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INDUSTRIALIZATION OF COFFEE PECTIN

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INSTITUTO CENTROAMERICANO DE INVESTIGACION Y TECNOLOGIA INDUSTRIAL (ICAITI)

APARTADO POSTAL 1552
Cables: ICAITI
Telex: 5312-ICAITI-GU

Avenida La Reforma 4-47, Zona 10
Guatemala, C. A.

TELEFONOS: 310431
317466

THIRD PROGRESS REPORT

INDUSTRIALIZATION OF COFFEE PECTIN

Presentado por el ICAITI a

ROCAP
Office of the Science Advisor AID/Washington
Energy Officer
Bureau of Science and Technology LAC/DR, AID/W

Guatemala, March 1985

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SUMMARY

In this report a complete set of analytical data is given for three coffee purified pectin samples and one crude pectin sample. The data is discussed and compared to commercial citrus pectin samples.

The purified pectin samples compare well with the commercial samples. However its molecular weight was found to be 3.6 times less. This low value prevented any setting in the jelly formation tests. The low value found may be the result of the processing-purifying methods or if may be representative of the true coffee pectin. This extremely important point is the subject of work in progress.

INDUSTRIALIZATION OF COFFEE PECTIN

1. BACKGROUND

The project started in July, 1983. Its general objective is to obtain experimental information required to determine the feasibility of using pectins from the coffee mucilage in food applications. The present progress report covers the third semester of the project.

2. ACTIVITIES

According to the work plan, the following activities were performed:

2.1 Procurement of research supplies:

Spare parts ordered for a pilot plant evaporator were received. Ethanol and acetone for pectin recovery were purchased locally.

2.2 Research work:

2.2.1 Extraction of pectin from coffee mucilage:

As informed in the last report, a sample of coffee brought from the coffee processing plant owned by the Federation of Coffee Producing Cooperatives of Guatemala (FEDECOCAGUA), in Palin, Escuintla, was depulped in a manual pulper machine and then processed in a Fukunaga pilot plant demucilaginator. Water was added to the Fukunaga (about 6:1 in vol/weight of mucilage) until the processed coffee beans plus the

mucilage solution flowed out of the machine. After separating the solids in a rotating screen, the diluted mucilage solution was concentrated in a vaccum pilot plant evaporator at about 60°C. This sample was identified as Palin A.

Another sample was obtained from a coffee plantation in Parramos, Chimaltenango, Guatemala; which was initially assumed to be from a single coffee variety. Later on it was found that it contained a mixture of at least 2 varieties, according to the different sizes of the coffee beans. About 90 kg of ripe coffee fruit from this sample were depulped and processed in the Fukunaga demucilaginator, but this time only an initial amount of water was added and then the liquid outlet of the machine was recirculated to prevent excessive dilution of the mucilage solution. The resulting concentrated solution was treated with ethanol to precipitate the marc (alcohol-insoluble material). This sample was identified as Parram.

2.2.2 Crude mucilage:

A portion of coffee mucilage from the Parram sample was freeze dried at 0°C in a Stokes laboratory freeze dryer. This sample was identified as Crude Mucil, Palin.

2.2.3 Extraction of pectin from coffee pulp:

As indicated in the previous progress report of the project, coffee pulp obtained from coffee fruit from Palin was extracted by boiling in alkaline sulfite solution by the method described by Calle, 1962. This treatment demethoxilated the pectin. The sample was labeled as Palin B.

2.2.4 Chemical determinations:

The marc (alcohol-insoluble solids) from the mucilage and pulp obtained from the samples of Palin was purified by Dr. Rombouts' method (Roumbouts, 1983) and, together with several commercial pectin samples, assayed for anhydrogalacturonic acid (% AAG) and ester methoxyl content; and the degree of methoxylation (DE) calculated. Other analysis performed were acetyl content, nitrogen, carbohydrates, moisture, total ash and acid insoluble ash.

Sulfur dioxide (SO_2), arsenic and lead contents were not determined because the available coffee pectin samples were too small.

Results from chemical analysis are reported, in dry basis, in Tables 1 and 2. Analytical methods and their references are listed in Table 3.

2.2.4.1 Anhydrogalacturonic acid:

As reported previously, two analytical methods were tested: carbazole and m-hydroxydiphenil. The m-hydroxydiphenyl method gave the best results for % AAG (Figure 1, Tables 1 and 2), because the data obtained is in accordance with the characteristics of the analyzed pectins; for example, unstandarized (purer) samples like those from coffee mucilage and coffee pulp, and Citrus pectin type 603 and Purple Ribbon Pure, gave the higher AAG contents.

Figure 1 compares the relative amounts of AAG in coffee pectins with a unstandarized citrus pectin. It can be seen that there is little difference between the samples, exceptuating the crude mucilage; which gave a lower amount of pectin substances than the values of 33 to 38% reported in the literature for coffee (Orozco, 1974).

2.2.4.2 Methoxyl and degree of esterification:

Two analytical methods: NaOH titration and gas chromato-

graphy were compared for these determinations. Results are showed in Tables 1 and 2 and Figures 3 and 4. It was concluded that the gas chromatographic method was better because it gave results in accordance with the literature for the commercial pectins, it did not give degrees of esterification higher than 76% as expected for natural pectins (Pedersen 1980) and was sensible enough to detect the low methoxyl content of the demethoxylated sample (Palin B).

Purple Ribbon D-075 L.M. and Purple Ribbon Pure (unstandarized) L.M. are low-methoxyl pectins and their % Methoxyl is what would be normally expected.

2.2.4.3 Acetyl:

Acetyl content was determined in coffee mucilage and coffee pulp pectin and in 9 commercial samples by a gas chromatography method (Rombouts, 1983), that was modified by esterification of the sample before injection. Results can be seen in Tables 1 and 2.

The acetyl content should be low for adequate gelling properties (Pippen et al, 1950). Pectin from the coffee mucilage gave an acetyl content within the same order of magnitude of that of commercial citrus pectin.

2.2.4.4 Nitrogen:

Nitrogen content was determined for samples of coffee mucilage and coffee pulp pectin, and for 9 commercial pectins. Results are shown in Tables 1 and 2.

All samples except one (Purple Ribbon D-075, L.M.) were within the FAO standard of 0.5% maximum nitrogen content (FAO, 1981).

2.2.4.5 Carbohydrates:

Total carbohydrates were analyzed for coffee mucilage, coffee pulp and 9 commercial pectins. Results are presented in Tables 1 and 2. Carbohydrate data presented in Table 5 of the previous progress report is incorrect.

Carbohydrate contents of coffee pectin samples are within the same order of magnitude as unstandarized (pure) commercial pectins.

2.2.4.6 Molecular weight:

The molecular weight (number average) of coffee mucilage pectin and that of a commercial citrus pectin was determined by the method described by Owens et al, 1952, and slightly modified by Rombouts 1983. Results are shown in Tables 1 and 3 and in Figure 5.

