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PROGRESS REPORT
CONTROL INDICES AND ANALYTICAL PROCEDURES

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SUMMARY

The present report covers the completion of one phase of the assigned objectives as described under "Control Index Studies. III. Determination of the Relationship of Specific Gravity to Stainless Steel Concentration." Data are presented for the specific gravities at 25, 40, 60, and 80°C of samples of originally 4M, 5M, 6M, and 7M sulfuric acid solutions, to which have been added 0, 20, 30, 40, 50, 60, and 70 grams per liter of type 347 stainless steel.

In addition to tabular presentation of the specific gravity as a function of steel concentration at each temperature, graphical presentation of such data is given together with the corresponding values of the absolute densities of the solutions and their temperature coefficients. Equations have been derived relating the observed specific gravity or density at each temperature to the stainless-steel concentration and the original sulfuric acid concentration. The possible precision of using specific gravity or density as an index of steel concentration is indicated. The experimental procedure and means of calculation are described in some detail.

PROGRESS REPORT

CONTROL INDICES AND ANALYTICAL PROCEDURES

SUMMARY OF RESULTS

CONTROL INDEX STUDIES. III. DETERMINATION OF THE RELATIONSHIP OF
SPECIFIC GRAVITY TO STAINLESS STEEL CONCENTRATION

A. Objective and Implementation

Objective. The object of the present study is the development of a relationship between specific gravity and concentration of dissolved type 347 stainless steel in sulfuric acid solutions at various temperatures. This relationship would indicate the analytical feasibility of determining by a specific gravity measurement the concentration of stainless steel present in a sulfuric acid solution. The 28 solutions investigated at temperatures of 25, 40, 60, and 80°C contained 0.0, 20.0, 30.0, 40.0, 50.0, 60.0, and 70.0 grams of stainless steel per liter of originally 4.0M, 5.0M, 6.0M, and 7.0M sulfuric acid.

In order to facilitate any attempt to repeat or to extend the work, an attempt has been made to describe the technic in sufficient detail to permit such repetition or extension. In addition, evaluation of the present report and the work on which it is based will rest on a better foundation relative to the merits and deficiencies of the results.

Preliminary Work. Early considerations led to the use of a Westphal-type specific gravity balance rather than an Ostwald or other type of pycnometer. Use of a pycnometer would have produced difficulties at the higher temperatures, and would have been more time-consuming. With the balance, determinations can be made accurately and in rapid succession, and, as subsequently indicated, numerous transfers are avoided. The balance arrangement is also suitable for the measurements to be made involving hydrofluoric acid solutions, whereas it would have been inadvisable to use glass pycnometers, and rather difficult to fabricate and use plastic pycnometers. The specific gravity balance used is accurate to 2 units in the fourth decimal place. A more sensitive type of specific gravity balance is available, but one could not have been obtained without delay when the work began.

B. Experimental Procedure

Preparation of Sulfuric Acid Solutions. The specific gravity of a large stock of concentrated sulfuric acid (chemical pure grade, 96% H_2SO_4) was determined. From this, the molarity of the stock acid could be ascertained by reference to the tables in a handbook such as Lange's Handbook of Chemistry. Knowing the molarity and specific gravity of the stock acid, the weight of it to be used for dilution to the desired amount of sulfuric acid solution of a given molarity is calculated by the following equation:

$$\text{Wt. in grams of stock acid needed} = \frac{\text{Molarity desired} \times \text{Density of stock acid} \times \text{Volume in ml. of solution desired}}{\text{Molarity of stock acid}}$$

The exact molarity of the resultant solution is determined by measuring its specific gravity and referring again to a handbook. In this manner, solutions of sulfuric acid were prepared which were 3.935M, 4.850M, 5.927M, and 6.882M.

Preparation of the Stainless-Steel Solutions. To prepare solutions of stainless steel of the desired concentrations, the amounts of stainless steel necessary for 225 ml of solution were weighed. To each sample, transferred to a 250-ml pyrex bottle, 225 ml of acid of the proper molarity was added. The resulting solutions were then in effect "molal" with respect to the steel. Thus, the acids themselves are molar solutions (moles of sulfuric acid present per liter of solution), while the steel concentration is expressed on an empirical basis (grams of steel added per liter of original solution). The equivalent of "20 grams per liter of solution" is actually 20 grams of steel plus 1000 ml of acid. The volume of the final solution would be increased by the addition of the dissolved steel constituents and decreased by the hydrogen and perhaps sulfate ion lost in the steel dissolution process.

The solutions were then allowed to stand for two days for dissolution to be completed and for more or less uniform aging before measurements were made, one day at room temperature and the second at an elevated temperature, i.e., between 60 and 80°C. Clear solutions were obtained in all but one or two instances as subsequently noted.

The steel used was supplied as a milled lot of metal by the resident representative of the American Cyanamid Company and was claimed to be representative of the type 347 stainless steel for which data were desired. The composition of stainless steel, designation 347, is given by the American Iron and Steel Institute as:

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Carbon	0.08% max.	Sulfur	0.030% max.
Manganese	2.00% max.	Chromium	17.00 to 19.00%
Silicon	1.00% max.	Nickel	9.00 to 12.00%
Phosphorus	0.040% max.	Columbium	10 (% C) min.

A sample of the particular lot used has been sent to the Idaho Falls installation of the American Cyanamid Company for emission spectrographic examination as well as for complete determination of composition. This information will be added to this report as an appendix or issued as a supplement when it is available.

Sample Containers. The container chosen was a 250-ml pyrex glass-stoppered reagent bottle with a standard taper joint (Fig. 1). The size of the bottle was determined by the dimensions and arrangement of the specific gravity balance. Use of the same bottle for the dissolution and the specific gravity determination eliminates the necessity for solution transfer, thus removing a possible source of error. The samples can be prepared, stored, brought to temperature, and measured and the storage-temperature-measurement cycle repeated without any solution transfer. The standard taper joint allows the sample bottle to be fitted with a condenser which is useful in measurements at the higher temperatures.

Specific Gravity Balance. The balance used was an inexpensive one of the chainomatic Westphal type manufactured by L. W. Hohwald, Jersey City, New Jersey. The balance (Figs. 2 and 3) did not have its own case, but was kept in a large wooden case with a glass door. The case was large enough to permit ready manipulation of the small sample constant-temperature bath resting on the balance floor and to facilitate interchange of sample containers. The thermoregulator was fastened to the rear inside wall of the case, several inches from the sample bath. All water and electrical leads were brought in through holes in the walls of the case.

As illustrated in Figs. 2 and 3, the beam of the balance is graduated in intervals of 0.1 from 0.0 at one end of the left arm, where the chain is fastened, to 2.0 at the end of the right arm, from which the plummet is suspended; 1.0 occurs over the central knife edge. A 10-gm roller weight moves on the beam and is used to obtain the unit and first decimal figures of the measurement. The second, third, and fourth decimal figures are read from the scale and vernier on the post (left side of the balance), up and down which the chain carrier moves.

