

FEASIBILITY STUDY ON ADDITION OF A COLOURANT
TO COMMERCIALY USED FIRE-RETARDANT FORMULATIONS
TO ENABLE VISUAL DISTINGUISHABILITY BETWEEN
TREATED AND UNTREATED MINE SUPPORT TIMBER

BY



C.B. DU TOIT

STUDENT NR. 80-2641-6

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PROMOTER: DR M BARISKA
DR G VAN DER KLASHORST

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DECLARATION

I, THE UNDERSIGNED, HEREBY DECLARE THAT THE WORK CONTAINED IN THIS THESIS/STUDY PROJECT IS MY OWN ORIGINAL WORK AND HAS NOT PREVIOUSLY IN ITS ENTIRETY OR IN PART BEEN SUBMITTED AT ANY UNIVERSITY FOR A DEGREE.

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(a)

(a) SUMMARY

The ability to visually distinguish between fire-retardant treated and untreated mining support timber, treated with ammonium sulphate and ammonium phosphate salts, is very difficult. In fact, once treated and allowed to dry for approximately three days, it visually becomes totally non-distinguishable from untreated timber.

The project objective is to identify a suitable colourant which can be incorporated into the fire-retardant formulation which will enable visual distinction between fire-retardant treated and untreated mining timber support on a cost effective basis. The following aspects were evaluated:

- Compatibility and stability within fire-retardant formulations.
- Colour effectiveness and fastness.
- Effect on combustion.

The results showed that:

- * Three of the chemical colourants proved to be compatible and stable at two ranges of temperatures. Colourants were Acid Green, Bazazol Red 76L and Malachite Green.
- * Of the colourants evaluated, Malachite Green and Bazazol Red 76L performed most satisfactorily over two exposure trials, i.e. controlled weather-o-meter and natural exposure.

(b)

a) SUMMARY (continued)

- * All three colouration chemicals had no effect on the combustibility of timber when included into the fire-retardant formulations.
- * The inclusion of a colourant into the fire-retardant can be performed in such a manner that fire-retardant treatment economy is hardly affected.
- * Large scale evaluations need to be performed to verify colour efficiency over time.

(c)

(b) OPSOMMING

Dit is uiters moeilik om visueel tussen mynhout wat behandel is met brandvertraag chemikalië, hoofsaaklik ammonium sulfaat en ammonium fosfaat soute, en onbehandelde mynhout te onderskei. Daar is bykans geen visuele onderskeid sodra die behandelde hout toegelaat was om vir drie dae lugdroog te word.

Die projek doelwit is om 'n geskikte kleurstof te vind wat tesame met die brandvertraag chemikalie gebruik kan word om sodoende die brandvertraag behandelde hout visueel kan onderskei van onbehandelde mynhout op 'n koste-effektiewe basis. Evaluasie wat die ondergenoemde insluit is uitgevoer:

- Aanpasbaarheid en stabiliteit wanneer gemeng met brandvertraag chemikalië.
- Kleur effektiwiteit en langdurigheid.
- Effek van kleurstof op brandbaarheid.

Die resultate toon dat:

- * Drie van die kleurstowwe wat ondersoek is, aanpasbaar en stabiel voorkom by twee blootstellings temperatuur reekse. Die kleurstowwe is Acid Green, Bazol Red 76L en Malachite Green.
- * Tydens blootstelling gedurende die twee metodes, gekontroleerde verwerking en natuurlike blootstelling, Malachite Green en Bazazol Red 76L die beste vertoon het.

(d)

(b) OPSOMMING (vervolgend)

- * Daar is gevind dat geen van die kleurstowwe 'n invloed op die brandbaarheid getoon het nie.
- * Die byvoeging van 'n kleurstof tesame met die brandvertraagmiddel dra minimaal by tot die koste van brand vertraag behandeling.
- * Groot-skaal se behandeling tesame met die kleurstof behoort uitgevoer te word om kleur effektiwiteit te bevestig.

1. INTRODUCTION

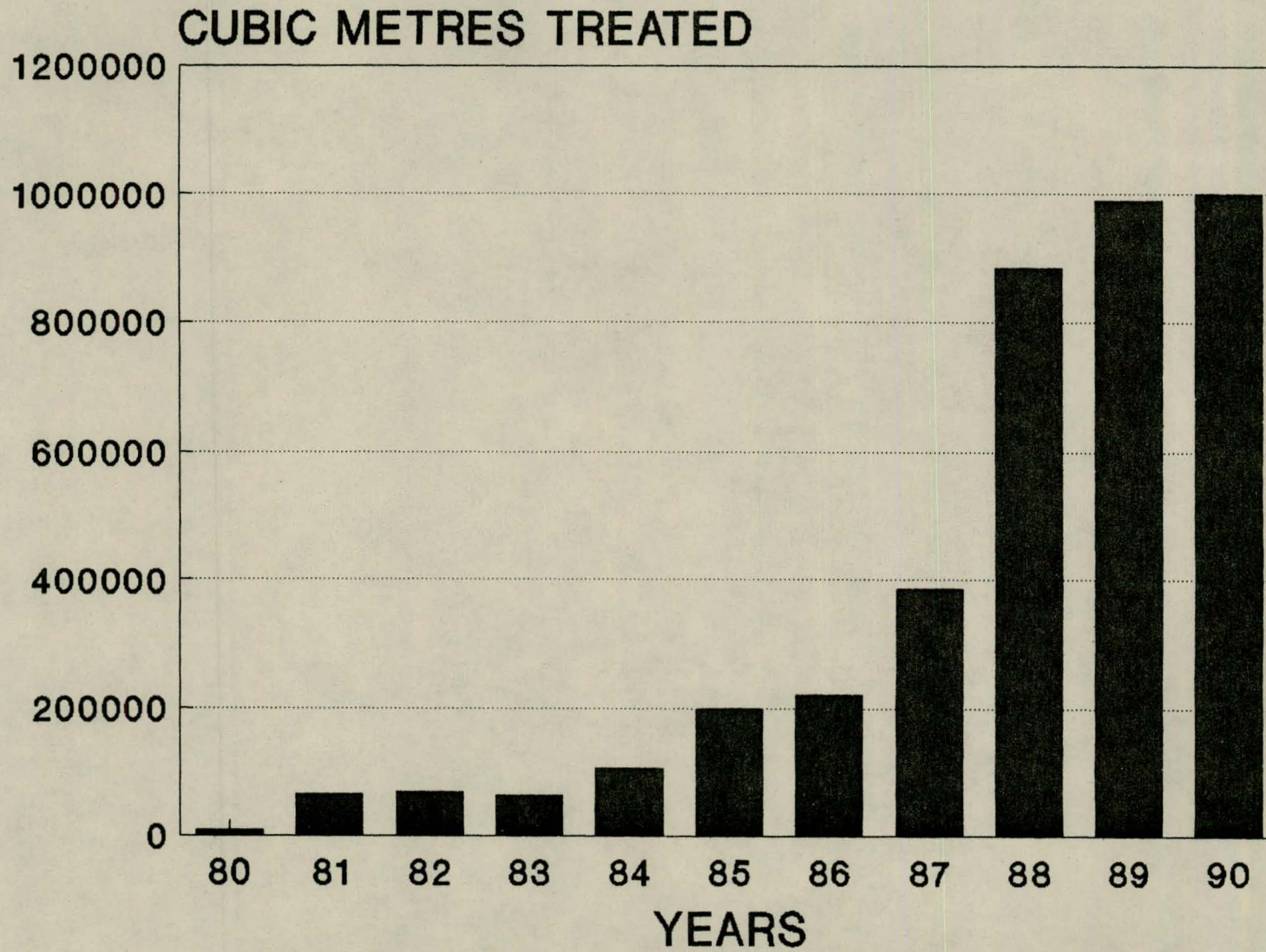
Fire-retardant treated mining support timber was introduced to the South African mining industry during 1979. Western Deep Levels mine was the first company to use fire-retardant treated timber support. During the following decade the use of fire-retardant treated timber increased substantially.¹ For detail, refer to figure 1.

The chemicals used in the pressure/vacuum impregnation system consists of ammonium sulphate and ammonium phosphate salts which are mixed in various ratios and dissolved in water, which acts as the carrier liquid during treatment. The proportional mixing of the abovementioned salts is related to cost effectiveness of the product. Currently, approximately 74 percent of the volume of timber treated is treated utilising a formulation consisting of 70 percent ammonium sulphate and 30 percent ammonium phosphate, whilst the remainder of the timber i.e. 26 percent, is treated using a 50 percent ammonium sulphate and 50 percent ammonium phosphate formulation.

Industry and customer specifications require fire-retardant treated timber to perform under comparative fire conditions in such a manner that when compared to untreated timber under given test parameters, the mass loss will be 33 percent less than that of untreated timber. In order to achieve the abovementioned performance, chemical retentions of 18,2 kg/m³ and 20,7 kg/m³ are required when using 50AS:50AP and 70AS:30AP formulations respectively.

As fire-retardant treatment adds approximately 9 to 12 percent to the timber cost, fire-retardant treated support timber is not used on all the mines. Currently, approximately 50 percent of support timber used is treated. Due to the low gold price (\pm \$358 per ounce), a

FIGURE 1: GROWTH IN TREATED VOLUME



number of mines have become marginal and some are even running at a loss.¹ For detail refer figure 2.

Within a mine some areas are classified as high risk areas, specifically referring to fire risk, i.e. support around shafts, winches and gulleys. As safety is of the utmost importance in the mining industry, fire retardants have a definite role to play; however, there is a cost associated and mines which do not have sufficient funds to treat 100 percent of their timber, choose not to treat at all.

One of the main reasons for the abovementioned happening is that once the fire-retardant treated timber has dried to 25-30 percent moisture content and has been exposed for ± one week, there is no visual distinction between treated and untreated timber. On average, timber (treated and untreated) stock of approximately 6 weeks is held above ground, prior to timber moving into the underground situation. It is therefore obvious that once at the mine, no visual distinction can be made between fire-retardant treated and untreated mining support timber.

Methods other than burning the timber to distinguish between fire-retardant treated and untreated timber exist, but are not totally practical, and are detailed below.

a) Spray timber with a (2N) NaOH solution. Ammonia gas is released and the distinctive ammonia smell will indicate the presence of either ammonium sulphate and/or ammonium phosphate.

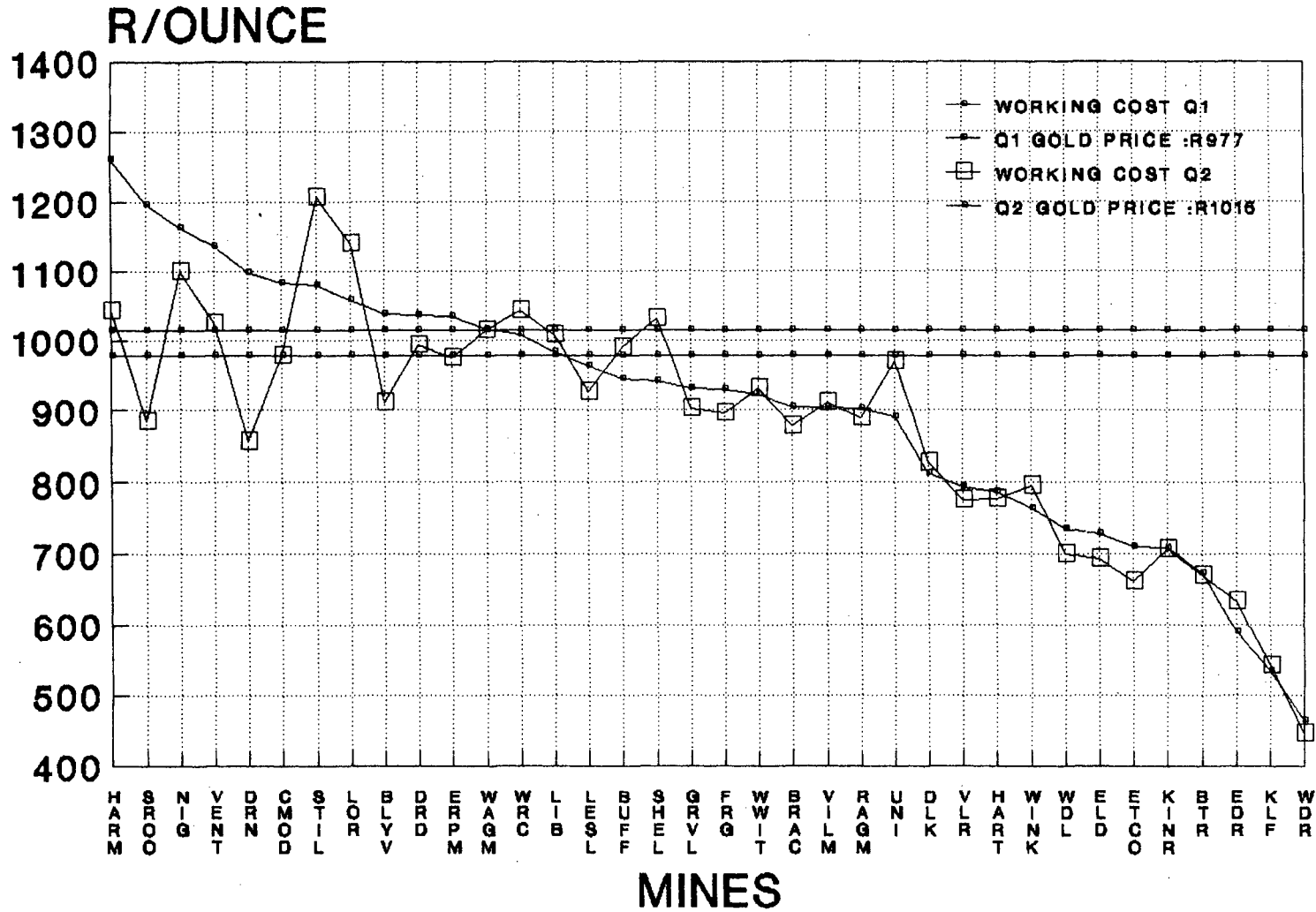
Ammonium can be prepared by the action of a strong base on an ammonium salt



Therefore:

Ammonium based fire retardant salt + (2N)NaOH -->
NH₃ + byproducts.

