TWO METHODS OF COMPARING EQUILIBRIUM MOISTURE OF GRAINS

by

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B. S., Faculdade De Agronomia Da Universidade Federal Do Parana, 1971

A MASTER'S THESIS

submitted in parital fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Grain Science and Industry

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1977

Approved by:

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Introduction

The equilibrium moisture content, hygroscopicity, adsorption and desorption are fundamental and important properties considered in the handling, drying and storing of grains and other biological materials. The concept of equilibrium moisture content sets up parameters (temperature, relative humidity) that determine the moisture content to which the materials can be dried and stored safely.

If materials are exposed to the vapor pressure of moist air, they are affected by the thermodynamic properties of water on contact. Each material has a different water vapor pressure characteristic at a certain air temperature, moisture content and other physical and chemical characteristics.

The vapor pressure of biological materials at various moisture contents and temperatures will determine the process of losing moisture (desorption) or gaining moisture (adsorption) which is described as a moisture equilibrium isotherm.

When the vapor pressure of the water, held by a biological material is equal to the water vapor pressure of the surrounding air, the moisture content of the material is the equilibrium moisture content. For materials such as grains the moisture content in equilibrium with a given relative humidity is higher when the product is desorbing than when it is absorging moisture. The difference between both isotherm values is known as hysteresis.

The hysteresis phenomenon has been explained by the concept of molecular shrinkage in the absorbent caused by absorbent activation.

A sigmoid shaped isotherm and the hysteresis effect have been observed for biological materials. That is, the equilibrium moisture content for a given relative humidity is higher during desorption than absorption and the difference is around 1% moisture, dry basis.

The variation in equilibrium moisture content is caused by differences in:

1. the kind of grain
2. the grain maturity
3. the grain history
4. the techniques for measuring relative humidity
5. the method used to determine equilibrium moisture content

In the course of investigation, temperature should be carefully controlled and the humidity should be controlled such as by aqueous acid or other solutions.

Aqueous sulfuric acid and ethylene glycol solutions of various concentrations can regulate the vapor pressure of the surrounding air and control the relative humidity from 0 to 100%. The vapor pressure depends on the chemical used, the concentration, and the temperature.

Many investigators have used sulfuric acid solutions to study biological materials in contact with controlled relative humidity and its effects, ethylene glycol is seldom used for this purpose. The objective of this research is to compare equilibrium moisture content of grains exposed to different vapor pressures of sulfuric acid and ethylene glycol solutions, fit the data in the Chung-Pfost equation, and supply some isotherm data.

Review of Literature

Many aspects of equilibrium moisture content for biological materials have been studied. Such materials exhibit characteristics of gaining and losing moisture because of their hygroscopicity.

Attempts to determine equilibrium moisture content were made in many areas, specifically with agricultural products. Some of these products, such as wheat and corn, were studied in detail. There is data available for almost all ranges of temperatures and relative humidities.

Many chemical compounds have been used to control relative humidity in studies of hygroscopicity.

Sulfuric acid and ethylene glycol solutions have been used for the purpose of vapor pressure calculation at various conditions and for maintaining constant humidity in a closed chamber to observe different hygroscopic characteristics and isotherm properties.

A booklet by Dow Chemical (15) shows that ethylene glycol is hygroscopic and this property is responsible for many applications of ethylene glycol as humectant and dehydrating agent.

Wilson (42) was one of the first researchers to work with sulfuric acid solution to keep a constant humidity in a closed chamber. He used homogeneous sulfuric acid solutions and he also described many advantages of this solution over other materials. The homogeneous solutions can be obtained by varying from 0 to 100% water. For this purpose, it was found that sulfuric acid solutions have no real competitor under ordinary conditions.

Greenwalt (18) showed that many investigators had used partial pressure of water out of aqueous solutions of sulfuric acid to determine parameters of vapor pressure. The available data on the partial pressures were divided into two groups, those for solutions up to 50% acid and those for solutions over 50% acid.

Many investigators have determined the vapor pressures of the weaker solutions and their data are concordant for all temperatures and concentrations. For acids stronger than 50%, the data is very sparse and in poor agreement.

Becker and Sallans (3) studied the desorption isotherms of wheat at 25°C and 50°C by means of sulfuric acid solutions to maintain the relative humidity.

Bushuk et. al., (8) used a sulfuric acid vapor pressure control system for studying hysteresis in wheat flour and its fractions. Breese (7) worked with sulfuric acid to demonstrate hysteresis in the equilibria of rough rice at 25°C. Relative humidities from 10 to 30% inclusive were controlled by H_2SO_4 solutions. It was also shown that equilibrium by adsorption is extremely rapid at relative humidities above 50% and, in desorption the exchange of water is not so rapid and, the equilibrium moisture content is higher. Chung et. al. (12) used the H_2SO_4 method with air removed from the sample container to study hysteresis of wheat. The relative humidities controlled by means of sulfuric acid solutions ranged from 8.9 to 88.9%. Guevara-Guio (17) worked with sulfuric acid solutions to obtain sorption isotherm for three different kinds of dry beans. In addition, the rate of sorption, densities, and the heats of desorption and adsorption of water were studied.

Gustafson et. al. (19) used saturated salt solutions (LiCl, MgCl, $Mg(NO_3)_2$, NaCl, $(NH_4)SO_4$, KNO_3 and K_2SO_4) to keep relative humidities controlled. Shelled corn was used and the equilibrium moisture content determined at temperatures ranging from 50 to 155^{o}F. The air-agitation system within humidity controlled chambers was used and Gustafson concluded that it does not produce a large reduction in the time required to reach equilibrium, as expected. Air agitation only reduces the required time at 50^{o}F.

Dunstan, Chung and Hodges (16) used propionic acid (0.4% by weight) to prevent molding of grain sorghum samples in high relative humidities. Proprionic acid didn't effect the moisture content equilibrium.

Troeger et. al. (40) used three insulated chambers designed and constructed for holding the samples under conditions of constant temperature and relative humidity for an extended period of time. These chambers were made of plexiglass with styrofoam insulation on all sides. Air was supplied by a compressed air line and saturated to the desired dew point by passing it upward through a packed tower with water at the dew point temperature trickling down. The saturated air was then heated to the desired dry bulb temperature before passing through the samples. The equilibrium moisture content in three varieties of peanuts was determined.

High relative humidities affect the moisture content because of mold growth. It could affect any method involving a change in weight.

On the basis of sorption isotherms for biological materials it is normally a sigmoidal shape and several theories have been described to explain the relationship between moisture content, relative humidity, and temperature.

Polanyi 1916 (36), Langmuir 1918 (28), DeBoer and Zwicker 1929 (14), McBain and Bakker 1935 (30), Bradley 1936 (4), Brunauer, Emmet and Teller 1938 (6), Hawkins and Jura 1944 (22), Othmer and Sawyer 1945 (32), Brockington, Dorin and Howerton 1949 (5), Larmour, Sallans and Craig 1949 (29), Hoover and Mellon 1950 (25), Henderson 1952 (23), Thompson and Shedd 1954 (39), Weston and Morris 1954 (41), Day and Nelson 1955 (13), Becker 1956 (3), Bushuk and Winkler 1957 (8), Hall and Rodriguez-Arias 1958 (21), Alam and Shove 1963 (2), Kuhn 1964 (26), Chung and Pfost 1967 (11), Strohman and Yoerger 1967 (38), Young and Nelson 1967 (44), Chen 1969 (9), Ngoddy and Bakker-Arkema 1970 (31), Henderson 1970 (24), Troeger and Butler 1970 (40), Chen and Clayton 1971 (10), Chung and Converse 1971 (12), Pixton and Warburton 1971 (33, 34) and Dunstan 1972 (16) are researchers who have proposed assumptions, theories and equations to contribute to the study of equilibrium moisture content. One isotherm equation will be worked with in this investigation. Chung and Pfost (11) developed a general isotherm equation for cereal grains and their products. This recent equation works with a wider range of relative humidities and temperatures.

The equation was verified by experimental data:

$$\ln(P/P_o) = -\frac{A}{RT}\exp(-BM)$$

where: P/P_o = equilibrium relative humidity

T = absolute temperature in degrees Rankin

R = universal gas constant

A, B = constants

M = moisture content, percent dry basis

More recently (35) the basic equation was modified to:

$$\ln(RH) = \frac{-A}{R_o\ (T + C)} \exp(-BM_D)$$

where: C = empirically determined constant

Also, the Chung-Pfost equation can be expressed for M_D as independent variables as shown:

$$M_D = E - F \cdot \ln\left[-R \cdot (T + C)\ \ln(RH)\right]$$

where: $E = \frac{\ln(A)}{B}$

$F = \frac{1}{B}$

To determine whether distinct differences exist between models obtained from sulfuric acid and ethylene glycol solutions we used a nonlinear, least-squares regression program called GAUSHAUS from Share Library (43) to test the hypothesis as follows:

$$H_o\text{: Class 1 = Class 2}$$

$$(\beta_1 = \beta_2)$$

The procedure (35) is to fit the equilibrium moisture model with "q" parameters to Class 1 and obtain a sum of squares error SSE (1). Then, again, fit the model to the Class 2 data and calculate a sum of squares error SSE (2). Pooling the two sums of squares together gives:

$$SSE = SSE\ (1) + SSE\ (2)$$

Combining sets of data and again fitting the model yields the sum of squares error SSE (0).

For the sum of squares hypothesis,

$$SS_{H_o}\ (\beta_1 = \beta_2) = SSE\ (0) - SSE$$

then the calculated $\underline{\underline{F}}$ statistic is

$$F_c\ (q,\ n-2q) = \frac{SS_{H_o}/q}{SSE/(n-2q)}$$

where: q = number of parameters in ERH-EMC model

n = total number of observations in Class 1 and Class 2

F_c = calculated F-ratio

the decision rule is:

Reject H_o if and only if $F_c \geq F_{.99}\ (q,\ n-2q)$

Materials and Methods

The study was conducted with five different kinds of grains: yellow dent corn, rough rice (medium grain), black beans, shelled peanuts, and soybeans. The yellow dent corn was air-dried and came from the Agricultural Grain Marketing Center, the soybeans were also air-dried and came from the Agronomy Department of Kansas State University. The black beans and peanuts were bought from local grocery stores while the rough rice came from the Department of Grain Science and Industry of Kansas State University.

All samples were tested at 38°C, 30°C, 25°C, 20°C, 15°C and 10°C temperatures and 30%, 45%, 60%, 75% and 90% relative humidities to obtain a wide range of equilibrium moisture content data. Samples of 5 to 20 grams were placed in fine wire baskets which were put over a wire screen above sulfuric acid solutions and also above ethylene glycol solutions in desiccators. The static method was used to allow the grains to come to equilibrium without air movement or without being mechanically moved. Concentrations of sulfuric acid were attained by mixing the compound with distilled water according to "International Critical Tables" (37) and Lange's Handbook of Chemistry (27) for sulfuric acid and concentrations of ethylene glycol solutions according to Dow (15) and the Handbook of Chemistry and Physics (20).

Desiccators were filled with 200 ml of solution to control the relative humidity. A controlled temperature room (chamber) was maintained to keep a constant temperature during the time needed to reach equilibrium moisture content or constant weight of samples. The period to attain the equilibrium moisture content varied from 15 to 34 days, depending on the temperature studied. Ten wire baskets containing duplicate samples for each of the five kinds of grain were placed in each desiccator and the samples were weighed every 48 hours to follow changes in weight and to determine the rate of sorption.

An ASAE standard: ASAE 352 oven method (1) was used for moisture determination after the samples had reached constant weight or equilibrium moisture content. For this purpose, whole grains were dried at 130°C (22 hours) for rough rice (similar to oats) and 103°C (72 hours) for the others. The final moisture content of each grain was calculated on wet and dry basis.

The final concentration of the solutions was determined by differences between initial and final specific gravity samples. Changes between initial and final concentrations were not significant as shown in Appendix A.

Results and Discussion

This investigation was conducted with 5 kinds of grain, 6 temperatures and 5 relative humidities. All these data were analyzed, principally to check the variation existing between the two solutions and to determine constants for equation. Each data point plotted represents average of duplicate samples for each grain, temperature, relative humidity and solution. Significant effects and interactions and the level of their significance are demonstrated in each table and figure.

Rate of Sorption

The rate at which the samples reached equilibrium moisture content or constant weight varied with temperature. Table 1 shows the time, in days, for the samples to reach equilibrium. Equilibrium was considered to be reached when the change in weight from the previous reading was .001% of the grain weight. At 30% and 45% relative humidities all the samples underwent desorption. The samples exposed to 60%, 75% and 95% relative humidities underwent adsorption.

As the equilibrium was approached the rates of sorption decreased considerably, because the vapor pressure of grain and environment approached equilibrium. The sorption rates appeared significantly higher during the first week for all temperatures studied.

Solution Concentration

The solutions were made by volume measurement for all temperatures and relative humidities. Appendix A shows calculated changes in concentration due to moisture moving to or from the solutions. According to

Table 1. Number of days to reach equilibrium for various grains at different temperatures.

	Temperature					
	38°C	30°C	25°C	20°C	15°C	10°C
Rough Rice	15	19	22	24	26	32
Yellow Dent Corn	15	19	22	24	26	32
Black Beans	17	21	24	26	28	34
Soybeans	17	21	24	26	28	34
Spanish Peanuts	17	21	24	26	28	34

Appendix A we can observe that all 30% and 45% relative humidity solutions gained moisture and all other relative humidity solutions lost moisture.

Differences in initial vs. final solution concentration were not considered significant.

Equilibrium Moisture Content for Sulfuric Acid and Ethylene Glycol Solutions

The experimental isotherm data for both solutions is shown in Tables 2 - 11 for rough rice, yellow dent corn, black beans, soybeans, and Spanish peanuts at 38°C, 30°C, 25°C, 20°C, 15°C and 10°C and relative humidities of 30%, 45%, 60%, 75% and 90%.

The experimental data agrees with early theories. There was a difference between the data for both solutions. Sulfuric acid solution data was always higher than ethylene glycol solution data at each of the temperatures and relative humidities. This difference may be attributed to the higher specific gravity of sulfuric acid (1.8305) as compared to ethylene glycol (1.1176) at standard conditions. Because of this difference, a slight error in the measurement of the acids may have resulted in a different concentration.

Comparison of Actual Data with Chung-Pfost Equation

The Chung-Pfost equation was used for studying sorption isotherms because it works with a wide range of temperatures and relative humidities. This equation is very easy to work and it is based on constants obtained from experimental data for each case and grain. Tables 12 - 13 show the Chung-Pfost equation constants for each grain. Figures 1 - 10 show the equilibrium moisture content predicted for a wide range of temperatures and relative humidities using the Chung-Pfost equation.

In Figures 11 - 20 we can observe a comparison of actual data with the Chung-Pfost equation model, tested at 30°C for five kinds of grains and for both solutions. Tables 12 and 13 show the standard error for moisture over all grains and solutions varied from a low of 0.5% for yellow dent corn to a high of 1.36% for black beans, dry basis

Comparison of Chung-Pfost Equation Data with Different Solutions

Figures 21 - 26 show yellow dent corn data for both solutions of sulfuric acid and ethylene glycol at 50°C, 40°C, 30°C, 20°C, 10°C and 0°C over a wide range of moisture content and relative humidities. Yellow dent corn was selected to demonstrate that the curves are very close to each other.

Table 14 shows that there was no significant difference between solutions for yellow dent corn. Meanwhile, Tables 15 - 18 show that for peanuts, black beans, rough rice and soybeans there was a significant difference between the solutions.

Figure 27 shows the predicted equilibrium moisture content of corn at 30°C as compared with observed data. There is a good agreement between the observed and predicted values at 30-95% relative humidity.

Table 2. Equilibrium moisture content for rough rice over sulfuric acid solutions at six temperatures and five relative humidities.

Temperature (°C)	Relative Humidity (%)	Equilibrium Moisture Content % (wet basis)	Equilibrium Moisture Content % (dry basis)
38	30	8.41	9.18
38	45	9.64	10.67
38	60	10.75	12.04
38	75	12.17	13.86
38	90	15.41	18.22
30	30	9.59	10.61
30	45	10.84	12.16
30	60	11.83	13.42
30	75	13.10	15.07
30	90	16.23	19.37
25	30	9.70	10.74
25	45	10.96	12.31
25	60	12.03	13.68
25	75	13.80	16.01
25	90	17.01	20.50
20	30	9.87	10.95
20	45	11.10	12.49
20	60	12.18	13.87
20	75	14.38	16.80
20	90	17.92	21.83
15	30	10.43	11.64
15	45	11.14	12.54
15	60	12.23	13.93
15	75	14.90	17.51
15	90	18.92	23.33
10	30	11.39	12.85
10	45	12.14	13.82
10	60	13.49	15.59
10	75	15.86	18.85
10	90	20.05	25.08

Table 3. Equilibrium moisture content for yellow dent corn over sulfuric acid solutions at six temperatures and five relative humidities.

Temperature (°C)	Relative Humidity (%)	Equilibrium Moisture Content % (wet basis)	Equilibrium Moisture Content % (dry basis)
38	30	7.50	8.11
38	45	8.99	9.88
38	60	11.36	12.82
38	75	13.13	15.11
38	90	16.46	19.70
30	30	8.51	9.30
30	45	10.13	11.27
30	60	11.98	13.61
30	75	14.03	16.32
30	90	17.03	20.53
25	30	8.89	9.76
25	45	10.76	12.06
25	60	12.73	14.59
25	75	14.13	16.46
25	90	17.96	21.89
20	30	9.23	10.17
20	45	11.23	12.65
20	60	13.00	14.94
20	75	14.46	16.90
20	90	18.35	22.47
15	30	10.11	11.25
15	45	11.70	13.25
15	60	13.22	15.23
15	75	15.20	17.92
15	90	19.21	23.78
10	30	11.09	12.47
10	45	12.96	14.89
10	60	14.83	17.41
10	75	16.70	20.05
10	90	21.12	26.77

Table 4. Equilibrium moisture content for soybeans over sulfuric acid solutions at six temperatures and five relative humidities.

Temperature (^oC)	Relative Humidity (%)	Equilibrium Moisture Content % (wet basis)	Equilibrium Moisture Content % (dry basis)
38	30	5.98	6.36
38	45	7.50	8.11
38	60	9.93	11.02
38	75	13.73	15.92
38	90	17.96	21.89
30	30	6.48	6.93
30	45	7.90	8.58
30	60	10.27	11.45
30	75	14.28	16.66
30	90	18.49	22.68
25	30	6.85	7.35
25	45	8.15	8.87
25	60	10.56	11.81
25	75	14.95	17.58
25	90	19.92	24.88
20	30	7.06	7.60
20	45	8.49	9.28
20	60	10.80	12.11
20	75	15.60	18.48
20	90	20.55	25.87
15	30	7.54	8.15
15	45	8.69	9.52
15	60	11.07	12.42
15	75	16.32	19.50
15	90	21.03	26.63
10	30	8.22	8.96
10	45	9.05	9.95
10	60	12.00	13.64
10	75	16.81	20.21
10	90	22.54	29.10

Table 5. Equilibrium moisture content for black beans over sulfuric acid solutions at six temperatures and five relative humidities.

