filtrate containing the alkoxybenzanilide saved. The precipitate was washed with two 10-ml. portions of benzene, and the washings were added to the benzene filtrate, which was washed successively with 20 ml. of water, 25 ml. of 5% hydrochloride acid to remove excess aniline, and 25 ml. of 5% sodium hydroxide. At this point some of the alkoxybenzanilide precipitated, and ether was added to redissolve it. The benzene-ether filtrate was finally washed with water, then dried over anhydrous potassium carbonate. Evaporation of the benzene and ether under vacuum left the crude alkoxybenzanilide, which was recrystallized from aqueous ethanol (Table II) until a constant melting point was obtained.

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Uranium Content of Uranyl Nitrate Solutions by Specific Gravity Measurement at Ambient Temperature

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SEVERAL ATTEMPTS have been made to correlate the specific gravity of aqueous solutions of uranyl nitrate with the corresponding uranium concentration of the solutions. An equation and nomograph have been reported (*5*) for uranium concentration determination from the specific gravity of uranyl nitrate solutions at 25° C. Also, specific gravity-uranium concentration data for uranyl nitrate solutions have been published (*1, 4*) for a temperature range of 10° to 90° C. with equations of correlation in terms of the specific gravity at 25° C.

Previously reported work does not provide an expression for obtaining the uranium concentration of a solution directly from a specific gravity reading made at temperatures of 10° to 50° C. Therefore, an effort was made to derive such an expression and to produce a nomograph based upon it capable of providing a rapid yet accurate means of uranium determination.

DERIVATION OF EQUATION

The data and equations reported by Perkins (*4*) furnish a basis to derive an equation relating specific gravity of a uranyl nitrate solution with its uranium concentration directly at temperatures ranging from 10° to 50° C. The equation presented by Perkins is of the form

$$\rho^{25}/_{4} = b_0 + b_1 C + b_2 N \tag{1}$$

where

C = uranium concentration of an aqueous uranyl nitrate solution, grams per liter
N = normality of free acid content in the solution
b_0, b_1, and b_2 = numerical constants at 25° C.

Inspection and analysis of Perkins' data show, however, that linear relationships exist between the following variables:

a. Constants b_0 and b_1 and the square of the temperature
b. Uranium concentration and constant b_2
c. Specific gravity and uranium concentration when the free acid content is zero

Substitution of relations *a* and *b* in Equation 1 results in an equation of the following general form to fit the experimental data:

$$\rho^{t}_{4} = x_0 + x_1 t^2 + (x_2 + x_3 t^2)\ C + (x_4 + x_5 C) N$$

or

$$\rho^{t}_{4} = x_0 + x_1 t^2 + x_2 C + x_3 C t^2 + x_4 N + x_5 C N \tag{2}$$

where

t = temperature of the uranyl nitrate solution, ° C.
x_1, x_2, x_3, x_4, and x_5 = numerical constants at any temperature t

To test the thesis that Equation 2 satisfactorily fits the experimental data of Perkins, the data were related in a multiple regression analysis programmed on a LGP-30 digital computer. Computer analysis showed that Equation 2 has a 99.6% multiple correlation and is the best of 13 possible relationships investigated. Stated with the values obtained by multiple regression

$$\rho^{t}_{4} = 1.0042 + 1.345 \times 10^{-3} C - 5.98 \times 10^{-6} t^2 - 1.12 \times 10^{-8} C t^2 + 2.51 \times 10^{-2} N + 3.14 \times 10^{-5} C N \tag{3}$$

Rearranged to express uranium concentration as the dependent variable, Equation 3 becomes

$$C = \frac{\rho^{t}_{4} - 1.0042 + 5.98 \times 10^{-6} t^2 - 2.51 \times 10^{-2} N}{1.345 \times 10^{-3} - 1.12 \times 10^{-8} t^2 + 3.14 \times 10^{-5} N} \tag{4}$$

EXPERIMENTAL EVALUATION

A limited amount of experimental work was performed to supplement the data of Burger and Perkins in the practical ranges of the variables and to test the reliability of Equation 4 (derived from Perkins' data) over these ranges. Several aqueous solutions of uranyl nitrate were compounded and tested. The methods of measurement duplicated normal procedures used in laboratory practice. Specific gravity measurements were made with glass hydrometers having a measuring precision of ±0.0005 gram per ml. Temperatures of the solutions were varied over a range of 20° to 50° C., and the free acid content ranged from 0.01 to 0.20N. The range of free acid content was not extended further because most values encountered in practice fall within these values. The uranium concentrations of the solutions varied from approximately 100 to 500 grams per liter. After specific gravity measurements had been made, the solutions were analyzed for actual uranium concentration by the colorimetric method described by Currah and Beamish (*2*). (This method with a precision of ±5% was the best available for uranium determination at the time that the analyses were performed.)

