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RESEARCH AND DEVELOPMENT ACTIVITIES ON DIRECT LIQUEFACTION TECHNOLOGY
QUARTERLY REPORT
January-March 1981

H. Davis, S. Ergun, C. Figueroa, C. Karatas,
D. Kloden, L. Schaleger, and N. Yaghoubzadeh

April 1981

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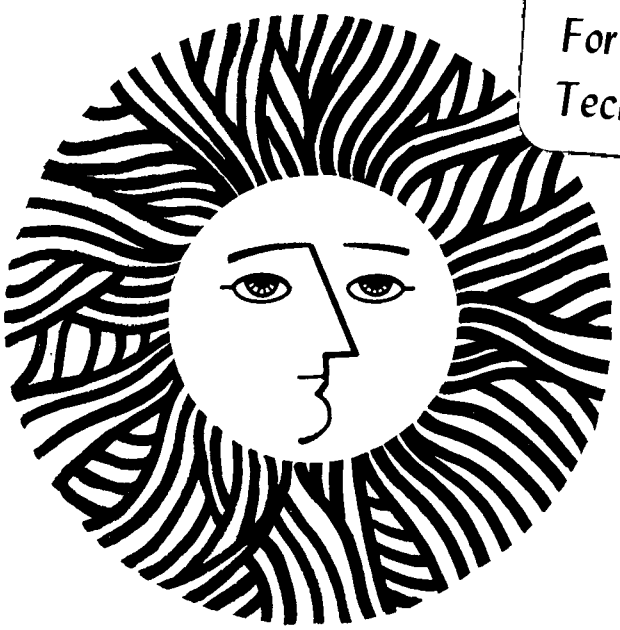

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Berkeley, California 94720

H. Davis, S. Ergun, C. Figueroa, C. Karatas,
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RESEARCH AND DEVELOPMENT ACTIVITIES ON DIRECT LIQUEFACTION TECHNOLOGY

HIGHLIGHTS

A partially successful initial operation of the continuous liquefaction unit (CLU) was terminated by a reactor plug. This is believed to have been caused by a series of gas flow stoppages.

Mechanical operation of the CLU system was otherwise excellent. Oil product was comparable in physical properties and molecular weight to the better PDU products.

Operable procedures for analytical characterization of oil and water soluble product were developed and applied.

Chromatographic and size exclusion "fingerprints" of oil product were developed and used to compare the CLU-1 product with previous oils.

Product water containing organic acids and other organics was successfully used in the hydrolysis of fresh wood. However, such a recycle process requires usage of sulfuric acid and sodium carbonate in amounts which appear to be excessive.

Organic content of water effluents was shown to fall with increasing severity of liquefaction.

A sample of process water from Albany PDU run TR-12 (PERC process) was shown to contain about 36% sodium salts of organic acids. From titration plus weight of salts recovered, the equivalent weight of acids is about 80 ± 3.

Task 1 - Operation of the Continuous Liquefaction Unit (CLU)

Operation of the CLU was initiated on March 13. During the heat-up period water alone was fed, and this operation was continued for about two hours after the reaction temperature of 350°C was reached. The water was being collected in pressure letdown vessel 2. (PV-2, see flow sheet). All motor valves and control equipment operated well. Synthesis gas flow was controlled by a hand-operated micrometer needle valve, since the thermal mass flow meter was at the manufacturer's for repair and recalibration. While the gas flow control was somewhat erratic, it was decided to switch to wood slurry at 10 PM. Operation continued smoothly except that gas flow became more erratic and required constant attention.

At 11:11 PM flow was initiated into pressure letdown vessel number 1 (PV-1) and operation was continued in an effort to get a steady state run. Gas flow continued erratic, averaging close to target but fluctuating from zero to several times target despite operator efforts at hand control. After one hour a plug developed in the reactor, apparently the result of a sequence of gas stoppages. Examination of the reactor tube later showed that the plug was in a length from about 4 to about 10% of the way into the reactor, with the section before and after this region being clean.

Product collection and recovery were smooth and effective. The oil recovered by chloroform extraction was liquid, though viscous, at room temperature -- in appearance like the better quality oils from the PDU operation. Conditions and results of product analysis are summarized in Table I.

While a minor adjustment in the method of slurry circulation will be made, it is concluded that the major limitation on successful operation was the gas flow control. The next runs will be made after the thermal flow meter is returned (expected receipt date April 6-7).

Task 2 - Support Activities

2a. Slurry Preparation and Characterization

The colloid mill -- small scale substitute for a refiner -- has been used to reprocess the necessary quantities of slurry for CLU operation. Pumping tests on slurries have been run. From the pressure drops through tubing equivalent to the CLU reactor section) effective viscosities can be calculated for this non-Newtonian fluid. A plot of log viscosity vs. reciprocal absolute temperature is reasonably linear, so that it is possible to extrapolate to higher temperatures (cf figure 1). If there were no reaction,

the effective viscosities at CLU reaction temperatures would be about 4 poise. With effective viscosity in this range, it seems reasonable to anticipate good contacting of the slurry by synthesis gas. As time permits, further data on slurry behavior with and without gas flow will be obtained, as well as data on slurries made from feedstocks other than Douglas fir. Initial efforts, for example to get pumpable slurries from two very different peat samples appear to be successful.

Experiments with using process effluent water in the slurry preparation steps have shown that this involves sodium carbonate and sulfuric acid requirements which appear to be excessive (see under Task 3). The large production of water-soluble carboxylic acids in both prehydrolysis and liquefaction is the cause of the problem. Experiments with possible variations on the prehydrolysis procedure are being started. The objective is to either minimize or somehow, to capitalize on the substantial quantity of wood acids generated. At this point, however, we have no really promising ideas.

In prehydrolysis the initial pH has been 1.7-1.8, obtained by adding about .075-0.10 wt % sulfuric acid to the water. After prehydrolysis, despite the generation of wood acids (about 0.12N in the water) the pH rises to about 2.4 or 2.5. This is about the pH we would obtain, if all the hydrogen ion from sulfuric were neutralized, from the wood acids alone. Further, when recycle waters are used (estimated wood acid normality in the water after hydrolysis about 0.7) the pH rises only to 2.1. Both these results are compatible with average pK's of $\sim$ 4.1 for the wood acids. Although acetic acid (pK - 4.72) is the most important single acid product, titration curves indicate that an average pK of 4.1 is reasonable. This is covered elsewhere in the report (Figures 2 and 3).