Temperature (°C)	Relative Humidity (%)	Equilibrium Moisture Content % (wet basis)	Equilibrium Moisture Content % (dry basis)
38	30	7.48	8.08
38	45	9.18	10.11
38	60	11.55	13.06
38	75	13.46	15.55
38	90	20.40	25.63
30	30	8.13	8.85
30	45	9.90	10.99
30	60	12.13	13.80
30	75	14.76	17.32
30	90	21.26	27.00
25	30	8.62	9.43
25	45	10.30	11.48
25	60	13.24	15.26
25	75	15.68	18.60
25	90	22.83	29.58
20	30	8.93	9.81
20	45	10.60	11.86
20	60	14.00	16.28
20	75	16.97	20.44
20	90	23.69	31.04
15	30	9.60	10.62
15	45	10.98	12.33
15	60	14.98	17.62
15	75	17.86	21.74
15	90	25.04	33.40
10	30	9.98	11.09
10	45	11.68	13.22
10	60	15.90	18.91
10	75	18.29	22.38
10	90	25.87	34.90

Table 6. Equilibrium moisture content for Spanish peanuts over sulfuric acid solutions at six temperatures and five relative humidities.

Temperature (^{o}C)	Relative Humidity (%)	Equilibrium Moisture Content % (wet basis)	Equilibrium Moisture Content % (dry basis)
38	30	4.05	4.22
38	45	4.64	4.87
38	60	5.77	6.12
38	75	7.78	8.44
38	90	10.81	12.12
30	30	4.32	4.52
30	45	5.08	5.35
30	60	6.01	6.39
30	75	7.89	8.57
30	90	11.16	12.56
25	30	4.52	4.73
25	45	5.46	5.78
25	60	6.23	6.64
25	75	8.12	8.84
25	90	12.07	13.73
20	30	4.79	5.03
20	45	5.76	6.11
20	60	6.34	6.77
20	75	8.29	9.04
20	90	12.98	14.92
15	30	5.12	5.40
15	45	5.92	6.29
15	60	6.52	6.97
15	75	8.89	9.76
15	90	13.93	16.18
10	30	5.90	6.27
10	45	6.32	6.75
10	60	6.99	7.52
10	75	9.74	10.79
10	90	14.25	16.62

Table 7. Equilibrium moisture content for rough rice over ethylene glycol solutions at six temperatures and five relative humidities.

Temperature (°C)	Relative Humidity (%)	Equilibrium Moisture Content % (wet basis)	Equilibrium Moisture Content % (dry basis)
38	30	9.07	9.97
38	45	9.49	10.49
38	60	10.47	11.69
38	75	12.02	13.66
38	90	15.05	17.72
30	30	9.19	10.12
30	45	10.20	11.36
30	60	11.19	12.60
30	75	12.72	14.57
30	90	15.82	18.79
25	30	9.22	10.16
25	45	10.62	11.88
25	60	11.31	12.75
25	75	12.90	14.81
25	90	16.04	19.10
20	30	9.71	10.75
20	45	10.86	12.18
20	60	11.69	13.24
20	75	13.10	15.07
20	90	17.12	20.66
15	30	9.98	11.09
15	45	11.00	12.36
15	60	12.05	13.70
15	75	13.46	15.55
15	90	18.23	22.29
10	30	10.39	11.59
10	45	11.96	13.58
10	60	12.86	14.76
10	75	14.62	17.12
10	90	19.50	24.22

Table 8. Equilibrium moisture content for yellow dent corn over ethylene glycol solutions at six temperatures and five relative humidities.

Temperature (^{o}C)	Relative Humidity (%)	Equilibrium Moisture Content % (wet basis)	Equilibrium Moisture Content % (dry basis)
38	30	7.49	8.10
38	45	8.99	9.88
38	60	10.78	12.08
38	75	12.21	13.91
38	90	16.17	19.29
30	30	8.48	9.27
30	45	9.69	10.73
30	60	11.84	13.43
30	75	13.03	14.98
30	90	17.01	20.50
25	30	8.84	9.70
25	45	10.20	11.36
25	60	12.23	13.93
25	75	14.08	16.39
25	90	17.56	21.30
20	30	9.23	10.17
20	45	11.05	12.42
20	60	13.00	14.94
20	75	14.26	16.63
20	90	18.02	21.98
15	30	9.90	10.99
15	45	11.66	13.20
15	60	13.21	15.22
15	75	15.12	17.81
15	90	18.99	23.44
10	30	10.90	12.23
10	45	12.85	14.74
10	60	14.69	17.22
10	75	16.10	19.19
10	90	20.83	26.31

Table 9. Equilibrium moisture content for soybeans over ethylene glycol solutions at six temperatures and five relative humidities.

Temperature (°C)	Relative Humidity (%)	Equilibrium Moisture Content % (wet basis)	Equilibrium Moisture Content % (dry basis)
38	30	5.96	6.34
38	45	6.81	7.31
38	60	7.89	8.57
38	75	11.86	13.46
38	90	16.78	20.16
30	30	6.41	6.85
30	45	7.24	7.81
30	60	8.94	9.82
30	75	12.21	13.91
30	90	17.56	21.30
25	30	6.84	7.34
25	45	7.63	8.26
25	60	9.16	10.08
25	75	13.72	15.90
25	90	18.38	22.52
20	30	7.00	7.53
20	45	8.02	8.72
20	60	9.98	11.09
20	75	14.47	16.92
20	90	19.26	23.85
15	30	7.80	8.46
15	45	8.41	9.18
15	60	10.89	12.22
15	75	15.29	18.05
15	90	20.23	25.36
10	30	8.22	8.96
10	45	9.03	9.93
10	60	11.45	12.93
10	75	16.72	20.08
10	90	21.05	26.66

Table 10. Equilibrium moisture content for black beans over ethylene glycol solutions at six temperatures and five relative humidities.

Temperature (°C)	Relative Humidity (%)	Equilibrium Moisture Content % (wet basis)	Equilibrium Moisture Content % (dry basis)
38	30	7.38	7.97
38	45	8.40	9.17
38	60	10.21	11.37
38	75	13.40	15.47
38	90	18.24	22.31
30	30	7.86	8.53
30	45	9.13	10.05
30	60	10.93	12.27
30	75	13.77	15.97
30	90	19.44	24.13
25	30	8.13	8.85
25	45	9.45	10.44
25	60	11.07	12.45
25	75	13.93	16.18
25	90	20.31	25.49
20	30	8.66	9.48
20	45	10.10	11.23
20	60	11.88	13.48
20	75	14.25	16.62
20	90	21.45	16.62
15	30	9.18	10.11
15	45	10.84	12.16
15	60	12.15	13.83
15	75	15.00	17.65
15	90	22.49	29.02
10	30	10.03	11.15
10	45	11.22	12.64
10	60	13.43	15.51
10	75	16.07	19.15
10	90	23.05	29.95

Table 11. Equilibrium moisture content for Spanish peanuts over ethylene glycol solutions at six temperatures and five relative humidities.

Temperature (^{o}C)	Relative Humidity (%)	Equilibrium Moisture Content % (wet basis)	Equilibrium Moisture Content % (dry basis)
38	30	4.06	4.23
38	45	4.51	4.72
38	60	5.48	5.80
38	75	6.11	6.51
38	90	10.33	11.57
30	30	4.24	4.43
30	45	4.65	4.88
30	60	5.93	6.30
30	75	6.83	7.33
30	90	11.02	12.38
25	30	4.37	4.57
25	45	5.10	5.37
25	60	6.20	6.61
25	75	7.16	7.71
25	90	12.04	13.69
20	30	4.73	4.96
20	45	5.43	5.74
20	60	6.24	6.66
20	75	8.29	9.04
20	90	12.98	14.92
15	30	4.91	5.16
15	45	5.82	6.18
15	60	6.50	6.95
15	75	8.89	9.76
15	90	13.47	15.57
10	30	5.29	5.59
10	45	6.31	6.73
10	60	6.90	7.41
10	75	9.33	10.29
10	90	14.22	16.58

Table 12. Equilibrium moisture content constants for sulfuric acid solution data.

Chung-Pfost Equation

$$M_D = E - F \cdot LN \left| -R \cdot (T + C) \; LN \; (RH) \right|, \text{ dry basis}$$

$$RH = \exp \left| \frac{-A}{R \cdot (T + C)} \cdot \exp(-B \cdot M_D) \right|$$

Grain	A	B	C	E	F	Standard Error Moisture
Black Beans	268.47900	11.774550	18.701290	0.474988	0.014929	0.01361
Yellow Dent Corn	521.77780	20.001150	6.578600	0.312844	0.049997	0.005176
Spanish Peanuts, Kernel	306.25240	25.738090	20.835490	0.222410	0.038853	0.008454
Rough Rice	810.88060	23.272470	7.544720	0.287813	0.042969	0.007552
Soybeans	324.25900	13.196280	34.472590	0.438119	0.075779	0.01070

Table 13. Equilibrium moisture content constants for ethylene glycol solution data.

Chung-Pfost Equation

$$M_D = E - F \cdot LN \left| -R \cdot (T + C) \; LN \; (RH) \right|, \text{ dry basis}$$

$$RH = \exp \left| \frac{-A}{R \cdot (T + C)} \cdot \exp \left(-B \cdot M_D\right) \right|$$

Grain	A	B	C	E	F	Standard Error Moisture
Black Beans	332.67450	14.304980	21.409600	0.405954	0.069906	0.01285
Yellow Dent Corn	498.24240	20.523970	5.119010	0.302626	0.048724	0.005100
Spanish Peanuts, Kernel	254.33250	26.242030	15.298990	0.211060	0.038107	0.009713
Rough Rice	977.87930	25.055305	10.362890	0.274807	0.039912	0.008362
Soybeans	244.96180	14.693820	18.451400	0.374382	0.068056	0.01171

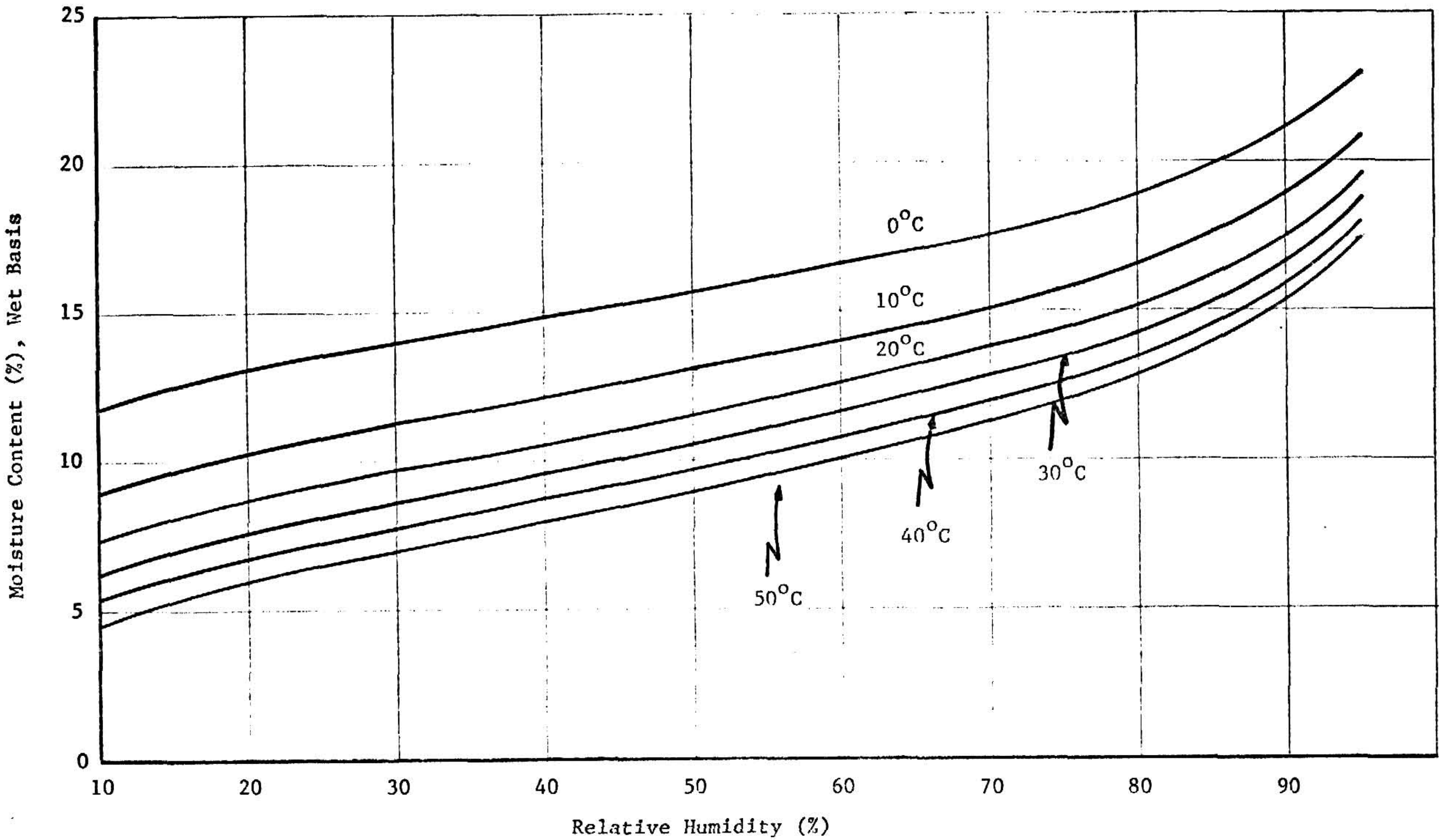

Figure 1. Calculated Isotherms Over Sulfuric Acid Solution, Rough Rice.

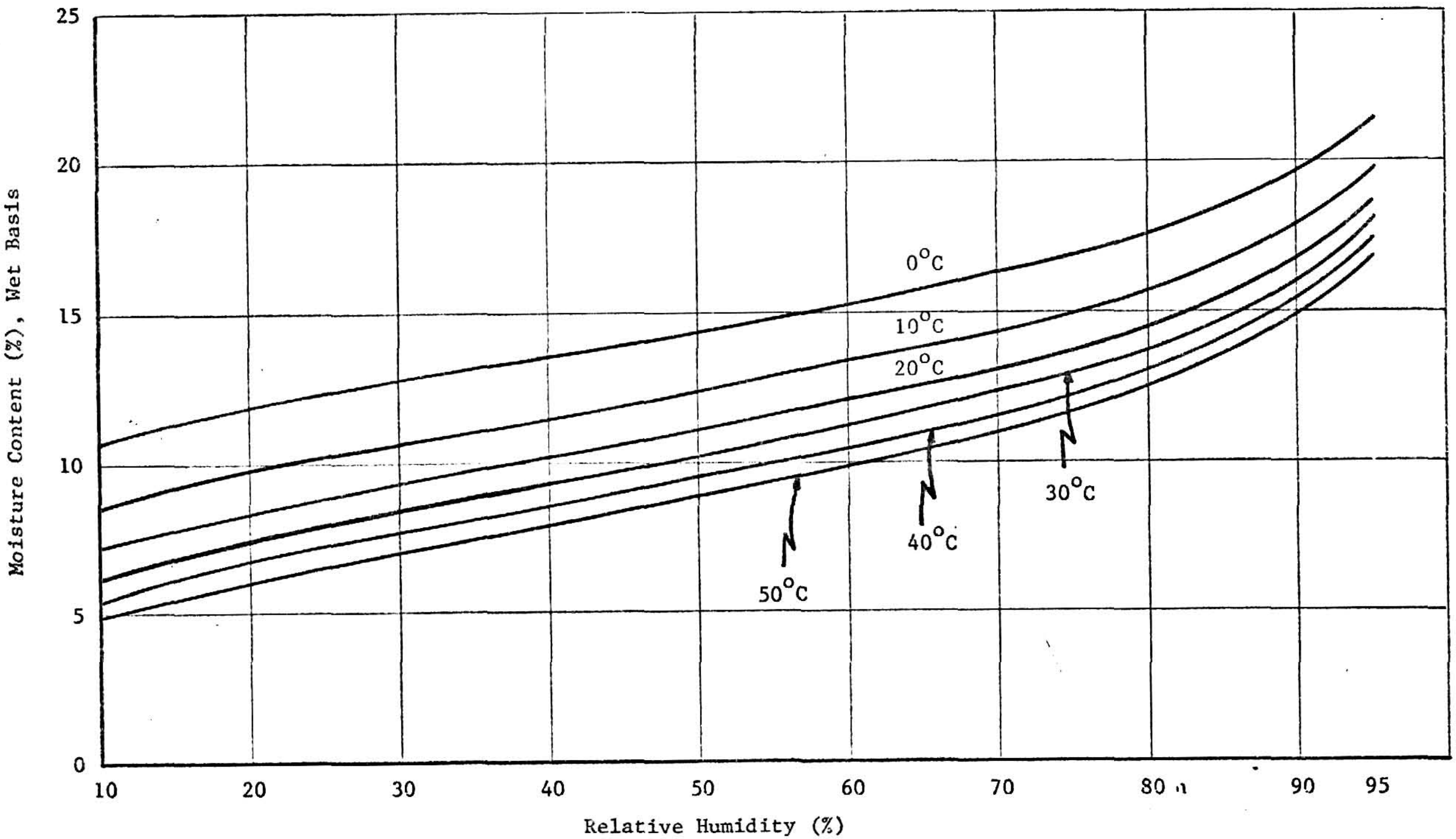

Figure 2. Calculated Isotherms Over Ethylene Glycol Solution, Rough Rice.