The coffee pectin samples used for this analysis was from mucilage extracted in a Fukunaga demucilaginator, from coffee brought from Palin.

The lower molecular weight of the coffee pectin samples was probably caused by enzymatic degradation during the extraction process of the mucilage in the Fukunaga machine, which took over 4 hours before adding alcohol to precipitate the pectin substances. Values reported in the literature for commercial citrus and apple pectins are higher then 50 000, while a citrus pectin sample degraded by heating at 119°C for 20 minutes gave a molecular weight of 20 000-30 000 (Owens et al, 1944).

This determination will be repeated for coffee pectin samples from mucilage obtained in a more adequate way to prevent degradation.

On the other hand, the values might reflect that coffee

pectin indeed is a low molecular weight polymer. This extremely important point will be further discussed in future reports.

2.2.4.6 Moisture:

Moisture content of all samples was determined to calculate results in dry basis. Moisture data is presented in Tables 1 and 2 and in Figure 2.

2.2.4.7 Total ash:

Total ash content was determined for all samples and is shown in Tables 1 and 2 and in Figure 6. Total ash content for coffee mucilage samples is higher than that of commercial citrus and apple samples, but it meets the Food Chemical Codex standard, 1981 (less than 10%); although not the Bulgarian standard of less than 5% (Bulgaria, 1980).

2.2.4.8 Acid insoluble ash:

This determination was made only for the crude coffee mucilage sample and its value can be seen in Table 1. It meets the FAO and Food Chemicals Codex standards that require a value of less than 1% (FAO, 1981; Food Chemicals Codex, 1981).

2.2.4.9 Equivalent weight:

Results for this determination are presented in Tables 1 and 2, and in Figure 7. Equivalent weight decreases with demethylation of pectins (Olsen et al, 1939). A significant coefficient of determination r, at the 5% level, was found for an exponential correlation of equivalent weight vs esterification degree obtained from experimental data for coffee and commercial pectins. A semi-log plot for these data can be seen in Figure 8. Equivalent weight results for coffee mucilage samples are within the same order of magnitude of the data for tangerine pell pectin reported by Moon et al, 1982.

2.2.4.10 Setting time:

Setting times were determined for most commercial pectin samples. Results were in accordance with the label designations (fast, mediu, slow). It was not possible to obtain setting times for coffee pectin because these samples didn't form a jelly, presumably because of its low molecular weight. Results are shown in Tables 1 and 2. Olsen et al, 1939, demonstrated that setting time is a function of equivalent weight. A significant coefficient of determination r, at the 5% level, was found for a power correlation of equivalent weight vs setting time obtained from experimental data for commercial pectins. A log-log plot for these data can be seen in Figure 9.

2.2.4.11 Jelly grade (°SAG):

Only the commercial pectin samples were graded, because the coffee mucilage samples didn't form a jelly.

Results can be seen in Tables 1 and 2. All standarized commercial pectin samples were labeled 150°SAG, but apple pectins averaged only 124.2°SAG; probably because of jelly grade losses during storage (Pedersen, 1980).

3. TRAVEL

After some mail and telephone constultations, the planned trip to Colombia was canceled because apparently there has not been new developments on coffee pectin utilization in this country. However, Mr. Juan Francisco Menchú from ICAITI traveled to Costa Rica in September 1984, where he visited a company (Subproductos de Café, S.A.) which has been created with support from a government financing institution (CODESA). He met the president of the company and other Costa Rican researchers. Subproductos de Café is planning to produce coffee pectin,

tannins and caffeine on a commercial scale, based on studies being carried on by two U.S. companies: Gibbs & Hill from San José, California and Parangon Technical Co. The studies have been financed by loans from the Interamerican Development Bank. USAID is considering financing a demonstration pilot plant. Pectin recovery will be performed by precipitation with $AlCl_3$, followed by acid-alcohol washing; neutralization and drying. The ELMU continuous demucilaginator will be used for mucilage recovery without any special modifications.

Mr. Menchú also visited Talleres Quezada Hnos., that manufacture ELMU demucilaginators, where he met the owner, Mr. Jorge Quezada. They are still producing the same type of machine, although the power was reduced from 20 to 12 HP without loosing performance.

4. WORK PLAN FOR FIRST SEMESTER 1985

4.1 Chemical characterization of pectin from coffee mucilage:

The remaining chemical analysis: SO_2, arsenic, and copper will be performed on the pectin from the coffee mucilage.

4.2 Physical tests:

Gel strenght, molecular weight, gelling time and setting temperature will be determined for pectin from coffee mucilage. A color comparison with commercial citrus pectins will be made with a Hunter Lab. color/difference meter.

About 20 g of dried coffee mucilage pectin will be sent to the Bulmer Co. in England, where it iwll be tested.

4.3 Coffee mucilage samples:

About 180 kg of coffee fruit each from 3 different coffee varieties: Bourbon, Caturra and Catimor will be collected from a private farm and processed in a batch demucilaginator (Sexilago) lent by a Guatemalan manufacturer. Ethanol will be immediately added to the recovered mucilage to prevent enzymatic deterioration.

4.4 Pectin extraction methods:

According to the original work plan, an evaluation of at least 3 pectin extraction methods is programmed for the second coffee season of the project which started in October 1984.

4.5 Evaluation of an industrial demucilaginator:

The planned evaluation of a continuous demucilaginator (ELMU) in an industrial installation was not feasible, because of the actual practice of recirculating process water, which is excellent for accelerating the rate of mucilage removal from the coffee beans due to its high content of enzyme producing microflora, but makes almost impossible to evaluate the machine for pectin recovery without major modifications in the process.

During the coffee season which started in October 1984, a batch demucilaginator will be evaluated for its mucilage yield and quality at ICAITI's pilot plant.

