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MINIMIZATION OF UNWANTED X-RAY EXPOSURES ON CONVENTIONAL PHOTOGRAPHIC FILMS BY DEVELOPER MODIFICATION

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Submitted To

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and Instrumentation

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Abstract

Experiments were done to reduce the influence of unwanted X-ray exposures on Kodak Plus-X film. Variations of surface developer components showed no favorable results. The concentration of KBr was varied up to ¹⁰ grams per liter with again, no favorable results. The incorperation of benzitriazole decreased base plus fog from values of .81 to .56 with a significant decrease in speed. Phenyl mercaptotetrazole was extreamly effective in decreasing both base plus fog and speed.

Introduction

Since the signing of the Limited Test Ban Treaty (I963) underground tests of nuclear devices have been 1 conducted. As a method of recording data, photographic film has been employed.

Although every possible precaution has been taken, the release of residual radioactivity has occured. Prior to processing, photographic film shows undesired effects when exposed to high energy radiation. The intent of this work is to devise a system whereby the effects of unwanted X-ray exposures may be minimized. The application of this work rests in recovering X-ray irradiated films.

Many workers have studied the effects of high energy radiation on photographic emulsions. Reports have been written by, Dutton, Hoerlin and Hamm, Charlesby, Pelc , Kornfield and others. Most of these studies however, deal with rather specific circumstances. Only experimental and theoretical information has been derived from them.

Hansen, McCue and Wycoff have suggested many means 2 by which irradiated films may be recovered. Some of these include special emulsions, rapid access, shielding of camera and film, storage, processing and image enhancement. Of these methods, variations in processing chemistry as well as the introduction of anti-foggants, were investigated.

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Background Information and Theory

Ionizing radiation is ^a term used to describe any radiation that upon absorption, produces the ionization of a material. In a most general sense, this type of radiation can be broken up into two major groups. The first involves propagation of mass and is termed corpuscular radiation. The second group, named electromagnetic radiation, propagates 3 no mass.

According to Dutton, the major types of corpuscular radiation (Alpha and Beta particles) can be eliminated by modest shielding. Therefore, assuming the presence of such protection, only electromagnetic radiation could be the source of image degradation.

Electromagnetic radiation can be broken up into two major classifications, X-rays and gamma rays. Since electromagnetic radiation of the same energy (wavelength) has the same properties, there are no fundamental differences between X-rays and gamma rays. The most useful method of differentiating between the two is by noting the source of radiation. ^A description and summary of X-rays and gamma rays follows.

Gamma radiation results from internal electron b transitions. An unstable nuclei catalyzes a cascading effect, i.e. electrons in outer orbitals fall into lower orbitals. The loss of energy during this process is

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emitted as gamma rays.

Gamma rays can be of a single discrete energy level or of many discrete levels. This is a function of the material and its decay process.

X-rays can be classified into two groups, again depending on their origin. In a general sense, Xxrays result from the interaction of high speed electrons 7 and a material. The material that has been traditionally used is tungsten.

Characteristic X-rays are produced from a similar cascading effect as described previously. The process differs in that a radioactive nuclei does not catalyze the reaction. If Beta radiation (high energy electrons) of suffici ent energy strikes a material and removes an electron, the cascading effect has been catalyzed. Electrons of higher energy orbitals cascade down to obtain the most energetically stable form. In this transition process, energy- is liberated in discrete levels.

Continuous X-rays, sometimes called 'Bremssthlung' , are produced when Beta rays are acted upon by Coulombic forces within the atomic structure. These forces deflect the electrons path, causing changes in acceleration. These changes result in the Beta ray loosing some of its energy in the form of continuous X-rays.

The radiation is continuous in nature because the

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loss in energy occurs in ^a random fashion. The unfiltered continuous radiation is sometimes referred to as a 'white spectrum' .

