Lehigh University Lehigh Preserve

Theses and Dissertations

1959

An apparatus for the determination of vapor-liquid enthalpy and phase equilibrium data

Arthur Henry Koeckert Lehigh University

Follow this and additional works at: https://preserve.lehigh.edu/etd



Part of the <u>Chemical Engineering Commons</u>

Recommended Citation

Koeckert, Arthur Henry, "An apparatus for the determination of vapor-liquid enthalpy and phase equilibrium data" (1959). Theses and Dissertations. 5008.

https://preserve.lehigh.edu/etd/5008

This Thesis is brought to you for free and open access by Lehigh Preserve. It has been accepted for inclusion in Theses and Dissertations by an authorized administrator of Lehigh Preserve. For more information, please contact preserve@lehigh.edu.

AN APPARATUS FOR THE DETERMINATION OF VAPOR-LIQUID ENTHALPY AND PHASE EQUILIBRIUM DATA

By

ARTHUR HENRY KOECKERT

AN APPARATUS FOR THE DETERMINATION

OF VAPOR - LIQUID

ENTHALPY AND PHASE EQUILIBRIUM DATA

рà

Arthur Henry Koeckert

A Dissertation

Presented to the Graduate Faculty

of Lehigh University

in Candidacy for the Degree of

Master of Science

Lehigh University
1959

The second of th

Alto Barrelline Contract

95 f 15 To 983 ...

ydinasvin "okop. 1989 This dissertation is accepted and approved in partial fulfillment of the requirements for the degree of master of Science.

(Date)

Professor An Charge

Head of the Department

TABLE OF CONTENTS

ARSTRACO	Pare
ABSTRACT	1
INTRODUCTION	2
DEVELOPMENT OF THE DESIGN	_
The General Design Equilibrium	5
Equilibrium	8
Enthalny Magairement	8
Equilibrium Enthalpy Measurement Adiabatic Operation	12
	18
DESCRIPTION OF APPARATUS Recirculating System	
Recirculating System	21
Recirculating System Adiabatic Walls Power Supply	21
Power Supply	32
Cooling Water System Thermoregulator and Controller	33
Thermoregulator and Controller Temperature Measurement	33
Temperature Measurement	38
Temperature Measurement Pressure Measurement and Control Composition Analysis	41
Composition Analysis	45
	48
OPERATING PROCEDURES Program of Test Runs	
Program of Test Runs Start-up	49
Start-up	49
Start-up Steady State Operation Data Collection	50
Data Collection	53
Shutdown	55
	56
RESULTS Measurements	57
Measurements Difficulties of Chematica	- •
	57
Performance of Equipment	61
Out of the contract of the con	64
CONCLUSIONS AND RECOMMENDATIONS System Design	71
System Design Instrumentation	
Instrumentation Equipment	
Equipment Scope of Investigation	16 74
Scope of Investigation	78
APPENDIX	,,
Calibrations Heat and Flow Rate Determination Insulation of the Calorimeter	84
Insulation of the Calermination	97
	99
	00

mi bevores our week sit was a sure

eyrade increamon

Javadnege de to ben.

Lo compete ear tot in control of the depres of

and the second desired.

TABLE OF SUPPRINTS

aWa I																									
Ţ		•	•	•	•		•			•	•	•			•		•						ι Ű;	ijΪ.	٤.
3	4	•	•	•	•	•	•.			•	,	•	•	,	·	•	•	•				÷	, U A	. U '	•
Ğ																	_							. 17	,
į																							- 44 x - 1 1 1 - 1 1 1 1 1	νΥ	·
e																							LIT.		
uI.	Ì		•						•	•	•	•	•	•	70	٠,	•	• •	• ••	4		L.A.	i ki ki ki Ki ki ki		
81																							, L.		
28	•		,			•					. ,							•					i	tartte e	·
ĬŜ																									•
17.7																						•			
ટ હ																							Not		
55				,										•	, ,	,	•	i¥		aria Par	•		000		
33	•	,		,						43	٠ .	· <u>.</u>			,			4.	i	ניסטו	اوم. دومورا	 .a. m	3. A.		
ŢΫ																									
34									10	TĴ	al O		.)	: .	 :	· •}	36	ui.	A3 1=1:	~~	3 64 F	ta e	~		
46	•			,	•		•	,	•				•		. !	٠,٠	. c	23.2		•	1	ر در در ا اورا	 نان		
49													•						(. •	ر ان	, .				ķi
A.F.																							٠٩.		
٥č																									
53	•	•	,				· •		,	.					;	٠.		•	· į.	. د د		4:	.) د.		
55				•		٠.	4				,	_					~	,	: 2.	1	'nς	. ;	i Ani		
36																							and the		
76		•				٠			;	•	ų				4										
Ya	•	4	•	•				.•*				٠	٠.					:					٠,		
In	•		•	٠		•	•,	•	•		•	1		4 %	ė	٠.		:	5 1	ر بائد ج					
Þĉ	•	r	٠	•		•	٠		•	,,*	,		Ю	r,	3 /	, i.			• 0	. '	.992	. 1			
25	•		•						•		٠		 	٠,	¥	e e	٠,						. ئ	٠	
<u>t/,</u>	•	-	٠.	•	4.		•	é		٠	٠,					, ·		:12.	: :	ðs.	į		77.6		
34	•	•	•	•	٠	•			•	ø:		٠		•	,		٠,	16	22.4	I(t)	a: L	1 9	in E		
74				•	٠	,		r				,			,				,	ij,	تانگ	Ĵ.,	17.4		
73	•	•	•	•	٠		•		•		•	•	r.	٥.	JE	(1) p.m.	ជ អ	(1 \	441	ζ.	J t	ن پڻ ا	3 4		
N.C																								· · · · · · · · · · · · · · · · · · ·	
64 97	٠	•	٠	i	•		•	٠	• .	•	•	•	• .	•	•	•	•	곱.	40	131	3.16	11	A.		
86 Le	•	•	¥	4	٠	*	•	•	.) <u>r</u>	JA	nl	Lil	øj.	91	Ö	Jái	<i>!</i> .	% O	<u>.</u>	الأ	(\mathcal{P})	JE	Ble		
-	•			•	•	•	:	•	٠,	T9	S O	mt	TO.	Liz		ыŲ	đ	LO	:5	OAG	î o I	ua.	πí		
100	, A	BL	U	12	Ite	100	ri.	0 (X	n	or	1	no.	tt	a i	cv.	Ĺ	<u> </u>	J B	9.1	dr.	es	ØL.		

		Page
•	Run 9	
I	Run 9 Run 9 Run 8	101
	Atra Heat Sources, Run 8 diabatic Enclosure	107
R	man in Plan Day	108
_	diabatic Enclosure Fror in Flow Rate Due to Hovement of Rotameter	100
P	TORRITA Change Com. T.	110
	in Equilibrium Flask illing and Setting the Thermoregulator eferences	
r	1111ng and Satting the ma	113
Re	eferences	115
		117
PIOURE	S .	
_		
1.		
٤.	Equilibrium Flask, Contacting Section, and	15
_	Adiabatic Walls Recirculating System Components Panel Board for Flow Control	
3.	Recirculating System Components	22
4,	Panel Board for Flow Control Sampling Equipment and Botlom Dane	25
5.	Sampling Fourthment and the second se	27
6.	The Cooling Water System Thermoregulator	31
7.	Thermoregulator Thyratron Relay Circuit Thermocouple Circuit	34
8.	Thyratron Relaw Canana	40
8.	Thermocouple Circuit	42
10.	Table I. Latent west line	44
11.	Temperature-Composition Discussion of	5 8
12.	Temperature-Liquid Composition Diagram Table II. Data of Bun 2	60
13.	Table II. Data of Run 8 Table III. Data of Run 8	62
14.	Table III. Data of Run 9 Thermoccuple Calibration for Entered	82
15.	Thermoccuple Calibration for Enthalpy	83
10		
16.	Table IV. Effect Upon Flow Rate of Error	87
1 77	in Position of Rotameter Float Calibration of Refractometer	0.0
17.	Calibration of hefractometer Table V. Design Date	93
18.	Table V. Design Data Rotameter Calibration Curve	95
19.	Rotameter Calibration Curve Length of Rotameter Scale	97
20.		111

ABSTRACT

An equilibrium still has been designed, built, and tested which may be used to determine liquid-vapor equilibrium compositions, the equilibrium pressure and temperature, and the isobaric heat of vaporization at the vapor composition. Investigations are limited by the apparatus to pressures near atmospheric and to miscible systems not highly volatile at room temperatures. The pressure may be held at one atmosphere within 0.7 mm lig. Equilibrium temperatures may be measured to within 0.1 degree (C). From the 480 ml of liquid required for operation, about 80 ml forms the vapor phase. The vapor is condensed in a reflux condenser where the latent heat is determined from the rise in temperature of the cooling water. A countercurrent contacting section below the condenser insures that only the latent heat is measured. The condensed vapor circulates by gravity through a rotameter at from 2 to 5 gm per minute and is revaporized in a tubular boiler. A bubble cap is used to contact the vapor with the equilibrium liquid held in a flask. Adiabatic walls of hoated aluminum pipe reduce the heat transfer between the system and the surroundings. The apparatus was tested with the ethanol-water system for which enthalpy-composition charts are available. The experimental latent heats fall within 5% of the literature values. The liquid line on a temperature-composition diagram agrees well with the literature, but the vapor line shows enrichment due to internal reflux. Design changes are proposed which will eliminate the reflux, improve the accuracy of the device and reduce the operating time.

TOAMTEGA

An equilibrius still has seen designed, built,

con tempted water by deed to determine liquid-vapor could braus dospositions, the equilibrium procesure and tonpresenting and the figures of valential and the property of the rate of the state of the transfer of the the transfer to Tide to don enclose Editoria o el trimogo que tala summinos. or office of the originating care agree of round a round as a life for today properties in the second statement of the In the originate and, course in the High of hermanian or gre was a treat for an approve another makes and action in the tennabios and a selection of range on their AMONTO TO STATE THE SOURCE STORE STORE STORES The second members of the second of the seco Some Trible of Time and empired control of the sound of the the property of a second country of the second of the seco the state of the s and the second of the same of the Live of the second \mathbb{Z}^{2} , \mathbb{Z}^{2} BOLL BERTHOLDS CONTROL OF THE CONTRO Section to a consistency of the figure of the And dark to the office and the second of the second of the office of the second of the and the **property of the second of the secon** and the property of the second drameirne sound amil cond and draw a thing this had not cho se internal reflux. Bosign charges are proposed which and to common and everynt this too common of the device and reduce the operating time,

INTHODUCTION

A knowledge of enthalpy values for vapor-liquid systems is necessary for the accurate determination of stage requirements in distillation column design. With phase equilibrium data the enthalpy values may be used to construct the entire enthalpy-composition diagram for graphical solutions or may be used in tabular form for analytic solution. In addition, the latent heat values make possible the application of rigorous thermodynamic theory to the testing of the consistency of the equilibrium data as shown by Dodge (1). The necessary data for the construction of the thermodynamic network even for binary systems is scattered throughout the literature and is frequently of doubtful accuracy, and for most systems is simply non-existent. Therefore, diagrams for only a few binary systems have been constructed. Graphical treatment and computer methods for multicomponent systems using enthalpy-composition data are currently undergoing study. To make such methods useful, enough data to permit the accurate construction of the network must be made available.

Four kinds of data are required to construct the thermodynamic network from experimental measurements. These are phase compositions and corresponding temporatures and pressures at equilibrium, heat of mixing, specific heat, and

THERUDUOCIUM

a Lapsiledge of anthalpy values for value to what is not deal of the cook of the cook is the second to the second oras cale ingles, resules melselables of the entropy ortimes of the solvent applier joundance out mother modified to -: Low froling or, to . . worder not leagues - to form of the continue to The establish of some and a second of the medical terms of the second second walls of the first and the value of the value of the first of the firs - Grisk of the Col Chamber Simmer of the Color of the Color of THE A. JO LESS TO SEE MADICALL TO BE SEEN TO SEE THE SECOND OF SAME OF SAME OF THE POST OF THE SAME AND THE SAME OF T with this contradiction as a street gaunic not gas a manage plant A the last the second of the figure of the second of the second of Total Committee of the State of the Committee of the Comm . His warned the form and the grade to yield be the form della discoppagne i della discoppagne della dell History (11) access the same of devices from the contract of the same sections. the starting with prolessor tookstop is a second subject to his TO A TO STORE MEDICANE SEE TO ARE LONGING TO SOME HELD STORE THE STORE OF THE STORE 48. 1. 1. 2013.

post with a number of the are required to a nontreasing the are placed as a number of the area of the

heat of vaporisation. All must be determined over the full range of compositions. The relationships among them may be visualized by considering the construction of an enthalpy-composition diagram, heat of mixing data determined along an isotherm provide a base line above which specific heats, bubble points and heats of vaporization are used to construct the saturated liquid and vapor enthalpy lines. The phase equilibrium data provide the tic lines which complete the diagram. The thermodynamic network so constructed is as accurate as the data. The only assumption is that the data points be close enough to permit interpolation between related points at adjacent compositions.

The apparatus described in this report is the result of a complete revision of equipment which was constructed and operated to test the basic features of the design. The present form was designed with more consideration for insulation, is more compact, and incorporates changes found necessary from operation of the original apparatus.

was omitted because no unique features would have been employed. Attention was directed to the more challenging problem of latent heat measurement. The heat of mixing was left unexplored simply to limit the scope of the investigation.

I want to thank the Texas Company for providing the fellowship which made this work possible, and the

near of vajorisation. All must be described over the full range of compositions. The constituential among them may no objection displayed by considering the construction of an enthelpy-objection displayed. Heat of mixing date determined along melocition displayed and includes a base line above which entering above to constitue above and the entering of the constituents. And if your mission is under the constituents and the state of the state of the constituents and the state of the state of the constituents. The constituents are constituents and the state of the second of the constituents of the constituents.

Code and the first time of relatives resident and the control of the code of the code

The Arthur of the second of th

A ward to them one forms Occasing for providing the followedly which mode this note possible, and one

faculty and staff of Lehigh University for their many help-ful suggestions.

4

faculty and staff of Lehich University for their many help-int suggestions.

DEVELOPMENT OF THE DESIGN

The General Design

Three of the four kinds of data needed to determine the enthalpy-composition network are so closely associated that when the conditions required to measure one are met, the other two may also be measured. These are the phase equilibrium compositions, the heat of vaporization of the saturated vapor, and its mean specific heat in the condensed state. Construction and operation of a single apparatus to determine all three, if not too complex, saves the time spent constructing and operating three pieces of equipment. In principle, a liquid mixture of the components at the composition to be tested is brought to its bubble point, then is vaporized and brought into contact with a liquid mixture of equilibrium composition at the same temperature. To obtain the necessary data an apparatus must be constructed in which the first mixture follows the precise thermodynamic path described above at constant composition. Appropriate measurements of changes in the surroundings permit the determination of the enthalpy values. It is evident that a bomb calorimeter or equilibrium cell is inadequate because the fluids must pass from point to point through the apparatus.

WORKER WY SU THERE WAY V

regimon ingress? was

uningered through the confident descending that wied a last the second was a man that RESIDE IN THE COURSE OF THE WORLD WINDOW A STREET offs to allowing the first per and general assence that it CONTROL OF A A CONTROL OF A CON of application of partial control of the control of and the second section of the second contraction of the second contrac April 2, me to the control of the State of the control of the cont which gift it was been a to be a selected to the good bound the in grate the little was the second of the second of the second of the second of Bright Color of Carlot Carlot and Carlot Car The second of th indonedance ed Jam - Her in de Løin innerender i de Seride bloomed was a consequence of a collection of board and a local collection The street will be a second of the second of San time a regulation of the content of the second sections and the live of a land region in the land of the street was a tout calors were or enuls offer cell is insidented demonds ando of dulo, modi ager Jean atlait odd cassaced tico accarrates.

A flow calorimeter may be chosen which provides either a single pass or a recirculating path for the phase at the Vapor composition. Both designs offer important advantages. In the single pass design, the moving phase flows from a holding tank into the enthalpy measuring and phase equilibrium regions; it then flows into a receiving tank. Since the mass flow rate may be accurately measured by weighing the receiver, the design has been the principle method used for enthalpy measurements. The design by Dana (2) and modified by Tallmadge, Schroeder, Edmister, and Canjar (3) is of this type, and both are examples of accurate heat measurement by electrical means. Both apparatus contained a stationary liquid phase through which the moving phase was passed, and the latter reference combined equilibrium measurements with the enthalpy values. But it was felt that the liquid did not represent the composition in equilibrium with the moving phase, that it was difficult to keep the volume of liquid constant, and that uncertainties, mentioned by Weissberger (4), in corrections for heat leaks prevented confidence in the enthalpy measurements. The advantage in flow rate measurement was exploited by McCracken and Smith (5) through the use of automatic valves in the product lines, and in highly accurate measurements by Callendar and Barnes (6), but neither design is applicable to equilibrium measurements. A variation of the once-through design by Rosanoff, Bacon, and White (7) and used by Cornell and Montonna (8) operated

a flow calorimeter may be chosen which provides ofther a single same or a recirculating eath for the phase the war account than, both designs offer important end of the same slucie one does on the moving place one ondringned to I have been outlied and a large of him week. inserted it is a section of the sect formered glodeness of the last tole there all the last adofgloaine cid in ed out a little one in who bods a little in-4. man, vd milsob office esminar base, time for been brider (a) the contract of the contra and offeren is a firement of the second of Deministrato estampera libora, esta la instituta de la contenta del contenta de la contenta de la contenta del contenta de la contenta del contenta de la contenta de la contenta de la contenta de la contenta del contenta de la contenta del contenta del contenta del contenta del contenta de la contenta de la contenta de la contenta de la contenta del conten CONTROL OF THE STATE OF THE STA Astronomic multiplication of contract to the contract of the c med does a fair how as the sound that it was a sound to dia de la marcial de **la compactació** de la compactació del compactació de la compac employ els geel of sireful to the the ways are a low to To been track and that was a fact of the contract of the contr Designation; where I real that encourage is the character of the conif opposite the later with none you will be all cold facult They is the control of the state of period or a second of the second of inger, a bus use of secondaries of the product lines, is oughly contacte descurements by Callender and James , (), but nearly decign is applicable to equilibrium measurements. A variation of the once-through design by losenoff, Bacon, and thite (7) and used by Jornell and Jonkonna (8) operated

can ever be reached with that form of apparatus, the design was dropped from consideration. The long operating time that most authors find necessary before phase equilibrium is considered established would seem to preclude the use of a once-through design because of the large quantities of liquid components which would be required to supply the moving phase for periods longer than a half hour.

The other alternative design, in which the moving phase is recirculated through the apparatus for as long a time as is necessary, has become widely accepted for making equilibrium measurements. This design, in which the recirculating phase continually repeats the exact thermodynamic path required for the desired enthalpy measurements, is the form used in the present study. The chief disadvantage appears in the attempt to make accurate flow rate measurement. Direct mass flow rate determination is not easily accomplished, since the flow of fluid must be diverted into a weighing tank for a period of time, before and after which a weighing must be made. The resulting depletion in some other section of the apparatus and the cessation of flow into parts downstream of the weighing point cause the steady state operation to be disrupted. Usually the recirculation is caused by a natural convection process and is maintained at a constant rate with constant liquid levels. If weighings were to be made, a pump would be required to maintain

As a differential distillation, and since no steady state can over be reached with that form of apparatus, the decign was dropped from consideration. The loop operation cinc the decign that necessary before phase equilibrians as considered and necessary before phase equilibrians as considered that seem to proclude the use of a concentration design because of the length enterties of the length of the length of the lings and the second of the length of the lings are considered to the length of the lings are the supply one will be a conserved to the length of the length of

graves has deter hi ingineh evisansesys corse s

o grai e voi emaracera eda agara d nos arergas el curre ya Bikan 1965 (1991) dan jilahik binda berin ya nanyabi bili bili bili ya k ent united to eronours. This during in which the rooters. plantagements forms and manager yills minima or a paint of ond all inductivations, yelled the leaders out is boulded did a Joseph and the respondent amplitudes, the same of all advantage La Morrentine entre confirmation entre and translation of the confirmation of Charlailignmans wilters were si wold, mismades of the wolf of the aparts guidelor a contactorate of the three contact a contact bear for a correct of them, before our after which a vehicle reduce amon at attitologo grade men est a con od istor yna odni well he neithreen one ban entaritys end is neither party development of the volgiting point ease the monday evade operation to be dismapped, Samalay the recirculation is caused by a namural convection process and is saintained et a constant rate with constant liquid levels. If reighlings were to be made, a pump would be required to maintain

The direct mass method for flow rate would unnecessarily complicate the equipment. Rate measuring devices which may be inserted into a fluid stream and which operate without disturbing a steady state flow were investigated. Of these, the ball type retameter was chosen as the most accurate of the direct reading devices for the low flow rate expected.

Equilibrium

The development of the design features of contemporary equilibrium stills is outlined in two excellent reviews by Fowler (9) and Midgway (10). In the design most favored today, vapor generated externally or in the equilibrium chamber is condensed into a vapor trap. The condensate recirculates either into a vaporizer attached to the equilibrium chamber or flows directly into the equilibrium flask to be revaporized.

(11) and by Murti (12, 10) suggest that the recirculating phase be vaporized before contacting the equilibrium liquid. With this provision the liquid in the equilibrium flask more nearly represents the true equilibrium liquid. The Othmer design (14, 15, 16) which employs the direct return of recirculating liquid to the boiling flask seems to prevent the establishment of the equilibrium liquid composition. The recirculating phase may be thought of as residing as liquid in the flask for some period of time before revaporize-

the constant rate necosary for accurate enthalpy values.
The direct mass method for flow rate would unnecessarily
out ligate the equipment. Rate measuring devices within any
be insarted into a fluid stream and which eparate without
distribution a seedy state flow were investigated. It there
the call expendence for well chosen as the mean accurate of

my religion.

The state of the s

(1) an explaned till, I see estitute to methodische chare in a constant prime in expression which is the contiller of the limits.

It this executation whe liquid in the equilibrium liquid. The dimensional manner of the constant proposed to the crack equilibrium liquid. The dimensional field the constant of the constant prime is the constant of the

ing. Depending upon the amount of flask liquid, the recirculating rate, and this indeterminate residence time, the flask liquid is more or less too highly concentrated in the volatile components to have the true equilibrium composition.

The comments of Swientoslawski (17) seem to indicate that the vapor formed by boiling a flask of liquid is superheated. He noted that his apparatus, using a Cottrell vapor lift to direct a vapor and liquid mixture onto an accurate thermometer, indicated a higher temperature when the boil-up rate was increased, even when the design provided a long contact time between vapor and liquid.

Vapor leaving the liquid surface within a heated flask would be in contact with the liquid for a far shorter time and would probably be much more highly superheated. For these two reasons, a separate vaporizing chamber was incorporated into the design.

It is sufficient in a study of binary systems to monitor the approach to equilibrium by noting the temperature of the equilibrium liquid and waiting for that temperature to become constant. But it may be possible for multicomponent systems to have a series of equilibrium points at the same temperature for a given total composition. By equilibrium points is meant the mass distribution between the phases, and the compositions. A still could operate at a constant temperature while the mass distribu-

ing. Depending upon the amount of flask liquid, the resirculating rate, and this independents residence time, the the flask liquid is more or less too highly concentrated in the volatile components to have the true equilibrius corposition.

And consequently of satisfactory of the control of

Associated and anticolous and the absociation of the proportions of the state of th

tion or the composition of one of the phases as well as the temperature.

A convenient method of watching the mass distribution is to restrict the system to two liquid phases by design, then monitor one of the liquid levels.

A check of the composition would require either periodic sampling with the chance of upsetting the operation or an in-line composition analyzer. Since it would be difficult to make sample analyses during a run and since no continuous analyzer was available, the method of liquid levels was chosen.

With these considerations in mind, a tubular boiler was chosen with inlet at a slightly higher level than the exit, as used by Jones, Schoenborn, and Colburn, and by murti. The liquid level was fixed at the inlet of the downward sloping boiler, and the amount of liquid in the recirculating loop was fixed by the liquid level at the point where the condensate collected from the condenser. As explained in the "Description of Apparatus," the latter level was brought outside the insulation so that the level could be observed. The single other liquid level was at the equilibrium flask. There were, therefore, only two liquid containers, and only two liquid levels capable of change. One level could be watched accurately, changes noted, and control action taken. The distribution of mass between the two phases was measured by the visible level, and a constant level would be indicative of a steady state operation.

tion or the composition of one of the phases as well no the composeture.

decign, such desired to the line appears to the the men distribution to be considered by decign, such desired as the line appears to the line layer.

unded on a continue of management could be sented of the continue of the conti

referred to contract and a contract needs that ands fovol cody of the gift of a fact of the test of the ve the investigation has a mode when a composition of the composition of wowoo and to doling and benil saw reast organic toll . Large waste to make the before the transport of the fact that the could be a weekeld the contract of the the latter level of the solution THE SECOND CONDENSES ON A SECURITOR SEC CONDENSES. ME werent and "the base of the first of the contract of the first of the following of Lovel edd or id on doldriner? ods oblacto ddiagodd waw fewal the do new Lovel bear I worke of while only they and the optimizations of the best of the control of the tree liquid containers, and cally two lifetic levels expaide of change. ene level sould be watehed coourately, obsumes coted, and control action taken. The distribution of mass between the tro phases was neasured by the visible level, and a constant lovel would be indicative of a steady state operation.

The validity of this analysis depends upon preventing the formation of any other liquid hold-up which cannot be measured and controlled in volume, such as an accumulation of liquid at the low end of the boiling tube.

operated in such a manner that superheating of the vapor could be prevented. Control of the heat input to the vaporiser would control the vapor temperature at its outlet. This temperature would then be matched with the temperature of the flask liquid and that of the vapor leaving the equilibrium flask. The equilibrium vapor would then be at its saturation point throughout its contact with the equilibrium liquid. The analysis of the control of the boiler proved to be incorrect, as is shown later.

an auxiliary boiling flask in which the vapor is generated prior to contact with the equilibrium liquid. Such a system was described by Chilton (18) and used by Scatchard, Raymond, and Gilmann (19). The system suffers from having three containers of liquid among which the contents of the still are distributed. In two of the containers, the boiling flask and the equilibrium flask, the liquid levels are turbulent and cannot be determined or controlled. Thus the distribution of liquid among the containers cannot be readily determined. Additional arguments against this design are presented by Othmer (16).

The validity of this analysis depends upon preventing the formation of any other liquid hold-up which cannot be assaured and controlled in volume, such as an accumulation of liquid at the low and of the beiling tube.

it was boomput that an expersel boiler could be openated in such a mannor that superhesting of the traction of the such vaporises with the superhest was the contract that on the compose with the best are interested of the contraction of the final limits and and of the final limits and and of the final limits and the openation of the contraction of t

The only discrete we willer notice consists at anterest a would be willied to continue the continue to continue appear and described by oblition (18) and used by demonsred, armond, and eliment (19). The system suffers from having three containers of the still are distributed, in two of the conteiners, the collection and the final final, the liquid levels are turbulent and cannot be determined or controlled. Thus the distribution of liquid enemy the conteiners cannot be readily determined. Additional arguments against this design are presented by Othmer (16).

a simple bubble cap. It was felt that this design would provide effective vapor-liquid contact, reducing the recirculation time required to achieve equilibrium. The vapor was forced to bubble through a two inch liquid head as it contacted the liquid, causing a pressure drop of about 3 mm Mg. For operation at 1 atmosphere this represented 0.3% change in pressure and had but a small effect upon the equilibrium. Noyes and Warfel (20) report for the ethanol-water system a change of 0.03 degree (C) in boiling point per mm change in pressure. Thus the change of 3 mm Mg in the equilibrium flask could be expected to produce a 0.1 degree (C) uncertainty in the reported equilibrium temperature.