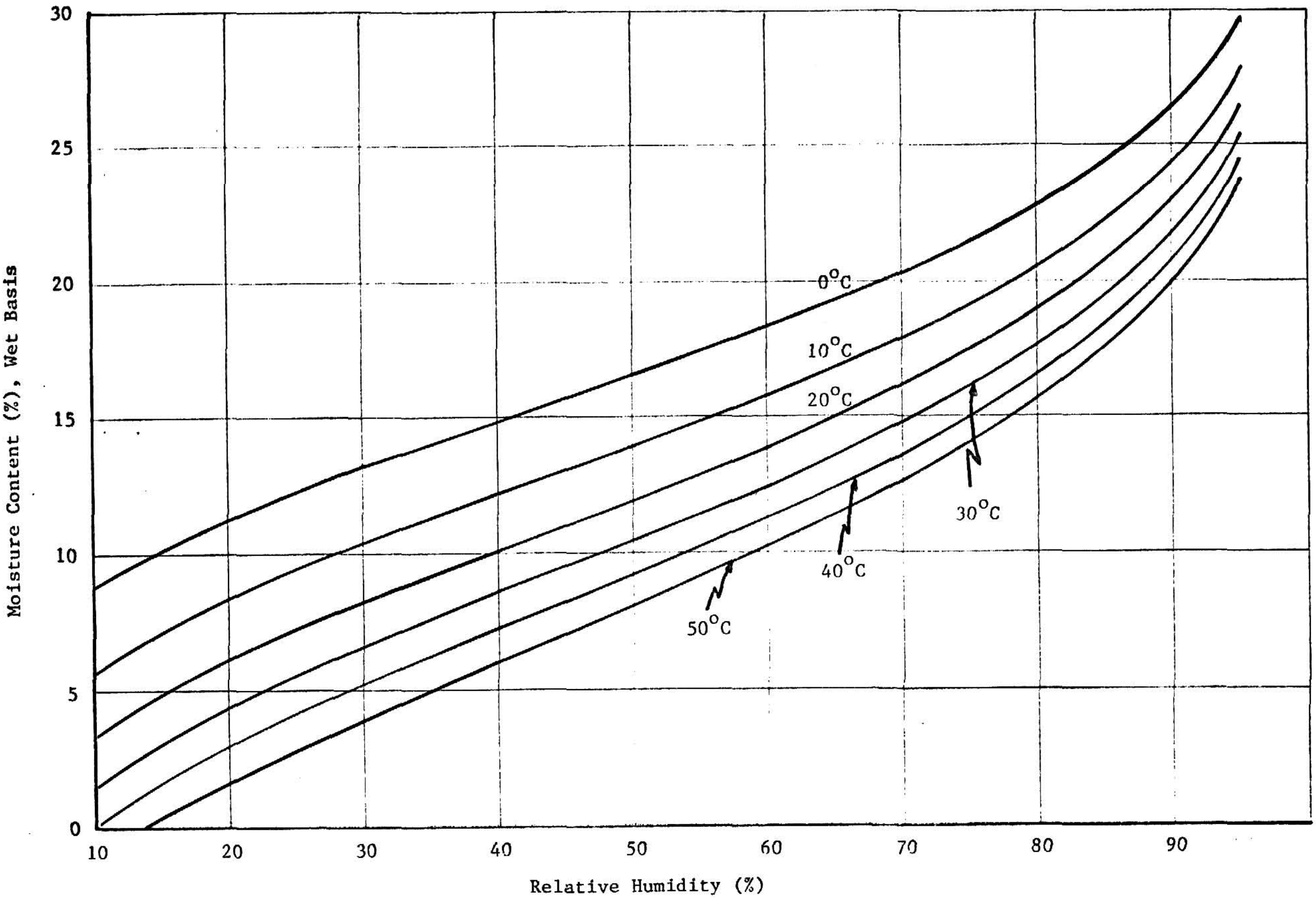

Figure 3. Calculated Isotherms Over Sulfuric Acid Solution, Black Bean.

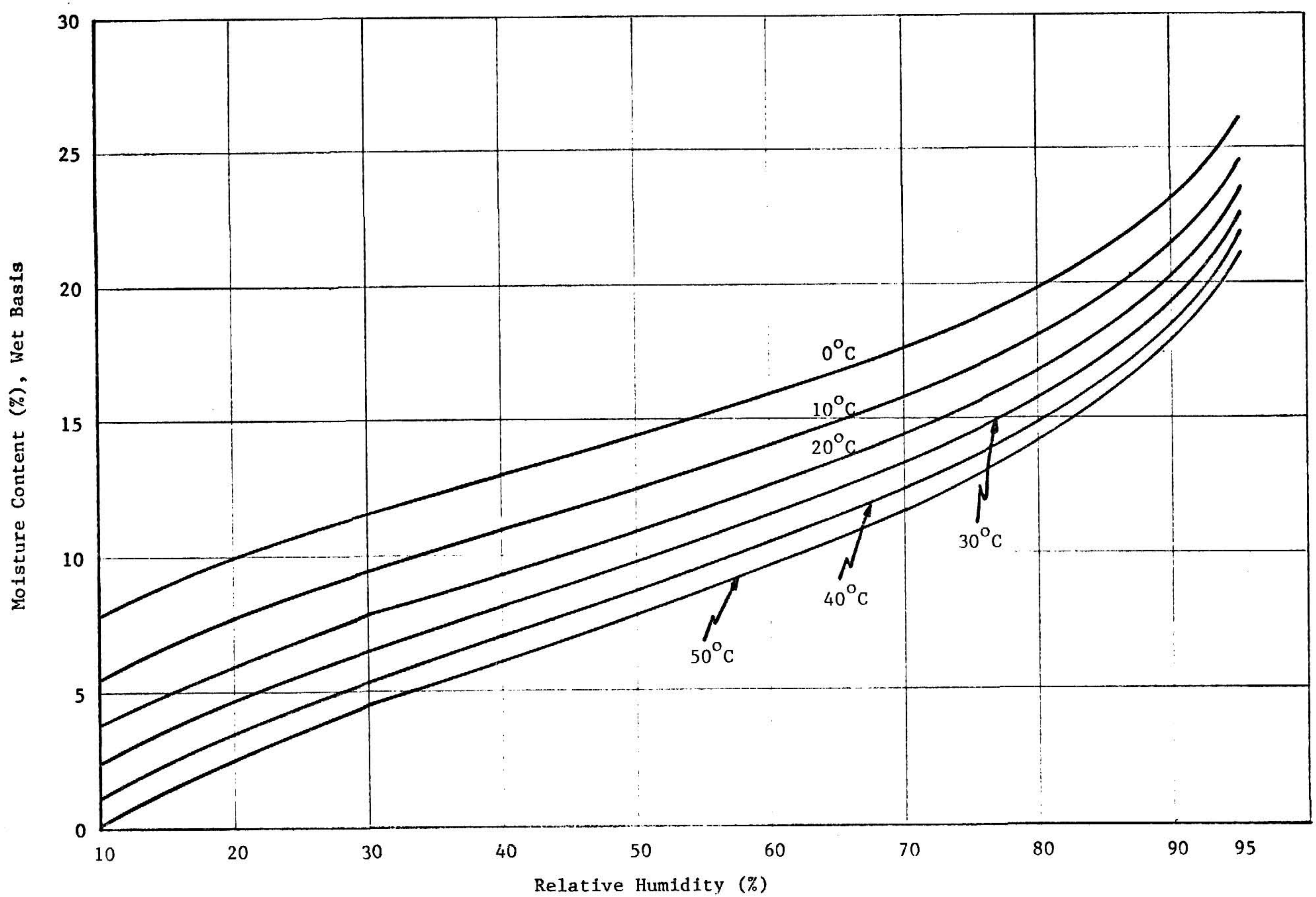

Figure 4. Calculated Isotherms Over Ethylene Glycol Solution, Black Bean.

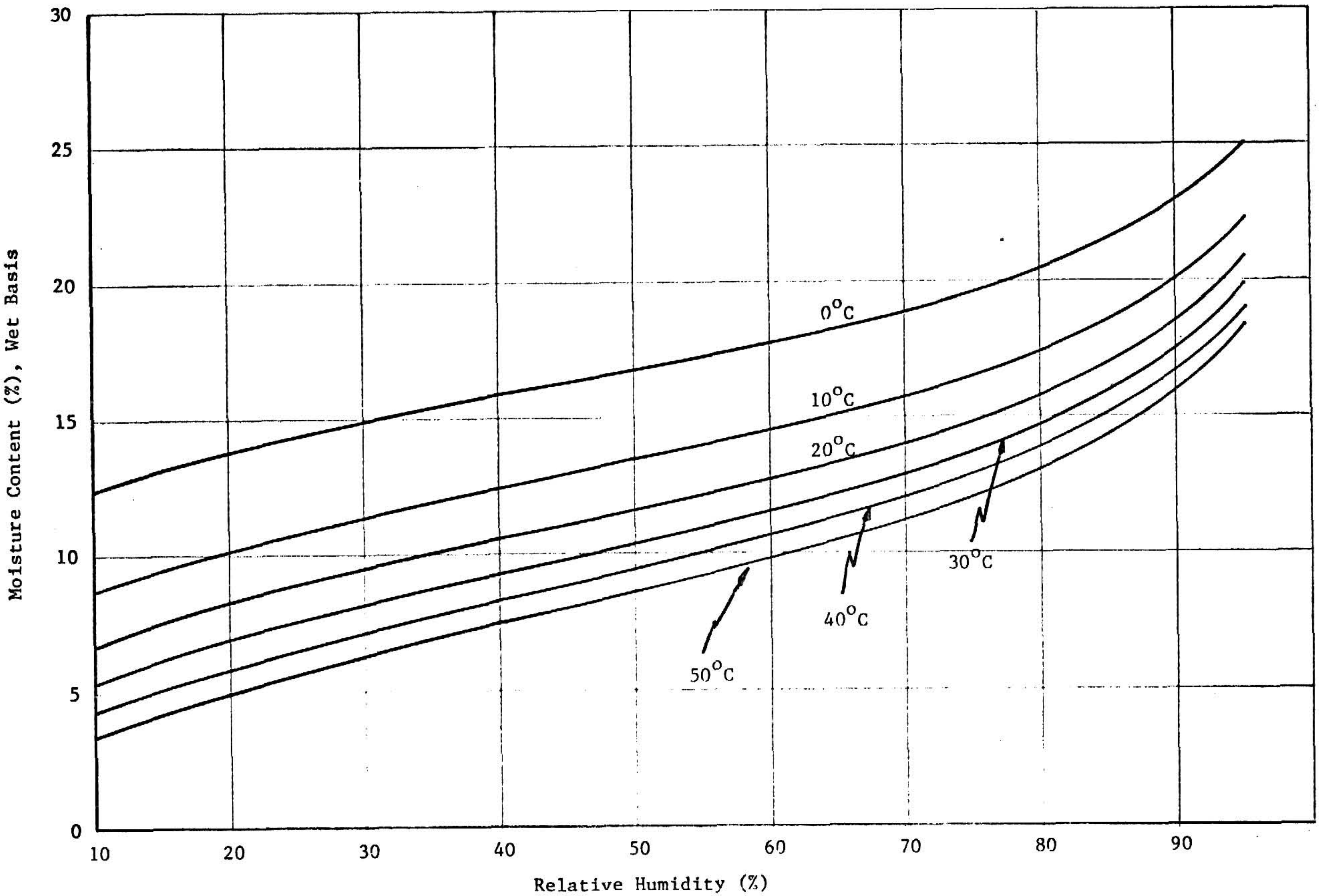

Figure 5. Calculated Isotherms Over Sulfuric Acid Solution, Yellow Dent Corn.

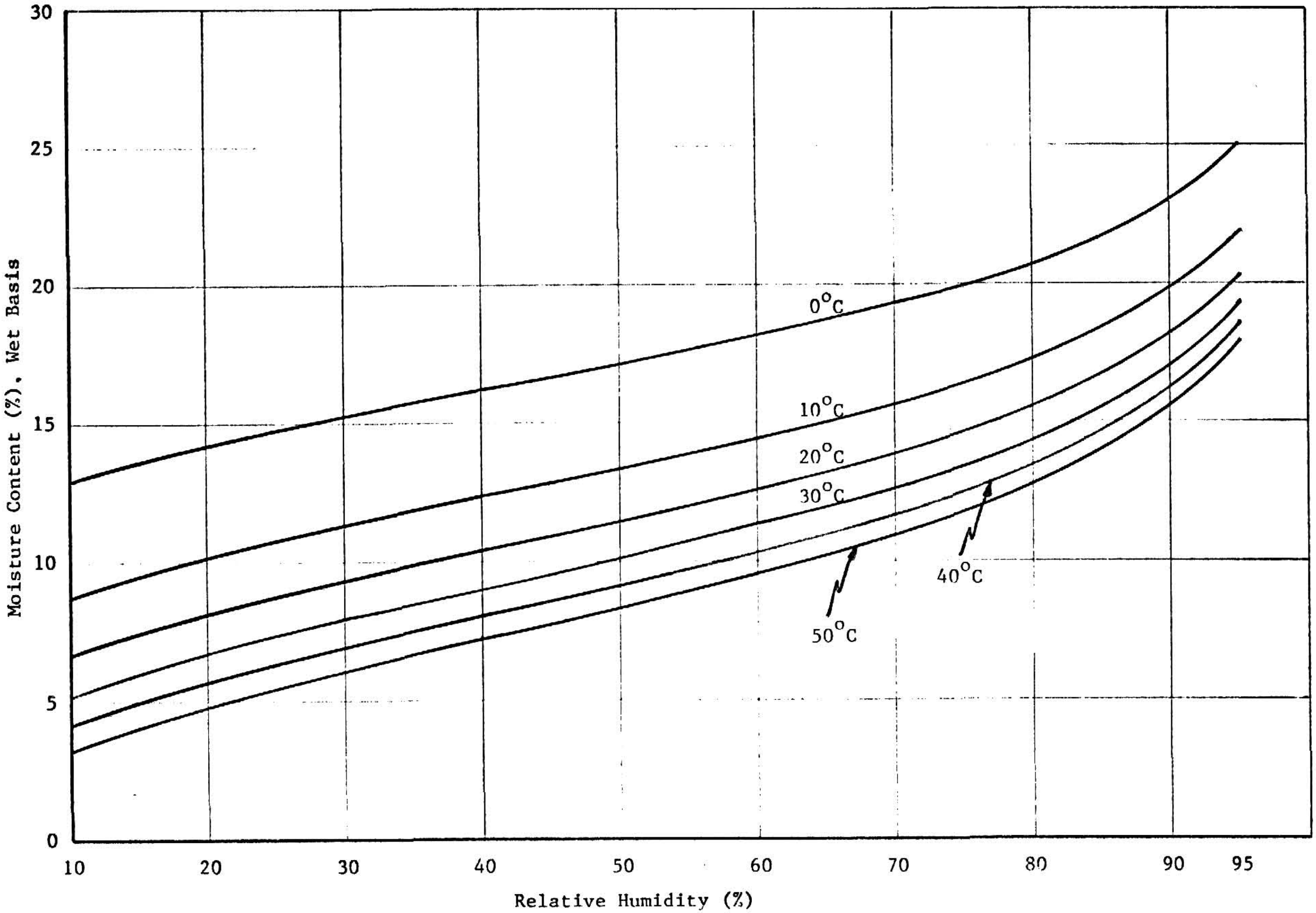

Figure 6. Calculated Isotherms Over Ethylene Glycol Solution, Yellow Dent Corn.

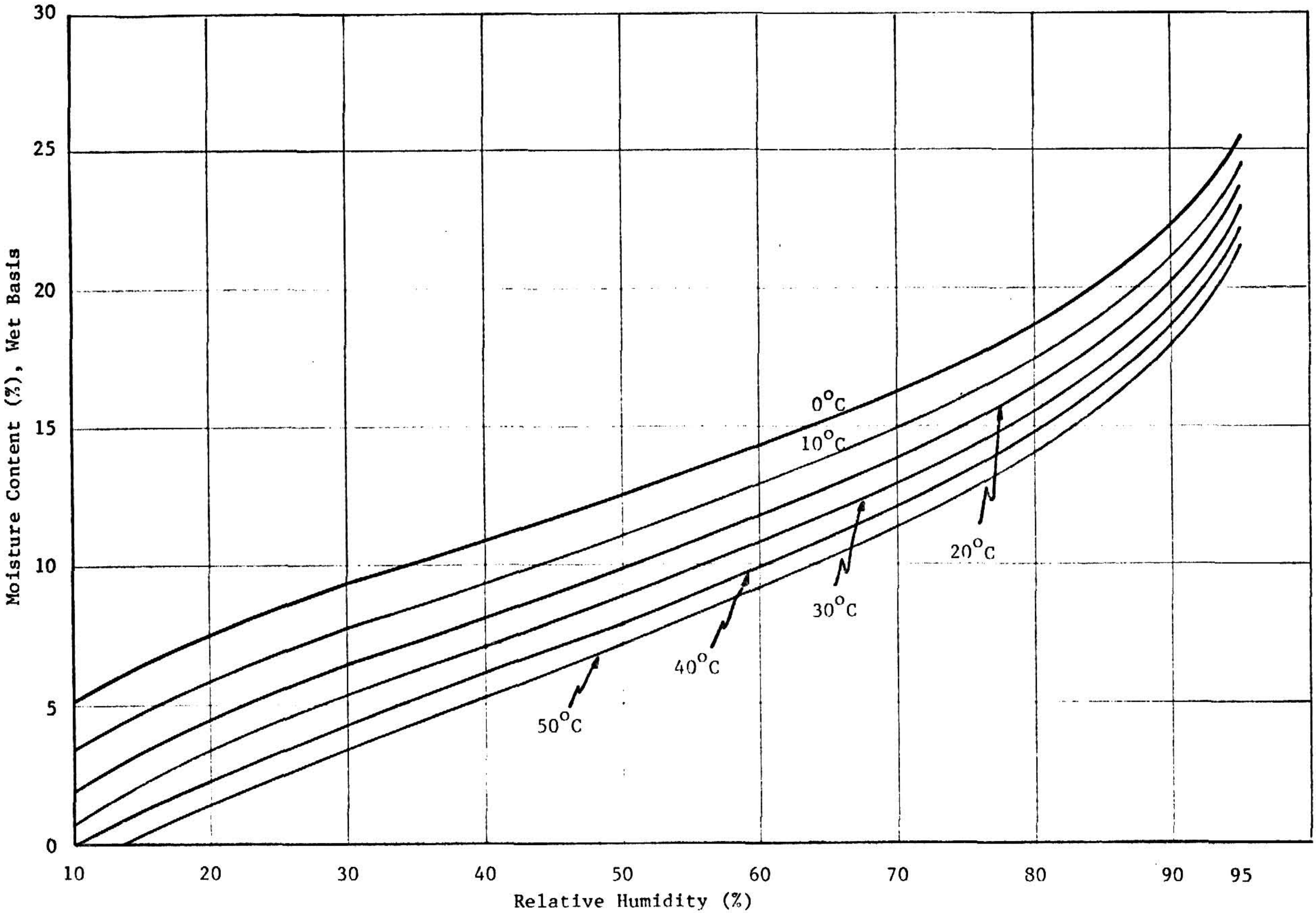

Figure 7. Calculated Isotherms Over Sulfuric Acid Solution, Yellow Soybean.