Due to this 'spectrum' nature of radiation, many terms have been defined to describe it. Effective energy, equivalent wavelength, radiation quality and half value layer may be used in this context.

Absorption Properties of X-Rays

The absorption of monoenergetic X-rays is governed by an equation very similar to that of Beer's Law. It is stated below:

 $I = I_0 e^{-\alpha}$ = = Linear Absorption Coef. This equation states that the intensity transmitte (I), equals the intensity incident (I_0) times an absorption factor. The absorptin factor is made up of the linear absorption coefficient and thickness of the material (d). This equation has the drawback in that it is dependent on the physical state of the material. Thus the following relationship is derived:

$$
I = I_0 e^{-(\n\pi/2)x}
$$
 π = Mass Absorption Coef.
\n $x = \text{Volume of cross sectional area of one cm}^2$ and x cm wide.

This equation is independent of the physical state of the material.

Both the linear and mass absorption coefficients of a material are a function of how that material interacts

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with radiation of that energy. Thus, to compute the total interaction between a spectrum of energy and matter one must sum the contributions at each wavelength. The types of interactions that exist between matter and high energy radiation is the topic of the next section.

Interactions Between X-Rays and Matter

There are four major types of interactions possible between X-rays and matter. They are: Thomsom (Incoherent) scattering, Compton (Coherent) scattering, Photoelectric Effect and Pair Formation. ^A description and summary of the basic properites of each follows.

When radiation is absorbed by ^a material and it is of sufficient energy to liberate an electron with some quantity of kinetic energy, the photoelectric effect is produced. The electron liberated, called a photo electron, may sometimes acquire very high kinetic energies. An equation for this process is given below:

h = W+ $\frac{1}{2}$ mv 2

Where 'W' represents the work function of the material, σ^2 represents the kinetic energy of the photo electron.

Compton scattering results when radiation absorbed does not give up all of its energy to the photo electron. The result is a change in wavelength and direction of the incident radiation, as well as the production of a recoil photo electron. An equation for this process is given

below:

h = h $+\frac{1}{2}mv^2$ h = Incident Energy h $\frac{1}{2}$ = Reflected Energy $\frac{1}{2}mv^2$ = Kinetic Energy an equation can be derived for the wavelength change as a result of Compton scattering. Shown belowi d = 0.024 (1-cos 0) 0 θ = Angle between incident and reflected beams. d ⁼ Change in Angstroms. The maximum wavelength occurs at an angle θ = Angstroms.
180 ⁰, and

is computed to be 0.048 λ .

Thomson scattering involves radiation emmited from an oscillating electric charge. Radiation is absorbed.by ^a material setting an electron in motion. The radiation emmitted by this electron is the same as the incident u radiation however its direction has changed. This is shown mathematically below.

> h = h '

Where h and h ' are the energies of the incident and reflected radiation respectively.

^A direct consequence of Einsteins energy-mass equivalence is the effect of pair formation. If radiation of sufficient energy passes through ^a nuclear field and electron and positron pair are formed. The Kinetic energy of the electron will wqual that of the positron, both equalling one half the incident radiation. This is shown

Pg- 6

below mathematically:

$$
h = (e^- + \frac{1}{2}mv^2) + (e^+ + \frac{1}{2}mv^2)
$$

$$
\frac{1}{2} mv^2 = h / 2
$$

This effect only occurs above radiation energies equal to 1.02 MeV. In general for incident radiation of that energy, pair formation comprises only a small percentage of the total interactions. As mentioned previously, the interactions are a function of the material and the radiation energy.

X-Ray Absorption by Photographic Films

*

When high energy radiation is absorbed by a material the primary action is the production of a free electron with high energy. In photographic emulsions the main interactions for lower energy photons (greater than .01 Angstroms) are the photoelectric effect and Compton scattering. For wavelengths shorter than .01 μ pair formation is also included as a mode of absorption.

The high energy electron produced, termed the primary electron, in travelling through the matter creates a track of ionizations. It is these ionizations which produce conduction band electrons.

