FACT SHEET
ILFORD NUCLEAR EMULSIONS

TECHNICAL INFORMATION FOR PARTICLE PHYSICS APPLICATIONS

1 INTRODUCTION

Nuclear research emulsions were first developed in the 1940s to meet the needs of physicists engaged in research on cosmic radiation. The range of materials was improved and extended throughout the following decade until the versatility of nuclear emulsions in the recording of charged particles and ionising radiation was recognised by workers in many other fields. In addition to the use in particle physics ILFORD nuclear emulsions are now extensively used in autoradiography - in medical and biological research, in metallurgy and in the study of chemically reactive surfaces.

ILFORD manufacture nuclear emulsions for a wide range of applications. The size of the silver halide crystals will determine the sensitivity of the emulsion - large grains are more sensitive to ionising radiation than small ones. In general, a low sensitivity emulsion is used to detect high-energy radiation or particles, as there is plenty of energy available to liberate electrons and form electron deficient bromine atoms. However, a more sensitive emulsion is required to detect very high-energy particles as they deposit very little energy along their tracks and travel too fast to be trapped by the silver halide crystals.

Other materials not listed here have been produced from time to time to meet the special requirements of individual workers. ILFORD are always pleased to discuss such special needs and, where possible, to develop new ways of applying photographic emulsions in scientific research.Email us

2 THE PHOTOGRAPHIC PROCESS

2.1 FORMATION OF THE LATENT IMAGE

A photographic emulsion is essentially a dispersion of silver halide crystals in a gelatin matrix. ILFORD nuclear emulsions are fundamentally the same as general purpose photographic emulsions, but have several distinguishing features:

- The silver halide crystals are very uniform in size and sensitivity.
- There are very few crystals that may be developed without exposure to a charged particle (very low chemical fog).
- The silver to gelatin ratio is much higher than in a conventional emulsion.

When such an emulsion is exposed to ionising radiation or light, clusters of silver atoms are produced. These are known as latent image centres, as they are not visible until the emulsion is developed, when all the crystals containing a latent image centre are reduced to metallic silver.

When a silver halide crystal absorbs light or ionising radiation, it has the effect of liberating mobile electrons and electron deficient bromine atoms. Transfer of an electron from an adjacent bromine ion, which in turn creates an electron deficiency, can overcome the electron deficiency of the bromine atom. In this way, a positive hole can move through the crystal lattice. This electron deficiency may also be known as a 'positive hole'.

It is important for latent image formation that a significant proportion of electrons and positive holes are trapped separately, otherwise they could recombine and regenerate halide ions. The silver halide crystal contains free (interstitial) silver ions, which can move through the lattice. When an interstitial silver ion encounters a trapped electron, the charges are neutralised and an atom of metallic silver is formed. The single atom of silver is unstable but, while it exists, it increases the efficiency of the site as an electron trap. In this way a stable nucleus of four or more atoms of silver can be built up. The site is then known as a latent image centre, and that entire crystal may be reduced to metallic silver on development.

2.2 DEVELOPMENT

Photographic development is the process by which the latent image contained in an emulsion is made visible by the reduction of silver ions in the silver halide crystal to metallic silver.

When developing ILFORD nuclear emulsions, a developer is usually chosen which reduces those crystals containing a latent image centre completely and leaves those not containing a centre unchanged. The development time used for processing material should be sufficient for those crystals with a latent image centre to be reduced completely, but not so long that unexposed crystals are developed. In practice, a certain number of crystals will be developed even though they do not contain a latent image centre. These grains, when developed, constitute what is known as fog or background.

Developing agents may be divided into two main groups, depending on the source of silver ions for reduction. In practice, most developers give a combination of the two sorts of development.

The first group is known as physical developing agents. In physical development, silver ions are provided from the solution in the form of a soluble complex. These are deposited on the latent image centre and are reduced to metallic silver. This produces spherical particles, the precise shape of which is affected by pH.