Task 2b - Analytical Support to CLU

The procedure needed to characterize the CLU-1 oil product was slightly modified from that given in the previous report. The revised procedure, as applied to the recovery and analysis of CLU-1 oil, follows.

Most of the product oil was collected in two-gallon plastic bottles along with a preponderance of aqueous phase. The water layer was decanted. The oil phase was taken up in chloroform and filtered through a tared Soxhlet thimble. Continuous Soxhlet extraction with chloroform for 8-hours (until the extracting solvent was almost colorless) followed by removal of solvent by rotary evaporation at 20 torr and 40-50°C yielded 45 g of chloroform-soluble oil. The oil was free-flowing at room temperature. Another 6 g of oil was recovered from within the pressure letdown vessels and the outlet of the reactor tube or from container walls.

The chloroform-insoluble residue amounted to 4.2% of the organic product on a moisture and ash-free basis. Half of this residue was extractable upon continued Soxhlet treatment with acetone. Combustion analyses of chloroform-soluble oil, chloroform-insoluble and acetone-soluble oil, and acetone-insoluble residue are given in Table I.

The use of chloroform as extracting solvent seems quite satisfactory. Water contained in the product is removed azeotropically and collects as a separate layer on top of the chloroform within the Soxhlet thimble. About 1.0 g of water was collected with 45 g of chloroform-soluble oil. Chloroform is slightly more selective than acetone. The 2.1% of material found to be soluble in acetone but insoluble in chloroform was shown by gel permeation chromatography to be almost entirely high molecular weight material, mol. wt. greater than or equal to 830, the exclusion limit of the column used.

Our initial efforts in the area of GPC gave a preliminary value for the number average molecular weight of CLU-1 oil of 3738. More significant than the actual value, which is at best a rough approximation, is the comparison of the GPC chromatograms of CLU-1, TR-7 and TR-10 oils shown in Figure 4. These traces suggest that CLU-1 is of a slightly higher quality than TR-7 oil, the maximum in the plot of retention volume vs. detector response being shifted to slightly lower molecular weights. TR-10 composite oil, which is solid at room temperature, not surprisingly shows no such maximum, only a shoulder on the peak due to excluded (i.e., high molecular weights, $\geq$ 830) matter.

Although this GPC work is definitely of a preliminary nature, there is no doubt that GPC will prove valuable in the characterization of whole oils and fractions. The use of several columns in series with beads having higher molecular weight exclusion limits should provide better resolution. Also a comparison of traces of the same oil using different detectors (refractive index, infrared or ultraviolet at wavelengths other than 280 nm) should provide chemical information as well.

"Fingerprints of Oil Samples"

A certain amount of "fingerprinting" of oils by capillary gas chromatography has also been done. On a fused silica capillary column 2.5% solutions of oil samples could be separated into at least 100 different compounds; under the same gas chromatographic conditions of temperature program and amount injected, oil samples of different runs under different conditions show distinctly different patterns on gas chromatograms.

By comparison of oil samples from runs TR-11, TR-10, TR-7 and CLU-1 (Figure 5) one observes different "fingerprints" of volatile constituents at column temperatures between 50° and 250°C. In spite of the fact most of these consituents have not yet been identified, it is evident that oils with a lower viscosity show much more material at lower column temperatures compared to "oils" which have an asphalt-like appearance.

The density of lower boiling compounds on the chromatograms with retention times lower than 22 minutes is much higher in the TR-7 and CLU-1 oils indicating a breakdown into more of the smaller fragments. Most of the major peaks are present in all four oils, but with different intensities, showing that the basic mechanism of formation from high polymer material must be similar under the reaction conditions.

Under the same gas chromatographic conditions standards have been run on the Hewlett-Packard 5880 to give some information about the range of boiling points one could expect. Examples were:

Substance	Boiling Point	Retention time
Phenanthrene	340	23.12
3,4-dimethoxy-phenylacetone	300	20.18
Isoeugenol, (2-methoxy-4-propenylphenol)	268	17.80
3,4-dimethoxy-benzaldehyde	281	18.23
resorcinol	280	14.54
catechol	245	12.30
guaiacol	205	9.53
phenol	182	6.96
cyclohexanone	156	4.40

So far it is not known how much of the product oil is shown on these gas chromatograms; quantitative separation procedures are underway to determine the percentage amount of the oils which these chromatograms represent. But is is evident that substances with a molecular weight up to at least 200 and boiling points up to 350°C will be represented by these "fingerprints".

The following information remains to be obtained: distillation characteristics (ASTM D-1160 or D-2887-70T (simulated)), viscosity and density.

Composition of Aqueous Effluent

Since very little had previously been done to characterize the water effluent, we have spent a fair amount of time in this area. The organic and inorganic products dissolved in water are of importance for several reasons:

- The organics are at concentrations high enough to strongly influence the material balance.
- Their overall yield is large enough to make recovery desirable.
- Both organic and inorganic constituents constitute a potentially serious disposal problem.
- Water-soluble organics are formed whether or not there is a wood prehydrolysis as in the LBL process. Thus they are important in an oil recycle process (like PERC).
- The water-soluble materials are related to the mechanism of liquefaction. At the very least, the carboxylic acids and anions formed must be intermediates in the always extensive carbon dioxide formation.

The work on water effluents has been along four lines. Simple acid-base titration of free acids, organic anions and bicarbonate ion (if present); gas chromatography on alkaline and acidic water samples or on extracts, coupled with GCMS to identify components; extraction of acids and non-acids separately followed by determination of recoverable quantities; derivatization followed by GCMS.

From the titration curves alone, it can be shown that several types of organic acid are present (figures 2 and 3). The average pK of the carboxylic acids present is, from the titration curves, close to 4.0 (cf 4.76 for acetic acid). The range of pK's must be from about 3 or less to 5 or more. Carboxylic acids with pK's near 5.0 are numerous (acetic acid and homologues, most dicarboxylic acids, such as adipic, some hydroxy and keto-acids). For pK's below 4 we must look for compounds like α-hydroxy or α-keto-acids such as glycolic or pyruvic and their homologues, or for polyfunctional acids (e.g. citric). A list of acids which are clearly identifiable is shown in Table II.

Acetic acid is the most abundant c arboxylic acid -- apparently over 25% of most samples. In fact, acetic acid may well be the single largest individual product. It is formed both in the hydrolysis step and in liquefaction.

A tentative scheme for following the effect of process variables on the water-soluble product follows.

