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AND DEVELOPMENT PARAMETERS FOR SODIUM C.P.
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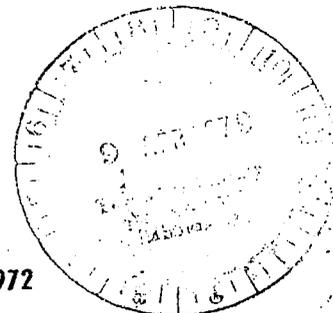
DEVELOPABLE IMAGES PRODUCED BY X-RAYS USING THE NICKEL-HYPHOPHOSHITE SYSTEM

II — Exposure and Development Parameters for Sodium Hypophosphite

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DEVELOPABLE IMAGES PRODUCED BY X-RAYS USING THE
NICKEL-HYPOPHOSPHITE SYSTEM
II - EXPOSURE AND DEVELOPMENT PARAMETERS
FOR SODIUM HYPOPHOSPHITE

by Charles E. May, Warren H. Philipp, and Stanley J. Marsik
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SUMMARY

Crystalline sodium hypophosphite was X-irradiated and then treated with an ammoniacal nickel hypophosphite solution. Treatment (development) resulted in the precipitation of nickel metal. The yield of nickel metal varied directly with particle size, sample weight, X-ray voltage, target current, exposure time, and development time. These findings show the process to be potentially useful in X-ray type photography (radiography and diffraction). The half-life for the latent image species was found to be relatively short (days); but this is not critical in most X-ray photography applications. Furthermore, our work can be interpreted on the basis that a hydrogen atom is involved in the mechanism and indicates that the autocatalytic development step may be self-poisoning.

INTRODUCTION

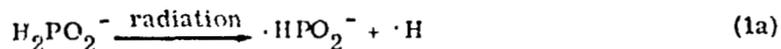
Current research at the Lewis Research Center led to the discovery of a photographic type process based on nickel (ref. 1). It resembled the common silver halide photographic process in that a latent image formed by irradiation could be amplified by development to produce a visible image of wide tonal gradation in the gray scale. Our photographic type emulsions contained nickel hypophosphite, and although they were not sensitive to visible light, they were sensitive to X-rays and electron radiation. This lack of sensitivity to visible light can be a distinct advantage in radiography and diffraction in that special lightproof cassettes and darkrooms are not necessary. A second advantage is that more abundant and less expensive nickel replaces the silver in the con-

ventional photographic emulsions. As yet the nickel hypophosphite emulsion is not as sensitive as the conventional photographic materials to X-rays: the exposure time for a good nickel image is about 5 minutes at about 20 000 roentgens per minute with a tungsten target tube at 250 kilovolts.

Further work (ref. 2) revealed that at least five salts in addition to nickel hypophosphite are potentially usable in emulsions for our X-ray sensitive and developable process. These are sodium hypophosphite, ammonium hypophosphite, lithium hypophosphite, sodium phosphite, and nickel formate. In each of these cases, the anion (HA^-) contains a covalently bonded hydrogen. This has led us to postulate (ref. 2) a mechanism for the overall process. According to this mechanism, hydrogen atoms are formed during the irradiation step as follows:

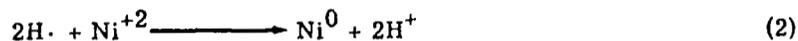


For example,



The hydrogen atoms react with the matrix to form a reducing species; this reducing species makes up the latent image. However, for simplicity, we refer to the reducing species as a "trapped hydrogen atom." The occurrence of reaction (1) is supported by electron paramagnetic resonance studies which have identified the various anion radicals ($\cdot\text{A}^-$) (refs. 3 to 5), but the trapped hydrogen atom has not been detected.

During the development, the trapped hydrogen atom reduces the nickel ion (Ni^{+2}) in the developer to nickel metal (Ni^0), with the formation of hydrogen ions (H^+):



The minute amounts of nickel metal thus formed then initiate the autocatalytic reduction of nickel ion by hypophosphite ion (ref. 2). In this reaction, the hypophosphite is oxidized to phosphorous acid (H_3PO_3):



Macroamounts of nickel metal are produced in the amplification reaction (3), and it is this metal that makes up the visible image.