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TABLE 1

PHYSICO-CHEMICAL CHARACTERIZATION OF COFFE PECTINS

ANALYSIS (All determinations in dry weight base).	CONFID. RANGE (0.05 LEVEL)	COFFEE MUCIL. PALIN A	COFFEE MUCIL. PARRAM.	COFFEE MUCIL. PALIN B	CRUDE MUCIL. PALIN	CITRUS PECTIN 60%
% ANHYDRO GALACTURONIC ACID						
CARBAZOL method	2.4509	67.18	73.74	77.60	10.28	85.22
m-PHENYL PHENOL method	1.0140	93.00	86.80	95.24	8.83	93.20
NaOH titration	0.5068	48.33	25.59		12.17	53.93
% METHOXYL CONTENT						
NaOH titration	0.1474	8.12	8.30			8.61
Gas Chromatography	0.6503	9.98	10.78	0.41		11.65
DEGREE OF ESTERIFICATION						
NaOH titration	0.9372	95.90	21.28			49.43
Gas Chromatography	4.2734	65.76	76.10	2.64		76.59
% ACETYL CONTENT						
Gas Chromatography	0.0363	0.38	0.09		0.34	0.30
% NITROGEN, micro Kjeldahl	0.0253	0.10	0.14	0.08		0.24
% CARBOHYDRATE CONTENT						
PHENOL-SULFURIC method	1.4330	49.53	47.75	51.73		42.60
% MOISTURE (oven method)	0.3036	13.74	16.85	14.57	26.87	8.35
% TOTAL ASH CONTENT	0.4641	6.20	5.63	1.97	5.50	2.96
EQUIVALENT WEIGHT						
NaOH titration	202.7070	4073.1	5611.4			2031.6
AVERAGE MOLECULAR WEIGHT						
viscosity method		20964.6	26880.8			86774.1
% ACID-INSOLUBLE ASH						
HCL boiling method					0.27	
SETTING TIME, sec.	9.7430					43.45
°SAG	3.4467					179.65

TABLE 2

PHYSICO-CHEMICAL CHARACTERIZATION OF COMMERCIAL PECTINS

ANALYSIS (All determinations in dry weight base).	CONFID. RANGE (0.05 LEVEL)	GENU PECTIN TYPE A CITRUS	GENU PECTIN TYPE B CITRUS	GENU PECTIN TYPE D CITRUS	PURPLE RIBBON PURE APPLE	BROWN RIBBON APPLE	PURPLE RIBBON D-075 APPLE	GREEN RIBBON APPLE	BLUE RIBBON APPLE
% ANHYDRO GALACTURONIC ACID									
CARBAZOL method	2.4509	63.05	58.70	67.26	88.97	66.04	61.36	72.66	77.57
m-PHENYL PHENOL method	1.0140	72.94	66.11	81.79	98.92	81.03	67.38	83.51	88.08
NaOH titration	0.5068	36.37	33.36	42.45	47.77	38.40	12.73	42.12	42.33
% METHOXYL CONTENT									
NaOH titration	0.1474	5.26	5.20	5.81	3.33	5.38	1.78	5.01	4.12
Gas Chromatography	0.6503	6.63	7.00	7.54	5.85	6.15	7.69	10.13	9.20
DEGREE OF ESTERIFICATION									
NaOH titration	0.9372	35.37	32.28	39.87	39.39	79.52	80.19	67.58	55.17
Gas Chromatography	4.2754	55.70	64.88	56.49	36.24	46.51	69.93	74.33	64.00
% ACETYL CONTENT									
Gas Chromatography	0.0363	0.30	0.32	0.36	0.35	0.28	0.35	0.38	0.38
% NITROGEN, micro Kjeldahl	0.0253	0.23	0.38	0.35	0.09	0.20	2.08	0.33	0.13
% CARBOHYDRATE CONTENT									
PHENOL-SULFURIC method	1.4330	55.65	55.36	54.99	51.69	62.60	59.06	62.05	59.44
% MOISTURE (oven method)	0.3036	2.74	3.24	6.08	9.87	5.55	8.39	10.02	10.19
% TOTAL ASH CONTENT	0.4641	1.49	1.08	2.21	5.12	1.01	4.01	4.16	1.63
EQUIVALENT WEIGHT									
NaOH titration	202.7070	1053.4	1772.1	839.1	299.53	914.44	990.97	572.11	410.09
AVERAGE MOLECULAR WEIGHT									
viscosity method						66521.8		44322.2	
SETTING TIME, sec.	9.7430	145.05	30.55	213.8		155.95		472.4	472.63
°SAG	3.4467	142.63	139.31	145.00		121.10		125.45	126.12

TABLE 3

ANALYTICAL METHODS

DETERMINATION	METHOD	REFERENCE
Anhydro galacturonic acid	Carbazole	Mc Comb & Mc Cready (1952)
Anhydro galacturonic acid	m-hydroxy-diphenyl	Kinter & van Buren (1982)
Anhydro galacturonic acid	NaOH titration	Schultz (1965)
Methoxyl	NaOH titration	Schultz (1965)
Methoxyl	gas chromatogr.	Walter et al.(1983)
Esterification degree	NaOH titration	Schultz (1965)
Esterification degree	gas chromatogr.	Walter et al.(1983)
Acetyl	gas chromatogr.	Rizzo (1980)
Nitrogen	micro Kjeldahl	AOAC (1980)
Carbohydrates	Phenol sulfuric	Hodge & Hofreiter (1962)
Moisture	Oven	FAO (1981)
Total ash	Calcination	AOAC (1980)
Acid insoluble ash	HCl boiling	FAO (1978)
Equivalent weight	NaOH titration	Schultz (1965)
Average molecular weight	viscosity method	Rombouts (1983); Owens et al.(1952).
Setting time	twist method	Joseph & Baier (1949)
°SAG	Ridgelimeter	IFT (1949)