Enthalpy easurement

The recirculating still causes a stream at the saturated vapor composition to follow a thermodynamic path ideally suited for the enthalphy measurements needed in the construction of enthalpy-composition diagrams. The two heat effects which can be measured are the isobaric latent heat of vaporization or condensation and the heat capacity of the liquid, all at the vapor composition. To understand the advantages of the various design alternatives, it is of help to consider the circulating fluid as a thermodynamic system operating in a cycle between two temperature levels as shown in Fig. 1. The system serves only to transfer heat between the hot and cold reservoirs. This it does by

THE EQUILIBRIUM STILL A THERMODYNAMIC CYCLE FIG. 1 COLD RESERVOIR (CONDENSER) LIQUID VAPOR HOT RESERVOIR (VAPORIZER)

The design chosen for the equilibrium Task was a simple bubble cap. It was felt that this design would crowide effective raper-inquid contact, reducing the rectired dulation time required to achieve equilibrium. The vacor was forced so abble chrongh a two inch liquid head as it contacted the liquid, canaing a two inch liquid head as it contacted the liquid, canaing a measure damp of about 3 mm of the contact or at a abmosphere with response the contact of the contact of the contact of the contact of the contact affect wother water system a change of 0.07 decree (0) in collier wother cate contact in pressure. The the contact of t

a con ounsee Termique

The reduced bing such because correspond to the substitute of the such and the such reduced of the such and the supposition of the such and supposition of the such as the substitute of the such as the substitute of the substitut

being heated to the bubble point, vaporized and superheated at the hot reservoir, and condensed and subcooled at the cold reservoir. The enthalpy change between vapor and liquid may then be measured by some known change in the surroundings at either reservoir. The problem is to separate the latent and sensible heat effects so that both may be measured independently.

At the hot reservoir, a subcooled liquid would have to be brought to its bubble point without producing vapor, using a measured heat input to determine the specific heat. This would prepare a saturated liquid for additional heating to determine the heat of vaporization. The temperature of a mixture undergoes a more or less regular rise from the subcooled liquid state to the completely vaporized state without the occurrence of a discontinuity in temperature or region of constant temperature. Supposedly the bubble point temperature is unknown, the purpose of the experiment being to determine it. The stream may not be viewed to notice bubble formation because of the necessity of insulating the measuring region. It would seem that no convenient characteristic of the stream could be used to establish the point where vaporization begins, and thus the latent and sensible effects could not be isolated at the hot reservoir.

A simple design has been devised to isolate the latent and sensible heat effects at the cold reservoir.

The equilibrium determination associated with the apparatus

being heated to the bubble point, vaporized and superheated at the het reservoir, and condensed and subsected at the cold reservoir. The onthalpy change become varies and liquid may then be seared by some theometically one, which come, and supported by some theorem one, and constant the search and the latest reservoirs. The search is the search of th

The second of th Single Committee of the and the state of t and the state of t na transport i de la companya de la La companya de la co the contract of the contract o $(x,y,y) \in \mathbb{R}^{n} \setminus \{0,1,2,3\}$, which is the $(x,y) \in \mathbb{R}^{n}$, $(x,y) \in \mathbb{R}^{n}$, $(x,y) \in \mathbb{R}^{n}$, $(x,y) \in \mathbb{R}^{n}$ The second of th and the second of the second o The property of the particular of the property Treatment or true cost which it was not a reason with provided the contract was all before also as a sold to categoristic. per a destable of the samp and good gradiged noor and to govern unser which committe effects could not be isolated to include the restructs. A single design has been devised to isolabe the

letent and sensible neat effects at the cold throwvoir. The equilibrium determination associated with the apperatus

AJ.

provides a supply of saturated vapor which may be fed to a condenser. The problem is to insure that a saturated liquid leaves the condenser, thereby removing no more heat than the integral isobaric heat of condensation. Vapor condensing on a condenser tube wall is subcooled as the liquid film builds in thickness. If the incoming vapor contacts the subcooled liquid leaving the condenser, some of the vapor will condense into the liquid and heat the condensate towards its bubble point.

A reflux condenser provides a means of contacting the vapor and condensate. The vapor condenses in the condenser, yielding its latent heat and some sensible heat to the water used for the enthalpy measurement. The condensate flows down the wall of the condenser countercurrent to the rising vapor. If an insulated section of tubing is provided below the condenser, then the downflowing subcooled liquid is exposed to the rising vapor and partially condenses until the liquid reaches its saturation point. Since a saturated vapor and liquid at the same composition are at different temperatures, the driving force to heat the liquid is substantial. If a trap is provided at the bottom of the contacting section, the liquid is prevented from returning to the equilibrium flask, and may be discharged to the recirculating system. Since a saturated vapor flows into the condenser and a saturated liquid of the same composition leaves the region, only the latent heat is removed by the condenser. The ability of this condenser arrangement to

provides a supply of saturated vapor which may be fed to a condenser. The problem is to insure that a naturated liquid leaves the condenser, thereby memoring no more heat than the invested declarate heat of condensation. Vapor condensing on a condenser take well is subscied as the injuid the injuid of the injuid of the injuid valor content that this injuid is subscieded injuid its absorbed the injuid of t

and condenses another a contrary as a contrary as the wallow and constants to the event condense in the comdonest, Alland the late of the or action of the restance elustaben in the construction with the end of the substitution and lot a down the real of the someones are proported two lists siding warms. If an invalition coupled of which is provided polow the sendencer, them the downloathe embodeded lightle og ceod to the glaing value, com captingly coor cases about the liquid reactor in earth total of litter setureted vastr and liquid as the cape composition are as different temeration, the original verse to meet the limit of is substantial. If a tran is egovided as the between of the contecting section, the liquid is provented from retarring to the equilibrium flesh, and may be discharged to the recirculating system. Since a naturated vapor flows into the condenser and a saturated liquid of the ease composition leaves the region, only the latent heat is removed by the condensor. The ability of this condenser arrangement to

degas the departing saturated liquid was noted in a recent article by Othmer (16), and the design forms the basis for a patent for gas-free distilled water (21).

The various designs of recirculating stills which use a sealed system and regulate the pressure by the adjustment of heating and cooling offer no advantages over the designs which use a direct control of a confining pressure, with the possible exception of operation at high pressures where the confining gas might have appreciable solubility in the condensed vapor at the bubble point. Indeed, these designs offer distinct disadvantages for enthalpy measurement at the condenser. During adjustment of the cooling rate, conditions unfavorable to accurate enthalpy measurements may be set up. Maintainance of the coolant AT within the desired measuring range may be impossible, with the resultant inaccuracy in measurement of AT and the opportunity for excessive heat leaks from the coolant to its surroundings. The problem is basically the fact that with a purged sealed system, the total inside condenser area must be used to condense vapor. The condenser may be operating in such a manner as to attempt to remove too much heat from the system, thereby tending to reduce the pressure. Various experimenters have attempted to correct for this by using only small portions of the outside area of the condenser (22) or by changing the coolant from water to air (22, 23) in order to reduce the ability of the coolant to remove heat from the system. One other possibility, which

degas the departing saturated liquid was noted in a recent article by Othmer (16), and the design forms the basis for a patent for gas-free distilled water (21).

The various designs of rectronisting stills watch use a seeled spaten and regulate the presence by the adjustment of heating and coeling effer as advantages even the designs union use to direct to threat of a contributing protection. with the possible exception of providition at high croscuror where the configin gas of at cover a recover sole and the in the condenge, vener as the mittle , sins, undaed, turner -nomestic veltation tole require values and take tello anglach mone at the condenset, butther to be thought of an conting -orangement tologies and consults to according to enablations and according minist to design on the constant of the constant of the second of the constant the demined areasy as years of the sease boarses the road bant inacouracy in regarises it of all wid wis octorbuniby for excessive hest leads from the covernme on its ANAM 3. AN COST and Wilkeriche to 1964: and the for Jordan a purged sealed ayang, in total list a condense a server a be used to concense value. The condensate and be of exciting companies de la companie de la compa the system, thereby tending to regues buy aresoner. Various experimenters asve accompted to correct for this by using only small portions of the outside area of the dondonser (22) or by changing the coolant from water to air (22, 25) in order to reduce the ability of the scolant to remove heat from the system. One other possibility, which

has not been tried, would be to adjust the recirculation rate to match the condensing capacity of the coolant. But such a method would prevent a much desired flexibility in operation.

The use of a reflux condenser connected at the top to a reservoir of confining gas automatically compensates for the changing demands of the system for cooling and permits simple measurement and control of the system pressure. The average coolant temperature and rate may be adjusted arbitrarily as long as vapor is confined to the condenser. At whatever conditions of recirculation rate or coolant rate are chosen, the vapor inside the condenser rises to just the height necessary for it to be condensed. In so doing it draws in or pushes out the confining gas in the reservoir. This phenomenon undoubtedly took place in the recent elevated pressure apparatus by Othmer, et. al. (23) although to a small degree. The reflux design with communication to a gas reservoir permits the use of a coolant whose specific heat is accurately known, namely water. Keeping the AT at a low value and the average temperature at room temperature reduces the heat leak without elaborate insulation. The use of cool water means that the upper section of the condenser may be kept at a low temperature to reduce the amount of vapor diffusion into the gas reservoir. A supply of room temperature water is easy to maintain at constant temperature and to supply for use in the enthalpy measurement.

has not been tried, would be to adjust the recirculation rate to match the condensing capacity of the coolant. But such a method would provent a much desired flexibility in operation.

The use of a refler sendence remiseded to be use top to a reservoir of confining gas automatically descendance. for the manning decimies at the system for ecoming one nowatts ourse mossage and control of the system or season. the average equipment a connection of the contraction of the seed AND ADDITION AND DO NOT TAKE HIS HOUSE OF A DOOR AN AT A DECIDENCE TOWN t amen was consitted of regimenter or race or race or race see chorum, and veget mitch i i minutus ili de marede see this noting accept the fell to be communitied in the communities. in draws to an electron on, the confidence of the second transfer of whateh with the training the companies with the companies of the companies elevated frequency to are the a level of the all olders. o religional construction of the contract of the contract of the religion of p pas coscreção e edite tado el o ecolor a mase apresidades heat is acqueately known, access water. complete the AT -creques and its equipmented against and ord puller tol a da inve soluces and look leak atthough elebrates anemics even The nee of cool water means that the namer section of the condenser may be kept at a low respective to reduce the amount of vapor diffusion into the apa redermit. A supply of room temperature water is easy to maintain at constant temperature and to supply for use in the enthalpy measurement.

one disadvantage involves the condensation temperature at the condensar. The condensation temperature depends upon the system under study and upon the confining pressure. For the ordinary solvent systems likely to be studied with this apparatus at one atmosphere pressure, the boiling points are substantially above room temperature and a water condensar operating near room temperature will condense the vapor. However, an example of a specific system system which presents problems is ethylene exide which boils at 10 0 and cannot be condensed using room temperature water. One other shortcoming, the opportunity for the diffusion of volatile components into the gas reservoir, must be mentioned.

Adiabatic Operation

Heat lost from the equilibrium flask would cause partial condensation of vapor. Such refluxing would destroy the equilibrium and deplete the amount of the recirculating phase. Heat lost from the contacting section would produce error in the enthalpy measurement. Such losses had to be brought below the allowable limits of error. The highest recirculation rate which would not produce excessive splashing from high vapor rate in the equilibrium flask was found by calculation (Appendix, pg. 97) to provide a heat flux of about 2000 cal per min and to represent a liquid flow of from 5 to 35 gm per min, depending upon the compounds chosen. Because of expected uncertainty approaching one

One disadvantage and components components to the components term produce at the condonser, to condonser, the components of deponds approach to the condition of the condition and a conditions and a conditions and a conditions and a conditions at the condition of the condition of the condition and a co

enter to an electrical de la companya del companya della companya

consists of the constant of th

persent in the measurements for enthalpy, it was proposed that heat losses not be reduced below one percent, or 20 cal per min. Both the equilibrium flask and contactor operate at up to 100 C, and a simple insulating material would produce too great a heat loss. The use of a silvered vacuum jacket would practically eliminate heat losses, but it was felt that an untried design which might require extensive remision should not be constructed in complex glassware. The construction and operation of an adiabatic wall seemed a simple expedient. In principle, an enclosure about the apparatus is brought to the same temperature as the apparatus. Since the region between apparatus and enclosure is then isothermal, no heat is transferred through the region, and the apparatus operates adiabatically. The use of good insulation between the apparatus and enclosure reduces the heat loss in case the two temperatures do not match. This method is used in an equilibrium still by Chao and Hougen (24). The principle was used for the precise determinations made by Callendar and Barnes and is applied to modern adiabatic calorimeters in which the adiabatic walls automatically follow the temporature of the calorimeter.

At the reflux condenser, since the cool water jacket lay between the hot vapor and the cool surroundings, little insulation and no adiabatic wall were necessary.

Assuming a 5 degree (C) difference between the water jacket and surroundings, and the use of three inch thick efficient

percent in the measurements for enthalpy, it was proposed tint heat lossed not be reduced below and sercont, on 80 cal nor win. Both the equilibration flesh and converted themplies will event about a ser in 10 to be an an edimento Now aville in the case of the control of the control of the bilitimal The Arrest for a little of the compared for respect the compared to Continues Street Street and instruction of good right are in got your provides a second of the first policy of the second enthaling for a light of the comment prosein to the problem of the process of the second control of the The Residence of the Control of the and the second of the second o ing and the second of the sec with the second of the second of the second of the second the state of the second of the ing the control of th (a) 1.22 (a) 1. (b) 1. (c) 1. (c) 1. (c) 1. (c) 1. (c) 1. And the second of the second o the state of the s The Source of the conference of the first present of the following the comment of the second of t NOT 30 1 10 1 1

at the refler continue, the test and all alors footest ley hebred the test of the cost suriculation.

[State legaleties and no adiabatic wall were necessary, assuming a 5 degree (0) difference between her sate factor and augmentage, and she uge of three into that this faithful and the third editions.

insulation, a heat loss of about 3 cal per min was estimated (Appendix, p. 14). This was less than 0.2% of the expected heat flux. The actual heat transfer probably amounted to only a small fraction of this value.

The condenser was stacked above the equilibrium flask as close to it as possible to make the path for the vapor as short as possible. Thus the opportunity for heat transfer with the surroundings near the region between the points of equilibrium and enthalpy measurement was reduced as much as possible while still leaving enough area for heat transfer.

insulation, a heat loss of about 3 oal per min was setimated (Appendix, p. 14). This was less than 0.2% of the capacity heat flux. The actual heat transfer probably securion to cally a small fraction of this value.

The condenser was atached above the emilibrium flack as close to it is possible to assist the test of the source of the source of the source of the site of the source of the site of the source of the source of and the source of and the source of the source of antithers and antique near the source of soullibrium and entending near that the source of t

DESCRIPTION OF APPARATUS

Recirculating System Components

In order to eliminate contamination problems, pyrex glass and Teflon were used to fabricate all equipment which might come in contact with the system under study. Since modification and flexibility were expected to be necessary, joints were used to attach the parts together. The use of Teflon tubing and ground glass ball joints and taper joints permitted ease in glassblowing, and flexibility in design and construction. However, once a successful design has been tested, it should be constructed without glass joints, thus eliminating the possibility of leaks. It was found necessary to use silicone grease to eliminate leakage in the ball joints at the rotameter and to make the large taper joints vapor-tight. The use of silicone grease is to be discouraged in apparatus for serious phase studies. Traces of the high molecular weight silicones have a pronounced effect on the equilibrium. Short sections of 0.25 inch ID Teflon tubing provided excellent flexible, leakproof sleeves with which to attach fire-polished 7 mm (0.27 in.) OD glass tubing.

The equilibrium flask (Fig. 2) had a vapor inlet thermocouple well, T2, dipping into the incoming vapor tube, and another well, T5, immersed in the equilibrium liquid.

EQUILIMIUM PLASK, MACCONTENT INDICATE OF TAIL OF CONTACTING SECTION AND CALCUIMETRIC CONDENSER ADIADATIC WALLS FIG. 2 ILT. 001, 7" HALF SCALE 10 24 24 GLASS WOOL L MM RCCE 30 MH-WOOL CONTACTING SECTION -10 11 7 10. PLYWOOD GLASS EQUILIBRIUM FLASK ROCK WOOL ALIC PUIM ALIA'S. 6 MM 30 MM 20 MM-

DESCRIPTION OF APPARATUS

Recirculation System Components

In order to eliminate contamination problems; pyrex glass and Teflon were used to fabricate all equipment which might come in contact with the system under study. Since modification and flexibility were expected to be necessary, joints were used to attach the parts together. The use of Teflon bubing and grand glass ball joints and taper joints permitted ease in Gleseblowing, and flexibility in design and construction, comever, once auccessful design has been tested, it should be constructed without. Slave juints, thus eliminating the possibility of leaks. it wha found necessary to hee allicone grease to chiminate leakage in the ball joints at the rotameter and to make the large taper joints vapor-tight. The nee of silicone grease is to be discouraged in apparatus for serious phase studies. Traces of the nigh molecular weight silicones have a pronounced effect on the equilibrium. Mort sections of 0.25 inch in Teflon tubing provided excellent flexible, leakproof sleeves with which to attach fire-polished 7 mm (0.27 in.) OD glass tubing.

The equilibrium flask (Fig. 2) had a vapor inlet thermocouple well, T2, dipping into the incoming vapor tube, and another well, T5, immersed in the equilibrium liquid.

A small bore sample tube from the bottom of the flack permitted the removal of the equilibrium liquid for analysis. The long tube length was necessary because of the thick adiabatic wall in that section. Slots were cut into the bubble cap in an attempt to break up the bubbles and cause better contact with the liquid. The flack held 530 ml before liquid overflowed into the inlet tube, and 400 ml was considered an adequate charge. The large open volume above the liquid level permitted the vapor to disengage from the liquid.

The condensate contactor fitted into the flask outlet where the vapor outlet thermocouple, T4, was located. Vapor passed through the inner tube and contacted the condensate which trickled down the side of the contactor. The adiabatic section reached to the lowest point of the condenser jacket, a point about one inch above the top of the contactor. A small thermocouple well, T7, dipped into the pool of condensate near the outlet of the collector. Five to ten small chips of broken ceramic measuring about 0.3 om in the largest dimension were placed in the bottom of the contactor to eliminate the possibility of the condensate being superheated. A side arm led the condensate through the adiabatic insulation. A thermocouple, T9, was taped against the outside of the contactor to give, along with T7, the average contactor temperature and to tell if the contactor was excessively long.

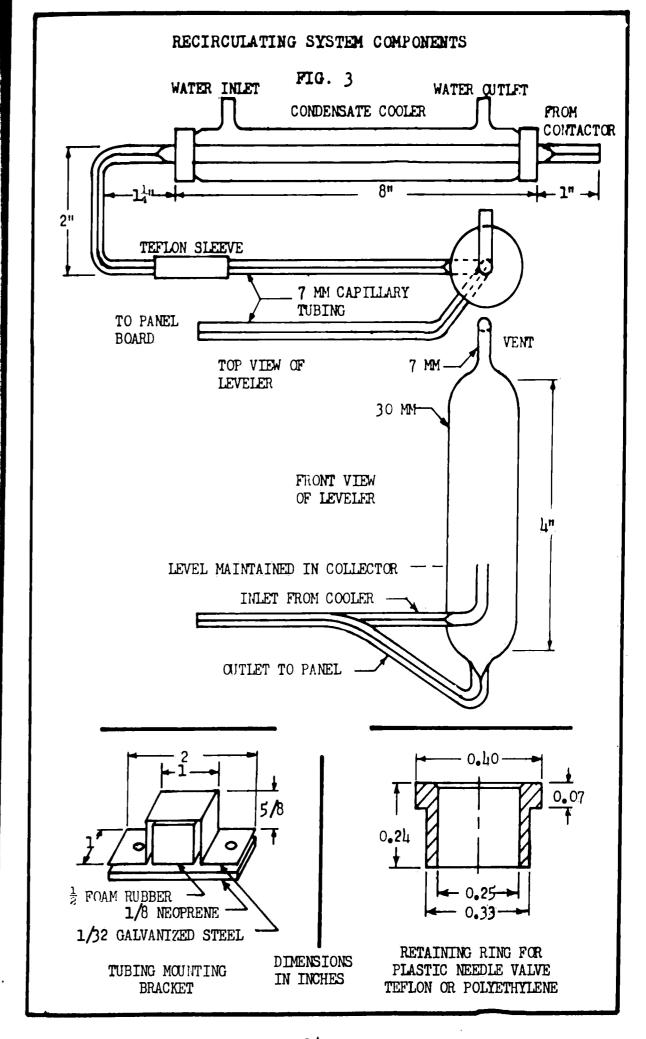
A small bord sample tube from the bottom of the flask permitted the removal of the equilibrium liquid for analysis. The long tube longth was necessary because of the thick adiabetic wall in that that necessary because out into the bubble one in an assume as and one bubbles and cause bubble contact contact the liquid. The sleek adiabeth and make action if will by white the color tenth of the liquid. The sleek adiabeth and cause actor of will be supplied to be seen to the committee of the liquid of the

SECTION OF SELECTION OF STAR OF SELECTION OF E Submood and the factor of the first and the design of the design of the second of -room mile manual for the large transfer of the decided and the court will procouding the substitute of the process of the contraction thereone is a structure of the contract of the property of the constation, and an analysis of the second second and analysis and owith induced for the colds with the community of the por and a state of the contract of the contract of the contract of we want to receive the service of the control of the control of the conanded education of the gold felleng and a salable of total superioated, .. with the life condernate dirough the adiabatic insultation. A thermodesquit, 19, was taped against the ourside of res contactor to give, along vity, $i^{\prime\prime}I_{i}$ the average contactor seapersture and to tell if the contactor was excessively long.

Heat was removed and enthalpy was measured with a standard 500 mm long West type condenser inserted into the contactor. Condensation took place inside a half inch diameter tube around which water flowed in a one inch diameter jacket.

Two clamps for large ball joints were filed oversize and used to clamp the tapered joints between the equilibrium flask and contactor, and between the contactor and condenser.

The condensate passed through a Teflon sleeve joint and into a small Liebig type water cooler (Fig. 3) which reduced the temperature to about 25 0 by the use of cooling water from the constant temperature bath. It is at this cooler that the sensible heat effect may be measured. Cooling at this point prevented a loss of volatile components out of the vent of the next element, the leveler (Fig. 3). The leveler permitted the liquid level in the condensate contactor to be measured, and controlled above a minimum. Teflon sleeves on the inlet and outlet provided flexibility for raising and lowering the position of the leveler. Thus the thermocouple well T7 was kept immersed in a pool of condensate and unexposed to warm vapor. Also vapor was prevented from entering the side arm where condensation would constitute a heat loss. By bringing the liquid level outside of the enshrouded condensate contactor, a sensitive measure of the operation of the still was brought to view. A rise or fall in this level meant that the mass of the



Heat was removed and enthalpy was meanable with a standard 300 mm long west type condenser inserted into the contactor. Condensation took place inside a half inch diameter tube around which water flowed in a one inch diameter jacket.

Two clamps for large bell joints were filed oversize and use to clamp one tapered joints between the
dand libratum flast and contactor, as between the contactor
and contactor as a

EVECTO COLIS. . . ANTEN COPPER STREET. OF SMI (8 .- 1st tab a small debte type sater cooler inte, 3) To say one to the evode of oruser that the trade of diolics water from the constant temperature beth. It is at Cas dor let that the sens 'il' seat a reat may be mensured. soulful at this ofer provinced a tork of voletile components All and your of the cast of asserts and to cook and to constannahano edu mi level bigoil and beurdan allevel ada continuous to use gared, and controlled above a mini and seffer allowers on the inlot and outlet provided fleribility for ruleing and lowering sta constiton of the leveler. Thus the thermocouple will fit her hopt impresed in a pool of condensate for unergoed to warm vapor, Also vapor sus prevented from antering the side arm where condensation would constitute a nest loss. By bringing the liquid level outside of the enshrouded sondersste contactor, a sensitive measure of the operation of the still was brought to view. A rise or fall in this level meant that the mass of the

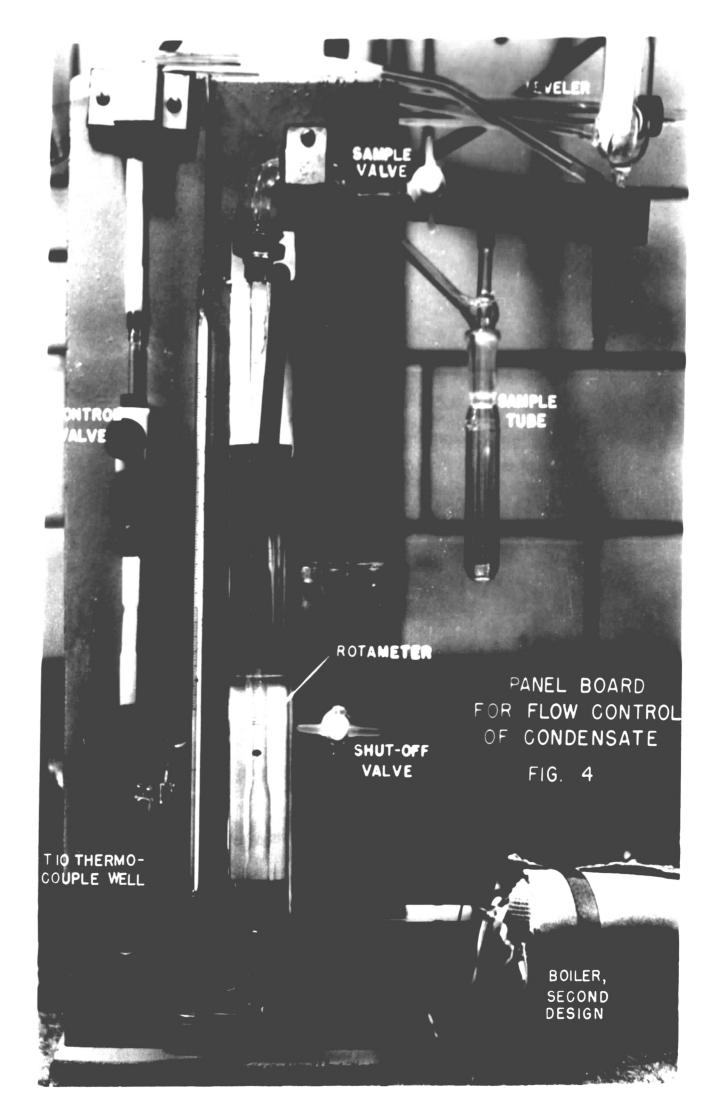
recirculating phase was changing, and that equilibrium was not established. A piece of tape on the leveler was marked in half centimeters so that levels could be recorded.