The purpose of this study was to determine the effect of various irradiation and de-

velopment parameters of the overall process in order to gain a greater understanding of the process. The 10 parameters studied were particle size, sample weight, X-ray voltage, target current, irradiation time, time lapse between irradiation and development, amount of developer, concentration of developer, time of development, and amount of agitation during development. For simplicity, a crystalline salt was used rather than an emulsion; the salt chosen was sodium hypophosphite. The developer was an ammoniacal nickel hypophosphite solution. The yield of nickel metal was determined gravimetrically; this yield was taken as a measure of the extent of reaction and would be indicative of the image density formed in a corresponding X-ray photograph. The results are discussed in terms of the mechanism and the use of the system in X-ray photography.

PROCEDURE

The effects of the 10 radiation and development parameters were studied by using the general procedure described in this section and varying only one parameter at a time. However, to verify the results, two different sample weights (0.05 and 0.10 g) were used in the study of each parameter.

Fisher certified grade sodium hypophosphite ($\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$) was used, and the general procedure was similar to that described previously (ref. 2). A weighed quantity (0.05 or 0.10 g, both ± 5 percent) of the as-received salt was placed in an aluminum planchet (having a 6-mm-deep, 12-mm-diam cavity). A tungsten target X-ray tube was used for irradiation, the standard conditions being 250 kilovolts, 10-milliampere target current, and 30-minute exposure at about 10 centimeters from the target. This dose corresponded to about 400 000 roentgens, uniform over the planchet area.

Within several minutes after irradiation the sample was added with no agitation to 2.00 ± 0.05 cubic centimeters of developer at room temperature. It consisted of 3 normal ammonium hydroxide (NH_4OH) containing 4 percent sodium hypophosphite ($\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$), 4 percent nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$), and 4 percent ammonium chloride (NH_4Cl) as a buffer. This developer was found to be stable, and the same batch of stock solution was used during this entire 5-month study. During the development the irradiated salt dissolved, and nickel metal slowly precipitated. The presence of the metal could generally be detected within 15 minutes from the start of development. The mixture of irradiated salt and developer was allowed to sit overnight to make sure ample time was provided for development. The metallic deposit was then filtered into a weighed crucible, washed, dried at 120°C , and weighed. The metal was shown to be essentially nickel metal by spectroscopic analysis and X-ray diffraction.

RESULTS AND DISCUSSION

The effects of the various parameters are best illustrated graphically. In figures 1 to 10 the yield of nickel is given as a function of the parameters being varied. The scatter of the data is greater than we had anticipated. Nevertheless, the scatter does not obscure the general trends of the dependence of nickel yield on the parameters studied. The reason for the scatter in the data is not understood at this time.

In many experiments, conditions were such that all the nickel present in the developer was reduced. In these cases the yield of metal would be essentially equal to the nickel originally present in the 2 cubic centimeters of developer used (0.0204 g/2 cm³ by gravimetric analysis). In all the figures, 1 to 10, such "exhausted developer regions" are indicated by dashed lines. Because no significant dependence of the yield on the various parameters can be expected in these regions, no discussion of such data is presented.

Particle Size

The two histograms in figure 1 show the yield of nickel metal for samples of various particle sizes. Three different samples were used: a powder (0.05 mm) obtained by grinding, crystals (2 mm) obtained by recrystallization, and the as-received salt (0.2 mm). In figure 1(a), the yield of nickel from the 0.05-millimeter size irradiated powder appears to be less than that from coarser samples. The results are indicative of the dependence on particle size one finds in the silver halide photographic system; that is, finer grains are less sensitive than larger ones. Substantiation of this conclusion would necessitate the use of a still finer particle size sample. The data in figure 1(b) are in the exhausted developer region.

