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DISSOLUTION RATES OF URANIUM DIOXIDE SINTERED PELLETS IN NITRIC ACID SYSTEMS

By R. F. TAYLOR, E. W. SHARRATT, L. E. M. de CHAZAL* and D. H. LOGSDAIL

The dissolution process for sintered uranium dioxide into nitric acid falls into two regimes of behaviour which depend on the overall transfer rate. The critical dissolution rate is approximately $8 \text{ mg. cm.}^{-2} \text{ min.}^{-1}$, its precise value depending upon the solvent composition. Below this critical value there is a large degree of chemical kinetic control, the dissolution rate being proportional to the cube of nitric acid concentration, the experimental activation energy being $15 \text{ kcal. mole}^{-1}$. Above the critical value the process appears to be controlled by the diffusion of a nitric acid species as the transfer rate is directly proportional to acid concentration with an activation energy of about $4 \text{ kcal. mole}^{-1}$.

In addition, the reaction is autocatalysed by the nitrite product so that forced convection can reduce rates by a factor of four times, and nitrite neutralisers can reduce them by a factor of one hundred times.

To a first approximation the dissolution rate depends simply on the total nitrate concentration although polyvalent nitrates and, in particular, ferric nitrate, tend to accelerate the reaction.

Introduction

The present work was undertaken to provide basic data for the design of plant for the continuous dissolution of uranium oxide fuel in nitric acid. Much information is available on the dissolution of this material¹⁻⁵ but the rates are often based on the time required for dissolution of 50% to 100% of a fuel pellet. For the design of continuous dissolvers an instantaneous reaction rate related to a definite surface area is required. The emphasis in the present investigation, therefore, has been to make weight-loss determinations over short time-intervals and to follow the change of effective surface area of a pellet as dissolution proceeds.

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A feature of the work is the number of individual determinations made. On each run the mean of six pellet-dissolution rates was taken because of the marked variation in behaviour of individual pellets.

Experimental

Materials used

The uranium dioxide was obtained in the form of sintered pellets, details of which are given in Table I. Pellets of batches 2—5 were fabricated from Grade 'A' oxide made by the calcination and reduction of ammonium diuranate which had been precipitated from a uranyl nitrate solution containing 150 g. of uranium/l. Pellets of batches 6 and 7 were made from Grade 'B' oxide which had a similar history except that the precipitating solution had only 60 g. of uranium/l. Reagents used were of A.R. quality except for a few of the nitrate additives which were of laboratory reagent standard. Nitric acid was always taken from freshly opened bottles.

Table I

Details of types of uranium dioxide used

Batch no.	Description	Final density, g./c.c.	Final composition
1	Non-stoichiometric sintered pellets	9.9 — 10.6	UO _{2.15} (approx)
2	Grade 'A' sintered pellets	10.4 ± 0.2	UO _{2.004} ± 0.003
3, 4, 5	Grade 'A' sintered pellets	10.4 ± 0.2	UO _{2.004} ± 0.003
6	Grade 'B' sintered pellets	10.6 ± 0.1	UO _{2.004} ± 0.003
7	Grade 'B' sintered pellets	10.74 ± 0.05	UO _{2.004} ± 0.003
8	Fused UO ₂ fragments	10.98	Variable

Apparatus

The dissolution cell consisted of a stainless steel beaker of 11 cm. dia. by 20 cm. high which was run with 700 ml. of acid to give a large excess of solvent. Liquor temperature was measured on a mercury-in-glass thermometer and control was maintained by immersing the beaker in an oil thermostat bath.

In unstirred runs the pellets were placed on their curved sides within each of six compartments of a stainless steel tray. Slots were put through each compartment wall to facilitate drainage and thus permit circulation by natural convection. The beaker was sealed by a polythene disc fitted with a reflux condenser for high-temperature runs.

For stirred runs the pellets were mounted on the six blades of a stainless steel paddle at a mean diameter of 1.3 in. An open cagework holder was used to give minimum shielding of the pellet surface from the acid. The paddle was rotated at controlled constant speed and formation of vortices in the acid was prevented by stainless steel radial baffles. Partial sealing was accomplished by a polythene collar on the paddle shaft which fitted within $\frac{1}{16}$ in. of an outer polythene lid.

