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# COMPREHENSIVE ANALYSIS OF ALUMINUM ALLOYS BY ATOMIC ABSORPTION SPECTROMETRY

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## BY ATOMIC ABSORPTION SPECTROMETRY

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# A B S T R A C T

In preparation for atomic absorption analysis, all categories of wrought and foundry aluminum alloys can be completely dissolved with a single solvent, consisting of a mixture of hydrochloric, nitric and hydrofluoric acids, in an acid digestion bomb. In particular, high-silicon alloys (up to 20%), which have previously proven difficult, are readily dissolved. A statistical study of a large variety of certified commercial reference standards in comparison with synthesized standards has verified the validity of the method for the determination of copper, iron, magnesium, manganese, nickel, chromium and zinc. The coincidence of results for the two sets of standards, confirmed by correlation coefficients approaching unity, revealed that interference effects are minimal, and analysis of the certified standards in relation to synthesized standard calibrations showed that average errors are less than ±5% for single determinations. Replicate analyses of several reference standards showed that reproducibility of results is in the order of 1 to 2%. Consequently, an accuracy of the same order is potentially attainable through multiple determinations.

# COMPREHENSIVE ANALYSIS OF ALUMINUM ALLOYS
# BY ATOMIC ABSORPTION SPECTROMETRY

### by K.I. McRae and C.A. Waggoner

## Introduction:

In recent years, there have been several composite schemes proposed for the determination of the principal constituents of aluminum alloys by atomic absorption spectrometry (1, 2, 3, 4, 5). Several different methods of sample dissolution and preparation have been reported in the literature. Wilson (1), for instance, suggested treatment with 10% sodium hydroxide followed by concentrated nitric acid for the determination of Cu, Mg, Ag and Zn. Mansell et al (2) proposed a 5:1 mixture of HCl and $HNO_3$, respectively, with the removal of undissolved residue by filtration. Dill (3) used concentrated hydrochloric acid followed by treatment with hydrogen peroxide, while Bell (4) used similar solvents but recommended the removal of residual silicon by volatilization with HF to prevent losses of Cu and Mg in the form of insoluble silicides, if silicon was suspected to exceed 1%.

All of the above techniques for aluminum alloy dissolution have shortcomings. Foremost is the fact that no single solvent treatment is sufficiently potent to completely dissolve all of the important alloying elements that are potentially determinable by atomic absorption spectrometry. Most of the elements are readily soluble in hydrochloric acid or nitric acid, but silicon, a relatively frequent constituent, is insoluble in these and other common solvents. A second disadvantage of these methods is that they require a considerable amount of time and effort in the preparation of the sample. Consequently, a single solvent mixture that could be applied universally would provide a substantial improvement in adapting the atomic absorption method to aluminum alloy analysis.

A technique has been developed by DREP by which all aluminum alloys, especially those with relatively high silicon content, may be completely dissolved. The procedure involves treatment of the alloy in an acid digestion bomb with a mixture of hydrochloric, nitric and hydrofluoric acids as the solvent (6). By this method, aluminum alloys containing as high as 20% silicon have been dissolved without loss (7).

Further to the above work, it remained to be demonstrated that the procedure would be applicable to other significant constituents of aluminum alloys, including copper, iron, magnesium, manganese, nickel, chromium and zinc. A review of the literature revealed that, with few exceptions, interference effects in the atomic absorption analysis of aluminum alloys are apparently minimal. The feasibility of applying the technique more generally was therefore investigated, using as a basis of validation a statistical evaluation of certified reference standards relative to primary synthesized standards.

## Experimental:

### A. Dissolution of Alloy:

For the purpose of this work, an acid digestion bomb of novel design (6) and a solvent consisting of a 3:1:1 mixture of hydrochloric, nitric and hydrofluoric acids, respectively, was used in order to completely dissolve all constituents that normally comprise aluminum alloys. The digestion bomb consists of an outer casing of stainless steel, similar in design to that used by the Parr Instrument Co. (Model 4745), and an inner close-fitting Teflon reaction vessel. The modified features of the reaction vessel are discussed in Reference 7.

Samples and standards are prepared by transferring 200 mg. quantities into a sample cup, also constructed of Teflon, and placing it in the bottom of the reaction vessel, to which has been delivered 8.0 ml of a 50% v/v acid mixture (3:1:1 mixture of HCl, $HNO_3$ and HF). The cell and outer casing are then carefully reassembled, ensuring that the acid mix and the alloy do not come into contact. After assembly, the digestion bomb is inverted and shaken vigorously, and then allowed to stand in a cool water bath for a period of approximately 30 minutes. The bomb is then carefully opened and the contents, including washings, are transferred to a graduated plastic (e.g. polypropylene) cylinder and diluted to a volume of 80.0 ml with distilled water. This yields a sample solution for analysis in which the final alloy concentration is 2.5 mg/ml in a 5% v/v acid mixture.

B. Instrumentation:

All determinations were carried out with a Jarrell-Ash Model 82-800 atomic absorption spectrophotometer. This instrument had been modified somewhat by replacement of the original burner assembly with a Varian-Techtron Model AB-50 unit, fitted with a variable flow nebulizer for controlling sample aspiration rate. In order to improve the reproducibility of the various flame conditions used, oxidant and fuel flow rates are measured by means of two Matheson Model 621-PBX high-accuracy rotameters. Instrumental parameters for the various elements are listed in Table 1.

C. Standards for Method Validation:

Two series of standards were prepared as a basis for validating the precision and accuracy of the method. For the preparation of primary calibration curves, individual solution standards containing only a single alloying element and aluminum were synthesized. Since no interfering constituents were present in these standards the corresponding calibration plots approached the ideal. To determine whether interelement or other anomalous effects might occur, commercial certified standards - supplied by the Aluminum Co. of Canada, the U.S. Bureau of Standards and the British Bureau of Analysed Samples - representative of all types of aluminum alloys were dissolved by the acid mixture in the digestion bomb, and were then evaluated analytically against the primary curves. Any deviations from the curves would thus be cause for suspecting possible interference effects, and could be specifically examined.

Stock solutions were prepared as follows from high-purity metals supplied by Spex Industries, Metuchen, N.Y.

(1) Fe, Mg, Mn, Zn, Cr - 1 gram of each metal dissolved separately in 20 ml of 50% v/v HCl and made up to 100 ml (10 mg/ml).

(2) Cu, Ni - 1 gram of each metal dissolved separately in 20 ml of 50% v/v $HNO_3$ and made up to 100 ml (10 mg/ml).

(3) Al - 2.5 g dissolved in 50% v/v HCl and made up to 100 ml (25 mg/ml).

Individual standards were then synthesized from the above stock solutions in the following manner: to 100-ml plastic graduated cylinders, 10 ml of aluminum

TABLE 1

## INSTRUMENT PARAMETERS

| Element | Cu | Fe | Mg | Zn | Mn | Ni | Cr | Ti | Si[a] |
|---|---|---|---|---|---|---|---|---|---|
| Line, nm | 324.7 | 372.0[b] | 285.2 | 213.9 | 279.5 | 341.5[b] | 375.9 | 364.3 | 251.6 |
| Oxidant | air | air | $N_2O$ | air | air | air | $N_2O$ | $N_2O$ | $N_2O$ |
| Lamp current, mA | 4 | 10 | 4 | 6 | 10 | 10 | 10 | 10 | 12 |
| Height of beam above flame, cm | 1.4 | 0.8 | 1.2 | 0.8 | 0.9 | 1.1 | 0.8 | 0.9 | |
| * Oxidant | 8.5 | 8.5 | 6.0 | 8.5 | 9.0 | 9.0 | 6.0 | – | – |
| * Acetylene | 3.0 | 2.5 | 5.0 | 2.5 | 3.0 | 3.0 | 5.2 | – | – |
| ** Auxiliary oxidant | 0 | 0 | 2.0 | 0 | 5.0 | 2.0 | 0 | – | – |
| Flame condition (oxidizing or reducing) | oxid. | oxid. | red. | oxid. | oxid. | oxid. | red.[c] | v.red. | red.[c] |

* Gauge setting on Matheson rotameter (Model 621PBX)

** Gauge setting on AA instrument

(a) Data from reference 7.

(b) Though weaker absorbing lines, these wavelengths were selected because of greater stability and lower background.

(c) "red feather" height $\simeq$ 2 cm.

stock, 10 ml of acid mix and appropriate volumes of analyte stock were added which, on making up to final volume, provided a total metal concentration of 2.5 mg/ml in a 5% v/v acid solution.