The outlet from the leveler lead to tubing mounted on a plywood panel board, Fig. 4, where the flow control valve, thermocouple well, rotameter, sample valve and shutoff valve were mounted. The rotameter produced a piping problem in that it had to be mounted vertically. To make the flow system compact, the fluid had to traverse an Sshape, passing down from the leveler, up through the rotameter, and finally down to the boiler inlet. Since the flow of the system depended upon the small hydraulic leg between the leveler and boiler, air bubbles which would stop the flow had to be eliminated at the start and prevented during a run. The use of capillary tubing, of proper placement of shutoff, sample, and control valves, and of cortain operating procedures (see p. 50) eliminated air bubbles. The use of capillary tubing reduced the volume of the recirculating phase, thereby causing a greater turn-over at a given flow rate and hastening the approach toward equilibrium. The components in the circulating system were designed in such a manner that the flow of condensate swept out all regions where liquid might lie. In this way, stagnant regions were prevented.

The Teflon and glass needed valve was manufactured by the Manostat Corp. The valve had a 1 mm bore and provided much closer control than could have been obtained

recirculating phase was charging, and there exilts being and was also well if the thin and a constitute of the constitut

ing reachers and evilor careful agents and the constant of a constant of a constant of a constant of a constant of agents and and agents agents and a constant of agents and agents and agents agents



and outlet. The slip rings originally sent were considered unsatisfactory and new ones shown in Fig. 3 were designed and constructed. Liquid still leaked past the new slip rings even after the plastic nuts were tightly screwed into the valve body. This is believed to be the result of bending forces on the inlet and outlet tubes. The use of reflen sleeves on both sides of the valve and the climination of rigid mounting for the valve eliminated leakage. It is to be noted that the new slip rings do not fit a manufacturer's design change in inlet and outlet glass tubes and that a return to the original slip rings will be necessary if the glass fittings are changed.

The thermocouple well, T10, Fig. 4, was to have measured the temperature at the rotameter so that the efficiency of the cooler could be checked and so that temperature could be duplicated during calibration of the flowmotor. Then the effect of temperature upon the indication of the flowmeter was discovered during calibrations, provision was made to jacket the flowmeter with water from the constant temperature bath. Thus the fluid inside the rotameter was brought to 25 C during operation and calibration of the flowmeter. The flowmeter was a Fisher and Porter Tri-Flat Rotameter with specifications O2F-1/8-12-5/36, pyrex tube and sapphire float. Practical range was 0.4 to 23 gm water per minute on a scale of 1 to 12. Ball joints were glassblown onto the rotameter ends to provide flexi-

Telmi add da Mael of monomou a dan Jud (1000,000 a mor)

wording a side of the state of the stat

Article of the control of the contro

meter for cleaning. A mirror was demented on the panel immediately behind the rotameter so that errors of parallax in reading the rotameter would be eliminated.

The condensate sample valve was connected to the point where the fluid turned downward toward the boiler. Bubbles collecting at this high point could be vented through the sample line to prevent the stoppage of circulation which would occur if bubbles became trapped in the lines through which flow was downward. The position provided clearance for sample tubes above the boiler. Placement of the sample line between the control valve and the boiler permitted the sample line to be used as an air intake at the time of shut-down. Without this vent, liquid from the equilibrium flask would be drawn into the boiler as vapor condensed in the boiler. This precaution was taken oy Jones, Schoenborn, and Colburn, and by Murti. A stopcook between the sample line and the boiler was used to shut off the path to the boiler when a sample of the recirculating phase was being taken. Both sample valves and the shut-off valve were Fisher and Forter Lau-Crest Stopcocks, the manufacturer's name for special stopcocks using Teflon plugs. They needed no lubrication, did not stick or leak, and were excellent for this application.

Condensed vapor samples of 10 cc were drawn by gravity or vacuum through the capillary tubing, Teflon stopcock and ground fitting into short tubes provided with

were presented to be the telester ends to the store and the there.

bility in the mounting and to sermit removal of the rotal meter for elemning. A mirror was demented on the panel function of the rote sees so that error is analyzed in read of the rote sees so that error. A analyzed in read of research of the rote is all and but there is a roce in the rote of the rote.

Section 1988 And the second section of the sectio

The same of the sa

March 1 St. Comment of the Comment o

The control of the co

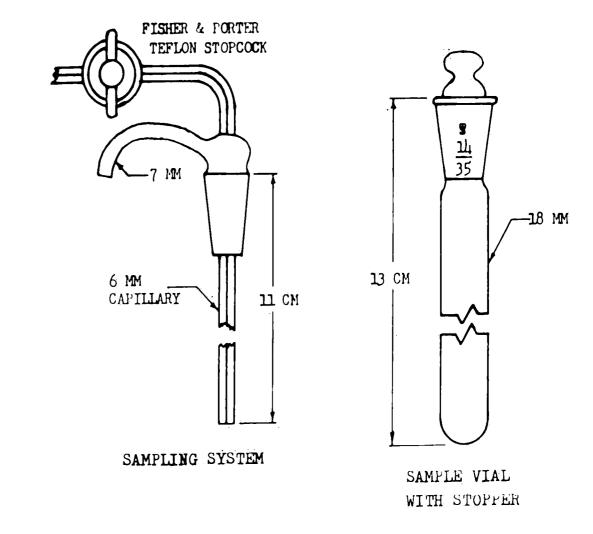
done made vacan asaptes of 10 co acre deam by

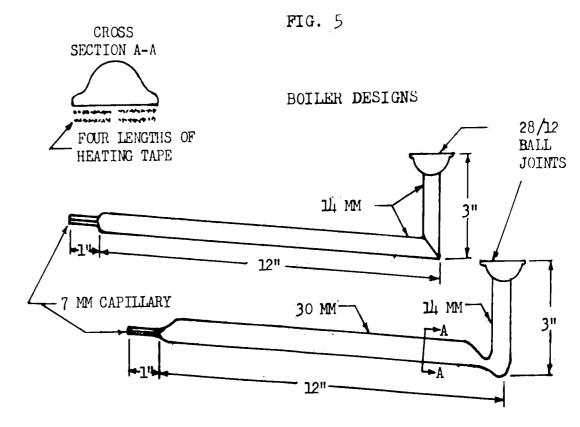
gravity or vecum through the capitlary tubing, leften atopoons and ground fitting into short moss provided with

introduced through the long capillary below the liquid level in the sample tube. Too in a beaker placed around the sample tube readily cooled the sample because of the small diameter of the sample tube. Both precautions were taken to reduce volatilization losses. A similar sampling system of capillary tubing, stopcook and ground joint was attached with a section of deficient tubing to the capillary tube of the equilibrium flash so that a sample of the equilibrium liquid could be taken.

The recirculating system held 30 ml in the region between condensate collector and boiler inlet. The liquid head which provided recirculation was a out 17 inches high.

tested. Miquic passing through a Teflon sleeve and a short 7 mm capillary tube entered the 14 mm tubing of the boiler where the condensate was vaporized as it trickled down the tube in a thin stream. An elbow at the end directed the vapor into the equilibrium flask. Seat was supplied by two four foot long heating tapes wound one on top of the other on the glass tube of the boiler. A 30 mm tube used for the second design was flattened along its length on the bottom side in an attempt to distribute the boiling liquid over a large area. The top half cylinder was insulated with asbestos, and an eight foot heating tape was laid longitudinally against the flat bottom so as to supply most of the heat to the bottom of the boiler in an attempt to eliminate





Bround joints, shown in Fig. & and B. The Bample was introduced through the long copiliary below the liquid love: in the two papers of the liquid the last the bank of the liquid the last the last the last the sample observed of the last the last

ADERTO BLUE DE LA COMPLEXA DEL COMPLEXA DE LA COMPLEXA DEL COMPLEXA DE LA COMPLEX

The control of the co

superheating of the vapor. The boilers were wrapped with asbestos and mounted in position to slope one inch in twelve.

an addition tube with side arm was inserted in the top of the condenser. The side arm provided an outlet for pressure measurement, and the addition tube and funnel and the Teflon and glass needle valve provided the means to introduce liquids for study.

ndiabatio malls

Two lengths of schedule forty eluminum sipe were used for the enclosures shown in ig. 2; a six inch diameter section around the equilibrium flask, and a four inch diameter section around the contractor. These were separated and supported by blywood circles and three wooden legs. Deep thermocouple wells were crilled into the tube wells. Glass wool was packed between the glassware and the lines. Two heating tapes, 3 ft and 4 ft long, were wound or the six and four inch pines respectively. Leavy blotting happr was used to hold rock wool insulation against the outside of the aluminum pines. This outside insulation was used to eliminate the effect of changes in ambient conditions. After an early run of the apparatus, rock wool was packed between the table and lower plywood circle, the region around the elbow at the end of the boiler. It was thought that accumulation of liquid at the end of the boiler was due to condensation in the tube running to the equilibrium

adjustice of the vapor. The outliers were arealised at the construction of the constru

.

empty of the second of the sec

flask and that insulation would prevent this. A test tube for a view port and a flashlight bulb were inserted into the insulation in order to observe the amount of liquid at the elbow.

rower Supply

Since power requirements were below 1000 watts, the 110 volt laboratory outlet was considered a satisfactory source. Voltage regulation became nucessary when it was discovered that the line voltage fluctuated, producing erratic operation of the equipment, a simple expedient requiring occasional operator attention was adopted. A single 110 volt powerstat transformer was plugged into the lab outlet, and its output voltage monitored with a vacuum-tube voltmeter. The output power was fou to three control powerstats. The voltage to the three control powerstats could then be adjusted to maintain a constant voltmeter reading. One control powerstat supplied power to two heating tapes in parallel for the boiler, and the other two supplied power to the two adiabatic walls.

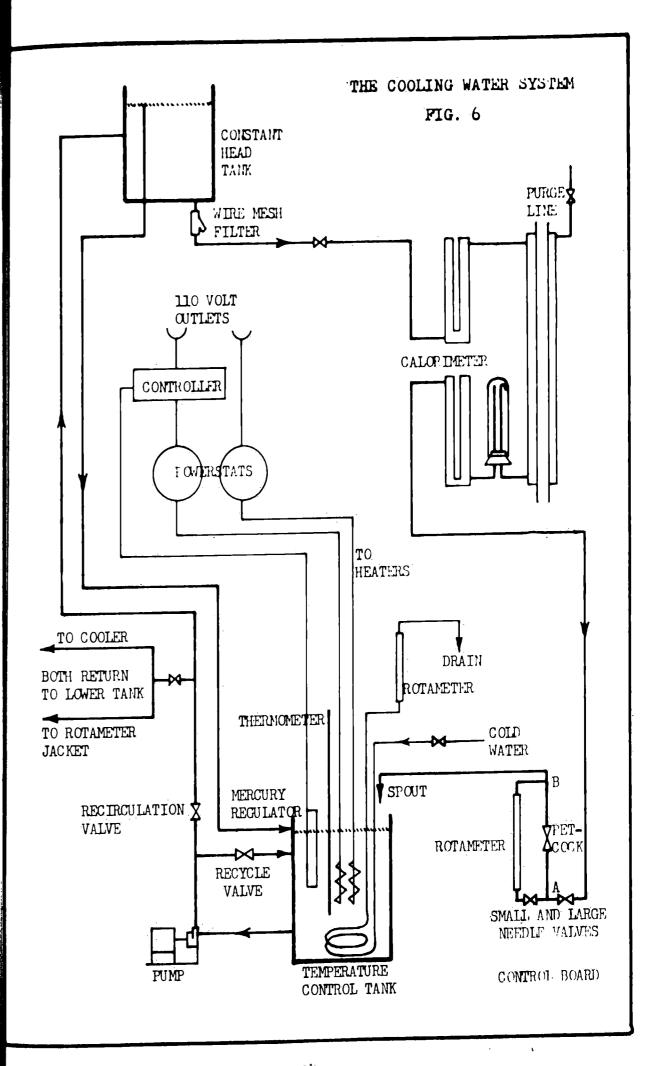
The Cooling later System

The purpose of the cooling water system was to provide water at 25 C and at a constant controlled rate to the calorimeter condenser. The system consisted of a temperature control tank with heaters and a cooling coil, a pump, valves, an auxilliary water supply, a constant head tank, the calorimeter and a control board shown in Fig. 6.

flack and that insulation would prevent this. A test tube for a view port and a flashlight buth were inserted into the from the front of the form of t

TO THE RESIDENCE OF THE PROPERTY OF THE PROPER

cor income and the control of the co



with appropriate pipe fittings and coated with "Tygon" paint in an attempt to prevent rusting. The temperature control tank was set at table height and contained two heaters, a cooling coil, and a mercury regulator. The regulator and a controller were used during operation to supply power through a powerstat to a 50 watt immersion heater. A 750 watt immersion heater, controlled with a powerstat, was used to bring the water system up to 25 C at the start of a run. Cold tap water was circulated through a cooling coil of 3 ft of 1/4 in. copper tubing. The rate of tap water was controlled by a 1/3 in, bore needle valve at the tap, and was observed on a rotameter of 20 gm per min capacity.

motor was connected to the piping system with short hose connections to reduce vibration. From the bottom of the temperature control tank, water could be pumped either in recycle to the top of the tank or to the constant head tank, The latter was supported on an angle iron frame at a height of five feet off the table. A half inch copper tube running through the bottom of the upper tank and into the lower tank served as a constant head device. Depending upon the setting of the two valves, the water could be recycled to the lower tank when a constant temperature bath was needed, or circulated through both tanks when a constant head, constant temperature water source was needed.

Por the the lander, five the It wash the trade of wit appropriate to since distinct a new design of "HOW I I I WE IT I Lypper ω is the μ sector ω . The sector ω varies of eigenstein a and San) wu; ru t o l 1.3 cincultation to the second sec

when it was discovered that a water jacket was necessary for the system rotameter, a stream was taken from a point above the recirculating valve for circulation through the jacket. This auxilliary water supply was also used for the jacket of the condensate cooler at the exit of the condensate cooler at the exit of the condensate collector. Mater from the jacket and cooler was returned to the lower tank.

Water for enthalpy measu rement was taken from the upper tank through a fine mesh filter. The water flowed through a section of neoprene tubing into the condenser calorimeter. The calorimeter consisted of two thermocouple wells, a mixing tube, and the west type reflux condenser described previously. Both wells were formed entirely from glass tubing. Each was formed of an 11 cm long 6 mm glass tube sealed at one end and jacketed with a 12 cm long 9 mm glass tube, both tubes having standard wall thickness. Inlet and outlet tubes were of 7 mm glass tubing. The mixing tube was used to reduce fluctuations in the temperature at the condenser outlet. It was constructed from a three inch long section of 30 mm glass tubing, sealed off at one end and plugged with a No. 6 two hole rubber stopper at the other end. Inlet and outlet tubes of 7 mm glass tubing, inserted through the stopper, reached to the top of the tube. The top of the inlet tube was bent over so that the incoming water could be directed downward into the tube for mixing.

An extra side arm with hose and pinch clamp was

stant to the end of the actions while the transfer

shen it was hiscovered that a water lacket was

THE JACK STATE OF THE STATE OF THE STATE AND ALL THE STATES AND ALL TH

The transmitted of the transmitt

an eater side arm with mest and place oleme was

entrapped air. Air collected there because of the decision to introduce water at the top of the condenser and to remove it at the bottom. Air bubbles would have reduced the available heat transfer area of the condenser.

around the calorimeter. These 4 x 12 x 13.5 inch blocks were hollowed out to accept the condenser, thermoscuple wells, mixing tu , and air purge line and to fit around the clamp holding the condenser and contactor together.

These components, attached with short sections of neoprene tubing, were sandwiched between the two blocks of Styrofoam. The blocks were bound and glued together. A 3/8 inch thick annulus between condenser and Styrofoam was packed with glass wool. When in place, the Styrofoam insulated the region extending from the top plywood circle to the pressure connection of the addition tube above the condenser.

a panel board where the rate was observed and controlled.

A 1/4 inch bore needle valve was used to control the rate.

The flow was divided and a side stream passed through a rotameter and small control valve to give a measure of the flow rate. Adjustment of the small control valve kept the rotameter float on scale for a wide range of flow rates through the calorimeter. A petcock on the main line provided a small pressure drop between points A and B of Fig. 6.

The flow through the rotameter was a measure of the pressure

installed at the top of the condenser jacket to remove mirropen sir, wir ocliested there because of the decision in although the top to the condenser and to remove the decision of the condenser and the available of the condenser.

BLOOKER CONTROL OF THE STATE OF

The control of the co

pressure drop is proportional to the square of the flow rate, the rotameter gave a sensitive indication of the constancy of the flow rate through the calorimeter. The rate could be controlled between 500 and 1000 gm per min.

Prom the top of the control board, a long spout returned the water to the lower tank. A compression fitting at the control board served as a swivel joint so that the spout could be swing horizontally over a 500 ml beaker for flow rate determination. Flow measurements taken in this manner prevented any upset in the flow through the calorimeter since no valves or liquid levels were disturbed. Direct mass flow determination eliminated any problems of calibration and accuracy which would be involved in the use of a rotameter.

Thermoregulator and Controller

A regulator and controller for the water system were constructed with a sensitivity of 0.01 degree (C) for use during calibration to check the potential vs. ΔT relation for the calorimeter thermocouples. The control was also used during runs of the apparatus in order to keep an isothermal bath in the jacket of the system rotameter.

Thermoregulator designs are available in profusion (6, 25, 26). Most rely upon the thermal expansion of a fluid in a bulb to force a capillary column of mercury into making or breaking contact with a fine wire inserted in the capillary tube. This switching action provides the

the they through the rotameter was a measure of the pressure

drop from A to B. Since the orifice equation shows that pressure drom is proportional to the square of the flow rate. the rotameter from a sansitive indication of the gonesianor of the flow rate that or the controller offwarm for and he gos and he gos and he gas on the same for the controller of warm for and he gas on the from the flow rate.

The respect to the control of the property of the property of the control of the

The state of the second se

TARGER TOUR AND THE PROPERTY OF A SECURE OF A SECURITY OF A SECURITY

this the souls to hope a still solution of merchant into hadred the fint into hadred of hadred of hadred into hadred in the continuous with a fine wire inserted in the capitlary bube. This switching action provides the

eignal to a controller, and the level of the fine wire determines the temperature level. The expansion coefficient of the fluid and the volume of the bulb in comparison to the capillary diameter determine the sensitivity.

chloride instead of the usual choice of toluene because of the greater expansion coefficient of carbon tetrachloride. The greater volatility of carbon tetrachloride was not considered a hindrance for room temperature operation. The bulb shown in Fig. 7 was fabricated from 1/2 inch strinless steel tubing instead or glass to avoid recidental breakage and distribution of mercury over the copper parts of the water system. A half inch stainless steel compression union was used to attach the bulb to a section of 7 mm glass capillary fit snugly into a lucite sleeve. To seat the lucite in the union, a short section of rubber tubing was used instead of the stainless steel compression sleeve.

soldered into a length of threaded brass rod. One section of one inch diameter knurled brass rod served as a turning knob, another section as a set screw. The contact screwed into an aluminum cap mounted on the lucite which also served as an insulator. The capillary tube extended above the bottom of the large hole in the lucite to form a well for excess mercury. One binding post was screwed into the aluminum cap and the other was silver soldered onto the stainless steel union. The procedure for assembling and

signal to a controller, and the level of the fine wire determines the temperature level. The expansion coefficient of some finish and but wolling of the finish and the volume of the one finished the confident to the confident of the confident o

Lunion approx לנוסו של בטוב ביו ביו בו בו בו בו סוף 36 July obdition to the first of a good one will be at all and HOOD I'M BOW O MILEDIAN CO DECIMAL TO CALL MANY A TO della maria della compania maria 1.1 The road was a sound from SAN a soul 1 ... 35% to the state of TOVERS SEE SEE SE a tropy brown, barrier of setting and page 17 to the analysis griduand to the commence of a second to the commence of Bernon Carlon C. C. Section Con Carlo Colored Co. Section C. here only noting official and no bestines our entered to of the linear core out thing the entruded above the most star derigo mode in the lucity to form a till com excens marcher, one canding post was screwed into the alicalnua our and the other was sliver soldered onto the stainless steel union. The procedure for assumbling and

THERMOREGULATOR COMPONENTS FIG. 7 SILVER NO. 22 DRILL WCITE-SULDER 3/8"3/16-32 TAP KNIRLED NO.56DRIIL BILASS 1/16-64TAI l" DIA 4311 3/4" 14^{1}_{2} n J+3 /4"> .56DRILL 1/16-64TAF 11" -> ALUMINUM CAL, 14" ROD -3/16-32 DIE 1 _STIVER SOLDER 7 MM: GLASS CAPILLARY MICHRONE 2-5/8"LONG CCMTACT **WCITE** ADJUSTABLE INSULATOR CHITACT TRON PLUG SILVER SOLDERED 12" 3 21 MERCURY LEVEL EXPANSION BULB THE STAINLESS STEEL TUBING

A variety of control circuits are available (27, 28, 29), and the design of control for constant temperature baths is analyzed qualitatively (6, 30) as well as analytically (28, 31). On-off control was deemed satisfactory. A standard Thyratron relay circuit, fig. 8, was chosen because of the low power carrying capacity of the regulator. Mercury regulators which directly switch the power to heaters are subject to erratic operation from arcing and mercury oxide formation at the contact. The relay could control 1000 watts at each control outlet, switching from one to the other as the control point was passed. A heater and cooler could both be operated. Capacitance in the circuit introduced a short time lag to reduce unnecessary switching as the control point was passed.

.omporature ..oasuroment

galvanometer were used with copper-constants; thermo-couples for all temperature measurements. The instrument, used with guarded lead and standard cells and with the elimination of stray potentials in the thermocouple circuit, was accurate to ±0.5 microvolt or 0.02 degree (C) for the onthalpy measurement, and to ±1.1 microvolt or 0.05 degree (C) for the equilibrium temperatures. The temperature tolerances are computed for a single junction.

Early difficulties over the stability of the

TO CONTROL ACTION SPDT RELAY 10-25 MA, 5000 A NEON BULB 20 TO CONTROL ACTION NEON BULB THYRATRON RELAY CIRCUIT FIG. 8 POWER POWER 110 VOLTS DPST SWITCH 8 MFD 250 V **┷**-- 8 MFTD + 25C V **E**150 V TC
MERCURY
RECULATOR 3 66.3 7 7 KV 10.1 MFD 7600 V ₹4.7 M & 2.2 M.A. RCA 2D21 AIL RESISTORS & WATT

~~

switching circuit was built as shown in Fig. 9. The copper bar and the switch, regions assumed to be isothermal, are shown surrounded by a dotted line. The junctions created inside an isothermal region yield no net potential as long as the same conducting material enters and leaves the region. Only constantan wires were connected to the copper bar, and only copper wires were attached to the switch. As long as the two regions were isothermal, no stray junction potentials could be generated in the thermocouple circuit. The compact mass of those two components and their enclosure in a small box made the fulfilment of the isothermal condition more likely. It should be pointed out that the two regions did not need to be at the same temperature, or at a constant temperature from day to day.

according to the design suggested in the operating manual for the potentiometer. The lead and the standard cells were placed in a plywood box which was lined with sheet aluminum. The negative terminal of the lead cell was attached to the aluminum and to shielding around the positive leads from both cells. It switch wired into the circuit of each cell prevented the potentials from being used except when needed. In shorting switch on the galvanometer protected the instrument against chance mechanical shock when not in use.

The potential at switch position I was a direct

₩. **(**)

杊

couples at condenser inlet and outlet, independent of the reference junction potential. Likewise, position 3 presented the potential difference between T2 and T4. rositions 2 and 4 through 11 used the reference junction. The circuits for thermocouples T8 through T11 have been omitted from the drawing.

Thermocouple junctions were formed by twisting the wire ends and soldering them together. About 6.5 cc of mercury was placed in each of the glass thermocouple wells to improve heat transfer in the vicinity of the junction. Glass backed admestive tape was used over the thermowells to prevent spillage of mercury and to keep thermocouples in place during assembly of the glassware. Thermocouples II, 2, 4, 5, and 7 were 4 3 a segage wire which barely fit into 4 mm glass tubing. Thermocouples IG, 3, 3, and 10 were 20 3 % 5 gage.

with soldered junction was immersed in a dewar flask along with ice from an ice machine.

Pressure Measurement and Control

The pressure was controlled by a volume of air connecting the top of the condenser to a water manometer.

A connection was also made to the top of the leveler. The trapped air exerted its pressure on the vapor in the condenser, the liquid in the leveler, and on the water in the

THE RESTRICT OF A STATE OF THE STATE OF THE

.

ACTION OF THE STATE OF THE STAT

instead of mercury because of the greater change in height with pressure. The pressure was controlled by introducing or removing air through a blow tube provided with a pinch clamp. The system pressure was kept at 760 mm mg using a barometric reading to determine the height of water required to increase or decrease the pressure of the atmosphere.

the vapor level in the condenser changed, a reservoir of air was provided by a one gallon bottle. A change in vapor level over the length of the condenser changed the air volume about C.1. and changed the pressure by the same amount. Therefore a change in the vapor level of the condenser, occurring as a changing demand by the system for heat exchange area, would effect neither the water level in the lanometer nor the pressure on the system. During early runs, rapid pressure fluctuations were observed at the water lanometer. To suppress these, a four inch section of drawn C mm capillary tubing was inserted in the neeprene hose. Communication between the condenser and leveler was not impeded by the restriction.

Composition Analysis

Chemical methods of analysis for ethanol-water mixtures are tedious and are accurate only at low alcohol concentrations (32). Refractive index undergoes a maximum

manomator, inter was ancient for the manomary fluid

that it of sprents on angle of no spreason change in hright

or to be seen in a spreading of a change of the problem.

or to be seen of the spreading of the spreading of the problem.

or to be seen of the spreading of the spr

- ,

.

ra Paul Rain Age de Marie Age.

dorman and the control of the state of the s

observed in the density. No Westphal balance sensitive to 0.1% in composition was available, and the pyonometer method was considered both too time consuming and likely to err from volatilization losses. Since an interferometer, which accurately and rapidly measures differences in refractive index, was available in the chemistry department, refractive index was chosen as the analytical method.

The Carl Wiss Jena Interferometer consists of optical plements and rewater bath contained in an insulated ovlinder set morizontali in an air bath controlle to within (.05 degree (C). Reasuring cells, mounted in mairs in cell holders, are equi med with cover glasses to reduce evaporation losses. Each cell has two windows which permit light from the instrument to hass through the cell to the optical section. When the holder is mounted in the water bath of the instrument, a light beam which masses through each cell and a second beam which masses through the water bath produce two interference patterns which are observed in an eyepiece. The positions of the patterns are brought into match by turning a thumbscrew linked to a mirror in the instrument. The thumbscrew is marked to give readings which are linear in the difference in refractive index between the liquids in the two cells. Sensitivity and range are directly and inversely proportional to the path length of light through each cell. A set of cell holders with cells of path lengths 0.5, 1.0, 2.0, and 4.0 cm are

at about G. a mass ifraction while no maxims of minims are observed in the constep. The test, half our and about the consteps of the following part the formation was not independent on the constant of the c

The second of th

with composition of chanol-water mixtures at compositions away from the maximum, the 0.5 cm cell which has the greatest range and least sensitivity was used. The effect of temperature upon the analysis is largely reduced because both unknown and known have about the same temperature coefficient.