Procedure

All pellets were first washed in acetone to remove adhering dust and traces of grease from the grinding operating and then thoroughly dried in air. It was then easy to detect surface imperfections and any faulty pellets were discarded. For every run, six pellets were each weighed to ± 0.1 mg., their lengths and diameters measured to ± 0.001 in. and these values used to calculate superficial areas and bulk densities.

In the case of a stirred run the motor was set to the required speed and the paddle connected to the drive so that rotation could be started immediately after immersion of the sample. At the end of the run the pellets in their container were plunged into two successive water-baths to stop any further dissolution, and finally into a beaker of acetone to remove surplus water. The precise temperature of the acid solution was noted at the beginning and the end of the run and the average taken.

After being dried for at least 2 h. at not more than 90°, the pellets were reweighed and the dissolution rate, per unit superficial area, for each pellet calculated. The average of the six dissolution rates was then taken.

To obtain point values of dissolution rate, pellets with surfaces ground smooth were used

and not more than 2% (usually 1%) of pellet dissolved. In this way changes in surface area were minimised so that the initial area could justifiably be used in calculating values of the mass flux. The change in surface area during overall dissolution was followed by making step-wise dissolutions of homogeneous pellets in fresh acid and estimating the available interfacial area from the rate of loss of weight.

Results

(1) Dissolution in nitric acid alone

(a) Unstirred runs

Influence of acid concentration and temperature

The results show that, for a given type of pellet, an increase in acid concentration or an increase of temperature below the boiling point leads to an increase in dissolution rate.

Non-stoichiometric oxide, such as the batch 1 pellets, dissolve 2-5 times as fast as stoichiometric material of the same density. In many cases the non-stoichiometric pellets crumbled before removal from the dissolution bath.

Results on pellets from batches 3-7 were much more consistent than those from the earlier batches, they are plotted in the graphs of Figs. 1-3. The dependence of dissolution rate upon temperature is shown in Figs. 1 and 3 in the form of Arrhenius plots. For unstirred dissolution rates below $4 \text{ mg. cm.}^{-2} \text{ min.}^{-1}$ at temperatures up to 95° , the plots are linear and have slopes which are essentially independent of acid concentration and are not very sensitive to the type of pellet used. These slopes represent experimental activation energies in the range $14.8 \pm 1.3 \text{ kcal. mole}^{-1}$. Above 95° the only temperature employed was the boiling point, the value of which depended on the solvent in use. The dissolution rate with boiling is typically a half to one-fifth of that expected at the same temperature in the absence of boiling. At the higher dissolution rates an abrupt change of slope occurs in the Arrhenius plots and the experimental activation energies are in the range 2-5 kcal. mole⁻¹. The point of change depends on the solvent, ranging between a rate of about $4 \text{ mg. cm.}^{-2} \text{ min.}^{-1}$ for 6 N-acid and about $11 \text{ mg. cm.}^{-2} \text{ min.}^{-1}$ for 14 N-acid. Dissolution rates for the batch 7 pellets (Fig. 3) did not reach these levels and show no breaks in the Arrhenius plots.

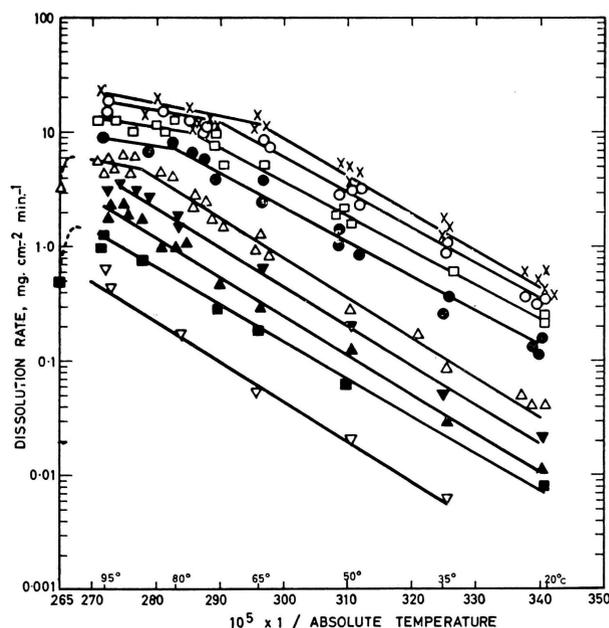


Fig. 1. Arrhenius plot of dissolution rates for ground Grade A oxide pellets
Concn. of nitric acid

