REPORT 7036

MOISTURE CONTENT DETERMINATION IN MILK POWDER.

A Report on Various Drying Ovens.

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1.0 INTRODUCTION

During a study on powdered milk, undertaken by the Food Research Institute and Production and Marketing, Dairy Division, certain anomalies appeared in the moisture content evaluation of samples. In an effort to determine the source of undue variation in evaluations, this study on the ovens, used for sample drying, was conducted.

Samples of the milk powder are graded for moisture content following accepted methods. In the method, (Sections 16.147, 16.148 and 16.149, A.O.A.C. Methods of Analysis) samples are dried for 5 hours in a vacuum oven at 100°C and at a pressure lower than 100 mm Hg, and the moisture content calculated from the loss in weight.

During initial testing, moisture content variations within homogeneous samples appeared to be associated with drying procedure. It was hypothesized that temperature variations within the drying ovens produced the observed variations, and tests to verify this were conducted on eight ovens used for the testing. Of the eight ovens, six were the vacuum type and two were forced air convection ovens. The latter two were included for comparative purposes.

2.0 TEST PROCEDURES

Temperatures recorded within the oven cavities were monitored using 36 ga. type T copper/constantan thermocouples located near the top surface at the centres of a 5 cm diameter, 0.635 cm thick aluminum discs. The top surfaces of the discs were painted white to simulate the radiant heat exchange of milk samples. Oven wall temperature measurements were monitored using thermocouples attached to the walls using an epoxy glue. Recording of temperature was with either a 24 point potentiometric recorder, or a 20 channel data logging system. Accuracy of recording was within at least $\pm 0.5^{\circ}C$.

Test 1. National #5840 Vacuum Oven.

Wall temperature test.

Twenty-four locations were monitored on the wall surfaces of the ovens with four thermocouples placed on each internal surface.

Two fix hour tests were conducted.

Test 2. National #5840 Vacuum Oven.

Spatial temperature distribution.

Five replications were run using ten thermocouple discs in a grid pattern on each of the two oven shelves. In one replication moisture contents were determined on twenty milk powder samples taken from a homogeneous batch.

Test 3. Gallenkamp B.S. O.V. 160 Forced convection air oven.

A 1.9 hour trial was conducted using this oven.

Fifteen thermocouples, five per shelf, were used in a pattern of one couple in each quadrant plus one in the centre.

Test 4. National #5840 Vacuum Oven.

Statistical trials.

Three replications of the standard five hour test were conducted. Eighteen thermocouple discs placed nine per shelf in rows of three were used to monitor temperature. Eighteen milk powder samples drawn from one homogeneous lot were used with each replication to evaluate moisture content variation. Each milk powder sample was placed beside a thermocouple.

Test 5. Cenco#95600A Oil Bath Vacuum Oven.

One five hour test was run recording the temperature at twenty locations within the oven. The oven had three shelves, and for the test 7 thermocouples were used on the top shelf, 8 on the second and five on the bottom. The shelves in this oven are of unequal size with the bottom one being quite small.

Test 6. National #5850 Vacuum Oven.

Three, five hour tests were run, two with narrow shelf spacing (the normal style) and one with wide shelf spacing. Temperatures were monitored at 18 locations, 9 on each shelf in a 3 x 3 grid.

Test 7. National #58501 Vacuum Oven

One five hour test was run as in Test 6 using the conventional shelf spacing.

Test 8. Precision Scientific - 1415 Vacuum Oven.

Two five hour tests were run using twenty thermocouples, ten on each shelf. In the first replication the oven had not fully stabilized at the start of the test. The second replication is rather idealized, as the oven was allowed to stabilize for two days before testing, and the oven was not opened prior to testing.

Test 9. National N-8607-1 Vacuum Oven.

This oven was similar to those of Tests 6 and 7 and the five hour test was run as in Test 6.

Test 10. Thelco Model 18 Forced Convection Air Oven.

One five hour trial was conducted using twenty thermocouples, ten per shelf.

Test 11. Napco #5861 Vacuum Oven.

Combined moisture and temperature test, as in Test 4 with three replications.

Test 12. Cenco #95600A Oil Bath Vacuum Oven.

Combined moisture and temperature test, as in Test 4 with three replications.