## D. Choice of Spectral Lines

The common spectral lines of the aluminum alloy elements were evaluated to determine what lines could be most suitably applied. Owing to the high atomic absorption sensitivity of some of the elements and, in addition, a limited choice of suitable lines, it was necessary to employ slightly different procedures for these elements. For example, adequate sensitivity was achieved for copper, magnesium and zinc by using a crossed burner which, at the same time, extended the analytical range to more practical limits. Further, to determine the high concentrations of copper, magnesium and zinc that occur in some alloys, five-fold dilutions of the original solution sample became necessary. The most sensitive lines of iron and nickel - 248.3 nm and 232.0 nm respectively - were found to lack stability, and lines 372.0 nm and 341.5 nm were selected as preferred alternatives. The conditions finally selected for the analyte determinations are outlined in Table II.

## Results and Discussion

### Calibration:

Preliminary evaluation of analytical calibrations for the individual analytes, based on a separate series of binary solution standards containing only the analyte and the aluminum base metal, showed that absorbance/concentration correlations were consistent and linear in all cases. With the essential validity of the procedure thus established, a large number of certified standards were then prepared and analyzed for comparison against the primary calibrations. In view of the wide variations in the alloy compositions of these standards, it was expected that any significant deviations in results would be indicative of possible interference effects. To ensure a precise comparison of all the data, graphic plots and statistical evaluations were made with a computer and plotter, based on a Fortran program [8] designed for linear regression correlation by the least-squares method. Separate correlations of the analytical data obtained for the synthesized standards and the certified standards established that they agreed closely (Table III). Computer graphs of the combined data were subsequently made, and the coincidence of these plots is illustrated in Figures 1 to 9. The corresponding statistical data are shown in Table IV. The graphs reveal little scatter in the results for the certified standards, which implies that interelement effects are minimal and that errors from this source are not, therefore, of practical significance. The absence of other factors that might cause error is further implied by the low intercept values for the graphs, shown in Table III. Excellent correlations between the two sets of standards are evident from the computer-calculated coefficients shown in Table IV. This verifies the coincidence of the calibration plots for the primary and certified standards, and thus proves the validity of the analytical method.

### Sensitivity:

The sensitivity thresholds for each of the graphs are indicated by the slope values shown in Table IV, and the calculated limits in percent and ppm

TABLE II

ANALYTICAL CONDITIONS

| Line (nm) | Conc. Range (%) | Burner Position | Alloy Conc.(mg/ml) |
|-----------|-----------------|-----------------|---------------------|
| Cu 324.7 | 0-7 | transverse | 2.5 |
| Cu 324.7 | 5-10 | transverse | 0.5 |
| Fe 372.0 | 0-2 | in-line | 2.5 |
| Mg 285.2 | 0-2 | transverse | 2.5 |
| Mg 285.2 | 2-10 | transverse | 0.5 |
| Mn 279.5 | 0-2 | transverse | 2.5 |
| Ni 341.5 | 0-2 | in-line | 2.5 |
| Cr 357.9 | 0-0.6 | in-line | 2.5 |
| Zn 213.9 | 0-2 | transverse | 2.5 |
| Zn 213.9 | 2-10 | transverse | 0.5 |

## TABLE III

### STATISTICAL COMPARISON OF ANALYTICAL DATA OBTAINED FOR SYNTHESIZED AND CERTIFIED REFERENCE STANDARDS

| | Synthesized Standards | Certified Standards |
|---|---|---|
| **COPPER** | $m = .086929$<br>$b = -.002507$<br>$r = .999935$ | $m = .088115$<br>$b = -.00479$<br>$r = .998055$ |
| **IRON** | $m = .189721$<br>$b = .002940$<br>$r = .999740$ | $m = .189362$<br>$b = .002932$<br>$r = .999579$ |
| **ZINC - 0-2%** | $m = .244567$<br>$b = -.001058$<br>$r = .999989$ | $m = .244817$<br>$b = -.000845$<br>$r = .999984$ |
| **ZINC - 0-10%** | $m = .044640$<br>$b = -.006500$<br>$r = .999870$ | $m = .043909$<br>$b = -.001837$<br>$r = .999883$ |
| **MANGANESE** | $m = .064625$<br>$b = -.001302$<br>$r = .999450$ | $m = .065578$<br>$b = -.001604$<br>$r = .999590$ |
| **NICKEL** | $m = .275770$<br>$b = -.002597$<br>$r = .999944$ | $m = .273838$<br>$b = .001289$<br>$r = .999932$ |
| **CHROMIUM** | $m = .876658$<br>$b = .000148$<br>$r = .999931$ | $m = .880294$<br>$b = .000009$<br>$r = .999880$ |
| **MAGNESIUM 0-2%** | $m = .317554$<br>$b = -.001410$<br>$r = .999960$ | $m = .325779$<br>$b = -.001150$<br>$r = .998320$ |
| **MAGNESIUM 0-10%** | $m = .034550$<br>$b = -.004300$<br>$r = .999730$ | $m = .032489$<br>$b = .001890$<br>$r = .999830$ |

$m$ = slope of linear regression curve.     $b$ = y-intercept

$r$ = correlation coefficient

## TABLE IV

### REGRESSION ANALYSIS RESULTS

| Analyte | Correlation Coefficient | Slope | Intercept | Sensitivity (%) | Sensitivity (ppm) |
|---|---|---|---|---|---|
| Cu | .99989 | .08735 | -.003172 | .087 | 2.2 |
| Fe | .99976 | .1897 | .002807 | .008 | 0.20 |
| Mg (0-2%) | .99904 | .3206 | -.000260 | .014 | 0.36 |
| Mg (0-10%) | .99903 | .03328 | -.000329 | .14 | 3.55 |
| Cr | .99991 | .8781 | -.000142 | .005 | 0.12 |
| Ni | .99992 | .2745 | -.000067 | .016 | 0.40 |
| Mn | .99951 | .0644 | -.001211 | .087 | 2.18 |
| Zn (0-2%) | .99998 | .2447 | -.000931 | .022 | 0.55 |
| Zn (0-10%) | .99988 | .04414 | -.002855 | .16 | 4.1 |

(based on the conventional definition of sensitivity: i.e., concentration that produces 1% absorption) are given in the right-hand column. Ultimate limits for the method apply only for iron, nickel and chromium, which were analyzed under standard conditions (with the burner in-line) designed to give near maximum sensitivity. The remaining elements - copper, magnesium, manganese and zinc - were determined under conditions intended to reduce sensitivity, for example, by rotating the burner and/or diluting the sample, in order to extend the upper limits of the working range. Estimated ultimate sensitivities for these elements, based on a sample concentration of 2.5 mg/ml, are approximately one-tenth the values obtained using the crossed burner, as follows: copper - .009% (0.2 ppm); magnesium - .0015% (0.04 ppm); manganese - .009% (0.2 ppm); zinc - .002% (.05 ppm). The determination of titanium was also attempted but the 364.3 nm line commonly employed was not sufficiently sensitive for the smallest amounts specified in some aluminum alloys ($\simeq 0.01\%$). It is likely that titanium could be determined by the present method, but a more concentrated sample (e.g., 10 mg/ml) would be necessary.

### Precision:

The reproducibilities of the various elemental determinations, taking into consideration the entire analytical procedure, were established by analyzing a series of certified standards that, as nearly as possible, spanned the concentration ranges of the particular constituents. Six replicates of each standard were prepared and the alloying elements were determined relative to the synthesized standard calibration graphs. Measurements were made in either a 3-second or 10-second integration mode, depending on the stability of the signal. The mean and the standard deviation of the six determinations were then calculated for each element. The results are presented in Table V. With only a few exceptions, relative standard deviations are less than 2%, which indicates a high order of precision. Only in two instances do the values reach approximately 5%. In one case - 0.05% zinc - a larger error is to be expected because of the low concentration level. In the other case - 0.73% manganese - it is not certain whether the error is in the method or the certified result.

### Accuracy:

The close coincidence of data obtained for the synthesized standards and the certified standards, illustrated in Figures 1 to 9 and Table III, suggest that the method is capable of producing very accurate results. A measure of the accuracy is indicated in Table VI, which shows the degree of reproduction of the certified standard results for single determinations when compared against calibrations based solely on the synthesized standards. Average errors are seen to be less than $\pm 4\%$ for all analyte elements except zinc in the 0 to 10% range, for which the error averages $\pm 5.4\%$. Extreme discrepancies in two of the results for nickel were omitted, as the concentrations approached the sensitivity limits and the errors were considered not representative of the determinations as a whole. In general, these results are indicative of the accuracy that can be achieved by the method on single determinations.