Chemical developing agents make up the second group and are more usually chosen when processing nuclear emulsions. However, the choice between a physical developer and a chemical developer will largely depend on the grain structure required in the processed image. In chemical development, silver ions are provided from the silver halide crystal containing the latent image centre. The action of a chemical developer produces a mass of filaments bearing little resemblance to the original crystal. If silver halide solvents such as sulphite are present in a chemical developer, an opportunity exists for some physical development to occur. In this case, the filaments in the processed plate will be shorter and thicker.

Chemical development, like many other chemical reactions, is dependent on temperature. In general, development occurs more rapidly at higher temperatures - below 10°C development virtually stops. For this reason it is important to keep the processing temperature constant during development, otherwise it will not be possible to assess the correct development time.

Chemical developers are also dependent on pH, and will only maintain a given activity within a narrow pH range. In general, the less alkaline the environment, the less active the developer will be. For this reason, the use of an acid stop bath such as ILFORD ILFOSTOP PRO is often recommended at the end of development. This stops development immediately so that the development time can be controlled precisely.

2.3 FIXATION

The purpose of fixation is to remove all the residual silver halide, leaving the metallic silver to form the image. If the silver halide was left in the emulsion, it would slowly go brown and degrade the image. The fixing agents most widely used are sodium or ammonium thiosulphate, which form thiosulphate complexes with the silver halide. Silver thiosulphate is soluble in water and so may be removed from the emulsion by washing.

It is important to use a fixer which has not been exhausted when processing nuclear emulsions, otherwise some silver halide will remain in the emulsion. To ensure that it is all removed a fixing time should be used which is twice the time it takes for the emulsion to clear.

After fixation, the emulsion must be washed very thoroughly. This is to remove all the silver thiosulphate complexes in the emulsion. If any do remain, they will eventually break down, forming silver sulphide which is brown and will obscure the image.

3 PRODUCT RANGE

3.1 TYPES OF EMULSION

There are currently three types of ILFORD nuclear emulsions, with a fourth under development specifically for Particle Physics.

ILFORD emulsion	Crystal diameter (μm)
G5	0.27
K	0.20
L4	0.11

ILFORD K emulsions are available in a range of sensitivities, which are defined by a number denoting increasing sensitivity from 0 to 5.

K0	Used in particle physics to record protons of energies up to 5MeV. Records thorium **α**-particles as nearly continuous tracks. Produced only by special order.
K2	Records protons to about 80 MeV. Slow electrons produce tracks of a few grains only.
K5	Exposure times tend to be shorter than with K2, especially where activity levels are low.

K5 emulsion is also available in a ready-to-use diluted form as K5D.

When using isotopes emitting particles of higher energy, use K5: when handling specimens of exceptionally low activity, use G5. For specialist applications using strongly ionising particles such as a-particles or fission fragments, K.0 can be made available.

A further range of emulsions is under development for particle physics experiments. These will be even more sensitive to minimum ionising particles than G5 emulsion. Preliminary samples can be made available by arrangement.

3.2 ORDERING

ILFORD nuclear emulsions are supplied in brown glass bottles of 50 and 100ml. It would be unpractical and generally undesirable to maintain large stocks of nuclear emulsion, but small quantities of the emulsions G.5, K.2, K.5 and L.4 are available from stock. Delivery of large quantities of emulsion is by arrangement. For further information on the availability of other nuclear emulsion products, please contact ILFORD. Email us

3.3 COMPOSITION

ILFORD nuclear emulsion contains 0.162g Ag/g emulsion and 0.042g gelatin/g emulsion. At least 65g of emulsion are contained in a 50ml bottle and 130g in a 100ml bottle.

The product is supplied in shreds for ease of handling.

The density of the material as supplied is around 1.3 g/cm3. On drying this rises to 3.8 g/cm3 in equilibrium with air at 58% relative humidity at 20°C.

4 PRODUCT USE

4.1 STORAGE BEFORE EXPOSURE

In general, nuclear emulsions should be protected from light and local radioactivity. Sensitised material should be stored in such a way that efficient stock rotation is possible to ensure that material is in optimum condition. It may be helpful to record the date when the emulsion was received on the bottle label.

