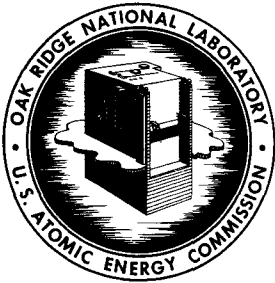X-822

OAK RIDGE NATIONAL LABORATORY
Operated by
UNION CARBIDE NUCLEAR COMPANY
Division of Union Carbide Corporation

UCC

Post Office Box X
Oak Ridge, Tennessee

DATE: August 12, 1959

COPY NO. 92

SUBJECT: Equilibrium Calculation in the System: Uranyl Nitrate, Nitric Acid, Water, TBP, and Kerosene Diluent Using the ORACLE Digital Computer

TO: F. L. Culler, Jr.

FROM: S. H. Jury and M. E. Whatley

ABSTRACT

A set of equations was written which allowed the calculation of equilibrium concentrations in the solvent phase of nitric acid and uranyl nitrate, given the initial TBP concentration in the solvent and the aqueous phase concentrations of acid and uranium. An ORACLE subroutine was written based on these equations which will calculate a set of equilibrium values in about 35 milliseconds. The subroutine was incorporated into a short program to calculate equilibrium points. A set of calculated points are presented in graphical form for 5, 20, 30, 40, and 100% TBP for nitric acid concentrations up to 7 $\underline{M}$ and uranyl nitrate concentrations up to 0.6 $\underline{M}$ in the aqueous phase. Since these calculations were based on limited data, it is the purpose of this memo to invite comment on their accuracy. The equations were reworked from a Russian paper by A. M. Rozen and L. P. Khorkhorina, using emperical polynomial fits for some poorly defined parameters, and were based principally on data from J. W. Codding. This project was undertaken to provide a subroutine to calculate equilibria in a general purpose ORACLE program to calculate the performance of multi-stage compound extraction cascades with more than one significant solute.

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DEVELOPMENT OF EQUATIONS

A number of investigators[1,2] have studied the aqueous uranyl nitrate-nitric acid system with TBP diluted with a petroleum hydrocarbon. McKay[3,4,5] and others have also made numerous contributions. There remains however the need for a generalized correlation of these works for column design. This work devotes itself to such an attempt.

Rozen[2] sets forth the argument that uranyl nitrate is extracted according to the equation:

$$UO_2^{2+} + 2NO_3^- + 2TBP = UO_2(NO_3)_2 \cdot 2TBP$$
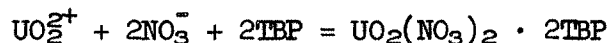
and thus:

$$Ku = \frac{[UO_2(NO_3)_2 \cdot 2TBP]}{[UO_2^{++}][NO_3^-]^2[TBP]^2} \tag{1}$$

where:

Ku = The thermodynamic equilibrium constant.

[] = Refer to activities.

The UO_2^{2+} and NO_3^- activities in the aqueous phase are used because at equilibrium their activities in the two phases are equal.

Nitric acid is apparently extracted according to the equation:

$$H^+ + NO_3^- + TBP = HNO_3 \cdot TBP.$$

For this equation the equilibrium relation may be written as:

$$K = \frac{[HNO_3 \cdot TBP]}{[H^+][NO_3^-][TBP]}. \tag{2}$$

Equation 2 may also be written in terms of concentrations and activity coefficients thus:

$$K = \frac{\gamma_{\Theta H}(HNO_3 \cdot TBP)}{\gamma_{H^+}(H^+)\,\gamma_{NO_3^-}(NO_3^-)\gamma_T(TBP)} \tag{3}$$

where:

() = Refer to concentrations. (See Table 1a for nomenclature).

Table 1a Nomenclature

W = aqueous phase acid concentration, moles/liter.

Z = organic phase acid concentration, moles/liter.

T_O = TBP concentration in the organic phase, moles/liter.

X = aqueous phase uranyl nitrate concentration, moles/liter.

Y = organic phase uranyl nitrate concentration, moles/liter.

γ = activity coefficient of uranyl nitrate in aqueous acid.

γ_T = the activity coefficient of TBP.

γ_Θ = the activity coefficient of $UO_2(NO_3)_2 \cdot 2TBP$.

f_H = factor defined by Equation (6).

$\gamma_{UO_2^{++}}, \gamma_{H^+}, \gamma_{NO_3^-}$ = ion activity coefficients.

$\gamma_{\Theta H}$ = the activity coefficient of $HNO_3 \cdot TBP$.

μ = ionic strength.

C_i = concentration of ith ion M/L.

Z_i = valence of ith ion.

f_u = factor defined by Equation (7).

Note: In the article by Rozen Nx was used to represent W and Ny to represent Z. In the ORACLE code Nx and Ny are used instead of W and Z.

If we define K_1 as:

$$K_1 = \frac{K \, \gamma_{H^+} \, \gamma_{NO_3^-} \, \gamma_T}{\gamma_{\theta H}} \qquad (4)$$

and note that

$$(HNO_3 \cdot TBP) = Z$$

$$(H^+) = W$$

$$(NO_3^-) = W + 2X$$

$$(TBP) = T_0 - 2Y - Z$$

then

$$K_1 = \frac{Z}{W(W + 2X)(T_0 - 2Y - Z)} \qquad (5)$$

If we now define f_H as:

$$f_H = K_1 W(2X + W) \qquad (6)$$

then:

$$Z = f_H(T_0 - 2Y - Z). \qquad (7)$$

Rozen used an average value of 0.2 for K_1. As will be seen later, K_1 is actually a function of ionic strength, μ, and the value of 0.2 is only a fair approximation.

Equation (1) may also be written in terms of activity coefficients and concentrations:

$$Ku = \frac{\gamma_\theta [UO_2(NO_3)_2 \cdot 2TBP]}{\gamma_{UO_2^{++}}(UO_2^{++}) \, \gamma_{NO_3^-}^2 (NO_3^-)^2 \, \gamma_T^2 (TBP)^2} \cdot \qquad (8)$$

Rozen defined the activity coefficient, γ, of uranyl nitrate in the aqueous phase as:

$$\gamma^3 = \gamma_{UO_2^+} \, \gamma_{NO_3^-}^2. \qquad (9)$$

He also defined $\bar{Ku}$ as:

$$\bar{Ku} = Ku \, \frac{\gamma_T^2}{\gamma_\theta} . \qquad (10)$$

Since:

$$[UO_2(NO_3)_2 \cdot 2TBP] = Y$$

$$(UO_2^{++}) = X$$

we may write:

$$\bar{K}u = \frac{Y}{(T_0 - 2Y - Z)^2 \, X\gamma^3(2X + W)^2} \qquad (11)$$

Rozen wrote this equation in the form:

$$Y = f_u(T_0 - 2Y - Z)^2 \qquad (12)$$

where f_u is defined as:

$$f_u = \bar{K}uX\gamma^3(2X + W)^2. \qquad (13)$$

Rozen's table of $\bar{K}u$ was fit by the emperical expression:

$$\bar{K}u = 86.01 - 25.59 \, T_0 + 2.718 \, T_0^2 \qquad (14)$$

Rozen's datum points as well as the fit given by equation (14) are plotted in Fig. 1.

