen in the cold tower. The actual overall catalytic exchange takes place with consecutive co-current hydrogen-ammonia gas streams that pass countercurrent to the liquid ammonia stream. The ammonia flows from the cold tower to the water

equilibrator tower where it is vaporized, passed counter-current to the feed water, stripped of its deuterium enrichment and fractionated from the water. The ammonia returns to the cold tower and recycles continuously. The feed water entering the equilibrator tower becomes enriched, and is separated from the ammonia. It flows through the hot tower countercurrent to the hydrogen and leaves the boiler as waste.

The product to be sent to the second stage is drawn either from the ammonia stream leaving the cold tower or from the water stream leaving the equilibrator. Waste from the second stage is returned to the system at a point beyond where it was drawn.

Essentially, this system is analogous to either a ammonia-hydrogen two tower system or a water-hydrogen two tower system, as far as extraction efficiency goes. In this case, however, the second liquid has been introduced in order to provide a means of producing a uniform pressure in the entire plant.

One of the advantages of this system over the hydrogen sulfide system is that it does not suffer from corrosion and can be constructed of ordinary steel.

At present, work is being done to determine the effective variables on the operation of the system.

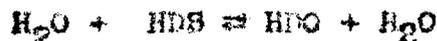
APPENDIX

Equilibrium Constants

The equilibrium constants for the gas phase exchange reaction between hydrogen sulfide and water have been calculated by Dr. Stockmayer from spectroscopic data and follow the equation

$\log K_g = 93.7/T - 0.0105$. Applying a correction for the effect of the relative volatility of HDO to H₂O, $\log P_{HDO}/P_{H_2O} = 0.0636 - 28.3/T$, the relationship $\log K_1 = 122.0/T - 0.741$ is obtained for the liquid-gas reaction.

Then for the reaction



Where
$$K = \frac{(HDO)(H_2S)}{(H_2O)(H_2S)}$$

<u>Temp. °C</u>	<u>K_g</u>	<u>K₁</u>
0	2.15	2.36
25	2.02	2.16
100	1.75	1.79
200	1.54	1.53

The equilibrium constants for the exchange reaction between ammonia-hydrogen, water-hydrogen, and ammonia-water have been calculated from spectroscopic data for the gaseous state by Drs. Kimball and Stockmayer and are as follows: For the gas phase, ammonia-hydrogen exchange $\log K_g = 0.0039 + 227.2/T$. Applying a correction for the relative volatility of NH₂D to NH₃, $\log P_{NH_3}/P_{NH_2D} = 15.42/T - 0.04668$, determined from data by I. Kirshenbaum, there is obtained the relationship for the liquid ammonia-hydrogen exchange $\log K_1 = 242.6/T - 0.0428$.

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Jerome Spevack

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Monthly Report
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The reaction



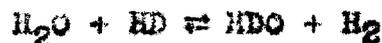
where
$$K = \frac{(\text{NH}_2\text{D}) (\text{H}_2)}{(\text{NH}_3) (\text{HD})}$$

<u>Temp. °C</u>	<u>K_K</u>	<u>c_g</u>	<u>K₁</u>	<u>c₁</u>
0	6.85	4.57	7.01	4.67
25	5.83	3.89	5.90	3.93
100	4.10	2.73	4.05	2.70

where (c) is the separation factor or distribution of deuterium between the ammonia and hydrogen phase and is equal to two-thirds of K.

For the gas phase water-hydrogen exchange $\log K_g = 205.4/T - 0.1307$. The relative volatility correction for HDO and H₂O is $\log P_{\text{HDO}}/P_{\text{H}_2\text{O}} = 0.0636 - 29.3T$, and the equilibrium constant between liquid water and hydrogen is expressed as $\log K_1 = 333.7/T - 0.1943$.

The reaction



where
$$K = \frac{(\text{HDO}) (\text{H}_2)}{(\text{H}_2\text{O}) (\text{HD})}$$

<u>Temp. °C</u>	<u>K_g</u>	<u>K₁</u>
25	3.62	3.89
100	2.63	2.71
200	2.01	2.00

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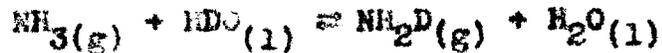
Jerome Spevack

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Monthly Report
11/11/42

The liquid water-ammonia gas exchange is determined from the relationship of the ammonia and water-hydrogen exchange reaction constants.

For the reaction



$$K_{g1} = K_{g(\text{NH}_3 - \text{H}_2)} / K_{1(\text{H}_2\text{O} - \text{H}_2)}$$

<u>Temp. °C</u>	<u>K_{g1}</u>	<u>α_{g1}</u>
25	1.50	1.000
100	1.51	1.007

Where (α_{g1}) is the separation factor or the distribution of deuterium between the ammonia and the water.