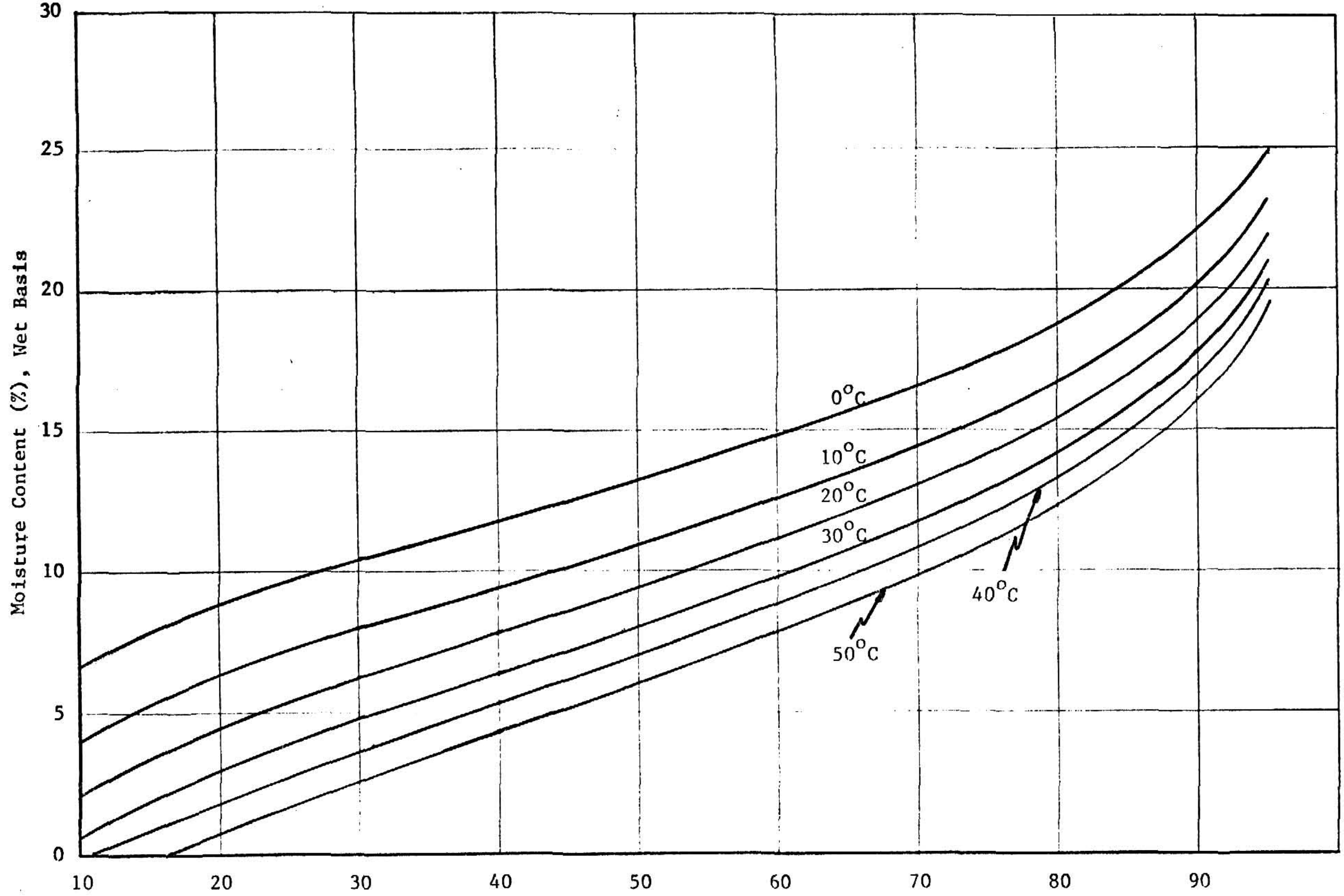

Figure 8. Calculated Isotherms Over Ethylene Glycol Solution, Yellow Soybean.

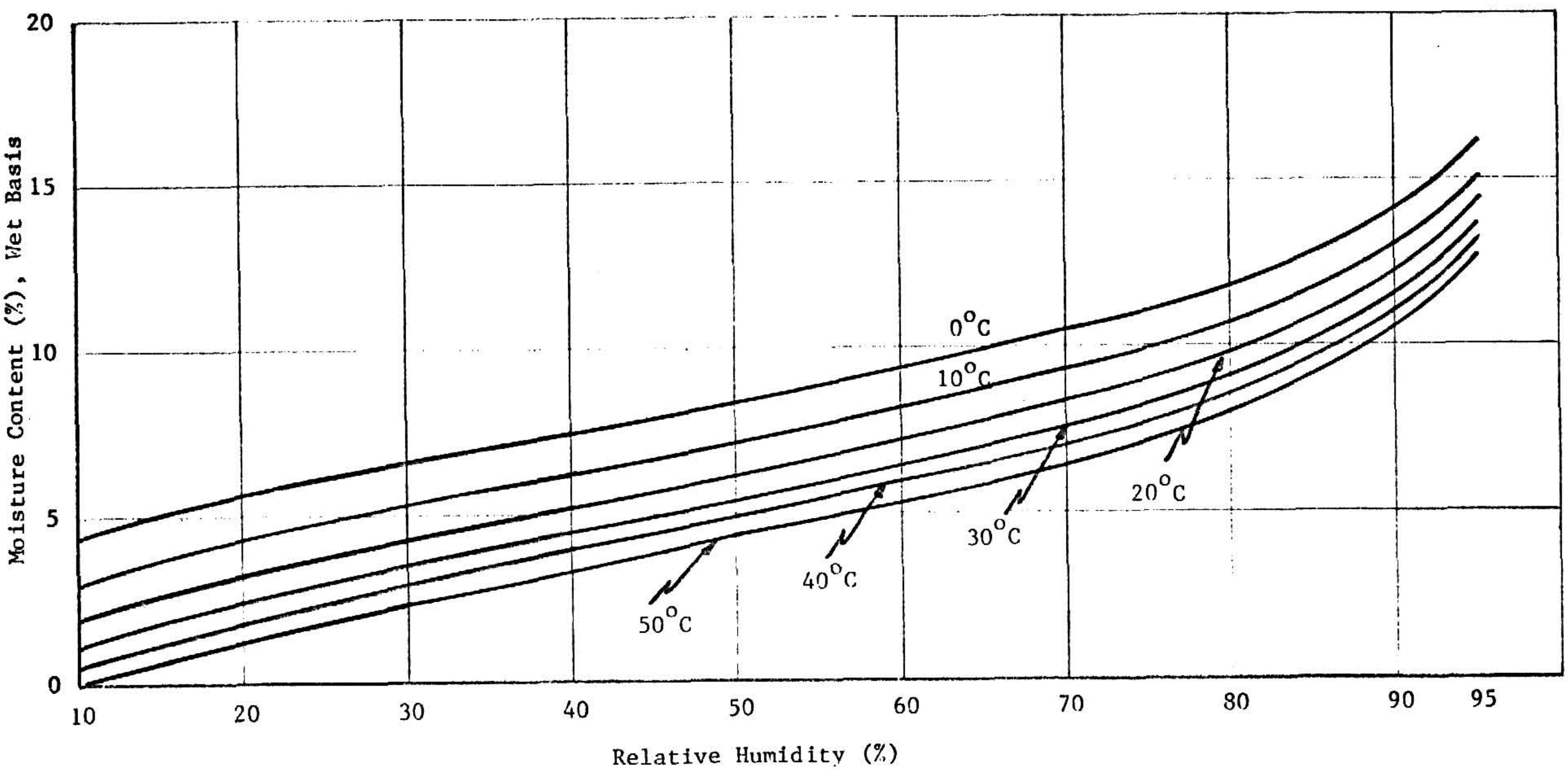

Figure 9. Calculated Isotherm Over Sulfuric Acid Solution, Spanish Peanuts.

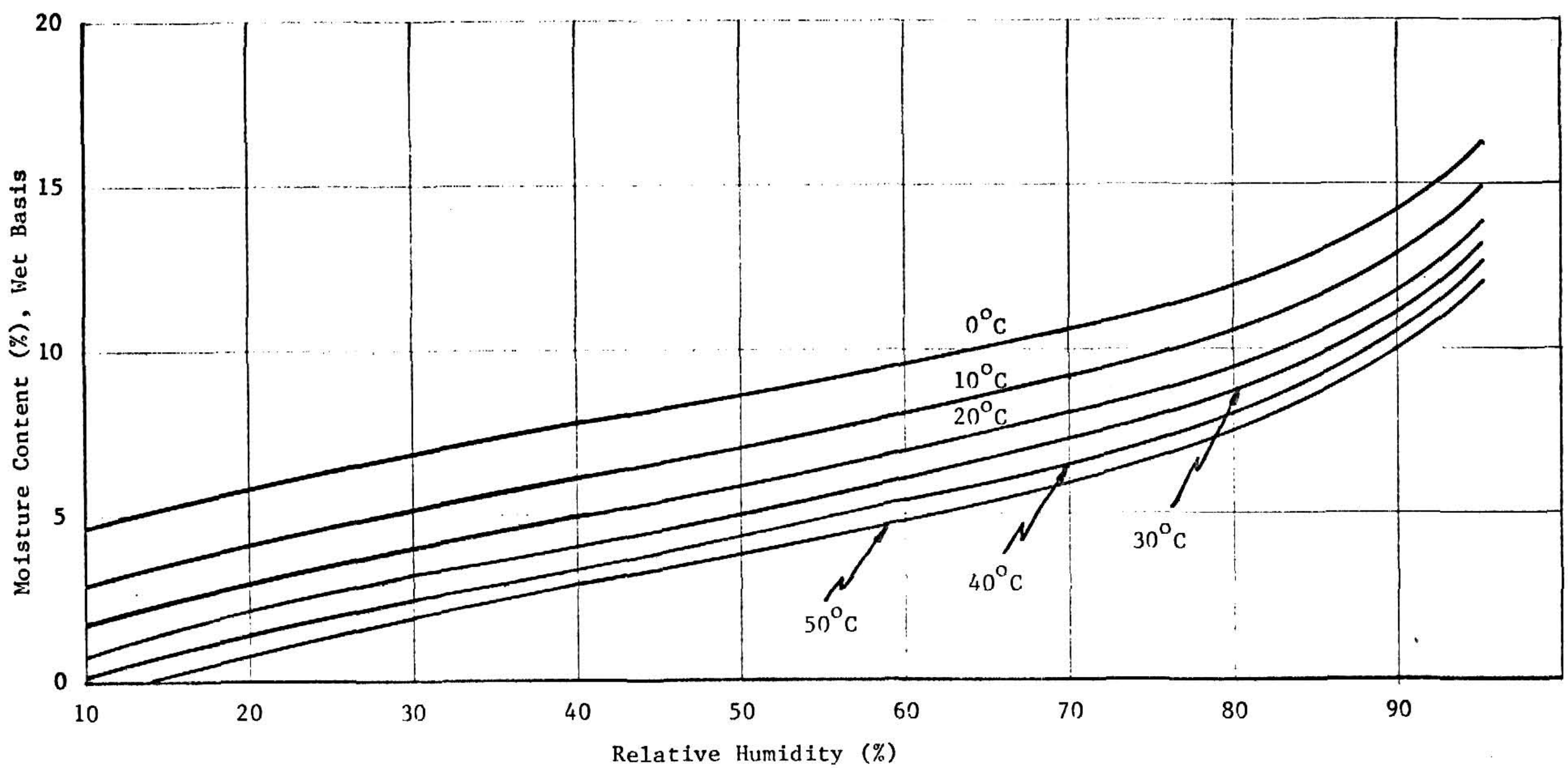

Figure 10. Calculated Isotherms Over Ethylene Glycol Solution, *Spanish Peanuts.*

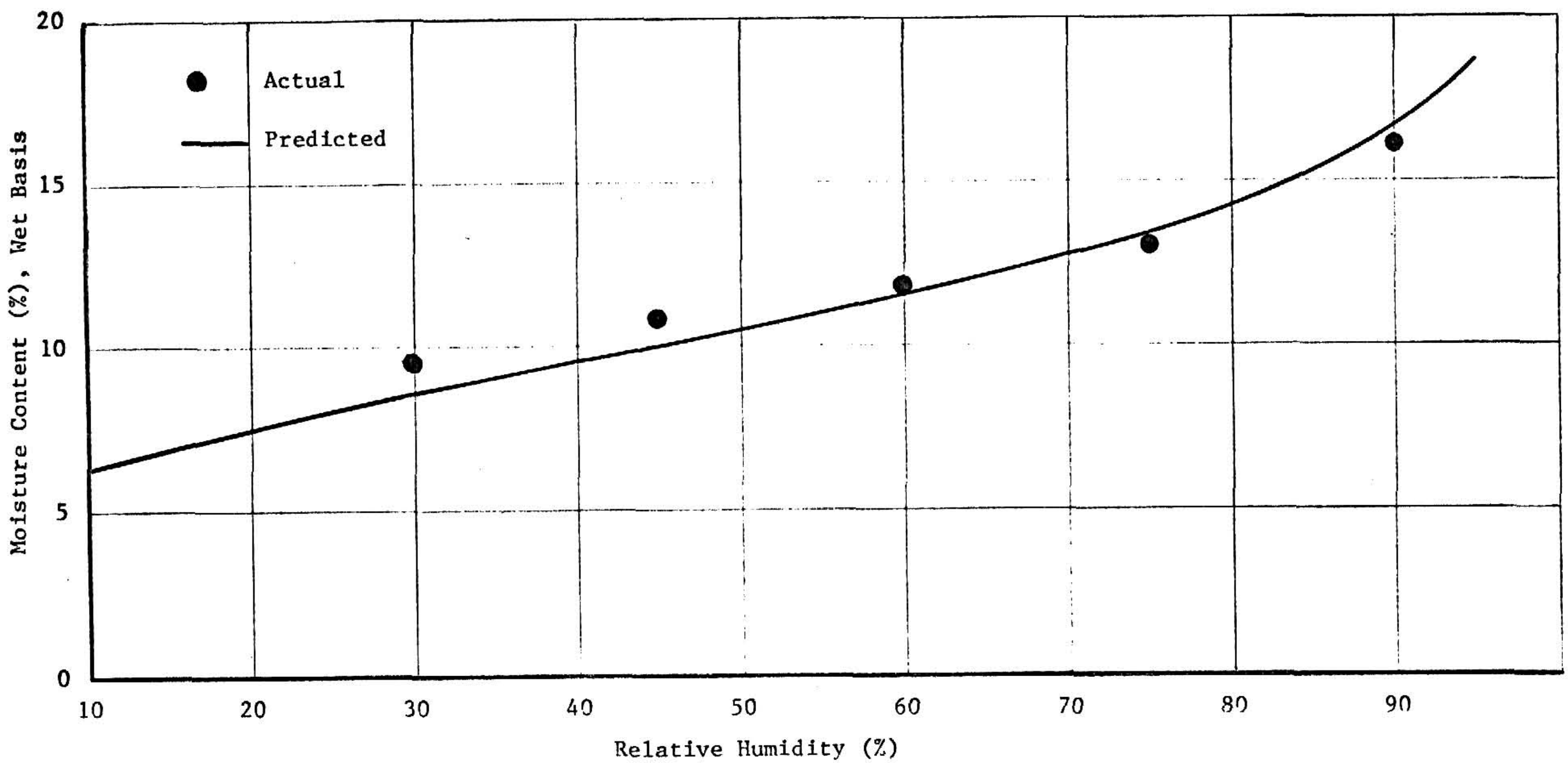

Figure 11. Comparison of Actual Data with Chung-Pfost Equation, Rough Rice Tested Over Sulfuric Acid at 30°C.

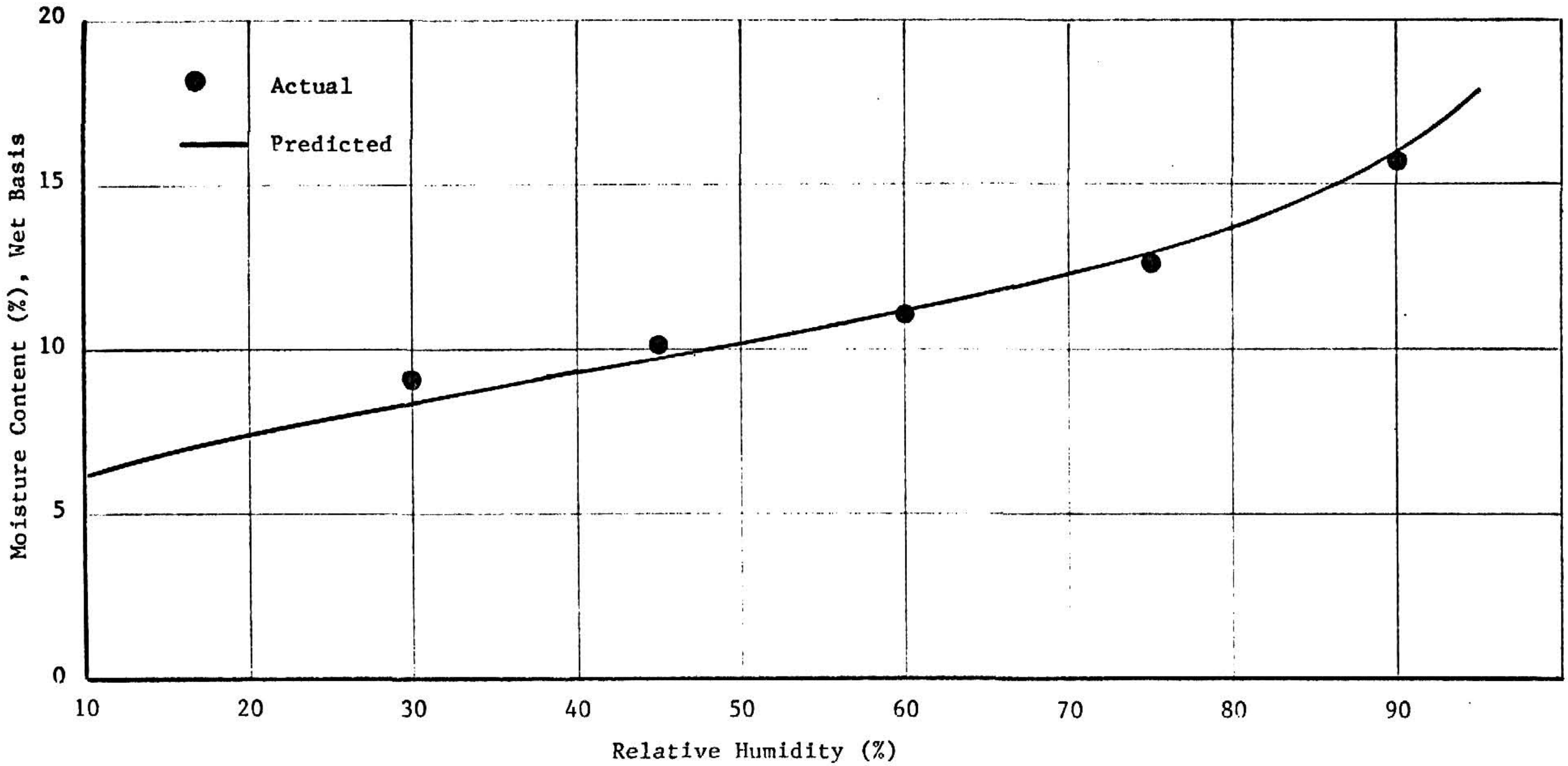

Figure 12. Comparison of Actual Data with Chung-Pfost Equation, Rough Rice Tested Over Ethylene Glycol at 30°C.