The K_1 and γ were computed from Codding's data using the previous resulting equations. They were plotted against ionic strength as shown in Fig. 2. The plots were fit to the empirical expressions:

$$K_1 = 0.385 - 0.155 \, (W + 3X) + 0.024 \, (W + 3X)^2 \qquad (15)$$

and

$$\gamma = 0.34 + 0.199 \, (W + 3X) \qquad (16)$$

The empirical curves are also shown in Fig. 2.

The $W + 3X$ terms in the empirical equations derive from the definition of ionic strength which is:

$$\mu = 1/2 \sum c_i z_i^2. \qquad (17)$$

Thus, this equation may be written:

$$\mu = 1/2[C_{UO_2^{++}}(2)^2 + C_{H^+}(1)^2 + C_{NO_3^-}(1)^2] = 1/2[4X + W + (2X + W)] = 3X + W \qquad (18)$$

The suggestion that γ and K_1 should be a function of μ comes from the work of McKay.

The scatter of the triangular and circle datum points in Fig. 2 suggested difficulty in Codding's measurement of acid and uranyl in the presence of each
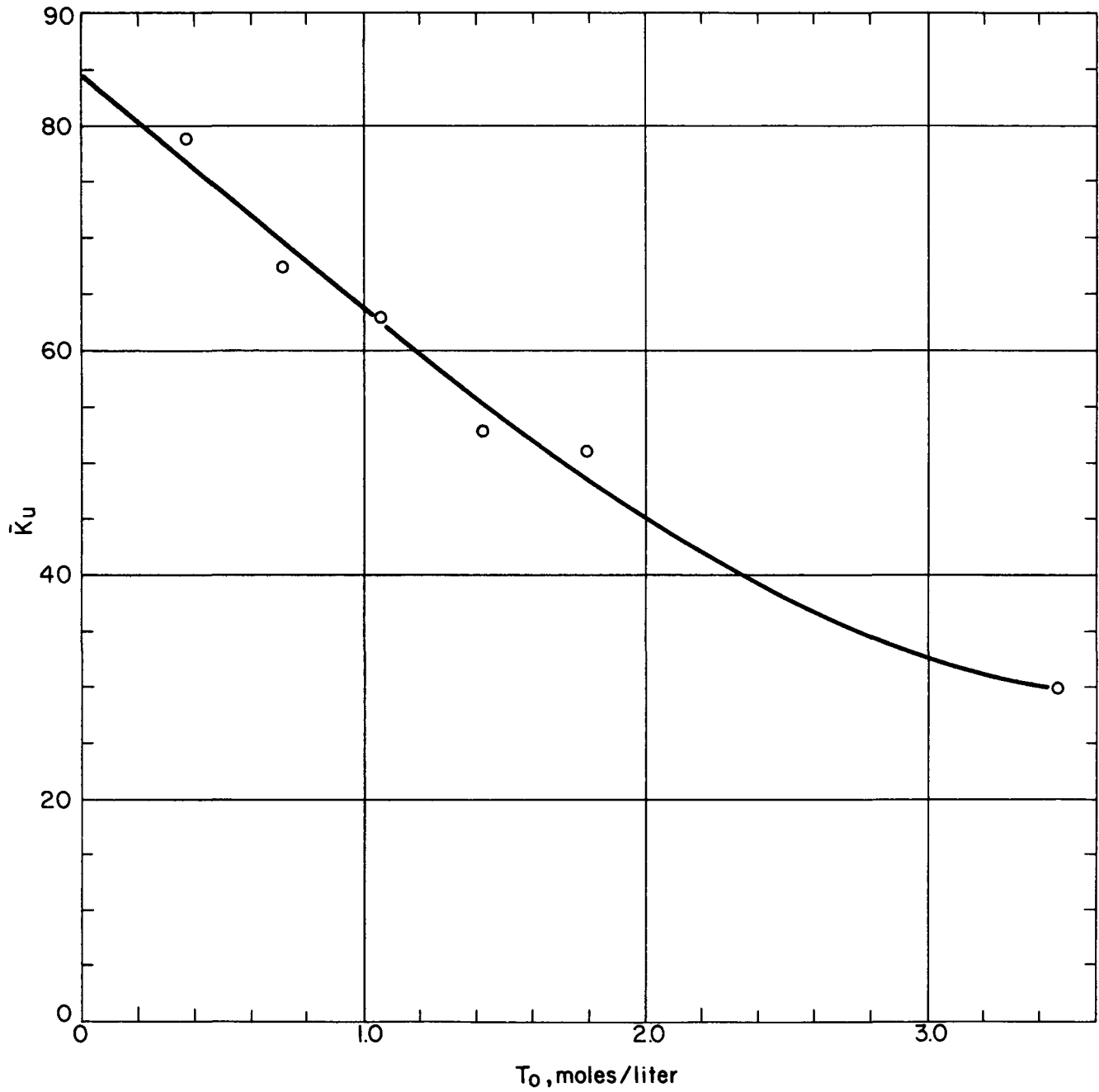
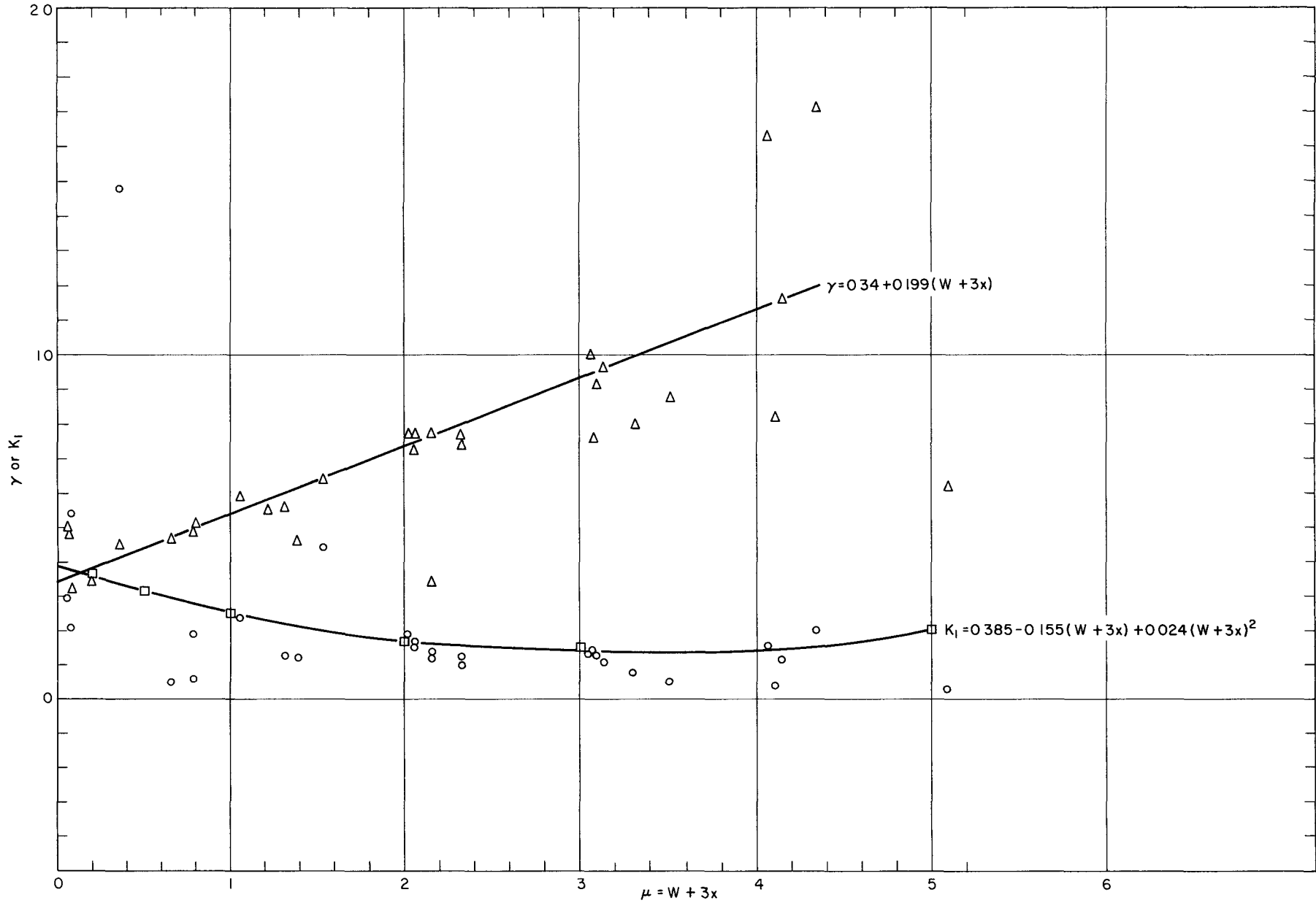Fig. I. A Plot of Data from Table I of Article by Rozen.