3.0 RESULTS

3.1 Temperature Variation

Wall temperature variations in the National Vacuum Oven #5840 were very pronounced at four of the twenty-four locations. The four thermocouples in the lower halves of the side walls showed cyclical variations in temperature of about 20°C at temperatures in the 125°C range. The wall temperatures toward the rear of the oven were higher than near the front by about 5°C at the lower level and by about 8°C at the higher level. On the ceiling and floor of the oven, cyclical variations were in the order of 2°C, but again with the 8°C differential between front and rear. Cyclical variation in the back wall temperature was low, but with 6°C differential between the upper and lower level. The location of higher temperatures along the lower areas with the increase towards the rear seems to correlate well with moisture readings obtained using this oven (Table 1).

A summary of the spatial temperature data obtained from the trialsfor the eight ovens is given in Table 2.

The vacuum ovens showed considerable spatial temperature variation, with the exception of the Cenco Oil Bath Oven. The Cenco oven has the vacuum chamber immersed in a thermostated oil bath to

achieve a very uniform and stable wall temperature. The spatial variation maximum of 2.5°C was better than the other ovens. The variation found with the Precision Scientific Oven (Test 08, Rep 02) was quite reasonable, however, this test is rather idealized, as the oven was not opened for loading prior to the test. The series of National Vacuum Ovens tested exhibited rather high degree of temperature variation. Oven #5840 of the Dairy Section at F.R.I. had variations in the order of 20°C which were excessive. The three National ovens of the Plant Products Division had average temperature spans between 11°C and 13°C with maximum span about 15°C. While still excessive in temperature variation, these latter three ovens were considerably improved over #5840.

In the force convection air circulation ovens (Test 03 & 10) much more uniform temperature distributions were observed with average temperature spans of about 1.5°C. The superior temperature uniformity is to be expected with this type of oven where the air circulates as a heat transfer media.

3.2 Temperature and Moisture Content

The spatial temperature variations within the ovens exhibit a definite influence on the moisture content readings of the milk powder, as recorded in different areas of the oven. Table 1 shows the pronounced differences obtained when 40 samples from a homogeneous lot were evaluated with one oven. The range of moisture content indicated from 3.31% to 3.91% is large. Table 3 presents the same data, but includes average representative temperatures in the various areas of the oven.

Compositing the data from Test 04, Reps 01, 02 & 03 in graphical form provides an indication of the influence of temperature on the apparent moisture content of a homogeneous milk powder sample (Fig. 1).

A strong trend is seen in the relationship between the temperature and the sample moisture content. A similar trend is observed in the data of the Napco Oven 5861 (Fig. 2). Statistical analysis of the data indicated that the temperature variation within the oven accounts for the differences in moisture contents observed. The analysis of covariance was performed on data adjusted for the variable temperature.

Tables 4, 5 and 6 show the summarized statistics for the three simultaneous moisture content/temperature trials with the three vacuum ovens. In the Cenco Oil Bath Oven the ranges for both temperature and moisture are quite low. The temperature spans on the average for the Cenco oven of 2.0°C were near to those of the forced convection air ovens (Test 03 & 10). For the other vacuum ovens the temperature variations were greater, as were the variations in moisture content. Maximum difference in moisture content found in the three ovens, when working with homogeneous samples, were: National 5840, 0.51%; Napco, 5861, 0.61%; Cenco Oil Bath, 0.32%. The regression coefficients (Tables 4, 5, 6) were found to be very low indicating that when the data was adjusted for temperature, almost all the variation was accounted for.

4.0 DISCUSSION

It is quite apparent that accurate temperature control is essential for adequate analysis of moisture in milk powder samples. While uniform temperature distribution within the chamber of force convection ovens is relatively easy to attain, vacuum ovens present a more difficult problem. Improvements have been made in the temperature distribution in some newer vacuum ovens. The stable and uniform temperature of the old Cenco Vacuum Oven is attributed to the thermostated oil bath which encloses the vacuum chamber.

Modifications to existing ovens to improve temperature uniformity are possible. In the air-jacketed ovens such as the National #5840, modifications to the heating system and a means to provide better air-jacket circulation should greatly improve temperature distribution.

Control of the spatial temperature uniformity within the ovens to a variation of less than 5°C would increase the accuracy of moisture content determination. Variations of no greater than 5°C by location within the oven cavity should reduce apparent moisture content variations in a homogeneous sample to about 0.2%.