48

available. Gooduse of the large charge in refractive index with commonition of cancel-water atxours at commonition was named that the large fine of carylang, the .b ca cell waich has the result of rungo and ledge a living of the carylang and ledge a living of the large and ledge a living of the large and ledge and ledge of the large and ledge a

OPERATING PROCEDURES

Program of Test Runs

The use of ethanol-water as a test system and of refractive index for analysis restricted the operation of the still to regions away from the refractive index maximum where compositions could not be accurately determined. It was felt desirable to operate in the region where there would be the greatest temperature difference between saturated vapor and liquid in the condensate collector, since runs with the pure components would enack operation in which vapor and liquid in the contactor were at the same temperature. These considerations limited operation to the range between 0 and 0.6 mass fraction ethanol, away from the ageotrope and refractive index maximum. Since the ethanol-water system was being used simply to test the operation and accuracy of the equipment, the entire composition range did not need to be covered.

Runs were first made with water to discover shortcomings requiring changes in the apparatus. After a successful run with water, the equipment was dried and then
charged and run with ethanol. Runs on mixtures were begun
by charging a single mixture to the entire still. Calculations showed that an initial charge of 480 ml at about 0.18
mass fraction would produce a vapor phase of about 0.6,

canto. Our his is a war de

المعمدة للفاريخ المرازات الإرافاتين

 $(x, \mathbf{R}) = (x + 3) \cdot (x + 2) \cdot (x + 2) \cdot (x + 3) \cdot (x$

 $\mathcal{C}(G)$. The second constant $\mathcal{C}(G)$ is the second constant $\mathcal{C}(G)$

•

Contract to the second of the

16.3

w · · · ·

*

the state of the s

Harrison to Control of the Control o

Hodova in the control of the control

THE LOUIS HAR STORY STORY SERVICES

agents of **menon** to an architecture to the construction of the co

on the contract of the contrac

of the actions of the property of the property of the about 0.11

.8.0 thode in sand, roger a couport bleer notioer? Teat.

Water added after each run to make up for sampling losses was used to change the point of equilibrium of the next run.

The distilled water tap in the laboratory was used as the source of water. Absolute ethanol of U. S. P. grade supplied by United States Industries was used without further purification.

Start-Up

The addition funnel was filled with the mixture to be tested, and the addition valve was opened to allow a trickle of liquid to flow into the condensate collector. The U-band between the cooler and leveler was temporarily elevated about one inch to prevent bubble accumulation in the cooler as liquid flowed from the collector to leveler. The control valve was used to keep the liquid level in the leveler above the outlet to prevent bubbles from entering the lines to the panel board. The shutoff valve was closed as liquid began to enter the boiler. A sample tube was used to collect a sample from the sample line, and lung pressure was used to reverse the flow, forcing the sample back into the lines. Subbles which had formed at the Teflon sleeves and in the thermocouple well could thus be forced back into the leveler to be eliminated. With all but the smallest bubbles eliminated, the control valve and sample valve were shut. The remaining 400 ml of the charge were rapidly added and quickly filled the condensate

the upper limit in mass fraction for accurate analysis.

Water added after each run to make up for sampling losses was used to charge the point of equilibrium of the next run.

Intentiable ten rater cap in the exponsions are new as the number of cuter, theoless stanked it. rade to place at a sure used at a light of form the column of the colu

11-31294

server to be a truly to the town of the truly and the first of the control of the The state of the s TOTALL TO THE STATE OF THE STATE OF A CONTRACT LAND WAS A PROPERTY OF The way is to the first of the contract of the Control of the state of the sta HART OF THE CONTRACT OF THE STATE OF THE STA Programme to the second add to the sound of notice of the decree the second of the second secon the material most as the case, on the material series to portable of the a form confirm on the contraction of the ecision rate for its the tharpoone were near acut. Sect. so foreign has then the Levelor to be attached and said before of ou, the smallest bubbles eliminated, the control valve and small volve for shut, the remaining 400 ml of the onarge were rapidly idded and quickly rilled the condensate

brium flask. The stopcock in the equilibrium liquid sample line was kept shut. The addition valve was shut after the charge was completely added.

To warm up the equipment the powerstats were turned on to heat the two adiabatic walls and insulation. The voltage to the two heaters was kept below forty voltate to prevent overheating.

It was necessary to have the switch to the lead cell turned on for about half an hour before the potentiometer standardization became stable. The lead cell required that time to reach a nearly constant potential. At no time was the switch to the standard cell left turned on for a time longer than that required to make a standardization.

The cooling water system was also brought into operation at this time. The cooling water system was filled with distilled water to eliminate any uncertainty as to the exact specific heat, and to avoid any tendency to foul the water system lines. The pump was started, and the valves set to recirculate water between the two tanks and through the calorimeter, cooler, and rotameter jacket. Either the auxiliary heater or the cooling coil was used to bring the water temperature to within a few tenths of a degree of 25 C. The controller was turned on and the heating circuit set to deliver full power to the 50 watt heater during the heating part of the control cycle. The adjustment screw on the regulator was raised or lowered to bring

collector, then ran down the vapor tube into the equiliprium flack. The stopcock in the noullibrium liquid each to the stute. The addition we are not singular to the course was kent shut. The addition we are no singular the course was not in a codect.

the control of the co

about control at 25,00 C. The loose nut on the adjustment sorew was used as a looknut against the aluminum cap to hold the screw in place at the set point. The auxiliary heater was used at about five volts to supply heat under conditions of low ambient temperatures.

The boiler heater was to be turned on and recirculation started when the flask liquid became heated to its boiling point. But the liquid in the equilibrium flask became heated only by overheating the lower aluminum tube for a few hours. To reduce the warm-up time, recirculation was begun before the flask liquid was completely heated.

The powerstat serving the heaters on the boiler was set at 40 volts for a few minutes to bring the boiler up to temperature. During the start of an early tun. 120 volts were applied with the result that the heater became red hot and the glass insulation became baked and embrittled. mediroulation was started by opening the shut-off valve to the boiler and regulating the flow rate with the needle valve. The recirculating phase vaporized in the boiler and condensed in the flask, thereby heating the liquid. The depletion of the recirculating phase was made up by drawing a full sample of the flask liquid and introducing it into the recirculating phase at the other sampling point. This was repeated a sufficient number of times to heat the flask liquid to its bubble point. At this point the vapor stopped condensing and began to travel the route through the calorimeter and leveler. Thus self-sustained recirculation was

about contained at 25,00 C. The loose nut on the adjustments acrew who used as a lookenut against to aluminum can to build our area of the surface of the su

- girkninger see in the first to the second second second see and a the straight of the state of th Can the second of the second o many regions of the second of The state of the s April 1980 Communication Communication may be something the The property of the property of the second The state of the s on the transfer of the second THE THE PARTY OF T . The stand of the stand of the standard of th The same of the sa softed the fitting production of the second second of the eld sonice quillende motor de de grene en allema en el Menily and that of senit is night a lostening to be before the

structed to 100 and tage, other to the report the calori-

moser and leveley, thus self-sustained recirculation was

brought about quickly despite the lack of direct heating at the flask.

Steady State Operation

many pairs of recirculation rates and heating rates at the boiler would yield steady state conditions. The choice to fix one rate and adjust the other to it had to be made. Operation at a fixed flow rate was chosen because the rotameter could not be read accurately at points between the etc. marks on the rotameter tube. The rate was maintained by adjustment of the lefton and glass needle valve.

ately held constant, and was used to gage the approach to steady operation. A change in the level would indicate an exchange of mass between the two phases and would also mean that the flow rate through the condenser was not the same as that through the rotameter. Thus neither equilibrium nor enthalpy measurem nts would be valid during such time.

ondensation within the equilibrium flask and/or accumulation of liquid at the end of the boiler. The two possibilities could be distinguished by looking at the dow of the boiler. Accumulation then could be corrected by temporarily increasing the power to the boiler. If there were no accumulation at the boiler, condensation at the equilibrium

brought about quickly despite the lack of direct heating at the flask.

tong . . tate vyere lon

Alles the Englan method of the condition of the particle of the conditions.

But I so the above the condition of the condition of the particle of the condition of the particle of the condition of the condition

Some first the second s

described the state of the collection of the state of the

flack could be the only alternative. It was thought that such condensation could only be caused by heat transfer to the surroundings and could be climinated by the proper adjustment of the adiabatic wall temperature.

A rise in level at the leveler would be due to evaporation of the flask liquid by either a superheated vapor leaving the boiler or improper adjustment of the adiabatic wall temperature. The cause could be determined by a comparison of the temperatures at the flask inlet, flask liquid and lower adiabatic wall. Adjustment of the heat to the boiler or adiabatic wall was to have corrected the problem.

It may be seen that indications of improper operation were available and that proper remedial action could be taken to correct any anticipated improper operation.

towards steady values, attention was turned toward the operation of the calorimeter and cooling system. It became necessary to turn off the auxiliary heater in the lower water tank and use the cooling coils to remove the heat picked up by the water in the calorimeter and cooler. The cooling water flow rate was decreased from its maximum until the potential difference Tl rose to a value within the calibrated range. The valve in the branch line to the rotameter on the panel board was adjusted to give a mid-range rotameter reading. The remainder of the operation consisted of adjusting the water temperature regulator, the system

Thek could be the only alternative. It was thought that such condensation could only be samed by heat transfer to the executings and could be eliminated by the proper time executings and could be eliminated by the proper time entry of the attention of the entry of

Description of nice to recommend to the recommendation of the reco

The country of the profit of the country of the cou

A construction of the construction construction.

flow rate valve, the powerstat settings, the supply voltage and the pressure; and of occasional checks of the barometric reading.

Data Collection

Data from huns 3 and 9 are shown in Table II and Table III, Appendix, p. 82 and 83. The time was recorded at the beginning and end of each set of data, after which new powerstat values were set and recorded. Changes in the two rotameter values, water temperature, and pressure were made as each value was read, and the new value was recorded. Standardization of the potentiometer was carried out before each set of potentials were read. The thermocouple potentials were read to the nearest J.Ol millivolt to save time at the beginning of a run, and all thermocounles were read to the nearest microvolt or C.1 microvolt as a steady state was approached and the final readings were being made. About six minutes were required to make a standardization and to read the ten potentials to 0.1 microvolt. The thermocouple readings were converted from potentials to temperatures. A section to the right of each table briefly indicates the observations made about operating conditions each time data were recorded.

When it was determined that a steady state had been reached, a complete set of data was taken without making changes of any variable. The calorimeter water rate was then determined by swivelling the spout from its place

flow rate walve, the powerstat settings, the supply voltage and the pressure; and of occusional checks of the barometric resultary.

And the state of

tara da de la companya de la company

المنظم المنظم والمنظم المنظم المن المنظم المنظ

•

•

• 1

Section 1988 And the section of the se

 $e^{-i E^{E_{i}}}$

La la servició de la como de la c

THE ROLLING AS COMPLETE BOT OLUM AND THE BELLEVIED FOR THE SERVICE AND THE SERVICE AND THE FOR FIRE OF THE SERVICE AND THE PROPERTY OF THE PRO

from half to one minute as measured by a stopwatch. The rotameter on the condenser water panel board, and the potential Tl were watched to certify their invariance throughout the determination. At the end of the time period, the spout was swivelled back over the lower tank, and the water was weighed. The measurement was repeated immediately.

Shutdown

The stopcock before the boiler and the three powerstats were shut off, and the system was opened to the atmosphere at the clow tube in the pressure system. Clean, dry, labeled sample tubes set in an ice bath were used to take samples of condensate and equilibrium liquid. The samle tubes were closed with ground stoppers and set aside in the ice bath for a few minutes before analyses were made. The Teflon needle valve was shut to prevent recirculation, and the condensate and shutoff stopcocks were opened to allow air into the boiler. This procedure was followed to prevent liquid from being drawn into the boiler from the flask. A half hour after the end of the run, the water circulating system was shut down. Flow through the calorimeter, condensate cooler, and rotameter jacket was shut off: the two valves at the pump outlet were reset to recycle the water to the lower tank, and the pump was shut off to end the run.

over the lower tank to a point over a 500 ml beaker for from half so one aimste as measured by a stopwatch, but so beamtor of the concensor water panel board, and the other time of the contiffer the framework of the contiffer the framework of the contiffer the framework of the contiffer the continuence of the contifer the continuence of the continuence

1 ...

Notice that

the state of the state of the state of many the second of the second the many that the party of the second of the second of the second of But the Company of the Committee of the AND THE RESERVE OF THE PARTY OF THE PARTY OF THE SECOND The contract to the first of the contract of t I will be supported to the control of the control o the first of the court of the first of the court of the contract of the court of th or complified the complete and complete and controlled the complete and controlled the controlle and ready may now and at history with with gather are given one for the controlled and the second and the Mudeming Langer of the commence of the commenc conjective and et, am tourness color and annihilative the and valves at a pump outlet were read to recycle the water to the Leave tank, are the sum wer sint off to end thu min

Measurements

The experimental latent heat values given in Table I, Fig. 10, fall within 5% of the reference values and show no systematic error. This large error may be explained by the failure of the apparatus to reach a steady state in the liquid level of the leveler and as a result of a surging condition in the condenser. Both conditions are discussed more fully under "CONCLUSIONS AND A COMMENDA-TIONS."

nent kuns 5, 6, and 7 was taken from Forry (33). The reference enthalpy data for mixtures was found in graphical form and in only two references, Bosnjakovic (34), and Smith, hong, Brown, and thite (35). The latter chart was used in the textbook by Brown, et. al. The method of calculation used by Bosnjakovic is not known, but Smith, et. al., used a variety of references on heats of mixing, A Hy of the pure components, boiling curves, equilibria, and Qp values. The two sources are compared numerically in Table I, the values having been interpolated from the graphs with the aid of a vernier caliper. The literature values are those given at the vapor composition determined in the present work. It is of interest to note a disagree-

ر.

CONTROL OF THE POSITION OF TWEET OF THE POSITION OF THE

at the state of the state of the

LO STREET DE

But Lapon

sec again body Lerseand and Bedver son ev

ह्मार्थित विकास अस्त्री स्थान निकास कर्णा विकास

E State of the Contract

e John

TABLE I

LATENT HEAT MEASUREMENTS

THE ETHANOL - WATER SYSTEM

AT 1 ATM.

FIG. 10

REFERENCE	BOSN- JAKOVIC	BRO	MN.	THIS	BOSN- JAKOVIC	BROWN	THIS
VAPOR PHASE COMPOSITION, MASS FRACTIO	N 0.597	0.59	97	0.597	0.572	0.572	0.572
SATURATED VAPOR TEMPERATURE, C	89.4	89.	. 4	90.4	90.7	90.2	91.1
SATURATED LIQUID TEMPERATURE, C	79.9	81.	• 0	83.4	80.2	81.3	81.9
SATURATED VAPOR ENTHALPY, CAL PER GM	408.8	410,	.8		418.7	419.1	
SATURATED LIQUID ENTHALPY, CAL PER GM	64.8	64.	.8		65.8	65.8	
LATENT HEAT, H _V , CAL PER GM	3144.0	346.	.0	33 0	352.9	353.3	360
			THIS WORK		REFERENCE	(33)	
LATENT HEAT, CAL PER GM, WATER, RUN 5 LATENT HEAT, CAL PER GM, ETHANOL, RUN 6			525 204		20 կ		

213

204

LATENT HEAT, CAL PER GM, ETHANOL, RUN 7

58

ment of 0.5% in the enthalpy values of the two references, appearing in the value of enthalpy of the saturated vapor.

No enthalpy measurements were made in Run 10. The method of calculation used in the present report is given in the Appendix, p. 100.

Fig. 11 shows the fairly large disagreement among the literature sources concerning the temperature-composition diagram. There are a large number of sources which list phase equilibrium concentrations, but only a few include the equilibrium temperatures. The few additional points from other sources not included here fall within the same gunshot patterns. The boiling point data of Noyes and Warfel (20), the oldest reference, and the equilibria of Otsuki and Williams (36), the most recent data, are the most complete and consistent sets in the desired range. The two form liquid lines which fall within 0.005 mole fraction of one another. Values for Run 8 and Run 9 fall within this narrow band, while the liquid composition for hun 10 falls somewhat lower. The vapor curve is much less well defined. The data of Otsuki and Williams form a set which falls from 0.02 to 0.04 mole fraction below the data of Carey and Lewis (37) and that of Jones, Schoenborn, and Colburn. The vapor compositions of the present study show considerable enrichment over those of any study except that of Evans (38). Other experimenters conclude that considerable reflux within Evans' apparatus caused the high values. It was concluded that internal reflux was the cause of the

eorginde vist considerable NOTATED TO SECTION TO SELECTION dudt digeoxe Toude 4 breacts affile abon Clear Treament caons J. TOI I'a'ı E True ent lo esuso ent saw xullon laurethl tant bebulonco saw JRT ebulo.. 9113 . 3 The Section 1. 3 the night values. o o no necesti 91CK ٢ A L MO'S BELLA LL! いるいとならい sai woller 500 52 som $\Gamma(i, b)$ 57.0 101dt The office 1. 1. Coole 16 c E EUE' BOLLCBD notur ast OUT. C . 1 1.67,61. •, ¥ Fort <u>:</u> τ, ين 3. . 6 -Seasso auteracce tenevo c C Ç entre itele CARDIT LIGHT 1.4.E. LIOLULAN Ö ; C 1:11. ť. .7 J. C. W. 133 77 1. TO 1.18 ્રં, ÷ 4.70 rellux willer 11.12 Sec. OUTSTON 61

in the enthalpy values of the two references,

0.0

To Jose

. Toger beservisc

eda

70

Adjusting to any and ut sutneeds

badter

00T

igun 10.

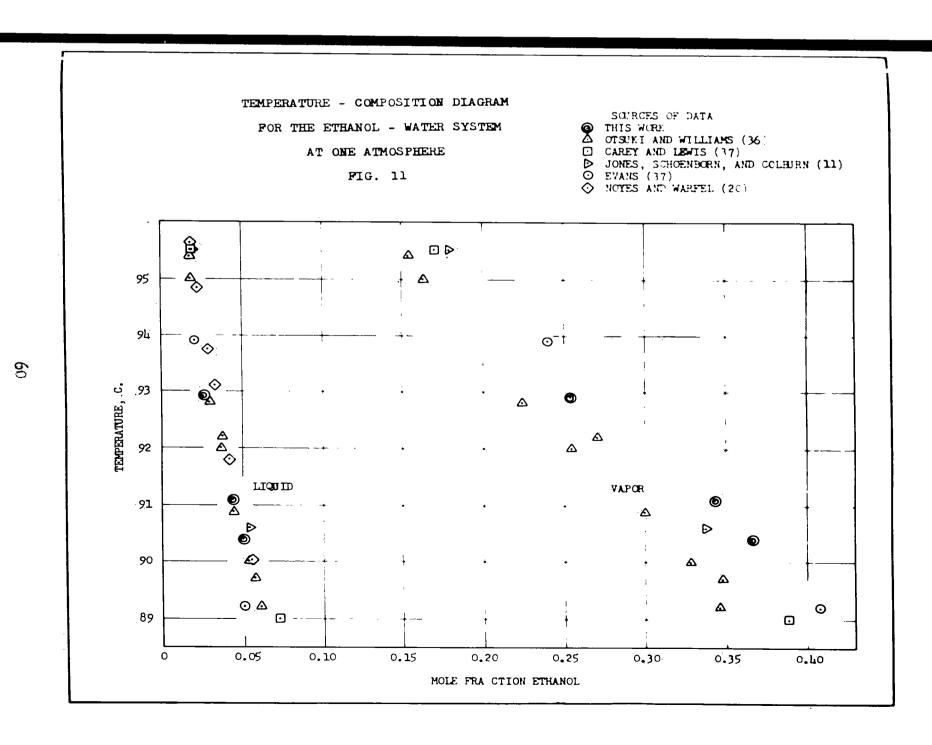
at

meanteneur a mete mage

cronding.

0.7

edu ni movin al drower innaore edu ni bear nelualizates



high vapor compositions found in the present study. Such reflux, causing the return of condensed vapor to the equilibrium flask would not have much effect on the liquid composition because of the large amount of liquid present. The source of reflux is considered in the next section.

The saturated liquid line in the composition range of the recirculating phase has been plotted in Fig. 12. The reference data form a curve definite to 0.01 mole fraction or 0.1 degree (C). The temperature T7 at the outlet of the condensate collector is the temperature plotted for the points of the present study. The temperatures appear to be too high by about 2.2 degrees (C) and 0.5 degree (C) for hun 8 and hun 9 respectively. It appears that the liquid was superheated as it left the contactor, but that the amount of superheat was substantially decreased in hun 9. The reasons are treated in the section on mechanical performance.

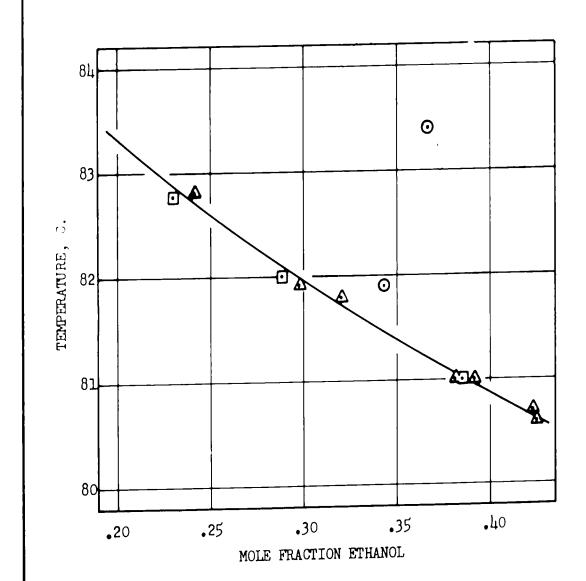
Difficulties of Operation

The operating variables in Run 8 differed significantly in two ways from the conditions set out as necessary for steady operation. They were the amount of superheat permitted in the vapor leaving the boiler and the temperature difference across the lower adiabatic wall. These have the effect of tending to increase the mass of the recirculating phase. The steady operation at the end of Run 8 indicates that these conditions were necessary to offset

TEMPERATURE - LIQUID COMPOSITION DIAGRAM

FOR THE ETHANOL - WATER SYSTEM AT 1 ATM.

FIG. 12



DATA SOURCES

O THIS WORK

☐ JONES, SCHOENBORN, AND COLBURN (11)

△ OTSUKI AND WILLIAMS (36)

high vapor compositions found in the present study. Such reflux, ocusieg the return of condensed vapor to the smill dim Clash would now have and, effect on the liquid don doly on 1 conus of the latter and in 11 and the cont. anoldens from all all memoritanes at authors of course car o menilida, et and al ori o collegio bira bel

1. 1. 1. 1. But also were and read of the standard of the plus for the transfer of the state of ACTUAL OF STATE OF ST Saginal environment of the constant of Company of the Company of the Company

042.00

-india standard on the open standard general of the two beautiful to the contract of Table to the contract of the contract of the -Big and a side of the flow of the side of the avr i sector. Illim officed in mower and the sector of the mos -worthour and he cann adv asserbed on animal in realite and lating chase. The atealy operation at the end of the indicates thet there conditions were necessary to offset

some condition which tended to reduce the mass of the recirculating phase. Operation during Run 9 with low superheat resulted in a steady depletion of the recirculating phase as shown by the dropping level in the leveler. The material could only have condensed inside the equilibrium flask or escaped from the apparatus. The latter seems unlikely. Calculations (Appendix, p. 101) show that the observed depletion was due to heat transfer at the vapor riser of the condensate collector. The lower temperature of the condensed binary vapor caused condensation of the vapor entering the contactor, and the condensate flowed back into the flask. Additional computations (Appendix, p. 107) on the steady operation of Run 8 show that the superheat of the incoming vapor and the heat supplied by the hot lower wall equate roughly with the internal condensation losses calculated for Run 9. The mechanism of depletion by condensation and return to the flask liquid is further confirmed by the high vapor compositions shown in Fig. 11.

operation was a cyclic fluctuation in the vapor flow rate through the equilibrium flask and calcrimeter. During Run 8, an uneven bubbling rate was heard coming from within the equilibrium flask. A surge of bubbles, lasting for about one second, could be heard at about 16 sec intervals. The surge of vapor condensing in the calcrimeter undoubtedly accounted for the large cyclic fluctuations in the temperature difference across the calcrimeter and in the pressure

seme condition which conded to reduce the mass of the red fattag phase. Openies during hun a with low superand control in a standy contestion of the catroulating than a main well and the property of the contraction of the and the little persons and the ways are a second of the condimineral results of the second CODE VICE TO STATE OF THE STATE TRUE CONTROL OF THE C Miller Committee of the Committee of the powofft state of The second of th -months of the control of the second Out stad to the first of the state of active to be a second of the second of the second of A STATE OF THE STA

The state of value of the state of the state

at the water manometer. There was no apparent fluctuation in the flow rate at the system rotameter or at the water rotameter. The restriction in the tube connecting the condenser with the pressure system reduced the fluctuations at the manometer. The mixing tank at the water outlet of the condenser substantially reduced the temperature fluctuation. In Run 6 the temperature varied between 3.6 and 5.4 degrees (0), but in Run 7 after the mixing tank had been inserted, the fluctuation was reduced and a reading of 5.90 ± 0.05 degrees (C) was obtained. The surging was more noticeable during the two component runs and during the early parts of all runs. At the end of Run 8, the temperature was 6.0 = 0.3 degrees (C), while the final value for hun 9 was 6.55 ± 0.07 degrees (C). The most plausible explanation for the surging is that vapor built up pressure in the boiler to overcome the liquid head in the equilibrium flask. Once the liquid head was overcome, vapor continued to bubble through the flask until the pressure in the boiler returned to some low value at which time bubbling practically stopped. The surging seemed to decrease as operation progressed toward a steady state. A comparison of the fluctuations in the value of ΔT in Runs 8 and 9 indicates that the amount of surging decreased when less superheating was present.

Performance of Equipment

The temperature measured at the liquid outlet of

at the water manometer. There was no apparent fluctuation in the flow rate at the system retameter or at the water rotameter. The restriction in the tube connecting the condender with the present system request the fluctuations at the maneron. The utains tenk at the water outlet of the someoneer entertaintent not aced the temperature fluctuatage, it and G and seconds to varied between 3,8 and 5.4 mood bad wash garining out to the missing fits research the matheur to the senator car nothern di wir incommit C. C. A. C. Corpogn of the analytical and. The surging res gairub bas a. w. there wie was a saire and each Sair of take in London South Common Common States Lanti old older (()) seen of the # 10.0 cm members will ರಾವರ ಕಟ್ಟ್ (೮) ಕಾಲಭಾರವ ಕ್ಷಮ್ ಕ್ಷಮ್ ನೀಡುವ ಅಂದು ರಾಜ್ಯಗಳ Aligned rooms would not be a work or a construction of thems of Bood blockfoods of our ready a from all of estamose * Supplementa for a serial particular to the serial series of the series of end filtrar trolly oda depend of the od hogether wegar Lough the outlaw holf of our of form were to the order. The results of lamene and and a second of the second and administration ្នុងវិស្ស៊ីស ឬវិសាធន៍ នេះ ១៩១ ខេត្ត ១០១៩ ២ ១៩ ១៦៤ ១៩៤១៩១៩ ១០១៤ ១៩, ១៩១៩ observables to the AT object the Mark the of the off Δ T in was 8 and 9 indicates to the amount of ourging decreased when less surectioneing var teacht.