× 14 N ○ 12 N □ 10 N ● 8 N
△ 6 N ▼ 5 N ▲ 4 N ■ 3 N △ 2 N

Points above 95° represent boiling reflux conditions

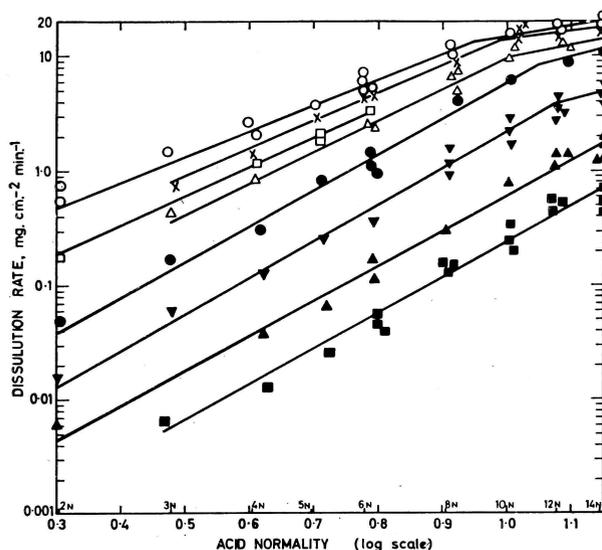


Fig. 2. Plots of dissolution rates vs nitric acid normality for Grade A oxide pellets

Temperature, °C ○ 95 × 85 □ 80 △ 75
 ● 65 ▼ 50 ▲ 35 ■ 20

Logarithmic plots of dissolution rate versus acid normality are given for Grade 'A' oxide pellets in Fig. 2. At rates below 4 mg. cm.⁻² min.⁻¹ the plots are linear with slopes in the range 2.3—3.3, but above this rate, at acid concentrations greater than 10 N, the slope in all cases falls to a value of approximately 1.0. The slopes of the main curves decrease progressively with increasing temperature. With Grade 'B' pellets it was found that the dependence upon temperature and nitric acid concentration was very similar to that for Grade 'A' material.⁶

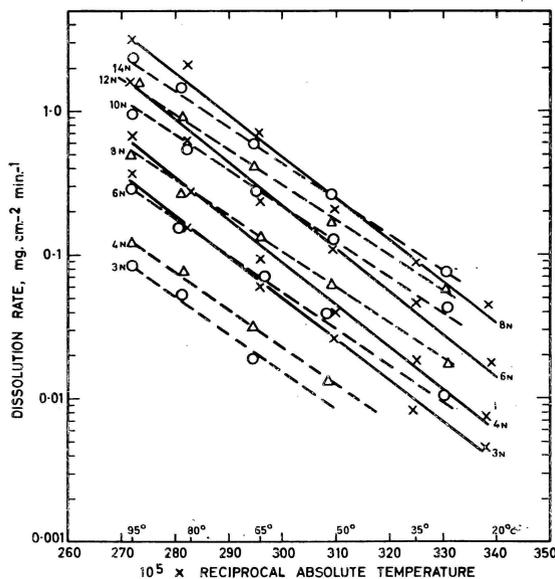


Fig. 3. Arrhenius plot for Batch 7 pellets in nitric acid

× ——— × } HNO₃ alone, unstirred in dissolving tray
 ○ ——— ○ } " " " " " " " " " " " "
 △ ——— △ } " " " " " " " " " " " "

Effects of pellet density

Summarising the results of 60 individual pellet dissolutions, it was found that the dissolution rate increased with decreasing pellet density in the following manner:

d 10.72 g./c.c.	gave a dissolution rate of	0.60 mg./cm. ² /min.	\pm 5%
d 10.64 "	" " " "	1.0 "	\pm 15%
d 10.56 "	" " " "	2.6 "	\pm 20%
d 10.50 "	" " " "	3.5 "	\pm 21%

(The variation in dissolution rate is expressed as the coefficient of variation, or fractional standard deviation.)

In contrast to this sensitivity to pellet density, it was found that fused oxide of d 10.98 g./c.c. had essentially the same dissolution rate as sintered oxide of d 10.74 g./c.c. However, uranium dioxide starts to dissociate above 1600° and the fused oxide contains metal inclusions and excess oxygen which probably give rise to its somewhat porous appearance after treatment with nitric acid.