## TABLE V

### PRECISION DATA

COPPER

| Standard | Content (%) | Mean Result (%) | Standard Deviation | Relative Standard Deviation (%) |
|---|---|---|---|---|
| 38S | 0.86 | 0.87 | .008 | 0.9 |
| 45243 | 2.4 | 2.40 | .019 | 0.8 |
| 85b | 3.99 | 4.00 | .042 | 1.0 |
| 216/1 | 4.42 | 4.37 | .014 | 0.3 |

IRON

| Standard | Content (%) | Mean Result (%) | Standard Deviation | Relative Standard Deviation (%) |
|---|---|---|---|---|
| 38S | .37 | .37 | .009 | 2.6 |
| B54S | .19 | .21 | .004 | 2.0 |
| 45243 | 1.0 | 1.04 | .014 | 1.4 |
| 216/1 | .40 | .42 | .002 | 0.5 |
| 29460-2 | 1.3 | 1.28 | .009 | 0.7 |
| 85b | .24 | .24 | .004 | 1.9 |

ZINC

| Standard | Content (%) | Mean Result (%) | Standard Deviation | Relative Standard Deviation (%) |
|---|---|---|---|---|
| 29460-2 | .04 | .033 | .0016 | 4.9 |
| 45243 | .050 | .050 | .0005 | 1.0 |
| 38S | .062 | .057 | .0004 | 0.7 |
| 85b | .030 | .026 | .0004 | 1.5 |
| B54S | .034 | .032 | .0004 | 1.6 |
| 216/1 | .11 | .099 | .0017 | 1.7 |

## TABLE V (Cont'd)

### MANGANESE

| Standard | Content(%) | Mean Result(%) | Standard Deviation | Relative Standard Deviation(%) |
|---|---|---|---|---|
| 45243 | .50 | .51 | .010 | 2.0 |
| B54S | .32 | .33 | .007 | 2.1 |
| 216/1 | .73 | .81 | .043 | 5.3 |
| 85b | .61 | .64 | .020 | 3.1 |
| 38S | .055 | .051 | .001 | 2.0 |
| 29460-2 | .03 | .028 | .0005 | 1.8 |

### CHROMIUM

| Standard | Content(%) | Mean Result(%) | Standard Deviation | Relative Standard Deviation(%) |
|---|---|---|---|---|
| 45243 | .03 | .029 | .0008 | 2.8 |
| 29460-2 | .04 | .049 | .0008 | 1.6 |
| B54S | .027 | .028 | .0011 | 3.9 |
| 38S | .038 | .037 | .0011 | 3.0 |
| 85b | .24 | .231 | .0018 | 0.8 |

### NICKEL

| Standard | Content(%) | Mean Result(%) | Standard Deviation | Relative Standard Deviation(%) |
|---|---|---|---|---|
| 38S | 0.84 | 0.89 | .004 | 0.5 |
| 181/1 | 2.04 | 1.95 | .014 | 0.7 |
| 87 | .59 | .60 | .006 | 1.1 |
| B54S | .029 | .040 | .0008 | 2.0 |
| 45243 | .05 | .057 | .0008 | 1.4 |
| 216/1 | .06 | .061 | .0007 | 1.1 |
| 85b | .084 | .098 | .0007 | 0.7 |
| 29460-2 | .03 | .035 | .0006 | 1.7 |

### MAGNESIUM

| Standard | Content(%) | Mean Result(%) | Standard Deviation | Relative Standard Deviation(%) |
|---|---|---|---|---|
| 38S | 1.13 | 1.11 | .004 | 0.3 |
| 181/1 | 1.42 | 1.41 | .012 | 0.8 |
| 216/1 | .74 | .74 | .011 | 1.4 |
| 87 | .39 | .39 | .004 | 1.1 |
| 29460-2 | .20 | .21 | .003 | 1.3 |

## TABLE VI

ANALYTICAL RESULTS FOR CERTIFIED REFERENCE
STANDARDS BASED ON SYNTHESIZED STANDARD CALIBRATIONS

COPPER

| Standard | Certified Content (%) | Result(%) | Relative Error (%) |
|----------|-----------------------|-----------|--------------------|
| 601      | 4.38                  | 4.40      | 0.46               |
| 75S      | 1.58                  | 1.54      | 2.53               |
| 16S      | 1.99                  | 1.97      | 1.00               |
| 183      | 2.99                  | 2.98      | 0.33               |
| 46820    | 0.90                  | 0.88      | 2.22               |
| A-111    | 1.64                  | 1.63      | 0.61               |
| 38S      | 0.86                  | 0.88      | 2.33               |
| 603      | 0.29                  | 0.28      | 3.45               |
| 45243    | 2.40                  | 2.42      | -                  |
| 85b      | 3.99                  | 4.05      | 1.50               |
| 216/1    | 4.42                  | 4.46      | 0.90               |
|          |                       | Average   | 1.39               |

IRON

| Standard | Certified Content (%) | Result(%) | Relative Error (%) |
|----------|-----------------------|-----------|--------------------|
| 38S      | 0.37                  | 0.36      | 2.70               |
| B54S     | 0.19                  | 0.20      | 5.26               |
| 45243    | 1.0                   | 1.01      | 1.0                |
| 216/1    | 0.40                  | 0.40      | -                  |
| 29460-2  | 1.3                   | 1.29      | 0.78               |
| 85b      | 0.24                  | 0.23      | 4.17               |
| 112      | 0.16                  | 0.16      | -                  |
| 601      | 0.52                  | 0.52      | -                  |
| 183      | 0.80                  | 0.80      | -                  |
| 86c      | 0.90                  | 0.91      | 1.11               |
| 492      | 0.63                  | 0.61      | 3.17               |
|          |                       | Average   | 1.65               |

## TABLE VI (Cont'd)

ZINC - 0-2%

| Standard | Certified Content (%) | Result (%) | Relative Error (%) |
|----------|-----------------------|------------|--------------------|
| 216/1 | .11 | .11 | -- |
| 44220W | .70 | .71 | 1.4 |
| 72s | .96 | .96 | -- |
| Z55S | 2.09 | 2.09 | - |
| 86c | 1.50 | 1.50 | - |
| 87 | 0.077 | .078 | 1.3 |
| | | Average | 0.45 |

ZINC - 0-10%

| Standard | Certified Content (%) | Result (%) | Relative Error (%) |
|----------|-----------------------|------------|--------------------|
| 86c | 1.50 | 1.62 | 8.0 |
| Z55S | 2.09 | 2.14 | 2.4 |
| 300 | 5.98 | 5.92 | 1.0 |
| 75s | 7.55 | 7.58 | 0.4 |
| 72S | .96 | 1.06 | 10.4 |
| 44220W | .70 | 0.77 | 10.0 |
| | | Average | 5.37 |

MANGANESE

| Standard | Certified Content (%) | Result (%) | Relative Error (%) |
|----------|-----------------------|------------|--------------------|
| 44220W | .40 | .39 | 2.5 |
| 45243 | .50 | .50 | - |
| 112 | 1.61 | 1.63 | 1.2 |
| 183 | 1.22 | 1.23 | .8 |
| 216/1 | .73 | .73 | - |
| 300 | .41 | .44 | 7.3 |
| 601 | .81 | .82 | 1.2 |
| 85a | .66 | .67 | 1.5 |
| 87 | .30 | .28 | 6.7 |
| | | Average | 2.36 |

TABLE VI (Cont'd)

NICKEL

| Standard | Certified Content (%) | Result(%) | Relative Error (%) |
|---|---|---|---|
| 87 | 0.59 | 0.59 | - |
| 38S | 0.84 | 0.84 | - |
| 604 | 2.00 | 2.00 | - |
| A-111 | 1.01 | 1.01 | - |
| 49220 | 1.2 | 1.21 | - |
| 1122 | 0.97 | 0.99 | 2.06 |
| 85a | 0.41 | 0.42 | 2.44 |
| 44220W | 0.15 | 0.16 | 6.67 |
| 45243 | 0.05 | 0.064 | 28.0 * |
| 85b | 0.084 | 0.100 | 19.0 * |