1. Titrate 20 g sample with 1.0N NaOH to pH ∿ 8.0 (free acid).

2. GC on alkaline solution. Then extract neutrals with chloroform (GC on extract).

3. Titrate raffinate with 1N HCl to pH ∿ 1.7. Correct for excess HCl and sulfate. Total carboxylic acid + anion (+ bicarbonate if present). GC on acid solution.

4. If bicarbonate was initially present, back titrate with 1N NaOH to determine net carboxylic acid + anion.

Exhaustive extraction of non-acids (from alkaline aqueous product) followed by acids (from the acidified raffinate) has been tried with several samples -- notably the water from PDU run TR-10. Some 25% or so of the acids are very resistant to extraction. These are largely compounds which do not give peaks in the gas chromatograph as we have been running it and are perhaps polyfunctional acids which are highly hydrophilic. By concentration and weighing of recovered extracts, we estimate the neutrals in the aqueous layer to be very approximately 40% of the weight of the acids. Also, the amounts of acid recovered are consistent with an earlier estimate of about 90 for their equivalent weight. The best estimate of equivalent weight, however, was obtained from the titration of the sodium salts remaining after extraction of neutrals from Albany PDU TR-12 water. Weight of sodium salts divided by equivalents of HCl required gave 107 as the equivalent weight for salt or 107-22 = 85 for the average equivalent weight of acid. A repeat analysis gave 77, so that about 80 $\pm$ 3 is a reasonable measure.

Since running aqueous salts or acids into the GC column causes a build-up it is necessary to have columns dedicated to each of the acid or alkaline samples. The GC's also give "fingerprints" which can be used to compare products obtained under different conditions.

Task 3 - Related Experimental Activities

Batch autoclave tests have been run to determine effects of severity and prehydrolysis on the aqueous layer and to investigate the possibility of recycling the water layer.

The effect of severity on total acid/anion content is shown in Table III There is a trend in content downward from the least severe (330°C - 0 min at temp) to the most (360°C, 30 min at temp), although little difference can be seen among the 3 middle-severity runs (330°C at 30 and 60 min and 360°C at 0 min). A run without prehydrolysis at 340°C, 0 minutes fits at about the expected place on the table, i.e., roughly the same quantity of acids is formed whether or not prehydrolysis is used.

Two series of recycle tests were made. In each, water from a liquefaction run was used in hydrolysis of fresh wood flour. Since the anions from the prior hydrolysis and liquefaction had to be acidified for the hydrolysis step and the acids then neutralized before liquefaction, increased amounts of sulfuric acid and sodium carbonate were required.

In the first series of runs, the amount of sodium carbonate was limited to 3% of the weight of dry wood hydrolyzed. The slurry pH before liquefaction was 3.2-3.4 and after liquefaction the aqueous phase was scarcely changed (3.3-3.6). The total acidity (free acid plus anion) was 0.7 equiv./kg in the water from the final phase. Yields of acetone insolubles were high ($\sim$ 25% of dry wood).

In the second series, the sodium carbonate necessary to bring the slurry to a pH of about 7.0 was added. In a typical run, with 70% of the product aqueous phase recycled (30% make-up water), the sulfuric acid required was about 8% of the weight of dry wood and the sodium carbonate about 18%. Final pH's of product water were about 5.0-5.3 and the total content of carboxylic acid plus anion was about 0.8 equivalents per liter (if the equivalent weight is 80, this corresponds to 6.4 wt% of the water).

It is easy enough to calculate the necessary usage of sulfuric acid and sodium carbonate from the anion and acid content of the aqueous phase. For example, assume a process in which 1000 kg dry wood, 1000 kg fresh water (as content of wet wood chips or of sodium carbonate solution) and 2500 kg recycle water, containing 0.7N anions and 0.15N free acids, are mixed before hydrolysis. Assume also, based on experience, organic acids equivalent to 0.12 eq/kg H_2O are generated in the hydrolysis stage. Calculations indicate that 88 kg H_2SO_4 is needed and 172 kg Na_2CO_3 to bring the slurry to pH = 8.0. This is 8.8% and 17.2% of the weight of dry wood, respectively, and is in the range found in the experiments. It appears to be a heavy load for the economics to bear.

One reason so much sodium carbonate is required is the fact that sodium bicarbonate is such a weak base. Figure 6 shows typical curves for the titration of prehydrolyzed slurry with 0.2N NaOH and 0.1 M (0.2N) Na_2CO_3. Beyond about pH = 5.1, sodium carbonate no longer contributes two equivalents per mol. If we go to pH = 8.0 with sodium carbonate, 0.1M Na_2CO_3 contributes effectively about 0.16 equivalents per liter.

An aqueous product sample with pH > 5.1 can be expected to have an appreciable content of bicarbonate ion. The amount can be estimated by titrating to pH $\sim$ 8 with NaOH, back titrating to low pH with HCl, then retitrating with NaOH after the carbon dioxide formed has evolved.

Task 4 - Evaluation of Process Improvements

The best news of this past quarter was the demonstration at Albany of the operability of the fired tubular reactor. There were problems with the LBL-mode runs (TR-10 and TR-11), but these were downstream of the tubular reactor. Clearly neither the stirred autoclave nor the standpipe reactor (as conditions forced them to be run) was satisfactory for adding the necessary additional residence time. The PERC run, TR-12, perhaps because the high oil recycle takes care of the additional residence time, was more satisfactory. The initial partially successful operation of the CLU, encourages us to believe the residence time question can be decided on our bench-scale -- possibly also the question of the value of a partial oil recycle.

We are looking at TR-10 and TR-11 oils in our analysis sequence and will look at TR-12 when it is received. TR-10 and TR-11 oils are clearly higher in molecular weight than TR-8 or the recent CLU product. Also, capillary gas chromatography indicates that the volatile components of TR-10 and TR-11 oils are less abundant.

One interesting piece of information came from titration and extraction of product water from PERC run TR-12. The initial pH of this phase was slightly alkaline. Titration with HCl and back-titration showed the water to be 4.04N in organic anions (carboxylates) and about 0.06N in bicarbonate ion. Extraction of the water at pH 9 to remove neutrals (including most phenols) and evaporation of the water gave a residue of sodium salts. By titration of this residue with HCl it was shown that the organic acids have an equivalent weight of about 80. They correspond to 29.2% of the water (the sodium salts were 36.7%). Since the density of the water was 1.175, the weight percent sodium salts is equivalent to (density - 1.0)·210, which might be a rough rule of thumb for estimating salts concentration.

