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Efficiency of chemical desiccants

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EFFICIENCY OF CHEMICAL DESICCANTS

by

Fred Charles Trusell

**A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of
The Requirements for the Degree of
DOCTOR OF PHILOSOPHY**

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Approved:

Signature was redacted for privacy.

In Charge of Major Work

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INTRODUCTION

"More attention should be paid to the choice of a desiccant and its condition than is usually given by the analyst, not only when the desiccant is to be used in absorption chains as in the determination of water or of carbon dioxide, but also for general drying purposes, as in desiccators."(14) When confronted with the problem of removing water from a gas the chemist has a wide range of desiccants from which to choose. Indeed, the real question becomes just how dry one wants the gas. All too often, however, the choice comes from habit: calcium chloride for general use, anhydrous magnesium perchlorate for greater drying power, and phosphorus pentoxide when the ultimate is needed.

As early as 1862 Pettenkofer (20), in a study of water in respiration and perspiration, established that calcium chloride did not quantitatively remove the water from a gas stream passing over it. Thirteen years later Laspeyres (16) showed that while calcium chloride would remove excess water from an air stream, hydrated calcium chloride would give up water to a dry air stream. A year later Dibbits (7) showed that the drying efficiency of calcium chloride decreased with an increase in temperature.

In 1865 Fresenius (10) determined the drying efficiency of anhydrous calcium chloride, calcium chloride containing 21% water, calcium oxide, and anhydrous copper sulfate, all relative to concentrated sulfuric acid. He also determined the efficiency of concentrated sulfuric acid relative to phosphorus pentoxide. The values which he found for the residual moisture left by each desiccant are, without exception, much higher than those found in later investigations.

In 1887 Morley, in a very long and tedious experiment, determined the weight of water plus phosphorus pentoxide which escaped in an air stream dried over phosphorus pentoxide. This was done by "drying the gas with phosphorus pentoxide, and then passing it through a weighed apparatus in which the gas was first slightly moistened, then much expanded, and lastly again dried by phosphorus pentoxide. If the weight of the apparatus decreases, the loss is due to the moisture left by the phosphorus pentoxide in that volume by which the gas passing out of the apparatus exceeds the gas entering it" (18). In a later work (19) he showed that in 900 liters of air, two hundredths of a milligram of phosphorus pentoxide escaped, almost exactly the same as the total weight of water plus phosphorus pentoxide. This lead him to state, ". . . no gravimetric experiments which the scientific world has in hand at present would need to take account of the moisture which phosphorus pentoxide leaves in a gas."

Beginning in 1911 Baxter undertook to evaluate several drying agents. In that year Baxter and Warren determined the moisture content of air dried over calcium bromide, zinc bromide, zinc chloride, calcium chloride, and sulfuric acid, all relative to phosphorus pentoxide (3). This work marks the beginning of a trend away from the practice of Morley of using large volumes of air for such determinations. In their investigations Baxter and Warren used 7.4 liters of air or less, with flow rates of one to four liters per hour. In 1916 Baxter and Starkweather evaluated sodium and potassium hydroxides, and re-evaluated calcium chloride. Again, small volumes of air were used (2).

The following year Dover and Marden investigated the efficiencies of several drying agents, but a uniform temperature was not employed, and some of the data given are extrapolated values obtained at temperatures other than those indicated (8).

The introduction of calcium chloride, sulfuric acid, and phosphorus pentoxide as drying agents seems to be lost in antiquity. In the years following 1912 several new desiccants were introduced: alumina by Johnson (15), anhydrous magnesium perchlorate by Willard and Smith (31), anhydrous barium perchlorate by Smith (23), boric acid anhydride by Walton and Rosenblum (29), barium oxide by Booth and McIntyre

and calcium sulfate by Hammond and Withrow (13).

In 1934 Bower published the most complete and critical survey of drying agents undertaken to that time, making all of his comparisons relative to phosphorus pentoxide (5). Bower returned to the tradition of Morley by using large volumes of air for each determination. In 1944 Bower re-examined three of the drying agents previously tested, alumina, silica gel, and anhydrous magnesium perchlorate. The amount of water remaining in air dried by the first two was revised downward, while that of the third remained unchanged (6).

Bower employed phosphorus pentoxide as his ultimate standard, basing his choice on the earlier work of Morley. However, all of his determinations were not made directly against phosphorus pentoxide. Frequently the determination was made against a secondary standard and a factor was added to correct for the efficiency of the secondary standard relative to phosphorus pentoxide. This makes possible a compounding of errors, and is a questionable technique to use in a critical study. Bower claimed that at the low flow rate used his values represent, for all practical purposes, equilibrium conditions. The fact that phosphorus pentoxide will not dry magnesium perchlorate below the trihydrate, even over a period of four months (31), lays this claim, and,

indeed, the validity of the entire determination, open to question.

In the light of the foregoing it was decided that a new study should be made to re-examine the efficiency of chemical desiccants. The mode of attack chosen was to pass humidified nitrogen over the desiccants and to trap the water which escaped in a cold trap immersed in liquid nitrogen. In this manner the weight of water escaping can be found. All determinations were made directly against liquid nitrogen, no secondary standards being employed. It can be shown that at equilibrium at the temperature of liquid nitrogen there is but one molecule of water vapor for each 1800 liters of gas (30). Even if equilibrium was missed by several orders of magnitude, a weighable amount of water would not escape this trap.

In recent years the field of drying agents has been clouded, as have several other fields, by the introduction of common names by the manufacturers of these products. The common names, along with the chemical formulae or compositions, are given in Table 1.

Table 1. Common names and chemical formulae or composition of desiccants

Common name	Chemical formula or composition
Anhydrocel	CaSO_4 prepared by dehydration of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$
Anhydrone	$\text{Mg}(\text{ClO}_4)_2$, variable amounts of water, 0.5 - 1.5 H_2O
Ascarite	90% NaOH, 10% asbestos
Desicchlora	$\text{Ba}(\text{ClO}_4)_2$
Drierite	CaSO_4
Hydralo	Al_2O_3
Mikohbite	68% NaOH, 32% fluffed mica
Molecular sieves	Synthetic zeolites

MATERIALS AND EQUIPMENT

The following materials and equipment were used in this work:

Alumina

Alumina was prepared by heating Baker and Adamson aluminum hydroxide powder to 400^o in a muffle furnace.

Anhydrocel

Anhydrocel was obtained from the G. Frederick Smith Chemical Company.

Anhydrone

Anhydrone was obtained from the G. Frederick Smith Chemical Company.

Ascarite

Ascarite was obtained from the Arthur H. Thomas Company.

Barium oxide

Porous barium oxide, carbide free, was obtained from Barium and Chemicals, Inc.

Barium perchlorate, anhydrous

Barium perchlorate, anhydrous, was prepared by vacuum drying J. T. Baker Desicchlora overnight at 127^o.

Calcium chloride

J. T. Baker calcium chloride, anhydrous, granular, eight mesh was used.

Calcium chloride, anhydrous

Calcium chloride was vacuum dried overnight at 127^o.

Calcium chloride, anhydrous, dried at high temperature

Calcium chloride was vacuum dried overnight at 245°.

Calcium oxide

J. T. Baker calcium carbonate was ignited six hours in a muffle furnace at 900°.

Drierite

Drierite was obtained from the W. A. Hammond Drierite Company.