In operation, the balance is first "zeroed" with distilled water at the temperature at which the measurements are to be made. This is done by setting the roller weight and chain at whatever value is to be assigned water in the ensuing measurements (1.0000 was used, as is indicated subsequently), and adjusting set-screw weights at the left and right ends of

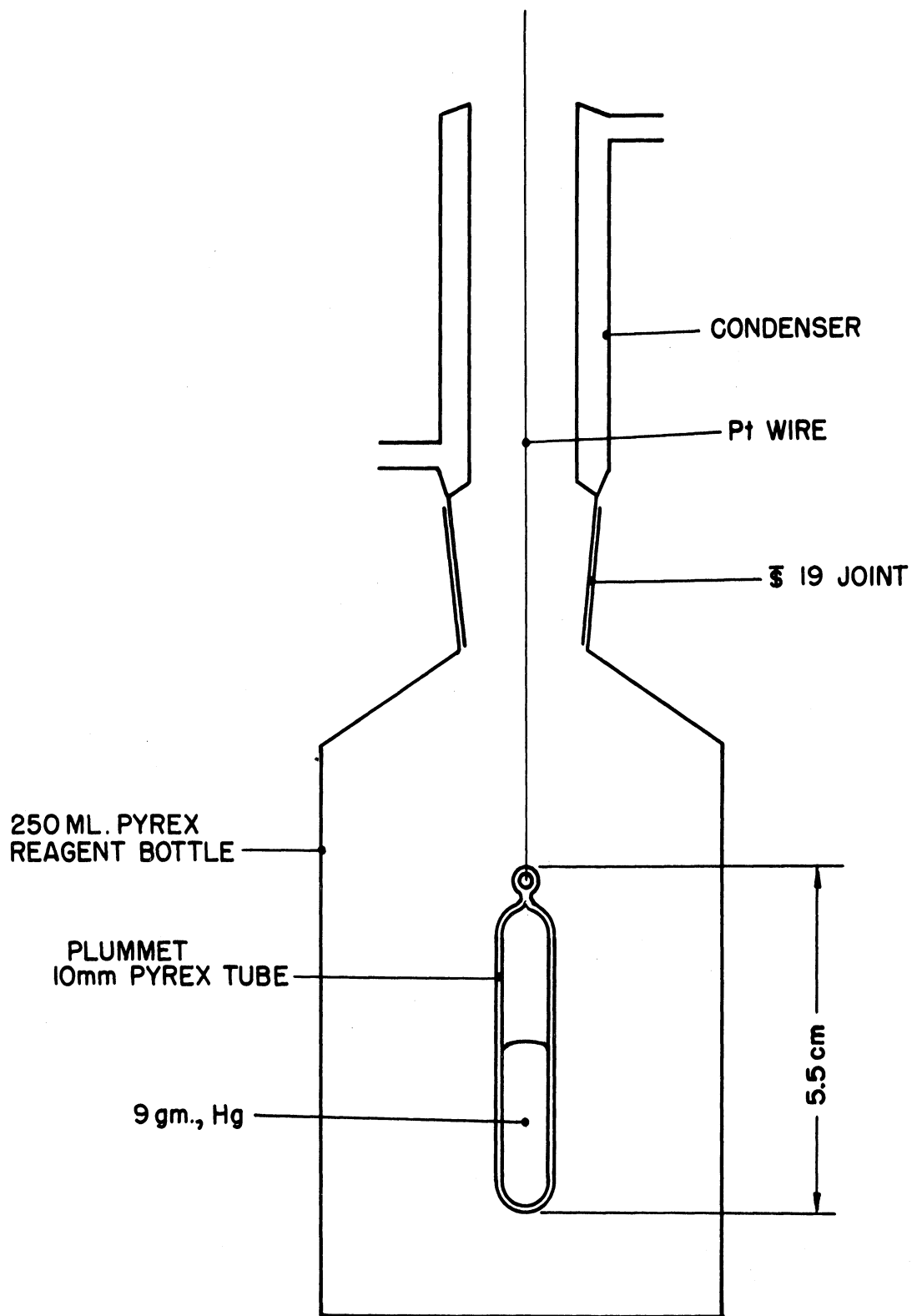


FIG. I.
SAMPLE CONTAINER

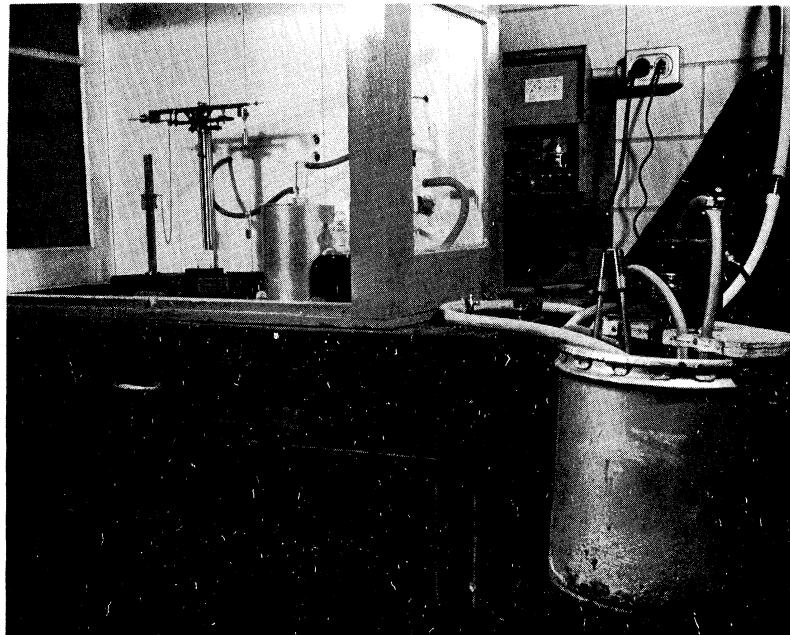
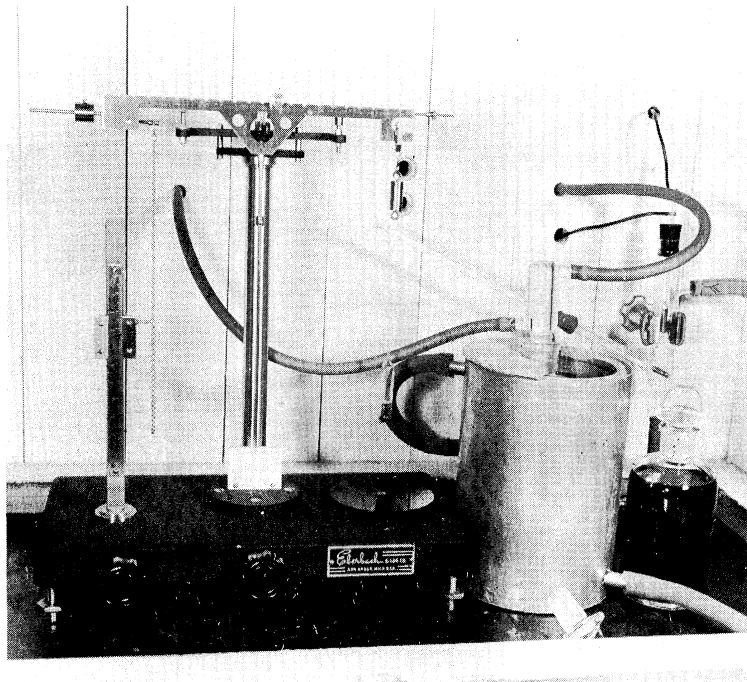


Fig. 2. Specific Gravity Balance and Constant-Temperature System.

