

Hygroscopicity of Complex Fertilizers

Determination of Critical Relative Humidity of Multicomponent Systems

Paul R. Geissler

A new vacuum line technique for the measurement of the equilibrium relative humidity of complex fertilizers consists of determining the vapor pressure of the saturated solutions that are formed on the surfaces of such fertilizers by the absorption of small amounts of water vapor. Applications of this method to systems of binary mixtures, reciprocal

salt pairs, and commercial fertilizers containing several components are discussed. The main advantage is that the method can measure critical relative humidities of samples over a range of measurable water concentrations at the low concentrations usually found under storage conditions.

Water-soluble fertilizers, which do not form crystalline hydrates, absorb moisture from the atmosphere by forming saturated solutions on exposed surfaces. Neglecting the small amount of water vapor that may be adsorbed in a monomolecular layer by van der Waal forces of attraction and also any water vapor adsorbed by capillary condensation, absorption of water will occur only when the partial pressure of water vapor in the atmosphere is greater than the partial pressure of water of a saturated solution of the fertilizer. Conversely, desorption will take place when the latter is greater than the former. The partial pressure of water in an atmosphere with which a solid and a saturated solution are in equilibrium is therefore equal to the vapor pressure of the saturated aqueous solution. This equilibrium vapor pressure divided by the vapor pressure of pure water at the given temperature is defined as the critical relative humidity (CRH) and is that relative humidity above which the fertilizer will absorb water from the atmosphere.

Critical relative humidities of pure fertilizer compounds and some binary mixtures have been previously determined by measuring the vapor pressures of saturated solutions with isotenoscopes, first by Edgar and Swan (1922) and later by Adams and Merz (1929) and Merz *et al.* (1933). The use of this method has many disadvantages for determining the critical relative humidity of a multicomponent or mixed fertilizer. The major disadvantage of the isotenoscope is that it is very difficult to determine at what point a solution is completely saturated with respect to all of the various components of the multicomponent fertilizer. It is also limited by the fact that in its usual application, it measures the vapor pressures of systems that contain much more water than that present in fertilizers under actual storage conditions. For single-component pure fertilizer compounds, the critical relative humidity will be independent of water concentrations, and for these the isotenoscope is an acceptable method of measurement. However, since the critical relative humidity of a given multicomponent fertilizer may not be the same at all water concentrations, incorrect values will be obtained if the isotenoscope method is applied to such systems. Yee (1944) described a method for measuring the hygroscopicity of fertilizers by measuring the relative

humidity of the air in equilibrium with the mixtures. This method is based on the use of an electric hygrometer to measure the relative humidity above a closed 16-ounce bottle about one-third full of the given fertilizer mixture. This procedure has several disadvantages in that it requires a large amount of fertilizer sample, frequent calibrations of the hygrometer, and relatively long times.

The vacuum line technique described here overcomes these difficulties by directly measuring equilibrium vapor pressures of saturated solutions that are formed on the surfaces of solid fertilizers by either the absorption of water from the gas phase or the desorption of water from a wet fertilizer in an evacuated system. The major advantage of the method lies in its ability to measure critical relative humidities of complex fertilizer systems at water levels that are found under typical storage conditions, with these water levels being easily determined. This and other advantages are discussed in greater detail by Geissler (1967).

EXPERIMENTAL PROCEDURE

The pertinent parts of the glass vacuum line used in this method are schematically shown in Figure 1. It consists of a water reservoir and thermostated sample containers that can be filled with water vapor, isolated by means of a stopcock from the rest of the vacuum line, and manometers to measure the vapor pressure inside the sample containers. The vacuum line is wrapped with Nichrome heating wire to prevent the condensation of water vapor on cold spots. The critical relative humidity of a fertilizer is determined by placing a 1- to 5-gram sample in a 25-cc. round-bottomed flask which is attached to the vacuum line by means of a greased standard-tapered joint. The sample is then allowed to come to thermal equilibrium at the de-