FIGURE 2: GOLD MINE PRODUCTION COSTS (EXCLUDING CAPEX) - Q1 & Q2 1991



- b) Spray timber with a Quimociac¹ solution and the presence of phosphate will ensure that the timber surface turns to a yellow colour. Product shelf-life, costs and lack of intensity of yellow colour are disadvantages related to the use of Quimociac in this application.

This study deals with qualitative evaluation on colourants and their ability to provide colour on a cost and performance effective basis when used in conjunction with ammonium sulphate and ammonium phosphate salt based fire-retardants.

Due to the qualitative nature of this study, reduced sample size was used and thus statistical interpretations were not performed.

2. OBJECTIVES OF THIS STUDY

The study objective is to:.

1. Qualitatively identify a suitable colourant which can be incorporated into the fire-retardant formulation, which will allow visual distinction between fire-retardant treated mining support timber and untreated mining support timber on a cost-effective basis.

2. The colourant once incorporated in the fire-retardant mixture will be evaluated for the following aspects:

- compatibility and stability within fire-retardant mixtures,
- colour effectiveness and fastness (moisture, UV, light, temperature);
- effect on combustion.

The study deals with the research done in achieving the objective.

2.1. ACKNOWLEDGEMENTS

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- * HL&H Mining Timber for financial support for this study.
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- * Mr M Erasmus and his staff from Vaal Reefs Gold Mine (No.9 Shaft) for assistance in exposure trials underground.

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- * Dr G van der Klashorst of the C.S.I.R. for project guidance.
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- * Lastly, sincere thanks to my family, Miriam in particular, for the motivation in completing this study.

2.2. DEFINITION AND VOCABULARY USED

- ..AS:..AP : Notation to depict a specific formulation with .. being representative of the actual active salt concentrations of ammonium sulphate and ammonium phosphate.
- End-matched : Samples are cut out of an individual specimen in such a manner that sample orientation is identical.
- Formulation : A given ratio of ammonium sulphate to ammonium phosphate salts.
- Freshly mixed Chemical : Mixed chemical containing raw material which has not been used to treat timber, i.e. consists of raw material and water only.
- Retention : Mass of active salt present per specified volume of timber.

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Slabs : The description of a mining timber pole which has two opposite parallel faces.

Trims : The description of a mining timber pole which has four opposite parallel faces.

Weather-o-meter : Apparatus used for controlled weathering.

Working solution: Fire-retardant solution which has been used in the treating process.

3. ESTABLISHED LITERATURE

An extensive literature survey was undertaken focusing on establishing whether technology exists to add colour to fire-retardant chemicals in order to generate a treated product which could be visually distinguished from untreated material. Simultaneously, literature which detailed information relating to means of colouring, colour sensing and colour measurement was scanned to achieve an understanding of colour.

The literature relating to the colouring of timber (reference 2,3,4 and 5) refers to timber which is treated with timber preservatives. No literature on addition of colourants to fire-retardant treated timber using ammonium sulphate and ammonium phosphate chemicals could be found.

3.1. Solid Timber Colouring: Related Literature

In an attempt to yield brown CCA treated timber, Poppen, H² (Germany) added azo or metal complex dyes to preservative salts which form stable aqueous solutions. A mixture containing 1% acid brown 237 and 99% salt blend composed of 35% $\text{CuSiF}_6 \cdot 6\text{H}_2\text{O}$ and 65% $(\text{NH}_4)_2\text{Cr}_2\text{O}_7$ was dissolved in H_2O to give a solution with 2-4% solid content for use in vacuum pressure impregnation of wood.

Altering the natural colour of low grade lumber was achieved by Watamanstu, K (Japan)³ utilising HNO_3 and NaClO solutions. Low grade cedarwood boards were immersed in 30% aq NaClO for 40-90 minutes at 16°C to colour the boards in yellow shades with an appearance of Japanese cedarwood.

In coloured board manufacture,⁴ woodlike shade variation and depth is obtained by impregnating wood

with a resin laminating with a board using an adhesive containing a colourant and coating the laminate with coloured compounds. The laminates are hot pressed, coated with a compound containing a colourant and dried to give a decorative veneer with a woodlike shade variation and depth.

Solid compounds containing polishing powder, red oxide and carbon black are useful for colouring wood in a woodlike reddish shade.⁵ 1000 grams polishing powder is mixed with 400 ml H_2O , 30 grams red oxide and 30 ml India ink, and dried to give a solid reddish colouring compound for wood.



3.2. Colour Science⁶

Details of the following sections, 3.2.1 to 3.2.7 are based on information extracted from: Encyclopedia of Chemical Technology, Volumes 6, 8, 17 and 22. The purpose of this inclusion is to give the reader a basic understanding of colour.

3.2.1 Colour Sensing

Colour is defined as part of the visual experience that deals with aspects of objects other than their size, shape and surface texture.

Most literature on colour assumes that only three variables are required for a complete description of colour. These are referred to as hue, luminosity and saturation. This is an over-simplification and theoretically only applies to unrelated colours which are seen as self-luminous stimuli, bearing no relation to the background and surrounding colour. For related colours, whose appearance depends on the surrounding and background, four variables are required to define colour. Re the following:

If the achromatic attributes present in all colours, including the whites, greys and black which do not possess a hue, are considered, the first perceptual attribute present is termed brightness. The brightness of any object or area relates to the amount of light emitted from such an object or area

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and is relevant for both related and unrelated colours. However, in related colours, a second attribute, lightness is defined which describes ability to reflect a greater or smaller fraction of incident light.

Of the chromatic attributes, the most easily defined term is hue, which denotes whether a colour is red, blue, yellow, etc. More difficult and controversial are the terms indicating the "strength" of the chromatic response by which the hue is recognised. Colourfulness as a definition is frequently used; however, saturation describes the sensation of colour sensing according to an area appearing to exhibit more or less chromatic colour. Saturation tends to remain constant as the level of illumination is changed.

Of the four attributes mentioned above, the visual task of recognising any object usually involves hue, lightness and saturation for related and unrelated colours. The term brightness is added for related colour recognition.

3.2.2 Physical aspects of colour

3.2.2.1. Sources of Light

Light is one type of electromagnetic radiation, limited to the visible region of the electromagnetic spectrum. Sources of light emit power and terms related to this power are called

radiometric terms. The response which such sources of light creates within the eye are called photometric terms. Both these phenomena are described by radiance and luminosity respectively. Radiometric quantities are considered as a function of the wavelength of radiation and referred to as the spectral radiant quantity. Photometric quantities are obtained by multiplying the corresponding spectral radiant quantity by the eye's response to power, the spectral luminous efficiency and integrating the product over the wavelength of the visible spectrum. The total radiant power emitted by a light source is the radiant flux measured in watts (W).

The effect of a light source on colour is accurately described by its spectral power distribution. For critical visual judgement of colour, standard sources are defined and made available in the literature.

3.2.2.2. Interaction of light with objects.

With interaction between light and an object, four aspects play a role in influencing the colour of the objects; (a) absorption, (b) fluorescence, (c) scattering and (d) reflection.

(a) Absorption: Absorption is the process in which radiant power is used to raise molecules in the object to a higher energy state. In their return to the ground state

energy level, molecules dissipate the power in the form of radiation. Spectrally, selective absorption is responsible for colour effects in transparent objects.

- (b) Fluorescence: Fluorescence is a process in which absorption is followed by re-radiation of the flux at only moderately longer wavelengths, still within the visible electromagnetic spectrum. The absorption, however, can occur in the ultra violet region, leading to visible fluorescence in amounts dependent on the spectral power of the source in the ultraviolet region.
- (c) Scattering: Scattering is the interaction in which radiation is redirected without change in wavelength. Atoms, molecules or colloidal particles when irradiated generate small amounts of radiation moving in directions determined by the size of the particle and the polarisation of such radiation. Scattered radiation can be observed directly and may be spectrally selective.
- (d) Reflection: On the macroscopic scale, radiation is reflected at the boundaries of objects where a change in refractive index occurs. Reflection from a rough surface, in

which reflected radiation propagates in a variety of direction, is termed diffuse reflection. Diffuse reflection can result from scattering within the body of an object.

The physical aspects of light vary between the underground and aboveground situation. Timber in the underground situation will be exposed to artificially created "light", whereas timber situated aboveground will be exposed to all aspects of light. Although the objective of this study was to visually distinguish treated timber from untreated timber aboveground, it could be of relevance once mines use more recycled timber.

3.2.3. Colour Vision

The complexity of colour vision via the human eye is great and in the majority of cases, theories are of a speculative nature. Some basic aspects of colour vision are well known.

The process of colour vision is briefly described. Light falling on the retina of the eye is detected for normal levels of illumination by three types of cone receptors, leading to photopic or colour vision. In dim light, a fourth type of receptor known as the rods is activated, but at higher levels of light they play no role in colour vision. The details of the photosensitive pigments in the cones within the eye, their mode of action and their

spectral responsitivity curves have not been unequivocally resolved. It is known that the majority of these cones are in the central region of the retina.

Beyond the retina, the neutral impulses transmitting colour vision information to the brain appear to utilise complex networks. Details as to the brain's method in interpreting colour visions are largely speculative.

As colour vision is critically important to the successful implementation of results of this study, the individual workers dealing with timber on a regular basis should be tested for colour-blindness. Adequate training should be provided to all workers involved with timber as to how to distinguish between untreated and colour treated timber.

3.2.4. Colourants

Colourants are chemical substances which are added to materials to produce colour effects. Colourants are classified into two categories i.e. dyes and pigments.

Dyes are substances that are molecularly dispersed in solutions which are transferred to materials and bound to it by intermolecular forces which will provide colour effects by spectrally selective absorption processes.

Pigments are larger than molecular-particle size and are held in place by their corresponding low mobility. Pigments tend to scatter and absorb light easily.

Traditionally, pigments are inorganic and dyes organic in nature, but many exceptions exist. Synthetic organic dyestuffs are used most frequently. In the paper industry, synthetic organic dyestuffs have been classified as follows, i.e. a) basic dyestuffs, b) acid dyestuffs, c) direct dyestuffs and d) organic pigments.

- a) Basic Dyestuffs: Basic dyestuffs are extremely brilliant and pure and have good tinctorial strength. They have a good affinity for material that contains lignin. Their light fastness is very poor; however, their fastness to water and steam is good. Basic dyestuffs are relatively difficult to dissolve in aqueous solutions. Chemically a further characteristic is the presence of triphenylmethane groupings.
- b) Acid Dyestuffs: Acid dyestuffs are generally represented by an alkali salt of a sulphonic acid of a dyestuff. A typical double bonded nitrogen atom, commonly referred to as an "azo bond", is present in most acid dyestuffs. Due to their anionic properties, acid dyestuffs have to be fixed onto cellulose. They dissolve very easily in water but have poor resistance to water and heat, as well as poor light fastness.

- c) Direct Dyestuffs: Direct dyestuffs are usually sodium salts of azo compounds containing sulphonic or carboxylic acid groups. Their solubility in water is poorer than that of acid dyestuffs. Fastness properties to heat, water and light are good. Direct dyestuffs have less affinity towards lignin than basic dyestuffs.
- d) Organic Pigments: Organic pigments are manufactured as insoluble colourants. Most organic pigment colourants have excellent fastness properties. They do not react with the cellulose fibre at all, but are physically deposited on material to provide colour.

3.2.5. Colourant Mixing

The mixing of dyes and pigments to produce desired colours is a major objective for colour technology, which applies to the colouring of materials in industry. Simple-subtractive mixing and complex-subtractive mixing of colourants occur.

- a) Simple-Subtractive Mixing: This term is reserved for the mixing of colourants in transparent systems in which only absorption is of importance.
- b) Complex-Subtractive Mixing: This term is referred to when colourant mixing takes place where scattering and absorption both play a role.

3.2.6. Colour Measurement

One aspect of colour measurement which is to be borne in mind is that when an individual examines an object, a wide variety of geometric conditions of illumination and view is possible. In the event of instrumentation being used for colour measurement, limitations in this regard exist. Spectrophotometry and Tristimulus colorimeters are most commonly used for colour measurement.

4. MATERIALS

4.1. Fire-Retardants

Ammonium sulphate and ammonium phosphate based fire-retardant salt solutions were used for this investigation. All experimental work was carried out, utilising a formulation consisting of 70 percent ammonium sulphate and 30 percent ammonium phosphate. This formulation is the most cost/performance effective based on the raw material costs. For chemical compatibility, effect on combustion, as well as colour fastness, a formulation consisting of ammonium sulphate and ammonium phosphate in equal ratios was included.

In industry, the average salt concentration of both formulations currently applied, ranges between 130 g/l and 210 g/l.¹ The concentration of made-up chemical was kept within these ranges and in all instances, except one, plant working solutions were used. Fresh raw material chemical was initially used to test for chemical compatibility.