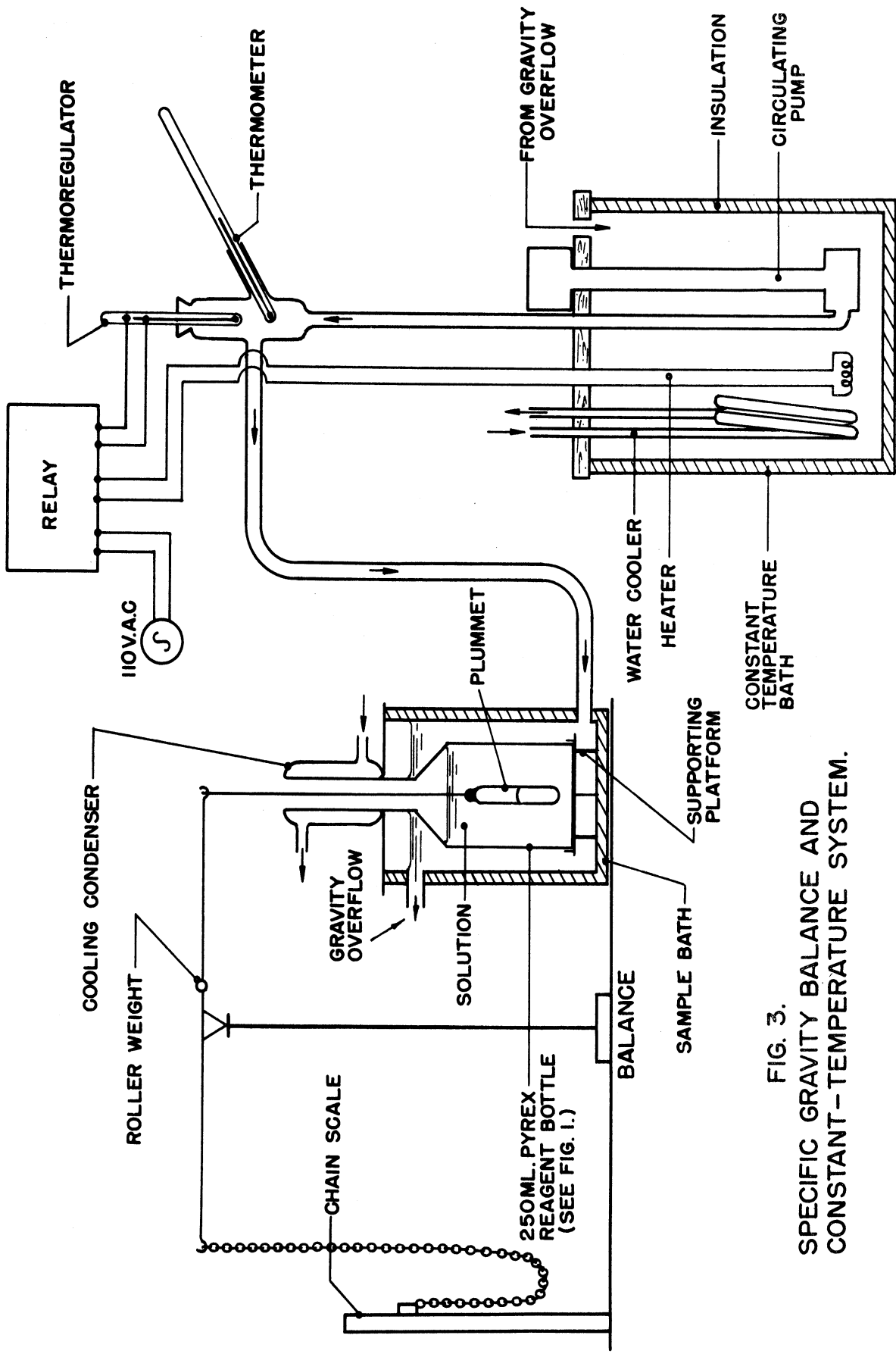


FIG. 3.
 SPECIFIC GRAVITY BALANCE AND
 CONSTANT-TEMPERATURE SYSTEM.

the balance beam until the scale pointer comes to rest at zero on the balance scale. The distilled water is then replaced by the solution to be measured. If a solution more dense than water is being tested, the plummet is buoyed up with a force equal to the difference in weight between the volume of water displaced by the plummet and the volume of solution displaced. This force is counteracted by adjustment of the roller weight and chain until the pointer again indicates zero. The balance is then calibrated to read directly in specific gravity units. However, for the present investigation a new plummet had to be designed and a correction factor determined which would convert the reading with the new plummet to specific gravity (see Section C).

Constant-Temperature System. The constant-temperature system consisted of four main units: (1) a constant-temperature bath, (2) a sample bath, (3) a thermoregulator, and (4) a relay.

The constant-temperature bath (lower right of Figs. 2 and 3) consisted of a 3-gallon copper can inserted in a 5-gallon metal container with vermiculite insulation between the walls. Immersed in the copper can were a copper coil carrying cooling water (building tap service water at about 15 to 18°C), a 1000-watt heater connected to the relay, and a circulating centrifugal pump. The can was kept almost full of water.

The water from the constant-temperature bath was pumped past a thermoregulator which was an H-B Thermometer Company No. 7910A fixed-point thermostat. The thermoregulator housing also contained a thermometer which was used as a check on the operation of the whole system.

The constant-temperature water then passed to the sample bath, from which it returned by gravity overflow to the constant-temperature bath. The sample bath consisted of an aluminum can approximately 4 x 6 inches in another aluminum can approximately 5 x 6.5 inches, with insulation between the walls. The sample container was positioned and held in the bath by a platform of 0.5-inch strips of aluminum. The top of the sample bath was covered by a split circle of Transite which fitted around the sample bottle neck.

The relay was a No. 7930 mercury relay manufactured by the H-B Thermometer Company. Temperature control of the sample was maintained to within 0.1°C of the desired temperature, i.e., 25, 40, 60, or 80°C. The rate of passage of tap water through the cooling coil was regulated so as to produce a relay cycle of a minute or less. The time required for a sample to reach the system temperature was determined by suitable experiments. In practice, the samples were brought to within a degree or two of the desired temperature before being transferred to the sample bath.

Procedure for Specific Gravity Determination. The sample solution was first heated by immersion in a large pan of water to approximately the temperature at which the determination was to be made. It was then immersed in the sample bath and left to reach temperature equilibrium. The

plummet was next immersed in the sample solution and was left for a minute or two until it came to approximate temperature equilibrium with the solution. Then several specific gravity readings were taken at 1/2-minute intervals until a constant reading was obtained. This reading was recorded, as it indicated temperature equilibrium. Generally, equilibrium was obtained in 2 or 3 minutes and about 3 or 4 readings were taken to establish constancy. The plummet was removed, washed in distilled water, rinsed with acetone and hung up to dry. Meanwhile, the next solution to be examined had been brought to temperature and was ready to be immersed in the sample bath a few seconds after the previous sample had been removed and placed in a bath (another large pan of water) for heating to the next higher temperature at which its specific gravity was to be measured.