5.0 RECOMMENDATIONS

Vacuum ovens are widely used for the determination of moisture contents in a wide variety of agricultural products. When these ovens are used in quality control and grading procedures, or other applications where accurate, repeatable results are required, it is advisable to carefully check each oven. Testing of the ovens for temperature uniformity over the duration of a normal drying test is necessary. Several locations on each shelf of the oven should be used during the test with continuous recording of the temperature. Ovens which exhibit wide temperature variations will indicate different moisture contents depending on sample location within the oven, and should not be used in critical applications. Additional testing of the ovens using homogeneous samples is also desirable. A drying test using twenty or more samples drawn from a homogeneous source will indicate to the operator the level of variation which can be expected with a given oven. Such testing should probably be done in triplicate with careful recording and placement of samples by location in the oven.

ACKNOWLEDGEMENTS

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TABLE 1

Moisture content variation with location
in the National #5840 Vacuum Oven

	TOP SHELF FRONT				BOTTOM SHELF FRONT			
	A	B	C	D	E	F	G	H
1.	3.37	3.31	3.33	3.40	3.52	3.58	3.53	3.57
2.	3.49	3.48	3.43	3.48	3.71	3.66	3.64	3.72
3.	3.54	3.54	3.49	3.56	3.84	3.71	3.78	3.81
4.	3.59	3.57	3.54	3.60	3.91	3.81	3.85	3.87
5.	3.63	3.63	3.61	3.62	3.89	3.82	3.84	3.91

TABLE 2

Summary of Spatial Temperatures during the testing of several drying ovens.

Test #	02	02	02	02	02
Rep #	01	02	03	04	05
Location	Dairy	Dairy	Dairy	Dairy	Dairy
Recorder	Esterline Angus	Esterline Angus	Esterline Angus	Esterline Angus	Esterline Angus
Oven	National #5840	National #5840	National #5840	National #5840	National #5840
Duration	5 hours	5 hours	5 hours	5 hours	5 hours
Oven set point	100°C	100°C	100°C	100°C	100°C
Oven start temp.	87°C	89.0°C	90°C	89°C	92°C
Oven finish temp.	98.25°C	98.25°C	98.5°C	100°C	100°C
Vacuum	30" Hg	30" Hg	29.75" Hg	29.75" Hg	29.75" Hg
Average* span	21.43°C	20.36°C	20.92°C	21.36°C	20.93°C
Maximum span	22°C	21.0°C	22.5°C	22.0°C	21°C
Minimum span	18.5°C	18.0°C	19°C	17°C	16°C

*Average span = Arithmetic mean of individual channel spans; temperature read from 2 hrs to 5 hrs.

TABLE 2 (Cont'd.)

Test #	03	04	04	04	05
Rep #	01	01	02	03	01
Location	Dairy	E.R.S.	E.R.S.	E.R.S.	Dairy
Recorder	Esterline Angus	Solartron Data Logger	Solartron Data Logger	Solartron Data Logger	Esterline Angus
Oven	Gallenkamp #OV160	National #5840	National #5840	National #5840	Cenco Oil Bath #95600A
Duration	1.9 hrs.	5 hrs.	5 hrs.	5 hrs.	5 hrs.
Oven set point	96°C	100°C	100°C	100°C	100°C
Oven start temp.	85°C	90°C	90°C	90°C	90°C
Oven finish temp.	97.0°C	100°C	100°C	100°C	99.5°C
Vacuum	Nil; forced circulation	29" Hg	29" Hg	29" Hg	27" Hg
Average span	1.375°C				2.0°C
Max. span	2.0°C				2.5°C
Min. span	1.0°C				1.5°C

TABLE 2 (Cont'd.)

Test #	06	06	06	07	08
Rep #	01	02	03	01	01
Location	Plant Products	Plant Products	Plant Products	Plant Products	Plant Products
Recorder	Esterline Angus	Esterline Angus	Esterline Angus	Esterline Angus	Esterline Angus
Oven	National #5850	National #5850	National #5850	National #58501	Prec. Scien. #1415
Duration	5 hrs.	5 hrs.	5 hrs.	5 hrs.	5 hrs.
Oven set point	100°C	100°C	100°C	100°C	100°C
Oven start temp.	92°C	89.5°C	90°C	91.0°C	85°C
Oven finish temp.	99.5°C	100°C	99.5°C	99.0°C	99.5°C
Vacuum	18.75" Hg	18" Hg	18.5" Hg	19" Hg	21" Hg
Average span	11.64°C	11.15°C	12.0°C	13.05°C	4.16°C
Max. span	15.0°C	13.0°C	13.0°C	14.5°C	7.0°C
Min. span	10.5°C	11.0°C	11.0°C	13.0°C	3.5°C

TABLE 2 (Cont'd.)

