MOISTURE DETERMINATION IN RAISINS AND PRUNES

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The most abundant material in agricultural commodities is water. Measurement of the amount of water in these commodities has occupied an important place in processing and marketing. The scientific and technological literature contains many reports of studies of moisture determination methods. We believe it can truthfully be said that the "best" method is yet to be found.

Of the various methods for determining the water content of dehydrated foods, the vacuum oven method is most often relied upon as the standard or reference procedure. It has proved to be accurate and precise on samples over a wide range of moisture levels. For commercial applications where rapid, simple methods are required for production and quality control, the vacuum oven method is considered by many to be too time-consuming and difficult.

It is the purpose here to report the results of moisture analyses of raisins and prunes determined by a number of methods. A wide range of moisture levels is represented in these samples. The objective of this continuing work is to seek techniques in moisture determination.

Five analytical methods for moisture determination are included in this report:

(1) Vacuum oven.

(2) Desiccation with magnesium perchlorate in vacuo.

(3) Nuclear magnetic resonance.

(4) Infrared moisture balance.

(5) Dried fruit moisture tester.

Experimental

Materials

Raisins and prunes were obtained from commercial sources in thirty-pound cartons. The moisture content was adjusted when desired by rehydrating in boiling water or dehydrating at 150° F. in a forced draft air oven. Enough of a master sample of each moisture level was prepared for the entire study. The fruit was ground whole through a common food grinder and kept in hermetically sealed glass containers for analysis.

Methods of Analysis

Vacuum oven: Triplicate five-gram samples were dried for thirty hours at 60° C. and a pressure of less than 5 mm. of mercury. The procedure is a slight modification of the method of the Association of Official Agricultural Chemists (2) for dried fruit.

Infrared Moisture Balance: This is a small self-contained unit, calibrated to read weight loss as percent moisture content. Heat from an infrared lamp, regulated by the amount of current supplied to the element, desiccates the fruit sample on the pan of the built-in balance. Time and temperature relationships for each fruit were determined in preliminary tests. Raisins containing 20% moisture required 20 minutes at 85 volts. At higher moisture levels, it was necessary to lengthen the time to 25 minutes. Replicate analyses were made at each moisture level.

Dried Fruit Moisture Tester: The method as described by the Dried Fruit Association was used (6). It relates conductivity of the sample with its moisture content as determined by the vacuum oven.

Nuclear Magnetic Resonance (NMR): The method employed for this study is similar to that used by Elsken and Shaw (3, 4) for determining moisture in substances of biological origin. Although NMR has been used, in some instances, without reference to a primary standard (5), it was necessary in analyzing dried fruit to correlate the results with a reference method. The vacuum oven method was employed for this purpose.

Results and Discussion

The comparative results of the analyses, with the exception of the NMR values, can be seen in Table I. For the purpose of the discussion which follows, the magnesium perchlorate method has been considered to be the standard or reference of comparison.

Drying by Desiccant: Extraction of water by magnesium perchlorate was considered to be complete in all samples after about ten months (see Figs. 1 & 2). At all moisture levels, this method gave lower results than the others, with the exception of analyses obtained with the D.F.A. moisture tester for samples R4 and R5. This may be due to the fact that dehydration by a desiccant at room temperature minimizes thermal destruction, loss of non-aqueous components, and chemical side reactions. It is thus possible that the method would give results nearest to the true moisture content of the dried fruits.

D.F.A. Tester:* For samples R4 and R5 the readings with the D.F.A. meter fell outside the limits of the existing tables which only go to 25% moisture. The moisture values therefore were obtained by extrapolation. Because these results are inconsistent with those obtained by other procedures employed in this study, it will be necessary to modify the electrical instrument if it is to be used for very high moisture raisins. Otherwise, this method provides

* For high moisture prunes the electrical circuit of the tester was modified. This provided reproducible results, but gave higher values than did the moisture balance and vacuum oven.

reproducible results with more speed than any other technique used.

Infrared Moisture Balance: The procedure gave reproducible results, but required special time-temperature adjustments for samples too high or too low in moisture content. This is an adequate instrument if it is used for a limited moisture range and is calibrated against an accepted reference method.

Vacuum Oven: This procedure gave higher values for moisture than the magnesium perchlorate method even when lower oven temperatures were used. As reported by Makower (1) removal of moisture in the temperature range used in this procedure can be accompanied by thermal decomposition and possible side reactions as well as other errors resulting from loss of volatile oils. It would be necessary thus to reduce the sample residence time or the temperature of the oven if results comparable to magnesium perchlorate values are to be obtained. Otherwise, the accuracy of the method is quite good.

Nuclear Magnetic Resonance: NMR as applied to the measurement of the water content of raisins and prunes did not provide an adequate method in this study. High results were obtained because of the presence of soluble solids, and the contribution of their hydrogen nuclei to the nuclear magnetic absorption. The instrument gave values that showed interference by pits, seeds, and oils; thus, only qualitative measurements of moisture of dried fruits were possible.