* omitted in average                Average  1.40

CHROMIUM

| Standard | Certified Content (%) | Result(%) | Relative Error (%) |
|---|---|---|---|
| 87 | .17 | .17 | - |
| 112 | .040 | .043 | 7.5 |
| 38s | .038 | .037 | 2.6 |
| 492 | .030 | .029 | 3.3 |
| Z55S | .23 | .23 | - |
| 300 | .15 | .15 | - |
| 601 | .020 | .018 | 10.0 |
| 57S | .30 | .30 | - |

Average  2.93

TABLE VI (Cont'd)

MAGNESIUM

| Standard | Certified Content (%) | Result(%) | Relative Error (%) |
|----------|----------------------|-----------|-------------------|
| 112 | .033 | .031 | 6.1 |
| 16S | .08 | .090 | 12.5 |
| A-111 | .10 | 0.11 | 10.0 |
| 183 | .25 | 0.21 | 16.0 |
| 601 | .39 | 0.39 | |
| 216/1 | .74 | 0.77 | 4.0 |
| 603 | 1.01 | 0.97 | 4.0 |
| 38S | 1.13 | 1.19 | 5.3 |
| 46820 | 1.2 | 1.24 | - |
| 85b | 1.49 | 1.51 | 1.3 |
| B54S | 4.40 | 4.35 | 1.1 |
| 262 | 10.57 | 10.02 | 5.2 |
| 263/1 | 4.92 | 4.87 | 1.0 |
| 57S | 2.61 | 2.61 | - |
| 350 | 10.30 | 9.94 | 3.5 |
| 75S | 2.60 | 2.58 | 0.8 |
| 300 | 2.76 | 2.76 | - |
| | | Average | 4.16 |

## Conclusions

An atomic absorption method previously developed to determine silicon in aluminum alloys has been shown to be applicable to all other common alloying constituents, including copper, chromium, iron, magnesium, manganese, nickel and zinc. The validity of the method, particularly with regard to the quantitative aspect of a novel acid digestion bomb dissolution technique and the possibility of interelement interference effects, was proven by a comparative statistical study of certified reference standards relative to synthesized primary standards. Excellent correlations and results of high precision and accuracy were demonstrated, thus confirming that potential errors are of minor significance. The method is unique in that, unlike existing methods, it is universally applicable without restriction to all categories of industrial aluminum alloys.

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        of Tabulated Data",  DREP Laboratory Note 73-6, May 1973.

## COINCIDENCE PLOTS OF CERTIFIED
REFERENCE STANDARDS VS. SYNTHESIZED STANDARDS

| Figure No. | Element | Spectral Line (nm) |
|---|---|---|
| 1 | Copper | 324.7 |
| 2 | Chromium | 375.9 |
| 3 | Iron | 372.0 |
| 4 | Magnesium (0-2%) | 285.2 |
| 5 | Magnesium (0-10%) | 285.2 |
| 6 | Manganese | 279.5 |
| 7 | Nickel | 341.5 |
| 8 | Zinc (0-2%) | 213.9 |
| 9 | Zinc (0-10%) | 213.9 |

Legend:   X - Synthesized Standards

◈ - Certified Reference Standards

Note:   Concentration is presented as the percent of alloying element in 2.5 mg/ml of the alloy in solution or of fractional concentrations employed.

For example:   1% alloying element   ≃.025 mg/ml (25 ppm) in 2.5 mg/ml solution.