Lithium perchlorate, anhydrous

Lithium perchlorate was prepared in this laboratory.

It was vacuum dried for 12 hours at 70° and for an additional 12 hours at 110°.

Magnesium oxide

Magnesium oxide was prepared by igniting Fischer Scientific Company magnesium carbonate in a muffle furnace for six hours at 800°.

Magnesium perchlorate, anhydrous

Anhydrous was vacuum dried for 48 hours at 245°.

Magnesium perchlorate, anhydrous, indicating

Magnesium perchlorate, anhydrous, indicating, was obtained from the G. Frederick Smith Chemical Company.

Magnesium sulfate, heptahydrate

J. T. Baker magnesium sulfate, heptahydrate, crystal, was used.

Mikohbite

Mikohbite was obtained from the G. Frederick Smith Chemical Company.

Molecular Sieve, 5A

Molecular Sieve, 5A, 1/16 inch pellets, was obtained from the Linde Company.

Phosphorus pentoxide

Baker and Adamson phosphorus pentoxide was used.

Potassium hydroxide

J. T. Baker potassium hydroxide pellets were used.

Silica gel

Silica gel was obtained from the Eagle Chemical Company, Inc.

Sodium hydroxide

Fischer Scientific Company sodium hydroxide pellets were used.

Moisture monitor

A Consolidated Electrodynamics Corporation Moisture Monitor, Type 26-302, was used.

Wet test meter

Precision Scientific Company wet test meters were used to measure the gas flow. The meters were calibrated before use.

EXPERIMENTAL PROCEDURES AND DATA

Gravimetric Determinations of Residual Moisture

Description of apparatus

The absorption train used in the gravimetric determinations in this work is shown in Figure 1. Compressed nitrogen in tank A supplied the carrier gas. Tube B was made of 10 mm borosilicate glass and was immersed in a Dewar flask of liquid nitrogen. At this point any contaminants in the carrier gas which will condense out at -196° are trapped. C was a distributor connection having one inlet and three outlets. Each outlet was fitted with a stopcock, permitting the flow rate of each stream to be regulated. From this point on there were three separate but parallel trains, permitting three determinations to be run simultaneously. Tubes D, E, and F were Kimax number 46050 Schwartz drying tubes fitted at the inlet end with a 12/5 ground glass socket and at the outlet end with a 12/5 ground glass ball. They were immersed in a 25° water bath. Tube D was filled with magnesium sulfate, heptahydrate, to feed a small amount of water back into the carrier gas. In the runs on Anhydrol, Ascarite, anhydrous barium perchlorate, calcium chloride, anhydrous calcium chloride, anhydrous calcium chloride dried at high temperature, calcium oxide, Drierite, magnesium oxide, Mikohbite, potassium hydroxide, and sodium

hydroxide, tube E was empty. During all other runs it contained calcium chloride as a preliminary desiccant. Tube F contained the drying agent under test. In cases where the desiccant was pre-treated by ignition or vacuum drying, it was loaded into tube F while still warm. A Pyrex wool plug was inserted on the outlet side of these tubes to prevent mechanical carry over. Tube G was immersed in liquid nitrogen during a determination to freeze out all of the water in the carrier gas which was not removed by the drying agent in tube F. A 10 mm borosilicate tube, H, containing Anhydron, prevented diffusion of water vapor back into the system. The volume of gas used is measured by the wet test meter, I. All ground glass joints, with the exception of the ball and socket joints on tube G, were lubricated with Apiezon N grease.

Before starting a determination the tubes were loaded as specified above, and six to ten liters of nitrogen were passed through the system to sweep it out. During this time the flow rate was regulated to approximately 225 ml per minute. Tube G was then taken to a 25° room for weighing. The relative humidity of this room was between 50 and 60 percent. Before removal from the train the inlet stopcock was closed, but the outlet stopcock was left open and allowed to remain open for three minutes. This permitted any oxygen which had condensed out to boil off harmlessly and escape to the atmos-

phere. The outlet stopcock was then closed. After one hour tube G was weighed to the nearest 0.1 mg, using an identical tube as a counterpoise. Before weighing the outlet stopcock was briefly opened to permit the pressure within the tube to become equal to the atmospheric pressure. Weighings were made at 30 minute intervals until two successive weighings agreed within 0.1 mg. Generally, two weighings sufficed. This gave the initial weight of tube G.

Tube G was then placed back into the train, and the determination was begun. Once a determination was started it was allowed to proceed without interruption. At the completion of a determination tube G was weighed in a manner identical to that described above. The gain in weight represented the amount of water not removed by the desiccant in tube F.

It was the original intention to use air as the carrier gas, pulling it through the absorption train by applying a slight vacuum at the outlet end. However, it was found that oxygen, boiling some 13° higher than nitrogen, was condensing out in excessive quantities. This indicates that the carrier gas was cooled to at least -183° as it passed through tube G. At this temperature the equilibrium vapor pressure of water over ice was calculated to be 2.3×10^{-19} mm Hg (30). Thus, in a determination using a total volume of 1000 l of carrier gas, not more than 4×10^{-16} g of water escaped detection.

Analysis of desiccants studied

When practical, the desiccants used were analyzed to determine their purity. Then, as an approximation, it was assumed that all of the impurities were water, and the degree of hydration was calculated. The original material was analyzed, as was a portion from the inlet side and the outlet side of the absorption tube F. From these analyses it can be shown that there was still unexhausted desiccant on the outlet side of the tube. These results, and the results of the individual determinations of residual moisture in the gas stream, are given in the tables at the end of this section.

With one exception, the desiccants which were salts of alkaline earth metals were analyzed by titrating the alkaline earth metal ion with (ethylenedinitrilo)tetraacetic acid at pH 10, employing Eriochrome Black T as the indicator. The single exception to this was the analysis of calcium sulfate (Anhydrocel and Drierite). In this case the weighed sample was stirred overnight in a beaker with a slurry of 10 g of wet Amberlite IR-120 cation exchange resin in the hydrogen form. The resulting sulfuric acid was then titrated with standard sodium hydroxide.

In the case of magnesium perchlorate, anhydrous, indicating, the potassium permanganate was reduced with hydroxyl-

ammonium chloride, and the total of manganese and magnesium was determined by titrating at pH 10 with (ethylenedinitrilo)-tetraacetic acid. The endpoint was markedly sharper if the solution was heated to about 40°. The manganese was determined colorimetrically as the permanganate in an aliquot of the same sample. The magnesium was found by difference.

Sodium and potassium hydroxides were analyzed by titration with standard hydrochloric acid.

Ascarite and Mikohbite were treated with hydrochloric acid and allowed to stand for an hour, with frequent swirling. The excess acid was titrated with sodium hydroxide.

Alumina was dissolved by refluxing for 20 minutes in concentrated perchloric acid. The perchloric acid was neutralized with ammonium hydroxide, excess (ethylenedinitrilo)tetraacetic acid was added, and the solution was warmed briefly. The excess (ethylenedinitrilo)tetraacetic acid was then titrated with copper nitrate, employing Calcein as the indicator. The end-point was detected by the quenching of the fluorescence of the indicator in ultraviolet illumination.

The results of the determination of the water vapor remaining in nitrogen after drying over the desiccants tested are summarized in Table 43. The results of individual determinations, along with the analyses of the desiccants, are shown in Tables 2 through 42.