Effect of changes in surface area

Fig. 4 shows how the weight loss rate varies through the period of total dissolution. Pellet homogeneity was checked by measuring dissolution rates of specimens reground to smaller diameters and it was demonstrated that dissolution rates were essentially constant with a coefficient of variation of 2%.

In general, the rate of weight loss rises to a maximum at about 20% of total dissolution and then decreases, passing through its initial value at about 60% total dissolution. The results for batch 7 material fall on a separate lower curve.

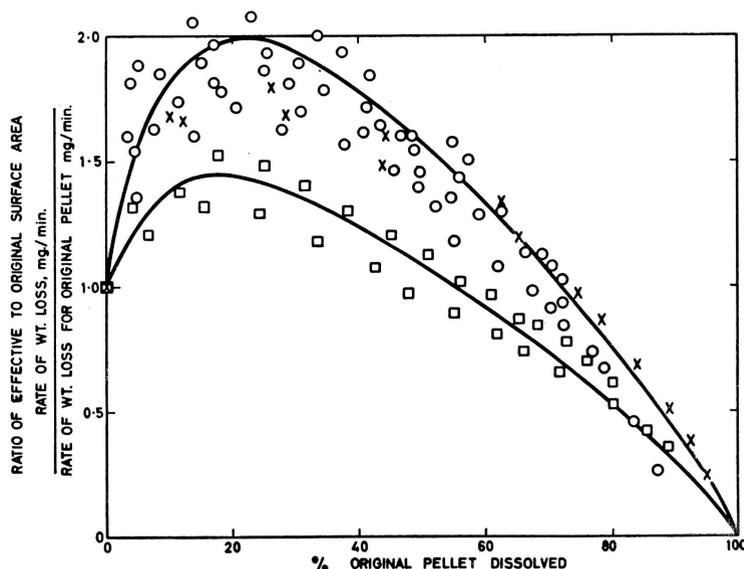


Fig. 4. Effective surface area vs % dissolution

× Grade A ○ Grade B, Batch 6 □ Grade C, Batch 7
Range of HNO₃ concn. 6–14N. Temp. 80–90°

(b) Stirred runs

The curves in Fig. 5 show that the dissolution rate decreases with increase of stirring speed up to about 600 r.p.m., after which further increase in stirring speed has no effect.

The results of a complete set of runs on batch 7 pellets at a stirring speed of 1500 r.p.m. are plotted together with the results of unstirred runs in Fig. 3. Rapid stirring is seen to reduce the dissolution rate by a considerable amount, typically a factor of 4, and the dependence on temperature appears to be slightly less than for natural convection (equivalent to an activation energy of 12 kcal. mole⁻¹ compared with 13.5 kcal. mole⁻¹).

Bubbling oxygen through the solution decreases the rate of attack to an extent similar to that for mechanical agitation, the limiting rate being reached on passing $1\frac{1}{2}$ l. of gas/min. through the dissolver pot. In a stirred system, the addition of an oxygen purge appears to accelerate slightly the dissolution process. A few exploratory tests with ultrasonic agitation of fixed frequency and energy (40 kc/s at 1 Wcm.^{-2}) again gave reduced dissolution rates.