or   ≃.005 mg/ml (5 ppm) in 0.5 mg/ml solution.
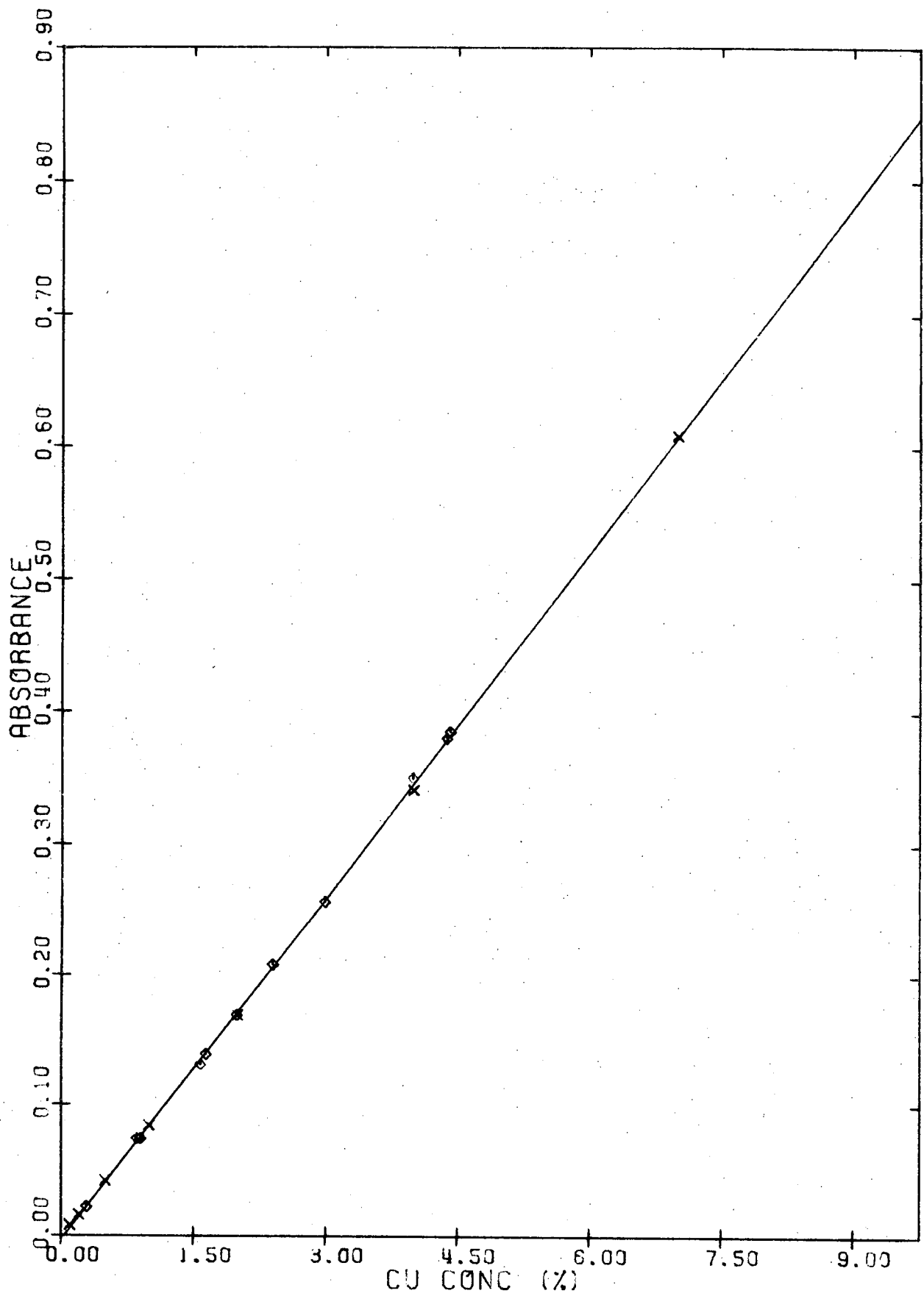
Figure 1.
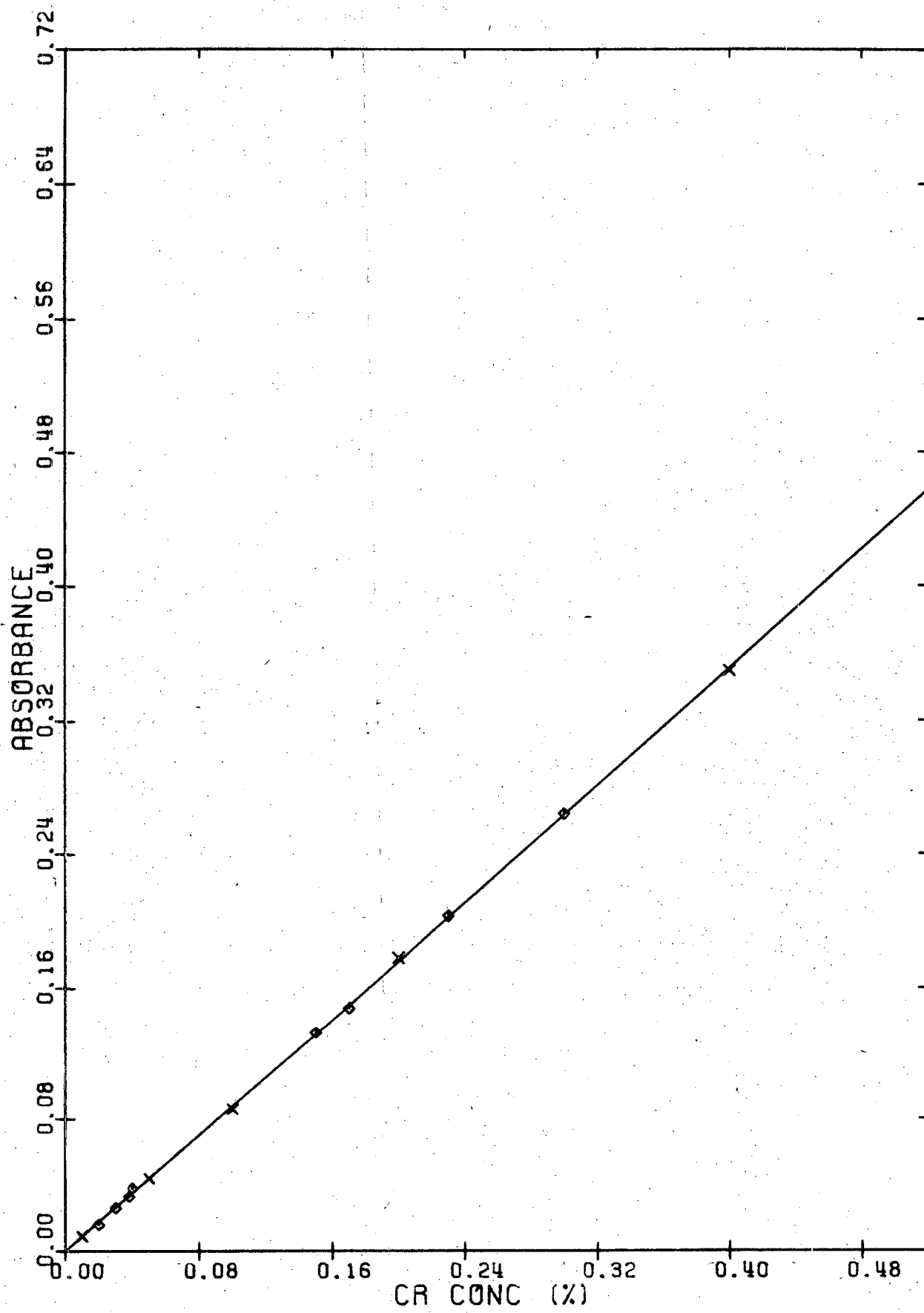
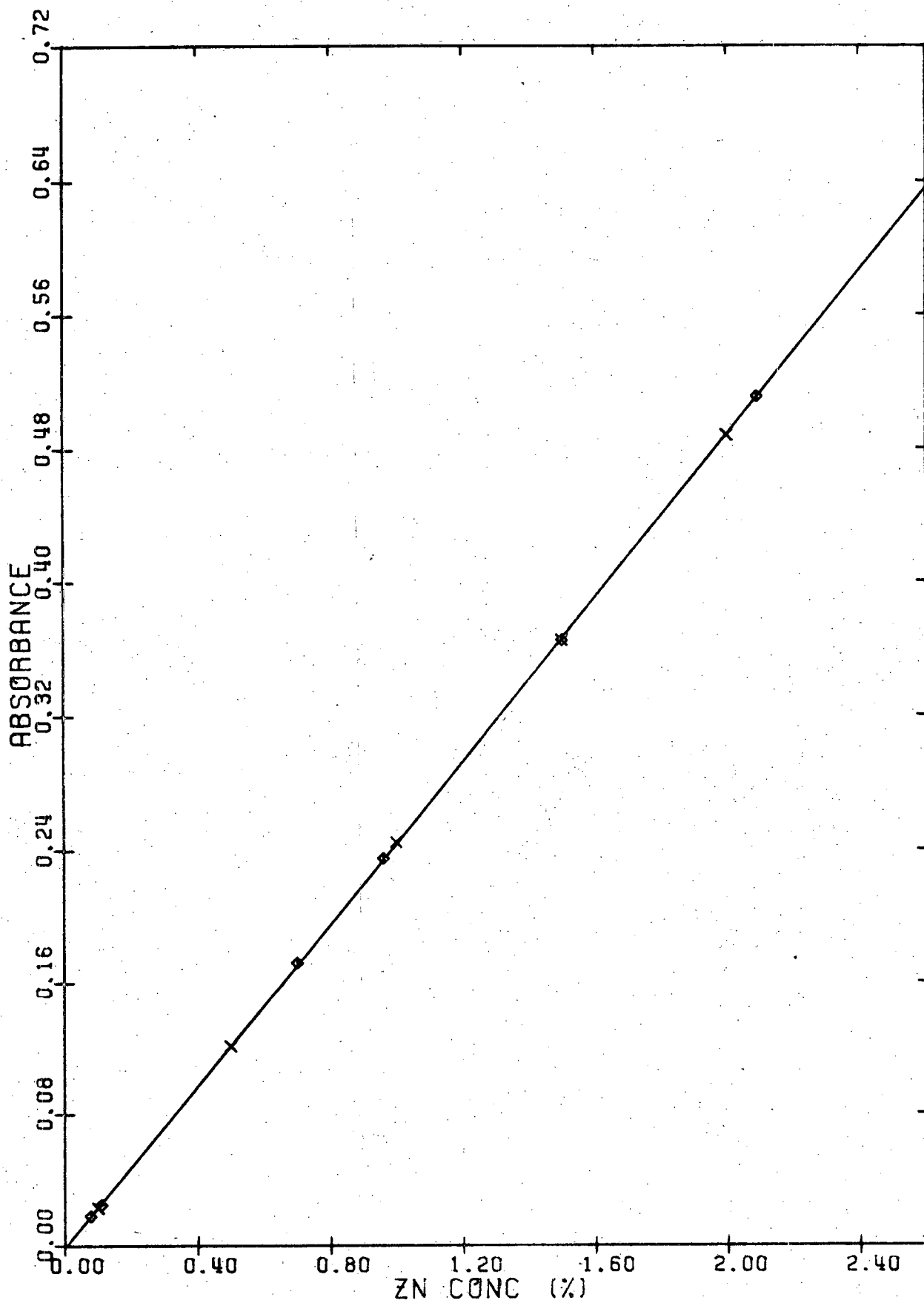Figure 2.