Figure 1. Absorption train used in gravimetric determinations of residual water in nitrogen dried over desiccants

- A. Compressed nitrogen
- B. Preliminary freeze-out tube in liquid nitrogen
- C. Three-outlet distributor
- D. Magnesium sulfate, heptahydrate
- E. Calcium chloride (when used) in 25° water bath
- F. Desiccant under test
- G. Freeze-out tube in liquid nitrogen
- H. Anhydrone safety tube
- I. Wet test meter

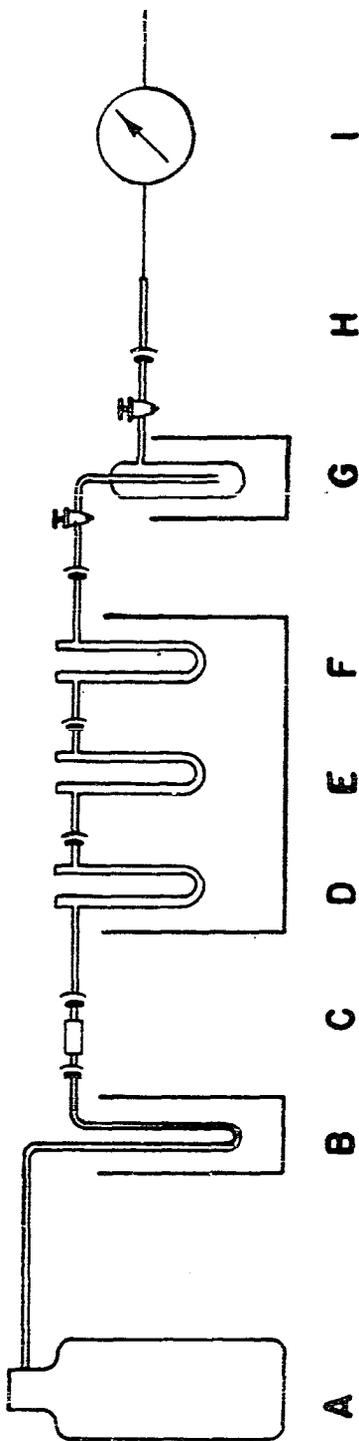


Table 2. Determination of water vapor remaining in nitrogen dried over alumina

	Run 1	Run 2	Run 3
Final weight	0.1989 g	0.4592 g	0.5273 g
Initial weight	0.1982 g	0.4585 g	0.5264 g
Weight gain	0.0007 g	0.0007 g	0.0009 g
Volume	267 l	251 l	275 l
Time	19 hr, 40 min	19 hr, 40 min	19 hr, 40 min
Flow rate	226 ml/min	213 ml/min	233 ml/min
Residual water	2.6 $\mu\text{g/l}$	2.8 $\mu\text{g/l}$	3.3 $\mu\text{g/l}$
Average residual water	2.9 $\mu\text{g/l}$		

Table 3. Analysis of alumina

	Original material	Inlet of tube F	Outlet of tube F
Ml of EDTA	50.00 ml	50.00 ml	50.00 ml
Normality of EDTA	0.1000 N	0.1000 N	0.1000 N
Ml of copper nitrate	13.44 ml	30.30 ml	27.72 ml
Normality of copper nitrate	0.0966 N	0.0966 N	0.0966 N
Sample weight	0.1889 g	0.1192 g	0.1192 g
Molecular weight	102.0	115.0	102.7
Corresponding formula	Al_2O_3	$\text{Al}_2\text{O}_3 \cdot 0.73 \text{H}_2\text{O}$	$\text{Al}_2\text{O}_3 \cdot 0.04 \text{H}_2\text{O}$

Table 4. Determination of water vapor remaining in nitrogen dried over Anhydrocel

	Run 1	Run 2	Run 3
Final weight	9.4047 g	9.5036 g	9.5614 g
Initial weight	9.2550 g	9.3585 g	9.4301 g
Weight gain	0.1497 g	0.1451 g	0.1313 g
Volume	720 l	692 l	646 l
Time	52 hr, 12 min	52 hr, 12 min	52 hr, 12 min
Flow rate	230 ml/min	221 ml/min	206 ml/min
Residual water	208 $\mu\text{g/l}$	210 $\mu\text{g/l}$	204 $\mu\text{g/l}$
Average residual water	207 $\mu\text{g/l}$		

Table 5. Analysis of Anhydrocel

	Original material	Inlet of tube F	Outlet of tube F
Ml sodium hydroxide	14.48 ml	24.17 ml	18.91 ml
Normality of sodium hydroxide	0.1163 N	0.1163 N	0.1163 N
Sample weight	0.1178 g	0.2038 g	0.1538 g
Equivalent weight	69.96	72.51	69.94
Molecular weight	139.9	145.0	139.9
Corresponding formula	$\text{CaSO}_4 \cdot 0.21 \text{H}_2\text{O}$	$\text{CaSO}_4 \cdot 0.49 \text{H}_2\text{O}$	$\text{CaSO}_4 \cdot 0.21 \text{H}_2\text{O}$

Table 6. Determination of water vapor remaining in nitrogen dried over Anhydrone

	Run 1	Run 2	Run 3
Final weight	9.2573 g	9.3632 g	9.4320 g
Initial weight	9.2557 g	9.3615 g	9.4303 g
Weight gain	0.0016 g	0.0017 g	0.0018 g
Volume	1258 l	1056 l	1177 l
Time	90 hr, 21 min	90 hr, 21 min	90 hr, 21 min
Flow rate	233 ml/min	195 ml/min	217 ml/min
Residual water	1.3 $\mu\text{g}/\text{l}$	1.6 $\mu\text{g}/\text{l}$	1.5 $\mu\text{g}/\text{l}$
Average residual water	1.5 $\mu\text{g}/\text{l}$		

Table 7. Analysis of Anhydrone

	Original material	Inlet of tube F	Outlet of tube F
Ml of EDTA	28.56 ml	29.00 ml	28.47 ml
Normality of EDTA	0.1005 N	0.1005 N	0.1005 N
Sample weight	0.7169 g	0.7791 g	0.7146 g
Equivalent weight	249.8	267.3	251.5
Corresponding formula	$\text{Mg}(\text{ClO}_4)_2 \cdot 1.48 \text{H}_2\text{O}$	$\text{Mg}(\text{ClO}_4)_2 \cdot 2.45 \text{H}_2\text{O}$	$\text{Mg}(\text{ClO}_4)_2 \cdot 1.53 \text{H}_2\text{O}$

Table 8. Determination of water vapor remaining in nitrogen dried over Ascarite

	Run 1	Run 2	Run 3
Final weight	9.1033 g	9.3630 g	9.4312 g
Initial weight	9.0995 g	9.3594 g	9.4271 g
Weight gain	0.0040 g	0.0040 g	0.0040 g
Volume	43 l	43 l	44 l
Time	3 hr, 13 min	2 hr, 13 min	3 hr, 13 min
Flow rate	228 ml/min	228 ml/min	223 ml/min
Residual water	93 $\mu\text{g/l}$	93 $\mu\text{g/l}$	93 $\mu\text{g/l}$
Average residual water	93 $\mu\text{g/l}$		