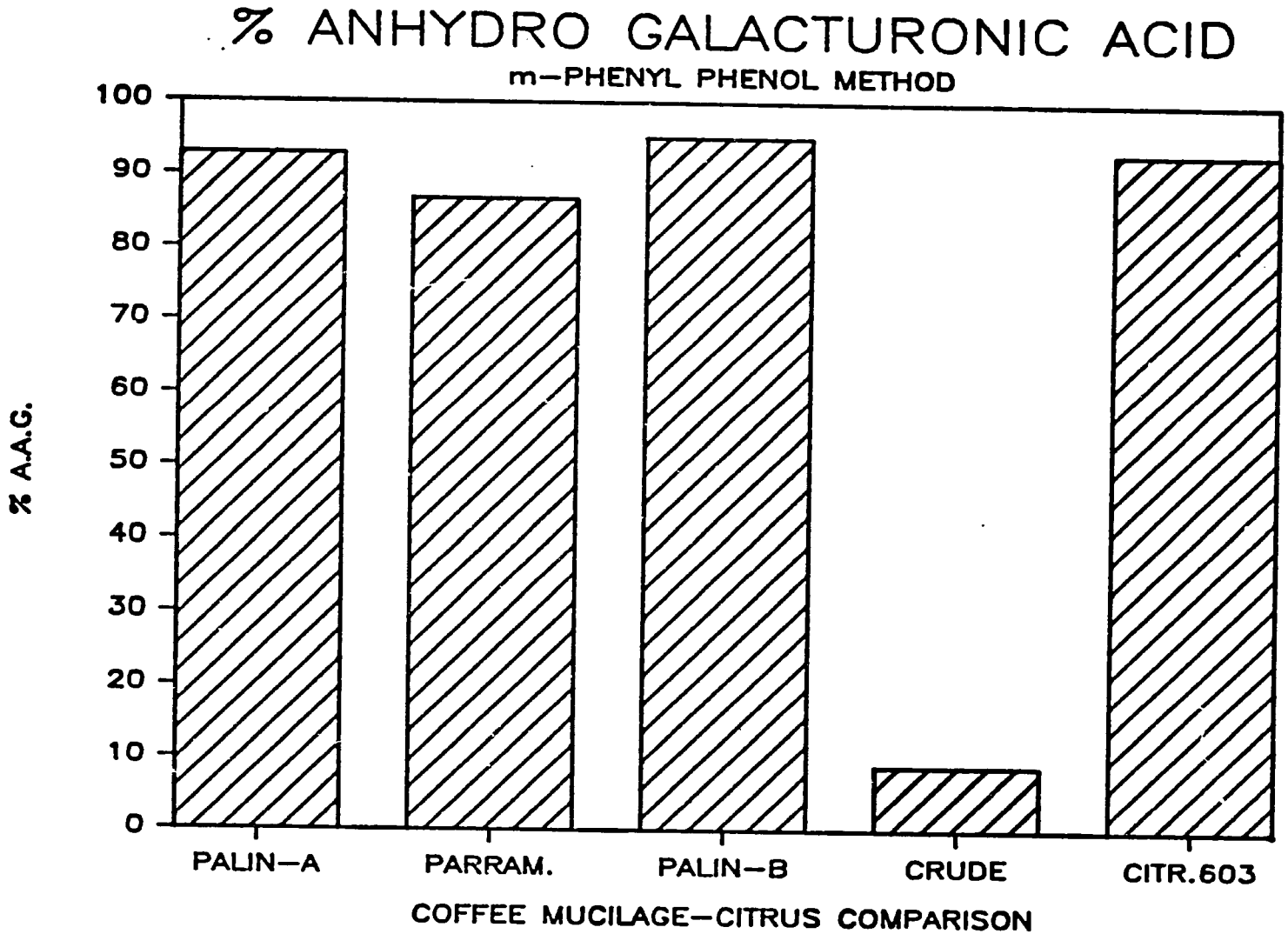
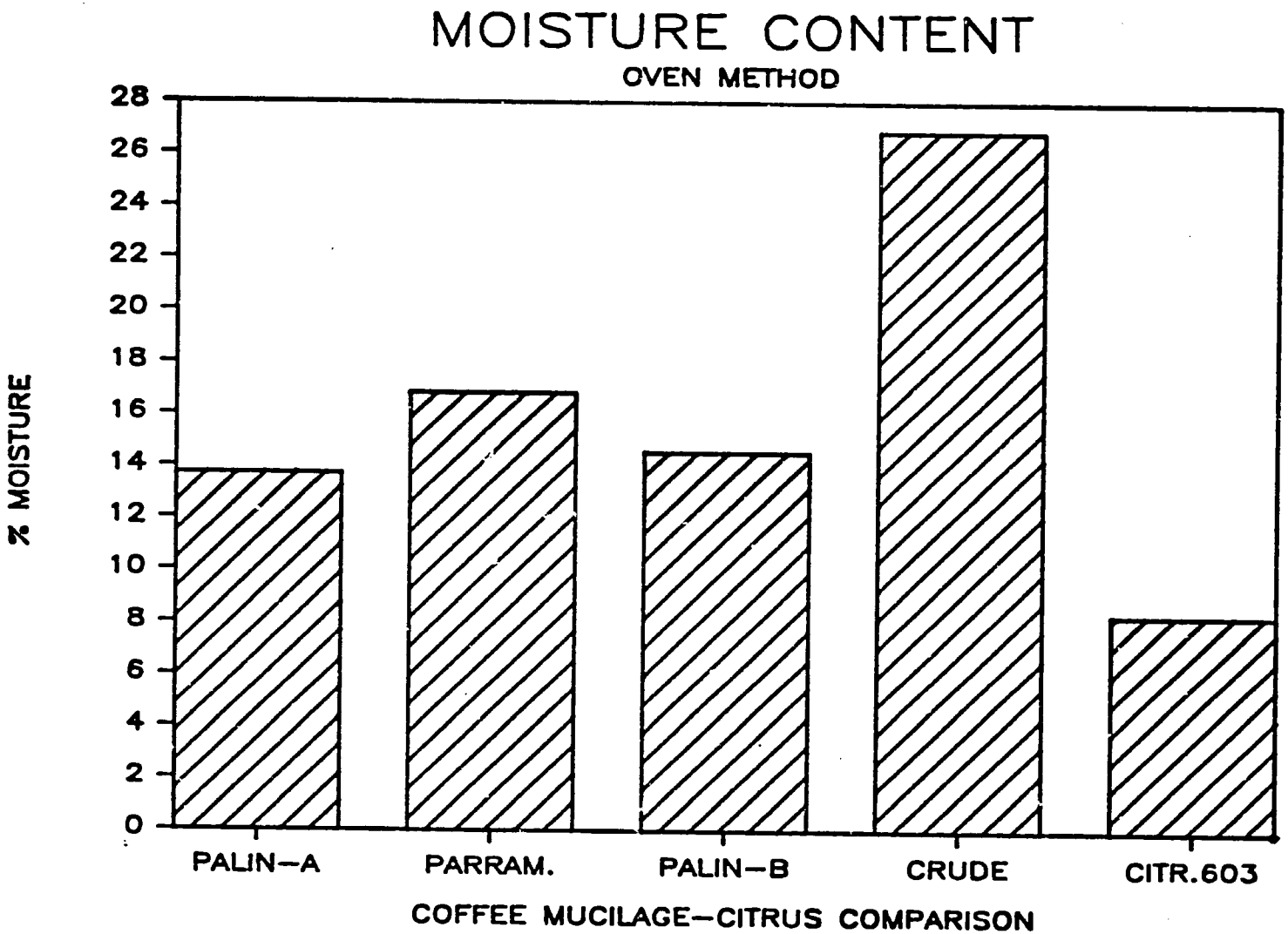

% ANHYDRO GALACTURONIC ACID
m-PHENYL PHENOL METHOD
% A.A.G.
100
90
80
70
60
50
40
30
20
10
0
PALIN-A
PARRAM.
PALIN-B
CRUDE
CITR.603
COFFEE MUCILAGE-CITRUS COMPARISON

