

provided, giving the amount of hydrochloric acid in 100 cc. of the ferric chloride solution used.

TABLE II.

Mg. Fe per cc. in solns. used.	No. of cc. HCl (sp. gr. 1.2) per 100 cc. FeCl ₃ soln.	Sensitiveness in Mg. C ₂ H ₃ O ₂ .		
		Volume 5 cc. ¹	Volume 50 cc.	Volume 200 cc.
5	0.5	10-20
10	1.0	25-35
5	3.0	...	20-30	20-30
10	3.5	...	25-35	30-40

The data in Table II apparently corroborate the original conclusions.

Summary.

1. It has been demonstrated that the ferric basic acetate test is not sufficiently sensitive.

2. The test, moreover, does not furnish a means of roughly estimating the amount of acetate present.

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GRANULAR CALCIUM CHLORIDE AS A DRYING AGENT.

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In measuring the efficiency of calcium chloride as a drying agent, Baxter and Starkweather² employed the fused salt. However, granular calcium chloride is much more commonly used, and is more rapid and efficient because its desiccating action is due to adsorption rather than to hydration, as is the case with the fused salt. Since the vapor pressure of an adsorbing substance is a function of the surface moisture that it already contains, it would be expected that granular chloride of calcium, when absolutely dry, would remove every trace of moisture from a gas passed over a sufficiently long column of it. Our experiments show this to be the case.

The method employed was to aspirate air containing a known amount of water vapor over a long column of absolutely anhydrous, granular calcium chloride, and absorb and weigh the residual moisture in a glass stoppered phosphorus pentoxide tube. To obtain the anhydrous chloride a tube containing a column of the chemically pure salt 100 cm. long and 2 cm. in diameter which was used for subsequent experiments was heated to 260-275°³ in a current of air dried over phosphorus pentoxide. Dry

¹ The experiments in a volume of 5 cc. were conducted in test tubes. The procedure as outlined above was followed except that only 1 cc. of the sodium chloride solution was added.

² THIS JOURNAL, 38, 2038 (1916).

³ Cf. Abegg, "Handbuch der Anorganischen Chemie," 2, 98.

carbon dioxide was passed through for some time to neutralize any traces of oxide that may have been produced by hydrolysis. A sample of calcium chloride removed from the tube at the close of the experiment gave a clear solution that was neutral to litmus. The tube was placed in a thermostat at 25°, air saturated with water vapor aspirated through, and the pentoxide tube weighed at intervals, with the following results:

EXPERIMENT I.

Temperature.....	25°						
Liters per hour.....	9.4	9.5	9.5	7.7	9.0	9.0	9.5
Liters of air.....	33.9	42.9	46.4	46.4	46.4	46.9	45.2
Mg. of residual water vapor...	+0.1	0.0	+0.2	+2.2	+5.5	+7.1	+9.2
Volume of air dried with maximum efficiency, 123 liters.							

Since the purpose of this investigation was to test the maximum efficiency of the drying agent, the introduction of a large quantity of water vapor was unnecessary, as most of the moisture was undoubtedly taken up by the first few centimeters of the granular calcium chloride, so, in other experiments, air, before entering the tube, was previously passed over a long column of the fused chloride. The desiccating action of the granular salt, then, was due only to adsorption. Following are two of several experiments which were conducted with results very similar to the above. The calcium chloride tube was dried after each experiment by heating to about 275° with a current of dry air passing through it.

EXPERIMENT II.

Temperature.....	25°					
Liters per hour.....	4.7	4.7	5.0	5.0	4.7	4.9
Liters of air.....	46.9	46.6	46.7	46.4	46.4	46.6
Mg. of residual water vapor....	+0.1	-0.3	+0.1	+0.1	+4.4	+7.5
Volume of air dried with maximum efficiency, 186 liters.						

EXPERIMENT III.

Temperature.....	25°			
Liters per hour.....	23	24	22	24
Liters of air.....	46.6	46.6	48.0	48.0
Mg. of residual water vapor.....	-0.2	+0.3	+1.7	+5.3
Volume of air dried with maximum efficiency, 93 liters.				

It may be noted from the above experiments that anhydrous chloride of calcium, in the porous state, removes at a rapid rate every weighable trace of moisture from a comparatively large volume of gas. The amount of air that may be dried with maximum efficiency decreases as the rate increases, but not in a direct proportion for at 23 liters per hour the volume was only about half as great as at 5 liters per hour. After the period of maximum efficiency the vapor pressure of calcium chloride rises at a moderately uniform rate; however, no attempt has been made to determine how it varies with the amount of moisture adsorbed. From a comparison of Expt. I, in which the aqueous pressure of the incurrent air was

23.76 mm., with Expts. II and III, in which the aqueous pressure was only 0.35 mm.,¹ it is evident that the efficiency of a calcium chloride drying tube does not depend on the quantity of water vapor in the gas that is being dried, but rather on the volume of gas and the rate of flow.

All the samples of granular chloride of calcium that we have examined contain a small amount of surface moisture, so their efficiency lies between that of the anhydrous, granular salt and the fused. The vapor pressures of several samples of Merck's "chemically pure," granular calcium chloride were determined by the method employed above, and were found to be constant for a given sample at a given temperature if no large amount of water were introduced into the tube or removed from it. In every case the vapor pressure of porous chloride of calcium was found to be less than that of the fused salt at the same temperature. For example, at 25° Baxter and Starkweather found the vapor pressure of fused calcium chloride to be 0.35 mm.; one sample of the granular showed, in six determinations, a vapor pressure between 0.23 mm. and 0.25 mm., while another sample was found to have a vapor pressure of 0.14 mm. to 0.16 mm. Thus, the efficiency obtained from granular chloride of calcium as it is ordinarily used is variable and far below that which may be had from the completely anhydrous salt. When calcium chloride is employed where an efficient drying agent is required it should be previously heated, preferably in the tube in which it is to be used, to drive off surface moisture.

F. M. G. Johnson² found that aluminium oxide was capable of practically perfect efficiency as a drying agent, and it is very probable that the desiccating action here, since it depends on the physical state of the oxide, is also one of adsorption rather than of hydration, as stated by Johnson.

It is a pleasure to acknowledge that this investigation was undertaken at the suggestion of Prof. J. B. Ford, of Trinity University, under whose direction some preliminary experiments were conducted.

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NOTES.

Vacuum-Jacketed Pycnometer for Liquids.—The author has used the Davis and Pratt³ type of pycnometer and found it very satisfactory from the point of view of manipulation. In this type, however, like all others, the proper temperature must be obtained after the instrument is filled by immersion in a constant temperature bath, which results in the troublesome task of drying and polishing before weighing. Believing that this

¹ *Loc. cit.*

² *THIS JOURNAL*, 34, 911 (1912).

³ *Ibid.*, 37, 1199 (1915).