Fig 2 Calculated Data and the Empirical Expressions for Them

other. In the case of K_1, it was decided therefore to fit K_1 to the pure acid runs, i.e., the square points, of Codding's data. These point show a much better consistency within themselves.

The equations for K_1, γ, $\bar{K}u$, etc, when rearranged and ordered, comprise a set from which equilibrium can be explicitly calculated. These equations are given in Table 1b.

DISCUSSION OF EQUATION DEVELOPMENT

It is a recognized fact that TBP and water have limited solubilities in each other and that these vary with uranyl and acid. Also the nitric acid associates to some extent in water as does uranyl nitrate. In this calculation these effects and others were taken into account indirectly through their effects on γ, K_1, and $\bar{K}u$. In other words, γ and K_1 were selected as functions of ionic strength so that Codding's experimental data at 30% TBP would be fit. Rozen had already determined $\bar{K}u$ as such a function of T_0 as to make the calculations fit at other values of T_0. More complete experimental information is badly needed to test the validity of the set of equations and resulting code presented. Although many investigators have worked with the subject system, Codding is one of the few who measured Y and Z for corresponding measured values of X, W, and T_0 at equilibrium.

SUBROUTINE CHARACTERISTICS

A digital computer subroutine which employes the equations in Table 1 is given in Appendix I. Since an iterative calculation of the performance of a 20 stage solvent extraction column might use this routine up to 200,000 times, it was essential that the routine be as fast as possible. An attempt to code it in the usual 8-32 pseudo code showed that a minimum time of about 300 milliseconds would be required per set of equilibrium values. This rate would result in over 16 hours of calculating time for equilibrium calculations alone. By coding in fixed point, and using many time savers, the calculating time was cut to about 35 milliseconds, which would require about two hours of equilibrium calculating time for the same problem.

Among the time saving devices employed was the avoidance of general purpose subroutines from the ORACLE compiler. This subroutine does its own conversion from an 8-32 input to fixed point and has its own loop for extracting a square root. It does use the compiler subroutine 00065 to convert the calculated numbers from fixed point to floating point.

The necessity for repeated recalculating $\bar{K}u$, which is a function of TBP concentration alone, was eliminated by storing the previous value of $\bar{K}u$ and using it if an examination of the TBP concentration revealed it to be unchanged.

There are eight constants which are specified in the set of equations. If consideration of more equilibrium data indicates it necessary, the values of these numbers in the code can be easily changed. The locations of these numbers are given in the Appendix.

Table 1b Equations for Calculating Uranyl Nitrate and Nitric Acid Equilibrium
Solvent Phase Concentrations from Initial TBP Concentration and
Aqueous Phase Concentrations

$$K_1 = 0.385 - 0.155(W + 3X) + 0.024(W + 3X)^2 \tag{19}$$

$$f_H = K_1W(2X + W) \tag{20}$$

$$\bar{K}u = 86.01 - 25.59T_0 + 2.718T_0^2 \tag{21}$$

$$\gamma = 0.34 + 0.199(W + 3X) \tag{22}$$

$$f_u = \bar{K}u\gamma^3X(2X + W)^2 \tag{23}$$

$$F = \frac{f_u}{(1 + f_H)^2} \tag{24}$$

$$Y = 1/2[T_0 - (1/4F)(\sqrt{1 + 8FT_0} - 1)] \tag{25}$$

$$Z = \frac{f_H}{1 + f_H}(T_0 - 2Y) \tag{26}$$

There are limits to the range of variables over which this routine will produce valid results. At low values of TBP concentration and high uranium concentrations it calculates negative values for the nitric acid concentration in the solvent phase . Some typical deviations are given in Table 2.

Table 2 Calculated Values Showing Limits of Subroutine

TBP % by vol	Aqueous Phase		Solvent Phase	
	HNO_3 $\underline{M}$	Uranyl Nitrate $\underline{M}$	HNO_3 $\underline{M}$	Uranyl Nitrate $\underline{M}$
40	1.0	0.8	- 0.023	0.760
30	2.0	0.6	- 0.031	0.558
20	3.5	0.8	- 0.043	0.386
5	3.0	0.5	- 0.017	0.108
5	2.0	0.5	- 0.011	0.108
5	2.0	0.8	-52.9	49.9
5	1.0	0.8	-32.2	63.97

The low negative values are inherent in the equations, and since they vary smoothly, an iteration procedure could use them. It is not, in fact, certain that the acid in the organic solvent remains positive in the real system. In any case, the effect was not noticed for aqueous uranium concentrations less than 0.5 $\underline{M}$ (120 gms U/liter) at TBP concentrations over 20%, and was noticed at only slightly lower uranium concentrations for the lower TBP concentrations.

The very large negative numbers for nitric acid concentration and the corresponding ridiculous solvent uranium concentrations found at 0.8 $\underline{M}$ uranium with 5% TBP represents a limitation of the code stemming from its fixed point nature. This is an unmonitored failure which, if allowed to occur, would ruin a calculation. Another manifestation of the fixed point limitation is the overflow from using aqueous phase uranium concentrations over 0.7 $\underline{M}$ (170 gms U/liter) with high (100%) TBP concentrations. The subroutine, however, will catch this error, type "Ouch" on the keyboard typewriter and stop the calculation.