The HCl titration curve is shown as Figure 3 . . Note that the half-way point, a rough measure of average pK, is at pH = 4.1.

If, as in run TR-12, sodium carbonate is added to the feed in sufficient quantity to keep the effluent water slightly alkaline, we can estimate the yield of organic acids by the relative equivalent weights;

$$\text{Yield acids} = (\text{lbs } Na_2CO_3/100 \text{ lbs dry wood}) \cdot \frac{\text{EQwt acids}}{\text{EQwt } Na_2CO_3}$$

$$= \%\ Na_2CO_3 \cdot 80/_{53} = \%\ Na_2CO_3 \cdot 1.51.$$

If the required Na_2CO_3 addition is 4%, the yield is 6%. By extraction and weighing of non-acids, we found these to constitute only 1.3% of the product water. However, Albany reports considerable organic carbon in the condensate water. This probably includes most of the non-acids plus some acids. So that the total yield of water-soluble organics must be much higher than 6%.

TABLE I

OPERATING CONDITIONS AND PRODUCT-RUN CLU-1

Slurry (20% total solids)	1.03 kg/hr
Gas (52.5% H_2, 47.5% CO)	0.8L/min
Outlet temperatures	335-358°C (345°C average)
Pressure	3050 PSIG inlet, 3000 PSIG outlet
Oil product recovery	53 g (= 28% of dry organic weight fed)
Water phase recovery	810 g
Residence time	∿ 15 min
Estimated Reynolds No.	960

Analyses - Oil Phase (maf basis)

% chloroform soluble — 95.8; C% 75.4, H% 7.7, O% 16.9
Calculated HHV: 14,600 Btu/lb

*% Chloroform insoluble, acetone soluble — 2.1; C% 77.8, H% 6.0, O% 19.8

*% Residence (acetone insoluble) — 2.1; C% 75.9, H% 5.2, O% 18.6.

* These calculated ash free; samples contained 3.0 and 4.6% ash respectively; also 0.4 and 0.3% nitrogen.

Water Phase (pH = 5.3)

Acidity 0.068N

Anions 0.125N

Total 0.193N (∿ 1.64 wt % of water) = 6 wt % of dry feed)

Total Organic Carbon 1.60%

Neutrals recovered from 100 ML water by chloroform extraction:
1.32 g = 1.32 wt % of water = 5.3 wt % of dry feed.

Gas Phase -- Final Analysis (Not yet steady state).

H_2 46.6%

CO 13.3%

CO_2 40.1%

TABLE II

SOME ACIDIC COMPONENTS OF AQUEOUS PRODUCT

A. Monocarboxylic Acids

Acetic	CH_3COOH
Propionic	C_2H_5COOH
Butyric	C_3H_7COOH
Isobutyric	C_3H_7COOH
C_5-C_8 Straight Chain + Isos	Probable

B. Dicarboxylic Acids

Succinic	$(CH_2)_2(COOH)_2$
Glutaric	$(CH_2)_3(COOH)_2$
Methyl Succinic	
Adipic	$(CH_2)_4(COOH)_2$
2-Methylglutaric	

C. Oxo - or Hydroxy-Acids

Levulinic	$C_5H_8O_3$
2,3 - Dihydroxybenzoic	$C_7H_6O_4$
4-Oxohexanoic	$C_6H_{10}O_3$
5-Oxohexanoic	$C_6H_{10}O_3$
γ-Valerolactone	$C_5H_8O_2$
(2 4-Hydroxyvaleric)	$C_5H_{10}O_3$

TABLE III

EFFECT OF PROCESS SEVERITY AND PREHYDROLYSIS ON CARBOXYLIC ACID FORMATION

Reaction Conditions		Total Carboxylic acid plus anion, equivalents/kg in process water
Temperature °C	Time at * Temperature, Minutes	
330	0	0.58
340	0	0.52 **
330	30	0.44
360	0	0.43
330	30	0.41
360	60	0.36

* Excluding heat-up time (∿ 25 min to 330°, ∿ 35 min to 360°) and cooling time.