Fire-retardant formulations used in this study are listed in table 1 below:

TABLE 1: FIRE-RETARDANTS

Formulation	Ammonium Sulphate (%)	Ammonium Phosphate (%)	Solution Concentration (g/l)	Solution Density (kg/l)	PH
1	70	30	165-200	1.084-1.103	+/- 6.1
2	50	50	165-204	1.096-1.119	+/- 6.1

4.2. Timber

E.grandis timber samples ranging in sizes as set out in Table 2 were used in this study. Timber samples were collected at the HL&H Orkney Timber Depot. As far as possible, timber containing bark and knots was eliminated due to possible interferences occurring during the treatment process.

TABLE 2. TIMBER SPECIMENS

Dimensions (cm)	Density Range (kg/m ³)	Sap- wood (%)	Heart- wood (%)	Quan- tity
2 x 2 x 1 (l,w,t)	500-550	100	0	180
5 x 10 x 1 (l,w,t)	500-550	100	0	45
9 x 15 (slab) ^a	Not determined	47-72	32-53	40
9 x 15 (trim) ^b	Not determined	43-86	14-57	40
9 x 55 (slab) ^c	Not determined	49-74	26-51	15
9 x 56 (trim) ^d	Not determined	29-85	15-71	15

l = length

w = width

t = thickness

- a. Non-conventional slab i.e. length 15 cm, distance between flat face 9 cm.
- b. Non-conventional trim i.e. length 15 cm, distance between flat faces 9 cm.
- c. Conventional slab i.e. length 55 cm, distance between flat face 9 cm.
- d. Conventional trim i.e. length 55 cm, distance between flat faces 9 cm.

4.3. Treatment Plant

An experimental timber treatment plant with treating cylinder dimensions of diameter 20 cm and length of 65 cm was manufactured. The plant was fitted with a vacuum pump capable of applying a vacuum of ± 60 kPa. A pressure pump with capabilities of ± 1500 kPa was installed.

A pressure relief valve, set to relieve at 900 kPa was installed to regulate maximum pressure.

4.4. Colourants

Eight colourants were evaluated in this study. For ease of reference, each colourant was allocated an alphabetical code. The colourant trade names are listed in table 3 below.

TABLE 3: COLOURANTS

Colourant	Alphabetical Code	Type of Dye
Acid Green	A	Acid
Tectilon Yellow	B	Acid
Fluorescent	C	Basic
BASAZOL Red 76L	D	Basic
Malachite Green	E	Basic
BASAZOL Violet 40L	F	Basic
BASAZOL C Black 21L	G	Basic
BASAZOL Brown 43L	H	Basic .

Colourants A,B,C and E were obtained from Spectrum Agencies whilst colourants D,F,G and H were supplied by BASF. As all colourant names are trade names, the chemical formulae and composition thereof are not commonly disclosed.

Due to their affinity to lignin containing substrates, their brilliancy and favourable costs, this study concentrates on basic dyestuffs. Acid dyestuffs have been included, but acid dyestuffs are normally more costly than the other types of dyes.

No evaluation on chemical additions to "bind" acid dyestuffs to cellulose were performed. Organic dyestuffs were not used due to the fact that organic dyestuffs do not react with cellulose fibre at all.

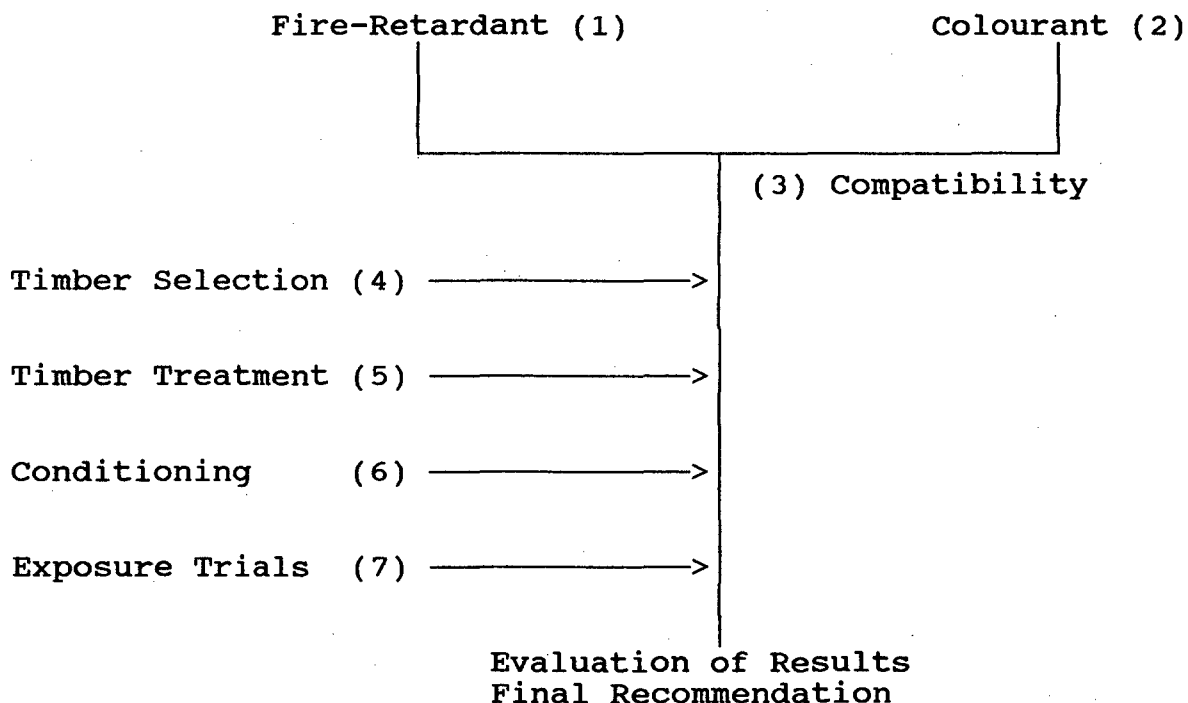
Due to availability and properties, mainly basic dyestuffs were evaluated.

5. METHODS OF PREPARATION AND EVALUATION

This chapter deals with preparation of all chemicals and timber specimens and includes the evaluation techniques used.

5.1. Flow Diagram

The experimental flow diagram is depicted below



Each numerically identified heading in the Flow Diagram will be addressed in the sub-section below, with specific reference to method of preparation and evaluation.

5.2. Preparation and Evaluation

5.2.1. Fire Retardants

Two fire-retardant formulations were utilised throughout the study. Fresh, unmixed industrial grade raw materials, as well as working solution, were used. For detailed application, refer to table 4 below.

TABLE 4: MATRIX OF FIRE-RETARDANTS USED

Formulation	Fresh Unmixed Chemicals	Solution Concentration +/- (g/l)	Working Solution	Solution Concentration +/- (g/l)
70AS:30AP	Chemical Compatibility	165	Chemical Compatibility	165
			Effect on Combustion	185
			Controlled Weathering Trials	185
			Uncontrolled Weathering Trials	185
50AS:50AP	Chemical Compatibility	165	Chemical Compatibility	165
			Effect on Combustion	185
			Controlled Weathering Trials	185

5.2.2. Colourant

During the testing process, colourants which failed any selection criteria were not included in the subsequent tests.

Visual effectiveness of colourant and coloured fire-retardant solutions were carried out using all colourants detailed in Table 3..

5.2.3. Compatibility

All colourants were used and solution concentrations of 1% m/m were made purely to establish the efficacy of the colourant in the fire-retardant formulation, and to ascertain possible complex formation and/or formation of sediment.

Colourant concentrations were lowered to a cost effective level and solutions were made up to ascertain whether ability to mix, complex formation, sedimentation and colour efficiency were affected. Samples were visually evaluated at room temperature and at 4 degrees Celsius for a period of up to 24 and 96 hours respectively.

5.2.4. Timber Selection

E.grandis timber samples were selected as outlined in Table 2. All specimens were end-matched with the

exception of a portion of the 55 x 9 cm trims and slabs. All small samples i.e. dimensions of 50 x 100 x 10 mm or smaller were fine sawn and sanded to accurate dimensions as stated in Table 2. As far as possible, timber containing large cracks and knots were discarded. All samples were individually marked for further reference.

5.2.5. Timber Treatment

Small samples, i.e. dimensions of 50 x 100 x 10 mm and smaller were treated together, using the same process parameters. The process parameters were established using samples of similar size, density and moisture content. Under set process parameters^a, the liquid volume uptake was determined and a process selected to treat all test specimens to the required dry salt nett retention in order to render a 33 percent fire-retardancy. The mass of each sample was determined prior to and directly after treatment.

Large samples, i.e. dimensions larger than 50 x 100 x 10 mm were treated in such a manner as to result in the spread of dry salt retentions and volumetric uptake, which resemble those obtained by the average everyday treatment operation. Process parameters were determined on a trial and error basis until the criteria for volumetric uptake spread and dry salt retentions matched those which are obtained on a daily basis at the treatment operation.

The mass of all samples was determined prior to and directly after treatment. Sapwood determination was performed on all large samples to determine what effect volumetric uptake has on colour efficiency.

5.2.6. Conditioning

Small scale samples, i.e. dimensions of 20 x 20 x 10 mm were conditioned to an average moisture content of 15 percent utilising a CSIR conditioning chamber set at 75% RH and 30° C.

All other samples were stored under cover for a period of five days prior to exposure trials. Sample moisture content was not determined.

5.2.7. Exposure Trials

a) Effect on Combustion

Samples measuring 20 mm x 20 mm x 10 mm were exposed to a heat source as stipulated in proposed SABS Test Method 1088, in order to determine whether the inclusion of the colourant had any influence on the fire-retardancy. The basic experimental procedure as stipulated in the test method comprises the following process:

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- Sample mass determined prior to exposure.
- Sample individually exposed to a heat source of $600 \pm 10^{\circ}$ C in a silica crucible.
Size (mm): 28 high x 56 diameter.
- Sample mass determined directly after a one minute exposure period.
- Percentage mass loss calculated.

Twenty end-matched samples per formulation per colour were tested.

b) Weather-o-Meter Trials

40 samples measuring 50 x 100 x 10 mm were exposed in a weather-o-meter. Three sets of uncoloured samples treated with a 70AS:30AP and 50AS:50AP were included, together with untreated samples as controls. A set of samples per colour per formulation were treated and evaluated. Each set consisted of five end-matched samples. One set of the end-matched samples was removed every 50 hours. Indications of colour loss were determined over a 200 hours exposure period. The samples were mounted on the circumference of a cylindrical tube which continuously rotated, in the process exposing the samples to UV radiation, daylight, infra-red radiation and water submerging.

Exposure Conditions:

- i. Rotation Speed - 1 cycle per 48 minutes.
- ii. UV Radiation - 2 x 30 W Fluorescent lights, 32 minutes exposure per cycle.
- iii. Daylight - 1 x 30 W Fluorescent light, 16 minutes exposure per cycle.
- iv. Infra-red radiation - 2 Inculoy elements. 17 minutes exposure per cycle.
- v. Water submerging - Slow running tapwater. 8 minutes exposure per cycle.

Due to temperature surrounding apparatus continuously changing, it was not monitored; however, it can be stated to be above room temperature. Relative humidity was not measured. Reflected rays were not brought into account.

The samples were evaluated using two techniques; firstly, determining whether the colour-treated sample could be visually distinguished from the treated sample containing no colour. Secondly, the amount of coloured area present on the end-matched

face was determined, using a point matrix where points were placed at 5 mm interval spacings. The area coloured was quantified and plotted against time to assess the rate of colour loss.

Wash water and timber samples were not analysed to determine quantity of colourant leached or remaining in the timber sample.

c) Natural Exposure Trials

Samples measuring 90 mm x 150 mm (slabs and trims) were exposed at two of HL&H's site depots, i.e. Westonaria and Welkom. Samples were exposed above ground and monitored for a period of nine weeks. During the exposure period, notes relating to weather conditions and distinguishability between colour treated and non- colour treated samples were made on a twice-a-week basis.

d) Underground Exposure Trials

Samples measuring 90 mm x 550 mm (slabs and trims) were allowed to air dry for a period of four weeks under cover. The samples were sent underground to Vaal Reefs No.9 Shaft, where conditions were monitored. The samples were mounted on the first row of packs adjacent to the face to ensure periodical

washing down. As soon as a new row of support packs were installed, the samples were moved to the newly installed row of support packs to ensure most severe washing down at the rock face. Samples were exposed in this manner for a period of five weeks. Samples were retrieved and evaluated as to whether any of the colouration was still present.

6. RESULTS AND DISCUSSION OF RESULTS

6.1. Chemical Compatibility

Sedimentation moults of both formulations at two concentrations at ambient temperature are detailed in figures 3, 4, 5 and 6 and at 4 degrees Celsius in figures 7,8, 9 and 10.

All colourants are referred to as in the alphabetical code set out in Table 3.

Colours F, G and H proved difficult in dissolving at the one percent concentration level; however, once concentration levels were lowered to 0,0078 percent, colour F dissolved successfully. This held true for both the 70AS:30AP and 50AS:50AP fire-retardant formulations. Colour C formed a complex as soon as mixing began in all the one percent solution concentrations.

The colour efficiency was good with the exception of colourants F, G and H. The reason for this can possibly be ascribed to the fact that the colourant did not totally dissolve.

Once mixed, all samples were allowed to settle out over a period of time at $20 \pm 3^{\circ}\text{C}$ and $4 \pm 2^{\circ}\text{C}$ for different periods. The samples at ambient were evaluated first and only selected samples were exposed at lower temperatures.