RESULTS OF CALCULATION

To test the subroutine a short program was written and used. The TBP concentrations used were 5, 20, 30, 40, and 100% by volume (0.19, 0.72, 1.06, 1.43, and 3.46 moles per liter respectively), and the aqueous phase nitric acid concentrations were 0.05, 0.5, 1.0, 2.0, 3.0, 3.5, 5.0, and, in some cases, 6.0 and 7.0 moles per liter. Ten values of aqueous phase uranium concentration were used ranging from 0.001 to 0.8 moles per liter. The results are shown graphically in Figs. 3 through 7. The plot of calculated curves for 30% TBP also includes Codding's data for purpose of comparison.

Beyond the insipid observation that these curves seem to follow the behavior of what is known about the system, there is no quantitative criteria for evaluating their accuracy. One of the purposes of this report is to invite those with equilibrium points from the many years of study of this system to comment on the validity of these curves.

THE ORACLE PROGRAM

The ORACLE program used to calculate the curves is available for calculating additional points. It is comprised of an administrative item, the subroutine to be tested, and a short edited output. The administrative item is given in Appendix II.

The input for the program is of the following form:

(a) Ten values for aqueous phase uranium concentration in moles per liter in "packed decimal" numbers.

(b) Any number of sets of TBP and nitric acid concentrations, in that order, in mole per liter in "packed decimal" numbers.

The code will read in the values of X and use them for every subsequent set of TBP and nitric acid concentrations which it will read as it calculates. The calculated answers will be punched out on paper tape, printing a separate table for each set of TBP and nitric acid concentrations, listing the values of X, Z, and Y (Appendix III).

REFERENCES

1. J. W. Codding, W. O. Haas, Jr., and F. K. Heumann, "Tributyl Phosphate-Hydrocarbon Systems", Ind.Eng.Chem.50, 145-52 (1958).