Table 9. Analysis of Ascarite

	Original material	Inlet of tube F	Outlet of tube F
Ml of hydrochloric acid	50.00 ml	50.00 ml	50.00 ml
Normality of hydrochloric acid	0.0969 N	0.0969 N	0.0969 N
Ml of sodium hydroxide	9.43 ml	20.33 ml	13.79 ml
Normality of sodium hydroxide	0.1050 N	0.1050 N	0.1050 N
Sample weight	0.1515 g	0.1312 g	0.1711 g
Percent sodium hydroxide	91.0 %	82.3 %	90.2 %

Table 10. Determination of water vapor remaining in nitrogen dried over barium oxide

	Run 1	Run 2	Run 3
Final weight	9.0983 g	9.3581 g	9.4261 g
Initial weight	9.0975 g	9.3574 g	9.4255 g
Weight gain	0.0008 g	0.0007 g	0.0006 g
Volume	254 l	233 l	249 l
Time	18 hr, 30 min	18 hr, 30 min	18 hr, 30 min
Flow rate	229 ml/min	210 ml/min	224 ml/min
Residual water	3.1 $\mu\text{g/l}$	3.0 $\mu\text{g/l}$	2.4 $\mu\text{g/l}$
Average residual water	2.8 $\mu\text{g/l}$		

Table 11. Analysis of barium oxide

	Original material	Inlet of tube F	Outlet of tube F
Ml of EDTA	36.71 ml	30.36 ml	35.41 ml
Normality of EDTA	0.0998 N	0.0998 N	0.0998 N
Sample weight	0.5834 g	0.5166 g	0.5591 g
Molecular weight	159.2	170.5	158.2
Percent barium oxide	96.2 %	89.8 %	96.6 %

Table 12. Determination of water vapor remaining in nitrogen dried over barium perchlorate, anhydrous

	Run 1	Run 2	Run 3
Final weight	9.0811 g	9.0781 g	9.0942 g
Initial weight	9.0648 g	9.0614 g	0.0781 g
Weight gain	0.0163 g	0.0167 g	0.0161 g
Volume	27 l	28 l	27 l
Time	2 hr, 3 min	2 hr, 3 min	2 hr, 3 min
Flow rate	220 ml/min	221 ml/min	220 ml/min
Residual water	604 µg/l	597 µg/l	596 µg/l
Average residual water	599 µg/l		

Table 13. Analysis of barium perchlorate, anhydrous

	Original material	Inlet of tube F	Outlet of tube F
Ml of EDTA	23.15 ml	19.10 ml	17.74 ml
Normality of EDTA	0.1005 N	0.1005 N	0.1005 N
Sample weight	0.7812 g	0.7108 g	0.5996 g
Equivalent weight	335.8	372.0	336.3
Corresponding formula	Ba(ClO ₄) ₂	Ba(ClO ₄) ₂ ·2H ₂ O	Ba(ClO ₄) ₂

Table 14. Determination of water vapor remaining in nitrogen dried over calcium chloride

	Run 1	Run 2	Run 3
Final weight	9.0540 g	9.0597 g	9.0653 g
Initial weight	9.0484 g	9.0540 g	9.0597 g
Weight gain	0.0056 g	0.0057 g	0.0056 g
Volume	57 l	57 l	57 l
Time	4 hr, 16 min	4 hr, 12 min	4 hr, 13 min
Flow rate	222 ml/min	226 ml/min	225 ml/min
Residual water	98 µg/l	100 µg/l	98 µg/l
Average residual water	99 µg/l		

Table 15. Analysis of calcium chloride

	Original material	Inlet of tube F	Outlet of tube F
Ml of EDTA	37.85 ml	41.10 ml	41.15 ml
Normality of EDTA	0.01005 N	0.01005 N	0.01005 N
Sample weight	0.04410 g	0.05615 g	0.04782 g
Equivalent weight	116.1	132.8	115.8
Corresponding formula	CaCl ₂ •0.28 H ₂ O	CaCl ₂ •1.21 H ₂ O	CaCl ₂ •0.23 H ₂ O

Table 16. Determination of water vapor remaining in nitrogen dried over calcium chloride, anhydrous

	Run 1	Run 2	Run 3
Final weight	9.0571 g	9.0591 g	9.0611 g
Initial weight	9.0547 g	9.0571 g	9.0591 g
Weight gain	0.0024 g	0.0020 g	0.0020 g
Volume	36 l	30 l	30 l
Time	2 hr, 31 min	2 hr, 10 min	2 hr, 12 min
Flow rate	231 ml/min	231 ml/min	227 ml/min
Residual water	67 $\mu\text{g/l}$	67 $\mu\text{g/l}$	67 $\mu\text{g/l}$
Average residual water	67 $\mu\text{g/l}$		

Table 17. Analysis of calcium chloride, anhydrous

	Original material	Inlet of tube F	Outlet of tube F
Ml of EDTA	39.64 ml	23.84 ml	36.61 ml
Normality of EDTA	0.1005 N	0.1005 N	0.1005 N
Sample weight	0.4569 g	0.4414 g	0.4221 g
Equivalent weight	114.6	183.9	114.6
Corresponding formula	$\text{CaCl}_2 \cdot 0.18 \text{H}_2\text{O}$	$\text{CaCl}_2 \cdot 4 \text{H}_2\text{O}$	$\text{CaCl}_2 \cdot 0.18 \text{H}_2\text{O}$

Table 18. Determination of water vapor remaining in nitrogen dried over calcium chloride, anhydrous, dried at high temperature

	Run 1	Run 2	Run 3
Final weight	9.1047 g	9.0540 g	9.0581 g
Initial weight	9.1006 g	9.0497 g	9.0540 g
Weight gain	0.0041 g	0.0043 g	0.0041 g
Volume	30 l	31 l	30 l
Time	2 hr, 12 min	2 hr, 15 min	2 hr, 11 min
Flow rate	227 ml/min	229 ml/min	228 ml/min
Residual water	136 $\mu\text{g/l}$	138 $\mu\text{g/l}$	137 $\mu\text{g/l}$
Average residual water	137 $\mu\text{g/l}$		

Table 19. Analysis of calcium chloride, anhydrous, dried at high temperature

	Original material	Inlet of tube F	Outlet of tube F
Ml of EDTA	28.37 ml	24.73 ml	23.83 ml
Normality of EDTA	0.1005 N	0.1005 N	0.1005 N
Sample weight	0.3174 g	0.3437 g	0.2666 g
Equivalent weight	111.3	138.3	111.3
Corresponding formula	CaCl_2	$\text{CaCl}_2 \cdot 1.5 \text{H}_2\text{O}$	CaCl_2

Table 20. Determination of water vapor remaining in nitrogen dried over calcium oxide

	Run 1	Run 2	Run 3
Final weight	0.7101 g	0.8892 g	1.0247 g
Initial weight	0.3716 g	0.5628 g	0.6940 g
Weight gain	0.3385 g	0.3264 g	0.3307 g
Volume	52 l	50 l	50 l
Time	3 hr, 45 min	3 hr, 45 min	3 hr, 45 min
Flow rate	331 ml/min	222 ml/min	222 ml/min
Residual water	650 $\mu\text{g/l}$	653 $\mu\text{g/l}$	664 $\mu\text{g/l}$
Average residual water	666 $\mu\text{g/l}$		