Considerations of Extraction Efficiency

An evaluation of the maximum deuterium extraction efficiency of the hydrogen sulfide-water system can be made by assuming equilibrium conditions at the ends of the two towers. If K₀ is the equilibrium constant for the exchange at the cold tower temperature and X_f is the concentration of deuterium in the feed water, then the concentration in the hydrogen sulfide leaving the cold tower is X_f/K₀. Similarly, the concentration of the water leaving the hot tower is X_fK_H / K₀, where K_H is the equilibrium constant for the reaction at the hot tower temperature. The fraction extracted is determined by the difference between the concentration of the feed and the waste liquor and is equal to (1 - K_H/K₀).

An evaluation of the maximum deuterium extraction efficiency of the ammonia-hydrogen water system can be made by assuming equilibrium conditions at the ends of the three towers. By the same treatment as was used for the hydrogen sulfide system, the concentration of the ammonia leaving the equilibrater tower is X_fα_w, where α_w is the separation factor for the ammonia-water exchange

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at that tower temperature. The concentration of the hydrogen leaving the cold tower is $X_1 a_w / a_c$ and similarly, the concentration of the water leaving the hot tower is $X_2 a_w K_H / a_c$. The fraction extracted is then $(1 - a_w K_H / a_c)$. Since the separation factor for the water-ammonia exchange is almost equal to one, the fraction extracted becomes $(1 - a_H / a_c)$.

It is important to note that in determining the ultimate extraction efficiency of either system, it is necessary to use the equilibrium constants of the liquid-gas reactions and not the gas phase equilibrium constants, although these may be used on calculations within the tower.

The First Stage of a Cascade System For the Hydrogen Sulfide-Water Exchange.

Calculations made on conditions within the tower are based on the assumption that the effective fractionation factor for the distribution of deuterium between the liquid and the gas phases is representative of equilibrium conditions in the tower and, therefore, determines the equilibrium curve for the tower operating under any temperature and pressure. The effective fractionation factor varies not only with the temperature but also with the solubility of the hydrogen sulfide in the liquid and the partial pressure of the water vapor in the gas.

$$\beta = \frac{(1 + H) (K_g + S)}{(1 + S) (1 + HK_g)p}$$

β = effective fractionation factor

H = molar ratio of water to hydrogen sulfide in the gas

S = molar ratio of hydrogen sulfide to water in the liquid

K_g = equilibrium constant for the gas phase exchange reaction

p = ratio of vapor pressures of H₂O to H₂S

Weight balances around the towers between the and the saturator sections determine the distribution of deuterium in both phases at the ends of each tower. Variation of these concentrations for varying liquid enables the selection of optimum flow conditions.

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Jerome Spevack

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Monthly Report
11/11/42

A plot of the effective fractionation factor as the equilibrium line and a plot of the operating line obtained from the concentrations at the ends of the towers enable the performance of a stepwise graphical determination of the number of theoretical plates required to produce a given enrichment of product. The result of such a determination are shown in the following table:

Cold tower temperature = 35°C
Hot tower temperature = 121°C
Total pressure = 25.5 atmos. (vp. of H₂S @ 35°C)
Partial pressure of H₂O in Hot tower = 12.75 atmos.
Mols circulating H₂S per mol water feed = 1.8
Concentration of deuterium in feed water = 1/7000 = 0.0143 mol % D₂O

Product Mol % D ₂ O	Concentration increase	Number of Theoretical Plates		
		Hot Tower	Cold Tower	Total
.0286 %	2 fold	27.0	15.2	42.2
.0428	3	35.7	19.3	55.0
.0572	4	41.6	21.7	63.3
.0715	5	45.9	23.4	69.3
.0857	6	49.2	24.7	73.9
.1000	7	52.1	25.8	77.9
.1143	8	54.5	26.8	81.3
.1286	9	56.7	27.5	84.2
.1429	10	58.6	28.2	86.8
.2144	15	65.9	31.5	97.4
.2860	20	71.0	34.4	105.4

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Attached to this memorandum is a report by R. H. Crist on the uranyl nitrate water-ether exchange system, reviewing briefly the work done at Yale, at the University of Pennsylvania, and at the DuPont Company. The work at the DuPont Company, looking to the large scale apparatus required for this work, has turned out very well indeed. A complete report will be prepared later, but it appears that sieve plate counter-current columns could be used to construct a plant with perhaps an equilibrium time of about 10 seconds. This would mean that the plant would require three or four months to come to equilibrium if it were producing material of double concentration. The plant would also be very inexpensive and could be constructed easily without a great drain on strategic materials.