2. A. M. Rozen and L. P. Khorkhorina, "On the Thermodynamics of Extraction with Tributyl Phosphate", AERE-Lib/Trans 792 (Translated by V. Beals) (1958).
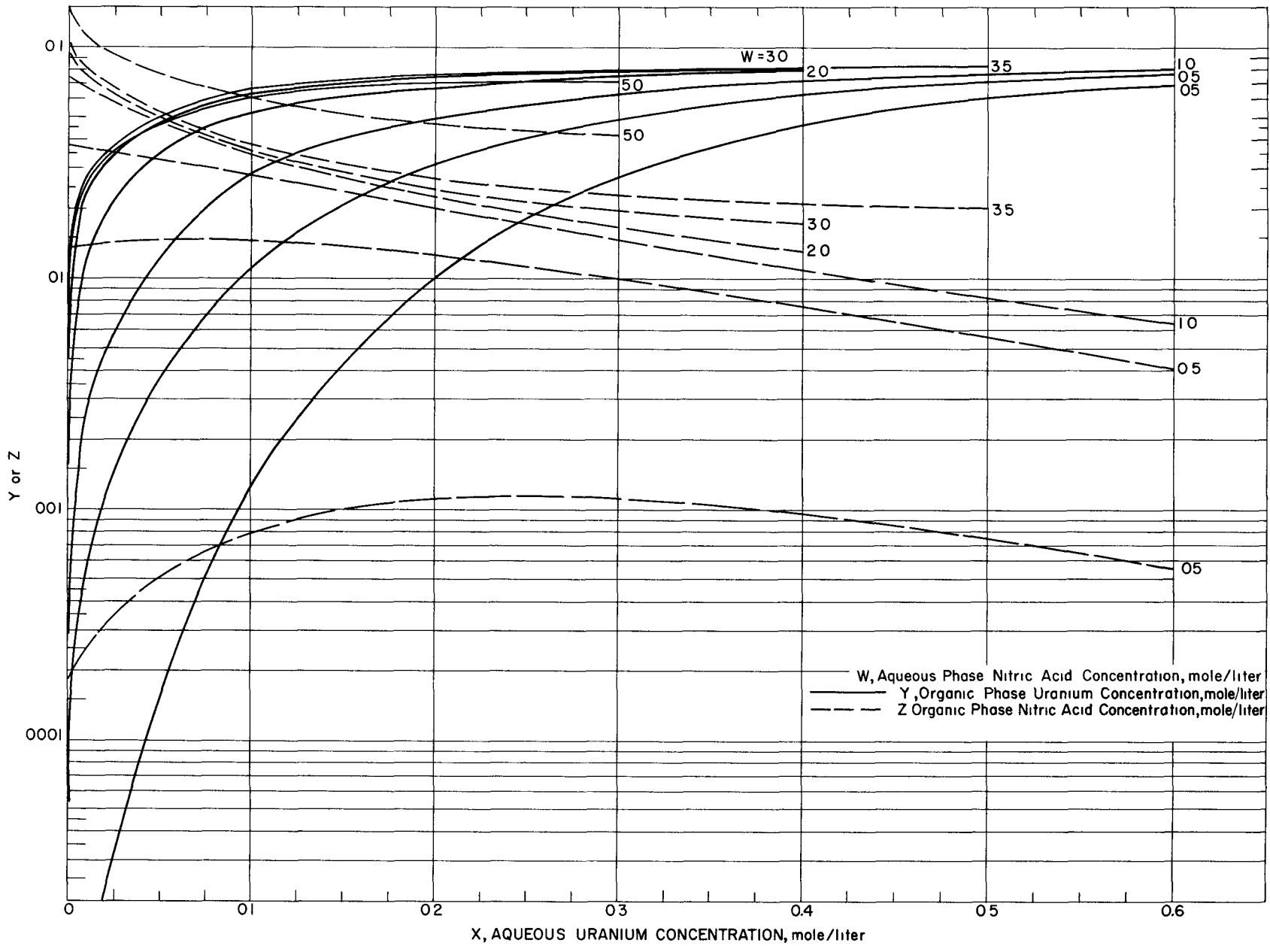
Fig 3 Calculated Distribution for 5% TBP
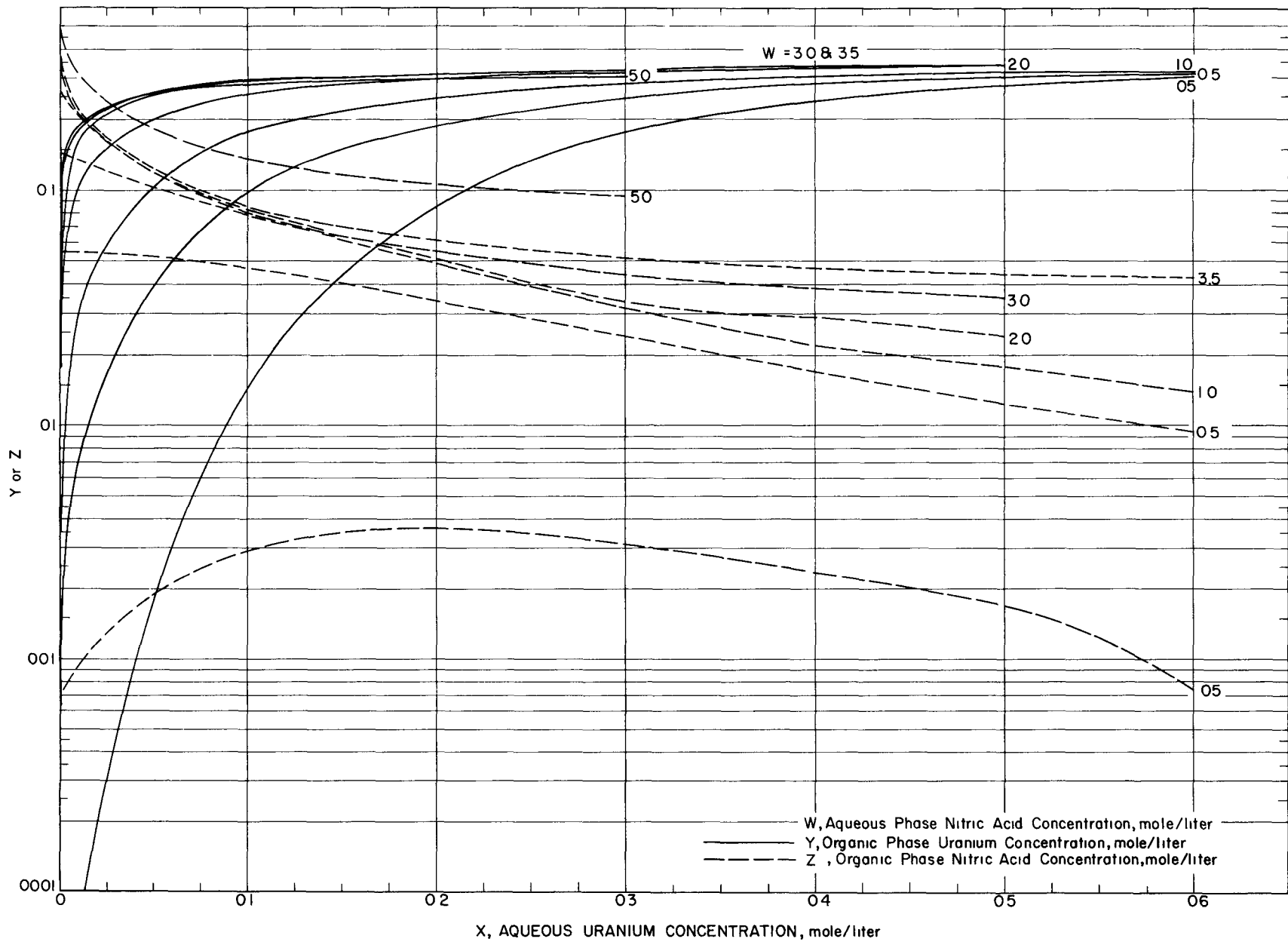
Fig 4 Calculated Distribution for 20% TBP
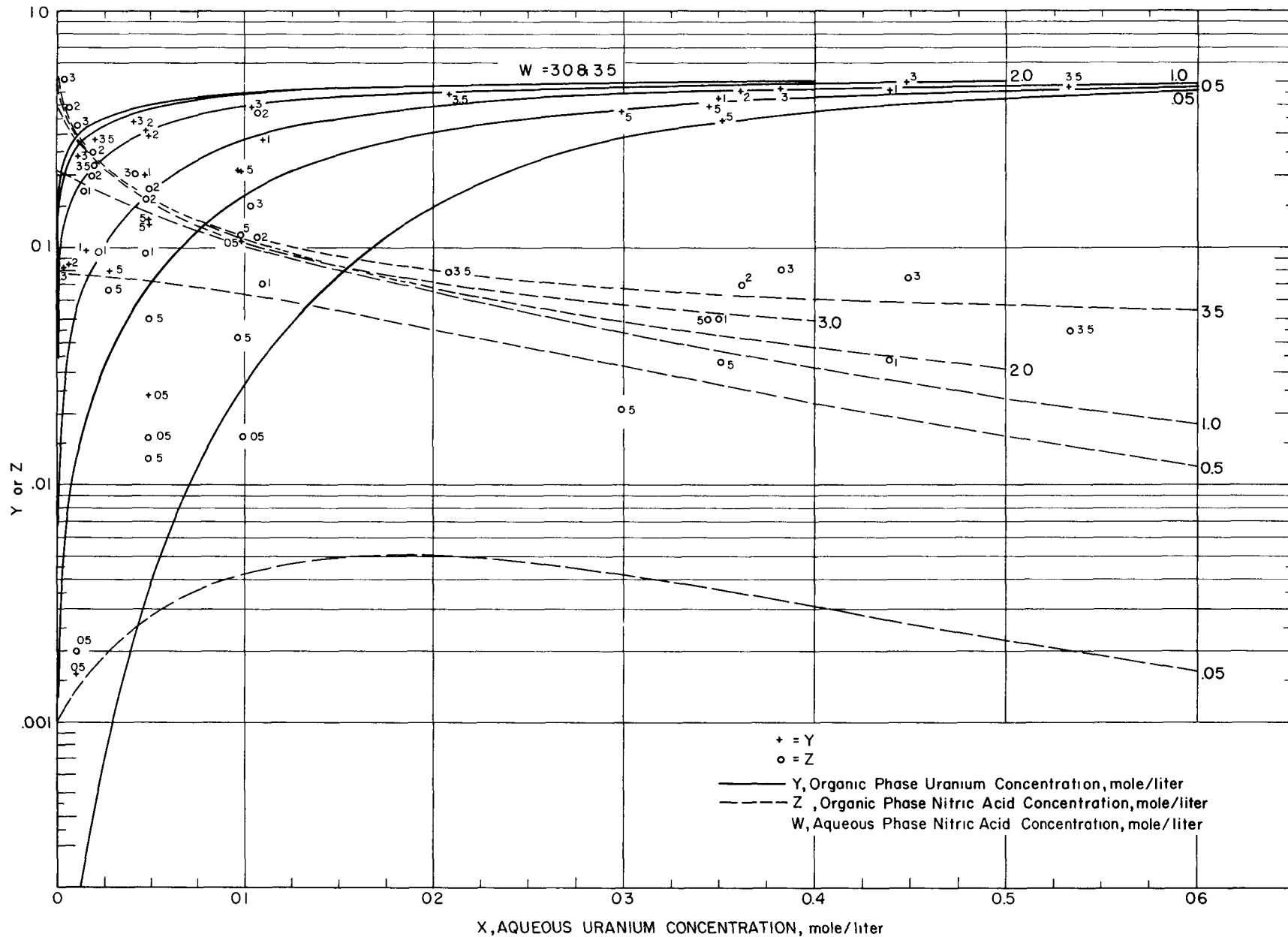
Fig. 5a. Calculated Distribution for 30% TBP.