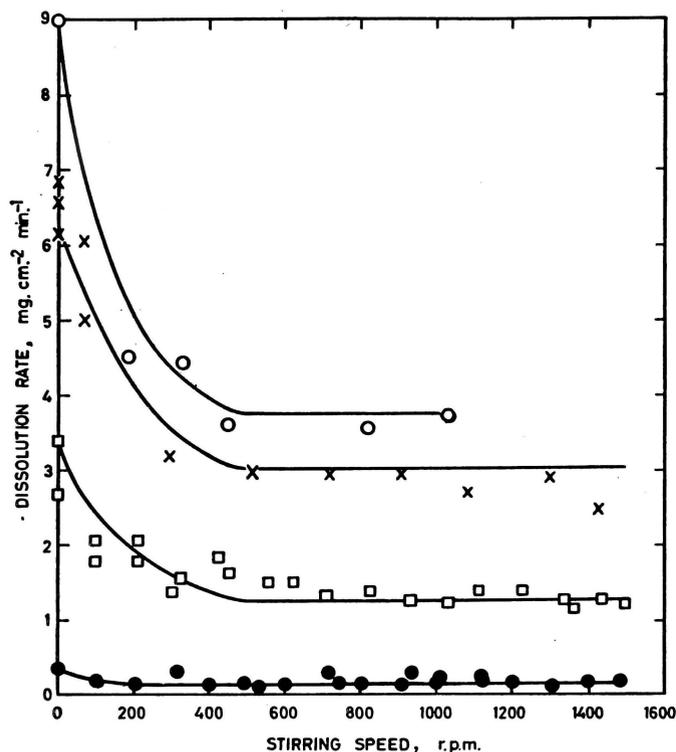


Fig. 5. Dissolution rate vs stirring speed for Grade A pellets

○ 80°, 6N-HNO₃ 2N in uranyl nitrate
 × 95°, 6N-HNO₃ □ 80°, 6N-HNO₃ ● 50°, 6N-HNO₃

(2) Dissolution in nitric acid with added reagents

(a) Unstirred runs

Fig. 6 demonstrates in a graphical form some results showing the effects of making additions of uranyl nitrate and other salts to the nitric acid solvent. The dissolution rate, relative to that in nitric acid, is plotted against the concentration of additive given in terms of nitrate normality or in terms of molarity, as indicated.

The effect of additions of monovalent nitrates is the same as for the corresponding amount of nitric acid over most of the range. In the case of lithium nitrate, 90% of the acid can be replaced by metal salt with no appreciable change in dissolution rate, but with no free acid the rate falls to a negligible value. Ammonium nitrate is exceptional and actually inhibits dissolution.

The influence of polyvalent metal nitrates is less simple. Ferric nitrate produces an increase in dissolution rate above that expected from the nitrate ion alone over the whole range of composition. Aluminium nitrate gives enhanced dissolution over most of the range, but in the absence of nitric acid the rate falls to one-third of that for acid alone. Up to a certain point, nitric acid can be replaced by uranyl nitrate (e.g. 6 N-acid plus 2 N-salt) with little effect, but the difference in rates between 6 N-acid and 3 N-acid plus 3 N-salt can be appreciable. Thus at low temperatures the uranium-bearing solution has the lower rate of attack but at temperatures above 50° it has the higher rate, so that the experimental activation energy is increased to 16 kcal. mole⁻¹.

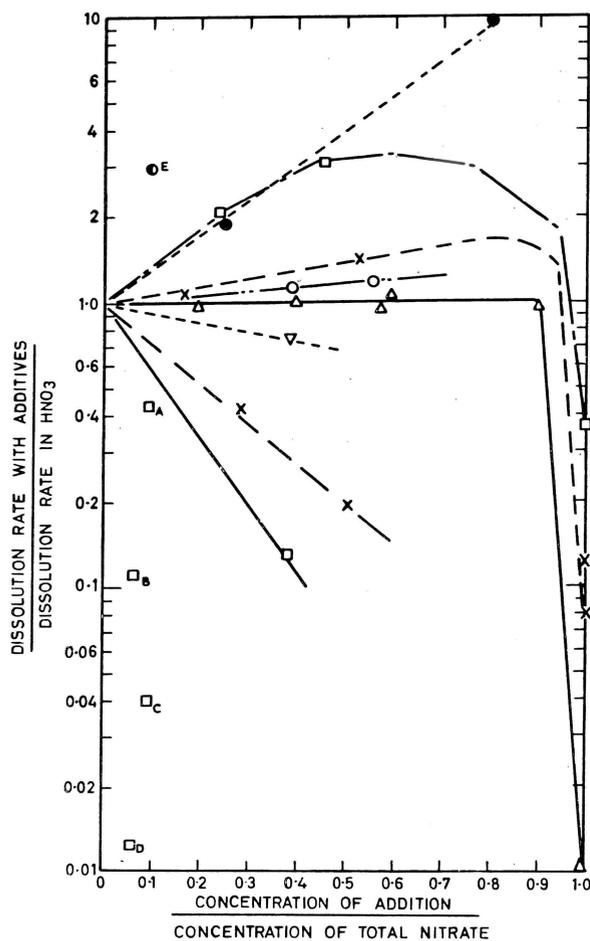


Fig. 6 Effect of various additives on dissolution rate in nitric acid (all not stirred unless specified, when rate was >600 r.p.m.)