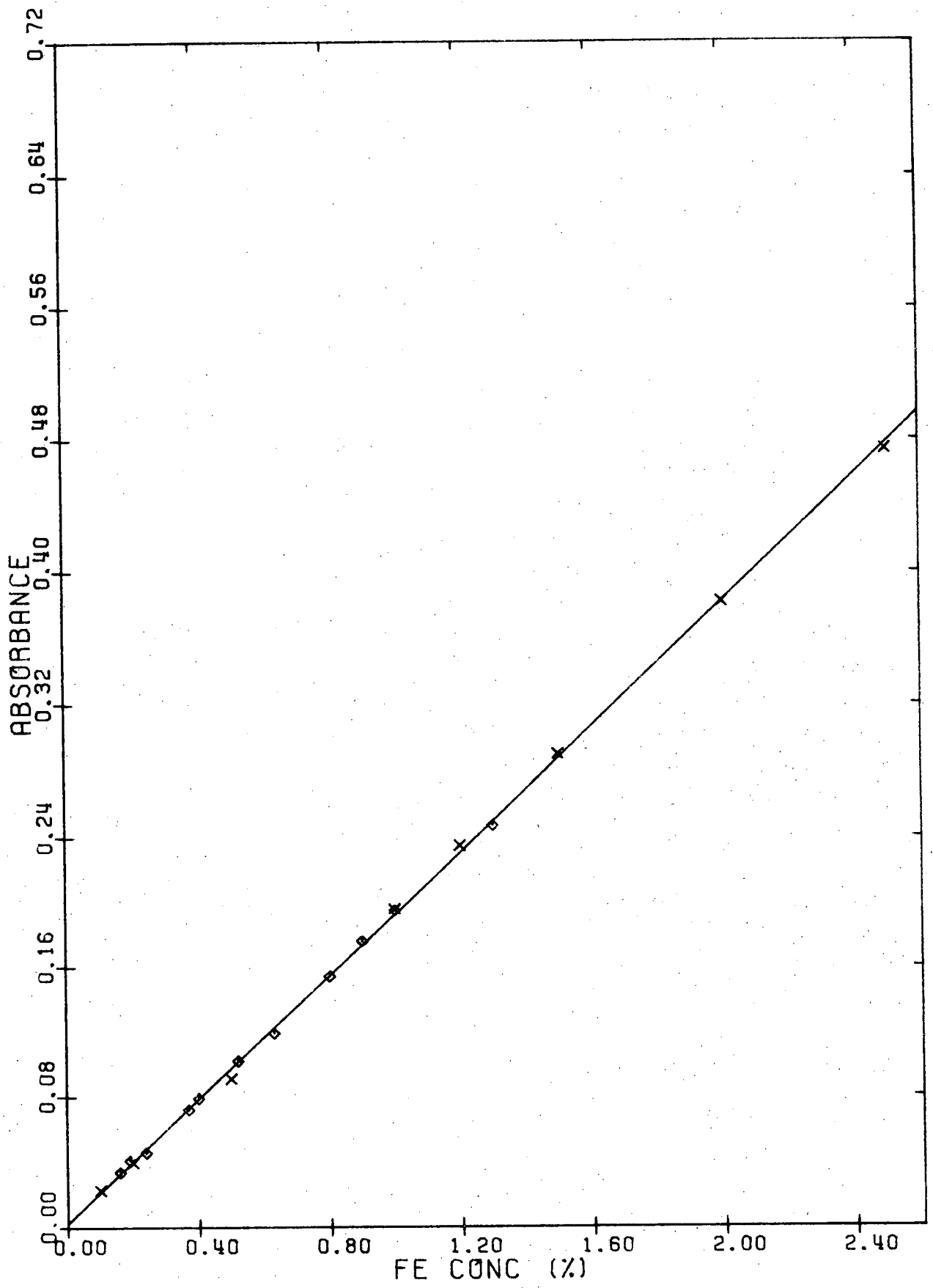
Figure 3.
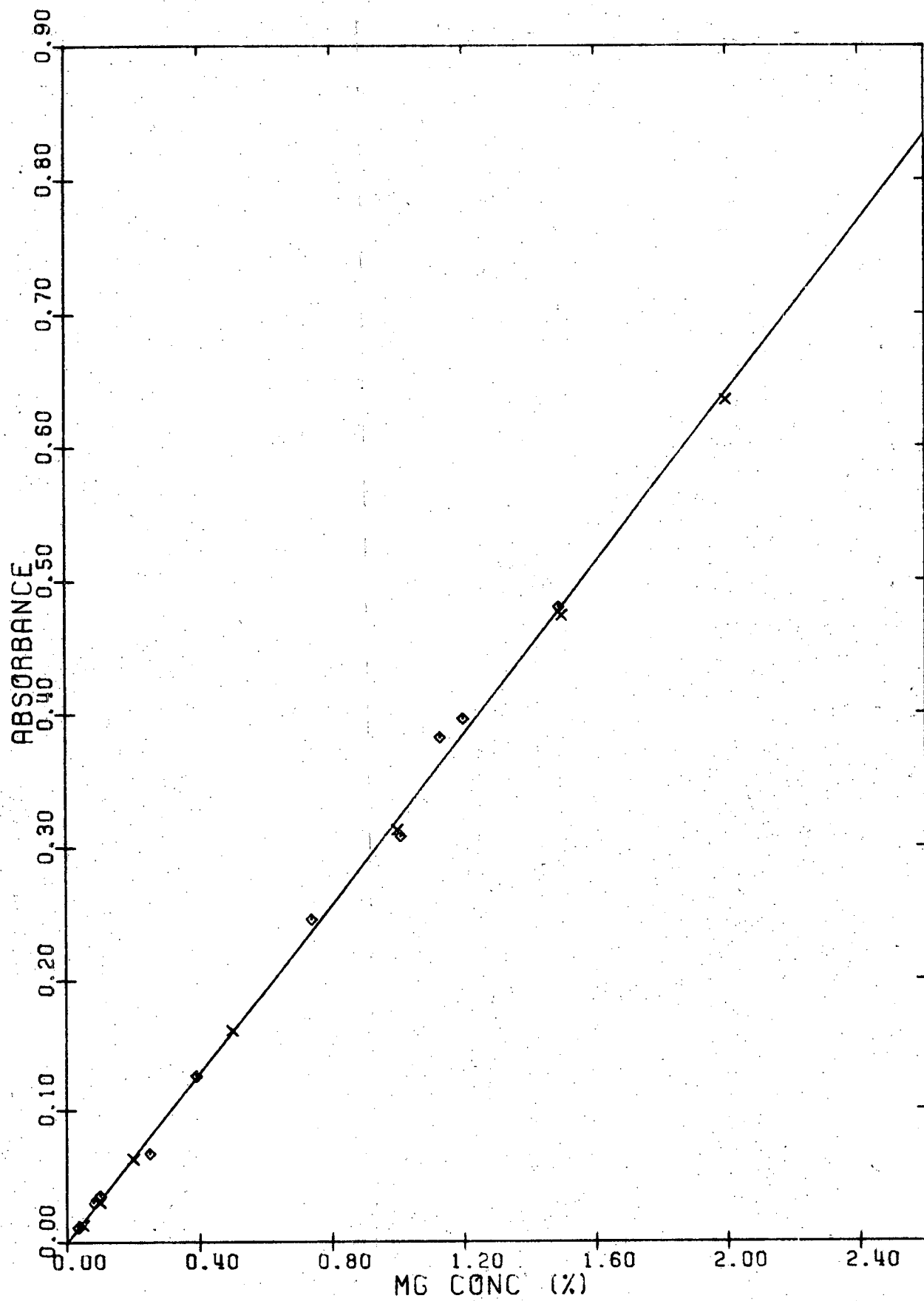
Figure 4.
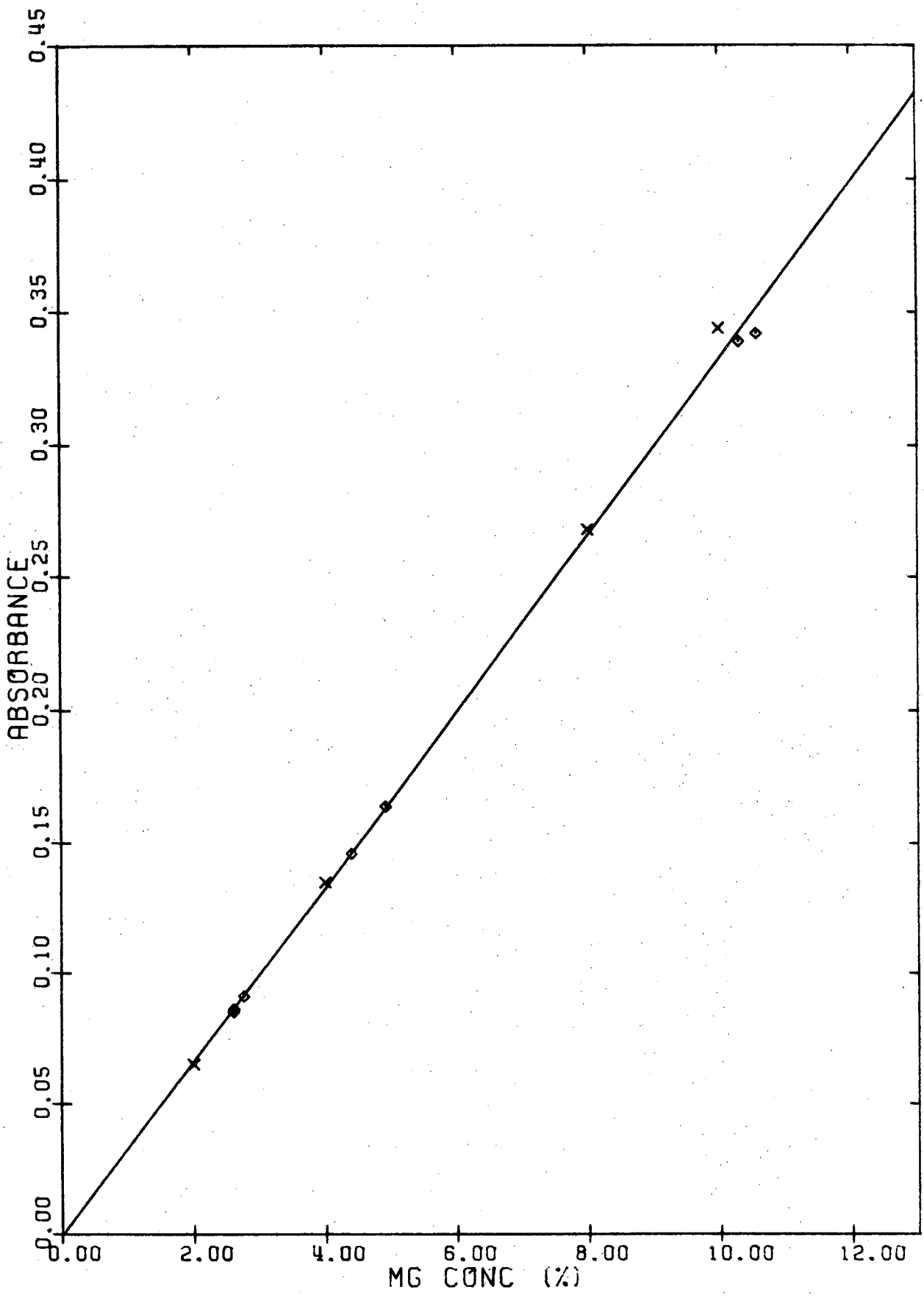