** Prehydrolysis omitted for this run.

FIGURE 1
EFFECTIVE VISCOSITY OF ALBANY SLURRY

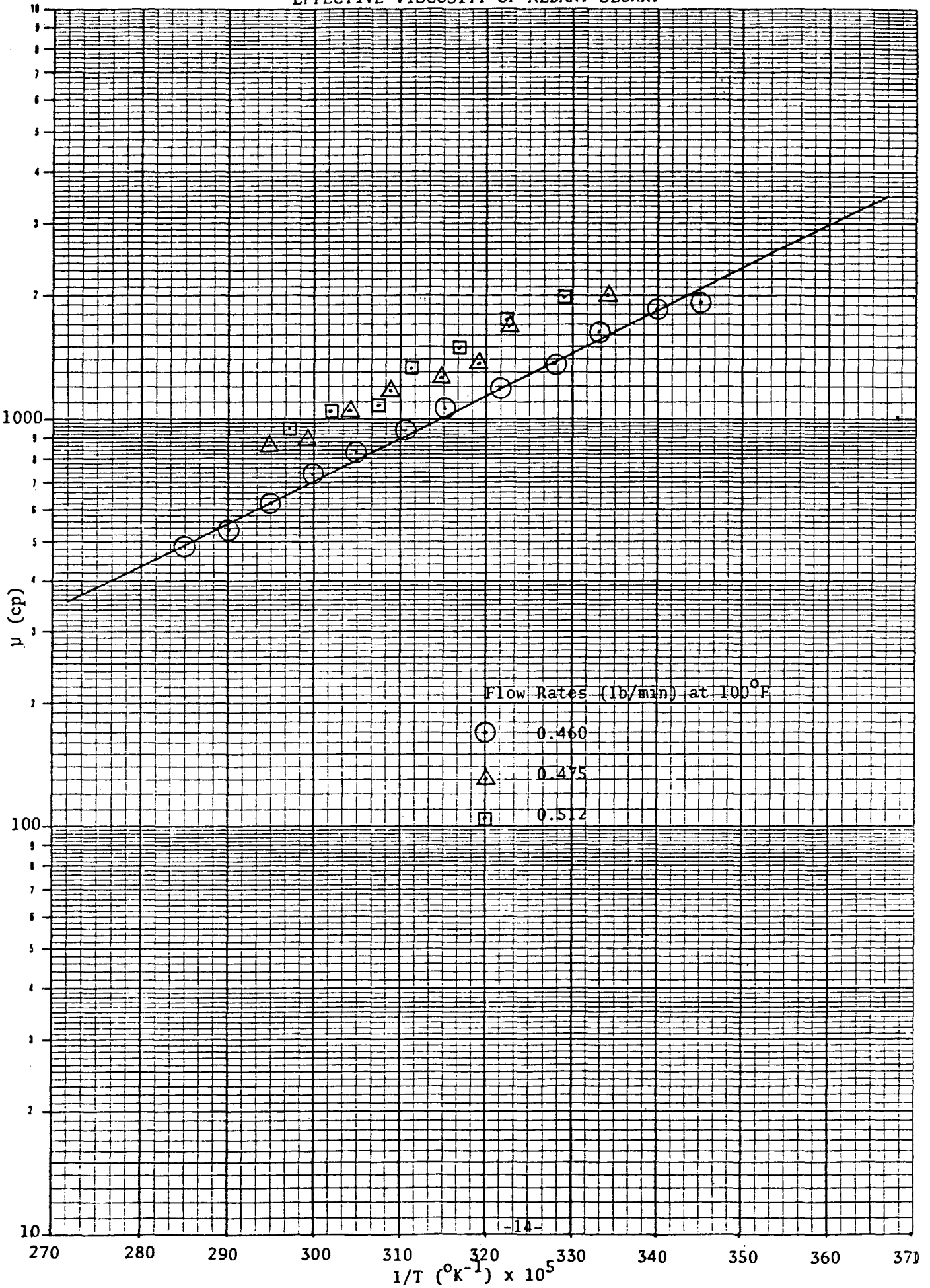


FIGURE 2
TITRATION OF PROCESS WATER

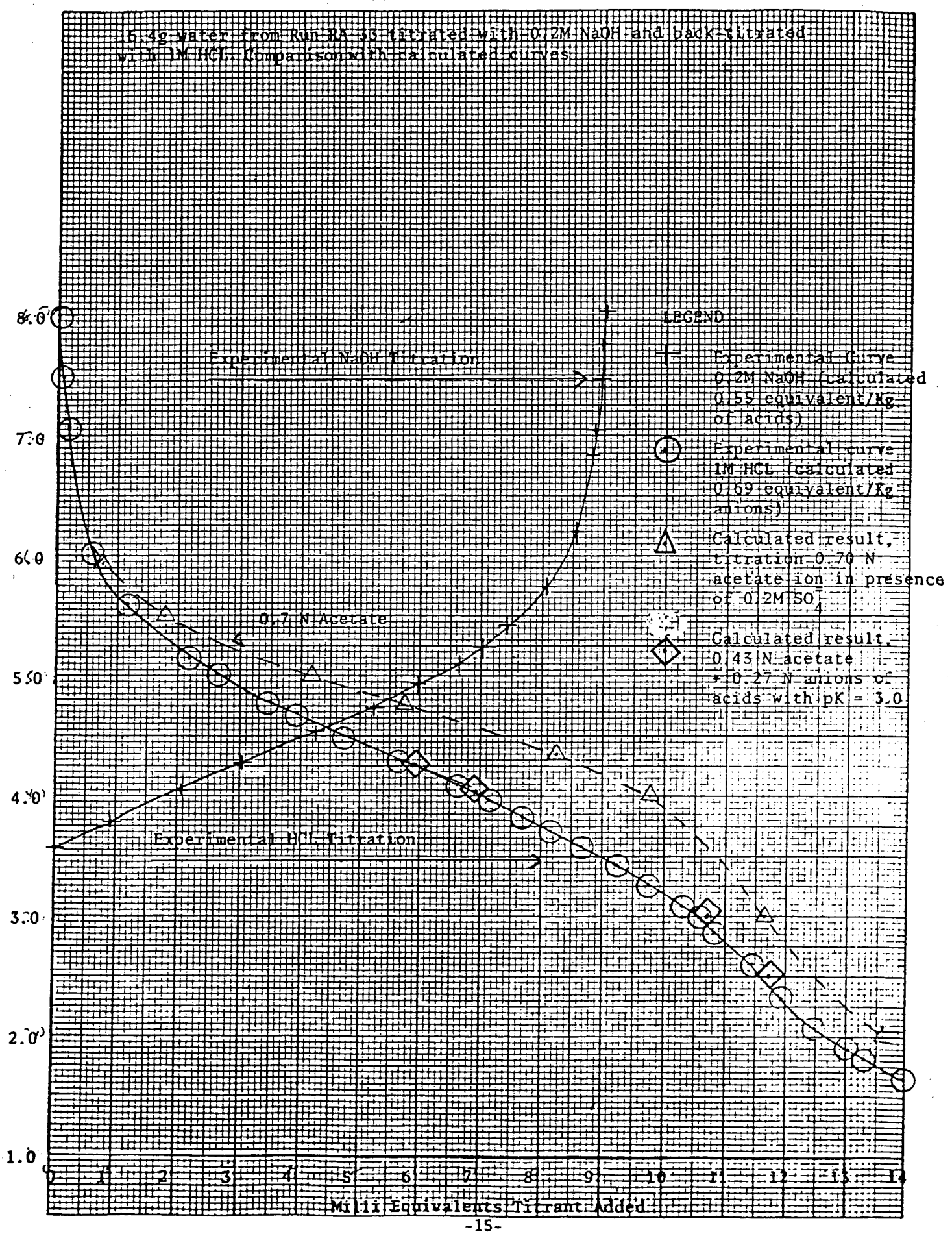


FIGURE 3

TITRATION OF SODIUM SALTS
FROM ALBANY WATER SAMPLE (TR-12)