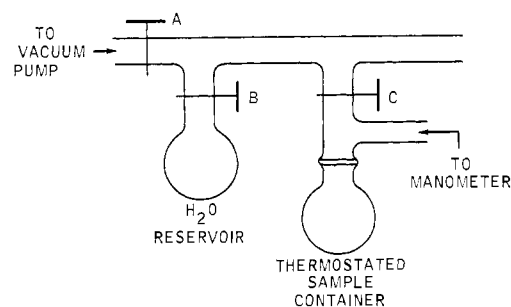


Figure 1. Vacuum line for measuring critical relative humidities of fertilizers

Esso Agricultural Products Laboratory, Esso Research and Engineering Co., Linden, N. J. 07036

sired temperature. With stopcocks *A* and *C* open and *B* closed, the system is evacuated to a pressure of less than 0.001 mm. of mercury. Stopcock *A* is then closed and *B* opened, filling the sample containers with water vapor. The temperature of the water reservoir must be at least as high as that of the thermostated sample to ensure that water vapor fills the sample containers to pressures greater than the vapor pressure of the saturated solution. The water in the reservoir must be previously degassed so that the pressure of water vapor alone is being measured in the sample containers. Stopcock *C* is closed at any time after *B* was opened and the decrease in vapor pressure is recorded as a function of time as the sample absorbs water. The pressure at equilibrium is taken to be the vapor pressure of the saturated solution of the sample, and its critical relative humidity is equal to that pressure divided by the vapor pressure of pure water at the temperature of the sample. The amount of water absorbed can be varied by changing the time interval during which the sample is exposed to the water reservoir. By weighing the samples before and after a determination, and knowing the absolute water concentration of the original sample, the critical relative humidity of a fertilizer can easily be studied as a function of per cent water over a wide range of water concentrations.

Equilibrium can also be approached by the desorption of water vapor from wet fertilizers. This is done by allowing the fertilizer sample to absorb water as above. Stopcock *B* is then closed and with *C* open, stopcock *A* is opened for a short time so as to evacuate the water vapor from the sample container. The sample is then isolated by closing stopcock *C* and the vapor pressure increase as water evaporates from the wet surface is recorded as a function of time until equilibrium is reached. The two methods of approach to equilibrium give identical results in all of the systems investigated, although the attainment of equilibrium by absorption of water vapor is the method used in the majority of the cases studied. At a given temperature, the rate at which equilibrium is approached depends on many factors, such as surface area, particle size, and water content of the sample. In actual practice, using 5-gram samples ground to pass through 80-mesh, equilibrium is reached at room temperature in less than 1 hour, when the amount of water absorbed is about 1% of the total sample weight. Using unground samples does not change the equilibrium values obtained, but does require longer times.

TREATMENT OF EXPERIMENTAL DATA

The equilibrium vapor pressure data for a given fertilizer obtained as described above, when plotted as the logarithm of the pressure *vs.* the reciprocal of the absolute temperature, gives straight-line relationships in the relatively narrow temperature range of 25° to 40° C. Figure 2 shows an example of such a plot. The lower line is typical of that obtained for many of the samples tested, both single- and multicomponent fertilizers. The upper line represents the Clausius-Clapeyron equation for pure water, and this usually has a slightly more negative slope than that for fertilizer systems. Experimentally, four determinations at different temperatures were usually sufficient to define the line for any fertilizer, unless the fertilizer water system was such that the 1 to 2% water present is