Primary electrons do not give uo their energy at ^a constant rate. Above a certain energy, around ²⁵⁰ eV's, the average rate of energy loss per micron is ^a constant. Below that value the energy loss increases as the

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electron energy decreases. This produces a higher image density as the electron slows down. See Figure I and Figure II (Next Page) .

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Figure 4.2. Average rate of energy loss of electrons per micron in X-ray emulsion as function of energy (keV) (calculated).

Figure I

Hoerlin and Hamm estimate that a 40 KeV electron may travel throgh a distance of about four grains rendering them developable. With an X-ray tube potential of ⁶⁵ Kvp, photoelectrons in the range 10 to 20 KeV are produced. From range and energy loss data it is estimated that ¹⁰ KeV's of energy are absorbed by eacg grain. Yamakawa derived a value of 5.8 eV's as the energy required for a charged particle to produce an ion pair in silver bromide. As a result of this energy data, approximately 1500 condution band electrons are being produced. It is estimated that the exposure time for this reaction is on the order of 10^{-14} seconds. As the electron traps Pg- 8

have to accommodate such a large number of elctrons, one would expect a large number of small latent images dispersed throughout the grain. Hoerlin and Hamm, Berg, Marriage and Stevens have verified this fact.¹⁷

Figure 4.3. Photomicrograph of an electron track as seen in a nuclear track emulsion.

Figure II

The quantum efficiency of X-ray exposures is ^a function of the emulsion and radiation energy. It would

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be fair to make the general statement that the quantum efficiency of the latent image forming process is usually grater than one. If the emulsion is of low sensitivity and the radiation long (low energy) it is possible for the Quantum efficiency to fall below one.¹⁴

The law of Quantum Equivelence states that for every photon absorbed a silver atom is produced. This law does not hold true for X-ray exposures as a result of their high energies. Since the Quanta is of much higher energy, it enables upon absorption, Many grainssto become developable. This implies an enormous amount of silver is being produced.

^E xperimental

 $\ddot{}$

The experimental portion of this thesis can be broken up into two main areas: general information experiments and evaluation of various developers. General information experiments include all preliminary work such as construction of all mecessary equipment, generation of D-logH curves for the various film developer combinations and determination of density as a function of attenuar step for X-ray exposures,

Before any processing was possible, certain supplies and materials had to be acquired. ^A list of chemicals and all pertinent information is given in the Appendix.

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The acquisition of materials delayed experimentation.,

^A method had to be devised to provide consistent agitation for up to five replicates. As development times of up to fifteen minutes can become very tedious and arduous, an agitation device was fabricated. Constructed of wood, this device (see appendix) made processing of up to five single reel stainless steel tanks possible.

Processing with five tanks was found, however, to be inefficient and inconsistent. Difficulty arose in filling and emptying the tanks. Thus only three reels were processed at one time.

At this time all films were exposed with a Kodak Model 101 sensitometer found in research darkroom R-10. An Inconel neutral density filter provided attenuation for Plus-X, Kodak neutral density filters amounting to .6 neutral density were added for the Tri-X exposures.

For convenience the use of premixed D-19 was employed. Chemistry to make five gallons was acquired from Chem Mix as was a used Kodak Cubitainer. After throughly cleaning the container the D-19 was mixed according to packaged instructions. (For the D-19 formula please see appendix,)

All Tech. Ops. solutions (Tech. Ops. refers to the developer solution devised by Bill Nam of Technical Operations Research. For the basic Tech. Ops. formula please see Appendix.) were mixed using the previously pg. 11 mentioned chemicals. Measurements were made using a Mettler H10 balance, located in research darkroom R-10. (For a listing of all apparatus used and pertinent information please see Appendix.) Adjustments in pH were made using the pH meters located in the photo-chem lab. All densities were read on the Mc Beth TD-504 densitometer fould in the computing center.