The key used for determination of quantity of sedimentation was as follows:

0 = Nil = Clearly less sedimentation than in uncoloured fire-retardant

2 = Small = Same amount of sedimentation than uncoloured fire-retardant.

4 = Med = More sedimentation than fire-retardant but supernatant liquid has distinct colour differential.

6 = Large = More sedimentation than fire-retardant with supernatant liquid showing little or no colour differential, (i.e. clear).

As vigorous mixing occurs in the treatment plant during start-up, the ease of re-mixing after sedimentation period was determined. No difference between the various formulations and colourants evaluated over time could be established and all samples, with the exception of F, G and H, were very easily brought back into suspension with minimal effort of mixing.

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There appears to be a correlation between the amount of sedimentation formed over a period of time and the actual colourant concentration. Due to lack of data points, this correlation has not been established.

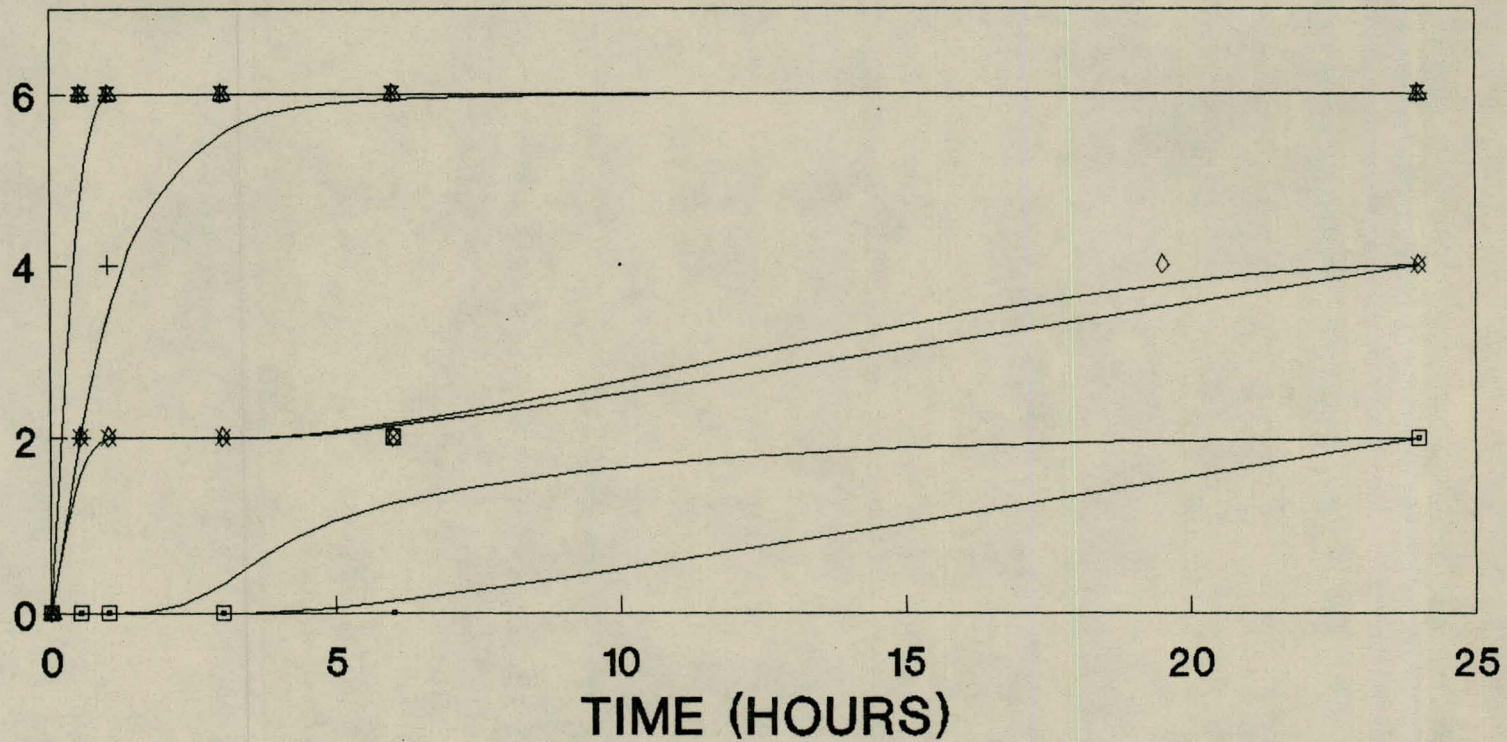
Fresh raw material was used in this experiment as the working solution normally contains a substantial quantity of foreign matter such as sawdust and dust.

Based on sedimentation results, all colours with the exception of A, D and E, were eliminated from the study.