Emulsions in gel form deteriorate rapidly above 5°C and must be kept under refrigeration, without freezing. The outer wrapper should be retained during storage. After coating and drying, slides may be stored at room temperature, refrigerated or frozen. When slides are stored below 5°C and are to be used at normal room temperature, they should be allowed to warm up in their packaging for about three hours to prevent condensation forming on the surface.

Sensitised materials should not be stored near to certain chemical solutions, such as ammonia, sodium sulphide or formaldehyde; or near fumes or vapours coming from volatile substances; gases, such as sulphur dioxide or coal gas; or some industrial solvents and cleaning fluids. Materials should not be stored on new or newly painted wood. Processing chemicals should be stored as far away as possible from sensitised material.

Its basic sensitivity and the exposure it receives from cosmic radiation and local radioactive sources determine the useful life of the fresh emulsion. If correctly stored, nuclear emulsions should remain in good condition for at least two months. Nuclear emulsions may be usable for considerably longer depending on the level of background acceptable in a particular application.

4.2 HANDLING

The shreds can normally be removed from the bottle with plastic tweezers. Wear suitable protective clothing such as laboratory overalls, safety glasses and gloves.

4.3 SAFELIGHT RECOMMENDATIONS

ILFORD nuclear emulsions are sensitive to blue light. For general darkroom illumination, the ILFORD 902 (light brown) safelight filter in a darkroom lamp fitted with a 15 watt bulb, is recommended. For direct illumination, the ILFORD 904 (dark brown) safelight filter is recommended. When maximum illumination is required, a sodium lamp with the correct safelight filters may be used. The safety of this type of lamp should be checked by a practical test before use.

ILFORD nuclear emulsion should not be exposed to safelighting for any longer than necessary. If preparations are to be manipulated before processing, great care should be taken to avoid prolonged exposure to safelight.

4.4 CHECKING THE EMULSION BEFORE USE

It is possible that ILFORD nuclear emulsion may have been exposed in transit to conditions causing the shreds to melt slightly and form a solid lump. These conditions may not have affected the performance of the emulsion. This should be tested before commencing an experiment. If the level of background is acceptable and the distribution of developed grains is uniform, the emulsion is undamaged and fit to use.

5 USING NUCLEAR EMULSION IN PARTICLE PHYSICS

The choice of emulsion for particle physics will always depend on the energy of the emitted particles and the discrimination required. ILFORD have a range of emulsions specially designed to satisfy a wide range of demands - see Section 3.1.

5.1 PREPARATION OF THE SURFACE

The substrate on which the emulsion is to be coated must be prepared such that the surface is hydrophilic. It is recommended that this surface is washed with acid and rinsed many times in distilled water. If required, the substrate may be subbed to provide a good adhesive surface between the section and the emulsion. A suitable subbing for dip coating is given below.

Gelatin	0.5g
Chrome alum (K2SO4.Cr(SO4)3.24H20)	5.0g
Distilled water to	1 litre

Make up the solution just before use. After dipping, drain the substrate and dry in a dust free atmosphere.

5.2 COATING THE SUBSTRATE

If a coating of emulsion onto glass is required this can be done by ILFORD. Coating onto other substrates will also be considered. Please enquire about feasibility and price.

It has been reported that heating the emulsion before exposure will increase its sensitivity. However, the temperature to which it is heated is critical, because after a certain point, which varies according to the emulsion, the corresponding increase in fog will overshadow any benefits of increased sensitivity.

Ideally, the environmental conditions in the darkroom are about 25°C and 75%RH.

Remove enough emulsion from the bottle for immediate requirements only. Melt in a glass or stainless steel vessel in a water bath at about 40°C, stirring gently to avoid local overheating. Deionised or distilled water can be added at this point to achieve the required viscosity.

When melting do not agitate so as to produce froth. If frothing does occur, larger air bubbles can be removed by filtering. Ideally, the emulsion should be allowed to melt undisturbed for an hour in the water bath. This has the advantage of reducing the background in the coated slides. Emulsion may be reheated after it has set, but contaminated emulsion must not be returned to the stock bottle.