● --- ● 2 N- Fe^{3+} □ --- □ 2 N- Al^{3+}
 × --- × 6 N- $\text{HNO}_3 + \text{UO}_2^{2+}$ at 95°
 ○ --- ○ 2 N-uranyl acetate
 △ --- △ 6 N- $\text{NO}_3^- + \text{Li}^+$ or Na^+
 ▽ --- ▽ 2 N-Na acetate □ --- □ 5 N- NH_4^+
 × --- × 3 N- UO_2^{2+} stirred
 □ A 0.5 M-urea stirred C 0.5 M-urea not stirred
 B 0.3 M-hydrazine stirred D 0.3 M-hydrazine not stirred
 ● E 0.5 M- NaNO_3

Ordinate, for comparable conditions and same total nitrate ion normality

Abscissa, molarity or nitrate ion normality, as indicated

The small positive effect of uranyl acetate is probably due to enhancement by uranyl and inhibition by acetate ions, since sodium acetate retards dissolution slightly, perhaps by virtue of its buffering action.

(b) Stirred runs

The decrease in dissolution rate on stirring is similar for solvent containing uranyl nitrate to that for acid alone (see above), and Fig. 5 shows that curves for the two types of solvent follow essentially parallel courses with increasing agitation.

Some results plotted in Fig. 6 give an indication of the extent to which additions of sodium nitrite and nitrite-neutralisers influence the dissolution process. Addition of 0.5 M-nitrite to 6 N-acid can increase the transfer rate threefold, while the addition of 0.5 M-urea can reduce the rate 3 times under forced convection and 16 times under natural convection. Hydrazine nitrate at 0.3 M concentration can reduce the rate 10 times in stirred solvent and nearly 100 times under conditions of natural convection. Hydrogen peroxide has little depressing action on the reaction although it is reported to slow down dissolution of copper in nitric acid by virtue of nitrous acid removal.

Mixtures of accelerators such as nitrites and polyvalent nitrates with nitrite-neutralisers generally gave results considerably lower than pure acid solvent.⁶ In the case of ferric nitrate, however, the reduction is comparatively small, probably because of the separate oxidising effect on uranium oxide as suggested by Nesmayanova.⁷ Banerji & Dhar⁸ reported that ferric salts act as catalysts in the dissolution of metals in nitric acid, it being suggested that ferric ion is reduced by metals to ferrous ion which then produces nitrous acid from the nitric acid.

Discussion of results

Pellet properties

The main curves of Figs. 1—3 together with very similar data for Grade 'B' pellets⁶ show that the dependence on temperature and nitric acid concentration does not change with pellet properties.

Bircumshaw & Riddiford⁹ point out that, for chemically-controlled reactions, the rate is a function of the true surface area, whereas for transport-controlled processes the rate is proportional to the apparent surface area for both forced and natural convection (see below). In principle this may be axiomatic, but for a typical practical case, where surface features range from shallow pitting to deep cracks and minute pores, the notion of a 'true' surface area is of doubtful utility. It is apparent that in very fine pores some diffusional control should always exist.

As dissolution rates above about 4 mg. cm.⁻² min.⁻¹ are transport-controlled (see below) they might be expected perhaps to be quite independent of pellet properties, but with coarse pitting the apparent surface area will probably vary to some limited extent. Although the difference between batches is relatively small (a factor ≈ 1.8) under these conditions, nevertheless it is a real one. Thus in Fig. 4 the results for batch 7 pellets fall on a curve distinct from that of other pellets in spite of the scatter resulting from the inclusion of results from both transport-controlled and kinetic-controlled systems.

Porosity of uranium dioxide pellets has been extensively studied¹⁰ and found to be related to density in the following manner. With increasing density up to about 10 g./c.c. the number of open pores decreases while the number of closed pores increases. Above this density, there are very few open pores and the closed pores, at first irregular in shape, become more compact. The dissolution rate for an oxide pellet with small discrete pores fairly regularly distributed will depend closely upon its density. Such a state of affairs could reasonably be assumed for a pellet of density 10.6 g./c.c. or greater. The abnormally high values of dissolution rate for bodies of lower density probably reflect some interconnection of pores.