With the chemistry and apparatus available, processing of sensi-strips proceeded. Density versus log exposure curves were plotted for Plus-X and Tri-X in both the Tech. Ops. formula and D-19. From these curves basic information was derived. The following development times were choosen as optimum taking into consideration base plus fog, speed and gamma (contrast). (Speed is defined as .8 divided by the exposure corresponding to .10 above base plus fog.)

Characteristic curves can be found in the Appendix.

It was during this branch of Experimentation that 1 many minor but troublesome difficulties arose. An unusual fogging was found to exist on the film. It did not impede experimentation and was erroneously ignored. Before proceeding any further, it became necessary to devise ^a system of negative identification. The table given below explains the identification system employed.

Exposures made to X-rays were conducted at Strong Memorial Hospital under the supervision of Dr. Donald Plewes. The X-ray Source is an Elema Schonander generator with outputs for general radiography and floroscopy. It is capable of generating 130 Kv peak and mA maximun of 600. The tube is ^a Picker rotating anode tube and the focal spot \mathbf{z} size is ² mm.

^A film holder was constructed out of cardboard as nothing else was available. The X-ray source was set at the levels given in table three.

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Note that the X-ray exposures were made on 12/16/78 yet processing did not occur until 2/10/79. This was a result of delays in chemistry aquisition.

Again an abnormal fogging pattern was found. The connection was not made between the previous fogging problem and this one. As a result the source of shielding necessary to protect the unexposed film was blamed. Tests were run noting the placement of shielding lead. Results showed that fog was present with more than adequate shielding. This left the film or storage containers to be at fault, ^A different film holder was used and experiments made resulted in the conclusion that the film was at fault. The probable origin was traced to a faint light leak found in my darkroom.

With the fogging problem resolved, experimentation proceeded. The films were exposed to 100 Kvp radiation with a stepped attenuator in the radiation path. The attenuator, made of aluminum, has sixteen steps each increasing in thichmess by 1/16 of an inch. ^A plot of the density produced as a function of step in the Tech. Ops. developer and D-19 was generated for the two films. (See Appendix for plot.)

It was decided after reviewing time considerations, that the investigation will proceed with only one film. There is no specific reasoning why Plus-X was choosen.

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^A decision had to be made concerning the radiation quality to be used for the developer variation portion of this thesis. From the previously mentioned graphs it was shown that Plus-X filtered with approximately ⁸ mm of aluminum produced a density of about one when processed in D-19. Since there was only seven millimeters of aluminum available, seven was used.

Film was exposed to the filtered radiation and processed in D-19 and Tech. Ops. to verify differentiation. 3 (Sets jfc and 2C) With differentiation proven, developer constituents can be varied.

Testing of Various Developers

The acquisition of a Kodak model ¹⁰¹ sensitometer made exposing sensi-strips much more convenient. From this point all exposures are made on this instrument.

Experimentation will consist of varying two of the developer constituents and the pH, each at two levels. The following chart gives an idea of the experimental design.

Table IV: Develpoer variation chart.

Metol

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High and low indicate variation in concentration plus and minus 50%. The pH at the low level is 10, (normal pH) and at the high, Ll. Variations in pH were achieved by adding a solution of one molar sodium hydroxide. ^A chart of the actual concentrations can be found in the Appendix.

In order to prevent oxidation of unused developer a nitrogen atmosphere was placed in the space remaining in the volumetric flask. Nitrogen was obtained from the nitrogen burst apparatus found in research darkroom R-10.

With this experimentation complete it was decided to investigate the effects of the potassium bromide concentration, The normal concentration of KBr in the Tech. Ops. developer is 1.0 grams per liter of solution. Solutions were mixed with KBr concentrations of .5, 1-5. 2.0, 2.5. gms per liter. The need for extended studies arose and KBr concentrations were varied up to 10.0 grams per liter.