C. Calculations and Treatment of Data

Equations for Use of Specific Gravity Balance. A new plummet had to be fabricated in order to fit the dimensions of the sample container. Since this plummet had a volume of displacement different from that of the plummet supplied with the balance, the calibration of the balance which was intended for use with a plummet of 10-ml displacement had to be corrected for use with the new plummet.

The equation derived, which relates V (volume displaced by the plummet), R (the balance reading) and the specific gravity based on "zeroing" with water, is:

$$\text{Specific gravity} = \frac{10 (R - 1)}{V} + 1.$$

By measuring several solutions of known specific gravity, readings were obtained from which the volume of the plummet could be determined empirically. The equation obtained for use with the new plummet was:

$$\text{Specific gravity} = \frac{10 (R - 1)}{3.493} + 1,$$

where R is the "observed value" recorded in Tables I to IV. Since the specific gravity is measured after the balance has been "zeroed" using water at the temperature concerned, the value obtained refers to the apparent density of water as 1.0000 at that temperature.

Conversion of Specific Gravity to Density. Since the density of water is unity at 4°C, in order to convert the specific gravity as observed under the conditions used to absolute density, the specific gravity must be multiplied by the density of water at the particular temperature at which the determination was made. The values for the density of the solutions

TABLE I

SPECIFIC GRAVITIES AND DENSITIES OF SOLUTIONS OF TYPE 347 STAINLESS STEEL IN
 ORIGINALLY 3.935M H₂SO₄ AT VARIOUS TEMPERATURES

Concn of Steel, gm/liter	Observed Value	Specific Gravity	Density	Observed Value	Specific Gravity	Density
	25°C (0.99707)			40°C (0.99224)		
0.0	1.0805	1.2305	1.2269	1.0770	1.2204	1.2109
20.0	1.0905	1.2591	1.2554	1.0874	1.2502	1.2405
30.0	1.0955	1.2734	1.2697	1.0924	1.2646	1.2548
40.0	1.1005	1.2877	1.2839	1.0973	1.2786	1.2687
50.0	1.1064	1.3046	1.3008	1.1026	1.2937	1.2837
60.0	1.1112	1.3182	1.3143	1.1084	1.3102	1.3000
70.0	1.1166	1.3338	1.3299	1.1138	1.3259	1.3156
	<u>60°C (0.98324)</u>			<u>80° (0.97183)</u>		
0.0	1.0758	1.2170	1.1966	1.0750	1.2147	1.1805
20.0	1.0859	1.2459	1.2250	1.0851	1.2437	1.2087
30.0	1.0907	1.2597	1.2386	1.0898	1.2571	1.2217
40.0	1.0956	1.2737	1.2537	1.0952	1.2726	1.2368
50.0	1.1013	1.2899	1.2682	1.1003	1.2871	1.2508
60.0	1.1069	1.3061	1.2842	1.1063	1.3043	1.2676
70.0	1.1122	1.3212	1.2990	1.1116	1.3195	1.2823

TABLE II

SPECIFIC GRAVITIES AND DENSITIES OF SOLUTIONS OF TYPE 347 STAINLESS STEEL IN
ORIGINALLY 4.850M H₂SO₄ AT VARIOUS TEMPERATURES

Concn of Steel, gm/liter	Observed Value	Specific Gravity	Density	Observed Value	Specific Gravity	Density
	25°C (0.99707)			40°C (0.99224)		
0.0	1.0967	1.2768	1.2731	1.0941	1.2693	1.2595
20.0	1.1077	1.3083	1.3045	1.1040	1.2977	1.2876
30.0	1.1127	1.3227	1.3188	1.1104	1.3160	1.3058
40.0	1.1170	1.3349	1.3310	1.1147	1.3273	1.3170
50.0	1.1220	1.3492	1.3452	1.1198	1.3429	1.3325
60.0	1.1270	1.3636	1.3596	1.1246	1.3567	1.3462
70.0	1.1320	1.3778	1.3738	1.1297	1.3713	1.3607
	<u>60°C (0.98324)</u>			<u>80°C (0.97183)</u>		
0.0	1.0921	1.2637	1.2425	1.0915	1.2619	1.2264
20.0	1.1022	1.2926	1.2709	1.1017	1.2911	1.2547
30.0	1.1079	1.3088	1.2869	1.1077	1.3083	1.2714
40.0	1.1134	1.3246	1.3024	1.1120	1.3206	1.2834
50.0	1.1178	1.3372	1.3148	1.1168	1.3343	1.2967
60.0	1.1225	1.3507	1.3281	1.1219	1.3489	1.3109
70.0	1.1274	1.3647	1.3418	1.1270	1.3636	1.3252

TABLE III

SPECIFIC GRAVITIES AND DENSITIES OF SOLUTIONS OF TYPE 347 STAINLESS STEEL IN
ORIGINALLY 5.927M H₂SO₄ AT VARIOUS TEMPERATURES

Concn of Steel, gm/liter	Observed Value	Specific Gravity	Density	Observed Value	Specific Gravity	Density
	25°C (0.99707)			40°C (0.99224)		
0.0	1.1168	1.3343	1.3304	1.1140	1.3263	1.3160
20.0	1.1265	1.3621	1.3581	1.1233	1.3528	1.3423
30.0	1.1312	1.3756	1.3716	1.1282	1.3669	1.3563
40.0	1.1358	1.3887	1.3846	1.1331	1.3810	1.3703
50.0	1.1407	1.4028	1.3987	1.1379	1.3946	1.3838
60.0	1.1458	1.4171	1.4129	1.1428	1.4086	1.3977
70.0	1.1507	1.4313	1.4271	1.1479	1.4232	1.4122
	<u>60°C (0.98324)</u>			<u>80°C (0.97183)</u>		
0.0	1.1119	1.3204	1.2983	1.1107	1.3169	1.2798
20.0	1.1213	1.3472	1.3246	1.1200	1.3436	1.3058
30.0	1.1261	1.3609	1.3381	1.1250	1.3578	1.3196
40.0	1.1307	1.3741	1.3511	1.1297	1.3712	1.3326
50.0	1.1358	1.3887	1.3654	1.1347	1.3857	1.3467
60.0	1.1408	1.4030	1.3795	1.1396	1.3996	1.3602
70.0	1.1455	1.4165	1.3928	1.1448	1.4143	1.3745

TABLE IV

SPECIFIC GRAVITIES AND DENSITIES OF SOLUTIONS OF TYPE 347 STAINLESS STEEL IN
ORIGINALLY 6.882M H₂SO₄ AT VARIOUS TEMPERATURES

Concn of Steel, gm/liter	Observed	Specific	Density	Observed	Specific	Density
	Value	Gravity		Value	Gravity	
	25°C (0.99707)			40°C (0.99224)		
0.0	1.1335	1.3821	1.3781	1.1303	1.3730	1.3623
20.0	1.1430	1.4093	1.4052	1.1405	1.4021	1.3912
30.0	1.1477	1.4229	1.4187	1.1450	1.4150	1.4040
40.0	1.1524	1.4361	1.4319	1.1497	1.4286	1.4175
50.0	1.1577	1.4513	1.4470	1.1548	1.4430	1.4318
60.0	1.1625	1.4651	1.4608	1.1597	1.4571	1.4458
70.0	1.1665	1.4763	1.4720	1.1632	1.4671	1.4557
	60°C (0.98324)			80°C (0.97183)		
0.0	1.1284	1.3676	1.3447	1.1277	1.3656	1.3271
20.0	1.1383	1.3960	1.3726	1.1374	1.3932	1.3540
30.0	1.1428	1.4088	1.3852	1.1419	1.4061	1.3665
40.0	1.1476	1.4227	1.3989	1.1473	1.4217	1.3817
50.0	1.1526	1.4368	1.4127	1.1518	1.4344	1.3940
60.0	1.1574	1.4502	1.4259	1.1567	1.4485	1.4077
70.0	1.1610	1.4608	1.4363	1.1599	1.4577	1.4166

appear in Tables I to IV, where the density of water at the temperature is given in parentheses next to the temperature figure.