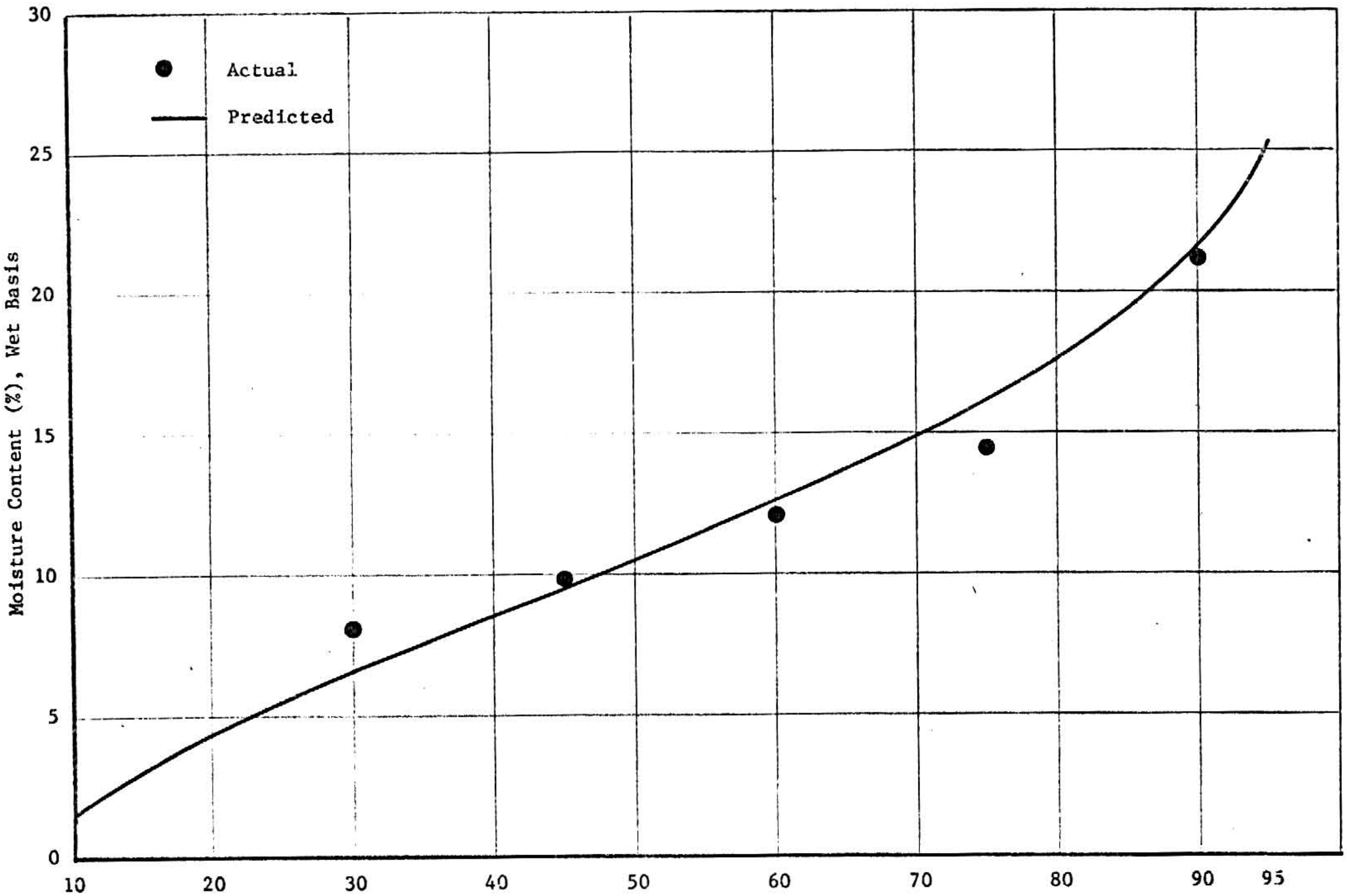

Figure 13. Comparison of Actual Data with Chung-Pfost Equation, Black Beans Tested Over Sulfuric Acid at 30°C.

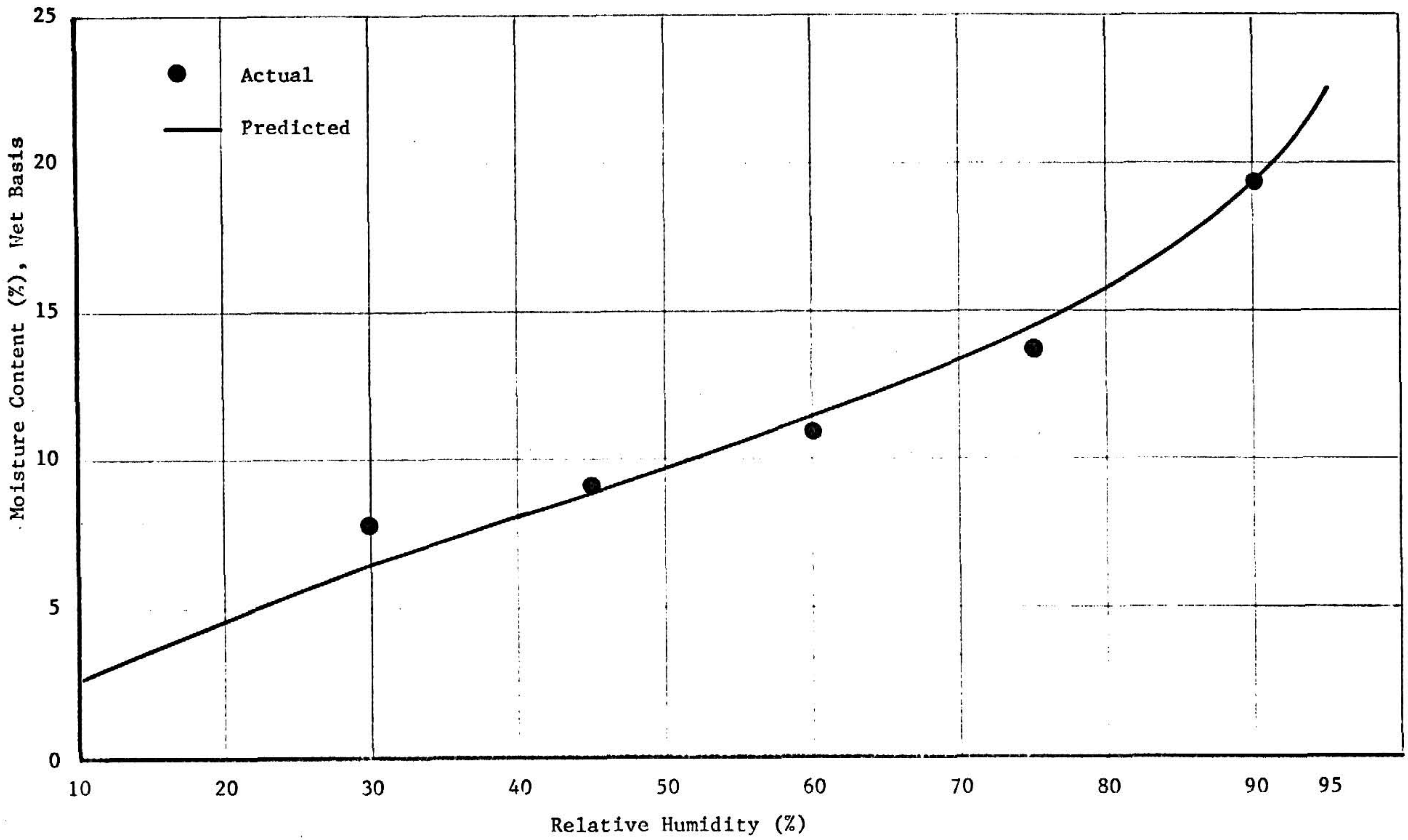

Figure 14. Comparison of Actual Data with Chung-Pfost Equation, Black Beans Tested Over Ethylene Glycol at 30°C.

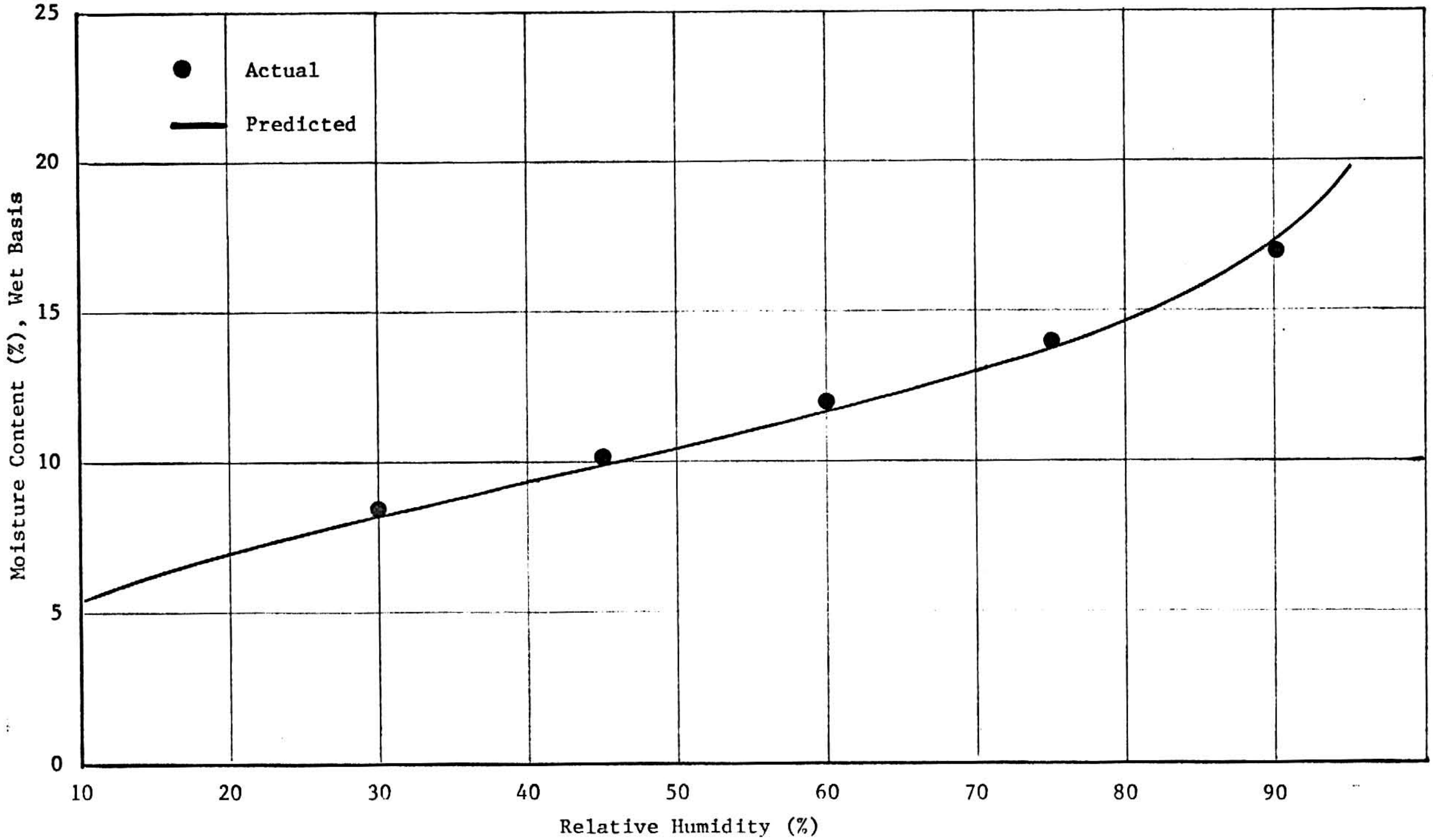

Figure 15. Comparison of Actual Data with Chung-Pfost Equation, Yellow Dent Corn Tested Over Sulfuric Acid at 30°C.

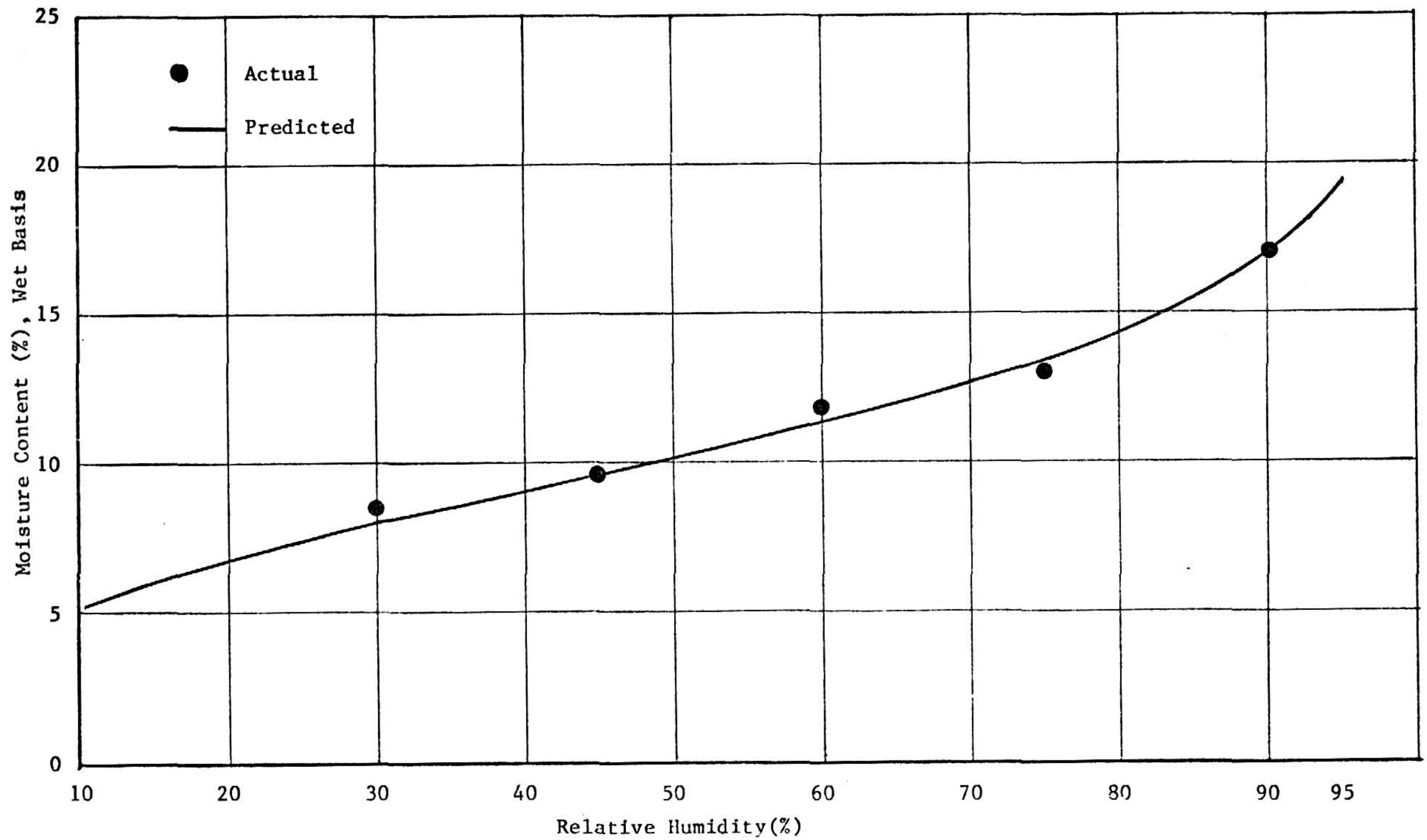

Figure 16. Comparison of Actual Data with Chung-Pfost Equation, Yellow Dent Corn Tested Over Ethylene Glycol at 30°C.

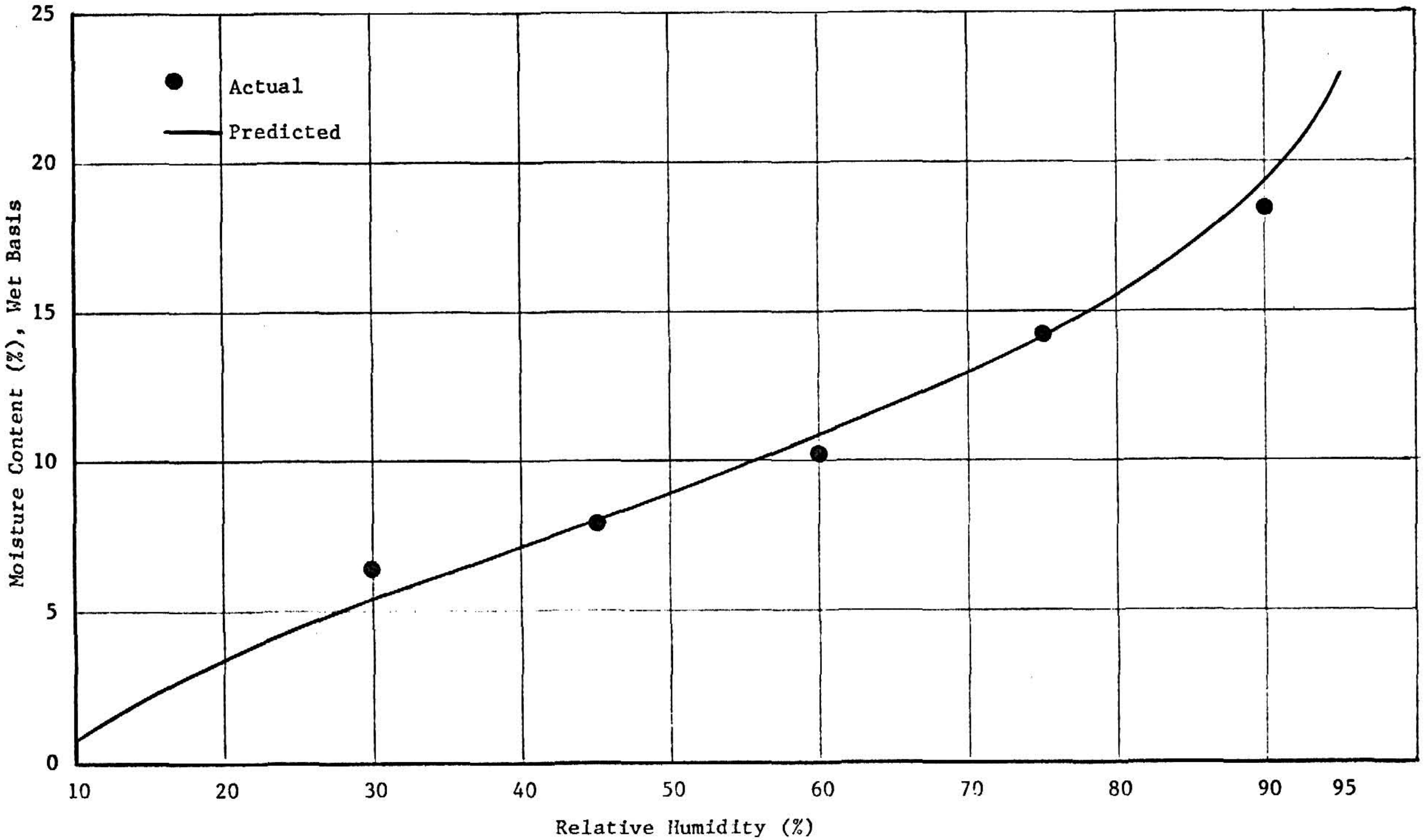

Figure 17. Comparison of Actual Data with Chung-Pfost Equation, Yellow Soybeans Tested Over Sulfuric Acid at 30°C.