The final portion of labwork consisted of examining the effects of antifoggants on the radiation fogged film. The antifoggants were used, benzitriazole (Kodak antifoggant #1) and Phenylmercaptotetrazole (PMT shall be used as an abbreiation for this material) .

^A standard solution of concentration equal to ² grams per liter of benzitriazole was mixed. In order to provide accurate dispense of this solution, pipets

 $pg.$ 1**b**

were used. Every ⁵⁰ ml of solution contained .1 grams of benzitriazole.

The concentration of Kodak antifoggant #1 was varied at nine levels ranging from .1 gms/l to 2.0 gms/l. The last two solutions (CD30 and CD31) containing ¹ and ² grams per liter respectively had the antifoggant introduced directly. One should note that ² grams per liter of benzitriazole required some heating and much agitation before going into solution.

The final experiments were conducted with varying concentrations of PMT. ^A standard solution prepared by Jim Kretchmer was used (concentration equal to 2.0 grams per liter) . The concentration of PMT was varied from .02 to .08 in .02 gms/l increments.

Results and Discussion

From the discussion earlier, one may conclude many stable latent images are being formed by ^a single X-ray photon. The minimization of this exposure can be approached in two ways. If latent images produced by high energy radiation are dispersed throughuot the grain, we can avoid most by using a surface developer. If the X-ray latent images are in fact smaller than those produced by visible light the we can attempt to increase the selectivity of development favoring the larger cites.

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The developer modification portion of this labwork was intended to investigate the effectiveness of various surface developers. As stated earlier the basic Tech. Ops. formula was altered, i.e. three factors at two levels.

It was found that varying the concentrations of metol and ascorbic acid, and changing the pH, had no significant effect with reguard to decreasing base plus fog or altering speed. A table of the ANOVA values can be found in the Appendix.

There may have been a few factors working against us with these experiments. In order to obtain reasonable density long development times were used. During the fifteen minutes of processing some AgX may have dissolved away exposing internal latent images to development.

^A second drawback involves the mechanism of latent image formation. All developer modification, KBr, benzitriazole and PMT concentration tests were carried out with the film exposed to light, then to X-rays. It may be possible that the visible light exposure produced latent images that are reasonably stable, yet would normally remain undeveloper. Photo electrons produced by the X-ray exposure may be drawn to these cites rendering them developable .

Taking these results into consideration it was decided that a method of discriminating between the various sized

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latent images was necessary. An investigation into the effects of varying the KBr concentration was made.

According to Mason, bromide in the developer has a much greater retarding effect on fog development than on image development. The action of excess bromide in 13 solution can be twofold. Excess halide ion in the vicinity of the AgX grain can decrease the thermodynamic activity of the silver ion at the surface and in solution. These actions would thus decrease the development action.²⁴

The excess bromide might also contribute to an effect known as the Negative Charge Barrier. Halide ions are absorbed from the developer to the crystal surface providing a negative charge around it. If the developing agent is negativly charged, it will be repelled.

With this as a basis for the possible reduction in fog levels, variations in KBr concentration were tested. It was found that varying the concentration of potassiun bromide did little in the way of improving film characteristics

The data shown in table six (next page) was plotted and linear regressions were run. Extreamly poor fits were found for the dependence of base plus fog on KBr concentration and the dependence of speed on KBr concentration,

For this, and the remaining experiments an efficiency factor has been defined. The developer's efficiency

These runs are erroneous and should be omitted

is defined as the speed (as calculated previously) divided by the base plus fog density. The efficiency factor is represented by the letter 'R'.

The highest 'R' values were found in the potassium bromide tests. These values did not unfortunatly, indicate a large decrease in base plus fog. These values showed only a minimal decrease in the speed obtained. Processing in the original Tech. Ops. formula yielded an 'R' value Of 7.0. Processing with KBr concentrations of five and

six grams yielded 'R' values of 11.9 and 11.5 respectively.