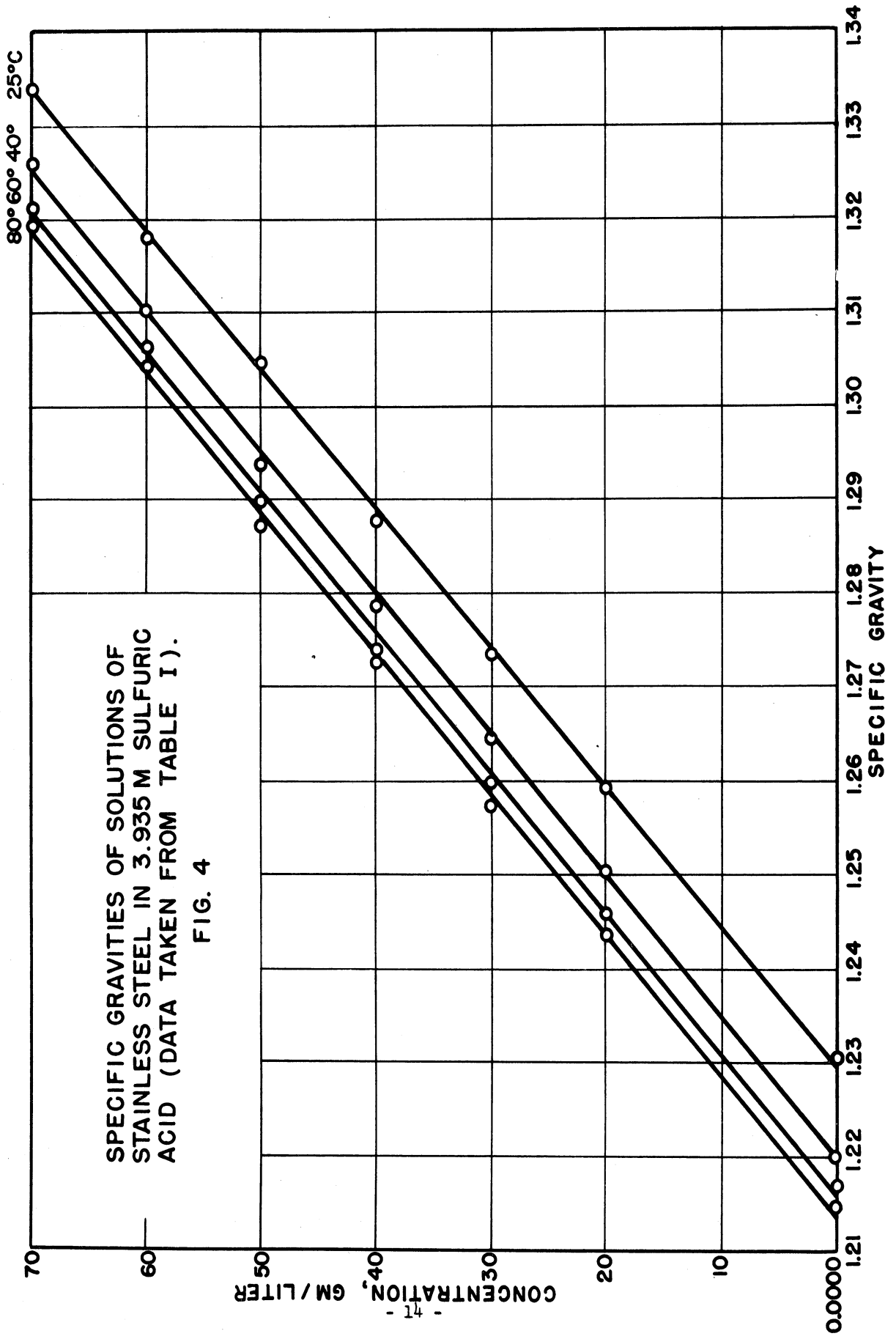
D. Data Obtained and Interpretation

Specific Gravity and Density Data. The basic data for the four original concentrations of sulfuric acid solution are given in Tables I to IV. These tables of data include three sets of values for each solution at each temperature. The "observed value" is the reading indicated on the specific gravity balance using the newly designed plummet. The "specific gravity" is the figure obtained by correcting the first value by the equation explained in Section C; it is the specific gravity of the solution referred to water as unity at each temperature. The absolute "density" is derived from the specific gravity by multiplying the specific gravity by the density of water at the temperature of measurement.

The specific gravities are plotted against concentration of dissolved steel in Figs. 4 to 7. Each figure pertains to a different original sulfuric acid molarity and has four sets of values, one for each temperature of measurement. The dimensions of the individual points on the graphs were originally drawn in accordance with the possible experimental error; thus, the marks consisted of a line whose horizontal dimension was ± 0.0007 specific gravity unit. This line has no appreciable thickness, since the weights of steel are accurate to four decimal places. The error on specific gravity is ± 0.0007 , since the sensitivity of the balance (0.0002) must be multiplied by the factor $10/3.493$, which was used in the conversion from the observed value to specific gravity. In the figures reproduced, the lines have been replaced by circles whose diameter was determined by their horizontal dimension of 0.0014 specific gravity unit. A straight line was drawn through the set of points for each temperature so as to represent the "best averaged values". The slopes of these lines are quite close to the slopes calculated by averaging the point-to-point slopes, discussed in the next section, which are given in Table V.