Kilpatrick's work at the University of Pennsylvania has given us a great deal of information with respect to the analytical procedures for uranyl salt solutions, and also the settling times of mixtures of ether and water solutions of uranyl nitrate.

The results secured at Yale University on the fundamental process are very difficult to understand. Neither I nor my colleagues see any way by which the analytical results can be in error. You will note from the table in Dr. Crist's report that all analyses either fall within a few hundredths of zero, with a mean deviation of the measurements of a few hundredths or else they fall in the neighborhood of 0.15, except one which is -0.23. These analyses are far outside the mean deviation. They do not, however, indicate as large an effect as previously reported.

Since our previous reports, Dr. T. Ivan Taylor, working at the Bureau of Standards, has presented a report - A314 - covering work which he has done on the exchange between uranyl solutions and zeolites. He finds in this case that his samples deviate from standard uranium samples by 0.8% in two experiments, and 0.4% in another, these analyses having been made by the counting method. That method has not proved very reliable, but it seems probable that such large differences as this are outside the errors of this method. Samples are now being prepared at Yale University, to be analyzed at Columbia University covering these experiments.

It is my conclusion at the present time that the effect that we thought we had observed in the difference of distribution constants of the isotopic uranyl nitrates, cannot properly be ascribed to any such difference. The experiments definitely show that this interpretation of the differences

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observed cannot be correct. It may be that the differences are due to analytical errors or some other unknown effect, such as a photochemical effect. Experiments are in progress to determine whether this is the case.

In view of this situation I have asked Mr. Chilton of the DuPont Company not to spend money for strategic materials on the pilot plant work until the laboratory experiments are cleared up. I am asking the Yale group to make conversions of uranyl samples for us. This technique they have now perfected to a high degree and can convert samples quickly. We are asking Professor Kilpatrick to finish up his work on the analytical and settling problems. Recommendations in regard to definite disposition of the Pennsylvania and Yale University work will be made at the next meeting of the Executive Committee.

Harold C. Urey

November 13, 1942

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MONTHLY REPORT
ON
URANYL NITRATE, WATER, ETHER EXCHANGE SYSTEM

by
R. H. Crist

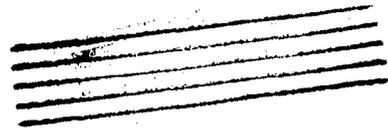
The significant developments have been with regard to (a) the methods of mixing and separating the two phases in such a manner that contact time and hold up are suitable and (b) the determination of the simple process separation factor.

In the work at Yale University on the simple process factor ten experiments have been completed and are reported in Table I.

TABLE I
Summary of Extraction Experiments

<u>Exp.</u>	<u>t°C</u>	<u>Analysis Pair</u>	<u>$\Delta \%$</u>	<u>a. d.</u>	<u>Remarks</u>
A	18-23	Et(40) - Orig	0.18	.02	Repetition of Columbia Experiment
B	0.2	Et(40) - Orig	-0.01	.05	0°C
		W(57-89) - W(1)	.02	.05	
		W(57-89) - W(1)	.00	.05	
C	25	Et(20) - CoDeEe	.15	.04	Solution 1/3 saturated
		Et(20) - DeEoFe	.17	.05	
D	18-23	W(2) - DeEoFe	-.01	.02	Solution contained nitric acid
		W(24) - W(2)	.00	.05	
		W(57-8-9) - DeEoFe	.01	.07	
		W(57-8-9) - DeEoFe	.01	.02	
		Et(40) - CoDeEe	.00	.05	
		Et(40) - DeEoFe	-.05	.05	
E	25	Et(40) - CoDeEe	.02	.07	
F	18-23	W(48) - W(2)	-.25	.02	Repetition Columbia Experiment
		Et(100) - DeEoFe	.14	.02	
G	18-23	W(24) - W(2)	-.01	.02	Solution alkaline with Na OH
		W(57-8-9-0) - W(2)	-.02	.03	
		Et(40) - DeEoFeGo	-.02	.04	
H	18-23	Et(40) - Orig	-.02	.02	30 Minute agitation
		W(57-40) - W ₂	.00	.01	
I	25	Et(40) - Orig	-.01	.04	Temp. 35°C
		W(57) - W ₂	.00	.01	
K	25-28	Et(50) - Orig	.01	.01	Further attempt to duplicate Columbia
		W(47) - W ₂	-.01	.04	

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of the
Unless otherwise indicated the temperature/experiment is 18 - 25°C and the time of shaking is 3 - 5 minutes. The symbols E and W refer to the ether and water layers respectively. The numbers in the parentheses refer to the number of single extractions or cycles involved. The solutions are saturated with respect to $UO_2(NO_3)_2$ except as indicated. Reduction in amount of solute during an experiment, of the order of 10,000 to 1.