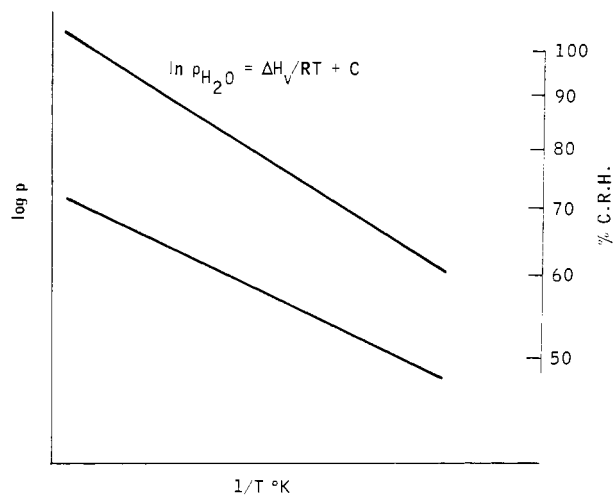


Figure 2. Determination of critical relative humidities from vapor pressure data

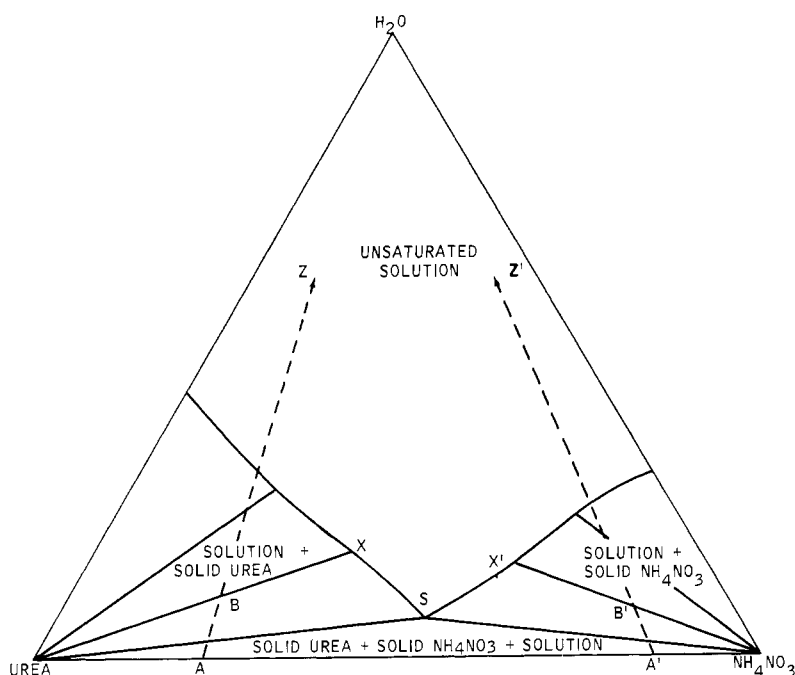
sufficient to dissolve completely one of the compounds present in the multicomponent fertilizer. In these cases, the critical relative humidity is dependent on the amount of water present, and the above straight-line relationship of the logarithm of the pressure *vs.* the reciprocal of the absolute temperature no longer holds.

Since the critical relative humidity of a fertilizer is equal to the vapor pressure of the saturated solution divided by the vapor pressure of water and since the logarithm of the critical relative humidity equals the difference between the logarithms of these two quantities, the differences between the two lines of Figure 2 are equal to the logarithms of the critical relative humidity. Thus, with the use of a logarithm scale (Figure 2) made from the same logarithmic graph paper as used in plotting the data, the differences can be read directly for any temperature in terms of critical relative humidity by placing the 100% point on the scale at the desired temperature on the upper line and reading the critical relative humidity from this scale where it intersects the lower line at the given temperature. If critical relative humidities are desired at one temperature only, they can be obtained simply by dividing the experimental equilibrium vapor pressure by the vapor pressure of pure water at that temperature.

RESULTS AND DISCUSSION

Binary Systems. To interpret the results of this part of the study, some reference to the phase rule chemistry of binary systems must be made. For this purpose, Figure 3 shows the solubility diagram of the urea-ammonium nitrate system at 30° C. based on data of Hardesty (1955). There are four areas of interest in this system. The upper region, which is high in water content, represents compositions which result in unsaturated solutions. The two fan-shaped areas are compositions which give two phases, saturated solution, and either pure solid ammonium nitrate or solid urea. In the fourth area, bounded by the corners of the triangle representing urea and ammonium nitrate and the eutectic solution represented by point *S*, all compositions result in the formation of two solid phases and one liquid phase. This eutectic solution is the most concentrated one possible in this system.