FIGURE 3 : SEDIMENTATION EVALUATION

70AS:30AP, COLOUR @ 1 %m/m, 20 DEG C

SEDIMENTATION

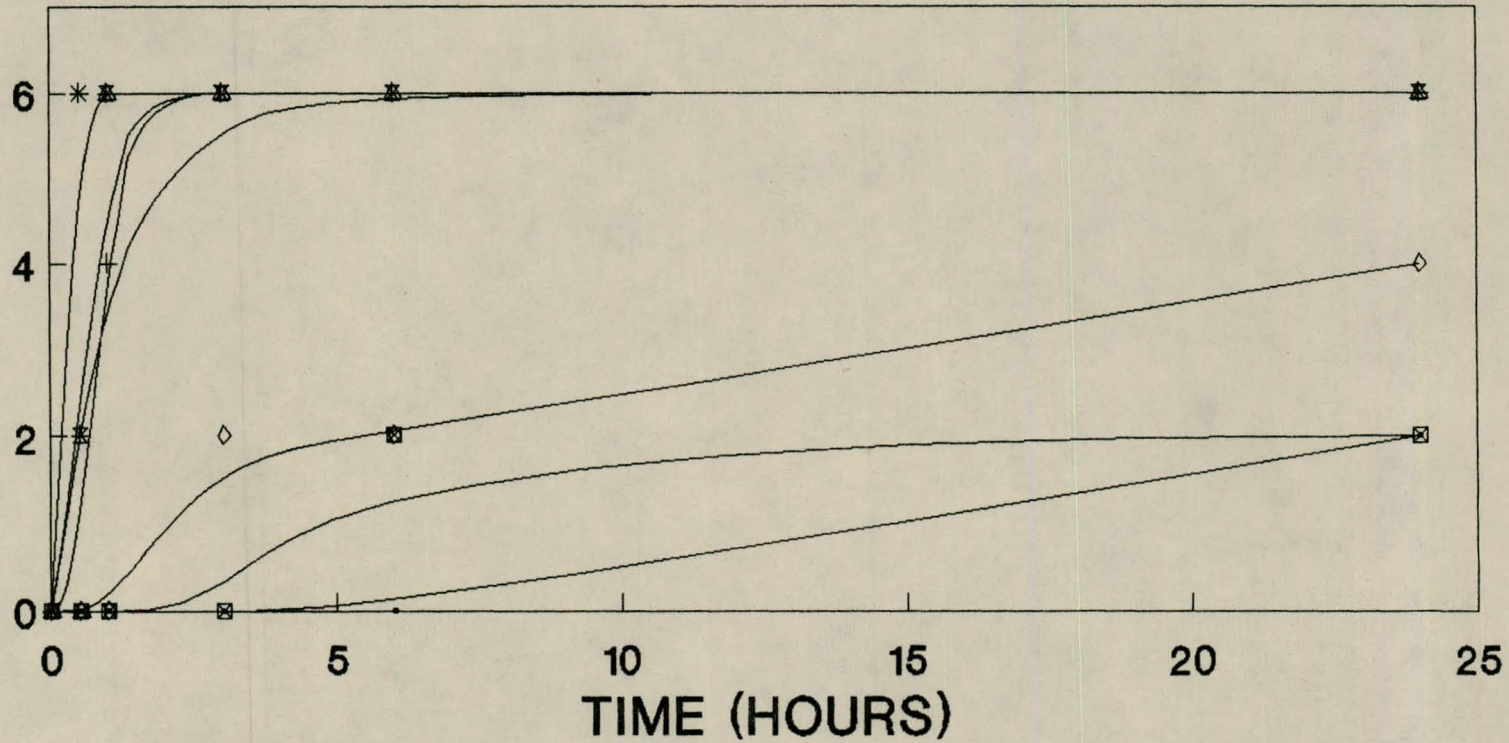


—•— COLOUR A —+— COLOUR B —*— COLOUR C —□— COLOUR D
—×— COLOUR E —◇— COLOUR F —△— COLOUR G —⊗— COLOUR H

FIGURE 4 : SEDIMENTATION EVALUATION

70AS:30AP, COLOUR @ 0.0078% m/m, 20 DEG C

SEDIMENTATION

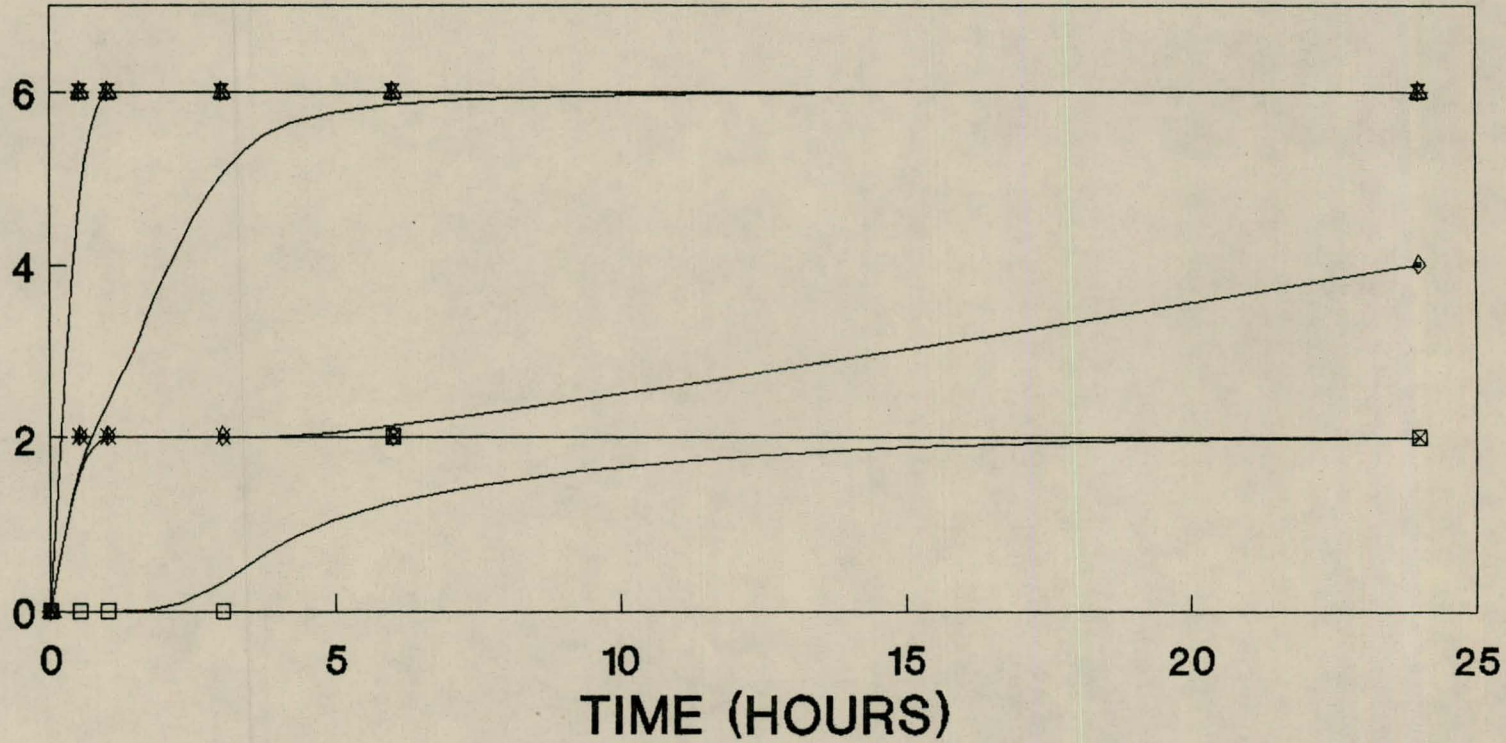


—●—	COLOUR A	—+—	COLOUR B	—*—	COLOUR C	—□—	COLOUR D
—×—	COLOUR E	—◇—	COLOUR F	—△—	COLOUR G	—⊗—	COLOUR H

FIGURE 5 : SEDIMENTATION EVALUATION

50AS:50AP, COLOUR @ 1.0 %m/m, 20 DEG C

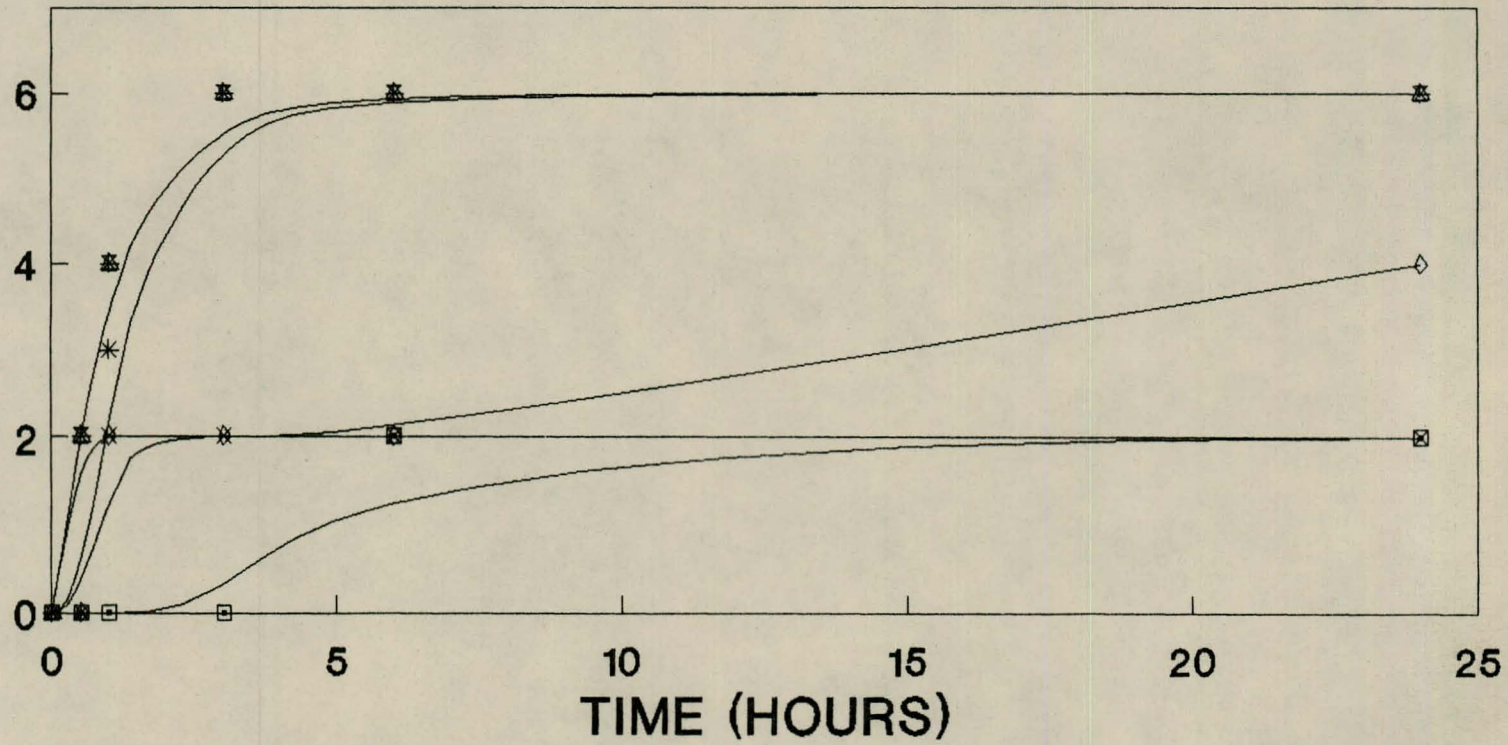
SEDIMENTATION



- COLOUR A
- +— COLOUR B
- *— COLOUR C
- COLOUR D
- ×— COLOUR E
- ◇— COLOUR F
- △— COLOUR G
- ⊠— COLOUR H

FIGURE 6 : SEDIMENTATION EVALUATION
 50AS:50AP, COLOUR @ 0.0078% m/m, 20 DEG C

SEDIMENTATION

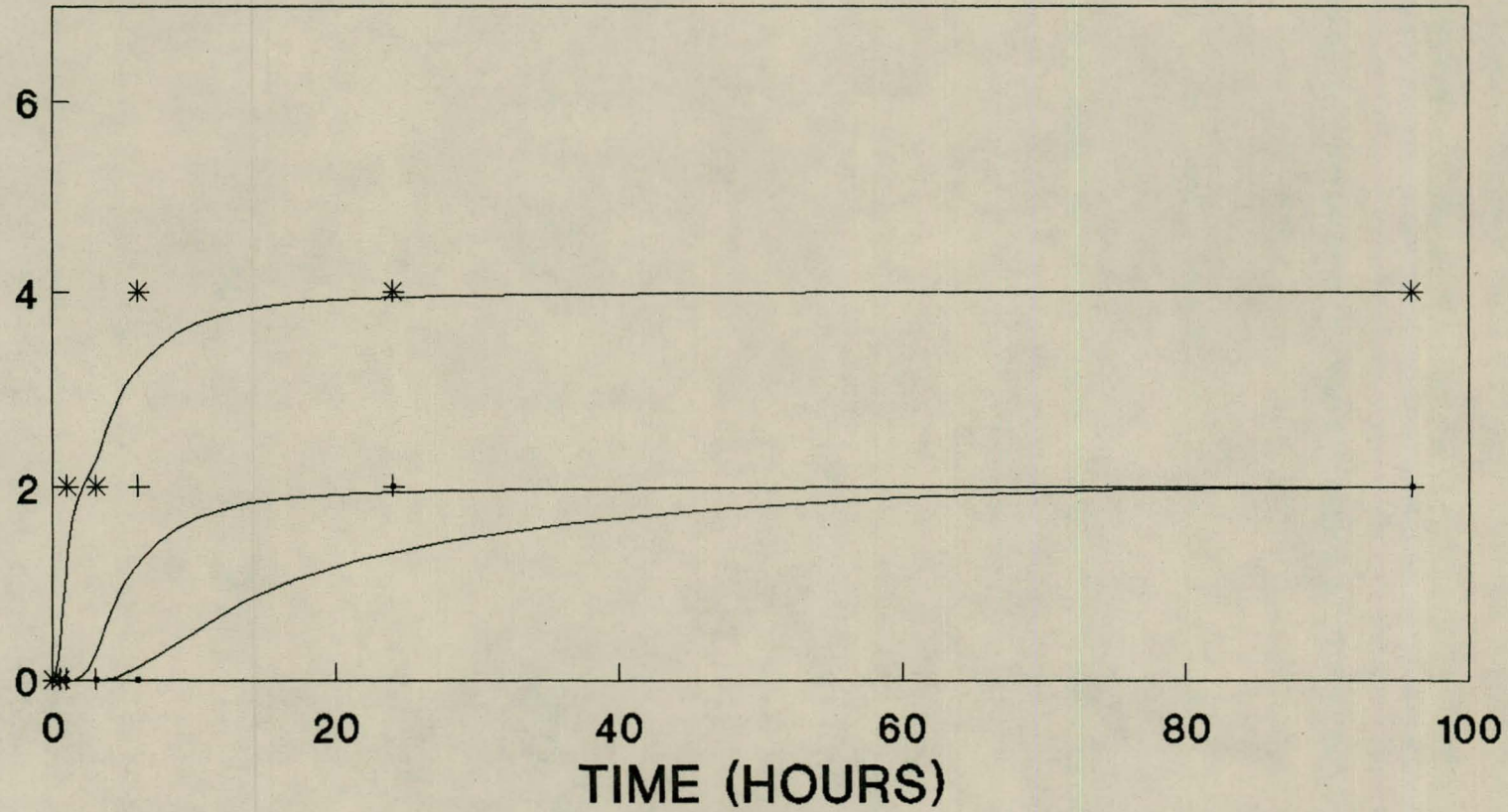


- | | | | |
|--------------|--------------|--------------|--------------|
| —●— COLOUR A | —+— COLOUR B | —*— COLOUR C | —□— COLOUR D |
| —×— COLOUR E | —◇— COLOUR F | —△— COLOUR G | —⊗— COLOUR H |