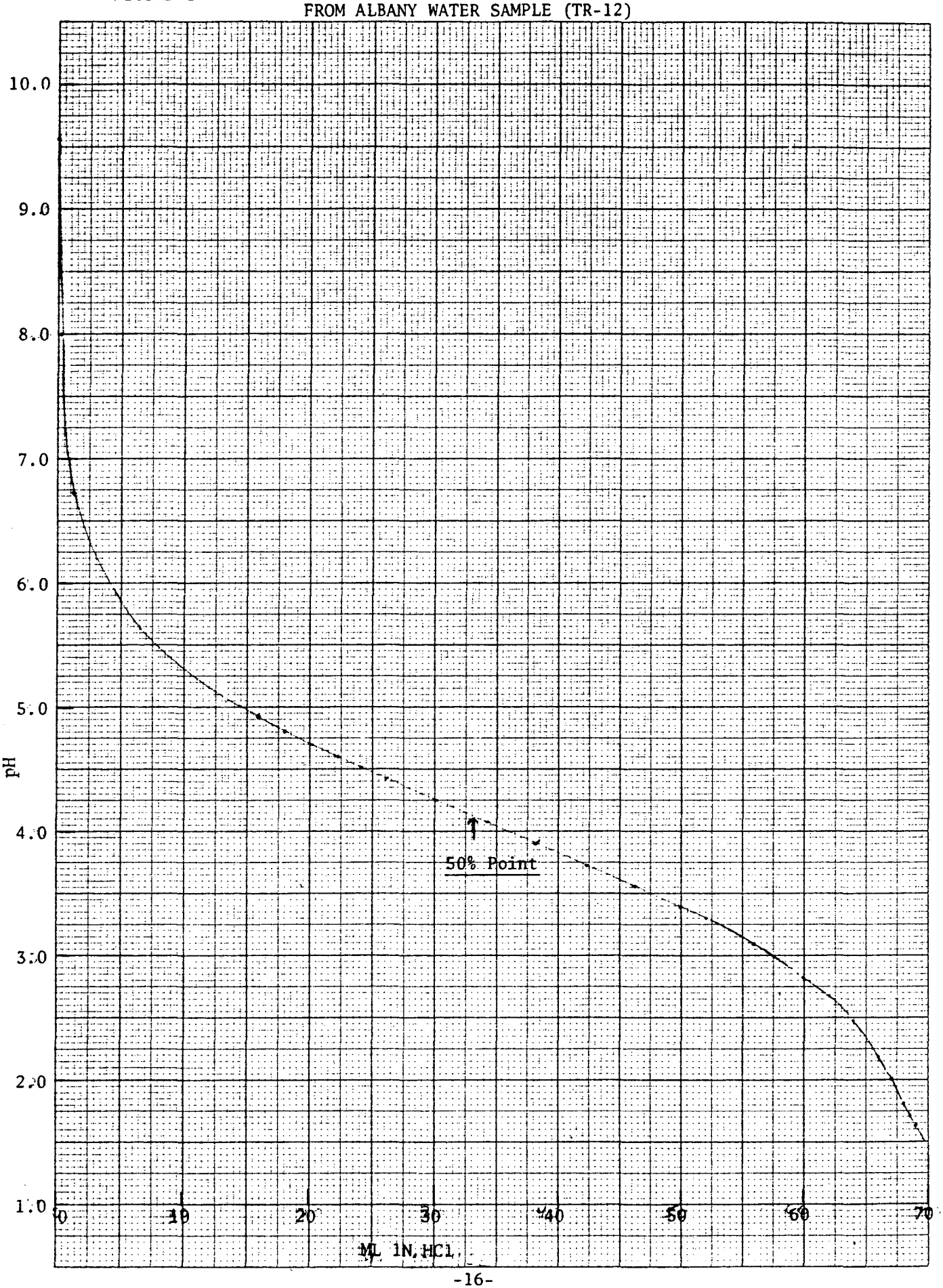


FIGURE 4

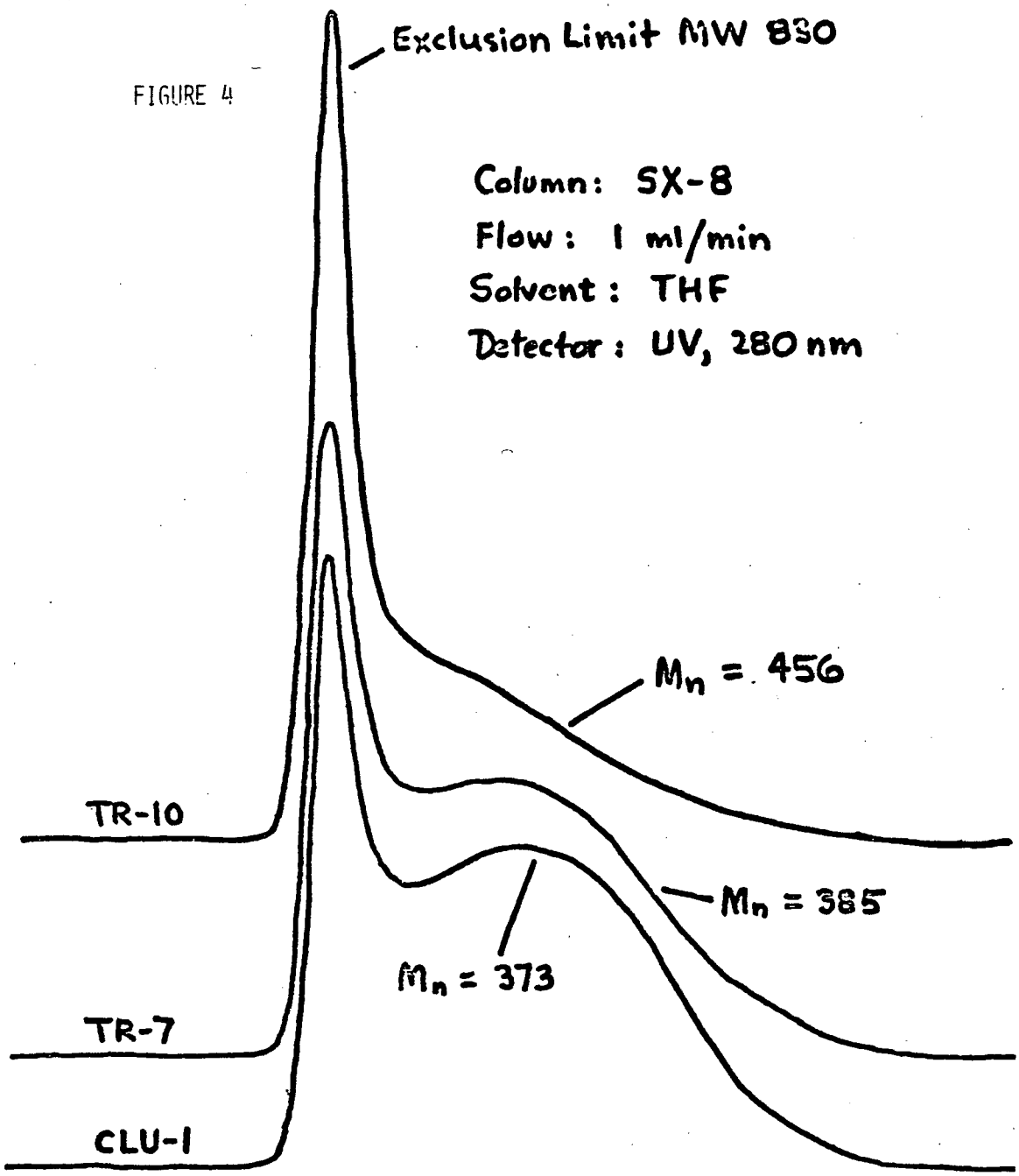