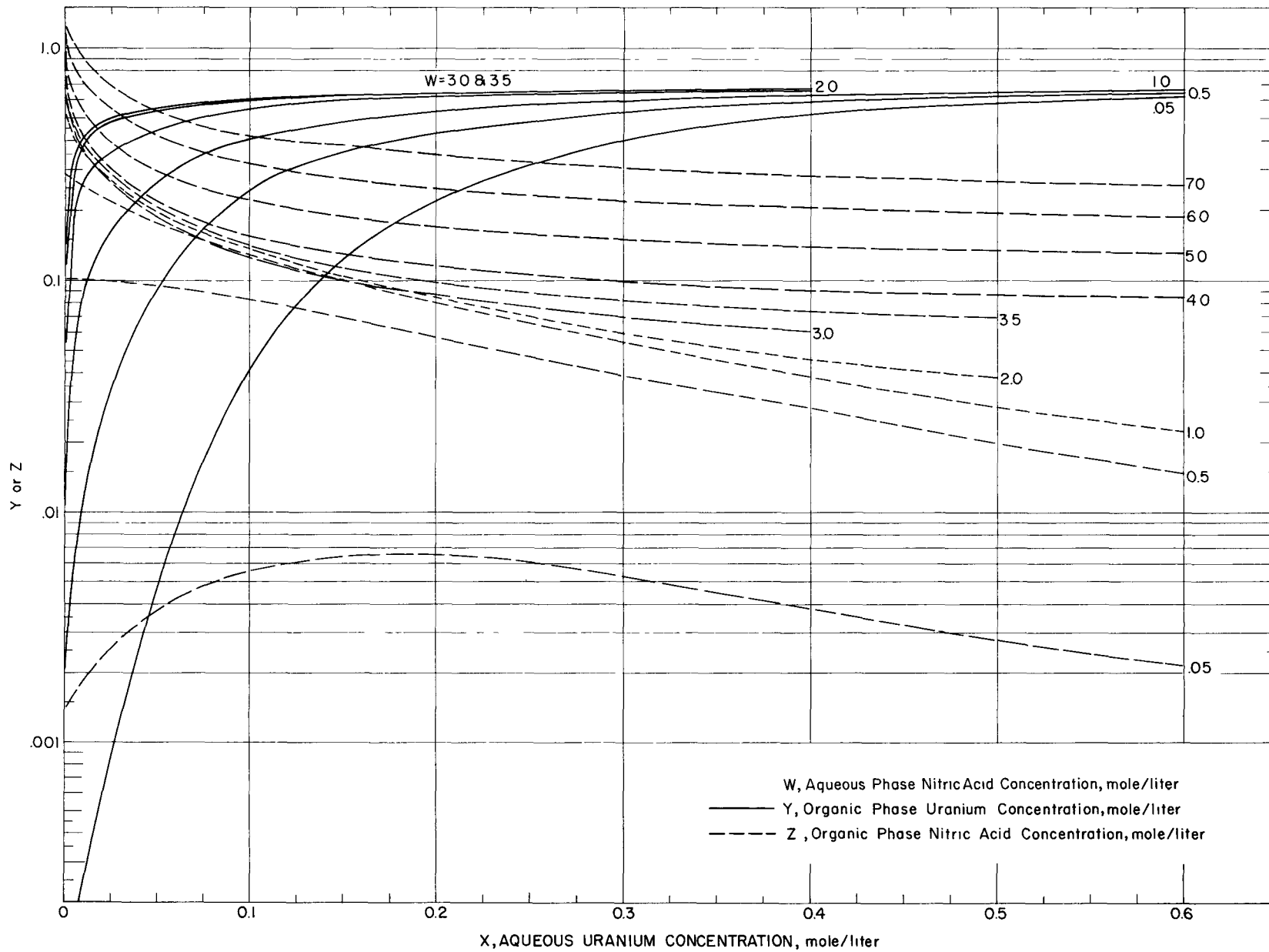
Fig 6a. Calculated Distribution for 40% TBP.
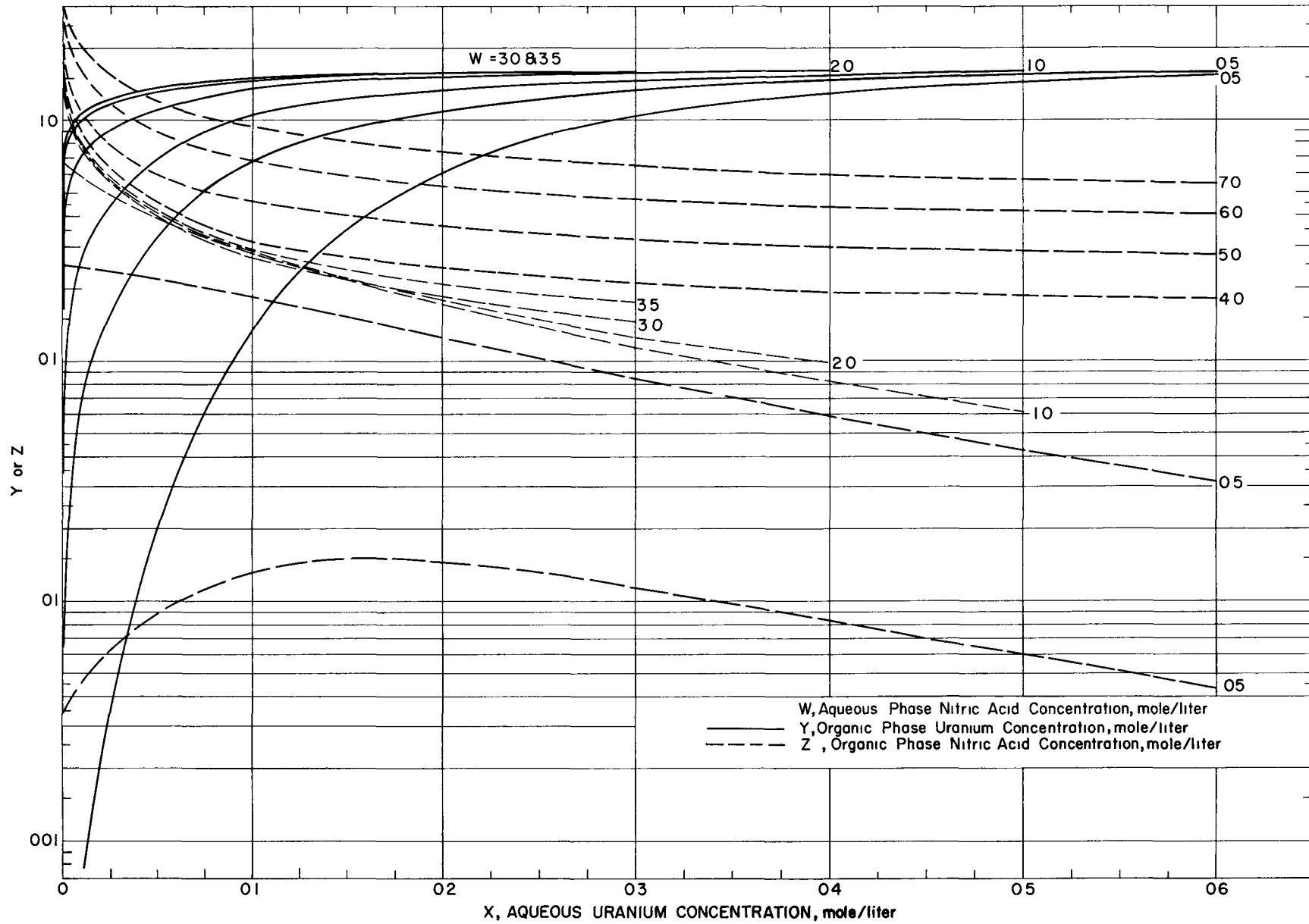
Fig 7a Calculated Distribution for 100 % TBP
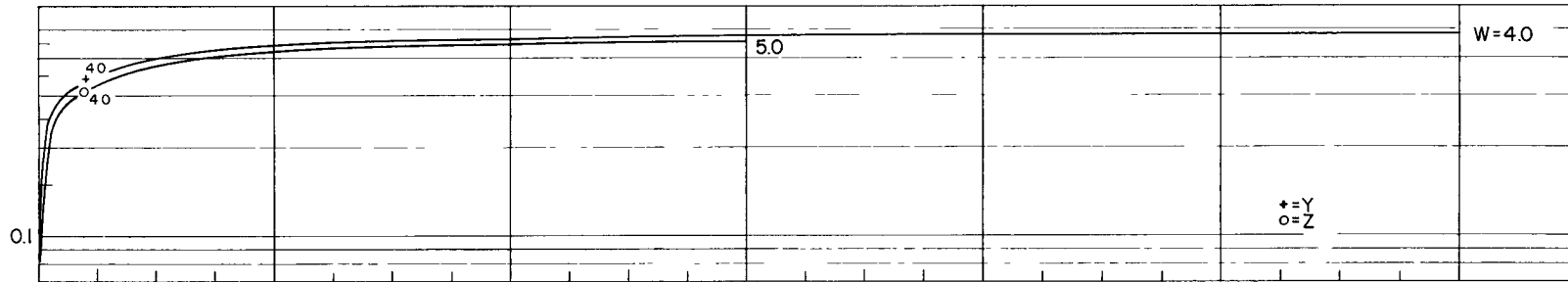
Fig. 5 b., 30% TBP
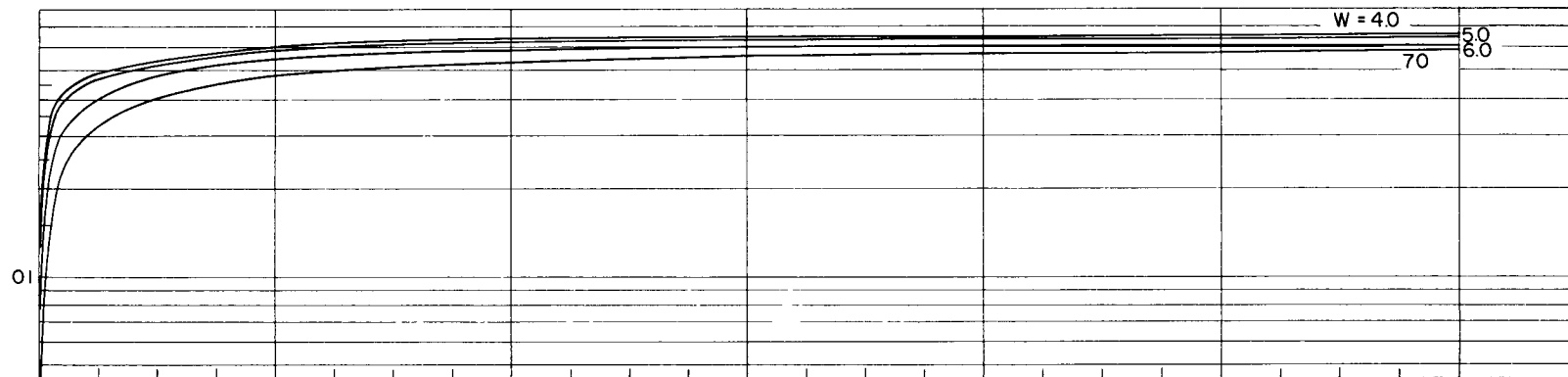
Fig. 6 b., 40% TBP
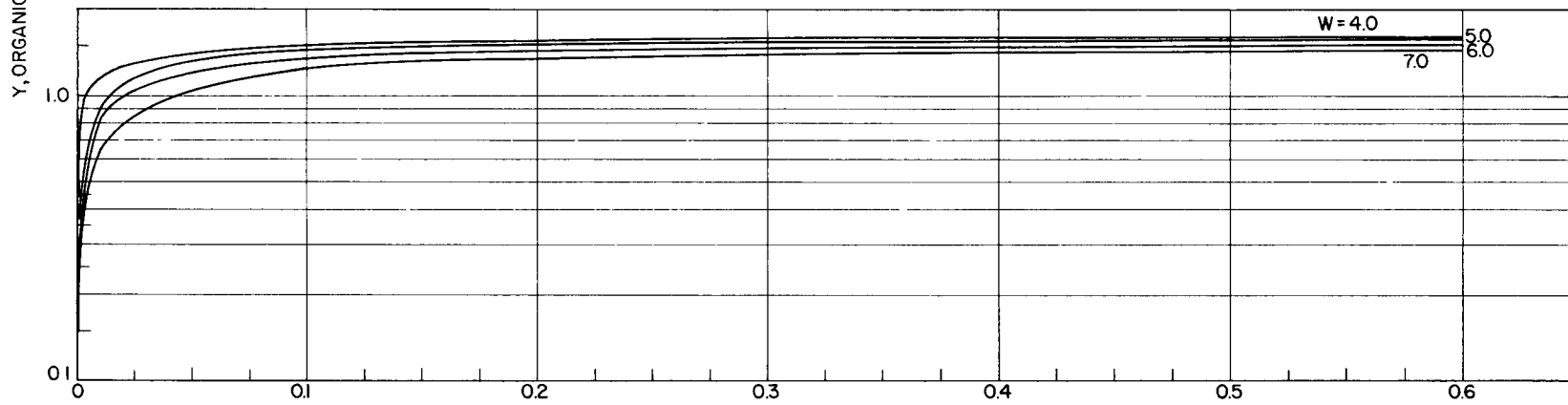
X, AQUEOUS URANIUM CONCENTRATION, mole/liter