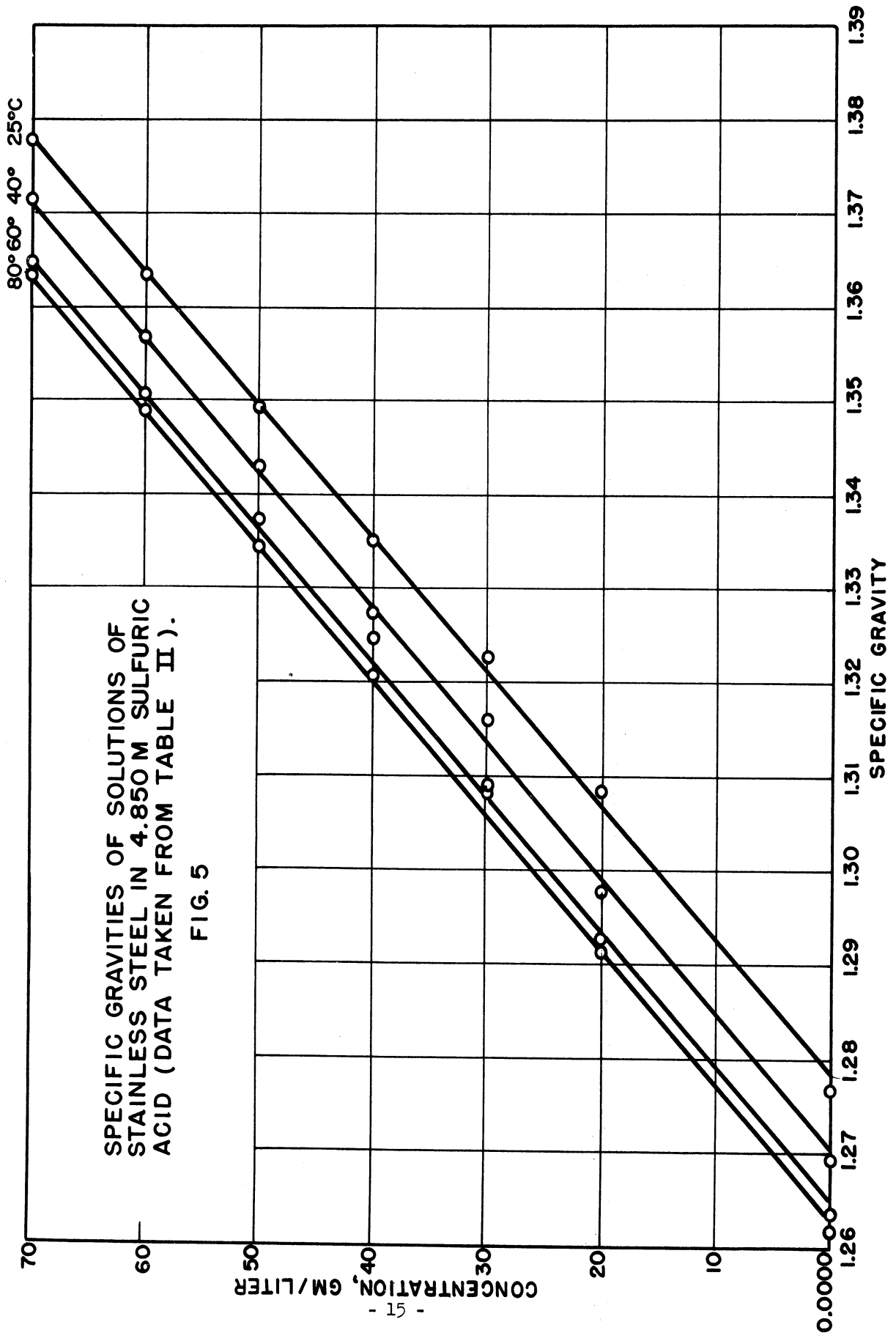
A fit of the lines to the data by means of a least-squares technic might be desirable, depending on the eventual accuracy of measurement desired. The possibility that a slightly different steel might be used which would result in a slight displacement of the data values was the deciding factor in not attempting a least-squares solution.

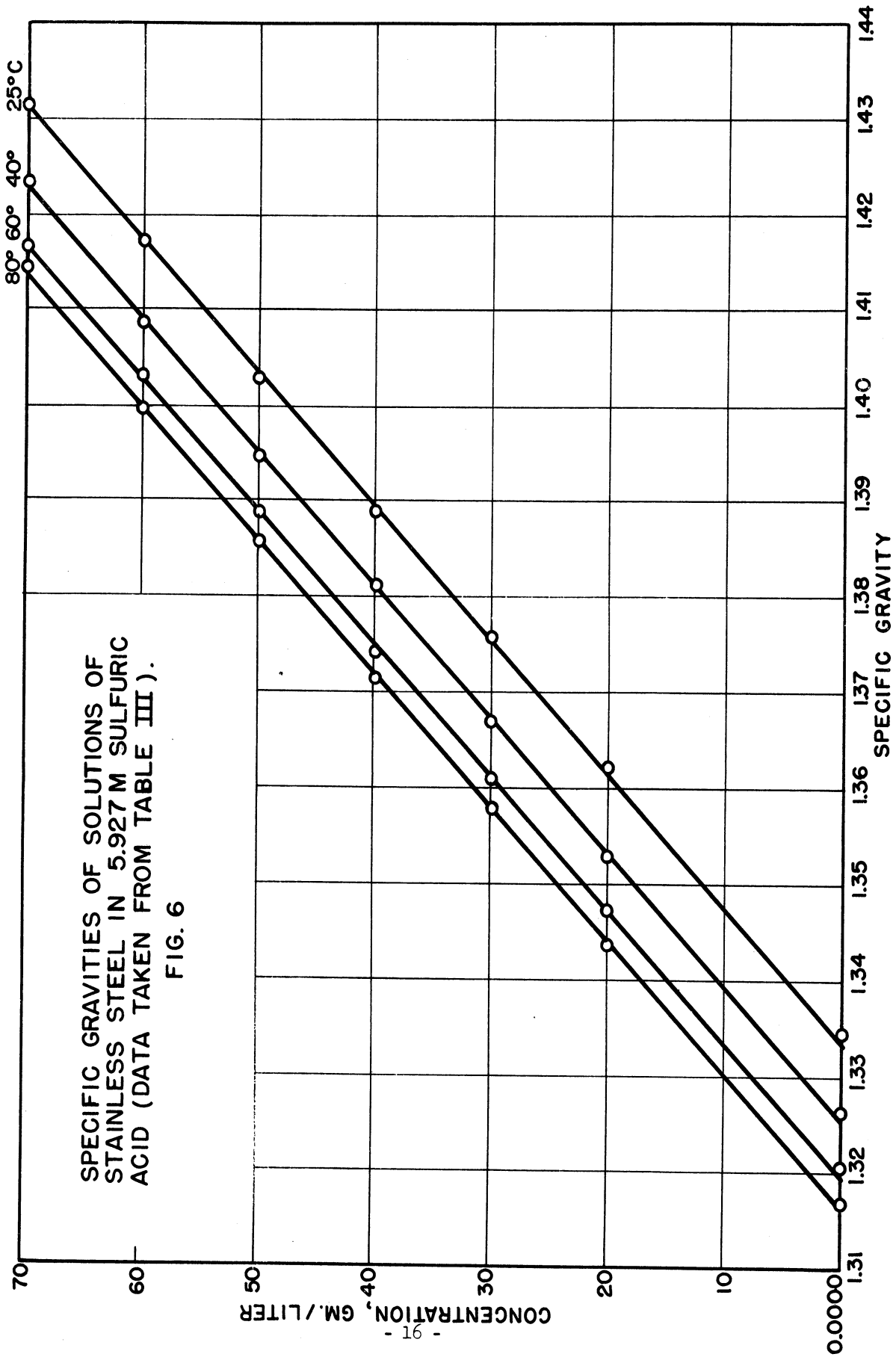
In view of the close fit of the experimental points to the average lines, no attempt was made to repeat any individual measurements, since the consistency of the data as a whole seemed to make individual discrepancies of minor importance. The cause of the deviation of the points for solution of 70 grams steel per liter in 7M acid is due to failure to retain all the material in solution, resulting in lower specific gravities than expected.



