Procedure for Processing Nuclear-Track Emulsions*

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Procedures have been investigated for processing exposed Eastman NTB Nuclear Track Plates which have thicknesses in the range of (a) 100–175μ and (b) 200–260μ. Processing recommendations are given for developing essentially all tracks of ionizing particles, including electron tracks. The process consists in initiating development at low temperature and allowing it to go to completion at a higher temperature, checking development in a cold acid stop bath, and fixing (with nitrogen agitation) first at low and later at a higher temperature.

THE increase in the use of special photographic emulsions for recording ionizing particles has led to a demand for emulsion coatings of thickness considerably greater than that encountered in ordinary photographic use. In addition, such emulsions have a considerably higher ratio of silver halide to gelatin than is found in most photographic materials. These special characteristics have required that particular procedures for their processing be worked out, and some experimental work has been done to provide recommendations for processing conditions which bring out the desired records of nuclear particles.

The basic problem in the development of any photographic emulsion, which applies equally to the processing of what will be called, for convenience, "nuclear-track emulsions," is to develop the exposed emulsion grains without a corresponding development of the unexposed or fogged silver halide. The important factors which affect this differentiation are both chemical and physical, but for emulsions of relatively great thickness the competing rates are largely controlled by physical factors. These factors are the rate of diffusion of the reacting developing agent into the exposed emulsion and the simultaneous diffusion of the reaction products away from the developing grains. It is well known that the products of development markedly affect the rate of development of photographic emulsions. In working out conditions for processing thick emulsions of nuclear-track type, it is necessary to adjust the rate at which the developing agent is supplied to the emulsion grains against the rate at which the products of development diffuse from the layer. It is very important that both the top and the bottom of the emulsion layer have as nearly the same degree of development as it is possible to obtain, since differences in development in these two regions might be misinterpreted in analyzing certain characteristics of the tracks of ionizing particles such as grain density.

In addition to the problem of insuring uniform and satisfactory development, there is the problem of retaining suitable physical characteristics of the developed emulsion layer. The emulsion should not swell or shrink unduly during processing, nor should it undergo localized reticulation which would tend to distort the character of the tracks observed. It is known that factors which affect the swelling and shrinking of gelatin also affect both the adhesion to a support and the physical homogeneity of photographic emulsions. Factors which influence the swell and shrink characteristics of a gelatin layer are the pH, salt content, and temperature of the solutions in which it is in equilibrium. Thus, as a general principle, it is well established that in order to retain maximum dimensional stability, with consequent adherence to the supporting layer, rapid changes in pH, in salt content, and in temperature must be avoided as much as possible at all stages in processing.

The above generalizations apply to developers used in photographic processing, to "stop baths" which are employed to stop the action of the developer, to fixing baths which are used to remove the undeveloped silver halide, and to the washing operations which follow fixation.

Suitable conditions have been worked out for processing nuclear-track plates having thickness in the range usually found for normal photographic products, and there is considerable information on this in the literature. However, special problems arise when the emulsion thickness becomes greater than about 100 microns. When it became apparent that special processing conditions would be needed for processing thick nuclear-particle plates, a program was undertaken in the Kodak Research Laboratories to work out the best recommendations for those using such special emulsions supplied by the several manufacturers of such materials: Ilford, Ltd., in England, Kodak, Ltd., in England, and Eastman Kodak Company in the United States.

When this work was in progress, several publications appeared which contained processing recommendations applying to thick nuclear-particle emulsions. A publication by C. C. Dilworth, G. P. S.

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Occhialini, and R. M. Payne[1] gave recommendations which were based principally upon work with Ilford plates, and more recently recommendations have been given in Technical Report OANAR-29-48[2] for processing Ilford and Kodak plates. Work done in the Kodak Laboratories was done with Eastman NTB Plates.

It can be stated, in general, that the results obtained in the present study have led to processing recommendations which are in remarkably close agreement with those given in the two publications previously mentioned. Similarly, the optimum conditions for the use of stop baths and fixing baths in processing have been found to agree very closely with those recommended. However, one innovation has been found to be advantageous, and that is the use of nitrogen agitation during the fixing operation which tends to shorten the fixing time appreciably.

Although the results obtained in the present work agree with those published, it was thought advisable to repeat in some detail recommendations arrived at, since at the present time many Eastman NTB Plates are being used and no specific directions have been given up to this time for processing such plates having a thickness of 100μ or greater.

During the course of the present investigation, a large number of variations were made in the composition of the processing solutions as well as in conditions, times, and temperatures of the processing steps. No attempt will be made to describe in detail the results of these variations.

The optimum developing process was found to be essentially that recommended by Dilworth *et al.*,[1] and in Technical Report OANAR-29-48.[2] This consists in initiating development in a low temperature developing solution and allowing the temperature to rise slowly to complete development, checking development by the use of a cold stop bath, and initiating fixation at low temperature and later allowing it to proceed at a higher temperature. Uniform development from the top to the bottom of the plate was generally (although not always) obtained.