Fig. 7 b., 100% TBP

3. I. L. Jenkins and H. A. C. McKay, _Trans. Faraday Soc._, 50, 107-19 (1954).

4. E. Glueckauf, H. A. C. McKay, and A. Mathieson, _Trans. Faraday Soc._, 47, 437-49 (1951).

5. A. W. Gardner, H. A. C. McKay, and D. T. Warren, _Trans. Faraday Soc._, 48, 997-1004 (1952).

APPENDIX I

Subroutine 00200

This subroutine calculates the equilibrium concentrations of uranyl nitrate and nitric acid in the organic solvent phase when the corresponding concentrations in the aqueous phase and the initial TBP concentration are given.

The subroutine entry in ORNL Compiler pseudo code is:

```
ff 000 00 200

-- ggg -- hhh

-- jjj -- ---

-- kkk -- mmm
```

where ggg and hhh are the addresses of the memory cells containing the aqueous phase concentrations of nitric acid and uranyl nitrate in mols per liter respectively.

jjj is the address of the initial concentration of TBP in the organic solvent in mols per liter.

kkk and mmm are the addresses which are to receive respectively the organic solvent phase concentrations of nitric acid and uranyl nitrate in mols per liter.

All numbers are in 8-32 floating point.

There are eight constants necessary to the calculation which appear in the equations:

$$\bar{K}u = a_1 T_0^2 - a_2 T_0 + a_3$$

$$K_1 = a_4 (W + 3X)^2 - a_5 (W + 3X) + a_6$$

$$\gamma = a_7 (W + 3X) + a_8$$

These constants are multiplied by scale factors and stored in the routine

Table 3 Location of Constants for Calculation

Constant	Scale Factor	Relative Address
1	2^{-2}	06a
2	2^{-9}	06b
3	2^{-7}	06c
4	1	06d
5	2^{-7}	06e
6	2^{-7}	06f
7	1	070
8	2^{-7}	071

All constants are used as binary fractions.