Figure 3. Solubility diagram for urea-ammonium nitrate-water at 30° C. on weight % basis



Thus, if one starts with anhydrous mixtures of urea and ammonium nitrate, such as given by points *A* and *A'*, and adds small amounts of water so that the over-all system compositions are in the invariant triangle bounded by *S* and the two lower corners, the same saturated solution will be formed by both mixtures. Since the vapor pressure of this saturated solution is what determines the system's critical relative humidity, both mixtures *A* and *A'* will have the same critical relative humidity. As more water is added so as to place the over-all composition points in either of the two fan-shaped areas, either the urea or the ammonium nitrate will disappear from the solid phase and there will remain only one solid phase in equilibrium with the saturated solutions. Thus, when the over-all composition points are at *B* and *B'*, the saturated solutions are given by points *X* and *X'* on the extremes of the tie lines. As more water is added, the percentage of salts in the saturated solutions decreases and according to Raoult's law the vapor pressures (and critical relative humidities) will increase. The critical relative humidity will only be constant for mixtures *A* and *A'* in the water concentration range from 0 to that required to place the over-all composition points within the fan-shaped area. The nearer the points *A* and *A'* are to the corners of the triangle—that is, the purer the mixture—the shorter the water concentration range in which the critical relative humidity is independent of the moisture level. Another way of stating this is that the critical relative humidity of a binary mixture will be a constant only when both of the components are present in the solid phase with insufficient water present to dissolve one of them completely.

These concepts were tested experimentally by measuring the critical relative humidity of two urea-ammonium nitrate mixtures over a range of water concentrations. For the two reagent grade mixtures used, 20 and 80% urea, the phase diagram shown in Figure 3 predicts that the critical relative humidity of both mixtures is constant up to about 3% total water. The results shown in Table I

are in good agreement with this. Below 3.6% water, the critical relative humidity of both mixtures is identical, equal to $17 \pm 2\%$, and at higher concentrations the critical relative humidity of both increases with increasing water concentration up to values of 35.2 and 43.2% at water levels of 7.5 and 10.4%, respectively. There is considerable experimental error in the water concentration values at the low levels because of the rapid absorption of water by each sample in the time interval between removal of sample from the vacuum line and subsequent weighing.

The effect of temperature on the critical relative humidity of urea-ammonium nitrate mixtures was also determined. These critical relative humidities are those for which both urea and ammonium nitrate are present in the equilibrium solid phase and are 20, 17, 8, and 5% at temperatures of 25°, 30°, 35°, and 40° C., respectively. This decrease in critical relative humidity with increasing temperatures is much larger percentagewise than that observed in other binary systems because of the very large temperature coefficient of solubility in this system.

Similar data on the effect of water and composition on critical relative humidity were obtained in a less soluble system, monoammonium-diammonium phosphate. Four different reagent grade mixtures were tested and the critical relative humidity values obtained at 30° C. and four different water levels are given in Table I. The critical relative humidities of all the mixtures are identical, with an average value of $76.8 \pm 1.6\%$, while pure monoammonium and diammonium phosphate have critical relative humidities of 92 and 82%. Although the tests were terminated before any change in critical relative humidity with increasing water concentration was observed, the solubility phase diagram for this system indicates that such a change would occur for the 9 to 1 MAP-DAP mixture at about 10% water, for the 1 to 1 mixture at 37% water, and for the 1 to 9 mixture at 13% water, none of which were exceeded.

Thus, from these two experiments on binary fertilizer mixtures, it can be concluded that unless the fertilizer

Table I. Critical Relative Humidities of Binary Mixtures at 30° C.