Performence of Louisians

The temperature measured at the liquid outlet of

the condensate collector was identical with the temperature in the equilibrium flask during runs on the pure components. It may be concluded that sufficient contacting area was provided over which the reflux from the condenser could be heated by the vapor. The superheating of the condensed liquid indicated in Fig. 12 for the binary runs may have been caused by the heat transfer through the vapor riser which caused refluxing. Ceramic chips introduced into the condensate contactor between Runs 8 and 9 may have depressed the tendency toward the superheating by providing sources for nucleation, thereby causing the closer agreement between the temperature in Run 9 and the reference values. The apparent superheating may be partly due to the position of the thermowell T7. The well extended through the path of the hot vapor entering from the equilibrium flask and may have been heated sufficiently to give an erroneous temperature reading.

The temperature near the top of the contactor,
T9, showed that the temperature of the contactor wall and
probably that of the liquid inside were below the saturation values. Thus the distance from the condenser and T9,
about two inches, did not supply sufficient contacting area
between liquid and vapor, and the rest of the contacting
area was needed to insure the elimination of subcooling
from the condensate. The difference between the two temperatures T7 and T9 (Appendix, Tables II and III) indicates
the impossibility of maintaining truly adiabatic conditions

the condensate collector was identical with the temperature in the equilibrium flesh during runs on the pure components. Baw some antibusince and tell numbers bebulence of year it analytical avan which the rather from the condensor could be houted by the vacor. The merenalmy of the condonaed birnia bunders will be the tell by the burger pare and menia goerv , is it comes to the and dam lead to be measure come witer called a figure at a committee and a strockned into the r**eagnigmb ov**id tim C hall to the products are well as denominagovernor vettelnostr - as less, a little to a top i it will be recorded transcer of the entry of the contract of the entry of the contract of SATE OF MENT SHOOM AND THE RESERVE OF A SECOND STATE OF A SECOND S to moddings and a company of the contract of the contract of To direct one discount to be with the sold of the form of the contract of the THE BLE MEDIC HOLD STORY STORY OF THE PROPERTY OF THE HANDON TO TO THE TOTAL OF THE STATE OF THE S 1.300

The street of the street of the street of the contrology of and reconcept of the street of the control of the street of the subcooling street of the control of the the two desired from the control of the street of the two two temperatures from the control of the street of the the street of the s

in the contacting section with the use of the present adiabatic wall.

match the temperature of the lower adiabatic wall with that of the equilibrium flask. The principle problem was the sensitivity of the aluminum wall temperature to small changes in the settings of the controlling powerstats. It became necessary to estimate and make one-tenth of a volt changes in the powerstat settings to reverse drifts of 0.1 degree per 10 min. The large number of other chores requiring the operator's attention, the time lag in heating and cooling the adiabatic walls, and the fact that no steady state was truly reached, caused the temperatures of the lower wall to differ from the flask temperature by a degree most of the time.

Movement of the position of the system rotameter float was due only to changes in liquid level in the leveler and to composition changes, although fluctuation was noted whenever there was enough liquid at the end of the boiler to cause bubbling. The change during the time interval between successive recordings of data rarely exceeded a few tenths of a millimeter. The Teflon needle valve proved to be a little coarse for the desired degree of control required. A stick-slip phenomenon at the valve stem caused small jumps in the rotameter reading, first to one side then to the other of the desired set point, as adjustment

in the contacting section with the use of the present adiabatte wall.

Considerable difficulty accompanied efforts to match the temperature of the level and the state that the could the temperature of the original flact. The principle problem was the campile view of the children walk to constant to small charges to small charges to the advance of the capetains and cooling the capetains of the capetains and cooling the capetains of the capetains and cooling the capetains of the capetain and cooling the capetains of the capetains of the capetains of the capetain and cooling the capetains of the capetains of the capetain of t

Clear course of the court of the system retention of levelor court court of the levelor of the court of the levelor of the court of court of court of the court of court of the court of th

was attempted. Because of this, it was difficult to keep the float at the desired position.

Difficulty was encountered in the attempt to vaporize totally the recirculating phase without superheating the vapor and without accumulating much liquid at the elbow. The temperature at the entrance to the equilibrium flask was found to fluctuate over a few tenths of a degree within a minute and to vary widely over a half hour period depending upon the amount of liquid at the end of the boiler, and the amount of heat supplied. The presence of liquid at the elbow cannot be considered proof that the vapor entering the equilibrium flask is at saturation. For example, at the 10,52 data entry during Run 8 (Appendix, Table II), superheating of 28 degrees (C) was noted at a time when the amount of liquid at the elbow was increasing. Run 9 was a deliberate attempt to reduce the amount of superheat despite the effect of such operation upon the attainment of a steady operation. Superheat was kept below 5 degrees (0), and uncontrollable increases of liquid at the elbow were not observed. This shows that the revised boiler design with wide area for boiling the liquid and with little or no contact between vapor and heated walls appears to have permitted a reduction in the amount of superheat not possible with the helically heated tubular boiler. But it should be mentioned that the flow rate could not be substantially increased without a return of the difficulties of superheating and incomplete vaporization. Difficulty was

was attempted. Because of this, it was difficult to heep the float at the desired position.

Difficulty was encountered in the attempt to vaporize totally the read rouletime cheer without superheatend the vener and withings recomminated much liquid at the olbow. The temperature of the district to the conflibrium land was found to fluctuate ever a few tentile of a degree bolter good thad a sexpo whether year od but about a rifet is undian room the amount of lings of the this the boiler. and the amount of heat supplies. The sessence of Hould at Tree titles and an action of the section of the tree trees on the trees ton the semiliteries fiest to st seriositors, for exemple, t ves 10:58 date onerw during Aug 8 (Acceptive Pable II). augerbostine of 0) degrees (0) were noted as a time whom the a sus C mail . antenered the woods out to the the dagens edigeob deedroome to the room edd sorber on dimental etch id in and activate of a control on the control of the atomical of a steady Superations, Surgeries was been been been of the end (C), and incommend to the second of the thing of the second of the compression is a minura that the movined botter design with Addo area for colling the light west with litetle or no con--wer evan of sinesch blick horned bee totov diemited ford eldistog for dandreaus to formers out at nother to bottle with the holloully heated tubular bollor, but it abould be mentioned that the flow rate could not be autentially ingregated without a reterm of the difficulties of superheating and incomplete vaporisation. Difficulty was

encountered with the lack of sensitivity of the powerstat used to supply power to the heater around the boiler.

lines at the start of a run disappeared after about an hour of operation by disolving in the recirculating fluid. This demonstrates the performance of the reflux design of the condenser in eliminating disolved gases from the recirculating phase. The subcooler operated effectively in reducing the temperature of the recirculating phase to room temperature and reduced the likelyhood of evaporation from the liquid level in the leveler.

About a half hour after the end of a run, liquid from the equilibrium flask filled the boiler despite the precautions taken to open the boiler to the atmosphere.

Since this did not effect the sampling or start-up, the situation was ignored.

The impossibility of draining the boiler was first noticed when the still was drained of water so that pure ethanol could be tested. The still was finally dried by attaching a vacuum pump with a trap to the top of the leveler. Both sampling valves and the top of the condenser were opened to the air and heat was supplied to the boiler. About half an hour was required for drying.

Ethanol vapor could be smelled at the blow tube of the pressure system when pressure changes were being made, indicating that ethanol was escaping from the condenser.

ensoundered with the last of venetaty of the powersum.

used to supply power to the heater around the botter.

subbles which were trapped in the recirculating

lines of the atent of a real Consequence after about on hour of operation by disolving in the restroudanting finite. This demonstrates the performance of the reflux demin of the confenser in whimingting disolved on an iron the restroit lating passe. The enhancing operated affectively in reducing the temperature of the restroitating chase to reducing the temperature of the restroitating chase to real temperature and reducing the like where the septembles. In the like where the overlying the structure and reducing the like the structure and the temperature.

About a half hour efter our and of a man, liquid from the equilibrium first fittied the boiler despite the presquetons taken to one the boiler to the atmosphere, binds this did not affect he man line on start-up, the situethan as ions on.

The is orgivality of appliedly the boiler one first netice what the still was desired of water to that pure othernal could be tested. The still as finally dried by attaching a versual pump with a temp at the ten of the land and the ten ten opidenser leveler. Note saidling valves and the ten ten dendenser ware observe to the six are heat war supplied to the boiler. Should built and hour was resulted for drying.

Etherof vapor could be emalled at the blow tube of the pressure of the pressure system when pressure changes were being made, indicating that ethered was escaping from the condenser.

ethanol and water, and, because of the surging which carried air out of and into the condenser, mixed with the rest of the air of the pressure system.

Leakage from various connections in the system was noticed at three sources. Some leakage was occasionally noticed around the tubing nuts of the Toflon needle valve, despite the precautions taken to eliminate bending thrusts on the tubing system at that point. Additional tightening of the mits with pliers was found offective in eliminating the leaks. The ball joints at the system retameter were the second source on leaks. Ethanol vapor could be detected in the region but liquid was rarely noticed. The third source may be inforred from the results of a pressure test made with the recirculating lines filled with water. The pressure was raised in the pressure system until the water leg registered full scale. The entire system was shut off from the atmosphere and the drop in pressure with time was noted. The pressure dropped from 30 to 20 cm water in about 10 sec. The source was not in the pressure system, for during a test with the pressure system sealed off from the glassware, the drop in pressure was negligible. The source was the tapered joints on the custom glassware. No leakage was noticed at the joints held with the Teflon tubing.

The needle value which controlled the flow rate of water through the calorimeter did not seem to provide sufficiently stable control. The mercury regulator was

Presumably, the air in the condenser bosses esturated with etherol and water, and, because of the surging which carried also out of and into the condenser, mixed with the rest of the air of the presence system,

Leakago from vanious commercions in the system was notioed at three sources. Lome leakage was occavionally noticed around and bubing zerbs of she suften needle valve, daspite the securious value to albuduate bending throusts on the buckles system at thet coint. Additional tightening or but mate with without was . Dune offered to eliginating the Acake. The Delt jointe and one committee with the word the second source on learn. Ethaner raper sould be detected is the respect that light was marked wetter we thavely the third source may be trieved for all provided or presents advanced wate with the craft adda and there allow with the water. The TOTAN SITUATION DE COME SECRETARIO DE LA LOCATION DE COME DE C log the same and the section of the entire system size of the tom such attraction of the coupling of the confidence and mostly 19 sec. The source was not in the pressure approar for during a teer with the erosure cycless coalest from the classwero, the drap in pressure was neglitible. The source was the tapered joints on the conton glassance. In leakage was noticed at the joints noid with the Perlon tubing,

The needle value which controlled the flow rate of water through the ealerdneter did not seem to provide sufficiently stable control. The mercury regulator was

found to change its set point from day to day as a result of evaporation of the carbon tetrachloride. No change was noticed during individual runs and the set point was restored when necessary by the addition of mercury. It was necessary to turn off the valve in the line leading from the pump to the constant head tank at the time the pump was shut off. This step prevented water from flowing back into the lower tank and there causing an overflow. During warm weather, the cooling coils were found to be barely adequate for removing the heat picked up from the condenser and from the warm room.

CONCLUSIONS AND RECOMMENDATIONS

System Design

It should be evident from the long run times required to operate the present apparatus and from the constant error due to heat loss which is characteristic of this type of equipment that as high a flow rate as possible should be used. The importance of the use of equipment as small as possible which has been designed for as high a flow rate as possible cannot be overstated. Potential errors stemming from uncertainties in the heat losses which cannot be calibrated must be eliminated at the design stage. Weissberger (4) should be consulted for the common errors in heat loss calibrations. The water flow rate determination and the calorimetric calculation may be better made when there is a higher recirculation rate, and the length of a run may be sharply decreased. The author suggests that a design be attempted which recirculates at about 20 gm per min and has about 50 gm in the recirculating phase so that an entire recirculation cycle is completed in less than 5 min. Some of the design features of Altscheller might be incorporated, especially the features which separate the phases after contact.

Since the limiting accuracy in the enthalpy measurement appears in the system flow rate, perhaps the

found be change its set point from day to day as a result of evaporation of the carbon tetrachloride. He change was noticed during individual runs and the cot point was restored when necessary by the addition of necessary. It was necessary to turn off the valve in the line leading from the cump to turn off the valve in the line leading from was come to the sear these tent at the time the pump was chil wit. This sear there water from flowing book into the circustow that consider the constant constant water from flowing book into the constant water from the burner series of the constant constant water from the burner series of the constant constant of the constant constant of the constant constant of the constant co

more than the story bed

CONCLUSIONS AND HOLCOMMISHINATIONS

4 5 4 1

Syntem Destgn

aemit nur gnot ens mort densive od bluede il recontred to operate the present apparetus and from the conobant error Ace so hear lone which is characteristic of this type of equi mant that as high a flow rate as pontible should be used. The importance of the use of squipment as a data as now benefits wood and autica wide and firms interest in the second of the contract of the contract of the second in the contract of the co orrors see ting feet erocciningles in the heat lease which de unob compatible tes and by all anntes at the design atage. action for the contract bedience of the common appealant or in could men collected in, one with they pay decoration tion are the delication of the medical and are not the medical we do the are for this or not more relief and the length of a ran car be superag above. The enthor suggests that a design be neberg of waits recirculates at about 20 gra per min and has about 30 gr in the recirculating phase so that an engine recimentation eyole is completed in less than 5 min. bome of the design features of Altscheller might be incorporated, especially the features which separate the phases after contact.

Since the limitime accuracy in the enthalpy measurement appears in the system flow rate, perhaps the

of using a smaller quantity of equilibrium liquid and a highly efficient device for contacting the vapor and liquid. Accurate direct mass flow measurement could then be utilised for the system flow rate as well as for the water rate.

explored in this investigation. Surging has been reported in the literature by Brown (39). But it appeared to occur only in apparatus which incorporated the direct return of condensate to a flask boiler, and the possibility of its occurence in the present design was not anticipated. It can merely be noted that surging appeared to lessen as the operation more nearly produced equilibrium conditions, that is, proper compositions and lack of superheating.

Instrumentation

Although the error analysis for the flow rate determination (Appendix, p. 10) points to errors of about 0.1% obtainable at higher flow rates when the float is maintained to within a tenth of a millimeter of the set point, the difficulty of keeping close tolerances in float position during runs of the whole apparatus must be considered. Calibrations at low rates were made to within 0.1%, suggesting even greater accuracies during calibrations at higher rates, but such flow control requires constant concentration to avoid steady drifts in the float position. Such precoccupation with flow control was not possible during

once-through design should be reviewed with the thought of using a masiler quantity of equilibrium liquid and a highly efficient device for concecting the vapor and liquid. Hoourate direct mass flow memeurement could then be utilized for the system flow rate as well as for the water rate.

Include the system flow rate as well as for the water rate.

explored in which invocations, ourging has been reported in the limenature by drown (39). Out it appeared to commonly in appearance which incorporated the direct return of academents of a fless bolier, and the possibility of its contracted in the present deal of the possibility of its courous in the present deal of was not unticipated. It can service we note that surpline appeared to lessen as the expertion more nearly produced conditioning conditions, some incorporation conditions.

The statement of E

Although the orrest aralysis for the flow rate color dination (appendin, p. 100) points to errors of about 0.1. cotainable at higher flow rates when the floot is maintained to althit a cents of a millimeter of the set point, the difficulty of becoing close telerances in float resibles daring vers of the whole spaceatus must be considered. Calabrations at low rates were made to within eldered. Calabrations at low rates were made to within a targesting even greater ecouracies during calibrations at higher rates, but such flow control requires constant of higher rates, but such flow control requires constant concentration to avoid steady drifts in the float position.

eperation because of the other factors requiring attention.

Perhaps an alternative in-line flow meter such as the thermistor, magnetic, acoustic, or pottermeter type could be used and the flow rate recorded and later integrated over the run time. One of these might prove to be inherently more accurate than the rotameter. If the jacketed rotameter is still to be used, the jacketing should extend over a greater length of tubing below the rotameter to insure that the recirculating liquid is at the desired temperature when it passes the float.

but was used with very low potentials from the thermocouples. The use of the thermopile arrangement given by
Weissberger would add significant figures for both the
enthalpy and the equilibrium determinations. The thermocouple wire should be a smaller size to reduce heat leaks
out the end of the wire and to make the wires more flexible.

If an automatic multipoint recorder were used to monitor
the approach to equilibrium, the operator would be freed
from time consuming data gathering and a better record of
the operation would be produced. The K-3 potentiometer
could then be used to make the final determinations. The
material such as ice used for the bath at the reference
junction must be pure so that the temperature may be
accurately known.

Devices which automatically control the pressure in an equilibrium still are widely used. A suitable design

operation because of the other factors requiring attention.

Perhaps an alternative in-line flow meter such as the thermiston, magnetic, accounting or pottermeter type could be used and the flow rate received and later integrated over the rate time, one of these might errore to be interestly more accurate than the retained that the potential received the flow of the following the fine factors and another should entered content to the factors and langth of the factor the retained of the factors over a greater langth of the factor the retained of the factors over a greater langth of the factor the retained of the factors over a greater langth of the factor the retained of the factors over a greater langth of the factor the readers.

The II-8 potentianter is depute of high accuracy was east as a set of the potentials from the thermal soughes. The new of the thermophis arrangement given by this sougher would add rightfount digmes for both the optimized with the couple where about the conditions. The thermal couple where about the conditions also be reduced feat leaks out the end of the wire and to make the wires more floxible. If an adversite multipoint recorder wore ased to remiter the the approach to applicable to optimize the condition of the this approach to applifying, and observation record of the this community date gathering and a better record of the condition he ased to make the final determinations. The saterial such as ice used for the bath at the reference of junction and to ourse so that the temperature may be securately known.

Devices which autometically control the pressure in an equilibrium still are widely used. A suitable design

should be picked from the literature and constructed, or purchased,

A question arose concerning the difference in pressure between the top of the condenser where the pressure was measured and the liquid level where the measurement was to apply. Calculations (Appendix, p. 1/3) show that the head of vapor and the drag of viscous flow produced no appreciable pressure gradient.

Equipment

The reflux design, apparently an innovation in latent heat measurements, has proved to be a promising device, isolating the latent effect with but two drawbacks. These are the problem of producing adiabatic conditions and the possibility of diffusion losses. Suggested improvements are the use of a double walled tube for the vapor inlet to the condensate chamber, the placement of the thermocouple well T7 out of the vapor region, and the use of a silvered vacuum jacket around the contacting and condensing section to form an adiabatic well. A good example of vacuum jacket design is found in Altsheler (32). Without the first change it is impossible to operate the apparatus in such a manner that it will both supply the proper conditions for enthalpy and equilibrium measurements and also reach a steady state operation.

The method used to establish equilibrium leaves much to be desired. The inherent uncertainties resulting

should be picked from the literature and constructed, or purchased.

be question arose concorning the discusses in pressure the top of the academos where the areanyre was massived and the invaluable broad where the areanyre are was massived and the invaluable broad while the research are the top was to apply, while of the figure that in the arose in a confident of the state of the st

Justine.

Account of the control of the contro

The mailed waed to establish scalinarium lanvas much to be desired. The inherent uncertainsies resulting

from the liquid head in the flask are too great for determinations of the highest accuracy. Probably some design in which the equilibrium liquid is also circulated should be adopted. Designs of this type usually employ the vapor lift principle. The apparatus of Altsheler is a good example, while more conventional Swientoslawski designs by Brown (39, 40) might be considered. In any event the amount of the liquid phase should be greatly reduced to about 100 ml. Some provision should be included to directly heat the equilibrium liquid. The liquid may then be quickly heated at the start of a run and during a run the distribution of mass between the phases may be adjusted.

The decision made at the outset, to monitor the phase distribution by observing liquid levels, is not an entirely satisfactory method. A far more sensitive monitor would be a composition analyzer mounted in the recirculating system line. Such a device would have the savantage that it would make part of the composition analysis which is required anyway. For instance, the fluid leaving the rotameter could be passed through a refractometer cell before entering the boiler. A record of the refractive index would show what changes in composition were taking place as the still was being guided to steady operation. The problems of acquiring, modifying, and installing a refractometer would have to be surmounted. Perhaps conductivity or some other property could be continuously measured instead.

From the liquid head in the flack are too great for determinations of the highest accuracy. Probably some design in which the equilibrium liquid is also circulated should be adopted. Designs of this type usually employ the vapor lift principle. The apparatus of litchelet is a good example, while more conventional element designs by rown (Mr.) 40) after be constanted. In any swent the more that the constanted, in any swent the amount of the limit onnes about a constanted of the limit onnes about a constanted of the limit onnes about a constanted of the limit on state of the receipt and any first on about the equilibrium liquid of the state of the liquid of th

phase distribution by observing the post is not executively extinfuceout rethod. Its means exciting a mitted would see a seminate that the excited in the service of the controling that it would the part of the executive and the control of the required array. The feather, which is required an exercise the control of passes the analysis of the property of the post of the control of the post of the problems of acquiring, modifying, and installing a refractometer would have to be surfounted. Forthaps conductivity or these other property could be continuously measured instead.

The problem of superheat and residual liquid at the boiler might be solved by providing holding time for the superheated vapor leaving the boiler. A length of tubing from elbow to entrance of the equilibrium flask could be used as a controlled heat leak to remove the superheat. A small heater at the low point of the apparatus, the elbow, is recommended for controlling the build-up of liquid at this point. Although the main boiler could be run at high superheat to prevent liquid buildup at the elbow, the extra heater is recommended so that the superheat may be eliminated at the boiler, leaving only slight superheat, say 1 to 5 degrees (C), to be removed by the proposed heat leak. Liquid which is not vaporized in the boiler or which trickles back from the heat leak will be vaporised by the small heater. In connection with this, it is recommended that a stopcock be placed at the lowest point in the equipment so that the entire still may be readily drained.

adiabatic walls used in this apparatus. They are the impossibility of using the heated adiabatic wall to match the temperature gradients along the condensate collector, the heat leak from heated wall into the calorimeter, and the difficulty of manipulating the temperature of the wall to the desired point. The silvered vacuum jacket mentioned above might be heated and insulated much like the adiabatic

The problem of superheat and residual liquid at the botter might be colved by providing bolding time for the superneated vapor leaving the boller. A length of tubing from elbow to entrance of the equilibrium flack ould be used on a montrolled here take to been ed bluce superbear, a small seaver at the low offit of the separaour, the older, is thoseworde to, consecultar the cold-up This to the second of the control of the second of the sec I read our thought of prevent in Capitalist to the sum ou of the distribution of the companies of the second of the distribution of the distribu thing the filter told the part to the term of the residence and there TO THE DISTURBED AT THE STREET OF THE PARTY OF A STREET AND A STREET with a commercial content and the file of the content of the conte DO Sign That are that since among antivities and a relien-VANDERS DY B . B . B . Cap. . B GOLLEG E. OR . B . PC B . S. CA is reach wide with stance. O is to a face comment point in significant to the will the significant and the read'ly arained.

established wells used in the authorise of the proof of the collaborate wells, they are the tempose interpretary of using the restence whicher is and to the the tempose the gradients along the condense collector, the heat load from heated we a theo acceptanter, and the difficulty of manipulating the tempose rathe of the desired point, the silvered vector is acceptanted monitoned show might be beated and insulated much like the adiabatic

walls in the present arrangement. The vacuum jacketed region should surround the equilibrium liquid.

blown onto all standard tubing at places where Teflon sleeves were used. It was felt that forces produced while attaching the sleeves might result in breakage of ordinary tubing. Use of the sleeves has shown that attachment is readily made and that the precaution was unnecessary.

Restriction of the line between the contactor and leveler by capillary tubing is undesirable since the same level is to be maintained in both.

The Teflon and glass needle valve should be eliminated. Other designs are available (41), and a more rugged and accurate type should be designed and constructed.

The use of a constant voltage power supply would further reduce the difficulty of operation and assure a more reliable supply. The powerstats controlling the boiler and adiabatic walls failed to give close enough control of power, and should be replaced by more sensitive controls, perhaps in the form of slidewire resistance.

The water system operated with but little difficulty. Tygon paint used to coat the two tanks was not
successful in preventing corrosion. A permanent system
should be constructed from copper. It may become necessary
to insulate the tanks and lines to prevent heating during
warm weather which would overload the cooling coil or
cause the delivery of water at the wrong temperature to

walls in the present arrangement. The vacuum jouketed region should surround the equilibrium limit.

shrift over middle the like the tree were rights

blown and all straight and are already to the some measured willed allowers form until . It was to be the some measured will to the admin the attention of the some first and the some f

The content of the co

which any open show the contraint two bent bent of the two dense of the angent of an observation. It may be occur necessarily to the angent of the may be occur necessarily to the times of the time the time the time weather witten would everywhere the continer witten would everywhere the continer of the content the content to the conte

regulator had a large time lag which was probably due to the use of stainless steel for the bulb. Flattened aluminum tubing would provide a much faster response.

Scope of Investigation

should place the most stringent conditions on both the equilibria and enthalpy measurement. Equilibrium determinations should be made on a system of high relative volatility and compared with other literature references, a method advocated by Brown (39). The operation of the still should be smooth during such operation. Heliability tests of the enthalpy determination are difficult to set up. Runs on pure components provide comparisons with accurate literature values, but the operation of the contacting section during multicomponent runs is difficult to check since highly accurate values, experimental or calculated, are not available.

No consideration has been given towards apparatus for immiscible systems. Fowler reports a variety of designs, and any which supply a sufficiently great recirculation rate could conceivably be modified with the addition of a calorimetric condenser. Flow metering of the vapor phase constitutes a problem because the condensate would split into two phases.

Construction of the enthalpy-composition diagram

the rotameter jacket or calorimeter. The mercury thermoregulator had a large time lag which was probably due to the use of stainless steel for the bulb. Plattered electrons tubing would provide a much factor response.

Soose of Investigation

enter que contidade en la contine diformente de comenticontrata de contrata de la consecuente de contrata de con

tus for immisciole systems. Toulor repersus a variety of designs, cad any which appears a variety of designs, cad any which supply a sufficiently great reciprounstion rate conceivably a modifical wire the conceivably a calerimetrus conceivably a thouse setting of the vapor passe constituted a problem passes the condensate would aplif into two phases.

Construction of the enthalpy-composition chagran

phere pressure. For plant design calculations where the pressure may be different from one atmosphere, the effect upon the enthalpy-composition chart should be determined. Liquid heats of mixing and specific heats are not affected, but equilibria and the heat of vaporisation show considerable pressure dependance (42). Thus design data must often be determined at the pressures to be encountered under process conditions.