The second column gives the pair of samples used for the analysis. In all cases the substance in an ether layer is compared to the original solid and substance from a water layer to that from another water layer. The difference in the percent content of rare substance for the analyses pair is shown in column three.

Examination of the data shows significant enrichment in experiments A, G and F. There are a number of obvious inconsistencies in the results. In (F) both positive and negative values were obtained; (I) at 30°C does not bear out the temperature effect indicated in (A) and (B); (K), designed to be a careful duplication of the original Columbia experiment shows no enrichment. The small effect for the acid (B) and alkaline (G) solutions might reasonably be a pH effect. On the other hand, the 30 minute shaking experiment (K) should have shown effects at least as great as experiment (A).

These evident inconsistencies might be explained on the basis of some unknown impurity or an accidental analytical error. Both possibilities are being thoroughly investigated.

Harned's group have perfected the method of conversion of uranium nitrate to Hex sufficiently to give up to 80% conversion on 150 mg samples.

The engineering group under Chilton reports favorably on the operation of the sieve plate column. Using a dummy solution, a 6 1/2 second holdup was obtained with a 2 1/2 inch plate spacing and 4 seconds for a 1 1/2 inch spacing. The column, 8.75 inches diameter and having a 10 1/2% free area of 0.11 inch holes, floods at a rate of 2 to 3 gal/min of water and 4 gal/min of isobutyl alcohol.

A horizontal separator made up of a pair of plates separated by 1/8 inch was fed through holes distributed along one edge at low feed rates corresponding to contact times of 6 seconds. The product streams contained 20% of the wrong phase and contamination increased with increasing feed rates.

The Columbia and du Pont engineering groups look with favor on the sieve plate column as a plant possibility. This is because of the

simplicity of construction and operation and the availability of materials. The Potbielniak contactor has none of these advantages and in addition would require redesign and development.

Cohen and Kaplan have made the necessary theoretical calculations for a continuous cascade system.

Kilpatrick has found that the highest settling rates of ether water emulsions are obtained with very pure substances. The rate is increased with increase of temperature, is little affected by small amounts of Na_2SO_4 and NaOH , and is increased to a greater or less degree by wetting agents, oils, organic acids and bases and some stopcock greases. The experiments show that all plant equipment must be scrupulously clean.

The Pennsylvania group also have perfected for general analytical purposes the refractive index, density and colorimetric methods for the determination of uranium in water and other solutions.

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Report on Photochemical Studies

by

S. I. Weissman
David Lipkin

University of California
November 10, 1942

In previous reports possible methods for the photochemical separation of the uranium isotopes have been suggested. In the past few months experiments designed to apply these suggestions have been carried out. Some 25 fractions obtained from these experiments have been subjected to isotopic analysis by measurement of their alpha and fission activities. Because of inconsistencies in the analytical data we can as yet draw no definite conclusions as to the success or failure of these methods. However, apparent deviations from normal isotopic composition of as high as 10 - 20% have been observed. Samples have been submitted for isotopic analysis by the mass spectrographic method in order to obtain confirmation or denial of the possibility that isotopic separation has been observed. In addition, a search for the sources of the above-mentioned deviations is being continued.

The ultraviolet absorption spectrum of UF_6 vapor has been obtained under high dispersion. In addition to the strong continuous absorption which sets in at about 3300 Å, there has been found a large amount of structured absorption in the region around 3650 Å. A more detailed analysis of the spectrum is being contemplated.

As suggested in previous reports, crystals of sodium uranyl acetate have been prepared in which the uranium consists of 16% U^{235} . The fluorescence spectrum of these crystals at 70°K. has been compared with that of similar crystals in which the uranium is practically pure U^{238} . Small differences in the relative intensities of certain of the weak lines appear in our photographs. Further studies are being made on a similar pair of rubidium uranyl nitrate crystals.

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Report on the Fluoro-Carbon Work

Harold C. Urey

Sept. 26, 1942

During the past month Dr. Rosen of Mr. Murphree's group, has made a very careful survey of the fluoro-carbon work, but I have not, because of pressure of other duties. The most important development in the field, however, has been the production of lubricating materials by direct fluorination procedures, by Dr. Grosse, Dr. Cady, and their group. The direct fluorination of lubricating oils from various sources has been carried on, with the production of a considerable variety of products. Lubricating oils from different crude materials give quite different fluoro-carbon compounds. Perhaps the most important observation, however, is that regardless of the source of the material and the melting points of the products, the viscosity of fluoro-carbons of the same boiling point is the same. This makes it impossible to select viscosity and volatility of the compounds independently.