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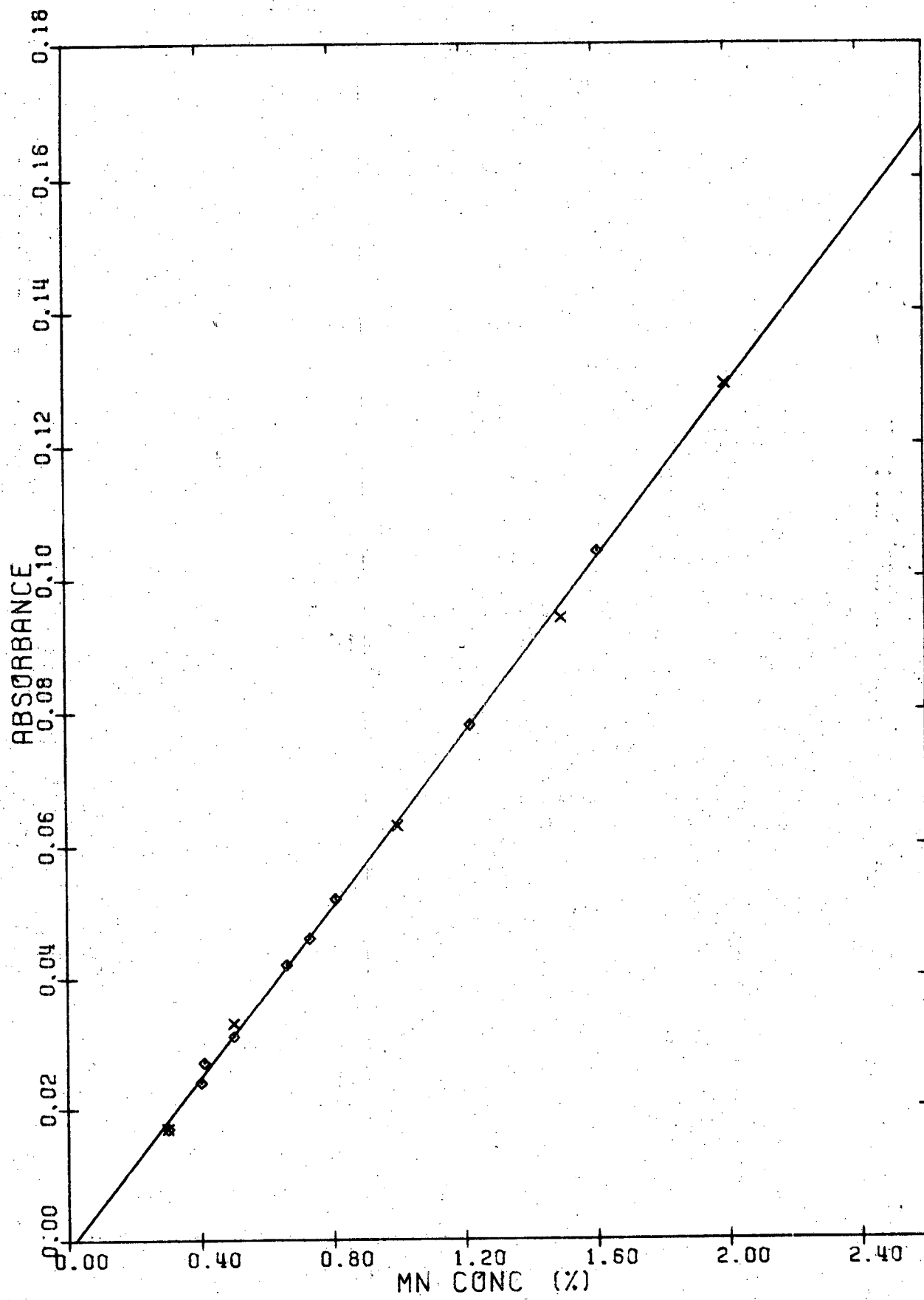
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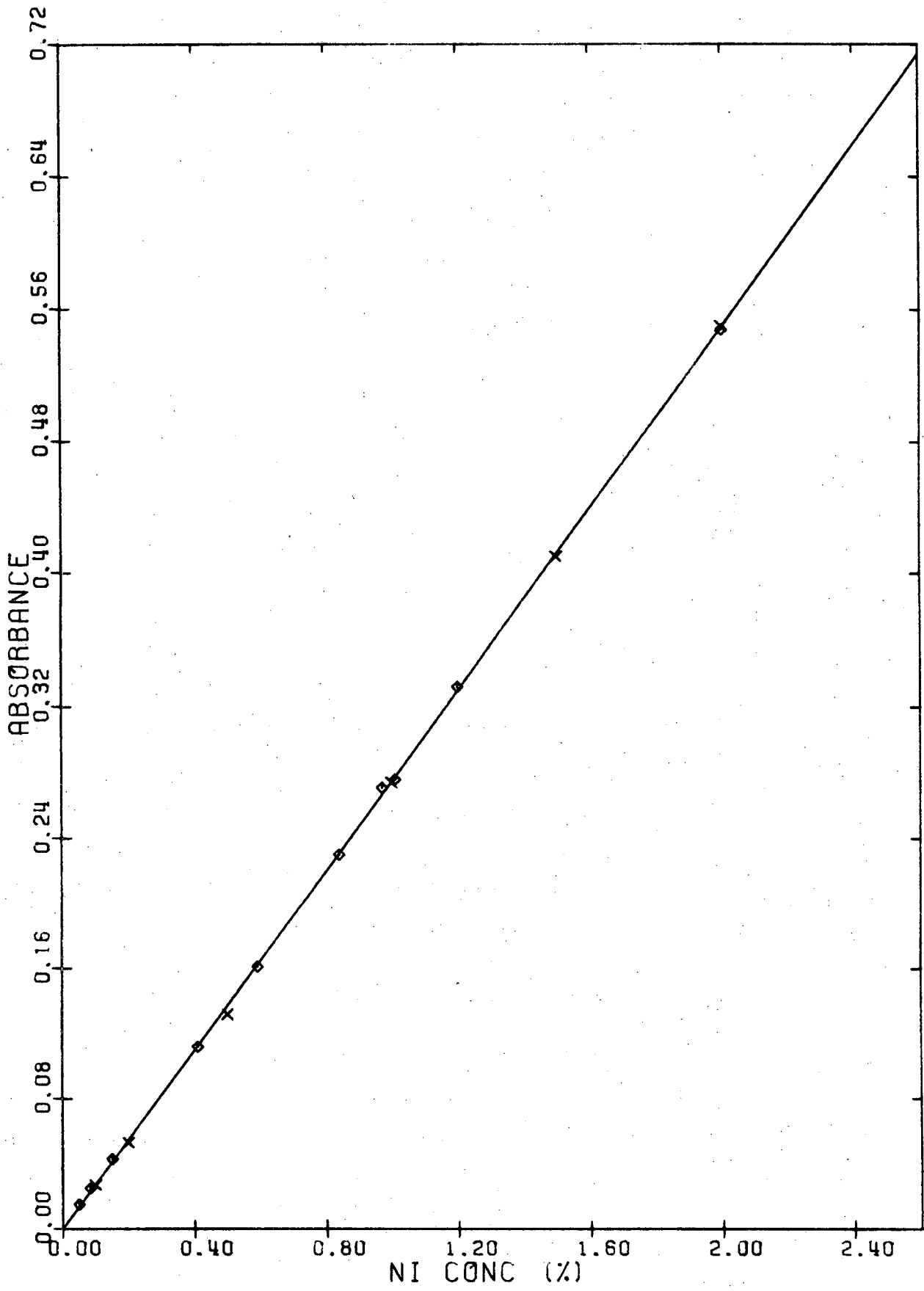
Figure 7.