SPECIFIC GRAVITIES OF SOLUTIONS OF STAINLESS STEEL IN 3.935 M SULFURIC ACID (DATA TAKEN FROM TABLE I).

FIG. 4





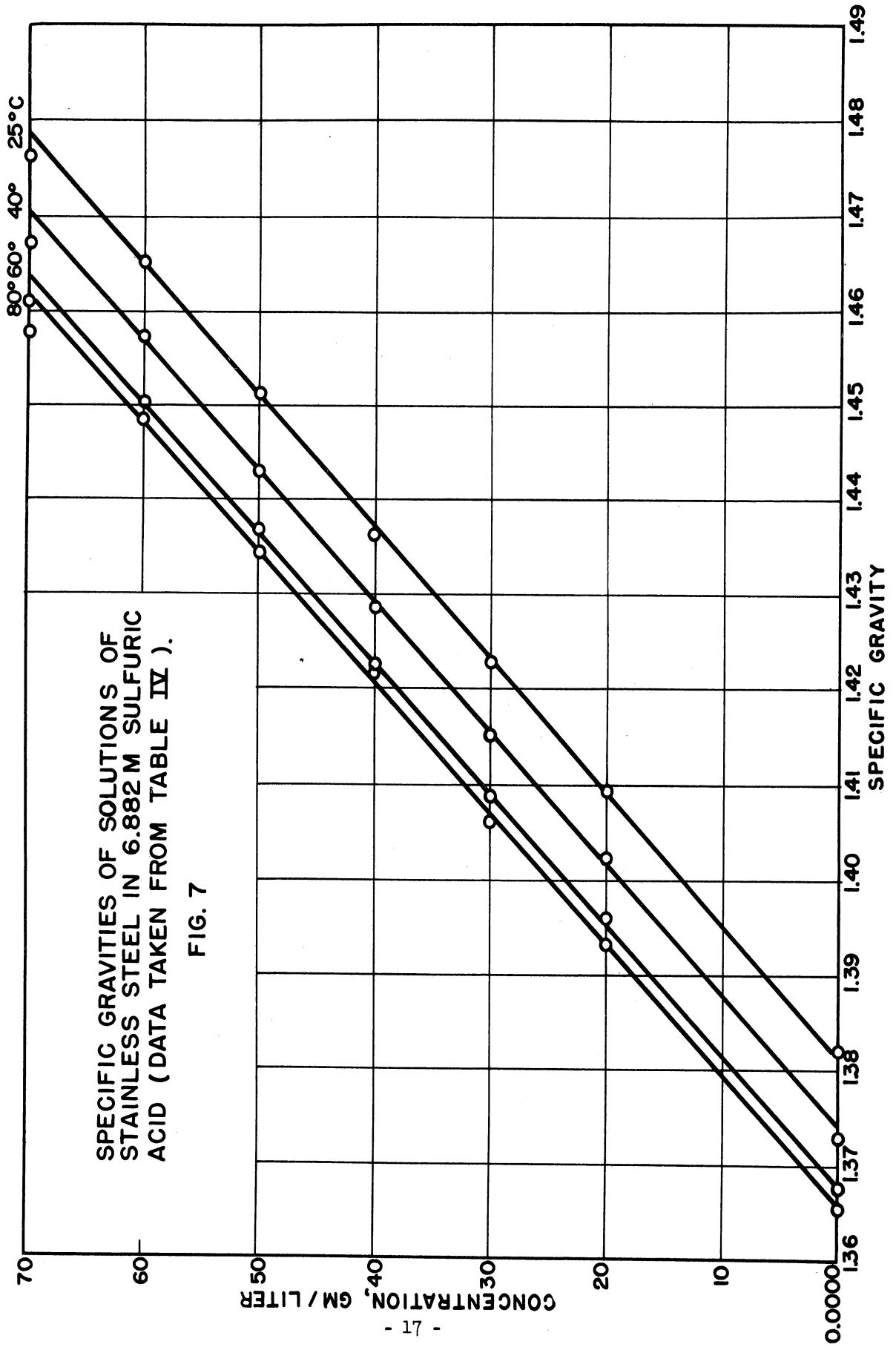


FIG. 7

TABLE V

SPECIFIC GRAVITIES, DENSITIES AND EQUATION CONSTANTS
FOR VARIOUS INITIAL ACID CONCENTRATIONS AND TEMPERATURES

Original H ₂ SO ₄ Concn	Temp. °C	Specific Gravity		Density	
		Acid	K σ	Acid	K σ
4M	25	1.2305	0.001452 ± .000023	1.2269	0.001447 ± .000024
	40	1.2204	0.001481 ± .000020	1.2109	0.001471 ± .000019
	60	1.2170	0.001453 ± .000030	1.1966	0.001432 ± .000022
	80	1.2147	0.001458 ± .000032	1.1805	0.001418 ± .000031
5M	25	1.2768	0.001483 ± .000056	1.2732	0.001477 ± .000056
	40	1.2693	0.001469 ± .000047	1.2595	0.001458 ± .000046
	60	1.2637	0.001472 ± .000034	1.2425	0.001448 ± .000034
	80	1.2619	0.001471 ± .000038	1.2264	0.001428 ± .000036
6M	25	1.3343	0.001377 ± .000011	1.3304	0.001375 ± .000011
	40	1.3263	0.001361 ± .000020	1.3160	0.001352 ± .000020
	60	1.3204	0.001358 ± .000016	1.2983	0.001335 ± .000016
	80	1.3169	0.001367 ± .000020	1.2798	0.001329 ± .000019
7M	25	1.3821	0.001367 ± .000011	1.3781	0.001362 ± .000015
	40	1.3730	0.001409 ± .000026	1.3623	0.001399 ± .000026
	60	1.3676	0.001386 ± .000019	1.3447	0.001363 ± .000018
	80	1.3656	0.001378 ± .000019	1.3271	0.001343 ± .000019

A small precipitate was noticed in the 60 grams per liter solution in 7M sulfuric acid. It was a light-colored curdy precipitate which settled out. Since the points plotted on the graph for this solution were consistent with the plotted lines, there was apparently no interference.

A larger amount of precipitate was present in the 70 grams per liter solution in 7M sulfuric acid. It was apparently of a nature similar to that mentioned in the previous paragraph, but the specific gravity points plotted for this solution show a wide variance from the plots of the rest of the points, indicating some sort of interference. Accordingly, the values for these points have not been used in the plotting of the average line nor included in the determination of the K values discussed in the next section.

Equations Derived from the Data. Since the graphs reveal straight-line functions between the specific gravity and the dissolved steel concentration for each particular original acid concentration and temperature, it is apparent that linear equations of the following form can be used to represent each set of experimental points:

$$\text{Sp. Gr. of Soln.} = \text{Sp. Gr. of Acid} + K (\text{Steel Concn}),$$

where

Sp. Gr. of Soln. = specific gravity of the solution at the temperature of measurement.

Sp. Gr. of Acid = specific gravity at the temperature of measurement of the original acid solution before any steel was dissolved in it.