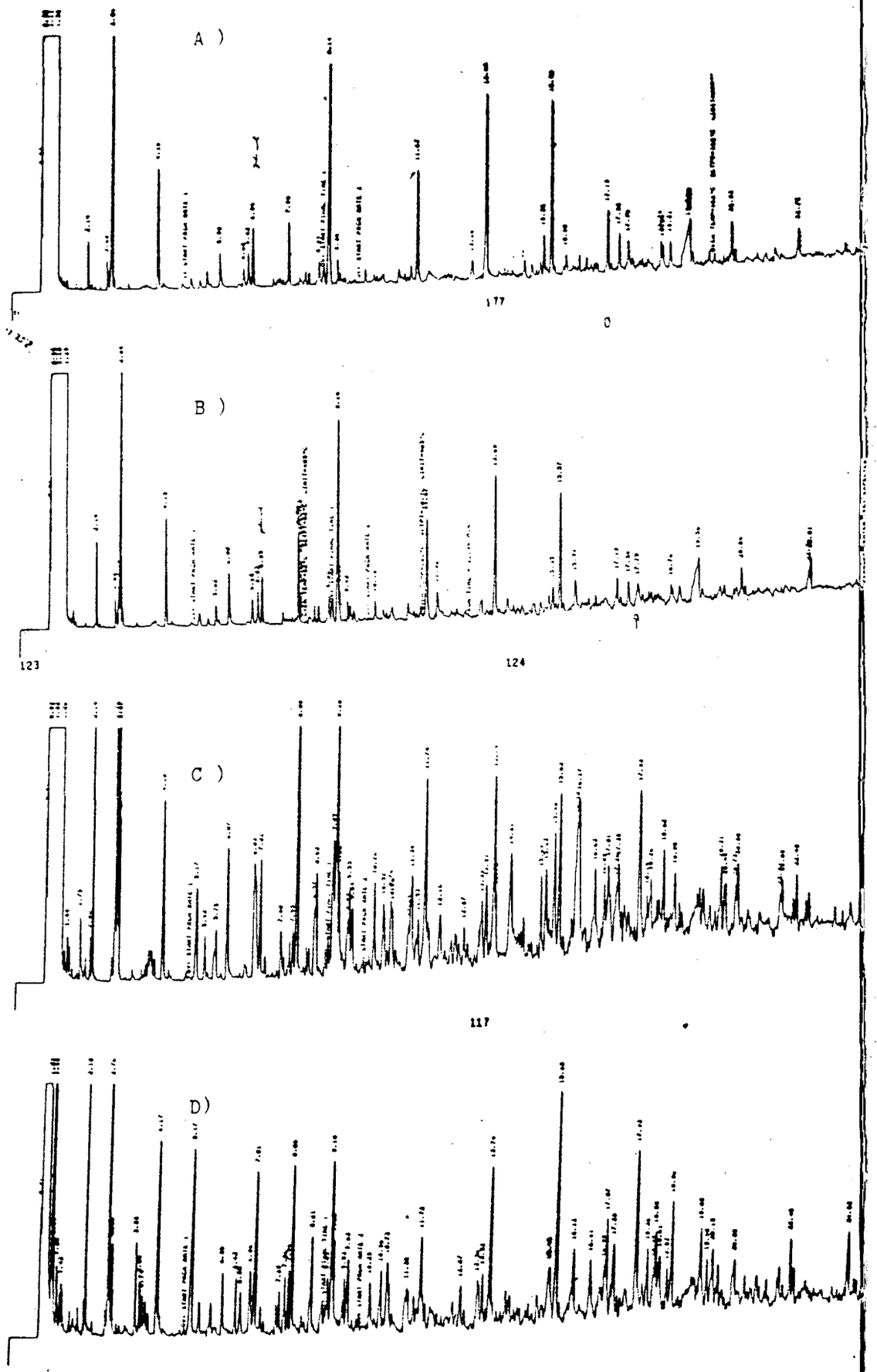
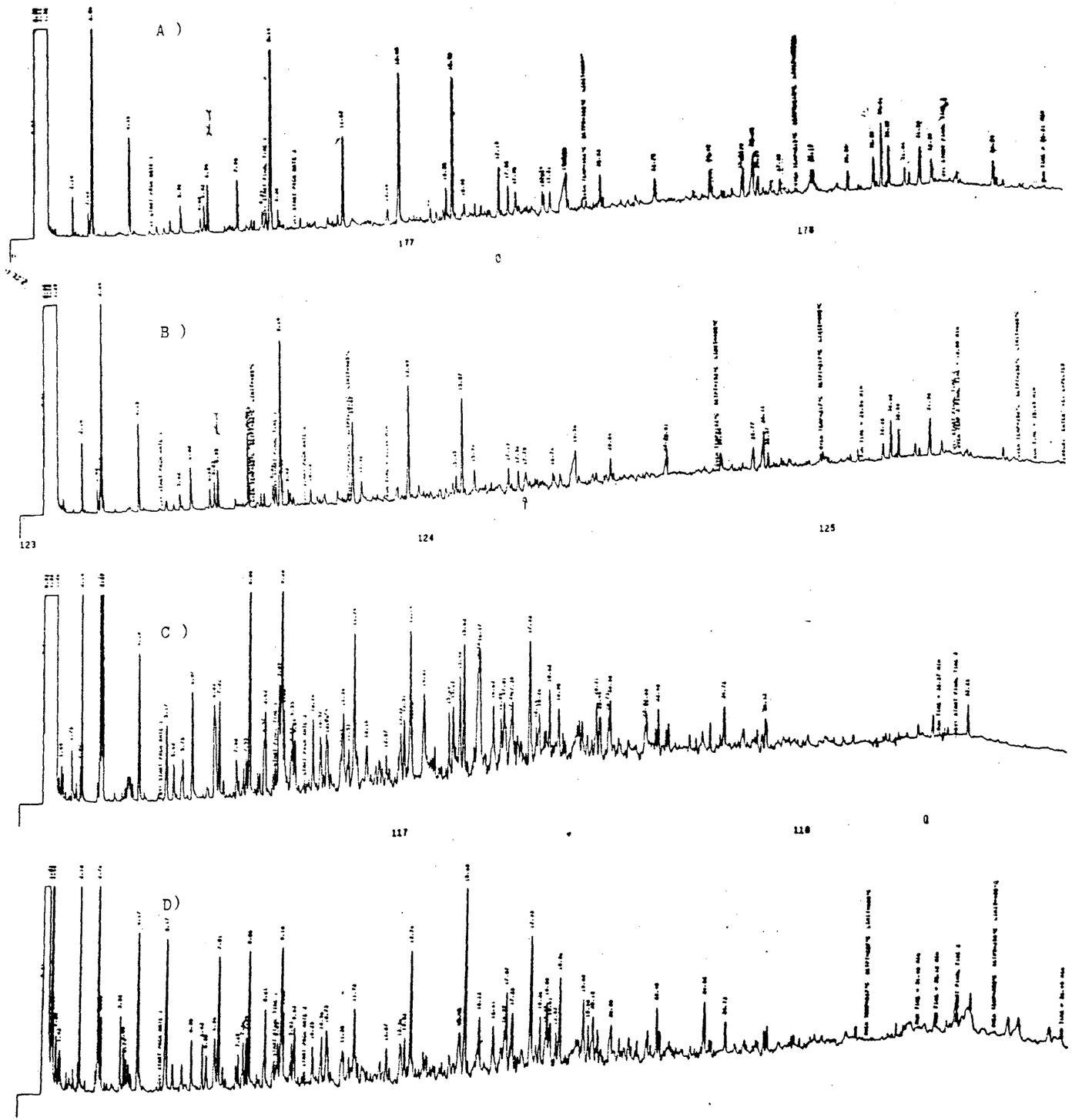