Table 21. Analysis of calcium oxide

	Original material	Inlet of tube F	Outlet of tube F
Ml of EDTA	40.20 ml	43.69 ml	39.96 ml
Normality of EDTA	0.0998 N	0.0998 N	0.0998 N
Sample weight	0.2247 g	0.2488 g	0.1899 g
Equivalent weight	56.01	57.06	56.01
Corresponding formula	CaO	CaO \cdot 0.04 H ₂ O	CaO

Table 22. Determination of water vapor remaining in nitrogen dried over Drierite

	Run 1	Run 2	Run 3
Final weight	9.2687 g	9.3731 g	9.4451 g
Initial weight	9.2530 g	9.3582 g	9.4295 g
Weight gain	0.0157 g	0.0149 g	0.0156 g
Volume	228 l	230 l	236 l
Time	16 hr, 32 min	16 hr, 32 min	16 hr, 32 min
Flow rate	230 ml/min	232 ml/min	238 ml/min
Residual water	69 µg/l	65 µg/l	66 µg/l
Average residual water	67 µg/l		

Table 23. Analysis of Drierite

	Original material	Inlet of tube F	Outlet of tube F
Ml of sodium hydroxide	24.64 ml	28.99 ml	19.08 ml
N of sodium hydroxide	0.1163 N	0.1163 N	0.1163 N
Sample weight	0.1958 g	0.2456 g	0.1151 g
Equivalent weight	68.33	72.84	68.07
Molecular weight	136.6	145.7	136.2
Corresponding formula	CaSO ₄ •0.02 H ₂ O	CaSO ₄ •0.53 H ₂ O	CaSO ₄

Table 24. Determination of water vapor remaining in nitrogen dried over lithium perchlorate, anhydrous

	Run 1	Run 2	Run 3
Final weight	9.1006 g	9.3608 g	9.4290 g
Initial weight	9.0971 g	9.3573 g	9.4252 g
Weight gain	0.0035 g	0.0035 g	0.0038 g
Volume	260 l	265 l	274 l
Time	21 hr, 0 min	21 hr, 0 min	21 hr, 0 min
Flow rate	223 ml/min	223 ml/min	228 ml/min
Residual water	13 $\mu\text{g}/\text{l}$	13 $\mu\text{g}/\text{l}$	12 $\mu\text{g}/\text{l}$
Average residual water	13 $\mu\text{g}/\text{l}$		

Table 25. Determination of water vapor remaining in nitrogen dried over magnesium oxide

	Run 1	Run 2	Run 3
Final weight	0.5604 g	0.7024 g	0.8279 g
Initial weight	0.3353 g	0.5371 g	0.6659 g
Weight gain	0.1711 g	0.1653 g	0.1620 g
Volume	22.14 l	22.20 l	22.00 l
Time	1 hr, 40 min	1 hr, 40 min	1 hr, 40 min
Flow rate	221 ml/min	222 ml/min	220 ml/min
Residual water	775 $\mu\text{g/l}$	752 $\mu\text{g/l}$	731 $\mu\text{g/l}$
Average residual water	753 $\mu\text{g/l}$		

Table 26. Analysis of magnesium oxide

	Original material	Inlet of tube F	Outlet of tube F
Ml of EDTA	25.55 ml	26.17 ml	24.10 ml
Normality of EDTA	0.0998 N	0.0998 N	0.0998 N
Sample weight	0.1050 g	0.1134 g	0.1027 g
Equivalent weight	39.41	43.42	42.70
Corresponding formula	MgO	MgO ·0.17 H ₂ O	MgO

Table 27. Determination of water vapor remaining in nitrogen dried over magnesium perchlorate, anhydrous

	Run 1	Run 2	Run 3
Final weight	9.0974 g	9.3574 g	9.4252 g
Initial weight	9.0972 g	9.3572 g	9.4250 g
Weight gain	0.0002 g	0.0002 g	0.0002 g
Volume	1200 l	1136 l	1199 l
Time	87 hr, 42 min	87 hr, 42 min	87 hr, 42 min
Flow rate	228 ml/min	216 ml/min	228 ml/min
Residual water	0.17 µg/l	0.17 µg/l	0.17 µg/l
Average residual water	0.17 µg/l		

Table 28. Analysis of magnesium perchlorate, anhydrous

	Original material	Inlet of tube F	Outlet of tube F
Ml of EDTA	22.16 ml	19.87 ml	18.79 ml
Normality of EDTA	0.0998 N	0.0998 N	0.0998 N
Sample weight	0.4983 g	0.6576 g	0.4239 g
Equivalent weight	225.4	330.0	226.1
Corresponding formula	Mg(ClO ₄) ₂ •0.12 H ₂ O	Mg(ClO ₄) ₂ •0.6 H ₂ O	Mg(ClO ₄) ₂ •0.16 H ₂ O

Table 29. Determination of water vapor remaining in nitrogen dried over magnesium perchlorate, anhydrous, indicating

	Run 1	Run 2	Run 3
Final weight	9.0993 g	9.3594 g	9.4271 g
Initial weight	9.0974 g	9.3574 g	9.4252 g
Weight gain	0.0019 g	0.0020 g	0.0019 g
Volume	414 l	431 l	455 l
Time	19 hr, 39 min	19 hr, 39 min	19 hr, 39 min
Flow rate	211 ml/min	220 ml/min	232 ml/min
Residual water	4.3 $\mu\text{g/l}$	4.6 $\mu\text{g/l}$	4.2 $\mu\text{g/l}$
Average residual water	4.4 $\mu\text{g/l}$		

Table 30. Analysis of magnesium perchlorate, anhydrous, indicating

	Original material	Inlet of tube F	Outlet of tube F
Ml of EDTA	22.19 ml	21.50 ml	20.93 ml
Molarity of EDTA	0.1000 M	0.1000 M	0.1000 M
Millimoles of $\text{Mn}^{++} + \text{Mg}^{++}$	4.438 m mol	4.300 m mol	4.186 m mol
Weight of KMnO_4	0.917 mg	0.974 mg	0.887 mg
Millimoles of KMnO_4	0.006 m mol	0.006 m mol	0.006 ml
Millimoles of $\text{Mg}(\text{ClO}_4)_2$	4.432 m mol	4.294 m mol	4.180 m mol
Sample weight	1.1156 g	1.0989 g	1.0470 g
Percent KMnO_4	0.80 %	0.89 %	0.85 %
Percent $\text{Mg}(\text{ClO}_4)_2$	86.6 %	87.0 %	89.7 %

Table 31. Second analysis of magnesium perchlorate, anhydrous, indicating

	Original material	Inlet of tube F	Outlet of tube F
Ml of EDTA	24.83 ml	20.00 ml	20.13 ml
Molarity of EDTA	0.1000 M	0.1000 M	0.1000 M
Millimoles of Mn ⁺⁺ + Mg ⁺⁺	4.6 m mol	4.000 m mol	4.026 m mol
Weight of KMnO ₄	1.113 mg	0.900 mg	0.912 mg
Millimoles of KMnO ₄	0.007 m mol	0.006 m mol	0.006 m mol
Millimoles of Mg(ClO ₄) ₂	4.959 m mol	3.994 m mol	4.020 m mol
Sample weight	1.2390 g	1.0221 g	1.0205 g
Percent KMnO ₄	0.91 %	0.88 %	0.89 %
Percent Mg(ClO ₄) ₂	89.4%	87.0 %	87.6 %