Sample Weight

In figure 2 the solid line shows the yield of nickel to increase monotonically with the sample weight. Because the number of trapped hydrogen atoms present should be proportional to the sample weight (assuming uniform radiation flux), we interpret the data to mean that the yield varies directly as the number of trapped hydrogen atoms. Thus, a particular yield of nickel metal corresponds to a specific number of trapped hydrogen atoms. This implies that the autocatalytic step, reaction (3), does not proceed indefinitely. Instead, the deposited nickel eventually loses its catalytic activity as the deposit grows. In other words, the deposit is poisoned. Because of this, radiographs using the

nickel-hypophosphite system have tonal gradations in the gray scale (ref. 1) instead of being merely black and white.

Even though figure 2 shows a well behaved dependence of nickel yield on sample weight, subsequent figures indicate unusual behavior. Under some conditions, a 0.10-gram sample acts as if it were an order of magnitude larger than a 0.05-gram sample; this is most apparent in figure 5 for about a 5-minute exposure, in figure 6, as discussed in the section Time Lapse Between Exposure and Development, and in figure 7 for 20 cubic centimeters of developer. At present we are unable to speculate about the exact cause of this unusual behavior; therefore, in subsequent sections we make no attempt to compare the results for 0.10-gram samples with those from 0.05-gram samples. However, we do suggest that such unusual behavior is more likely a result of the development step than a result of the irradiation.

Exposure Parameters: X-Ray Voltage, Target Current, and Time

The solid-line curves in figures 3 to 5 show that the yield of nickel metal increases monotonically with each of the exposure parameters: X-ray voltage, target current, and exposure time. On the upper abscissa of each of these figures is indicated the irradiation dose in roentgens (obtained by calibration); thus, for all three figures, the yield of nickel is essentially plotted as a function of irradiation dose. Because the yield is a measure of the number of "trapped hydrogen atoms," one can interpret all of these curves as merely showing a direct dependence of the number of trapped hydrogen atoms on the irradiation dose. The data in figure 5 further indicate that there may be an induction period involved in the process, as is known to exist in the conventional photographic process.

Time Lapse Between Exposure and Development

In figure 6 is plotted the yield of nickel metal as a function of the time lapse between exposure and development. Such a plot allows one to infer the "half-life" for the trapped hydrogen atom. The half-life for a 0.05-gram sample appears to be less than 12 hours, while that for a 0.10-gram sample appears to be about 24 days. Both values are quite short compared with that for the modern silver halide photographic process. Nevertheless, such a lifetime for a latent image is sufficiently long for most X-ray photographic applications (radiography and diffraction).

The large difference in the half-life between the irradiated 0.05- and 0.10-gram samples is unique. At present, no logical explanation for this is offered.

Amount of Developer

Figure 7 shows the yield of nickel as a function of the amount of the developer. The solid line (extending beyond 2.5 cm^3 for the smaller $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$ sample, 0.05 g) represents finality in development. The nickel deposit is incapable of catalyzing the reduction of any more nickel. Thus, the excess nickel present in a quantity of developer above 2.5 cubic centimeters is not deposited in the case of the smaller weight sample.

The dashed curves, in contrast, correspond to complete depletion (exhaustion) of the developer. All of the nickel present in the developer is deposited; thus, the yield of nickel is limited by the quantity of nickel in the developer. The larger $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$ sample (0.10 g) can use up more than 20 cubic centimeters of developer; this signifies the enormous chemical amplification obtainable by the development of a relatively few chemically trapped hydrogen atoms.

Developer Concentration

The effect of the developer concentration was investigated by diluting the stock solution with various amounts of water; the relative proportions of the active chemicals in the developer used were thus constant.

Figure 8 is a plot of nickel yield against developer dilution. Except for the dropoff in nickel yield as one approaches zero dilution for the smaller weight sample, the yield of nickel is constant over the entire range of the dilutions tested (zero to 20-fold dilution of the stock solution). This constancy of nickel yield is, of course, due to the fact that the total nickel content of the developer is being deposited. The drop in nickel yield at low dilutions (high concentrations) of developer may be due to catalytic poisoning by a higher hypophosphite concentration. In any case, this dropoff does indicate that lesser amplification may result from a too concentrated developer.