FIGURE 1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OVEN METHOD
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6
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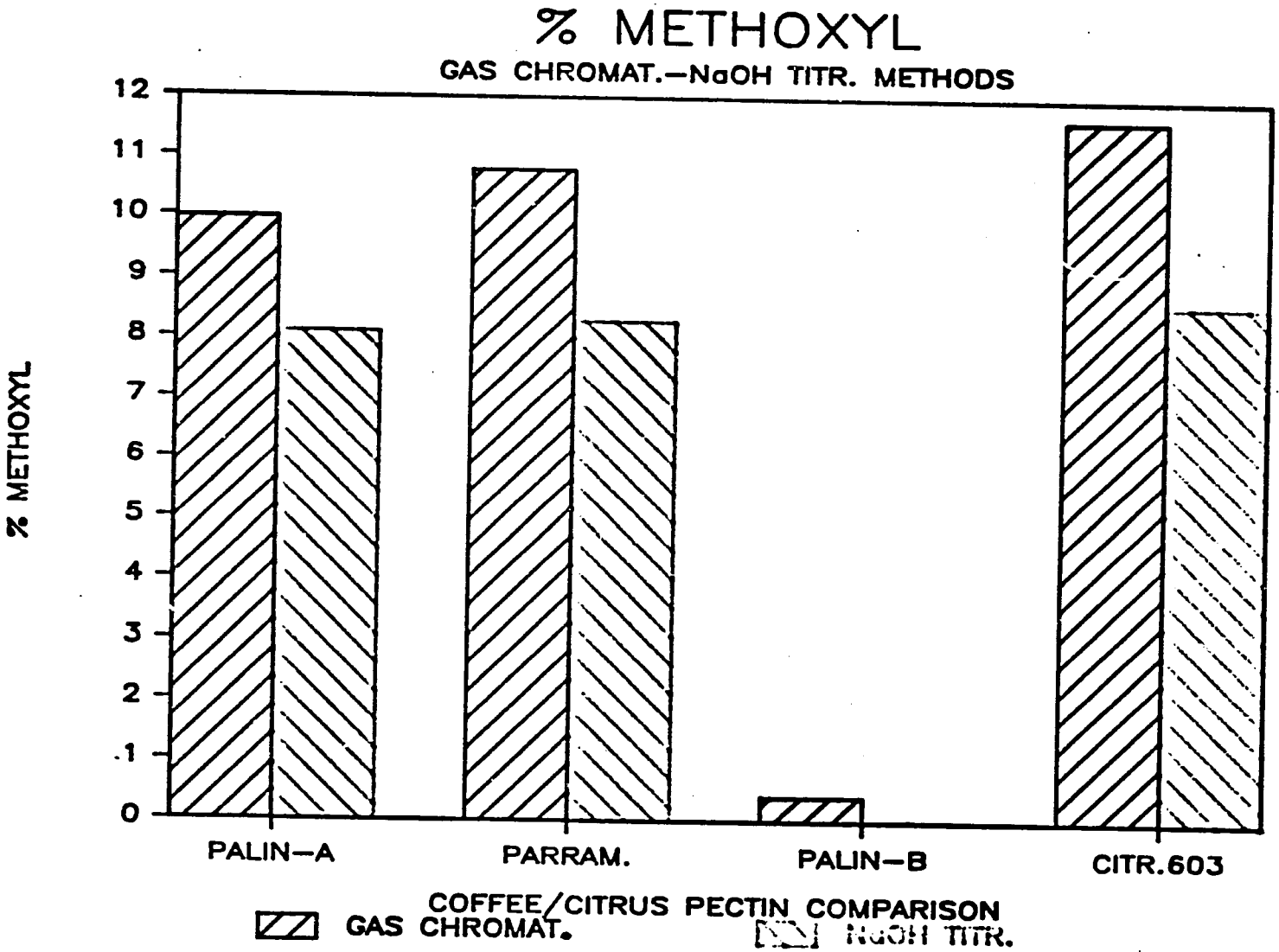
PARRAM.
PALIN-B
CRUDE
CITR.603
COFFEE MUCILAGE-CITRUS COMPARISON

FIGURE 2


% METHOXYL
GAS CHROMAT.—NaOH TITR. METHODS
% METHOXYL
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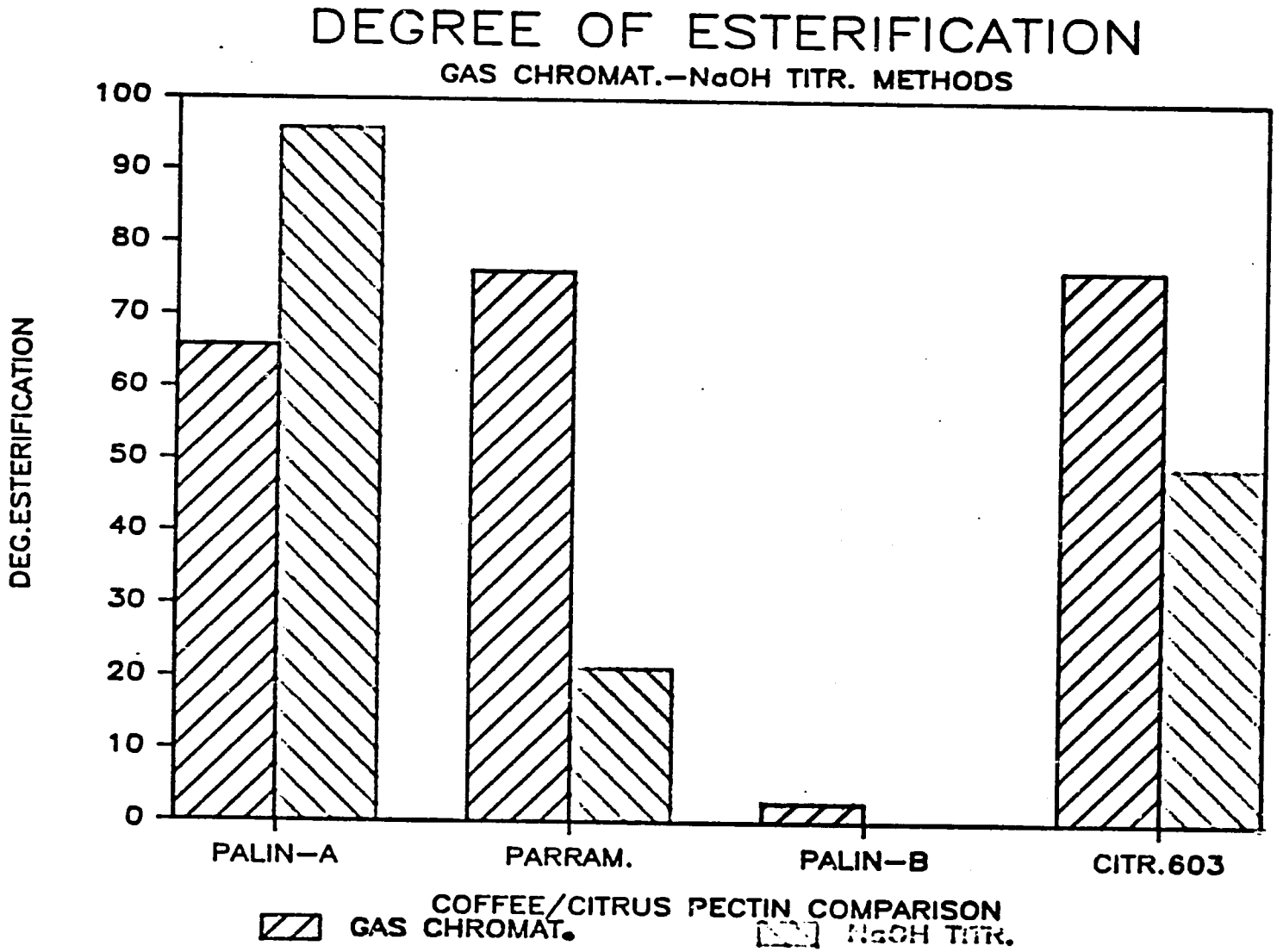
10
9
8
7
6
5
4
3
2
.1
0
PALIN—A
PARRAM.
PALIN—B
CITR.603
COFFEE/CITRUS PECTIN COMPARISON
GAS CHROMAT.
NaOH TITR.