This study is proceeding and will continue to be an important part of our dried fruit research program. Modifications of present methods are being investigated; different methods for determining moisture in dried fruits are being sought. Careful study of new techniques reported in the literature is an important part of our work on this problem. Water is certainly one of the most important constituents of all agricultural commodities. A great deal of scientific talent for many decades has been directed toward devising means for measuring it accurately. As the work moves along, we shall keep the industry informed through reports such as this and through contacts with technical personnel.

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6. Dried Fruit Association of California (unpublished data).

TABLE I

MOISTURE CONTENT OF RAISINS AS DETERMINED BY FOUR METHODS
(averages of three analyses)

SAMPLE #	DESICCATION (Perchlorate)	MOISTURE Balance (IR)	VACUUM Oven	D.F.A. Meter
R_1	12.29	13.3	13.14	13.0
R_2	9.18	10.3	9.92	10.6
R_3	18.28	21.2	21.09	19.9
R_4	27.28	29.0	29.1	25.9*
R_5	32.38	33.6	34.25	27.8*
R_6	14.06	16.2	16.12	16.0

*Extrapolated beyond charts

TABLE 1

MOISTURE CONTENT OF PRUNES AS DETERMINED BY FOUR METHODS
(averages of three analyses)

SAMPLE #	DESICCATION (Perchlorate)	MOISTURE Balance (IR)	VACUUM Oven	D.F.A. Meter
P_2	15.70	18.4	17.8	18.8
P_3	22.37	24.5	24.5	26.1
P_4	24.96	27.1	27.17	28.2
P_5	29.33	30.6	31.18	32.2

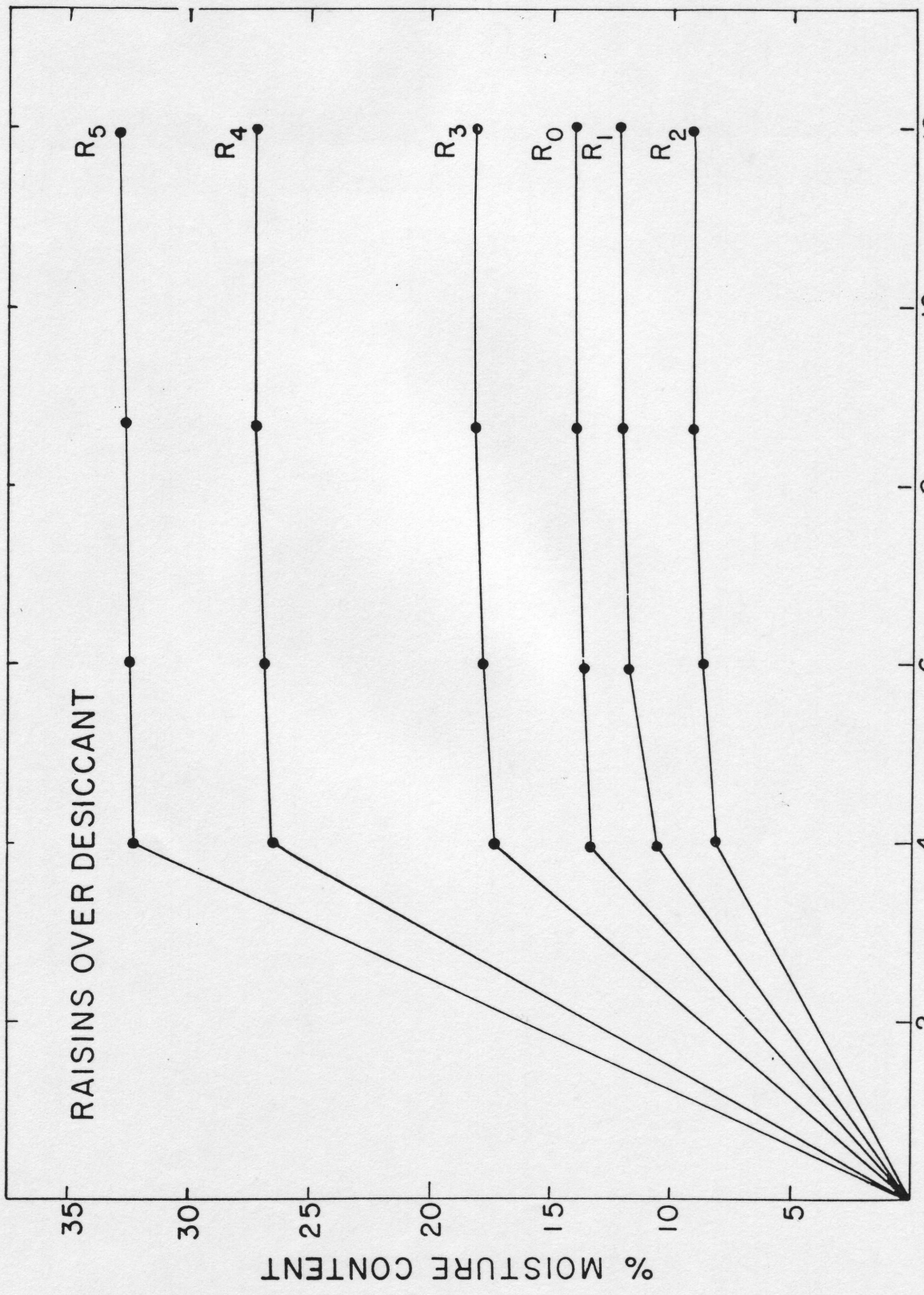
RAISINS OVER DESICCANT
R_5
R_4
R_3
R_0
R_1
R_2
% MOISTURE CONTENT
35
30
25
20
15
10
5
TIME IN MONTHS
2
4
6
8
10
12