Development Time

Figure 9 shows the dependence of yield on development time with no agitation. For both sample weights, there is first a rapid increase in nickel yield extending to roughly 3 to 6 hours and then a much slower development rate. Our standard development time, overnight, is therefore more than adequate for complete development. The long development time found would indeed be expected to be a disadvantage when the method is used in X-ray type photography; however, in practice we found long development times are not necessary. Radiographs using a nickel hypophosphite emulsion require only a 5-minute development.

Agitation

Because the degree of agitation of a salt in a developer is difficult to measure, our experiment was limited to the two extremes: no agitation and a very vigorous manual agitation. This vigorous agitation for both sample weights caused complete dissolution of the irradiated salt within 15 seconds with no accompanying nickel deposition (fig. 10). The fact that no nickel forms on vigorous agitation is probably an effect of the rapid dissolution of the sodium hypophosphite; the same result (no nickel deposit) would be anticipated for the other highly soluble developable hypophosphites if vigorous agitation were used during their development. Rapid dissolution of such irradiated hypophosphites would be expected to allow the trapped hydrogen atoms to escape from the matrix before they would be able to produce sufficient catalytic nickel to initiate the autocatalytic development step, reaction (3). We believe that the trapped hydrogen atoms that are liberated from the crystals dissipate finally in the form of hydrogen gas.

In the development of irradiated nickel hypophosphite emulsions containing gelatin as a support, the degree of agitation is not crucial (unpublished data obtained at the Lewis Research Center). The gelatin support probably inhibited the dissolution of the hypophosphite, and thereby sufficient time was allowed for the active nickel sites to form.

CONCLUDING REMARKS

This study concerning the effect of various parameters on the nickel-hypophosphite developable system has provided insights into the use of such a system in X-ray type photography. The data indicate that this system possesses many of the same general characteristics that the silver halide one does. However, there are still some physical differences. For example, the common photographic emulsion differs from ours in that its irradiated salt (silver halide) is insoluble and the metal (silver) making up the visible image is derived from the irradiated salt.

A slight deficiency of our system brought to light in this report is the comparatively short lifetime of the latent image species. However, this does not impair its usefulness in most X-ray photographic applications (radiography and diffraction). The nature of this decay has not as yet been established.

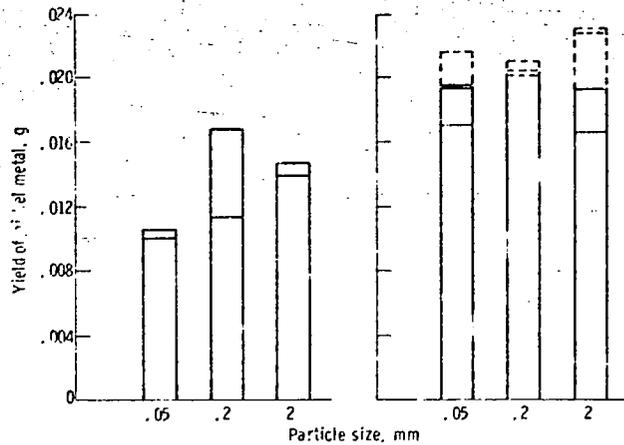
Although our work indicates that the nickel-hypophosphite system is usable in an X-ray photographic process, it raises many questions concerning the mechanism. The most apparent question concerns the tremendous effect that the weight of the irradiated sample has on the nickel yield and the half-life of the latent image. Another question concerns the effect of the dissolution rate on the yield of nickel (visible image). In spite of the fact that such questions have arisen, our findings are interpretable in terms of

our previously proposed mechanism (ref. 2) involving a chemically trapped hydrogen atom. Furthermore our present work indicates that a poisoning mechanism is operative in the autocatalytic deposition of the nickel. Apparently, the degree of poisoning is related to the developer concentration.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, January 20, 1972,
114-03.