The uranium concentrations reported for the experimental study are compared in Table I with those computed from Equation 4. The comparison shows that there is good agreement at all levels between the reported and computed uranium values and thus demonstrates that use of the equation is legitimate over the ranges of variables cited.

Table I. Experimental Determinations of Specific Gravity and Uranium Concentration for Uranyl Nitrate Solutions

Temp.,	Specific Gravity		Free Acid	Uranium Concn., Grams per Liter	
° C.	$t/25$	$t/4$	Normality	Reported[a]	Equation
20	1.140	1.143	0.01	102	102
25	1.138	1.141			
30	1.1365	1.140			
40	1.133	1.136			
50	1.130	1.133			
20	1.208	1.212	0.02	150	151
25	1.206	1.210			
30	1.204	1.208			
40	1.200	1.204			
50	1.194	1.198			
20	1.237	1.241	0.02	169	172
25	1.2345	1.238			
30	1.2325	1.236			
40	1.228	1.232			
50	1.224	1.228			
20	1.2715	1.275	0.08	200	196
25	1.271	1.275			
30	1.268	1.272			
40	1.264	1.268			
20	1.409	1.413	0.12	300	301
25	1.4065	1.411			
30	1.403	1.407			
40	1.400	1.404			
20	1.546	1.551	0.16	400	401
25	1.542	1.547			
30	1.540	1.545			
40	1.534	1.539			
20	1.7055	1.711	0.20	509	520
25	1.702	1.707			
30	1.6985	1.703			
40	1.6905	1.695			

[a] Analytical determination of uranium by colorimetric method (*5*). Precision of method is ±5%.

NOMOGRAPH

Because the dependent variable, uranium concentration, is not separable from the independent variables, Equation 4 cannot be expressed directly as a nomograph and thus requires modification. Such modification can be accomplished by substitution from relation *c* of the linearly related specific gravity for the concentration term combined with temperature in Equation 3. With this modification and after rearrangement, Equation 3 is stated as

$$1.345 \times 10^{-3} C = \rho_4^t - 1.0042 + (8.4 \times 10^{-6} \rho_4^t - 2.4 \times 10^{-6}) t^2 - (3.14 \times 10^{-5} C + 2.51 \times 10^{-2}) N \qquad (5)$$

Equation 5 is translatable to nomographic form according to recognized procedures (*3*). The nomograph constructed from Equation 5 is presented in Figure 1 and its use is illustrated by the following example:

GIVEN: A uranyl nitrate solution with a specific gravity of 1.2652 at 35° C. and a free acid content of 0.20N.

FIND: The uranium concentration of the solution.

METHOD: Join 35° C. on the t scale with 1.2652 on the ρ_4^t scale by means of a straight-edge rule (line A, Figure 1). Note the point of intersection on the α scale.

Join the point on the α scale to 0.20 on the N scale by straight edge (line B, Figure 1).

Read the uranium concentration at the point of intersection with the C scale. The uranium value so found is 194 grams per liter.

DISCUSSION

The equations presented by Burger and others (*1*) and Perkins (*4*) were combined to obtain an expression for direct determination of uranium content from specific gravity measurement at a given ambient temperature

$$C = \frac{\rho_4^t - 1.0101 + 3.556 \times 10^{-4} t - (3.135 \times 10^{-2} - 1.548 \times 10^{-5} t) N}{1.3517 \times 10^{-3} - 6.675 \times 10^{-7} t} \qquad (6)$$

To compare the precisions of uranium determination by Equations 4 and 6 and the nomograph, limits of precision were calculated using Perkins' data as a standard of comparison. Values of uranium content were computed from the equations and the nomograph for combinations of temperature (10° to 40° C.) and free acid content (0 to 1.0N) spanning the range of experimental data. After calculation of the variance between computed and experimental values of uranium content, the limits of precision were obtained by multiplication of the standard deviation of the average difference and the proper t value taken from a table of such values for a 95% confidence level. The limits of precision calculated by this method for uranium concentrations ranging from 170 to 480 grams per liter are as follows for the three types of calculation:

Equation 6. ±5 grams per liter
Figure 1. ±2 grams per liter
Equation 4. ±1 gram per liter

It is evident from these limits of precision that the use of the derived equation or the nomograph for computation of uranium concentration is more accurate than the use of the modified equation derived from Burger and Perkins.