Table 32. Average of first and second analyses of magnesium perchlorate, anhydrous, indicating

	Original material	Inlet of tube F	Outlet of tube F
Average percent KMnO ₄	0.86 %	0.89 %	0.87 %
Average percent Mg(ClO ₄) ₂	88.0 %	87.0 %	88.6 %

Table 33. Determination of water vapor remaining in nitrogen dried over Mikohbite

	Run 1	Run 2	Run 3
Final weight	0.4310 g	0.6275 g	0.7546 g
Initial weight	0.3320 g	0.5377 g	0.6631 g
Weight gain	0.0990 g	0.0898 g	0.0915 g
Volume	63.1 l	71.7 l	70.0 l
Time	6 hr, 15 min	6 hr, 15 min	6 hr, 15 min
Flow rate	200 ml/min	228 ml/min	222 ml/min
Residual water	1570 $\mu\text{g/l}$	1250 $\mu\text{g/l}$	1310 $\mu\text{g/l}$
Average residual water	1378 $\mu\text{g/l}$		

Table 34. Analysis of Mikohbite

	Original material	Inlet of tube F	Outlet of tube F
Ml of hydrochloric acid	50.00 ml	50.00 ml	50.00 ml
Normality of hydrochloric acid	0.1040 N	0.1040 N	0.1040 N
Ml of sodium hydroxide	25.40 ml	29.85 ml	22.80 ml
Normality of sodium hydroxide	0.1159 N	0.1159 N	0.1159 N
Sample weight	0.1170 g	0.1415 g	0.1510 g
Percent sodium hydroxide	68.7 %	49.2 %	67.7 %

Table 55. Determination of water vapor remaining in nitrogen dried over Molecular Sieve 5A

	Run 1	Run 2	Run 3
Final weight	9.0975 g	9.3577 g	9.4255 g
Initial weight	9.0966 g	9.3569 g	9.4247 g
Weight gain	0.0009 g	0.0008 g	0.0008 g
Volume	220 l	200 l	230 l
Time	16 hr, 23 min	16 hr, 23 min	16 hr, 23 min
Flow rate	224 ml/min	205 ml/min	234 ml/min
Residual water	4.1 $\mu\text{g}/\text{l}$	4.0 $\mu\text{g}/\text{l}$	3.5 $\mu\text{g}/\text{l}$
Average residual water	3.9 $\mu\text{g}/\text{l}$		

Table 36. Determination of water vapor remaining in nitrogen dried over phosphorus pentoxide

	Run 1	Run 2	Run 3
Final weight	9.2537 g	9.3594 g	9.4317 g
Initial weight	9.2516 g	9.3574 g	9.4296 g
Weight gain	0.0021 g	0.0020 g	0.0021 g
Volume	555 l	560 l	576 l
Time	42 hr, 10 min	42 hr, 10 min	42 hr, 10 min
Flow rate	220 ml/min	222 ml/min	224 ml/min
Residual water	3.8 $\mu\text{g}/\text{l}$	3.5 $\mu\text{g}/\text{l}$	3.6 $\mu\text{g}/\text{l}$
Average residual water	3.6 $\mu\text{g}/\text{l}$		

Table 37. Analysis of washings from tube G for phosphorus pentoxide

Sample	A ₃₆₀	$\mu\text{g P}_2\text{O}_5/100 \text{ ml}$	$\mu\text{g P}_2\text{O}_5/\text{l}$ of nitrogen
Standard 1	0.057	50	---
Standard 2	0.112	100	---
Standard 3	0.173	150	---
Standard 4	0.227	200	---
Run 3	0.093	83	0.14

Table 38. Determination of water vapor remaining in nitrogen dried over potassium hydroxide

	Run 1	Run 2	Run 3
Final weight	0.3844 g	0.5804 g	0.7002 g
Initial weight	0.3677 g	0.5625 g	0.6832 g
Weight gain	0.0167 g	0.0179 g	0.0170 g
Volume	18.6 l	18.8 l	18.2 l
Time	1 hr, 22 min	1 hr, 22 min	1 hr, 22 min
Flow rate	227 ml/min	229 ml/min	222 ml/min
Residual water	928 $\mu\text{g/l}$	955 $\mu\text{g/l}$	935 $\mu\text{g/l}$
Average residual water	939 $\mu\text{g/l}$		

Table 39. Analysis of potassium hydroxide

	Original material	Inlet of tube F	Outlet of tube F
Ml of hydrochloric acid	25.40 ml	21.50 ml	22.33 ml
Normality of hydrochloric acid	0.0969 N	0.0969 N	0.0969 N
Sample weight	0.1610 g	0.1439 g	0.1411 g
Equivalent weight	65.4	67.4	65.3
Corresponding formula	KOH •0.52 H ₂ O	KOH •0.63 H ₂ O	KOH •0.51 H ₂ O

Table 40. Determination of water vapor remaining in nitrogen dried over silica gel

	Run 1	Run 2	Run 3
Final weight	9.2753 g	9.3808 g	9.4555 g
Initial weight	9.2537 g	9.3594 g	9.4317 g
Weight gain	0.0216 g	0.0214 g	0.0238 g
Volume	315 l	304 l	330 l
Time	22 hr, 50 min	22 hr, 50 min	22 hr, 50 min
Flow rate	230 ml/min	222 ml/min	241 ml/min
Residual water	69 $\mu\text{g}/\text{l}$	70 $\mu\text{g}/\text{l}$	72 $\mu\text{g}/\text{l}$
Average residual water	70 $\mu\text{g}/\text{l}$		

Table 41. Determination of water vapor remaining in nitrogen dried over sodium hydroxide

	Run 1	Run 2	Run 3
Final weight	0.3609 g	0.5601 g	0.6808 g
Initial weight	0.3513 g	0.5512 g	0.6718 g
Weight gain	0.0096 g	0.0089 g	0.0090 g
Volume	18.0 l	17.4 l	18.2 l
Time	1 hr, 19 min	1 hr, 19 min	1 hr, 19 min
Flow rate	228 ml/min	221 ml/min	230 ml/min
Residual water	534 $\mu\text{g/l}$	511 $\mu\text{g/l}$	495 $\mu\text{g/l}$
Average residual water	513 $\mu\text{g/l}$		

Table 42. Analysis of sodium hydroxide

	Original material	Inlet of tube F	Outlet of tube F
Ml of hydrochloric acid	46.86 ml	40.77 ml	50.00 ml
Normality of hydrochloric acid	0.0969 N	0.0969 N	0.0969 N
Sample weight	0.1840 g	0.1652 g	0.1968 g
Equivalent weight	40.4	41.8	40.6
Corresponding formula	$\text{NaOH} \cdot 0.03 \text{H}_2\text{O}$	$\text{NaOH} \cdot 0.1 \text{H}_2\text{O}$	$\text{NaOH} \cdot 0.03 \text{H}_2\text{O}$

Table 43. Summary of water vapor remaining in nitrogen dried over the desiccants tested by the gravimetric method

Desiccant	Residual water µg/l
Magnesium perchlorate, anhydrous	0.17
Anhydrone	1.5
Barium oxide	2.8
Alumina	2.9
Phosphorus pentoxide	3.6
Molecular Sieve 5A	3.9
Magnesium perchlorate, anhydrous indicating	4.4
Lithium perchlorate	13
Calcium chloride, anhydrous	67
Drierite	67
Silica gel	70
Ascarite	93
Calcium chloride	99
Calcium chloride, anhydrous, dried at high temperature	137
Anhydrocel	207
Sodium hydroxide	513
Barium perchlorate, anhydrous	599
Calcium oxide	666
Magnesium oxide	753
Potassium hydroxide	939
Mikohbite	1378

Attempts to Determine Water in Gases Using
The Ionization Gauge

The determinations in the preceding section were all made under dynamic conditions, that is, a flow rate of approximately 225 ml per minute. Equilibrium may, or may not, have been reached between the desiccant and the water vapor in the nitrogen, depending upon the kinetics of the desiccant under consideration. It had been the original intention, in addition to the above project, to determine the water in a gas over the desiccants after sufficient time had elapsed for equilibrium to be attained.