Throughout the experimental part of this study, the assistance provided by thermodynamic methods in the construction of the enthalpy-composition chart has been overlooked. It is not always necessary to have all four kinds of data available over the composition range. In particular, if the heats of vaporization of mixtures are unavailable, the dew point curve may still be constructed by the method outlined by Dodge (43). The heat of vaporigation of the pure components, the specific heats of the pure component vapors and the dew point temperature of mixtures form the basis of computation, with the assumption that the heat of mixing of the vapor mixtures is negligible. Other assumptions are usually incorporated into the values of the latent heats of the pure components when computed from vapor pressure data. These assumptions become the subjects for investigation by researchers with such apparatus as that of the present study. An experimental and thermodynamic analysis of the terms comprising the latent

is usually made with data taken or computed at 1 atmosphere pressure. For plant design calculations where the pressure may be different from one atmosphere, the effect upon the emthalpy-sumposition chart chould be determined. Liquid hears of mixing and specific hears are not not affected. Dut equilibria and rise hear of vaporisation show considerable pressure to endance ((P)). The attribute the endance of research at the pressure of endance ((P)). The attribute the endance of and one attribute and other attributes at the pressure of attribute at the pressure of attributes.

Pares house the oxportage ball in the a budge Copy of the second of the second of the second in the second of the seco counting, of the artistance of the material continues error is a version personautour agents for el de l'oblemisero itinds of detailers to see the companies of the endiagon of the seed of the companies of the seed of the companies of the seed of the companies of the companie and author, in the energy of which is the first of the THE CONTRACTOR OF A CONTRACTOR OF A CAREER OF A CAREER CONTRACTOR OF THE CONTRACTOR and the control of the opposite of smile a control of the cablen of the jame orgonocts, the specific rather a cale party component vacans and objective was policy and another anamous of the tures form the bases of commitation, with the assumption that the acut of mixing of the varior mistricular to the lightbis. withou assumptions are neutily incorporated incorporate of the lateou seeks of the pure commones when compated from vapor presente data. These assumptions become the subjects for investigation by researchers with such appens. tus as that of the present study. An expertaental and thermodynamic analysis of the terms comprising the larent

work on the experimental values of the pure component latent heats and the experimental values of the pure component latent heats and the assumption of negligible heat of mixing to get the calculated values. But the validity of the assumption with other systems appears to be unchecked by anyone else.

Perhaps the most crucial question to be answered in regard to continuing experimental measurement of enthalpy involves knowing what accuracy of the enthalpy values is necessary for the use to which they are to be put. If the purpose be a thermodynamic investigation of the ideality assumptions, then the experimental investigations should be continued, remembering that the latent heat may have to be determined to within 0.1% before non-idealities are detected. If the purpose is the accumulation of enough information to construct enthalpy-composition diagrams with accuracy sufficient for column design applications, then the calculation method, combined with equilibrium determinations, may give satisfactory results.

of thermodynamic investigation, the more recent literature should be consulted for methods of calculating the heat of mixing and the other excess thermodynamic properties from equilibrium measurements. Work has been done in this

heat of any two-component mixture was made by Dana. His work on the exponitivegen system led to the conclusion that latent heats at intermediate compositions sould be determined to withhin 0.1% of the exportmental values, using the exportmental values of the pure component latent heats and the ascumption of nogliable heat of whithey of the naturation of got the calculated values. But the values of the naturation of objects of the casumante.

With other spread operate we as unchecked of entermoler.

In Pegard to convincing at the content that of the property of the property of the contents of

Af the decision in adds to continue at the level level of thermodynamic investigation, the more recent literaums should be consulted for methods of calculating the heat of mixing and the other excess thermodynamic properties from equilibrium measurements. Nork has been done in this

field by Murti, and by Scatchard and co-workers (44).

This work should be investigated anyway to see if experimental determination of the heat of mixing of the liquid may be made unnecessary.

to usually made with date taken or computed at I obman phone processes. For plant dost a carrelettema wante the presente war bu different from one ambenden, the in the state of the states and the state of the states of Start the second of the second Burney Committee of the The second of th The state of the s Section 15 Company of the ϕ (4.55) ϕ MINE TO THE RESIDENCE OF THE STATE OF THE ST the control of the co and the second of the second o the first of the control of the cont The second of th The following the spinor will add and arread dramages on the mores form a bessa of commission, with the asymptotic took in the case of the contract of the second of the seco manter and our! become created williams and week amess works holigant near state of the page occurrence when to added the from vapor preseure deta, These assumptions besent the subjects for investigation by researchers with such soffintus as that of the present study. An expertaching and

work on the oxygen-nitrogen system led to the conclusion that latent heats at intermediate compositions could be determined to within 0.1% of the experimental values, using the experimental values of the pure component latent heats and the assumption of negligible heat of mixing to get the calculated values. But the validity of the assumption with other systems appears to be unchecked by anyone else.

remaps the most crucial question to be answered in regard to continuing experimental measurement of enthalpy involves knowing what accuracy of the enthalpy values is necessary for the use to which they are to be put. If the purpose be a thermodynamic investigation of the ideality assumptions, then the experimental investigations should be continued, remembering that the latent heat may have to be determined to within 0.1% before non-idealities are detected. If the purpose is the accumulation of enough information to construct enthalpy-composition diagrams with accuracy sufficient for column design applications, then the calculation method, combined with equilibrium determinations, may give satisfactory results.

of thermodynamic investigation, the more recent literature should be consulted for methods of calculating the heat of mixing and the other excess thermodynamic properties from equilibrium measurements. Work has been done in this

thermodynamic analysis of the terms comprising the latent

heaf of any two-component mixture was under by sund. Its work on the expension eyesel led to the consideration that the expension of the consideration that the bests of interesting the consistent of the consideration of

A TELL CONTROL OF CONT

At the decision is add to continue at the love level of thermodynamic invatigation, the more recent liters cure chould be consulted for methods of estaulating the heat of mixing and the other excess thorsodynamic propagates from equilibrium measurements. North has been done in this

field by Murti, and by Scatchard and co-workers (44).

This work should be investigated anyway to see if experimental determination of the heat of mixing of the liquid may be made unnecessary.

APPENDIX

This work should be investigated unyway to see if experinental determination of the heat of mixing of the limit may be made unnecessary.

field by Mirts, and by Soatchard and co-werkers (44).

	SYSTEM Rotameter.	leveler number in cm	WATER ROTAMETER	water temperature In Lover tank	PRESSURE WATER LEG CM OF WATER	SI	VERSTAT ETTINGS VOLTS	1			тнеж	OCCUPI CENTI	E REAI	DINGS					NEW POMERSTAT SETTINGS VOLTS			COMMENT ON CONDITIONS OF OPERATION
TIME PM	SYS	IN EE	MAT	WAT	PRE WAT CM	ı	II	III		2	14	5	6	7	8	9	10	TIME	:	11	111	
2:38	0	13		24.6	Ö	0	30	25			63.8		101	56.4	81.4			2:41	0	40	25	
2:52	0	13.1		24.8	0	50	140	25	·		64.7	18.5	10L		83.1			2:54	50	30	25	FOUR FILL LIQUID SAMPLES
	2.6	9.8			0	70	30	25		77		55.2	103	62.9	85.0	59.0	214	3:27	7≎	1"	25	TRANSFERRED FROM FLASK T' RECIRCULATING LINE
3:18	4	8.1			0	70	15	25			87.5	87.8	96.8	82.3	87.2	75.9		3:25	7C	25	25	
1	5		8	24.95	10	65	25	25	4	98.0	88	89.9	95.8	81.3	89.5	79.3	24.5	3:41	65	25	15	
#=#0	5.00	11.7	7•3 7•3	24.90 25.00	11.2	65 52	25 1 5	15 20	5.6-6.3 4.5-5.4	111.6 170	91.4 91.5	92.1 91.7	9!1•2 89•9	811.8	82.1	79.5	271	h:31	60 40	15 22	1.ª	LIGHTO SYLVE BIRPLING LEVEL IN ELECH OF BOILER
7:02	4-98	10.2	6.7	24.97	8.1	50	2 2	18	4.4-4.5	156	90.9	91.0	86.5	84.1	81.3	7'	212	7:U	1.7	25	19	THE METT,
7:33	5.00.	9.9	6.6	25.00	11.0	47	25	19	և.3:	135.1	90.9	90.9	87.7	84.1	81.5	75.7	213	7:141	140°2	?<.<	1°.5	FEN DY 15 TY 7:31, AP NY 1.5 NY AY 7:33.
7:47	5.00	9.6	6.6	25.02	11.7	48.5	30	18.5	خصت	131	90.8	90.8	88.6	814.C	81.8	79.7	2:2	7:55	1,8.5	3≘	19.5	APIT 1 ML.
8:21	5.00	9.2	6.5	25.02	11.1	48.5	30	18.5		132.2	90.7	90.7	93.2	83.8	81.7	79.7	24.4	8:30	148.0	24	la.c	ELF-W EMERTY OF LIVE ED
8:43	5.00	9.0	5	25.00	10.8	1.8	25	18.6	4.6-5.4	131	90.7	90.7	92.7	83.9	92.0	79.7	213	A:42	1,0.0	20	La.	55% DE 5.5~7.15 AT 9:02
9:04	5.00	. 8.9	4.8	25.01	11.7	47.7	20	18.5		127.7	90.6	90.5	91.0	83.7	81.9	79.5	213	ទ:ប	1.7.9	214.5	1	A FTG ER ES AT 9:14
9:29	5.00	8.6	4	25.02	11.6	47.8	24.5	18.4		121.0	90.5	90.5	91.0	83.6	91.9	79.7	2:5	6:35		74.5	La . 3	ELBON ENGTY
9:47	5.01	8.4	3.2	25.00	11.1	47.7	24.5	18.2		125	90.5	00,€	90.9	83.5	81.9	79.7	21.4	G: \</td <td>47.4</td> <td>24.5</td> <td>17.9</td> <td></td>	47.4	24.5	17.9	
10:19	5.00	8.1	3.1	25.01	11.9	47.5	40	17.9	5.3-6.1	122	90.5	90.5	101	83.5	81.4	79.7	214.3	10:29	1.7.0	1.0	17.8	FEW DRITS HEATING LIMER FLASE TO FUT
10:52	5.00	8.0	3.2	25.00	11.6	47.1	110	17.8	5.6-6.2	119	90.5	90.4	111	83.4	81.9	79.8	216	h1: 0	47.1	148	17.5	LIGITO IS INTREASING MORE MATIL
17:16	5.00	8.2	3.4	25.00	11.2	147.2	110	17.8	5.96	118	90.6	90.5	יותנו	83.6	1			11:25	l l	C	17.0	LEVEL IS ABOUT 4" INTO RETIR- BELOW BURDLING AND CHATING PLASE
11:110	4.98	8.2	3.7	25.02	5.	47.2	0	17.0	<u> </u>	1176	90.11	90.4	100	83.1	82.5	79.7	211	11:49	0	OFF	OFF	IMPREASING.

FIG. 13 TABLE II. DATA OF HUN 8

FIG. 11.
TABLE III. DATA OF RUN 9.

	TIME	SYSTEM ROTAMETER	leveler nubber in cm	WATER ROTAMETER	WATER TEMPERATURE IN LOWER TANK	PRESSURE WATER LEG CM OF WATER	POWERSTAT SETTINGS VOLTS			IGS THERMOCOUPLE READINGS CENTIGRADE						TDE	NEW POWERSTAT SETTINGS VOLIS			COMMENT ON COMEDITIO OF OPERATI	xwes				
	PM.	88	25	R SC	WAT IN	E & S	I	II	III	_ 1	2	4	5	6	7	8	c	10	PM	I	11	III	·-·		
	-	5.0	8	-	25	0	60	25	20		97.8	91.4	91.5	85.4	83.3	80.3	79.8	24.8	2:02	6 0	26	19	ELBOW FULL OF LIQUI	В	
	2:32		10.2	-	25.6	0	60	26	19	 -	9 t	93.1	93.2	87.3	82.7	81.0	80.0	24.3	2:39	50	30	17	BARFLY BUBBLING	!	
	2:56		10.2	-	25.05	-6.5	50	30	17		105	92.8	92.9	88.0	82.4	80.0	79.6	24.6	3:06	50	30	17	JUST BELOW LEVEL OF		
	կ։կ2	5.00	5	` -	23.6	-8.5	50	30	17		99.5	91.4	91.5	97.7	81.9	79.1	79.5		4:47	50	25	18	SAMPLE LIME BROKEN, BUN WAS NOT STOFFED		
3	6:46	4.8	1.8	-	24.4	- 9	50	25	1.8		بابلا	90.9	90.9	93.7	81.7	81.5	79.5	25.0	6:51	146	2 C	17	LEVEL ALMOST OUT OF STORED, SAMPLE LIN SAMPLES FED INTO RE	E ATTACHED,	
		5.0	9.5	-	25.15	0	71	20	17		93.1	92.5	92.7	97.5	82.4	79.3	79.9	25.7	8:01.	50	25	20	RAISE LAVEL, RUN RE		
		5.00	9.9	-	24.95	4	50	24	20		95.5	92.5	92.6	88.3	82.3	81.3	79.7	25.6	8:29	148	28	18	BARELY BUPBLING-STO	TFED-BECAN AGAIN	
	8 :3 5	5.00	9.5	-	24.97	li.	8. کیا	28	18		99.1	92.3	92.4	80.5	82.2	81.3	79.7	25.3	8:12	l ₄ 7	28	17.5			
	8:15	5.00	9.0	7	24.97	4.5	47	28	17.5	4.25	94.7	92.0	92.2	90.7	82.1	81.2	79.6	25.0	8:52	1.7	25	17.5	BARELY BURNLING		
		6.00	6.8	6.6	25.00	5	54	25	17.5	6.2-6.6	91.2	91.5	91.5	91.0	82.0	80.6	79.6	21.0	9:103	514	25	17.5	RAISED REDIFO, RATE	E TO INCREASE TI.	
	10:01	5.8	4.4	6.5	24.98	4	60	25	17.5	6.68	91.1	91.1	91.2	91.0	81.9	8c.5	79.7	25.1	lo:ce	55	218	17.5	BARELY BURRLING FRO		
	10:29	5.8	5	6.4	25.00	1.5	70	24.8	17.7		93.6	91.5	91.6	9C.8	82.2	BC.L	79.5	25.2	10:35	7¢	25.1	15.1	S . W	OUR SAMOULES ED INTO	
	10:39	6.01	8.9	-	25.00	2	60	30	18		93.9	92.3	92.4	92.0	82.5	80.7	80.1	25.1	10:53	6 C	29	18.5	BARELY BUPHLING RE	SCIPCULATING LINE	
	10:56	6.∞	8.9	7	25.01	2	60	29	.18.1:	7.4-8.0	9h	92.3	92.3	92.9	82.5	80.8	80.	25.0	भाःक	6.	25	18.1.	BARELY BURBLING		
	11:0h	6.00	8.8	7	24.95	2;	60	25	18.1.		95.3	92.1	92.2	2.8	82.1.	81.0	8c.c	25.0	11:09	<8	25	1F.L	BARELY BURBLING	NG	
	11:23	6.00	8.4	7	24.95	-2.6	57	25	18.4	· -	98.6	91.8	91 9	92.6	82.3	91.2	79.8	217	11:27	57.5	26	1º.3	NO PUBLING		
	17:14O	6.00	7.6	7.1	25.02	-2.6	57.4	25.1	18.2		94.8	91.6	91.6	92.5	82.1	81.3	79.8	211.		57.1	25.1	17.5			
	12:10	5•79	4.9	7.1	25.00	-5.5	57.4	25.1	17.5		92.5	91.1	91.2	92.1	81.9	L		L	12:17	OFF	OFF	OFF	T1 STEADY		

CALIBRATIONS

Calorimeter Thermocouples

The pair of thermocouples used for the enthalpy determination was calibrated for a 5 to 10 degree (C) difference with the lower temperature at 25 C. Therefore two controllable constant temperature baths were required. The thermocouple to be used at the calcrimeter inlet was inserted with a drop of mercury into a scaled six mm. glass tube and immersed six inches into the lower tank of the cooling water system. The pump was used to recycle the water directly back to the tank to provide agitation, and the tank was controlled at 25 C. The other thermocouple was similarly immersed in a four gallon constant temperature oil bath. A motor driven agitator and a 500 watt immersion heater, the latter operated through a manually controlled powerstat, were used to maintain the bath at levels between 30 and 35 C.

Two Brooklyn Thermometer Co. mercury in glass thermometers, range 19 to 35 C, interval to 0.01, with auxiliary 0 C scale, and supplied with factory certification, were used to determine the two temperatures. Corrections reported in the certificates were applied, and were no greater than 0.01 degree. Emergent stem corrections were found to be of significance in only one case, for

and the same

ngagan gapang kenalagang gapang kenalagan kenalagan gapang kenalagan gapang kenalagan gapang berasal berasa ke Tanggang penalagan kenalagan gapang kenalagan gapang kenalagan gapang kenalagan gapang kenalagan gapang kenala

en en la companya de la co

na dia mandria di Kalendaria. Ny INSEE dia mampiasa ny kaominina dia mandria mpikambana ao amin'ny fivondronana amin'ny fivondronana amin'n

 $\mathcal{F}_{i} = \{ i, j \in \mathcal{F}_{i} : i \in \mathcal{F}_{i} : i \in \mathcal{F}_{i} : i \in \mathcal{F}_{i} \}$

nurilishme with a second control of the control of

which a correction of 0.01 degree was applied. It is estimated that the temperature difference between the two baths could be determined to 0.01 degree, an error no greater than 0.2% for the 5 to 10 degree range of temperature difference.

The water tank could be maintained to within 0.01 degree indefinitely, and the oil bath could be maintained to within 0.1 degree indefinitely and to 0.005 degree for a half minute. The change in temperature difference between the two baths was on the order of 0.005 degree during the time required to read both thermometers, standardize the potentiometer, make the potential determination twice, and record the data.

measurements, a series of thirteen determinations was made over a seven hour period with the baths at 25 and 35 c. The actual temperature difference was not more than 0.05 degree different from 10 degrees, and each potential was corrected by 0.4 µv for each 0.01 degree between the actual temperature difference and 10 degrees. The corrected values of potential showed a spread of 1.1 µv or 0.3% from the average potential value of 412.1 µv. An additional check on stability of the circuit was made two days later, and three determinations averaged 412.2 µv with a spread of 0.6 µv. It was felt that confidence in the stability and reproducibility of the thermocouple and

which a correction of 0.01 decree are applicable. The obtained which the back is not the more than the back and the back is not the area of the area.

The state of the s

potentiometer circuits had been established.

couple potential corresponding to the condenser water ΔT would be, for a given ΔT , independent of the temperature at the inlet of the condenser. Another set of calibrations was made to determine the effect of a change in the temperature levels in the two baths at a constant temperature difference. The potential was determined for a five degree difference at temperature pairs of 24.50 C and 29.50 C, of 24.90 C and 29.90 C, and of 25.00 C and 30.00 C. The potentials at all three 5 degree intervals were identical, showing that determination of the inlet temperature and corrections for its variations would be unnecessary.

Calibration of the thermocouples at intermediate temperature differences in the range between 5 and 10 degrees established the relation between potential and temperature difference. The factor k in the relation $\Delta T = k \mathcal{E}$ has been plotted as a function of \mathcal{E} , the potential, in Fig. 15. The relation may be seen to be not quite linear.

The calibration showed that the potential at Tl could be relied upon as an accurate measure of the temperature difference across the calorimeter, and would be independent of small changes in the temperature of the cooling water system.

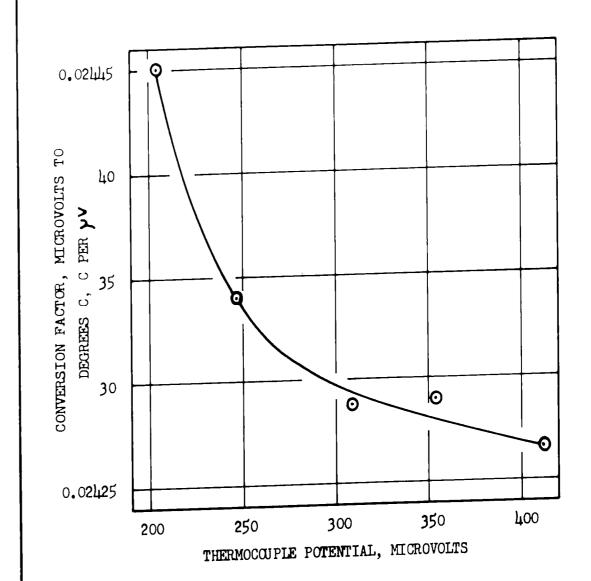
Equilibrium Temperature Thermocouples

The oil bath was used to calibrate the potentials

the stability and reproducibility of the thrusepounts and

THERMOCOUPLE CALIBRATION FOR ENTHALPY MEASUREMENT FIG. 15

COPPER-CONSTANTAN JUNCTIONS (TI COOLER JUNCTION AT 25 ± 0.5 C.



notentians e mos our sulmores or demoisened.

The contract of the contract

Fig. outstoom whores the case process to pare country to pare country of the country outstoom as an accurate control of the country of the country of the control of the co

Mout Hbrium Tomografure Thermocouples

The oil bath wes used to calibrate the potentials

 \sim

which a correction of 0.01 degree was replied. The continue of the representation that the temperature differentiac be well the two baths acade by defention of the correction of the correction

The state of the control of the gradient of the control of the con

potentiometer circuits had been established.

couple potential corresponding to the condenser water ΔT would be, for a given ΔT , independent of the temperature at the inlet of the condenser. Another set of calibrations was made to determine the effect of a change in the temperature levels in the two baths at a constant temperature difference. The potential was determined for a five degree difference at temperature pairs of 34.50 C and 29.50 C, of 24.90 C and 29.90 C, and of 25.00 C and 30.00 C. The potentials at all three 5 degree intervals were identical, showing that determination of the inlet temperature and corrections for its variations would be unnecessary.

Calibration of the thermocouples at intermediate temperature differences in the range between 5 and 10 degrees established the relation between potential and temperature difference. The factor k in the relation $\Delta T = k \mathcal{E}$ has been plotted as a function of \mathcal{E} , the potential, in Fig. 15. The relation may be seen to be not quite linear.

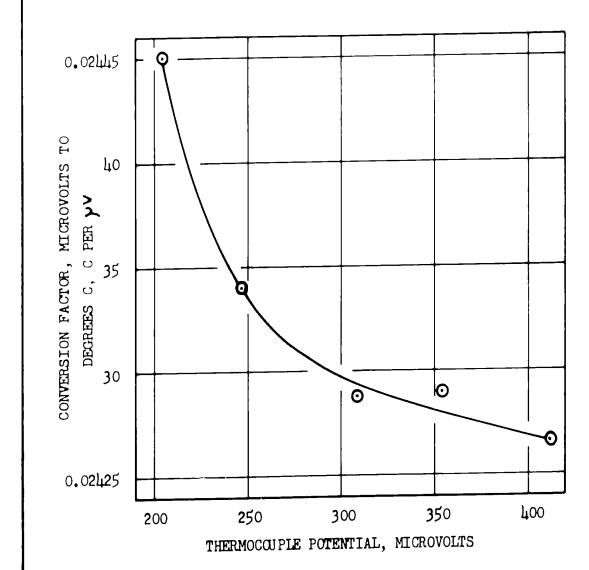
The calibration showed that the potential at Tl could be relied upon as an accurate measure of the temperature difference across the calorimeter, and would be independent of small changes in the temperature of the cooling water system.

Equilibrium Temperature Thermocouples

The oil bath was used to calibrate the potentials

THERMOCOUPLE CALIBRATION FOR ENTHALPY MEASUREMENT FIG. 15

COPPER-CONSTANTAN JUNCTIONS (TI COOLER JUNCTION AT 25 ± 0.5 °C.



potentioneter ofrontto had been established.

It was nothered a sire whother whother who the strate of the property of the construction of the strate of the construction o

Can specify a constant of the rest of the rest of the constant of the 20 map 20

The state of the s

Moullibrium femography Thermocouples

The oil bath was used to callbrate the potentials

of the seven thermocouples used at the equilibrium flask, contacting section and adiabatic walls. The thermocouples were inserted with a drop of mercury in individual six inch long glass tubes and immersed in the bath with the agitator, 750 watt heater, and two unstandardized ASTM moroury in glass thermometers with 50 C to 100 C ranges in one tenth graduations. The thermometers agreed to within 0.05 degree or closer over their ranges. They were read with magnifiors to 0.01 degree and stem corrections of as much as 0.3 degree were applied. The seven potentials were recorded to the nearest microvolt at calibration points of 70, 30, 90, and 100 C. The potentials agreed to within 3 µ v at each temperature, which indicated uniformity of composition in the thermocouple wire. The calibration followed practically a straight line displaced from the calibration values found in handbook tables (45) by 20 microvolts, equivalent to 0.4 degree. The probable cause is a difference in the composition of the thermocouple wires from those used in the handbook determinations. It seems unlikely that impurities in the ice at the reference junction could cause a 0.4 degree difference from 0 C, that the thermometers used in the calibration could be that inaccurate, or that the potentiometer would be in error by that much. The displacement demonstrates the necessity for calibrating thermocouples for accurate work.

of the seven thermonouples used of the equilibrium flesh, contracting or other additional additional additional and additional additionaly additional additional additional additional additional addition

A commence of the contract of

Pressure

to bring the pressure in the system to 760 mm Hg, a barometer and two correction and conversion charts were used. The reading of a mercury in glass barometer was corrected for temperature effects, and the difference between the barometric reading and 760 mm Hg was converted to the equivalent water leg height. The water leg was adjusted using the blow tube in the pressure system. The pressure could be maintained to within 1 cm water or 0.7 mm Hg of one atmosphere as a conservative estimate. This variation would cause a change of no greater than 0.05 degree in the boiling point, so that operation at 760 mm Hg could be assured to within the expected accuracy of the temperature measurement of 0.1 degree.

Heat Leak

was opportunity for heat transfer between the upper aluminum cylinder and the water in the condenser jacket through the plywood and Styrofoam. The heat transfer was independent of the vapor flow rate into the condenser.

Heating rates of the condenser water were determined with the aluminum walls at 80, 90, and 100 C and with no fluid recirculating in the system. The heating amounted to 13, 18, and 23 cal per min at the three temperatures, or a correction of about 1% to be applied to the expected

Prosecure

To defending the problem is the system to 760 money, a bandto bring the problem is the system to 760 money, a bandmother and two correction are convenience charte were used.
The resultan of a charte in I have some sure according to the problem of the problem is a convenience.
The resultan of a charte in I have a first and a convenience of the conven

The consideration for the second seco

latent heat flux of 2000 cal per min. Since the correction was accurately known and was taken into account during enthalpy calculations, it could not be a cause of inaccuracy or uncertainty.

System Plow Rate

Calibration of the rotameter was made after the run of the apparatus so that the same composition could be used in the calibration. After analysis of the vapor sample, about 500 cc of an ethanol-water solution were prepared to within 0.1% of the vapor concentration for use in the calibration. The recirculating line was dismantled above the Teflon needle valve. The solution was charged to a flask provided with a two hole stopper. An inverted U-tube of capillary tubing, in sections joined by Teflon sleeves for flexibility, ran from the bottom of the flask, out of the flask, and down to the inlet of the needle valve. A bit of glass wool in a short Teflon sleeve, which was slipped over the capillary tubing at the bottom of the flask, served as a filter to prevent solid material from interfering with the operation of the needle valve or rotameter. The flask was mounted above the panel board so that about a two foot liquid head was established between liquid level in the flask and the outlet from the vapor sample line. Liquid flowed from the flask through the filter, U-tube, Teflon needle valve. thermocouple well, rotameter, Teflon sample valve, and

latent host flux of 2000 onl per min. Since the correction was accurately known and was taken into account during enthalpy onloulations, it could not be a cause of incouracy or according.