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(a) 0.05-Gram sample.

(b) 0.10-Gram sample.

Figure 1. - Effect of particle size

Sample: $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$.
 Irradiation: tungsten target X-ray tube, 250 kV, 10 mA, 30 min.
 Developer: 3 N NH_4OH , 4% $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 4% $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 4% NH_4Cl .
 Development: 2 cm^2 of developer, sitting overnight, no agitation.
 Multiple samples run for each particle size; dashed portions of bars indicate exhausted developer region.

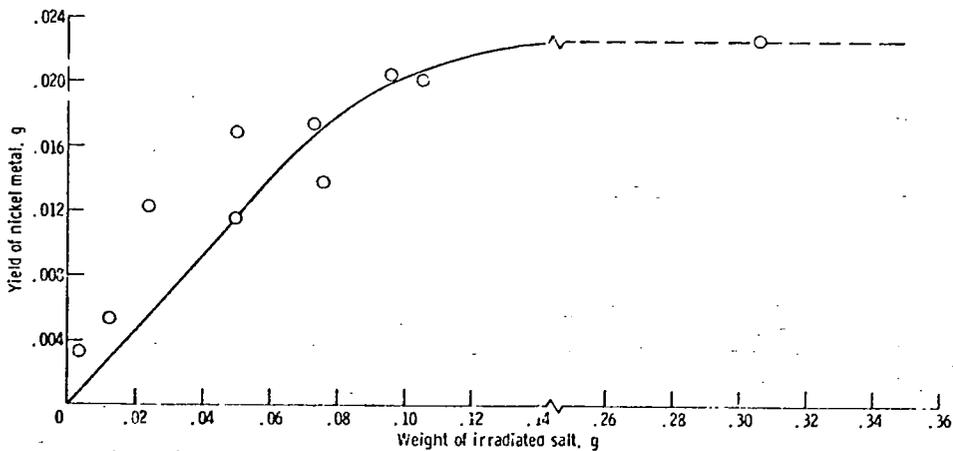


Figure 2. - Effect of sample weight.

Sample: $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 0.2-mm particle size.
 Irradiation: tungsten target X-ray tube, 250 kV, 10 mA, 30 min.
 Developer: 3 N NH_4OH , 4% $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 4% $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 4% NH_4Cl .
 Development: 2 cm^2 of developer, sitting overnight, no agitation.
 Dashed curve indicates exhausted developer region.

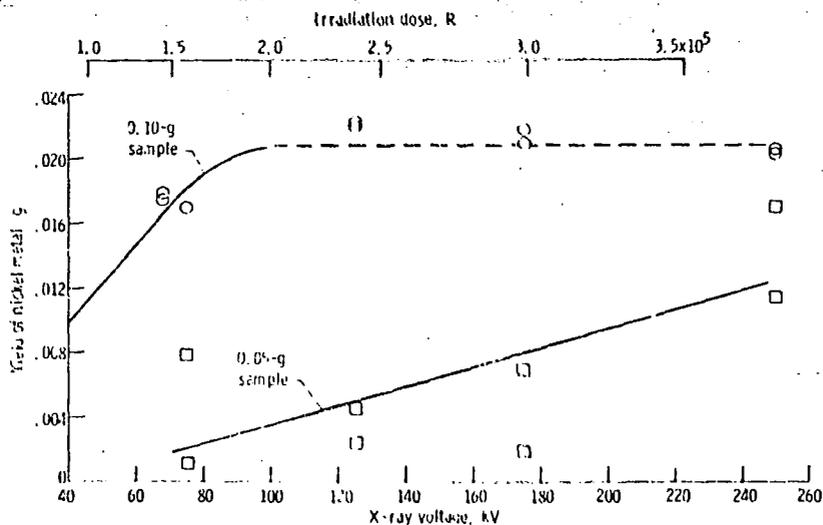


Figure 3. - Effect of X-ray voltage.