FIGURE 3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50
40
30
20
10
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PALIN—A
PARRAM.
PALIN—B
CITR.603
COFFEE/CITRUS PECTIN COMPARISON
GAS CHROMAT.
NaOH TITR.

FIGURE 4

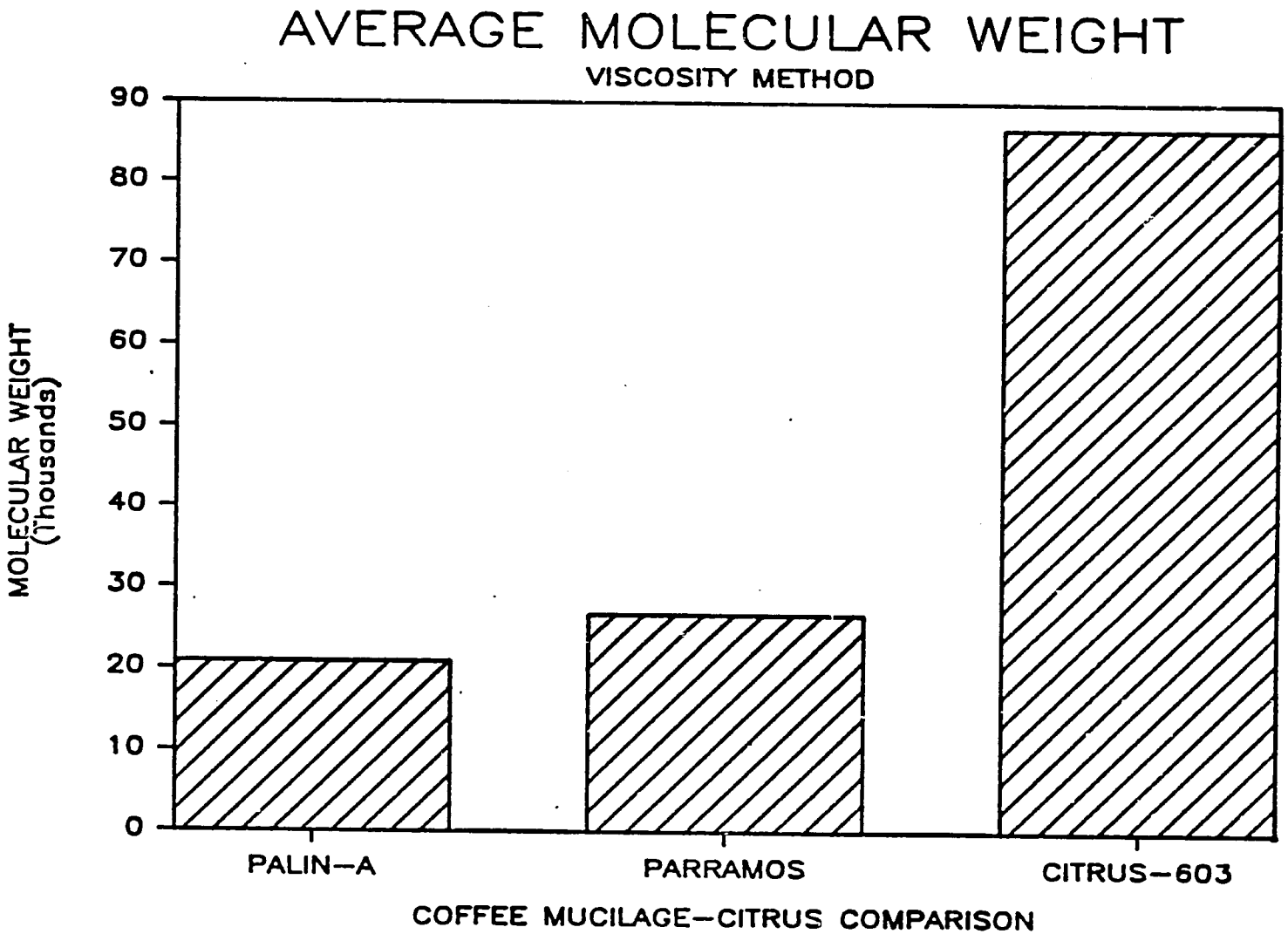

AVERAGE MOLECULAR WEIGHT
VISCOSITY METHOD
MOLECULAR WEIGHT
(Thousands)
90
80
70
60
50
40
30
20
10
0
PALIN—A
PARRAMOS
CITRUS—603
COFFEE MUCILAGE—CITRUS COMPARISON