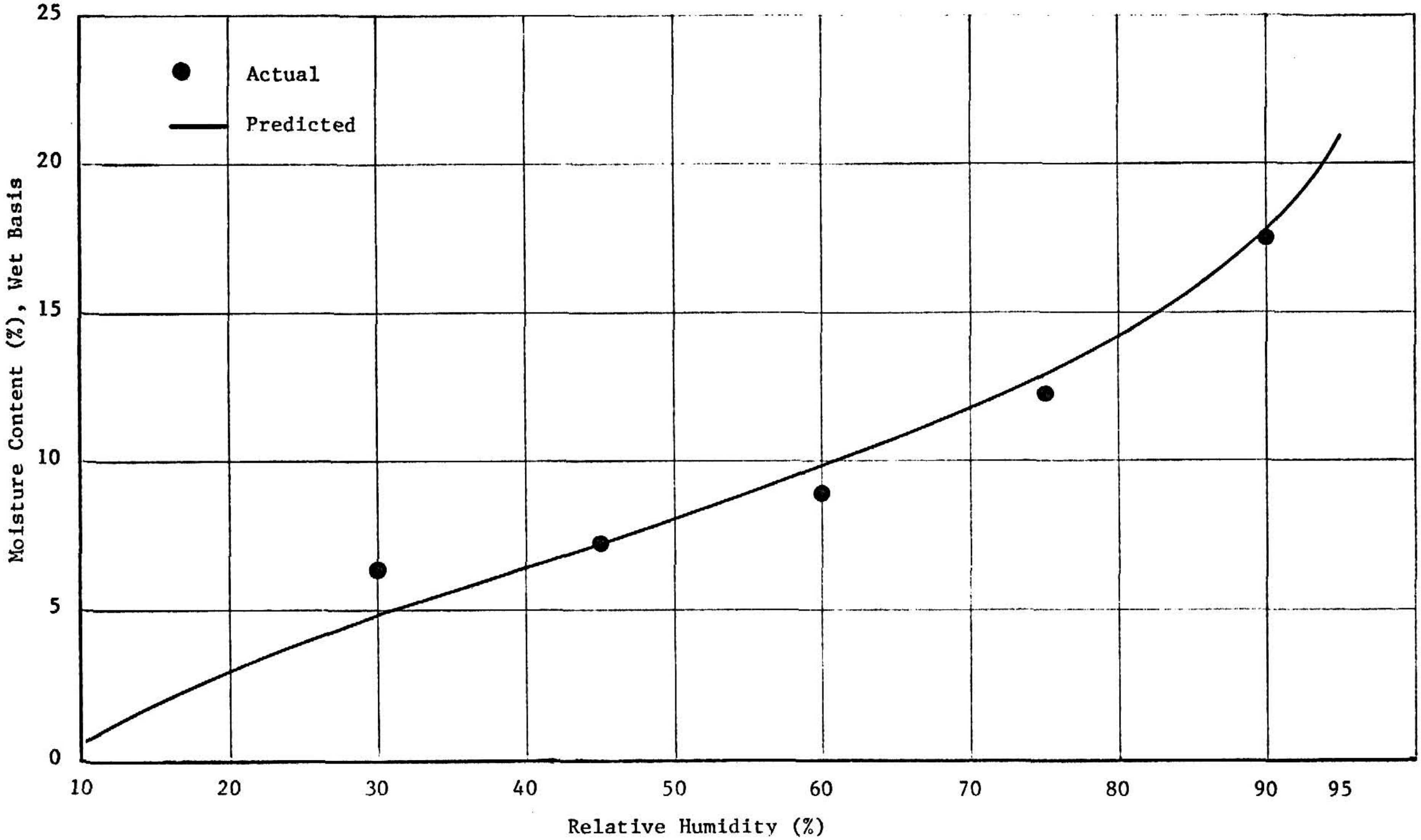

Figure 18. Comparison of Actual Data with Chung-Pfost Equation, Yellow Soybeans Tested Over Ethylene Glycol at 30°C.

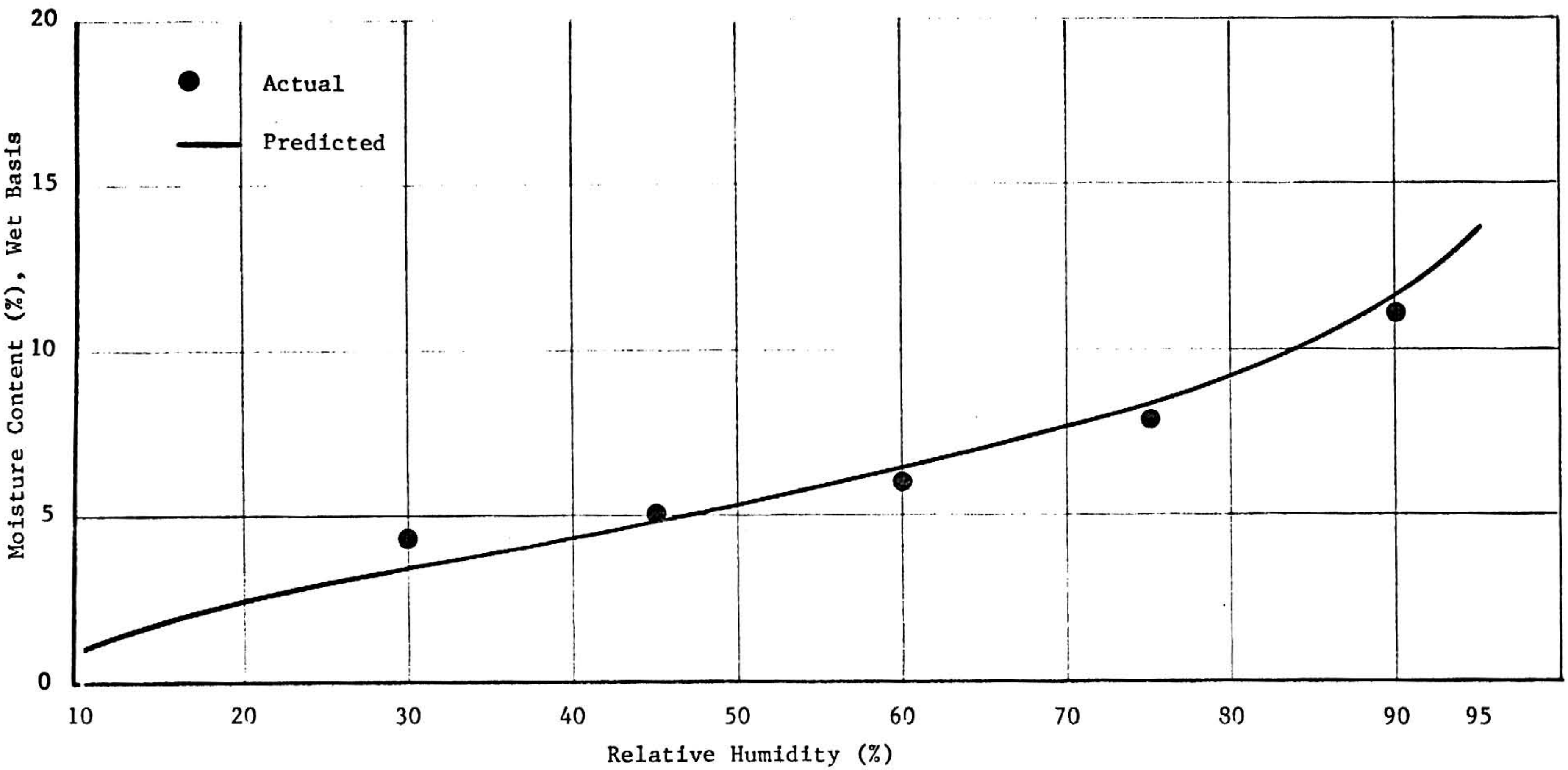

Figure 19. Comparison of Actual Data with Chung-Pfost Equation, Spanish Peanuts Tested Over Sulfuric Acid at 30°C.

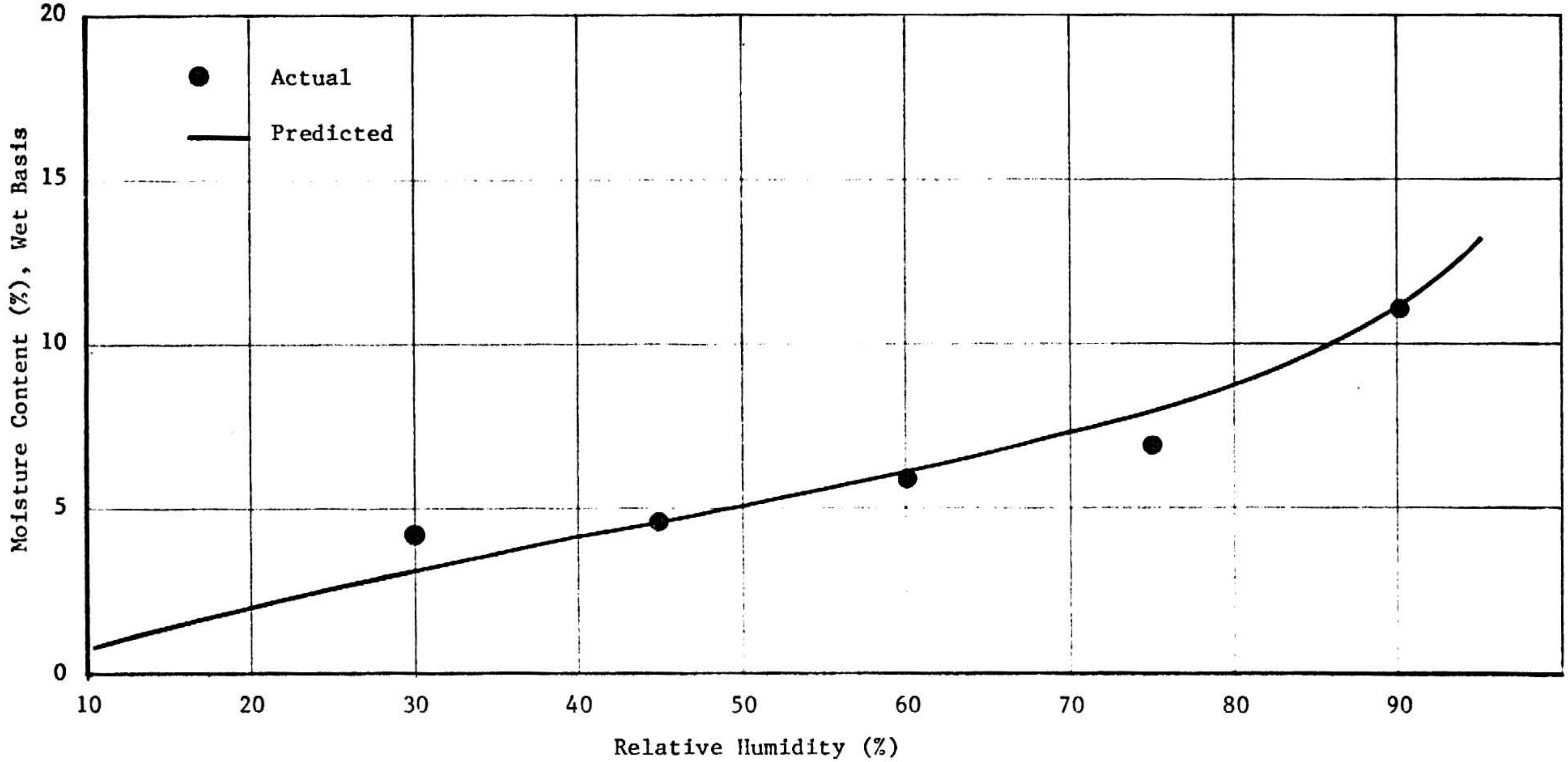

Figure 20. Comparison of Actual Data with Chung-Pfost Equation, Spanish Peanuts Tested Over Ethylene Glycol at 30°C.

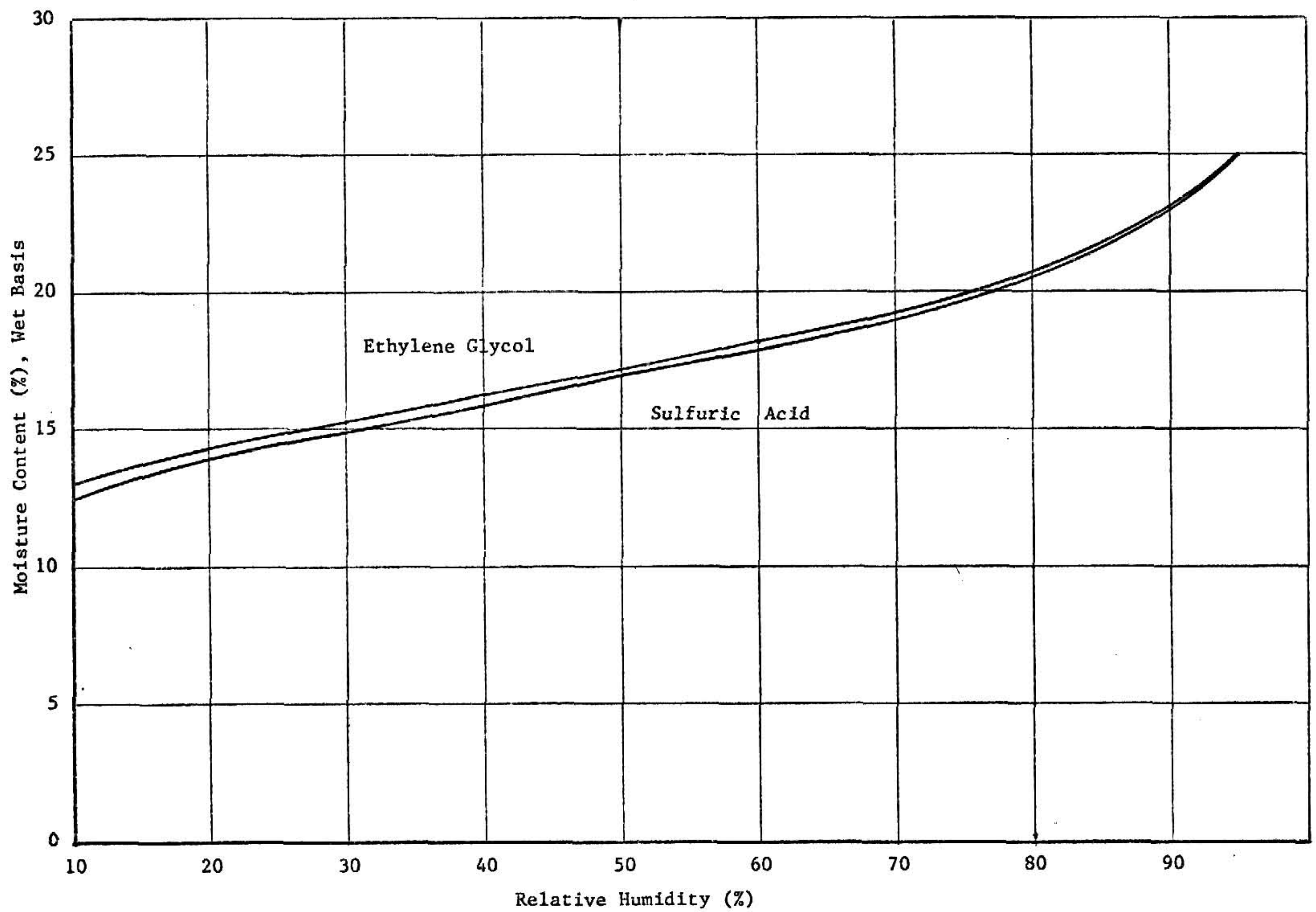

Figure 21. Comparison of Results with Different Solutions, Yellow Dent Corn Tested at 0°C.