FIG. 1

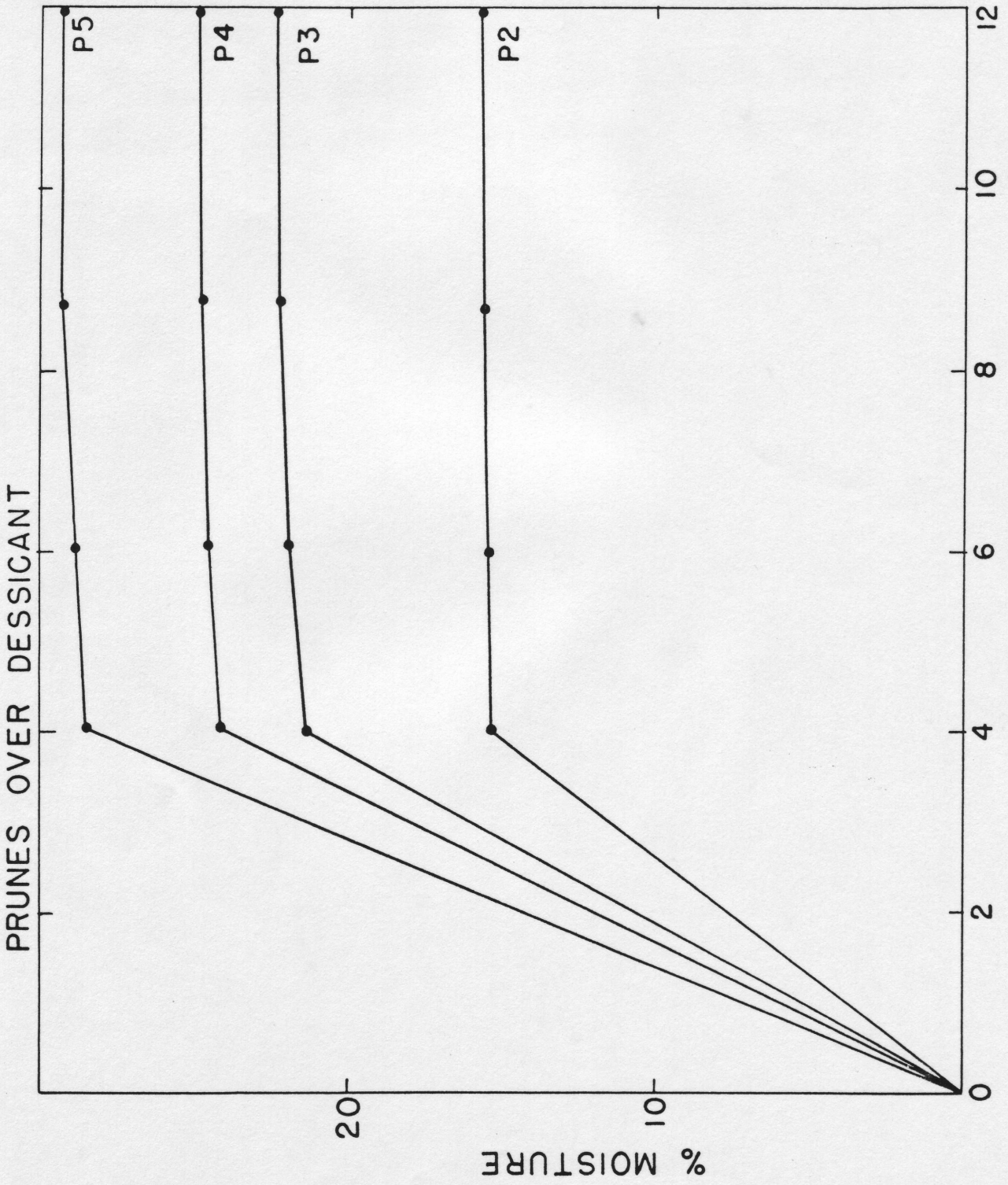
PRUNES OVER DESSICANT
P5
P4
P3
P2
% MOISTURE
20
10
0
2
4
6
8
10
12
TIME IN MONTHS

FIG.1