FIGURE 5

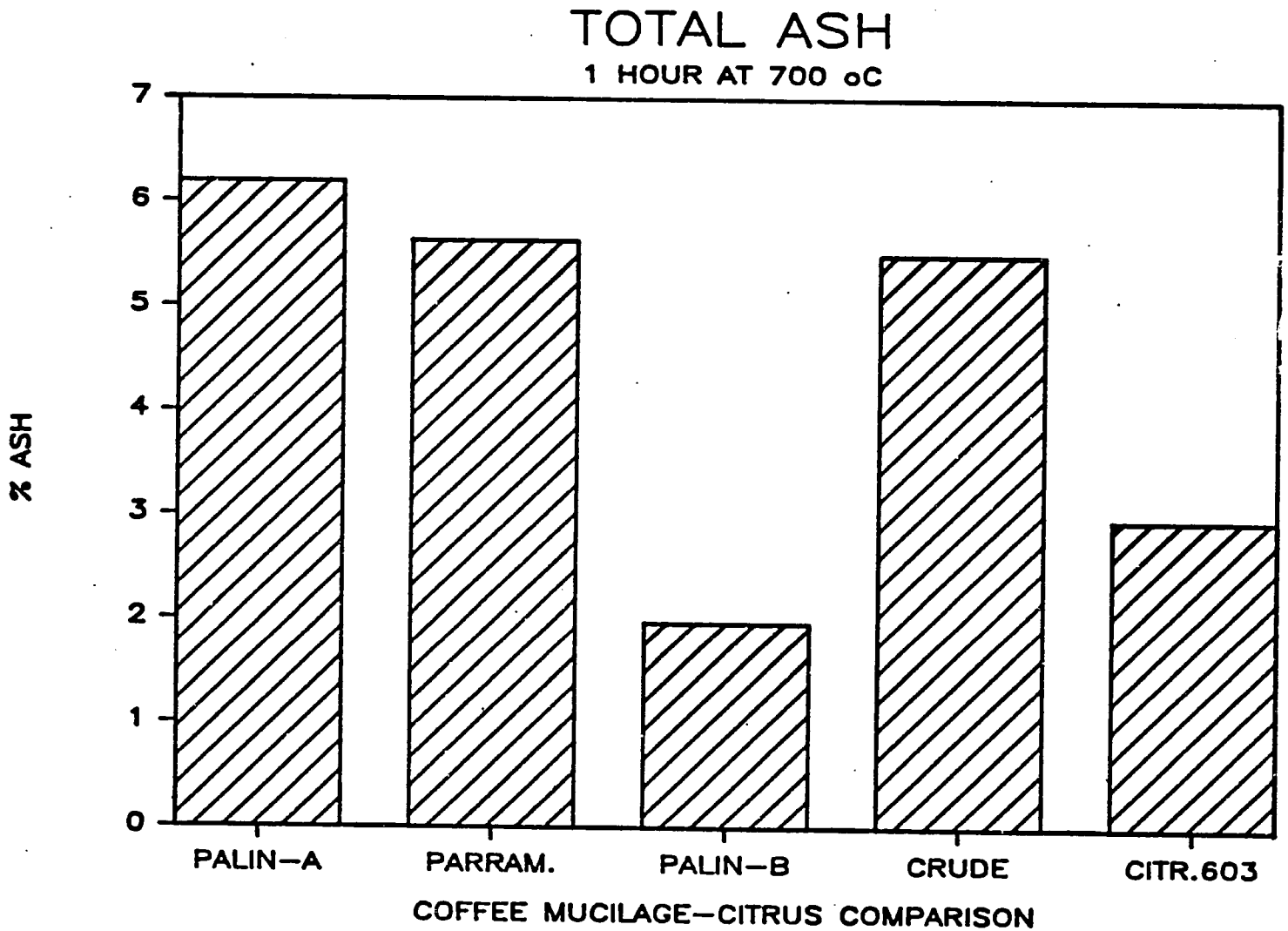

TOTAL ASH
1 HOUR AT 700 oC
% ASH
0
1
2
3
4
5
6
7
PALIN—A
PARRAM.
PALIN—B
CRUDE
CITR.603
COFFEE MUCILAGE—CITRUS COMPARISON

FIGURE 6

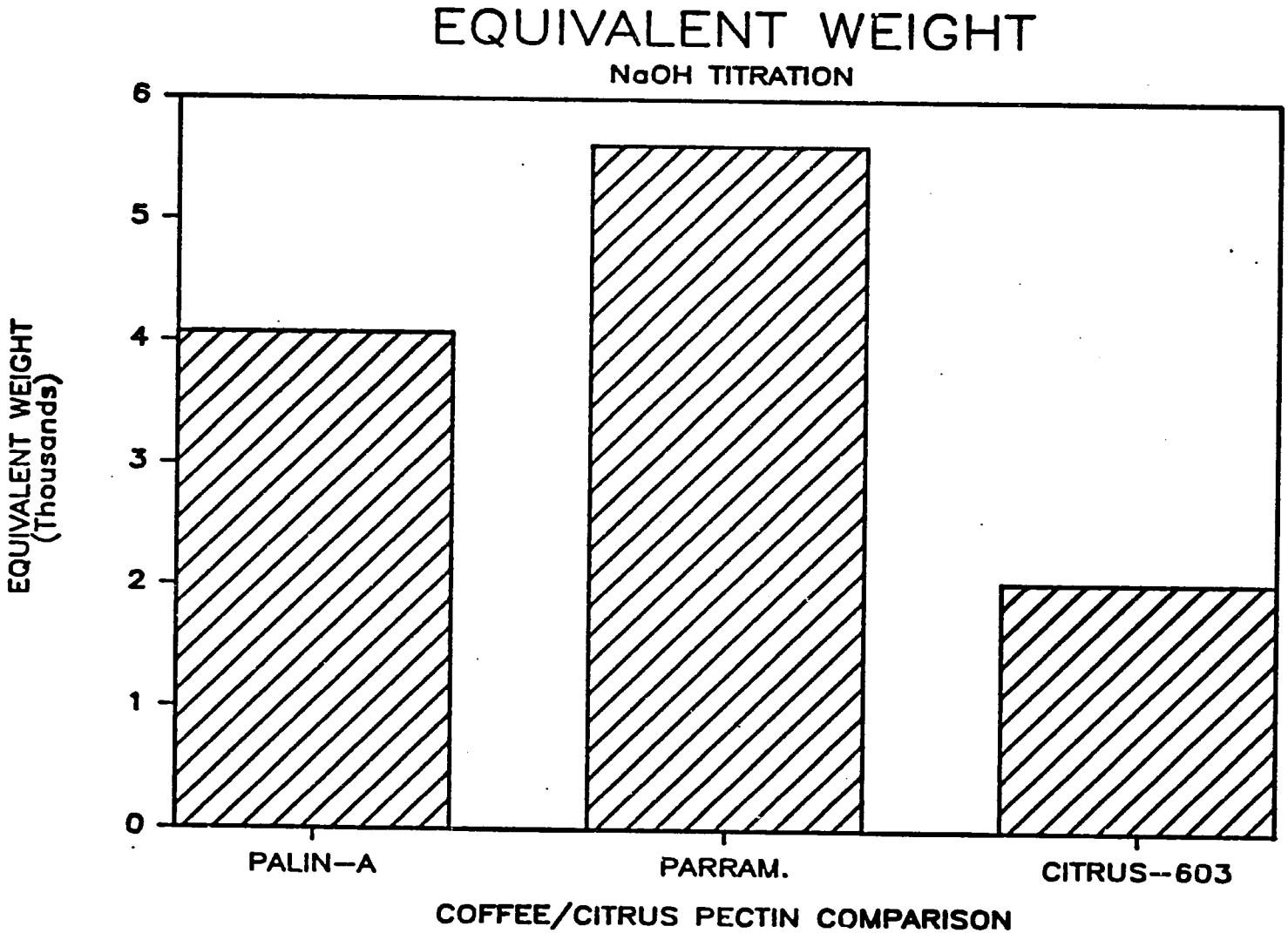
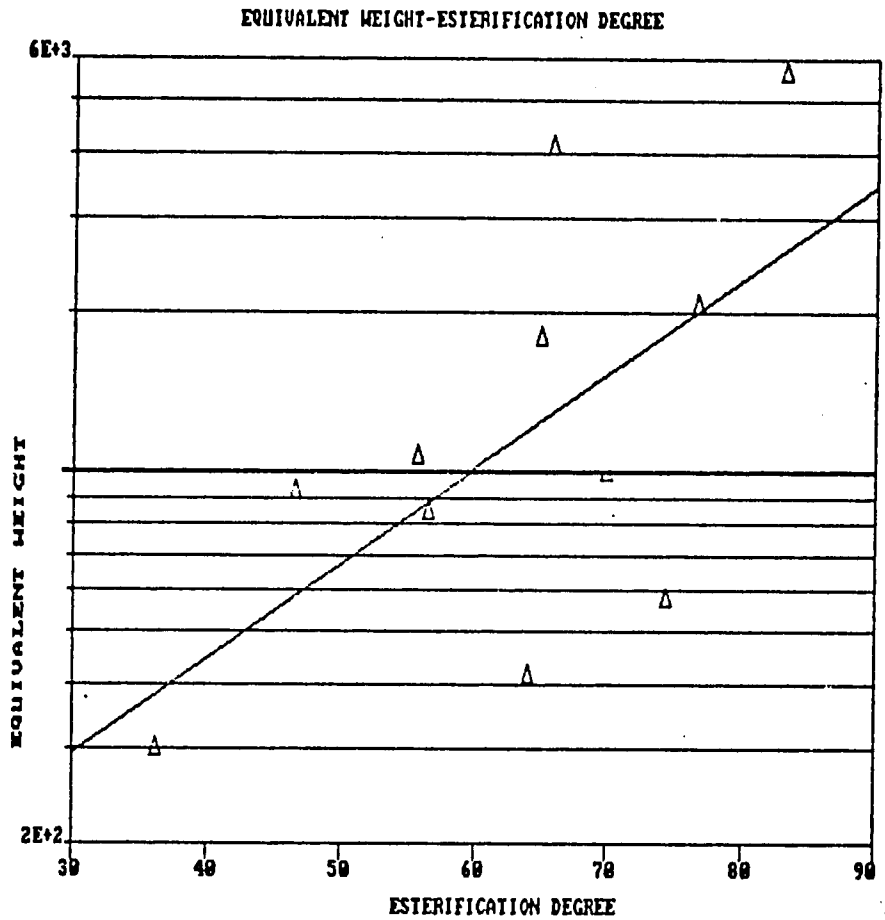