Sample: $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$, 0.2-mm particle size.
 Irradiation: tungsten target X-ray tube, 10 mA, 30 min.
 Developer: 3 N NH_4OH , 4% $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$, 4% $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 4% NH_4Cl .
 Development: 2 cm² of developer, sitting overnight, no agitation.
 Dashed curve indicates exhausted developer region.

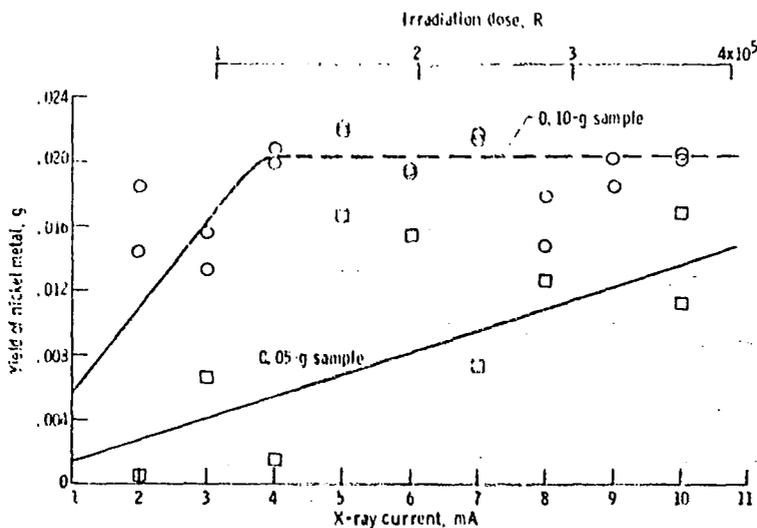


Figure 4. - Effect of target current.

Sample: $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$, 0.2-mm particle size.
 Irradiation: tungsten target X-ray tube, 250 kV, 30 min.
 Developer: 3 N NH_4OH , 4% $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$, 4% $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 4% NH_4Cl .
 Development: 2 cm² of developer, sitting overnight, no agitation.
 Dashed curve indicates exhausted developer region.

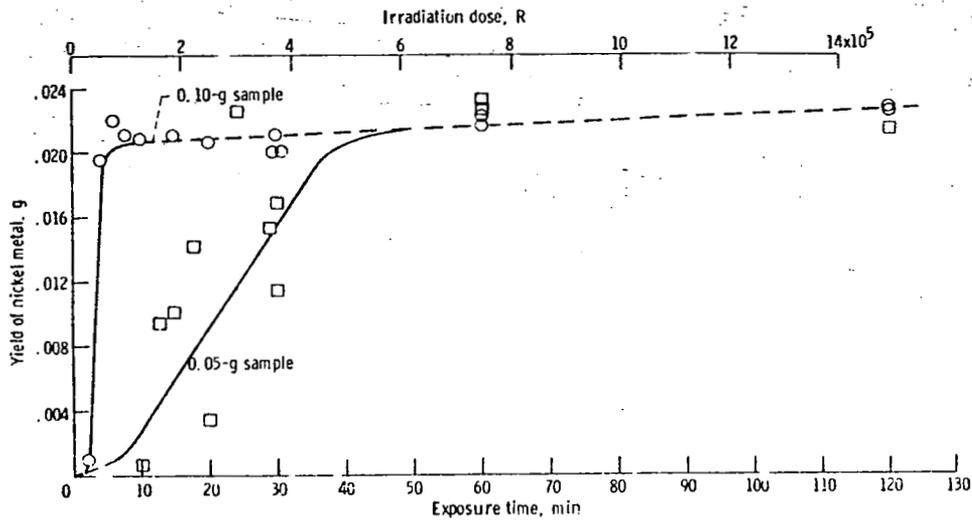


Figure 5. - Effect of exposure time.

[Sample: $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 0.2-mm particle size.
 Irradiation: tungsten target X-ray tube, 250 kV, 10 mA.
 Developer: 3 N NH_4OH , 4% $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 4% $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 4% NH_4Cl .
 Development: 2 cm³ of developer, sitting overnight, no agitation.
 Dashed curves indicate exhausted developer region.]