Figure 8.
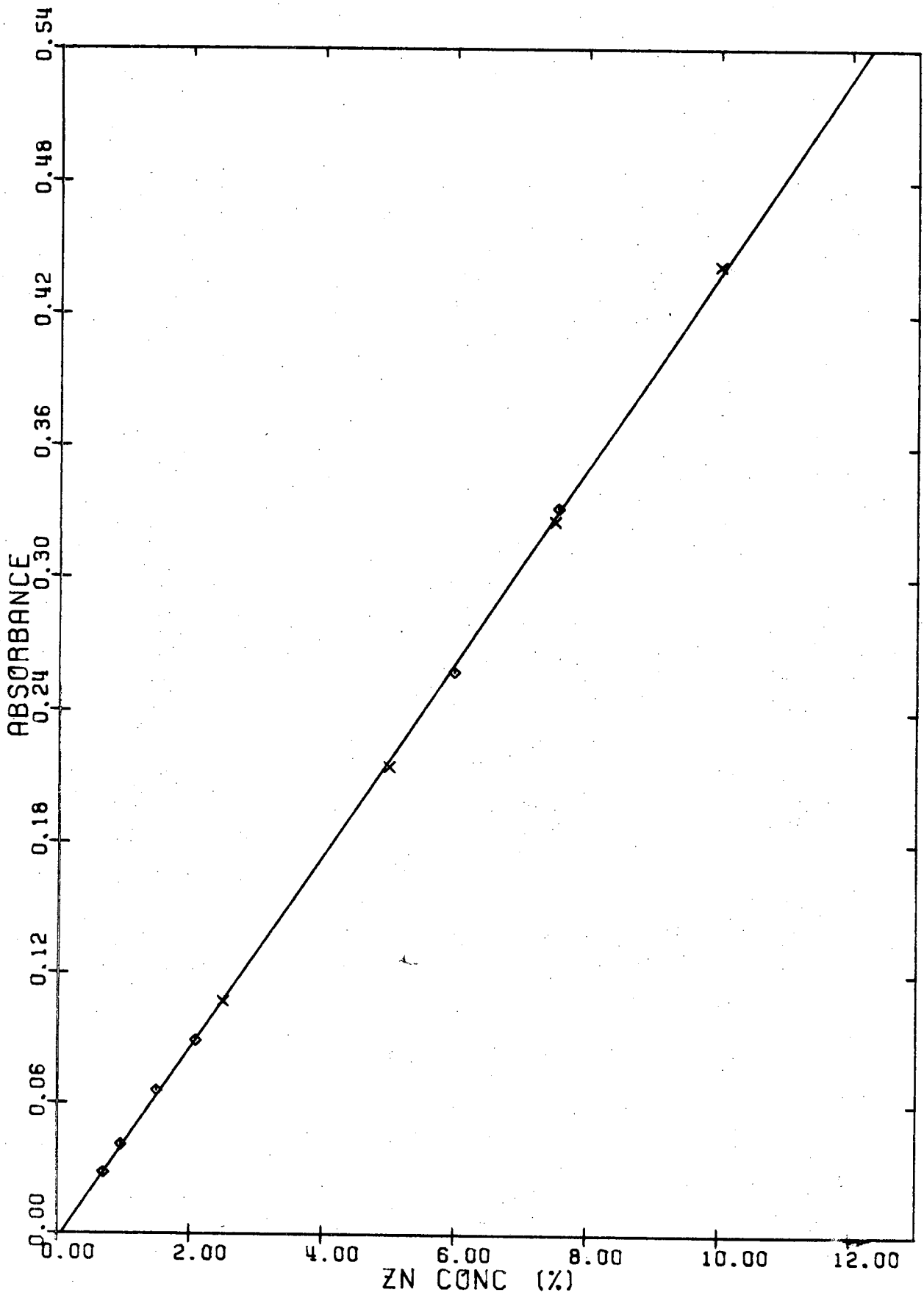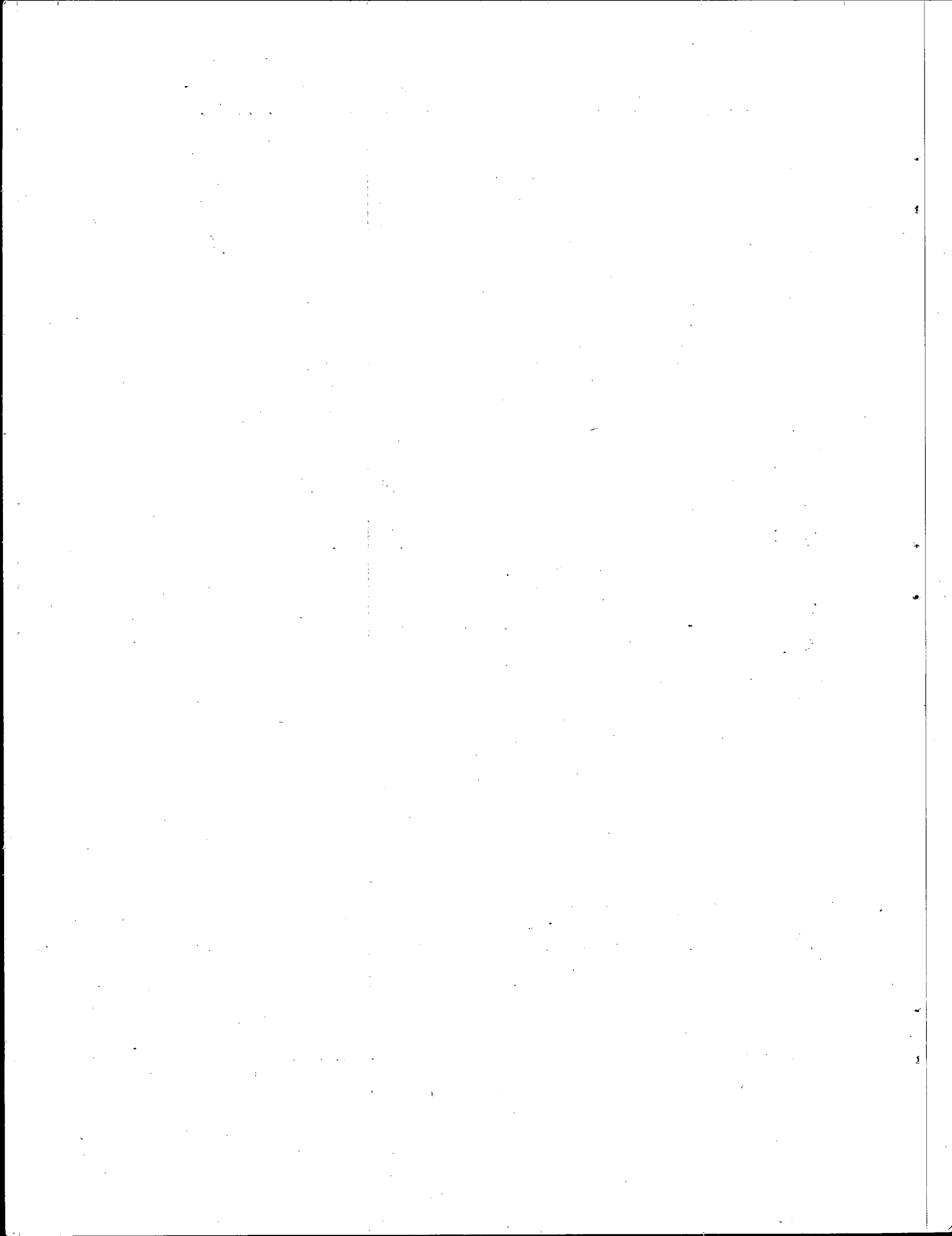
Figure 9.

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