EQUIVALENT WEIGHT
NaOH TITRATION
EQUIVALENT WEIGHT
(Thousands)
0
1
2
3
4
5
6
PALIN—A
PARRAM.
CITRUS--603
COFFEE/CITRUS PECTIN COMPARISON

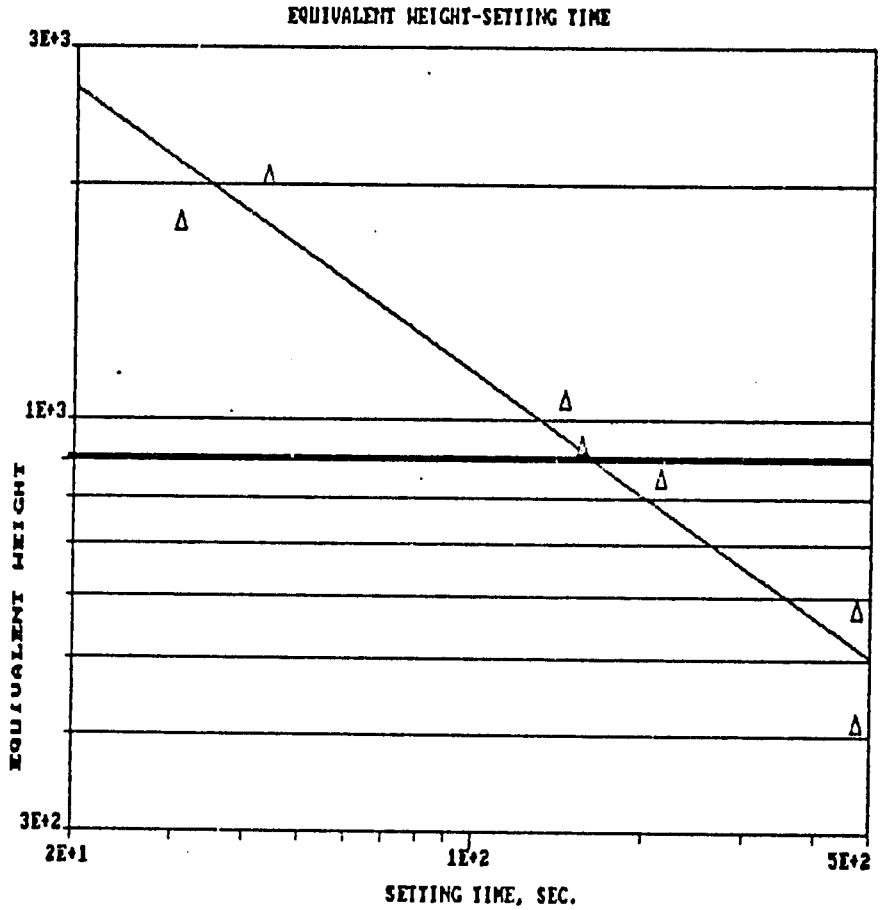
FIGURE 7



THE REGRESSION POLYNOMIAL OF LINE 1 -

(1.936E+00) + (1.783E-02)*X
THE VARIANCE - 8.618E-02

FIGURE 8



THE REGRESSION POLYNOMIAL OF LINE 1 -

(4.096E+00) + (-5.172E-01)*X
THE VARIANCE - 3.546E-03

FIGURE 9

INSTITUTO CENTROAMERICANO DE
INVESTIGACION Y TECNOLOGIA INDUSTRIAL
(ICAITI)

CENTRAL AMERICAN RESEARCH INSTITUTE FOR INDUSTRY
Avenida La Reforma 4-47, Zona 10
GUATEMALA, C. A.

COSTA RICA
EL SALVADOR
GUATEMALA
HONDURAS
NICARAGUA

APARTADO POSTAL 1552
Cables: ICAITI
Telex: 5312-ICAITI-GU
TELEFONOS: 310631 y
317466

THIRD PROGRESS REPORT

INDUSTRIALIZATION OF COFFEE PECTIN

Presented by ICAITI to:

ROCAP
Office of the Science Advisor AID/Washington
Energy Officer
Bureau of Science and Technology LAC/DR, AID/W

ICAITI, Personnel

María del Carmen de Arriola
Delia Arriola

Ricardo García
Project Leader

Carlos Rolz
Head, Applied Research Division

Francisco Aguirre B.
Director