A copy of the subroutine is included (see following pages).

Item Number: 00200   Page   1

Key Words: 00200, 69,   0,   9,   0,   0,   0,   0,   0

Code: 000:   20   eb1   54   004

               001:      20   eb1   54   006

               002:      20   eb1   54   00a

               003:      20   eb1   54   068

               004:      60   000   74   005

               005:      24   000   5f   f01

               006:      24   000   71   007

               007:      54   009   24   000

               008:      5f   f02   40   000

               009:      24   000   5f   f03

               00a: 24   000   54   066

               00b: 51   067   24   f03

               00c: 22   018   5f   f00

               00d: 27   f00   4a   01b

               00e: 24   f03   5f   018

               00f: 1c   020   22   ea7

               010:      4a   04d   5f   f00

               011:      25   f00   51   012

               012:      24   e80   1c   000

               013:      7f   01a   38   06a

               014:      22   06b   5f   f00

               015:      60   f00   38   01a

               016:      1b   009   20   06c

               017:      5f   019   43   01b

               018:      00   000   00   000

               019:      00   000   00   000

01a:	00	000	00	000
01b:	24	f01	1c	020
01c:	22	ea7	4a	04d
01d:	5f	f00	25	f00
01e:	51	01f	24	e80
01f:	40	000	1c	000
020:	7f	f01	24	f02
021:	1c	020	22	ea7
022:	4a	04d	5f	f00
023:	25	f00	51	024
024:	24	e80	1c	000
025:	7f	f02	24	f02
026:	20	f02	20	f02
027:	20	f01	5f	f03
028:	60	f03	38	06d
029:	22	06e	5f	f00
02a:	60	f00	38	f03
02b:	40	000	1b	007
02c:	4f	04d	20	06f
02d:	4f	04d	5f	f00
02e:	24	f02	20	f02
02f:	20	f01	5f	f04
030:	60	f04	38	f01
031:	40	000	1b	007
032:	4f	04d	5f	f05
033:	60	f05	38	f00
034:	40	000	1b	007
035:	4f	04d	5f	f00

036:	60	f03	38	070
037:	20	071	4f	04d
038:	5f	f03	60	f03
039:	38	f04	40	000
03a:	1b	007	4f	04d
03b:	5f	f05	60	f05
03c:	38	f02	40	000
03d:	1b	007	4f	04d
03e:	5f	f06	60	f06
03f:	38	f03	40	000
040:	1b	007	4f	04d
041:	5f	f06	60	f06
042:	38	f03	40	000
043:	1b	007	4f	04d
044:	5f	f05	60	f05
045:	38	f04	5f	f03
046:	24	f00	20	e01
047:	4f	04d	5f	f04
048:	22	f03	4b	04d
049:	24	f03	3a	f04
04a:	38	019	3b	f04
04b:	7f	f02	40	000
04c:	38	01a	43	04f
04d:	8a	04e	43	04e
04e:	00	210	c1	238
04f:	1b	007	20	069
050:	4f	04d	5f	f03
051:	60	ec5	12	001

052:	4f	054	1c	001
053:	12	002	4c	051
054:	7f	f05	25	f03
055:	10	001	3a	f05
056:	7f	f06	24	f05
057:	10	001	20	f06
058:	5f	f06	60	f06
059:	22	f05	5f	f06
05a:	25	f06	22	eb0
05b:	7f	f05	48	054
05c:	24	f05	18	002
05d:	22	e01	18	009
05e:	3b	f02	7f	f02
05f:	38	f00	3b	f04
060:	7f	f05	24	01a
061:	22	f02	5f	f06
062:	ff	000	00	065
063:	40	f05	50	f05
064:	80	f06	40	f06
065:	24	f05	20	e07
066:	5f	000	24	f06
067:	20	e06	5f	000
068:	43	000	00	000
069:	00	200	00	000
06a:	56	f9c	10	000
06b:	06	65d	c2	000
06c:	56	028	10	000
06d:	03	014	60	000

06e:	00	27a	e1	400
06f:	00	628	f5	000
070:	19	78d	60	000
071:	00	570	80	000

APPENDIX II

ORACLE Program to Calculate Test Points

This program was written for the express purpose of testing subroutine 00200. It reads in ten values of X and uses them for calculating equilibrium values for any number of sets of values of TBP concentration and nitric acid concentration. All input numbers must be in moles per liter in packed decimal form. The code is available in paper tape form from the author on request. The operating instructions are as follows:

1. Load code to 000.

2. Place input tape under reader.

3. 43 000. The ten values of X will be read in, the first set of T_0 and W will be read in, and the answers will be punched out as the calculation proceeds. It will continue to read pairs of T_0 and W as long as they appear.

4. If the limits of the fix point scaling are exceeded the word "Ouch" will be typed on the console typewriter and the calculation will stop. To read in the next case, 43 020.

A copy of the adminstrative item is included (see following pages).

Item Number: 00001   Page   1

Key Words: 00001, 22, 3, 0, 0, 0, 0, 0, 0

Code:				
000:	ff	000	00	035
001:	40	800	40	809
002:	ff	000	00	035
003:	40	80a	40	80b
004:	24	008	51	013
005:	24	008	51	00f
006:	88	fff	43	008
	00	000	a0	001
007:	04	014	88	80a
008:	60	007	40	800
009:	ff	000	00	098
00a:	88	fff	43	00c
	00	000	a0	002
00b:	04	014	88	80b
00c:	60	00b	40	000
00d:	ff	000	00	098
00e:	88	fff	43	010
	00	000	a0	003
00f:	04	014	88	800
010:	60	00f	40	000
011:	ff	000	00	098
012:	ff	000	00	200
013:	40	80b	40	800
014:	40	80a	40	000
015:	40	80c	40	80d
016:	60	017	43	018

Item Number: 00001   Page   2

017:	04	014	80	80c
018:	ff	000	00	098
019:	60	01a	43	01b
01a:	04	014	80	80d
01b:	ff	000	00	098
01c:	8c	e35	24	013
01d:	20	eb1	51	013
01e:	51	00f	20	ea3
01f:	22	015	4b	010
020:	8c	e35	8c	e35
021:	8c	e2f	43	002

APPENDIX III

Format for Output of ORACLE Equilibrium Calculation of Uranyl Nitrate Nitric Acid Solvent Concentrations from Aqueous Concentrations

TBP =   1.429

HNO3 =   6.000

x	Ny	y
0.1000E-2	1.158	0.8204E-1
0.1000E-1	0.7265	0.3186
0.9999E-1	0.3189	0.5429
0.1500	0.2753	0.5672
0.1999	0.2496	0.5817
0.3000	0.2207	0.5981
0.3999	0.2052	0.6071
0.5000	0.1961	0.6126
0.6000	0.1903	0.6161
0.7999	0.1846	0.6199

Ny = Z   x = X   y = Y

All concentrations are in moles per liter. E-2 means times 10^{-2}, etc.

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