Test #	08	09	10
Rep #	02	01	01
Location	Plant Products	Plant Products	Plant Products
Recorder	Esterline Angus	Esterline Angus	Esterline Angus
Oven	Prec. Scien. #1415	National #N-8607-1	Thelco #18
Duration	5 hrs.	5 hrs.	5 hrs.
Oven set point	100°C	100°C	96.0°C
Oven start temp.	100.5°C	90°C	82.0°C
Oven finish temp.	100.5°C	100.5°C	96.0°C
Vacuum	20" Hg	19" Hg	Nil; forced circulation
Average span	3.33°C	12.86°C	1.5°C
Max. span	3.5°C	15.0°C	1.5°C
Min. span	2.0°C	12.5°C	1.0°C

TABLE 3

Temperatures and Moisture Content evaluation
by areas within National Oven #5840

	TOP SHELF				BOTTOM SHELF			
	A	B	C	D	E	F	G	H
1	3.37% 89°C	3.1	3.33 88°	3.40	3.52	3.58 96°	3.53	3.57 97°
2	3.49	3.48 92°	3.43	3.48 93°	3.71 103°	3.66	3.64 101°	3.72
3	3.54 97°	3.54	3.49 96°	3.56	3.84	3.71 104°	3.78	3.81 107°
4	3.59	3.57 97°	3.54	3.60 98°	3.91 108°	3.81	3.85 106°	3.87
5	3.63 100°	3.63	3.61 99°	3.62	3.89	3.82 106°	3.84	3.91 108°C

TABLE 4

National Vacuum Oven #5840

VARIABLE	MEAN	MINIMUM	MAXIMUM
2 P.M.	3.6889	3.4300	3.9400
1 Temp	99.9752	86.4330	111.1270

Analysis of Covariance Adjusted for Variable(s) Temp

	UNADJUSTED			ADJUSTED			
SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE	D.F.	SUM OF SQUARES	MEAN SQUARE	F
REPS (R)	2	.000433333	.000216667	2	.000689235	.000344617	.325 N.S
H (H)	1	.739674043	.739674043	1	.022175246	.022175246	20.8 **
W (W)	2	.021700000	.010850000	2	.017610829	.008805415	8.30 **
D (D)	2	.311233333	.155616667	2	.005880000	.002940000	2.77 N.S
H*W	2	.012448153	.006224076	2	.004786748	.002393374	2.25 N.S
H*D	2	.021137047	.010568524	2	.021615756	.010807878	10.2 **
W*D	4	.008333334	.002083333	4	.008725801	.002181450	2.06 N.S
ERROR	38	.039774075	.001046686	37	.039293885	.001061997	
TOTAL	53	1.154733318					

Regression Sum of Squares .00048018979

Constant Term of Regression Equation 3.3821022

Regression Coefficients with their Standard Errors

	COEFF.	S.E.
Temp	.0030686	.0045635

TABLE 5

Cenco Oil Bath Vacuum Oven

VARIABLE		MEAN	MINIMUM	MAXIMUM
1	PCM	4.8866	4.7300	5.0500
2	Temp	97.3807	95.7774	98.4571

Analysis of Covariance Adjusted for Variable(s) Temp

	UNADJUSTED			ADJUSTED			
SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE	D.F.	SUM OF SQUARES	MEAN SQUARE	F
REPLICATIONS	2	.0001264	.0000632	2	.0005370	.0002685	.079N.S.
H (H)	1	.1020937	.1020937	1	.1019353	.1019353	29.9**
D (D)	2	.0053804	.0026902	2	.0054301	.0027150	.796N.S.
W (W)	2	.0029158	.0014579	2	.0032846	.0016423	.483N.S.
H*D	2	.0202304	.0101152	2	.0206626	.0103313	3.04 N.S.
H*W	2	.0010797	.0005399	2	.0014047	.0007023	.021N.S.
W*D	4	.0145162	.0036290	4	.0139049	.0034762	1.02 N.S.
ERROR	37	.1229358	.0033226	36	.1225035	.0034029	
TOTAL	52	.2692784					