The nomograph is subject to limitations with respect to free acid content and uranium concentration. The use of the full free acid scale from 0 to 1.0N is valid on the basis of the work of Burger and Perkins. However, the most accurate results will be obtained when the range from 0 to 0.2N is used, since it is further substantiated by the experimental studies and is the practical range of acid content variability. As pointed out above for uranium concentrations, limits of precision were calculated for the range from 100 to 480 grams per liter. Uranium concentrations less than 100 grams per liter are subject to less precision and the use of the nomograph for values less than 10 grams per liter is not recommended.

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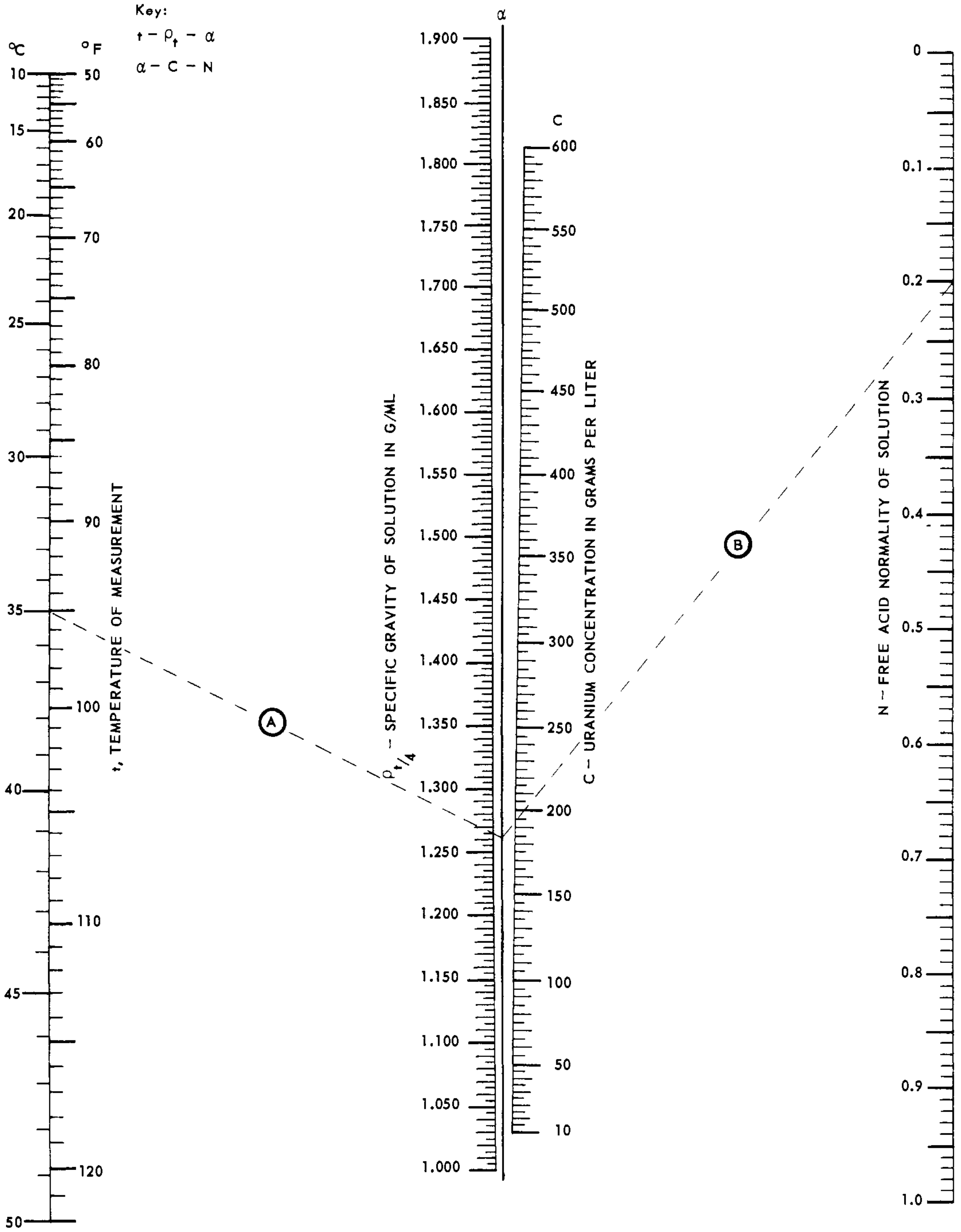

$$0.001345\,C = \rho_{t/4} - 1.0042 + (8.4\rho_{t/4} - 2.4)10^{-6}t^2 - (0.00314\,C + 2.51)10^{-2}N$$

Figure 1. Uranium concentration of uranyl nitrate solutions

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