A preliminary survey would indicate that suitable lubricants for use in the diffusion plant can be secured, but that it is very doubtful whether suitable lubricants for the centrifuge plant can be obtained.

The above are tentative conclusions which may be modified by future experiments.

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The Production of Volatile Uranium Compounds

Harold C. Urey

Sept. 26, 1942

Prof. Schlesinger of Chicago is now preparing about 20 grams of the uranium tetrade and samples of this material will be sent to Columbia for test on diffusion apparatus to see whether they are stable under these conditions. Prof. Schlesinger's experimental work indicates continued stability in the neighborhood of 75°, with apparently only a small decomposition at the beginning of the test. These tests were made in glass. No information in regard to the stability in metals is yet available. The method of preparation is still impractical for large scale production. Progress is being made with other methods, however. The most hopeful seems to be the use of lithium hydride and the boron trimethoxy compounds to produce lithium borohydride and lithium methylate directly. The source of the hydrogen in this case is the hydrogen of the lithium hydride. Difficulty has been encountered in getting lithium hydride because of its extensive use by the Army.

It would seem to me that within the next month we shall have rather definite information as to whether these compounds can contribute anything of importance to our program.

1257757

The University of Chicago
Department of Chemistry

November 9, 1942

Professor H. C. Urey
Columbia University
New York City

* 19950001276 *



Urey, H. C. (NY 10027)

Dear Professor Urey:

In view of your statement last Saturday that you intend, when our project comes up for reconsideration by the N.D.R.C., to propose that it be extended in some form beyond its present expiration date (February 22, 1943), I am taking the liberty of summarizing our discussion of the arguments in favor of such an action:

(1) All doubts concerning the feasibility of producing uranium borohydride in quantity have been removed by the development of new, simplified methods of preparing diborane and borohydrides.

(2) The compound does not seem to be affected by or to act upon mild steel, thus avoiding the necessity of using expensive special steels now in great demand for other war purposes.

(3) For processes for which relatively low pressures and correspondingly low temperatures (50° - 55°C.) might be adequate, the substance is unquestionably sufficiently stable.

(4) Decomposition has been more extensive than desirable at temperatures above 60°C., but there are numerous indications that the observed decomposition may be due to the effect of glass surfaces or to traces of impurities. We are now working on the elimination of both possible causes of decomposition.

(5) Now that we have developed methods of obtaining the borohydride, in the quantities needed for rapid expansion of our work, we have started our search for derivatives which might be inherently more stable or more volatile than the borohydride itself.

(6) Preliminary investigations carried out in cooperation with the Metallurgical Project indicate that there is a possibility that the borohydride may be very valuable in overcoming certain difficulties that have been encountered by that group.

(7) From the preceding it is obvious that the borohydride or its derivatives have very considerable prospects of success. But even if it should prove desirable to start with the hexafluoride, the borohydride possibilities should still be explored in order to have a second procedure, should at present unexpected obstacles to the use of the fluoride appear during the course of its future development.

DECLASSIFICATION RECOMMENDED

George Volin
Name (ADC) - Organization *DOE-ORO*
Date *10 August 1995* 1257758

DECLASSIFICATION AUTHORIZED

MALCOLM THEISEN, EASI
Name (ADD) - Organization

Date *8-18-95*

1.13.15
OR-25

Prof. H. C. Urey
11/9/42

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In this connection, two points should be seriously considered:

(a) Before the expiration of our present contract we shall have completed our work on the simplification of preparative methods. Furthermore, we shall have trained a group of younger men to replace those loaned to us by industry; when the latter return to their former positions, the younger men can take over at less cost to the project. For these reasons, we would expect to proceed with a somewhat smaller and considerably less expensive group. A six months' extension adequate for the purpose could be financed by from \$40,000 to \$50,000, or perhaps even less, depending on the unexpended balance of the present appropriation.

(b) Should the work not be continued, a group trained with considerable difficulty to this very specialized work would have to be disbanded. There would be a tremendous loss of time in reassembling and retraining another group should it later be decided to take up the borohydride studies once more.

For these reasons, a six months' extension, which would probably enable us to complete the work, is considered extremely desirable.

I hope this resume will be of value to you in presenting the matter to the Committee.

Sincerely,

(Sgd)

H. I. Schlesinger

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