K = a constant characteristic of the original acid concentration and of the temperature of measurement (see below).

Steel Concn = the concentration of dissolved type 347 stainless steel in the grams per liter of original acid solution.

Obviously, similar equations can be formulated for the densities of the stainless steel solutions:

$$D \text{ of Soln.} = D \text{ of Acid} + K' (\text{Steel Concn}).$$

In Table V are given the data necessary to use these equations, i.e., specific gravities and densities of the original sulfuric acid solutions at the various temperatures, and the corresponding K and K' values. Use of these values and the equations just given will facilitate calculations based on measurements made at intermediate temperatures, and steel acid concentrations.

The values of K given in Table V were obtained as follows: Equations of the type indicated were set up for each individual experimental point (original acid specific gravity and observed specific gravity of solution for each steel concentration at each temperature). The six values of K obtained for each steel concentration range (same original acid concentration at one temperature) were averaged and the standard deviation calculated in the usual way:

$$\sigma = \sqrt{\frac{\sum(x_i - \bar{x})^2}{n - 1}}$$

These values are given in the table. In general, the individual values were lower than the average K at the lower steel concentrations and higher at the higher steel concentrations. This may be a real effect due to the difference in temperature coefficient of expansion of the original acid solution as compared to that obtained by dissolving increasing amounts of steel, which results in lowering the actual acid concentration of the test solution. This effect may well vary in relative magnitude and be the cause of apparent differences in the K values of Table V.

No attempt was made to obtain values of K, which are obviously the slopes of the lines of Figs. 4 to 7, by a least-squares treatment of the data. The reason for this omission has been indicated.

Possible Precision of Control. The average ranges of specific gravity change in solutions containing from 0 to 70 grams of steel per liter are about 0.105, 0.102, 0.097 and 0.093 for 4M, 5M, 6M, and 7M sulfuric acid solutions. The measurements as made have a probable maximum error of ± 0.0007 specific gravity unit, which corresponds to a probable maximum error of 0.5 gram of steel per liter. Since a change in steel concentration of 0.07 grams per liter corresponds to 0.0001 specific gravity unit, use of a larger specific gravity plummet or of any specific gravity device readable to ± 0.0002 specific gravity unit would result in a probable maximum error of about 0.14 gram of steel per liter.

Information concerning a commercially available indicating density apparatus has been turned over to the resident representative of the American Cyanamid Company.

Coefficients of Cubical Expansion. To facilitate the use of the data as well as to indicate the magnitude of the effect of temperature on the volume of different steel-sulfuric acid solutions, the coefficients of cubical expansion (actually temperature coefficients of density change) were calculated and are given in Table VI. These coefficients represent the fractional change in density of a given solution (original sulfuric acid and dissolved type 347 stainless steel concentrations specified) per °C. The coefficients were calculated by the equation:

TABLE VI

TEMPERATURE COEFFICIENTS OF CUBICAL EXPANSION (FRACTIONAL CHANGE OF DENSITY)
OF SOLUTIONS OF VARIOUS SULFURIC ACID AND STEEL CONCENTRATIONS*

Concn of Steel gm/liter	Original Acid Concentration											
	4M			5M			6M			7M		
	25-40°C	40-60°C	60-80°C	25-40°C	40-60°C	60-80°C	25-40°C	40-60°C	60-80°C	25-40°C	40-60°C	60-80°C
0	8.8	5.9	6.8	7.2	6.8	6.5	7.3	6.7	7.2	7.7	6.5	6.6
20	8.0	6.3	6.7	8.7	6.5	6.4	7.8	6.6	7.1	6.7	6.7	6.8
30	7.9	6.5	6.9	6.6	7.3	6.1	7.5	6.8	6.9	6.9	6.7	6.8
40	7.9	5.9	6.8	7.1	5.6	7.4	6.9	7.1	6.9	6.7	6.6	6.2
50	8.8	6.1	6.9	6.3	6.7	6.9	7.1	6.7	6.9	7.0	6.7	6.7
60	7.3	6.1	6.5	6.6	6.8	6.5	7.2	6.6	7.0	6.9	6.9	6.4
70	7.2	6.3	6.5	6.4	7.0	6.2	7.0	6.9	6.6	7.4	6.7	6.9

*The values given are to be multiplied by 10^{-4} to obtain the actual coefficient. Temperatures given are in °C.

$$\text{Coeff.} = \frac{1}{0.5(D_{T_1} + D_{T_2})} \times \frac{D_{T_1} - D_{T_2}}{T_1 - T_2}$$

where

D_{T_1} = density of the solution at temperature T_1 , and

D_{T_2} = density of the solution at a higher temperature T_2 .

Since the density decreases with increasing temperature, the coefficients will be negative as defined. Three values of the coefficient are presented for each individual solution, representing the three adjacent temperature intervals, i.e., 25-40, 40-60, and 60-80°C. The variation of the coefficients over the temperature range may represent the conflicting tendencies due to acid depletion and salt increase previously mentioned.

An attempt was made to obtain from the literature data on the coefficient of cubical expansion for sulfuric acid solutions. From the data given in Lange's Handbook of Chemistry, for concentrated sulfuric acid in the temperature range of 0 to 30°C, the volume at any temperature, V_t , is related to the volume at 0°C as follows:

$$V_t = V_0 (1 + 0.5785 \times 10^{-3}t - 0.864 \times 10^{-6}t^2).$$

The corresponding equation for 10.9% sulfuric acid is:

$$V_t = V_0 (1 + 0.2835 \times 10^{-3}t + 2.580 \times 10^{-6}t^2).$$

The solutions used, 4 to 7M sulfuric acid, correspond to 31.3 to 48.7% sulfuric acid at 25°C. The coefficient of cubical expansion on a per °C basis can be computed as 5.8×10^{-4} for the concentrated (presumably 96%) sulfuric acid in the range around 25°C, as compared to the somewhat higher values which we found.

The coefficients given in Lange for three percentages of sulfuric acid at 20°C are (a) 1.4%, 2.3×10^{-4} ; (b) 5.4%, 3.1×10^{-4} ; and (c) 10.9%, 3.87×10^{-4} . Extrapolation to 31.3% and 48.7% gives 6.6 and 9.5. No claim is made for such extrapolation except to indicate that the data of Table VI are of the probable magnitude indicated.

Volume of the Plummet. As was indicated in the previous section, the value observed on the specific gravity balance was converted to an actual specific gravity value by an expression which involved division by the volume of the plummet. The volume of the plummet was determined to be 3.493 cc at 20°C by measurement of several solutions of known specific gravity. The question arose as to the possible effect of the change in this volume due to temperature. On the basis of dimensions of 1.0 cm external diameter, 6.0 cm external length and 0.1 cm wall thickness which were approximately those of the plummet, the increase in volume in going from 20° to 80° is about 0.0004 cc; the linear coefficient of expansion of Pyrex was taken as 3×10^{-6} cm per °C.

The negligible effect expected was verified by zeroing the specific gravity balance with water at 25° (balance set at 1.0000) and by then measuring the specific gravity of water at 60° and 80°. The values obtained checked the values given in the literature within the experimental error of reading the specific gravity balance.

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