System Plon Late

delibration of the robanethe was write after this nound the to middle out the properties of THE STATE OF THE OWNER OF A RESOLUTION OF THE STATE OF TH TO CONTRACT TO LEAD TO CONTRACT OF THE PROPERTY OF THE PARTY OF THE PA water or we wanted the contract of the contrac ಯಾಗು ವರಚಿಸಿದ್ದಾರೆ. ಎಂದು ಸಂಗೀತ ಕಾರ್ಯ ಕರ್ಮ ಕರ್ಷ ಗಳುರಾಗು ಅಂತಿ ಮುಂದು ಗುರ್ತಿ ಎಂದು ಅಂತಿ ಮುಂದು and grant to the control of the cont restrict and the contract of manager to take a commentation THE ST WITH SEC. LET . T. S. S. MARCHES S. B. MARCHES (210) THE representation of the contraction of the contractio with the track of the term of the contract of the contract of the the religious of the ser were becalle and delike . 2001e the very of colling or hevers , i ell and le modice and add to anidarous with defin artesty dail work halveden biles needle valve or november, The Shirk has region above the panel board so that about a two woot landid terms and setabliched setween light level in the theat and the outlet from the vapor ample line. Liquid Clored from the flask throngh the filter, J-tube, Seflon needle valve, thermocomple well, rotameter, forlon easele valve, and

into a 100 ml Erlenmeyer flask set on a laboratory pan balance. The Teflon valve in the line to the boiler was kept shut. An Ohaus 311 gm capacity balance reading to 0.01 gm was used. Water was circulated in the rotameter jacket at 25.00 0.

Bubbles were removed with difficulty by replacing the Erlenmeyer flask with a sample tube, draining a full sample, then using lung pressure to force liquid from the sample tube back into the system. Eventually the bubbles were forced up the U-tube and into the flask where they escaped from the system.

rate with the needle valve to the same rotameter reading as observed during operation. Liquid flowing from the sample outlet was caught in the Erlenmeyer flask on the balance. As the balance arm passed the rest point, a stopwatch was started. The balance counterweight was increased by 30 gm, and the stopwatch was stopped as the balance arm passed the rest point. The sample valve was then shut off until the next determination. The time required for each calibration ranged from eight to twenty minutes. Throughout each calibration, the needle valve was adjusted to maintain the proper rotameter reading.

Early difficulty in obtaining reproducible calibrations led to jacketing the rotameter to make the temperature of the fluid constant. Calibrations differed by 2% when water was tested at 25.00 and 26.00 C at a into a 100 ml Brisnmeyer flack set on a laboratory pan balance. The Teflon valve in the 11mo to tun boiler was kept shut. An Ohans Sil go oa setty belonce reading to 0.01 cm was used. Teter were invalinged to the retaratory facket at 25.00 c.

Chairmagner Timek with a tamble officiently by replaced the importance of the importance of the importance of the importance of the case o

Delegation of the control of the con

Drations led to jacksting old rotameter to make the temperature of the fluid constant. Jailbrations differed by 2% when water was tested at 25.00 and 26.00 0 at a

changes 2% per degree at room temperature, while the density changes 0.03%. After the effect of temperature was eliminated, a series of four successive calibrations with water agreed within 0.14% of one another. Calibrations at intermediate compositions agreed to within 0.3%.

losses was made for the calibrations with ethanol and with mixtures. Ethanol was left standing in the rlenmeyer flack on the balance for a period of time until a change in weight could be noted. Based on this evaporation rate, a loss was calculated for the length of time of each calibration. The loss was added to the value of mass collected and the sum divided by the time of the calibration to determine a corrected rate. Corrections amounted to about 0.3%. The determination of the error and was probably low because the mass transfer from a droplet falling into the Erlenmeyer flack would be greater than the transfer from a stagnant liquid surface.

The position of the float in the rotameter was maintained to within a few tenths of a millimeter during runs and calibrations. The calibration chart from the manufacturer has been used with the dimensions of the rotameter scale to estimate the rate of change of flow rate with float position at various float positions (Appendix,

rotaneter reading of five. The viscosity of mater ohanges for dogress at room temperature, while the that density changes 0.00m. There the effect of experience of the state of experience of the state of experience of the first area of the state of experience of the state of the

isomery of the control of the contro

das Lagitons of him is to a childmeter and maintained on a childmeter and maintained on a childmeter and caring runs and caliberations, the orliberation show Lean the manufactures has been used with the dimensions of the rote sets aster coals to estimate the wate of cherge of flow rote with float position at various float positions (appendix,

p. //o). The following table shows the error in flow rate corresponding to an error of 1 mm in the position of the rotameter float from the desired reading.

TABLE IV. EFFECT UPON FLOW RATE OF ERROR IN POSITION OF ROTALETER FLOAT

FIG. 16

Scale Reading	Nominal Flow Rate, ou om per min (water)	Error in Plow Rate, ou om per mm change in float position	Error, Percent of Flow Rate
3	1.0	0.093	9.3
5	3.7	0.17	4.6
8	10.6	0.21	2.0
10	16.4	0.20	1.2

The third and fourth columns show that although the rotameter is more sensitive at low flow rates, the accuracy of the device for flow measurement is breater at the higher flow rates.

during a run and to achieve reproducibility of float position between runs and calibrations a somewhat unusual spacial relation between float and rotameter markings was chosen as a convention for making readings. The top of the ball float was chosen to represent the float position, and the top of each etch mark on the rotameter glass was chosen as the level represented by that etch mark. With the aid of the needle valve the top of the ball was brought tangent to the top of the etch mark, and the flow rate was considered to be set at the value corresponding to

p. //O). The following table shows the error in flow rate corresponding to an error of 1 mm in the position of the rotanebur float from the desired reading.

MARKE IV. SEPTION UPON 1950 SEPTS OF EMINOR

THE WALL CONTRACTOR OF THE PROPERTY OF A

32 .0 9

nonel Inconer noll lo one	woll at words on the second of	forthmod policy mode modew) nice (nodew) nice	oealo Som³°ny
0.0 0.0 0.0	0.17 0.17 0.00 0.00	1.0 V.5 O.01 A.01	6 6

LEU DE LEGO DE CONTRER COMMENT STOR DELLE SESTION DE PORTES DE PROPERTO DE LE CONTRER DE PORTES DE COMMENTA DE COMPANS DE CONTRER DE CONTRE DE CONTRER DE CONTRER DE CONTRER DE CONTRE DE CONT

Alliano es impiamos el sigles distillado es ser esta es

carded a man and we do diese the selectivity of float confidence confidence to select the selection of the confidence of

the etch mark. Since the etch marks were about 0.2 mm wide, the choice among levels represented by the etch mark was important.

Composition Analysis

The interval in refractive index between mixtures and either of the two pure components extended past the largest interval covered with the smallest interferometer cell. Therefore mixtures of known concentrations were required as standards for the determinations of samples having refractive indexes outside the range covered by the pure components. Although the relation between the refractometer reading and refractive index is linear, the relation between refractive index and composition is not, and unknown compositions could not be determined by simple interpolation of the interferometer reading. To determine the direct relation between interferometer reading and composition, a series of six known concentrations was prepared in the range 0.1 to 0.6, in 0.1 increments of mass fraction. Interferometer readings were determined for successive pairs beginning with distilled water vs. 0.1 ethanol, then 0.1 ethanol vs. 0.2 ethanol, and so on. A reading for distilled water vs. distilled water was used as a zero correction. Cumulative values of readings were plotted on a large graph against composition, a small reproduction of which appears in Fig. 17. To more accurately determine the shape of the curve between

Š

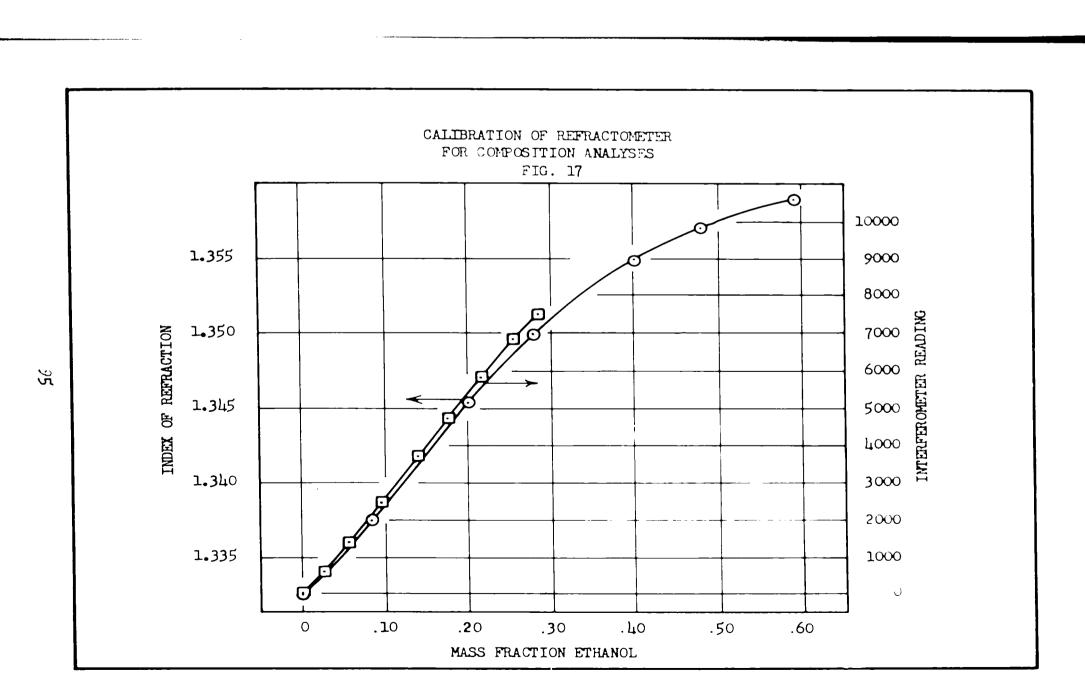
Terror server bear to

The control of the co

Wanter Total Bound

atter thioretent.

Atter the opotes emoni teast representely of the efor work



experimental points, refractive index data (46) for the system were plotted on the same graph. The shape of the reference curve was followed by eye as the calibration points were connected. Components were taken from the same sources used for operation of the still. The known solutions were prepared on the Ohaus balance to 0,1 in composition. The samples, of 50 gm each, were weighed into four ounce bottles having screw caps with polyethylene seals to reduce composition changes through evaporation. It is believed that the composition of samples taken from runs of the apparatus could be determined to the nearest 0.001 mass fraction.

Oxperimental points, refractive index data (40) for the stem were piotted on the scale of the index in the scale of the sc

HEAT AND FLOW RATE DETERMINATION

A test run with air blowing through a tube inverted in a beaker of water showed that a rate of about 9000 oc per min was the greatest rate above which excessive splashing occurred. Since the geometry of the equilibrium flask was similar and splashing of liquid up into the condensate contactor was considered undesirable, this figure was taken as the maximum for design purposes. From that number and physical data of some common solvents, the following table has been prepared:

TABLE V. DESIGN DATA

F10. 18

Compound	CC Vapor rer CC Liquid	$_{\Delta}$ H $_{ m w}$ Cal/Om	Heat Flux Cal/Min at 9000 CC Vapor Per Min	Liquid Flow Hate CC/Min
Ethanol	521	204.3	2780	17.3
Water	1700	540	2850	5.3
Me thanol	723	262.8	2590	12.4
Benzene	344	94.1	2160	26.2
Acetic Acid	59 0	96.7	1550	14.7
Ethyl Ether	256	83.8	2110	35.1

From this calculation it was concluded that the recirculating liquid could be expected to transfer at least 2000 cal per min to the condenser. Heat exchange between the surroundings and the recirculating saturated vapor would produce an error in the 2000 cal per min rate.

so that figure was used as a basis for percentage calculations.

Control of the Contro

grand the grand with the latter was a solution.

and the second of the second o

 Δ

	·	Δ	garage (v. 1800) State (v. 1800)	
i di selata Garage di Selata Garage di Selata	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	e t Le e t		•
timotis Diba Sideos Sandi Ugʻidi	. 6	1 • 1 1 · 1 · 1 • 1	,	1 , A 1 , A 1 ,

rectrousting of the control of the sounding saturated vapor would proceed an error in the 2000 cal por min rate.

so that figure was used as a basis for percentage calculations.

INSULATION OF THE CALORIMETE.

Heat could only be transferred from the water jacket to the surroundings. Assume that the cooling water is at 30 C, that the ambient temperature is 25 C. The inside diameter of the insulation, the outside diameter of the condenser jacket, is 0.75 inches = 0.0625 ft. The length of the insulation is 17.5 inches = 1.46 ft, and the thickness is 3 inches. Assume that the insulation has k = 0.02 Stu/hr-ft-F. For heat transfer through a cylinder,

$$q = \frac{2\pi lk \Delta T}{\ln (r_2/r_1)}$$

 $\Delta T = 5$ degrees (C) = 9 (F).

$$\ln r_2/r_1 = \ln \frac{3.375}{0.375} = \ln 9 = 2.20$$

q = 3.2 cal per min.

Since the expected heat flux through the condenser was about 2000 cal per min, this represents an uncertainty, in the form of a possible error of about $\frac{3.2}{2000}$ x 100 = 0.16%.

INSULATION OF THE CALORIDATA.

neat could only be transferred from the water

jacket to the authorndings, when the the occiling mater is at a subject that the above the confidence is a subject of the character alians of the confidence of the character of the subject of the character of the confidence of the character of the confidence of the character o

$$\frac{1}{\sqrt{3}} \frac{\Delta}{\sqrt{3}} \frac{A(m)}{\sqrt{2}} = 0$$

- 1913日 - 1913日 (2) 1913日 (ANA) ANA(The Franch Committee Commit

when addit C is the property of the second $\frac{3.6}{3.000}$ and the content of the content of

LATENT HEAT CALCULATION

PROM EXPERIMENTAL DATA

The latent heats were determined by a heat balance around the calorimeter, made as follows:

$$m_{\psi}$$
 C_{D} $\Delta T = \Delta H_{\psi} m_{B} + \lambda$

where $m_{W,S}$ = the mass of water and system flowing through the calorimeter in a given time interval,

Cn = specific heat of water,

ΔT = the difference in inlet and outlet water temperatures,

 ΔH = the latent heat,

= the heat leak as described on pg. 89.

The equation may be arranged as follows for the purpose of calculation:

$$\Delta H_{V} = \frac{m_{W}}{m_{S}} \quad C_{D} \quad \Delta T - \frac{\sqrt{2}}{m_{S}}$$

LATENT HEAR CALCULATION

ATAG WAT ALMIN YOUR WITH

The latent beats were detirained by a beat ball of

around but helprineter, unde en follower

 $m_{\rm e} = 0_0 \Delta x = \Delta H_{\rm vec} + 1$

where $x_{n+3}=1$ the maps of sates and syetem files by which records the first state of the first state of

control of the californ was the

, 115 Opt

= w + 1 + v = 1

red of colony of dear to the models of a m

The state of the s

The second second second

 Δ_{i} , Δ_{i} Δ_{i}

SOURCES OF LOSSES IN

THE RECIRCULATING PHASE, RUN 9

Amount of Loss

The level in the leveler was recorded with time as part of the data of all runs. Since the dimensions of the leveler and contactor are known, the change in level may be converted to a change in mass of recirculating phase. Then the level in the leveler was above the 7.5 cm mark, the level could change in both the leveler and the contactor (Fig. 2 and 3). Below that level, only the leveler could drain. The cross-sectional area of liquid above that level was $A_1 = 7r(r_1^2 + r_2^2 - r_3^2 - r_4^2)$, where r_1 and r_2 are the inside diameters of the leveler and contactor respectively, and r_3 and r_4 are the outside diameters of the tube in the leveler and the vapor riser in the contactor respectively.

 $r_1 = r_2 = 1.4$ cm, $r_3 = 0.35$ cm, $r_4 = 0.5$ cm, $A_1 = 11.1$ cm². The cross sectional area of liquid below that level was $A_2 = \gamma_1 (r_1^2 - r_3^2) = 5.78$ cm².

From the data of Run 9, the liquid level dropped faster after the value of 7.6 cm at 11:40 PM so a different height change corresponded to each area.

Drop in height at level over 7.5 is $\frac{8.9 - 7.6 \text{ cm}}{45 \text{ min}} = 0.029 \text{ cm/min}$.

Drop in height at level under 7.5 is $\frac{7.6 - 4.9 \text{ cm}}{30 \text{ min}} = 0.09 \text{ cm/min}$.

Sourchs of Losses in

The RECLICULATING PRASE, AUN 9

amount of Los.

The level in the propositive state three the correct fixed as the fixed art of the constant of the levelet and states of all runs. Since the discrete in laws the levelet and so it checked as the constant of the levelet and the laws the constant of the constant of the levelet and the levelet and the levelet and the constant of the levelet and the constant of the constant of the constant of the levelet and the constant of the c

 $r_1 = r_1 = 1.4$ and $r_2 = 0.50$ and $r_4 = 0.50$ and $r_5 = 1.51$ and the areas sectional area of items, below they later was $h_2 = r_1 (r_1) - r_3^2) = 0.72$ and.

From the data of and i, the lighted and allierant noiont change corresponded to each area.

Drop in height at level under 7.5 is $\frac{3.8 - 7.6 \text{ cm}}{40 \text{ min}} = 0.029 \text{ cm/min}$. Drop in height at level under 7.5 is $\frac{7.6 - 4.8 \text{ cm}}{50 \text{ min}} = 0.09 \text{ cm/min}$.

Rate of loss of mass = 7 r2ha

= (11.1 cm²) (0.029 cm/min) (0.887 gm/cm³) = 0.286 gm/min for a level over 7.5 cm.

 $=(5.78 \text{ cm}^2) (0.09 \text{ cm/min}) (0.887 \text{ gm/cm}^3) = 0.461 \text{ gm/min}$ for a level below 7.5 cm.

The values do not agree because of the inaccuracy of the measurements and the changes in operating variables which took place during the run. This computation gives an average loss of 0.37 gm per min during kun 9.

The condensate formed from heat losses would be at about the composition of the saturated liquid. The saturated liquid in hun 3 was at 0.043 mole fraction ethanol, with a latent heat of 480 cal per gm as determined from the two references (34, 35). The heat transferred to produce this mass loss was (480 cal/gm) (0.37 gm/min) = 178 cal/min = 42 3tu/hr.

Source of neat Leak, hun 9

The depletion was not a result of overflow from the condensate contactor into the flask since the liquid level in the leveler never became high enough to cause overflowing. There are three regions where the loss could have taken place.

Source 1. Expansion of Vapor while Bubbling
Through the Liquid Phase. The drop in pressure through
2 in. of liquid is about 0.069 psi. The Holier chart for
water shows that an idiabatic expansion of saturated steam
produces a superheated vapor, and would result in a tem-

date of loss of mass = Ir rene.

= (11.1 cm^2) (0.029 cm/min) (0.637 gm/om^3) = 0.236 gm/min for a level over 7.5 cm.

= (5.78 cm²) (0.09 cm/min) (0.887 gm/or³) = 0.461 gm/min for a level below σ_{ee}

The values do not the december of the one intends witon deal and the contract of the miles took the contract of the contract o

en la regentation de la company de la compan

The grade of the configuration

And depose that we have not and the state of the control of the co

pource 1. Areansion of vapor of the duption and the service the court of the chart of the chart of the court of the chart for water shows that an idiabatic expansion of saturated steam produces a superheated vapor, and would rosult in a tem-

Although the material undergoing the expansion was only

O.4 mass fraction water, the behavior would be similar

and the expansion would cause no condensation.

Source 2. Condensation on the Wall of the Equilibrium Flask. During the part of Run 9 under consideration, the aluminum pipe surrounding the equilibrium flask was 0.4 to 0.8 degrees (C) above the temperature of the flask. Therefore condensation on the flask wall was not possible, and vaporization was slight.

Vapor hiser Tube of the Condensate Contactor. Vapor at 91.5 C rose within the tube towards the contacting section and condensate at 32 C lay in a pool around the tube. A temperature drop of 10 degrees (C) or 13 (F) existed across the tube wall. For simplicity of computation, the problem has been turned around to ask: What temperature drop would be necessary to transfer the estimated 42 Btu per min across the tube wall, and how well does the calculated drop compare with the actual 13 degree (F) drop?

Symbols and Their Values

h₁ = coefficient for condensing vapor on the inside wall.
 h₂ = coefficient for stagnant liquid with water-ethanol properties.

The thickness of the glass tube is given as 0.1 ± 0.018 cm in the Corning catalogue.

perature drop of 0.5 degree (0) under the present situation. Although the daterial undercoing the expandion was only 0.6 mass fraction water, the behavior would be similar and the expanden would cause to condensation.

Source 2. Condensation on the act of the

entilbring Flack, such the east of unsubmood object - stick from the success of unsubmood object - stick from the success of t

The second of the second secon

Combols and Coir Values

h = ceefficient .c. chaers a ceefficient .c. chaers a ceefficient for stagmant ifouff with vertar-conand properties.

The thickness of the glass tube is given as 0.1 ± 0.018 cm in the Corning catalogue.

 D_2 = outside diameter of the tube = 1.0 cm.

 D_1 = inside diameter of the tube = 0.76 to 0.84 cm.

 $D_2/D_1 = 1.20$ to 1.31; In $(D_2/D_1) = 0.182$ to 0.269

L=H = height of tube immersed in condensate = 1 inch = 0.083 ft.

 $A_m = 7.8 \times 10^{-3} \text{ft}^2$ average.

k = 0.63 Btu/hr-ft-F for glass.

q = 42 Btu/hr.

Vapor composition may be taken as 0.6 ethanol and 0.4 water. The liquid which condensed to form the inside film was 0.1 ethanol and 0.9 water, all mass fractions. The constants are evaluated at 195 F.

kg: k = 0.390 Btu/hr-ft-F for water at 195 F (MoAdams (47), p. 456).

k for ethanol decreases with increasing temperature.

Use $k_f = 0.3$ Btu/hr-ft-F.

 $\rho_{\mathbf{f}}: \rho_{\mathbf{w}} = 60.1 \text{ lbm/ft}^3 \text{ at 195 F.} \text{ (Steam Tables)}$

Use $\rho_f = 60 \text{ lbm/ft}^3$.

 $g = 32.2 \text{ ft/sec}^2.$

$$\lambda = \frac{510 \text{ cal}}{\text{gm}} \frac{\text{Bto}}{252 \text{ cal}} \frac{454 \text{ gm}}{\text{lbm}} = 920 \text{ Btu/lbm}.$$

 $y_f = 0.4$ op (idcAdams, p. 466)

A. What ΔT_g must occur through the glass tube to cause the transfer of 42 Btu/hr?

Do = outside diameter of the tubs = 1.0 om.

 $D_1 = inside$ diameter of the tube = 0.76 to 0.84 em.

02/D1 = 1.20 to 1.31; 14 (Dg/D1) = 0.182 to 0.269

L= H = belyht of tabe fumeredd in sondensate = 1 inch

= 0.083 ft.

 $\lambda_{1} = \pi \, D_{10} = \frac{\pi \, |_{0.8} \, e_{tt}}{R_{s} \, D_{4} \, e_{tt}} = \frac{1 \, 1n_{s}}{1 \, e_{tt}} \frac{1 \, 1n_{s}}{1 \, e_{tt}} \frac{r_{tx}}{1 \, e_{tt}} = 0. \, 49^{x} 10^{-2} \, r_{t}^{2} E_{s}.$ $\lambda_{1} = \pi \, D_{10} = \frac{1 \, 1n_{s}}{R_{s} \, D_{4} \, e_{tt}} \frac{1 \, 1n_{s}}{1 \, e_{tt}} \frac{1 \, 1n_{s}}{1 \, e_{tt}} \frac{r_{tx}}{1 \, e_{tt}} \frac{1}{1 \, e_{tt}} \frac{r_{tx}}{1 \, e_{tt}} = 0. \, 49^{x} 10^{-2} \, r_{t}^{2} E_{s}.$ $\lambda_{1} = \pi \, D_{10} = \frac{1 \, 1n_{s}}{R_{s}} \frac{1 \, 1n_{s}}{1 \, e_{tt}} \frac{1 \, 1n_{s}}{1 \, e_{tt}} \frac{r_{tx}}{1 \, e_{tt}} \frac{1 \, n_{s}}{1 \, e_{tt}} \frac{r_{tx}}{1 \, e_{tt}} \frac{r_$

 $\lambda_{\rm cr} = 7.3 {\rm km}^2 {\rm Mt}^2$ whenever.

ೂರು ೯೮೮ ಕನ್ನೇ ಚ=ದಿ ಕು ಸಾಧಾ ≕ ಷ

.2. \13 = 6

go tara in the election and the metal care out

The transfer most and an armore as were the terminated w

The first bulk arms of the secretary of the second secretary

garder dig Amilia en son in De La grama en en en en en de son de la companya de la seguidad de la companya della companya de la companya della companya dell

. In the observe examples were the constant of the properties $dss_{ij}=0.0000 \, dt/dt/dt$

PF1 PW = 50.1 1 10/11 0 120 1 1 100 = 19 13

. Con μιος του = 39 θατο

R = 1.1. 12/800.

λ = 200 cal 8to | 454 km = 320 Hcu/1bm.

Pr = 0.4 op (descame, p. 466)

A. That $\Delta T_{\rm g}$ must occur through the glass tube to cause the transfer of 42 Btu/hr?

$$\Delta r_{\rm g} = \frac{0 \ln(D_{\rm g}/D_{\rm l})}{2 \pi k l} = \frac{42 \text{ Btu}}{\text{hr}} = \frac{0.168 \text{ to } 0.269 \text{ hr-ft-7}}{0.68 \text{ Btu}}$$

20.083 ft = 23.3 to 34.3 degrees (F), an average of 29 degrees (F). If, as is suspected, the 10 mm tube in the condensate contactor was thinner than the Corning catalog value, then the prediction for Δ Tg would be reduced substantially.

B. What ATil can be calculated for the inside film?

$$h_1 = 0.943 \left[\frac{kf^3 \rho f^2 g \lambda}{\gamma f L (\Delta T_{f1})} \right]^{\frac{1}{4}}$$
 (McAdams, p. 331)

which is to be used in $q = h_1 A_1 \Delta T_{fl}$. These are combined to give $(\Delta T_{fl})^{3/4} = \frac{q}{0.943 A_1 \left[\frac{k_f^3 \beta f^2 g \lambda}{\mu_f L}\right]^4}$

Evaluating the bracketed group gives

0.027 Btu ³ :				<u>-</u> 1	
1488 op-ft-sec	0.083 ft	3600 sec hr	² = (46.	5x ₁₀ 12)	units
= 2610 Btu/hr-					
Substituting,	$(\Delta T_{fl})^{3/4} = -$	42 Btu 0.	943 6.88	10-3 ft ²	-
$\frac{\text{hr-ft}^2 - (F)^{3/4}}{2610 \text{ Btu}}$	$= 2.47 (F)^3$	3/4.			