Regression Sum of Squares .00043233021

Constant Term of Regression Equation 2.626771

Regression Coefficients with their Standard Errors

	COEFF.	S.E.
Temp	.019757	.055428

TABLE 6

Napco Oven #5861

VARIABLE		MEAN	MINIMUM	MAXIMUM
1	PCM	4.0724	3.7900	4.4000
2	Temp	102.3215	98.1667	108.9633

Analysis of Covariance Adjusted for Variable(s) Temp

	UNADJUSTED			ADJUSTED				
SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE	D.F.	SUM OF SQUARES	MEAN SQUARE	F	
REPLICATIONS	2	1.216937100	.608468550	2	.044267881	.022133941	4.71	*
H (H)	1	.000816669	.000816669	1	.002756161	.002756161	8.584	N.S
D (D)	2	.015937028	.007968514	2	.002298165	.001149082	.244	N.S
W (W)	2	.017403699	.008701849	2	.019761501	.009880750	2.1	N.S
H*D	2	.008211115	.004105557	2	.016376648	.008188324	1.74	N.S
H*W	2	.008011111	.004005556	2	.015941488	.007970744	1.70	N.S
W*D	4	.018062963	.004515741	4	.023627358	.005906840	1.26	N.S.
ERROR	38	.182607451	.004805459	37	.174436171	.004714491		
TOTAL	53	1.467987136						