FIGURE 7 : SEDIMENTATION EVALUATION

70AS:30AP, COLOUR @ 1.0 %m/m, 4 DEG C

SEDIMENTATION

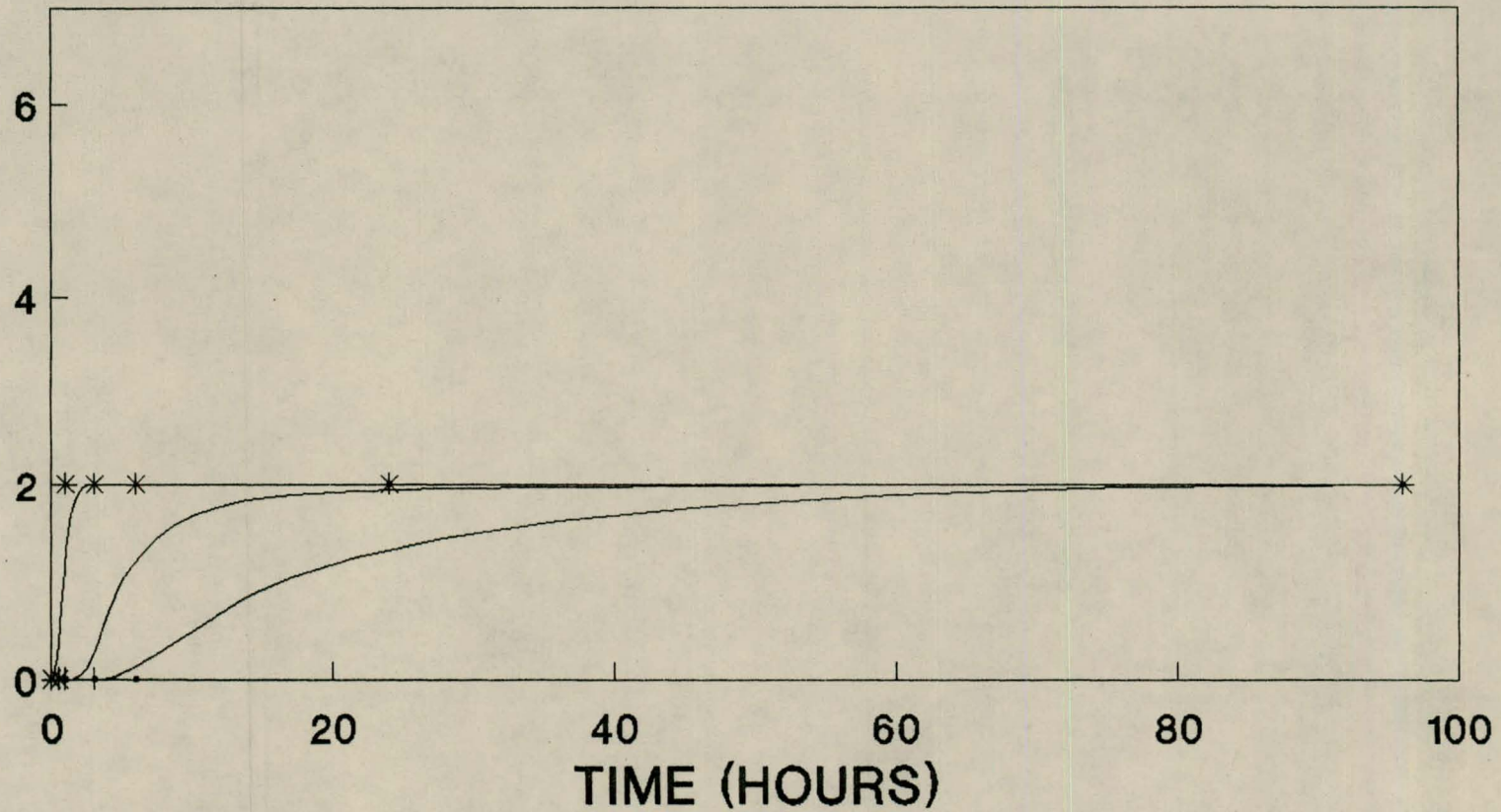


—•— COLOUR A —+— COLOUR D —*— COLOUR E

FIGURE 8 : SEDIMENTATION EVALUATION

70AS:30AP, COLOUR @ 0.0078% m/m, 4 DEG C

SEDIMENTATION



— COLOUR A + COLOUR D * COLOUR E

FIGURE 9 : SEDIMENTATION EVALUATION

50AS:50AP, COLOUR @ 1.0 %m/m, 4 DEG C

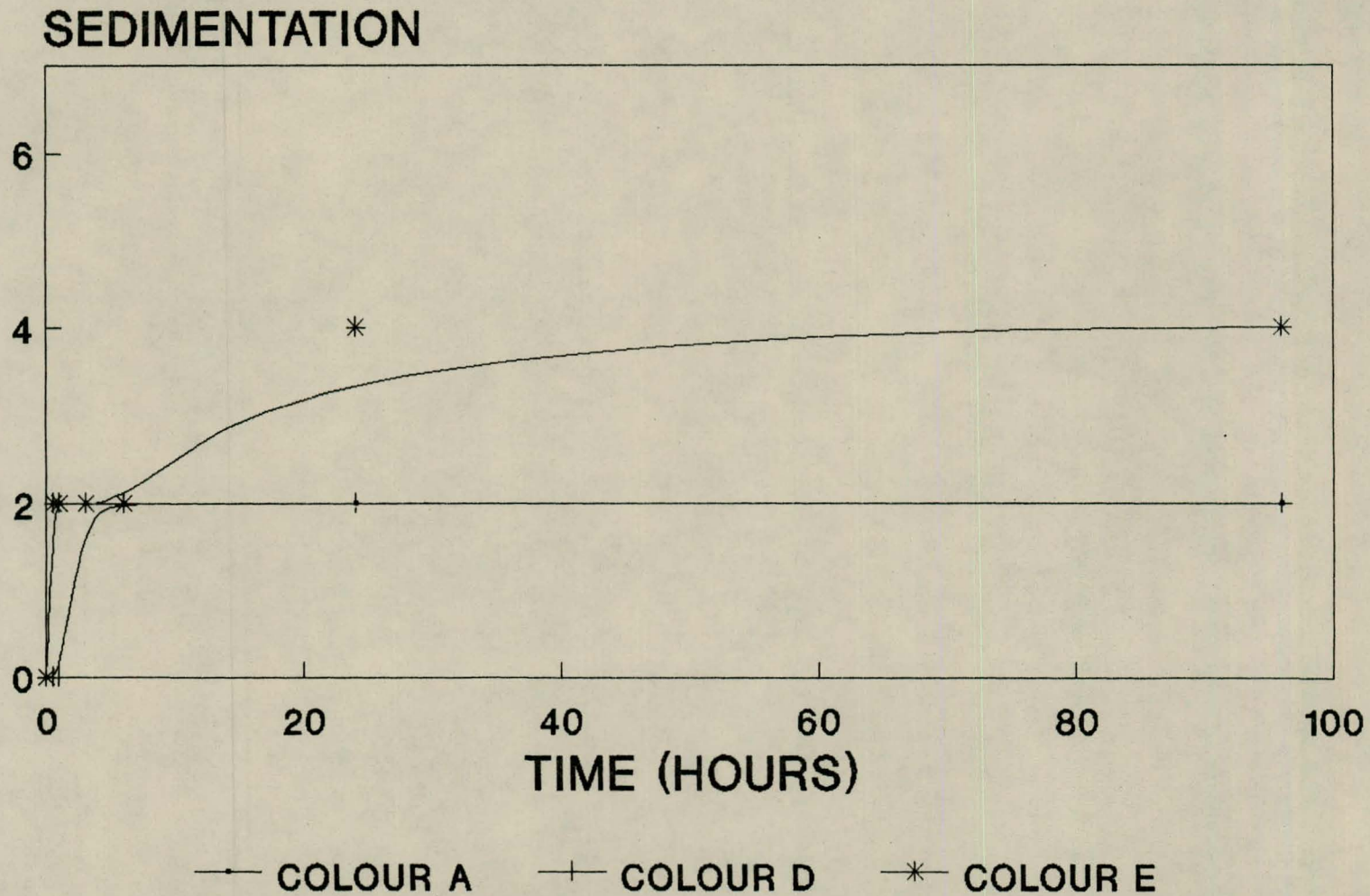
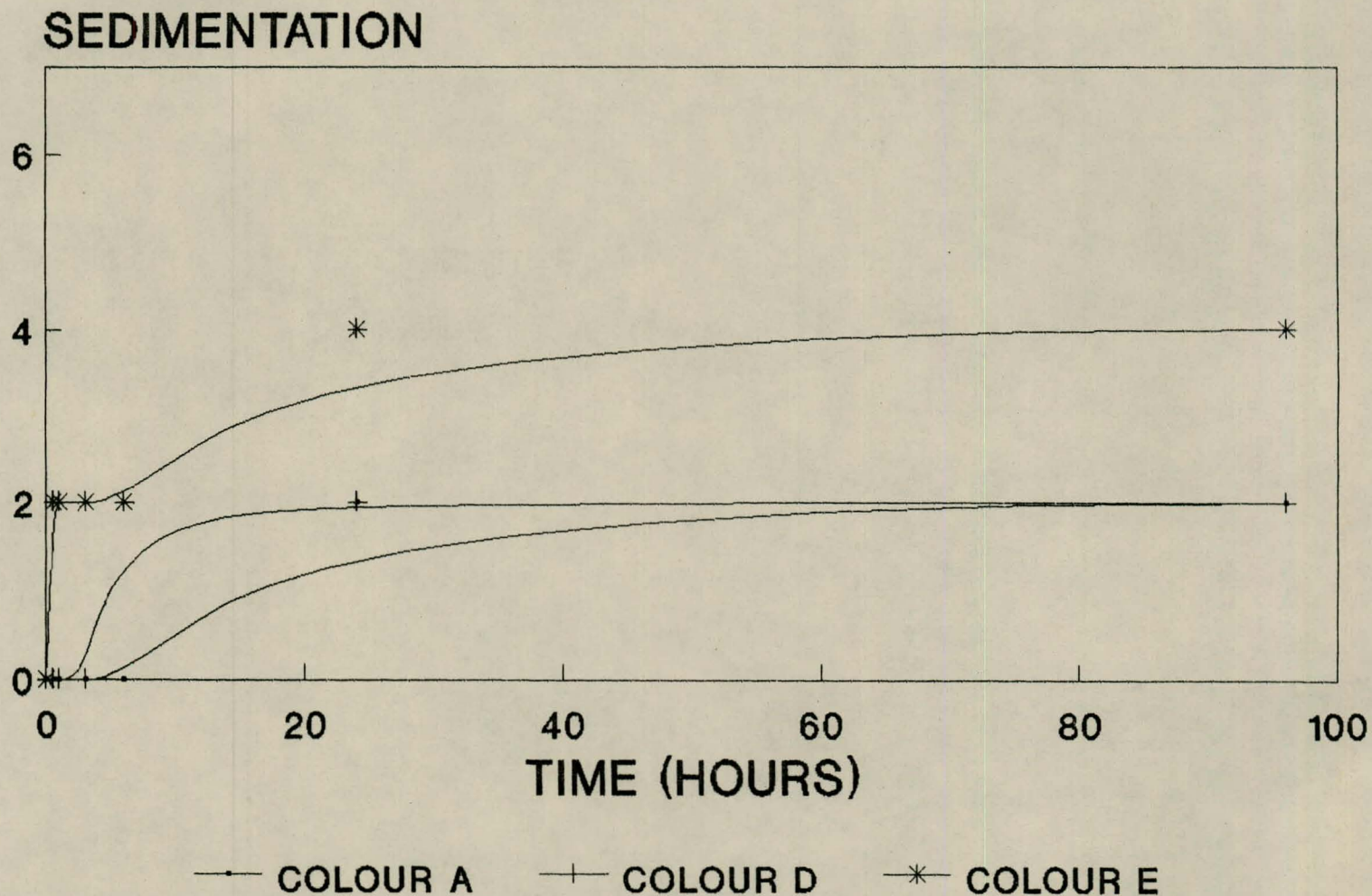


FIGURE 10 : SEDIMENTATION EVALUATION

50AS:50AP, COLOUR @ 0.0078% m/m, 4 DEG C



6.2. Effect of Colouration on Combustion

End-matched samples with dimensions of 20 x 20 x 10 mm were treated to target retentions as stipulated in Table 5 below. Fire retardant and colouration concentrations were kept constant and samples were conditioned to a final moisture content of 15 percent. A conditioning chamber with parameters set at 30°C and a relative humidity of 78 percent was used to ensure samples were at 15 percent moisture content.

In order to achieve the given dry salt retentions, process parameters were established by experimenting with similar untreated samples. The parameters finally used were:

- submerge samples under chemical for 1 minute;
- place the submerged samples inside the autoclave (samples are weighted down) and seal autoclave;
- subject the autoclave containing the samples to a vacuum of 60 kPa for a period of 4 minutes (an initial period of 3,5 minutes is required to allow vacuum to increase from 0 to 60 kPa gauge pressure);
- relieve vacuum, retrieve samples and determine their mass on an individual basis.

Due to the samples being 100 percent sapwood, chemical uptake was high during the vacuum cycle and a pressure cycle could be eliminated.

TABLE 5: TREATMENT RESULTS

Sample Code	Quantity	Description	Timber Density (kg/m ³)	Fire Retardant Salt Concentration (g/l)	Target Sapwood DSNR (kg/m ³)	Actual Sapwood DSNR (kg/m ³)
B	20	70AS:30AP + No Colour	538 (+/- 5.97)	188	41.4	36.8 (+/- 0.98)
C	20	50AS:50AP + No Colour	587 (+/- 11.89)	193	36.4	36.4 (+/- 1.31)
D	20	70AS:30AP + Colour A*	525 (+/- 4.52)	195	41.4	44.7 (+/- 0.70)
E	20	70AS:30AP + Colour D*	522 (+/- 4.79)	195	41.4	41.2 (+/- 2.14)
F	20	70AS:30AP + Colour E*	545 (+/- 7.04)	191	41.4	40.7 (+/- 2.24)
G	20	50AS:50AP + Colour A*	590 (+/- 7.51)	197	36.4	41.1 (+/- 1.77)
H	20	50AS:50AP + Colour D*	539 (+/- 10.45)	191	36.4	42.1 (+/- 1.21)
I	20	50AS:50AP + Colour E*	530 (+/- 15.30)	204	36.4	38.2 (+/- 1.59)

*Colour concentration used was 0.0078% m/m.

Figures in parentheses represent standard error of mean (95% confidence limits).

Sample conditioning and burning trials were performed as described.

TABLE 6: COMBUSTION RESULTS

Sample Code	Quantity	Description	Moisture Content Prior to Burn %	% Mass Loss During Exposure
B	20	70AS:30AP + No Colour	15.2 (+/- 0.04)	34.7 (+/- 1.77)
C	20	50AS:50AP + No Colour	15.1 (+/- 1.21)	35.1 (+/- 1.03)
D	20	70AS:30AP + Colour A*	14.9 (+/- 1.35)	31.8 (+/- 1.21)
E	20	70AS:30AP + Colour D*	15.0 (+/- 1.07)	32.0 (+/- 2.01)
F	20	70AS:30AP + Colour E*	15.7 (+/- 1.35)	31.0 (+/- 1.91)
G	20	50AS:50AP + Colour A*	14.4 (+/- 1.68)	28.0 (+/- 1.91)
H	20	50AS:50AP + Colour D*	15.4 (+/- 1.07)	26.9 (+/- 0.42)
I	20	50AS:50AP + Colour E*	15.9 (+/- 1.12)	30.4 (+/- 2.24)

*Colour concentration used was 0.0078 % m/m.

Figures in parentheses represent standard error of mean
(95% confidence limits)

Due to the known influence which density has on mass loss of untreated sapwood during exposure, the sample ranges containing higher density samples, i.e. C and G, should be interpreted bearing this in mind. The equation which describes the effect of density was established by Conradie W.E. and Thobye C.J. in 1989.⁷ It is represented by:

$$y = 99,26 - 0,080 x \quad (r = 1.0)$$

where

y = estimated mass loss percentage

r = density at $\pm 9,8\%$ moisture content expressed in kg per m³

c = correlation coefficient

It would thus be expected that sample ranges C and G would produce lower mass loss results. This unfortunately does not transpire for the test results, and is complicated by the higher than target retentions for sample range G.

As the sapwood retentions obtained with the control sample range, i.e. B is slightly below target, established fitted curves relating to the performance of 70AS:30AP and 50AS:50AP fire-retardant formulations were used. These equations were established by Conradie W.E. and Thobye C.J.⁹ in 1989, using a range of salt retentions and determining percentage mass loss after exposure.

Relationship between mass loss as a result of thermal degradation and chemical retention for various formulations as determined by Conradie W.E. and Thobye C.J. are given in Table 7 below:

TABLE 7: RELATIONSHIP BETWEEN MASS LOSS PERCENTAGE AT VARIOUS CHEMICAL RETENTIONS.

Formulation	Equation	r
70AS:30AP	$y = 79.09 - 12.72 \ln x$	0.93
50AS:50AP	$y = 66.56 - 10.50 \ln x$	0.90

Where y = estimated mass loss percentage.

x = chemical retention (kg/m^3 sapwood).

r = correlation coefficient.

Utilising equations in Table 7 above, and calculating estimated mass loss percentages of sample range D to I, the following is obtained and depicted in Table 8 below.

TABLE 8: ESTIMATED VS ACTUAL MASS LOSS PERCENTAGES

Sample	Estimated Mass Loss (%)	Actual Mass Loss (%)	Difference (%)
D	30.8	31.8	3.2
E	31.8	32.0	0.6
F	31.9	31.0	- 2.8
G	27.5	28.0	1.8
H	27.3	26.9	- 1.5
I	28.3	30.4	7.4

Bearing in mind the correlation coefficients of the equations in Table 7, as well as the role influence which density plays in the mass loss equations, differences as calculated in Table 8 could be ignored.

It can therefore be deduced that the addition of the colourant at the very low levels of 0,0078 percent has no significant effect on the mass loss of timber under exposure to heat source as described earlier.

6.3. Weather-O-Meter Trial Results

Eight sample strips, each consisting of five end-matched samples measuring 50 x 100 x 10 mm of clear sapwood were treated as described in section 5.2. (5). The same samples were used to evaluate the colour effectiveness over time and the results are reported and discussed below.

As the percentage sapwood to heartwood volume ratio in mining timber is approximately 1:1, a sapwood retention of liquid of approximately 180-220 l/m³ was regarded as average based on actual charge sheet analysis!

The treatment results of samples are detailed in table 9 below:

TABLE 9: TREATMENT RESULTS OF SAMPLES TO BE USED IN THE WEATHER-O-METER EXPOSURE TRIALS

Sample Range	Description	Volumetric Chemical Uptake (l/m ³ Sapwood)
A	Untreated Control	-
B	70AS:30AP + No Colour	202 (+/- 39.3)
C	50AS:50AP + No Colour	230 (+/- 20.0)
D	70AS:30AP + Colour A*	192 (+/- 5.9)
E	70AS:30AP + Colour D*	183 (+/- 9.5)
F	70AS:30AP + Colour E*	162 (+/- 11.3)
G	50AS:50AP + Colour A*	198 (+/- 11.8)
H	50AS:50AP + Colour D*	190 (+/- 10.3)
I	50AS:50AP + Colour E*	216 (+/- 6.7)

*Colour concentration used was 0,0078 % m/m.

Figures in parentheses represent standard error of mean. (95% confidence limits).

All sample blocks were allowed to air-dry for 72 hours prior to being exposed as described in section 5.2 (7b). After exposure, sample blocks were evaluated using two methods as described in section 5.2. (7b).