As the pellets were shown to be reasonably homogeneous throughout, the curves in Fig. 4 represent the change in effective surface area as dissolution proceeds. This change appears to be relatively independent of acid concentration and temperature, but density would be expected to have some effect. Thus batch 7 pellets with densities ≥ 10.7 g./c.c. fall on a lower curve than those having densities between 10.3 and 10.6 g./c.c. In similar work at Oak Ridge⁴ pellets of density 10.35 g./c.c., when 35% had been dissolved, had effective surface areas four times as great as the initial areas.

Variation of experimental conditions

The main portions of the Arrhenius plots have slopes equivalent to experimental activation energies of about 15 kcal. mole⁻¹, a value which lies in the range exhibited by catalysed and heterogeneous chemical reactions although an appreciable component of diffusional control could be present. It is of interest that Lacher & Salzman¹¹ report similar values for the dissolution of uranium metal in stirred nitric acid.

At the higher dissolution rates the activation energies fall to between 2 and 5 kcal. mole⁻¹ which may be compared with the value of 4 kcal. mole⁻¹ for the energy of diffusion for many electrolytes in aqueous solution. It is, therefore, suggested that, above a certain limiting rate, the process is diffusion controlled and that below the limit there is a large degree of chemical kinetic control (see below for the influence of nitrous acid diffusion). The limiting rate is in the region of 4 mg. cm.⁻² min.⁻¹, its precise value depending upon the solvent composition. In the overall transfer process the mass flux, identical with the dissolution rate per unit area of the pellet envelope, would be expected to become diffusion-controlled above a certain value because of the lower activation energy for the transport process.

This interpretation is supported by the dependence on nitric acid concentration. At the lower rates, the dissolution rate is proportional to the nitrate concentration raised to the power 2.8 ± 0.5 , the precise value depending upon the temperature. This high degree of dependence on nitrate concentration strongly suggests chemical control. But beyond certain limits, corresponding to the breaks in the Arrhenius plots, the dependence becomes first-order. It, therefore, seems likely that the controlling factor at high rates is the diffusion of nitric acid or nitrate ion.

Above it was shown that rates of dissolution are strongly dependent on nitrite concentration in the solvent and since nitrous acid is a dissolution product it follows that the reaction is autocatalytic. Nitrous acid is known to play a similar rôle in the dissolution in nitric acid of a number of metals¹² including uranium.¹¹ Under conditions of low dissolution rate, at least, it is apparent that the reaction does not behave as a diverging chain reaction but operates in the quasi-stationary regime¹² as would be expected with complete kinetic control. A marked reduction in dissolution rate on increasing the degree of agitation is typical of this type of autocatalytic process.

The direct dependence on total nitrate concentration, to a first approximation, confirms the findings of previous workers.^{1, 3} Catalytic effects of the dissolution products have been attributed to the presence of uranyl nitrate¹³ and in the present work it was observed that, at low temperatures, a yellow mantle resembling uranyl nitrate forms around the pellets and is progressively dispersed with increasing agitation. At higher temperatures the layer has a darker colour. Moreover, uranyl acetate increases the dissolution rate slightly despite the inhibiting action of the acetate ion, and uranyl ion itself appears to have a positive effect at higher temperatures. Nevertheless, the present results on the quantitative effects of the various additives clearly indicate that nitrite ion is the key autocatalytic agent. Uranyl ions may have a second-order effect by influencing the nitrous acid concentration at the interface.

When the transport conditions are changed, by altering the agitation or the solvent viscosity or by some other means, the diffusion of nitrite will change and the concentration at the reacting interface will move to some other quasistationary value. It would therefore, appear that if nitrite concentrations were altered markedly, for example by operating under appreciable hydrostatic head or by air sparging, the internal relationships shown in Figs. 1 and 2 would, in general, still hold, but the absolute values of dissolution rate would be shifted by a factor depending on the change in interfacial nitrite concentration.

Such a case is illustrated in Fig. 3, although a slight decrease in activation energy does seem to have been introduced by stirring. In an uncatalysed reaction any effect of stirring would be to accelerate the process and also to increase the activation energy. In the present case, however, increased diffusion rate lowers the local concentration of nitrite and the dissolution rate. Decreases in the nitrite concentration might well have a greater overall effect at higher temperatures and thereby give rise to a lower activation energy for the stirred system and to the relationships shown in Fig. 3.

Acknowledgments

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