Regression Sum of Squares .0081712806

Constant Term of Regression Equation 8.0127455

Regression Coefficients with their Standard Errors

	COEFF.	S.E.
Temp	-.038509	.029251

MOISTURE CONTENT Vs. TEMPERATURE

NATIONAL OVEN 5840

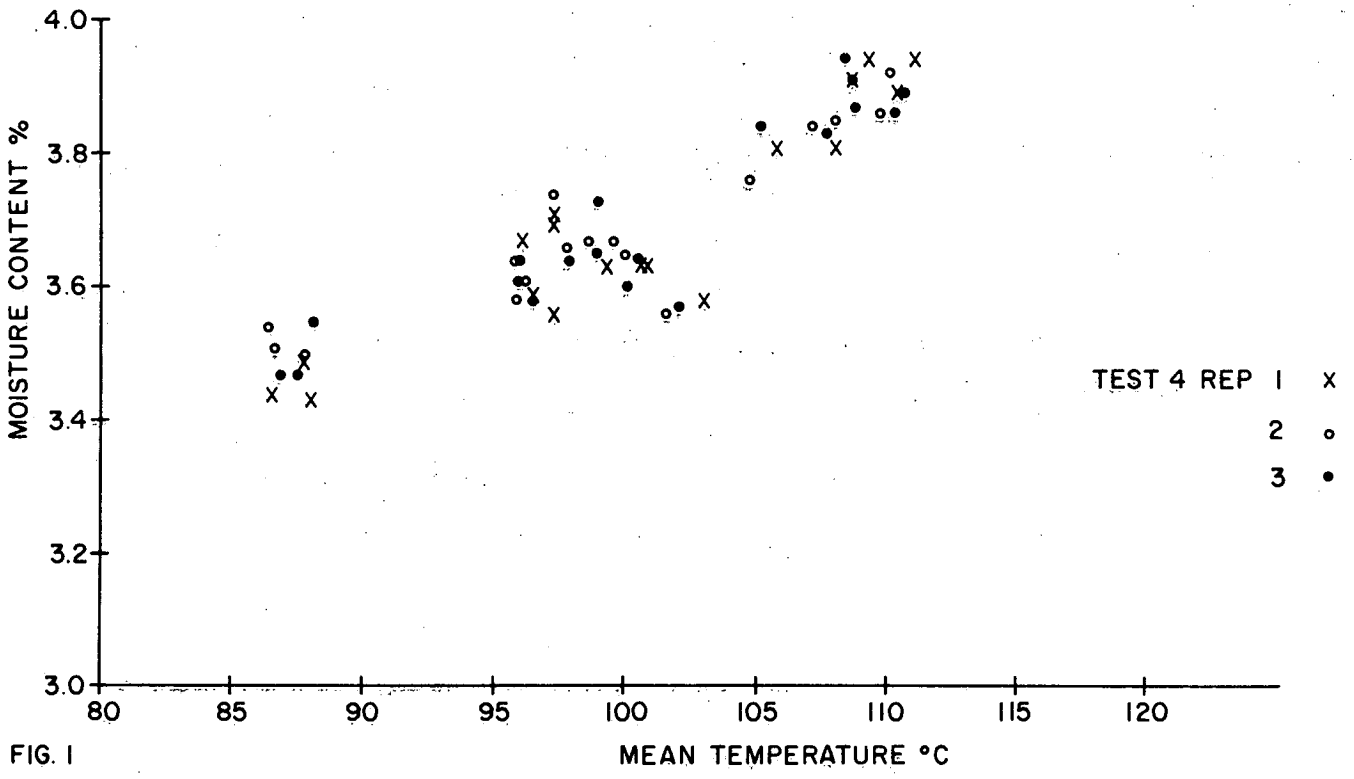

FIG. 1

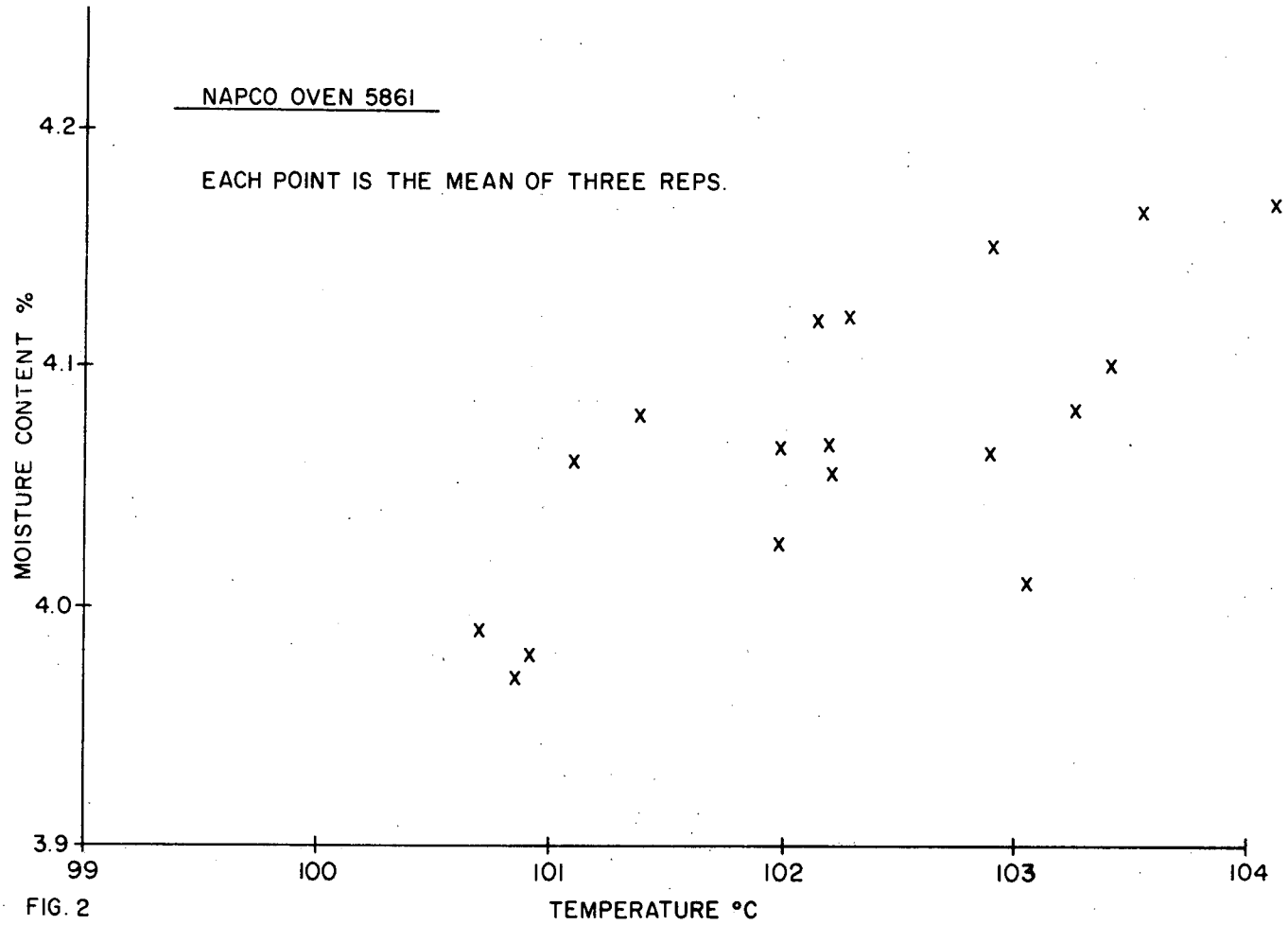
NAPCO OVEN 5861
EACH POINT IS THE MEAN OF THREE REPS.
MOISTURE CONTENT %
4.2
4.1
4.0
3.9
99
100
101
102
103
104
TEMPERATURE °C

FIG. 2