There are a few general observations which may be of interest. It was observed that prehardened emulsions had a greater tendency than untreated plates to strip off the glass base. Presoaking plates in water before low temperature development caused additional background fog in some cases, and there appeared to be no advantage gained by the presoak. No special equipment was used in the developing; the plates were bathed in photographic trays resting in a low temperature brine bath at the start, and the trays were then removed from the brine bath and were either allowed to warm up or were placed in a warmer bath to bring them up to the desired temperature. In the stopping, fixing, and washing operations, the plates were held in a vertical position in a small stainless-steel rack. In some cases during the stopping operation, the stop bath was agitated by bubbling through it nitrogen which had been passed through an ordinary jacketed Büchner funnel in order to produce a stream of small bubbles.

The experiments indicate that strong nitrogen agitation can be generally recommended during the fixing operation. The rates of fixation were increased as much as twofold by its use, and in no case were any harmful effects observed. Some experiments indicate that the use of air agitation during fixation is inadvisable because of possible oxidation of the smaller developed silver grains. When fixation was carried out at other than room temperature, water or brine at the desired temperature was pumped through the jacket of the Büchner funnel. A few experiments were made with ammonium thiosulfate, but this agent appeared to dissolve some of the finer grains and is not recommended.

In the present work, exposures were made to the following sources of ionizing particles: (1) protons of energy 1 to 2 and 7 Mev,[3] (2) neutrons from a paraffin-jacketed radium-beryllium source, (3) electrons from beta-rays in the range 0.3 Mev as well as those obtained from 190-kv x-rays, and (4) alpha-particles having an energy of 5.3 Mev derived from polonium.

The following procedures should develop to a fairly high degree every track, including electron tracks, in an exposed Eastman NTB Plate having thickness in the range 100 to 175μ:***

Development.—Soak in D-19**** (1:1) at 5°C for 30 minutes. No agitation. Add 2 parts of water at 20°C to D-19 (1:1) and allow to stand in a 20°C bath for 30 minutes.

Stop Bath.—Two percent acetic acid at 5°C. 30 minutes' strong agitation.

Fixation.—Soak in 30 percent sodium thiosulfate solution (hypo), with no agitation, at 5° for 15 minutes. Then continue in 30 percent hypo

[1] Dilworth, Occhialini, and Payne, Nature **162**, 102 (1948).

[2] Unclassified. Office of Naval Research, London Branch; Office of the Assistant Naval Attaché for Research, American Embassy, London, England.

[3] We are indebted to the University of Rochester for the use of their cyclotron for making these exposures and, in particular, to Mr. J. F. Norton, of the Physics Department.

*** Eastman NTB Plates are light-sensitive and should be handled in the light of a Wratten Series 2 Safelight.

**** Formula for D-19 Developer:

Water, about 125°F (50°C)	500 cc
Elon	2.2 grams
Sodium sulfite, desiccated	96.0 grams
Hydroquinone	8.8 grams
Sodium carbonate, monohydrated	56.0 grams
Potassium bromide	5.0 grams
Cold water to make	1.0 liter

Dissolve chemicals in the order given.

at 20°C, with strong agitation (nitrogen), for 5 minutes longer than time required to clear. Approximate time required to clear, with strong agitation:

100μ NTB (81 percent Ag halide) $\frac{1}{2}$ hour;
160μ NTB (81 percent Ag halide) 1 hour.

Wash.—Wash in cool running water at least one hour. (An even longer wash is helpful in removing dye stain.)

Dry.—Dry at 20°C, preferably where there is circulating air.

In some cases it is desirable to weaken development in order to bring out certain desirable characteristics of tracks for identification or counting purposes and also to leave undeveloped tracks caused by particles having lower energy than those being specifically studied. Several procedures for developing a plate in which the grains of a 7-Mev proton track are resolvable are as follows:

Develop in D-19 (1:1) at 5°C for 10 minutes—no agitation. Add 2 parts water; let stand at 20°C for 5 minutes; stop, fix, and wash according to the recommended procedure.

Develop in D-19 (1:3) at 5°C for 60 minutes—no agitation. (No development at higher temperature.) Stop, fix, and wash according to recommended procedure.

Development conditions intermediate in activity between the recommended procedure and those just given can be most easily obtained by varying the time of development at either or both temperatures given. By suitable variation it should be possible to develop tracks made by particles of widely different energies.

Experiments were made with Eastman NTB Plates in the thickness range 200 to 260 μ. Recommendations for their development are similar to those made for the thinner plates and are given below:

Development.—Soak in D-19 (1:1) at 5°C for 45 minutes—no agitation. Add 2 parts water at 20°C. Allow to stand at 20°C for 45 minutes.

Stop Bath.—Two percent acetic acid at 5°C—30 minutes' slight agitation.

Fix.—Soak in 30 percent hypo at 5°C for 30 minutes—no agitation. 30 percent hypo at 20°C, with strong nitrogen agitation, for 5 minutes longer than time required to clear. Approximate time required to clear NTB Plates (81 percent Ag halide), using strong nitrogen agitation:

200μ NTB (81 percent Ag halide) $1\frac{3}{4}$ hours;
250μ NTB (81 percent Ag halide) 3 hours.

Wash.—Wash in cool running water at least two hours.

Dry.—Dry at 20°C, preferably with circulating air.

The above procedure should give satisfactory electron tracks. For proton tracks, the period of development should be reduced by approximately 60 to 70 percent.

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