Component 1 in Mixture, Wt. %	Water, Wt. %	Critical Relative Humidity, %
Urea ⁽¹⁾ -Ammonium Nitrate ⁽²⁾		
20	2.2	15.5
	3.3	14.5
	2.9	18.5
	3.8	21.5
	7.4	32.4
80	7.5	35.2
	3.0	14.9
	3.6	18.9
	3.3	19.5
	5.1	27.9
	9.1	39.2
	10.4	43.2
Monoammonium Phosphate ⁽¹⁾ -Diammonium Phosphate ⁽²⁾		
10	~1	75.4
	~2	77.0
	~4	77.0
25	7.6	79.3
	~1	75.6
	1.4	79.7
50	~3	77.0
	6.6	77.1
	~1	75.1
90	~2	76.2
	~3	76.7
	6.2	77.2
90	~1	75.9
	~2	76.5
	~4	76.5
	8.5	78.7

mixture contains a relatively small amount of a highly soluble component or one that forms a highly soluble eutectic mixture, the critical relative humidity of this mixture will be independent of the amount of water present in the range of low water concentrations usually found in fertilizers and also independent of the ratio of the components in such binary mixtures.

Reciprocal Salt Pairs. Merz *et al.* (1933) have discussed in detail the hygroscopicity of reciprocal salt pair fertilizer mixtures. They measured the critical relative humidities of several such systems using the isotenscope method along with petrographic identification of the solid phases present. The following discusses such systems, but unlike the previous work does so from a phase rule point of view.

In this discussion, reciprocal salt pairs can be treated as systems composed of water and four ions, two of which are positive and two negative. These can undergo double decompositions by the interaction $AB + CD \rightleftharpoons AD + CB$. Since such systems have four components, the phase rule demands that there can be only one temperature at which all four salts of the given system co-exist as solid phases in equilibrium with solution and water vapor. Above this temperature, one salt pair is stable—that is, capable of being in equilibrium with the saturated solution—and below it the reciprocal pair is the stable one. Van't Hoff and Reicher (1889) have shown that the pair which has the smaller product of solubility products at a given temperature ($K_{AB} \times K_{CD}$ as compared with $K_{AD} \times K_{CB}$) is the stable pair at that temperature.

This reversal of stability of salt pairs is depicted by the sequence of isothermal diagrams shown in Figure 4 for the simplest case in which no double salts or solid solutions are formed. These are Janecke projections of a square pyramid in which the apex represents pure water and the four corners of the square represent the pure salts, salts with a common ion being placed at adjacent corners of the square. Ricci (1951) has discussed the use of this type of diagram. Along lines AB to AD and CB to CD the ratio of B/D varies linearly from 1 to 0 and likewise along AD to CD and AB to CB , A/C varies from 1 to 0. Gram equivalent percentages rather than weight percentages must be used. In the first isotherm, t_1 of Figure 4, points a , b , c , and d are the binary eutectics in the CD - CB , CD - AD , AD - AB , and AB - CB water systems. The arrows on these lines denote the direction of decreasing water content or increasing water solubility for the saturated solutions. Along the line aQ_1 , the saturated solutions are in equilibrium with both CD and CB ; along cQ_2 , the solid phases are AD and AB . Along line Q_1Q_2 the equilibrium solid phases are AB and CD , which means that they are the stable salt pair at this temperature. At Q_1 , there exists an isothermally invariant point at which the saturated solution is in equilibrium with the stable salt pair AB and CD and also solid CB . At Q_2 , the third salt present in addition to the stable salt pair is AD . Point m at the intersection of the AB - CD diagonal with the curve Q_1 - Q_2 is a saddle point with respect to water content, and compositions on the diagonal AB - CD behave like ternary systems consisting of water, AB , and CD . Solution m is termed a congruently saturated solution, signifying that it is in equilibrium with the stable salt pair alone and can be prepared from water and these two salts.