The ability to distinguish visually between the exposed sample was based on a criteria of colour difference, i.e. could one without doubt recognise the colour in the colour treated sample when compared to the non-coloured treated sample. Results are depicted in Table 10 below.

TABLE 10: COLOUR DISTINCTION BETWEEN COLOUR TREATED AND NON COLOUR TREATED SAMPLES

Sample Range	Description	Exposure Period (Hours)				
		0	50	100	160	200
D	70AS:30AP + Colour A*	Y	Y	Y	N	N
E	70AS:30AP + Colour D*	Y	Y	Y	Y	Y
F	70AS:30AP + Colour E*	Y	Y	Y	Y	Y
G	50AS:50AP + Colour A*	Y	Y	N	N	N
H	50AS:50AP + Colour D*	Y	Y	Y	Y	N
I	50AS:50AP + Colour E*	Y	Y	Y	Y	Y

*Colour concentration used was 0,0078 % m/m.

Y = Yes. Able to distinguish between colour treated and non colour treated samples.

N = No. Not able to distinguish between colour treated and non colour treated samples.

The indication Y in table 9 above does not quantify the amount of colour fading or intensity of colour, but merely indicates whether one can distinguish between colour treated and non colour treated samples.

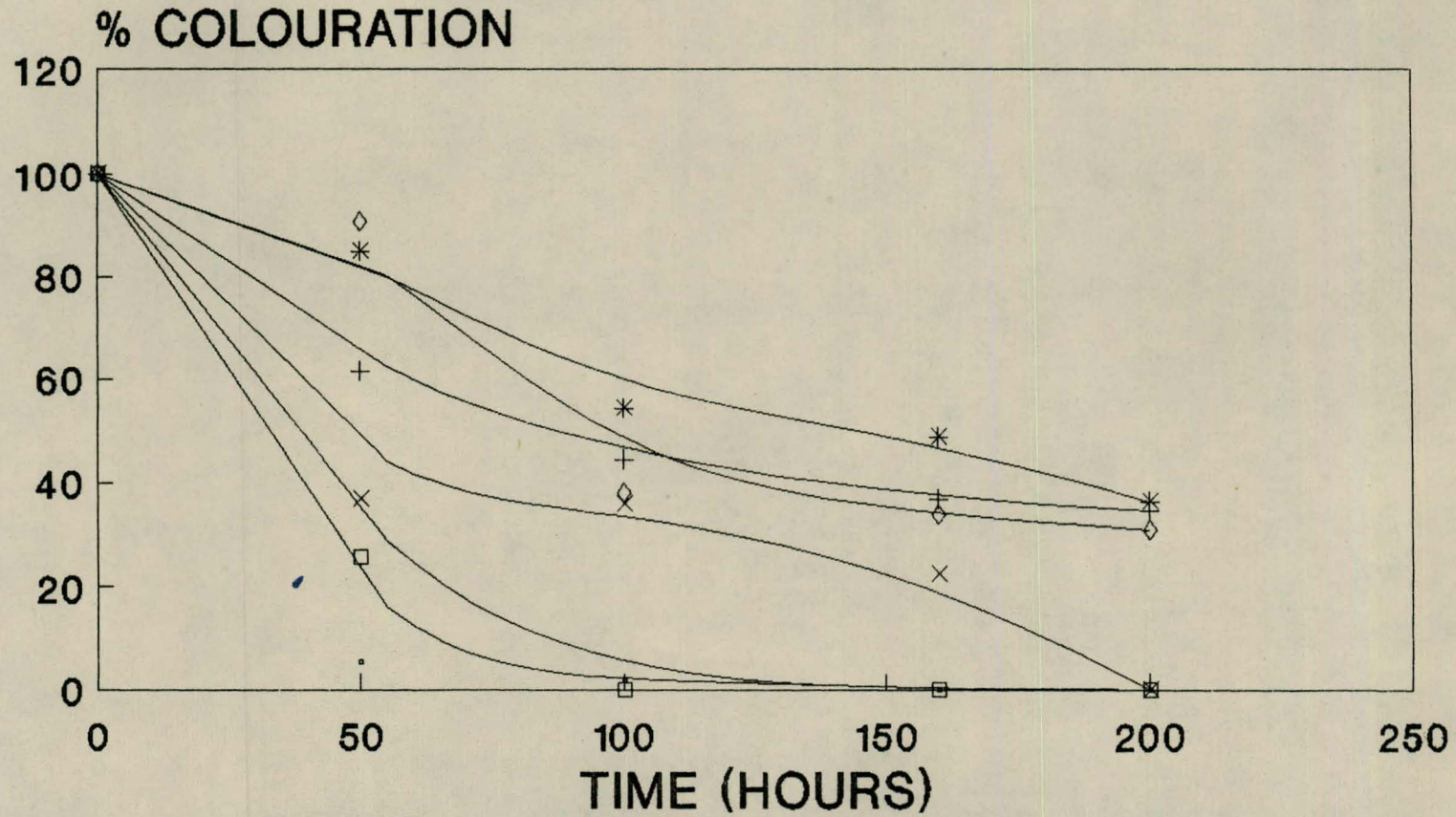
Based on this evaluation, Colour E proved to be the best performer followed by Colour D and then A.

To quantify the extent to which colour efficiency decreased during the weather-o-meter exposure trial, the area of colouration on the exposed surface was calculated using a 5 mm point matrix. As samples were end-matched, the effect of the timber could be regarded as a variable of no significance. The results are graphically displayed in figure 11.

From figure 11, it is evident that a substantial amount of colouration is lost during the first 50-hour exposure period. The rate at which the colour is lost after 50 hours exposure shows a significant decrease. Colour E was the best performer, closely followed by Colour D. Colour A was the worst performer and blocks had lost all colour between 100 and 150 hours.

The intensity of the colour was not brought into account during the area evaluation. It must be pointed out that the intensity of colour was substantially reduced as exposure period increased. Although the colour intensity was reduced, it was still easy to distinguish between colour treated and non colour treated samples over the 200 hour exposure period when referring to samples D and A. This applies for both formulations used.

FIGURE 11 : WEATHER -O- METER TRIAL



—•— 70AS:30AP +A

—+— 70AS:30AP +D

—*— 70AS:30AP +E

—□— 50AS:50AP +A

—×— 50AS:50AP +D

—◇— 50AS:50AP +E

Due to the combination of conditions sequentially experienced within the weather-o-meter, it is not possible to ascribe the loss of colour to any particular exposure condition.

6.4. Natural Exposure Trials

All timber samples to be used in the natural exposure trials were treated as described in section 5.2.5. and evaluated as described in section 5.2.7(c).

A sample range consisting of 16 samples further divided into five end-matched samples per range were selected and used. The objective was to cover as broad a range of sapwood ratios as possible within the sample, to eventually have a large spread in volumetric uptake and during exposure, to determine what effect these aspects have on colour intensity and efficiency.

Variation of the vacuum and pressure period was used to obtain the desired effect of large distribution within the treatment results. The treatment process parameters are described in Table 11 below (min = minutes applied).

TABLE 11: PROCESS PARAMETERS

Process	Vacuum		Pressure		Samples Treated
	kPa	min	kPa	min	
Untreated	0	0	0	0	A1-P1
1	-60	5	0	0	A(2-5), B(2-5), C(2-5), D(2-5)
2	-60	2,5	0	0	E(2-5), F(2-5), G(2-5), H(2-5)
3	-60	5	500	1,0	I(2-5), J(2-5), K(2-5), L(2-5)
4	-60	5	500	0,5	M(2-5), N(2-5), O(2-5), P(2-5)

For colour reference purposes, the following schedule has been compiled and detailed in table 12.

TABLE 12: SAMPLE CODING

Sample Code Containing Numerical	Formulation and Colour Description
1	Untreated
2	70AS:30AP Formulation + No Colour
3	70AS:30AP Formulation + Colour A*
4	70AS:30AP Formulation + Colour D*
5	70AS:30AP Formulation + Colour E*

*Colour concentration used was 0,0078 % m/m.

The samples were treated and exposed. Details are depicted in Tables 13, 14, 15 and 16. An untreated sample was included in the exposure trial to ensure that the discolouration rate of untreated timber under exposure conditions could be monitored. A freshly treated sample was air-dried for future reference as a control.

TABLE 13: SAMPLE DATA, TREATMENT RESULTS & EXPOSURE LOCATION:
NON-COLOURED SOLUTION 70AS:30AP FORMULATION

Process Applied	Sample Number	% Sapwood Volume	% Heartwood Volume	Chemical Uptake (l/m ³ Sapwood)	Location of Exposure
1	A2	68	32	164	WELKOM
1	B2	61	39	190	WELKOM
1	C2	71	29	189	WESTONARIA
1	D2	71	29	242	CONTROL
2	E2	72	28	283	WELKOM
2	F2	47	53	170	CONTROL
2	G2	63	37	147	WESTONARIA
2	H2	58	42	169	WESTONARIA
3	I2	43	57	88	WELKOM
3	J2	78	22	166	WESTONARIA
3	K2	79	21	71	CONTROL
3	L2	64	36	120	WELKOM
4	M2	46	54	190	WESTONARIA
4	N2	86	14	28	WELKOM
4	O2	61	39	120	WESTONARIA
4	P2	66	34	163	CONTROL

TABLE 14: SAMPLE DATA, TREATMENT RESULTS & EXPOSURE LOCATION:
COLOUR A + 70AS:30AP FORMULATION

Process Applied	Sample Number	% Sapwood Volume	% Heartwood Volume	Chemical Uptake (l/m ³ Sapwood)	Location of Exposure
1	A3	68	32	149	WELKOM
1	B3	61	39	185	WELKOM
1	C3	71	29	182	WESTONARIA
1	D3	71	29	210	CONTROL
2	E3	72	28	203	WELKOM
2	F3	47	53	127	CONTROL
2	G3	63	37	118	WESTONARIA
2	H3	58	42	130	WESTONARIA
3	I3	43	57	84	WELKOM
3	J3	78	22	152	WESTONARIA
3	K3	79	21	61	CONTROL
3	L3	64	36	123	WELKOM
4	M3	46	54	192	WESTONARIA
4	N3	86	14	35	WELKOM
4	O3	61	39	128	WESTONARIA
4	P3	66	34	181	CONTROL

TABLE 15: SAMPLE DATA, TREATMENT RESULTS & EXPOSURE LOCATION:
COLOUR D + 70AS:30AP FORMULATION

Process Applied	Sample Number	% Sapwood Volume	% Heartwood Volume	Chemical Uptake (l/m ³ Sapwood)	Location of Exposure
1	A4	68	32	148	WELKOM
1	B4	61	39	167	WELKOM
1	C4	71	29	183	WESTONARIA
1	D4	71	29	225	CONTROL
2	E4	72	28	192	WELKOM
2	F4	47	53	166	CONTROL
2	G4	63	37	147	WESTONARIA
2	H4	58	42	154	WESTONARIA
3	I4	43	57	83	WELKOM
3	J4	78	22	159	WESTONARIA
3	K4	79	21	65	CONTROL
3	L4	64	36	128	WELKOM
4	M4	46	54	215	WESTONARIA
4	N4	86	14	54	WELKOM
4	O4	61	39	142	WESTONARIA
4	P4	66	34	205	CONTROL

TABLE 16: SAMPLE DATA, TREATMENT RESULTS & EXPOSURE LOCATION:
COLOUR E + 70AS:30AP FORMULATION

Process Applied	Sample Number	% Sapwood Volume	% Heartwood Volume	Chemical Uptake (l/m ³ Sapwood)	Location of Exposure
1	A5	68	32	187	WELKOM
1	B5	61	39	222	WELKOM
1	C5	71	29	235	WESTONARIA
1	D5	71	29	298	CONTROL
2	E5	72	28	215	WELKOM
2	F5	47	53	147	CONTROL
2	G5	63	37	133	WESTONARIA
2	H5	58	42	153	WESTONARIA
3	I5	43	57	73	WELKOM
3	J5	78	22	132	WESTONARIA
3	K5	79	21	61	CONTROL
3	L5	64	36	103	WELKOM
4	M5	46	54	161	WESTONARIA
4	N5	86	14	37	WELKOM
4	O5	61	39	102	WESTONARIA
4	P5	66	34	143	CONTROL

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The ability to distinguish between colour treated and non-colour treated samples was performed by means of a visual evaluation on a comparative basis. All samples were evaluated twice a week. At the end of the nine-week evaluation period, most colour treated samples could be distinguished from non-colour treated samples. The only failing samples were Samples N and I, which in all colour treatments appeared to have lost their colour after 4-5 weeks.

One of the reasons for Samples N and I losing their colour was possibly due to the low chemical solution uptake, which was below average, i.e. (35-54 l/m³) and (73-84 l/m³) respectively. Furthermore, sample N contained a large percentage of sapwood volume which means chemical distribution could have been scattered.

One aspect which was noted, was the pattern of colour fading. The "bottom" of all samples, i.e. not totally exposed, showed a significantly reduced amount of colour fading/loss. Colour efficiency on the end-grain sections was confined to the sapwood areas midway through the exposure test. This implies that the surface coated heartwood sections which were coloured after fresh treatment, was of such low concentrations that leaching out and/or fading away, was fairly easy.

Although the evaluation is not of a quantitative nature, it does strongly provide support for the idea of colour fire-retardant treated samples in order to distinguish them from untreated or non-coloured fire-retardant samples.

As the data was analysed in a qualitative manner, it is not possible to distinguish between the two locations of exposure.