Since the determination of a small amount of a gaseous component in the presence of large amounts of carrier gas is a commonplace determination in vapor phase chromatography, the literature of this field was surveyed in search of a sensitive detector. Of the many described, most were eliminated on the grounds of insufficient sensitivity or lack of long term stability. The most promising was the electron tube ionization gauge of Ryce and Bryce (22).

This device was used, feeding the output of the tube plate to a differencing amplifier, as in Figure 2a. This is much the same as was done by Ryce and Bryce. It was also used in the same manner as described by Farrington et al., (9) as shown in Figure 2b. It was found that comparatively

large amounts of nitrogen or natural gas could be detected with this circuit, but no response was obtained from water vapor, even in amounts many times larger than those anticipated.

The circuit shown in Figure 3 was also employed. Any ions drawn to the plate of the ion gauge caused a current, I_1 , to flow as shown, and the plate of the ion gauge became more positive. The Heathkit Direct Current Operational Amplifier then gave rise to I_2 so that $I_1 R_1 = I_2 R_2$, returning the plate of the ion gauge to ground potential. The current I_2 also flowed through the resistor R_3 , giving rise to a voltage which was proportional to the original current. The instrument noise exceeded the signal level, however, and quantitative measurements were not possible.

Svec* suggested that the pressure inside the ion gauge was too high, causing the mean free path of any water ions formed to be shorter than the interelectrode distance. On his recommendation a mercury diffusion pump was used to maintain the pressure within the ion gauge at five microns of mercury. A copper capillary tube of 0.005 inch inside diameter was used to replace the needle valve in the design of Ryce and Bryce, and was constricted to maintain the proper

*Svec, H. J., Chemistry Department, Iowa State University of Science and Technology, Ames, Iowa, Suggestions on the operation of ionization gauges. 1960.

pressure within the ion gauge. The circuits previously described were all tried again, but with no improvement in results.

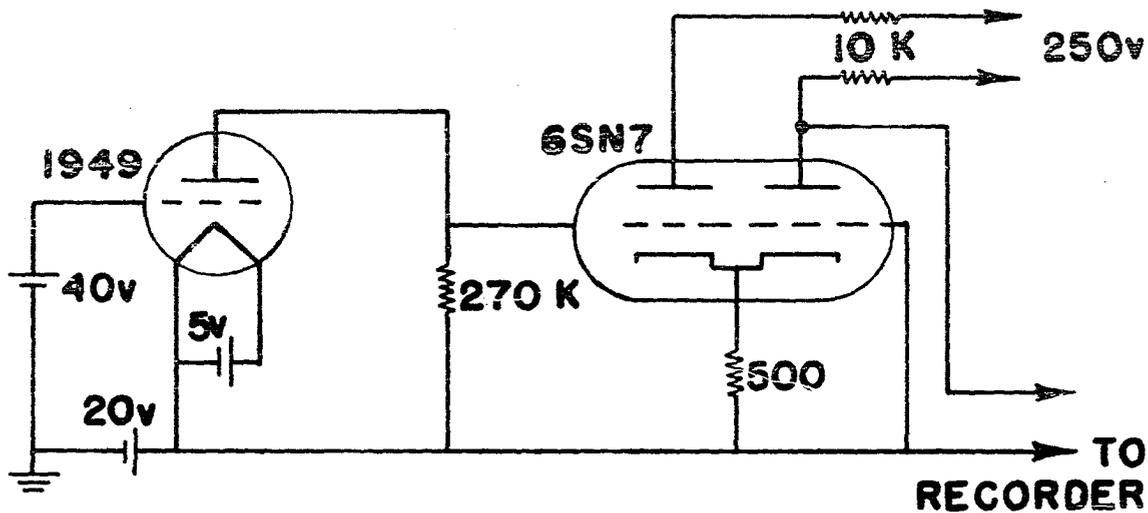
At this point it was decided to verify the value of 12.56 electron volts as the ionization potential for water. In most of the references the values obtained, whether by electron bombardment (1, 17, 24, 25) or by spectral data (21, 26) ranged between 12 and 13 electron volts. One worker (11, 12) using the electron bombardment method found the value to be 18 electron volts.

Upon raising the ionizing potential above 18 volts a response to water was obtained using the circuit shown in Figure 2b. However, at water levels of less than 30 ug per liter of gas the instrument noise level would have exceeded that of the signal. In addition to this, the response was sluggish, the baseline fluctuated excessively, and quantitative measurements were impossible.

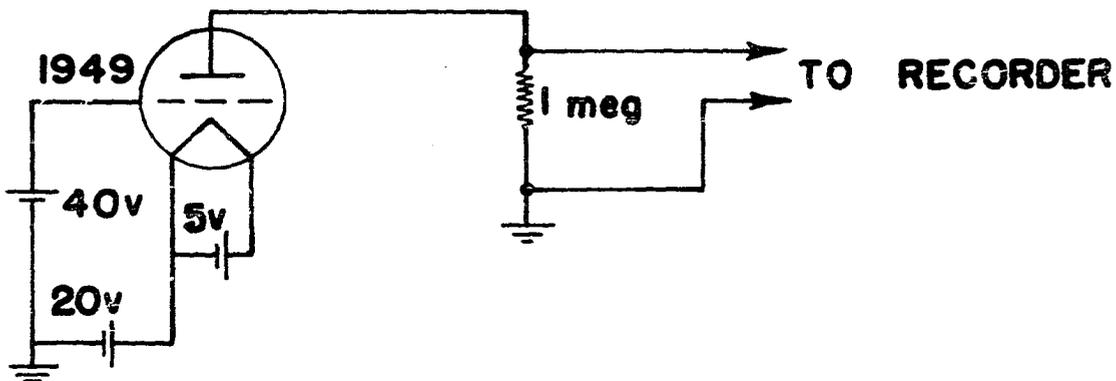
The plate current of the tube was also fed into an electronic circuit designed by Svec (27). In this circuit the plate current was dropped across a 4×10^{10} ohm resistor, the resulting voltage being amplified by a FP-54 electrometer tube. This circuit would not produce a stable zero reading, and the response to water was extremely sluggish. Quantitative measurements were impossible.