Emulsion may be applied to the substrate either by dipping the material into the emulsion or by allowing a few drops of emulsion to fall onto it. The thickness of the layer when dry will be 1/8 of its thickness at this stage. For example, 0.8ml/dm^2 will produce a layer 10µm thick when dry.

To produce a thick layer, pour the warm emulsion onto prepared ILFORD glass plates, supported on a level glass slab. A glass or stainless steel spatula may be used to spread the emulsion over the required area. Wipe the back of the plate before the emulsion dries. It is important not to dry thick layers too quickly or the emulsion surface will crack.

If a very thin layer of emulsion is required (such as in autoradiography) then dipping is the best coating option. The emulsion is diluted with an equal volume of water and after dipping allow the emulsion to drain off the glass as completely as possible. Hold the substrate vertically on a gauze pad for draining and place horizontally for setting and drying.

Allow the emulsion to set in the dark, then dry it with a gentle current of clean air. The temperature and humidity of the air current are not critical, but drying at room temperature will avoid stress marks in the emulsion. Alternatively, allow the emulsion to dry on a cold, metal plate. This will slow down drying, but increase the gelling speed. Slides may also be dried in a carbon dioxide atmosphere.

Thinner layers of emulsion can be produced by diluting the emulsion with additional water, or by the addition of glycerol, which has the effect of reducing any fog resulting from stress between the gelatin and the silver halide crystals. In certain circumstances, it may be better to dilute the emulsion with gelatin. This has the effect of decreasing signal and background. This can be an advantage when examining a specimen with very high activity. In addition, emulsions diluted with gelatin are more sensitive than undiluted ones (in relation to their dilution).

Pressure marks:
Rapid drying or brushing of the plate before it is completely dry causes these.

5.3 STORAGE DURING EXPOSURE

If exposure times are short, i.e. up to two days, the storage conditions during exposure are not important. The exposures encountered in some experiments may be very much longer, perhaps months or even years. In these circumstances, it is essential that the emulsion be kept under much the same conditions as emulsion stored before use. High humidity can cause increased fog and accelerated latent image fading, so it may be important to maintain low humidity conditions during the exposure period. If latent image fading still remains a problem, it may be slowed down by exposing the preparation in an atmosphere of inert gas.

It is extremely important to process the materials immediately after the exposure time has been completed. The latent image fading is progressively more severe as the crystal size of the silver halide decreases in the order G5, K and L4. If there has to be a delay before the emulsion can be processed, then the preparation should be stored with proper protection from radiation at a temperature between 5-10°C and at 50%RH.

6 PROCESSING THICK EMULSION LAYERS

A number of processing techniques are described below. These are intended as a guide and may be modified to suit individual working conditions and experimental aims. During the course of an experimental programme, processing techniques should be standardised.

These processing recommendations are made on the basis of dry layer thickness before processing.

The method of developing ILFORD Nuclear emulsions is largely determined by the thickness of the emulsion layer. This is because the time taken for the developer to diffuse through the layer should be an insignificant fraction of the total development time. If this were not the case development would be uneven through the layer.

The development time to be used is the shortest time that will achieve complete development of all the latent image centres (see Section 2). Always ensure the developer is fresh and has not started to turn brown.

6.1 Layer thickness UP TO 50 microns

The best results will be obtained using ILFORD PHENISOL diluted 1+4 at 18°C. Alternatively use ID-19 (Section 8.1) or Kodak D-19 diluted 1+1 at 20°C.

For the thinnest layer (below 10 microns) a development time of around 4 minutes should be sufficient. As layer thickness rises towards 50 microns this may have to be extended to 15 minutes. However, for the less sensitive emulsions (K0 and K2) 5 minutes may be adequate.

6.2 Layer thickness around 100 microns

There are 2 options available here. The first, at a uniform temperature of 20°C is undoubtedly the most convenient.