6.5. Underground Exposure Trials

Samples for this exposure trial were prepared and treated as detailed in 5.2.7(d) and 5.2.5. Individual samples of similar colour were bound together after treatment to ensure larger samples for easy handling in the underground situation.

The samples were classed into two types, i.e. slabs and trims, and the following treatment parameters were applied.

Slabs - 5-minute initial vacuum (wet vacuum).
- Pressurise cylinder to 250 kPa for 30 seconds.
- Relieve vacuum.

Trims - 5-minute initial vacuum (wet vacuum).
- Pressurise cylinder to 500 kPa for 30 seconds.
- Relieve vacuum.

Treatment results are detailed in Table 17 below.

TABLE 17: TREATMENT RESULTS OF SAMPLES FOR UNDERGROUND EXPOSURE

Sample Number	Colour Applied	% Sapwood Volume	% Heartwood Volume	Chemical Uptake (l/m ³ Sapwood)
SLAB 7	A	63	37	331
8	A	63	37	280
9	A	52	48	201
10	D	52	48	277
11	D	74	26	123
12	D	74	26	143
13	E	55	45	207
14	E	55	45	197
15	E	63	37	240
TRIM 7	A	50	50	138
8	A	50	50	155
9	A	54	46	89
10	D	54	46	102
11	D	35	65	69
12	D	35	65	69
13	E	70	30	115
14	E	70	30	128
15	E	69	31	120

For the larger scale treatment trials, it was evident that colour efficiency on the cross cut ends was more pronounced when compared to the colour effect on circumference of the timber. The reason for this phenomenon is possibly due to the large surface area available for colourant adhesion on the roughly sawn cross cut faces when compared to the circumference.

Colour efficiency evaluation was not performed on the samples placed underground.

7. ECONOMICAL OVERVIEW

As a large number of mines currently find themselves in a non-profit or marginal-profit situation due to working costs exceeding the revenue generated by gold sales (see figure 1), any further financial expenditure is mostly out of the question. Furthermore, additional costs incurred due to the added cost of colouration of fire-retardant treated timber, could possibly increase the number of non-profitable or marginally profitable mines.

In analysing timber support costs and the impact which timber treatment has, the following transpires.

Average timber cost to mines = R360 per ton
 Average timber treatment cost to mines = R35 per ton

From the above, it can be seen that the total timber and treatment costs amount to R395 per ton, which contributes approximately 6 percent to average working costs of a mine.

The addition of a colourant at the concentration levels as used in this study would add to the cost as detailed in Table 19.

TABLE 19: COST OF COLOURATION

Colour	Cost (R/kg)	Concentration Used %	Cost per Ton Treated (R/ton)	Increase in Timber Related Cost %
A	140.00	0.0078	1.09	0.303
D	49.21	0.0078	0.38	0.106
E	47.00	0.0078	0.37	0.103

- 60-

Should HL&H Mining Timber not pass on these additional costs for colour treated timber to their various customers, their additional cost based on 750 000 tons of timber would be as stated below:

Using colourant A, additional costs of R817 500.

Using colourant D, additional costs of R285 000.

Using colourant E, additional costs of R277 000.

One aspect which should be borne in mind is that some additional business could be gained from those who wish to use fire-retardant treated timber in high risk areas, but have declined to do so due to the non-distinguishability between treated and untreated timber. On the negative side, a substantial amount of business could possibly be lost due to mines which are currently using all treated timber, moving to a situation where only high risk areas will be treated, due to the fact that treated and untreated timber can now be distinguished from one another.

On the operating side of HL&H Mining Timber, additional costs of approximately R22 000 using colour A; R8 000 using colour D; R7 300 using colour E, to commission all treatment plants to using the colouration. Once all plants have been commissioned, no additional costs relating to timber handling, treatment cycle times, chemical analysis and mixing will be incurred, as the colourant will be added into the mixing trough in small quantities as fresh treating solution is prepared daily.

8. CONCLUSION AND RECOMMENDATION

8.1. Conclusions

Based on results obtained and discussed in section 5 of this document, the addition of a colourant to the fire-retardant chemical in order to achieve a final treated product which is visually distinguishable from non coloured fire-retardant treated timber is feasible.

Results on chemical compatibility proved that colourant Acid Green, Bazazol Red 76L and Malachite Green are compatible with both commercially used fire-retardant formulations. Colour efficiency in these cases was very good. The influence of temperature of solution on chemical stability, as well as stability over time up to 96 hours, was tested and results obtained were good:

- * The effect on combustion when the colourant was included in both commercially used fire-retardant formulations was not relevant when differences experienced with using the test method are borne in mind.
- * The controlled weathering trials indicated that Malachite Green performed the best, closely followed by Basazol Red 76L. Acid Green did not perform well and is therefore not recommended. It is important to note that the initial loss in colour during the weathering trials was substantial, but thereafter the rate of colour loss decreased significantly.

- * The uncontrolled (open air) weathering test supports the findings of the controlled (weather-o-meter) weathering test to an extent. The significant initial colour loss was also experienced, followed by a reduction in the rate of colour loss. Due to the variance in sapwood chemical uptake, the excessive colour loss when using Acid Green was not as pronounced as expected. A trend which was noted was the close correlation between the rate of colour loss and low sapwood chemical uptake.

- * Based on this evaluation, Malachite Green and Basazol Red 76L performed the best. The only criticism in using Malachite Green, which results in a green tint treated product, is the fact that it visually resembles CCA treated timber (copper-chromium-arsenic wood preservative). This could lead to confusion in the timber treatment industry.

8.2. Recommendations

Due to its distinct colouring properties, it is recommended that Basazol Red 76L be used at a 0.0078 percent concentration.

As a scaling up exercise, it is further recommended that a few colour trials are performed on a large scale at the small HL&H Mining Timber plant at Orkney, using colour D only. This will have to be performed to enable HL&H to gain information relating to full scale treatment efficacy of the colourant.

Toxic gas emission and skin irritation tests were not included in this study, due to the extremely low concentrations being used. It is advisable to perform these evaluations as a matter of course prior to full scale implementation.

The individual exposure trials represent conditions which timber comprising the outside of packs will experience. The inner portions of a timber pack will be protected from the elements by the outside layers and it is anticipated that under these circumstances, the colour efficiency could be extended well beyond the exposure period used in this study. The effect of the abovementioned should be quantified.

On a full scale basis, the longevity of the colourant should be tested until there is no evidence of colour remaining in the timber. This should be performed to establish the "shelf life" of colour treated timber.

Cost effectiveness should be re-evaluated once large scale samples have been treated and evaluated on a quantitative basis.

The possible negative influence related to current customers using less treated timber due to the easy distinguishability of treated timber for high risk areas only, should be quantified and followed up.

Underground exposure trials should be re-done to establish the longevity on colour efficiency in the underground environment.

9. LITERATURE

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ANNEXURE

PHOTOGRAPHS

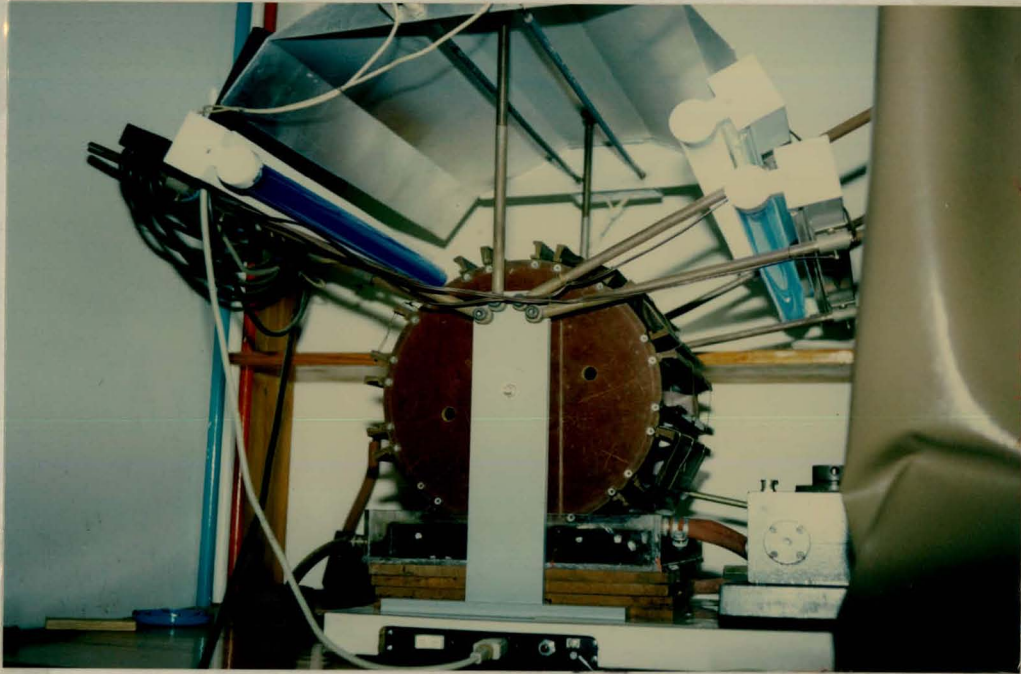


PLATE 1. WEATHER-O-METER APPARATUS.

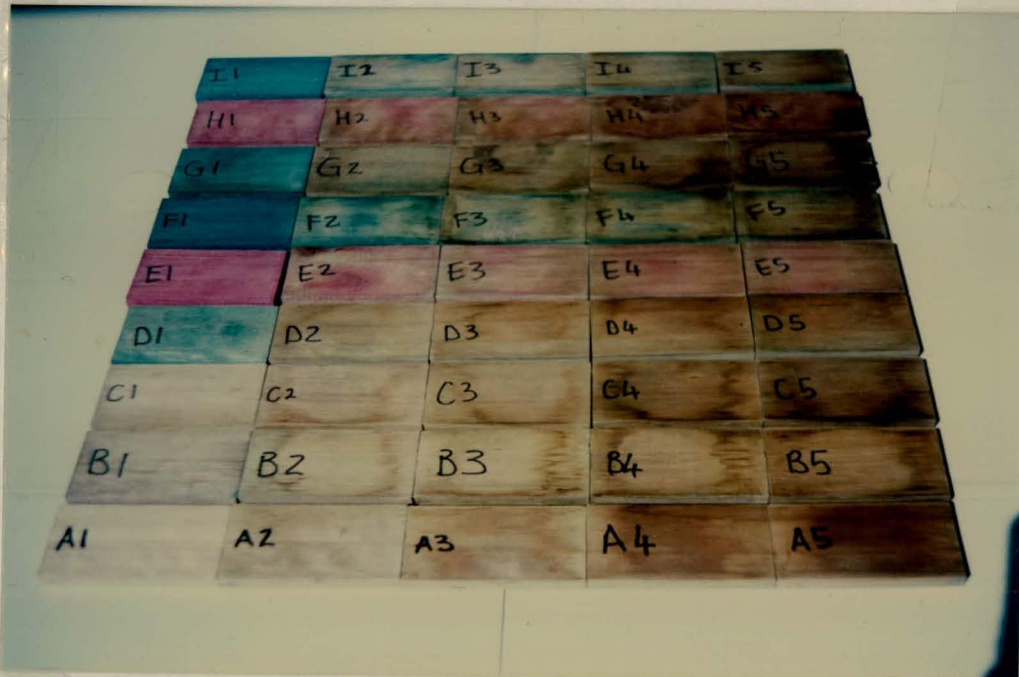


PLATE 2: SAMPLES EXPOSED FOR 0,50,100,160 AND 200 HOURS.

ANNEXURE

PHOTOGRAPHS



PLATE 3. SAMPLE SET A,B,C AND D AFTER TREATMENT.



PLATE 4: SAMPLE SET E,F,G AND H AFTER TREATMENT.

ANNEXURE

PHOTOGRAPHS



PLATE 5. SAMPLE SET I, J, K AND L AFTER TREATMENT.



PLATE 6. SAMPLE SET M, N, O AND P AFTER TREATMENT.

ANNEXURE

PHOTOGRAPHS



PLATE 7. OPEN-AIR EXPOSURE.



PLATE 8: OPEN-AIR EXPOSURE. NOTE HEARTWOOD UNCOLOURED.

ANNEXURE

PHOTOGRAPHS



PLATE 9. SAMPLE SET A,B,C AND D AFTER EXPOSURE (9 WEEKS)



PLATE 10: SAMPLE SET E,F,G AND H AFTER EXPOSURDE (9 WEEKS)

ANNEXURE

PHOTOGRAPHS



PLATE 11 SAMPLE SET I, J, K AND L AFTER EXPOSURE (9 WEEKS)

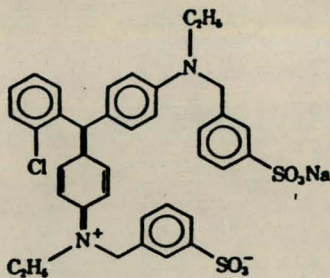


PLATE 12: SAMPLE SET M, N, O AND P AFTER EXPOSURE (9 WEEKS)

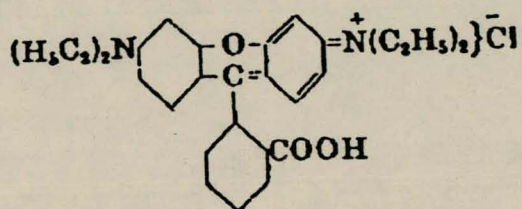
ANNEXURE

COLOURANTS STRUCTURES ⁶

**Acid Green
(Colour A)**



**Basazol Red 76 L
(Colour D)**



**Malachite Green
(Colour E)**