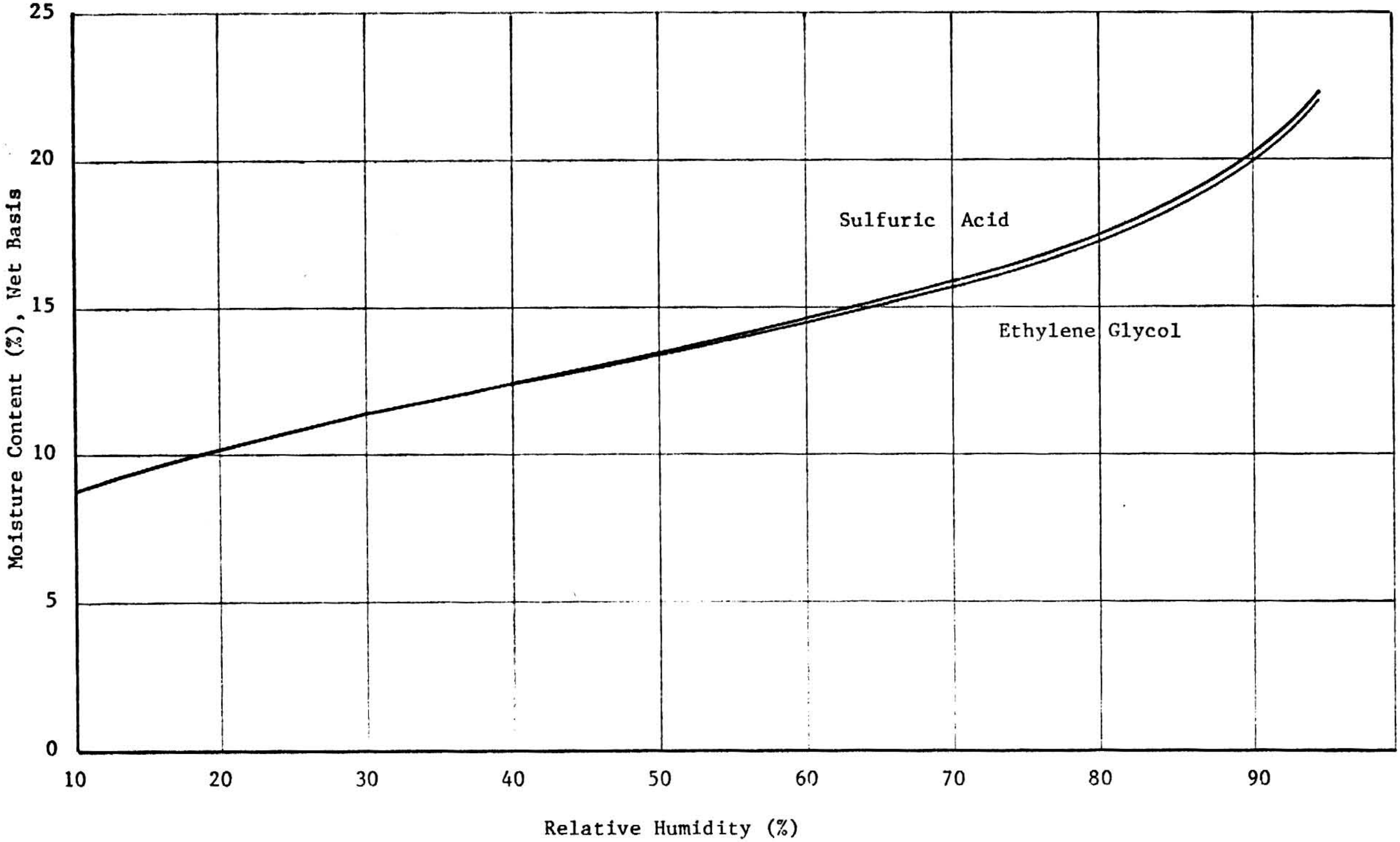

Figure 22. Comparison of Results with Different Solutions, Yellow Dent Corn Tested at 10°C.

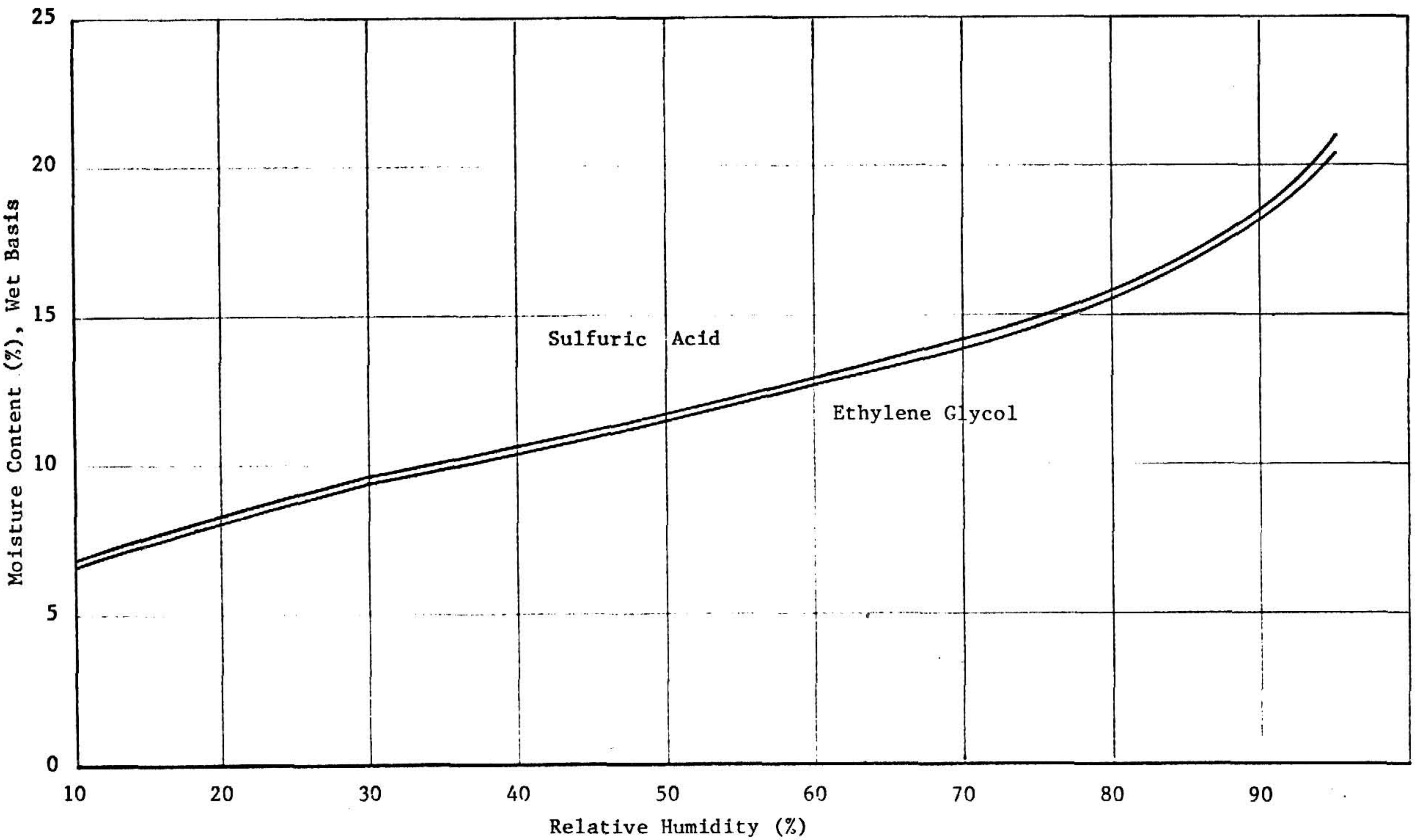

Figure 23. Comparison of Results with Different Solutions, Yellow Dent Corn Tested at 20°C.

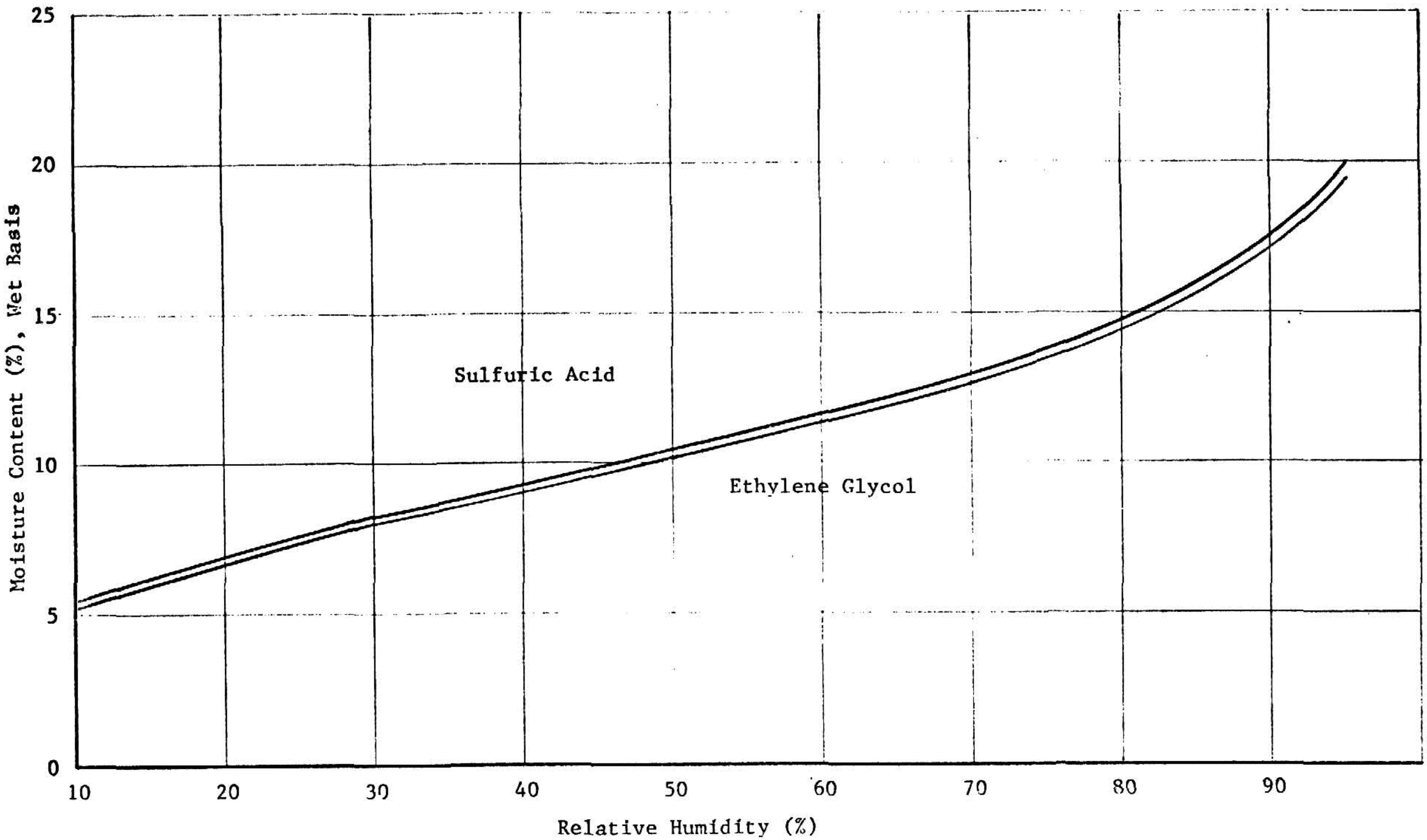

Figure 24. Comparison of Results with Different Solutions, Yellow Dent Corn Tested at 30°C.

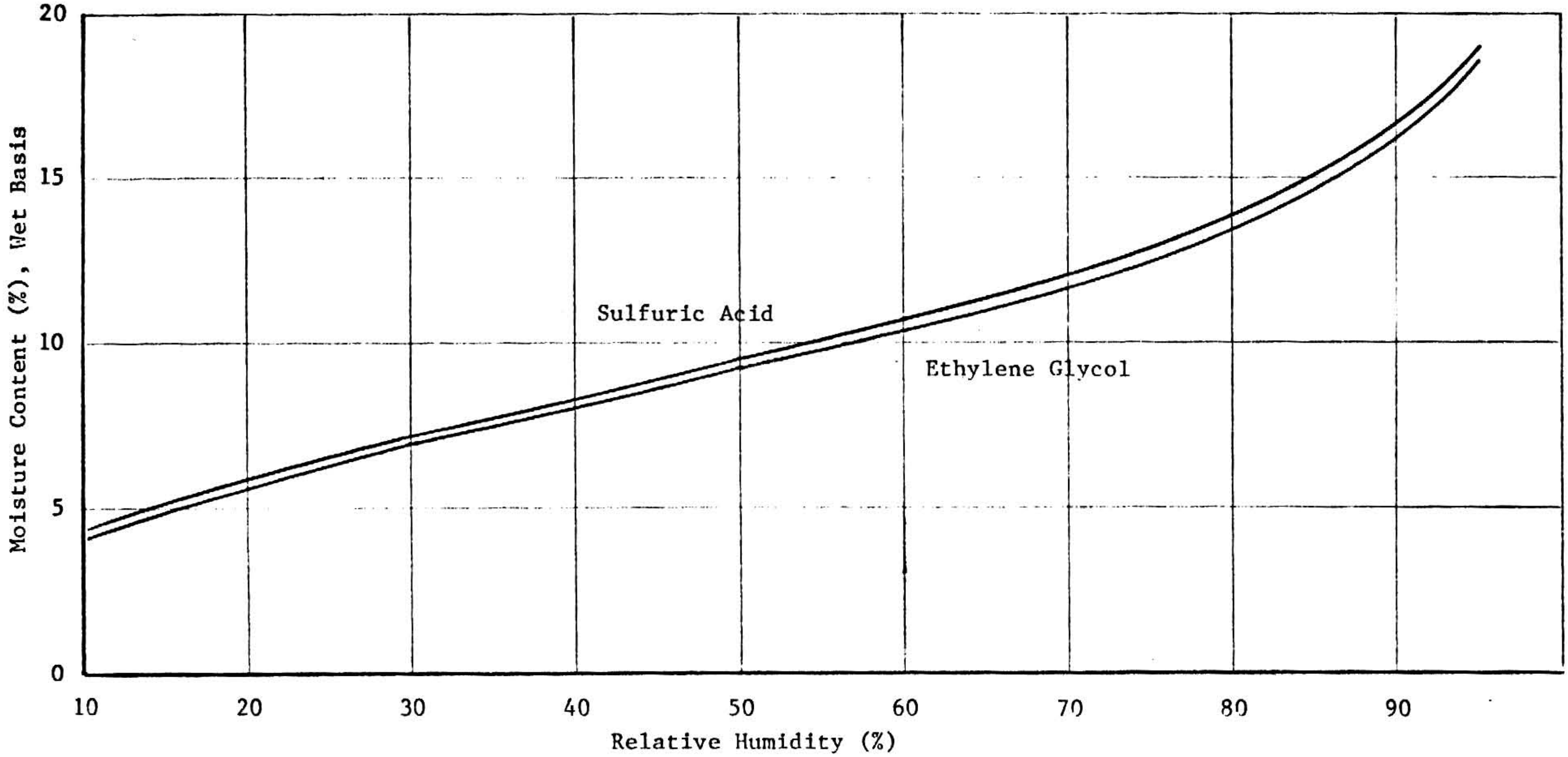

Figure 25. Comparison of Results with Different Solutions, Yellow Dent Corn Tested at 40°C

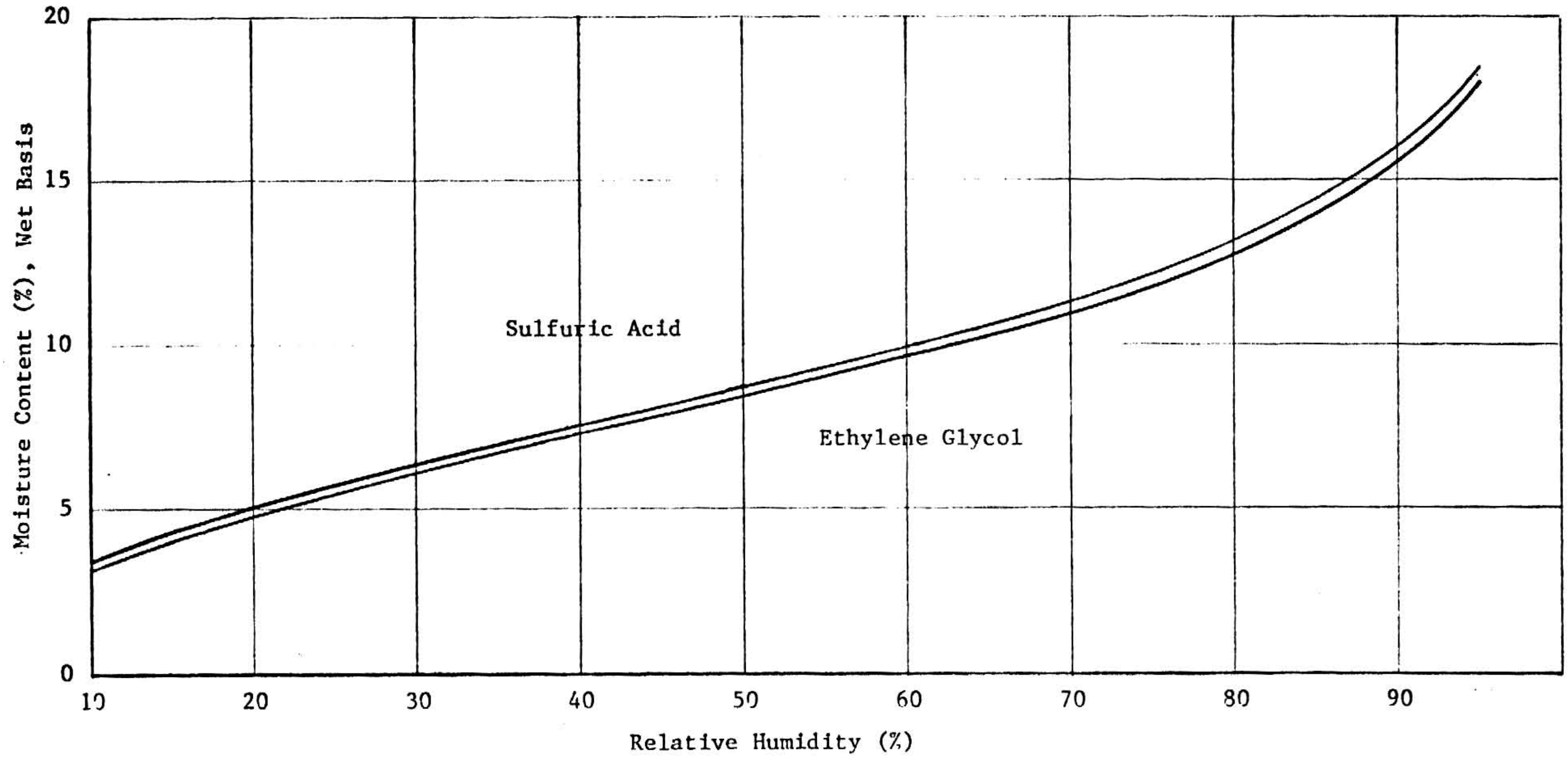

Figure 26. Comparison of Results with Different Solutions, Yellow Dent Corn Tested at 50°C.

Table 14. Statistical test for yellow dent corn.

Combination	d.f.	SS
EYDC	27	0.00070246
SYDC	27	0.00072359
EYDC + SYDC	~~27~~ 56	0.00162190

H_o: EYDC = SYDC

H_A: EYDC ≠ SYDC

Reject H_o if and only if $F_c \geq F_{.99}$ (3, 54)

SSE (1) = EYDC (SS) = 0.00070246

SSE (2) = SYDC (SS) = 0.00072359

SSE (0) = (EYDC + SYDC) = 0.00162190

SSE = SSE (1) + SSE (2) = 0.00142605

SS_{H_o} = SSE (0) - SSE = 0.00019585

$$F_c = \frac{0.00019585/3}{0.00142605/54} = 2.29$$

$F_{.99}$ (3, 54) = 4.13

We accept H_o

Conclusion:

Sulfuric acid and ethylene glycol solution data for yellow dent corn at 30°C is not significantly different at 99% confidence level.