Figure 2. Ionization guage tube with amplifiers

- a) Circuit utilizing a difference amplifier
- b) Circuit of Farrington et al. (9)

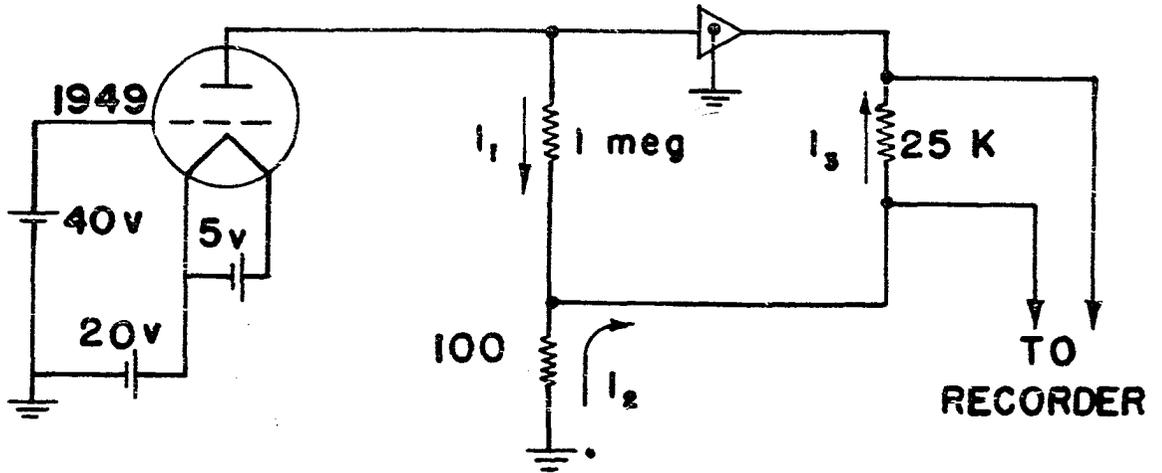


a



b

Figure 3. Ionization guage tube with the Heathkit Analog
Computer Operational Amplifier



Further work along these lines was not undertaken.

The Consolidated Electrodynamics Corporation
Moisture Monitor and the Determination of
Water in Gases

The Consolidated Electrodynamics Corporation Moisture Monitor, Type 26-302, was employed to measure the water vapor in a gas stream. This instrument is essentially the one described by Taylor (28). The sensing element of the instrument is two interwound but separate helices of platinum wire connected by a thin coating of phosphorus pentoxide. The phosphorus pentoxide removes the water vapor from the gas stream, and it is electrolyzed by impressing a potential between the platinum electrodes which exceeds the decomposition potential of water. The current from this electrolysis is proportional to the water in the gas stream. The instrument, as received from the manufacturer, is calibrated in parts per million of water vapor by volume at a flow rate of 20 ml of gas per minute.

A covar glass-to-metal joint was used to attach the glass U-tube to the stainless steel connection into the instrument. The water content of a tank of nitrogen was determined gravimetrically as 325 ug per liter. This tank was connected directly to the U-tube containing the drying agent through a lubricated 12/5 ball and socket joint. Ni-

trogen was permitted to flow for 24 hours before a final reading was taken. The data thus obtained is shown in Table 44.

A comparison of the values thus found with those determined gravimetrically shows no simple relationship between them. Most of the differences can be attributed to the fact that the gas flow in the gravimetric determinations was approximately 11 times faster than that of the determinations employing the moisture monitor.

It should be noted that the moisture monitor did not give a reading of zero when phosphorus pentoxide was tested. A zero reading would have been expected, since the sensing element of the monitor is based on phosphorus pentoxide. More significantly, when the U-tube, without desiccant, was immersed in liquid nitrogen and helium was employed as the carrier gas, the instrument still did not produce a reading of zero. These facts cast serious doubt on the accuracy of the instrument at low moisture levels.

A stainless steel U-tube was employed in the hope that the adsorption of water on the walls of the tube would be less of a problem on this material than on glass. However, in no case was a stable reading obtained any faster, or was the final reading significantly different from that obtained using the glass tube.

Table 44. Determination of water vapor remaining in nitrogen dried over various desiccants using the Consolidated Electrodynamics Corporation Moisture Monitor, Type 26-302

Desiccant	P.p.m. water by volume	μg water/l	μg water/l gravimetric method
Anhydrocel	680	503	207
Anhydrone	6.2	4.6	1.5
Barium oxide	2.7	2.0	2.8
Barium perchlorate, anhydrous	132	98	599
Calcium chloride	150	111	99
Calcium chloride, anhydrous	12	9	67
Calcium oxide	5.0	3.7	666
Drierite	153	113	67
Liquid nitrogen ^a	2.3	1.7	
Lithium perchlorate	28	21	13
Magnesium perchlorate, anhydrous	4.4	3.3	0.17
Molecular Sieve 5A	3.3	2.4	3.9
Phosphorus pentoxide	3.2	2.4	3.6
Silica gel	180	133	70

^aHelium used as carrier gas instead of nitrogen.

In the light of the foregoing findings it was decided that the moisture monitor was not sufficiently accurate for the purposes of these determinations.

SUMMARY AND CONCLUSIONS

The efficiency of several chemical desiccants has been studied by passing humidified nitrogen over the desiccants at a flow rate of 225 ml per minute and freezing out the residual water in a tube immersed in liquid nitrogen. By measuring the total volume of gas used and by weighing the water in the cold trap, the residual moisture in the gas stream in terms of micrograms of water per liter of gas has been calculated.

It has been shown that as a desiccant, anhydrous magnesium perchlorate stands in a class by itself, being nearly 10 times as efficient as the next best drying agent tested, Anhydron $[\text{Mg}(\text{ClO}_4)_2 \cdot 1.5 \text{H}_2\text{O}]$. Following Anhydron in efficiency are barium oxide, alumina, phosphorus pentoxide, Molecular Sieve 5A (a synthetic zeolite), and magnesium perchlorate, anhydrous, indicating. All of these leave less than five micrograms of water per liter of gas.

The common practice of following Ascarite (sodium hydroxide on asbestos) and Mikohbite (sodium hydroxide on fluffed mica) with a desiccant when these materials are used as absorbants for carbon dioxide has been justified. The futility of using calcium chloride for this purpose has been indicated.

The gravimetric method employed was shown to be quantitative, as oxygen condensed in the freeze-out tube when air was employed as the carrier gas. This indicates that the carrier gas was cooled to at least -183° , at which temperature the equilibrium vapor pressure of water over ice is 2.3×10^{-19} mm Hg. Such a vapor pressure would permit the loss of only negligible amounts of water.

In accord with the findings of Morley (19), it was determined that a negligible weight of phosphorus pentoxide was vaporized when that substance was utilized as a desiccant. However, contrary to his findings, a distinctly measurable amount of water remained in the nitrogen stream dried over this reagent.

It should be noted that the low efficiency of calcium oxide and magnesium oxide, and the apparent low capacity of the latter, make them unsuitable as drying agents under the conditions tested.

Unsuccessful attempts were made to determine the efficiency of the desiccants by detecting the residual water in helium dried over them with an ionization gauge. Several circuits were tried, but none was satisfactory.

The Consolidated Electrodynamics Corporation Moisture Monitor Type 26-302, was employed to measure the water vapor

remaining in nitrogen dried over the various desiccants. The results from this instrument are discordant with those of the gravimetric method. It should be noted, however, that the flow rates of the carrier gas was not the same for both methods.

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