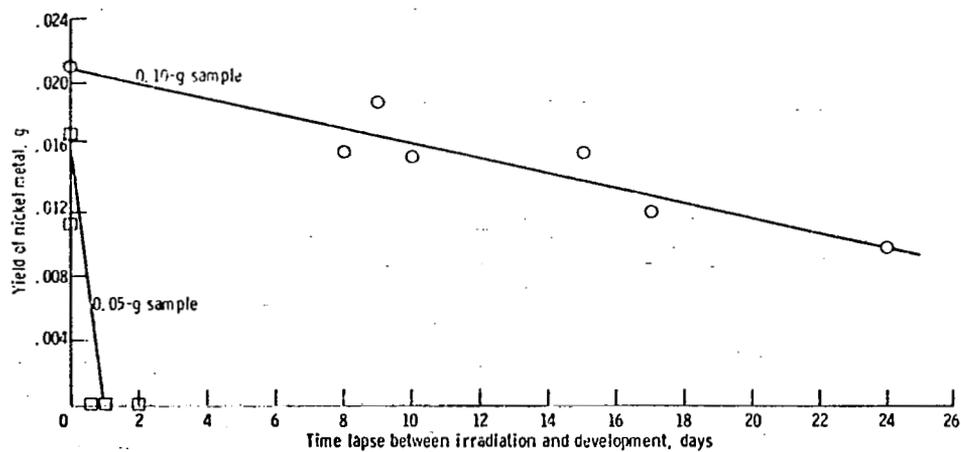


Figure 6. - Life of chemically trapped hydrogen atoms.

[Sample: $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 0.2-mm particle size.
 Irradiation: tungsten target X-ray tube, 250 kV, 10 mA, 30 min.
 Developer: 3 N NH_4OH , 4% $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 4% $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 4% NH_4Cl .
 Development: 2 cm³ of developer, sitting overnight, no agitation.]

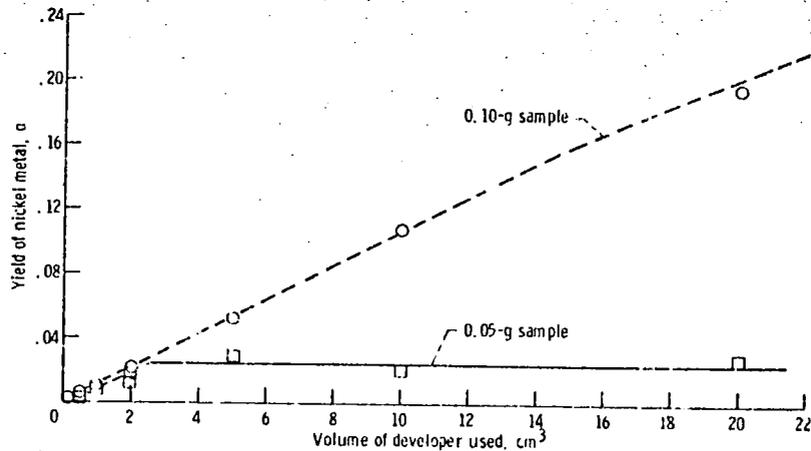


Figure 7. - Effect of amount of developer.

Sample: $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 0.2-mm particle size.
 Irradiation: tungsten target X-ray tube, 250 kV, 10 mA, 30 min.
 Developer: 3 N NH_4OH , 4% $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 4% $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 4% NH_4Cl .
 Development: sitting overnight, no agitation.
 Dashed curves indicate exhausted developer region.