At a higher temperature t_2 , the system is incongruently saturated with respect to salts AB and CD , since a solution that is in equilibrium with only solid AB and CD cannot be made from water and these two salts without the addition of solid AD . The system designated by point Q_1 must have a higher water content and therefore lower solubility than point Q_2 , as indicated by the direction of the arrow along line Q_1Q_2 . At the transition temperature

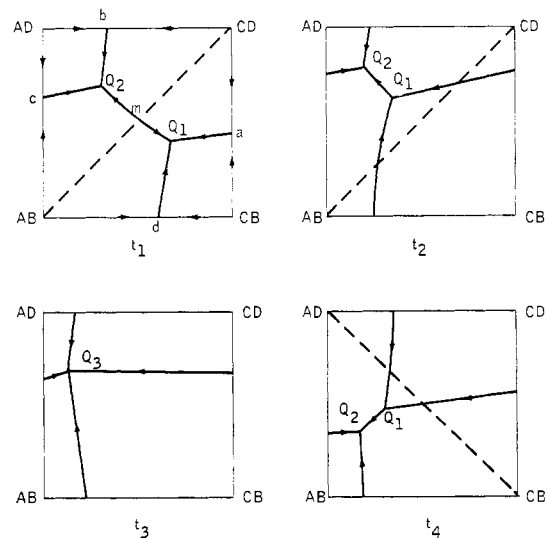


Figure 4. Inversion of stability in reciprocal salt pairs with changing temperatures

t_3 , point Q_3 represents a solution that is saturated with all four salts present in the solid phase. This solution is also one of maximum solubility at that temperature. At a higher temperature t_4 , the curve Q_1Q_2 for saturation with a pair of reciprocal salts reappears, but in such a way that the opposite pair, AD and CB , are the stable ones.

When an anhydrous reciprocal salt pair system absorbs a small amount of water, the saturated solution that is formed has a composition that can be determined if the phase diagram for the system is known at the desired temperature. Thus, if the phase diagram is similar to the t_4 isotherm of Figure 4, all systems within the area $AD-CD-CB$ and on the diagonal $AD-CB$ form solutions of composition Q_1 and the remaining systems form solution Q_2 . In all cases of this type, Q_1 has a higher water content than Q_2 and therefore a higher critical relative humidity. For a t_1 isotherm, all systems within $AB-CB-CD$ form solution Q_1 , those within $AD-AB-CD$ form Q_2 , and those on the diagonal $AB-CD$ form solution m . In this latter case, m has a higher critical relative humidity than either Q_1 or Q_2 . For a t_3 isotherm, only one solution, Q_3 , is possible on the addition of water to any mixture of the components of the reciprocal salt pair system. Therefore, only one critical relative humidity is possible for a system at its transition temperature.

The effect of composition on the hygroscopicity of such systems was verified by measuring the critical relative humidity of four separate reagent grade mixtures of the potassium nitrate-ammonium chloride-ammonium nitrate-potassium chloride system at 30° C. The phase diagram for this system, based on the data of Aronova and Luskaza (1935), is shown in Figure 5 and is similar to the t_4 isotherm of Figure 4. The critical relative humidity values obtained for each of the 1 to 9 salt pair mixtures are also shown in Figure 5. As discussed above, the three mixtures that form solution Q_1 on the absorption of water have the same critical relative humidity of about 68%. This is higher than the 55% critical relative humidity of solution Q_2 , which is formed by the absorption of water by the high ammonium nitrate sample. These critical relative humidities were measured at sufficiently low water concentrations so that the values are independent of the amount of water present. They also agree with the two values of 67.9 and 54.7% reported for this system by Merz *et al.* (1933).

The above concepts predict that there are only two critical relative humidity values possible at low water concentrations for mixtures of the unstable salt pair at 30° C., ammonium nitrate-potassium chloride. One will be the critical relative humidity of solution Q_2 in Figure 5, and this value will apply to all mixtures in which there is a molar excess of ammonium nitrate. In those binary mixtures in which potassium chloride is in equimolar or greater amounts, the critical relative humidity for these systems will be that of solution Q_1 . To test this, the critical relative humidity of a series of ammonium nitrate-potassium chloride mixtures, using reagent grade materials, was measured at 30° at water concentrations of less than 1%. The results shown in Figure 6 verify the above prediction. A somewhat gradual increase in critical relative humidity in the region of equimolar mixtures of ammonium nitrate and potassium chloride is observed