 $\Delta T_{11} = 3.34$ degrees (F), the temperature drop through the condensing film.

C. The calculation of a suitable film coefficient for the stagnant liquid is difficult. However, a chart in McAdams, Fig 14-15, p. 383, may be used as a guide.

105

 $\frac{q}{1000A} = \frac{48}{1000} = \frac{48}{8.6 \times 10^{-3}} = 4.89$. A value of h = 400 to 800 1s indicated. $\Delta T_{f2} = \frac{q}{hA} = \frac{48}{400 \text{ to } 800} = \frac{8.6 \times 10^{-3}}{8.6 \times 10^{-3}}$

= 6 to 12 degrees (F).

The three temperature drops may be added to get the total estimated drop required to transfer 42 Btu per min. $\Delta T_g + \Delta T_{fl} + \Delta T_{f2} = 29 + 3.3 + 9 = 41.3 \text{ degrees (F)}.$ This calculated value of 41.3 degrees (F) must be compared with the value of 13 degrees (F) taken from the experimental data. Although the values do not compare well, it must be remembered that the heat loss is only a rough estimate. The calculation demonstrates that heat transfer through the vapor riser tube caused the major portion of condensation within the region of the equilibrium flask.

 $2\pi 0.033 \text{ fb}$ = 23.3 to 50.3 degrees (F), an average of 29 degrees (F). If, we is susheated, the 10 mm tube in the condensate contactor was thinner that the contactor cathles value, then the rediction for $\Delta T_{\rm g}$ would be redicted and stanticity.

B. that APri can be calculated for the inside film?

$$\frac{1}{11} = 0.343 \frac{3}{11} \frac{$$

 $\frac{1}{\sqrt{2}} \frac{1}{\sqrt{2}} \frac{1}{\sqrt{2}} \frac{1}{\sqrt{2}} \frac{1}{\sqrt{2}} = \frac{1}{\sqrt{2}} \frac{1}{\sqrt{$

CANNELL OF THE REAL ADMINISTRAL CLASSIFICATION

 $\Delta \Gamma_{\Gamma 1} = 3.59$ lighter (i.e., income and the product with solutions of the condensity of the cond

0. The calculation of a suitable film coefficient for the atagnam liquid is difficult. Accepts, a chart in academs, Fig 14-15, p. 385, may be used as a guide.

1000A = 1000 8.6x10-5 = 4.69. A value of h = 600 to 800 1s indicated.

ATES = 4.69. A value of h = 600 to 800 | 9.6x10-5 |

= 6 to 18 degrees (12).

The caree temperature dryps war to adop to get the total estimated two required to trensfor 40 3th con min. $\Delta T_g + \Delta T_{f_1} + \Delta T_{f_2} = 93 + 2.5 + . = 41.5 \text{ degrees (F).}$ with deformated rates of 41.7 degrees to the conserved after the value of 13 degrees to the total value of 13 degrees to the the conserved after the value of 13 degrees to the the the theorem and the value of 13 degrees to the theorem and the theorem

EXTRA HEAT SOURCES, RUN 8

Adiabatic Wall Heating

There was a differential of 20 degrees (C) across the lower adiabatic wall in Run 8. From the calculation on the adiabatic enclosure, rependix, p. 708, a 0.15 degree (C) Δ T produced an exchange of no more than 0.785 cal per min. $\frac{20 \ (0)}{0.15 \ (C)} \frac{0.785 \ cal}{min} = \frac{104 \ cal/min}{104 \ cal/min}$

Superheat of Entering Vapor

The vapor temperature was 118 C, at the inlet to the equilibrium flask, the liquid temperature 90.5 C. Superheat amounted to 28 degrees (C). The vapor temperature at the outlet of the equilibrium flask was 90.6 C, showing that all the superheat was removed by contact with the liquid. The approximate specific heat of vapor was 0.5 cal per gm-C. The mass flow rate was about 3 gm per min.

$$\frac{3 \text{ gm}}{\text{min}} = \frac{0.5 \text{ cal}}{\text{gm-C}} = \frac{23 \text{ (C)}}{\text{min}} = \frac{42 \text{ cal/min}}{\text{min}}$$

Extra Heat from Both Sources

104 + 42 = 146 cal/min = 35 Btu/hr.

This value is of the same order as the 42 Btu/hr calculated as the loss for hun 9. Therefore the two extra sources of heat were just sufficient to vaporize enough of the liquid to offset the condensation loss at the vapor riser in the condensate collector.

EXTRA HEAT SOURCES, HUN 8

Adiabatic sall heating

There was a differential of to degrees , or across the lower adiabatic rell in the section that act about to another the substitution of the act about to another and the substitution of the substitution of

ouserboar of intering lupor

the equilibrium of the city of the mattheway.

The equilibrium of the city of the mattheway.

The equilibrium of the city of the mattheway of the constitution of the

Extra iona from contactor

104 + 40 = 140 0A1/120 = 30 starter

fals value is of the same of the in the #2 ota/or calculated as the lose for han a. Alarmost the use entra sources of deck were just sufficient to vaporize enough of the liquid to offset the condensation lose at the vapor riser in the condensate collector.

ADIABATIC ENCLOSURE

How much heat will be transferred across the adiabatic walls as a result of error in matching the temperature of the heated wall with that of the equipment inside?

Assume that thermocouples are calibrated to within 0.1 degree (C), that the aluminum tubes can be maintained to within 0.15 degree (C) of the temperature of the glass. Assume that the insulation has k=0.03 Btu per hr-ft-F.

At the Contacting Section

The contacting section is 1.13 inches in diameter and 7 inches long. Four inch aluminum pipe with a inch walls was used for the adiabatic wall. How much heat is lost through a cylinder of insulation 1.13 in. ID and 3.5 in. OD, 7 in. long and with 0.15 degrees (C) drop across it?

$$q = \frac{2\pi k l \Delta T}{\ln(r_2/r_1)} = \frac{2\pi 0.03 \text{ Btu}}{\ln r_1 - \text{ft-F}} = \frac{7 \text{ in}}{12 \text{ in}} = \frac{0.15 \text{ (C)}}{5 \text{ (C)}} = \frac{9 \text{ (F)}}{5 \text{ (C)}}$$

$$\frac{252 \text{ cal}}{8 \text{ tu}} = \frac{100 \text{ min}}{100 \text{ min}} = \frac{0.125}{1.13} = \frac{0.11 \text{ cal/min}}{1.13}$$

$$\ln(3.5/1.13) = \ln 3.10 = 1.13$$

At the Equilibrium Flask

How much heat is lost through insulation around 108

ADIABATIC ENGLOSURE

down much beat will be trensferred across the adiabatic walls as a result of orpor in lace, include temperature of the needed wall rith that of the enteront inside?

ABBRURG COAR COAR COARDOCALISM NOT CONTONATOR TO WELLWithin .1 degree (C), trat coardinate or the artist tailing to a coardinate or the artist tailing to a coardinate or the artist tailing the artist are artist tailing the artist are artist tailing the artist tailing the artist are artist and are artist are

at the ocateorias action

And I thought the second of th

it the Louiltorium Wlask

How much heat is lost through insulation sround

the equilibrium flack kept within 0.15 degrees (C) of the flack temperature? Assume cylindrical insulation 5 in. ID, 6 in. OD, and 3 in. long.

$$q = \frac{0.125 \mid 8 \mid}{7 \mid \ln(6/5)} = 0.785 \text{ cal/min.}$$

This calculation indicates greater heat transfer than was likely because of the thin cylindrical geometry assumed for the calculation.

the equilibrium flask kept within 0.18 degrees (0) of the flask temperature; assume cylindrical insulation 5 in.

1D, 6 in. etc. and 3 in. long.

This calculation indicates of the title ransfer than was likely cooked that a the calculation of the calculation of the calculation.

RRROR IN FLOW RATE

DUE TO MOVEMENT OF ROTAURTER PLOAT

the position in the rotameter float at various float positions, two charts were required. One, the manufacturer's calibration chart, Fig. 19, was used to determine the rate of change in flow rate with the scale reading on the glass tube of the rotameter. Slopes at four scale readings were determined. The second chart, a plot of the scale readings was, the height in mm of each scale reading, Fig. 20, was used to determine the rate of change in scale reading with height. Slopes at the same four points were taken. The products of slopes at corresponding points gave the errors in flow rate corresponding to a one mm change in float position from the set point.

Example:

Scale reading of 5. Flow rate = 3.7 cu cm/min.

Tangents are shown at the scale reading of 5.

From Fig. 19,
$$\frac{10.8 - 3.7}{9.0 - 5.0} = \frac{7.1}{4} = 1.77$$
 ou cm/min per scale reading.

From Fig. 20,
$$\frac{6.7 - 2.8}{55 - 15} = \frac{3.9}{40} = 0.097$$
 scale readings per mm.

$$(1.77)(0.097) = 0.173$$
 cc/min per mm.

$$\frac{0.173}{3.7} (100) = 4.6\%$$

BIUROIN IN PLON KATE

LANGE LETTERS OF SOME BELLEVOR DE SOUL

The figure of the state of the

From the translation of the state of the sta elicent mark to the region of the continue to both the continue to the continu with a least to be as the first that the first and the second of the second o ্ল বুলুলে ও প্ৰাণ তেওঁ বিভাগ and the second s A GO THE STATE OF Commence of the second

of the first of the second of

and the state of t

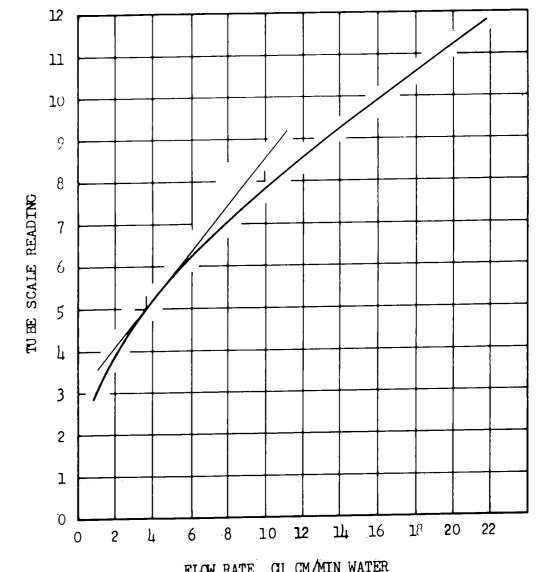
 $\frac{1}{2} \int_{\mathbb{R}^{n}} \frac{dx}{dx} dx = \frac{1}{2} \int_{\mathbb{R}^{n}} \frac{dx}{dx} = \frac{1}{2} \int_{\mathbb{R}^{n}} \frac{dx}{d$

The margheon of which is $=\frac{5.6}{0.0} = \frac{6.5 - 1.0}{10.0}$ (1). 10^{-1}

(1.77)(0.097) = 0.173 co/alo der ma.

$$\frac{0.173}{3.7}$$
 (100) = 4.6%

TUBE - 02F-1/8-12-5 FLOAT - SAPHIRE



FLOW RATE, CU CM/MIN WATER

PRESSURE CHANGE

PROM TOP OF CONDENSAR

TO LIQUID LEVEL 14 EQUILIBRIUE PLASF

Pressure Due to Column of Vapor

The height of vapor was not over 20 inches.

Vapor density of ethanol at 1 atm. is 0.00165 gm/cm³, from the International Critical Tables.

Pure water at 1 atm has a density of 0.0372 lbm/ft³. For computation, use $\rho = 0.1$ lbm/ft³. ressure from the weight of vapor: $\Delta p = \frac{R}{80} \rho h$; $\frac{R}{80} = 1$ lbf/lbm.

$$\Delta p = \frac{0.1 \text{ lbm}}{\text{ft}^3} \frac{1 \text{ lbf}}{\text{lbm}} \frac{20 \text{ in}}{\text{l2 in}} \frac{\text{ft}}{0.0193 \text{ psi}} \frac{1 \text{ ft}^2}{144 \text{ in}^2}$$

 $\Delta p = 0.06 \text{ mm Hg.}$

Error in pressure measurement from this source is negligible.

Pressure Drop Lue to Flow of Vapor

The only substantial restriction to vapor flow is the inlet to the condensate collector. The shape is cylindrical, 0.8 cm diameter, and 3 in. long. D=0.0263 ft; L=0.25 ft.

Area =
$$\frac{\pi D^2}{4} = \frac{\pi 0.0263^2}{4} = 5.44 \times 10^{-4} \text{ ft}^2$$
.

rassons. Mands.

. White A. S. Carlotte

and the second of the second o

to main a media of the superior.

The control of the co

The form of the first of the fi

, the

 $\phi_{ij} = \Delta$

 $\Delta_{i} = \sum_{i=1}^{n} A_{i} = \sum_{i=1}^{n} A_{i}$

The state of the s

en de la companya de Esta companya de la c

.31 . : . 2 = .

1930.0 T = 40 17 = 3017.

The flow rate was about 2 gm/min. The μ for ethanol vapor is 0.0105 cp, for water vapor is 0.0125 cp. Use $\mu = 0.013$ cp. From the previous calculation, $\rho = 0.1$ lbm/ft³.

$$G = \frac{W}{\Lambda} = \frac{2 \text{ gm}}{\text{min}} \frac{1 \text{bm}}{5.44 \times 10^{-4} \text{ ft}^2} \frac{1 \text{bm}}{464 \text{ gm}} \frac{\text{min}}{60 \text{ sec}} = 0.135 \text{ lbm/sec-ft}^2$$

$$V = \frac{0}{8} = \frac{0.135 \text{ lbm}}{800-912} = \frac{1.35 \text{ ft/sec.}}{0.1 \text{ lbm}} = 1.35 \text{ ft/sec.}$$

$$R_0 = \frac{DG}{\mu} = \frac{0.0263 \text{ ft}}{0.013 \text{ cp}} = \frac{0.0263 \text{ ft}}{800 \text{ ft}^2} = \frac{1488 \text{ cp}}{1 \text{ bm}} = 405$$

Flow was laminar. Therefore, eqn. (12-25), p. 322, of McAdams may be used.

$$-\frac{dp}{dL} = \frac{32MV}{g_c} = \frac{32 |0.013 \text{ cp}| 1.35 \text{ ft}}{8ec} \frac{8ec^2 - 1bf}{32.17 \text{ lbm-ft}}$$

$$\frac{|\text{lbm}|}{0.0263^2 \text{ ft}^2} \frac{|\text{in}^2 - \text{atm}|}{1488 \text{ cp-ft-sec}} \frac{\text{ft}^2}{14.7 \text{ lbf}} \frac{|\text{lt}^2|}{144 \text{ in}^2}$$

$$\frac{\text{dp}}{-\frac{\text{dp}}{\text{dl}}} = 8.03^{\text{x}}10^{-6} \text{ atm/ft.}$$

$$-\Delta p = \frac{0.25 \text{ ft} \mid 8.03 \times 10^{-6} \text{ atm}}{\text{ft}} = 2 \times 10^{-6} \text{ atm} = 1.5 \times 10^{-3}$$

The pressure drop from the flowing vapor was negligible.

It can be seen that the pressure at the surface of the liquid in the equilibrium flask was essentially the same as the pressure at the top of the condenser.

The flow rate was about it gn/min. The μ for sthanol vapor is 0.0105 cp, for water vapor is 0.0125 co. the μ = 0.015 cp. From the travelens calculation, ρ = .1 lbm/ft³.

$$v = \frac{0}{800 - 10^{2}} = \frac{100}{100 - 10^{2}} = \frac{0}{100} = v$$

$$= \frac{\text{one } f(t) + 0 + ct}{\text{mol}} = \frac{\text{of } collect}{\text{open}} = \frac{32 + collect}{\text{open}} = \frac{31}{4} = 64$$

អាស្រីសាស្ត្រី ខេត្ត នៅ នេះ នេះ នេះ នេះ ស្រីសាស្ត្រី មានស្រែង ស្រីសាស្ត្រី មានស្រែង និងស្រែង និងស្រែង និងស្រែង

. 48. " ", 3

$$\frac{1}{10}$$

a on the torace of the Mark

PILLING AND SETTING

THE THERMOREGULATOR

The stainless steel bulb was firmly attached to the union and completely filled with carbon tetrachloride. With the bulb in the horizontal position shown in Fig. 7. mercury was added through the union, displacing the carbon tetrachloride until the mercury level reached to about one quarter inch from the top of the union. The plugged end of the bulb was lowered a few angular degrees to permit mercury to displace more carbon tetrachloride so that a level would be finally reached as shown in the figure. Additional mercury was used to bring the mercury level to its original occition. It was felt that if the bulb were kept level from them on, the plug of mercury would prevent the carbon tetrachloride from escaping. The lucite section with the capillary tube was then attached to the union in the same manner as a section of tubing. Mercury was forced up the capillary and over into the well. The union with both tubes was mounted in the water bath, and the temperature adjusted to within 0.1 degree (C) of the set point. If the capillary remained full of mercury, the temperature was raised a few tenths and the excess mercury knocked into the well. Reduction of the temperature to the set point then brought the mercury level into the capillary. If

PHILING AND SETTING

The That son dualton

ine stainiess steel out ass firmly attached to the union on apparent filled of the our on retructionide. "I wi meds o late a lastner the west of company that animary was axion computed the inition, isolated the outbon Bornes Control Control Control Control Control Control Control in dark to look to the top of the staged with the stagged -450 OJ 8097760 THEN I ST TO VE US THE with the to all door now alcoholist acidonide or trat Programme and the second of the second of the of lovel years. The second of the second orion filt the state of the s The VERT CONTROL STANS COME A CONTROL OF THE RESIDENCE OF THE PARTY OF noldoes solded was the an include of the lacity The Moline and the respondence on the second and the second of hearst and more against on a level of a man bout sit. the state of the s -aregras has a first the second of the second Jalou Territoria de la Companya del companya de la companya del companya de la co GTUIRES mot the grant of the and about the state of the said was reised a few contra and the excess mercury incoked into the well, weather of the test weather to the set point then brought the mercury lavel into the earlilary. If

insufficient mercury was in the regulator, additional could be added either by removing the lucite and refilling the union, or by forcing mercury into the capillary with the use of a small long-nosed plastic syringe. When the mercury level had been brought into range at the set point temperature, the aluminum cap and adjustment scrow were placed on the lucite, with the adjustment contact wire running inside the capillary. Air bubbles in the mercury column, introduced when the syringe was used, were removed by running the contact wire up and down the length of the capillary. The set screw on the aluminum cap was used to fit the cap firmly on the lucite. Leads from the two regulator contacts were brought into the jack plug of the controller. Secause of polarity in the controller and the fact that one wire was grounded to the water through the regulator bulb, only one of the two possible wiring arrangements worked.

insufficient moroury was in the regulator, additional could be added without by removing the lucite and refilling the antion, or by foreing meroury into the capitlary with the antion, or by foreing meroury into the capitlary with the creary isval and benefit syringe. Then the errory isval and been controlled being one of the control of the following the point to a great the control of the control of the runtil of the control o

The main the production of the second to see the second testing of the main testing of the second testing of t

··· TOW ISSUED AS MEDICAL COMMON

REPERBNCES

- (1) Ibl. N. V., and Dodge, B. F., Chem. Eng. Science, 2, 120 (1953).
- (2) Dana, L. I., Amer. Acad. Arts and Sciences, Proc., <u>60</u>, 241 (1925).
- (3) Tallmadge, J. A., Schroeder, D. W., Edmister, W. C., and Canjar, L. N., Chem. Eng. Prog. Symposium Ser., Vol. 50, No. 10 (1954).
- (4) Weissberger, A., Physical Methods of Organic Chemistry, Vol. 1, 2nd Ed. Interscience Publishers, 1949.
- (5) McCracken, P. G. and Smith, J. M., Journal Amer. Inst. Chem. Eng., 2, 498 (1956).
- (6) Callendar, H. L. and Barnes, H. T., Phil. Trans. Royal Soc., Alsy, 55, 149 (1902).
- (7) Rosanoff, M. A., Bacon, C. W., and White, R. H., J. Amer. Chem. Soc., 36, 1803 (1914).
- (8) Cornell, L. a. and Montonna, R. L., Ind. Eng. Chem., 25, 1331 (1933).
- (9) Fowler, R. T., Ind. Chemist and Chem. Mfgr. 24, 717, 824 (1948).
- (10) Ridgway, K., Ibid., 32, 59 (1956).
- (11) Jones, C. A., Schoenborn, E. M., and Colburn, A. P., Ind. Eng. Chem., 35, 666 (1943).
- (12) Murti, P. S., PhD. Dissertation, University of Texas (1956).

HEP LHENGES

- (1) Ibl. N. V., and Lodge, B. F., Chem. Eng. Science, 2, 125 (1353).
- (f) Jame, L. I., Amer. Acad. Arts and Sciences, Proc., <u>60</u>, 141 (1085).
- (3) is imaches, ..., ..., schroeder, ..., Edulater, a. C., and Janier, ..., chan, ..., chan, ..., cress, symposius Ser., Vol. 30, i.e., it (1954).
- ve, attendar, e., rynicul sethòda of organic Chemistry, vol. i. for ad. interaciones unitabers, 1940.
- - ر بروهها باز و دور المحادي ال
- - (9) .caler, ..., ind. oligadat en olig. fgm. 24, 717, 226 (1945).
 - (10) Midgray, ... (bic., 32, 55 (1956).
 - (11) denus, v. ..., sencerosen, v. b., and Selburn, A. P., Ind. ang. shem., 35, 666 (1943).
 - (12) Murtl, .. 2., PhD. Dissertation, University of Texas (1956).

- (15) Murti, P. S., and Van Winkle, M., Journal Amer. Inst. Chem. Eng., 5, 517 (1957).
- (14) Othmer, D. F., Ind. Eng. Chem., 20, 743-6 (1928).
- (15) Othmor, D. F., Ind. ang. Chem., Anal. Ed., 4, 232 (1932).
- (16) Othmor, D. F., Anal. Chem., 20, 763 (1948).
- (17) Swientoslawski, Ebulliometric Measurements, Rheinhold Publishing Corporation, 1945.
- (18) Chilton, T. H., Proc. 4th Symposium, Chem. Eng. Educ., 68 (1935).
- (19) Scatchard, G., Maymond, C. B., and Hilmann, R. R., J. Amer. Chem. Soc., 60, 1275 (1938).
- (20) Moyes, A. A. and Marfle, A. R., Ibid., 23, 463 (1901).
- (21) Smith, V. C., Patent C. S. 2,816,064. Dec. 10, 1957.
- (22) Griswold, J., Andres, D., and Klein, V. A., 2et. Hefiner 22, No. 6 (1943).
- (23) Othmer, D. F., Moeller, W. P., Englund, S. W., and Christopher, R. G., Ind. Eng. Chem. 43, 707 (1951).
- (24) Chao, K. C. and Hougen, C. A., Chem. Lng. Science 7, 246 (1958).
- (25) Yee, J. Y. and Davis, h. O., Ind. Ing. Chem., Anal. Ed. 8, 477 (1936).
- (26) Weber, R. L., Temperature Measurement and Control, Chap.
 IX, 1941.
- (27) Scott, R. B. and Brickwedde, F. G., Bureau of Stds.

 Journal of Research, Vol. 6 (1931). RP 284.
- (28) American Institute of Physics, Temperature -- Its Measurement and Control in Science and Industry.

- (13) Eurti, r. S., and Van sinkle, M., Journal Amer. Inst. Chem. Sng., 3, 517 (1957).
 - (14) othmer, D. F., Ind. Lng. Chom., 20, 743-6 (1928).
- (13) otnoer, J. E., Ind. ung. Chem., Anal. ed., 4, 232 (1932).
 - (18) others, D. J., andl. Chem., 20, 763 (1948).
 - (17) owientoglawail, conflictor commonts, Weinhold amblishing Jordenston, 16sh.
- caller to the first of the first of the state of the stat

 - A Common of the second of the
 - (10) May be a more at the second of the factor of the second of the seco
- (23) American Institute of Physics, temperature--its Messurement and Control in Science and Industry.

- (29) Heisig, G. B. and Gernes, D. C., Ind. Eng. Chem., Anal. Ed. 6, 155 (1934).
- (30) Forziati, A. F., Beveridge, J. M., and Rossini, P. D., Bureau of Standards Journal of Research, Vol. 35, RP 1685.
- (31) Ansley, A., Temperature Control. London, Chapman and Hall. 1942.
- (32) Altsheler, W. B., Unger, E. D., and Kolachov, P., Ind. Eng. Chem. 43, 2589-64 (1951).
- (33) Perry, J. H., Chem. Eng. Handbook, Third Edition, 1950.
- (34) Bosnjakovic, F., Technische Thermodynamic. T. Steinkopff, Leipzig, 1935. Supplementary Charts.
- (35) Smith, D. A., Auong, J., Brown, G. G., and White, R. R., Pet. Ref. 24, No. 8, 296 (1945).
- (36) Utsuki, A. and Williams, F. C., Chem. ng. Prog., Symposium Ser. 49 No. 6, Phase Equilibria -- Collected Research Papers for 1953, 55 (1953).
- (37) Carey and Lewis, Ind. Eng. Chem. 24, 382 (1932).
- (38) Evans, r. N., Ind. Eng. Chem. B, 260 (1916).
- (39) Brown, I., Australian Journal of Scientific Research 5, 530 (1952).
- (40) Brown, I., and Ewald, A. H., Australian Journal of Scientific Research, Ser. A. Physical Science, Vol. 3-4, 306 (1950-1).
- (41) Hipkin, H. G. and Myers, H. S., Ind. Eng. Chem. 46, 2524 (1954).

- (29) Heisig, G. B. and Gernes, D. C., Ind. Eng. Chem., Anal. Ed. 6, 158 (1954).
- (30) Forsiati, A. P., Severidge, J. M., and Rossini, P. D., Suresu of Standards cournal of Research, Vol. 35, RP 1685.
- (31) Antley, A., Jempersture Control. London, Chapman and Pall. 1342.
- (32) Alteneter, d. B., Unger, u. D., and acidadov, P., Ind. ang. Chem. 43, 255-64 (1951).
- (t) corry d. t., Onem, lane, tandbook, lared addition, 1950.
- ca) cartakovio, .. 'somiso's incresciptatio, 1. Steinkopff, _05 1/1, 1450. Capishostari Sharts.
- - - with Carey and extend when the new we will be added
 - (3161) was a second of the same of the same (1316).
 - .59 proved 1.3 sustraited courns. Contentific Research 5, 500 (1.52).

 - (41) Alpkin, m. G. and Myers, H. D., Ind. ong. Chem. 46, 2624 (1954).

- (42) Beebe, and Coulter, Ind. Eng. Chem. 34, 1501 (1942).
- (48) Dodge, B. F., Chemical Engineering Thermodynamics, p. 400-1.
- (44) Scatchard, G., Tichnor, L. B., Goates, J. R., and McCartney, E. R., J. Am. Chem. Soc. 74, 3721 (1952).
- (45) Handbook of Chemistry and Physics, 35th Ldition.
- (46) International Critical Tables, 1st Ed., Vol. 7, p. 67, 92.
- (47) McAdams, A. H., Heat Transmission, 3rd Ed., McGraw Hill, 1954.