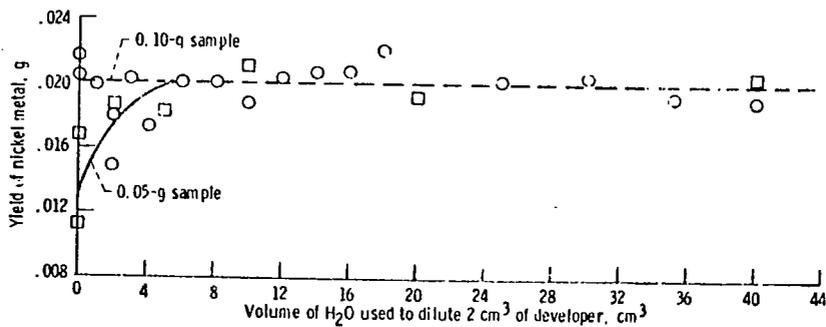


Figure 8. - Effect of diluting developer.

Sample: $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 0.2-mm particle size.
 Irradiation: tungsten target X-ray tube, 250 kV, 10 mA, 30 min.
 Developer: 3 N NH_4OH , 4% $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 4% $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 4% NH_4Cl .
 Development: 2 cm³ of developer, sitting overnight, no agitation.
 Dashed curves indicate exhausted developer region.

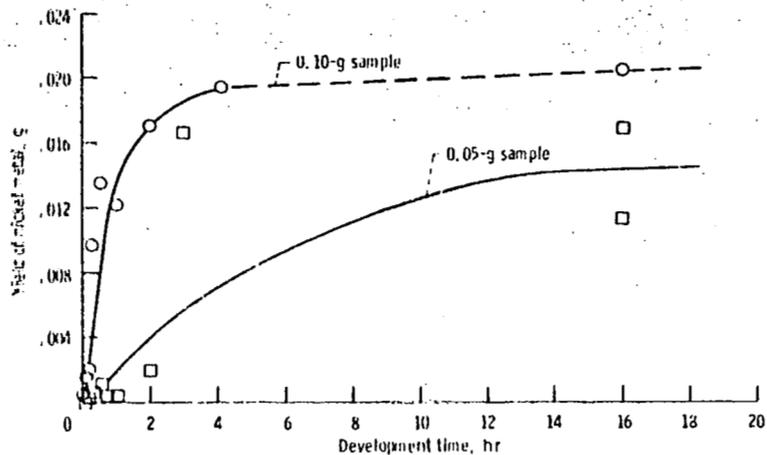


Figure 9. - Effect of development time.

Sample: $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 0.2-mm particle size.
 Irradiation: tungsten target X-ray tube, 250 kV, 10 mA, 30 min.
 Developer: 3 N NH_4OH , 4% $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 4% $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 4% NH_4Cl .
 Development: 2 cm^3 of developer, no agitation.
 Dashed curve indicates exhausted developer region.

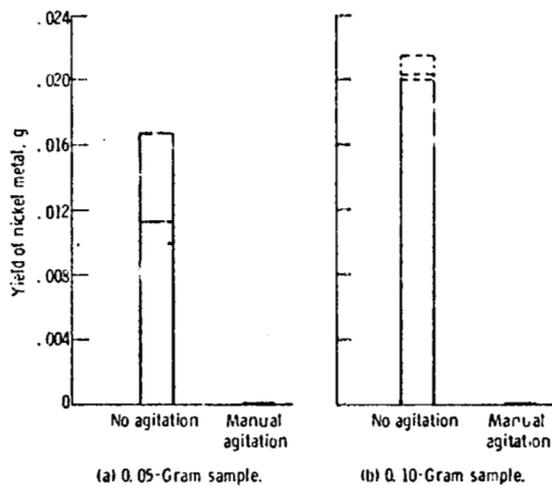


Figure 10. - Effect of agitation.

Sample: $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 0.2-mm particle size.
 Irradiation: tungsten target X-ray tube, 250 kV, 10 mA, 30 min.
 Developer: 3 N NH_4OH , 4% $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, 4% $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 4% NH_4Cl .
 Development: 2 cm^3 of developer, sitting overnight.
 Specimens agitated manually dissolved in 15 seconds; multiple samples run for each particle size; dashed portions of bar indicate exhausted developer region.

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