instead of the expected stepwise increase. This is due to the fact that in those mixtures in which there is a slight molar excess of ammonium nitrate, a consideration of the three-dimensional phase diagram for this system shows that the critical relative humidity will increase with increasing water concentration at very low water levels. This is very similar to the phenomenon observed in simple binary mixtures discussed earlier, in which the critical relative humidity of a mixture is dependent on the water concentration when a very small amount of a highly soluble component is present. This deviation from the stepwise increase in critical relative humidity is not observed at slight molar excesses of potassium chloride, because the relative insolubility of this compound makes the critical relative humidity independent of water concentration at the low ranges used.

Another reciprocal salt pair system that was studied consisted of potassium sulfate-potassium chloride-ammonium chloride-ammonium sulfate. Hill and Loucks

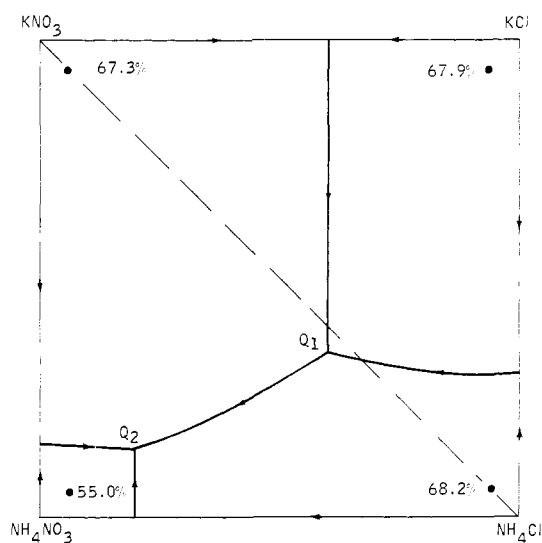


Figure 5. Critical relative humidities at 30° C. in ammonium nitrate-potassium chloride reciprocal salt pair system

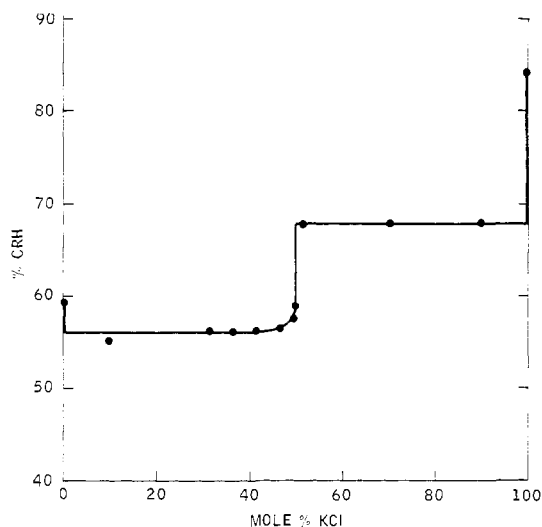


Figure 6. Critical relative humidities of ammonium nitrate-potassium chloride mixtures at 30° C.

(1937) have shown that only three solid phases are possible in this system—that is, solid solutions of ammonium and potassium sulfate, potassium chloride in ammonium chloride, and ammonium chloride in potassium chloride. For such a system there is only one solution that can be formed on the addition of small amounts of water to anhydrous mixtures—the saturated solution that is in equilibrium with the three solid phases. Thus, only one critical relative humidity would be expected for any composition within this system. This was verified by measuring the critical relative humidity of four separate mixtures, each containing more than 90% of one of the four possible salts. At 30° C., an average critical relative humidity value of 71.6% was obtained experimentally with a maximum deviation of 0.4%. Again this agrees both with the above predictions from phase rule concepts and with the single value of 71.3% reported for this system by Merz *et al.* (1933).

Multicomponent Systems. Table II lists some of the critical relative humidities that have been measured for commercially available multicomponent, complex fertilizers. Unless otherwise stated, the data are for the initial absorption of water by these fertilizers with the final water content being less than 1.5%. In all of these cases, the critical relative humidity does not depend on the amount of water present at these low water concentrations.