The second, the "temperature cycle" method of Dilworth, Occhailini and Vermaesen (Section 9) gives better discrimination between tracks and background but is somewhat less convenient.

6.2.1 Uniform temperature method

Pre-soak the layer in distilled water for 20 minutes at 20°C before development.
Develop in ID-19 (Section 8.1) diluted 1+9 at 20°C with agitation such as mechanical rocking. For G5, develop 60-70 minutes; for K2 or K0, 15-35 minutes.

6.2.2 Temperature cycle method

In this method the emulsion takes up developer at a low temperature at which it is inactive. It is then removed from the developer and maintained at the required temperature for the time needed for development to occur.
Pre-soak the layer in distilled water for 30 minutes before development. Start at 20°C cooling to 5°C over this time.

Soak in Brussels Amidol developer (see Section 8.2) for 50 minutes at 5°C. Take out of the developer and remove surface liquid by blotting with filter paper.

Warm the plate to 25°C and hold at this for 50 minutes. You can do this by placing in a stainless steel disk maintained at 25°C by a surrounding water bath.

6.3 Layer thickness above 100 microns

For a 200 micron layer the method in Section 6.2.2 can be modified to a 50 minute soaking time.

For thicker layers users are referred to the work of Dilworth, Occhailini and Vermaesen (Section 9).

6.4 STOP BATH

After development, transfer the material to an acid stop bath. This may be made up with 0.2-2% acetic acid solution or ILFORD ILFOSTOP PRO diluted 1+19 with water.

Like development, stop bath times vary with layer thickness. A time of 1 minute will suffice for thinner layers, rising to around 10 minutes for a 100 micron layer.

6.5 FIXING

The emulsion should then be fixed. Use any standard non-hardening fixer, e.g. ILFORD HYPAM diluted 1+4 for 4 minutes at room temperature. Hardening fixers are not recommended because of the difficulty of ensuring an efficient wash.
Thick layers of emulsion will take longer to fix. Fix the material for twice the time it takes the emulsion to clear.

6.6 WASHING

After fixation, thin emulsions may be exposed to normal room light. The required washing time will depend on the thickness of the emulsion. A time of about 10 minutes is needed for layers of up to 10 microns but at 100 microns 4 hours is necessary. Slides may be washed in tap water, but give a final rinse in distilled water. While not essential, it is recommended that the wash water temperature be about the same as the temperature of the processing solutions.

Slides may be washed in tap water, but give a final rinse in distilled water. This will ensure that any materials dissolved in the tap water are not concentrated in the emulsion on drying. Failing to do this is a common cause for a lack of permanence in the final image.

7 STORAGE OF PROCESSED MATERIALS

After nuclear emulsions have been processed, the storage conditions are less critical than before. If plates and slides are properly processed, with adequate fixing and washing, and are correctly stored they will keep in good condition for many years. Store processed material that is to be kept for a long time at about 10°C and 50%RH.

Variation in temperature and humidity should be kept to a minimum. Store preparations in the dark while they are not being examined.

8 DEVELOPER FORMULATIONS

8.1 ID-19

Metol	2.2g
Sodium sulphite, anhydrous	72g
Hydroquinone	8.8g
Sodium carbonate, anhydrous	48g
Potassium bromide	4g
Water to	1 litre

8.2 Brussels Amidol

Sodium sulphite, anhydrous	18g
Potassium bromide	0.8g
Amidol	4.5g
Boric acid	35g
Water to	1 litre

9 USEFUL REFERENCES

Dilworth C C., Occhialini G P S. and Vermaesen L. 1951
"On Processing Nuclear Emulsions"

Proceedings of a symposium held at the University of Bristol published in
"Fundamental Mechanisms of Photographic Sensitivity"

Butterworths Scientific Publications
Powell C F, Fowler P H and Perkins D H, 1959

The study of Elementary Particles by the Photographic Method, Pergamon Press.
(The first section of this book provides a general introduction to nuclear emulsions.)

HARMAN technology Limited, Town Lane, Mobberley, Knutsford, Cheshire WA16 7JL, England
www.ilfordphoto.com