This experiment, providing no positive results led into an investigation of two antifoggants, benzitriazole and phenyl mercaptotetrazole (PMT). The action of these antifoggants is not entirely understood, however it is presumed that they inhibit development by absorption to the grain or latent image surface.

Benzitriazole was included into the developer at nine levels in an effort to reduce fog. The data, shown in table seven below was analyzed using linear regressions.

The relationship between benzitriazole concentration and base plus fog and benzitriazole concentration and speed are summerized below: following page.

> Base plus fog = $-1.50(Ben) + .812$ $r^2 = .97$ Speed = $-1.50(Ben) + 7.53$ $r^2 = .40$

$$
\texttt{pg. } 21
$$

One can see that for a given benzitriazole concentration one can vary the base density with a relatively high degree of accuracy. It must be noted that ^a change in speed and contrast will also occur.

The mosy efficient run with reguard to benzitriazole concentration was the one containing one gram per liter of the antifoggant. Its 'R' value was calculated to be 11.2 and is a result of modest decreases in both base plus fog and speed. (.65 and 7-30 respectively) The final benzitriazole run (2.0 gms/l) showed an important decrease in base plus fog, a value of .51 was recorded. The speed on this run also dropped producing an 'R' value of 7.5.

The final experiment, varying the PMT concentration produced more strikeing results. As the PMT concentration was increased from .02 to .08 grams per liter the base plus fog decreased from .56 to .30 . Again the undesireable effects of decreased contrast and speed were also produced.

The action of PMT is theorized as absorbing on the crystal surface and as a result, impeeding development. This had such a large effect that the 'R' values produced were extreamly low. With only .02 grams per liter of PMT in the developer the fog was reduced by one third to a value on .56.

A similar regression analysis was run on the PMH₂. 22

data with the following results

Base plus fog = $-4.45(PMT)$ +.635 r^2 =.96 Speed = $-5.51(PMT) +4.40$ $r^2 = .96$

Graphs of the PMT data can be found in the Appendix.

Conclusions

With the preceeding results one can conclude that varying the developer constituents does little in the way of improving developer selectivity with reguard to X-ray and visible light produced latent images. Variations is the potassium bromide concentration provided only minor differences. The introduction of antifoggants made the greatest change with reguard to decreasing base plus fog. The maximum amount of benzitriazole tested was effective in decreasing base plus fog without ruining speed. PMT drastically reduced both base plus fog and speed.

To within limits, the minimum desired fog can be obtained by choosing the proper amount of antifoggant. One must however, be aware of the consequences, loss in contrast and speed.

For future experimentation I would suggest a more detailed look into the action of antifoggants as a mechanism to reduce high energy radiation fog.

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Agitation Apperatus

D-19 and Tech. Ops. Formulas

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D-19
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Chemicals Used

Apparatus

Kodak Model ¹⁰¹ Sensitometer (RIO) Serial numbers not available .

Kodak Model 101 Sensitometer (R5) S.N. 903

R.I.T. No. 62741'

Mc Beth TD504 Sensitometer (Computing Center) S.N. 2622A

R.I.T. No. 113368

Elema Schonander X-ray generator

Picker rotating anode tube

Concentration of Developer Components, CD1-CD8

Developer	Metol	A.A.	Na Meta	KBr	pH
CD1	.702	1.65	1.00	5.00	10.01
CD2	.701	4.95	1.00	5.00	10.01
CD3	2.10	1.65	1.00	5.00	10.00
CD4	2.10	4.95	1.00	5.00	10.01
CD5	.704	1.65	1.00	5.00	11.13
CD6	.703	4.96	1.00	5.00	11.01
CD7	2.10	1.65	1.00	5.00	11.02
CD8	2.10	4.96	1.00	5.00	11.00
T.0.	1.40	3.30	1.00	5.00	10.00

Base plus fog

Speed

 $\hat{\mathbf{v}}$

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Footnotes

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