The low critical relative humidities of normal and triple

superphosphate on the initial absorption of water are due to the presence of free sulfuric acid or phosphoric acid in each. As the free acids initially absorb water forming more dilute acid solutions, the critical relative humidities increase continuously with increasing amounts of water absorbed. Therefore, the variation of critical relative humidity of NSP and TSP with water concentration will depend on the amount of free acid present. In both of these tests, the fertilizers appeared dry and free-flowing at the completion of the absorption of water. Thus, superphosphates can absorb small amounts of water which will raise their critical relative humidity to very high values and their physical structure is such that they are “dry” at these water concentrations. After this absorption of water, their high critical relative humidity prevents the further absorption of any water, insuring that the material will remain free-flowing. These high critical relative humidities are consistent with the fact that normal superphosphate is principally monocalcium phosphate and gypsum and that triple superphosphate is principally monocalcium phosphate.

In comparing similar fertilizers (Table II), the incorporation of highly soluble nitrogen sources such as ammonium nitrate or urea greatly decreases the critical relative humidity of complex fertilizers. On the other hand, the less soluble ammonium sulfate does not cause such a marked decrease. The stepwise change in critical relative humidity of urea, potash, and ammoniated triple superphosphate with degree of ammoniation is due to the disappearance of particular compounds from and appearance of new compounds in the fertilizer as the ammoniation proceeds. Since the critical relative humidity of the system depends only on the components present and not on their relative ratios, it will change only when a new composition in terms of components results.

Comparison of critical relative humidity values obtained for pure single-component and binary fertilizers by the above-described method with values reported in the literature indicates that the vacuum line technique is accurate within less than two critical relative humidity units. A large amount of data on replicate samples shows that the method is also reproducible within the same limits. The main advantage of the vacuum line technique is that it is rapid, simple to use, and can determine critical relative humidities over a large range of measurable water concentrations, including those concentrations usually found in fertilizer storage practices.

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Table II. Critical Relative Humidities of Complex Fertilizers

N/P/K Ratio	Components ^a	Critical Relative Humidities			
		25° C.	30° C.	35° C.	40° C.
0-1-0	NSP at initial H ₂ O absorption	47	47	47	47
	NSP at 0.5% H ₂ O		50		
	NSP at 0.8% H ₂ O		70		
0-1-0	TSP at initial H ₂ O absorption	43	42	42	42
	TSP at 1.1% H ₂ O		67		
	TSP at 2.0% H ₂ O		90		
	TSP at 2.2% H ₂ O		99		
1-0.22-0	U, AS, MAP, DAP	52	51	49	48
1-0.87-0.83	AS, MAP, DAP, KCl	68	65	60	
	U, AS, MAP, DAP, TSP, KCl		45		
1-0.44-0.83	AS, MAP, DAP, KCl	72	67	63	
	AN, MAP, DAP, KCl	51	49	47	46
	U, MAP, DAP, KCl	44	43	42	41
	U, KCl, TSP ammoniated at 0 to 0.5 lb. NH ₃ /unit P ₂ O ₅		22		
	U, KCl, TSP, ammoniated at 0.5 to 2.5 lb. NH ₃ /unit P ₂ O ₅		40		
	U, KCl, TSP ammoniated at >2.5 lb. NH ₃ /unit P ₂ O ₅		54		
1-0.44-0	AN, KCl, NSP ammoniated at 3.0 lb. NH ₃ /unit P ₂ O ₅	54	50	44	39
	AS, MAP, DAP	68	65	60	
	AN, MAP, DAP	50	48	47	46

^a Major constituents of commercially available fertilizers which contain various impurities. NSP = normal superphosphate; TSP = triple superphosphate; U = urea; AS = ammonium sulfate; MAP = monoammonium phosphate; DAP = diammonium phosphate; KCl = potassium chloride; AN = ammonium nitrate.