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obs: EYDC = Ethylene glycol solution, yellow dent corn

SYDC = Sulfuric acid solution, yellow dent corn

Table 15. Statistical test for Spanish peanuts.

Combination	d.f.	SSE
E Peanuts	27	0.00254764
S Peanuts	27	0.00192981
E Peanuts + S Peanuts	57	0.00465870

H_o: E Peanuts = S Peanuts

H_A: E Peanuts $\neq$ S Peanuts

Reject H_o if and only if $F_c \geq F_{.99}$ (3, 54)

SSE (0) = 0.00465870

SSE = 0.00447745

SSH_o = 0.00018125

F_c = 18.00

$F_{.99}$ (3, 54) = 4.13

We reject H_o

Conclusion:

Sulfuric acid and ethylene glycol solution data for Spanish peanuts at 30°C are significantly different at 99% confidence level.

Table 16. Statistical test for black beans.

Combination	d.f.	
E B Bean	27	0.00445893
S B Bean	27	0.00500272
E B Bean = S B Bean	57	0.01838675

H_o: E B Bean = S B Bean

H_A: E B Bean ≠ S B Bean

Reject H_o if and only if $F_c \geq F_{.99}$ (3, 54)

SSE (0) = 0.01838675

SSE = 0.00946165

SS_{H_o} = 0.0089251

F_c = 16.98

$F_{.99}$ (3, 54) = 4.13

We reject H_o

Conclusion:

Sulfuric acid and ethylene glycol solution data for black beans at 30^oC are significantly different at 99% confidence level.

Table 17. Statistical test for rough rice.

Combination	d.f.	SSE
E R Rice	27	0.00188795
S R Rice	27	0.00154011
E R Rice + S R Rice	57	0.00433968

H_o: E R Rice = S R Rice

H_A: E R Rice ≠ S R Rice

Reject H_o if and only if $F_c \geq F_{.99}$ (3, 54)

SSE (0) = 0.00433968

SSE = 0.00342806

SS_{H_o} = 0.00091162

F_c = 4.79

$F_{.99}$ (3, 54) = 4.13

We reject H_o

Conclusion:

Sulfuric acid and ethylene glycol solution data for rough rice at 30°C are significantly different at 99% confidence level.

Table 18. Statistical test for soybeans.

Combination	d.f.	SSE
E Soy	27	0.00370475
S Soy	27	0.00309562
E Soy + S Soy	57	0.00930378

H_o: E Soy = S Soy

H_A: E Soy ≠ S Soy

Reject H_o if and only if $F_c \geq F_{.99}$ (3, 54)

SSE (0) = 0.00930378

SSE = 0.00680037

SS_{H_o} = 0.06250341

F_c = 6.3

$F_{.99}$ (3, 54) = 4.13

We reject H_o

Conclusion:

Sulfuric acid and ethylene glycol solution data for soybeans at 30°C is not significantly different at 99% confidence level.

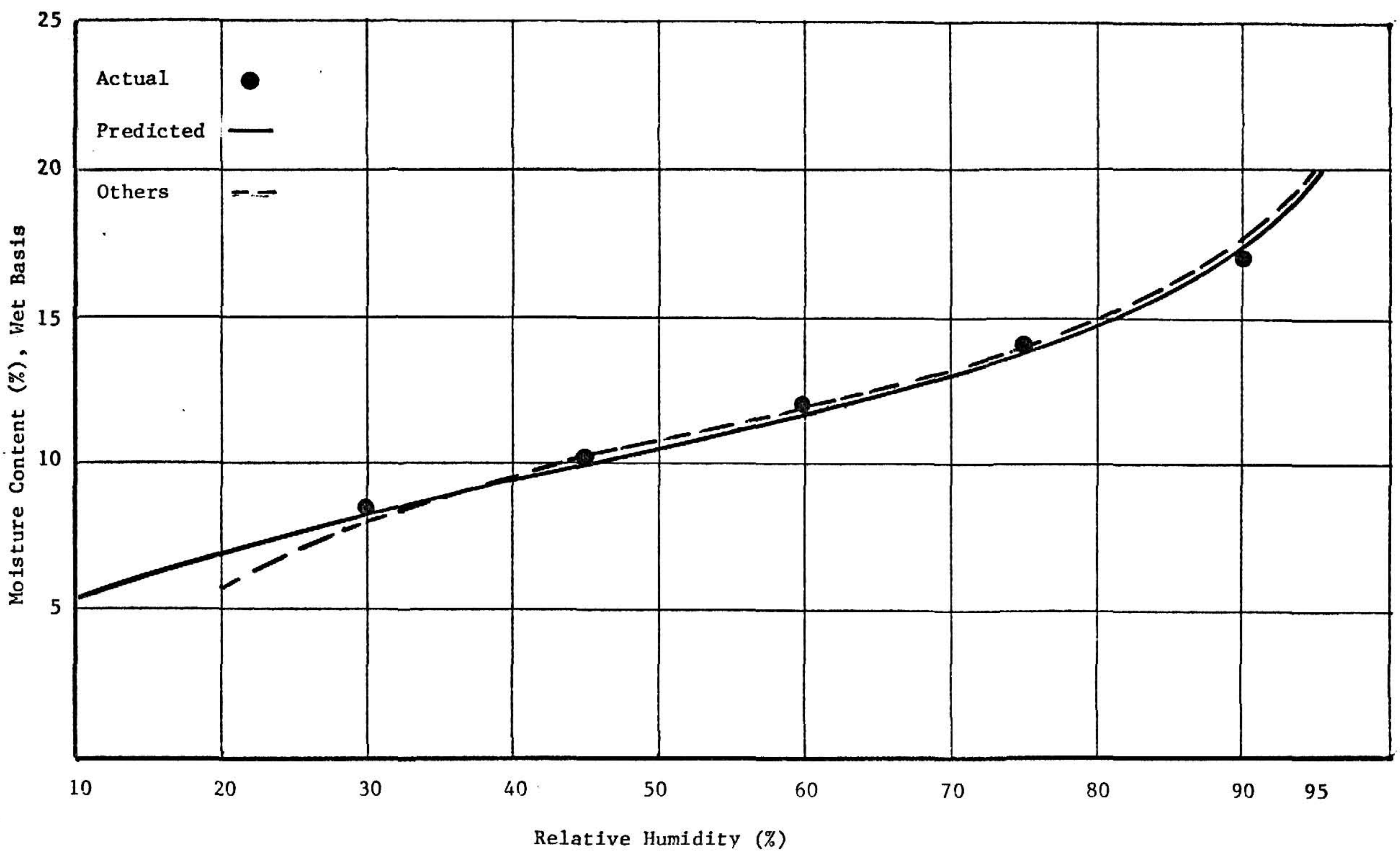

Figure 27. Predicted Equilibrium Moisture Contents of Corn at 30°C as Compared with observed data.

Conclusions

From the results of the study the following conclusions can be made.

1. That the sulfuric acid solution gave a higher equilibrium moisture data than that of the ethylene glycol solution at the temperature range studied (10^o - 38^oC).
2. The equilibrium moisture content of yellow dent corn in either sulfuric acid or ethylene glycol solutions were statistically similar. Meanwhile, the equilibrium moisture content in either solution of rough rice, black beans, soybeans and peanuts were significantly different.
3. That ethylene glycol can be conveniently used to provide the desired relative humidity of air in an enclosed container. The added advantage is its non-corrosiveness.
4. Because of the relative closeness of the specific gravities of ethylene glycol and water (1.1176 and 1.0, respectively) as compared to 1.8305 for sulfuric acid, their actual concentrations for a desired relative humidity might have differed from the expected, hence the equilibrium moisture deviations.

Recommendations

It is recommended that future studies on the equilibrium moisture content of grains using solutions, the actual relative humidity in the chamber be determined rather than assuming that the desired relative humidity existed.

Acknowledgements

The author wishes to express his deep gratitude and appriciation to Companhia Brasileira de Armazenamento-Cibrazem for their support in his training and in the completion of this work.

Sincere appreciation and heartfelt gratitude goes to the author's major professor, Dr. Harry B. Pfost whose guidance and encouragement led to the completion of his studies. He is grateful to the other members of his advisory committee: Drs. Do Sup Chung and David Sauer for their assistance and advice through the course of his program.

A special thanks to the U. S. D. A. Grain Marketing and Research Center, Manhattan, Kansas for allowing him to use their facilities in this study and to the various technicians and personnel for their assistance.

To Carolyn Beaudet for typing this thesis and to his friends who in one way or another helped him in this endeavor, a special appreciation.

Finally, to his beloved wife Vera and daughter Rafaela for their moral support and encouragement throughout the course of his work a deepest sense of gratitude.

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APPENDIX

```
C     THIS PROGRAM CALCULATES CHANGE IN THE CONCENTRATION
C      OF THE SOLUTIONS USED TO MAINTAIN HUMIDITY IN CLOSED SPACES
       DIMENSION VOLA(30),WTI(10),WTF(10),DELTAW(30),TEMP(30),HUM(30)
      1,SUMI(30),SUMF(30),CONSF(30),CONSI(30)
   10 FORMAT(3F10.3)
   11 FORMAT(10F8.3)
   12 FORMAT('1','CHANGE IN THE CONCENTRATION OF SOLUTIONS AFTER EXPERME
     1NT')
   14 FORMAT('0','SULFURIC ACID SOLUTION')
   15 FORMAT('0',3X,'S-NO',4X,'TEMP-C',3X,'HUMIDITY',3X,'INTIAL-WT',3X,
     1'FINAL-WT',3X,'DIFFERENCE',3X,'INTIAL-CONC',3X,'FIAL-CONC')
   16 FORMAT('0',3X,I2,2(6X,F5.2),5(4X,F8.3))
      WRITE(6,12)
      WRITE(6,14)
      WRITE(6,15)
C     INTIALIZE THE SPECIFIC GRAVITY OF THE SOLUTON, TOTAL VOLUME
      SPGA=1.84
      VOLS=1000.
C     INPUT DATA SEQUENCE
C     TEMPERATURE, RELATIVE HUMIDITY, VOLUME OF THE SOLUTION
C     INTIAL WEIGHTS, FINAL WEIGHTS, OF THE SAMPLES
      N=30
C     N IS THE NUMBER OF SOLUTIONS
      DO 100 I=1,N
      READ(5,10) TEMP(I),HUM(I),VOLA(I)
      SUM=0.0
      SUMIT=0.0
      M=10
C     M IS THE NUMBER OF SAMPLES ABOVE EACH SOLUTION
      READ(5,11) (WTI(J),J=1,M)
      READ(5,11) (WTF(J),J=1,M)
C     ADD UP INTIAL AND FINAL WEIGHTS
      DO 101 K=1,M
      SUM=SUM+WTI(K)
      SUMIT=SUMIT+WTF(K)
  101 CONTINUE
      SUMI(I)=SUM
      SUMF(I)=SUMIT
C     TAKE THE DIFFERENCE
      DELTAW(I)=SUMF(I)-SUMI(I)
C     FIND THE WEIGHT OF SOLUTION
      DUMY=VOLA(I)*SPGA
C     WTWA IS WEIGHT OF WATER IS SAME AS VOLUME
      WTWA=VOLS-VOLA(I)
C     WTFIN IS WEIGHT OF WATER AFTER ADDING THE CHANGE
C     FIND INTIAL CONCENTRATION
      CONSI(I)=DUMY/(DUMY+WTWA)
      WTFIN=WTWA-DELTAW(I)
C     FIND FINAL CONCENTRTION
      CONSF(I)=DUMY/(DUMY+WTFIN)
  100 CONTINUE
      DO 102 L=1,N
      WRITE(6,16) L,TEMP(L),HUM(L),SUMI(L),SUMF(L),DELTAW(L),CONSI(L),
     1CONSF(L)
  102 CONTINUE
      STOP
      END
```

Appendix A. **Change in solution concentration due to moisture movement between samples and solutions.**

Sulfuric acid solutions

S-NO	Temperature °C	Humidity	Total Sample Weight, gm. Initial Weight	Total Sample Weight, gm. Final Weight	Difference	Initial Concentration	Final Concentration
1	38.00	90.00	124.29	132.22	7.94	0.199	0.201
2	38.00	75.00	116.97	120.28	3.31	0.319	0.320
3	38.00	60.00	111.54	112.17	0.63	0.407	0.407
4	38.00	45.00	114.34	113.56	- 0.77	0.484	0.484
5	38.00	30.00	116.13	114.39	- 1.74	0.551	0.550
6	30.00	90.00	122.30	132.28	9.98	0.168	0.170
7	30.00	75.00	125.52	129.26	3.73	0.313	0.315
8	30.00	60.00	131.83	132.81	0.98	0.398	0.398
9	30.00	45.00	138.39	136.95	- 1.45	0.480	0.480
10	30.00	30.00	139.66	137.04	- 2.63	0.551	0.550
11	25.00	90.00	141.93	151.15	9.22	0.168	0.170
12	25.00	75.00	114.61	117.57	2.96	0.314	0.315
13	25.00	60.00	128.20	129.01	0.81	0.398	0.398
14	25.00	45.00	135.99	124.58	-11.41	0.476	0.472
15	25.00	30.00	125.60	123.40	- 2.20	0.551	0.550
16	20.00	90.00	116.29	125.03	8.74	0.168	0.170
17	20.00	75.00	125.69	129.37	3.68	0.314	0.315
18	20.00	60.00	113.21	113.98	0.77	0.401	0.402
19	20.00	45.00	125.39	124.70	- 0.69	0.477	0.476
20	20.00	30.00	131.70	129.48	- 2.23	0.546	0.545

Sulfuric acid solutions

S-NO	Temperature °C	Humidity	Total Sample Weight, gm. Initial Weight	Final Weight	Difference	Initial Concentration	Final Concentration
21	15.00	90.00	203.64	216.62	12.98	0.188	0.191
22	15.00	75.00	214.90	221.19	6.29	0.314	0.315
23	15.00	60.00	209.93	212.36	2.43	0.401	0.402
24	15.00	45.00	210.24	210.04	- 0.20	0.469	0.469
25	15.00	30.00	186.36	184.29	- 2.07	0.546	0.545
26	10.00	90.00	189.61	193.53	3.92	0.188	0.189
27	10.00	75.00	208.71	210.71	2.00	0.314	0.314
28	10.00	60.00	178.56	178.72	0.16	0.401	0.401
29	10.00	45.00	173.21	167.62	- 5.59	0.469	0.467
30	10.00	30.00	211.34	204.41	- 6.93	0.546	0.543

Appendix A. Change in solution concentration due to moisture movement between samples and solutions.

Ethylene glycol solutions

S-NO	Temperature °C	Humidity	Total Sample Weight, gm.		Difference	Initial Concentration	Final Concentration
			Initial Weight	Final Weight			
31	38.00	90.00	125.71	132.70	6.99	0.471	0.473
32	38.00	75.00	113.96	116.06	2.10	0.596	0.697
33	38.00	60.00	100.72	101.34	0.62	0.810	0.809
34	38.00	45.00	108.46	107.66	- 0.79	0.880	0.880
35	38.00	30.00	105.79	104.35	1.44	0.926	0.926
36	30.00	90.00	143.28	141.92	7.64	0.477	0.479
37	30.00	75.00	141.98	145.01	3.02	0.702	0.703
38	30.00	60.00	122.93	123.07	0.14	0.813	0.813
39	30.00	45.00	125.16	124.00	- 1.16	0.883	0.882
40	30.00	30.00	130.68	127.83	- 2.85	0.927	0.926
41	25.00	90.00	136.38	144.47	8.09	0.482	0.485
42	25.00	75.00	118.54	120.45	1.91	0.733	0.734
43	25.00	60.00	128.09	128.22	0.13	0.818	0.818
44	25.00	45.00	122.27	120.98	- 1.29	0.886	0.886
45	25.00	30.00	116.89	114.41	- 2.48	0.928	0.927
46	20.00	90.00	127.41	135.97	8.56	0.490	0.493
47	20.00	75.00	130.01	131.84	1.83	0.718	0.719
48	20.00	60.00	130.42	130.49	0.07	0.826	0.826
49	20.00	45.00	139.04	137.69	- 1.35	0.888	0.887
50	20.00	30.00	137.23	134.43	- 2.80	0.929	0.928

Ethylene glycol solutions

S-NO	Temperature	Humidity	Total Sample Weight, gm.		Difference	Initial Concentration	Final Concentration
			Initial Weight	Final Weight			
51	15.00	90.00	154.21	162.56	8.35	0.492	0.495
52	15.00	75.00	161.86	165.52	3.66	0.725	0.727
53	15.00	60.00	161.74	162.31	0.57	0.832	0.832
54	15.00	45.00	181.87	180.64	- 1.23	0.890	0.889
55	15.00	30.00	179.06	177.01	- 2.05	0.930	0.929
56	10.00	90.00	185.09	198.32	13.23	0.519	0.524
57	10.00	75.00	187.51	190.17	2.66	0.738	0.719
58	10.00	60.00	192.50	194.48	1.98	0.836	0.837
59	10.00	45.00	197.86	196.58	- 1.28	0.893	0.892
60	10.00	30.00	188.65	186.40	- 2.25	0.930	0.929

TWO METHODS OF COMPARING EQUILIBRIUM MOISTURE OF GRAINS

by

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B. S., Faculdade De Agronomia Da Universidade Federal Do Parana, 1971

AN ABSTRACT OF A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Grain Science and Industry

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1977

Many aspects of equilibrium moisture content for biological materials have been studied. Such aspects are fundamental and important in the handling, drying and storing of cereals, legumes and oilseeds. Insect, fungi and bacteria control in the handling, processing and storage of flour and other grain products are related also to moisture content.

Attempts to determine equilibrium moisture content were made specifically on agricultural products because they gain or lose moisture depending on certain conditions of temperature and relative humidity.

Chemical compounds have been used to create an atmospheric condition to make possible the study hygroscopicity through relative humidity control.

Sulfuric acid and ethylene glycol solutions were used to control the relative humidity, although their behavior is different, the cause of this phenomenon is unknown.

Our research is designed to show the suitability of sulfuric acid and ethylene glycol solutions to control the relative humidity in closed chambers, to supply data under different conditions, and to determine the fit of our data to existing concepts and models. The research resulted in isotherms for rough rice, black beans, yellow dent corn, Spanish peanuts and soybeans.