Fig. 4. Gel permeation chromatograms of biomass oils. M_n is the number average molecular weight calculated by means of the GPC II computer program provided by Perkin-Elmer Corp. Eight calibration standards ranging in mol. wt. from 78 to 659 were employed.



"FINGERPRINTS" of oil samples: A) TR-11 -HEWLETT PACKARD 5880A - tempe
B) TR-10 -fused silica capillary column
C) CLU-1
D) TR-7

FIGURE 5

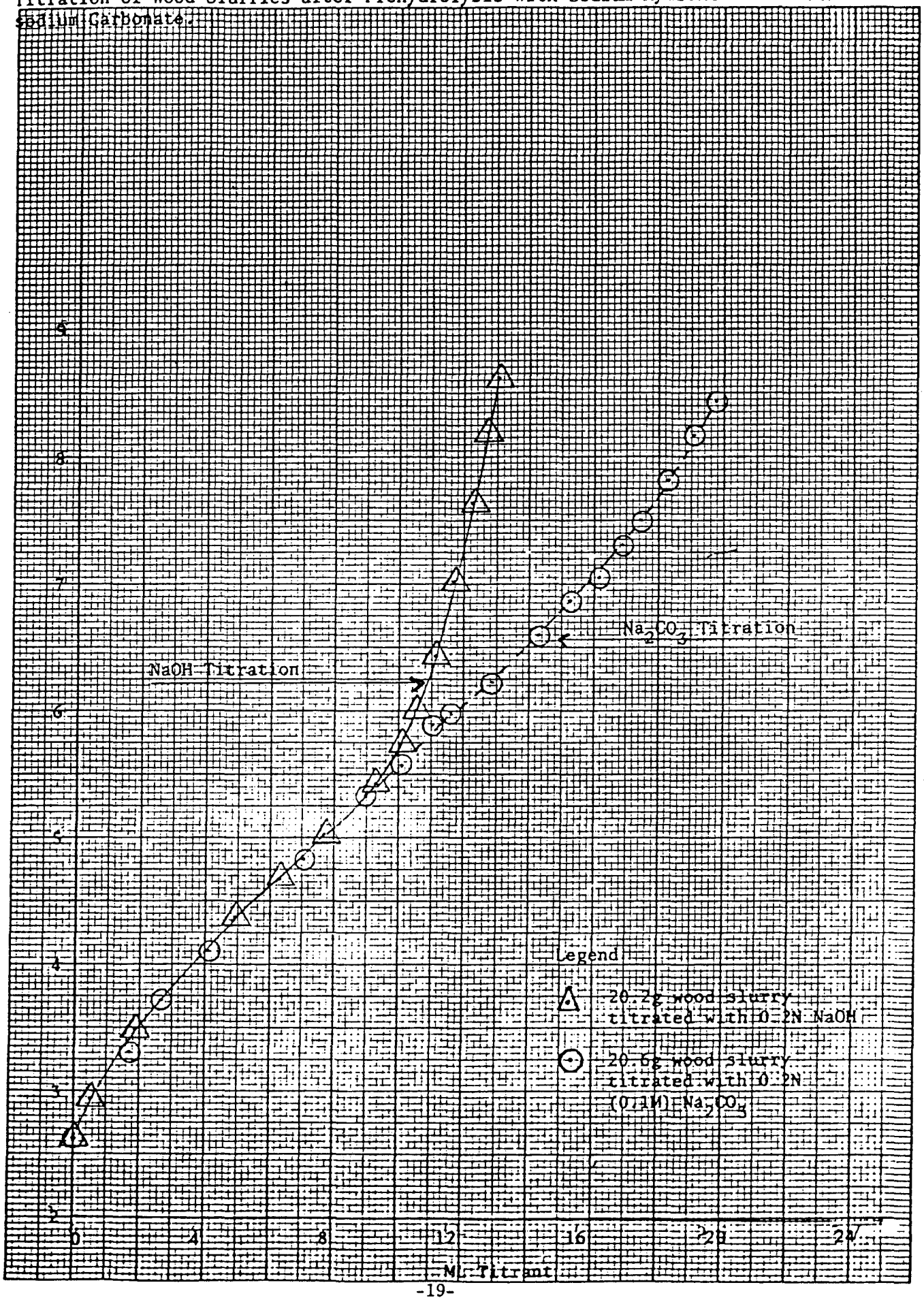


"FINGERPRINTS" of oil samples: A) TR- 11 -HEWLETT PACKARD 5880A - temperature range: 50 to 250°C
B) TR-10 -fused silica capillary column, 30mx0.32 mm, liquid phase DB5 .
C) CLU-1
D) TR-7

FIGURE 5

FIGURE 6

Titration of Wood Slurries after Prehydrolysis with Sodium Hydroxide and with Sodium Carbonate.



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